WINLAB32 FOR ICP
INSTRUMENT CONTROL SOFTWARE

Software Guide

PerkinElmer®
For the Better

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Welcome to WinLab32!

Important Features of the WinLab32 Software

- This highly usable product was developed by a team of application experts, laboratory managers, graphic designers, cognitive psychologists, and users from all over the world. Sample analysis, scheduling, and data reporting can be handled easily and efficiently.

- Operation is simplified. Based on the familiar Microsoft Windows menus and graphics, the software has easy-to-use graphics and familiar pull-down menus.

- Complete system controls and useful online information are at the user's fingertips, making procedures simple and routine, even for new operators with minimal training in ICP lab procedures. Less training is required and time can be saved, hence increasing lab productivity.

- The automation of many routine tasks not only saves time, but also avoids expensive, time-consuming errors.

- Comprehensive data reprocessing and reporting capabilities include: built-in QC automation routines; full data reprocessing from stored spectra; methods and data stored in a commercial database; and easy generation of customized reports using off-the-shelf word processor or spreadsheet programs.

- The flexibility of WinLab32 should allow you to comply with quality control protocols and regulatory procedures associated with Good Laboratory Practice (GLP) and Good Automated Laboratory Practice (GALP) requirements.

Related Applications

In addition to the WinLab32 software, PerkinElmerPerkinElmer provides:

- The Data Manager software application lets you archive, copy, delete, and rename the data sets created by WinLab32. Within this application, you can select a subset of the data from a results data set and save it in a file format that can be read by many off-the-shelf applications, including spreadsheet and database management programs. In addition, you can create printed reports of data where you control the design of and the information included in the report.

- WinLab32 Offline is a second copy of the WinLab32 application that does not control the instrument. You can use WinLab32 to edit methods, enter sample

information, examine data, build IEC or MSF models, or reprocess data in the software without operating the instrument. This is useful when you wish to work with methods and data while an analysis is in progress.

Important Prerequisites

Before you use WinLab32 software, you should:

• Understand ICP principles and practices. If you need background information, you should consult one of the many textbooks available.

• Know how to use a personal computer. If you need further information, consult your computer manual.

• Understand Windows fundamentals. If you have questions, refer to manuals, books or CD-ROM tutorials on Microsoft Windows.

WinLab32 Software

Organization

The WinLab32 software contains a series of windows, each of which has been optimized to help you perform some part of the overall analytical task. This model was a refinement of one of our current systems that has been well accepted in the analytical community. Each window in the software provides a specialized, task-oriented view into the system. To perform a task, the analyst displays one or more windows that provide the appropriate controls or views of the data.

These windows are grouped in the following functional categories:

Data Analysis and Control:

Editors for Parameter Input	Use these windows to create or edit new methods and sample information files. These are the Method Editor, Periodic Table, Wavelength Table, and Sample Information Editor.

Controlling the Analysis

These windows enable you to select a method, sample information file, and make decisions about the analysis, whether it is a manual or automated analysis. These are the Manual Analysis and Automated Analysis windows. In addition, a Data Reprocessing window allows you to recalculate data.

Displaying Information and Correcting Interferences

Windows are available to view calibration and spectral and tabular results during an analysis. Take a close look at a spectrum with the Examine Spectra/MSF window, where you can correct interferences using the MSF technique. An IEC Model Builder lets you apply IEC correction factors.

Handling Data: Data Manager application

A full range of data management capabilities is available using this separate software application. Use Data Manager to copy, archive, delete, results data sets. You can also export data to other file formats and create reports of data. Step-by-step wizards are available.

Instrument Control:

Controlling the Plasma, the Spectrometer and FIAS	Before, during, and after an analysis, use these windows as needed to control hardware parameters. These include the Plasma Control, the Spectrometer Control, and FIAS Control windows.
Adjusting and diagnostics	When the instrument requires adjusting or diagnosing, check the Diagnostics window. In addition, System Monitors keep you informed of actions in the spectrometer, plasma and other hardware.
Online Assistance: **Finding Information**	Throughout WinLab32, there are several information sources: online help topics, messages from the instrument, a tip of the day, and tool tips.

Overview of Windows for Data Analysis and Control: Specialized Editors for Parameter Input

There are several key windows for entering parameters for the analysis. Here is an overview.
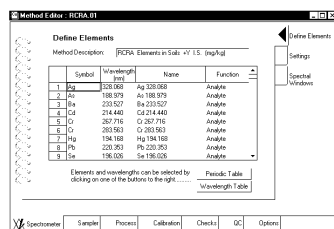
Method Editor. In the Method Editor, you enter all of the analytical information needed to perform an analysis: parameters for instrument settings, directions to convert spectral data into intensity data, calibration information and autosampler settings. Spike concentrations for matrix check samples, location of quality control solutions and instructions for performing QC procedures are also included here as well as several options for printing and storing data.
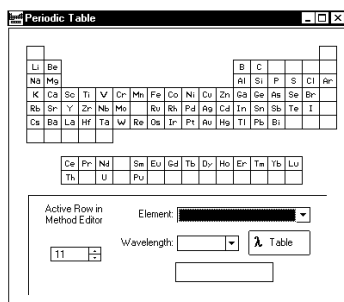
Periodic Table. The Periodic Table presents a graphical, easy-to-use means of selecting elements and their recommended wavelengths for your method. The elements and wavelengths that you select will be entered automatically in the Method Editor: Spectrometer, Define Elements page.

Wavelength Table. Use the Wavelength Table to search for all of the elements that emit a signal at a particular wavelength. This window also lets you search for wavelengths for your method beyond those that appear in the recommended wavelengths list found in the Periodic Table.

Sample Information Editor. Use this window to enter all of the information regarding your samples. You enter information to describe the samples, information on how the samples were prepared, and set up an analysis schedule. In addition, you have the flexibility to note parameters that vary by sample, such as weight, and those that may be common to all samples, such as analyst name.

Overview of Windows for Data Analysis and Control: Windows for Controlling Analyses
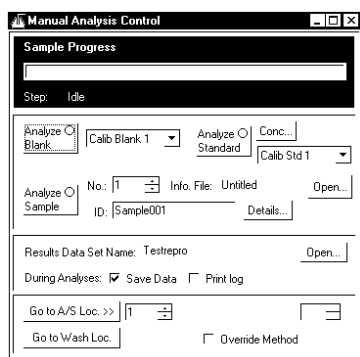
Manual Analysis Control. Use the Manual Analysis window to analyze each blank, standard and sample manually. You select a method, sample information file, and if you choose to save data, a results file. When you analyze each item, the current status is reported here as well as in the Spectrometer monitor. Performing a manual analysis is useful when you wish to analyze only a few samples.
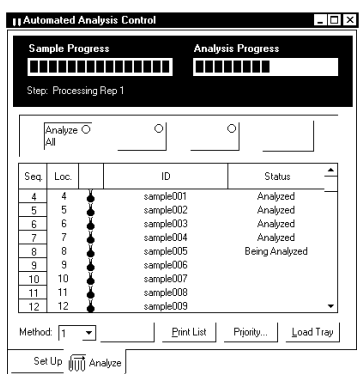
Automated Analysis Control. For analyses with many samples or when an autosampler is used, you can set up an automated analysis. On the Set Up page, you select the method, sample information file, and if you chose to save data, a results data file. On the Analysis page, you start the analysis and can then observe the current status of each blank, standard and sample as well as the progress of the overall analysis. (The Spectrometer and Autosampler Monitors report the status of their respective hardware.) The Automated Analysis window lets you analyze up to the full capacity of your autosampler tray.
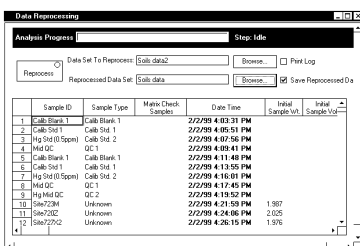
Data Reprocessing. Use this window to recalculate existing data in a results data set. This is useful if you wish to set different analytical conditions than those in the initial analysis, select different peak parameters, or change or correct method parameters without the need to reanalyze the samples.

Overview of Windows for Data Analysis and Control:
Windows for Displaying Information and Correcting Interferences
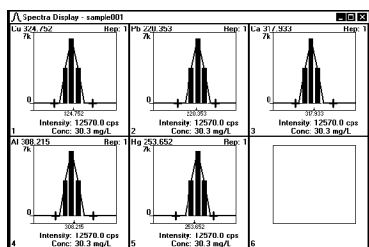
Spectral Display. Use the Spectral Display window to view the spectra as it is generated during an analysis or spectra reprocessed from stored data. This graphical window shows subarray pixels, background correction points, intensity and concentration data. This window can be printed, resized, and, if desired, spectral data can be saved to the results data file. Color and layout can be adjusted. This window updates as each sample is analyzed.

Results Display. Use the Results Display window to view the data generated from the analysis in a tabular form. This information can be written to a log file and printed. This data, saved in a results data set, can then be manipulated in the Data Manager as needed to create reports and to be exported to other formats.
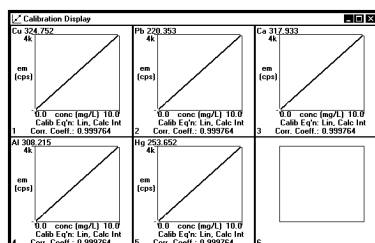
Calibration Display. This window shows the calibration curve for the elements being analyzed in each sample. It also provides the concentration of elements in the calibration standard, the calibration equation being used, and the correlation coefficient. (You set up the calibration and select the calibration equation in the method.) This window can be printed and resized, and color and layout can be adjusted. This window updates as the calibration analysis progresses.
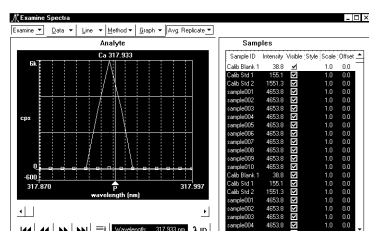
Examine Spectra/MSF. The Examine Spectra/MSF window has two modes: In Examine Spectra mode, you can take a closer look at any spectrum or group of spectra for anomalies. Use it to make changes, such as updating the peak wavelength or changing background correction points. Should interferences be present, the Multicomponent Spectral Fitting mode of this window lets you create MSF models (blank, analyte, interferent) that you can use to make comparisons with your spectrum results to identify and subtract out interfering signals.
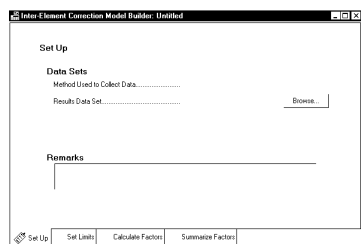
IEC Model Builder. Use the IEC Model Builder when you suspect interferences that are direct spectral overlaps. This window takes you through set up, setting limits, calculating and summarizing factors that, when applied, reallocate emission intensities to correct for interferences.

Overview of Windows for Instrument Control: Windows for Plasma and Spectrometer Control
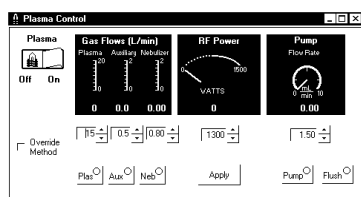
Plasma Control. Use this window to modify plasma parameters, such as gas flows, radio frequency power and pump flow rate. You also use this window to monitor the status of major components affecting the plasma and sample introduction system, and to ignite the plasma and shut it off. In addition, you can override the method parameters with those contained in this window. (Recommended settings for plasma parameters and for nebulizers are contained in the Plasma Control Window section.)
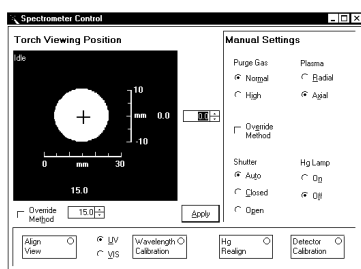
Spectrometer Control. Use this window to control key parameters for the spectrometer. You can adjust the torch viewing position, set spectrometer parameters manually (such as purge gas and plasma viewing mode) and override parameters in the method as well as perform a detector calibration. For simultaneous spectrometers, you can also perform a system wavelength calibration or a mercury realignment using this window.

FIAS Control. Use the FIAS Control window to view the FIAS status and set valves, pumps , and remotes independently of any other ICP functions

Overview of Windows for Instrument Control: Windows for Adjusting the Instrument and for Diagnostics

Diagnostics. When a question arises in instrument operation, the Diagnostics window can help by providing information about system connections (the System page) and about the status of the spectrometer, plasma generator and autosampler. Each of these component pages also contains a history showing the messages between the component and the software. In addition, each page contains a reconnect button for the associated component.

Status Panels. Several Status Panel windows provide updated information on instrument operation. During an analysis, these status panels report the position of the autosampler probe, the status of the spectrometer and plasma, and the status of instrument startup/shutdown. They are located at the top of the WinLab32 main screen but can be moved to any position on screen as desired.

Overview of Windows for Handling Data: Data Manager Application

The Data Manager application is a useful tool for maintaining and using data generated by the instrument and the WinLab32 software application. See the WinLab32 Data Manager Software Guide for further information.
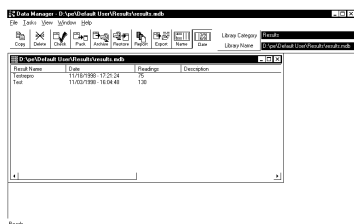
Data Manager window. Use the Data Manager window to perform various data handling maintenance tasks. You can create, check, rename, copy, delete, and restore results data sets as desired. It is important to periodically delete and archive data sets to prevent libraries from becoming too large. Also, if you are running WinLab32 Enhanced Security use the Data Manager utility to view a master event log of all significant actions performed by a user as well as the revision history on all files and data objects.
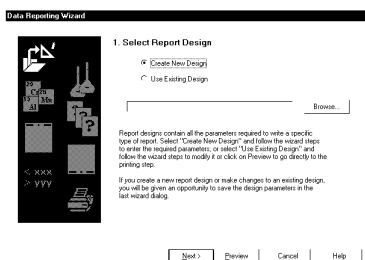
Reporting Wizard. Use Data Manager's Reporting Wizard to create printed reports of selected data. This wizard is an interview-style series of dialogs to help you easily choose the data and the format in your report.
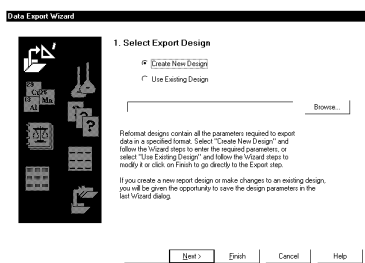
Export Wizard. Data Manager's Export Wizard lets you select a subset of the data contained in a results data set and write it to a file that can be read by many other software applications, including spreadsheet and database management programs.

Overview of WinLab32 Online Assistance
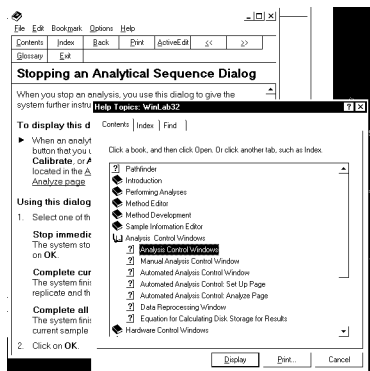
Help topics. To answer your questions as you operate the instrument, WinLab32's online help file contains step-by-step procedures, parameter descriptions, graphics, labeled screen shots, examples, a glossary and a comprehensive index as well as a full text search. You can print an individual topic or an entire book. (To do so, open the help contents window, click on a book or topic of interest and select Print in the bottom right hand corner of the contents window.)
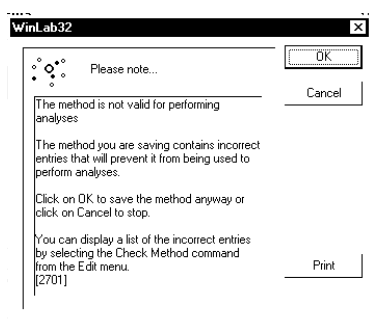
System Message. When there is an inconsistency in input information, WinLab32 alerts you with a system message dialog. This identifies the concern and provides an appropriate action to enable you to make a correction, or it may present you with a choice of actions. These messages can be printed for your reference.
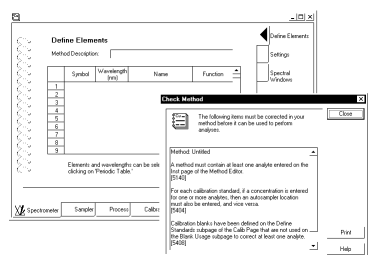
Check method. When an inconsistency in a method occurs, a check method dialog appears. This identifies the concern and provides an appropriate action to enable you to make a correction, or it may present you with a choice of actions. It also provides other useful information pertaining to the method. These messages can be printed for your reference. If you wish to check a method for errors or inconsistencies, in the Edit menu, click on Check Method. The active method will be checked and problems reported.
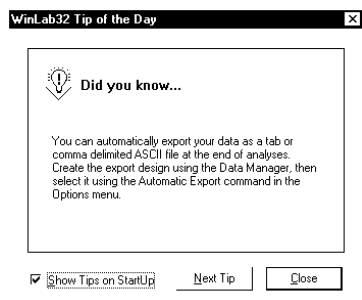
Tip of the Day. When you start the WinLab32 application, a Tip of the Day message appears with additional information that you may find helpful as you operate the instrument.
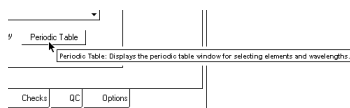
Tool tips. Throughout WinLab32, information is available for window features such as buttons, entry fields, options, etc. When you place the mouse cursor on a feature, a one-line description, action, or other useful information relating to the feature selected appears.
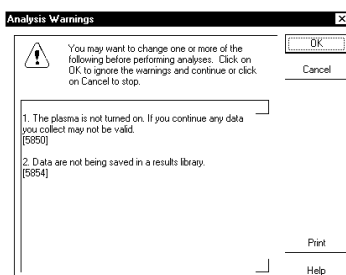
Analysis checking and error (warning) messages. When you have entered analysis information and begin the analysis, WinLab32 checks the entries to make sure that everything is set up properly. Depending on the type of error found, the software will display an analysis check dialog, which provides general information, or an analysis warning dialog, which alerts you to the possible collection of invalid data, so that you can make corrections.

Workspaces

Workspaces let you store a group of selected windows for later use. The workspace saves all of the entries you have made in the windows for your methods and analyses. It also saves the location of each window on the screen. The workspace is saved in a workspace file, which has the extension .wsp. Using workspaces, you can fill in and arrange windows once, saving time whenever you set up methods or perform analyses.

Entries that you can save using a workspace include the names of the method and sample information file, information contained in the Automated Analysis window (Set Up page), and any information concerning multi-method analysis. It also saves

decisions that you have made for your analysis, such as if data is to be saved and where, if it is to be printed, and if the method in memory is to be used.

To create a workspace:

1. Open the desired windows, fill in the entries for your methods and analyses and arrange the windows on the screen. For example, suppose you have run an analysis and arranged the windows as follows:
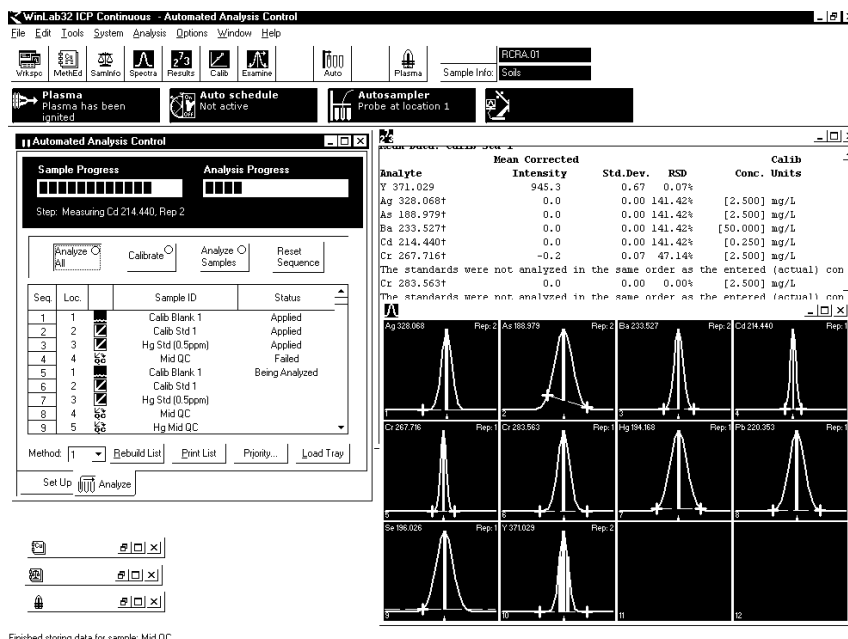
You can save the entire screen with all of the displayed windows and entries in a workspace file to redisplay later.

2. To save the position of these windows and the entries within them, in the **File** menu, click on **Save As... Workspace**... In the dialog that appears, type a name for the workspace and click on **Open**. For example, type "analysis1".

3. When you wish to view (or use) these windows and their entries again, in the **File** menu, click on **Open Workspace**. In the dialog that appears, select **analysis1.wsp** and click on **Open**. The windows with the entries appear exactly as before.

Libraries and Files In Data Manager

Libraries and Data

The WinLab32 Data Manager application uses database technology to organize information in a structured format that is easy to manipulate. This information includes: parameters needed to perform analyses; data collected during analyses; and parameters used by related WinLab32 applications.

The data generated before, during, and after analyses is stored in libraries. Each library is a group of related tables and indices that contain data in a structured format.

Data Manager

The Data Manager is a WinLab32 application that helps you organize and manage the data stored in libraries. You can rename, copy, archive, and delete data sets.

Important Note Library maintenance should be done periodically to prevent your Results Library from becoming too large. A large Results Library can lead to disk problems. Maintenance procedures include archiving, deleting, and packing data sets. For additional information, please refer to Data Manager Help: *What's Important About Managing Data*.

Files

Not all WinLab32 information is stored in database libraries. For example, sample information is stored in ASCII files, so that you can easily create compatible sample information files using off-the-shelf applications such as spreadsheet programs. You can use the Windows Explorer to copy, rename, move, or delete these files.

Different Libraries

You may want to create separate libraries for each person using the system. You can do this by creating new libraries for the methods and results using Data Manager. Refer to the Data Manager Help file for information.

Safety Information and Conventions

This help file contains procedures for setting up and performing analyses using WinLab32 and detailed descriptions of the windows in the software. Be advised of the following safety information and aware of the conventions used in this help file.

Safety Information

Caution

Before setting up and operating the instrument, carefully read the safety precautions described in the Hardware Guide and observe these precautions at all times.

Conventions Used in the online help and software guide

The following special formats are used to set apart important information and warnings when dealing with the instrument hardware.

Warning

A warning indicates an operation that could cause *personal injury* if precautions are not followed.

Caution

A caution indicates an operation that could cause *instrument damage* if precautions are not followed.

Note   Notes emphasize significant information in a procedure or description.

Performing Analyses

2

Performing Analyses Overview

This chapter explains the procedures that you will use to operate your instrument with WinLab32. It contains eight sections that guide you through setting up and optimizing the instrument, setting up the software and performing analyses.

Observe Safety Precautions

△

Caution

Before setting up and operating the instrument, carefully read the safety precautions described in the Hardware Guide and observe these precautions at all times.

To perform analyses

▶ Use sections to select the group of steps required for your analyses.

Performing Analyses Section	Results
Setting up the Instrument	• system checked • major components turned on • plasma ignited successfully
Optimizing and Verifying Performance	• hardware optimized and ready to go
Selecting a Workspace, Method, and Sample Information File	• workspace created with required windows • method or methods designated for your analyses • file created for samples in your analyses
Setting Options in Control Window	• results data set named • such as turning options on or off, automatically selected

Arranging Samples	• solutions prepared
	• samples placed in the autosampler
Analyzing Samples	• concentrations of all unknown samples determined
Generating Reports	• data from analyses collected and then printed using WinLab32 Data Manager
Shutting Down the Instrument	• plasma extinguished
	• instrument shut down or put in an automatic shutdown/startup mode

For information on the system, installation, maintenance, and troubleshooting, refer to the Hardware Guide.

Setting Up the Instrument

To set up the instrument

Setting up the instrument requires these basic steps.

Setup Step	What's Required
Checking the System	Check these items regularly to make sure that the system is set up properly.
Starting the Instrument and Accessories	Switch on power to the various components of the system.
Starting the Computer and Software	Start the software and verify instrument configuration.
Setting Up and Starting the Peristaltic Pump	Install the pump tubing so the pump is ready to start automatically once the plasma is ignited.
Preparing to Ignite the Plasma	Make important checks and changes to avoid ignition problems.

Setup Step	What's Required
Igniting the Plasma	
Examining the Plasma	Observe the plasma to determine its stability.
Correcting Unsuccessful Ignition	Troubleshoot problems if the plasma does not light.

Checking the System

Proper setup and routine maintenance are required to keep your instrument in proper working condition so that you will get acceptable performance.

To check the system

Before starting the system, use this checklist to make sure that the system is in proper working order. Consult your Hardware Guide for additional information, as well as for information on cleaning and maintaining your system.

Exhaust vent

▶ Check that your vent system is switched on, is working properly, and is not blocked.

Argon supply

▶ Be sure that an adequate supply of argon is available and is connected to the system. Check that a spare tank is ready if necessary. For more information, see the Installation chapter in the Hardware Guide.

▶ Check that the cylinder valve is open and the regulator for the argon outlet pressure is set within the range specified in the Hardware Guide.

▶ Check for leaks at the gas connections.

Nitrogen purge gas supply

▶ Be sure that an adequate supply of nitrogen is available and connected to the system. Check that a spare tank is ready. For more information, see the Installation chapter in the Hardware Guide.

▶ Check that the cylinder valve is open and the regulator for the nitrogen outlet pressure is set within the range specified in the Hardware Guide.

▶ Check for leaks at the gas connections.

Shear gas supply (Axial or Dual View only)

▶ The shear gas typically used is compressed air, although nitrogen may be used. If nitrogen is used, be sure that an adequate supply is available and connected to the system. Check that a spare tank is ready. For more information, see the Installation chapter in the Hardware Guide.

▶ Check for leaks at the gas connections.

Cooling water supply

The cooling water (house water or chiller) should meet the required specifications and be installed according to the information in the Installation chapter in the Hardware Guide.

▶ Check that the cooling water supply is connected to the instrument.

▶ If using a chiller, check the electrical connections. Be sure the chiller is turned on and has been filled. Periodically check the liquid level. See the Hardware Guide for the appropriate coolant and for other details.

▶ If using house water, check that the supply is turned on and is being filtered.

▶ Check for any leaks in the plumbing connections.

Sample introduction system

▶ Inspect the torch, glassware, and aerosol injector tube. The glassware should be clean. Small amounts of deposits are acceptable in most cases. Also check for signs of melting. Refer to the Maintenance chapter in the Hardware Guide for instructions on cleaning the torch.

▶ Check that the RF coil is clean. This prevents arcing across the coils. Also, if the humidity in your lab is high, be sure that the coil is dry. If necessary, use a soft cloth to dry it.

Leakage of air into any part of the torch, nebulizer or spray chamber may cause ignition problems. Therefore, be sure to regularly check the following items:

▶ Check the torch gas connections. For more information, see the Hardware Guide.

▶ Check that the nebulizer/end cap is tightly secured to the spray chamber.

▶ Check that the sample capillary tubing is attached to the nebulizer sample inlet. The tubing should be clean and in good condition.

▶ Check that the drain fitting is secured on the spray chamber drain. A loose-fitting drain can cause pressure leaks and consequent plasma instability.

Peristaltic pump and tubing

▶ Check that the pump tubing is in good condition, has no flat spots, and is correctly installed around the pump head. See the Hardware Guide for details.

▶ Replace tubing regularly, after eight to sixteen hours of continuous use. If you are routinely using organic solvents, you will need special tubing, since standard tubing may not last as long as with aqueous solutions. Refer to the Hardware Guide for a list of replacement tubing.

▶ Check that the pump rollers are clean and can move freely.

Drains

► Check that the spray chamber drain is properly set up on the pump so that waste is pumped out of the spray chamber. Replace the drain tubing if it has deteriorated.

► Check the spray chamber drain leads to the drain bottle. Empty the drain bottle if necessary. Dispose of waste properly.

Autosampler

► Install the tray in the autosampler and place the wash beaker in the rear left of the tray (location 0).

► Check that the sampling probe is installed at the correct height and the probe capillary is attached to the pump tubing for the sample.

Printer

► Check that the printer has an adequate supply of paper to print your results.

Starting the Instrument and Accessories

We recommend that you leave the Main Instrument switch on even when the instrument is not in use. If the Main Instrument switch is turned off, the system will need to reinitialize when power is switched back on. The initialization sequence, automatically carried out by the system, can take from 15 minutes to over an hour, depending on how long the instrument power remained off. For more information on the initialization sequence and the location of system components such as switches, refer to the Hardware Guide.

To conserve the purge gas for the detectors, you can use Automatic Shutdown in the Automatic Shutdown/Startup dialog. This feature is an alternative to turning off the power to the main instrument, which also then shuts off the purge gas.

You can also specify Automatic Startup in this dialog, indicating the time to start up, whether the plasma is to be ignited, and if the pump should begin operating upon startup.

To start up the instrument

1. If you have not already done so, turn on the gases and the cooling water supply or chiller. See the checklists in *Checking the System* earlier in this chapter.

2. If the Main Instrument switch has been turned off, turn it on. Usually, this switch should remain on to avoid a waiting period for instrument startup.

3. Close the doors to the sample compartment. Be sure that the front door is fully closed and secured.

4. Turn on the autosampler and any other accessories.

Note   If you turn on any of the devices under GPIB control after you start the software, you will need to reset communication to the device in the Diagnostics window by clicking on the **Reconnect** button. (The devices under GPIB control include the instrument and the autosampler.)

Starting the Computer and Software

To start the computer and software

1. Switch on the computer, monitor, and printer.

2. Click on the **Start** button. Select **Programs** ▶ **PerkinElmer WinLab32** ▶ **WinLab32**.

   The WinLab32 software starts.

To verify instrument configuration

1. For all instruments:
   Check that the autosampler type and tray are correct. Change, if required. To change the autosampler, exit the software and run the Reconfigure utility. To run the Reconfigure utility, click on **Start** ▶ **Programs** ▶ **PerkinElmer WinLab32** ▶ **Reconfigure.**

   To change the tray, use the Autosampler... command in the **Options** menu.

2. For all instruments:
   Check that the peristaltic pump and tubing type are correct. Change if required, using the Pump... command in the **Options** menu.

3. For the Dual View (DV) instruments:
   In the Spectrometer Control window, check that the correct plasma viewing mode (axial or radial) is set. Change the viewing mode if required, using the options in the Spectrometer Control window

Setting Up and Starting the Peristaltic Pump

Before setting up the pump, be sure you have made a daily check of the system, started the instrument and accessories, and started the computer and software. For more details on the pump, see the Hardware Guide.

To set up the peristaltic pump

1. Check that the sample tubing and the drain tubing leading from the spray chamber are properly set up on the pump, as shown in the Hardware Guide.

2. If the pump tubing is new, gently stretch it. Position the clips on the tubing in front of the tubing stops.

3. Replace the tubing clamps for each channel and swing the cam levers over to apply tension to the clamps.

To start the pump

1. Place the sample capillary tubing in a container of a solution whose matrix matches that of the samples in your application (for example: deionized water, diluted acid, or organic solvents).

2. In the Toolbar, click on the Plasma icon or in the Tools menu click on **Plasma Control** to Display the Plasma Control window. Click on the **Pump** button to turn on the pump.

3. Adjust the flow rate to 1.5 mL/min for aqueous analysis. (The aqueous analysis default value is 1.5 mL/min.) For organic analysis adjust the flow rate to 0.8 mL/min. (The organic analysis default value is 0.8 mL/min.)

4. If necessary, adjust the tension on the pump tubing, one channel at a time. For the sample tubing, gradually tighten the adjustment screw until the liquid flows smoothly without bubbles. The drain tubing should have a segmented flow of liquid leading to the drain bottle. Bubbles in the drain tubing are normal. See the Hardware Guide for a more detailed adjustment procedure.

Preparing to Ignite the Plasma

Before beginning this step, be sure the system, instrument, computer, and pump are ready. These preliminary steps are outlined in Set Up: Summary of Steps.

Before igniting the plasma

► Check that a method with the appropriate plasma conditions has been selected. Use the **Method** button on the toolbar to open a different method.

► Close and secure the doors to the sample compartment. An interlock prevents you from igniting the torch if the doors are not fully closed.

Caution When the humidity in your laboratory is high (>75%), water can condense on the RF coil of the torch when the torch is not in operation. To prevent damage to the RF coil, use a soft cloth to dry the coil before igniting the torch.

If switching from organic to aqueous solutions

► To avoid ignition problems when switching from organic to aqueous solutions, purge organic vapors that may remain in the torch. If needed, see *Correcting Unsuccessful Ignition* later in this chapter.

Note PerkinElmer recommends having two torch and sample introduction systems, one for use with agueous solutions and one for use with organic solutions.

If using wavelengths below 190 nm

▶ If wavelengths below 190 nm will be measured, you must use the Spectrometer Control window to do a high nitrogen purge of the optical system. A high purge time of approximately 15 minutes is usually sufficient if the instrument has been purging for a long time. Additional purge time may be necessary if the intensity readings are not satisfactory.

▶ Before running samples, return the purge rate to normal and take a measurement to make sure that you have achieved a steady-state signal, indicating that the high purge time was sufficient.

Igniting the Plasma

Before beginning this step, be sure the system, instrument, computer, and pump are ready, and that you have made the correct preparations to ignite the plasma. These preliminary steps are outlined in Set Up: Summary of Steps.

The Ignition Process

During the ignition process, argon gas flows through the torch and spray chamber, purging the sample introduction system of air. With the argon continuing to flow, power is applied to the RF coil. Then, a high voltage spark is injected into the argon flow causing the argon to ionize. The free electrons that are created then interact with the applied RF field to cause further argon ionization and form a plasma.

Messages appear in the Plasma Status display to inform you of the ignition status. The system sets the plasma gas flows. Then a message reads "Initial Purge" and the system counts down in seconds. The plasma is ignited, the plasma gas flows are then set to the starting plasma conditions of the active method in the Plasma Control Window, or if the override method is selected to those specific set points. At the end of this process, a message reads "Plasma has been ignited."

To ignite the plasma

1. Read the entire procedure before igniting the plasma.

2. Observe the following precautions when igniting the plasma.

⚠ **Caution**

Viewing the Plasma During Ignition

When you ignite the plasma, be sure to observe it closely through the viewing window. If the plasma is unstable, immediately click the **Plasma** switch to **Off** in the Plasma Control window (or press F9) to turn off the plasma; or, press the red **Emergency Plasma Off** button above the sample compartment.

An unstable plasma, as shown in the diagram, can cause the end of the torch to become overheated and to deform. If the deformity is severe, the argon gas flow patterns will be altered, and the torch must be replaced.

Note   Pressing the red **Emergency Plasma Off** button allows you to shut off the plasma directly and bypasses the software. This button can be used if the software hangs up or if communication between the instrument and the computer is interrupted. Using this button does not harm the instrument. If you use this button, you must reset the system before you can reignite the torch. Be sure that the red button is in the "out" position, and select the **Reset RF Emergency Off** command in the **System** menu.

3.   If you have not yet started the pump, place the sample capillary tubing in a container of a solution whose matrix matches that of the samples in your application (for example: deionized water, diluted acid, or organic solvents).

The pump automatically shuts off at the beginning of the ignition sequence and is restarted at the end of the ignition sequence.

4.   Click the **Plasma** switch to **On** in the Plasma Control window (or press F9) to turn on the plasma.

The Plasma Status display indicates when the plasma has been ignited.

5.   Immediately examine the plasma through the viewing window.

Examining the Plasma

After igniting the plasma, you must examine it through the viewing window to determine whether it is stable or unstable.

To examine the plasma

► Carefully note the characteristics of the plasma. A stable plasma will be situated just above the inner quartz tube in the torch and will have a bright discharge of the shape as shown. An unstable plasma has an irregular shape and may have an air gap underneath.

A stable plasma

An unstable plasma
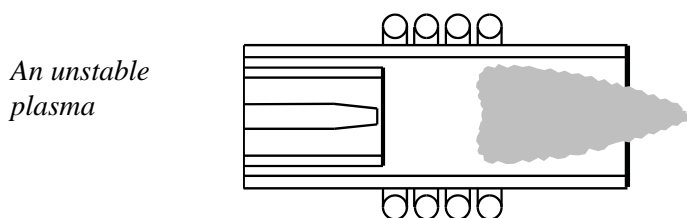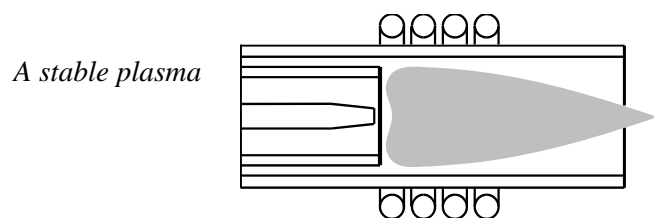
To proceed if the plasma is stable

1. Wait one hour before running samples. This ensures accuracy in your results, since it allows the temperature of the sample introduction system to fully stabilize.

2. Proceed to Optimizing and Verifying Performance.

To shut off the plasma if the plasma is unstable

1. Click the **Plasma** switch to **Off** in the Plasma Control window (or press F9) to turn off the plasma, or press the red **Emergency Plasma Off** button that is located above the sample compartment.

2. Proceed to Correcting Unsuccessful Ignition.

Note Pressing the red **Emergency Plasma Off** button allows you to shut off the plasma directly and bypasses the software. This button can be used if the software hangs up or if communication between the instrument and the computer is interrupted. Using this button does not harm the instrument. If you use this button, you must reset the system before you can reignite the torch. To reset the system, be sure that the red button is in the "out" position, and select the **Reset RF Emergency Off** command in the **System** menu.

To reignite the plasma after the plasma has been shut off

An unstable plasma is usually caused by air leaking into the system. To correct this problem, do the following:

1. Check the torch gas connections. For more information, see the Hardware Guide.

2. Repeat the ignition procedure. Click the **Plasma** switch to **On** in the Plasma Control window (or press F9).

   The Plasma Status window indicates the ignition status.

3. Immediately examine the plasma as before. If no plasma is formed, see *Correcting Unsuccessful Ignition*.

Correcting Unsuccessful Ignition

If the plasma fails to ignite during the ignition sequence, the plasma switch will go to the off position, indicating that ignition has failed but may be attempted again. Simultaneously, an error message is displayed in the Plasma Status display.

To correct unsuccessful ignition

Check this list of possible reasons for unsuccessful ignition. Review the Instrument Message history which is found on the Plasma Tab of the Diagnostics window. Correct the problem if possible, and then try to ignite the plasma again. See *Igniting the Plasma* earlier in this chapter.

Plasma Conditions

▶ Be sure you are using the correct plasma conditions for your application. Check the **Sampler:Plasma** page in the Method Editor.

Ignitor Cable or Contact

▶ On instruments with an ignitor cable, check to make sure that the cable is plugged in. On instruments with an ignitor contact, check that the copper strip on the torch is in close proximity to the ignitor contact finger.

Exhaust vent

▶ Be certain that your exhaust vent is operating properly and is not blocked. Operation of the exhaust vent is required to establish proper argon gas flow patterns as well as for safety reasons.

Air leaks

An unstable plasma is usually caused by air leaking into any part of the torch, nebulizer, or spray chamber. To correct this problem, do the following:

▶ Check the torch gas connections. For more information, see the Hardware Guide.

▶ Check that the torch O-rings are in good condition. Replace cracked or worn O-rings.

Drain

▶ Check that the fitting on the spray chamber drain is secure. Be sure that the pump is properly draining the spray chamber and that the drain liquid is not backing up into the spray chamber.

Nebulizer end cap

▶ Check that it is tightly secured to the spray chamber.

Sample capillary and tubing

Caution

When you release the tension on the peristaltic pump tubing (that leads to the nebulizer), be sure that the free end of the sample capillary or sample probe is not immersed in the solution. Failure to make this check may result in the solution being siphoned into the spray chamber and torch.

▶ Check that one end is attached to the nebulizer and that the other end is immersed in solution.

Organics

▶ If you are analyzing organic solutions and cannot ignite the plasma, turn on the nebulizer argon for two minutes with the pump off to purge the spray chamber.

To purge organic vapors from the sample introduction system

If you are switching from organic to aqueous solutions, purge organic vapors using the following steps.

1. Open the Plasma Control window. Be sure **Override Method** is checked so you can make the following adjustments.

2. Start the nebulizer argon flow at 0.50 L/min by clicking on **Neb** and setting 0.50 in the box above.

3. Start the auxiliary argon flow at 1.0 L/min by clicking on **Aux** and setting 1.0 in the box above.

4. Start the plasma argon flow at a rate of 15 L/min by clicking on **Plas** and setting 15.

5. Place the capillary tube from the peristaltic pump in a beaker of deionized water.

6. Change the pump rate to the high flush speed (approximately 2.5 mL/min) by clicking on **Flush**.

7. Continue aspirating water for approximately five minutes to thoroughly purge the sample introduction system of organic vapors.

    Alternative procedure for purging organics: Aspirate an approximately 1% liquid soap solution for about 15 minutes at the normal pump rate, followed by deionized water for another five minutes. If this does not improve the plasma condition and the precision, remove the sample introduction system and give it a thorough cleaning.

Note PerkinElmer recommends having two torch and sample introduction systems, one for use with agueous solutions and one for use with organic solutions.

Optimizing and Verifying Performance

Before proceeding, be sure you have completed instrument setup as outlined in *Setting Up the Instrument* earlier in this chapter.

Performance checks

Performance checks should be done regularly to help assure acceptable performance. The frequency of using these checks depends on how critical analytical precision is in your application. To help assure acceptable performance and to monitor instrument condition, it is suggested that a log be maintained. These tests include:

•   **Sodium bullet test**, where the aspiration of a solution of 1000 mg/L or more of sodium should produce a defined "bullet" in the center of the plasma discharge. This test allows you to visualize the sample flow in the plasma, so that you can check that the sample introduction system is working correctly.

- **Background Equivalent Concentration (BEC) test,** which is a useful indicator of the relative sensitivity of the instrument for a particular emission line**.** The BEC value is the concentration of an element which would produce the same emission intensity as the plasma background measured at the analyte wavelength. The BEC checks torch alignment, plasma viewing height (only meaningful in radial view), nebulizer gas flow rate, and incident RF power.

- **Precision Test,** which expresses the short-term precision for several measurements for a strong emission line. This test indicates the Relative Standard Deviation (RSD) of the instrument's analyte emission intensity or concentration measurements. The RSD may also be referred to as the CV (Coefficient of Variation). A high RSD or CV is usually indicative of a problem with the sample introduction system such as improper drainage, leaks, improper tension on the pump tubing, worn pump tubing, or nebulizer problems.

- **Detection Limit Test,** which measures the noise of the baseline signal in concentration units to give an indication of the lowest concentration of an element which can be measured. The detection limit is calculated as three times the standard deviation of the blank.

To make performance checks

▶ Use the following procedures in sequence or use the Browse buttons to move through each procedure.

1. Sodium Bullet Test

2. Creating a Method for the Performance Checks

3. Setting Up for the Performance Checks

4. Performing the Background Equivalent Concentration (BEC) Test

5. Performing the Precision Test

6. Performing the Detection Limit Test

Troubleshooting

▶ If acceptable performance is not found with these tests, see the Hardware Guide for troubleshooting performance problems. The performance tests can often pinpoint the cause of the problem.

When you have completed the performance tests, refer to *Selecting a Workspace, Method, and Sample Information File* later in this chapter.

Sodium Bullet Test

To run the sodium bullet test:

1. For aqueous analyses, aspirate a 1000 mg/L solution of sodium. For organic analyses, aspirate an appropriate organic blank.

2. Examine the plasma through the viewing window in the sample compartment door.

A yellow-orange bullet (or green, in the case of organic compounds) should be visible in the center of the discharge, and should extend from the base of the discharge to about 2-3 mm past the RF coil. If the bullet height is unsatisfactory, adjust the nebulizer argon flow in the Method Editor or Plasma Control windows.

If no bullet appears or the bullet is faint:

• Check that your sample does contain sodium at the required concentration.

• Check that sample is being pumped to the nebulizer. Make sure that the pump lever is engaged and that the pump tubes are connected appropriately.

• Check that the drain is being pumped properly. You should see a segmented flow of bubbles through the tubing.

If the above checks fail to make an improvement, turn off the plasma, then check the following:

• Check that the nebulizer end cap is connected tightly.

- Check the nebulizer spray pattern: run deionized water for several minutes, then remove the nebulizer end cap. If the nebulizer spray is sputtering or uneven, inspect the nebulizer tips for clogging and clean or replace them as necessary.

Warning

If the plasma has been on, allow five minutes for the torch to cool before checking the following.

- Check that the injector is not clogged. You will need to disassemble the torch as described in Chapter 4, Maintenance in the Hardware Guide for your instrument.

Creating a Method for the Performance Checks

The following procedure describes how to create the method you will need for the BEC, Detection Limit, and Precision tests. This method can be stored and recalled later for periodic performance checks at whatever interval is desired.

To create the method for the tests:

1. In the **File** menu, click on **New ▶ Method...**

2. In the Create New Method dialog, select default conditions for an aqueous analysis.

3. When the Method Editor appears, click on **Periodic Table.** In the Periodic Table, select manganese using the 257.610 wavelength. Select other elements you may be interested in using for the performance checks. If available, cadmium or titanium (in addition to manganese) would be good choices to get an overall indication of the instrument's performance. For cadmium, select 226.502 nm; for titanium, select 334.940 nm.

4. Type a description for the method. In the **File** menu, select **Save ▶ Method**. Type a name for your method such as "perftest" and click on **OK**.

5. On the **Spectrometer: Settings** page, select:

    Read Time: Min 10 sec.; Max 20 sec.
    Read Delay: 60 sec. for manual sampling; approx. 120 sec. for an autosampler
    Replicates: 3

6. Click on the Sampler tab. On the **Plasma** page, select **Same for all Elements** and use the parameters listed below for axial viewing:

	Axial Viewing
Plasma Flow:	15 L/min
Aux Flow:	0.5 L/min
Neb Flow:	0.75 L/min
RF Power:	1450 W
View Dist:	15 mm

7. Click on the **Sampler** tab. On the **Peristaltic Pump** page, select:

    Sample Flow Rate:1.50 mL/min

8. Click on the **Process** tab. On the **Peak Processing** page, select:

    Peak Algorithm: Peak Area
    Points/Peak: 3

9. Click on the **Calibration** tab. On the **Define Standards** page, you need only one Calibration Blank and one Calibration Standard. On the **Calib Units and Concentration** page, select 10 mg/L of Manganese. If there are other elements in your method, select the standard concentrations for these elements. Typically, use a concentration 100 times greater than the expected detection limit.

10. Click on the **Options** tab. For the Results Display and Printed Log, select:

    Analytical Header
    Replicate Data
    Means and Statistics

11. In the **File** menu, select **Save** ▶ **Method**.

Setting Up for the Performance Checks

Solutions You'll Need

To do the performance checks, you will need the following solutions:

- Manganese solution: 10 mg/L (in 1% HNO_3 or another appropriate acid). Optionally, you can use a multielement solution containing other elements of interest (at a concentration 100 times greater than the recommended detection limit If available, cadmium or titanium (in addition to manganese) would be good choices to get an overall indication of the instrument's performance.

- Blank solution containing deionized water and an appropriate acid to match the sample matrix.

- Rinse solution containing deionized water and an appropriate acid. The acid concentration should match the standard and samples.

Setting Up for the Tests

To run the tests, you will need a method. Refer to *Creating a Method for the Performance Checks* earlier in this chapter.

Before running the tests, you should have completed the following procedures:

- Detector Calibration

- Wavelength Calibration

- Torch View Optimization

Performing the Background Equivalent Concentration (BEC) Test

How is the BEC Determined?

In performing the BEC test, you determine what concentration of analyte is equivalent to the plasma background at the analyte wavelength. This provides an indication of the sensitivity of the instrument. To do the test you first perform a calibration by running a blank followed by a standard. The calibration establishes the relation between emission intensity and concentration. A calibration also takes into account the dark current noise.

Once the calibration is complete, you close the shutter, thereby blocking the light from the plasma source from reaching the detector. At this point, you take a reading (at zero intensity). This extrapolates the calibration line backward until it intercepts the concentration axis. The resulting concentration value is negative. By changing the sign to positive, you obtain the BEC value.

Example of a calibration curve used in determining the BEC value.
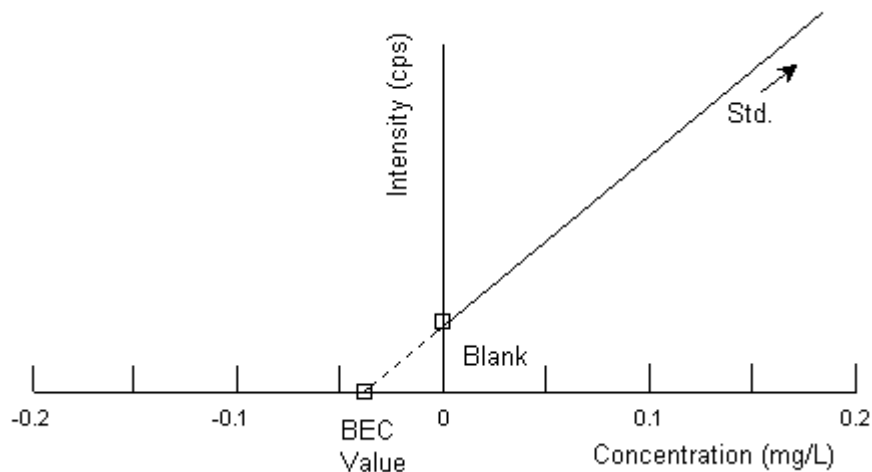
Performing the BEC Test

Before starting, you should have already set up the system for the tests. If not, see *Setting Up for the Performance Checks* earlier in this chapter.

1. Check that the method you created for the performance tests (for example, "perftest", is open. If not, in the **File** menu, click on **Open ▶ Method.**

2. Click on the **Manual** icon to open the Manual Analysis Control window.

3. In the Manual Analysis Control window, select **Print Log**. In addition, if you want to save your results, click on **Open...** and select a Results Data Set Name.

4. Aspirate the blank and click on **Analyze Blank**.

5. Aspirate the manganese or multielement solution and click on **Analyze Standard**. This completes the calibration.

6. Aspirate the rinse solution.

7. In the **Tools** menu, click on **Spectrometer Control**.

8. In the Spectrometer Control window, close the shutter by selecting the "Closed" option.

9. Click on the Manual Analysis Control window to bring it to the front. For the Read Delay, select **Override Method** and set the Read Delay to **0**.

10. While continuing to aspirate the rinse solution, click on **Analyze Sample**. (This will give you results in concentration units.)

11. Click on the Spectrometer Control window to bring it to the front. Change the shutter from Closed to the Auto position, which returns it to instrument control.

Check the results. By taking the negative value and making it a positive value, this gives you the BEC for Mn 257.610. It should be less than or equal to 0.04 mg/L. If it is not, refer to *Performance Problems* in Chapter 5, Troubleshooting, in your Hardware Guide.

Performing the Precision Test

Before starting, you should have already set up the system for the tests. If not, see *Setting Up for the Performance Checks* earlier in this chapter. For convenience, you may want to run this test after the BEC test. You can use a solution of 10 mg/L manganese for the test or, optionally, a multielement solution containing other elements of interest.

1. Check that the method you created for the performance tests (for example, "perftest") is open. If not, in the **File** menu, click on **Open ▶ Method.**

2. If you have just run the BEC test, the Manual Analysis Control window should be displayed. If not, click on the **Manual** icon.

3. In the Method Editor, click on the **Spectrometer: Settings** page and specify 10 replicates.

4. In the Manual Analysis Control window, disable **Override Method** to use the Read Delay specified in the method. To disable it, click on the box to remove the check mark.

5. Aspirate the manganese or multielement solution.

6. In the Manual Analysis Control window, click on **Analyze Sample**.

Check the results. The RSD should be less than 1.0%. If it is not, refer to *Performance Problems* in Chapter 5, Troubleshooting, in your Hardware Guide.

Performing the Detection Limit Test

Before starting, you should have already set up the system for the tests. If not, see *Setting Up for the Performance Checks* earlier in this chapter. For convenience, you may want to run this test after the BEC or Precision test. You can use a solution of 10 mg/L manganese for the test or, optionally, a multielement solution containing other elements of interest.

1. Check that the method you created for the performance tests (for example, "perftest", is open. If not, in the **File** menu, click on **Open ▶ Method.**

    Make the following changes to the method:

    On the **Spectrometer: Settings** page, select:

    Read Time: Min 50 sec.; Max 50 sec.
    Replicates: 20

Note Increasing the Read Time in the method to 50 seconds will provide better detection limits.

---

2. If you have just run the BEC or Precision test, the Manual Analysis Control window should be displayed. If not, click on the **Manual** icon.

3. Aspirate the rinse solution and rinse sufficiently, particularly if you have just done a Precision test (rinse for two minutes).

4. If you have already done the BEC test, this included a calibration so you can skip to Step 5. If you have *not* yet done a calibration:

    •   Aspirate the blank and click on **Analyze Blank**.

    •   Aspirate the manganese or multielement solution and click on **Analyze Standard**. Rinse for at least 2 minutes after the standard is analyzed. A full 5-minute rinse is recommended.

5. Aspirate the blank solution.

6. While continuing to aspirate the blank, press **Analyze Sample**. (This will give you results in concentration units.)

Check the results. To determine the detection limit, multiply the standard deviation of the blank by three. Compare your results with the required detection limits for the elements.

Detection limits for the three elements we suggested for the test are:

Analyte	Wavelength	Detection Limit
Mn	257.610 nm	1.0 µg/L
Ti	334.940 nm	0.5 µg/L
Cd	226.502 nm	1.6 µg/L

If your results are too high, refer to *Performance Problems* in Chapter 5, Troubleshooting, in your Hardware Guide.

Selecting a Workspace, Method, and Sample Information File

Before you can analyze samples, you need: windows suitable for your analysis; one or more methods; and a sample information file.

To select these items for automated analyses

1. In the **File** menu, click on **Open** ▶ **Workspace...** and select the workspace desired.

    The group of windows (a workspace) that appears enables you to carry out your analysis. The Automated Analysis Control window must appear in this workspace. For example, you can set up a workspace using the following windows; Spectra Display, Results Display, Calibration Display, and Plasma Control or any other windows of your choice.

    If you do not select a workspace, you can select these windows individually from the Windows and Tools menus. The selected windows can be saved as a workspace by using **Save As** ▶ **Workspace...** in the **File** menu.

2. Select a method on the Automated Analysis Control window Set Up page by double-clicking on the first empty cell in the Method column. In the Open Method dialog, select the method that you wish to use. For more information, see *Opening and Saving Methods and Results* in Chapter 11, *Menus and Toolbar*.

    If an appropriate method is not there, see *Creating a Method* earlier in this chapter.

3. To select more than one method, you must first select **Open Methods in List** on the Setup page. This option is located below the Methods and Sample Locations table. Repeat Step 2 to select additional methods.

4. Select a sample information file if desired. By using a sample information file, information you supply about the samples (for example, sample weights or dilutions) is used in the analysis. In the Automated Analysis Control window Set Up page, click on **Open…** and select a sample information file in the Open Sample Information dialog.

    If an appropriate file is not present, see *Creating a Sample Information File* in Chapter 4, *Sample Information Editor* for details.

5. If you want to run samples from a sample information file, first select the check box for **Use Sample Information.** In the table, under the Sample Info File column, three options appear in a drop-down list. If you want to analyze all of the samples in the sample information file, select **All Defined** from the drop-down list. If you want to select only certain autosampler locations, select **Locations**. If you want to select samples by the sample numbers listed in the sample information file, select **Sample Nos.** If you select Locations or Sample Nos., be sure to enter the autosampler locations or sample numbers in the appropriate columns. Example: **10-15,18,20,22,25-30**

    - or -

    If you are not using a sample information file, deselect the check box for **Use Sample Information** below the name of the sample information file. Then enter the autosampler locations in the Locations column.

    Type individual locations or a range of locations. Use commas to separate the locations and ranges. It is not necessary to enter the locations of blanks, QC's, check or calibration solutions. Enter locations for these if you want them to be treated as a sample within the analysis. Example: **10-15,18,20,22,25-30**

6. The settings you have made can be saved in a workspace by using **Save As** ▶ **Workspace...** in the **File** menu.

To select these items for manual analyses

1. In the **File** menu, click on **Open** ▶ **Workspace...** to select the workspace.

    The group of windows (a workspace) that appears enables you to carry out your analysis. The Manual Analysis Control window must appear in this workspace. Optional windows include: Spectra Display, Results Display, Calibration Display, and Plasma Control.

    If you do not select a workspace, you can select these windows individually from the Windows and Tools menus. The selected windows can be saved as a workspace by using **Save As** ▶ **Workspace...** in the **File** menu.

2. In the **File** menu, click on **Open** ▶ **Method...** to select a method,

    If an appropriate method is not there, see *Creating a Method* earlier in this chapter.

3.   A sample information file is optional. You may want a sample information file if you want to enter all the sample information before an analysis begins. See *Creating a Sample Information File* in Chapter 4, *Sample Information Editor* for details.

If you do not select a sample information file, the software uses the default file called "Untitled." Be sure to open the Sample Information Editor and check that entries in the "Untitled" file are appropriate for your analysis. If some entries are completed, but others are not, the sample concentration may not be reported in sample units. See *Sample Preparation Parameters* in Chapter 4, *Sample Information Editor* for details.

4.   The settings you have made can be saved in a workspace by using **Save As ▶ Workspace...** in the **File** menu.

Setting Options in Control Window

Before you can analyze samples, you need to set certain options in the control window that you are using. These options are not specified in the method and include: specifying the results data set where you will save data; specifying whether you want the results printed during the analysis; specifying special automatic startup and shutdown options; and specifying automatic export of data.

For all analyses, to select options for the analyses

1.   In the Automated Analysis Control window Set Up page or the Manual Analysis Control window, click on **Open...** next to the **Results Data Set Name** information field. Select the name of the data set where you will save the results. If this data set already exists, new data will be added to it. When you select a results data set name, the Save Data box is selected automatically (a checkmark appears), confirming that data will be saved.

Note   Archive result data sets periodically to avoid filling up space on the hard disk. For more information, please refer to the Maintaining Data section of the Data Manager Help. To calculate the amount of hard disk storage space that is required for storing result data sets, see *Equation for Calculating Disk Storage for Results* in Chapter 5, *Analysis Control Windows*.

2. To print a log of intensity counts, analytical results, and other information about the analysis, select the **Print Log During Analyses** box.

For automated analyses only, to select other options

1. If you want certain components to be automatically switched off when the analysis is finished, first select the **Automatic Shutdown** box, then click on **Set...** to open the Automatic Shutdown/Startup dialog. Complete this dialog, and click on **OK.**

2. If you want Automatic Startup to be enabled, use the above dialog to schedule an automatic startup. You can also specify whether the plasma is to be ignited and if the pump is to begin operating upon startup.

3. During the analyses, you can automatically export data contained in the results data set and write it into a file that can be read by many other programs, including spreadsheet and database management programs. To do this, select **Auto Export**, then click on **Set...** and select the Export Designs. You must first create the Export Design(s) in the WinLab32 Data Manager. An export design defines a subset of data items that you want to export from a data set. For more information, refer to WinLab32 Data Manager Help.

4. On simultaneous ICP instruments only, you can select automatic wavelength realignment to occur at regular intervals. Select the **Auto Wavelength Realign** box on the Automated Analysis Control window Set Up page and, then click on **Set...** to set a time interval.

5. The settings you have made can be saved in a workspace by using **Save As ▶ Workspace...** in the **File** menu.

For more information on analyses using an autosampler, see the *Arranging Samples* section below. For more information on manual analyses, see *Analyzing Samples* later in this chapter.

Arranging Samples

Before arranging samples in the autosampler using the autosampler loading list, you must have entered all information for your samples and solutions in your method or methods and in the sample information file if you have one.

For details on where to enter this information, see: Manual Analysis Control window, Method Editor, and Sample Information Editor.

To get samples and solutions ready for analyses

▶ Prepare the required samples and solutions according to your standard laboratory procedures.

To load samples and solutions in the autosampler using the Automated Analysis Control window

1. In the **Automated Analysis Control** window, click on the **Analyze** tab.

2. In the **System** menu, click on **Autosampler Loading List** to see a list of the locations of your samples and solutions.

3. Load samples and solutions in an empty tray.

4. Click on **Load Tray**.

5. Place the loaded tray in the autosampler.

6. In the **Analysis** menu, click on **Autosampler** ▶ **Go to Wash**.

To load samples and solutions in the autosampler when the Manual Analysis window is open

1. In the **System** menu, click on **Autosampler Loading List** to see a list of the locations of your samples and solutions.

2. Load samples and solutions in an empty tray.

3. In the **Analysis** menu, click on **Autosampler** ▶ **Load Autosampler Tray** or press Shift-F11.

4. Place the loaded tray in the autosampler.

5. Click on **Go to Wash Loc**. in the Manual Analysis Control window.

Analyzing Samples

Before analyzing samples, be sure you have performed all preliminary steps as summarized in *Performing Analyses: Overview* earlier in this chapter.

Three types of analyses

▶ Select how you will analyze samples from the options below.
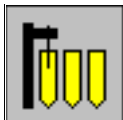
Automated Analyses, using an autosampler to analyze all samples and solutions

Manual Analyses, manually presenting samples and solutions to the instrument

Manual Analyses Using the Autosampler, requiring both manual operations and an autosampler to analyze samples and solutions. In this case, often used for method development, you want direct control of the autosampler, so the autosampler is just holding samples for you.

Automated Analyses

Automated analyses are performed using the Automated Analysis Control window. Other windows such as the Results window are optional.

To perform automated analyses

Step	What it does
Examining the Run List	Before analyzing samples, you should check the run list. If any changes are required, you must change either the method or the sample information file, then rebuild the run list.
Calibrating and Analyzing Samples	Solutions and samples are analyzed. A calibration is performed according to your specifications, and you can recalibrate if desired.
Stopping and Restarting	During the analysis, you may need to stop operations and restart the analysis, according to these procedures.
Analyzing Additional Samples	While an automated analysis is in progress, you can add samples to the sample sequence, enter information on a new sample, or analyze additional samples at the end of an analysis.

Note During an analysis, the shutter automatically opens and closes for each sample. If you have an extensive number of samples to analyze, however, you may wish to set the shutter to the open position manually to increase analytical throughput. You do this in the Spectrometer Control Window. Be sure to close the shutter when the analysis is complete.

<table>
<tr><td rowspan="2">⚠
Warning</td><td>Leaving the shutter in the open position with the plasma on could lead to deterioration of the optics and eventual degradation of UV performance. It is recommended that the shutter be closed when analyses are not being performed.</td></tr>
</table>

Examining the Run List

To examine the run list before analyzing samples

1. Examine the list of solutions in the run list on the Analyze page of the Automated Analysis Control window. If you have changed the method or sample information file, click on **Rebuild List** to view an updated run list. Verify that your run list is correct. If it is correct, skip steps 2 and 3 below.

   A sample run list is shown below.
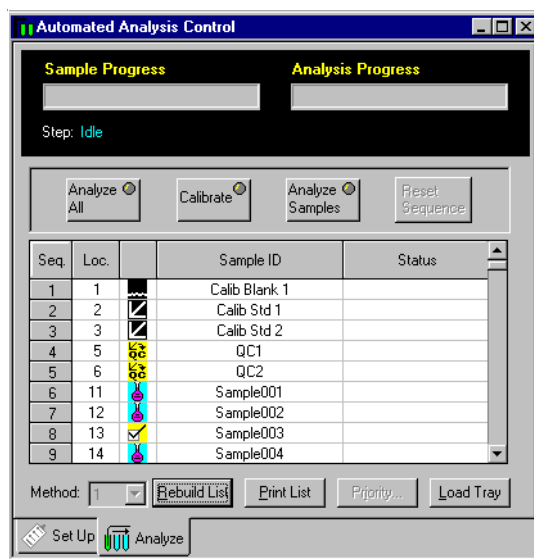
2. To make changes to the run list for unknown samples, go to the Sample Information Editor and make the changes in the Sample Information file. To make changes to QC samples, go to the Method Editor and make changes in

the QC section of the method. If you have matrix check samples and need to make changes, these samples are defined in the method and scheduled in the sample information file.

3. To make changes to the run list for calibration solutions, go to the Method Editor Calibration: Define Standards Page and make the changes.

4. To analyze a selected group of samples that are a subset of samples in the sample information file, type the locations or sample numbers on the Automated Analysis Control window Set Up page. Example: **10-15,18,20,22,25-30**

Calibrating and Analyzing Samples

According to your calibration choices, select the appropriate steps below. All controls (**Analyze All**, **Calibrate**, and **Analyze Samples**) are located on the Automated Analysis Control Analyze page. You select the calibration parameters in the Method Editor Calibration pages and view the calibration curves in the Calibration Display window. You can also view a summary of the calibration data in the Results Display Window.

Note	Directly before analyzing aqueous solutions, we recommend that you aspirate a solution of deionized water with an acid concentration that matches the calibration blank and standards for at least 10 minutes. This procedure stabilizes the sample introduction system.

Select a calibration option and analyze mode

To do this	Use this
To generate a new calibration and continue with samples (overrides Initial Calibration page of the Method Editor)	Click on **Analyze All**.
To generate a new calibration, examine it, and continue with samples	Click on **Calibrate**. Examine the calibration and recalibrate if desired. When ready, click on **Analyze Samples**. Continued on the next page.

To recalibrate	Click on **Calibrate**.
To analyze samples when a calibration is automatically recalled with the method (as directed on the Initial Calibration page of the Method Editor)	Click on **Analyze Samples** to analyze samples using this calibration.
To manually recall a calibration and then analyze samples	In the **Analysis** menu, click on **Recall Calibration**... Select the results data set that contains the desired calibration. Then, click on **Analyze Samples**.

Stopping and Restarting

Note When you stop an analysis, the autosampler probe automatically goes back to the wash.

Select stop and restart options from the following

To do this	Follow these steps
To stop an analysis	• In the Automated Analysis Control window Analyze page, click on the button that you used to start the analysis, press **F8** (**Cancel**), or select **Cancel Analysis...** from the Analysis menu. • In the Stopping an Analytical Sequence dialog select the option that indicates how you want to stop.

Continued on the next page.

To change Set Up options after an analysis has begun.	• Click on the tab for the **Set Up page**.
	• Change any available set up option. For example, you can select whether or not to save data, change the sample information file or program an automatic shutdown. On simultaneous ICP instruments, you can select an auto wavelength realign.
	• Some options are locked. To change any locked option, you must first click on the tab for the Analysis page and then click on Reset Sequence. For example, you cannot change a data set until you click on Reset Sequence.
To restart an analysis from a selected solution	• In the Automated Analysis Control window Analyze page, click on the button that you originally used to start the analysis. The other buttons will be disabled.
	• In the Continuing an Analytical Sequence dialog, select where you want the analysis to continue.
	Tip: The Run List on the Analyze page of the Automated Analysis Control window shows the sample, highlighted in yellow, that was being run when the analysis stopped.
To restart an analysis from the beginning	In the Automated Analysis Control window Analyze page, click on **Reset Sequence** and then on a button (**Analyze All**, **Calibrate**, or **Analyze Samples**) that starts an analysis.

Analyzing Additional Samples

Several options are available when you need to analyze additional samples while an analysis is in progress:

▶ To add samples to the sample sequence while an automated analysis is in progress, click on **Priority...** on the Analyze page of the Automated Analysis Control window. The Add Sample Dialog appears.

▶ To enter information on a new sample that you want to insert in the analytical sequence, in the Analysis menu, click on **Automated Analysis ▶ Append Samples to Analysis List** while an automated analysis is in progress. The Append to Analysis List Dialog appears.

▶ To analyze additional samples at the end of an analysis, in the **Analysis** menu, click on the **Automated Analysis ▶ Append Samples to Analysis List** command to append samples from your sample information file to the end of the run list while an automated analysis is in progress. In the Append to Analysis List Dialog that appears, enter the sample numbers from the sample information file (**do not** enter autosampler locations).

After an automated analysis is completed, if you need to analyze additional samples, use the following procedure.

1. Check that the calibration curve still appears in the Calibration Display window. If not, select **Recall Calibration...** from the Analysis menu, then select the results data set that contains the calibration.

2. If you are using a Sample Information file, list the additional samples and autosampler locations.

3. In the Automated Analysis Control Set Up page, select the autosampler locations for the additional samples.

4. Click on the **Analyze** tab in the Automated Analysis Control window.

5. Click on **Analyze Samples.**

Manual Analyses

Manual analyses are performed using the Manual Analysis Control window. Other windows such as the Results window are optional.

To perform manual analyses

Step	What it does
Calibrating	A calibration is performed according to your specifications, and you can recalibrate if desired.
Analyzing Samples	Solutions and samples are analyzed.
Stopping and Restarting	During the analysis, you may need to stop operations and restart the analysis, according to these procedures.

Calibrating

You select calibration information in the Method Editor Calibration page, analyze each calibration standard, and view the resulting calibration curves in the Calibration Display window. You can also view a summary of the calibration data in the Results Display window by clicking on the Calibration Summary command in the Analysis menu.

Note Directly before analyzing aqueous solutions, we recommend that you aspirate a solution of deionized water with an acid concentration that matches the calibration blank and standards for at least 10 minutes. This procedure stabilizes the sample introduction system.

To generate a new calibration

1. To analyze each calibration blank:

    ▶ Select the calibration blank name from the list next to **Analyze Blank** in
      the Manual Analysis Control window.

    ▶ Click on **Analyze Blank**.

2. To analyze each calibration standard, select the calibration standard name from
    the list next to **Analyze Standard**.

    ▶ If you want to view the concentration for a given calibration standard, click
      on **Conc...**

    ▶ To change these concentrations, go to the Method Editor Calibration:
      Calib Units and Concentrations page.

3. When you are ready to analyze each calibration standard, click on **Analyze
    Standard**. After you have analyzed a blank and a calibration standard, you can
    view a summary of the calibration data in the Results Display Window by
    clicking on **Calibration Summary** in the Analysis menu.

4. To analyze each reagent blank, select the reagent blank name from the list next
    to **Analyze Blank**. Click on **Analyze Blank**.

5. Use the Calibration Display window to examine the calibration curve and
    recalibrate if desired.

To recall a calibration

1. Open a method that has the same analytes as the calibration curves you are
    interested in viewing.

2. In the Analysis menu, click on **Recall Calibration...**

3. Select the results data set that contains the desired calibration.

To recalibrate

▶ Repeat the steps above for generating a new calibration.

Analyzing Samples

Manual analyses can be performed with or without a sample information file.

To analyze samples with a sample information file

The name of the sample information file you have selected is displayed in the Manual Analysis Control window next to **Info. File.**

1. In the Manual Analysis Control window, select the number (**No.**) of the sample you want to analyze.

2. Click on **Analyze Sample.**

To analyze samples without a sample information file

1. If desired, type a sample ID (**ID**) in the Manual Analysis Control window.

2. If desired, enter other information (such as sample weight and dilution values) about the sample. To do this, click on Details...

Note   If you want different categories of information to appear in the Sample Details dialog, you may open a sample information file that contains these categories.

3. Click on **Analyze Sample**.

Stopping and Restarting

To stop an analysis

▶ In the Manual Analysis Control window, click on the button that you used to start the analysis, press **F8** (**Cancel**), or select **Cancel Analysis...** from the Analysis menu.

To restart an analysis

► Click on a button that starts an analysis (**Analyze Blank, Analyze Sample, Analyze Standard)**.

