Agilent 7700 Series ICP-MS MassHunter Workstation

Quick Start Guide

This guide describes how to use the Agilent 7700 Series ICP-MS MassHunter Workstation.

Agilent Technologies

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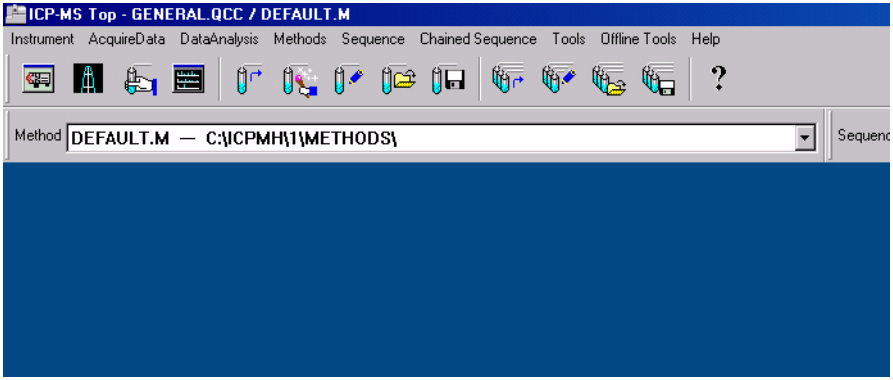
Startup

To start the Agilent 7700 Series ICP-MS MassHunter Workstation software:

- Double-click the ICP MS Top desktop icon  , *or*
- Select **Programs > ICP-MS MassHunter Workstation > ICP-MS Top** from the Windows Start menu.

The ICP-MS Top window opens:



Toolbar

The following shortcuts are available on the ICP-MS MassHunter Workstation toolbar:

Button	Action	Button	Action
	Display Instrument Status Panel		Display Data Acquisition Panel
	Display Tuning Panel		Display Data Analysis Panel
	Run Current Method		Run Current Sequence
	Run Method Wizard		Edit Sequence
	Edit Method		Load Sequence
	Load Method		Save Sequence
	Save Method		Display online help window

Preparing for Analysis

The following topics will help you prepare the instrument for analysis with Agilent 7700 Series ICP-MS MassHunter Workstation.

Things to check before analysis

Utilities

- Argon gas pressure: 500 to 700 kPa
- Cell gas (Helium): 90 to 130 kPa
- Cell gas (Hydrogen): 20 to 60 kPa
- Exhaust duct (on)
- Cooling water (Chiller or heat exchanger on)
- Drain and rinse tank (not full)

Peristaltic pump tubing

- Sample, drain, and internal standard lines

Prepare tuning solutions

You will need the following standards and solutions for ICP-MS operation:

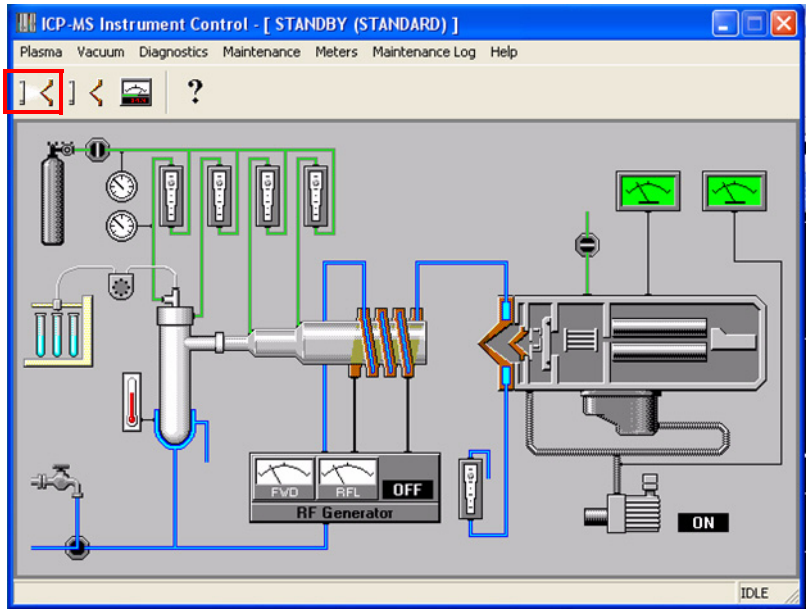
- Tuning solution (1ppb Li, Co, Y, Ce, Tl)
- Internal standard solution
- Dilute the P/A factor tune solutions 75x, then mix the two solutions 1:1 to prepare a P/A factor tune solution for use in the lab. PA factor tuning is used to achieve linear functionality across the detector's pulse and analog modes. Pulse mode is used to quantitate relatively low concentrations (ppb range). Analog mode is used to quantitate relatively high concentrations (ppm range).