Manual Analyses Using an Autosampler

Manual analyses using an autosampler are performed using the Manual Analysis Control window. Other windows such as the Results window are optional.

To perform manual analyses using an autosampler

Step	What it does
Calibrating	A calibration is performed according to your specifications, and you can recalibrate if desired.
Analyzing Samples	Solutions and samples are analyzed.
Stopping and Restarting	During the analysis, you may need to stop operations and restart the analysis, according to these procedures.
Analyzing Additional Samples	If you have completed a manual analysis using an autosampler and need to analyze additional samples, refer to this procedure.

Calibrating

Note Directly before analyzing aqueous solutions, we recommend that you aspirate a solution of deionized water with an acid concentration that matches the calibration blank and standards for at least 10 minutes. This procedure stabilizes the sample introduction system.

To generate a new calibration

1. To analyze each calibration blank:

   ▶ Select the autosampler location from the values next to **Go to A/S Loc.>>** in the Manual Analysis Control window.

   ▶ Click on **Go to A/S Loc.>>**.

   ▶ Select the calibration blank name from the list next to **Analyze Blank**.

   ▶ Click on **Analyze Blank**.

2. To analyze each calibration standard, select the calibration standard name from the list next to **Analyze Standard**.

   ▶ Select the autosampler location from the values next to **Go to A/S Loc.>>**.

   ▶ Click on **Go to A/S Loc.>>**.

   ▶ If you want to view the concentration for a given calibration standard, click on **Conc...**

   ▶ To change these concentrations, go to the Method Editor Calibration: Calib Units and Concentrations page.

3. When you are ready to analyze each calibration standard, click on **Analyze Standard**.

Tip   Use **Go to Wash Loc.** between the calibration standards and the reagent blank to avoid carryover.

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4. To analyze each reagent blank:

   ▶ Select the autosampler location from the values next to **Go to A/S Loc.>>**.

   ▶ Click on **Go to A/S Loc.>>**.

► Select the reagent blank name from the list next to **Analyze Blank**.

► Click on **Analyze Blank**.

5. Use the Calibration Display window to examine the calibration curve and recalibrate if desired.

To recall a calibration

1. In the Analysis menu, click on **Recall Calibration...**

2. Select the results data set that contains the desired calibration.

To recalibrate

► Repeat the steps above for generating a new calibration.

Analyzing Samples

Tip Use **Go to Wash Loc.** before analyzing samples to avoid carryover from your calibration standards.

To analyze samples with a sample information file

The name of the sample information file you have selected is displayed in the Manual Analysis Control window next to **Info. File.**

1. In the Manual Analysis Control window, select the autosampler location from the values next to **Go to A/S Loc.>>**.
    Note: In a manual analysis, the autosampler location in a sample information file is not used by the software; therefore, you must use **Go to A/S Loc.>>** to select the location each time you analyze a sample.

2. Click on **Go to A/S Loc.>>**.

3. Select the number (**No.**) of the sample you want to analyze.

4. Click on **Analyze Sample**.

To analyze samples without a sample information file

1. In the Manual Analysis Control window, select the autosampler location from the values next to **Go to A/S Loc.>>**.

2. Click on **Go to A/S Loc.>>**.

3. If desired, type a sample ID (**ID**).

4. If desired, enter other information (such as sample weight and dilution values) about the sample. To do this, click on Details...

Note   If you want different categories of information to appear in the Sample Details dialog, you need to add the categories to the sample information file. For more information, see *Customizing the Sample Information Editor* in Chapter 4, *Sample Information Editor*.

5. Click on **Analyze Sample**.

Stopping and Restarting

To stop an analysis

▶ In the Manual Analysis Control window, click on the button that you used to start the analysis, press **F8** (**Cancel**), or select **Cancel Analysis...** from the Analysis menu.

Note   When you stop an analysis, be sure to click on **Go to Wash Loc.** The autosampler probe returns to the wash location.

To restart an analysis

▶ Click on a button that starts an analysis (**Analyze Blank, Analyze Sample, Analyze Standard)**.

Analyzing Additional Samples

If you have completed a manual analysis and need to analyze additional samples, use the following procedure.

1. Check that the calibration curve still appears in the Calibration Display window. If not, select **Recall Calibration…** from the Analysis menu, then select the results data set that contains the calibration.

2. If you are using a Sample Information file, list the additional samples and autosampler locations.

3. In the Manual Analysis Control window, select the autosampler location, Sample Number (No.) and ID for the additional sample that you want to analyze.

4. Click on **Analyze Sample**.

Generating Reports

If you saved data in a results data set (as requested in an analysis control window), you can use the WinLab32 Data Manager to generate reports in a wide variety of formats.

To start the WinLab32 Data Manager

► In the **File** menu, click on **Utilities** ► **Data Manager**.

or

► Click on the **Start** button in Windows. Select **Programs** ► **PerkinElmer WinLab32** ► **Data Manager**.

Shutting Down the Instrument

There are two ways to extinguish the plasma. You can extinguish the plasma manually or you can extinguish it automatically using Automatic Shutdown. Automatic Shutdown can be selected in the Automatic Shutdown/Startup dialog, which is accessed from the Automated Analysis Control Set Up page or using the Auto Shutdown/Startup command in the System menu.

Another mode is Automatic Startup, which can also be selected in the Automatic Shutdown/Startup dialog, as described above. If you selected Automatic Startup to restart your system, this affects the procedures that you follow when you shut down.

Note   If you have scheduled an Automatic Shutdown or Startup, you must leave the WinLab32 software on. Do not exit the software.

To automatically extinguish the plasma and put the spectrometer on standby

If you have selected Automatic Shutdown and indicated how you want the system to shut down, the system will automatically shut down and flush the sample introduction system according to your choices. The system next extinguishes the plasma and puts the spectrometer into standby if that option was selected. The software remains on.

To manually extinguish the plasma

1. Flush the sample introduction system for five minutes with the plasma on. During this five minutes, if you analyzed aqueous solutions, either flush with deionized water only or flush with a dilute acid solution, followed by deionized water. After analyzing organic solutions, flush the system with an appropriate clean solvent.

2. Extinguish the plasma by clicking on the **Plasma Off** switch in the Plasma Control window.

| **Note** | Pressing the red **Emergency Plasma Off** button on the instrument allows you to shut off the plasma directly and bypasses the software. This button can be used if the software hangs up or if communication between the instrument and the computer is interrupted. Using this button does not harm the instrument. If you use this button, you must reset the system before you can reignite the plasma. To reset the system, be sure that the red button is in the out position, and select the **Reset Emergency Off** command in the **System** menu. |

When the plasma is extinguished, the plasma off/on switch in the Plasma Control window changes from blue to white and the peristaltic pump stops pumping solution to the plasma.

3. Proceed with this step depending upon the conditions of **Automatic Startup**.

   If Automatic Startup is not being used

   -or-

   If Automatic Startup has been selected and does not include turning on the plasma and the pump

   ▶ Take the sample capillary out of the flush solution, and then run the pump for a few minutes to clear solvent from the sample capillary and pump the spray chamber.

   ▶ Release the clamp lever and remove the tubing. (This will increase the lifetime of the pump tubing.)

Caution

Always remove the sample capillary or the autosampler probe from the solution when you are finished using the pump. Otherwise, the solution can siphon into the spray chamber and flood it.

If Automatic Startup has been selected and includes turning on the plasma and the pump

- ► Leave the sample capillary in the wash solution.

- ► If the level of the wash solution reservoir in the autosampler is above the spray chamber, leave the peristaltic pump tubing clamped. This prevents the wash solution from being siphoned into the spray chamber.

  -or-

- ► If the level of the wash solution reservoir in the autosampler is below the spray chamber, release the clamp lever on the pump tubing. This prevents the solution from being pumped when the system automatically starts up.

4. If desired, you can manually put the spectrometer on standby as described below.

To manually put the spectrometer on standby

1. In the **System** menu, click on Auto Shutdown/Startup.

2. In the dialog, select **Enable shutdown...** and **Immediately on OK**. Be sure **Put Spectrometer into Standby** is selected.

3. If the plasma has already been extinguished, be sure that the **Wash before shutdown** option is not selected and that **Turn off Plasma and Pump** is not selected.

4. Be sure that you have enabled Automatic Startup and set the date and time.

Using the Method Editor 3

Method Editor Overview

You use the Method Editor to create a new method or to modify the parameters in an existing method. You must have one or more methods to perform analyses.

What is a method?

All analytical information needed to perform an analysis and report the results of an analysis is contained in a method (and a sample information file, if it is used). You can use an existing method as it is, modify an existing method to suit the purposes of your application, or create a new method.

Modified or new methods must be saved in a methods library if you wish to use them at a later date. You can delete, copy, or rename methods using the Data Manager application.

Methods in the Results Library

When you perform analyses, a copy of the method can be saved automatically in a results library as part of the results data set (if you so designated on the Method Editor Options page). You might want to do this so that you can recall such methods from a results library if, for example, you wanted information about the method used for a certain sample or wanted to save a particular method in a methods library for later use.

The active method

The "active" method, which is displayed in the toolbar, is the method that is open. When you start an analysis, the active method defines the parameters of the analysis.

The pages of the Method Editor

The Method Editor window contains bottom tabs, and each tab provides access to a set of pages.

The contents of the pages are described here:

Tab/Pages	Contents
Spectrometer	Parameters for Spectrometer settings.
Sampler	Parameters for the plasma, peristaltic pump and autosampler wash.
Process	Directions to convert spectral data into intensity data, correct for interferences and assign internal standards.
Calibration	Calibration information, autosampler locations, and details on how sample results will appear.
Checks	Recovery tab - Spike concentrations for matrix check samples.
	Sample Limits tab - Specifications for performing sample limit checks.
QC	Locations of quality control solutions and instructions for scheduling and performing quality control procedures.
Options	Remarks about the method and options for the results display, printed log, and results data set.

Using the Method Editor

To display this window

▶ In the **Tools** menu, click on **Method Editor,** or on the **Toolbar** click on **MethEd**

▶ If you do not have an open method, in the **File** menu, click on **Open** ▶ **Method**.

- or -

▶ In the Method /Sample Info bar, click on **Method:**
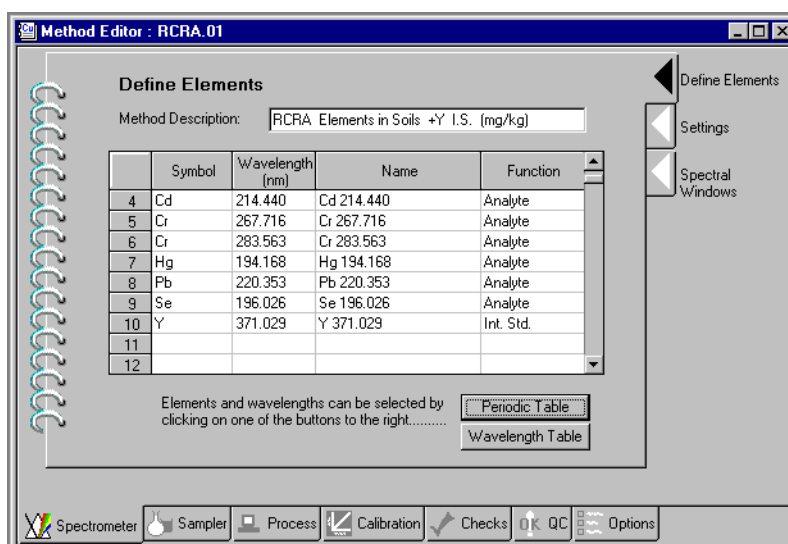
Using methods

There are several ways to get methods for your analyses. You can open an existing method and use it as it is, modify it to suit your application, or create a new method.

Summary procedure: using methods

1. Open an existing method or create a new one.

2. If you are creating a new method or modifying an existing method, open the Method Editor. On the pages of the Method Editor window, modify the parameter settings to suit the analysis.

3. If you want to use the method later, save the method under a suitable name. The method will be stored in the methods database located in your user directory.

To delete, copy, rename methods

▶ In the **File** menu, click on **Utilities** ▶ **Data Manager** and use that application to perform these tasks. For additional information, please refer to the Data Manager Help.

Useful conventions

Within the tables located in the Method Editor, entries that are in a normal font can be modified in that table. Entries that are in a bold font are for view only and cannot be modified from within that table.

Creating a New Method

To create a new method

1. In the **File** menu, click on **New** ▶ **Method**.

   The Create New Method dialog appears where you select the plasma conditions. If you wish, you can use a copy of any existing method.

2. Decide what elements you want in your method and what wavelengths you wish to use. This information will be recorded in the table in the Method Editor Spectrometer: Define Elements Page.

   ▶ To place this information directly into the Method Editor, enter the elements and wavelengths desired on the Method Editor Spectrometer: Define Elements Page. Proceed to step 3.

      -or-

   ▶ Use the Periodic Table to select the elements and their associated wavelengths. Your entries will automatically appear in the table on the Method Editor Spectrometer: Define Elements Page.

3. In the Method Editor, complete the entries on the Spectrometer pages, clicking on the side tabs to view each page.

4. Display the remaining pages of the Method Editor by clicking on the tabs at the bottom and side of the window and by completing the entries.

5.   To save the method, use **Save** ▶ **Method** in the **File** menu.

Periodic Table

You can use this table to select the elements and their associated wavelengths for your method. The elements and wavelengths that you select will automatically be entered on the Method Editor Spectrometer: Define Elements Page. You can also enter elements and wavelengths directly into the Method Editor.

The Periodic Table showing elements included in an example method prepared for a soils analysis.
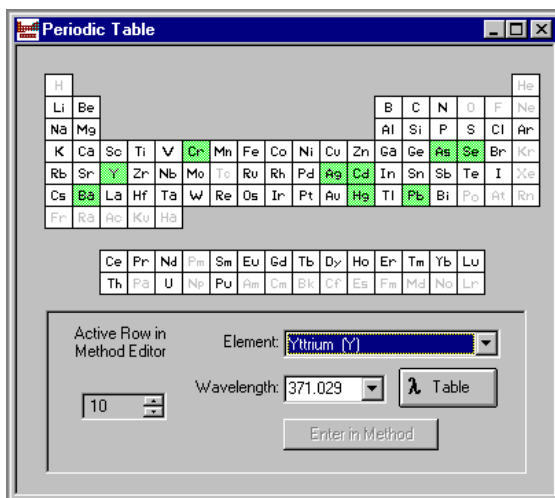
To display this table

▶ In the **Tools** menu, click on **Periodic Table**.

   - or -

▶ On the **Method Editor Spectrometer: Define Elements Page,** click on the **Periodic Table** button.

To select elements and wavelengths for your method from the periodic table

On scanning CCD ICP spectrometers, you can select any element at any wavelength. On simultaneous ICP spectrometers, elements and wavelengths that are not available on the detector are indicated by grey typeface.

1. Double-click on an element symbol. This enters the element and its default wavelength directly into the active row in the table on the Method Editor Spectrometer: Define Elements Page. The **Active Row in Method Editor** automatically increments to the next row. Continue making your selections.

   - or -

   Click on an element symbol using the right mouse button. From the list of recommended wavelengths, double-click on the desired value. (If you want to select several wavelengths for one element, click on the wavelengths and then click on **Enter in Method**.) This enters the element and the recommended wavelength directly into the method. The **Active Row in Method Editor** automatically increments to the next row. Continue making your selections.

2. When you have completed making your choices, click on the window's close box.

To select elements and wavelengths for your method from the alphabetical element list

1. Select each element and its associated wavelength from the drop down lists. This enters the element and its recommended wavelength directly into the active row in the table on the Method Editor Spectrometer: Define Elements Page. The **Active Row in Method Editor** automatically increments to the next row. Continue making your selections.

2. When you have completed making your choices, click on the window's close box.

To change the values in any row

► Change **Active Row in Method Editor** to the desired row, select the new element and wavelength, and click on **Enter in Method**.

To search for elements at a particular wavelength or to choose wavelengths not on the wavelengths list

► Click on **Wavelength Table** and complete the entries as desired.

Wavelength Table

You use this table to search for elements at a particular wavelength or to choose wavelengths not on the recommended wavelengths list that is found on the Method Periodic Table. Additional information such as intensities and BEC's are given for certain elements.

The elements and wavelengths that you have selected appear on the Method Editor Spectrometer: Define Elements Page.

On scanning CCD ICP spectrometers, you can select any element at any wavelength. On simultaneous ICP spectrometers, elements and wavelengths that are not available on the detector are indicated by grey rows.

Tip Double-click on a column header to sort the table by that parameter. For example you can sort by element, state, wavelength, BEC, signal-to-noise, intensity, or any other parameter in the column headers. Also see the table of references at the end of this section. These publications were used as sources for the Wavelength Table.

The Wavelength Table.

	Elem.	State	Wavelength (nm)	Pref. Order	BEC (mg/L)	DL mg/L (W, P, &F)	Rel Sens(B)	Sig/Noise (S & T)	Intensity (W)
1	Ag	I	328.068	1	0.26	0.0070	170000	2040.2	4200.0
2	Ag	I	338.289	2	0.43	0.0130	82000	1261.2	2200.0
3	Ag	II	243.778	3	4.00	0.1200	4200	28.2	23.0
4	Ag	II	224.874	4		0.5000	880		3.0
5	Al	I	396.153	1	0.95	0.0280	64000	268.4	2050.0
6	Al	I	308.215	2	1.52	0.0450	23000	79.2	780.0
7	Al	I	394.401	3	1.59	0.0470	32000	145.8	1050.0
8	Al	I	237.313	4	1.00	0.0300		28.8	130.0
9	Al	I	309.271	5	0.77	0.0230		141.0	1400.0
10	Al	II	167.022	6	0.05				
11	Ar	I	420.069	1				297.2	750.1
12	Ar	I	363.268	2					23.0

To display this table

▶ In the **Tools** menu, in the Method Editor, or in the Periodic Table click on **Wavelength Table.**

    - or -

▶ In the Examine Spectra/MSF window, click on 🏃 **ID**.

    - or -

▶ In the IEC Model Builder Calculate Factors page, click on **Display Wavelength Table.**

References used in the Wavelength Table

Parameter	Reference
DL mg/L (W, P & F): Radial Detection Limits (mg/L)	Winge, Peterson, and Fassel, *Applied Spectroscopy* 33, p. 206, 1979
Rel Sens (B): Relative Sensitivity	Boumans, *Line Coincidence Tables for ICP -AES*, Pergamon Press, 1980
Sig/Noise (S &T): Signal-to-Noise Ratio	Schierle and Thorne, *Spectrochimica Acta* 50B, pp. 27-50, 1995
Intensity (W): Intensity	Wohlers, *ICP Information Newsletter* 10, No.8, pp. 593-688, Jan 1985

To view the wavelengths for one element

1. Select an element.

2. Click on **All Wavelengths** or **Recommended Wavelengths** to specify what values you want to see. If you have a simultaneous ICP spectrometer, you have an **On Arrays** option as well. The On Arrays option will narrow down the search to just those wavelengths that appear on the subarray.

3. If the **Center Wavelength** entry field is not clear, double-click on this box and press **Delete** to clear it.

4. Click on **Search.**

    The specified wavelengths for that element appear in the table.

To display data for all elements

1. Select **All Elements** in the **Element to Include** entry field, or click on the **All** button. (All Elements means all elements in the wavelength table.)

2. Click on **All Wavelengths** or **Recommended Wavelengths** to specify what values you want to see. If you have a simultaneous ICP spectrometer, you have an **On Arrays** option as well. The On Arrays option will narrow down the search to just those wavelengths that appear on the subarray.

3. Type a number in the **Center Wavelength** entry field or double-click on a wavelength in the table.

4. Indicate the range in the **Wavelength Range** entry field. The default range is 0.5nm.

5. Click on **Search**.

    All wavelengths that fall in the wavelength range appear in the table.

**To copy an element and a wavelength into the method
from the Wavelength Table**

If you opened the Wavelength Table from the Method Editor, you can copy entries
directly into the method.

1. Select the element and wavelength you want to add by clicking in that row.

    Active Row in Method Editor defaults to the first blank row in the Method
    Editor Spectrometer: Define Elements page. This is where the new information
    will be added.

    ► If you wish to overwrite an existing Method Editor row, change **Active
      Row in Method Editor** as desired.

2. Click on **Enter Selected Wavelength in Method.** The information is added to
    the Method Editor.

**To copy several elements and wavelengths into the method from the
Wavelength Table**

If you opened the Wavelength Table from the Method Editor, you can copy entries
directly into the method.

1. Select the elements and wavelengths:

    ► To enter a range, click on a row and then hold down the Shift key and click
      on another row. All of the rows in between will be selected.

    ► To enter information from noncontiguous rows, click on a row, then hold
      down the Control key and click on additional rows to select them.

    ► **Active Row in Method Editor** defaults to the first blank row in the Method
      Editor Spectrometer: Define Elements page. This is where the new
      information will be added.

    ► If you wish to overwrite information in existing Method Editor rows,
      change **Active Row in Method Editor** as desired. The row that you select
      will be where the first element and wavelength in the group you selected
      in the Wavelength Table will appear in the Method Editor.

2. Click on **Enter Selected Wavelength in Method.** The information is added to
    the Method Editor.

Opening an Existing Method

A method is required for an analysis. It is used to set up the conditions under which the analysis will be run. Methods that you have saved are stored in a methods library (a large capacity structured database) so that you can use them at a later date. You can also get a copy of a method used in a particular analysis; this method is stored in a results library as part of the results data set (if you so designated on the Method Editor Options page).

To open an existing method from a methods library

1. In the **File** menu, click on **Open** ▶ **Method...**.

2. In the Open Method dialog, select the method you want, then click on **OK.** If the method you want is stored in a different methods library, click on **Browse** and select the correct library location. Return to the Open Method dialog, and select the desired method

   If the Method Editor is open, the method name appears in the title bar. The method name also appears in the Toolbar of the WinLab32 main window, next to the Method button. This is the active method, which controls your analysis. (You do not need to open the Method Editor window to open an existing method.)

3. Use this method for your analysis with no modifications if desired. To change parameters in this method, see *Modifying a Method* later in this chapter.

To get a copy of a method associated with a stored results data set

1. In the **File** menu, click on **Import from Results Library...**.

2. Select the results data set and click on **OK**.

3. When the Import from Results Library dialog appears, select the method you want to import and then, at the bottom of the dialog, select the **Method** check box and click on **OK**.

   If the Method Editor is open, the method name appears in the title bar. The method name also appears in the Toolbar of the WinLab32 main window, next to the Method button. This is the active method, which controls your analysis.

4. Use this method for your analysis with no modifications if desired. To change parameters in this method, see *Modifying a Method* in the next section. To save this method to a method library, use **Save** or **Save As...** from the **File** menu.

Modifying a Method

You may want to make modifications to an existing method.

To modify a method

1. Open a method.

2. In the **Tools** menu, click on **Method Editor**. Modify the entries as desired on the Spectrometer pages, using the side tabs to view each page.

3. Display the remaining pages of the Method Editor by clicking on the tabs at the bottom of the window and modifying the entries.

4. To save the changes, click on **Save** ▶ **Method** in the **File** menu to save the method with its current name or **Save As** ▶ **Method** in the **File** menu to save the method with a new name. The method is saved in the Method database.

Selecting Options and Entering Values

- Some parameters have fixed options denoted by an option button. You may select only one of these options for any one parameter.

Do Calib Blanks contain
internal standards?
⦿ Yes ○ No
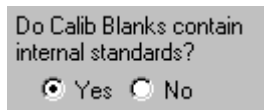
- Some options are associated with a check box. You may select as many of these as you wish. When a check box is selected, it contains a check mark.

Save with Results
☐ Spectral Data
☑ Copy of Method
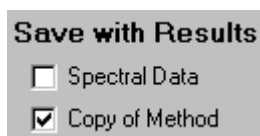
- Options for some parameters are contained in drop-down lists. Click on the arrow at the right-hand side of the drop-down list to see the choices available.

Peak Algorithm

Peak Area ▼
Peak Ht.
Peak Area
MSF
Peak Area
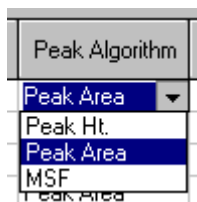
- Spin boxes are often used for numerical parameter settings. To increase or decrease the value, click on the appropriate arrowhead. You can also click on and type an acceptable value.
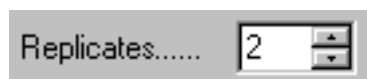
- To enter information in a text box, click on the text box, then type the necessary value or characters.

- To open another page of the Method Editor, click on the tab containing the name of the page at the bottom or side of the window.

Tabs across the bottom of the Method Editor group parameters into several main areas.

Side tabs further group parameters into individual pages.
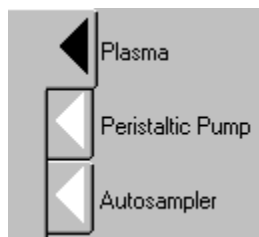
Method Editor Pop-Up Menus

The following pop-up menus contain several convenient commands that are useful as you work with the Method Editor.

Method Editor pop-up menu

To display this pop-up, click with the right mouse button in the gray background area of the Method Editor.

```
New Method
Open Method...
Save Method
Save Method As...
Save Method as Text...

Periodic Table

Print...
Print Preview...
```
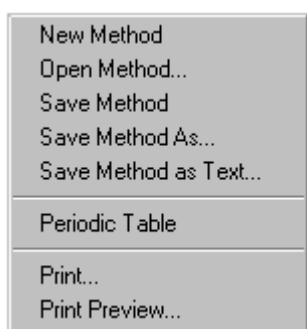
Command	Description
New Method	Lets you select the starting conditions to include in the new method.
Open Method...	Opens a stored method.
Save Method	Saves the contents of the Method Editor using the same file name.
Save Method As...	Saves the contents of the Method Editor using a new file name.
Save Method As Text...	Saves the contents of the Method Editor to a text file or comma-delimited ASCII file.
Periodic Table	Displays the Periodic Table, where you can select elements and wavelengths.
Print...	Prints the contents of the Method Editor.

Command	Description
Print Preview...	Displays the formatted Method report in a preview window. Use the Zoom In and Zoom Out buttons to enlarge or reduce the report. Click on **Close** to return to the Method Editor.

Pop-up menu for Tables with Column Fill dialogs

To display this pop-up, click with the right mouse button in a spreadsheet table. This pop-up only appears in tables that have column fill dialogs and is most useful when you are working with a block of selected entries.
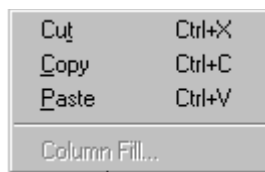
Command	Description
Cut	Removes the contents of the selected entry fields and places it onto the Windows clipboard**.**
Copy	Copies the contents of the selected entry fields and places it onto the Windows clipboard.
Paste	Inserts contents from the Windows clipboard into an entry field.
Column Fill...	Displays the Column Fill dialog for the selected column. To use the Column fill command, select a block of entry fields and then right click on the table and select this command.

Pop-up menu for individual text entries

To display this pop-up, double-click in an entry field to select the contents, then click with the right mouse button. In some text boxes within the Method Editor, a right click will display this pop-up.
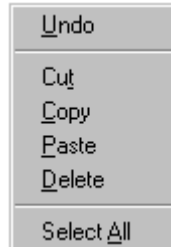
Command	Description
Undo	Reverses the last command.
Cut	Removes the contents of the selected entry field and places it onto the Windows clipboard.
Copy	Copies the contents of the selected entry field and places it onto the Windows clipboard.
Paste	Inserts contents from the Windows clipboard into an entry field.
Delete	Removes the contents of the selected entry field.
Select All	Selects all of the text in an entry field.

Checking a Method

Before you use a method, you can let the software review all of the entries in the Method Editor pages using the Check Method dialog. This dialog alerts you to any existing problems or inconsistencies in the method that must be corrected before the method can be used for an analysis. When you save a method, the method will be checked automatically.

▶ To display the Check Method message dialog, on the **Edit** menu, click on **Check Method**.

The Check Method dialog appears.

▶ To print this information, click on **Print** in the Check Method dialog.

Saving a Method

You must save a method if you want to use it later. When you save a method, a copy of the method is placed in the Methods database. If you selected the Copy of Method option in the Method Editor Options page, a copy of the active method that was used to obtain the results of your analyses is stored with the results data set.

Note When you save a method, the software reviews all of the entries automatically. If it finds any problems or inconsistencies, the Check Method message dialog is displayed to inform you of the error.

To save a method with its current name

▶ In the **File** menu, click on **Save** ▶ **Method**.

To save a method with a new name

1. In the **File** menu, click on **Save As** ▶ **Method**.

2. Type a file name up to 25 characters in length, and click on **OK**.

| Note | To save a method as a text file for making notations or printing, see *Printing a Method*. |

Printing a Method

You can print a method summary, which contains all of the parameters in the method. You can also save a method summary in a text file, then open it in a text editor such as Notepad and make notations or print selected sections.

To print a method summary

1. Click on the Method Editor window to make it active. Note that the title bar of an active window is a different color from other windows.

2. In the **File** menu, click on **Print** ▶ **Active Window**.

3. In the Print dialog that appears, check that the correct printer is shown. If you need to select another printer or change other setup options such as the paper size, click on **Setup**.

4. Click on **OK** to start printing.

To save a method summary in a text file

1. Click on the Method Editor window to make it active.

2. In the **File** menu, click on **Save As Text** ▶ **Method**.

3. Type a name for the file, and click on **OK**.

    You can now open the file in a text editor such as Notepad and make notations or print selected sections.

Method Editor: Spectrometer Pages Overview
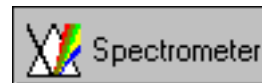
You use these pages to define the parameters that control how the hardware is set up and how the system collects data.

To display the Spectrometer pages

▶ Click on the **Spectrometer** tab at the bottom of the Method Editor window.

Spectrometer Pages

▶ To set the following parameters, click on the appropriate tab on the right-hand side of the window.

Define Elements Page

Settings Page

Spectral Windows Page

Method Editor Spectrometer Pages: Define Elements Page

You use this page to define the symbol, wavelength, name, and function of an element. In the Method Editor, an "element" represents a combination of parameters associated with a measurement at a particular wavelength. If you are measuring an element at more than one wavelength, you must repeat the element on this page for each separate wavelength.

Detail of the Define Elements page showing entries included in an example method prepared for a soils analysis.

Define Elements

Method Description: RCRA Elements in Soils +Y I.S. (mg/kg)

	Symbol	Wavelength (nm)	Name	Function
1	Ag	328.068	Ag 328.068	Analyte
2	As	188.979	As 188.979	Analyte
3	Ba	233.527	Ba 233.527	Analyte
4	Cd	214.440	Cd 214.440	Analyte
5	Cr	267.716	Cr 267.716	Analyte
6	Cr	283.563	Cr 283.563	Analyte
7	Hg	194.168	Hg 194.168	Analyte
8	Pb	220.353	Pb 220.353	Analyte
9	Se	196.026	Se 196.026	Analyte

Elements and wavelengths can be selected by clicking on one of the buttons to the right..........

Periodic Table

Wavelength Table

Method Description

This short description of your method will appear next to your method name in the Open Method dialog. It will also appear in the Method Description field of the Analysis Header displayed in the Results Display and Printed Log.

▶ Type up to 80 characters.

Symbol

This is the symbol of the element to be measured.

▶ To change or add an element, type the letters for the symbol in the entry field, or click on Periodic Table. When the Periodic Table appears, select the desired elements.

Wavelength

This is the wavelength in nm that the Spectrometer will use to measure the associated element.

To add or change a wavelength:

▶ Type an element symbol in the **Symbol** field to get the default wavelength for an element.

- or -

▶ Type the value for the wavelength in the entry field.

- or -

▶ Click on **Periodic Table.** When the Periodic Table appears, select the desired wavelength.

Name

This is the name that describes the element. The default entry is the element symbol followed by the wavelength. No two elements in a method can have the same name.

▶ If you want to change the default name, type up to 20 characters, including spaces and punctuation. For example, if you are determining analytes using different plasma viewing modes or different plasma conditions, you may want to add information to the default name such as "Al 396.152–Axial" or "Ca 317.933—0.8 neb").

Function

This is the role of the element in the analysis.

Analyte
The element's concentration will be determined based on a valid calibration.

Internal standard (Int. Std.)
The element will act as an internal standard.

▶ Click on the text and select Analyte or Int. Std. from the drop-down list.

Periodic Table

▶ Click on this button to display the Periodic Table, from which you can select elements and their associated wavelengths for your method.

Wavelength Table

▶ Click on this button to display the Wavelength Table, where you can search for elements at a particular wavelength or choose wavelengths not in the recommended wavelength list.

Method Editor Spectrometer Pages: Settings Page

You use this page to set spectrometer parameters that control how the elements will be measured.

Detail of the Settings page. This shows entries included in an example method prepared for a soils analysis.

Spectral

Purge Gas Flow
This is the spectrometer purge gas flow rate. The time taken to purge air from the spectrometer depends on various factors, including how long the instrument has been in a low purge state.

Normal
A normal purge is used during routine operation.

High
The high purge is normally used during the analysis for wavelengths below 190 nm. A high purge performed before an analysis clears the system of air,

which contains moisture and oxygen, before analyzing elements below 190 nm. See the *Spectrometer Control Window* in Chapter 6, *Hardware Control Windows*.

Spectral Profiling

This option is only available on simultaneous ICP spectrometers. If Spectral Profiling is selected, the slit image is scanned, which yields greater data density and shows smoother peak profiles. Using spectral profiling takes four times as long per measurement as when profiling is not being used. Spectral profiling is most useful for identifying spectral interferences and collecting data when the wavelength requires adjustment. In some circumstances, it is useful for analyses using Multicomponent Spectral Fitting.

Resolution

This option is only available on simultaneous ICP spectrometers.

Fixed
The same resolution is used for all elements. There are three settings: High, Normal, and Low. The selection you make sets the slit width and determines how the detector will be read.

▶ Select **Low** for slightly better detection limits and analyte precision. This setting gives the poorest resolution. The slit width is at its widest and each pixel in the detector subarray is read as a whole. This gives the highest sensitivity per element, but poor freedom from spectral interferences.

▶ Select **Normal** to provide performance that has the optimal combination of sensitivity and freedom from spectral interferences for most applications. The slit width provides the spectral bandpass equal to one pixel width and each pixel in the detector subarray is read as a whole.

▶ Select **High** to provide the greatest freedom from spectral interferences, but lower sensitivity than Normal or Low. This setting gives the greatest resolution, but the lowest intensities. The slit width is at its narrowest and each pixel in the detector subarray is read as two separate half-pixels.

Variable
Different resolutions are used for each analyte.

▶ Click on **Set...** and in the Set Element Resolution dialog that appears, select the resolution for each element.

Time

This is the time in seconds that the signal is measured.

▶ For automatic integration, click on **Auto**. The software automatically sets the integration time individually for each analyte based on intensity. Select the minimum and maximum times that you want to use for all analytes.

▶ For manual control, click on **Set...** next to **Manual.** In the Set Manual Integration dialog that appears, select the integration and read times for each analyte. (Integration time is the time period in seconds in which the detector collects photons before transferring the charge and reading out the signal. The read time is the total measurement time in seconds per replicate.)

Delay Time

This is the number of seconds that elapse before the instrument begins to measure the signal.

Valid entry range: 0 - 999.

Replicates

This is the number of read cycles to be performed on each sample and calibration solution.

Valid entry range: 1 - 999.

If you are using the Optima 2000/2100, you can Measure by: Element or Replicate.

- Element – Selecting Measure by Element tells the spectrometer to stay at the wavelength of the element for the number of Replicates selected. For example, if you selected six (6) replicates for Cu and Pb it will measure Cu six times before it changes wavelength to measure Pb six times.

- Replicate – Selecting Measure by Replicate tells the spectrometer to measure each element once then repeat for the number of replicates. For example, if you selected six (6) replicates for Cu and Pb, it will measure

Cu once then change the wavelength and measure Pb once for replicate one, then change the wavelength and measure Cu once then change the wavelength to measure Pb once for replicate two until it completes the cycle six times.

Method Editor Spectrometer Pages: Settings Page - FIAS Technique

You use this page to set spectrometer parameters that control how the elements will be measured.

Settings

Spectral

Purge Gas Flow . . . . ⊙ Normal   ○ High

Read Parameters

Integration Time: 0.010   BOC Time: 2

	Element	Transient Read Delay	Transient Read Time
1	Cu 327.393	0.0	15.0
2	Pb 220.353	0.0	15.0

Replicates: 1

Spectral Settings

Purge Gas Flow: This is the spectrometer purge gas flow rate. The time taken to purge air from the spectrometer depends on various factors, including how long the instrument has been in a low purge state.

Normal: A normal purge is used during routine operation when all wavelengths are greater than 190 nm.

High: The high purge is normally used during the analysis for wavelengths below 190 nm. A high purge performed before an analysis clears the system of

air, which contains moisture and oxygen, before analyzing elements below 190 nm. See the Spectrometer Control window.

Read Parameters

Integration Time: This is the integration time used by the spectrometer to collect each spectrum of data. Enter an integration time between 0.001 and 50 seconds.

BOC Time: The amount of time to measure the baseline just before the beginning of the read step. Enter a time between 0 and 5 seocnds

Element: The name of the analyte entered on the Spectrometer page.

Transient Read Delay: For each analyte this is the amount of time to wait after the read step begins before processing the signal. Double clicking this column heading displays a column fill dialog.

Transient Read Time: For each analyte this is the amount of time to be spent processing the signal.

Replicates: The number of measurements that should be made of each analytical line.

Transient Read Delay Column Fill

Enter the periodof time that exists between the start of time-based data and the point where peak height/area processing begins. (Enter a value between 0 and 120 seconds to wait after the read step begins before processing the signal.)
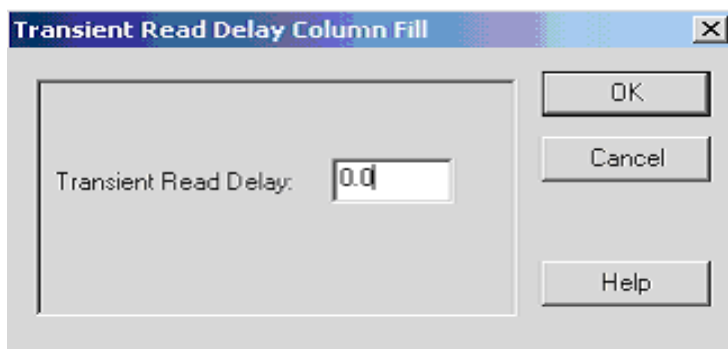
Transient Read Time Column Fill

Enter a period of time-based data on which peak height/area processing is done. (Enter a value between 0.1 and 120 seconds to be spent processing the signal.)
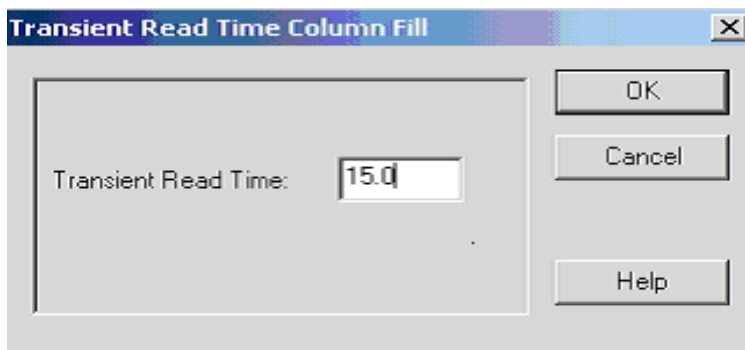
Method Editor Spectrometer Pages: Set Element Resolution Dialog

Use this dialog to select different resolutions for each element. Different resolution settings are only available on simultaneous ICP spectrometers.

To display this dialog

► Click on **Set...** next to **Resolution** on the **Settings** page.

Using this dialog

► Select the resolution you want for each analyte from the drop-down list.

F'n (Function)

This view-only column shows parameters that describe the kind of action the instrument will take for this element.

- A = Analyte

- IS = Internal Standard

To change the parameter, use the Function entry field on the Spectrometer: Define Elements page.

Element

This view-only column shows all names of the elements selected for the current analysis. To change the element, click on the **Periodic Table** button on the Spectrometer: Define Elements page. In the Periodic Table that appears, select the elements and wavelengths that are to be added.

Resolution

This is the resolution for the element.

There are three settings: High, Normal, and Low. The selection you make sets the slit width, which affects how the detector will be read.

> ► Select **Low** to provide the lowest resolution. The slit width is at its widest and each pixel in the detector subarray is read as a whole. This gives the highest sensitivity per element, but poor freedom from spectral interferences.

> ► Select **Normal** to provide resolution that has the optimal combination of sensitivity and freedom from spectral interferences for most applications. The slit width provides the spectral bandpass equal to one pixel width and each pixel in the detector subarray is read as a whole.

> ► Select **High** to provide the greatest freedom from spectral interferences, but lower sensitivity than Normal or Low. The slit width is at its narrowest and each pixel in the detector subarray is read as two separate half-pixels.

Note   Using multiple resolution settings in your method will cause measurements to be made sequentially in groups as defined by resolution type during the analysis.

Method Editor Spectrometer Pages: Set Manual Integration Dialog

Use this dialog to select different integration and read times for each element.

To display this dialog

▶ Click on Set... next to Manual on the Settings page, Read Time parameter.

Using this dialog

▶ Type the integration times and read times that you want to specify.

Notes **#1**: The read time divided by the integration time must be a whole number.

#2: On simultaneous ICP spectrometers, using multiple integration and read times in your method will cause measurements to be made sequentially in groups as defined by integration and read time during the analysis.

F'n (Function)

This view-only column shows the kind of action the instrument will take for this element.

- A = Analyte
- IS = Internal Standard

To change the parameter, use the **Function** entry field on the Spectrometer: Define Elements page.

Element

This view-only column shows all the elements selected for the current analysis. To change the element, click on the **Periodic Table** button on the Spectrometer: Define Elements page. In the Periodic Table that appears, select the elements and wavelengths desired.

Integration Time

This is the time period in seconds in which the detector collects photons before transferring the charge and reading out the signal. The maximum integration time is 8 seconds for a scanning CCD spectrometer and 50 seconds for a simultaneous spectrometer.

> **Note** This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Read Time

This is the total measurement time in seconds per replicate. Read time divided by integration time gives you the number of integrations performed per replicate.

> **Note** This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Method Editor Spectrometer Pages: Spectral Windows Page

You can use this page to set the survey window and auto integration window. The survey window is the wavelength range in which the plasma emission data is collected. The auto integration window is the wavelength range in which the integration time is calculated, based on the signal of highest intensity.

By default, the survey window and auto integration window are set to the same wavelength range. On simultaneous ICP spectrometers, this range is determined by the wavelength range of the subarray on which the wavelength of interest falls. On scanning CCD ICP spectrometers, the wavelengths are all directed to a single detector array, and the software sets the wavelength range to default values.

On either type of spectrometer, you can adjust the wavelength range for the survey window or the auto integration window. In some cases, it may be advantageous to set the auto integration window to be smaller than the survey window. For example, if your interferent signal is higher than the analyte signal, the auto integration time may be shorter than appropriate for your analysis. This is because the software calculates the auto integration based on the highest signal, which, in this example, would be the interferent. Therefore, you may want to limit the auto integration window to exclude the interferent, so the software will calculate a longer integration time.

Detail of the Spectral Windows page showing entries included in an example method prepared for a soils analysis.

Spectral Windows

	Element	Wavelength (nm)	Survey Lower	Survey Upper	Auto Lower	Auto Upper
1	Ag 328.068	328.068	327.998	328.148	327.998	328.148
2	As 188.979	188.979	188.939	189.025	188.939	189.025
3	Ba 233.527	233.527	233.423	233.588	233.423	233.588
4	Cd 214.440	214.440	214.236	214.491	214.236	214.491
5	Cr 267.716	267.716	267.532	267.786	267.532	267.786
6	Cr 283.563	283.563	283.498	283.628	283.498	283.628
7	Hg 194.168	194.168	194.126	194.215	194.126	194.215
8	Pb 220.353	220.353	220.304	220.405	220.304	220.405
9	Se 196.026	196.026	195.984	196.074	195.984	196.074
10	Y 371.029	371.029	370.954	371.125	370.954	371.125
11						
12						
13						
14						

Element and Wavelength

These view-only columns show the elements and wavelengths selected for the current analysis. To change the element, click on the **Periodic Table** button on the Spectrometer: Define Elements page. In the Periodic Table that appears, select the elements and wavelengths desired.

Survey Lower and Survey Upper

This sets the wavelength range in which plasma emission data is collected from the lowest to highest wavelength. The survey window must be set equal to or greater than the auto integration window.

Auto Lower and Auto Upper

This sets the wavelength range in which the integration time for the analyte is calculated.

When automatic integration is used, the software automatically sets the read time individually for each analyte based on the signals of highest intensity. However, for some analyses, you may want to select a smaller measurement range to exclude a strong interfering peak when the integration time is being determined. This will give you improved signal-to-noise characteristics for the analyte of interest.

Method Editor: Sampler Pages Overview

You use these pages to define the parameters that control the plasma, peristaltic pump and autosampler wash.

To display the Sampler pages

▶ Click on the Sampler tab at the bottom of the Method Editor window.

Sampler Pages

▶ To set the following parameters, click on the appropriate tab on the right-hand side of the window.

• Plasma Page

• Peristaltic Pump Page

• Autosampler Wash Page

Method Editor Sampler Pages: Plasma Page

You use this page to set the plasma gas flows, RF power, viewing distance, and source equilibration delay. You also set the plasma aerosol type, and the nebulizer startup conditions. These can be the same for all elements or different for one or all elements.

Detail of the Plasma page showing entries included in an example method prepared for a soils analysis.

Plasma

Source Equilibration Delay (sec) . . . . . . [15 ⇅]

Plasma Conditions: ⊙ Same For All Elements   ○ Vary by Element

Plasma Aerosol Type: ⊙ Wet   ○ Dry

Nebulizer Start-up   ⊙ Instant   ○ Gradual

	F'n	Element	Plasma (L/min)	Aux (L/min)	Neb (L/min)	Power (watts)	View Dist	Plasma View
	--	**All**	15	0.5	0.60	1400	15.0	Axial
1	A	Ag 328.068	15	0.5	0.80	1300	15.0	Axial
2	A	As 188.979	15	0.5	0.80	1300	15.0	Axial
3	A	Ba 233.527	15	0.5	0.80	1300	15.0	Axial
4	A	Cd 214.440	15	0.5	0.80	1300	15.0	Axial
5	A	Cr 267.716	15	0.5	0.60	1400	15.0	Axial
6	A	Cr 283.563	15	0.5	0.80	1300	15.0	Axial
7	A	Hg 194.168	15	0.5	0.80	1300	15.0	Axial
8	A	Pb 220.353	15	0.5	0.80	1300	15.0	Axial
9	A	Se 196.026	15	0.5	0.80	1300	15.0	Axial

Source Equilibration Delay

This is a delay in seconds, which can be used to allow the plasma to stabilize after changes have been made to the plasma (source) conditions.

Plasma Conditions

You can assign the same gas flows, RF power, and viewing distance values to all elements in the method or you can vary these values for each element individually. If you have a dual-view instrument, you can also assign plasma view (axial or radial) in this way.

Same for All Elements
Select this option to use the same gas flows, RF power levels, viewing distances and plasma view for all elements.

Vary by Element
Select this option to vary the parameters for each element. Enter the values you want in the table.

Plasma Aerosol Type

You can select the type of aerosol to use for the analysis.

Wet
Use when the sample aerosol is produced by nebulizing a solution using a pneumatic nebulizer, such as the cross-flow nebulizer or the GemCone nebulizer.

Dry
Use when the sample aerosol is desolvated, i.e., by an ultrasonic nebulizer or is produced directly from a solid sample, e.g., using laser sampling.

Nebulizer Startup Conditions

You can choose to have the nebulizer startup instantly or gradually.

Instant
This option turns the nebulizer argon flow on quickly after ignition. Use this for most cases.

Gradual
This option turns the nebulizer argon flow on slowly after ignition. Use this for igniting while aspirating high solids sample matrices or when the first procedure does not work.

F'n (Function)

This view-only column shows the kind of action the instrument will take for this element.

- A = Analyte

- IS = Internal Standard

To change the parameter, use the **Function** entry field on the Spectrometer: Define Elements page.

Element

This view-only column shows all the elements selected for the current analysis. To change the element, click on the **Periodic Table** button on the Spectrometer:

Define Elements page. In the Periodic Table that appears, select the elements and wavelengths desired.

Note   The following parameters have Column Fill dialogs that can be accessed by double-clicking on the parameter's column header.

Plasma

This is the argon flow rate for the ICP torch plasma gas (also known as coolant or outer flow).

Valid entry range: 0 - 20 L/min. However, most torch designs will require a minimum flow rate of about 10 L/min to achieve a stable plasma.

Aux

This is the argon flow rate for the ICP torch auxiliary gas.

Valid entry range: 0 - 2.0 L/min.

Neb

This is the argon flow rate for the nebulizer or sample carrier gas.

Valid entry range: 0 - 2.0 L/min. However, many nebulizer designs will allow a maximum gas flow rate of less than 2.0 L/min. See the information provided with your nebulizer to determine its maximum gas flow rate.

Power

This is the amount of RF power delivered to the plasma discharge.

Valid entry range: 750 - 1500 watts.

View Dist

This is the nominal position within the plasma for the light that is measured by the spectrometer. For radially viewed plasma, it is the distance from the load coil. For axially viewed plasma, the 15-mm position represents the center of the plasma discharge.

Plasma View

If you have a dual-view instrument, this column is present. Plasma view refers to the viewing position of the plasma torch, which can be set to radial or axial independently for each analyte.

Radial
Light emitted from the side of the plasma passes through the torch slit and is directed to the spectrometer optics.

Axial
Light emitted along the axis of the plasma is directed to the spectrometer optics. The observation zone of the plasma is circular and the spectrometer views a column of light from the central channel of the plasma.
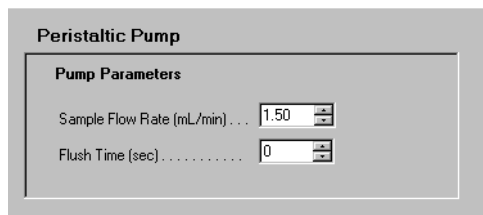
Attenuated Radial or Attenuated Axial
These settings are available only for scanning CCD ICP spectrometers. Use these settings when lower intensities are desired, for example, for samples of high concentration that would normally saturate the detector and for sensitive wavelengths, typically in axial mode.

Method Editor Sampler Pages: Peristaltic Pump

You use this page to select the rate of sample flow. You also select whether or not a sample flush time should be added to the analysis delay time.

Detail of the Peristaltic Pump page.

Peristaltic Pump

Pump Parameters

Sample Flow Rate (mL/min) . . . 1.50

Flush Time (sec) 0

Sample Flow Rate

Select the rate in mL/min that the sample solution will be pumped into the plasma during an analysis. As you change this value, the pump speed that appears will be changed appropriately. The value range and ratio of pump rate to pump speed depend on both the pump tubing and the pump model selected.

Flush Time

This is the time (if any) between the wash time and the read delay, when the pump will aspirate sample solution at the Sample Flush Rate.

Valid entry range: 0 - 999. This is normally set to equal the time it takes for the sample to travel from the sample vessel to the nebulizer (while usually being pumped at a higher rate).

Method Editor Sampler Pages: Autosampler Page

You use this page to schedule when and for how long the sample delivery tubing will be washed, and to set the wash pump rate.

Note You specify the type of autosampler that you are using when you install the software. If needed, you can change this setting using the Reconfigure utility: in Windows select Start, Programs, PerkinElmer WinLab32, Reconfigure and use the wizard to change the autosampler. To set the tray type, see *Autosampler Configuration dialog* in Chapter 11, *Menus and Toolbar*. If you have an Optima 3000 Family instrument, you set the pump type from the Options menu, Configure Pump dialog.

Detail of the Autosampler page.

Wash

A wash step is often used during an automated analysis to clean off the autosampler probe and rinse out the sample delivery tubing.

Wash Frequency

Never
Select this option to be sure that no washes will be performed.

Between Samples
Select this option to perform a wash between each sample.

Only after samples whose concentrations exceed limits
Select this option to specify a wash for any analyte when its concentration exceeds a specified limit.

After every sample + extra time if sample concentrations exceed limits
Select this option to increase the length of a wash for any analyte when its concentration exceeds a specified limit.

Rate

This is the flow rate in mL/min that will be used during the Wash Time.

Normal Time

This is the time in seconds that the autosampler probe is immersed in the wash solution. The value range and ratio of pump rate to pump speed depend on both the pump tubing and the pump model selected.

Wash Location

This is the location of the wash solution in the autosampler tray, usually zero (0).

Extra Time

This is the amount of extra wash time in seconds that will be added to a wash. Enter the amount of wash time directly or if you wish, you can increase the wash time based on the concentration of a sample.

Method Editor Sampler Pages: Extra Wash Concentration Limits dialog

Use this dialog to increase the length of a wash for any analyte when its concentration exceeds a specified limit. In automated analyses, this is useful if you are concerned that a particular analyte concentration may not be rinsed fully from the autosampler probe unless the wash is extended.

To display this dialog

▶ In the **Autosampler** page, select one of the following options: **Only after samples whose concentrations exceed limits** or **After every sample + extra time**, and then click on the **Set...** button.

Using this dialog

▶ Type the concentration limits you want for each analyte. The units shown are calibration units. These are selected on the Calibration Units and Standard Concentrations page.

The total wash time will be the Normal Time + Extra Time selected on the Autosampler Wash page.

When the concentration of any analyte exceeds the entered value, the wash time will be increased by the amount of time you selected.

| Note | The Concentrations parameter has a column fill dialog that can be accessed by double-clicking on the column header. |

Method Editor Sampler Pages: Autosampler Page - FIAS Technique

You use this page to tell the system when to wash the autosampler sample tube and sample probe, and for how long. You can make the wash procedure dependent on the concentration of the analyte in the solution.

You also select the autosampler location of the emission setup solution when you are using the flame emission technique.

You select the autosampler tray in the **Options menu > Autosampler**.

Frequency

Never: No wash steps during the analysis. When this option is selected, the entry fields for the options below are inactive.

Between Samples: The system washes the sample tube and probe after every solution that it analyzes. Select a suitable option for Normal Cycles to tell the system how long to wash the sampling system.

Only after solutions exceeding limit: The system washes the sample tube and probe after it has analyzed a solution where the measured concentration or absorbance exceeds the value you select for Limit.

After all solutions + extra time if solution exceeds limit: The system washes the sample tube and probe, after it has analyzed each solution, for the time you select for Normal Time or the number of Normal Cycles. If the measured concentration or absorbance of a solution exceeds the value you select for Limit, the system will wash the sample tube and probe after analyzing this solution for an additional period of time that you select for Extra Time or Extra Cycles.

Wash Location: The sample tray location of the wash solution.

Normal Cycles: This parameter appears only with flow-injection techniques. The system pumps wash solution through the system using the FIAS program steps that you select below, for the number of times you select here.

Extra Cycles: Enter a value here if you want to set extra wash time after solutions where the measured concentration or absorbance exceeds the value you select for Limit. The system pumps wash solution through the system using the FIAS program steps that you select below, for the number of times you select here in addition to the number of times you select for Normal Cycles. Use this option together with the option above: After all solutions + extra time if solution exceeds limit.

Use FIAS steps: Select the first and last steps in the FIAS program that you want the system to use for the wash cycle. You can set step 0, the Prefill step, as the first step in the wash cycle.

FIAS Page

Use the FIAS page to enter the event versus time program that controls the flow injection system.

Operation

Sample Volume: Enter the volume, in microliters, of the sample loop you intend to use. The settings in the Recommended Conditions window are usually based on a volume of 500 µL.

Flow Injection Program

Step: This column shows the steps in the FIAS program. Every program contains a Prefill step and between one and eight additional steps.

Use the Prefill step to ensure that the sample tube that leads to the FIAS-valve is filled with sample solution. This step is used only for the first replicate. The other program steps are performed for each replicate.

Time (sec): This is the duration of the program step in seconds.

The duration of the steps before the read step must be at least as long as the BOC time that you select on the Spectrometer page.

Pump 1 and Pump 2 Speed (rpm): This is the speed of the pump during the program step. To switch off the pump during a program step, enter 0.

Valve Fill/Inject

Fill -- The sample loop is filled with sample.

Inject -- The sample in the sample loop is injected into the carrier stream.

Remotes: Remotes 2 through 10 are relays that can be used to control accessories that are connected to the Instrument Interface on the rear of the instrument. Remote 1 is reserved for triggering the signal measurement on the spectrometer. Select the box in the Remote column for the step in which the accessory should be triggered. The system activates the relay for the duration of the step.

Default: Use this button to enter the recommended FIAS program, for the element that you have selected, into the Method Editor.

Read Step: Select the FIAS program step where you want to start the signal measurement. Usually, this will be the inject step. The signal measurement starts at the beginning of the step and continues for the Read Time selected on the Spectrometer page.

Steps to Repeat: Select a range of FIAS program steps that you want to be repeated, for example, with the amalgam technique to make multiple injections.

If you do not want to repeat any steps, enter 0 for Number of Repeats.

Number of Repeats: This is the number of times the system will repeat the steps selected for Steps to Repeat. If you do not want to repeat any steps, enter 0.

Method Editor: Process Pages Overview ![Process]

You use the entries on the Process pages to control the data processing part of the analysis. Data processing involves converting spectral data into intensity data for each element and correcting for spectral overlaps.

To display the Process pages

▶ Click on the **Process** tab on the bottom of the Method Editor window.

Process pages

▶ Click on the appropriate tab on the right-hand side of the window to select the pages.

• Spectral Peak Processing Page

• Spectral Corrections Page

• Assign Internal Standards Page

Method Editor Process: Spectral Peak Processing Page

You use this page to select how the intensity peaks for your analytes will be measured. If you wish, you can set a different peak algorithm for each analyte.

Detail of the Spectral Peak Processing page showing entries included in an example method prepared for a soils analysis.

Spectral Peak Processing

	F'n	Element	Peak Algorithm	Points per Peak
1	A	Ag 328.068	Peak Area ▼	1
2	A	As 188.979	Peak Area	1
3	A	Ba 233.527	Peak Area	1
4	A	Cd 214.440	Peak Area	1
5	A	Cr 267.716	Peak Area	1
6	A	Cr 283.563	Peak Area	1
7	A	Hg 194.168	Peak Area	1
8	A	Pb 220.353	Peak Area	1
9	A	Se 196.026	Peak Area	1
10	IS	Y 371.029	Peak Area	3

F'n (Function)

This view-only column shows the action that the instrument will take for this element.

- A = Analyte

- IS = Internal Standard,

To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Element

This view-only column shows all the elements selected for the current analysis. To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Peak Algorithm

Select the mathematical algorithm that will be used to process the analytical data on this item.

Peak Area

The system uses the sum of the intensities recorded by the pixels on the detector used for the read. The number of data points used is defined by the Points Per Peak entries on this page. Since the total number of data points for each spectrum increases by a factor of four when spectral profiling is turned on, you have to use four times as many points/peak to measure the same area under the peak if spectral profiling is on than you would if spectral profiling were off. You also must also quadruple the amount of time allowed for this to occur.
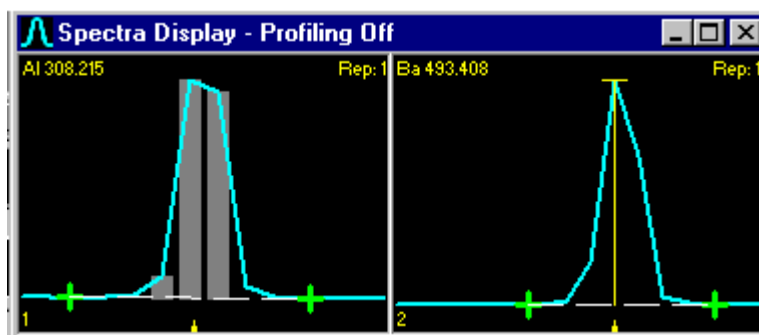
Peak Height

If you are not using spectral profiling, the number that you enter in Points Per Peak defines the region that the software will search in order to locate the peak maximum. For example, if you entered 5 in Points Per Peak, the software will search 5 points around the calibrated center. When the software finds the point that produces the highest intensity, that point will be defined as the Peak Height.

If you are using spectral profiling, the peak height is determined in different ways depending on the number of points chosen:
1 point: uses theoretical wavelength or wavelength calibration point
2 points: uses highest of the 2
3 or 4 points: 3 point parabolic fit
5 or more points: 5 point cubic fit

The total number of data points for each spectrum increases by a factor of four when you use spectral profiling. Therefore, if spectral profiling is on, you have to use four times as many points per peak to define the same search region if spectral profiling is on than you would if spectral profiling were off. You also must also quadruple the amount of time allowed for this to occur.

Comparison of Peak Area and Peak Height algorithms with profiling off.

Comparison of Peak Area and Peak Height algorithms with profiling on.
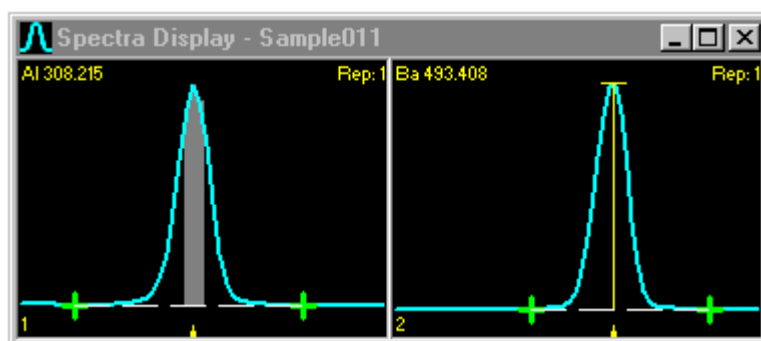
MSF (Multicomponent Spectral Fitting)

The system uses a multiple linear regression to fit the spectra from the analysis to predetermined models. If there is no MSF model stored for the element being read, a warning message appears during the analysis. Because Points per Peak is not relevant when MSF processing is used, the Points Per Peak column is dimmed.

Spectral profiling is not necessary for MSF. However, there may be certain instances where spectral profiling may be useful as a means of improving results for elements that have spectral interferences.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Points Per Peak

This is the amount of data to be captured depending on the type of algorithm that you selected:

- If you selected Peak Area, the value in the Points Per Peak entry is the number of data points to be summed. On simultaneous ICP spectrometers, this equals the number of subarray pixels that will be summed when spectral profiling is off. If spectral profiling is on, you need to use about four times as many points to cover the same spectral area.

- If you selected Peak Height, this entry is the width of the search window used for the peak height determination. On simultaneous ICP spectrometers, you need to use about four times as many points to cover the same spectral area when spectral profiling is on.

- If you selected MSF, this entry is dimmed.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Method Editor Process Pages: Spectral Corrections Page

You use this page to select the corrections to be made for spectral overlaps and shifting background emission. Two kinds of overlap correction are possible, Interelement Correction and Multicomponent Spectral Fitting.

Detail of the Spectral Corrections page showing entries included in an example method prepared for a soils analysis.

Spectral Corrections

	F'n	Element	Overlap Correction	Backgrnd Correction	BGC1	BGC2
1	A	Ag 328.068	None	1- Point	-0.030	0.000
2	A	As 188.979	None	2- Point	-0.012	0.017
3	A	Ba 233.527	None	2- Point	-0.021	0.021
4	A	Cd 214.440	None	2- Point	-0.020	0.020
5	A	Cr 267.716	None	2- Point	-0.025	0.025
6	A	Cr 283.563	None	2- Point	-0.026	0.026
7	A	Hg 194.168	None	2- Point	-0.018	0.018
8	A	Pb 220.353	None	2- Point	-0.020	0.020
9	A	Se 196.026	None	1- Point	-0.018	0.000

IEC Model: (None)   MSF Model: (None)

F'n (Function)

This view-only column shows the action that the instrument will take for this element.

- A = Analyte

- IS = Internal Standard

To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Element

This view-only column shows all the elements selected for the current analysis. To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Overlap Correction

Select a mathematical method that will be used to process the analytical data on this item for spectral overlaps. It is possible to use one, two, or all three settings in a method, although each individual element can only use one setting. The selections that appear for Overlap Correction depend upon the peak algorithm that you selected on the Method Editor Process: Spectral Peak Processing page, as shown below.

If the Peak Algorithm is:	Then, Background Correction is:
Peak area	None or IEC
Peak height	None
MSF	MSF

None
No overlap correction will be made for this item.

IEC (Interelement Correction)
Interelement correction is a technique of correcting for spectral interferences that uses mathematical correction factors to reallocate element concentrations. See *Interelement Correction Model Builder* in Chapter 8, *Interferences & Interelement Correction* for more information. This option is only available if Peak Area is selected as Peak Algorithm on the Process: Peak Algorithm, Points/Peak page. If IEC is selected for any element, an IEC Table name must also be selected on this page.

MSF (Multicomponent Spectral Fitting)
A multiple linear regression is done to fit the spectra from the analysis to predetermined models. It can be used for spectral interference correction or for improving detection limits. See the *Examine Spectra/MSF* window in Chapter 9, *Using the Examine/MSF Window* for more information. This option is automatically selected if MSF was selected as Peak Algorithm on the Process: Peak Algorithm, Points/Peak page. If MSF is selected for any element, an MSF Table name must also be selected on this page.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Background Correction

Select a method that will be used to correct for background changes for this element.

None
No background correction will be made for this element.

1-point
One-point background correction will be made for this element (using BGC1).

2-point
Two-point background correction will be made for this element (using BGC1 and BGC2).

MSF (Multicomponent Spectral Fitting)
A multiple linear regression is done to fit the spectra from the analysis to predetermined models. See *Examine Spectra/MSF* window in Chapter 9, *Using the Examine/MSF Window* for more information. This option is automatically selected if MSF was selected as Peak Algorithm on the Process: Peak Algorithm, Points/Peak page.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

BGC1 (Background Correction Point 1)

This entry is dimmed unless 1-point or 2-point background correction is selected. It sets the offset from the wavelength selected on the Spectrometer: Define Elements page to be used for the first background point.

▶ Type a number for the offset. BGC1 can be on either the left or right side of the peak wavelength. If you wish to enter a negative offset to specify a point on the left side of the peak wavelength, type a minus sign (-) and then a number.

- or -

▶ You can select background points in the Examine Spectra/MSF window. For more information, see *Using the Background Correction Point Option* in Chapter 9, *Using the Examine/MSF Window*.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

BGC2 (Background Correction Point 2)

This entry is dimmed unless 2-point background correction is selected. It sets the offset from the wavelength selected on the Spectrometer: Define Elements page to be used for the second background point.

▶ Type a number for the offset. BGC2 can be on either the left or right side of the peak wavelength. If you wish to enter a negative offset to specify a point on the left side of the peak wavelength, type a minus sign (-) and then a number.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Method Editor Process Pages: Assign Internal Standards Page

This page is used to assign internal standards to the elements they will be correcting. To assign internal standards, you must define at least one element to be an Internal Standard on the first page of the Method Editor.

Detail of the Assign Internal Standards page showing entries included in an example method prepared for a soils analysis.

Assign Internal Standards

	Analyte	Internal Standard
1	Ag 328.068	Y 371.029
2	As 188.979	Y 371.029
3	Ba 233.527	Y 371.029
4	Cd 214.440	Y 371.029
5	Cr 267.716	Y 371.029
6	Cr 283.563	Y 371.029
7	Hg 194.168	Y 371.029
8	Pb 220.353	Y 371.029
9	Se 196.026	Y 371.029

Internal Standard Options

Do Calib Blanks contain internal standards?

⦿ Yes ○ No

Report apparent concentrations for internal standards:

⦿ No

○ As A Percent

○ As Concentrations

Set...

Assign Internal Standards

Assign an internal standard for each analyte that you want to be corrected using internal standardization during the analysis. If an analyte does not have an internal standard assigned to it, internal standardization will not be performed for that analyte.

Note   This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Internal Standard Options

Do Calib Blanks contain internal standards?

Set this to "yes" if the calibration blanks in the method contain internal standards, otherwise set this to "no."

If you select "yes," then the first calibration blank analyzed will be used to initialize the internal standardization correction routine. If you select "no," the software uses the internal standard intensity in the first calibration standard to initialize the internal standard routine.

With either choice, you have the option of entering an ID and autosampler location for an Internal Standard blank on the Calibration: Define Standards page. Scroll down to the Int Std Blank entry line. The software subtracts this blank intensity from the internal standard intensities measured in all solutions, i.e. the calibration standards and samples.

Report apparent concentrations for internal standards:

No
Internal standards are reported using intensities only.

As a Percent
During an analysis, the internal standards will be reported as a percentage of an initial concentration of 100 and units will be "%." This lets you compare the intensity of the internal standard to the initial value to see if drift has occurred.

As Concentrations
Define the initial (or actual) concentrations of the internal standards by clicking on **Set..**. and entering the information in the Internal Standards Concentrations dialog. Then internal standard results will be reported in concentration during analysis.

Using Internal Standardization

Using internal standardization

Internal standardization can be applied to an analysis to correct for physical interferences that affect your results.

The two types of physical interferences that internal standardization can correct for are:

1. Sample viscosity variations which affect the pumping efficiency and hence the amount of sample delivered to the nebulizer.

2. Sample surface tension variations of the solutions being aspirated which affect the nebulization efficiency, and hence the amount of sample being vaporized into the spray chamber.

How internal standardization works

Internal standardization uses a non-analyte element as an internal standard. This element must be absent from your original samples, or at the same, known, concentration in all samples, so that the reference signal it generates can be used as a measure of sample being delivered to the plasma. The element selected as the internal standard and the elements you are determining should have similar excitation characteristics so that other interferences do not invalidate the relationship between analyte and internal standard signals. The concentration of internal standard added to or present in all samples, standards and blanks must be identical.

The software takes the intensity observed for the internal standard elements in the solution and references this with the intensity recorded for either the blank or the first calibration standard (depending on whether the calibration blanks contain the internal standard as specified on the Internal Standards page of the method). It then divides the analyte intensities it observes by the result. For instance, if the amount of sample delivered to the plasma drops in half, both the internal standard and the analyte intensities drop in half. When the internal standard correction is applied, the effect of the reduced sample flow is canceled out. Hence, an accurate result can be obtained. For information on the equations used in Internal Standardization, see *Internal Standardization* in Chapter 12, *Calibration and Calculations*.

Selecting internal standards

We recommend that you select background correction when using internal standardization to correct for any background contribution to the signal. The theoretical basis for internal standardization requires the use of the net analyte signal only, as opposed to the gross uncorrected analyte signal.

Ideally, the internal standard element you select for your analysis should satisfy the following requirements:

- Not present in the sample, or present in all at exactly the same known concentration.

- Similar excitation requirements.

- Free from contaminants.

- Readily available.

- No spectral lines that would interfere with the spectral lines of the analyte. Iron, for example, can be a poor choice at higher concentrations due to its complex emission spectrum.

- Present at a concentration from 500 to 1000 times the detection limit concentration or from 1 to 10 mg/L.

For calibration, the software uses internal-standard-corrected emission intensities rather than analyte-to-internal standard ratios. This allows stored calibrations with or without internal standards to be used.

Specifying internal standardization in the method

Decide on the following selections in your method when using internal standards:

1. Select the element(s) you want to use as internal standard(s) and define their function as "Int. Std." on the Spectrometer: Define Elements page.

2. Select the Process tab of the method, then select the Internal Standards page. On this page:

   - Assign the internal standard(s) to the analyte(s) in the table.

   - Select whether or not the calibration blanks contain the internal standard. The internal standard must be in all calibration solutions and samples. You can choose to use a special internal standard blank. The internal standard blank intensity is subtracted from the internal standard intensity measured in all solutions. An Internal Standard blank is useful if the internal standard selected has a comparatively low intensity and the blank's intensity is

significant relative to that of the internal standard. It may also be useful if there is structure in the blank such as an OH-band.

- • Select if you want to report the apparent concentrations of the internal standard element. If so, you can report the concentrations as a percent (with the initial percentage assigned as 100%) or as a concentration referenced to a value you enter.

Results corrected for internal standardization

Results that have been internal standard corrected are marked with a footnote symbol. The $\dagger$ symbol is placed just after the analyte name.

Method Editor Process Pages: Internal Standards Concentrations Dialog

Use this dialog to define the initial (or actual) concentrations of the internal standards.

To display this dialog

▶ Click on **Set...** beneath **Report apparent concentrations for the internal standards** on the **Assign Internal Standards** page.

Using this dialog

▶ Type the concentration for each internal standard and select the units from the drop-down list. You cannot change the Internal Standards listed here. You select these on the Method Editor Spectrometer: Define Elements page.

Method Editor: Calibration Pages Overview

You use the Calibration pages to define calibration standards and concentrations and to select parameters such as the type of calibration equation and the autosampler locations for blanks and standards. You also select how blanks will be used, and whether or not to use a stored calibration.

To display the Calibration pages

▶ Click on the **Calibration** tab on the bottom of the Method Editor window.

Calibration pages

▶ Click on the appropriate tab on the right-hand side of the window to select these pages:

• Define Standards Page

• Calibration Units and Standard Concentrations Page

• Calibration and Reagent Blank Usage Page

• Calibration Equation and Sample Units Page

• Initial Calibration Options Page

• Multiline Calibration Page

Method Editor Calibration Pages: Define Standards Page

You use this page to select the IDs and autosampler locations that you want to use for the blanks, standards, and other calibration solutions.

Detail of the Define Standards page showing entries included in an example method prepared for a soils analysis.

Method of Additions

Check this option to analyze samples using method of additions. When Method of Additions is checked a drop down list of method of additions equations becomes enabled. From the drop down select the appropriate method of additions equation to use in your method. You can select from the following equations: **Method of Additions, Sample Intercept**, **Method of Additions, Calculated Intercept**, **Method of Additions Calibrate**. Please note that the equation you select here will be applied to all analytes specified in your method.

ID

This is the name that you give to each calibration solution. It will be printed on the log and saved in the results data set. If you do not type a name, the system uses pre-set IDs such as Calib. Blank, Calib Std 1, etc.

A/S Location

This is the autosampler location you wish to use for each calibration solution. For autosampler trays that have multiple size tube capacities, the larger containers located in positions 1 through 8 typically are used for blanks, calibration standards, and QC samples, if any. The next available autosampler location is automatically assigned by the software when a new calibration blank, calibration standard, or reagent blank ID is entered. The user may modify the settings.

Calibration Blank 1-5

You can use up to 5 calibration blanks. The calibration blanks are analyzed before the standards in an automated analysis. How the calibrations blanks are used in the analysis is defined on the Blank Usage page. (It is useful to have 5 calibration blanks available, for example, if you need to create a blank to match each matrix in each standard.)

▶ Type the ID for each calibration blank.

Internal Standard Blank

If you have entered an internal standard, this option appears on the bottom of the scrolling list of Calib Blanks. (You may need to scroll to reach it.) If you select an internal standard blank for the method, you decide which internal standards it is used for on the Blank Usage page.

▶ Type the ID for the internal standard blank.

Calibration Standard 1 - 30

You can use up to 30 calibration standards. These standards will be analyzed in sequence (following the calibration blanks) in an automated analysis, beginning with Calibration Standard 1.

▶ Type the ID for each calibration standard.

Addition 1-30

You can use up to 30 additions.

▶ Type the ID for each addition

Note   When you select **Method of Additions**, the software assumes that the
aliquots of sample containing the additions (addition 1, addition 2, etc)
immediately follow the orginal sample in the autosampler tray. If there are
two or more samples to analyze, each sample has its own additions. The
samples should be placed in the autosampler in the following order: blank,
sample, A1 (sample + addition 1), A2 (sample + addition 2), etc. You run
a blank for the first sample only. This blank will be used for all other
samples.

Reslope Standard

Note: This option is disabled if you checked Method of Additions.

You use the reslope standard to make small corrections to the slope of the
calibration curve using a single calibration standard following small changes in
instrumental conditions.

► Type the ID for the reslope standard.

Note   You must also schedule when the Reslope Standard should be analyzed.
You schedule the Reslope Standard using the Recalibrate Before
parameter in the Sample Information Editor.

Reagent Blank 1-5

Note: This option is disabled if you checked Method of Additions.

You can use a reagent blank to compensate for differences in composition between
the reagents used to prepare the samples and those used to prepare the calibration
standards. Reagent blanks are analyzed immediately after the calibration standards
in an automated analysis. The reagent blank concentrations measured are
subtracted from all subsequently analyzed samples. If multiple reagent blanks are
analyzed, their concentrations are averaged and the average concentration is
subtracted from each sample.

► Type the ID for each reagent blank.

Deleting Blanks and Standards

It is important to clear the information for blanks and standards that are not to be included in an automated analysis. All blanks and standards that are present in the method will be analyzed in an automated analysis.

▶ To clear the information for unwanted blanks and standards, click on the left-most column to highlight the rows for blanks and standards you want to remove and press the **Delete** key.

The rows for the blanks and standards will remain in the tables, but the information in them will be cleared along with any information relating to them (including concentrations) in the remainder of the pages in the Calib section of the Method Editor.

Method Editor Calibration: Calibration Units and Standard Concentrations Page

You use this page to select the units and concentrations for your calibration standards or additions, if Method of Additions is checked on the Define Standards page of the Method Editor.

Detail of the Calibration Units and Standard Concentrations page showing entries included in an example method prepared for a soils analysis.

Calibration Units and Standard Concentrations

	Analyte	Calib Units	Calib Std 1	Hg Std	
1	Ag 328.068	mg/L ▼	2.500		
2	As 188.979	mg/L	2.500		
3	Ba 233.527	mg/L	50.000		
4	Cd 214.440	mg/L	0.250		
5	Cr 267.716	mg/L	2.500		
6	Cr 283.563	mg/L	2.500		
7	Hg 194.168	mg/L		0.500	
8	Pb 220.353	mg/L	2.500		
9	Se 196.026	mg/L	0.250		

Analyte

This view-only column shows all the elements designated as analytes for the current analysis. To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Calib Units

This column shows the units in which the concentrations of your calibration solutions/additions solutions are expressed.

▶ To change the units for an analyte, select an option from the drop-down list.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

To select your own units

The units available in the drop-down lists are stored in an ASCII file called units.ini located in the C: (or chosen drive) \Program Files\WinLab32 directory. You can use any text editor to add units to the file. You can also rearrange the units in the file so that those used frequently are on the top of the list. The units.ini file contains information on the format and content of the file. If the units.ini file contains improper information, the software may fail.

Standard Concentrations

These columns show the standard concentrations for each analyte in each calibration standard. If the concentration entry for an analyte in a standard is left blank, then that analyte will not be measured when that standard is analyzed. Do not enter zero for a calibration standard.

▶ To enter or change the standard concentration for an analyte, type the new concentration in the entry field.

▶ To clear the standard concentration for an analyte, click on the entry field and press **Delete**.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Method Editor Calibration Pages: Calibration and Reagent Blank Usage Page

You use this page to select which calibration blanks should be used in calibrating the elements in your analysis. You can also decide whether the blanks should be subtracted from the analyte intensities, used for calibration, or both.

Detail of the Calibration and Reagent Blank Usage page showing entries included in an example method prepared for a soils analysis.

Calibration and Reagent Blank Usage

	F'n	Element	Use Calibration Blank	Subtract Reagent Blank
1	A	Ag 328.068	Calib Blank 1	n/a
2	A	As 188.979	Calib Blank 1	n/a
3	A	Ba 233.527	Calib Blank 1	n/a
4	A	Cd 214.440	Calib Blank 1	n/a
5	A	Cr 267.716	Calib Blank 1	n/a
6	A	Cr 283.563	Calib Blank 1	n/a
7	A	Hg 194.168	Calib Blank 1	n/a
8	A	Pb 220.353	Calib Blank 1	n/a
9	A	Se 196.026	Calib Blank 1	n/a
10	IS	Y 371.029	None	n/a

F'n (Function)

This view-only column shows the action that the instrument will take for this element.

- A = Analyte

- IS = Internal Standard

To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Element

This view-only column shows all the elements selected for the current analysis. To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Use Calibration Blank

This column shows which calibration blank should be used as part of the calibration for this element. Having multiple calibration blanks allows you to select different calibration blanks for different elements to correct for differences in baseline of each blank solution. The calibration blank is usually analyzed before the standards.

The way in which the calibration blank is treated in the calibration is based on the calibration equation. For more information, see Chapter 12, *Calibration and Calculations*.

▶ To change the calibration blank for one element, select an option from the drop-down list.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Subtract Reagent Blank

This column allows you to use reagent blanks to correct for variations in analytes. The normal way of using a reagent blank is to subtract the results for all elements. By turning off reagent blank subtraction for certain elements that you know are not present in the reagent blank, you can reduce the noise or bias that subtracting a near-zero reagent blank concentration would otherwise add to a measurement. The column is active only if one or more reagent blanks have been defined on the Define Standards page.

Note This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Method Editor Calibration Pages: Calibration Equations and Sample Units Page

You use this page to select the type of calibration equation you will use and the units and concentration reporting format for your samples.

Note: If you checked Method of Additions on the Define Standards page, then the Calibration Equation cells are read-only. The Calibration Equation that is displayed for each element, when Method of Additions is enabled, is the equation that is selected on the Define Standards page.

Detail of the Calibration Equations and Sample Units page showing entries included in an example method prepared for a soils analysis.

Calibration Equations and Sample Units

	F'n	Element	Calibration Equation	Sample Units	Max. Dec. Places	Max. Signif. Figs
1	A	Ag 328.068	Lin, Calc Int	mg/kg	3	4
2	A	As 188.979	Lin, Calc Int	mg/kg	3	4
3	A	Ba 233.527	Lin, Calc Int	mg/kg	3	4
4	A	Cd 214.440	Lin, Calc Int	mg/kg	3	4
5	A	Cr 267.716	Lin, Calc Int	mg/kg	3	4
6	A	Cr 283.563	Lin, Calc Int	mg/kg	3	4
7	A	Hg 194.168	Lin, Calc Int	mg/kg	3	4
8	A	Pb 220.353	Lin, Calc Int	mg/kg	3	4
9	A	Se 196.026	Lin, Calc Int	mg/kg	3	4
10	IS	Y 371.029	n/a	mg/kg	3	4

F'n (Function)

This view-only column shows the kind of action that the instrument will take for this element.

- A = Analyte

- IS = Internal Standard

To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Element

This view-only column shows all the elements selected for the current analysis. To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Calibration Equation

This column shows the calibration equation used to process the analytical data on this item, defining the relationship between intensity and concentration for your samples. See Chapter 12, *Calibration and Calculations* for further details.

▶ Select the calibration equation for an analyte.

Linear (Lin, Calc Int)
This equation creates a calibration curve that relates concentration to intensity linearly.
Linear equations are used when you expect a linear relationship between concentration and intensity. Generally, this is 4 to 6 orders of magnitude greater than the detection limits and will depend on wavelength region and your sample matrix. In this equation, the calibration blank is subtracted from all solutions and included in the calculation of the calibration curve. The intercept with the intensity axis is calculated rather than fixed at zero intensity and zero concentration.

Linear-thru-zero
This creates a linear calibration curve, but forces the curve through the origin of the graph. Linear equations are used when you expect a linear relationship between concentration and intensity. Generally, this is 4 to 6 orders of magnitude greater than the detection limits and will depend on wavelength region and your sample matrix. In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve. The calibration curve is forced to go through zero intensity and zero concentration.

Linear (Bracketing)
This is a linear calibration technique designed for use over a restricted concentration range. The calibration is performed with at least two standards that bracket the calibration range of interest. The use of a blank is optional. If you use a blank, it is subtracted from all solutions, but is not included in the calculation of the calibration curve.

Non-linear (Non Lin, Calc Int)

Non-linear equations are used when you expect your sample concentrations to be outside of the linear range of the technique. This option covers the widest concentration range and requires that you use a blank and at least three standards. In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve. The intercept with the intensity axis is calculated rather than fixed at zero intensity and zero concentration.

Non-linear (Bracketing)

Non-linear equations are used when you expect your sample concentrations to be outside of the linear range of the technique. Bracketing is a calibration technique designed for use over a restricted concentration range. The calibration is performed with at least three standards that bracket the calibration range of interest. The use of a blank is optional. If you use a blank, it is subtracted from all solutions, but is not included in the calculation of the calibration curve.

Note   This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Sample Units

This column shows the units in which the sample results are expressed. Conversion from sample results determined in Calib Units to results expressed in Sample Units involves corrections made for weight, volume, and dilution information supplied for samples in a Sample Information File (or the Sample Details dialog in a manual analysis), e.g. converting from mg/L to mg/kg. Corrections are also automatically made for converting units with order-of-magnitude differences, e.g. converting mg/L to µg/L.

▶   To change the units for an analyte, select an option from the drop-down list.

Note   This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

You can select either Weight/Volume units or Weight/Weight units. There are, however, some restrictions on using combinations of certain Calib Units with

certain Sample Units with respect to information that needs to be supplied in the Sample Information File (SIF) for each sample. Failure to include the proper information with samples will cause sample results to be reported in Calib Units only. See *Sample Preparation Parameters* in Chapter 4, *Sample Information Editor* for more details about the sample quantity information required in the Sample Information Editor.

To select your own sample units

The units available in the drop-down lists are stored in an ASCII file called units.ini located in the C: (or chosen drive) \Program Files\WinLab32 directory. You can use any text editor to add units to the file. You can also rearrange the units in the file so that those used frequently are on the top of the list. The units.ini file contains information on the format and contents of the file. If the units.ini file contains improper information, the software may fail.

Max. Decimal Places

This column shows the maximum number of figures to the right of the decimal point that are displayed for the concentrations reported in sample units or calibration units.

It is possible that the final result will not include the number of decimal places specified here, depending on the value that is specified for Max. Significant Figures. In general, when the result is small, the Max. Decimal Places value is used. However, when the result is large, the Max. Significant Figures is used.

Example 1

Max. Decimal Places = 3, Max. Significant Figures = 2
Calculated Concentration: 11.001
Reported Concentration: 11

The calculated concentration has three decimal places, but it exceeds the maximum number of significant figures. Therefore, the result is reported as 11 with two significant figures.

Example 2

Max. Decimal Places = 3, Max. Significant Figures = 4,
Calculated Concentration: 0.03587
Reported Concentration: **0.036**

The calculated concentration has four significant figures, but it exceeds the maximum number of decimal places. Therefore, the result is reported as 0.036 with three decimal places.

Note   This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Max. Significant Figures

This column shows the maximum number of significant figures that are displayed for the concentrations reported.

It is possible that the final result will not include the number of figures specified here, depending on the value that is specified for Max. Decimal Places. See the examples under Max. Decimal Places for more information.

Note   This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Method Editor Calibration Pages: Initial Calibration Options Page

You use this page to decide whether to create a new calibration using standard and blank solutions, or to use a calibration that has been stored.

Detail of the Initial Calibration Options page.

Initial Calibration Options

When opening this method manually:

☐ Load the calibration curve set selected below:

[   ]   Browse...

When using this method in a multimethod sequence:

⦿ Start by constructing new calibration curves.

○ Start using the existing calibration curve set.

○ Start using the stored calibration curve set selected below:

[   ]   Browse...

When opening this method manually:

Load the calibration curve selected below:
All calibration curves will be cleared when the method is opened, and the calibration curves you select will be recalled from a stored results data set. If you are performing a manual analysis, the calibration curves that you have selected here will be used for the analyses. If you are performing an automated analysis, you can use the calibration curves that you have selected here instead of generating a new calibration. When you click on Analyze Samples in the Automated Analysis Control window, the calibration curves selected here will be used.

When using this method in a multimethod sequence:

When two or more methods are entered in the Set Up page of the Automated Analysis Control window, the options are:

Start by constructing new calibration curves
New calibration curves will be created for each method by analyzing the

blanks and standards specified in the method.

Start using the existing calibration curve set
The calibration curves currently appearing in the Calibration Display window from the previous methods used will be carried over.

Start using the stored calibration curve set selected below
The calibration curves you select will be recalled from a stored results data set. If several calibrations are contained in the results data set, the last set of calibration curves generated will be recalled.

Method Editor Calibration Pages: Multiline Calibration Page

You use this page to select various reporting options when using two or more wavelengths for the same element. You can choose to:

• select the appropriate wavelength based on the concentration of the sample. You must have a different concentration range for each wavelength. See the Select Line action described below.

• report the average concentration of two or more wavelengths. You must have the same concentration range for each wavelength. See the Average Lines or Report and Average actions described below.

The Multiline Calibration Page. For easy reference, blue highlighting helps distinguish between each element group.

☑ **Multiline Calibration**

Seq.	Elem.	Action	Analytes	Low	High	Units
->1	Cu	Always Report ▼	Cu 324.752	0.00	20.00	mg/L
2	Cu	Always Report	Cu 224.700	0.00	20.00	mg/L
3	Cu	Always Report	Cu 327.393	0.00	20.00	mg/L
1	Fe	Select Line	Fe 238.204	0.00	100.00	mg/L
2	Fe	Select Line	Fe 234.349	100.00	500.00	mg/L
3	Fe	Select Line	Fe 273.955	500.00	2000.00	mg/L
1	Ti	Average Lines	Ti 334.940	0.00	20.00	mg/L
2	Ti	Average Lines	Ti 336.121	0.00	20.00	mg/L
3	Ti	Average Lines	Ti 337.279	0.00	20.00	mg/L
1	Zn	Report & Average	Zn 206.200	0.00	20.00	mg/L
2	Zn	Report & Average	Zn 213.857	0.00	20.00	mg/L
3	Zn	Report & Average	Zn 202.548	0.00	20.00	mg/L

Move Up   Move Down

The list of analytes on this page is taken from the Method Editor Spectrometer: Define Elements page, but only the analytes to which multiline calibration applies appear here. The results will appear in the Results Display window.

Multiline Calibration

Note: Multiline Calibration is disabled when **Method of Additions** is checked on the D**efine Standards** page.Select this checkbox to use multiline calibration. Note: If the spreadsheet table is dimmed, you must select this checkbox to activate it.

Sequence

This column shows the order in which each analyte will be considered within each element group.   This is meaningful for only the Select Line action and can be changed using the Move Up and Move Down buttons.

Element

This view-only column shows the elements in the current method that meet the criteria for multiline calibration, that is, two or more wavelengths have been assigned to this element. The elements are sorted first alphabetically and then by concentration range.

Action

This is how you want to have the concentration reported for each analyte.

Note: This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Select an action for each analyte.

Always Report
Use this action to report the concentration of an element at each wavelength assigned, without the use of the multiline calibration feature. In the example page shown at the beginning of this section, the individual concentration will be reported for copper at each wavelength. In the Results Display window, this action shows the concentrations at each wavelength, as in normal reporting.

Select Line
Use this action to determine an element at two or more wavelengths, but report the concentration at only one wavelength, based on the element concentration

found in the sample.

In the example page shown at the beginning of this section, the Select Line action has been assigned to iron at three wavelengths. Suppose that, when the analysis is performed, the concentration of iron is found to be 150 mg/L. This concentration will be reported at the 234.349 wavelength.

The software compares the 150 mg/L result with the concentration range of the first wavelength in the sequence, 238.204. Because the concentration does not fall within the 0 to 100 mg/L range, the next wavelength in the sequence, 234.349, is considered. The 150 mg/L concentration does fall within the 100 to 500 mg/L range, which was used for this wavelength. Therefore, the Results Display window will show only the result at Fe 234.349, 150 mg/L.

Note: If the concentration range does not fall within the range of any of the wavelengths, then the last wavelength in the sequence is reported.

You can change the sequence using the Move Up and Move Down buttons. The software will consider each wavelength in the sequence you specify.

Average Lines
Use this action to report an average concentration for an element at two or more wavelengths. In the example page shown at the beginning of this section, the concentration of titanium is determined for each wavelength. The three concentration values are averaged and the result is reported. To use this action, you must have the same concentration range and units assigned to each analyte. However, you can assign different calibration equations. One average concentration per element is calculated.

In the Results Display window, only the calculated average concentration is shown. The average concentration is displayed with a new analyte name using the element symbol and the word "Average." For example, "Ti Average."

Note: A saturated wavelength will not be included in the calculation.

Report & Average
Use this action to report individual concentrations for an element at two or more wavelengths as well as the average concentration. In the example page shown at the beginning of this section, the concentration of zinc is determined for each wavelength, an average calculated and all information reported. To use this action, you must have the same concentration range and units assigned to each analyte. However, you can assign different calibration equations. The

Results Display window shows the concentration of the element at all wavelengths and the calculated average of these concentrations.

Analytes

This view-only column shows all of the analytes in the current analysis that meet criteria for multiline calibration. This information was entered on the Method Editor Spectrometer: Define Elements page.

Low

This view-only column shows the lower limit of the calibration range. A zero appears here for any calibration equation except linear or nonlinear bracketing. For either bracketing equation, the lower limit that you entered in the Method Editor Calibration: Calibration Units and Standard Concentration page will appear here.

High

This view-only column shows the upper limit of the calibration range. You entered this information on the Method Editor Calibration: Calibration Units and Standard Concentration page.

Units

This view-only column shows the units in which the concentration will be expressed.

Move Up

When you choose Select Line, you use this button to move an analyte up one place within the sequence of an element group. The software will consider each wavelength in the sequence you specify.

Move Down

When you choose Select Line, you use this button to move an analyte down one place within the sequence of an element group. The software will consider each wavelength in the sequence you specify.

Method Editor: Recovery Checks Page
(Analyte Concentrations added to Recovery Check)

You use this page to enter the concentrations of analytes added to matrix recovery check (i.e., "spike") samples analyzed using this method. Scheduling of matrix recovery check samples for an analysis is performed using the Sample Information Editor.

Detail of the Analyte Concentrations page.

Analyte Concentrations Added to Recovery Check

	Analyte	Conc Units	Recovery Set 1	Recovery Set 2	Recovery Set 3
1	Ag 328.068	mg/kg			
2	As 188.979	mg/kg			
3	Ba 233.527	mg/kg			
4	Cd 214.440	mg/kg			
5	Cr 267.716	mg/kg			
6	Cr 283.563	mg/kg			
7	Hg 194.168	mg/kg			
8	Pb 220.353	mg/kg			
9	Se 196.026	mg/kg			

Concentration Units: ○ Calibration   ◉ Sample

To display the Checks page

▶ Click on the **Checks** tab at the bottom of the Method Editor window.

Analyte

This view-only column shows all the analytes selected for the current analysis. To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Conc Units

This view-only column shows Calibration Units or Sample Units, depending on the selection made at the bottom of this page.

Recovery Set 1 to Set 6

These are the concentrations of analytes added to samples to create matrix recovery check (i.e. "spike") samples to be analyzed using this method. You may enter up to six different sets of concentrations for each method. In an analysis, a recovery set can be used more than once. For example, you may analyze three recovery check samples that are spiked with high analyte concentrations as defined in Set 1 and three other recovery check samples that are spiked with low analyte concentrations as defined in Set 2.

To perform spike recovery measurements, you must enter information about the matrix check samples in the Sample Information Editor using the Matrix Check Sample Entry Dialog. The equation used to calculate recovery checks is shown in the help topic for the Matrix Check Sample Entry Dialog.

Note   This parameter has a Column Fill dialog that can be accessed by double-clicking on the parameter's column header.

Concentration Units

Calibration
Select this option to perform recovery calculations using the matrix check sample concentrations expressed in Calib Units.

Sample
Select this option to perform recovery calculations using the matrix check sample concentrations expressed in Sample Units.

Method Editor: Sample Limits Checks Page
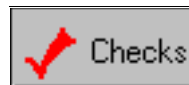
The Sample Limits page allows you to quickly and easily set up sample limit checks. When a sample result falls above or below a sepcified range, a message is displayed in the Results window to indicate the failure
.

Detail of the Sample Limits Checks page.

	Analyte	Units	Conc	Lower	Upper	
1	Cr 205.560	ug/L	1.25	1.125	1.375	
2	Zn 206.200	ug/L		0.99		
3	Ni 221.648	ug/L	1.25	1.125	1.375	
4	Cu 224.700	ug/L	1.1	0.99	1.21	
5	Co 228.616	ug/L				
6	Fe 238.204	ug/L				
7	Mn 257.610	ug/L				
8	Mg 285.213	ug/L				
9	V 290.880	ug/L				
10	Ti 334.940	ug/L				

☑ Sample Limits

Concentration Units: ○ Calibration   ● Sample

Calculate Limits...

☐ Show sample limit range along with out of limit remark

Using the Sample Limits Page

On this page you specify limits for analytes that are defined in your method. These limits will be used to determine whether or not a flag is displayed when a sample result falls above or below a specified limit. You have the option to enter a Concentration value for a particular analyte/s and then use the Calculate Limits button to automatically calculate upper and lower limits for the analytes where a concentration is specified. Or, you can simply enter a value in the Upper and/or Lower Limit cells for a particular analyte.

To display the Sample Limits page

1. Open the Method Editor.

2. Click on the **Checks** tab, which is located on the bottom of the Method Editor window.

3. Click on the **Sample Limits** tab page.

To automatically calculate limits:

1. Enter the sample **Concentrations**.

2. Click on the **Calculate Limits...** button.
   The Calculate Limits dialog appears.

3. On the Calculate Limits dialog, specify the upper and lower limits in percent to be used to calculate the limits.

4. Click **OK**.
   You are returned to the Sample Limits page and limits are only calculated for analytes that have a Concentration specified.

5. Select whether you want concentrations expressed in **Calibration** units or **Sample** units.

6. Check the Show range along with limit remark option to have the range displayed on the results screen along with the upper/lower limit fail message, if a sample falls above/below the specified limit.

Note: Whenever you change the parameters on the Calculate Limits dialog and click **OK**, the Upper and Lower Limits, displayed on the Sample Limits page, are automatically updated for all analytes that have a concentration specified.

To specify an upper and/or lower limit:

1. Type in the sample **Upper** and/or **Lower** limit values.

2. Select whether you want concentrations expressed in **Calibration** units or **Sample** units.

3. Check the Show range along with limit remark option to have the range displayed on the results screen along with the upper/lower limit fail message, if a sample falls above/below the specified limits.

Method Editor: QC Pages Overview

You use the QC pages to enter parameters that describe quality control samples. QC samples are used to perform runtime checks of instrument performance. You schedule when and under what conditions the QC samples are to be analyzed. QCs can be scheduled after the initial calibration, after any recalibration, periodically throughout the analysis and at the end of the analysis.

The periodic QCs also can be scheduled in the Sample Information Editor. If periodic QCs are scheduled in both the Method Editor and the Sample Information Editor, then the Sample Information Editor takes precedence.

To display the QC pages

► Click on the **QC** tab on the bottom of the Method Editor window.

To perform QC analyses

To enter the required QC information, you must perform these steps:

1. Enter descriptive information about each QC sample including the QC sample ID and autosampler location on the first QC page.

2. Enter the concentration and the upper and lower limits for each QC sample on the second QC page. The software can calculate the upper and lower concentration limits, or those limits can be entered manually.

3. Select when each QC sample is to be analyzed on the third QC page. The frequency can be the same for all QC samples or different for each one.

Note   QC Samples can also be scheduled using the Sample Information Editor.

---

4. Select the action to be performed when the measured concentration is outside of the acceptable limit for each QC sample on the fourth and fifth QC pages.

5. Select the maximum number of times a group of standards or standards and samples may be reanalyzed to prevent the system from going into an infinite loop on the Actions: End and Retry page.

QC Pages

▶ Click one of the tabs on the right side of the Method Editor to select a page.

•   QC Sample Definition Page

•   QC Sample: Concentration and Limits Page

•   Schedule for QC Analyses Page

•   Failure Actions for After-Calibration and Periodic QC's Page

•   Failure Actions for At-End QC's

QC Flow Charts

There are four QC flow charts. The first flow chart shows an example of how a QC schedule might look. The second flow chart shows the sequence of an initial calibration. The third flow chart shows the analysis of an initial calibration QC. Lastly, the fourth flow chart shows an example of a failure action. The failure action used is Recalibrate and Continue.

Flow Chart 1:   An Example of a QC Schedule

```
┌─────────────┐      ┌──────────────┐      ┌──────────┐
│ Initial     │ ───► │ Analyze After│ ───► │ Analyze  │
│ Calibration │      │ Initial Calib QC│   │ Unknown  │
└─────────────┘      └──────────────┘      │ Samples  │
  See Flow              See Flow           └──────────┘
  Chart #2              Chart #3

┌─────────┐      ┌──────────┐      ┌───────────┐
│ Analyze │ ───► │ Analyze  │ ───► │ Analyze at│
│ Periodic│      │ Unknown  │      │ End QC    │
│ QC      │      │ Samples  │      └───────────┘
└─────────┘      └──────────┘
```
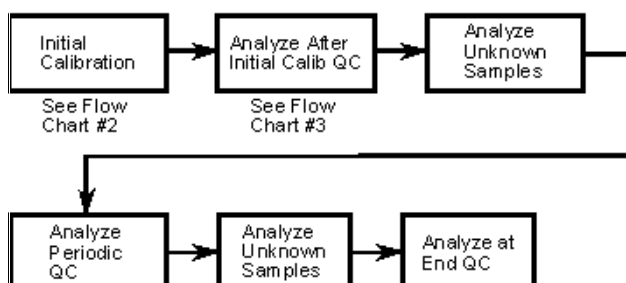
Flow Chart 2:   Initial Calibration

```
┌───────────┐   ┌────────┐   ┌────────┐   ┌────────┐   ┌────────┐   ┌──────────┐
│Calibration│──►│Standard│──►│Standard│──►│Standard│──►│Standard│──►│ Reagent  │──► See Flow
│ Blank     │   │   1    │   │   2    │   │   3    │   │  ...n  │   │ Blank    │    Chart #3
└───────────┘   └────────┘   └────────┘   └────────┘   └────────┘   │(optional)│
                                                                    └──────────┘
```
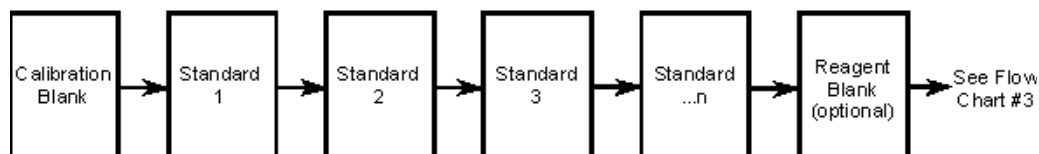
Flow Chart 3:   The Analysis of Initial Cal QC
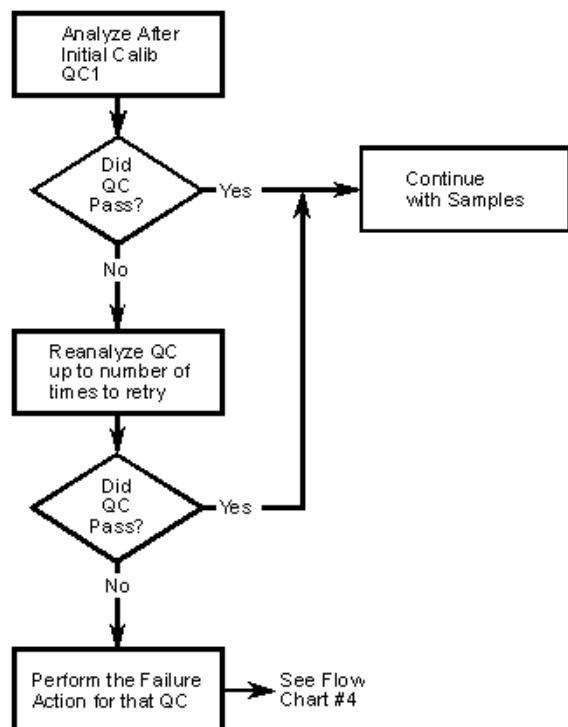
Note If the QC fails and there are zero retries specified, then the instrument will perform the failure action for that QC.

Flow Chart 4:   An Example of a Failure Action

```
        ┌─────────────────────┐
        │ Execute Recalibration│
        │ and Continue Action  │
        └─────────────────────┘
                   │
                   ▼
        ┌─────────────────────┐
        │ Analyze After        │
        │ Recalibration QC     │
        └─────────────────────┘
                   │
                   ▼
               ╱ Did ╲
              ╱  QC   ╲──Yes──────────┐   ┌──────────────┐
              ╲ Pass? ╱               │   │ Continue     │
               ╲     ╱                │   │ with Samples │
                  │                   │   └──────────────┘
                 No                   │
                   │                  │
                   ▼                  │
        ┌─────────────────────┐       │
        │ Reanalyze QC         │       │
        │ up to a number of    │       │
        │ times and retry      │       │
        └─────────────────────┘       │
                   │                  │
                   ▼                  │
               ╱ Did ╲                │
              ╱  QC   ╲──Yes──────────┘
              ╲ Pass? ╱
               ╲     ╱
                  │
                 No
                   │
                   ▼
        ┌─────────────────────┐
        │ Recalibrate QC Failed│
        │ All Retries          │
        │ Action is Stop       │
        └─────────────────────┘
```
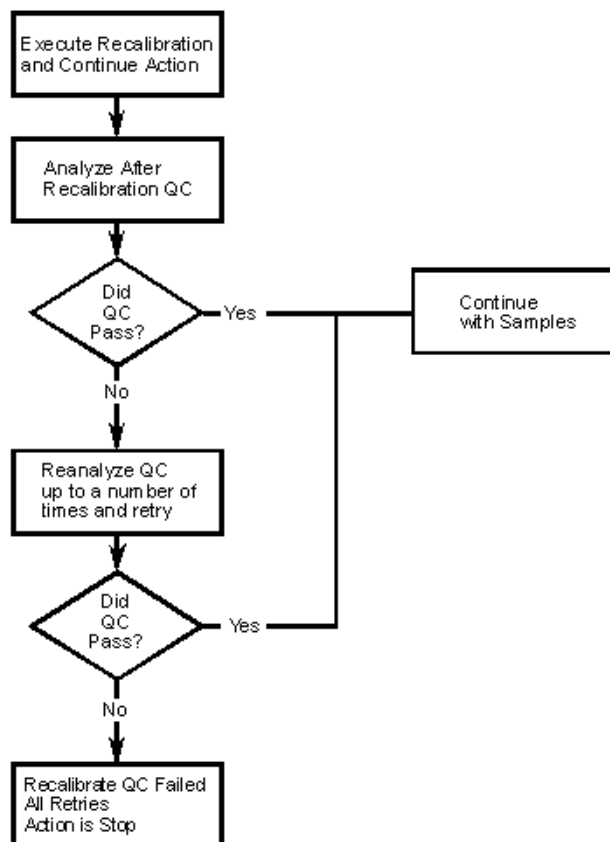
Method Editor QC Pages: QC Sample Definition Page

You use this page to enter the names and autosampler locations for the QC samples. You also indicate whether to count a QC as a sample and whether to subtract reagent blanks from the QC samples. This page contains a table with columns for up to 20 QC samples and includes rows for the following parameters.

Detail of the Sample Definition page showing entries included in an example method prepared for a soils analysis.

QC Sample Definition

	QC 1	QC 2	QC 3
QC Sample ID	Mid QC	Hg Mid QC	
Autosampler Loc	4	5	
Count as Sample	☐	☐	☐
Subtract Reagent Blank	☐	☐	☐

QC Sample ID

This is the quality control sample ID that will be displayed in the Results Display window and in the results data set and printed or sent to a log file. You must first enter an ID in order to enter any other information for a QC. The maximum number of characters is 20.

Example: **EPA Check #1**

Autosampler Location

The software will automatically assign the next available autosampler location when a QC Sample ID is entered. Enter the autosampler location for each quality control sample if the location selected by the system is not appropriate.

► Click on an entry field and type a value between 0 and the maximum location number on your autosampler tray.

Count as Sample

If you want the QC to be considered part of the sample count, select this box. For example, for a frequency of every 10 samples, there are nine unknown samples and one QC sample.

Subtract Reagent Blank

Selecting this box for this parameter will cause the reagent blank concentrations to be subtracted from the measured QC Sample concentrations.

If the QC Sample is to be analyzed as an unknown sample, then you would normally subtract the reagent blank. If the QC Sample is a calibration standard that is similar in matrix to the unknown sample and was reanalyzed to check the calibration, then you would not subtract the reagent blank. This is because the reagent was not subtracted from the calibration standard when the reagent was originally analyzed.

Method Editor QC Pages: QC Samples: Concentrations and Limits Page

This page is used to enter QC sample concentrations and the upper and lower limits which trigger failure actions by the software.

Detail of the Concentrations and Limits page showing entries included in an example method prepared for a soils analysis.

QC Sample Concentrations and Limits

QC Sample ID: QC 1: Mid QC

	Analyte	Units	Conc	Lower	Upper
1	Ag 328.068	mg/L	1.25	1.13	1.38
2	As 188.979	mg/L	1.25	1.13	1.38
3	Ba 233.527	mg/L	25	23	28
4	Cd 214.440	mg/L	0.125	0.113	0.138
5	Cr 267.716	mg/L			
6	Cr 283.563	mg/L	1.250	1.125	1.375
7	Hg 194.168	mg/L			
8	Pb 220.353	mg/L	1.250	1.125	1.375
9	Se 196.026	mg/L	0.125	0.113	0.138

Concentration Units: ⦿ Calibration   ◯ Sample

Calculate Limits...

QC Sample ID

This specifies the QC sample whose parameters are displayed.

Analyte

This view-only column shows all the analytes selected for the current analysis. To change the parameter, use the entry field on the Spectrometer: Define Elements page.

Units

This view-only column shows Calibration Units or Sample Units, depending on the selection made at the bottom of this page.

Concentration/Upper Limit/Lower Limit

This specifies the action limits for each QC sample. If the measured concentration is between the specified upper and lower limits, no action will be taken. If the measured concentration is above the upper limit or below the lower limit, the appropriate failure action, which is defined on the appropriate Actions page, will be performed.

To automatically calculate the upper and lower limits:

1. Enter the QC sample concentrations.

    Since the significant figures and decimal places in the calculated limits are based on those in concentration entries, you must enter the concentrations using enough significant figures and decimal places so that the limits to be calculated have enough significant figures and decimal places for proper limit checking. For example, if you only use one figure for the concentration entry, then the limits will also have only one figure.

2. Click on the **Calculate Limits...** button.

3. When the Calculate Limits dialog appears, specify the upper and lower limits in percent to be used to calculate the limits.

4. Click on **OK**.

Automatic limit calculation can only be done on a percentage basis of the entered QC sample concentration. Limits based on detection limits have to be entered manually since the QC sample concentration is usually zero.

Calculate Limits...

This button displays the Calculate Limits dialog, which you use to calculate the upper and lower limits for QC samples.

Calculate Limits Dialog

Use this dialog to calculate the upper and lower limits for QC samples.

To display this dialog

▶ Click on the **Calculate Limits...** button on the **QC Sample Concentrations and Limits** page of the Method Editor.

Using this dialog

1. Specify the upper and lower limits in percent to be used to calculate the limits.

2. Click on **OK**.

Automatic limit calculation can only be done on a percentage basis of the entered QC sample concentration. Limits based on detection limits have to be entered manually since the QC sample concentration is usually zero.

Method Editor QC Pages: Schedule for QC Analyses Page

This page is used to enter the schedule for the QC analyses.

Note	QC samples can also be scheduled using the Sample Information Editor. If scheduling is done using the Sample Information Editor, this takes precedence over any scheduling in the Method Editor.

Detail of the Schedule for QC Analyses page showing entries included in an example method prepared for a soils analysis.

Schedule for QC Analyses

	QC 1	QC 2		
QC Sample ID	Mid QC	Hg Mid QC		
After Initial Calib	✔	✔		
After Recalibration	✔	✔		
Periodically	✔	✔		
Frequency	1	1		
At End	✔	✔		

Periodic timing of Analyses

Frequency: ⦿ Same for all QC's: 8   ○ Variable

☐ Maximum time between QC's: 1   minutes

Schedule for QC Analyses

QC Sample ID

This view-only row specifies the QC sample whose parameters are displayed.

After Initial Calib

The QC will be analyzed after the initial calibration has been performed.

Note	If you schedule one or more **After Initial Calib QC** samples and then perform an Automated Analysis using the **Analyze Samples** button, the instrument will analyze the QC as part of the sample list. This is useful if you wish to use an existing calibration curve, analyze the **After Initial Calib QC** sample as a check (without reanalyzing your calibration standards), then take action based on the pass or fail status of the QC.

After Recalibration

The QC will be analyzed after any recalibrations that are part of the analysis.

Periodically

The QC will be analyzed at specified intervals during the current analysis, based on the frequency you select.

Frequency
This is the frequency at which the periodic QC samples are analyzed. For example, if you select a frequency value of 20, then every 20 samples the QC sample will be analyzed.

If you use the same frequency for all periodic QC samples, the values in the Frequency row in the table will have a grey background and cannot be changed.

If you select **Variable** for the frequency, the values displayed in the table will have a white background. You may then enter different frequency values for each QC sample.

At End

The QCs will be analyzed at the end of the current analysis.

Periodic Timing of Analyses

Frequency

This is the frequency at which QC samples are analyzed. You can use the same frequency for all periodic QC samples, or vary the frequency for each QC sample.

Maximum time between QC's

This is only available when **Same for All QC's** option has been selected for the frequency.

To specify a maximum amount of time between periodic QC samples, click on the check box, then select the time.

Method Editor QC Pages: Failure Actions for After-Calibration and Periodic QC's Page

You use this page to define the actions to be taken if any of the QC samples that are analyzed after a calibration or are analyzed periodically fail. You may select from one of several actions.

Detail of the Failure Actions for After-Calibration and Periodic QC's page showing entries included in an example method prepared for a soils analysis.

Failure Actions for After-Calibration QC's

	QC 1	QC 2	
QC Sample ID	Mid QC	Hg Mid QC	
Times to retry QC	None	None	
When All Tries Fail	Recal & Cont	Recal & Cont	
Additional Message			

Failure Actions for Periodic QC's

	QC 1	QC 2	
QC Sample ID	Mid QC	Hg Mid QC	
Times to retry QC	None	None	
When All Tries Fail	Recal & Rerun	Recal & Rerun	
Additional Message			

Failure Actions for After-Calibration QC's

QC Sample ID

This view-only row shows the name that you assigned to each individual QC sample.

Times to retry QC

Select an option from the drop-down list. If it fails initially, the QC sample is reanalyzed up to the specified number of times. If it passes at any one time, the analysis continues. If all tries fail, the failure action will be performed. If you select None from the drop-down list, the QC will not be reanalyzed. Instead, the system will go directly to performing the failure action.

When All Tries Fail

Select a failure action from the drop-down list:

Note If there are any after-calibration QCs, they will be analyzed after the recalibration action. If the QCs pass, then the rest of the action is performed (continue or rerun samples).

Continue
The system prints a failure message and any optional message that you typed and continues with the analysis.

Recal & Cont
The system prints a failure message and any optional message that you typed. The system performs a complete recalibration as defined on the Calibration page including the reagent blank if selected, then continues with the analysis.

Rslp & Cont
The system prints a failure message and any optional message that you typed. The system analyzes the calibration blank and reslope standard as defined on the Calibration page including the reagent blank if selected, then continues with the analysis.

AZ & Cont
The system prints a failure message and any optional message that you typed. The system performs an autozero using the calibration blank and the reagent blank (if you are using one), then continues with the analysis. An autozero sets the reading of the calibration blank or reagent blank to zero automatically.

Alarm and Pause
The system prints a failure message and any optional message that you typed. An alarm is activated and the analysis pauses. At this point, you will get an

option to stop or continue the analysis. If an automatic shutdown is scheduled, Alarm and Pause will prevent system shutdown.

Next Method
The system prints a failure message and any optional message that you typed. The current method is stopped and the system proceeds to the next method, if any. If there is no next method, the system stops the analysis. If an automatic shutdown is scheduled, Next Method will prevent system shutdown.

Stop
The system prints a failure message and any optional message that you typed. The system stops the analysis. If an automatic shutdown is scheduled, Stop will prevent system shutdown.

Additional Message

The system will automatically print standard messages indicating what is happening if the measured QC concentration does not fall within the range specified by the limits.

You can type an optional message up to 60 characters in length in this entry field that will be printed when a failure action is performed in addition to the standard message.

Failure Actions for Periodic QC's

QC Sample ID

This view-only row shows the name that you assigned to each individual QC sample.

Times to retry QC

Select an option from the drop-down list. If it fails initially, the QC sample is reanalyzed up to the specified number of times. If it passes at any one time, the analysis continues. If all tries fail, the failure action will be performed. If you select None from the drop-down list, the QC will not be reanalyzed. Instead, the system will go directly to performing the failure action.

When All Tries Fail

Select a failure action from the drop-down list:

Note If there are any after-calibration QCs, they will be analyzed after the recalibration action. If the QCs pass, then the rest of the action is performed (continue or rerun samples).

Continue

The system prints a failure message and any optional message that you typed and continues with the analysis.

Recal & Cont

The system prints a failure message and any optional message that you typed. The system performs a complete recalibration as defined on the Calibration page including the reagent blank if selected, then continues with the analysis.

Rslp & Cont

The system prints a failure message and any optional message that you typed. The system analyzes the calibration blank and reslope standard as defined on the Calibration page including the reagent blank if selected, then continues with the analysis.

AZ & Cont

The system prints a failure message and any optional message that you typed. The system performs an autozero using the calibration blank and the reagent blank if selected, then continues with the analysis. An autozero sets the reading of the calibration blank or reagent blank to zero automatically.

Alarm and Pause

The system prints a failure message and any optional message that you typed. An alarm is activated and the analysis pauses. At this point, you will get an option to stop or continue the analysis. If an automatic shutdown is scheduled, Alarm and Pause will prevent system shutdown.

Recal & Rerun

The system prints a failure message and any optional message that you typed. The system performs a complete recalibration as defined on the Calibration page including the reagent blank if selected. Then, the samples are rerun, starting after the last QC that passed.

Reslp & Rerun

The system prints a failure message and any optional message that you typed.

The system analyzes the calibration blank and reslope standard as defined on the Calibration page including the reagent blank if selected. Then, the samples are rerun, starting after the last QC that passed.

AZ and Rerun
The system prints a failure message and any optional message that you typed. An autozero is performed using the calibration blank and the reagent blank if selected. Then, the samples are rerun, starting after the last QC that passed. An autozero sets the reading of the calibration blank or reagent blank to zero automatically.

Next Method
The system prints a failure message and any optional message that you typed. The current method is stopped and the system proceeds to the next method, if any. If there is not a next method, the system stops the analysis. If an automatic shutdown is scheduled, Next Method will prevent system shutdown.

Stop
The system prints a failure message and any optional message that you typed. The system stops the analysis. If an automatic shutdown is scheduled, Stop will prevent system shutdown.

Additional Message

The system will automatically print standard messages indicating what is happening if the measured QC concentration does not fall within the range specified by the limits.

You can type an optional message in this entry field that will be printed when a failure action is performed in addition to the standard message.

Method Editor QC Pages: Failure Actions for At-End QC's

You use this page to define actions to be taken for QC's run at the end of an analysis. You select the number of times to retry the QC sample before a failure action is performed. When the At-End QC samples pass, the analysis ends. If all tries fail, or if no retries are scheduled, then the failure action that you select is performed immediately.

Detail of the Failure Actions for At-End QC's showing entries included in an example method prepared for a soils analysis.

Failure Actions for At-End QC's

	QC 1	QC 2	
QC Sample ID	Mid QC	Hg Mid QC	
Times to retry QC	None	None	
When All Tries Fail	Recal & Rerun	Recal & Rerun	
Additional Message			

Maximum Retries After QC Failure

After a group of standards or unknowns has been

reanalyzed [1] [⇅] times, then [Continue ▼]

Failure Actions for At-End QC's

QC Sample ID

This view-only row shows the name that you assigned to each individual QC sample.

Times to retry QC

Select an option from the drop-down list. If it fails initially, the QC sample is reanalyzed up to the specified number of times. If it passes at any one time, the analysis continues. If all tries fail, the failure action will be performed. If you select None from the drop-down list, the QC will not be reanalyzed. Instead, the system will go directly to performing the failure action.

When All Tries Fail

Select an failure action from the drop-down list.

Note   If there are any after-calibration QCs, they will be analyzed after the recalibration action. If the QCs pass, then the rest of the action is performed (continue or rerun samples).

Continue
The system prints a failure message and any optional message that you typed and continues with the analysis.

Recal & Rerun
The system prints a failure message and any optional message that you typed. The system performs a complete recalibration as defined on the Calibration page including the reagent blank if selected. Then, the samples are rerun, starting after the last QC that passed.

Rslp & Rerun
The system prints a failure message and any optional message that you typed. The system analyzes the calibration blank and reslope standard as defined on the Calibration page including the reagent blank if selected. Then, the samples are rerun, starting after the last QC that passed.

AZ & Rerun
The system prints a failure message and any optional message that you typed. An autozero is performed using the calibration blank and the reagent blank if selected. Then, the samples are rerun, starting after the last QC that passed. An autozero sets the reading of the calibration blank or reagent blank to zero automatically.

Alarm and Pause
The system prints a failure message and any optional message that you typed. An alarm is activated and the analysis pauses. At this point, you will get an option to stop or continue the analysis. If an automatic shutdown is scheduled, Alarm and Pause will prevent system shutdown.

Next Method
The system prints a failure message and any optional message that you typed. The current method is stopped and the system proceeds to the next method, if any. If there is no next method, the system stops the analysis.

Stop
The system prints a failure message and any optional message that you typed. The system stops the analysis. If an automatic shutdown is scheduled, Stop will prevent system shutdown.

Additional Message

The system will automatically print standard messages indicating what is happening if the measured QC concentration does not fall within the range specified by the limits.

You can type an optional message up to 60 characters in length in this entry field that will be printed when a failure action is performed in addition to the standard message.

Maximum Retries After QC Failure

You use this parameter to select the maximum number of reanalyses of samples before an action is taken. You select the action to be taken when the number of times you run the sample is equal to the maximum number of retries. See *Flow Chart 4: An Example of a Failure Action in QC Flow Charts* earlier in this chapter.

1. Type a value for the maximum number of retries that you want the system to perform.

2. Select an option from the drop-down list.

    Next Method
    The system prints a failure message and any optional message that you typed. The current method is stopped and the system proceeds to the next method, if any. If there is no next method, the system stops the analysis. If an automatic shutdown is scheduled, Next Method will prevent system shutdown.

    Continue
    The system continues with the next unknown sample as if no failure occurred.

    Stop
    The system prints a failure message and any optional message that you typed. The system stops the analysis. If an automatic shutdown is scheduled, Stop will prevent system shutdown.

    Alarm and Pause
    The system prints a failure message and any optional message that you typed. An alarm is activated and the analysis pauses. At this point, you will get an option to stop or continue the analysis. If an automatic shutdown is scheduled, Alarm and Pause will prevent system shutdown.

Method Editor: Options Page

You use the entries on the Options page to set up your Results Display and Printed Log. You also set some options for information to be stored with a Results Data Set for an analysis.

Detail of the Options page showing entries included in an example method pre-pared for a soils analysis.
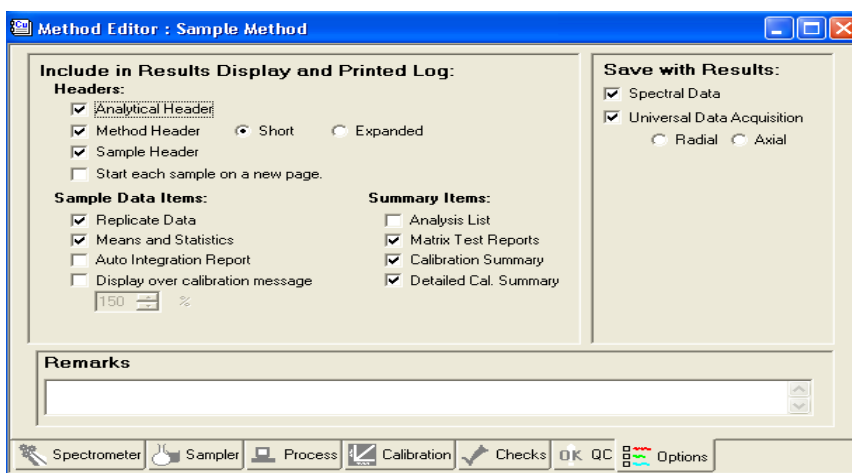
Headers to include in Results Displays and Printed Log:

Select the type of information you wish to include in the Results Display and the Printed Log for your analysis. For detailed descriptions of the following headers, see *Results Display Window* in Chapter 7, *Information Display Windows*.

Analytical Header
Select this to include a header when a new analysis is begun. This header includes the analysis start time, technique, the sample information file name, the results data set name, the results library name, and other general information. Each time the current method is modified and another sample is analyzed, a new analytical header is displayed.

Method Header
Select this to include a header that contains the name of the method, the method description, the date the method was last saved, and names of IEC and MSF files. The expanded header includes information from the method on the calibration equation, peak processing, plasma viewing, and spectral corrections. You can select from a **Short** or **Expanded** method header.

Sample Header
Select this to include a header with sample information, including sample ID, sample weight, dilution, and sample prep volume. You have the option to start each sample on a new page.

Sample Data Items:

Select the type of information you wish to include in the Results Display and the Printed Log for your analysis.

Auto Integration Report
Select this option to include an Auto-Integration Report for each measurement when auto integration is being used. Information includes the integration time, number of integrations and read time.

Replicate Data
Select this option to include data for each replicate.

Means and Statistics
Select this option to include the mean values of each set of replicates, the standard deviation, and the relative standard deviation.

Analysis List
Select this option to include a summary of the operations that were performed on all of the samples.

Calibration Summary (Auto Analysis only)
Select this option to include a calibration summary in the Results Display window. If this is selected, the calibration summary is automatically included for automated analyses after the last standard is analyzed. **Note:** For manual analyses, you must select the Calibration Summary command in the Analysis menu.

Sample Data Items:

Select the type of information you wish to include in the Results Display and the Printed Log for your analysis.

Replicate Data
Select this option to include data for each replicate.

Means and Statistics
Select this option to include the mean values of each set of replicates, the standard deviation, and the relative standard deviation.

Auto Integration Report
Select this option to include an Auto-Integration Report for each measurement when auto integration is being used. Information includes the integration time, number of integrations and read time.

Note: In the FIAS technique, Auto Integration Report is not displayed.

Display over calibration message
Select this option to display an over calibration message when the sample concentration is greater than the entered percentage of the highest standard. When this option is checked the % spin box is enabled. In the % spin box enter a value between 100 and 999 %.

Summary Items:

Analysis List
Select this option to include a summary of the operations that were performed on all of the samples.

Matrix Test Reports
Select this option to include information about matrix check samples (duplicates, recovery samples and spikes) analyzed.

Calibration Summary (Auto Analysis only)
Select this option to include a calibration summary in the Results Display window. If this is selected, the calibration summary is automatically included for automated analyses after the last standard is analyzed.

Note: For manual analyses, you must select the Calibration Summary command in the Analysis menu.

Detailed Cal. Summary
Select this option to display a detailed summary of each calibration curve, including the calculated concentration of each standard. This summary will be displayed after the final standard has been run.

Save with Results

Spectral Data
Select this to include the spectral data in the results data set. The spectral data must be stored in a results data set if it is to be displayed in the Examine Spectra window or used for setting up MSF models or Data Reprocessing.

Universal Data Acquisition
Select this option to run in Universal Data Acquisition (UDA) mode. When this option is checked, the Optima records all of the emission spectra for every sample, regardless of the elements being determined. This UDA capability allows you to retroactively use data that was not in the original method to determine additional elements or measure at alternative wavelengths.

Note: If you have a dual view method, then the Radial and Axial radio buttons will become enabled when Universal Data Acquisition is checked. You can then select the Radial radio button to collect Universal Data in radial viewing mode, or you can select the Axial radio button to collect Universal Data in axial viewing mode.

Remarks

Use this entry field to type your comments regarding the method.
(duplicates, recovery samples and spikes) analyzed.

Universal Data Acquisition (UDA)

Universal Data Acquisition (UDA) is an option available on the Options tab page of the Method Editor. When this option is checked, the Optima records all of the emission spectra for every sample, regardless of the elements being determined. This UDA capability allows you to retroactively use data that was not in the original method to determine additional elements or measure at alternative wavelengths.

Enabling Universal Data Acquisition Mode

This section shows you how to record all of the spectral data for each sample.

Note: When Universal Data Acquisition mode is enabled all of the spectral data is recorded behind the scenes and only the data for elements defined in your method are reported. If, at a later time, you wish to determine additional elements or measure at alternative wavelengths, you can select the UDA data from a results data set and reprocess the data with a method that contains the elements you want to measure. For more information on reprocessing UDA data see Reprocessing Universal Data.

To set up a method with Universal Data Acquisition enabled:

1. Open the Method Editor and set up a method to suit your analysis. When setting up your method, make sure that steps 2-5 are followed, so that the emission spectra for every sample are recorded.

2. From the **Method Editor Options** tab page, check **Spectral Data**.

3. Check **Universal Data Acquisition**.

Note: If you have a dual view method, then the Radial and Axial radio buttons will become enabled when Universal Data Acquisition is checked. You can then select the Radial radio button to collect Universal Data in radial viewing mode, or you can select the Axial radio button to collect Universal Data in axial viewing mode.

4. Click on the **Spectrometer** tab **> Settings** tab.
   The Spectrometer Settings are displayed.

5. On the **Settings** tab, make sure that **Resolution** is set to **Fixed** and **Time(sec)** is set to **Auto**.

6. Set up the rest of your method as required for your analysis and then save your method.

7. Analyze your samples.
   All of the spectral data is captured in the data set you specified and is available for reporting via the Reprocess command. For more information on reprocessing UDA data, refer to the next section of this manual.

Note: You cannot perform an analysis with a method that has all three of the following parameters enabled: Spectral Profiling, High Resolution, and Universal Data Acquisition. At least one of these parameters must be unchecked in order for your method to be valid. For example, you can perform an analysis using a method that has Spectral Profiling and Universal Data Acquisition enabled.

Reprocessing UDA Data

If you collected data with Universal Data Acquisition enabled, you have the ability to retroactively use data that was not in your original method to determine additional elements or measure at alternate wavelengths. The steps outlined below show you how to reprocess UDA data.

To reprocess UDA data:

1. On the Toolbar click on the **Reprocessing** icon.
    OR
    From the **Tools** menu, click on **Data Reprocessing**.
    The Data Reprocessing window is displayed.

2. Next to **Data Set to Reprocess**, click on **Browse**... and select a results data set that containst the data you want to reprocess.
    Results data sets that contain Universal Data are marked with a > in the row number.

In the Data Reprocessing window, the results from the reprocessed data will be saved to the data set shown in the **Reprocessed Data Set** field. This field defaults to the data set from the most recent analysis. A cehck mark appears in the **Save Reprocessed Data** box, confirming that the reprocessed data will be saved. If you do not want to store reprocessed data, uncheck Save Reprocessed Data.

3. To change to a different data set, click on **Browse...** (next to Reprocessed Data Set).
   The Select Results Data Set dialog appears.

4. Type in a new data set name or select an existing one and then click **OK**.

5. Open the method that you want to use for reprocessing.

6. Confirm that hte method in use contains the correct settings for the results data set that is to be reprocessed.

7. In the **Data Reprocessing** table, review the information. The IDs in the selected results data set are listed along with the sample information that was used when the data was orginally collected.

8. To edit this information, type the new information into the fields as desired. To access a pop-up menu containing commands for editing the table, right click anywehre in the table.

Note: There is a column fill dialog available for each of the parameters in teh table, except Sample Type and Original method. To access a column fill dialog, doulbe-click on the parameter's column header.

9. To hide columns that do not contain data, check **Hide Empty Columns**.

10. To only display samples with UDA data, check Only show samples with Universal Data.

11. To select items of interest for reprocessing, select the associated row numbers in the table. To select a series of rows, click on the first row number of interest, hold down the Shift key, and click on the last row number of interest. YOu can also drag over the row numbers column using hte mouse cursor. To select non-consecutive rows, hold down the Ctrl key and click on each row number.

Tip: The order in which samples are selected is the order in which reprocessing occurs. As you select each row, the reprocessing sequence appears in the Sequence column.

12. Click on **Reprocess**.

Sample Information Editor 4

About the Sample Information Editor

We recommend that you use a sample information file to store information about the samples. The system uses this file to label the data from your samples both on the printed log of the results and in the results data set. Information in this file is also often needed by the system to calculate final concentrations.

Enter IDs only for samples and matrix check samples in the sample information file. Enter IDs for the blanks, standards, and QC samples in the Method Editor. If you enter IDs for blanks, standards, or QC samples in the Sample Information Editor, these solutions are analyzed as samples.

The Sample Information Editor showing entries for a soils analysis with solid sample units.

Sample Information Editor : Soils

Parameters Common to All Samples

Batch ID	
Volume Units	mL
Weight Units	g

File Description

For Solid Sample Units

*Parameters marked with an asterisk override settings in the method.

Parameters That Vary By Sample   Append to Analysis List

Sample No	A/S Location	Sample ID	Initial Sample Wt.	Sample Prep. Vol.	Aliquot Volume	Diluted To Vol.
1	6	Site723M	1.987	100		
2	7	Site720Z	2.025	100		
3	8	Site727X2	1.976	100		
4	9	Site727X3	2.056	100		
5						
6						
7						
8						
9						
10						

To display this window

► On the **Toolbar**, click on **SamInfo**.

- or -

► In the **Tools** menu, click on **Sample Information Editor**.

File format

The sample information file is written in comma-delimited ASCII, a format that you can also create using other software such as a BASIC program, a spreadsheet program, or a database program. You can also create this file on a laboratory information management system and load it into your instrument control computer to perform analyses.

Using the Sample Information Editor

Customizing the Sample Information Editor

You can select the exact parameters that you need to describe your samples and add them to the Sample Information Editor. You select the sample description, preparation, and scheduling parameters from a list of available parameters.

For the sample description and sample preparation parameters, you decide whether the information will be the same for all samples or will vary for particular samples:

•   If the Information you enter will be the same for all samples, you assign the parameter to the **Parameters Common to All Samples** table.

•   If the information you enter will vary for individual samples, you assign the parameter to the **Parameters That Vary By Sample** table.

In addition, you can define your own parameters by selecting **User Defined** in the list of parameters.

Once you have included all the parameters that you need, you can save this configuration as a sample information design. In the File menu, select **Save As ► Samp Info Design** to save as a .sid file. By saving the design, you can reuse it later for similar sample information files.

Editing Information in the Sample Information Editor

To edit information about the samples or to remove the assigned parameters from either table, you use commands in the Edit menu. In the Parameters That Vary By Sample table, you can click with the right mouse button in the table and a pop-up menu appears. This pop-up menu contains commands similar to those found in the Edit menu.

Appending Samples to an Automated Analysis

You can modify and append samples to an automated analysis.

1. While the instrument is performing an automated analysis, in the Sample Information Editor modify the information in samples as desired, then click on the **Append to Analysis List** button.

2. When the Append to Run List dialog appears, type the range of samples that you want to append and click on **OK** to add the samples to the end of the run list in the Automated Analysis Control window.

Note You can drag the mouse cursor through a range of samples in the Sample Information Editor and, when you click on the Append to Analysis List button and the Append to Run List dialog appears, the range of the selected samples already appears in the dialog.

To delete, copy, or rename sample information files or sample information designs

▶ Use the Windows Explorer. These files are stored in the pe\username\sample information directory, where *username* will be replaced automatically with the name that you used to log in to the computer. For example, if your login name is "smith," the directory name will be pe\smith\sample information. For more information, see your Windows documentation.

Creating a New Sample Information File

To create a new sample information file

1. In the **File** menu, click on **New** ▶ **Sample Info File...** The New Sample Information File dialog appears.

2. Select a design from the Design List and click **OK**. This list includes a default design and any other designs that have been previously created and stored. To create a name for the sample information file, in the **File** menu, click on **Save** ▶ **Sample Info File**. Type a name for the new sample information file, then click on **Save**.

3. You can customize the Sample Information Editor by selecting the parameters you need to describe your samples. See *Customizing the Sample Information Editor* below.

4. Enter the sample information. For example, autosampler locations, Sample ID, Initial Sample Weight, etc.

5. If desired, annotate the file using the File Description field to provide further descriptive information. When you move the mouse cursor to this text area and click with the right mouse button, a pop-up menu of editing commands appears.

6. In the **File** menu, click on **Save As** ▶ **Sample Info File...** The Save As dialog appears. Type a name for the file, then click on **OK**.

Customizing the Sample Information Editor

To fully describe your samples, you can add or remove parameters in the Sample Information Editor and then save the configuration as a sample information design to use again.

To add parameters to the Sample Information Editor

1. If you have not created a name for the new sample info file, do the following:

   • In the **File** menu, click on **New** ▶ **Sample Info File...** The New Sample Information File dialog appears.

   • Select a design from the Design List and click **OK**. This list includes a default design and any other designs that have been previously created and stored. To create a name for the sample information file, in the **File** menu,

click on **Save** ► **Sample Info File**. Type a name for the new sample information file, then click on **Save**.

2. In the **Edit** menu, click on **Parameter List...**

- or -

Click with the right mouse button anywhere on the background area in the Sample Information Editor. In the pop-up that appears, select **Parameter List...**

The Sample Information Parameters dialog appears.

3. In the **Available** list, click on a parameter to select it. (To deselect the parameter, click again.) The parameters are shown in alphabetical order.

4. Click on the arrow button that points to the list where you wish to move the selected parameter. Parameters marked with an asterisk (*)override settings in the method.

   ► If the entry for the parameter is common to all samples, move the parameter to the **Common** list.

   ► If the entry for the parameter varies for each sample, move the parameter to the **Variable** list. For example, the analyst name might be the same for all samples, while the sample IDs would vary for each sample.

      The parameter is removed from the **Available** list and appears in the list you chose.

   ► To change your selections, click on a parameter in the **Common** list or in the **Variable** list, then click on an arrow pointing back to the **Available** list.

      The parameter is returned to the **Available** list.

5. Select additional parameters, if desired, and move them to the appropriate lists. When you are finished, click on **OK**.

In the Sample Information Editor, use the scroll bars to see the changes you made. Parameters that you added to the **Common** list appear in the **Common Parameters** table, and those added to the **Variable** list appear in the **Parameters That Vary by Sample** table.

To remove parameters from the Sample Information Editor

To remove parameters from the Parameters Common to All Samples table:

▶ Click with the right mouse button anywhere on the background area in the Sample Information Editor. In the pop-up that appears, select **Parameter List...** Click on a parameter in the **Common** list, then click on an arrow pointing back to the **Available** list.

To remove parameters from the Parameters That Vary By Sample table:

▶ Click with the right mouse button anywhere on the background area in the Sample Information Editor. In the pop-up that appears, select **Parameter List...** Click on a parameter in the **Variable** list, then click on an arrow pointing back to the **Available** list.

To save the sample information design

1. In the **File** menu, click on **Save As** ▶ **Sample Info Design...** The Save As dialog appears.

2. Type a name for the design, then click on **OK**.

Opening a Stored Sample Information File

1. In the File menu, click on **Open** ▶ **Sample Info File...**. The Open Sample Information dialog appears.

2. Select the sample information file that you want to open, then click on **Open**.

Modifying a Stored Sample Information File

1. In the File menu, click on **Open** ▶ **Sample Info File...**

2.   Select the sample information file that you want to open, then click on **Open**.

3.   You can customize the Sample Information Editor by selecting the parameters you need to describe your samples. See *Customizing the Sample Information Editor* earlier in this chapter.

4.   Change the sample information as desired.

5.   Change the information in the File Description field as desired, using the pop-up menu for editing commands.

6.   Save the file:

▶ To save the file with the same name, click on **Save** ▶ **Sample Info File** in the **File** menu.

▶ To save the file with a new name, click on **Save As** ▶ **Sample Info File...** in the **File** menu. The Save As dialog appears. Type a name for the file, then click on **Save**.

Note   If you modify a sample information file and then attempt to create a new one, a message appears asking if you wish to save the changes to the first sample information file. This also happens when you attempt to exit the WinLab32 software.

Printing a Sample Information File

To print the contents of a sample information file:

1. Click on the Sample Information Editor to make it active. Note that the title bar of an active window is a different color from other windows. (The colors that appear on the screen depend on the colors you have selected in your Windows® application.)

2. In the **File** menu, click on **Print Active ▶ Window**.

3. In the Print dialog that appears, check that the correct printer is shown. To select a different printer, click on the drop-down arrow in the **File Name** field and make a selection. To make other changes, such as paper size or graphic attributes, click on the **Properties** button.

4. Click on **OK** to start printing.

Editing Sample Information Parameters

You can edit the entry fields and add or delete rows in the Parameters that Vary by Sample table.

To clear information in the table

When you clear information, the content in an entry field is removed, not the entry field itself.

1. Click on the entry field that contains the information you want to remove.

2. In the **Edit** menu, click on **Clear**.

   - or -

   Click with the right mouse button. A pop-up menu appears. Click on **Clear**.

   - or -

   Press the Delete (**Del**) key.

To insert rows in the table

When you insert rows, the new rows are inserted before the first row you select in the table.

1. Select a row in the table by clicking on a number in the Sample Number column of the table. The entry fields in the row become highlighted to indicate that the row is selected.

2. If you want to insert more than one row, you select the same number of rows in the table. For example, to insert three new rows, you would select three existing rows. To select additional rows, hold down the Shift key while clicking on additional row numbers.

3. In the **Edit** menu, click on **Insert Rows**.

    - or -

    Click with the right mouse button. A pop-up menu appears. Click on **Insert Row**.

To delete rows in the table

When you delete rows, the contents of the row and all the entry fields in the row are removed.

1. Select a row in the table by clicking on a number in the Sample Number column of the table. The entry fields in the row are highlighted to indicate that the row is selected.

2. To select additional rows, hold down the Shift key while clicking on additional row numbers.

3. In the **Edit** menu, click on **Delete Rows**.

    - or -

    Hold down the right mouse button. A pop-up menu appears. Click on D**elete Rows**.

Sample Information Editor Pop-Up Menus

The following pop-up menus contain several convenient commands as you work with the Sample Information Editor.

To display the pop-up menus

▶ Click with the right mouse button in the Sample Information Editor.

> ▶ When you click with the right mouse button in the Parameters that Vary by Sample table, a pop-up menu containing editing commands for the table appears.

> ▶ When you click with the right mouse button anywhere on the background area in the window, the Sample Information Editor pop-up menu appears.

Sample Information Editor pop-up menu
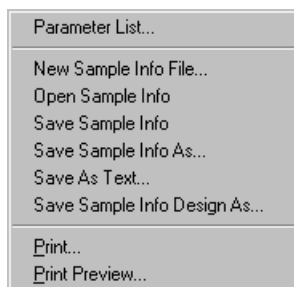
Command	Description
Parameter List...	Adds new parameters to the Sample Information Editor.
New Sample Info File...	Selects a design and a creates a name for the new sample information file.
Open Sample Info	Opens a stored sample information file.

Command	Description
Save Sample Info	Saves the contents of the Sample Information Editor using the same file name.
Save Sample Info As...	Saves the contents of the Sample Information Editor using a new file name.
Save As Text...	Saves the contents of the Sample Information Editor to a text file or comma-delimited ASCII file.
Save Sample Info Design As...	Saves the configuration of the Sample Information Editor as a design file. You can use a design file to quickly customize the Sample Information Editor with the parameters you need to describe your samples.
Print...	Prints the contents of the Sample Information Editor.
Print Preview...	Displays the formatted sample information report in a preview window. Use the Zoom In and Zoom Out buttons to enlarge or reduce the report. Click on **Close** to return to the Sample Information Editor.