Ignite the plasma

1 Open the Instrument Control panel in *either* of the following ways:

- Select **Instrument Control** from the Instrument menu, *or*
- Click the Instrument Control icon.

The ICP-MS Instrument Control panel opens:



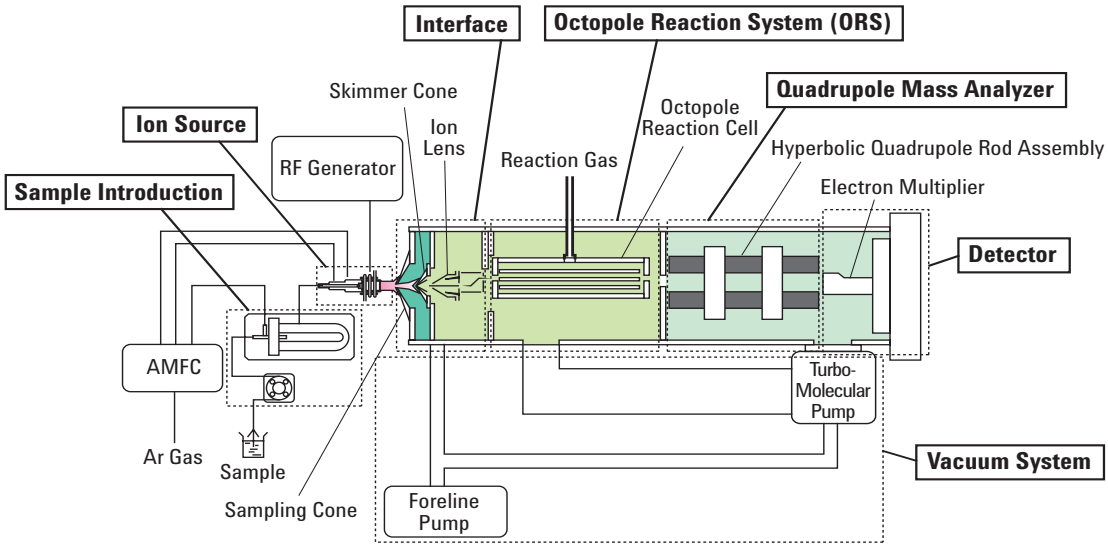
2 Confirm that the instrument is in Standby mode by checking the title bar of the Instrument Control panel.

3 Ignite the plasma by clicking the Ignite Plasma icon in the toolbar.

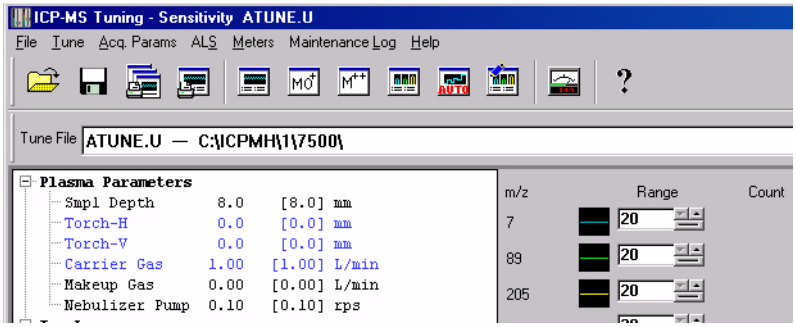
4 Once the plasma is on and the instrument goes from standby to analysis mode, check tuning as described in the following section.

Check tuning

The following diagram labels the parts of the ICP-MS instrument. You can refer to this diagram during the discussion of tuning parameters in this section.



Open the Tuning panel by selecting **Tune** from the Instrument menu, or by clicking the Tune icon on the ICP-Top toolbar.



Toolbar The following items (shortcuts) are available on the Tuning panel toolbar:

Button	Action	Button	Action
	Load tuning parameters from file		Save tuning parameters to file
	Generating tuning report		Print current graph
	Tune sensitivity		Tune Oxide Ion
	Tune Doubly Charged Ion		Resolution and Axis
	Select Mode & start Autotune		Set Acquisition parameters for tuning
	View meter control panel		Display online help window

Tip See the *"Tuning 7700"* in online help for more information on individual tuning parameters.

Plasma Correction

Execution of plasma correction is necessary in the following cases:

- When the instrument is installed
- When the nebulizer has been changed
  (However, if the same nebulizer unit is reused repeatedly by detaching and reattaching, it is not necessary to conduct the correction each time as long as the nebulizer is installed correctly.)
- When the sampling cone is changed
- When the torch is changed
- When the matrix tolerance has changed after the instrument has been used for 6-12 months and normal maintenance procedures have been followed

  Once plasma correction is performed, there is no need to conduct it again in most cases, provided the instrument is used for normal measurement.