Table editing pop-up menu
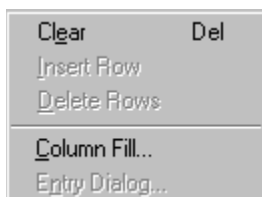
```
Clear          Del
Insert Row
Delete Rows
Column Fill...
Entry Dialog...
```

Command	Description
Clear	Removes the contents of the selected entry fields.
Insert Row	Inserts a new row before the currently selected row.
Delete Rows	Removes the selected rows in the table.

Command	Description
Column Fill...	Displays the Column Fill dialog for the selected column. You use this dialog to automatically enter information for a range of samples.
Entry Dialog...	Displays the Entry dialog for the selected entry field. You use this dialog to type information for a text entry field.

Sample Information Parameters and Dialogs

Sample Information parameters

The parameters in the Sample Information Editor fall into three different categories:

Parameter	Description
Sample Description parameters	These parameters include the sample and batch names used to identify the sample, the autosampler location, and other optional information for describing the sample.
Sample Preparation parameters	These parameters include information about the way in which the sample was prepared and the units in which the final sample concentrations will be reported.
Analysis Schedule parameters	These parameters are used to schedule QC's, periodic recalibration, and matrix check samples. You can also select a Read Delay or Wash Time for a sample that differs from the method.

Sample Information dialogs

Aliquot Volume Column Fill Dialog
Analyze QC's Before Column Fill Dialog
Autosampler Location Column Fill Dialog
Diluted to Volume Column Fill Dialog
Initial Sample Volume Column Fill Dialog
Initial Sample Weight Column Fill Dialog
Matrix Check Sample Entry Dialog
Remarks Entry Dialog
Sample ID Column Fill Dialog
Sample Information Editor Column Fill Dialogs
Sample Information Parameters Dialog
Sample Units Column Fill Dialog
Solids Ratio Column Fill Dialog
Units Column Fill Dialogs
User Defined Entry Field Dialog

Sample Description Parameters

Batch ID

This is the name you give to this batch of samples.

▶ Type up to 25 characters for each sample. You can use any combination of letters and numbers.

- or -

▶ To automatically enter a sequence of IDs, double-click on an entry field and use the Column Fill dialog.

Sample ID

This is the name you give to each sample.

▶ Type up to 25 characters for each sample. You can use any combination of letters and numbers.

- or -

▶ To automatically enter a sequence of IDs, double-click on an entry field and use the Sample ID Column Fill dialog.

Remarks

Click on the entry field. The Remarks Entry dialog appears, in which you can type up to 50 characters.

Autosampler Location

This is the location of the solution in the autosampler tray.

▶ Type the location for each solution.

- or -

▶ To automatically enter the locations for a sequence of samples, double-click on an entry field and use the Autosampler Location Column Fill dialog.

Analyst Name

This is the name of the person setting up or performing the analysis.

▶ Type up to 20 characters for each sample. You can use any combination of letters and numbers.

- or -

▶ To automatically enter a sequence of names, double-click on an entry field and use the Column Fill dialog. Note: If the name is the same for all samples, assign this parameter to the **Parameters Common to All Samples** table.

User Defined...

Use this parameter to define your own parameter. For example, you may want to record an account name for the batch of samples. You can customize up to five sample information parameters. Select User Defined... in the list of parameters, then type a name for the parameter using up to 20 characters in the User Defined Entry field dialog.

Sample Preparation Parameters

Use the following guidelines when entering information about sample preparation in the Sample Information Editor.

		Sample Preparation Parameters		
If your Calibration Units are...	**And your Sample Units are...**	**Initial Sample Weight and Sample Prep Vol.**	**Initial Sample Vol. and Sample Prep Vol.**	**Aliquot Volume and Diluted to Volume.**
Wt/Vol	Wt/Wt	Required *		Optional (but must enter both)
Wt/Vol	Wt/Vol		Enter both or omit both*	Optional (but must enter both)
Wt/Wt	Wt/Wt	Not used in conversion	Not used in conversion	Optional (but must enter both)
Wt/Wt	Wt/Vol	The software will not convert Wt/Wt calibration units to Wt/Vol sample units. The software will only report the sample concentration in calibration units, not sample units.		

Notes * Without the required entries, the software will only report the sample concentration in calibration units, not sample units. If both entries are required and you only enter one, again, the concentration in sample units will not be reported.

If you enter the Aliquot Volume, you must also enter the Diluted to Volume for correct reporting of results.

You select calibration units and sample units in the Method Editor on the Calibration Units and Concentrations page. Or, you can select sample units in the Sample Information Editor. If the sample units in the Sample Information Editor differ from those in the Method Editor, the sample units in the Sample Information Editor will be used.

Sample Preparation Parameters

The Sample Preparation parameters are shown below. For an example using Initial Sample Weight, Sample Prep Volume, Aliquot Volume, and Diluted to Volume see, *Sample Preparation: An Example* later in this chapter.

Aliquot Volume

If an aliquot of the sample solution is taken and diluted to a final volume, record the aliquot volume for this entry and the final volume for the Diluted to Volume entry.

Enter the volume using the units selected for the Volume Units. To automatically enter the same value for a sequence of samples, double-click on an entry field and use the Aliquot Volume Column Fill dialog.

Diluted to Volume

This is the final volume of solution obtained by diluting an aliquot of the sample solution.

You can also record the dilution as the ratio of the original sample volume to the final sample volume. For example, if 10 mL of sample is diluted to 200 mL, this

ratio would be 10:200 or 1:20. Enter 20 for the Diluted to Volume entry and 1 for the Aliquot volume.

Enter the volume using the units selected for the Volume Units. To automatically enter the same value for a sequence of samples, double-click on an entry field and use the Diluted to Volume Column Fill dialog.

Solids Ratio

This is the ratio of the wet and dry weights for the sample. This ratio is used to correct the sample concentration. For more information, see the *Solids Ratio Column Fill dialog* later in this chapter.

Initial Sample Weight

If you weigh the sample during sample preparation, you must enter the Initial Sample Weight and the Sample Prep Volume.

Enter the weight using the units selected for the Weight Units. To automatically enter the same value for a sequence of samples, double-click on an entry field and use the Initial Sample Weight Column Fill dialog.

Initial Sample Volume

If you measure the sample volumetrically during sample preparation, you can enter the Initial Sample Volume and the Sample Prep Volume.

Enter the volume using the units selected for the Volume Units. To automatically enter the same value for a sequence of samples, double-click on an entry field and use the Initial Sample Volume Column Fill dialog.

Weight Units

Select the units for the weight value(s) you enter for each individual sample or for all samples. These weight units are used for the Initial Sample Weight and the Nominal Sample Weight entries.

To automatically enter the same value for a sequence of samples, double-click on the column header and use the Units Column Fill dialog.

Volume Units

Select the units for the volumes you enter for each individual sample or for all samples. These weight units are used for the Initial Sample Volume, the Sample Prep Volume, the Aliquot Volume, and the Diluted to Volume entries.

To automatically enter the same value for a sequence of samples, double-click on the column header and use the Units Column Fill dialog.

Sample Prep Volume

After a solid sample has been dissolved or a liquid sample has been acidified (or otherwise treated), the resulting solution is prepared to a specific volume. This is the Sample Prep Volume.

Enter the volume using the units selected for the Volume Units. To automatically enter the same value for a sequence of samples, double-click on an entry field and use the Column Fill dialog.

Nominal Sample Weight

This is the target weight when samples are weighed. This entry is required only if corrections are being made to weight/volume measurements to compensate for weight variations among samples. The Nominal Sample Weight is divided by the Initial Sample Weight to correct the final concentration value.

Enter the weight using the units selected for the Weight Units. To automatically enter the same value for a sequence of samples, double-click on an entry field and use the Column Fill dialog.

Sample Units

These are the units used to report the concentrations of the samples. If the sample units in the Sample Information Editor differ from those in the Method Editor, the sample units in the Sample Information Editor will be used. You can select different sample units for different samples in the Sample Information Editor, but concentrations for all elements will be reported using the same units.

To automatically enter the same value for a sequence of samples, double-click on the column header and use the Sample Units Column Fill dialog.

To select your own units

The units available in the drop-down lists are stored in an ASCII file called UNITS.INI located in the WinLab32 directory. You can use any text editor to add units to the file. You may also rearrange the units in the file so that those used frequently are near the top of the list.

To modify the UNITS.INI file.

1. In Windows operating system, start the Windows Explorer.

2. Display the WinLab32 directory.

3. Double-click on the **units.ini** file.
   The Notepad application is automatically started and the UNITS.INI file is opened.

   The UNITS.INI file contains four sections: volume units [Vol], weight units [Wt], weight/volume units [Wt/Vol] and weight\weight [Wt/Wt] units. The first unit in each section is the base unit and has a conversion factor of 1.0. **Do not change or move the base unit.**

4. Add the units and the conversion factor to the appropriate section of the file using the following format.

   Units12=new_units,conversion factor

   The conversion factor is multiplied by the value in new units to convert it to a value in base units. For example, to convert milliliters to the base unit of liters, 1 mL = 1.0E-3 L, so the conversion factor is 1.0E-3.

5. In the **File** menu, click on **Save.**

6. Restart the WinLab32 software.

Sample Preparation: An Example

The following is an example of sample preparation:

An analyst weighs 1.54 grams of sample into a beaker. This is the Initial Sample Weight.

Next, hydrochloric acid is added to the sample and the solution is heated so that the sample is dissolved. After the solution is allowed to cool, the analyst quantitatively transfers it into a 250 mL flask and fills the flask to the 250 mL mark. This is the Sample Prep Volume.

A 10 mL aliquot of this solution (the Aliquot Volume) is pipetted into a 100 mL volumetric flask and diluted to the 100 mL mark. This is the Diluted to Volume.

To summarize, for the above example, you would enter the following values:

Initial Sample Weight	1.54 grams
Sample Prep Volume	250 mL
Aliquot Volume	10 mL
Diluted to Volume	100 mL

Note   You can also record the Diluted to Volume as the ratio of the original sample volume to the final sample volume. In this example, 10 mL of sample is diluted to 100 mL, so the ratio would be 10:100 or 1:10. You can enter 1 for Aliquot Volume and 10 for Diluted to Volume.

Analysis Schedule Parameters

Recalibration Before

Select a recalibration, reslope, or autozero before the solution identified in this row is analyzed. Select **None** or leave the entry field blank if you do not want any of these options performed before this solution. To automatically enter the same information for a sequence of samples, double-click on an entry field and use the Recalibrate Before Column Fill dialog.

Matrix Check Samples

Use this parameter when analyzing a pair of matrix check samples. The first sample in the pair is the reference sample and the second sample is the matrix check sample. The first sample must be scheduled for analysis before the second sample. Double-click on the entry field for the second sample in the pair of matrix check samples. The Matrix Check Sample Entry dialog appears, in which you select options for the matrix check calculation and identify the two samples in the matrix check pair.

Analyze QCs Before

Use this parameter to analyze quality control samples before the selected samples. Type the numbers of the QC samples in the entry field. These numbers are found in the QC section of the method. Type individual QC numbers or a range of QC numbers. Use commas to separate the numbers and ranges. Example: **1, 3-5**.

To analyze QC samples at a specified frequency, double-click on the entry field. The Analyze QCs Before Column Fill dialog appears.

Note Periodic QC Scheduling in the sample information file will override Periodic QC Scheduling in the Method Editor.

Read Delay

Use this parameter to use a Read Delay that differs from the read delay that is entered in the method. Enter the Read Delay in seconds. To automatically enter the same value for a sequence of samples, double-click on the column header and use the Column Fill dialog.

Wash Time

Use this parameter to use a Wash Time that differs from the Wash Time that is entered in the method. Enter the Wash Time in seconds. To automatically enter the same value for a sequence of samples, double-click on the column header and use the Column Fill dialog. Note that the Wash Frequency is selected in the method. You must select **Between Samples** for the Wash Frequency in order for a wash to occur.

Sample Information Parameters Dialog

You use this dialog to select the exact parameters that you need to describe your samples. You also modify the maximum number of samples displayed in the Sample Information Editor.
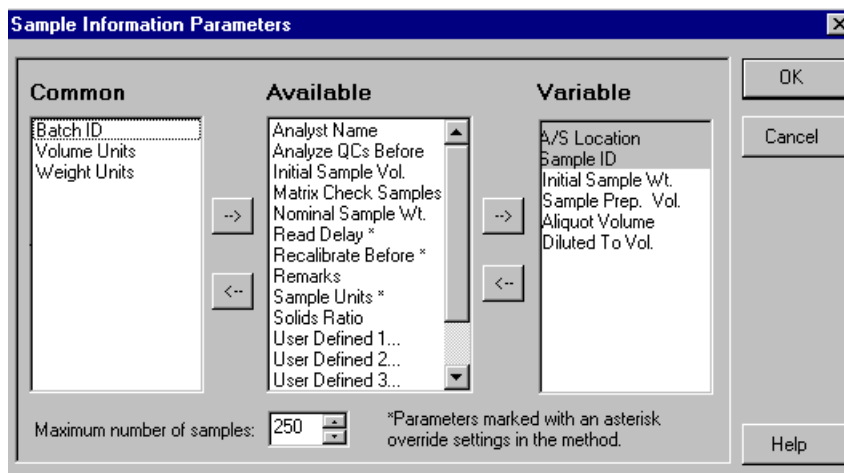
To display this dialog

1. Display the Sample Information Editor.

2. In the **Edit** menu, click on **Parameter List...**

    - or -

    Click with the right mouse button anywhere on the background area in the Sample Information Editor. In the pop-up that appears, select **Parameter List...**

    The Sample Information Parameters dialog appears.

To add parameters to the Sample Information Editor

1. In the **Available** list, click on a parameter to select it. (To deselect the parameter, click again.) The parameters are shown in alphabetical order.

2. Click on the arrow button that points to the list where you wish to move the selected parameter. Parameters marked with an asterisk (*)override settings in the method.

    ▶ If the entry for the parameter is common to all samples, move the parameter to the **Common** list.

    ▶ If the entry for the parameter varies for each sample, move the parameter to the **Variable** list. For example, the analyst name might be the same for all samples, while the sample IDs would vary for each sample.

    The parameter is removed from the **Available** list and appears in the list you chose.

    ▶ To change your selections, click on a parameter in the **Common** list or in the **Variable** list, then click on an arrow pointing back to the **Available** list.

    The parameter is returned to the **Available** list.

3. Select additional parameters, if desired, and move them to the appropriate lists. When you are finished, click on **OK**.

    In the Sample Information Editor, use the scroll bars to see the changes you made. Parameters that you added to the **Common** list appear in the **Common Parameters** table, and those added to the **Variable** list appear in the **Parameters that Vary by Sample** table.

To specify the maximum number of samples:

▶ Use the up or down arrows to specify the number of samples you have and then click on **OK**. In the Sample Information Editor, the Parameters that Vary by Sample table will contain this number of rows, one for each sample.

User Defined Entry Field Dialog

You use this dialog to enter a name for a parameter you want to add to the Sample Information Editor. You can select a common user defined entry field for all samples or you can vary the information in this entry field by sample. For more information, see *Customizing the Sample Information Editor* earlier in this chapter.

To display this dialog

This dialog automatically appears when you select a User Defined Entry field in the Sample Information Parameters dialog.

To define the entry field

Type a name for the entry field using up to 20 characters, then click on **OK**. The entry field is added to the Sample Information Editor.

Sample Information Editor Column Fill Dialogs

To automatically enter information for a range of consecutive samples, the Sample Information Editor contains Column Fill dialogs.

To display a Column Fill dialog

▶ Double-click on either an entry field in the column or the column header.

To automatically enter information for a range of samples

1. Type the information in the entry field in the dialog. Enter values or text, as appropriate for the entry field. For a definition of the sample parameter, see *Sample Information Parameters and Dialogs* earlier in this chapter.

2. Select the sample number range for your group of samples:

    ▶ In the Start box, select the sample number for the first sample in the group.

    ▶ In the End box, select the sample number for the last sample in the group.

3. Click on **OK.** The software enters the values in the Sample Information Editor.

To display a Column Fill dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

▶ Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Autosampler Location Column Fill Dialog

You use this dialog to enter autosampler locations for a range of consecutive samples in the Sample Information Editor.

To display this dialog

▶ Double-click on either an entry field in the column or the column header.

To automatically enter autosampler locations for a range of samples

1. Select the first autosampler location for this range of samples in the **Starting Location** box.

2. Select the increment for the autosampler locations. For example, if you want to fill each autosampler location, select **1**. If you want to fill every other location, select **2**.

3. Select the sample number range for your group of samples:

   ▶ In the Start box, select the sample number for the first sample in the group.

   ▶ In the End box, select the sample number for the last sample in the group.

4. Click on **OK.** The software enters the IDs in the Sample Information Editor.

To display this dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

▶ Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Sample ID Column Fill Dialog

You use this dialog to enter Sample IDs for a range of consecutive samples in the Sample Information Editor. The sample IDs have a common prefix, followed by a variable number, followed by a common suffix. For example: water1march95 and water2march95.
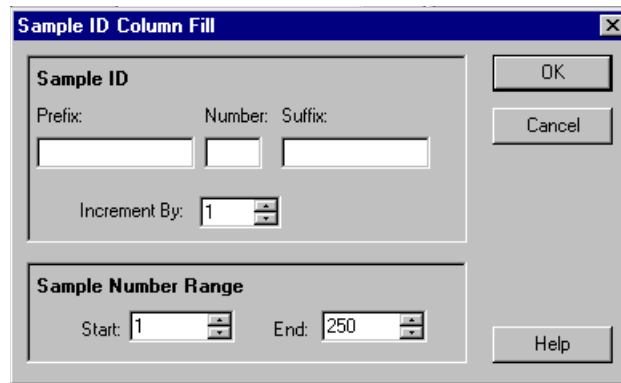
To display this dialog

▶ Double-click on either an entry field in the column or the column header.

To automatically enter sample IDs for a range of samples

1. Type the sample ID prefix. This is a combination of numbers or letters that will precede the sample ID number. The total number of characters allowed for the prefix, number, and suffix is 25.

2. Type the sample ID number. This number will be incremented by the value you select in the **Increment by** box.

3. Type the sample ID suffix. This is a combination of numbers or letters that will follow the sample ID number.

4. Select a value for the increment. The Sample ID number is incremented by this value.

5. Select the sample number range for your group of samples:

   ▶ In the Start box, select the sample number for the first sample in the group.

   ▶ In the End box, select the sample number for the last sample in the group.

6. Click on **OK.** The software enters the IDs in the Sample Information Editor.

To display this dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

▶ Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Sample Units Column Fill Dialog

You use this dialog to enter the same sample units for a range of consecutive samples in the Sample Information Editor. The sample units that you enter in the Sample Information Editor will be used in place of the units selected in the method.

To display this dialog

▶ Double-click on either an entry field in the column or the column header.

To automatically enter sample units for a range of samples

1. Select the units in the list.

2. Select the sample number range for your group of samples:

   ▶ In the Start box, select the sample number for the first sample in the group.

   ▶ In the End box, select the sample number for the last sample in the group.

3. Click on **OK.** The software enters the units in the Sample Information Editor.

To display this dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

▶ Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Units Column Fill Dialogs

You use these dialogs to enter the same Weight Units or Volume Units for a range of consecutive samples in the Sample Information Editor.

To display these dialogs

▶ Double-click on the column header for **Weight Units** or **Volume Units**.

To automatically enter units for a range of samples

1. Select the units in the list.

2. Select the sample number range for your group of samples:

   ▶ In the Start box, select the sample number for the first sample in the group.

   ▶ In the End box, select the sample number for the last sample in the group.

3. Click on **OK.** The software enters the units in the Sample Information Editor.

To display these dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

▶ Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Initial Sample Weight Column Fill Dialog

If the final sample concentrations are being reported in weight/weight units, you must enter the sample weights in the Sample Information Editor. You use this dialog to enter the same Sample Weights for a range of consecutive samples.

To display this dialog

► Double-click on either an entry field in the column or the column header.

To automatically enter initial sample weights for a range of samples

1. Type a value for the initial sample weight.

2. Select the sample number range for your group of samples:

    ► In the Start box, select the sample number for the first sample in the group.

    ► In the End box, select the sample number for the last sample in the group.

3. Click on **OK.** The software enters the values in the Sample Information Editor.

To display this dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

► Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Initial Sample Volume Column Fill Dialog

You use this dialog to enter the same Initial Sample Volumes for a range of consecutive samples in the Sample Information Editor.

To display this dialog

▶ Double-click on either an entry field in the column or the column header.

To automatically enter initial sample volumes for a range of samples

1. Type a value for the initial sample volume.

2. Select the sample number range for your group of samples:

    ▶ In the Start box, select the sample number for the first sample in the group.

    ▶ In the End box, select the sample number for the last sample in the group.

3. Click on **OK.** The software enters the values in the Sample Information Editor.

To display this dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

▶ Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Aliquot Volume Column Fill Dialog

You use this dialog to enter the same aliquot volume for a range of consecutive samples in the Sample Information Editor.

To display this dialog

▶ Double-click on either an entry field in the column or the column header.

To automatically enter aliquot volumes for a range of samples

1. Type a value for the aliquot volume.

2. Select the sample number range for your group of samples:

    ▶ In the Start box, select the sample number for the first sample in the group.

    ▶ In the End box, select the sample number for the last sample in the group.

3. Click on **OK.** The software enters the values in the Sample Information Editor.

To display this dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

▶ Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Diluted To Volume Column Fill Dialog

You use this dialog to enter the same final sample volume for a range of consecutive samples in the Sample Information Editor.

To display this dialog

▶ Double-click on either an entry field in the column or the column header.

To automatically enter final sample volumes for a range of samples

1. Type a value for the final sample volume.

2. Select the sample number range for your group of samples:

   ▶ In the Start box, select the sample number for the first sample in the group.

   ▶ In the End box, select the sample number for the last sample in the group.

3. Click on **OK.** The software enters the values in the Sample Information Editor.

To display this dialog and pre-select a range of samples

If desired, you can pre-select the range of samples in the Sample Information Editor by selecting a range of entry fields in the column. When the dialog is displayed, this range will automatically be entered.

▶ Click on the first row in the range of samples and drag the mouse cursor over the entry fields that you want to fill in the column. The rows are highlighted. In the **Edit** menu, click on **Column Fill...**

Sample Information Editor: Recalibrate Before Column Fill Dialog

You use this dialog to enter the same recalibration information for a range of consecutive samples in the Sample Information Editor.

To display this dialog

▶ In the Sample Information Editor, double-click on either an entry field in the **Recalibrate Before** column or the column header.

To automatically enter recalibration information for a range of samples

1. Select a recalibration, reslope, or autozero before the solution identified in this row is analyzed. Select **None** or leave the entry field blank if you do not want any of these options performed.

2. Select the frequency in which you want to recalibrate, then select the sample numbers for a range of samples. For example, to recalibrate before every five samples in a range numbered 10 through 20, select **Schedule after every 5 samples,** then select **10** for the Start number and **20** for the End number. In this example, the software would automatically recalibrate before Sample Numbers 15 and 20.

Sample Information Editor: Analyze QCs Before Column Fill Dialog

You use this dialog to schedule quality control samples.

To display this dialog

► In the Sample Information Editor, double-click on either an entry field in the **Analyze QCs Before** column or the column header.

To schedule the QC samples

1. Type the numbers of the QC samples in the entry field. These numbers are found in the QC section of the method. Type individual QC numbers or a range of QC numbers. Use commas to separate the numbers and ranges. Example: **1, 3-5**.

2. Select the frequency in which you want to analyze the QC samples, then select the sample numbers for a range of samples. For example, to analyze the QCs before every five samples in a range numbered 10 through 20, select **Schedule after every 5 samples,** then select **10** for the Start number and **20** for the End number. In this example, the software would automatically enter the QC sample numbers to be analyzed before Sample Numbers 15 and 20.

Sample Information Editor: Solids Ratio Column Fill Dialog

You use this dialog to enter the ratio of the wet and dry weights for the sample.

To display this dialog

▶ In the Sample Information Editor, double-click on an entry field in the **Solids Ratio** column.

To enter the solids ratio

1. You may enter this ratio in one of two ways, depending on how you want to report the results.

   ▶ If you are analyzing a dry sample and want the result converted to a wet basis, enter the ratio of dry weight divided by wet weight.

   - or -

   ▶ If you are analyzing a wet sample and want the result converted to a dry basis, enter the ratio of wet weight divided by dry weight.

   In either case, the concentration in calibration units is multiplied by the solids ratio as part of the conversion from concentration in calibration units to concentration in sample units.

2. Select the sample number range for your group of samples:

   ▶ In the Start box, select the sample number for the first sample in the group.

   ▶ In the End box, select the sample number for the last sample in the group.

3. Click on **OK.** The software enters the values in the Sample Information Editor.

To convert a measured concentration in calibration units to a final concentration in sample units:

$$C_s = C_c f_2 \ \times \ \frac{V_p}{Q} \times \ \frac{V_d}{V_a} \times \ SR \times \frac{1}{f_1}$$

where:

C_s = concentration in sample units

f_1 = convert sample units to wt / wt (mg / g) or wt / vol (mg / L) base units

C_c = concentration in calibration (standard) units

f_2 = convert calib units to wt / vol (mg / L) base units

Q = initial sample quantity (wt or vol) converted to wt (g) or vol (L) base units

V_p = sample preparation volume converted to vol (L) base units

V_d = diluted to volume converted to vol (L) base units

V_a = aliquot volume converted to vol (L) base units

SR = solids ratio

Note: All f_n values are taken from the units.ini file.

Sample Information Editor: Remarks Entry Dialog

You use this dialog to enter comments about a sample or batch of samples.

To display this dialog

▶ In the Sample Information Editor, click on the Remarks entry field.

To enter remarks

▶ Type up to 50 characters of text, then click on **OK**.

Sample Information Editor: Matrix Check Sample Entry Dialog
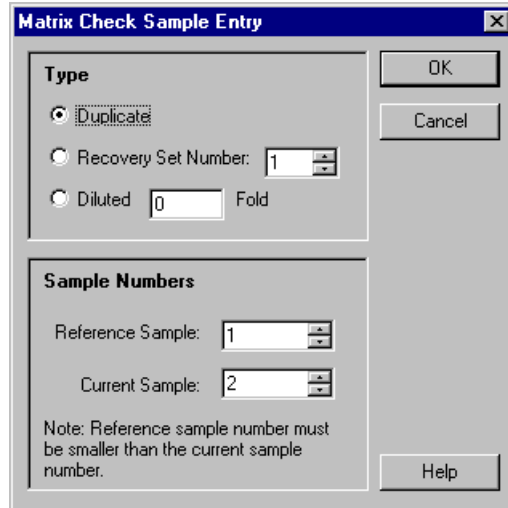
To display this dialog

▶ In the Sample Information Editor, double-click on an entry field in the Matrix Check Sample column. You should select the entry field for the second sample in the pair of matrix check samples.

- or -

▶ In the Sample Information Editor, click on an entry field in the Matrix Check Sample column, then click using the right mouse button and in the pop-up menu that appears, click on **Entry Dialog...**.

To select options for the matrix check calculation

1. Select an option:

Duplicate
A matrix check duplicate measurement is performed. The software calculates the relative percent difference using the equation:

$$\text{Relative \% Difference} = \frac{|\text{conc. \#1 - conc. \#2}|}{(\text{conc. \#1 + conc. \#2}) \div 2} \times 100$$

where:
conc #1 = the result obtained for the concentration of the reference sample
conc #2 = the result obtained for the concentration of the duplicate sample

Recovery Set Number
A spike recovery measurement is performed. Select the number of the recovery set on the Checks page of the Method Editor. The recovery set number indicates the analyte concentrations added to the reference sample to create the recovery sample. The software calculates the spike recovery for each analyte using the equation:

$$\text{Spike \% Recovery} = \frac{(\text{conc. \#2 - conc. \#1})}{\text{spike conc. added}} \times 100$$

where:
conc #1 = the result obtained for the concentration of the reference sample
conc #2 = the result obtained for the concentration of the recovery sample

Diluted *x* fold

A matrix check dilution measurement is performed. Enter the dilution factor. This is the ratio of the original sample volume to the final sample volume. For example, if 1 mL of sample is diluted to 10 mL, type 10. The software calculates the percent difference using the equation:

$$\% \text{ Difference} = \frac{|\text{conc. \#1} - (\text{conc. \#2} \times \text{DF})|}{\text{conc. \#1}} \times 100$$

where:

conc #1 = the result obtained for the concentration of the reference sample
conc #2 = the result obtained for the concentration of the diluted sample
DF = dilution factor

2. Select the sample number of the Reference Sample. This is the first sample in the pair of matrix check samples. This sample must be scheduled for analysis before the second sample in the pair.

3. Check the sample number for the Current Sample. Change the number, if necessary, so that it is the matrix check sample, which is the second sample in the pair.

Analysis Control Windows   5

About Analysis Control Windows

You can operate the instrument in either a manual mode (with or without an autosampler), or in an automated mode (with an autosampler only). When you operate in manual mode, you define the order and control the analysis. When you operate in automated mode, the application runs Blanks, Standards and Samples in a pre-defined order. You can also reprocess data stored in the Results library using different analytical conditions such as wavelength, peak processing algorithm, background correction, standard concentrations, units of concentration or QC concentrations.

▶ Select the appropriate analysis control window depending on the task you wish to perform.

To do this	Use
Analyze a few samples manually.	Manual Analysis Control window
Analyze many samples using an autosampler.	Automated Analysis Control window
Recalculate stored data using different analytical conditions.	Data Reprocessing window

Manual Analysis Control Window

You use this window to set up and perform an analysis manually.
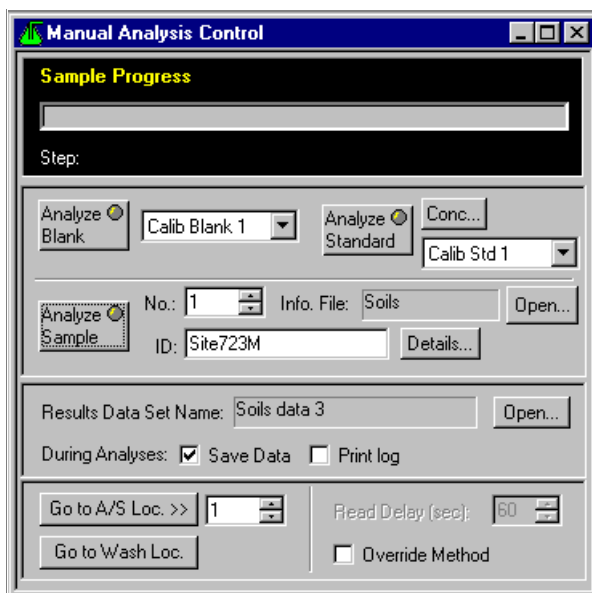
To display this window

▶ On the **Toolbar**, click on **Manual**.

-or-

▶ In the **Tools** menu, click on **Manual Analysis Control**.

Window Displays

Sample Progress

This display shows the status of the analysis.

Sample Progress   shows how much (the approximate percentage) of the analysis has been completed for the current sample.

Step   shows the current operation being performed.

Window Controls and Entries

Analyze Blank

► Click on this button to analyze the calibration or reagent blank solution shown in the entry field. When you do this, you are defining the calibration or reagent blank for the analysis. To interrupt the blank analysis, click again on **Analyze Blank**.

► To change the blank shown in the entry field, select an option from the drop-down list. Only the blanks that you have defined in the Calib: IDs and Locations page in the active method appear here.

Analyze Standard

► Click on this button to analyze the calibration standard shown in the entry field. The results of this analysis are used to set up a calibration. To interrupt the standard analysis, click again on **Analyze Standard**.

► To change the standard shown in the entry field, select an option from the drop-down list. The list shows all of the standards, including reslope, that you defined in the active method. A calibration curve is created after all blanks and standards defined in the method are run. To view a calibration curve, click on the Calib button, or from the Options menu, click on Calibration Display.

► To check the concentration values that were entered for the standard in the method, click on **Conc...**. In the View Standard Concentrations dialog that appears, the concentrations and units of each standard are shown. To change

the concentration of a standard, use the Method Editor-Calibration-Calib Units and Concentration.

Analyze Sample

► Click on this button to analyze the sample indicated by the No: (Number) entry field. To interrupt the analysis, click again on **Analyze Sample**.

No.

This shows the sample number of the current sample in the sample information file.

► Enter a different sample number, if needed. The Sample ID entry field shows the ID of the sample you selected.

ID

This identifies the sample that you are currently analyzing. If you are using a Sample Information file, the ID shown in the file appears here. Enter a different ID, if needed.

► To add or change dilution or other data for the sample, click on **Details...** In the Sample Details dialog that appears, change the appropriate entries by replacing the existing text or selecting an item from a drop-down list. For example, type a new Batch ID, then make a selection in the list of Volume Units.

Info File

This shows the sample information file that describes the sample sequence. The next sample ID appears in the ID box so you can analyze each sample in the proper sequence.

► Click on **Open...** and, in the Open Sample Information dialog that appears, select an existing sample information file. If you wish to create a new file, use the Sample Information Editor.

Note   The sample information file should contain entries for samples only. All other solutions, such as blanks and standards, are defined in the Method Editor.

Results Data Set Name

This shows the name of the results data set where the analysis results will be stored. Saving data is optional.

▶ To save data, select a results data set name where the data will be stored: Click on **Open** and in the Select Results Data Set dialog that appears, type a new data set name or select an existing one. A check mark appears in the Save Data box next to During Analysis, confirming that data will be saved.

▶ If you do not want to save data, disable Save Data, or do not specify a results data set name.

Note   To be able to reprocess data, it must be saved.

During Analyses

This shows two options for handling data generated in an analysis.

Save Data
When you select a results data set name, this box is selected automatically (a check mark appears) confirming that data will be saved.

Note   For information on the amount of disk storage space that is required for your results, see *Equation for Calculating Disk Storage for Results* later in this chapter.

Print Log
Select the Print Log box to print a log of signals, analytical results, and other information about the analysis. The log includes an analytical header and, for each sample, a sample header and the data that is shown in the Results Display window. Look at all of the selections on the Method Editor Window Options page to get an overview of the selected analytical results that will appear in the printout.

Go to A/S Loc>>

▶ Click on this button to move the probe to the selected autosampler location shown in the entry field. The Auto Sampler Monitor reflects the change.

Go to Wash Location

▶ Click on this button to move the probe to the wash location. The Auto Sampler Monitor reflects the change.

Read Delay (sec)

This is the time, in seconds, that the system waits after it receives an analyze command before it starts to measure the signal. This delay allows the sample to reach the plasma before measurement begins. If the method contains a flush time that is greater than zero, the system will perform a sample flush before beginning the read delay. By default, the read delay from the method will be used.

▶ To use a read delay that is different than that shown in the method, select the Override Method box and enter a new read delay value.

Override Method

When using the Read Delay process, this box controls whether the instrument will use the values in the active method or the changes you make in this window. If it is left blank, the values in the active method are used. If the Override Method box is checked, the values entered in this page are used. The values entered in the method are not changed.

View Standard Concentrations Dialog

This is an information-only dialog that provides the concentrations and units for the standards in the active method. You cannot make changes in this dialog. You must use the Method Editor, Calibration, Calib Units and Concentration to make any changes.

To display this dialog

▶ In the Manual Analysis Control window, click on **Conc....**

Using this dialog

Select the standard of interest from the drop-down list. The table shows the ID, concentration and units of the analytes present in the standard, according to the active method.

To change any of these parameters, go to the Method Editor, Calibration pages: Define Standards and Calib Units and Concentration.

Sample Details Dialog

You use this dialog to view and change parameters in the current Sample Information file during manual analysis.

To display this dialog

▶ In the Manual Analysis Control window, click on **Details**.

Using this dialog

▶ Review the parameter values and make changes as desired. For example, to enter a Batch ID, double-click in the Batch ID entry field and type a name. To enter Volume Units, click in the Volume Units entry field and select units from the drop-down list.

▶ Changes made using this dialog will be reflected in the active sample information file. If you want to save the parameters entered using this dialog, save the active sample information file.

Parameters Common to All Samples

These are parameters that are the same for all samples such as the Batch ID and volume and weight units. The parameters that appear in this dialog depend on the parameters in the active Sample Information file. Refer to the Sample Information for details on the individual parameters.

Parameters that Vary by Sample

These are parameters that vary for individual samples, such as the sample ID, A/S location and the initial sample weight. The parameters that appear in this dialog depend on the parameters in the active Sample Information file. Refer to Sample Information for details on the individual parameters.

Select Results Data Set Name

You use this dialog to create a new, or select an existing data set for saving results or reprocessing data.

To display this dialog

► In the Manual Analysis Control window, the Automated Analysis Control window: Set Up page, or the Interelement Correction Model Builder, click on **Open...** next to Results Data Set Name.

► In the Data Reprocessing window, click on **Browse...** next to Reprocessed Data Set or Data Set to Reprocess.

Check the Results Database Library location

The current database library location is shown. This is where the results data set will be stored.

To change the library location:

1. Click on **Browse.**

2. In the Open dialog that appears, select the directory and click Open**.**

3. In the Select Results Data Set dialog, click on the name of the data set you want to use.

4. Click on **OK**.

To select a results data set for saving data

▶ To create a new data set, type a name in the Name entry field and click on **OK**. The Results Data Set dialog closes and the name you typed appears in the window where you initiated the command.To save results to an existing data set:

▶ Click on the name of the results data set in the table, then click on **OK.**

-or-

▶ Double-click on the results data set name.

Automated Analysis Control Window

When you want to perform an automated analysis, (the application runs blanks, standards and samples in a pre-defined order) use this window to control the system and autosampler.

To display this window

▶ On the **Toolbar**, click on **Auto**.

-or -

▶ From the **Tools** menu, click on **Automated Analysis Control.**

Note   You can only have the Manual Analysis or Auto Analysis or Reprocessing window open at one time.

Automated Analysis Control window pages

Click on the labeled tabs in the Automated Analysis Control window to display the following pages.

•   **Set Up**. This page defines the methods and sample information files to be used, where to store analytical data, and other setup parameters, such as wavelength recalibration, automatic shutdown and automatic export. You are not required

to enter any information on this page. By default the method in memory will be used for the analysis. Note the other settings that are selected. You can change any of them that you wish.

• **Analyze**. This page indicates instrument analysis status, controls the instrument and autosampler and contains options for starting and stopping an analysis.

Using a sample information file

Usually, you enter information in the method for analyzing blanks, calibration standards, and QC samples. If, however, you wish to analyze any of the blanks, calibration standards, or QC samples as samples, you must enter their IDs in the sample information file. As a result, they will be treated like samples in addition to the usage specified in the method.

Automated Analysis Control: Set Up Page

You use this page to select methods and a Sample Information file. You also can select a results data set to store the data, print an analysis log, and select other actions to occur during an analysis.

Note You can change setup options even after an analysis has begun by returning to the Set Up page.

*The Automated
Analysis
Control window:
Set Up Page.*

To display this page

▶ Click on the **Set Up** tab on the bottom of the Automated Analysis Control window.

Methods and Sample Locations

Method

You use this entry field to select the methods for the automated analysis.

▶ Double-click on an entry field in the Method column. In the Open Method dialog that appears, select a method.

Note   To select more than one method, you must first select **Open Methods in List** on the Setup page. This option is located below the Methods and Sample Locations table. You can select up to 50 methods.

Delay

This is the time, in minutes, that the system waits before it starts the method. (Maximum of 99 minutes)

Sample Info File

By selecting samples from a sample information file, information you have supplied about the samples (for example, sample weights or dilutions) is used in the analysis. Use this column to specify the samples you want to use from a sample information file. First, open a sample information file. Then, select the check box for **Use Sample Information** below the name of the sample information file. Then three options appear in the Sample Info File column in a drop-down list. If you want to analyze all of the samples in the sample information file, select **All Defined** from the drop-down list. If you want to select only certain autosampler locations, select **Locations**. If you want to select samples by the sample numbers listed in the sample information file, select **Sample Nos.**

Locations

If you do not want to analyze all of the samples in the sample information file, use this entry field to select only the autosampler locations of the samples that you want to analyze. If you have selected All Defined or Sample Nos. in the Sample Info File drop-down list, you cannot enter locations here. If you have not selected the check box for **Use Sample Information** below the name of the sample information file, the locations do not apply to a sample information file.

▶ Type individual locations or a range of locations. Use commas to separate the locations and ranges. It is not necessary to enter the locations of blanks, QC's, check or calibration solutions. Enter locations for these if you want them to be treated as a sample within the analysis.

Example: **10-15,18,20,22,25-30**.

Sample Nos.

If you do not want to analyze all the samples in the sample information file, use this entry field to select only the sample numbers that you want to analyze. If you have selected All Defined or Locations from the Sample Info File drop-down list, you cannot enter sample numbers here.

Example: If you only want to analyze #s 1, 3, 4, and 5, but not #2, in the Sample Info File column, select **Sample Nos**. This opens the Sample numbers field so that the sample numbers can be entered.

Method Source

Choose Use Method in Memory to use the Method file currently active (and displayed on the toolbar.) Choose Open Methods in List if you want to choose one or more methods to use during analysis.

Sample Information File

A sample information file stores information about the samples. The system uses this file to label the data from your samples and often needs it to calculate final concentrations. The sample information file also specifies the autosampler locations of the samples.

▶ To select a sample information file, click on **Open**. In the Open Sample Information dialog that appears, select the name of the file. Then make a selection in the Sample Info File column.

Note If a sample information file is not entered, the "Untitled" sample information file that is currently open in the editor is listed.

Use Sample Information

Select this box to specify the samples you want to use from a sample information file and to use the information specified in the Sample Information File (initial weight, dilution, volume, etc.) during the analysis. WinLab32 uses the information to calculate corrections for final results.

Results Data Set Name

This shows the name of the results data set where the analysis results will be stored. Saving data is optional.

▶ To save data, select a results data set name where the data will be stored: Click on **Open** and in the Select Results Data Set dialog that appears, type a new data set name or select an existing one. When you click on **OK**, the dialog closes

and on the Automated Analysis Set Up page (Method and Sample Locations), a check mark appears in the Save Data box, confirming that data will be saved.

▶ If you do not want to save data, do not specify a results data set name, or click on the Save Data check box to clear the check mark.

Save Data During Analyses

When you select a results data set name, this box is selected automatically (a check mark appears) confirming that data will be saved. To select options for the type of data to be saved and for the type of results to be displayed and printed, use the Options page in the Method Editor.

Note For information on the amount of disk storage space that is required for your results, see *Equation for Calculating Disk Storage for Results* later in this chapter.

Additional Options

Auto Shutdown
To schedule an automatic shutdown, click on **Set...** In the Automatic Shutdown/Startup dialog that appears, select options controlling time of and events to occur at shutdown and startup. This feature allows you to shut down the plasma at the end of the analysis and start it up again at a later specified time.

Auto Export
To automatically export data and write it into a file that can be read by many other programs, including spreadsheet and database management programs, select this box and click on **Set...** to select one or two export designs. For more information, see the Data Manager Help.

Auto Wavelength Realign
It may be important to correct for small changes in wavelength alignment that may occur during an analysis. This is done using the autowavelength realign feature. Select this box to have the system perform an auto wavelength realign. For more information, see *Automatic Wavelength Realignment* later in this chapter.

Print Log During Analyses
Select this box to print a log of signals, analytical results, and other information about the analysis. The log includes an analytical header and, for each sample, a sample header and the data that is shown in the Results Display window as selected in the Options page of the Method Editor. Look at the selections on the Method Editor Window Options page to get an overview of the selected analytical results that will appear in the printout.

Sample Info File Column Fill dialog

You use this dialog to automatically enter the samples you want to use from a sample information file when using several methods in an analysis.

To display this dialog

▶ On the Set Up page of the Automated Analysis Control window, select the **Open Methods in List** option and then double-click on the **Sample Info File** column header.

To automatically enter sample information for a range of methods

1. In the Automated Analysis Control window, select the methods for the analysis. You must have more than one method listed.

2. In the Automated Analysis Control window, open a sample information file.

3. Select the check box for **Use Sample Information**.

4. On the Set Up page of the Automated Analysis Control window, double-click on the **Sample Info File** column header.

5. In the dialog, select one of the following options:

   All Defined
   Select this option to analyze all of the samples in the sample information file.

   Locations
   To analyze only certain autosampler locations listed in the sample information file, select this option and enter the locations.

Sample Nos.
To analyze only certain sample numbers listed in the sample information file, select this option and enter the sample numbers.
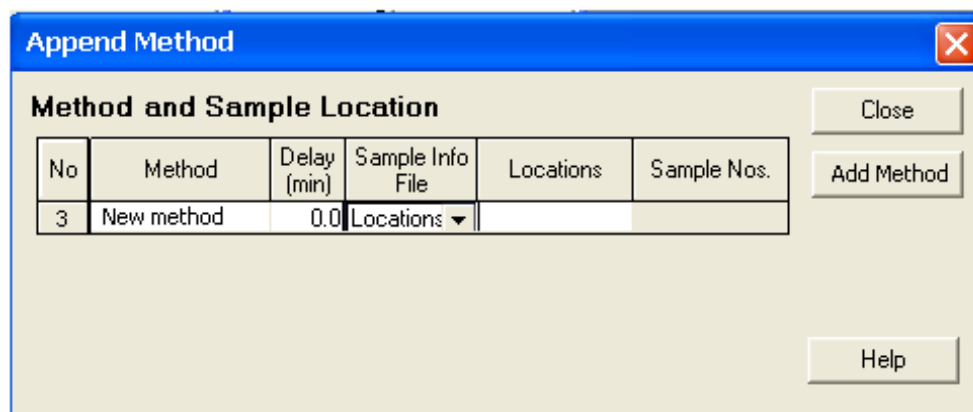
Automatic Wavelength Realignment

To set the interval time for performing a wavelength realignment, on the Automated Analysis Control dialog, click the Set button next to Auto Wavelength Realign. In the Automatic Wavelength Realignment dialog that appears, enter the interval for the auto wavelength realign.

To select Start of Analysis or Start of Each Method, click the radio button next to these options. To set a different time, click in the check box next to Periodic and enter the interval time in minutes. Click on OK to close the dialog.

Append Method

You use this dialog to add methods to a run list after an analysis has started.

To append a method to a run list:

1. After an analysis has started click the **Append Method** button, which is located on the Automated Analysis Control Window.
    The Append Method dialog appears.

2. Double-click on an entry field in the **Method** column.
    The Open Method dialog appears.

3. In the Open Method dialog, select a method.

4. Specify a **Delay time** for the method you just added.
    Delay is the time, in minutes, that the system waits before it starts the method.
    The maximum delay you can specify is 99 minutes.

5. Use the **Sample Info File** column to specify the samples you want to use from a sample information file. By selecting samples from a sample information file, information you have supplied about the sample (for example, sample weights or dilutions) is used in the analysis.

    You can select from three options. If you want to analyze all of the samples in the sample information file, select **All Defined** from the drop-down list. If you want to select only certain autosampler locations, select Locations. If you want to select samples by the sample numbers listed in the sample information file, select **Sample Nos**.

    Note: For this column to be enabled you must have checked Use Sample Information on the Set Up tab of the Automated Analysis Control dialog.

6. If you selected **Locations** from the Sample Info File drop down, then you can use this entry field to select only the autosampler locations of the samples that you want to analyze. In this field type individual locations or a range of locations. Use commas to separate the lcoations and ranges. It is not necessary to enter the locations of blanks, QCs, check, or calibration solutions. Enter locations for these if you want them to be treated as a sample within the analysis.

    Note: If you have selected **All Defined** or **Sample Nos.** in the Sample Info File drop-down list, you cannot enter locations here. If you have not checked **Use Sample Information** on the Automated Analysis Control Set Up tab, the locations do not apply to a sample information file.

7. If you selected **Sample Nos.** from the Sample Info File drop down, then use this entry field to select only the sample numbers that you want to analyze.

   Note: If you have selected **All Defined** or **Locations** from the Sample Info File drop-down list, you cannot enter sample numbers here.

8. Click the **Add Method** button.

   WinLab32 performs a method and analysis check for the new method. The method now appears in the next available row on the Set Up tab of the Automated Analysis Control dialog.

   Note: Each time you click the Add Method button, the Append Method screen is cleared thereby allowing you to add another method.

9. The Append Method dialog will remain open until you click **Close**.

Automated Analysis Control: Analyze Page

You use this page to start or stop an analysis, and view and make changes to the analytical sequence and monitor the progress of the analysis.

The Automated Analysis Control window: Analyze Page showing a soils analysis in progress.
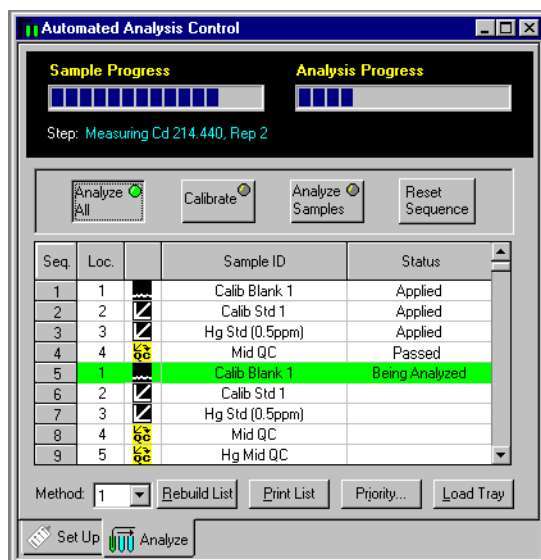
To display this page

▶ Click on the **Analyze** tab on the bottom of the Automated Analysis Control window.

Note Before you begin an analysis, make sure you have selected a method and defined the location on the Automated Analysis Control: Set Up page.

Window Displays

Sample Progress

This display shows how much of the analysis (percent complete) has been performed for the current sample.

Analysis Progress

This display shows how much of the entire analysis (percent complete) has been performed.

To view autosampler operation during an analysis:

1. In the Options menu, click on General Preferences.

   The General Preferences dialog appears.

2. Click on the View tab.

3. Select Autosampler Monitor.

4. Click on **OK**.

Window Controls and Entries

Analyze All

▶ Click on this button to analyze all of the samples in the analytical sequence.

▶ To interrupt the analysis sequence, click again on **Analyze All**. The Stopping an Analytical Sequence dialog appears, so that you can select when the analysis will stop.

Usually, **Analyze All** first calibrates the system, then analyzes the samples identified on the Set Up page. If, however, you stopped the automated analysis before all the samples were analyzed (and did not click on **Reset Sequence**), you may resume the analysis at a particular place in the analytical sequence by clicking again on **Analyze All.** The Continuing an Analytical Sequence dialog appears so that you can select the point where the interrupted analysis will restart.

Calibrate

▶ Click on this button to start the calibration sequence. The system uses the blank and standards defined in the method.

▶ To interrupt the calibration sequence, click again on **Calibrate**. The Stopping an Analytical Sequence dialog appears so that you can select when the blank and standard analysis will stop. If you stopped the blank and standard analysis, (and you did not click on **Reset Sequence**), you may resume at a particular place in the calibration sequence by clicking again on **Calibrate**. The Continuing an Analytical Sequence dialog appears so that you can select the point where the interrupted analysis will restart.

When calibration is complete, you may want to examine the calibration curves in the Calibration window before analyzing samples.

Analyze Samples

▶ Click on this button to start analyzing samples after the instrument has been calibrated.

▶ Click on the button a second time to stop the analyses. The Stopping an Analytical Sequence dialog appears, so that you can select when the sample analysis will stop.

The analysis usually starts at the beginning of the sequence that is shown on the Set Up page. If, however, you stopped the automated analysis before all samples were analyzed, (and you did not click on **Reset Sequence**), you may resume the analysis at a particular place in the analytical sequence by clicking

again on **Analyze Samples**. The Continuing an Analytical Sequence dialog appears so that you can select the point where the interrupted analysis will restart.

Note   If you schedule one or more **After Initial Calib QC** samples in the method, **Analyze Samples** will analyze the QC as part of the sample list. This is useful if you wish to use an existing calibration curve, analyze the **After Initial Calib QC** sample as a check (without reanalyzing your calibration standards), then take action based on the pass or fail status of the QC. For more information on QCs, see the Method Editor: QC Pages.

Reset Sequence

This button resets the analytical sequence back to the beginning.

▶ Click on this button to reset the analytical sequence to 1. The next time you click on Analyze All, Analyze Samples or Calibrate, the sequence will be rebuilt and the system will start with the first item in the list.

Analytical Sequence

Method

If more than one method is listed on the Set Up page, you can view which method is being used for the current analysis. The methods are numbered on the Set Up page. Type in, or click the arrow, to choose the Method number.

Rebuild List

▶ To refresh the display (to show changes you have made to the method or sample information file), click on this button.

Print List

▶ To print the sequence, location, sample type, ID, and status information shown, click on this button. The printed report also shows the current date and time as well as the method name.

Priority...

▶ To insert a new sample into the sequence (only during an analysis), click on this button and in the Add Sample dialog that appears, enter new sample information.

Load Tray

▶ Click on this button to raise the autosampler probe and move it to the back so that the sample tray can be removed or installed. Click on **Load Tray** again to restore the probe to its operating position.

Stopping an Analytical Sequence Dialog

When you stop an analysis, you use this dialog to give the system further instructions.

To display this dialog

▶ When an analytical sequence is in progress, click on the button that you used to begin the analysis: **Analyze All**, **Calibrate**, or **Analyze Samples**. These buttons are located in the Automated Analysis Control window: Analyze page.

Using this dialog

1. Select one of the options.

   Stop immediately
   The system stops the analysis immediately when you click on **OK**.

   Complete current replicate
   The system finishes the measurement for the current replicate and then stops the analysis.

   Complete all replicates for current sample
   The system finishes all the replicate measurements on the current sample and then stops the analysis.

2. Click on **OK**.

   Or, if you decide not to stop the analysis, click on **Cancel**.

Continuing an Analytical Sequence Dialog

When you stop an analysis and then decide to continue it, you use this dialog to give the system instructions.

To display this dialog

▶ After you stop an analysis, click on the button that you originally used to start the analysis. Do not click on **Reset Sequence** unless you want to start a new analysis. These buttons are in the Automated Analysis Control window: Analyze page.

Using this dialog

1. Select one of the options.

   Continue with next sequence #
   The analysis will continue with the next solution in the analytical sequence. The next sequence number is the one that follows the last completed sequence number.

   Reanalyze previous sequence # and continue
   The analysis will continue with the previous solution in the analytical sequence. The previous sequence number is the one that was being analyzed when the analysis stopped.

   Continue with sequence # *n*
   The analysis will continue with a selected solution in the analytical sequence. Type the autosampler location of the sequence in the entry field.

   Restart current Method
   The analysis will restart the method that was being performed when the analysis was interrupted. This performs the same action as clicking on **Reset** and then on **Analyze All**, **Analyze Samples** or **Calibrate**.

Note If you have multiple methods, only the current method is rerun.

2. Click on **OK**. The system continues the analysis, based on the option selected.

   Or, if you decide not to continue the analysis, click on **Cancel**.

Reset Automated Analysis Dialog

After you have stopped an automated analysis, you can use this dialog to reset it to the beginning of the analytical sequence. The analytical sequence indicates the order in which the solutions will be analyzed. Calibration curves will be cleared when the analysis is restarted.

Using this dialog

1. After you stop an automated analysis, click on **Reset Sequence** in the Automated Analysis Control window.

2. In the dialog, select:

    ▶ **OK** to reset the analysis to the beginning of the analytical sequence.

    -or-

    ▶ **Cancel** if you decide that you do **not** want to reset the analysis.

    Other options are available for restarting the analysis. For example, you may want to continue the analysis from the next solution in the analytical sequence. For more information, see the *Continuing an Analytical Sequence Dialog* earlier in this chapter.

Add Sample Dialog

You use this dialog to enter information on a new sample that you want to insert in the analytical sequence.

To display this dialog

▶ In the Automated Analysis Control window: Analyze page, click on **Priority... The Priority button is active only while an analysis is in progress.**

Using this dialog

1. Enter the information on your sample in the same way you usually do. The parameters shown are the same as those in the Sample Information Editor.

2. Select an option from the When to Analyze drop-down list.

3. If you want to add another sample, click on **Add Sample**. This button adds the sample to the run list and refreshes the dialog.

Go To Location Dialog

You use this dialog to analyze samples in selected locations or move the autosampler probe to a particular location.

To display this dialog

▶ In the **Analysis Menu**, select **AutoSampler**. Then select **Go to Location**.

Using this dialog

Click on an option button, then click on **OK**.

Go to wash
This moves the autosampler probe to the location defined in the Method Editor and lowers it.

Go to location
This moves the autosampler probe to the location shown and lowers it. To change the location, type a location number or use the spin buttons.

Data Reprocessing Window

You use this window to reprocess sample data that is stored in a results data set. There are several reasons why you might want to reprocess data:

- You can "peak" or adjust the wavelength and change the analytical conditions of the method. For example, the line for Co 228.616 is showing up at 228.652, but all the concentrations are being calculated at 228.616. After "peaking" and updating the method, the wavelengths are changed to 228.652. Reprocessing allows the stored data to be recalled and the concentrations to be corrected according to the new and proper wavelength.

- You chose peak height and wanted peak area.

- • You chose 3 points per peak and want 5 or 7.

- • You created a MSF model to correct for some overlapping peak and want to obtain the true concentration of your analyte.

- • You entered 0.1 for a concentration of a standard and it is actually 1.0.

- • You chose mg/mL but you need the results to print out in ppb or oz/ton.

- • You chose a linear calibration equation instead of nonlinear.

- • You ran just the samples and forgot to recall the calibration from another data set. Therefore, no concentrations are given and you need that information.

- • You entered the wrong QC concentration or wrong QC limits the first time.

Reprocess cannot be accessed unless the Analysis Control Windows are closed.

The Data Reprocessing window.

To display this window

► On the Toolbar, click on **Reproc**.

    -or -

► From the **Tools** menu, click on **Data Reprocessing**.

Using this window

1. Next to Data Set to Reprocess, click on **Browse...** and select a results data set that contains the data you want to reprocess.

2. In the Data Reprocessing window, the results from the reprocessed data will be saved to the data set shown in the entry field. This field defaults to the data set from the most recent analysis. A check mark appears in the Save Reprocessed Data box, confirming that reprocessed data will be saved.

   To change to a different data set, click on **Browse...** and in the Select Results Data Set dialog that appears, type a new data set name or select an existing one. A check mark appears in the Save Results in Data Set box, confirming that reprocessed data will be saved. If you do not want to store reprocessed data, click the check box to clear it.

3. The Method should be open in the Method Editor so if you want to reprocess using different method parameters, that information can be used. Confirm that the method in use contains the correct settings for the results data set that is to be reprocessed.

4. In the table, review the information. The IDs in the selected results data set are listed along with the sample information that was used when the data was originally collected. To edit this information, type the new information into the fields as desired. To access a pop-up menu containing commands for editing the table, right click anywhere in the table. To hide columns that do not contain data, select the Hide Empty Columns check box.

Note   There is a column fill dialog available for each of the parameters in the table, except Sample Type and Original Method. To access a column fill dialog, double-click on the parameter's column header.

5. To select items of interest for reprocessing, select the associated row numbers in the table. To select a series of rows, click on the first row number of interest, hold down the Shift key, and click on the last row number of interest. You can also drag over the row numbers column using the mouse cursor. To select non-consecutive rows, hold down the Ctrl key and click on each row number.

Tip The order in which samples are selected is the order in which reprocessing occurs. As you select each row, the reprocessing sequence appears in the Sequence column.

6. Click on **Reprocess**.

Data Reprocessing Display

This shows a status of the reprocessing operation.

Reprocess

Click on this button to begin reprocessing. Click again to interrupt reprocessing.

Save Reprocessed Data
This box is selected automatically (a check mark appears), indicating that the reprocessed data generated will be saved to the results data set shown in the entry field. To select a different results data set, click on **Browse...**, and in the Select Results Data Set dialog that appears, type a new data set name or select an existing one. Note that the spectral data is not saved with the reprocessed results and does not change with reprocessing.

Print Log
Select this box to print a log of signals, analytical results, and other information about the analysis. The log includes an analytical header and, for each sample, a sample header and the data that is shown in the Results Display window as selected on the Options page of the Method. Look at all of the selections on the Method Editor Window Options page to get an overview of the selected analytical results that will appear in the printout.

Hide Empty Columns
Select this box to hide columns that do not contain data.

Data Set to Reprocess

Select the results data set that contains the data you want to reprocess. Click on **Browse...** and in the Results Data Set dialog that appears, select an existing data set name.

The sample parameters from the results data set you selected are brought into the Data Reprocessing window. The column heads in bold indicate that this information from the original analysis cannot be modified.

Note   For an example using Initial Sample Weight, Sample Prep Volume, Aliquot Volume, and Diluted to Volume see, *Sample Preparation: An Example* in Chapter 4, *Sample Information Editor*.

Sequence

This is the sequence in which samples will be reprocessed. This sequence appears as you select each row number.

Sample ID

This is the name you gave to each sample.

Sample Type

This is the solution type: blank, standard, sample, etc.

Omit Replicates

This is the entry for replicate numbers that you do not wish to reprocess for a sample.

Matrix Check Samples

This shows any samples that are designated as matrix checks.

Date/Time

This shows the date and time that each sample was analyzed.

Initial Sample Wt.

This is the quantity of sample weight before sample preparation.

Initial Sample Vol.

This is the quantity of sample volume before sample preparation.

Sample Units

Use this column to change the sample units on a per sample basis before reprocessing data. The concentration will be calculated based on the new units. (All of the analytes in a sample must have the same sample units to use this parameter.)

Weight Units

This shows the units of the Initial Sample Quantity. These are either weight or volume units.

Sample Prep. Volume

After a solid sample has been dissolved or a liquid sample has been acidified (or otherwise treated), the resulting solution is prepared to a specific volume. This is the Sample Prep Volume.

Aliquot Volume

If an aliquot of the sample solution is taken and diluted to a final volume, record the aliquot volume for this entry and the final volume for the Diluted to Volume entry.

Diluted to Volume

This is the final volume of solution obtained by diluting an aliquot of the sample solution. You can also record the dilution as the ratio of the original sample volume to the final sample volume. For example, if 10 mL of sample is diluted to 200 mL, this ratio would be 10:200 or 1:20. Enter 20 for the Diluted to Volume entry and 1 for the Aliquot volume.

Volume Units

If the final sample concentrations are being reported in weight/weight units, you must enter the units for the Sample Volume.

Solids Ratio

This is the ratio of the wet and dry weights for the sample. This ratio is used to correct the sample concentration. For more information, see the *Solids Ratio Column Fill dialog* in Chapter 4, *Sample Information Editor*.

Nominal Sample Weight

This is the target weight when samples are weighed. This entry is required only if corrections are being made to weight/volume measurements to compensate for weight variations among samples. The Nominal Sample Weight is divided by the Initial Sample Weight to correct the final concentration value.

Batch ID

This is the name you give to this batch of samples.

Original Method

This shows the method you used in the analysis. This information cannot be modified.

Remarks

The remarks you entered in the Sample Information file before the initial analysis.

Data Reprocessing Window Pop-Up Menu

The following pop-up menu contains several convenient commands as you work with the Data Reprocessing window.

To display the pop-up menus

▶ Click with the right mouse button in the Data Reprocessing window table. The Table editing pop-up menu appears:

Clear	Del
Insert Row	
Delete Rows	
Column Fill...	
Entry Dialog...	

Command	Description
Clear	Removes the contents of the selected entry fields.
Insert Row	Inserts a new row before the currently selected row.
Delete Rows	Removes the selected rows in the table.
Column Fill...	Displays the Column Fill dialog for the selected column. You use this dialog to automatically enter information for a range of samples.

Command	Description
Entry Dialog...	Displays the Matrix Check Sample Entry dialog for the selected entry field. You use this dialog to type information for a text entry field.

Reprint Data Window

You use this window to specify which results data you want to reprint. The Reprint function will allow you to create an exact copy of your original results data printout.

Note: Data can only be reprinted if it was obtained using versions 4.0 and higher of the ICP WinLab32 software.

To display this window:

▶ On the Toolbar, click on **Reprint**

-or -

From the **Tools** menu, click on **Reprint Data**.

Using this window

Note: If you plan on using the Reprint Data function, we recommend that you enable the Spectral Data option when collecting data. You enable Spectral Data by checking this option on the Options tab page of the Method Editor. If Spectral Data is not checked and you run an analysis, the samples will still be stored in a batch and will be available for reprinting, however, no data will be shown for those samples.

1. Next to **Results Data Set Name**, click on **Open...** and select a results data set that contains the data you want to reprint.

2. Next to **Analysis Batch Date/Time**, click **Open...** to select a batch that you want to reprint.
   The Select Analysis Batch dialog appears and displays the batches that are available in the data set you selected. Batches are identified by the date and time they were initially created.

   The rules for how batches are created are as follows:

   When you are performing **Manual Analyses**, a batch is created when you run the first solution after opening the Manual Analysis window. The batch will end when you close the Manual Analysis window. All samples that are run in between are stored as part of the same batch.

   When you are performing **Automated Analyses** a new batch is created when you first select one of the analyze buttons (Analyze All, Calibrate, and Analyze Samples). The batch will end when you close the Automated Analysis Control window. If the analysis is paused and then continued, the continuation of the analysis will be part of the same batch.

   When you are **Reprocessing Analyses** a new batch is created when you first open the Data Reprocessing window and click the Reprocess button. The batch will end when you close the Data Reprocessing window.

3. Click **Preview** to preview the selected data in the Results window, without printing.

4. Click **Print** to print the selected data.

   Output is sent to the Results window. It should be noted that no new data is stored to the Results database when you use the Reprint function.

Equation for Calculating Disk Storage for Results

To roughly calculate the amount of hard disk storage space that is required for storing results data sets, use the following equation:

Database Size = (Analytes * Samples) * 550 + (Analytes * Samples * Replicates) * Type-Factor Increase

Database size in bytes

Samples = actual samples + number of standard analyses + number of blank analyses + number of QC analyses (I.E. number of entries in analysis list)

Type-Factor = 300   Not Storing Spectra
500   Storing Spectra, normal resolution, no slit scanning
1100   Storing Spectra, normal resolution, 4-position slit scanning
Storing Spectra, high resolution, no slit scanning

Analytes	Samples	Replicates	Type-Factor	Approximate Size Increase (Bytes)
10	54	1	500	567,000
20	54	7	500	4,374,000
20	54	3	300	1,566,000
40	29	3	1100	4,466,000

Hardware Control Windows   6

About Hardware Control Windows

These windows are used to manually control specific parts of the hardware. Because a method usually controls some of these functions automatically during an analysis, these windows will be most useful to you during method development and for setup.

▶ Select the appropriate hardware control window depending on the task you wish to perform.

To do this	**Use**
Manually adjust the plasma and monitor the settings.	Plasma Control Window
Perform special spectrometer operations, including adjusting alignment.	Spectrometer Control Window

Plasma Control Window

You can use the Plasma Control window to modify plasma parameters, to monitor the status of major components affecting the plasma and sample introduction system, and to ignite the plasma and shut it off.

The following table shows tasks related to the Plasma Control Window.

For information on:	See:
Making the correct preparations to ignite the plasma and correcting unsuccessful ignition.	Igniting the Plasma
Performance checks that should be done periodically.	Optimizing and Verifying Performance
Parameters in the method that correspond to the Plasma Control window.	Method Editor
Selecting different types of pump tubing.	Pump Configuration Dialog

Plasma Control Window: A Quick Look
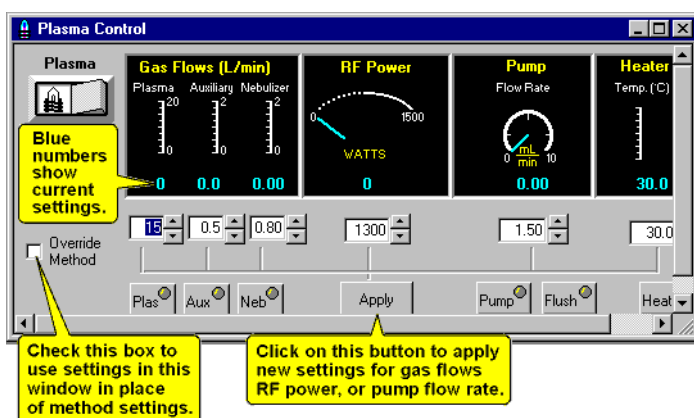
Note   The heater control shown above is not available on Optima 3000 Family spectrometers.

To display this window

▶ On the **Toolbar**, click on **Plasma**.

   - or -

▶ In the **Tools** menu, click on **Plasma Control.**

Plasma Control Window: Controls and Displays

Plasma Switch

▶ Click on this switch (or press **F9**) to turn the plasma on and off.
To turn the plasma off, you can also use the **Emergency Plasma Off** button
located on the instrument above the sample compartment.

Hint You can press **F9** to turn the plasma on and off even if the Plasma Control
window is not open.

The Plasma Status window indicates if ignition is successful or if there is a
problem. For more information, see *System Monitors* in *Chapter 10* and see
Igniting the Plasma, for details about the ignition process. Wait one hour before
analyzing samples. This warm-up time ensures accuracy in your results since it
allows the temperature of the sample introduction system to fully stabilize.

Override Method

If this box is checked, the instrument will use values for your next analysis as
specified in this window. If the box is not checked, the instrument will use the
values in the active method. Selecting Override Method does not change the values
in the active method or the method stored in the results library.

Gas Flows Display and Controls: Plas, Aux, and Neb Buttons

If you select the Override Method checkbox, the **Plas**, **Aux**, and **Neb** buttons allow
you to change the post-ignition flow rates of the argon gases and to monitor their
current values. **Plas**, **Aux**, and **Neb** refer to the plasma, auxiliary, and nebulizer
gases. The blue numbers in the display indicate the current flow rate.

▶ To start or stop gas flow, click on the desired button beneath the display. The green light on a gas button indicates that the gas is flowing.

⚠ If you stop the plasma gas flow while the torch is ignited, the torch will go out.

Caution

▶ To change a gas flow rate, select or type a value in the box beneath the appropriate gas display. For more information, see *Recommended Settings for Plasma Parameters and Recommended Settings for Nebulizers* later in this chapter. The maximum flow rate that you can enter cannot actually be attained by some nebulizers. If the nebulizer cannot reach the highest value, it will return the value it reached to the display. An error message will be recorded on the Plasma tab of the Instrument Diagnostics window.

▶ Click on **Apply** after you have adjusted the setting for the flow rates.

RF Power Display and Controls: Apply Button

These allow you to change the radio frequency power that is dissipated in the plasma and to monitor the current power.

▶ To change the power setting, select or type a value. For more information see *Recommended Settings for Plasma Parameters* later in this chapter. Click on **Apply** after you have adjusted the RF power.

Pump Gauges and Controls: Pump, Flush, and Apply Buttons

These allow you to change the sample flow rate and to monitor the status.

▶ To change the sample flow rate, select or type the appropriate value. The software adjusts the pump speed based on the type of pump tubing you selected in the Pump Configuration dialog. To change the pump tubing, in the **Options** menu, click on **Pump**. A typical sample flow rate is 1.5 mL/min for all instruments. Ranges for sample flow rates vary with the pump and pump tubing you are using. For example, values are from 0.6 to 5.7 mL/min for the

PerkinElmer pump (with 0.76 mm i.d. tubing) and from 0 to 2.5 mL/min for the Gilson pump. Then, click on **Apply**. For more information, see the *Pump Configuration dialog* in Chapter 11, *Menus and Toolbar*.

► Click on **Pump** to start or stop the peristaltic pump. The green light indicates that the pump is turned on.

► Click on **Flush** to set the pump speed to the flush setting in the Pump Configuration dialog.

Heater Control: Heat and Apply Buttons

These allow you to set the temperature of the spray chamber enclosure to provide a constant temperature for the spray chamber. For aqueous analyses, set the temperature to a few degrees above the lab temperature. A setting of 38 °C is typically used. If you set the temperature much higher than the lab temperature, it will take longer for the system to stabilize. For organic analyses, the spray chamber should be as cool as possible, therefore we recommend that you do not use the heater.

► To change the setting, select or type a value. Click on **Apply** after you have set the temperature.

► Click on **Heat** to turn the heater on or off. The green light indicates that the heater is turned on.

Overriding Method Settings

If you modify the settings in the Plasma Control window and want to test them for your next analysis, you do not need to modify the method. To override the method settings, see the illustration.

Modify settings in the Plasma Control Window as desired. Click on **Apply.**
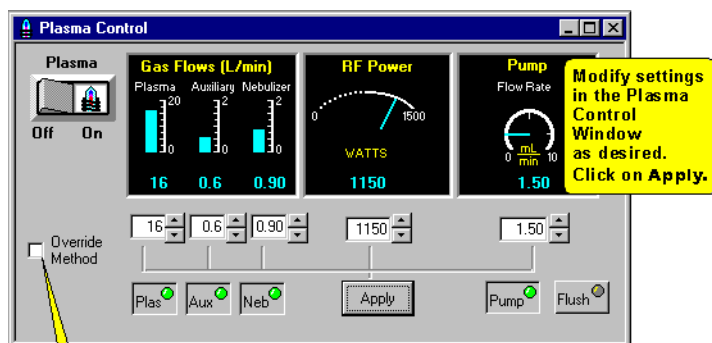
Select this box to use the Plasma Control settings in place of the method settings in the active method circled below.
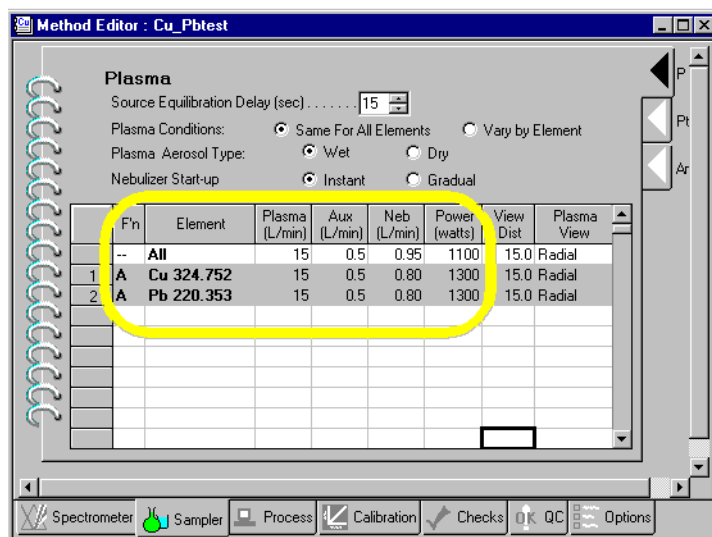
Recommended Settings for Plasma Parameters

Parameter	Aqueous	Organic
Plasma Flow:	15 L/min	15 L/min
Auxiliary Flow:	0.5 L/min (for Optima 3000 Family ICP spectrometers)	1.0 L/min
	0.2 L/min (for other simultaneous ICP spectrometers or for scanning CCD ICP spectrometers)	
Nebulizer Flow: (for Low-Flow GemCone or the Concentric Glass nebulizer)	0.80 L/min	0.50 L/min
RF power:	1300 watts	1300 watts
Sample Flow Rate	1.50 mL/min	0.80 mL/min

Recommended Settings for Nebulizers

Nebulizer	Part number	Argon Flow Rate	
		typical value	operating range
GemTip Cross-Flow	N068-0503	0.8 L/min	0.7 - 1.3 L/min
Low-Flow GemCone	N069-0671	0.6 L/min	0.4 - 0.95 L/min
High Solids GemCone	N069-0670	1 L/min	0.7 - 1.0 L/min
Concentric Glass (MEINHARD) Type A	0047-2020	0.8 L/min	0.7 - 1.3 L/min
Concentric Glass (MEINHARD) Type C	0047-2022	0.8 L/min	0.7 - 1.3 L/min
Concentric Glass (MEINHARD) Type K	N068-1574	0.6 L/min	0.4 - 0.7 L/min
Ultrasonic (115 V)	N069-1709	1.0 L/min or less	n/a
Ultrasonic (230 V)	N069-1710	1.0 L/min or less	n/a

Spectrometer Control Window

You can use the Spectrometer Control window to adjust and align the position from which the plasma is viewed, control various spectrometer manual settings, and perform calibrations of the spectrometer.

The following table shows tasks related to the Spectrometer Control Window.

For information on:	**See:**
Adjusting the wavelength, a procedure which allows you to fine-tune the peak maximum for a wavelength for a particular data set or for subsequent analyses on the instrument.	Adjusting the Wavelength
Performance checks that should be done periodically.	Optimizing and Verifying Performance
Parameters in the method that correspond to the Spectrometer Control window.	Method Editor: Spectrometer Pages Overview

Spectrometer Control Window: A Quick Look

The Spectrometer Control window is shown as it appears for simultaneous ICP spectrometers and is set for axial viewing.

Manual settings for spectrometer controls

Spectrometer Control

Torch Viewing Position

Idle

You can modify the coordinates for the torch viewing position for specialized use.

10
mm   0.0   0.0
-10

0   mm   30

15.0

☐ Override Method   15.0

Manual Settings

Purge Gas
- ⦿ Normal
- ○ High

☐ Override Method

Shutter
- ⦿ Auto
- ○ Closed
- ○ Open

Plasma
- ○ Radial
- ⦿ Axial

Hg Lamp
- ○ On
- ⦿ Off

Apply

| Align View | ⦿ UV   ○ VIS | Wavelength Calibration | Hg Realign | Detector Calibration |

Aligns the torch viewing position

See Table of Spectrometer Alignment & Calibration Controls.

The Spectrometer Control window is shown as it appears for scanning CCD ICP spectrometers and is set for radial viewing.

Manual settings for spectrometer controls

Spectrometer Control

Torch Viewing Position

Idle

You can modify the coordinates for the torch viewing position for specialized use.

10

mm   0.0   0.0

-10

0   mm   30

15.0

Override Method   15.0   Apply

Manual Settings

Purge Gas
- ○ Normal
- ○ High
- ● Off
- ☐ Override Method

Plasma
- ● Radial
- ○ Axial
- ○ Attn Radial
- ○ Attn Axial

Shutter
- ● Auto
- ○ Closed
- ○ Open

Align View ○

Initialize Optics ○

Detector Calibration ○

Aligns the torch viewing position

See Table of Spectrometer Alignment & Calibration Controls.

To display this window

▶ In the **Tools** menu, click on **Spectrometer Control.**

Spectrometer Control Window: Adjusting the Torch Viewing Position

In the Spectrometer Control window, you can:

▶ modify the coordinates for the torch viewing position

-or-

▶ automatically align the torch viewing position for the highest signal intensity by clicking on **Align View**. The spectrometer uses a computer-controlled mirror in the transfer optics to adjust the area of the plasma viewed by the optical system.

Spectrometer Control Window: Modifying the Coordinates for the Torch Viewing Position

Due to the various temperature zones in the plasma and differences in analyte ionization processes, slight adjustments to the torch viewing position may give higher intensity signals for particular analytes. Adjustments should be made on an empirical basis. After verification of the desired results, the new settings may be used to override the Viewing Distance setting in the method for all analytes. Keep in mind that, alternatively, the software can automatically align the torch viewing position for a particular analyte using Align View.

The crosshair on the Torch Viewing Position display shows what part of the plasma is currently being viewed by the spectrometer. After modifying one or both coordinates, click on **Apply** to set the new viewing position. The crosshair then moves to the new position and the blue numbers in the display indicate the current settings for the coordinates.
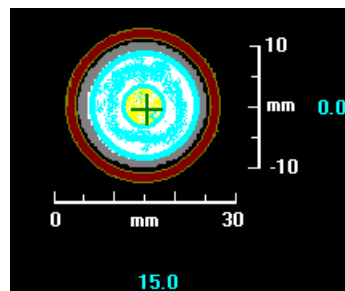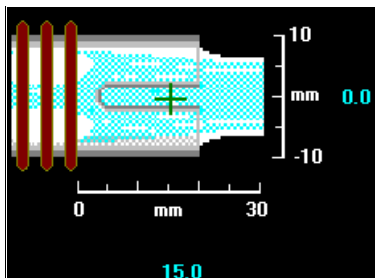
For radial viewing, you can modify:
1) the viewing position from the RF coil
to the tip of the plasma. (horizontal scale
0 to 30 mm)

For axial viewing, you can modify
the viewing position relative to the
plasma center on two axes.

2) the position relative to the plasma
center (vertical scale -10 to 10 mm)

To use the new settings in place of the settings in the method, select **Override Method.** The settings for both coordinates will be used. If you do not select Override Method, the system will use the setting for the Viewing Distance parameter. This parameter is in the method on the Sampler: Plasma page. It has only one coordinate and assumes the other coordinate to be 0. For example, a setting of 15.0 indicates the coordinates of 15.0 and 0.0.

If you want to set the torch viewing position individually for each analyte, you can do this using the Viewing Distance parameter in the method.

Spectrometer Control Window: Automatically Aligning the Torch Viewing Position

Perform this procedure when:

- the instrument or software is first installed

- the instrument is moved to a new location

- the torch is removed or replaced

- the RF coil is replaced

- you want to optimize intensities for an individual analyte

1. In the **Tools** menu, click on **Spectrometer Control**.
   The Spectrometer Control window appears.

2. On Dual View instruments, select Axial or Radial. We recommend that you first do the alignment in one viewing mode, then select the other viewing mode and repeat the procedure.

3. To view spectra collected during the procedure, open the Spectra Display window.

4. Click on **Align View** in the Spectrometer Control window to open the Align View dialog.

5. Select the element and wavelength. This is the wavelength that the system will use to measure the intensities at different viewing positions and locate the position that gives the highest intensity. You have the following choices.

   Click on the **Select Analyte** option and then select Manganese, which is typically used as the alignment wavelength for the majority of analyses.

   -or-

   Click on the **Select Analyte** option and then select another element from the drop-down list. This feature is for specialized analyses where you want to optimize the intensity for an individual analyte of interest. This may adversely affect the intensities of other analytes.

   -or-

   Click on the **Other** option and type in an element and/or wavelength not in the drop-down lists.

6. Set the Read Delay time.

7. Aspirate a solution containing the element you have selected. If you are using Manganese (Mn), use 10 mg/L for radial viewing or use 1 mg/L for axial viewing.

When using radial viewing, the system adjusts the lateral viewing position only.

When using axial viewing, the system adjusts both coordinates.

The system will determine the intensity at the selected wavelength, while adjusting the viewing position in incremental steps. In the Results window, a report is generated that gives the intensities found at each position. The system changes the **Torch Viewing Position** to the position that gives the highest intensity. In the Spectrometer Control window, the software resets the coordinates so that the 0 and 15 position is at the location of highest intensity.

Extended Search

For axial viewing only: If you have performed the procedure above, but are not satisfied with the results, you can extend the search over a larger area. This search takes longer than the usual procedure. Before using Extended Search, you should perform the sodium bullet test to make sure that the sample is reaching the plasma.

▶ To perform an Extended Search, select the **Extended Search** checkbox and then repeat the procedure above. A search is performed over a larger area of the torch.

If any of the following conditions are found, a message dialog appears to give you an opportunity to confirm that you wish to use the new alignment (or cancel it) if desired:

- The distance from the last alignment is greater than 5 mm.

- The value of the peak is less than 10,000 counts.

- The element wavelength used for the last optimization is not the same as the one used for the previous optimization.

Align View Dialog

You use this dialog to automatically align the torch viewing position for the highest signal intensity.

To display this dialog

1. In the **Tools** menu, click on **Spectrometer Control**.
    The Spectrometer Control window appears.

2. On Dual View instruments, select Axial or Radial. We recommend that you first do the alignment in one viewing mode, then select the other viewing mode and repeat the procedure.

3. To view spectra collected during the procedure, open the Spectra Display window.

4. Click on **Align View** in the Spectrometer Control window.

Using this dialog

You have the following choices.

1. Click on the **Select Analyte** option and then select Manganese, which is typically used as the alignment wavelength for the majority of analyses.

    -or-

    Click on the **Select Analyte** option and then select another element from the drop-down list. This feature is for specialized analyses where you want to optimize the intensity for an individual analyte of interest. This may adversely affect the intensities of other analytes.

    -or-

    Click on the **Other** option and type in an element and/or wavelength not in the drop-down lists.

2. Set the Read Delay time.

3. Aspirate a solution containing the element you have selected.

The system will determine the intensity at the selected wavelength, while adjusting the viewing position in incremental steps.

▶ For axial viewing only: If you have performed the procedure above, but are not satisfied with the results, you can perform an extended search. To do so, click on the Extended Search check box and repeat the procedure above. A search is performed over a larger area of the torch.

Note   This search takes longer than the usual procedure. Before using Extended Search, you should perform the sodium bullet test to make sure that the sample is reaching the plasma.

For more information, see *Automatically Aligning the Torch Viewing Position* earlier in this chapter.

Spectrometer Control Window: Manual Settings

Override Method

If this box is checked, the instrument will use the value for your analysis as specified in this window. If the box is not checked, the instrument will use the value in the active method. Selecting Override Method does not change a value in the active method or in the method stored in the results library.

Purge Gas

Nitrogen is used to purge the optical system during operation to remove oxygen and moisture. Usually, the purge should be set to Normal. The High setting may be used to improve sensitivity for elements with wavelengths below 190 nm since oxygen absorbs below 190 nm. On scanning CCD ICP spectrometers, you have the option of turning the purge gas off.

View Mode (Dual View instruments only)

Select the plasma viewing mode.

Radial
Light emitted from the side of the plasma passes through the torch slit and is directed to the spectrometer optics.

Axial
Light emitted along the axis of the plasma is directed to the spectrometer optics. The observation zone of the plasma is circular and the spectrometer views a column of light from the central channel of the plasma.

Attenuated (Attn) Radial or Attenuated (Attn) Axial
These settings are available only for scanning CCD ICP spectrometers. Use these settings when lower intensities are desired, for example, for samples of high concentration that would normally saturate the detector and for sensitive wavelengths, typically in axial mode.

Shutter Auto/Closed/Open

A shutter in the transfer optics allows light from the plasma to enter the spectrometer optics. This shutter may be set to the Auto, Closed, or Open position. The Auto setting indicates that the shutter is under instrument control**.**

Usually, the shutter is set to **Auto**. If you have an extensive number of samples to analyze, however, you may wish to set the shutter to the open position manually to increase analytical throughput. Be sure to close the shutter when the analysis is complete.

! Warning	Leaving the shutter in the open position with the plasma on could lead to deterioration of the optics and eventual degradation of UV performance. It is recommended that the shutter be closed when analyses are not being performed.

To measure dark current, take measurements with the shutter in the closed position.

Hg Lamp Off/On

This control appears only for simultaneous ICP spectrometers. An internal mercury lamp, under system control, is used for the mercury realignment procedure and periodic mercury recalibration. Manual control of this lamp is available for diagnostic purposes to verify that the signal is reaching the detector and that the mercury lamp is working.

Spectrometer Control Window: Overriding Method Settings

In the Spectrometer Control window, the following settings can override method settings.

Setting in Spectrometer Control Window	Setting in Method
Torch Viewing Position: To change the position, select new coordinates and click on **Apply**.	The **View Dist (Viewing Distance)** parameter is set in the method on the Sampler: Plasma page.
For either axial or radial viewing, both coordinates will be used.	The Viewing Distance parameter has only one coordinate and assumes the other coordinate to be 0. For example, a setting of 15 indicates the coordinates of 15 and 0.
Purge Gas	The **Purge Gas Flow** parameter is set on the Spectrometer: Settings page.

To override a setting in the method, select the **Override Method** box next to the setting in the Spectrometer Control window.

Table of Spectrometer Alignment and Calibration Controls

Control	What it Does	When to Use
Align View	Optimizes the torch viewing position for the highest signal intensity.	Perform this procedure when: • the instrument or software is first installed • the instrument is moved to a new location • the torch is removed or replaced • the RF coil is replaced • you want to optimize intensities for an individual analyte
Wavelength Calibration **(simultaneous spectrometers only)**	Gathers data for UV or VIS emission lines to compensate for slight variations in optical alignment. At the end of the UV or VIS wavelength calibration, the system determines the wavelength offset of an emission line from the internal mercury lamp and records the slit position as a reference point for future mercury realignments.	The instrument must be operated with the plasma on for at least one hour before initiating wavelength calibration on Optima 3000 Family spectrometers. Perform this procedure: • when the instrument or software is first installed • when the instrument is moved to a new location • after reinstalling or upgrading the software • if analyte peaks in the Examine Spectra window are significantly offset from the central line for the wavelength on a consistent basis

Control	What it Does	When to Use
Initialize Optics *(scanning CCD spectrometers)*	Performs a prism scan to optimize wavelength for the slit in the instrument optics. The system takes an intensity reading, scans the surrounding intensity range and locates the midpoint. If this midpoint varies from the initial intensity measurement, the system will optimize optics for use in subsequent analyses.	The instrument must be operated with the plasma on for at least 10 minutes before initializing optics. Initialize Optics is performed automatically during analyses. However, if there are wide fluctuations in ambient laboratory temperature that appear to affect intensity, you can initialize optics manually. To do this, click on the Initialize Optics button in the Spectrometer Control window.
Hg Realign *(simultaneous spectrometers only)*	To compensate for spectrometer drift (for example, changes in barometric pressure), the system compares the mercury slit position recorded at the end of the wavelength calibration to the current position found on the detector.	The spectrometer must be in the ready mode before initiating a mercury realignment on Optima 3000 Family spectrometers. Use before adjusting the peak wavelength, a procedure which allows you to fine-tune the calibration for a particular wavelength. For more information, see *Adjusting the Wavelength* in Chapter 9, *Using the Examine/ MSF Window*. Use before collecting data for MSF models.

Control	What it Does	When to Use
Detector Calibration	The system collects and processes the detector signal bias and dark current data in order to quantify signals from the detector properly.	Perform this procedure when: • the instrument or software is first installed • the instrument is moved to a new location • after reinstalling or upgrading the software • before checking detection limits **Note:** On scanning CCD ICP spectrometers, a detector calibration is performed automatically when you start the software.

Performing a System Wavelength Calibration

This section covers the procedure for simultaneous ICP spectrometers.

Perform this procedure:

- when the instrument or software is first installed

- when the instrument is moved to a new location

- after reinstalling or upgrading the software

- if analyte peaks in the Examine Spectra window are significantly offset from the central line for the wavelength on a consistent basis

Notes   If your peaks are slightly offset, a system wavelength calibration is usually not necessary. Instead, fine adjustments to wavelengths can be made using the Examine Spectra window. For more information, see *Examine Spectra Window* in Chapter 9, *Using the Examine/MSF Window*.

If using MSF and a wavelength calibration is performed, you must recreate your MSF models before performing an analysis for accurate results.

Important   Since spectrometers are sensitive to thermal effects, it is important that the instrument be operated with the plasma on for at least one hour before initiating wavelength calibration on Optima 3000 Family spectrometers.

Solutions You'll Need for Wavelength Calibration

• Calmix 3 solution (Part No. N058-2152) available through PerkinElmer, which contains 100 mg/L of P, K, S; and 20 mg/L of As, La, Li, Mn, Mo, Ni, Sc, Na.

• For spectrometers that have a visible wavelength channel on the detector, you will need VIS Wavecal mix (Part No. N930-2946) available through PerkinElmer, which contains 1 mg/mL Ba, Ca; and 10 mg/L of La, Li, Mn, Na, Sr; and 50 mg/L of K.

• Rinse solution

To initiate a wavelength calibration

1. Make sure that the plasma is lit and has been on for at least one hour on Optima 3000 Family spectrometers. On other PerkinElmer spectrometers, wait one-half hour before starting the wavelength calibration.

2. In the **Tools** menu, click on **Spectrometer Control**.
   The Spectrometer Control window appears.

To perform UV channel wavelength calibration:

1. Aspirate the Calmix 3 solution (Part No. N058-2152).

Note   On Axial (XL) instruments, you may need to dilute the wavecal solution
1:10 to keep certain emission lines from saturating the detector. On Dual
View (DV) instruments, select radial viewing. If you have selected axial,
a message appears informing you to change to radial.

2. Select **UV**, then click on **Wavelength Calibration** in the Spectrometer Control
    window. In the dialog that appears, click on **OK** to perform the calibration.

The UV calibration may take several minutes. You may view the spectra used for
calibration by clicking on **Spectra** in the toolbar. When the UV calibration is
complete, the UV coefficients and RMS (Root Means Square) value will be
displayed in the Results window. These values should meet the following
specifications.

UV Coefficients and RMS	Specification
First Coefficient	absolute value < 2.0
Second Coefficient	absolute value < 2.0
Third Coefficient	absolute value < 8.0
RMS measures variability of wavelength offsets)	< 2.0

If these specifications are not met, repeat the wavelength calibration and make sure
all peaks can be seen in the Spectra Display window. If the specifications are still
not met, a complete optical alignment may be required. Contact your PerkinElmer
service engineer.

For spectrometers that have a visible wavelength channel on the detector, to
perform VIS channel wavelength calibration:

1. Aspirate VIS Wavecal mix (Part No. N930-2946).

Note On Axial (XL) instruments, you may need to dilute the wavecal solution 1:10 to keep certain emission lines from saturating the detector.On Dual View (DV) instruments, select radial viewing. If you have selected axial, a message appears informing you to change to radial.

2. Select **VIS**, then click on **Wavelength Calibration** in the Spectrometer Control window.

The VIS calibration may take several minutes. You may view the spectra used for calibration by clicking on **Spectra** in the toolbar. When the VIS calibration is complete, the VIS coefficients and RMS (Root Means Square) value will be displayed in the Results window. These values should meet the following specifications.

VIS Coefficients and RMS	Specification
First Coefficient	absolute value < 3.0
Second Coefficient	absolute value < 3.0
Third Coefficient	absolute value < 12.0
RMS measures variability of wavelength offsets	< 2.0

If these specifications are not met, repeat the wavelength calibration and make sure all peaks can be seen in the Spectra Display window. If the specifications are still not met, a complete optical alignment may be required. Contact your PerkinElmer service engineer.

Wavelength Calibration Dialog

You use this dialog to perform wavelength calibration for the spectrometer.

Wavelength Calibration Dialog for Simultaneous ICP Spectrometers

This dialog appears only for simultaneous ICP spectrometers.

The Wavelength Calibration dialog for Simultaneous ICP Spectrometers.
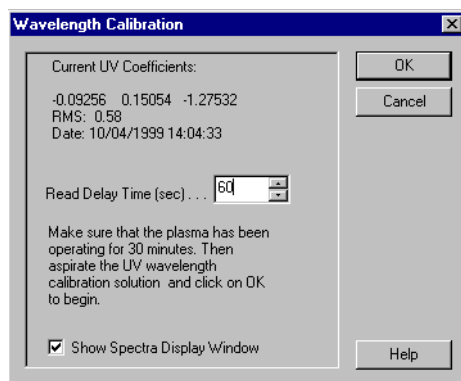
To display this dialog

▶ In the **Spectrometer Control window,** click on **Wavelength Calibration.**

Information in this dialog

Current values for the UV or VIS coefficients and RMS (Root Means Square) value are displayed in this dialog. These values indicate whether the current wavelength calibration meets specifications. You may want to note these values before clicking on **OK** to begin the wavelength calibration.

For more information, see *Performing a System Wavelength Calibration* earlier in this chapter.

Performing a Mercury Realignment

This function is available only on simultaneous ICP spectrometers.

A mercury realignment compares the known wavelengths of the internal mercury lamp with the found positions on the detectors and is used to compensate for spectrometer drift. It is especially useful before collecting data that you will use for a Wavelength Adjustment (Examine Spectra window) or MSF.

Note   In the Automated Analysis Control window, you can specify a mercury realignment (called Auto Wavelength Realign) to occur at defined intervals during an automated analysis (one-hour intervals are recommended). For more information, see *Automated Analysis Control: Set Up Page* in Chapter 5, *Analysis Control Windows.*

To perform a mercury realignment

Important   Since spectrometers are sensitive to thermal effects, it is important that the spectrometer is turned on for at least one hour before initiating mercury realignment on Optima 3000 Family spectrometers.

1. In the **Tools** menu, click on **Spectrometer Control**.
   The Spectrometer Control window appears.

2. Click on **Hg Realign** to display the Hg ReAlignment dialog. Click on **OK** to start the mercury realignment.
   The routine takes about one minute.

Mercury Realignment Dialog

This function is available only on simultaneous ICP spectrometers.

During a mercury realignment, the spectrometer compares the mercury slit position recorded at the end of the wavelength calibration to the current position recorded on the detector.

To display this dialog

▶ In the **Spectrometer Control** window, click on **Hg Realign**.

Information in this dialog

This dialog displays the peak offset for the mercury realignment performed during Wavelength Calibration and the peak offset, drift, and slit adjustment for the last mercury realignment. You may want to note these values before clicking on **OK** to begin Hg Realign.

For more information, see *Performing a Mercury Realignment* earlier in this chapter.

Performing a Detector Calibration

The system collects and processes the detector signal bias and dark current data in order to quantify signals from the detector properly.

Note On scanning CCD ICP spectrometers, a detector calibration is performed automatically when you start the software and takes 15 to 30 seconds.

Perform this procedure when:

• the instrument or software is first installed

• the instrument is moved to a new location

• after reinstalling or upgrading the software

• before checking detection limits.

The plasma does not need to be on for this procedure.

To perform a detector calibration

1. In the **Tools** menu, click on **Spectrometer Control**.
   The Spectrometer Control window appears.

2. Click on **Detector Calibration** in the Spectrometer Control window to display
   the Detector Calibration dialog. Click on **OK** to begin Detector Calibration.
   The system automatically closes the shutter to measure signal bias and dark
   current. The detector calibration routine may take up to twenty minutes.

Detector Calibration Dialog

This dialog displays information about detector calibration.

To display this dialog

▶ In the **Spectrometer Control** window, click on **Detector Calibration**

For more information, see *Performing a Detector Calibration* earlier in this
chapter.

Detection Limit Diagnostics - Performance Check

The Detection Limit Performance Check calculates detection limits for analytes, the results of which are displayed in the Mean Data and Replicate Data sections of your analysis report. You can use this Performance Check to evaulate how well your instrument is reading.

To display this dialog:

1. From the **Tools** menu, select **Spectrometer Control**.
   The Spectrometer Control dialog is displayed.

2. Click on the **Performance Checks** button.

To use this window:

1. Use the **Replicate** spin box to select the number of replicates.

2. Use the **SD** spin box to enter the factor to multiply by the standard deviation to calculate the detection limit.

3. In the **ID** text box, specify a name to identify the sample.
   By default "DL Check" is used as the sample ID.

4. Select which set of **Concentration Units** will be used for detection limit calculations. Your choices are: **Calibration** units or **Sample** units.

5. Select the Manual radio button if you wish to run manually.

   OR

   Select the **Autosampler** radio button if you wish to run with an autosampler.
   If you select Autosampler, the **Location** spin box becomes enabled.
   From the Location spin box, specify the sample location.

6. Open a method that contains at least one analyte with a valid calibration curve. The equation used cannot be Bracketing or Method of Additions.

7. Click **OK** on the Performance Checks dialog to begin the Detection LImit analysis.
    The Performance Checks dialog closes and the Detection LImit Checks analysis begins.
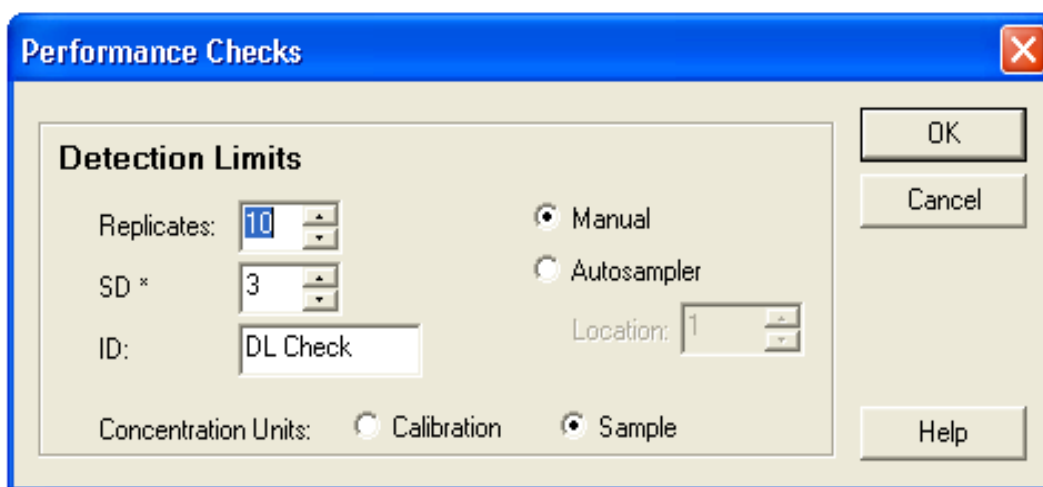
FIAS Control Window

You use the FIAS Control window to manually control the FIAS pump module and view the progress of the FIAS program. The status of the FIAS pump module and the FIAS program are shown at the top of this window. The FIAS On/Off and Goto Next Step buttons will always be disabled since there is no FIAS program in this configuration.

FIAS Control Window: A Quick Look
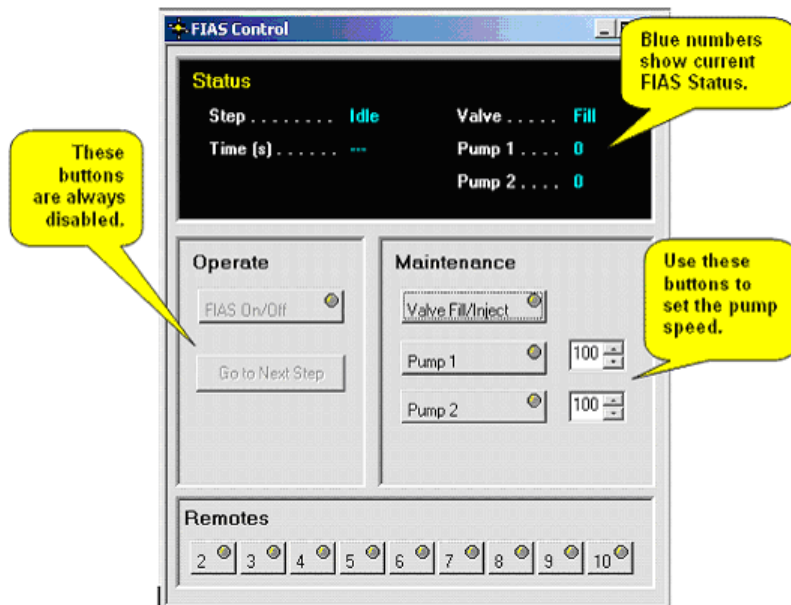
To display this window

▶ On the Toolbar, click on **FIAS** or In the Tools menu, click on FIAS Control.

Controls and Displays

You use the FIAS Control window to manually control the FIAS pump module and view the progress of the FIAS program. The status of the FIAS pump module and the FIAS program are shown at the top of this window.

You can only use these controls when there is no analysis in progress.

Valve Fill/Inject

To change the position of the valve, click on this button.

Fill -- The sample loop is filled with sample.

Inject -- The sample in the sample loop is injected into the carrier stream.

Pump 1

To start or stop the pump, click on this button. To the right of the button you can select a value for the speed of pump 1.

Pump 2

To start or stop the pump, click on this button. To the right of the button you can select a value for the speed of pump 1.

Remotes

Remotes 2 through 10 are switches that you use to control instruments that are connected to the Remote contacts on the rear of the flow-injection unit. Remote 1 is always used to trigger the Read function on the spectrometer.

Select the box to switch the remote on; a cross appears in the box. Clear the box to switch off the remote.

Controlling a FIAS Pump

When a FIAS is configured, its pumps, valve, and remotes are controlled directly via the FIAS Control window with one exception. When the plasma is extinguished for any reason (turned off by the user, or due to a hardware error), the FIAS pumps (if a FIAS is configured and connected), will turn off.

The following processes are not affected by the presence of a FIAS:

• "ICP peristaltic pump. If you do not want to use the ICP peristaltic pump while using the FIAS, you must turn it off manually by setting the pump speed to 0. In this case, 0 should be considered a valid speed; no functionality should be disabled due to a speed of 0.

• Startup/Shutdown. The Pumps Off At End feature (which automatically turns the FIAS pumps off at the end of an analysis), is not used in ICP. Of course, as described above, if the plasma is automatically shut down at the end of an analysis, the FIAS pumps will be turned off as part of that process.

Information Display Windows 7

About Information Display Windows

An overview of the Information Display Windows follows:

Spectra Display Window

View spectra and associated information, as each spectrum is generated during an analysis.

The Results Display Window

View a list of the analytical results as they are generated from an analysis.

You can also print a paper copy report of the analytical results shown in the Results Display window.

Calibration Display Window

View the calibration curves:

- during routine analysis to visually check the calibrations.

- during Method Development to ascertain an appropriate curve fitting algorithm.

Autosampler Loading List

View the samples listed according to the autosampler location number.

Spectra Display Window

This window displays the spectra of analytes by sample either generated from the analysis or reprocessed from stored data. You can specify the number of panes (and thereby spectra) you wish to view at one time.

Use this window to:

- Evaluate the spectral data from your solutions during an analysis.
- Check the progress of an analysis.

Note Since the y-axis of every signal is expanded automatically, even baselines can look like analyte signals. You must look carefully at the emission scale to distinguish background from actual spectra. A convenient alternative is to view representative spectra (of blanks, standards, and samples) that are overlaid in the Examine window. See the *Select Scale & Offset Dialog* in Chapter 9, *Using the Examine/MSF Window,* where you can maintain a constant y-axis scale, if desired.
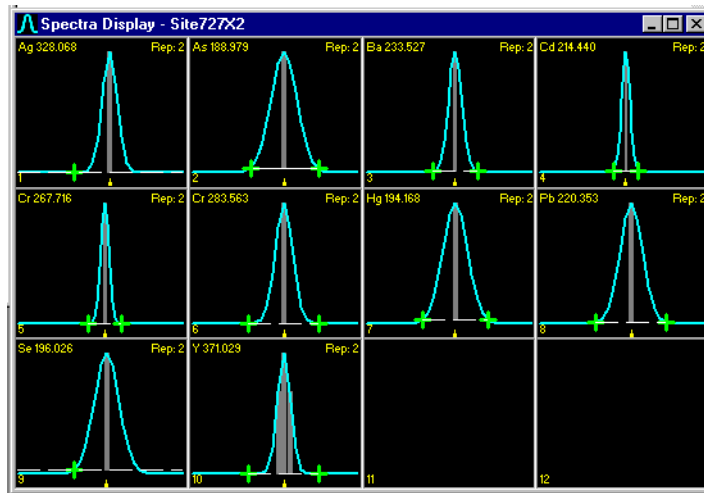
The Spectra Display window showing the spectral results of a soils analysis.

To display this window

▶ On the **Toolbar**, click on **Spectra**.

- or -

▶ In the **Tools** menu, click on **Spectra**.

Selecting Layout and Colors

You use the Spectra Display Options dialog to select the layout and colors for both the on-screen and printed Spectra window. To select this dialog, double-click anywhere inside the Spectra Display window or select Options menu ▶ Spectra Display.

Using the Spectra Display Options dialog, you can select the layout and number (and, therefore, spectra to be viewed at one time). You can also select colors to display on screen and for printing the Spectra display window.

In addition, if you are using Multicomponent Spectral Fitting, you can select whether or not to display the MSF analyte spectra along with the uncorrected spectra.

Resizing, labeling, and order of spectra

You can resize the Spectra Display window by placing the cursor on any side, or corner, of the window until the cursor changes to a double-headed arrow shape. Click and hold the left mouse button as you drag the mouse and change the window size.

- Resizing the window changes the size of each pane but not the number of panes.

- As the size is increased, you can view additional information, which includes the Intensity and Concentration values for every element to be measured in that solution. Also, you can see the concentration units and range for the x- and y-axes.

- The number in the lower left-hand corner of a pane corresponds to the row number of the element in the Method Editor. To rearrange the order of the elements in the panes, you can reorder the elements in the Method Editor.

- The number in the upper right-hand corner of a pane represents the most recent replicate analyzed.

Note   If an analyte is displayed as "saturated," you can correct for the saturated data. See *Correcting Saturated Samples* later in this chapter for more information.

Spectra Display Window: Printing Spectra

You can print the spectra that is shown in the **Spectra Display** or **Examine Spectra** windows. To adjust the printout colors, from the Options menu select Spectra Display or Examine Spectra/MSF and change the colors in the Options dialog. If the printer color selections have been modified, and you wish to return to the default colors, click on **Printer** under the default color settings. Click **OK** to close the dialog.

To print spectra:

1. Click on the Spectra Display or Examine Spectra window to make it the active window. Note that the title bar of an active window is a different color from other windows. (The colors displayed on your screen depend on the Windows colors you have defined for your PC.)

2. In the File menu, click on **Print** ▶ **Active Window**.

3. In the Print dialog that appears, check that the correct printer is shown. If you need to select another printer, click the drop-down arrow next to the Printer Name field. Click on the **Printer name** to select it. To change other setup options such as the paper size, click on **Properties**.

4. Click on **OK** to start printing.

The Spectra Display window always uses a layout of three rows and two columns for printouts. Changing the layout on the screen will not affect the printout. Up to six spectra are printed on one page.

When you print the Examine Spectra window, the spectra are overlaid in one graph on the page.

Spectra Display Options Dialog

Using the Options menu ▶ Spectra Display ▶ Spectra Display Options dialog, you can select the layout and number of spectra to be viewed at one time. You can also select colors to display on screen and for printing a screen of the Spectra Display window. In addition, if you are using Multicomponent Spectral Fitting, you can select whether or not to display the MSF analyte spectra along with the uncorrected spectra.

To select a display layout

▶ To use the default layout of all spectra, place a check mark next to Auto-Layout. An appropriate number of rows and columns will automatically be selected to display all the spectra. For example, if there are 11 analytes, Auto Layout displays a 4 x 3 grid.

▶ To customize row and column layout, first remove the check mark next to Auto-Layout. Then click on the counter spin buttons to specify the number of rows and columns in the display. The number of rows and columns each range from 1 to 16, which means that 1 to 256 panes can be displayed at once.

Selecting Colors

To change Spectra Display screen colors:

1. In the Screen column, click on the drop-down menu for the item you want to change and select a color.

2. To change the colors without closing the Spectra Display Options dialog, click on **Apply**. You can see the effect of the screen color changes in the Spectra Display window and modify them if desired.

   - or -

   If you are satisfied with the colors, click on **OK** to apply the changes and close the dialog.

Note   To return to the original screen colors, click on the Screen button under Default Color Settings.

To change colors for a printout of the Spectra Display screen:

1. In the Printer column, click on the drop-down menu for the item you want to change and select a color.

2. To change the colors without closing the Spectra Display Options dialog, click on Apply. To see the effect of the printer color changes, you must print the Spectra Display window on a color printer.

    - or -

    If you are satisfied with the colors, click on **OK** to apply the changes and close the dialog.

Printing Spectra

▶ To create a printout of the spectra, select File ▶ Print ▶ Active Window. The spectra are printed in black and white or in color, depending on the type of printer you have. See *Printing Spectra* earlier in this chapter for more information.

Displaying MSF Analyte Spectra

▶ To display the corrected MSF analyte spectra, select the **Enabled** checkbox on the bottom of the screen. You can then view the corrected MSF spectra in comparison with the uncorrected spectra.

Selecting line widths

▶ To select line widths for displaying spectra, type a number from 1 to 10 to designate the relative line width for MSF Spectra and for All Other Spectra. MSF Spectra Line Width defaults to 1 and All Other spectra Line Width defaults to 2.

Smoothing

Check this option to smooth the displayed spectra. Analytical values are not effected.

Return to the Default Colors

1. To change all of the screen colors or printer colors back to their default settings, select the **Screen** or **Printer** buttons under Default Color Settings.

2. To change the colors without closing the Spectra Display Options dialog, click on **Apply**.

   - or -

   To apply the change and close the dialog, click on **OK**.

Return to the Default User-Configurable Options

1. To change all of the user-configurable options (other than the colors) in this dialog back to their default settings, select **Default** under the Default Options Settings section.

2. To change the user-configurable options without closing the Spectra Display Options dialog, click on **Apply**. You can see the effect of the changes in the Spectra Display window and modify them if desired.

   - or -

   If you are satisfied with the user-configurable options, click on **OK** to apply the changes and close the dialog.

Results Display Window

The Results Display window shows the measurements made on the current samples, such as the calculated concentration values and statistical information.

You use this window to display results that are generated from the Manual Analysis Control window, the Automated Analysis Control window, the Spectrometer Control window or the Data Reprocessing window. The format of the Results is not variable in this window, but you can create custom reports from the Data Manager Utility. (In the **File** menu, select **Utilities**, then **Data Manager**.)

The Results Display window showing the results of a soils analysis. Here, Yttrium is the internal standard. (The † symbol indicates results that have been corrected using internal standardization.)

```
2³₃ Results                                                        _ ▢ ✕
===========================================================================
Sequence No.: 8                          Autosampler Location:
Sample ID: Site720Z                      Date Collected: 1/15/99 2:49:45
Analyst:
Sample Wt: 2.025 g                       Sample Prep Volume: 100 mL
Dilution:                                Data Type:  Original

---------------------------------------------------------------------------
Mean Data: Site720Z
                  Mean Corrected       Calib                    Sample
Analyte             Intensity     Conc. Units    Std.Dev.   Conc. Units
Y 371.029            263433.7
Ag 328.068†            8771.7     1.789 mg/L      0.0277     88.35 mg/kg
As 188.979†               8.5     2.324 mg/L      0.3285     114.8 mg/kg
Cd 214.440†             215.3     0.203 mg/L      0.0002     10.04 mg/kg
Cr 283.563†            8553.4     2.064 mg/L      0.0009     101.9 mg/kg
Hg 194.168†               1.8     0.006 mg/L      0.0036     0.300 mg/kg
Pb 220.353†             109.4     2.059 mg/L      0.0168     101.7 mg/kg
Se 196.026†               5.2     0.246 mg/L      0.0126     12.15 mg/kg
```

To display this window

▶ On the **Toolbar**, click on **Results**.

- or -

▶ In the **Tools** menu, click on **Results**.

To print this window

All information shown in this window can be printed in one of three ways:

* automatically (See *Printing Results* later in this chapter for further information)

* by selecting **File ▶ Print ▶ Active Window** (while the Results Display window is opened). Then click **OK** on the Print dialog that appears.

* by selecting **File ▶ Active Window Preview** (while the Results Display window is opened).Click Print on the dialog that appears, then click **OK** on the Print dialog.

Tip When you clear the Results Display (Analysis ▶ Clear Results Display), the contents are written to a log file called results.log in the user's home directory (for example, C:\pe\Default User\Temporary Files\Results.log). This log file is erased each time you restart WinLab32. A log file is an ASCII text file that can be printed, modified in a text editor (e.g., Notepad) or saved.

Note The Results Display window can only contain 2000 lines of information at a time (approximately 12 pages). When the 2000-line limit is exceeded, new lines are added at the end of the display and old lines are removed from the beginning. The lines removed from the display are stored in results.log under the user's home directory, for example: C:\pe\Default User\Temporary Files\Results.log.

Using this window

Customize the Results Display by selecting the items you wish to include from Tools ▶ Method Editor ▶ Options page:
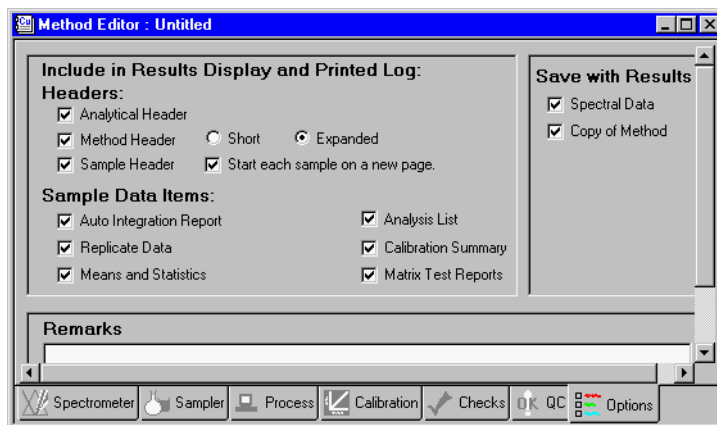
| Tip | To obtain the most abbreviated list of results, select only Means and Statistics from the Method Editor ▶ Options page. |

Select the type of information you want to include in the Results Display and the Printed Log for your analysis.

Analytical Header

Select this to include a header when a new analysis is begun. A new analysis header is also displayed each time the current method is modified and another sample is analyzed.

Method Header

Select this to include a header that contains information on the method. Select short or expanded.

Sample Header

Select this to include a header with sample information. Click the checkbox if you want to start each sample on a new page.

Auto Integration Report

Select this to include auto integration data for each measurement when auto integration is being used.

Replicate Data

Select this to include data for each replicate.

Means and Statistics

Select this to include the mean values of each set of replicates and to include the standard deviation and the relative standard deviation. You must have means selected in order to display statistics.

Analysis List

Select this option to include a summary of the operations that were performed on all of the samples.

Calibration Summary (Auto Analysis only)

Select this to include a calibration summary in the Results Display window. If this is selected, the calibration summary is automatically included for automated analyses after the last standard is analyzed.

Note   For manual analyses, you must select the Calibration Summary command in the Analyses menu.

Matrix Test Reports

If there is a sample matrix, mark this box to include matrix reports.

Save with Results: Spectral Data

Select this to include the spectral data in the results data set. The spectral data must be stored in a results data set if it is to be displayed in the Examine Spectra window or used for setting up MSF models or Data Reprocessing.

Save with Results: Copy of Method

Select this to include a copy of the method used to generate results in the results data set. A new copy of the method is included with the results each time the method is modified and another sample is analyzed. The method stored with the results can be recalled into memory by using the Import from Results Library... command in the File menu.

Remarks

Use this entry field to type your comments regarding the method.

Saving this window

When you clear the Results Display (Analysis ▶ Clear Results Display), the contents are written to a log file called results.log in the user's home directory (for example C:\pe\Default User\Temporary Files\Results.log). This log file is erased each time you restart WinLab32. A log file is an ASCII text file that can be printed, modified in a text editor (e.g., Notepad,) or saved.

Note   The Results Display window is not cleared automatically when you start a new analysis. To clear the window, select Analysis ▶ Clear Results Display.

An overview of this window

The following are the major sections that may appear in the Results Display window:

• Analytical Header

• Auto Integration Header and Information

• Replicate Header and Information

- Mean Header and Information

- Calibration Summary Header and Information

- Sample Header

- Method Header

For information on saturated samples, see *Correcting Saturated Samples* later in this chapter.

Results Display Window: Analytical Header

A discussion of each entry from the Analytical Header appears below:

Start Time

The date and time when the current analysis began.

Note   To set the format for Date/Time, from the Windows Control Panel click the Date/Time icon and enter the information in the dialog box.

Logged In Analyst

The name of the Analyst taken from the Windows system log in.

Spectrometer Model

The Spectrometer Model selected in the current configuration.

Plasma On Time

The time the plasma was ignited.

Technique

The name of the sampling technique selected in the Select Sampling Technique dialog. (ICP, Continuous, ETV, AA - Flame, fumes.)

Autosampler Model

The type of Autosampler model you are using.

Sample Information File

The name of the Sample Information File (.SIF file) and the full pathname.

Results Data Set

The name of the Results Data Set. A Data Set contains the essential components of an analysis, i.e., the Method, the spectral data, Sample Information and the analytical results. You specify the Results Data Set name when any one of the following actions is performed:

*   Manual Analysis Control window ► Results Data Set Name (Open button) ► Results Data Set dialog

*   Automated Analysis Control window Set Up tab ► Results Data Set Name: (Open... button) ► Results Data Set dialog

*   Data Reprocessing window ► Save Results in Data Set (Open... button) ► Results Data Set dialog

Note   Look at all of the selections on the Method Editor window ► Options page to get an overview of the selected Analytical Results that will appear in the Results data set or the Results window.

Results Library

The name and location of the Library where the Results are saved.

Results Display Window: Auto Integration Header and Information

Auto Integration entries:

Analyte

The name of the analyte.

Integration Time

The time period in seconds in which the detector collects light from the plasma before dumping the charge and reading out the signal.

Number of Integrations

Read time divided by integration time gives you the number of integrations performed per replicate.

Read Time

The total measurement time in seconds per replicate.

Refer to the Method Editor for a full discussion of each parameter.

Results Display Window: Replicate Header and Information

Following each sample header is the replicate header along with the results for each analyte. (A replicate is a repeated read cycle performed on a sample and calibration solution.)

Replicate Data

The identification name you gave to the sample solution.

Replicate Number

The number of the current replicate in a series of replicates performed on each analyte. You select the number of total replicates for the analytes in the:

- Method Editor ▶ Spectrometer ▶ Settings tab

Analyte

The name of the element analyzed. The element symbol is shown along with the emission wavelength that was used. The element and wavelength are taken from the active method.

Net Intensity

The resulting intensity from the element analysis minus the background signal.

Corrected Intensity

The resulting intensity after an internal standard correction (for example, blanks, reagent blanks, internal standards, IEC models) has been applied.

Calibration Concentration Units

The concentration of the element in calibration units. You select these units in the:

•   Method Editor ▶ Calibration pages ▶ Calibration Units and Standard Concentrations page.

Concentration Sample Units

The concentration of the element in sample units. Conversion from sample results determined in Calib Units to results expressed in Sample Units involves corrections made for weight, volume, and dilution information supplied for samples in a Sample Information File (or the Sample Details dialog in a manual analysis), e.g., converting from mg/L to mg/kg. Corrections are also automatically made for converting units with order-of-magnitude differences, e.g. converting mg/L to µg/L.

You select these units in the:

•   Method Editor ▶ Calibration pages ▶ Equations and Sample Units tab or in the Sample Information Editor.

If the sample units in the Sample Information Editor differ from those in the Method Editor, the sample units in the Sample Information Editor will be used.

Analysis Time

The time when the current sample was analyzed.

Note   To set the format for Date/Time, open the Windows Control Panel and double-click on the Date/Time icon.

Results Display Window: Mean Header and Information

An explanation of the Mean information appears below:

Analyte

The name of the element analyzed at a specified wavelength. The element symbol is shown along with the emission wavelength that was used. The element and wavelength are taken from the active method.

Mean Corrected Intensity

For each sample, the mean corrected intensity. The mean concentration is expressed in standard units or sample units.

Concentration in Calibration Units

For each sample, the concentration calculated using the mean corrected intensity value.

Standard Deviations

For each sample, the standard deviation of the mean standard concentration values expressed in calibration units or sample units.

Sample Concentration Units

For each sample, the mean sample concentration expressed in sample units.

%RSD

For each sample, % RSD of either the corrected signal and the mean standard (or sample) concentration.

Information Line

Additional information about the sample measurements that may be of interest to you, e.g., standard applied, QC messages (including QC recoveries), etc.

Results Display Window: Sample Header

A discussion of each entry from the Sample Header appears below:

Sequence No.

This is the sequence in the run list.

Sample ID

The name you give to each sample. Can be any combination of letters and numbers up to 25 characters.

Analyst

The name of the person performing the analysis. This is entered in the Sample Information file.

Nebulizer Back Pressure

This is the argon flow rate for the nebulizer or sample carrier gas.

Valid range: 0 - 2.0 L/min.

(This option is not available on the Optima 3000 series instruments.)

Sample Wt:

When working with weight units, this is the sample weight entered in the Sample Information File.

Dilution

If a sample was diluted, the dilution factor (10ml to 100 ml is a factor of 10).

Autosampler Location

The location of the solution in the autosampler.

Date Collected

The date and time when the current sample was analyzed.

Note	To set the format for Date/Time, from the Windows Control Panel click the Date/Time icon and enter the information in the dialog box.

Sample Prep Volume

After a solid sample has been dissolved or a liquid sample has been acidified (or otherwise treated) the resulting solution is prepared to a specific volume. This is the Sample Prep Volume. The information is from the Sample Information File.

Data Type

Original or Reprocessed.

Results Display Window: Method Header

You can display a short or expanded method header in the Results Display window. Make a selection in the Method Editor ▶ Options tab.

A discussion of each entry from the Method Header appears below.

Method Name

The method name used for the analysis. You specify this name when you select

- File menu ▶ Save As... ▶ Method ▶ Save Method As dialog

IEC

The name of the Interelement Correction File (.IEC file) will appear only if you make the appropriate selections:

- Method Editor ▶ Process page ▶ Spectral Corrections tab (select IEC for an element under the Overlap Correction Column and select an IEC Table) and Results are saved.

Method Description

A brief description of the method used for the analysis. You specify this name when you select

- Method Editor window ▶ Spectrometer page ▶ Define Elements tab ▶ Method Description

Method Last Saved

The date and time of the method was last saved.

MSF

The name of the Multicomponent Spectral Fitting File (.MSF file) will appear only if you make the appropriate selections:

- Method Editor ▶ Process page ▶ Peak Algorithm..., tab (select MSF for an element under the Peak Algorithm Column). Also select an MSF Table on the Spectral Corrections page.

Method Header Expanded

The Expanded Method Header contains all the above entries, plus the additional entries listed below.

Analyte

The name of the element analyzed. The element symbol is shown along with the emission wavelength that was used. The element and wavelength are taken from the active method.

Calib Eq'n

This is the calibration equation that is specified in the current method. (A list of calibration types is shown on the Calibration page of the Method Editor window.) After the calibration curve has been determined, you can clear (delete) the calibration by clicking on Clear Calibration Blank from the Analysis menu.

Processing

Peak area, peak height or MSF.

View

Plasma viewing condition: Attenuated Axial or Attenuated Radial.

Internal Standard

Lists the name of the Internal Standard as it appears on the Spectrometer pages, Define Elements tab. If there is no Internal Standard, then None or N/A is displayed.

IEC

InterElement Correction, Yes/No.

Results Display Window: Calibration Summary Header and Information

The following information is shown in the Calibration Summary:

Analyte

The name of the element used for the calibration. The element symbol is shown along with the emission wavelength that was used for the calibration. The elements and wavelengths are taken from the active method.

Standards

The total number of standards applied. The standards are taken from the active method.

Equation

The type of calibration equation used to calculate the calibration curve, taken from the active method. Possible choices are linear, linear-bracketed, linear-thru-zero or non-linear. See the *Method Editor Calibration pages: Calibration Equations and Sample Units page* in Chapter 3, *Using the Method Editor*.

Intercept

The calculated y-intercept of the calibration curve. The y-intercept is the point where the calibration curve crosses the y-axis of the calibration plot. It represents zero concentration.

Slope

The calculated slope of the calibration curve. Slope represents the relationship of the increase in intensity to the increase in concentration. It is the ratio of the change in the y-axis to the change in the x-axis.

Curvature

The calculated curvature is the "a" coefficient in the equation (b = slope and c = intercept):

$$y = ax^2 + bx + c$$

where x is the concentration and y is the intensity for a non-linear calibration curve.

Correlation Coefficient

The calculated correlation coefficient. The correlation coefficient shows how well the data fit the calibration curve. A value of 1.000000 indicates a perfect fit.

Note The value will always be 1.000000 if your calibration curve has only two points (calibration blank and 1 calibration standard).

Reslope

The reslope standard is used to make small corrections to the slope of the calibration curve. Usually, a calibration blank is analyzed just before the reslope standard. The new reslope value is then compared with a multipoint curve.

Reslope is a time-saving device that lets you adjust a curve without running multiple calibration sites. The application uses the new curve (reslope) to determine the concentration values.

Results Display Window: Printing Results

You can automatically send a hard copy report of the analytical results to the printer as the results are generated.

Note If you want to be certain that all of the analytical results generated by the Optima are documented you are encouraged to print a log during the analysis.

To print a log of your results

▶ Make sure that there is a checkmark in the Print Log box in the Manual Analysis Control, Automated Analysis Control, or Data Reprocessing window. If it is blank, click on the checkbox if to select it. You must have a printer online. The default printer is automatically selected. Options for the printed log can also be selected on the Options tab of the Method Editor.

Correcting Saturated Samples

One or more of the samples you analyzed may indicate an intensity of "saturated." If an analyte saturates, you will either see the indication of "Saturated" in the Results Display window (listed under Net Intensity) or the Spectra Display window. In the Examine Spectra window, the sample ID will be crossed out and either the saturation intensity value or the word "Saturated" will appear in the Intensity column.

When an analyte saturates, it means that the measurement at the chosen wavelength could not be made because the intensity of the emitted light was too high to measure.

The reasons for an analyte saturating are:

• The sample contained too high a concentration of the analyte

• The wavelength chosen for the analyte was too sensitive

In some cases, saturation may occur if the read delay is not sufficient when the instrument takes an initial measurement (called a pre-shot) to determine the auto

integration time. Try increasing the read delay to allow the signal to be fully stabilized in the plasma before a measurement is taken.

The software displays a saturation error code or message to give further information. Refer to Saturation Codes.

To correct for saturation, do one of the following:

▶ Choose a less intense wavelength for the analyte.

To change the wavelength, open the Method Editor, and select a different wavelength for the element. Make this selection from the Spectrometer page. Save the method and then reanalyze the samples.

▶ On Dual View instruments, you can change the plasma viewing type from Axial to Radial.

By changing the viewing type to Radial, the intensity at the wavelength of interest may decrease.

Change the viewing type on the Sampler: Plasma page of the Method Editor. Save the method and then reanalyze the samples.

Note   On scanning CCD ICP spectrometers, you have the option of using Attenuated Axial or Attenuated Radial as the plasma viewing type in the method. Use these settings to provide lower intensities for samples of high concentration that would normally saturate the detector and for sensitive wavelengths.

▶ Dilute the sample.

To decrease the concentration of the element, you can dilute the sample. If you choose to dilute the sample, be sure to update the dilution information in the Sample Information File and save the file before reanalyzing the samples. See *Using the Sample Information Editor* in Chapter 4, *Sample Information Editor*, for more information.

Saturation Codes

The software provides the following saturation error codes or messages. In some cases, saturation is indicated during "preshot." This is an initial reading that is taken to determine the optimum auto-integration time. On the Options page of the method you can select Auto Integration Report which will give you the exact integration times determined during "auto-integration" or "pre-shot."

Code	Description
2	"Saturated2" appears in the Net Intensity column in the Results Display. This information also appears in the Spectra Display window. This code and message applies only to simultaneous ICP spectrometers. The intensity measured during pre-shot is too high for several wavelengths and the detector cannot read out the data. You may be able to rerun the analysis and select only one of the wavelengths to prevent saturation from occurring.
3	"Saturated3" appears in the Net Intensity column in the Results Display. This information also appears in the Spectra Display window. The intensity measured for the analyte during pre-shot is too high, even for a 1 ms integration (the shortest integration time available). Either the wavelength chosen for the analyte was too sensitive or the sample contained too high a concentration of the analyte.

4   "Saturated4" appears in the Net Intensity column in the Results Display. This information also appears in the Spectra Display window.

The intensity measured for the analyte during the replicate readings is too high. This may have occurred because the signal wasn't stabilized before the pre-shot measurement, so the signal intensity changed between the "pre-shot" reading and the time the replicate was measured.

For the Optima 2000/2100, in manual integration, the message "Saturation within survey window" appears in the Results Display. For either type of integration, the message "Saturated. Code = 4" appears in the Spectra Display.

First, try increasing the read delay and rerunning the analysis. If saturation still occurs, the wavelength chosen for the analyte was too sensitive or the sample contained too high a concentration of the analyte.

5   In auto integration, the message "Saturation outside of integration window" appears in the Results Display. A peak with too high an intensity was found outside of the auto integration window but within the survey window.

For the Optima 3000/ 4000/5000, in manual integration, the message "Saturation within survey window" appears in the Results Display. For either type of integration, the message "Saturated. Code = 5" appears in the Spectra Display.

For more information on the integration and survey windows, refer to the Spectral Windows page in the Method Editor. The saturated peak may not be the analyte of interest, but an interferent. If you are using background correction, be sure to check that the background correction points are not placed in the range where saturation is occurring, which can invalidate your results.

6　　　In auto integration, the message "Saturation outside of integration window" appears in the Results Display. In manual integration, the message "Saturation outside survey window" appears in the Results Display. A peak with too high an intensity was found outside of the survey window. For either type of integration, the message "Saturated. Code = 6" appears in the Spectra Display.

For more information on the integration and survey windows, refer to the Spectral Windows page in the Method Editor.

The saturated peak may not be the analyte of interest, but an interferent. If you are using background correction, be sure to check that the background correction points are not placed in the range where saturation is occurring, which can invalidate your results.

Calibration Display Window

The Calibration Display window shows the active calibration as it is being generated and when it is being used by the system. If this window is empty, there is no active calibration curve.

You use this window to**:**

- Determine whether the standards lie on the curve and compare this graphic display of the calibration to the correlation coefficient.

- Check that you have selected the correct algorithm (linear, linear-bracketing, inear through zero, and nonlinear) for the calibration.

- View the effect on the calibration after selection of different algorithms or other Method parameters (e.g., subtraction).

- Print this window for your reports to present a graphical display of the calibration.

- Reslope. Run a calibration curve with two or three standards (a multipoint curve). Use Reslope to determine a new value for the curve to compare it with the multipoint curve.

The Calibration Display window showing calibration curves for a soils analysis.)
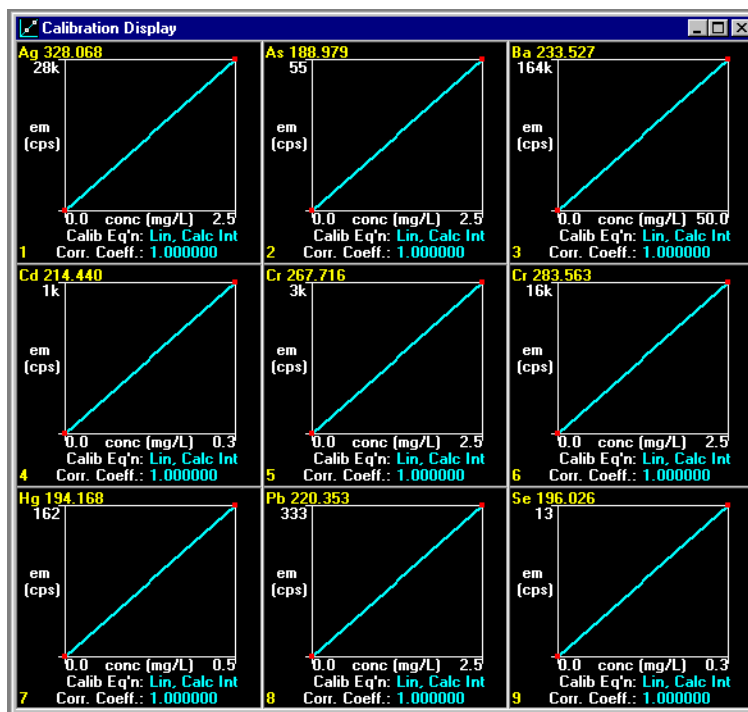
To display this window

▶ On the Toolbar, click on the **Calib** button .

- or -

▶ In the **Tools** menu, click on **Calibration**.

Selecting Layout and Colors

To select the layout, number of calibration curves to be viewed at one time, and colors for both the on-screen and printed Calibration window, you use the Calibration Display Options dialog. To display this dialog, double-click anywhere inside of the Calibration Display window. Or, in the Options menu, click on

Calibration Display. For information on printing, see *Printing the Calibration Display window* later in this chapter.

Reslope

Run a calibration curve with two or three standards (a multipoint curve). Use Reslope to determine a new value for the curve to compare it with the multipoint curve. The reslope curve, which appears as a dashed line, lets you adjust a curve without running multiple calibration sites. The application uses the new curve (reslope) to determine the concentration values.

Resizing, labeling and order

You can resize the Spectra Display window by placing the cursor on any side or corner of the window until the cursor changes to a double-headed arrow shape. Click and hold the left mouse button as you drag the mouse and change the window size.

- Resizing the window changes the size of each pane but not the number of panes.

- As the size is increased, you can view additional information, which includes the Intensity and Concentration values for every element to be measured in that solution. Also, you can see the concentration units and range for the x- and y-axes.

- The number in the lower-left corner of a pane corresponds to the row number of the element in the Method Editor. To rearrange the order of the elements in the panes, you can reorder the elements in the Method Editor.

Calib Eq'n

This is the calibration equation that is specified in the current method. (A list of calibration types is shown on the Calibration page of the Method Editor window.) After the calibration curve has been determined, you can clear (delete) the calibration by clicking on **New Calibration** from the Analysis menu.

Corr. Coeff.

The correlation coefficient indicates how well the data fit the generated calibration curve. A value of 1.0000 would indicate a perfect fit.

Calibration Display Window: Printing Calibration Curves

You can print the calibration curves shown in the Calibration Display window. To adjust the printout colors, from the Options menu select **Calibration Display** and change the colors in the Calibration Display Options dialog.

If the printer color selections have been modified and you wish to return to the default colors, click on the **Printer** button under Default Color Settings.

To print calibration curves:

1. Click on the Calibration Display window to make it active. Note that the title bar of an active window is a different color from other windows. (The colors that appear depend on the Windows settings you have chosen on your PC.)

2. In the **File** menu, click on **Print** ▶ **Active Window.**

3. In the Print dialog that appears, check that the correct printer is shown. If you need to select a different printer, click the drop-down arrow next to the Printer Name field. Click on the Printer name to select it. To change other setup options, such as the paper size, click on **Properties**.

4. Click on **OK** to start printing.

The calibration curves are always printed in a layout of two columns and three rows. Changing the layout on the screen will not affect the printout. Up to six calibration curves are printed on one page.

Calibration Display Options Dialog

Using this dialog, you can select the layout and number of calibration curves to be viewed at one time. You can also select colors to display on screen and for printing a screen of the Calibration Display window.To display this dialog:

▶ In the **Options** menu, click on **Calibration Display**.

- or -

▶ Double-click anywhere inside of the Calibration Display window.

To select a display layout

▶ To use the default layout of all calibration curves, select Auto-Layout. An appropriate number of rows and columns will automatically be selected to display all the calibration curves. For example, if there are 11 analytes, Auto Layout displays a 4 x 3 grid.

▶ To select rows and columns, first click the check box next to Auto-Layout to clear the check mark. This makes the row and column fields available. Then click on the counter spin buttons to specify the number of rows and columns in the display. The number of rows and columns each range from 1 to 16, which means that 1 to 256 panes can be displayed at once.

Selecting Colors

To change individual screen colors:

1. In the Screen column, click on the drop-down menu for the item you want to change and select a color.

2. To change the colors without closing the Calibration Display Options dialog, click on **Apply**. You can see the effect of the screen color changes in the Calibration Display window and modify them if desired.

    - or -

    If you are satisfied with the colors, click on **OK** to apply the changes and close the dialog.

Note   To return to the original screen colors, click on the **Screen** button under **Default Color Settings**.

To change colors for a printout of the Calibration Display screen:

1. In the Printer column, click on the drop-down menu for the item you want to change and select a color.

2. To change the colors without closing the Calibration Display Options dialog, click on **Apply**. To see the effect of the printer color changes, you must print the Calibration Display window on a color printer.

   - or -

   If you are satisfied with the colors, click on **OK** to apply the changes and close the dialog.

To Print Calibration Curves

To create a print out of the calibration curves, in the File menu, click ▶ Print ▶ Active Window. The calibration curves are printed in black and white or color, depending on the type of printer you have.

Return to the Default Colors

1. To change all of the screen colors or printer colors back to their default settings, select the **Screen** or **Printer** buttons under Default Color Settings.

2. To change the colors without closing the Spectra Display Options dialog, click on **Apply**.

   - or -

   To apply the change and close the dialog, click on **OK**.
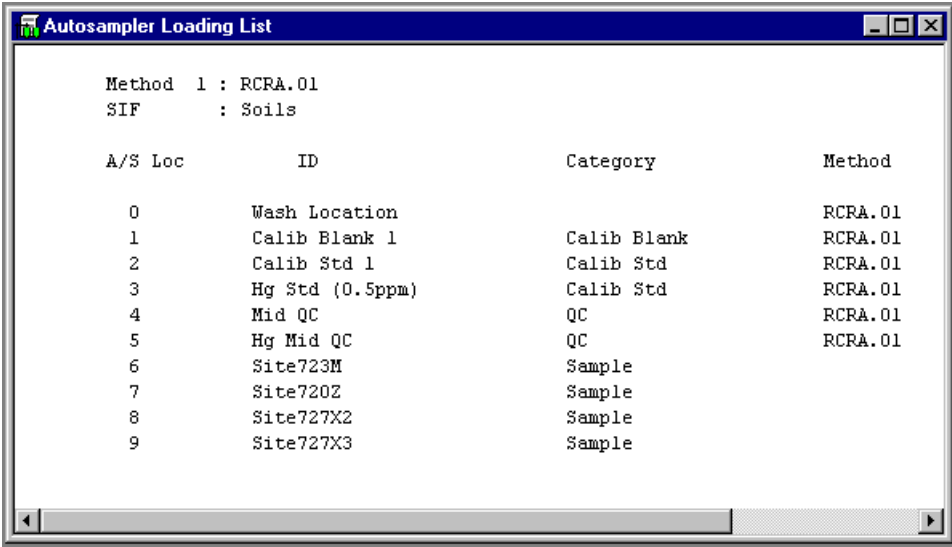
Autosampler Loading List

Use this window to view and print the list of solutions you need to fill the autosampler tray. The information shown is taken from the active method and the current sample information file.

To display this window

▶ Make the appropriate entries on the Set Up page of the Automated Analysis Control window and then click on the **Analyze** tab.

▶ In the System menu, click on **Autosampler Loading List.**

An example of an Autosampler Loading List window follows:

```
 Autosampler Loading List                                        _ □ ✕

      Method  1 : RCRA.01
      SIF       : Soils

      A/S Loc          ID                  Category          Method

         0          Wash Location                            RCRA.01
         1          Calib Blank 1          Calib Blank       RCRA.01
         2          Calib Std 1            Calib Std         RCRA.01
         3          Hg Std (0.5ppm)        Calib Std         RCRA.01
         4          Mid QC                 QC                RCRA.01
         5          Hg Mid QC              QC                RCRA.01
         6          Site723M               Sample
         7          Site720Z               Sample
         8          Site727X2              Sample
         9          Site727X3              Sample

```

Using this window

1. Set up an automated or manual analysis by selecting a method and sample information file.

2. In the Autosampler Loading List, review the following information: the name of the method used, the sample information file used and the blanks,

calibration standards and samples in the order that they are to be placed in the autosampler tray.

Note   In the list of autosampler locations and solutions, the method is not shown for samples (unknowns).

3.   To print the list, in the **File** menu, select **Print Active Window,** and in the print dialog that appears, click on **OK**.

Interferences & Interelement Correction

8

Spectral Interference Overview

The presence of Spectral Interferences can cause the results of an analysis to be incorrect. The software provides several techniques for minimizing or completely removing spectral interferences.

For information on WinLab32 interference correction techniques, see *About Interference Correction* later in this chapter.

About Spectral Interferences

Spectral interferences encountered in ICP-OES fall into four different categories, each with its own causes and remedies.