Operating Method

When performing plasma adjustment, use a 1pbb tuning solution containing Ce. The following explains the method, refer to the online help.

Settings for Preset Mode

Select the nebulizer and tuning type, and set to the Preset mode. Under the Preset mode, parameters such as RF power, sampling position, carrier gas flow rate, makeup gas flow rate, and nebulizer pump speed are fixed to the preset values.

1 In the ***Tuning*** window, select ***Preset Plasma*** >> ***Select Plasma***.

The ***Plasma Select*** dialog appears.

2 Check the ***Preset Mode*** box.

3 Select the ***type of nebulizer*** to be used from the drop-down menu (For 7700x).

4 Select ***Robust*** or one of the 3 levels of ***Ultra Robust*** (For 7700x). Select ***Hot Plasma*** (For 7700s).

If the matrix concentration exceeds 1%, select ***Ultra Robust-Level: High*** (if the desired sensitivity is not achieved, select Level ***Medium*** or ***Low***).

5 Click ***OK***.

The ***Tuning*** window becomes active.

Checking the Tuning

Check the tuning as follows:

1 Check the each parameters:

- Open the last-used tuning file (typically **nogas.u**, **h2.u** or **he.u**).
- Select **Acquisition Parameters** from the Acq. Params menu. Set the mass numbers to be measured as 7 (Li), 89 (Y), 205 (Tl), and 156/140 (Ce oxide formation rate) and click **OK**.
- Check the following values displayed on the Tuning window:

Table 1 Typical Sensitivity Values (For 7700x)

Mass	Sensitivity (cps/ppb)		
	No Gas Mode	He Gas Mode (3.6ml/min)	H_2 Gas Mode (4.0ml/min)
^{7}Li	30,000	---	---
^{59}Co	---	24,000	---
^{89}Y	100,000	---	60,000
^{205}Tl	60,000	---	---
Oxide ratio ≤ 1.5% (≤2% for MicroFlow Nebulizer)			

2 If the desired sensitivity is not achieved, select **Autotune** from Tume menu. Refer to the Help and execute Autotune.

3 After completing autotune, check the sensitivity parameters on the ICP-MS Tuning - Sensitivity window.

4 Save the tune file by selecting **Save** from the File menu. In general you will want to overwrite the tune file that was open, so that it has the updated values.

5 (*optional*) Generate a tune report by selecting **Generate Report** from the File menu. Enter optional comments if desired and click **OK**. A report showing the following information is sent to your printer:

- Sensitivity data
- Resolution/Axis data
- Lens parameter settings

Depending on how your system is configured, tuning results will also be recorded in the maintenance log.

Tip You can also get a hard copy of current tune parameters by clicking the **Stop** button and selecting **Print** from the File menu.

Reference: For 7700x

The recommended tuning parameter values for 7700x are shown below.

Table 2 Recommended Values for 7700x (When Using a MicroMist Nebulizer)

Parameter	No Gas Mode		Cell Gas Mode		High Energy Collision Mode *2	
	Recommended Value	Recommended Range	Recommended Value	Recommended Range	Recommended Value	Recommended Range
RF Power [W]	1550	Fixed	1550	Fixed	1550	Fixed
Smpl Depth [mm]	8.0	Fixed	8.0	Fixed	8.0	Fixed
Carrier Gas [L/min]	1.05	1.01 to 1.11	1.05	1.01 to 1.11	1.05	1.01 to 1.11
Makeup Gas [L/min]	0	0 to 1.11	0	0 to 1.11	0	0 to 1.11
Dilution Gas [L/min]	0	Fixed	0	Fixed	0	Fixed
Neb Pump [rps]	0.1	Fixed	0.1	Fixed	0.1	Fixed
S/C Temp [degC]	2	Fixed	2	Fixed	2	Fixed
He or H_2 gas [ml/min]	0	Fixed	3.6 (He) 4.0 (H_2)	3.2 to 4.0 (He) 3.6 to 4.4 (H_2)	10 (He)	7 to 10 (He)
Extract 1 [V]	0	Fixed	0	Fixed	0	Fixed
Extract 2 [V]	-180	-200 to -160	-180	-200 to -160	-180	-200 to -160
Omega Bias [V]	-80	-110 to -70	-80	-110 to -70	-80	-110 to -70
Omega Lens [V]	10	7 to 12	10	7 to 12	10	7 to 12
Cell Entrance [V]	-30	-40 to -30	-40	-40 to -30	-130	-150 to -110
Cell Exit [V]	-50	-60 to -40	-60	-60 to -40	-150	Fixed
Deflect [V]	10	8 to 15	0	-5 to 4	-80	-90 to-70
Plate Bias [V]	-40	-50 to -30	-60	Fixed	-150	Fixed
OctP RF [V]	180	150 to 200	180	150 to 200	190	180 to 200
OctP Bias [V] *1	-8	-10 to -6	-18	Fixed	-100	Fixed
QP Bias [V] *1	-3	-7 to -3	-15	Fixed	-96	-97 to -90

*1 Generally, set the QP Bias at least 2 to 3V higher than OctP Bias.