Simple Background Shift

	Cause	**Remedy**
A shift in background that is essentially constant over a given range. The background may shift either up or down.	A high concentration of the interfering analyte may emit a continuum of radiation in the given wavelength range	Set a **background correction point** somewhere near, but not falling on, the profile of the analyte you are measuring. Or select a different wavelength that is not affected by the interferent.
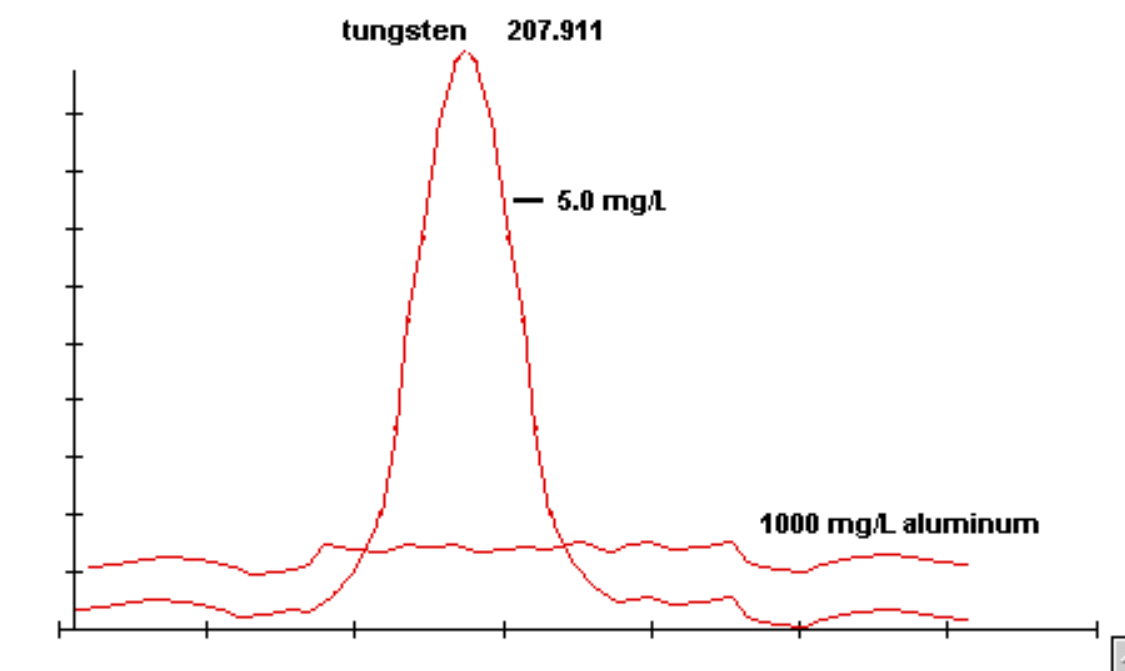
Sloping Background Shift

	Causes	**Remedy**
A shift that occurs on only one side of the given wavelength range	(1) Overlapping of an analyte line by one wing of another severely broadened line nearby	Since the slope of the interferent is constant on either side of the peak, set **two background correction points**, one on either side of the peak of the analyte you are measuring.
	(2) Molecular emission bands are sometimes present in the ICP discharge, especially when the plasma is not shielded from the ambient atmosphere.	

cadmium 214.438

1.0 mg/L cadmium

1000 mg/L aluminum
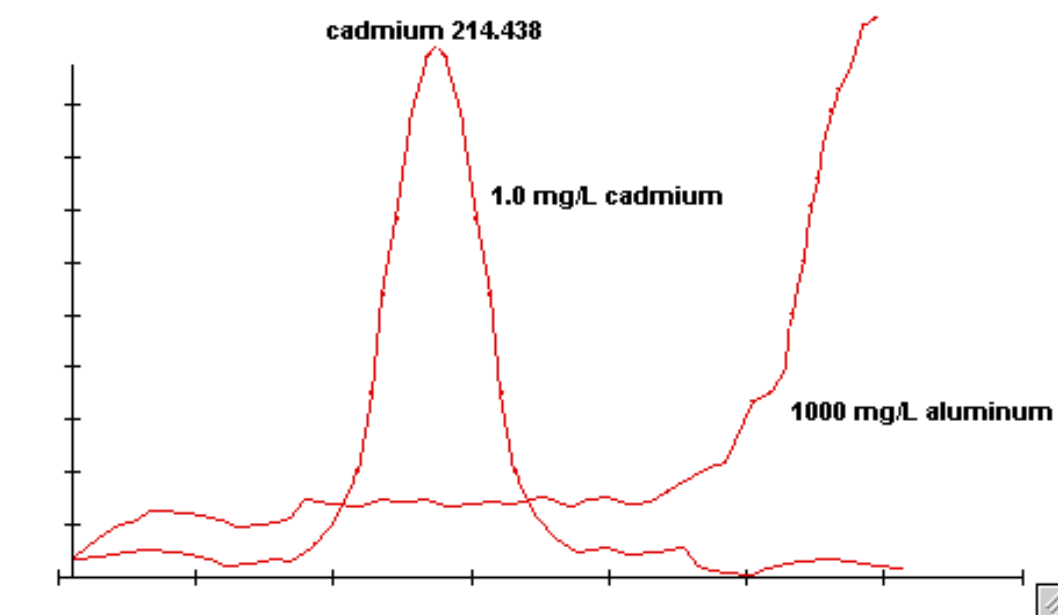
Direct Spectral Overlap

	Cause	**Remedy**
Ideally no interfering emission line can fall directly on the analyte emission line; however, because spectral lines have a finite width and measurement systems are imperfect, direct spectral overlap does occur.	Two lines may appear to be overlapped when they cannot be resolved by the spectrometer.	Use an alternate wavelength for the analyte you are measuring, if available. Or use the Inter-Element Correction (**IEC**) technique.

1000 mg/L platinum
platinum 267.715

5.0 mg/L chromium
chromium 267.716
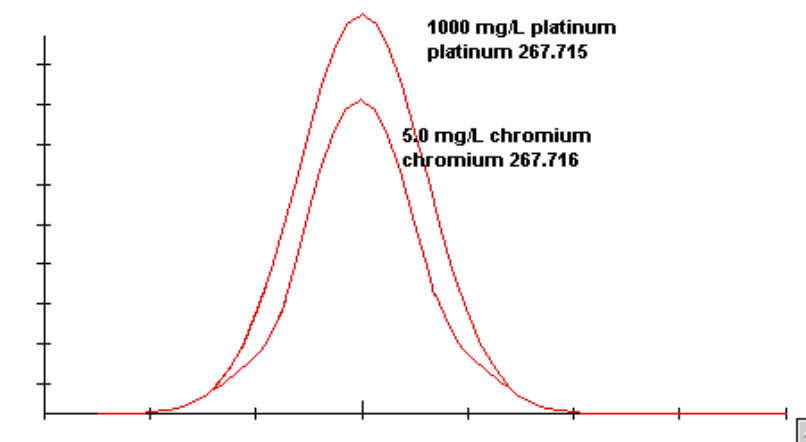
Complex Background Shift

	Cause	**Remedy**
This type of shift is represented by a shift in background intensity that varies significantly on either side of the analyte line.	This interference is usually caused by the occurrence of a number of intense, closely spaced emission lines nearby, and perhaps directly overlapping, the analyte wavelength.	Use an alternate wavelength for the analyte you are measuring, if available. Or use the Multi-spectral component Fitting (**MSF**) technique.

1.1 mg/L cadmium
cadmium 228.802

31 mg/L arsenic
arsenic 228.812
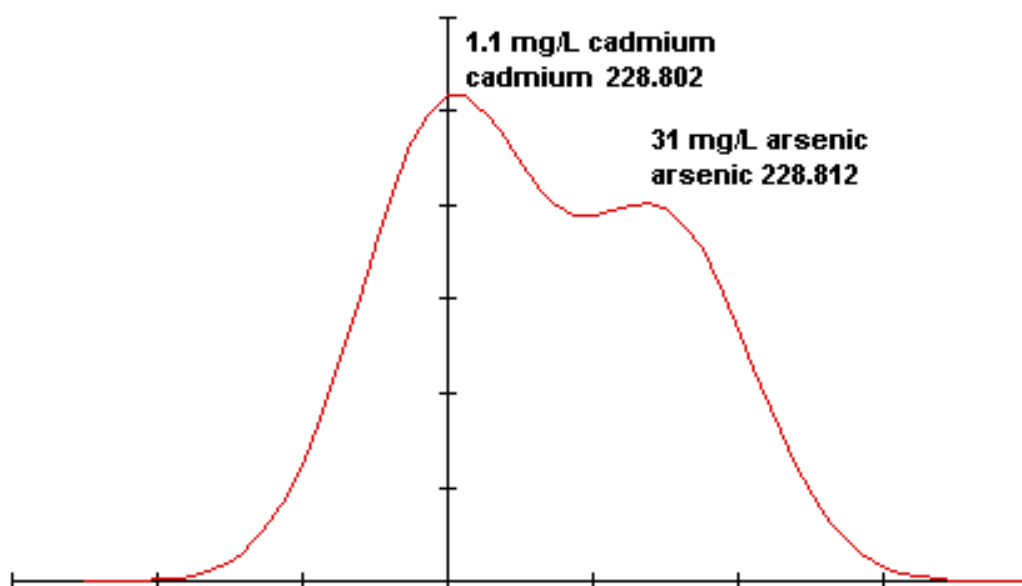
About Interference Correction

WinLab32 offers several methods to correct spectral interference.

Choose a topic that describes a method you are interested in learning more about:

- **Setting Background Correction Points**

- **Using Inter-Element Correction (IEC)**

    IEC corrects interferences as long as the concentration of the interfering element remains within the linear concentration range, the matrix remains constant, and the plasma conditions remain unchanged.

    For information on the similarities and differences between IEC and MSF, see *IEC or MSF: Which to Use* later in this chapter.

- **Using MSF (Multicomponent Spectral Fitting)**

    MSF uses mathematical models to distinguish analyte spectra from interfering spectra. MSF involves a very different process for interference correction as compared to IEC.

    As well as correcting for interferences, MSF can improve detection limits and precision. MSF uses a multiple linear regression to fit the models created to unknown spectra during an analysis. Since, only the peak shapes need to remain constant, typically, the models are independent of concentration, plasma conditions, and matrix effects.

    For information on how to apply MSF, see *Using the MSF Menu* in Chapter 9, *Using the Examine/MSF Window*.

    For background and tutorial information about MSF, see *Overview of How MSF Functions* in Chapter 9, *Using the Examine/MSF Window*.

IEC and MSF: Which To Use

IEC and MSF are two techniques you can use to minimize or remove spectral interferences. Inter-Element Correction (IEC) uses mathematical correction factors to reallocate emission intensities. Multicomponent Spectral Fitting (MSF) uses multivariate calibration to determine the concentration of an analyte.

Use IEC if...

- You expect direct spectral overlaps.
- You are taking data for use by an organization that requires Interelement Correction.

Use MSF if...

- You want better interference correction, better detection limits, and better precision than are normally obtained with IEC.

IEC and MSF: Similarities, Differences, and Restrictions on Use

Similarities

Both IEC and MSF:

- Involve mathematical adjustment/recalculation of intensity data

- Require an initial analysis to create a mathematical model

- Save the model as a file for use in the Process Page/Spectral Corrections tab of the method.

Differences

	IEC	**MSF**
Model created and edited	IEC Model Builder window	Examine MSF window
Model stored	IEC file	MSF file
Model used in the method	Overlap Correction set to IEC and IEC table name selected in method	Processing mode is set to MSF and MSF table name selected in method
Restrictions on use	You must use the same resolution, profiling, background correction points, and plasma parameter entries for IEC data collection as for end use.	You must use the same resolution and profiling settings for data collection as for end use.

Overview of the IEC Process

Interelement Correction (IEC) is a method of correcting for spectral interferences. It uses mathematical correction factors to reallocate emission intensities. Use the Interelement Correction Model Builder window to set up and update IEC corrections for the elements and wavelengths you are analyzing. IEC can be run using Data Reprocessing or in an analysis.

Before you use this window, you must complete the Preliminary Steps described below. Once the data have been collected, use the IEC window as a tool to interactively determine the factors. This window also gives you the option of changing certain method parameters and having the new settings incorporated into the calculated IEC factors.
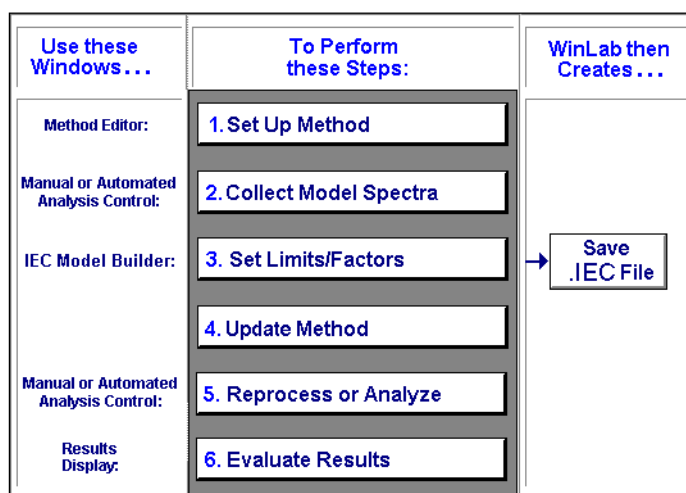
Preliminary Steps

Before you create an Interelement Correction Table (IEC), you must:

- Open the Method Editor and, in your method, define the calibration standards and a pure solution for each interfering analyte.

- Run an analysis, making sure you store the data so that it can be reprocessed using IEC.

- Once analysis is completed, set up an IEC model to calculate factors and set correction limits.

Using IEC

Here is a summary of how to use the IEC model.

- Open the IEC Model Builder from the Tools menu

- Create and save the model (the .IEC file)

- Specify the IEC file in the method and save the method

- Analyze your samples or reprocess your data

Notes   You should have the same Resolution setting, background correction points, and plasma parameter entries for IEC data collection as for the analysis you want to correct. If you change any of these parameters in the method, you should recreate IEC factors.

If you have some elements for which you want to create IEC factors for multiple wavelengths, refer to Multiple Wavelengths per Element.

If you do not follow the procedures in Multiple Wavelengths per Element, you will get errors if you try to collect IEC data for any element with more than one wavelength in the method.

Interelement Correction Model Builder

How IEC Works

When the calibration standards and interfering analyte solutions are read, WinLab32 builds a mathematical matrix that defines the concentrations observed for each analyte. This matrix, which is stored in the IEC table file, is a model of the spectral interferences expected from the elements you analyzed.

Each IEC standard solution is measured for the analytes you designate in the active method. (It is possible to deselect or "disable" elements from this analysis using the Enable/Disable Elements dialog.) The IEC correction factors are the ratios of the apparent concentration for each analyte to the concentration measured for the IEC reference element.

You do not have to analyze an IEC Standard solution for every element in your analysis, only those that produce interferences you want to correct for using IEC. If you skip IEC Standard solutions, WinLab32 assumes that there is no interference from these elements and will use zeros for these elements in the IEC table.

Creating an Interelement Correction Table

You create an Interelement Correction Table by entering information on the pages of the IEC Model Builder window. The following steps provide an overview of this process. For complete information about using each page, click on the page name in the steps below.

To create a new IEC table:

Note Before you set up the IEC model, run an analysis as you normally would and save the data.

1. In the Tools menu, select IEC Model Builder.

    The IEC Model Builder window Set Up page appears.

2. If another IEC model already exists, and you want to start a new one, select **New** from the File menu. Then select IEC Model. (This option is grayed out unless the IEC Model Builder window is open.)

3. On the Set Up page, click on **Open...** to select the Results Data Set to be used in determining the factors. Enter any remarks you want to save with the file.

4. On the Set Limits page you can enter correction limits for each analyte in the Minimum Concentration for Interference Correction column. Note that you can skip this page and set low factors to zero after calculation using the Calculate Factors page.

5. On the Calculate Factors page, select samples (the pure solutions of the interferents), and then match the various analytes requiring interference correction with the samples that contain the pure interfering element. Factors are automatically calculated after you choose an interfering analyte.

6. On the Summarize Factors page you can print the factors. You can also edit the factors on this page before saving.

7. Save the IEC model by selecting **Save** from the File menu. Then select **IEC Model**.

8. Click **Update Method** to update analytes in the method which require IEC correction.

To modify and re-save the IEC factors:

- If you modify the IEC factors and want to save the changes, on the **File** menu, select **Save** ▶ **IEC Model**.

- If you modify the IEC factors and want to save the changes but also retain the original file, from the **File** menu select **Save As** ▶ **IEC Model**.

- To save the information as a text file, from the File menu, select **Save As Text** ▶ **IEC Factors**.

To use an existing IEC model with a new result data set

You can recalculate the factors of an existing IEC model to use with a different results data set, i.e., a results data set other than the one you used to create the IEC model.

1. Create a new IEC table or open an existing one.

2. On the Set Up page, click **Open…** to select a different results data set.

   A warning message appears asking if you want to recalculate IEC factors.

3. Click on **Yes** to recalculate IEC factors based on the selected result data set. (If you click **No**, the current model is cleared.)

   The samples in the selected results data set are checked against samples in the IEC model.

   If the new results data set does not contain samples that are in the current IEC model, a message appears informing you that samples are missing.

   If the new results data set contains the same samples as in the IEC model, the new results set is checked for duplicate sample IDs. If duplicate sample IDs exist, you are given an opportunity to choose one sample from each group of duplicates. Samples from the new result data set are checked if they contain analytes that are part of the current IEC model. A message appears that describes each sample along with its missing analytes.

Modifying an Interelement Correction Table

You can modify an existing IEC table by reanalyzing IEC standards, analyzing new IEC standards or directly editing the table.

To edit IEC factors directly:

1. In the Tools menu, click on **IEC Model Builder.**
    The IEC window, Set Up page appears.

2. In the **File** menu, click on **Open ▶ IEC Model...** and select the IEC file name, or on the Set Up page, click on **Open…** to find and select the results data set used.

3. Enter new factors on the Calculate Factors Page.

To save the changes:

1. If you modified the IEC factors and want to save the changes, on the **File** menu, select **Save ▶ IEC Model**.

2. If you modified the IEC factors and want to save the changes but also retain the original file, from the File menu select **Save As ▶ IEC Model**.

3. To save the information as a text file, from the **File** menu, select **Save As Text ▶ IEC Factors**.

Multiple Wavelengths per Element

Refer to the following guidelines when you want to create IEC factors for elements with multiple wavelengths.

Note   If you do not use these guidelines, you will get errors if you try to collect or use IEC data for any element with more than one wavelength in the method.

▶   For a given analyte, you cannot have IEC factors for more than one Reference Element per interfering element.

For example, if multiple Fe lines are used in an analysis and the analyst needs to correct for Fe interference on an analyte line, only one of the Fe lines (i.e. Reference Elements) can have a non-zero IEC factor in the row for that analyte.

▶ Also, it is important when an IEC Standard for an interfering element with multiple lines in a method is analyzed, that the other "elements" defined for the interfering element are not analyzed as analytes. Otherwise, you would be making a correction for an element on itself.

For example, you would be correcting for the interference of Fe on Fe.

▶ To ensure that only one Reference Element per interfering element has IEC factors associated with a given analyte, it is best to analyze only one IEC Standard per interfering element. To avoid the situation where an element is corrected for interference upon itself, you can disable data collection for those elements by using the Enable/Disable Elements function. See the Analysis Menu in Chapter 11, *Menus and Toolbar*.

Example 1

As an example, suppose you have a method that contains three Fe lines named Fe1, Fe2, and Fe3. For the simplest case, we will use Fe1 as the Reference Element for all IEC corrections. To create IEC factors for Fe interference correction:

1. Calibrate using the method as usual using the Calib page.

2. When ready to analyze the Fe IEC Standard on the Analyze page:

   • Select Fe1 as the Reference Element.

   • In the Analysis menu, open the Enable/Disable function and disable Fe2 and Fe3.

   • Analyze the Fe IEC Standard.

Note Remember to re-enable Fe2 and Fe3 before measuring IEC Standards for other elements.

Example 2
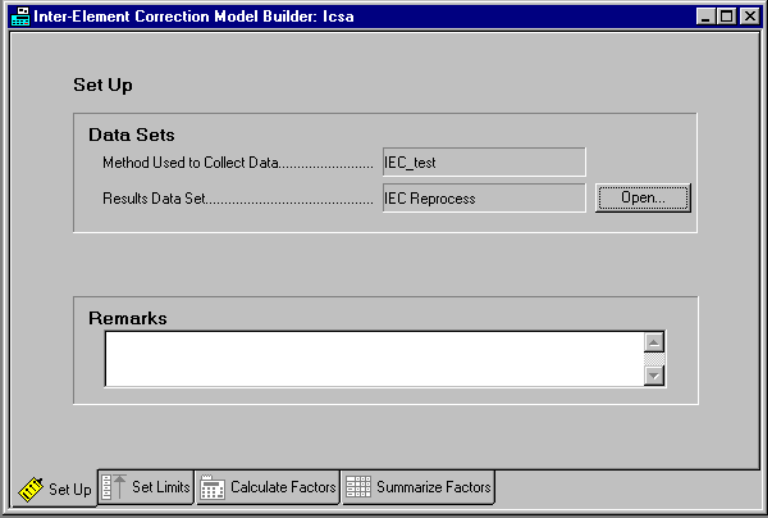
In a more complex example, suppose that you are using the same method containing three Fe lines, but you want to use Fe1 as the Reference Element for correction of Fe on As and Fe2 for correction of Fe on Cr. You may want to do a correction of this sort so that you can match an interferent atom line with an analyte atom line and interferent ion line with an analyte ion line to improve the quality of the interference correction. To create the Fe IEC factors for this situation:

1. Calibrate as usual.

2. To create the factor for correction of Fe on As,

    •   Select Fe1 as the Reference Element.

    •   Disable Fe2, Fe3, and Cr.

    •   Analyze the Fe IEC Standard.

    •   Select Fe2 as the next Reference Element.

    •   Re-enable Fe2 and Cr and disable Fe1 and As (Fe3 should still be disabled).

    •   Analyze the Fe IEC Standard.

Remember to enable the disabled elements before measuring other IEC Standards.

Interelement Correction Model Builder:
Set Up Page

Use this page to select the Results Data Set containing the data to be used in determining the factors. Enter comments in the Remarks field.

To display this page

▶ In the **Tools** menu, click on **IEC Model Builder**. The Inter-Element Correction Model Builder opens with the Set Up page displayed.

To use this page

1. If you already have a model that you want to change, from the **File** menu, click on **Open - IEC Model**.

   The Open dialog appears.

2. Select a file name, or enter a new file name.

3. Click on **OK**.

To open a data set:

1. Click the **Open** button on the Set Up page to search for an existing data set. The Select Results Data Set window appears.

2. Click the **Browse** button if you need to search for another data base. The Open dialog appears.

3. Search for and select the file.

4. Click on **Open**.

   The data sets are listed in the Select Results Data Set window.

5. Select a data set.

6. Click on **OK**.

After you have selected a data set, the file name appears in the Results Data Set field. The Method Used to Collect Data field is automatically filled in.
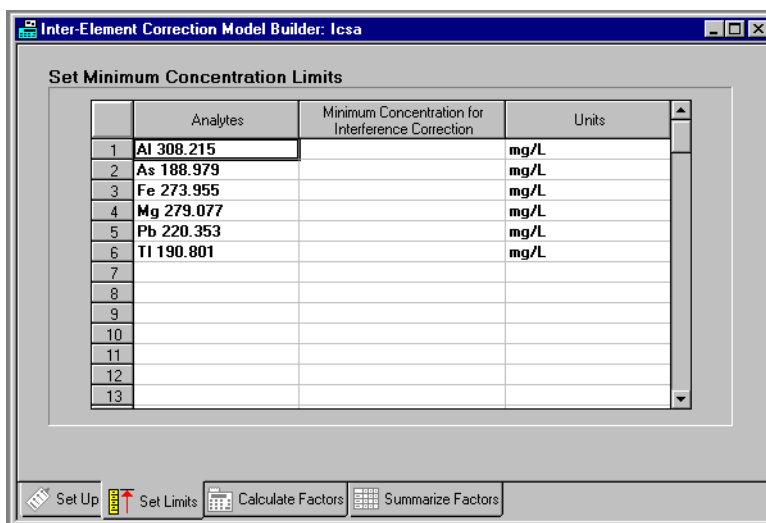
Remarks

Use this entry field to type your comments regarding the IEC table.

Interelement Correction Model Builder: Set Limits Page

On the Set Limits page of the IEC Model Builder you can define the correction limit for elements that you are measuring. For example, you can enter the detection limits of the analytes. Any apparent concentration from an interfering element at or below the value entered should be set to zero. Note that the units should be the same as the calibration units entered on the Calibration page in the method.

Note You may choose to skip this page and simply set low factors to zero after calculation using the Calculate Factors page.

To display this page

1. On the **Tools** menu, select IEC Model Builder.

2. Click the **Set Limits** tab.

To Use This Page

Analytes

This column automatically lists the analytes from the results data set chosen on the Set up page. Read through this list to make sure that these are the analytes for which IEC factors should be calculated.

Minimum Concentration for Interference Correction

You can manually change any entry in the Minimum Concentration column by typing in a new entry on the Set Limits page.

You can also double-click on the Minimum Concentration heading to display the Minimum Concentration Column Fill dialog. The concentration limit you enter in this dialog will be entered for all analytes on the Set Limits page. Click OK to close the dialog.

You can also select a range of entries or select noncontiguous entries and then display the Minimum Concentration Column Fill dialog.

▶ To select a range of entries, click on an entry, press the Shift key and click on another entry. All of the entries in between these two will be selected.

▶ To select non-contiguous entries, click on an entry, then press the Control key and click on additional entries.

Once your entries are selected, right click. When the pop-up Edit menu appears, click on **Column fill...** and the Minimum Concentration Column Fill dialog will appear. You can also display the Minimum Concentration Column Fill dialog by clicking on the Edit menu and selecting **Column fill...**.

Units

The units are the standard units, not the sample units.

Using Correction Limits

The correction limits are applied automatically and are used during IEC factor creation and during sample analysis.

During IEC factor creation:

• If an apparent analyte concentration (concentration measured at the analyte line, but due to the interferent instead of the analyte) is **greater than or equal to** the analyte's correction limit, an IEC factor for the analyte/interferent pair is calculated and entered into the IEC factors table.

• If an apparent analyte concentration is **less than** the correction limit, the IEC factor entered into the IEC table for the analyte/interferent pair is zero. This is because the effect of the interferent on the analyte was not sufficient to warrant a correction.

During sample analysis:

• If an apparent analyte concentration is **greater than or equal to** the correction limit, the software then applies the IEC factor to the analyte concentration and a final result is calculated and appears in the Results Display window.

• If an apparent analyte concentration calculated by applying IEC is **less than** the correction limit, correction for this analyte/interferent pair is not performed. This is because the effect of the interferent on the analyte was not sufficient to warrant a correction.

Minimum Concentration Column Fill Dialog

Use this dialog to enter a correction limit for each analyte before you collect IEC standard data. When you make an entry in this dialog, it fills in the Minimum Concentration for Interference Correction column for all the analytes listed on the Set Limits page of the IEC Model Builder.

To display this dialog

1. On the **Tools** menu, select **IEC Model Builder**.

2. Click the **Set Limits** tab to display the **Set Minimum Concentration Limits** page.

3. If desired, select a range of rows by shift-clicking in the column.

4. Double-click on the heading of the second column**, Minimum Concentration for Interference Correction**. (You can also right-click the column and choose Column Fill.) The Minimum Concentration Column Fill dialog appears.

Using this dialog

1. Enter the Limit number you want to appear in the Minimum Concentration for Interference Correction column.

   Usually, you type a value that is based on the detection limit for the analyte. This is the minimum apparent analyte concentration from an interferent for which interference correction should occur.

2. Click **OK** to close the dialog.

   The number you entered is listed in the Minimum Concentration for Interference Correction column for every analyte listed (or for the ones you selected).

3. You can change individual analyte limit numbers by clicking on the number listed in the Minimum Concentration for Interference Correction column and typing in a new number.

Note	You may choose to skip this page and simply set low factors to zero after calculation using the Calculate Factors page.

Interelement Correction Model Builder: Calculate Factors Page

The Calculate Factors page is used to match the analytes requiring interference correction with the samples that contain pure interfering element.

The formula for calculation is:

$$\text{Factor} = \frac{\text{Apparent concentration of analyte due to the interfering element measured at the analyte wavelength}}{\text{Actual concentration of interfering element measured at another wavelength characteristic of the interfering element}} \times 1000$$

For an example of how factors are calculated, see *Interelement Correction Model Builder: Summarize Factors Page* later in this chapter.
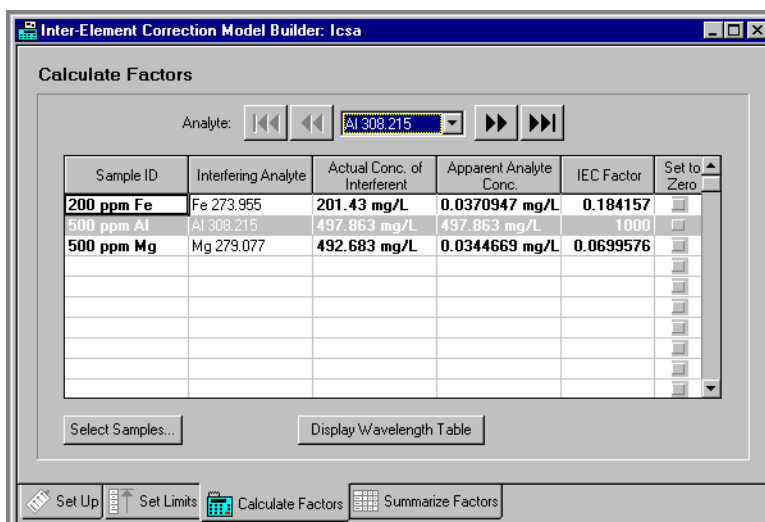
To display this page

1. On the **Tools** menu, select **IEC Model Builder**.

2. Click on the **Calculate Factors** tab.

Using this page

1. Select an analyte that requires IEC correction.

   Use the control buttons on the top of the page to scroll through the names of all the analytes in the active method.

2. Click on the Select Samples button or double-click on the Sample ID header. In the Select Samples dialog that appears, select the samples (that is, the pure solutions of the interferents) which will appear on the Calculate Factors page. Click on OK to return to the Calculate Factors page.

3. For each sample, designate the interfering analyte by selecting the analyte name from the drop-down list in the Interfering Analyte column. Select None if you want to turn off that sample during the calculation of factors for the current analyte.

4. Click on Display Wavelength Table to display the Wavelength Table dialog. Use this Table to search for elements at a particular wavelength or to choose wavelengths not found on the Method Periodic Table. Additional information such as intensities and BEC's are given for certain elements.

5. WinLab32 automatically calculates factors for all combinations of interfering analyte with the active analyte.

    The Actual Concentration of Interferent and Apparent Analyte Concentration and IEC factor are automatically entered.

6. Any factors that are very small can be set to zero by clicking on the check boxes under Set to Zero. Any factors not calculated are set to zero.

Note Any errors encountered are displayed in the IEC Factors column. Error types are:
#No sample: Sample ID not found.
#No analyte: Interfering Analyte not found.
#DIV/0: IEC Factor calculation encountered a divide by zero error.

User Entered IEC Factor Dialog

Use this dialog to set automatic calculation or manually enter factors for the Interferent Reference Analytes listed on the Summarize Factors page of the IEC Model Builder.

If the automatically calculated factor is not yielding the results you expect, you can enter an IEC factor manually. Also, your analysis may require that you enter a factor manually, such as an EPA factor (realizing that factors vary depending on plasma conditions, instrument type, etc.)

To display and use this dialog

1. On the **Tools** menu, select **IEC Model Builder**.

2. Click the **Summarize Factors** tab.

3. Double-click on the IEC factor shown for an Interferent Reference Analyte. For example, if P 213.617 is shown in Interferent Reference Analyte column with an IEC factor of 7.50, double-click on 7.50

   The User Entered IEC Factor dialog appears.

4. Click the radio button next to **Manually Set Factors** and enter a number. The number can be a predetermined factor, or it can be an offset of the automatically calculated factor, that may produce better results for the solution of a known concentration.

5. Click **OK** to return to the Summarize Factors page.

   The number you entered appears in the Interferent Reference Analyte column and row you selected.

Note   To reset the IEC factor back to automatically calculated, open the User Entered IEC factor dialog and click the radio button next to Automatic Calculation.

Interelement Correction Model Builder: Summarize Factors Page

Use this page to view the IEC table selected on the Setup Page, and, if needed, edit factors directly. The values calculated on the Calculate Factors page are transferred automatically to this summary page. You can update the method to use the factors and print the factors. You can also edit the factors on this page before you save them.

To display this page

1. On the **Tools** menu, select **IEC Model Builder**.

2. Click on the Summarize Factors tab on the bottom of the Interelement Correction Window.

Using this page

Each factor is applied to the analysis results of the analyte shown in the row to compensate for interference caused by the Reference Element in the column. If you want to change a factor, you can either edit directly (by double-clicking it), reanalyze standards or analyze new standards.

IEC Factors

Analytes

These are analytes from the method used to create the IEC table.

Interferent Reference Element

These are the names for the elements whose signals may be interfering with the analyte signal. Each analysis element is also a potential Interferent Reference Element.

For a given analyte, you cannot have IEC factors for more than one Reference Element per interfering element. For example, if multiple Fe lines are used in an analysis and the analyst needs to correct for Fe interference on an analyte line, only one of the Fe lines (i.e. Reference Elements) can have a non-zero IEC factor in the row for that analyte.

Double-click on an Interferent Reference Element IEC factor to display the User Entered IEC Factors dialog, where you can choose Automatic Calculation or Manually Set Factors.

Note   You can also enter a factor manually into the spreadsheet: Click on the entry and type the new factor.

Show

Click the radio button to either **Show All Analytes** and all factors, even the ones that are zero; or **Only Non-zero Factors**, which limits the display to only those analytes and interfering elements where at least one value in every row and column is not zero.

Update Method

After you save the IEC file (File/Save command), click the **Update Method** button to update the method in the Method Editor. When you click Update Method, the name of the IEC file is entered in the Method Editor, Process Page, Spectral Corrections page of the active method. The Interference Correction on all analytes with nonzero factors will be set to IEC. IEC is automatically selected to perform Overlap Correction for analytes with nonzero factors.

Print Factors

Click the **Print Factors** button to print the table of factors as currently displayed. Print the complete table showing all analytes, or the table that contains only analytes and interfering elements where each row and column contains at least one nonzero IEC factor.

Select the radio button to print in either Portrait (vertical) or Landscape (horizontal)

How Factors are Calculated

The value of each factor is equal to the apparent analyte concentration divided by the interferent concentration. The apparent analyte concentration is the concentration measured at the analyte line, but due to the interferent instead of the analyte.

$$\frac{\textbf{Apparent Analyte Conc.}}{\textbf{Interferent Conc.}} \textbf{ = IEC Factor}$$

When you analyze samples, the software multiplies the interferent concentration by the IEC factor. It then subtracts this product from the measured analyte concentration to result in the corrected analyte concentration.

$$\textbf{Measured Analyte Conc. - } \left(\textbf{Interferent Conc. } * \textbf{ IEC Factor}\right) \textbf{ = Corrected Analyte Conc.}$$

For example, suppose you are determining As and you know that Fe is creating an interference. You find that:

2000 ppm of Fe will create a reading of 20 ppb of As

The IEC factor is calculated at:

$$\frac{\textbf{20 As ppb}}{\textbf{2000 Fe ppm}} \textbf{ = 0.01 ppb As / ppm Fe}$$

You then analyze a sample with the measured results of:

As = 60 ppb and Fe = 4000 ppm

Multiplying:

4000 ppm Fe ∗ 0.01 ppb As / ppm Fe = 40 ppb As

40 ppb is the signal in As that is actually due to Fe.

The corrected concentration of:

As = 60 ppb (measured) - 40 ppb (due to Fe) = 20 ppb.

Select Samples Dialog

This dialog is used to select the samples that appear in the Sample ID column of the Calculate Factors page of the IEC Model Builder.

To display this dialog

1. From the **Tools** menu, select **IEC Model Builder**.

2. Click the **Calculate Factors** tab.

3. Click the **Select Samples** button or double-click on the Sample ID header. The Select Samples dialog appears.

Using this dialog

1. Click on the sample name to select it. To select all the samples listed, click **Select All**, or press the Shift or Ctrl key as you click to select a group or individual samples.

2. Click **OK** to close the dialog.

Using the Examine/ MSF Window 9

Overview of Examine/MSF

The Examine Spectra/MSF (Multicomponent Spectral Fitting) window serves a dual purpose:

• You can examine the spectra that have been saved to a data set.

• You can correct for spectral interference by setting background correction points or by using the MSF technique.

Refer to *About Interference Correction* in Chapter 8, *Interferences & Interelement Correction*, for more information on the different ways you can use the software to correct interferences.

To display this window

▶ On the **Toolbar**, click on **Examine**.

-or-

▶ In the **Tools** menu, click on **Examine/MSF**.

Using the Examine Spectra/MSF window for the first time

By default, this window opens in the Examine Spectra mode. In this mode, the word Examine is displayed in the top-left corner of the window. In Examine Spectra mode, you can:

• modify method parameters directly from the Examine window.

• modify the appearance of the spectra to aid in method development.

• set up spectral displays for reports.

For more information on using this window in Examine Spectra mode, see the next section, *Overview of the Examine Spectra Window* later in this chapter.

▶ To change this window to Examine MSF mode, select MSF from the drop-down list in the top-left corner of the window. For more information on using MSF, see *Overview of How MSF Functions* later in this chapter.

Overview of the Examine Spectra Window

Note	For information about using multicomponent spectral fitting (MSF), see *How to Use the MSF Window* later in this chapter.

The main function of the Examine Spectra window is to display spectra that were saved to a Data Set so that you can:

1. Modify Method parameters:

    •   Select Peak Wavelength and Background Correction Points directly from the graphic display.

    •   Select Peak Algorithm (peak height or peak area) and the Points/Peak based on your observation of spectra in the Examine Spectra window.

    Refer to the *Update Method Parameters dialog* later in this chapter.

2. Change the presentation of the spectra to aid in Method Development so that you can identify interferences and select appropriate wavelength(s) for an element, etc.

3. Set up spectral displays for your printouts.

4. Troubleshoot possible hardware problems by evaluating standard spectra. (e.g., not enough sample entering plasma, clogs, etc.)

5. Fine-tune the instrument wavelength calibration.

6. Correct saturated data. See *Correcting Saturated Samples* in Chapter 7, *Information Display Windows*, for more information.

To display this window

▶ On the Toolbar, click on **Examine** .

     - or -

▶ In the Tools menu, click on **Examine Spectra/MSF**.
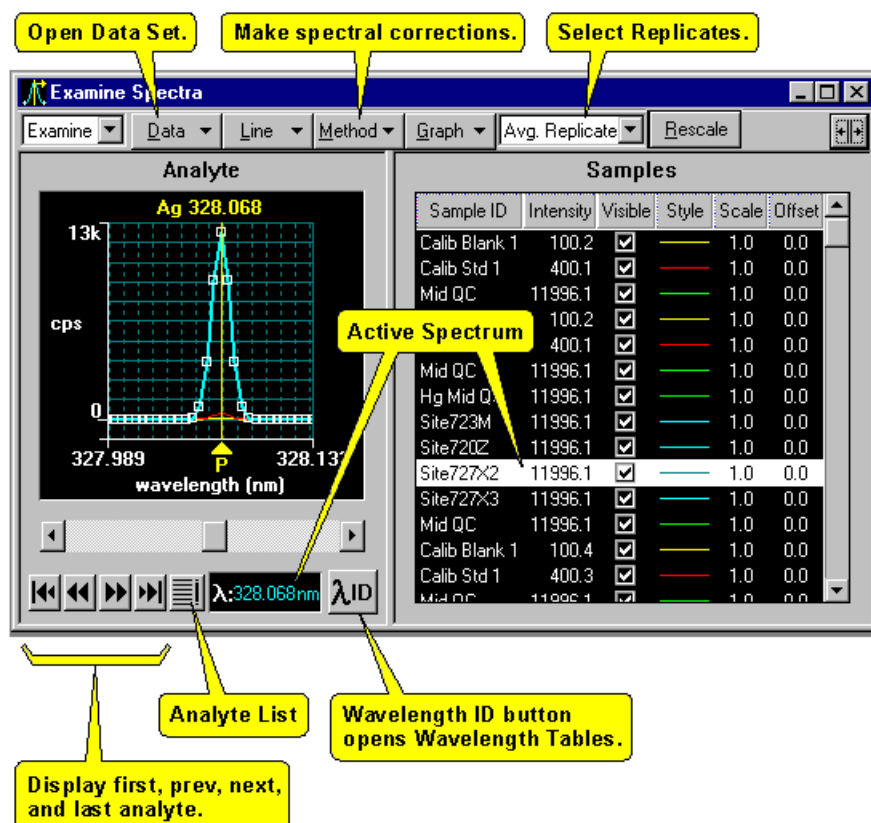
Using this window

Tip 1 If you want to modify the method, open it before you begin using the Examine Spectra/MSF window. In order for you to interact with the method, this method must contain the same elements and wavelengths as the one used for data collection of the samples (data set). To ensure that you save both the spectral data and the method used to collect them, make sure to select both the Spectral Data check box and the Copy of Method check box in the Method Editor window Options page. Optionally, you can open a data set from the Data Reprocessing window and determine the appropriate method from the Original Method column. You would then open the appropriate method (using the MethEd button on the Toolbar).

Tip 2 Save the method, if you wish to retain the changes made to method parameters from the Examine Spectra/MSF window.

Tip 3 Save spectra to the same data set if you will want to overlay these spectra in the same display because you can select only one Data Set per Examine session.

The lists and menus along the top of this window are:

Menu/List	Description
Examine or MSF:	Select either Examine or MSF to toggle the window between two different modes
Data menu	Select data set, samples, and analytes to work with
Line menu	Modify the spectra display.
Method menu	This menu is present only in Examine mode. Set and adjust peak wavelength, update method parameters.

Menu/List	Description
MSF Menu	This menu is present only in MSF mode. Set blank, analyte, interferents, mask for interference correction
Graph menu	Modify display options.
Replicates List	Display Average, First, or Last replicate

Analyte and Wavelength selection buttons:

• First, Previous, Next, Last analyte in the list. Use these buttons to change the currently displayed analyte.

• List of analytes button. Click this button to view a list of selected analytes. You can select any analyte from the list to display it.

• Wavelength display. Displays the wavelength of the cursor position.

• Wavelength ID button. Opens the Wavelength Table dialog.

How to Use the Examine Spectra Window

Note   For information on using MSF, see *How to Use the Examine MSF Window* later in this chapter.

Before you begin using the Examine Spectra window, open the method that was used to generate the spectra. If you are not sure which method was used, select Import from Results Library... in the File menu, then select the results data set. If you want to use a different method, it must contain some (but not all) of the elements and wavelengths that were originally used when the spectral data was collected.

Selecting spectra to display

1. Open the Examine Spectra/MSF window (in the Toolbar, click on the **Examine** button). Select **Examine** from the top-left drop-down list, if necessary.

2. In the Examine window, click on the Data menu, and then click on **Data Set**.

3. Complete the Data Selection Wizard to select samples and analytes.

The Examine Spectra window displays the first analyte.

What You See in the Examine Spectra Window

The window is split vertically with the **graph** on the left and the **legend** on the right. The graph shows all spectra of a chosen analyte for the sample selected.
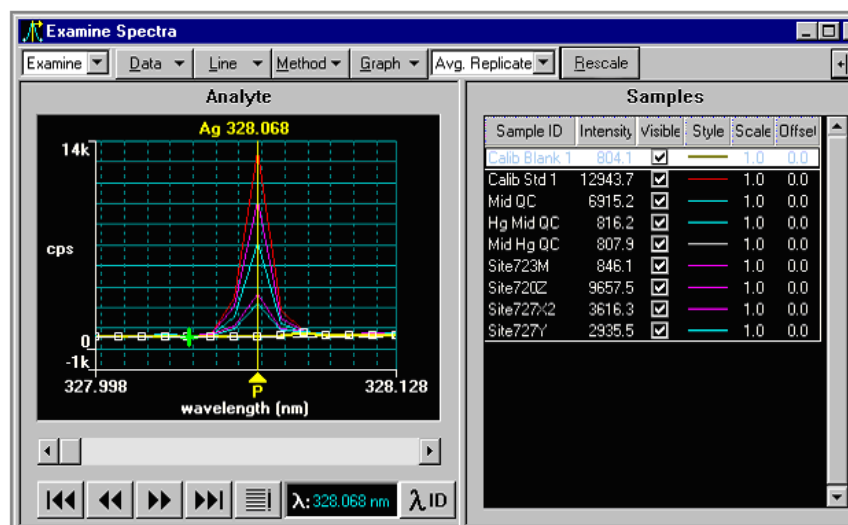
The legend shows you the list of samples for each analyte. It displays columns to indicate and set the hidden attribute, scaling, and offset. Since there is an entry for each sample, it is easy to tell at a glance how these settings are adjusted relative to all of the samples. More importantly, all of the samples' intensities are listed together in one column. Also, you can click on one sample and it is highlighted in the graph.

Along the **bottom of the graph** are buttons for Select First Analyte, Select Previous Analyte, Select Next Analyte, Select Last Analyte, Select Analyte and Select a Wavelength. (You can also access these commands from the Data menu.)

Select:	**To display:**
	The Analytes list, which lists the wavelength for each element that was selected from Go to Analyte.
	Double-click on one of the analyte names. You can then view that wavelength for all of the sample(s) listed in the legend.
	The first analyte in the Analytes list.
	The prior element in the Analytes list (starting from the analyte that was displayed before you clicked on this button). If you continue to press this button, you will make analyte selections that **move back up toward the top** of the Analytes list. At the top of the list, this button ceases to function. Then, you can press the Next button or Last button to move down the list.
	The next analyte in the Analytes list (starting from the analyte that was displayed before you clicked on this button). If you continue to press this button, you will make analyte selections that **move downward toward the bottom** of the Analytes list. At the last analyte in the list, this button ceases to function. Then, you can click on the Back button or First button to move back up the list.
	The last analyte in the Analytes list.
	Select a Wavelength.

At the **top of the graph**, there is a drop-down list to select which replicate mode is displayed. There are four replicate modes: Average Replicate, First Replicate, Last Replicate and All Replicates. All Replicates displays all of the replicates for a particular sample. This is different than the other modes, which display the first, the last, or the average of all replicates for all samples.

By default, the system automatically positions the **splitter bar**, which separates the graph from the legend, so that the legend is exactly wide enough to display the text it contains. This is constantly updated as that text changes. However, it is possible to allow more space for the graph or legend.

To allow more space for the graph or legend

▶ When you place the mouse pointer over the splitter bar, it changes to a double-headed arrow. Drag left or right to increase the size of the graph or legend pane.
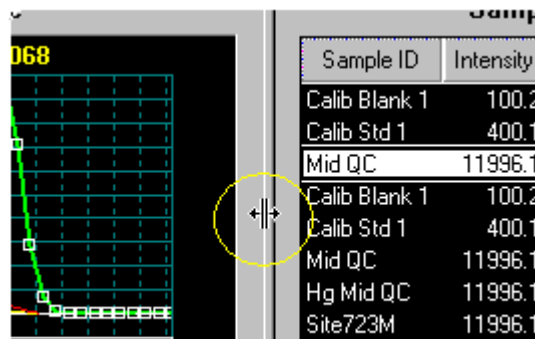
Note   If your cursor does not change to a double-headed arrow, you must disable the **auto-position splitter bar** check box in the Examine Display Options dialog. In the Options menu, click on Examine Spectra/MSF.... At the bottom of the Examine Display Options dialog, click on the **auto-position splitter bar** check box to clear the check mark.

If you want the software to position the splitter bar for you automatically, click on the **auto-position splitter bar** check box to display a check mark.

Printing from the Examine Spectra/MSF Window

You can print the graph and legend displayed in the Examine Spectra/MSF window as follows:

1. From the File menu, choose Print.

2. From the Print menu, choose Active Window (or Active Window Preview).

Examine Spectra/MSF window: Keyboard Shortcuts

The following keyboard shortcuts exist in the Examine Spectra/MSF window:

In Examine and MSF modes:

f   First Analyte

<   Previous Analyte

>   Next Analyte

e   Last Analyte

g   Go to Analyte

v   Toggles Hide/Visible for the active sample

<del>   Deletes the active sample from the current analyte's sample list

← Moves the Wavelength Cursor to the left by one picometer

→ Moves the Wavelength Cursor to the right by one picometer

The rest of the cursor keys (up, down, page up, page down, home, end) always apply to the legend. For example, to advance to the next sample in the legend, press the down-arrow key. Provided that there is indeed another sample to advance to, that will become the active sample and be highlighted.

In Examine mode only:

p   Set Peak Wavelength

b   Add Background Point

m   Opens the Update Method Parameters dialog

w   Opens the Adjust Wavelength dialog

Hold down the CTRL key and click in the graph area to assign a new Background Correction Point at that position, provided that there are not already 2 of them assigned and you do not click at the exact Peak Wavelength. (To remove a Background Correction Point, drag it (press and hold down the left mouse button) out of the graph window. When you release the mouse button outside the graph window, the Background Correction Point will be removed).

In MSF mode only:

b   Assign as Blank

a   Assign as Analyte

i   Assign as Interference

c   Clear Assignment

m   Mask

n   No Mask

In addition to keyboard shortcuts, if you double-click in the following areas of the Examine Spectra/MSF window the following dialogs will appear:

If you double-click here:	This dialog will open:
left of the y-axis in the graph area	Define Y-axis Options Dialog
right of the y-axis in the graph area	Examine Spectra/MSF Display Options Dialog
in the header of visible column of the legend	Visible Column Fill Dialog
anywhere in the scale or offset columns of the legend	Select Scale & Offset Dialog

Selecting Data for the Examine/MSF Window

Before you begin using the Examine Spectra window, open the method that was used to generate the spectra. If you are not sure which method was used, in the **File** menu, select **Import from Results Library...**, then select the results data set. If you want to use a different method, it must contain some (but not necessarily all) of the elements and wavelengths that were originally used when the spectral data was collected**.** To ensure that you save both the spectral data and the method used to collect them, make sure to check both the Spectral Data check box and the Copy of Method check box from the Method Editor window Options page.

Save the method, if you wish to retain the changes made to Method parameters from the Examine Spectra window.

Save spectra to the same data set if you will want to overlay these spectra in the same display. This is because you can select only **one** Data Set per Examine Spectra session.

To display data in the Examine/MSF window, select Select Data Set from the Data menu. A three-step Wizard walks you through the data selection process.

Select Results Data Set

Use the Data Selection Wizard to perform any of the following functions:

Select a results library from which to retrieve a data set

▶ Click on the Browse button to select a path from the Results Path dialog. (The default pathname appears.)

Select spectra to display in the Examine Spectra Window

▶ Click on a data set name to select a data set from which you wish to display spectra. (You will specify the sample(s) and analyte(s) in subsequent dialogs.)

Note   You can select only **one** data set per Examine Spectra session.

Sort the data set list

▶ Click on the appropriate option button to sort the Data Set list by Name or Date/Time.

Select samples and elements from a data set

▶ After you choose a data set, click **Next** to display Step 2., the Select Samples page of the Data Selection Wizard.

Select Samples

Open the Data Selection Wizard by choosing **Select Samples** from the **Data** menu in the Examine Spectra/MSF window. If this option is not available, choose Select Data Set.

Note   If you are selecting samples for MSF, select at least one analyte and either a blank or an interference. For MSF, each sample selected must have a unique name (multiple samples with the same name cannot be selected for use with MSF.)

Use the Select Samples dialog to perform any of the following functions:

Select every sample from the data set

▶ Click on the **Select All** button and then click on **Next**. By default, all samples are initially selected (highlighted). To deselect the samples, click on any sample in the list.

Select a specific sample from the data set

▶ Click on a specific sample from the list.

Note   If you wish to view the replicates for a sample:
1) Select only one sample from this dialog.
2) From the Replicates drop-down list in the Examine/MSF window, select **First Replicate**, **Last Replicate** or **Average Replicate**. See *Selecting Replicates* later in this chapter.

Select a set of samples that are listed sequentially

▶ Click on a sample, then press and hold the **Shift** key and click on another sample. All samples between these two will be selected (highlighted). Click on the **Next** button.

Select a set of samples that are NOT listed sequentially

▶ Press and hold the **Ctrl** key while clicking on any samples you wish to highlight. Release the **Ctrl** key, confirm your selections, and then click on the **Next** button.

Note 1   If you accidentally select (highlight) a sample, simply deselect it by **Ctrl/clicking** on that sample again. (You can use this technique to deselect no matter how you originally chose to select an item).

Note 2	Once you have made selections, click **Next** to display Step 3., the Select Analytes page of the Data Selection Wizard.

Select Analytes

Open the Data Selection Wizard by choosing Select Analytes from the Data menu in the Examine Spectra/MSF window. If this option is not available, choose Select Data Set.

Use the Select Analytes dialog to perform any of the following functions:

Select every analyte in the data set

▶ Click on the **Select All** button then click on **Finish**. (By default, all samples are initially highlighted.)

Select a specific analyte in the data set

▶ Click on a specific analyte from the list.

Select a set of analytes that are listed sequentially

▶ Click on a sample, then press and hold the **Shift** key and click on another sample. All samples between these two will be selected (highlighted). Click on the **Finish** button.

Select a set of analytes that are NOT listed sequentially

▶ Press and hold the **Ctrl** key while clicking on any analytes you wish to highlight. Release the **Ctrl** key, confirm your selections, and then click on the **OK** button.

Note	If you accidentally highlight an analyte, simply unhighlight it by **Ctrl/ clicking** on that analyte again. (You can use this technique for **unhighlighting** no matter how you originally chose to highlight an item.)

Exporting Spectra

Open the Export Spectra dialog by selecting **Export Spectra** from the Data menu in the Examine MSF window. You must have a data set open to use this option.

You can export spectral data, which includes all the data points collected in the spectral measurement range for each analyte to construct the peak that represents the intensity values. Then you can view the exported data in a spreadsheet or database program of your choice and graph the data points, if desired.

For example, after exporting the spectral data, here is an excerpt from the resulting spreadsheet:

Data Set: test1			
Analyte: Cu 324.752			
Wavelength	Calib Blank 1	Calib Std 1	Sample001
324.689	10	10	10
324.699	10	10	10
324.709	10	10	10
324.719	10	10	10
324.729	10	10	10
324.739	27.6	110.1	2090
324.749	50.2	200.2	3800
324.759	27.6	110.1	2090
324.769	10	10	10
324.779	10	10	10
324.789	10	10	10
324.799	10	10	10
324.809	10	10	10
324.819	10	10	10

The first column lists the wavelengths in the spectral measurement range for the analyte, Cu 324.752. The number of wavelengths will depend on how the peak was processed as defined by the method parameters (peak algorithm, points/peak, and, on simultaneous ICP spectrometers, whether signal profiling was used). The remaining columns list the intensities for each data point in counts per second

(cps). By using the spreadsheet program to graph the following data points, you can re-create the peaks from the Examine Spectra/MSF window.

Fill in the appropriate choices in the Export Spectra dialog:

- ► **Format:** Choose the column format you wish to use for displaying the wavelength data for each sample. If all samples share the same wavelengths, select the option for Single Wavelength Column, and the first column in the resulting spreadsheet will display the wavelengths in the spectral measurement range as shown above. If one or more of the samples uses a different set of wavelengths, select the option for Wavelength Column for Each Sample, and the wavelength column in the spreadsheet will be repeated for each sample.

- ► **Analytes:** Choose to export data for the analyte currently displayed in the Examine window, or choose all analytes.

- ► **Replicates:** Choose to include data from the first or last replicate, the average (mean) values, or all replicates.

- ► **Data Delimited by:** Choose the delimit character preferred by your spreadsheet or database program. Columns of data can be separated by commas, tabs, or semicolons.

- ► **File name:** Select a name for the export file. Choose the same name as the data set or create a different name.

- ► **Extension:** Choose the extension preferred by your spreadsheet or database program.

- ► **Directory:** Use the default Reports directory or browse to locate the directory where you want to save the exported file.

Selecting Replicates

You can choose the replicates you wish to view in the Examine Spectra/MSF window. The Replicates list is next to the Graph menu. Click on the drop-down arrow to make a selection, as described below.

View spectra of the first replicate only

▶ Click on **First Replicate** in the Replicate drop-down list.

Note You may wish to view a single replicate when you are optimizing instrument parameters for the maximum signal/noise which will be critical for detection limit measurements.

View spectra of the last replicate only

▶ Click on **Last Replicate** in the Replicate drop-down list.

View spectra of the average of all replicates

▶ Click on **Avg. Replicate** in the Replicate drop-down list.

Note During routine analysis you will most often just want to view an average of the replicates, which is the default setting. An exception to this may be if the Replicate Data from the Results Display window shows any significant discrepancies in replicate results.

Using the Examine/MSF Line Menu Options

Choose from the Line menu options described in the following sections.

Select Line Styles Dialog

Use the Line Styles dialog to customize the appearance of the spectra by color or line style. Before you display this dialog you must open a data set in the Examine Spectra/MSF window.

To display this dialog:

▶ In the Line menu of the Examine Spectra/MSF window, click on **Styles**.

   - or -

▶ Double-click anywhere in the Styles column of the legend.

Selecting analytes

▶ Select whether the line styles that you set in this dialog will apply to either the **Current Analyte** or **All Analytes**.

Selecting color(s) for the unknown sample spectra

To select a range of colors:

▶ In the Colors panel, select a color from the drop-down list for the **First Unknown** and select another color from the drop-down list for the **Last Unknown**, either under the Screen or Printer headings.

This specifies the range's starting and ending colors. A corresponding range of colors appears. This range will be used to display the various unknown sample spectra. The colors closest to the First Unknown value designate the first samples. The sample color gradually changes until it approaches the Last Unknown color, which designates the last sample.

Note The settings on your computer display affect how the range appears:

If the setting is 256 colors or less, the number of colors available to create the range is extremely limited between the first and last colors.

If the setting is greater than 256 colors, you should see a smooth transition in the color range between the first and last colors.

To select one color for all unknown sample spectra:

▶ In the Colors panel, select the same color from the drop-down lists for both the **First Unknown** and **Last Unknown**, either under the Screen or Printer headings. The color you selected will be used for all unknown sample spectra.

Note The colors for blank spectra, standard spectra, and Hg/QC spectra are set using the Spectra Display Options Dialog and always correspond to the same colors in the Spectra Display window.

Selecting a line style

▶ In the Styles panel, for each Sample ID listed, select a line style from the drop-down lists in the Styles column. This is useful if you need to differentiate between various samples when printing to a black and white printer. The available styles are: Solid, Dashed, Dotted, Dash Dot, or Dash Dot Dot.

Note You must use a line width of 1 in order to use line styles. When line widths of greater than 1 are selected, the line style is always displayed as Solid. If you selected line styles, but cannot display them, make sure that Line Widths for the Current Sample and/or Other Samples are set to 1.

Selecting line widths

▶ To select line widths for displaying spectra, type a number from 1 to 10 to designate the relative line width for the Current Sample (the currently selected sample) and for Other Samples. The Current Sample defaults to 2 and Other Samples defaults to 1.

Return to the Default Colors

1. To change all of the screen colors or printer colors back to their default settings, select the **Screen** or **Printer** buttons under Default Color Settings.

2. To change the colors without closing the Line Styles dialog, click on **Apply**.

    - or -

    To apply the change and close the dialog, click on **OK**.

Return to the Default Style Settings

1. To change all of the style settings (other than the colors) in this dialog back to their default settings, select **Default** under the Default Options Settings section.

2. To change the style settings without closing the Line Styles dialog, click on **Apply**. You can see the effect of the changes in the Examine Spectra/MSF Display window and modify them if desired.

    - or -

    If you are satisfied with the style settings, click on **OK** to apply the changes and close the dialog.

Select Scale & Offset Dialog

To display this dialog:

▶ In the Line menu of the Examine Spectra/MSF window, click on **Scale and Offset...**

- or -

▶ Double-click anywhere in the Styles or Offset column of the legend.

Note   If the dialog does not appear, you must first open a data set.

Tip 1   Select an Active Spectrum BEFORE YOU SELECT THIS DIALOG.

To apply a Scale Factor and/or a Baseline Offset to a spectrum, you must first select that spectrum by clicking on one of the sample names in the legend (i.e., the sample list in the right-hand side of the Examine Spectra window). Notice that the spectrum name will be highlighted and data points (which are indicated by small squares) appear on the associated spectrum to designate it as the active spectrum. (Data points will only appear if the data points option is selected from Select Graph Options Dialog.)

Tip 2   View the current status of an active spectrum by noting the right-hand side of the Examine Spectra window. You can check the intensity of the peak at the current cursor position, as well as the offset value and the scale factor that have been applied to the active spectrum. (The values for the offset and scale will be zero and one respectively, when you first select an active spectrum).

For example, if you use this dialog to apply both an offset and scale factor, then you will see those values listed.

Use the Select Scale & Offset dialog to perform any of the following functions:

Apply the Scale and Offset to the current sample only or to all samples

Select a scale factor to apply to the sample(s)

▶ **Type a value** (0.001 to 1 million). The appearance of the spectrum will be scaled larger or smaller based on the specified value. Click on the **Apply** or **OK** button to apply this factor to the scale for the active spectrum.

Note 1 Check the box next to **Automatically determine full scale factors** and the software fills in the scale and offset factors that cause the sample(s) to appear to utilize the entire y-axis dynamic range. If the **All Samples** is selected, the factors for each sample will be optimized (separately.)

Note 2 A Scale factor only changes the appearance of the spectrum; the original data (stored in the data set) remains unchanged.

Note 3 It is possible to detect low level contaminants by using scale factors, since an expansion factor is applied uniformly across the spectrum. Also, if a spectrum has an interference, this scale factor can help you to evaluate the active spectrum in comparison to any other spectra displayed. For example, you may want to compare the scaled spectrum (as the unknown mixture) with a spectrum containing only the suspected interfering component.

Select an offset to apply to the sample(s)

▶ **Type a value** (- one billion to + one billion). Click on the **Apply** or **OK** button to move the baseline position of the spectrum up or down based on the specified value.

Note 1   An Offset only changes the appearance of the y-axis of the spectrum and so the original data (stored in the data set) remains unchanged.

Note 2   When you have a group of spectra that are closely spaced (and sometimes overlaid on top of each other), you can apply an offset to move the active spectrum up or down (which does not change the scaling) so that you can make a direct comparison to the other spectra.

Reset the Scale and Offset factors for the active sample or all samples.

With the appropriate option selected for **Current Sample Only** or **All Samples**, click on the **Reset** button. A scale factor of 1.0 and an offset of 0.0 will be displayed. Click **Apply** or **OK** to apply the factors.

Add or Subtract Spectra Dialog

Open the Add or Subtract Spectra dialog by choosing Add from the Line menu in the Examine Spectra/MSF window. If this option is not available, you must first open a data set.

Use this dialog to add or subtract spectra by following the steps below:

1. **Select the operation to perform: addition or subtraction.**

    ► Click on either the **Add** or **Subtract** option button.

2. **Select the spectra you wish to add or subtract.**

    ► Identify Spectrum 1 and Spectrum 2 by opening each selection box and clicking on the desired spectrum name.

3. **Select the name of the resulting spectrum from the addition or subtraction.**

▶ **Type a name** in the **New Spectrum Name** entry (if you do not enter a new spectrum name the system specified name, "Sample" will appear in the Samples list).

4.   Perform the addition or subtraction and view the spectrum.

▶ Click on the **OK** button to apply the selections you have made in the dialog.

The resulting spectrum appears in the Examine Spectra window. Also, the new spectrum name is included in the Samples list and appears as the active spectrum (as designated by the box).

Note: When used appropriately, here are some of the uses for spectral addition or subtraction**:**

- Detect possible contaminants

- Evaluate concentrations required for the analysis.

- Identify chemical and spectral interferences

- Evaluate peak wavelength selection (by elimination of a peak (subtraction) or by spiking (addition)).

- Make an assessment of instrument parameters and possibly use as an aid in troubleshooting

- Make an assessment needed to select parameters during Method Development (e.g. Background correction points, determine appropriate data treatments (such as IEC), etc.)

Using the Hide/Visible Line Option

In the Line menu of the Examine Spectra/MSF window, click on the Hide/Visible option to perform the functions listed below. Also you can perform the same functions in the right side of the Examine window, under the Hide/Visible column by checking or unchecking the box next to each sample.

Remove a spectrum from the display but retain the name in the samples list

▶ Select the active spectrum and then click on Line menu and then click on the **Hide** option. The active spectrum is automatically removed from the display.

Note 1   The spectrum name remains in the Samples list but the line showing its color is removed to indicate that this spectrum is "hidden."

Note 2   You can hide any number of spectra.

Re-display a previously hidden spectrum

▶ Select a Hidden spectrum from the samples list (it will have a spectrum name but there will be no line color next to it) and then click on Line menu ▶ Hide/Visible (or click the box in the Visible column of the legend). The active spectrum reappears in the display.

Remove or display all samples

▶ Double-clicking on the word "visible" at the legend's visible column heading will open a column-fill dialog. There is a single checkbox in the dialog, label "visible". Checking or unchecking that checkbox and clicking the **OK** button will cause all samples to be either visible or hidden, respectively.

Using the Delete Line Option

From the Line menu in the Examine Spectra/MSF window, click on the Delete option to perform the following function:

Delete a spectrum from the display

▶ Select the active spectrum and then in the Line menu, click on **Delete**. The active spectrum is removed from the current analyte's display (but not from the data set).

Note 1 To redisplay a "deleted" spectrum you will need to reselect it: In the Data menu, click on Select Samples.

Note 2 You can delete any number of spectra.

Using the Examine/MSF Method Menu Options

Choose the options described in the following sections to set method parameters for examining spectra.

Setting a New Peak Wavelength for the Method

When you view the peak for an analyte, the peak's highest intensity may not occur exactly at the wavelength that you specified in the method. In this situation, you can modify the peak wavelength in the method directly from the Examine Spectra window.

Tip See *Example: Setting a New Peak Wavelength for the Method* later in this chapter for more information.

To set and update the peak wavelength

1. Display the spectra for the analyte of interest in the Examine Spectra/MSF window.

2. Open the method that was used to generate the data. If you are not sure which method was used, select **Import from Results Library...** in the File menu, then select the results data set.

3. In the Examine Spectra/MSF window, click on the name of the sample in the Samples list to designate the active spectrum on the graphics display.

4. Drag the vertical cursor to the point of maximum intensity on the peak. The peak maximum can be determined by observing the value in the Intensity column on the right side of the window. The Wavelength display shows the cursor position.

5. In the **Method** menu, select **Set Peak Wavelength.**
   The Wavelength display shows the chosen wavelength, and the peak wavelength symbol is repositioned to align with the cursor.

6. In the **Method** menu, select **Update Method Parameters...**
   The Update Method Parameters dialog appears. The current wavelength from the method and the revised wavelength are shown.

7. Be sure a check mark appears next to Peak Wavelength, then select one of the following:

   Update
   Click on the **Update** button to update the method according to the revised wavelength and any other selections you have made in this dialog. However, the method changes will not be saved to disk.

   Update and Save Method
   Click on the **Update and Save Method** button to modify the method according to the revised wavelength and any other selections you have made in this dialog and save the method to disk.

8. You can view the change in the Method Editor on the Spectrometer: Define Elements page.

Note	The analyte name specified in the method usually contains the reference wavelength (for example As193.696), but can be renamed if desired.

Example: Setting a New Peak Wavelength for the Method

For example, let's say you are collecting data using a method that contains the analyte Cadmium at the wavelength of 228.802. When you display the Cadmium spectrum in the Examine Spectra window you may find that while the method expects the wavelength to be at exactly 228.802 nm, shown by the peak wavelength symbol, the vertical cursor is not aligned with the center of the peak.
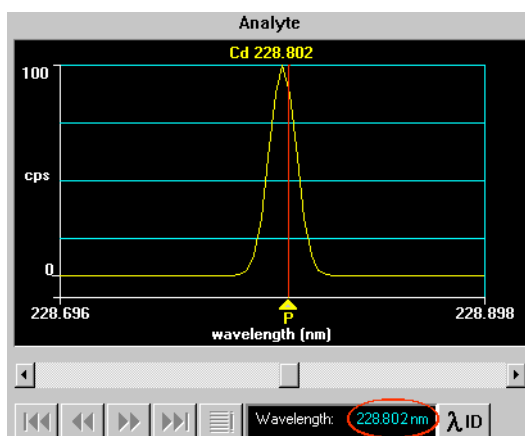
You can move the cursor so it is aligned with the center of the peak and a readout of the cursor's wavelength position (at the bottom of the Examine Spectra window) shows that the peak is actually appearing at another wavelength, for example 228.799 as shown below. Then, you can choose to set the peak wavelength to the peak maximum at 228.799.
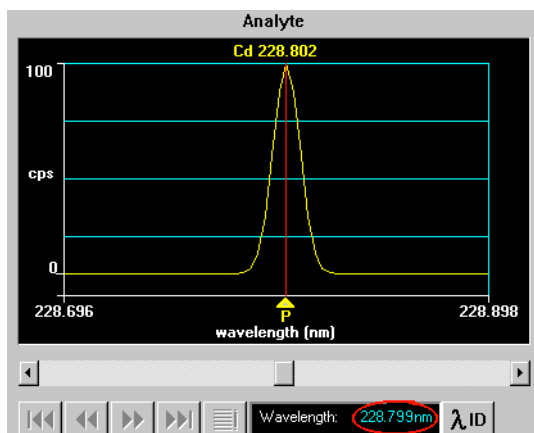
After setting the new peak wavelength, you can update the method with the revised wavelength using the Update Method Parameters dialog. For more information, see *Setting a New Peak Wavelength for the Method* earlier in this chapter.

Note   In some special situations, you may want to update the instrument and/or data set with the new wavelength, as described in the next section.

Adjusting the Wavelength

You can choose to fine-tune the wavelength for future analyses and/or for a particular data set.

Important   This adjustment should only be done when determined to be fully necessary. Before making this adjustment, see *Tips: Adjusting the Wavelength* later in this chapter.

To adjust the wavelength

1. Display the spectra for the analyte of interest in the Examine Spectra/MSF window.

2. Click on the name of the sample in the Samples list to designate the active spectrum on the graphics display.

3. Drag the vertical cursor to the point of maximum intensity on the peak. The peak maximum can be determined by observing the value in the Intensity column on the right side of the window. The Wavelength display shows the cursor position.

4. In the **Method** menu, select **Adjust Wavelength...**
    The Adjust Wavelength dialog appears. The original wavelength and the revised wavelength are shown.

5. Choose how you want to use the revised wavelength value:

    Update Instrument and Data Set
    The revised wavelength will be entered in an instrument offset table, which is used to correct for wavelength differences in all subsequent analyses. On simultaneous ICP spectrometers, the entire subarray for this wavelength will be updated to compensate for offsets for other wavelengths in the range of this subarray. On scanning CCD ICP spectrometers, this modification applies only to the specific wavelength you have revised. In addition to updating the instrument offset table, the revised wavelength will be applied to the current data set open in the Examine Spectra window. Updating the data set is useful when you need to make adjustments for method development (for example, adjusting background correction points) or if you will be reprocessing the data set at a later time.

    Update Instrument
    The revised wavelength will be entered in an instrument offset table, which is used to correct for wavelength differences in all subsequent analyses. On simultaneous ICP spectrometers, the entire subarray for this wavelength will be updated to compensate for offsets for other wavelengths in the range of this subarray. On scanning CCD ICP spectrometers, this modification applies only to the specific wavelength you have revised.

Update Data Set (only)
The revised wavelength will be applied to the current data set open in the Examine Spectra window. Select this option if the revised wavelength applies only to the current data set and you do not wish to revise the method or the instrument for future analyses. Updating the data set is useful when you need to make adjustments for method development (for example, adjusting background correction points) or if you will be reprocessing the data set at a later time.

Tips: Adjusting the Wavelength

To determine accurate peak wavelengths during Method Development, consider the following:

Consider the Peak Algorithm

Depending on the Peak Algorithm, peak wavelength has different effects:

- If **Peak Ht**. processing is chosen, it defines the center of the window in which the software will search for a peak.

- If **Peak Area** processing is chosen, it defines the center pixel that will be summed in the spectral measurement range.

Updating the Instrument Offset Table

The revised peak wavelength can be used in an instrument offset table, which is used to correct for wavelength differences in all subsequent analyses. It is recommended to keep a log book (or file) of the dates and pertinent information such as the element(s) and wavelength(s) that were revised.

When determining peak wavelengths, be sure you are using a pure single-element standard for the analyte of interest at a reasonable concentration (for example 1 ppm).

On simultaneous ICP spectrometers, be sure to perform a Hg Alignment before updating the instrument offset table. For more information, see *Table of Spectrometer Alignment and Calibration Controls*. The Adjust Wavelength dialog allows you to fine-tune the wavelength calibration for each subarray.

After updating the instrument offset table, all previous methods may be invalidated for use on the newly "calibrated" instrument. Methods that were used prior to the new offsets may need to be revised for selected wavelengths. In addition, if you have set up previous methods that already compensate by using slightly different wavelengths, then changing the instrument offsets will cause these wavelengths to be incorrectly adjusted.

Spectral Profiling

On simultaneous ICP spectrometers, if you are not achieving the desired results with spectral profiling off, you may want to turn on Spectral Profiling while you are determining peak wavelengths. It is sometimes easier to discern the peak wavelength position when the peak is symmetrical and has a smooth shape. It is recommended that you overlay both the lowest and highest concentration standards (and, of course, some samples) to determine peak wavelength. Then, for routine analysis, you can turn off Spectral Profiling.

Using the Background Correction Point Option

To set a background correction point, choose Add Background Point from the Method menu in the Examine/MSF window.

Tip 1   When the standards and samples are not matrix matched, it is considered good practice to use at least a one-point background correction.

Tip 2   For any type of sloping background, use a two-point background correction, and select an alternate wavelength for the element whenever it is possible.

To select Background Correction (BGC) points follow the steps below:

1. **Select the active spectrum**

   ▶ Click on the spectrum name in the samples list to designate the active spectrum on the graphics display.

2. **Graphically select a BGC point**

    ▶ Use the drag technique (or the horizontal scroll bar) to position the cursor wherever you want to place a BGC point. Alternately, you can place a BGC point with your mouse. To do so, hold down the Ctrl key, point your mouse where you want a BGC point, and click.

    ▶ Click on the Method menu and then click on Add BGC Point. Note that green crosshairs appear on the graph to indicate the position of the BGC point. To reposition an existing BGC point, simply drag it with the cursor.

Note A red BGC point located at either end of the X axis indicates that the background offset in the method is outside of the wavelengh range shown in Examine. To reposition the BGC point within the x axis, simply drag it with the mouse cursor. The color returns to green.
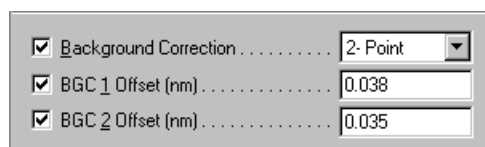
3. **For a second BGC point repeat step 2, otherwise skip to step 4**

    ▶ You may only select up to two BGC points per spectrum.

Note To remove a BGC point, drag it to any position outside of the graphic display.

4. **Update the Method**

    ▶ In the **Method** menu, click on the **Update Method Parameters** option to display the Update Method Parameters dialog. Note that up to three check marks will appear when you have selected two BGC points and the offset values for selected BGC point(s) are listed automatically. For example:

▶ Click on the Update button to enter the new BGC points into the Method.

Note **Save the Method** if you wish to save the updated BGC offset value(s) and correction type (i.e., 1-Point, 2-Point or None).

Identifying Elements Using the Button

λ ID

To help you identify an element responsible for an interfering peak:

1. Move the vertical cursor to the interfering peak, note the wavelength and click on **ID**.

2. The Wavelength table appears showing a list of elements and wavelengths in the same spectral measurement range as the current peak.

Update Method Parameters Dialog

To update method parameters, choose Update Method Parameters from the Method menu in the Examine/MSF window.

The Update Method Parameters dialog has several different uses described below:

For using this dialog to:	See:
Set the peak wavelength and update the method.	*Setting a New Peak Wavelength for the Method* earlier in this chapter.
Adjust Background Correction	*Using the Background Correction Point Button* earlier in this chapter.
Modify the Peak Algorithm and Points/Peak	*Method Editor Process: Spectral Peak Processing Page* in Chapter 3, *Using the Method Editor*, for information on these parameters.

Update

▶ Click on the **Update** button to modify the method according to the selections you have made in this dialog. However, the method changes will not be saved to disk. If you have selected that you want to update the method to a new peak wavelength, be sure to select the check box for Peak Wavelength, then click on **Update**.

Update and Save Method

▶ Click on the **Update and Save Method** button to modify the method according to the selections you have made in this dialog and save the method to disk.

Show Analytical Points

Select **Show Analytical Points** from the Method menu in the Examine Spectra window.

This feature allows you to view a highlighted region of points along the active spectrum that were used to calculate intensity.

To hide the highlighted analytical points region, reselect Show Analytical Points from the Method menu.

Using the Examine/MSF Graph Menu Options

Choose the options described in the following sections to modify options for the spectral display area.

Define Y-axis Options Dialog

Open the Define Y-axis dialog by choosing Define Y-axis from the Graph menu in the Examine Spectra/MSF window. If this option is not available, you must first open a data set.

Note	Double-clicking on the graph area (the left-hand side) of the Examine Spectra/MSF window, but to the left hand side of the y-axis, is a convenient way to open the Define Y-axis Options dialog.

Use this dialog to perform the following functions:

Change the y-axis options for ONLY the Current Analyte or All Analytes in the data set

▶ Click on the appropriate option button at the top of the dialog.

Set the scale to linear or log

▶ To display graphs using a logarithmic scale (to compress large values without compressing small values) select Logarithmic.

Select the maximum value for the y-axis

▶ To automatically scale the y-axis so the maximum Y value is at least 100, click on the Automatic (min 100) option button.

▶ To automatically scale the y-axis for the highest intensity peak in the window, click on the Automatic option button.

▶ To enter a specific maximum value for the y-axis scale, click on the Manual option button and enter a value (that ranges from -1 billion to 1 billion, inclusive).

Select the minimum value for the y-axis

▶ To automatically scale the y-axis so that the zero point on the Y-axis must be shown, click on the Automatic with zero axis option button. If log scale mode is selected, then the minimum y-axis value is 1, so the zero point is not shown.

▶ To automatically scale the y-axis for the minimum intensity in the window, click on the Automatic option button.

▶ To enter a specific minimum value for the y-axis scale, click on the Manual option button and enter a value (that ranges from -1 billion to 1 billion**;** inclusive).

Select Label Options Dialog

Open the Label Options dialog by choosing Label Options from the Graph menu in the Examine Spectra/MSF window. If this option is not available, you must first open a data set.

Use this dialog to perform the following functions:

Apply label options to the Current Analyte Only or All Analytes in the data set

▶ Click on the appropriate option button at the top of the dialog.

Select the desired label options:

▶ Click on the appropriate option button for the desired graph title (i.e., to use the analyte name, to enter your own title, or no title at all.)

▶ Click on the appropriate option button to display x-axis and/or y-axis labels.

Display Options Dialog

Open the Examine Spectra/MSF Display Options dialog by choosing Display Options from the Graph menu in the Examine Spectra/MSF window. If this option is not available, you must first open a data set.

Note You can choose Printer colors in this dialog and then print the graph displayed in the Examine Spectra/MSF window as follows:

▶ From the File menu, choose **Print** ▶ **Active Window.**

Select the Examine Display Options dialog to perform the following functions:

Apply color selections to the Current Analyte Only or All Analytes in the data set

Select colors for the following items:

▶ Axes and Label

▶ Graph Title

▶ Examine Graph Background

▶ MSF Graph Background

▶ BGC Point Mark(s)

▶ Wavelength Cursor (if enabled)

▶ Y-Axis Grid Divisions (if enabled). The number of Y-Axis Grid Divisions can also be selected (from 1 to 100.)

▶ Data Points on Active Spectrum (if dialog has Active Spectrum points enabled)

Enable or Disable the following items:

▶ X-Axis Grid Divisions (When enabled, x-axis grid divisions are displayed at every tenth picometer.)

▶ Auto-position splitter bar (Disabling this option lets you resize the Graph and Legend panes of the Examine window by positioning your mouse on the bar and dragging left or right.)

Return to the Default Colors

1. To change all of the screen colors or printer colors back to their default settings, select the **Screen** or **Printer** buttons under Default Color Settings.

2. To change the colors without closing the dialog, click on **Apply**.

   - or -

To apply the change and close the dialog, click on **OK**.

Add Label Dialog

Open the Add Label dialog by choosing Add Label from the Graph menu in the Examine Spectra/MSF window. If this option is not available, you must first open a data set.

Use this dialog to add text labels to an active spectrum by following the steps below:

1. **Select the active spectrum.**

    ► Click on the spectrum name.

2. **Open the Add Label dialog.**

    ► Click on the Graph menu and then click on Add Label.

3. **Enter the desired title for the active spectrum.**

    ► Type a label name for the active spectrum and click on **OK**. The text appears in the window.

    ► To move the text, position the mouse cursor where you want the text to appear. Hold down the Shift key and click on the desired position.

    ► To label another spectrum on the window repeat steps 1 through 3. See below, for an example showing a label for each spectrum.
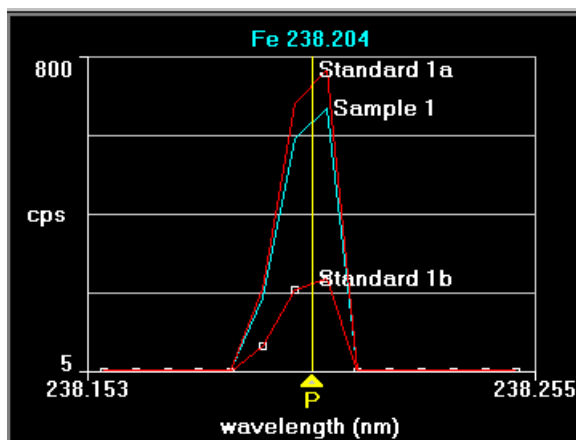
Automatically use the spectrum Sample ID as the label text:

Click the Use Sample ID button to automatically enter the Sample ID as the label text.

Label all samples with their Sample ID's as their label text:

Check the Label all samples with their Sample ID's checkbox to enable this feature. If this checkbox is checked when the OK button is pressed, all of the samples for the current analyte will be assigned labels and each will be the Sample ID for the respective spectrum.

Clear all labels:

Check the Clear labels from all samples checkbox to enable this feature. If this checkbox is checked when the OK button is pressed, any existing labels will be removed from the display of the current analyte. This does not refer to the axes labels or the graph title label – to clear them, use the Select Label Options Dialog.

How to Use the Examine MSF Window

Before using the Examine MSF window, select MSF from the list at the top left of the Examine Spectra window. The window toggles from Examine mode to MSF mode. The two modes share much of the same functionality. However, several differences exist between the Examine Spectra window and the Examine MSF window. These differences are shown in the following pictures and also described below.
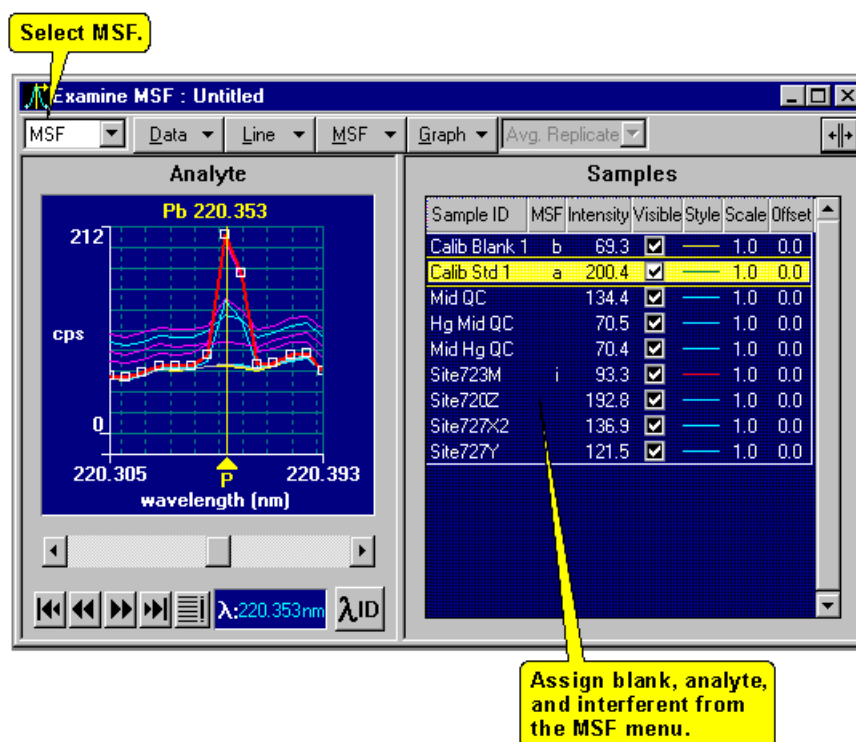
To start using the Examine MSF window, select spectra to display:

1. Click on the Data menu, and then click on **Data Set**.

2. Complete the Data Selection Wizard to select samples and analytes.

The Examine MSF window displays the first analyte. You can now use the MSF menu to assign values for an MSF model (blank, analyte, interferent). You save the model as an MSF file, to be used during analysis or reprocessing to correct for spectral interferences.

What You See in the Examine MSF Window

The window is split vertically with the graph on the left and the legend on the right. Notice that the default background color in MSF mode is different than Examine Spectra mode to help you tell at a glance which mode you are using. The graph shows you the spectrum for the selected analyte. The legend shows you the list of samples for each analyte.

The legend displays columns to indicate and set the MSF values, as well as the visible attribute, line style scaling, and offset. Since there is an entry for each sample, it is easy to tell at a glance how these settings are adjusted relative to all of the samples. More importantly, all of the samples' intensities are listed together in one column. Also, you can click on one sample and its spectrum is identified in the graph with data points (which are indicated by small squares along the line).

In Examine MSF mode, there is no Method menu. Instead there is an MSF menu which you use to assign values for an MSF model that you can use to correct for spectral interferences.

Along the bottom of the graph are buttons for Select First Analyte, Select Previous Analyte, Select Next Analyte, Select Last Analyte, and Select Analyte.

The replicates list is grayed out in Examine MSF mode.

You can easily allow more space for the graph or legend.To do so, position your mouse over "splitter bar" that separates the left and right panes. Your cursor changes to a double-headed arrow. Drag left or right to increase the size of a pane. (If your cursor does not change to a double-headed arrow, you must disable the "auto-position splitter" option in the Examine Display Options dialog.)

Note	The Examine MSF window is used to create the MSF model. To view the results of using MSF in an analysis, you must open the Results Display and the Spectral Display windows. The Results Display window lists the analytical results and indicates that the sample was processed by MSF (as applicable, the **.msf** file name will be listed for each analyte in the sample). The Spectra Display window shows both the uncorrected data and the MSF-corrected analyte data.

Using the MSF Menu

Once you have collected spectra of the blank, analyte, and individual solutions for each interferent (if any), then you can build the MSF model that will be used to correct for spectral interferences when you analyze samples.

Before using the MSF menu, use the Data menu to open a data set. Then use the analyte selection toolbar, located at the bottom of the graph, to display the first analyte for which you want to correct for interferences.

About the MSF Menu

Once the blank, analyte and individual spectra for each interferent are displayed, you can use the MSF menu to make assignments to the spectra:

- blank or global blank

- analyte or global analyte

- interferent(s) or global interferent

You can also mask (and unmask) a portion of the model spectrum.

As you develop the MSF model for each analyte in the data set, you can use the MSF Viewer/Editor to view your blank, analyte, and interference selections and make changes if you wish. For more information on the Viewer/Editor see *Using the MSF File Viewer* later in this chapter.

How to Use the MSF Menu

1. Make sure you have a data set open and that the analyte you wish to model is displayed.

2. To designate the blank as the active spectrum, click on its spectrum name in the legend.

3. Click on the MSF menu and then on **Blank** or **Assign Global Blank**. A **b** will appear next to the blank spectrum name in the legend.
   To remove the **b**, select Blank (or Assign Global Blank) again.

4. To designate the sample as the active spectrum, click on its spectrum name in the legend.

5. Click on the MSF menu and then on **Analyte** or **Assign Global Analyte**. An **a** will appear next to the analyte spectrum name in the legend.
   To remove the **a**, select Analyte (or Assign Global Analyte) again.

6. To designate an interferent as the active spectrum, click on its spectrum name in the legend.

7. Click on the MSF menu and then on **Interfence** or **Assign Global Interferent**. An **i** will appear next to the interferent spectrum name in the legend.
   To remove the **i**, select Interference (or Assign Global Interferent) again.

Note   You can repeat steps 6 and 7 for each interferent spectrum.

8. If desired, mask any area of the spectrum that you do not wish to include in the spectra profile of the MSF model. Click on the **MSF** menu and then on **Mask**, and drag the vertical cursor that appears across the area to be ignored in the MSF calculation. A series of X's appears confirming the area to be masked. For details on when to use masking, see *Masking* later in this chapter. (To remove the masking, click on the **MSF** menu and then on **No mask**.)

Save the Model

In the File Menu, click on **Save** ▶ **MSF Model** and specify a name. Note that this MSF file name will appear as a selection in the **MSF Table** drop-down list in the method (on the Process page, Spectral Corrections tab).

Note   Once you have created and saved an MSF file, you can automatically reuse the model with a new data set as long as the Sample IDs are the same.

▶ To reuse the model, first open the new data set in the MSF window. Then, in the File Menu, click on **Open** ▶ **MSF Model** and select the MSF file that has the assignments you want to use. The Sample IDs must be the same.

Analyze samples

Analyze check samples (to evaluate results) and unknowns.

Note   In the Method make sure to select:

Process page, Peak Processing tab, select **MSF** as the Peak Algorithm for the appropriate Element.

Process page, Spectral Corrections tab, select **MSF** as the Overlap Correction. Then from the **MSF Table** drop down list, select the .msf file that has been saved as the MSF model for that element.

Save the Method and repeat the steps above, as necessary, to optimize the MSF model.

About Masking

An area of a model spectrum can be selectively excluded from the spectral profile of the model. For example, if you have a spectrum of an analyte solution that has an interferent with minimal or no overlap then you can use the Mask button to selectively remove the interferent from being included in the model of the analyte. Masking makes it possible to avoid adding an unnecessary component to the matrix model and this means **you do not have to collect a spectrum of the component to be masked**.

Masking:
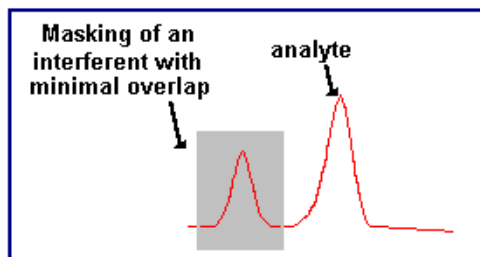
To use masking appropriately, the interferent must have **minimal or no overlap** with the analyte. (When you make this judgment, be sure that you are viewing the maximum concentration of the interferent). Under these circumstances when you apply masking, you can expect the same precision as if the interferent was included in the MSF model.

Be cautious about masking an area. Before you apply masking, check to see whether the area will help you distinguish the analyte, as in the following example.
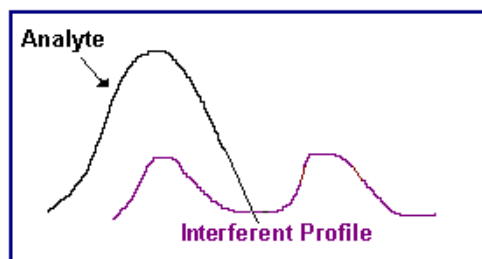
Here, the first peak of the interferent profile occurs at the same wavelength as the analyte. Because the analyte completely overlaps this peak, it is difficult to discern the interferent peak intensity. The second interferent peak occurs at a slightly higher wavelength and provides information on intensity that may help you to discern the intensity of the overlapping interferent peak. Therefore, the spectral region where the second interferent peak occurs should **not** be masked.

In the following example, the first peak in the interferent profile partially overlaps the analyte and should be included in the MSF model. A second peak does not overlap and the spectral region where the peak occurs can be masked.
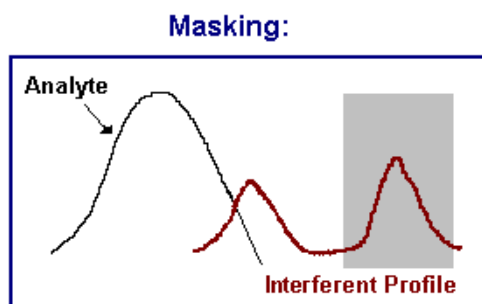
About Global Assignments

When you are assigning a blank, analyte, or interferent to an MSF model (in the Examine MSF window), you can make either a single assignment or a global assignment. You use the global assignments to streamline the model creation process.

For example, if your blank is common to all analytes, you can first select a data set that includes the blank and the standard containing the analyte(s). Then you can highlight the blank in the Examine MSF legend and choose Assign Global Blank from the MSF menu to assign that sample as the blank for the MSF model for each analyte in your data set.

Similarly, you can highlight the standard containing the analyte(s) in the legend and choose Assign Global Analyte from the MSF menu. This will assign that standard as the analyte in the MSF model for your data set.

In addition, if you have an interference that is common to all analytes, choose Assign Global Interference from the MSF menu.

To view or edit your blank, analyte, and interference selections, open the MSF Viewer/Editor.

Using the MSF File Viewer/Editor

Open the MSF File Viewer/Editor from the MSF menu in the Examine Spectra/ MSF window.

This dialog lists the analytes currently modeled. For each analyte, the sample ID's and their assignments in the model are listed.

A green check mark indicates an assignment was made. For example, if you have assigned a blank, it is identified with a check mark and the word "blank" in parentheses.

If a sample does not have an assignment, a red X appears next to it.

Use this dialog as follows:

- Click the plus or minus sign next to an analyte to show (or hide) its blank, analyte, and interferent.

- Click **Expand All** to show all blanks, analytes, and interferents for each analyte model.

- Click **Collapse All** to show only a list of analytes.

- To delete a specific analyte or an individual sample ID from a particular model, highlight it and click on **Delete**. For example, to delete a blank, highlight the name of the blank, and click on Delete.

You must save the .MSF file after you make any changes in the File Viewer/Editor. To save the file, choose Save from the File main menu, and then choose MSF.

An Overview of How MSF Functions

Multicomponent Spectral Fitting (MSF) uses multivariate calibration to determine the concentration of an analyte. Multivariate calibration is analogous to a mathematical filter that can distinguish between the components of a complex spectral profile. The signal contribution of three components can be separated from the analyte:

- interference
- background
- noise

Although MSF is very useful for correction of spectral overlap it **requires** that you have knowledge of the expected components in the real samples (for an exception, see *Masking* earlier in this chapter). In practice, this means you will need to prepare individual solutions for the blank, interferent(s), and analyte.

Note 1   MSF is not suitable where there is complete overlap of analyte and interferent (In that case, use the Inter Element Correction (IEC). For all other types of spectral overlap, MSF is a powerful technique that can be used to improve detection limits and precision.

Note 2   Even when there are no interferences, you can use MSF's ability to improve detection limits and precision. (Simply create a two-component model that is composed of just the blank spectra and the analyte spectra.) MSF is beneficial when:

- it is difficult to assess BGC points**,** or

- the noise contribution is significant, such as in measurements near an analyte's detection limit

- For a two-component model, MSF may save you time because:

    - MSF can automatically correct for the background so there is **no need to select BGC points.**

    - MSF uses all of the information about the peak so there is **no need to select peak wavelength.**

Note 3 MSF may be used along with internal standardization to further improve precision, which may be especially important for measuring analytes in a complex matrix.

Perform Steps to Use MSF

An overview of how to perform Multicomponent Spectral Fitting is shown below, along with information about how the software uses input data to calculate analyte concentration:

Use these Windows...	To Perform these Steps:	WinLab then Creates...
Method Editor:	1. Set Up Method	
Manual or Automated Analysis Control:	2. Collect Model Spectra	
Multicomponent Spectral Fitting:	3. Assign Spectral Profiles	→ .MSF File
Method Editor:	4. Modify Method	↓
Manual or Automated Analysis Control:	5. Perform an Analysis	→ Matrix Model
Results Display:	6. Evaluate Results	←
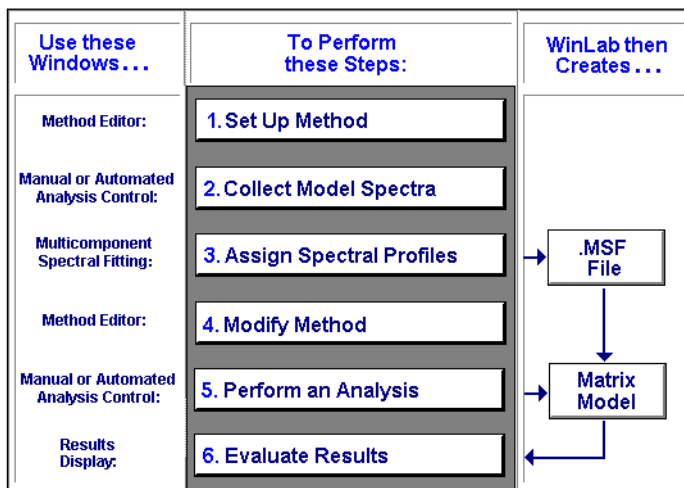
Step 1. Set Up Method

When you choose to perform MSF for an analyte, it is assumed that you have already acquired some knowledge of your samples:

- You have determined that there is a need for MSF (i.e., usually because of spectral interference and that no other interference-free wavelength is available).

- You have qualitative information about the sample (i.e., you have identified the analyte and the major interferent). If the interferent has minimal overlap then you can use masking (and in that case, it is unnecessary to know the identity of the interferent). Refer to the *Masking* topic earlier in this chapter for more information.

- You know what instrumental conditions are needed to run your application.

Next, you need to set up a method to collect spectra of individual solutions that will be used to model the spectral profile of each of the components (blank, analyte, and interferent(s)).

Note   For multiple analytes you will have to make assignments for each analyte.

Tip 1   All solutions can be analyzed as samples. Make sure to identify every spectrum with a unique sample ID.

Tip 2 Theoretically, it is "unnecessary" to prepare quantitative solutions for the model, since the underlying assumption is that the spectral profile is independent of the concentration, and you will not be entering concentration values into the method. However, in practice, if you wish to optimize the ability of the model to predict unknown concentration of the analyte, then you should still consider the following suggestions when preparing solutions for the model:

Concentration of the analyte solution should be 100 times its detection limit at that wavelength.

Concentration of the interferent solution should be the maximum concentration of interferent that will be present in the analysis.

Set up and save a Method that specifies the following parameters:

• Algorithm set to Peak Ht. or Peak Area

Note At this point, do not specify MSF in the Method when you are first collecting the spectra for the model.

• Multiple replicates to reduce noise

• Spectral profiling (**yes** or **no**).Set this to match your analysis. (Spectral profiling is not required; however, in some instances, it may improve results, although it will take longer to process.)

Tip Optionally, you may decrease the maximum read time to compensate for the increased time that is required for spectral profiling.

Step 2. Collect Model Spectra

Use the Automated Analysis Control Window or the Manual Analysis Control Window to perform an analysis.

Make sure to...

•   Save the sample and standard spectra to the same data set as the spectra used for the MSF model.

•   Before you collect spectra, make sure that the Save Data and Print Log boxes are selected. in the analysis window.

•   Set up the Sample Information file as required for your samples.

Step 3. Assign Spectral Profiles

Note   For multiple analytes you can use the MSF menu to make global assignments.

Tip   **Save spectra to the same data set** so that any spectra used to create the MSF model can be displayed at the same time in the MSF window (this is a requirement for creation of the MSF model since you can select only **one** data set per MSF session).

Once you have collected the model spectra, then you will create the model incorporating each spectral profile (for blank, analyte, and interferent(s)).

Display the spectra in the Examine MSF Window

1.   In the Examine MSF window, choose a data set by selecting Data Set from the Data menu and following the Data Selection Wizard.

2.   The spectra appear in the Examine MSF window and now you can build a Model.

Build the MSF Model

For specific steps on using the MSF menu to build the model, see *Using the MSF Menu* earlier in this chapter.

Save the Model of the Spectral Profile

In the File menu, click on **Save** ▶ **MSF Model** and specify a name. Note that this MSF file name will appear as a selection in the MSF Table drop down list in the Method.

Note   Once you have created and saved an MSF file, you can automatically repeat the assignments for the blank (b), analyte (a), and interferent (i) on a new data set as long as the Sample IDs are the same for both files.

▶ To repeat current assignments for new data, first open the new data set in the MSF window. Then, in the File menu, click on **Open** ▶ **MSF Model** and select the MSF file that has the assignments you want to use. The Sample IDs must be the same.

Create .MSF File

Once you have assigned each spectrum as

(a) Analyte

(b) Blank

(i) Interferent(s)

You must save this information as a .**msf** file.

While still in the MSF Window**:** In the File menu, click on **Save** ▶ **MSF Model** to enter a file name (that must have a **.msf** extension). This file name is automatically added to the **MSF Table** drop-down list in the Method Editor (Process page, Spectral Corrections tab).

A summary of Steps 3 through 6 as they relate to how MSF functions in WinLab32:

In **Step 3: Assign Spectral Profiles**, first, you will assign the model spectra as blank (b), analyte (a), and interferent (i). Next, when you save the .**msf** file, the software will create the .**msf** file that contains the profile information.

In **Step 4: Modify Method**, you will select the **.msf** file that is appropriate for that method.

In **Step 5: Perform an Analysis**, the software will input both the modeled spectra (from **Step 3:**) and the spectra collected for analysis (blanks, standards, and samples) to create a Matrix equation that will output unknown analyte concentration.

In **Step 6: Evaluate Results**, you can view the MSF-corrected results that were output from the Matrix Model. The Results Display window lists the analytical results and indicates that the sample was processed by MSF (as applicable, the **.msf** file name will be listed for each analyte in the sample). The Spectra Display window shows both the uncorrected data and the MSF-corrected analyte data.

Step 4. Modify Method

| Note | You must use the same spectrometer Options (Resolution, Profiling) for data collection of the model spectra as for the standards and samples. |

The purpose of modifying the method is to collect the spectra for analysis by applying MSF processing and selecting the **.msf** file that was created from the MSF window (i.e., saved as the profile model for that element/wavelength).

In the Method make sure to select:

• Process page, Peak Processing tab, MSF as the Peak Algorithm for the appropriate element/wavelength

• Process page, Spectral Corrections tab, MSF, then from the **MSF Table** drop down list select the **.msf** file name.

| Note | BGC points and Pts/Peak are irrelevant to the MSF processing mode and so the system deselects (shades a light gray) those parameters in the Method that are inappropriate, and therefore unavailable when MSF processing is selected. |

• Options page, Results Display/Printed Log Options, check all appropriate boxes so that you will have all the information required to evaluate the analytical results.

• Options page, check all boxes so that you can perform data reprocessing and view spectra in the Examine MSF window, as needed.

Step 5. Perform an Analysis

Tip   **SAVE SPECTRA TO THE SAME DATA SET** so that any spectra used to create the MSF model can be displayed in the Examine MSF window along with the spectra collected for the analysis.

Collect spectra of the:

- blank

- standards

- unknown samples

Step 6. Evaluate Results

The Results Display window lists the analytical results and, in this case, (if you selected to display the Expanded Method header) indicates that the sample was processed by MSF (as applicable, the **.msf** file name will be listed for each analyte in the sample). The Spectra Display window shows both the uncorrected data and the MSF-corrected analyte data.

With Multicomponent Spectral Fitting, you can expect the detection limit will be improved by, at least a factor of 2 (sometimes as large as 5) compared to peak height processing. (For further details refer to *Spectrochimca Acta* Vol.48B. No. 10 pp. 1265-1273, 1993.)

Note Before you analyze an unknown sample with a new model, you should first validate the model by analyzing several samples (These samples should actually be standards with a known quantity of analyte). In this way you will be able to assess whether the model will be valid for the actual samples.

Validate the Model

A common approach to testing the validity of the MSF model is to analyze a solution containing a known amount of the interferent at the analyte wavelength. The resulting concentration should be zero. The zero amount verifies that the MSF model is applying the appropriate correction to your results.

The Matrix Model for MSF

Matrix Notation is used to express the model. (For further details refer to: (1) U.S. patent 5,308,982; Ivaldi et al. (2) N. Draper and H. Smith, Applied Regression Analysis, Wiley New York (1981))

$$Y = X \beta + E$$

Y is the (nx1) vector of the unknown sample spectrum The model of the unknown spectrum is treated as a vector and the analyte concentration is determined for each unknown sample.

X is an (nxp) matrix of the model components (analyte, interferent(s), blank). See more about X below.

β is a (px1) vector of parameters (coefficients) See more about β below.

E is an (nx1) vector of errors (residuals). See more about E below.

(n = number of wavelength positions, p= number of components)

About X

As input from the **.msf** file, the modeled spectral profiles (for blank, analyte, and interferent(s)) are included in matrix X as vectors.

Linear models can be used because...
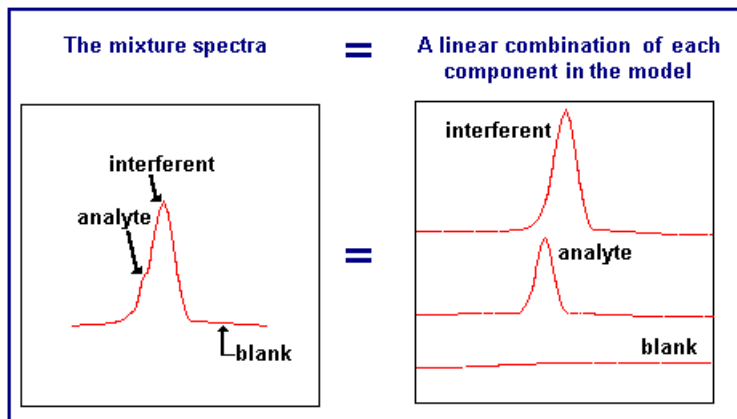
No background correction is needed because the blank is included as a component in the X matrix. This means that background correction is derived automatically from the regression.

About β

β is the regression coefficient. The method of least squares is used to estimate β such that the calculation apportions the relative contribution of the corresponding model to the unknown spectrum.

As illustrated below, this apportionment becomes a quantitative measure of the corresponding element that is present in the unknown sample spectrum.

This "apportionment" is based on the assumption that peak shape remains constant for different concentrations.

This is due to the inherent linearity of the ICP signal and the spectrometer's excellent wavelength stability (registration).

β is directly related to concentration.
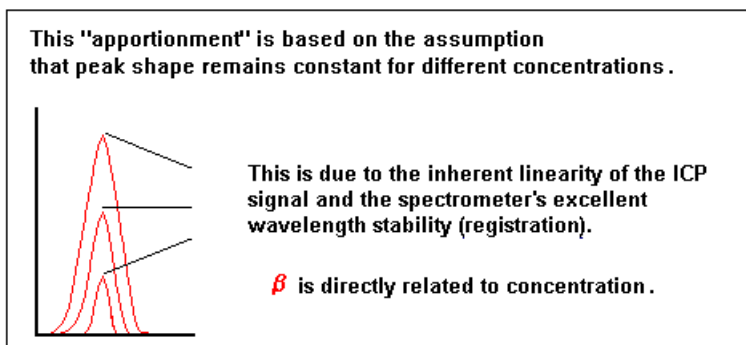
About E

E is the unexplained error that occurs when the model estimates concentration as a function of peak response (intensity and shape). The error term is often called the residual (i.e., **Prediction** from the calibration curve **minus** the **Actual** measured response).
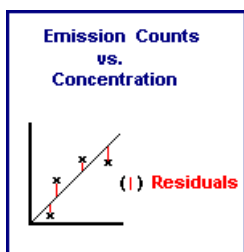
The calibration curve is constructed by applying a linear regression to the actual data. The regression minimizes the residuals to result in the "best" straight line through the data points for each standard.

When a calibration curve is generated there will always be some residual (error). A model can never perfectly match reality and this is why you must use regression equations rather than simply solving a series of simultaneous equations. Multivariate calibration is highly effective in minimizing random error in order to "reality."

Note that the residual applies to the emission counts if the concentration is known.

About Univariate and Multivariate Calibration

Any ICP calibration will try to estimate (or model) the relationship between peak response and standard concentration.

- In univariate calibration (e.g., using Peak Height processing) peak intensity is taken at **one** wavelength.

- In multivariate calibration (such as used in MSF), matrix algebra is used to process peak intensity at **many** wavelengths. When many wavelengths are used, the peak response is not just the intensity but the shape of the peak and this shape becomes an inherent part of the information that is used to calculate the sample concentration. Also, as summarized below, using more of the information from the spectra means that the multivariate calibration models can yield more precise estimates of the unknown concentration and can improve detection limits.

Display of Univariate Versus Multivariate Calibration
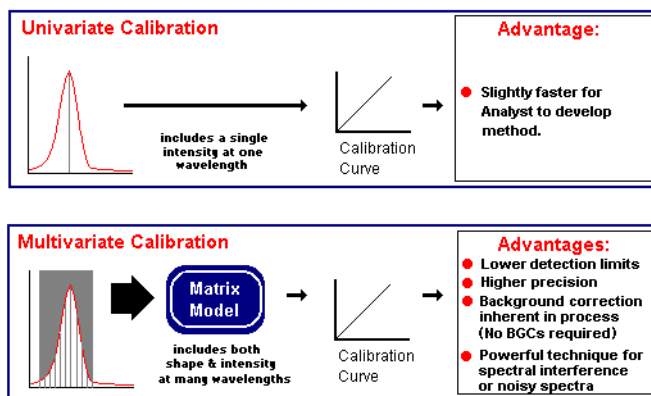
Diagnostics & System Monitors Windows 10

Diagnostics and System Monitor Windows

You can use various windows to monitor your instrument, perform diagnostics, and review system (error) messages.

☛ Select the appropriate window, depending upon the task you wish to perform.

Use	To do this
System Monitors	Check the status of the major components of the system.
Instrument Diagnostics window	Review detailed status information and messages for the spectrometer, plasma generator, and autosampler. Re-establish communication between the system components.

System Monitors

You use the system monitors to check the status of the following:

• Plasma Torch

• Spectrometer

• Autosampler

• Auto Shutdown/Startup

The system monitor displays both a status icon and a system message:
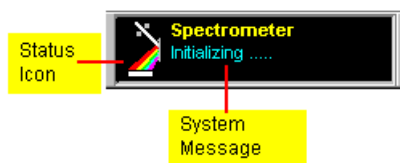
By double-clicking on the status icon for the Plasma, Spectrometer, or Autosampler, you can get further information about the system component in the Instrument Diagnostics window. By double-clicking on the status icon for Auto Shutdown/Startup, you open the Automatic Shutdown/Startup dialog.

Plasma Status Icons

The following icons are used for instrument models with a horizontally mounted torch.

Axial Plasma Off	Axial Plasma On	Radial Plasma On	Radial Plasma Off	Plasma Error

For instrument models with a vertically mounted torch, the icons have a different orientation but the meaning is the same.

Spectrometer Status Icons

Shutter Closed	Shutter Open	Spectrometer Error

Autosampler Status Icons
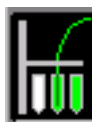
Probe Down	**Probe Up**	**Error**

Auto Shutdown/Startup Status Icons

Not Enabled	**Automatic Shutdown Enabled**	**Automatic Startup Enabled**

System Messages

The System Monitor windows display the most recent messages pertaining to the system component.

Instrument Diagnostics Window

You use this window to review the value of parameters for the overall system and the spectrometer, plasma, and autosampler. This window is also used to reset communication between the system components.

To display this window

▶ In the System menu, click on **Diagnostics.**

Instrument Diagnostics

The Instrument Diagnostics window contains bottom tabs, and each tab provides access to information on each system component, including the status of the component and a log of messages.

Click on the appropriate tab on the bottom of the window to select these pages:
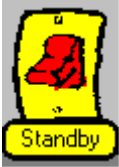
- System Page

- Spectrometer Page

- Plasma Page

- Autosampler Page

Diagnostics: System Page

This page shows a graphic representation of the system, which includes:

- the instrument showing the two major components, the plasma generator and the spectrometer optics;

- the computer

- the autosampler; and

- connections between the system components representing communication through the GPIB (General Purpose Interface bus) interface.

The System page indicates the operational status problem of the system components in the following ways:

Status	Icon	Meaning
Fault		A problem has occurred within this part of the system. Read the Message History by clicking on the appropriate tab in the Diagnostics window. This problem needs to be fixed before you can operate the instrument.
Marginal		This status only pertains to the Optima 2000, 2100, and 7000 and is displayed when the neon lamp is not functioning properly.
Standby		This status pertains to the Optima 3100, 3200, 3300, 4100, 4300, 5100, 5200, 5300, 7100, 7200, and 7300 spectrometers. When this symbol is displayed the spectrometer is in standby mode, which means the argon purge and cooling water are shut off. Standby mode is entered by the user via the **System > Auto Shutdown/Start-up…** menu command.

For example, a red fault is shown in the screen below. If a fault is indicated, obtain more information by clicking on the tab for the system component and reading the Message History. For example, for the screen below, a fault is indicated for the spectrometer so you would click on the **Spectrometer** tab to get more information.
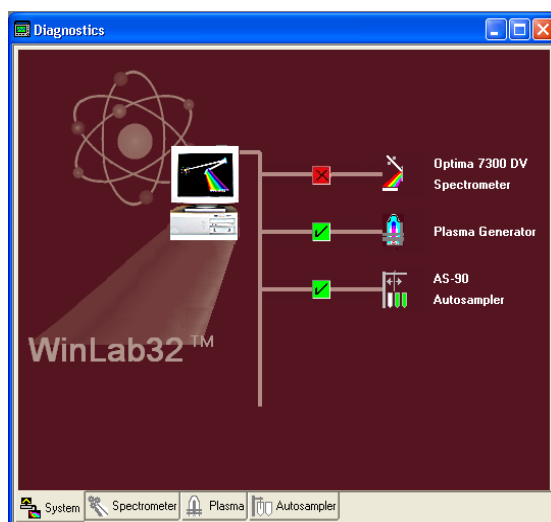
If there is a communication error through the GPIB interface, the connection lines are shown in red.

System Communication

The computer communicates with the instrument and accessories through the GPIB (General Purpose Interface Bus) interface.

The System page of the Diagnostics window indicates the system communication status in the following ways:

Communication Status	Color	Meaning
Connecting	Blue	The system is establishing system communication between components.
Connected	Green	System communication has been successfully established.
Not Connected	Red	A communication problem has occurred.

If there is a communication error through the GPIB interface, the connection lines on the System page of the Instrument Diagnostics window are shown in red. For example, this is indicated for communication between the computer and the plasma generator and spectrometer in the screen below.
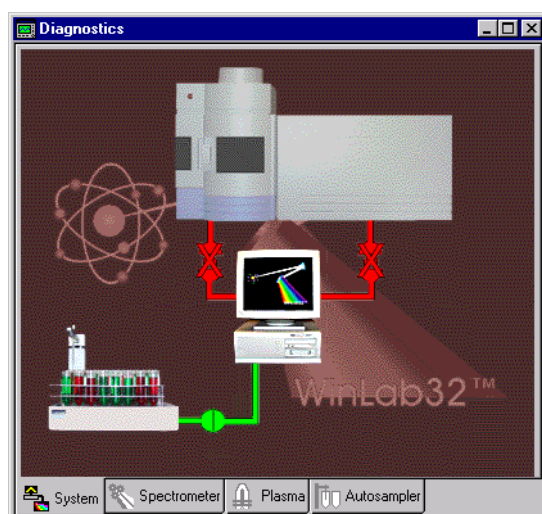
Before starting the software, you should always check that the following system components are on:

- spectrometer

- RF generator

- autosampler

- printer

In addition, check that the Emergency Plasma Off switch on the front panel is not pressed in.

If you turn on any of the devices under GPIB control *after* you start the software, a communication problem may occur. For example, if the autosampler has been disconnected, then reconnected; the autosampler power was off, then turned back on when the software was started; or if you have selected a different type of autosampler, use the **Reconnect** button on the Autosampler page of the Diagnostics window.

If a communication problem should occur, check the following:

1. Check that the power cable is connected and the power is on for the system component.

2. Check that the GPIB cables (also called "IEEE" cables) are connected.

3. If using an autosampler, check that the DIP Switch settings are correct. For more information, see the Hardware Guide for your spectrometer.

4. Click on the tab for the component in the Instrument Diagnostics window. Click on the **Reconnect** button.

5. If a problem still exists, check the connector on the cable. If the pins are broken, order a replacement cable.

In some cases, after clicking on the **Reconnect** button, you may need to exit then restart WinLab32. In addition, it may also be necessary to turn off power to the component, wait a few seconds, then turn it back on.

You may also need to use the Reconnect button when you have a problem related to the instrument status (cooling water, temperature, gas flows). Once you have fixed the status-related problem, it may be necessary to click on **Reconnect** to reset the communication.

Diagnostics: Spectrometer Page

This page displays status information and the Instrument Message History for the spectrometer.

Status

This includes information on the spectrometer model, serial numbers, base module software and firmware versions, and the current status of the instrument. To view all of the information, use the scroll bar. To print the information, click on the **Print** button.

Instrument Message History

This includes a log of all of the messages that have been displayed in the System Monitor for the spectrometer. The most recent message is listed first. All messages listed here pertain to the spectrometer hardware. Messages marked with a question mark (?) indicate a "marginal" problem such as a temperature or voltage is too low or too high. Messages marked with an asterisk (*) indicate a "fatal" problem that would cause the spectrometer to shut down. Refer to *Operating Status Problems* later in this chapter for common status problems. For information on the different spectrometer error messages, refer to the Hardware Guide for your spectrometer.

Configure IP

Click on this button to launch the Spectrometer IP Configure dialog. (This is not available when the Simulation mode is configured.)

Note   This button only appears for Optima spectrometers connected via Ethernet (Optima 5X00 series).

Initialize

This button appears only for Simultaneous ICP spectrometers and is used to start up the spectrometer when it is in standby mode.

Firmware

Click on this button to install the firmware used when the current version of WinLab32 was developed.

Note   This button only appears for users who have administrator privileges for the local Windows Workstation. You can check this under **Start ▶ Programs ▶ Administrative Tools ▶ User Manager**. This button does not appear on Optima 3000 series instruments

Reconnect

Click on this button to reset communication between the system components if a problem has occurred. For more information, see *System Communication* earlier in this chapter.

Print

Click on this button to print the instrument status information and the message history. This information is useful for PerkinElmer service engineers in troubleshooting a problem.

Enable Update

This checkbox updates the instrument status information and instrument message history. When you display this page, this checkbox is selected automatically.

Diagnostics: Plasma Page

This page displays status information and the Instrument Message History for the plasma generator.

Status

This includes information on the generator model, base module software and firmware versions, and the current status of the plasma generator. To view all of the information, use the scroll bar. To print the information, click on the **Print** button.

Instrument Message History

This includes a log of all of the messages that have been displayed in the System Monitor for the plasma. The most recent message is listed first. All messages listed here pertain to the plasma generator. Messages marked with a question mark (?) indicate a "marginal" problem such as a temperature or voltage is too low or too high. Messages marked with an asterisk (*) indicate a "fatal" problem causing the plasma generator to shut down. Refer to *Operating Status Problems* later in this chapter for common status problems. For information on the different plasma generator error messages, refer to the Hardware Guide for your spectrometer.

Firmware

Click on this button to install the firmware used when the current version of WinLab32 was developed.

Note   This button only appears for users who have administrator privileges for the local Windows Workstation. You can check this under **Start** ▶ **Programs** ▶ **Administrative Tools** ▶ **User Manager**. This button does not appear on Optima 3000 series instruments

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Reconnect

Click on this button to reset communication between the system components if a problem has occurred. For more information, see *System Communication* earlier in this chapter.

Print

Click on this button to print the status information and the message history. This information is useful for PerkinElmer service engineers in troubleshooting a problem.

Enable Update

This checkbox updates the instrument status information and instrument message history. When you display this page, this checkbox is selected automatically.

Diagnostics: Autosampler Page

This page displays status information and the Instrument Message History for the autosampler.

Status

This includes information on the autosampler model, base module software and firmware versions, autosampler tray, and the current status of the autosampler. To view all of the information, use the scroll bar. To print the information, click on the **Print** button.

Instrument Message History

This includes a log of all of the messages that have been displayed in the System Monitor for the plasma. The most recent message is listed first. All messages listed here pertain to the autosampler. Refer to *Operating Status Problems* later in this chapter for common status problems.

Firmware

Click on this button to install the firmware used when the current version of WinLab32 was developed.

Note	This button only appears if you have an autosampler that uses downloadable firmware. Also, you must have administrator privileges for the local Windows Workstation. You can check for such privileges under **Start ▶ Programs ▶ Administrative Tools ▶ User Manager**.

Reconnect

If the autosampler has been disconnected, then reconnected; the autosampler power was off, then turned back on when the software was started; or if you have selected a different type of autosampler, use the **Reconnect** button on the Autosampler page of the Diagnostics window. For more information, see *System Communication* earlier in this chapter.

Print

Click on this button to print the status information and the message history. This information is useful for PerkinElmer service engineers in troubleshooting a problem.

Enable Update

This checkbox updates the instrument status information and instrument message history. When you display this page, this checkbox is selected automatically.

Diagnostics: FIAS Page

This page shows important information about the flow-injection system that is useful for troubleshooting and service purposes.

Status

This information identifies your flow-injection system. You should have this information available when you discuss problems with your PerkinElmer service engineer.

Instrument Message History

This is a list of all the messages sent to and received from the computer. This is very useful diagnostic information for the service engineer.

Reconnect
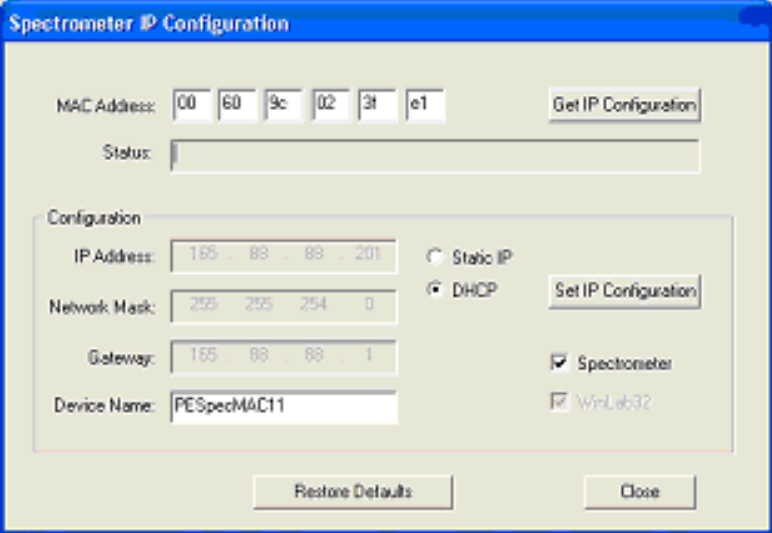
Use this button to reset the communication between the flow-injection system and the computer.

Print

Use this to print the information shown in this window. This page displays status information and the Instrument Message History for the FIAS.

Diagnostics: Spectrometer Page - IP Configure

This dialog enables you to set the IP address configuration for your spectrometer.

MAC Address

Enter the Media Access Control (MAC) Address that is specific to your Optima spectrometer and is a unique identifier. It is located on the spectrometer board, and on a sticker that is located on the back of your Optima instrument.

Get IP Configuration

Click the Get IP Configuration button to retrieve the IP configuration from the spectrometer. The Status area displays the status of selected action.

Configuration Section

Network node configuration of the spectrometer.

IP Address

An identifier for a device on a TCP/IP network. Enter the IP Address when Static IP configuration is selected.

Network Mask

Groups IP addresses together to form a sub net. Enter the Network Mask when Static IP configuration is selected.

Gateway

IP address of your network Gateway. Enter the network address of your network gateway when Static IP configuration is selected.

Device Name

The name used to identify your Optima spectrometer rather than IP address, when DHCP is selected. Enter the Device Name to identify your Optima spectrometer when DHCP is selected.

Static IP

Click to select a fixed Internet Protocol address.

DHCP

Click to select Dynamic Host Configuration Protocol (DHCP), a protocol for assigning a dynamic IP address on a network.

Set IP Configuration

Click this button to set the entered IP configuration into WinLab32 and optionally the spectrometer when the Spectrometer checkbox is checked.

Restore Defaults

Restores default values into all entry fields.

Note: This button does not configure the spectrometer. Only the dialog fields are affected.

Close

Exits the dialog. No other action is taken.

Spectrometer

Check to configure your Optima spectrometer in addition to WinLab32.

WinLab32

This is always checked to select WinLab32 configuration.

Diagnostics: Spectrometer Page - Configuring IP

When you open this dialog, a default MAC address is entered into the MAC Address entry field. The first four fields are populated with the best known starting address for Optima spectrometers. The last two are populated with zeroes. The MAC address will be persisted by WinLab32, and will only require re-entry upon: Re-installation of WinLab32, or when connecting the PC to another Optima instrument. Default values for all other dialog fields are entered into the dialog. Those fields are: IP Address, Network Mask, Gateway, Device Name, Static IP, and DHCP. Values for all these fields are persisted by WinLab32.

The WinLab32 checkbox is always checked and always disabled. Its purpose is to indicate that WinLab32 is always configured whenever the 'Set IP Configuration' button is selected. The Spectrometer checkbox is unchecked when the dialog is opened.

1.   Before proceeding enter the MAC address for the spectrometer the first time WinLab32 is installed. The MAC address is unique to the spectrometer and is located on the spectrometer board, and on a sticker that is located on the back of your Optima instrument.

2.   Click Get IP Configuration once a valid MAC Address has been entered to retrieve the configuration currently set into the spectrometer. The configuration is displayed in controls within the Configuration group box. The status display informs you of the operation sequence and the outcome.

3.   Messages are displayed describing operations performed.

Note: The Status display is cleared upon entry of any value into any field.

4.   Change or set a Static or Dynamic IP address

   •   Change or set a static IP address: Click the Static IP radio button. Enter the IP Address, network mask, and gateway IP address into their respective entry fields, and click Set IP configuration. Monitor the Status display area for the "IP configuration set… Now retrieving IP configuration" message. Then monitor the Status display area for the "IP configuration retrieved" message. This completes the operation. Perform other actions or exit the dialog by selecting the Exit button.

   •   Set a dynamic IP address: Click on the DHCP radio button. Enter a Device Name (See description below). The IP address, network mask, and gateway IP address will be assigned by a server configured on the network. Monitor the Status display area for the "IP configuration set… Now retrieving IP configuration" message. Then monitor the Status display area for the "IP configuration retrieved" message. This completes the operation. Perform other actions or exit the dialog by selecting the Exit button.

Note: Check the Spectrometer checkbox to configure your Optima spectrometer in addition to WinLab32. Only the Device Name is required to configure WinLab32.

Device Name Allowable Settings

The Device Name will be used by ICP WinLab32 for connection to your Optima spectrometer when using DHCP. The DHCP server on your network assigns an IP address and will map the Device Name to that IP address when your application software (ICP WinLab32) connects to the spectrometer. The name is determined by you. Syntax rules for the name are: The name must be unique within your network. One (1) to twenty five (25) characters can be used. The characters can be either letters or numbers, and the first character must be a letter. Upper and lower case letters may be used, although no distinction is made between upper and lower case by a DHCP server when mapping a Device Name.

5.   Recommended Setup for Optima 5X00: Although the TCP/IP connection described in this document is flexible enough to support most LAN topologies, it is recommended that a direct, dedicated, static, single cable connection is used to connect the PC to the Optima spectrometer. The main driver for this decision is that performance can be affected by a DHCP server, and other LAN traffic.

The Optima 5X00's PC can be connected to a LAN and/or Internet via a second network adapter. The IP configuration of this adapter, being separate from the spectrometer network, can be configured to the requirements of the customer's LAN and/or internet provider. A USB adapter is available for this purpose as PerkinElmer part number 0940-4826.

The spectrometer network will have a class B Network mask "255.255.0.0" with the network portion of the IP set to "64.1".

Spectrometer Setup

All Optima 5X00 Spectrometers will have the same IP configuration:

Static IP address "64.1.3.1", Network mask "255.255.0.0". Gateway

is not used and can be set to "0.0.0.0". Device name is not used and can be set to blank.

PC Setup

All Optima 5X00 PC's will have the same IP configuration for the network card used for connection to the spectrometer: Static IP address "64.1.3.2", Network

mask "255.255.0.0", Gateway is not used and can be set to "0.0.0.0", Device name is not used and can be set to blank.

Operating Status Problems

The following table describes common problems associated with the system status. For more information about specific error messages or corrective actions, refer to the Hardware Guide for your spectrometer.

A red fault icon appears in the Instrument Diagnostics window.

The system has been shut down due to a "fatal" error. Click on the tab for the system component and check the Instrument Message History for error messages that describe the cause(s).

Check the operating status problems given below. Refer to the Hardware Guide for your spectrometer for a description of error messages.

Cooling Water Status

Cooling water may not be flowing properly.

Check that the cooling water is on. Check that the cooling water hoses are connected to the back of the instrument and that the hoses are not kinked. Check that the cooling water drain is not obstructed.

If using a chiller, check that it is filled and working properly. Check the chiller water temperature. Check if algae is building up in the cooling lines, causing a blockage.

If using a cooling water supply, check the flow rate and pressure. Check the water filter and change the filter element if it is plugged.

After you fix the problem, you may need to reset the instrument in the Instrument Diagnostics window by clicking on Reconnect. If this does not work, exit and restart the WinLab32 software.

Spectrometer Temperature Status

The spectrometer temperature may not be within the required range.

Check the cooling water as described previously. Monitor the temperature in the Instrument Diagnostics window. The temperature affects instrument stability, so if you operate the system in marginal temperature status, your results may be affected.

Interlock Status

The doors to the sample compartment may not be closed securely. Make sure that the doors are properly closed. Wait for error message to clear.

Interlocks for the argon or water flow may not be satisfied. Check the cooling water supply as described previously. Check the argon supply (see below).

Argon Status

The argon pressure (monitored for the detector purge) may not be in the required range.

Check that the argon supply is on. Check the argon hose connections. Make sure that they are not obstructed. Check the pressure at the cylinder regulator.

If this does not solve the problem, contact a PerkinElmer service engineer to check the argon pressure setting on the instrument.

After you fix the problem, you may need to reset the instrument in the Instrument Diagnostics window by clicking on Reconnect. If this does not work, exit and restart the WinLab32 software.

Nitrogen Status

The pressure monitored for the spectrometer purge may not be in the required range. The purge gas may be nitrogen or argon.

Check that the purge gas supply (nitrogen or argon) is on. Check the gas hose connections. Make sure that they are not obstructed. Check the pressure at the cylinder regulator.

After you fix the problem, you may need to reset the instrument in the Instrument Diagnostics window by clicking on Reconnect. If this does not work, exit and restart the WinLab32 software.

The spectrometer is not ready.

The spectrometer may not have completed the initialization routine. In this case, the software will indicate how long the initialization will take.

All interlocks may not be satisfied. See the information on Interlock Status described previously.

The GPIB cables (also called "IEEE" cables) may be disconnected or there may be a system communication problem. Check all GPIB cables.

Double-check the cooling water, argon, and nitrogen supplies.

Open and close the front door to the sample compartment to try resetting the door interlock.

After you fix the problem, you may need to reset the instrument in the Instrument Diagnostics window by clicking on **Reconnect.** If this does not work, exit and restart the WinLab32 software.

Software Troubleshooting

This section includes general software maintenance and troubleshooting information.

Database Management Tips

On a regular basis, it is important to archive your results data sets on floppy disks, tape, or other storage medium for long term storage. After archiving data sets, you should then remove the data sets and pack the database (library). If you allow the database (library) to become too large, all database operations in WinLab32 such as performing an analysis or reprocessing data may take longer. It is also important to periodically archive your methods, although the method library fills up much more slowly than the results library.

For instructions on archiving data sets and methods, see the *WinLab32 Data Managers User's Guide* or online help.

Exiting the Software and Shutting Down the Computer

Before you turn off your computer, always close all applications and exit Windows properly to avoid damaging files, including the database. To exit Windows operating system, select the shut down option as described in your Windows documentation. A screen message informs you when you can safely turn off your computer.

Software Recovery Options

Occasionally, the keyboard or mouse may "lock up" or a software error message that reads "General Protection Fault" may appear. If one of these situations occurs, refer to the software recovery options that follow.

▶ If you get a "General Protection Fault" message, it is not usually necessary to exit Windows, however, you should exit and restart WinLab32.

▶ If the software "locks up" do the following:

1. Hold down both the **Ctrl** and **Alt** keys and press **Del**. This will usually give you a Windows dialog that allows you to directly shut down the system. Click on **Shut Down...** in this dialog.

   - or -

   If the keyboard is locked and **Ctrl-Alt-Del** has no effect, try using the mouse

   to select **Start** ▶ **Shut Down....**

2. If the plasma was on, it is normal for it to remain on during shutdown. Restart WinLab32 to either resume work or extinguish the plasma. If you need to shut off the plasma, you can also press the red Emergency Plasma Off switch on the front of the instrument.

▶ If you cannot shut down the computer properly as described above, *as a last resort*, do the following:

1. Press the Reset button on the computer.

   - or -

   Turn off the computer and wait 30 seconds (for the components to fully discharge), then restart the computer.

2. If the plasma was on, it is normal for it to remain on during shutdown. Restart WinLab32 to either resume work or extinguish the plasma. If you need to shut off the plasma, you can also press the red Emergency Plasma Off switch on the front of the instrument.

Technical Support

Should you require assistance with a software- or hardware-related problem, contact your local PerkinElmer representative. In addition to a detailed description of the problem, the following files are helpful.

- archived method (see Data Manager help file for instructions)

- archived results library (see Data Manager help file for instructions)

- message history (print out from Instrument Diagnostics window.)

Enable/Disable Service Log Files Dialog

This dialog enables you to provide a PerkinElmer service engineer with information about your instrument for diagnostics and troubleshooting. The service logs record software actions and intermediate results. Software actions concern the commands issued and the control of system devices. Intermediate results or calculations show, for example, how a concentration value was calculated or how data was acquired or normalized.

Note Service logs are not involved in routine procedures. They are for diagnostics and troubleshooting only.

To display this dialog

▶ Hold down both the **Ctrl** and **Shift** keys and press **L**.

Note You cannot display this dialog while an analysis is in progress.

To enable or disable the service logs

1. Hold down both the **Ctrl** and **Shift** keys and press **L** to display the Enable/Disable Service Log Files dialog.

2. Select the service log files that you want to record.

   The service logs will be recorded in the temporary files folder in the user directory, for example /pe/Default User/Temporary Files. The log files are named according to the selections in the dialog. For example, the Auto Sampler Controller Log file is named "AutoSamplerController.Log."

Note When you first enable a service log, the software erases any information that was previously in the log and begins recording new information. When you exit the software, the log files will remain unchanged. When you restart the software then begin recording new information, previous information in the log file will be erased.

Types of Service Logs

The following types of service logs are available:

Controller Logs: There are three Controller logs that record communication initiated by the Controllers to the three system devices: autosampler, plasma generator, and spectrometer. The Controllers send messages and pick up responses to and from the three device servers or "base modules." The device server or "base module" for each of the three system devices is a separate application that controls its respective device.

Device Server Logs: There is a device server or "base module" for each of the three system devices: autosampler or FIAS, plasma generator, and spectrometer. The Device Server logs record the communication between the device server and its respective device.

Analysis Log: This log can record detailed information about the analytical results including pairs of data points; a break-down of analytes according to when they are processed in groups; wavelength calibration calculations; and spectral resolution values.

Result Storage Log: This log shows how results are sent to the results data set.

Stability Log: This log records a peak index value and actual peak wavelength indicating where the peak is located.

Description of Available Log Files

Auto Sampler Controller Log

This log records communication between the controller and the device server (or "base module") for the autosampler or FIAS. The letter "O" indicates OLE commands sent by the controller. The letter "B" indicates low level commands if the option for **Low Level (All Controllers)** is selected in the dialog. The time that each command is sent or received is given in milliseconds.

Plasma Generator Controller Log

This log records communication between the controller and the device server (or "base module") for the plasma generator. The letter "O" indicates OLE commands sent by the controller. The letter "B" indicates low level commands if the option for **Low Level (All Controllers)** is selected in the dialog. The time that each command is sent or received is given in milliseconds.

Spectrometer Controller Log

This log records communication between the controller and the device server (or "base module") for the spectrometer. The letter "O" indicates OLE commands sent by the controller. The letter "B" indicates low level commands if the option for **Low Level (All Controllers)** is selected in the dialog. The time that each command is sent or received is given in milliseconds. Select **Data Xfer Block** to record a block of information for each replicate containing spectral data for each analyte. Errors shown indicate saturation codes.

Analysis Log

Select **Detailed** to record data points for intensity and wavelengths for each survey window. The detailed log also includes calibration coefficients and number of calibration standards.

Select **Process Groups** to record how analytes are grouped according to processing parameters. Since instrument settings can vary by analyte, the instrument separates the analytes into groups that have the same settings. This log shows the number of processing groups, the analytes in each group, and a list of the parameters used.

Select **Wave Cal.** To record the peak offset values generated during a wavelength calibration. This log shows the equation: Difference = Measured - Theoretical. It also lists the Result Vector and RMS (Root Mean Square) values.

Select **Spectral Resolution** to display the resolution for each analyte peak. The resolution (in nanometers) is equal to the peak width at half of the peak height. **Note:** This value is only valid if the Peak Height algorithm is used.

Results Storage Log

This log records the analytical data sent to the results data set, including the result index, sample index, mean index, replicate number, corrected intensity, the number of data points in the spectral data.

Stability Log

For each replicate, this log records the sample name, analyte name, corrected peak intensity, peak index indicating where the peak was found in a series of data points, and actual peak position in nanometers.

AsFIAS32 Log

This log records the communication between the device server and its respective device (autosampler or FIAS). Select **Server** to record OLE commands from the controller. Select **Device** to record commands from the device server. These commands are labeled "C" for a device server command or "A" for an answer from the device. Select **Errors** to display any error messages.

Generator32 Log

This log records the communication between the device server for the plasma generator and the firmware. Select **Server** to record OLE commands from the controller. Select **Device** to record commands from the device server. These commands are labeled "C" for a device server command or "A" for an answer from the device. Select **Errors** to display any error messages.

SimSpec32 Log or SeqSpec32 Log

This log records the communication between the device server for the spectrometer and the firmware. The SimSpec32 log applies to simultaneous ICP spectrometers. The SeqSpec32 log applies to scanning CCD ICP spectrometers. Select **Server** to record OLE commands from the controller. Select **Device** to record commands from the device server. These commands are labeled "C" for a device server command or "A" for an answer from the device. Select **Errors** to display any error messages.

SimSpec32 Log (Device Qualifiers)

For simultaneous ICP spectrometers, you can also select Device Qualifiers to be recorded in the SimSpec32 Log. To do this, you must select **Device** under the SimSpec32Log, then select the Device Qualifiers.

Select **Auto Integration Groups** to record information about the different analyte groups used in auto integration. Since auto integration times can vary by analyte, the instrument separates the analytes into groups that have the same integration times. This log shows the number of auto integration groups and the analytes in each group. Select **Detailed** to record the grouping algorithm in process and the maximum intensity within the integration window.

Select **Fixed Integration Groups** when Manual settings were selected in the method to record information about groups of analytes with fixed integration times.

Select **Spectra Acquisition** to record the data block sent back from the spectrometer to the base module.

Select **Normalization** to record the treatment of each data point as it is converted to counts per second (cps). The normalization routine subtracts the signal bias (SB), applies the linearity constant, convert to cps, and subtracts the dark current (DC).

Select **Data Xfer Block** to record a block of information for each replicate containing spectral data for each analyte. This data transfer block is the counterpart to the data transfer block for the Spectrometer Controller Log and is in an identical format.

WinLab32 Message Dialogs

During WinLab32 operation, messages may appear to provide you with information to correct entries in the software and ensure that valid data is collected. These messages can be printed.

WinLab32 General Messages dialog

When there is an inconsistency in input information, WinLab32 alerts you with a system message dialog. This identifies the concern and provides an appropriate action to enable you to make a correction, or it may present you with a choice of action.

WinLab32 messages can include the following message dialogs:

- Check Method dialog
  For more information see Check Method dialog in Chapter 11, *Menus and Toolbar*.

- Analysis Checking and Error dialogs (discussed on the next page.)

Analysis Checking dialog

After you have entered the analysis information and begin the analysis, the software checks the entries to make sure everything is set up properly.

A partial list of the checks performed is given below:

- if used, recovery standards are defined

- results library specified is not too large to use

- sample information file entries are complete and consistent

Analysis Error (Warning) Messages dialog

Depending on the error found, the software may display a software warning message. (In the Hardware Guide for your instrument, a warning means there is potential personal injury. The analysis error (warning) messages here alert you to possible collection of invalid data.)

A partial list of the checks that may result in a software warning message are:

- reslope standards, QCs, recovery sets are specified correctly

- the method selected for reprocessing data is the same method used to collect the original data.

- plasma is not turned on

- peristaltic pump is not turned on

- nebulizer gas is not turned on

Progress Timer Dialog

During the operation of many processes throughout instrument operation, a Progress Timer dialog appears. This dialog reports the time remaining until the specified event occurs and gives you an opportunity to cancel, if desired.

For example, this dialog appears when you have selected **Immediately on OK** in the Shutdown/Startup dialog and click on OK. It shows the amount of time until

shutdown.

Menus and Toolbar 11

About the Toolbar and Menus

You use the toolbar for quick access to key windows and dialogs available in the software.
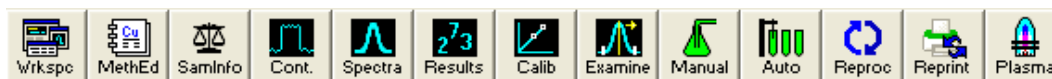
You use the menus to access the windows, commands and dialogs available in the software.
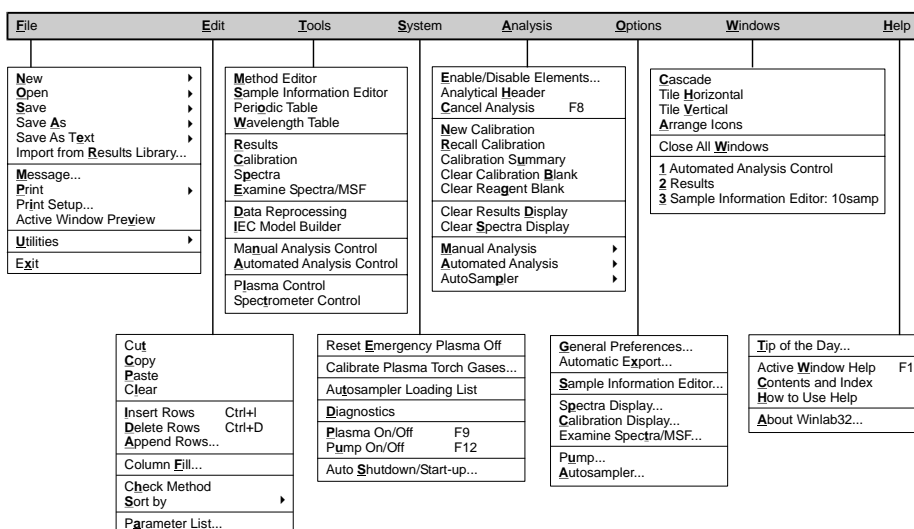
Toolbar

The Toolbar is located below the menu bar in the application window. You can use the buttons on the toolbar as a shortcut to accessing key windows and dialogs in the software.

Note   If you do not see the toolbar: In the Options menu, click on General Preferences and then click on the Toolbar checkbox to select it.

You can place the toolbar anywhere along the edge of the WinLab32 window with the toolbar buttons aligned either horizontally or vertically. Click on the background area surrounding the toolbar buttons and drag (hold down the mouse button and move the mouse) the toolbar to the location you wish.

To resize the toolbar, place the mouse cursor along any edge of the toolbar so that a double arrow appears. Then hold down the mouse button and resize the toolbar.
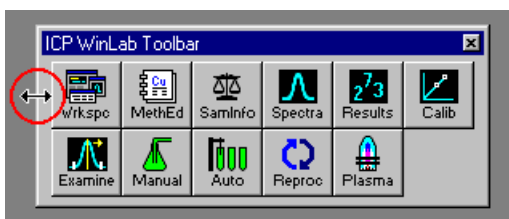
Toolbar Buttons

When you click on the Workspace (Wrkspc) button, the Open Workspace File Open dialog appears, which lists stored workspaces. To create a new workspace, see *Workspaces* in Chapter 1, *Introduction to WinLab32*.
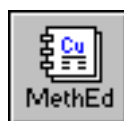
When you click on the Method Editor (MethEd) button, the Method Editor appears, which you use to enter analytical parameters.

When you click on the Sample Information (SamInfo) button, the Sample Information Editor appears, which you use to enter information about your samples.

When you click on the Spectra button, the Spectra Display window appears, which you use to view spectra during analyses.

When you click on the Results button, the Results Display window appears, which you use to view analytical data during analyses.

When you click on the Calibration (Calib) button, the Calibration Display window appears, which you use to view the calibration curves. The calibration curves displayed are from the most recent analysis. You can clear the calibration curves or recall a stored calibration. For more information, see the *Analysis Menu Commands* later in this chapter. In addition, you can specify the calibration to be used in the method on the Calibration: Initial Calibration Options page.

When you click on the Examine button, the Examine Spectra window appears, which you use to examine the details of spectra one element at a time or use Multicomponent Spectral Fitting (MSF). Peak wavelength and background correction points may be fine-tuned here.

When you click on the Manual button, the Manual Analysis Control window appears, which you use to perform analyses with manual sampling. You can also use this window to control the autosampler manually during an analysis.

When you click on the Auto button, the Automated Analysis Control window appears, which you use to perform automated analyses with an autosampler.

When you click on the Reprocessing (Reproc) button, the Data Reprocessing window appears, which you use to reprocess previously gathered data.

When you click on the Reprint button, the Reprint Data screen appears, which you use to specify which results data you want to reprint. Reprinting data will create an exact copy of your original results data printout..

When you click on the Plasma button, the Plasma Control window appears, which you use to ignite and adjust the plasma conditions and peristaltic pump.

When you click on the Method button, a dialog that you use to open a stored method appears. The name of the method that is currently open appears in the status display.
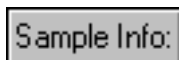
When you click on the Sample Info button, the Open Sample Information dialog appears, which you use to open a stored file. The sample information file that is currently open appears in the status display.

File Menu Commands

File Menu Command	Description
New ▶ Method...	Use this command to create a new method. The Create New Method Dialog appears, where you can select a set of plasma conditions in a new method for different types of analyses, for example, organic or aqueous. Or, you can choose to use a copy of the active method or another stored method as a starting point.
New ▶ Sample Info. File...	Use this command to create a new sample information file. The New Sample Information File dialog appears, in which you select the default design (default.sid) or a design you have created. The design (.sid) file is a template that contains the sample parameters that you want to use. To create a design file, see *Using the Sample Information Editor* in Chapter 4, *Sample Information Editor*.
New ▶ MSF Model...	Use this command to create a new Multicomponent Spectral Fitting model.
New ▶ IEC Model...	Use this command to create a new Interelement Correction model. The Interelement Correction (IEC) Model Builder appears showing the Set Up page.
Open ▶ Method...	Use this command to select a stored method for editing or performing manual analyses. The Open Method dialog appears.
Open ▶ Sample Info File...	Use this command to select a stored sample information file for editing or performing manual analyses. An Open dialog appears, where you can select a sample information file.

File Menu Command	Description
Open ▶ MSF Model...	Use this command to open an existing Multicomponent Spectral Fitting model. An Open dialog appears, where you can select an MSF model.
Open ▶ IEC Model...	Use this command to open an existing Interelement Correction model. An Open dialog appears, where you can select an IEC model.
Open ▶ Workspace...	Use this command to open a workspace, which contains a layout of stored windows and entries. An Open dialog appears, where you can select a workspace.
Save ▶ Method	Use this command to save changes to the active method. The first time you save a Method, the Save As Method dialog will appear prompting you for a name. Note: If, for any reason, a method is not ready to be saved, a check method dialog will appear.
Save ▶ Sample Info File	Use this command to save changes to the active sample information file. The first time you save a sample information file, the Save As Sample Information File dialog will appear prompting you for a name.
Save ▶ MSF Model...	Use this command to save changes to the active Multicomponent Spectral Fitting model.
Save ▶ IEC Model...	Use this command to save changes to the active Interelement Correction model.
Save As ▶ Method...	Use this command to save the active method with the latest changes in the methods database. The Save As Method dialog appears, where you can enter a name for the method.

File Menu Command	Description
Save As ▶ **Sample Info File...**	Use this command to save the active sample information file with the latest changes in a separate file. A Save As dialog appears, where you can enter a name for the sample information file.
Save As ▶ **Sample Info Design...**	Use this command to save the active sample information design with the latest changes in a separate file. A Save As dialog appears, where you can enter a name for the sample information design.
Save As ▶ **MSF Model...**	Use this command to save the active Multi-component Spectral Fitting model with the latest changes in a separate file. A Save As dialog appears, where you can enter a name for the MSF File.
Save As ▶ **IEC Model...**	Use this command to save the active Interelement Correction model with the latest changes in a separate file. A Save As dialog appears, where you can enter a name for the IEC File.
Save As ▶ **Workspace...**	Use this command to save the active workspace, which contains a layout of stored windows and entries, with the latest changes in a separate file. A Save As dialog appears, where you can enter a name for the workspace.

File Menu Command	Description
Save As Text	Use this command to save the contents of the Method Editor, a sample information file, Autosampler Loading List, or IEC Factors to an ASCII text file. You can then use a text editing application to view, modify or print the information.
Import from Results Library...	Use this command to open the method, MSF Model, or IEC model that was used to generate a data set. First, select the results data set, then select the method or model(s) you want to import. The method or model(s) will then be opened in the appropriate windows.
Message	Use this command to print or save a message with your results. The Message dialog appears, where you enter the message.
Print ▶ Active Window	Use this command to print the contents of the active window such as the Method Editor, the Calibration Display window, the Spectra Display window, the Examine Spectral/MSF window, a sample information file, or any text display window, such as the Results Display window or Autosampler Loading List. Click on the window of interest and then select this command. The Print dialog appears, where you can select the pages to print. Note: Printouts of spectra and calibration curves are always in the layout of 2 columns and 3 rows. (See your Windows manual for further information on printing, if needed.)

File Menu Command	Description
Print ▶ **Reset Page Numbers**	When information in the Results window is printing, use this command to reset the page number of the next page to be printed to 1. This overrides the usual situation where the page numbers are consecutive when the Results window is printed.
Print ▶ Skip Line	In the Results window, use this command to add a blank line after the end of the text. When the Results window is printed, it will contain the added space.
Print ▶ New Page	When information in the Results window is printing, use the command to force a page to print, even if the page is only half filled with information. Usually, a page does not print until it is completely filled.
Print Setup...	Use this command to set options for printing, such as printer name, paper size and orientation. (See your Windows manual for further information on printing, if needed.)
Active Window Preview	Use this command to view the contents of the active window before printing.

File Menu Command	Description
Utilities	Use this command to view a submenu where you can start companion WinLab32 applications. Use Data Manager to archive and maintain Method and Results Data Sets, create printed reports of data, and convert data for use with spreadsheet and database management programs. Use WinLab32 Offline while the instrument is analyzing samples on-line to begin a new session of WinLab32. You can use WinLab32 Offline for entering information or reprocessing data. WinLab32 Offline does not allow you to control the instrument.
Exit	Use this command to exit the WinLab32 software application. This command prompts you to save data and to confirm that you want to exit, then it shuts down the plasma and the WinLab32 software.

Create New Method Dialog

This dialog gives you the option of selecting a set of recommended plasma conditions in a new method for different types of analyses, for example, organic or aqueous. It also gives you the option of selecting either the active method or another stored method as a starting point for creating a new method.

To display this dialog

▶ In the File menu, click on **New** ▶ **Method…**

To select plasma conditions in a new (default) method

1. Click on the Default option to start with a new "Untitled" method.

2. Select one of the following plasma conditions:

    Aqueous (see conditions given below)
    Organic (see conditions given below)
    Add Conditions

    - or -

    Select **Modify.**

    When you select **Add Conditions** or **Modify** you can set up conditions for a customized type of analysis. The Plasma Default Conditions Dialog appears. Enter a Plasma Conditions Name and select the appropriate settings for your analysis.

    The software will open the Method Editor with the settings you selected.

Plasma Conditions for aqueous and organic analyses

Parameter	Aqueous	Organic
Plasma Flow:	15 L/min	15 L/min
Auxiliary Flow:	0.5 L/min (for Optima 3000 Family ICP spectrometers)	1.0 L/min
	0.2 L/min (for other simultaneous ICP spectrometers or for scanning CCD ICP spectrometers)	
Nebulizer Flow: (for Low-Flow GemCone or the Concentric Glass nebulizer)	0.80 L/min	0.50 L/min
RF power:	1300 watts	1300 watts
Sample Flow Rate	1.50 mL/min	0.80 mL/min

To select the active method or a stored method to use as a starting point

When creating a new method, it may be useful to start with a copy of the active method or another stored method as a basis for creating the new method. By selecting **Copy of active method** or **Copy of another method**, a copy of the method is opened in the Method Editor called "Untitled." Modify the settings as needed, then save it under a different name using **Save As** ▶ **Method** in the **File** menu.

Plasma Default Conditions Dialog

You use this dialog to select settings for plasma conditions, aerosol type, and nebulizer startup when creating a new method. Recommended settings are displayed for Aqueous and Organic analyses.

To display this dialog

▶ In the File menu, click on **New** ▶ **Method** and in the Plasma Default Conditions Dialog that appears, click on **Modify..**.

Plasma Conditions Name

This shows the name of the set of plasma conditions that you selected in the Create New Method dialog. When defining a new set of conditions, enter a name here.

Post-ignition Plasma Conditions

Plasma Gas Flow (L/min)

This is the flow rate of the argon gas that is contained between the outer and intermediate injector tubes in the torch.

Auxiliary Gas Flow (L/min)

This is the flow rate of the auxiliary argon gas, which is contained between the intermediate and center injector tubes in the torch.

Nebulizer Gas Flow (L/min)

This is the flow rate of the nebulizer gas, which carries the sample aerosol into the plasma.

RF Power (Watts)

This is power in watts transmitted from the RF generator to the ICP.

Sample Flow Rate (mL/min)

This is the flow rate of the solution pumped into the nebulizer by the peristaltic pump. Its range depends on the type of pump and pump tubing being used, but is typically 1 to 3.

Aerosol Type

This shows the type of aerosol that will be used for the analysis.

Wet
Use when the sample aerosol is produced by nebulizing a solution using a pneumatic nebulizer, such as the cross-flow nebulizer or the GemCone nebulizer.

Dry
Use when the sample aerosol is desolvated, i.e., by an ultrasonic nebulizer or is produced directly from a solid sample, e.g. using laser sampling.

Nebulizer Start

These are two procedures that control the post-ignition nebulizer argon flow differently.

Instant turns the nebulizer argon flow on quickly after ignition. Use this for most cases.

Gradual turns the nebulizer argon flow on slowly after ignition. Use this for igniting while aspirating high solids sample matrices or when the first procedure does not work.

For more information, see *Recommended Settings for Plasma Parameters* in Chapter 6, *Hardware Control Windows*.

New Sample Information File Dialog

You use this dialog to create a new sample information file based on a selected design.

To display this dialog

▶ In the File menu, click on **New Sample** ▶ **Information File...**

Using this dialog

Select a sample information design name from the Design List. This list includes a default design and any other designs that have been previously created and stored. A sample information design includes the exact parameters that you need to

describe your samples. For information on creating a sample information design, see *Using the Sample Information Editor* in Chapter 4, *Sample Information Editor*.

Opening and Saving Methods and Selecting Results

This topic covers the Open Method, Save Method As and Select a Results Data Set dialogs.

Check the database library location

The database library is where the methods or results are stored. The current library location is shown. If the stored methods or results data set does not appear, you may look for it in a different database library location.

1. To change the library location, click on **Browse...**

2. In the Open dialog that appears, select the directory path and file name for the library.

Methods

To open a stored method (Open Method dialog)

▶ Click on the name in the table, then click on **OK.**

-or-

▶ Double-click on the name in the table.

To save a method (Save Method As dialog)

▶ Type a name of up to 25 characters in the **Name** entry field, then click on **OK.**

Results Data Sets

To select a stored results data set (Select Results Data Set dialog)

▶ Click on the name in the table, then click on **OK.**

-or-

▶ Double-click on the name in the table.

To create a new results data set

▶ Type a name of up to 25 characters in the **Name** entry field, then click on **OK.**

To sort the method or data set list

▶ Click on the appropriate option button to sort by Name or Date/Time.

Open Dialog

You use this dialog to select a stored file. This can be either a sample information file, a Multicomponent Spectral Fitting model, an Interelement Correction model, or a workspace file.

To display this dialog

▶ In the File menu, click on **Open,** then select either **Sample Info File..., MSF Model...**, **IEC Model...** or **Workspace...**

Using this dialog

The default folder where files are stored appears in the Look In box, and the extension for the file type you are opening automatically appears in the Files of Type entry field.

For example, if you are opening a sample information file, the default folder that appears contains existing sample information files. The .sif extension appears in the File of Type entry field.

▶ Click on the name of the file you want to open, then click on **Open.**

Save As Dialog

Use this dialog to save the active file with the latest changes in a separate file. This can be either a sample information file, a sample information design file, a workspace file, an Interelement Correction model or a Multicomponent Spectral Fitting file.

To display this dialog

▶ In the File menu, click on **Save As** then select the type of file you want to save.

To save the file

The default folder where files are stored appears in the Look In box.

1. Type a name in the File name entry field.

| Note | Windows allows you to use filenames longer than the 8-character names required by MS-DOS conventions. |

The software automatically adds the appropriate extension.

2. Click on **Save.**

Import from Results Library Dialog

Use this dialog when you need to open the method, MSF Model, or IEC model that was used to generate a data set. For example, if you need to reprocess data, you would open the method that was used to generate the data set before starting reprocessing.

To display this dialog

▶ In the File menu, click on **Import from Results Library.**

To import a method, MSF Model, or IEC Model

1. In the **File** menu, click on **Import from Results Library.**

2. Select the results data set and click on **OK.**

3. When the Import from Results Library dialog appears, select the method or model(s) you want to import and then at the bottom of the dialog, select the corresponding check boxes to describe your selection. Click on **OK**.

The method is opened in the appropriate windows.

The MSF model is opened in memory so that the results may be reprocessed with this MSF model. However, the MSF model is not available in the MSF/Examine window, nor is it available for use with other methods.

The IEC model is opened in memory and will also appear in the IEC Model Builder window, if you have that window open. If desired, this model can then be saved, so that a copy resides in the IEC directory. The IEC model can then be used in other methods.

Message Dialog

Use this dialog to print a message regarding the analysis in your results. You can also save this message to the results library.

To display this dialog

► In the File menu, click on **Message…**

Using this dialog

1. Type the message in the dialog.

2. Check that you have the desired option(s) checked:

    If you select **Print Message**, the message will appear in the Results window and be printed in the log.

    If you select **Save Message**, the message will be saved in the active results library.

Edit Menu Commands

Edit Menu Command	Description
Cut	Use this command to remove selected text or values in a table and place in the Windows clipboard.

Edit Menu Command	Description
Copy	Use this command to copy selected text or values in a table and place in the Windows clipboard.
Paste	Use this command to insert text or values from the Windows clipboard into a table.
Clear	Use this command to remove selected text or values in a table.
Insert Rows	Use this command to insert an empty row into a table of parameters such as those in the Method Editor or Sample Information Editor. Select a position in the line after the one you want to insert and then select the command. A line will be inserted before the line containing the cursor. All entries below the inserted line move down one position.
Delete Rows	Use this command to delete selected rows from a table. All entries below move up respectively one position.
Append Rows	Use this command to add rows to the end of the Sample Information table. In the Sample Information Parameters Dialog that appears, select the number of rows you want to append by using the spin box labeled **Maximum number of samples.** You can also modify the sample information parameters in this dialog.
Column Fill...	Use this command to fill a selected column in the Method Editor or Sample Information Editor. When you click on this command, a dialog appears where you enter values to fill the selected column.

Edit Menu Command	Description
Check Method	Use this command to check for any problems or inconsistencies in the method.
Sort by... ▶ **Element Symbol**	When the Method Editor Instrument page is open, use this command to sort the elements in the method alphabetically by the element symbol.
Sort by... ▶ **Wavelength**	When the Method Editor Instrument page is open, use this command to sort the elements in the method by the wavelength.
Sort by... ▶ **Analyte Name**	When the Method Editor Instrument page is open, use this command to sort the elements in the method alphabetically by the element name.
Sort by... ▶ **Function**	When the Method Editor Instrument page is open, use this command to sort the elements in the method by the function (as indicated by "analyte" or "internal standard").
Parameter List....	Use this command to open the Sample Information Parameters dialog, where you can add parameters to a sample information file. Parameters can be common to all samples or they can vary by sample.

Check Method

This message dialog lets you know the results of the Check Method command in the Edit menu. This command checks to make sure that the method is self-consistent and, if any problems are found, the dialog displays information about what corrections are needed. When you save a method, the method will be checked automatically.
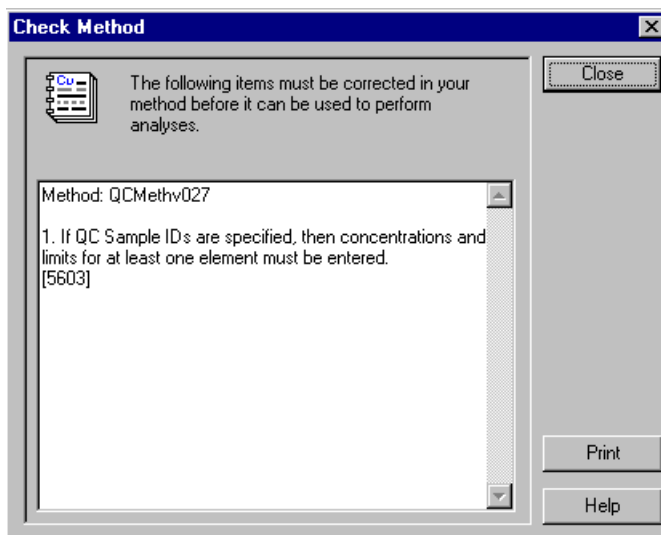
When a method is checked, the software examines the parameters to make sure that the information you have entered is fully complete. A partial list of the checks performed is given below:

• standard concentrations are entered

• the reslope standard concentrations are valid

• autosampler locations for calibration standards

• wavelengths are available

• survey and auto integration windows are properly defined

- plasma conditions are valid

- internal standard assignments

- IEC selections

- MSF selections

- Quality Control samples (concentrations, schedule, failure actions)

Tools Menu Commands

Tool Menu Command	Description
Method Editor	Use this command to view the Method Editor, where you enter analytical parameters.
Sample Information Editor	Use this command to view the Sample Information Editor, where you enter the location of the samples in the autosampler, sample IDs, sample weights and sample volumes.
Periodic Table	Use this command to open the Method Periodic Table, where you enter elements and wavelengths to create a new method or modify an existing method.
Wavelength Table	Use this command to view the Wavelength Table window. You use this window to select wavelengths when creating or editing a method or identifying interferences viewed in the Examine Spectra window.
Results	Use this command to view the Results Display window, which shows the measurements made on the current sample, the calculated concentration values, and statistical information.
Calibration	Use this command to view the Calibration Display window, which shows the active calibration curves.
Spectra	Use this command to view the Spectra Display window, which shows spectra acquired during an analysis.

Tool Menu Command	Description
Examine Spectra/MSF	Use this command after data is acquired and saved to view the Examine Spectra window, where you can examine the spectra in detail, one element at a time. You can use this window to overlay spectra and to select peak wavelength and background points. You also use this window for Multicomponent Spectral Fitting (MSF) by creating models for analytes and interferents. You must save the data to perform these functions in this window.
Data Reprocessing	Use this command to view the Data Reprocessing window. You use this window to reprocess existing data.
IEC Model Builder	Use this command to view the Interelement Correction Model Builder. You use this tool to create factors that correct for spectral interferences.
Manual Analysis Control	Use this command to view the Manual Analysis Control window. You use this window to perform analyses with manual sampling.
Automated Analysis Control	Use this command to view the Automated Analysis Control window. You use this window to perform automated analyses with an autosampler.
Plasma Control	Use this command to view the Plasma Control window. You use this window to ignite and adjust the plasma, control the peristaltic pump and check instrument status.
Spectrometer Control	Use this command to view the Spectrometer Control window. You use this window to align the torch viewing position and select spectrometer settings.

System Menu Commands

System Menu Command	Description
Reset RF Emergency Off	Use this command to reset the RF Emergency Off switch after it has been used to extinguish the plasma.
Calibrate Plasma Torch Gases…	Use this command only when a new torch has just been installed or if a message has been displayed indicating that there is a problem. Performing this calibration at other times is unnecessary. The calibration procedure shuts off the plasma and takes approximately six minutes to complete.
Autosampler Loading List	Use this command to view information about the solution in each autosampler location. When you click on this command, the Autosampler Loading List appears.
Diagnostics	Use this command to review the current status of parameters for the spectrometer, plasma and autosampler. When you click on this command, the Instrument Diagnostics window appears.
Plasma On/Off	This command opens the Plasma Control window and turns on the plasma. If the plasma is already on, this command turns it off, but does not close the Plasma Control window.

System Menu Command	Description
Pump On/Off	This command opens the Plasma Control window and turns on the pump. If the pump is already on, this command turns it off, but does not close the Plasma Control window.
Auto Shutdown/Startup...	Use this command to select options for automatically shutting down or starting up the instrument. When you click on this command, the Automatic Shutdown/Startup dialog appears, where you make your selections.

Automatic Shutdown/Startup Dialog

You use this dialog to select options for automatically starting up or shutting down the instrument.
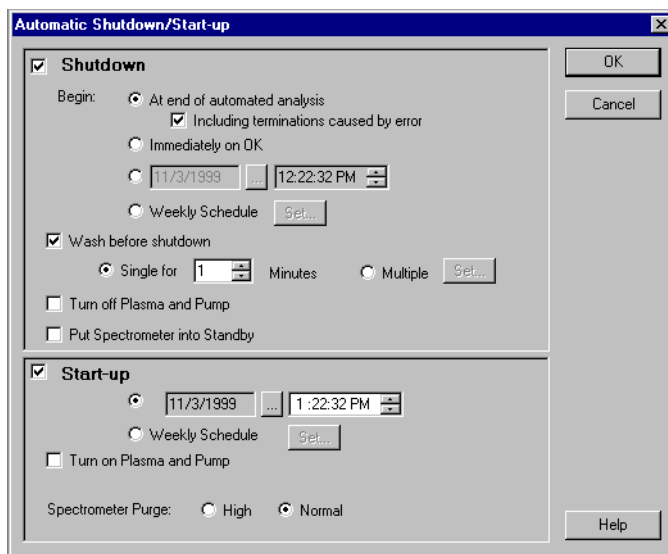
To display this dialog

▶ In the System menu, click on **Auto Shutdown/Startup...**

-or-

▶ In the Automated Analysis Control window Set Up page, click on **Set...** next to **Auto Shutdown**.

Shutdown

▶ To program an automatic shutdown, select **Shutdown** and select one of the following options:

At end of automated analysis
To schedule the automatic shutdown for the end of the next automated analysis, select this option. If you want the shutdown conditions you select to occur if the analysis is terminated by a hardware error or QC stop action, select **Including terminations caused by error**.

Immediately on OK
Select this option to schedule the automatic shutdown to occur immediately after you click on **OK** to close this dialog. A timer dialog appears and counts down the time to shutdown. It also allows you a brief opportunity to cancel the shutdown, if desired.

Date/Time entry fields
To schedule the automatic shutdown for a specific date and time, enter the date, hour and minute when the shutdown is to occur. Base the time on a 24-hour clock. For example, 8:30 p.m. would be entered as 20:30.

Weekly Schedule
To schedule times during the days of the week for automatically shutting down the instrument, click on **Set…**

Note The WinLab32 software must be left on before the time when the automatic shut down is scheduled.

Wash before shutdown
Select this box to specify the time in minutes that you want the wash to continue before the instrument automatically shuts down. You must be using an autosampler for this option. You can use single or multiple wash solutions:

Single for x minutes
Select this option if you have a single wash solution and enter the wash time. The location of the wash solution is selected in the method on the **Sampler: Autosampler Wash** page.

Multiple
Select this option if you have multiple wash solutions, for example, if you want to wash with nitric acid in autosampler location 2 for 10 minutes and then wash with deionized water in location 3 for 8 minutes. Click on **Set..** to enter the wash times and autosampler locations of the wash solutions.

Shutdown Options

Turn off Plasma and Pump
Select this box to turn off the plasma and pump upon shutdown.

Put Spectrometer into Standby
Select this box to put the spectrometer into a standby mode. This turns off the detector argon purge and cooling water.

Start-up

▶ To program an automatic startup, select **Start-up** and select one of the following options:

Date/Time entry fields
Enter the exact date and time that the instrument is to begin operation. Base the time on a 24-hour clock. For example, 8:30 p.m. would be entered as 20:30.

Note   If you do not schedule an automatic start-up, you can start the spectrometer manually from standby mode. In the System menu, click on Diagnostics. When the Diagnostics window appears, click on the Spectrometer tab and then on the Initialize button.

Weekly Schedule
To schedule times during the days of the week for automatically starting up the instrument, click on **Set…**

Note The WinLab32 software must be left on before the time the automatic start-up is scheduled.

Start-up Options

Use these options to select whether the plasma is to be ignited and the pump is to begin operating upon startup. You can also select whether to perform a high purge for the spectrometer upon startup.

For more information, see *Performing Analyses: Setting Up the Instrument* in Chapter 2, *Performing Analyses*, and *Performing Analyses: Shutting Down the Instrument* also in Chapter 2, *Performing Analyses*.

Weekly Schedule Dialog

You use this dialog to schedule times during the days of the week for automatically starting or shutting down the instrument.

To display this dialog

▶ In the Automatic Shutdown/Startup dialog, select **Weekly Schedule** and click on **Set…**

To set a weekly schedule

1. Select the day(s) of the week when you want to automatically shut down or start up the instrument.

2. Select the shutdown or start-up time(s).

Note The WinLab32 software must be left on before the time when the automatic shutdown or start-up is scheduled.

For more information see *Performing Analyses: Setting Up the Instrument* in Chapter 2, *Performing Analyses*, and *Performing Analyses: Shutting Down the Instrument* also in Chapter 2, *Performing Analyses*.

Multiple Wash Settings Dialog

Use this dialog if you want to use multiple wash solutions before the instrument automatically shuts down. For example, you can wash with a 5% or 10% nitric acid solution in autosampler location 2 for 10 minutes and then wash with deionized water in location 3 for 8 minutes.

To display this dialog

1. In the System menu, click on **Auto Shutdown/Startup...**

2. Select **Enable shutdown** and **Wash before shutdown.**

3. Select the **Multiple** wash option, then click on **Set…**

Using this dialog

Enter the wash time and autosampler location for each wash solution.

Analysis Menu Commands

Analysis Menu Command	Description
Enable/Disable Elements...	Use this command to include or exclude an element from your method in an analysis. When you click on this command, the Enable/ Disable Elements dialog appears, where you can make your selections.

Analysis Menu Command	Description
Analytical Header	Use this command to print the analytical header. This is particularly useful when you have reset a Manual Analysis. The analysis header will be displayed in the Results window and will also be printed provided that a printed log is selected. The analysis header includes the start time, name of analyst, sample information file name, results data set name, and results library name, and other general information about the analysis.
Cancel Analysis...	Use this command to cancel an analysis in progress in either the Manual Analysis Control or Automated Analysis Control windows. If you cancel an automated analysis, the Stopping an Analytical Sequence dialog appears.
New Calibration	Use this command to clear the existing calibrations from the Calibration Display window. You can generate a new calibration by analyzing the calibration standards.
Recall Calibration...	Use this command to recall a stored calibration from the results library. After you click on this command, select the results data set where the calibration is stored. If several sets of calibrations have been run, the most recent one will be recalled.

Analysis Menu Command	Description
Calibration Summary	When you are performing a Manual Analysis, use this command to view a summary of the calibration data. When you click on this command, a calibration summary is added to the information in the Results Display Window. (In an Automated Analysis, the calibration summary appears automatically in the Results Display Window after the last standard is analyzed, provided that this option is selected on the Options page of the Method Editor.)
Clear Calibration Blank	Use this command to clear the calibration blank concentrations from the analysis. If multiple calibration blanks have been analyzed, all calibration blank concentrations are cleared.
Clear Reagent Blank	Use this command to clear the existing reagent blank concentrations from the analysis. If multiple reagent blanks have been analyzed, all reagent blank concentrations are cleared.
Clear Results Display	Use this command to clear tabular analysis results from the Results Display window.
Clear Spectra Display	Use this command to clear spectral and text information from the Spectra Display window.
Manual Analysis ▶ Analyze Next Blank	After you have set up the Manual Analysis Control window, use this command to analyze the next blank. This is the same as clicking on **Analyze Blank** in the Manual Analysis Control window.

Analysis Menu Command	Description
Manual Analysis ▶ **Analyze Next Standard**	After you have set up the Manual Analysis Control window, use this command to analyze the next standard. This is the same as clicking on **Analyze Standard** in the Manual Analysis Control window.
Manual Analysis ▶ **Analyze Sample**	After you have set up the Manual Analysis Control window, use this command to analyze a sample. This is the same as clicking on the **Analyze Sample** in the Manual Analysis Control window.
Automated Analysis ▶ **Analyze All**	After you have set up the Automated Analysis Control window to start analyzing a sequence, use this command to begin analyzing the blanks, standards and samples. This is the same as clicking on **Analyze All** in the Automated Analysis Control window.
Automated Analysis ▶ **Calibrate**	After you have set up the Automated Analysis Control window to start analyzing a sequence, use this command to begin analyzing the blanks and standards. This is the same as clicking on **Calibrate** in the Automated Analysis Control window.
Automated Analysis ▶ **Analyze Samples**	After you have set up the Automated Analysis Control window to start analyzing a sequence, use this command to begin analyzing the samples. This is the same as clicking on **Analyze Samples** in the Automated Analysis Control window.

Analysis Menu Command	Description
Automated Analysis ▶ **Reset Sequence...**	Use this command after you have set up the Automated Analysis Control window, have interrupted the analysis and wish to reset the analysis to the beginning. Calibration curves are cleared when the analysis is restarted. This is the same as clicking on **Reset Sequence** in the Automated Analysis Control window.
Automated Analysis ▶ **Priority Sample...**	Use this command to add samples to the sample sequence while an automated analysis is in progress. When you click on this command, the Add Sample dialog appears in which you enter sample parameters and select when to analyze the sample. This command is the same as clicking on **Priority…** in the Automated Analysis Control window.
Automated Analysis ▶ **Append Samples to Analysis List...**	Use this command to append samples from your sample information file to the end of the run list while an automated analysis is in progress. In the Append Samples to Analysis List dialog that appears, enter the sample numbers from the sample information file (**do not** enter autosampler locations).
Autosampler ▶ **A/S Probe Up/Down**	Use this command to move the autosampler probe up or down, depending on its current position.
Autosampler ▶ **Go to Wash**	Use this command to go to the autosampler location of the wash solution. The location of the wash solution is selected in the method on the Sampler: Autosampler Wash page.

Analysis Menu Command	Description
Autosampler ▶ **Go to Location**	Use this command to select the location where the autosampler probe will move. When you click on this command, the Go To Location dialog appears.
Autosampler ▶ **Load Autosampler Tray**	Use this command to move the autosampler probe up and then to the back left position. This is the same as clicking on **Load Tray** in the Automated Analysis Control window.

Enable/Disable Elements Dialog

You use this dialog to include or exclude elements in the active method from an automated analysis, manual analysis or data reprocessing. This dialog applies only to single methods run from memory.
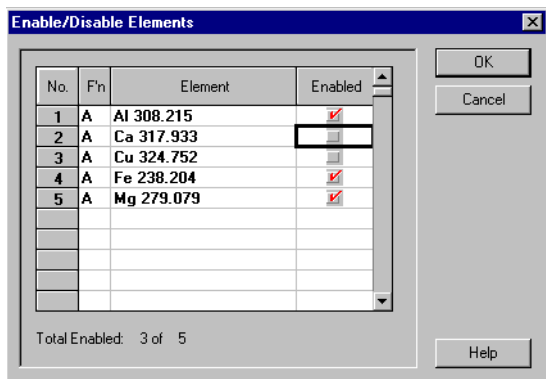
To display this dialog

► In the Analysis menu, click on **Enable/Disable Elements...**

Using this dialog

1. Review the list of elements and decide which to include or exclude. These elements are taken from the active method.

► To include an element in an analysis, click on the check box so that a check appears.

► To exclude an element from an analysis sequence, click on the check box so that the check is cleared.

Note The Enabled parameter has a column fill dialog that can be accessed by double-clicking in the column header.

2. Click on **OK**.

Note Elements will remain disabled until re-enabled using this dialog or until a new method is loaded.

Recall Calibration Dialog

You use this dialog to select stored calibration data for performing analyses. This is so that you do not have to calibrate each time you analyze samples.

To use this dialog

▶ Before opening this dialog, open a method that contains the elements and wavelengths that you want to use with the recalled calibration. Typically, this would be the method that you originally used to collect the calibration data. If you are not sure which method was used, select **Import from Results Library…** from the File menu, then select the results data set. If you want to use a different method, it must contain some (but not necessarily all) of the elements and wavelengths that were originally used when the calibration data was collected.

▶ In the Analysis menu, click on **Recall Calibration...**

Check the library location

The database library is where the calibration data is stored, as part of a results data set. The current library location is shown.

1. To change the library location, click on **Browse…**

2. In the Open dialog that appears, select the directory path for the library.

To select stored calibration data

In an analysis window, when the Save Data box next to During Analysis is selected, calibration data is automatically stored with results. When you display an analysis window and select a results data set, this box is selected automatically (an X appears).

▶ To recall a particular set of calibration data, click on the name of the results data set where the results were stored when the calibration was performed, then click on **OK.**

The recalled calibration will be used for the next manual or automated analysis, which you can now start by clicking on **Analyze Sample(s)** in the Manual or Automated Analysis Control window.

Note   In a situation where calibration was performed more than once for a results data set, the data for the last calibration performed are recalled.

Append Samples to Analysis List Dialog

Use this dialog to add samples to be analyzed at the end of an automated analysis.

To display this dialog

▶ In the Analysis menu, click on **Automated Analysis** ▶ **Append Samples to Analysis List** while an automated analysis is in progress.

To append samples to the end of an automated analysis

1. Before using this dialog, you need to add new samples to the sample information file.

2. In the **Analysis** menu, click on **Automated Analysis** ▶ **Append Samples to Analysis List** while the automated analysis is in progress. In the dialog, click on the entry field so the cursor appears. Type the sample numbers from the sample information file (for example **7,8,9** or **7-9**). Use commas to separate the locations and ranges.
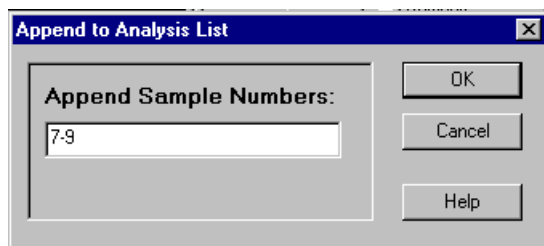
The new samples are added to the end of the run list in the Automated Analysis Control window.

Options Menu Commands

Options Menu Command	Description
General Preferences...	Use this command to set up the software with the desired toolbar icons and system monitors. See *General Preferences Dialog: View Page* later in this chapter for more information. In addition, use this command to select sounds to play at the end of certain tasks to signal completion or to signal the user's attention. See *General Preferences Dialog: Sounds Page* later in this chapter for more information.
Automatic Export...	Use this command if you want to automatically export data contained in a results data set and write it into a file that can be read by many other programs, including spreadsheet and database management programs. The Automatic Export dialog appears. The export file contains ASCII data records.
Sample Information Editor…	Use this command to select the exact parameters that you need to describe your samples, including sample description, preparation, and scheduling parameters, and add them to the Sample Information Editor. For more information, see the *Sample Information Parameters Dialog* in Chapter 4, *Sample Information Editor*.
Spectra Display...	Use this command to change the number of panes and the colors that appear in the Spectra Display window. You can also optimize colors for printing screens of this window in a 2-column by 3-row format. When you click on this command, the Spectra Display Options dialog appears.

Options Menu Command	Description
Calibration Display...	Use this command to change the number of panes and the colors that appear in the Calibration Display window. You can also optimize colors for printing screens of this window in a 2-column by 3-row format. When you click on this command, the Calibration Display Options dialog appears.
Examine Spectra/MSF…	Use this command to change the colors that appear in the Examine Spectra/MSF window. You can also optimize colors for printing screen captures. When you click on this command, the Examine Spectra/MSF Display Options dialog appears.
Pump...	Use this command to select options for the peristaltic pump. When you click on this command, the Configure Pump appears, where you make your selections.)
Autosampler...	Use this command to view the autosampler model and select tray type that you are using. (To change the autosampler model, exit the software and click on Start ► Programs ► PerkinElmer WinLab32 ► Reconfigure.)

General Preferences Dialog: Sounds Page

Users may elect to play sounds at various intervals or at the end of certain tasks to signal completion or to signal the user's attention. Sounds are included in the software and are located in the same drive in which the application was installed. In order to use the Sounds Page, you must have a sound card installed in your computer.

To display this dialog

▶ In the Options menu, click on **General Preferences**. Select the Sounds tab.

To select sounds

Note   You may use any Windows sound files with a .wav document extension or any .wav sound files that you might have installed.

1. Select **Play Sounds**, which activates the dialog choices.

2. Select from the following choices:

    End of Analysis
    This signals when an automated analysis is completed.

    End of Wave Calibration
    This signals when the wavelength calibration is completed.

    End of Align View
    This indicates the end of a torch view alignment.

    End of Detector Calibration
    This indicates when the detector calibration is completed.

    QC Failure
    If the Quality Control sample fails and the analysis is stopped, you can be alerted by a pre-selected sound.

    QC Paused
    If the Quality Control sample fails and you have selected to pause the system (using the Alarm & Pause failure action) you can be alerted by a pre-selected sound.

Hardware Error
If a hardware error occurs such as the plasma gases are turned off, you can be alerted by a pre-selected sound.

Plasma Extinguished
You will be alerted if the plasma is unexpectedly turned off (not a manual shut-off).

3. Click on **Browse…** to modify the selection of a sound file.

   Sounds are set to defaults and are interchangeable. For example, you may replace the End of Detector sound with that of the QC Failure.

4. After you have checked the appropriate sounds and their related .wav files, click on **Apply**.

5. Click on **OK** to close the dialog.

General Preferences Dialog: View Page

You may select viewing preferences for your screen that serve as short-cuts for certain purposes. These viewing icons are a quick alternative for often-used functions.

Viewing preferences that you may choose to appear on the screen include:

Toolbar
This displays commonly used icons across the top of your screen.

Method/Sample Info Bar
This displays the buttons that allow you to quickly modify both methods and sample information.

Status Bar
This displays the information line at the bottom of the screen.

System Monitors
The System Monitors allow you to monitor the status of the plasma, spectrometer, autosampler, and startup/shutdown activities. The four System Monitors are the Plasma Monitor, Spectrometer Monitor, Autosampler Monitor, and Startup/Shutdown Monitor.

Show Tool Tips
This displays tool tips, which appear when you place the mouse cursor on a WinLab32 feature such as a button, entry field, option, etc. When you do so, a one-line description, action, or other useful information relating to the feature selected appears. If you wish to turn off tool tips, click on this checkbox until the checkmark disappears.

To display this dialog

▶ In the Options menu, click on **General Preferences**. Select the View tab.

To select viewing options

1. Click on the check box for each item you wish to view.

2. After you have made your selections, click on **Apply**.

3. Click on **OK** to close the dialog.

Automatic Export Dialog

You use this dialog if you want the software to automatically export data contained in a results data set and write it into a file that can be read by many other third-party software packages, including spreadsheet and database management programs. The export file contains ASCII data records.

To display this dialog

▶ In the **Options** menu, click on **Automatic Export...**

-or-

▶ On the Automated Analysis control window click on Set... next to Auto Export.

To automatically reformat data:

Note You must first create the Export Design(s) in the Data Manager. An export design defines a subset of data items that you want to export from a data set. For more information, refer to Data Manager Help. When exporting data, you have the ability to select two of your designs as Export Design 1 and Export Design 2. You may want to do this if you want different information to be exported or you want the data to be exported to two separate file locations. Remember, in each Export Design created you assign the directory path where you want your results stored.

1. In the Export Design 1 entry field type the name of the design you created in the Data Manager or click on **Browse...** next to the entry field and when the dialog appears select the name of the design (*.xpt) and click on **OK**.

2. Repeat step 1 if an Export Design 2 was created in the WinLab32 Data Manager.

3. Click on **OK**. A checkmark appears in the Auto Export checkbox indicating that for each analysis you perform, data will be exported automatically. To stop exporting data in this manner, click in the checkbox to clear the checkmark.

Configure Pump Dialog

You use this dialog to select options for the peristaltic pump.

To display this dialog

▶ In the Options menu click on **Pump...**

Using this dialog

▶ Select the options that correspond to your instrument configuration.

Pump Settings

Pump Tubing

Select the tubing type from the drop down list, for example:

    black/black

    orange/red

    orange/blue, etc.

The pump tubing selected will determine the pump speed needed to achieve a given sample flow rate.

Pump Flush Rate

Enter the default flush speed. This is the pump speed that will be used when you click on the Flush button in the Plasma Control window. A sample flush is normally used to deliver sample from the sample vessel to the nebulizer at a faster rate than the normal sample flow rate. This decreases the total amount of time it takes to analyze each sample.

Enable Tubing Saver

Select this checkbox to use the Tubing Saver feature, which extends the lifespan of the peristalic pump tubing by operating the pump at a slower speed than normal.

Note This feature is not available for the Optima 3000 instruments.

Pump Type

Optima
Select this option to specify the Optima pump.

Gilson Minipulse
This option appears only when the software is configured for the Optima 3000 Family spectrometers. Select this option to specify the Gilson Minipulse pump.

Autosampler Configuration Dialog

You use this dialog to view the autosampler model and select the tray type that you are using.

To display this dialog

▶ In the Options menu, click on **Autosampler...**

Using this dialog

▶ Select the options that correspond to your autosampler configuration.

Autosampler Model

The autosampler model is set to its original configuration, which was set up when the software was installed.

You may not change the autosampler model in the Autosampler Configuration dialog. To change the model, you need to exit the software and run the Reconfigure utility.

To run the Reconfigure utility, click on **Start** ▶ **Programs** ▶ **PerkinElmer WinLab32** ▶ **Reconfigure.**

Autosampler Tray

▶ Click on Browse to select a different autosampler tray.

▶ Highlight the tray you are using and click on **Open** to select the tray. Different trays are available that will appear in this dialog depending upon the autosampler model you are using.

Turn peristaltic pump off while moving autosampler

Select this checkbox to turn off the pump while the autosampler probe arm moves horizontally between samples.

Autosampler Configuration Dialog AS-93

You use this dialog to view the autosampler model and select the tray type that you are using.

To display this dialog

1.   In the Options menu, click on Autosampler...

Using this dialog

Select the tray that corresponds to your autosampler configuration.

Autosampler Model

The autosampler model is set to its original configuration, which was set up when the software was installed.

To change the model, exit the software and run the Reconfigure utility. To run the Reconfigure utility, click on Start Programs PerkinElmer WinLab32 Reconfigure.

Autosampler Tray

1.   Click on Browse to select a different autosampler tray.

2.   In the dialog that appears, the trays are listed that can be used with the current autosampler configuration.

3.   Click on the tray you are using and then click on Open.

Rinse

The AS-93 autosamplers have a rinse location used to rinse the sample probe. If you want to change the rinse solution continuously, select one of the options where the pump is switched on. Use a higher pump speed to change the rinse solution more quickly.

Window Menu Commands

You use the Window menu to rearrange open windows or icons on the application window.

Window Menu Command	Description
Cascade	Use this command to resize and overlap open windows so that each title bar is visible.
Tile Horizontal	Use this command to resize and arrange open windows horizontally across the screen.
Tile Vertical	Use this command to resize and arrange open windows vertically down the screen.
Arrange Icons	Use this command to bring icons of minimized windows to the lower left-hand corner of the screen.
Close All Windows	Use this command to close all of the open windows in the application.

Help Menu Commands

Help Menu Command	Description
Tip of the Day…	Use this command to display tips about using the software when starting the software.
Active Window Help	Use this command to display help information about the window that is currently active. Shortcut: **F1.**
Contents and Index	Use this command to access a table of contents and the index for online Help.
How to Use Help	Use this command to access information that explains how to use online Help.
About WinLab32...	Use this command to view information about the software. In the dialog that appears, click on **Details** to view current version numbers for the main application, the base modules, and the firmware.

Calibration and Calculations 12

Calibration

Linear Calibration Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is linear and that the following equation describes this relationship:

$Y = MX + B$

where:

    X = concentration

    Y = intensity

    M = slope of the calibration curve

    B = y-axis intercept

Given 2 or more data points, the values for M and B are calculated using the following equations [1, 2].

    n = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

$$M = \frac{(n)\sum_{n}^{i=1}(X_i Y_i) - \sum_{n}^{i=1}(X_i)\sum_{n}^{i=1}(Y_i)}{(n)\sum_{n}^{i=1}(X_i^2) - (\sum_{n}^{i=1} X_i)^2}$$

$$B = \frac{\sum_{n}^{i=1}(X_i^2)\sum_{n}^{i=1}(Y_i) - \sum_{n}^{i=1}(X_i Y_i)\sum_{n}^{i=1}(X_i)}{(n)\sum_{n}^{i=1}(X_i^2) - (\sum_{n}^{i=1} X_i)^2}$$

An example of this calculation is described below:

The WinLab Report shows the following information for ZN 206.200:

Mean Data: Calib Blank 1

Analyte	Mean Corrected Intensity	St. Dev.	RSD	Conc.	Calib. Units
Zn 206.200	-2.6	0.00	0.00%	[0.00]	mg/L

Calibration Summary

Analyte	Stds.	Equation	Intercept	Slope	Curvature	Corr. Coef.
Zn 206.200	1	Lin, Calc Int	0.0	5.780	0.00000	1.000000

Replicate Data: QC1

Repl#	Analyte	Net Intensity	Corrected Intensity	Conc.	Calib Units
1	Zn 206.200	30210.0	30212.6	5227	mg/L

Where:

Intensity is calculated as: $5227 * 5.780 + 0 = 30212.06$ using the equation $Y = MX + B$.

The intensity of the blank is then subtracted to get the Net Intensity which is calculated as: $30212.6 - 2.6 = 30209.46$

Please note that the numbers derived from the calculation above may differ slightly from the intensity value displayed in the WinLab report, because WinLab calculates Intensity values using additional significant figures for the Concentration value; these additional significant figures are not displayed on the printed WinLab report.

Linear Forced through Zero

A calibration curve defined using this equation is forced to go through zero intensity and zero concentration.

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is linear and that the following equation describes this relationship:

$Y = MX$

where:

X = concentration

Y = intensity

M = slope of the calibration curve

The intercept is forced to be zero.

Given 2 or more data points, the values for M are calculated using the following equation.

n = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

$$M = \frac{\sum_{n}^{i=1}(X_i Y_i)}{\sum_{n}^{i=1}(X_i^2)}$$

Linear Bracketing Calibration

The bracketing standards calibration option is applicable over a restricted concentration range. The calibration is performed with at least two calibration standards that bracket the concentration range of interest.

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is linear and that the following equation describes this relationship:

$$Y = MX + B$$

where:

X = concentration

Y = intensity

M = slope of the calibration curve

B = y-axis intercept

Given 2 or more data points, the values for M and B are calculated using the following equations [1, 2].

n = number of standards (includes the blank)

The use of a blank is optional. If a blank is not used, a zero intensity is assumed. In this equation, the blank is subtracted from all solutions, but is not included in the calculation of the calibration curve.

$$M = \frac{(n)\sum_{n}^{i=1}(X_i\,Y_i) - \sum_{n}^{i=1}(X_i)\sum_{n}^{i=1}(Y_i)}{(n)\sum_{n}^{i=1}(X_i^2) - (\sum_{n}^{i=1}X_i)^2}$$

$$B = \frac{\sum_{n}^{i=1}(X_i^2)\sum_{n}^{i=1}(Y_i) - \sum_{n}^{i=1}(X_i\,Y_i)\sum_{n}^{i=1}(X_i)}{(n)\sum_{n}^{i=1}(X_i^2) - (\sum_{n}^{i=1}X_i)^2}$$

Non-Linear Calibration Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is a curvilinear one that can be described by the following polynomial equation:

$$Y = AX^2 + BX + C$$

X = concentration

Y = intensity

A = curvature

B = slope

C = y-axis intercept

Given 3 or more data points, the values for A, B and C are calculated using the following equations [1, 2, 3].

n = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

$$A = \frac{\left[\dfrac{a_2}{a_0} - \dfrac{a_5}{a_3} \right]}{\left[\dfrac{a_1}{a_0} - \dfrac{a_4}{a_3} \right]}$$

$$B = \frac{a_5}{a_3} - \frac{(A \times a_4)}{a_3}$$

$$C = \frac{c_2}{n} - \frac{(B \times c_0)}{n} - \frac{(A \times c_1)}{n}$$

Where:

$$a_0 = \frac{c_0}{n} - \frac{c_1}{c_0}$$

$$a_1 = \frac{c_1}{n} - \frac{c_3}{c_0}$$

$$a_2 = \frac{c_2}{n} - \frac{c_4}{c_0}$$

$$a_3 = \frac{c_1}{c_0} - \frac{c_3}{c_1}$$

$$a_4 = \frac{c_3}{c_0} - \frac{c_5}{c_1}$$

$$a_5 = \frac{c_4}{c_0} - \frac{c_6}{c_1}$$

$$c_0 = \sum_{n}^{i=1} (X_i)$$

$$c_1 = \sum_{n}^{i=1} (X_i^2)$$

$$c_2 = \sum_{n}^{i=1} (Y_i)$$

$$c_3 = \sum_{n}^{i=1} (X_i^3)$$

$$c_4 = \sum_{n}^{i=1} (X_i Y_{ii})$$

$$c_5 = \sum_{n}^{i=1} (X_i^4)$$

$$c_6 = \sum_{n}^{i=1} (X_i^2 Y_{ii})$$

Non-Linear Bracketing Algorithm

This calibration curve is established by assuming that the relationship between concentration (the X values) and intensity (the Y values) is a curvilinear one that can be described by the following polynomial equation:

$$Y = AX^2 + BX + C$$

X = concentration

Y = intensity

A = curvature

B = slope

C = y-axis intercept

Given 3 or more data points, the values for A, B and C are calculated using the following equations [1, 2, 3].

n = number of standards (includes the blank)

In this equation, the blank is subtracted from all solutions and included in the calculation of the calibration curve.

The use of a blank is optional. If a blank is not used, a zero intensity is assumed. In this equation, the blank is subtracted from all solutions, but is not included in the calculation of the calibration curve.

$$A = \frac{\left[\dfrac{a_2}{a_0} - \dfrac{a_5}{a_3}\right]}{\left[\dfrac{a_1}{a_0} - \dfrac{a_4}{a_3}\right]}$$

$$B = \frac{a_5}{a_3} - \frac{(A \times a_4)}{a_3}$$

$$C = \frac{c_2}{n} - \frac{(B \times c_0)}{n} - \frac{(A \times c_1)}{n}$$

Where:

$$a_0 = \frac{c_0}{n} - \frac{c_1}{c_0}$$

$$a_1 = \frac{c_1}{n} - \frac{c_3}{c_0}$$

$$a_2 = \frac{c_2}{n} - \frac{c_4}{c_0}$$

$$a_3 = \frac{c_1}{c_0} - \frac{c_3}{c_1}$$

$$a_4 = \frac{c_3}{c_0} - \frac{c_5}{c_1}$$

$$a_5 = \frac{c_4}{c_0} - \frac{c_6}{c_1}$$

$$c_0 = \sum_{n}^{i=1} (X_i)$$

$$c_1 = \sum_{n}^{i=1} (X_i^2)$$

$$c_2 = \sum_{n}^{i=1} (Y_i)$$

$$c_3 = \sum_{n}^{i=1} (X_i^3)$$

$$c_4 = \sum_{n}^{i=1} (X_i Y_{ii})$$

$$c_5 = \sum_{n}^{i=1} (X_i^4)$$

$$c_6 = \sum_{n}^{i=1} (X_i^2 Y_{ii})$$

Correlation Coefficient

Correlation coefficients are calculated in WinLab32 using three different formulas, based on the current calibration equation.

Note The correlation coefficient is the same as Pearson's "r" value.

Linear Calculated Intercept / Linear Bracketing

$$
r = \frac{n \sum_{i=1}^{n} x_i y_i - \sum_{i=1}^{n} x_i \sum_{i=1}^{n} y_i}{\sqrt{n \sum_{i=1}^{n} x_i^2 - \left(\sum_{i=1}^{n} x_i \right)^2} \cdot \sqrt{n \sum_{i=1}^{n} y_i^2 - \left(\sum_{i=1}^{n} y_i \right)^2}}
$$

where:

X = concentration

Y = intensity

n = number of standards+1

Based only on concentration (intensity) points, not the calculated curve.

Coefficient may be 1.0 even if points are not on the curve.

For Linear Calculated Intercept, the blank (0,0) point is included, which increases the number of standards (n) by 1.

Linear Zero Intercept

$$r = \frac{\sum_{i-1}^{n} x_i y_i}{\sqrt{\sum_{i-1}^{n} x_i{}^2 \cdot \sum_{i-1}^{n} y_i{}^2}}$$

where:

X = concentration

Y = intensity

n = number of standards

Based only on concentration (intensity) points, not the calculated curve

Coefficient may be 1.0 even if points are not on the curve

Nonlinear (Calculated Intercept / Zero Intercept / Bracketing)

$$r = \sqrt{1.0 - \frac{\sum_{i-1}^{n}(x_i - y_i)^2}{\sum_{i-1}^{n} x_i{}^2 - \frac{\left(\sum_{i-1}^{n} x_i\right)^2}{n}}}$$

where:

X = concentration (entered by user)

Y = concentration (measured from calibration curve)

n = number of standards

Based on actual and measured concentrations (the delta between the user-entered value and the calculated curve).

Will work for linear or non-linear curves

Coefficient will be 1.0 only if all points are on the curve.

For Nonlinear Calculated Intercept, the blank (0,0) point is included, which increases the number of standards (n) by 1.

Method of Additions Sample Intercept

Use the analyte addition technique when the matrix causes an interference that varies from sample to sample.

The calibration curve is forced to go through the point defined by the sample with no addition added, and in the other the sample with no addition is treated as just another point by the least squares routine.

The equation used for the sample intercept option is:

$$C = -K_1 I$$

C is the concentration added to an aliquot of sample and I is the difference between the intensity for the aliquot with added standard and the intensity measured for the sample. The final sample concentration is calculated by multiplying the slope ($-K_1$) times the intensity of the sample.

Method of Additions Calculated Intercept

A least squares technique is used to determine the K1 coefficient when two or more standards are used for calibration. This calibration curve is not forced to go through the point defined by the unspiked sample and the intercept with the intensity axis is calculated.

The equation used for the calculated intercept option is:

$$I = K_1 + K_2 C$$

The sample with no addition is treated as just another point by the least squares routine that determines the K1 and K2 coefficients.

Method of Additions Calibrate

The system uses method of additions, with the zero intercept equation option for the first sample then analyzes subsequent samples using the calibration curve generated for the first sample. This is appropriate when the matrix causes an interference that is the same for all the samples.

This calibration curve is forced to go through the point defined by the unspiked sample.

Calculations

Standard Deviation and Precision

The equation used to calculate Standard Deviation (SD) is as follows:

$$SD = \sqrt{\frac{\sum(c_i - c_{mean})^2}{n-1}}$$

where:

c_i is each replicate measurement

c_{mean} is the mean value

n   is the number of measurements

The equation used to calculate precision or the Relative Standard Deviation (RSD) is:

$$RSD(\%) = 100 * \frac{SD}{mean}$$

Reslope

The reslope feature of the software allows you to make small corrections to the calibration curve using a single calibration standard following small changes in instrumental conditions. You might also want to use a reslope standard with a matrix similar to your samples, for example Standard Reference Materials from the United States National Institute of Standards and Technology, to compensate for small matrix effects after you have performed the initial calibration with aqueous calibration standards. When a reslope recalibration is performed, the slope coefficient is adjusted to make the concentration calculated by the equation equal to the actual concentration of the reslope standard. A reslope standard near the middle of the calibration range will usually give the best results.

A single reslope standard, however, will not adequately compensate for major changes in instrument parameters. You should do a complete recalibration following parameter changes that are likely to alter the shape of the calibration curve, such as instrument drift, changes in plasma conditions, or large matrix differences.

Internal Standardization

When performing a calibration in ICP, the sample intensity is expressed as a function of concentration:

$$I_{Sample} = f\,(\,C\,) \qquad [1]$$

When using internal standards, the sample intensity is divided by the intensity of the internal standard line as follows:

$$\frac{I_{Sample}}{I_{Internal\ Standard}} = f\,(\,C\,) \qquad [2]$$

In addition, the WinLab32 software uses an internal standard initial intensity value (IS Initial value). The first time the internal standard intensity is measured, the value is remembered and added to equations 1 and 2 as follows:

$$\left(\frac{I_{Sample}}{I_{Internal\ Standard}} I_{IS\ Initial\ Value} \right) = f\,(\,C\,) \qquad [3]$$

By looking at the corrected analyte intensity value and comparing this with the raw value, you can see how much the intensity has drifted.

The internal standard initial intensity value (IS Initial value) is stored with the calibration curve.

The following information gives more details about how the IS Initial value is handled in the WinLab32 software.

- The IS Initial value is determined during measurement of the first solution that contains the internal standard(s). If the calibration blanks contain internal standard, then the IS Initial Value is measured as part of the blank. If the internal standard(s) are not in the blank, then the IS Initial Value is measured in the first calibration standard.

- If the calibration curve is cleared, the IS Initial value is also cleared. This can occur under two circumstances:

  by selecting the New Calibration command from the Analysis menu, or

  by loading a method that does not also load a stored calibration curve.

- The IS Initial value is stored with calibration curves and recalled when the calibration curve is recalled.

- The IS Init value is not reinitialized when any type of recalibration occurs.

- Results that have been internal standard corrected are marked with a footnote symbol. The † symbol is placed just after the analyte name.

- For data reprocessing the IS Initial value is cleared when the calibration curve is cleared exactly the same as for normal sample analyses. Generally, you should clear the calibration curve before reprocessing data; if existing calibration curves are not cleared, then any calibration operations, even if at the beginning of the samples to be reprocessed, would be treated as recalibrations and the IS Initial value would not be reinitialized.

Weighted Linear Calibration

When working at low level concentrations it provides an alternative calibration scheme that weighs the low standards to a greater degree.

Determine the slope, intercept, and correlation coefficient for the equation:

$$y = b_0 + b_1 x$$

where x is the measured net corrected intensity (blank subtracted). The weights are applied by multiplying the intensity by the weighting factor for each standard. In this calibration the weighting factor is the reciprocal of the square of the user entered concentration value for each standard.

Where:

x is the Concentration value of the standard

y is the measured intensity of the standard

n is the number of standards

i is the index for the standards

b0 is the intercept

b1 is the slope

C is the correlation coefficient

$$w_1 = \sum \frac{1}{x_i^2} \qquad\qquad w_4 = \sum \frac{y_i}{x_i^2}$$

$$w_2 = \sum \frac{x_i}{x_i^2} \qquad\qquad w_5 = \sum \frac{y_i^2}{x_i^2}$$

$$w_3 = \sum \frac{x_i^2}{x_i^2} = n \qquad\qquad w_6 = \sum \frac{x_i y_i}{x_i^2}$$

Intercept:
$$b_0 = \frac{(w_4 w_3) - (w_6 w_2)}{(w_1 w_3) - w_2^2}$$

Slope:
$$b_1 = \frac{(w_1 w_6) - (w_4 w_2)}{(w_1 w_3) - w_2^2}$$

Correlation Coefficient:
$$C = \frac{(w_1 w_6) - (w_2 w_4)}{\sqrt{\left[(w_1 w_3) - w_2^2\right]\left[(w_1 w_5) - w_4^2\right]}}$$

Glossary

A

Analyte. The element whose concentration is determined in an analysis.

Array detector. A silicon chip that is manufactured with a one or two-dimensional array of photosensitive material on its surface. The photons that are absorbed by the units (*pixels*) of the array are converted to electrons which are collected and read out. Examples include Photodiode Arrays (PDAs), Charge-coupled Devices (*CCDs*), Charge-injection devices (CIDs) and Segmented-array Charge-coupled Devices (*SCDs*).

Aspiration. A high speed gas flow is directed across an open tube to draw solution into the nebulizer.

Auxiliary flow. The flow of argon gas that is contained between the intermediate and the injector tubes of the ICP torch.

B

Background correction. Process of correcting the intensity recorded for a particular wavelength for intensity due to the ICP continuum background and other *spectral interferences.* This is normally done by choosing one or two points at one or both sides of the peak you want to correct. Points can be chosen manually. The software then measures the intensity at these points, interpolates an intensity at the peak wavelength, and then subtracts that intensity from the intensity recorded at the peak.

BEC test. Background Equivalent Concentration test. A test of instrument sensitivity. The concentration of a solution whose intensity is equivalent to the intensity of the background at the same wavelength is determined. The lower the concentration of this solution, the more sensitive the instrument for that element.

Blank (solution). A solution that contains none (or rather, an undetectable amount) of the analyte you are interested in. Used for a zero point in calibration.

C

Calibration curve. The plot or equation showing intensity versus concentration for an *analyte*. Once this is established, the concentration of any solution containing this analyte within the concentration range of the calibration can be found.

Calibration equation. Mathematical function used to convert a sample's measured emission intensity into a concentration reading. *See also* Curve Type.

CCD. Charge-coupled Device. A two-dimensional array detector that uses a charge-coupled readout of the photoelectrons generated by light absorption. This type of detector is used in PerkinElmer scanning CCD ICP spectrometers. *See also* Array detector, SCD.

Correlation coefficient. Indicates the quality of the fit between the calibration curve and the points it is fitting. A value of 1.000 indicates a perfect fit.

Curve type. Describes the calibration curve made using a particular *calibration equation.* Using different curve types for the same standard and blank measurements may give different analytical results.

CV test. Coefficient of Variation test. *See* Relative Standard Deviation (RSD).

D

Default settings. The instrument software contains standard settings which are the most generally used settings. There are default settings for aqueous and organic analyses that are available to use as a starting point when creating a new method.

Detection limit. The lowest concentration of an *analyte* whose presence can be determined with a specified degree of certainty. Often defined as the concentration of an analyte that results in signal intensity that is three times (or twice) the *standard deviation* of the background intensity at the wavelength measured.

Detector. A photosensitive device that absorbs photons and converts them into electrons with a certain efficiency. These photoelectrons can be measured to indicate the intensity of the incoming light, that is, the number of photons from the emission source. *See also* Array detector, CCD, and SCD.

Difference spectrum. Spectrum that results from subtracting one spectrum from another.

E

Echelle grating. Coarsely ruled diffraction grating used in multiple orders to achieve high spectral *resolution.*

Excitation characteristics. The amount of energy required to promote an electron to a higher energy level in an atom or ion. This excited state is necessary for the atom or ion to emit light. Different elements have different excitation characteristics.

F

Firmware. Programmable silicon chips that form an interface between the computer software and the instrument hardware that it controls. Firmware has a restricted set of commands but operates extremely fast, thus allowing the software to be simpler and speed up instrument response.

G

GPIB. General-Purpose Interface Bus. A commonly used protocol (agreement) on how computers and electronic devices should communicate. The computer uses a GPIB card to allow it to communicate with the instrument *firmware.*

I

IEC. *See* Interelement Correction.

IEEE-488. Common interface protocol for communications between computers and other electrical devices.

Integration time. The time for which the *detector* absorbs photons from the plasma before a signal is read out. The integration time multiplied by the number of *reads* specified equals the *read time*.

Interelement Correction (IEC). A spectral interference correction technique in which emission contributions from interfering elements that emit at the analyte wavelength are subtracted from the apparent analyte emission after measuring the interfering element at another wavelength.

Intercept. Where two lines meet. Commonly used in spectroscopy in calibration work, for example, the y-intercept, meaning the point where the *calibration curve* cuts the x= 0 axis of the calibration plot.

Interference. Anything that causes the *analyte* signal in a sample to differ from the analyte signal in a calibration solution at the same concentration. *See also* Spectral interference.

Internal reference. IUPAC name for *internal standard.*

Internal standard. A nonanalyte element, present in all standards, blanks, and samples, that is used to correct for physical or matrix *interferences* in an analysis.

L

Linear fit. Type of *calibration curve* where a straight line is drawn through the standard and blank emission/concentration values.

M

Matrix. The components of the sample other than the elements of interest. The matrix can substantially change results obtained, through *spectral interference* or viscosity change.

Mercury lamp. A lamp inside PerkinElmer simultaneous ICP spectrometers that emits light at a mercury wavelength. This is periodically used to return the instrument to the current wavelength calibration.

Method. Controls instrument settings and how the instrument acquires and processes data in an analysis.

MSF. *See* Multicomponent Spectral Fitting.

Multicomponent Spectral Fitting (MSF). A processing mode that distinguishes analyte spectra from interfering spectra by the use of stored mathematical models. It uses a multiple linear regression technique to fit the models created to unknown spectra during an analysis. Since only the peak shapes need to remain constant, typically, the models are independent of concentration, plasma conditions, and matrix effects.

> Sources of information on MSF include the following two articles:
>
> Ivaldi, Juan C. et al., "Multivariate methods for interpretation of emission spectra from the inductively coupled plasma," *Spectrochimica Acta*, Vol. 47 B, No. 12, pp. 1361-1371, 1992.
>
> Ivaldi, Juan C. and Barnard, Thomas W., "Advantages of coupling multivariate data reduction techniques with simultaneous inductively coupled plasma optical emission spectra," *Spectrochimica Acta*, Vol. 48 B, No. 10, pp. 1265-1273, 1993.

N

Nebulizer Flow. The flow of argon gas that enters the nebulizer and carries the sample in the form of an aerosol into the plasma.

Neon Correction. A neon reference spectrum is measured for use in wavelength correction on PerkinElmer scanning CCD ICP spectrometers.

O

Optimizing. The process of determining the best conditions and settings for an analysis.

Outer Flow. IUPAC term for *plasma flow*.

P

Peak algorithm. The way in which the emission data from the spectrometer is converted into emission counts. This can be done in three ways: (1) by searching for a peak and estimating its height (*Peak height processing);* (2) by summing the output of several detector pixels (*Peak area processing*); or (3) by treating the output mathematically and relating it to existing spectral models (*Multicomponent Spectral Fit processing*).

Peak area processing. A processing mode that sums the intensities recorded by specified *pixels* on a given *subarray* for an element.

Peak height processing. A mode of processing emission data where the highest intensity points are searched for, and then an algorithm is applied to approximate the peak height. *See also* Area processing and Multicomponent Spectral Fitting.

Pixel. Photosensitive unit of an *array detector* whose output can be read individually.

Polychromator. A type of spectrometer that can detect and process data for two or more wavelengths at the same time.

Pre-shot. A quick "snapshot" of the plasma emission which is made by the spectrometer when Auto Integration is selected. This is used to determine optimum integration times.

Precision. A measure of how reproducible a set of measurements is. Commonly used statistical measurements are *standard deviation (SD)* and *relative standard deviation (RSD),* also called *coefficient of variation (CV).*

R

Read time. The amount of time per *replicate* for which the *detector* measures light from the plasma for the current sample. The total read time equals the *integration time* multiplied by the number of integrations specified.

Relative Standard Deviation (RSD). The *standard deviation* of a group of measurements divided by the mean of those measurements. A test used to measure relative precision for a group of measurements. Often expressed as a percent. Also known as Coefficient of Variation (CV) test.

Replicate. A sample can have one or more replicates, meaning that there will be repetitive measurements taken.

Reslope. When there are small changes in instrumental conditions, the reslope feature of the software allows you to compensate by making small corrections to the calibration curve using a single calibration standard.

Resolution. The measure of a spectrometer's ability to separate adjacent wavelengths. The High setting for resolution maximizes the resolving power on PerkinElmer simultaneous ICP spectrometers.

RF Power. The amount of energy per unit time transmitted from the RF generator to the ICP discharge. Measured in watts.

S

Sample uptake rate. IUPAC term for *sample flow rate.*

Scanning CCD ICP Spectrometer. A spectrometer that detects and processes data for one wavelength at a time.

Simultaneous ICP Spectrometer. A spectrometer that detects and processes data for two or more wavelengths at the same time.

SCD. Segmented-array Charge-coupled device Detector. A specialized type of array detector (developed for and used in PerkinElmer simultaneous ICP spectrometers) that has groups of photosensitive pixels, called *subarrays*, on the detector surface where important atomic emission lines appear. The SCD uses charge-coupled readout like a CCD, but the subarrays can be individually addressed. *See also* Array Detector, Detector, CCD.

Slit Steps. The number of positions used by the spectrometer entrance slit during data collection for each replicate on simultaneous ICP spectrometers. The slit can be moved under software control. When the spectral profiling is set to off, the spectrometer uses one slit step (entrance slit position); when spectral profiling is on, the spectrometer uses four.

Shutter. A mechanical device used to block the plasma emission from entering the spectrometer.

Spectral interference. An interference caused by the emission from elements or compounds other than those of interest.

Spectral profiling. On PerkinElmer simultaneous ICP spectrometers, the spectrometer entrance slit is adjusted by ¼ pixel steps during data collection, thus moving the slit image across the detector *subarrays.* This increases the data density across the spectral peaks and produces smoothed spectral displays.

Split-pixel. Reading a detector *array* so that information is taken from each half-pixel rather than from each *pixel*.

Standard. A solution with accurately known concentrations of the elements to be determined in an analysis. Two or more standards are used to make a *calibration curve.*

Standard deviation. A statistical definition of the variation in a group of similar measurements.

Structured background. Spectral interference that is non-random, thereby altering analytical results for nearby analyte lines.

Subarray. On PerkinElmer simultaneous ICP spectrometers, this is a group of *pixels* on an *SCD* which are positioned to measure a small, specific wavelength range.

T-Z

Weighted linear fit. A *curve type* where the importance given to a calibration point in the fitting process (the weighting) is directly related to the concentration of the standard measured for that point.

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