*2 High-Energy Collision mode allows for enhanced reduction of interference under the Helium mode. However, as it will also result in reduced low-mass sensitivity, it should be used in accordance with the purpose of the analysis.

Reference: For 7700s

The recommended tuning parameter values for 7700s are shown below.

Table 3 Recommended Values for 7700s (When Using a MicroFlow Nebulizer)

Parameter	High Sensitivity		Cell Gas Mode		High Energy Collision Mode [*2]		Cool Plasma	
	Recommended Value	Recommended Range	Recommended Value	Recommended Range	Recommended Value	Recommended Range	Recommended Value	Recommended Range
RF Power [W]	1500	fixed	1500	fixed	1500	fixed	600	fixed
Smpl Depth [mm]	8	7 to 10	8	7 to 10	8	7 to 10	18	fixed
Carrier Gas [L/min]	0.7	fixed	0.7	fixed	0.7	fixed	0.7	fixed
Makeup Gas [L/min]	0.5	0.3 to 0.7	0.5	0.3 to 0.7	0.5	0.3 to 0.7	0.75	0.6 to 1.2
Dilution Gas [L/min]	0	fixed	0	fixed	0	fixed	0	fixed
Neb Pump [rps]	0.1	fixed	0.1	fixed	0.1	fixed	0.1	fixed
S/C Temp [degC]	2	fixed	2	fixed	2	fixed	2	fixed
He or H_2 gas [ml/min]	0	fixed	3.6(He) 4.0(H_2)	3.2 to 4.0(He) 3.6 to 4.4(H_2)	10(He)	7 to 10(He)	0	fixed
Extract 1 [V]	4.5	3 to 7	4.5	3 to 7	4.5	3 to 7	-120	-200 to -40
Extract 2 [V]	-100	-170 to -60	-100	-170 to -60	-100	-170 to -60	-5	-30 to 5
Omega Bias [V]	-70	-100 to -30	-70	-100 to -30	-70	-100 to -30	-70	-120 to -30
Omega Lens [V]	11	5 to 15	11	5 to 15	11	5 to 15	6	3 to 10
Cell Entrance [V]	-30	-40 to -30	-40	-40 to -30	-130	-150 to -110	-30	-40 to -30
Cell Exit [V]	-50	-60 to -40	-60	-60 to -40	-150	fixed	-60	fixed
Deflect [V]	12	8 to 15	0	-5 to 4	-80	-90 to -70	9	5 to 13
Plate Bias [V]	-40	-50 to -30	-60	fixed	-150	fixed	-60	fixed
OctP RF [V]	180	150 to 200	180	150 to 200	-190	180 to 200	150	100 to 200
OctP Bias [V] [*1]	-8	-12 to -6	-18	fixed	-100	fixed	-18	-30 to -10
QP Bias [V] [*1]	-3	-5 to -3	-15	fixed	-96	-97 to -90	-5	-5 to -3

*1 Generally, set the QP Bias at least 2 to 3V higher than OctP Bias.

*2 High-Energy Collision mode allows for enhanced reduction of interference under the Helium mode. However, as it will also result in reduced low-mass sensitivity, it should be used in accordance with the purpose of the analysis.

Setting Quantitative Method Parameters

The following topics will help you create an acquisition method for quantitative analysis with the Agilent 7700 Series ICP-MS MassHunter Workstation.

Set up a Quantitative Method

Create a quantitative method for environmental analysis as described below:

1 Select **Edit Entire Method** from the Methods menu. This initiates a series of dialog boxes, so you can set the parameters for your method. See the online help for a full description of the items on these dialog boxes.

Tip See also "Use the Method Wizard to create a method" on page 19.

2 On the Edit Method dialog box, mark the following options, then click **OK**.

3 On the Method Information dialog box:

a Enter a comment

b Mark the **Data Analysis** check box to run the data analysis automatically after data acquisition. For instructions on setting up a Data Analysis method, see "Open an existing batch for data analysis" on page 26.

c Click **OK**.

4 On the Select Sample Types (or Select QC Items, if the Intelligent Sequence software is installed) dialog box:

   a Configure the list of **Selected QC Items** on the right to contain **CalStd** and **Sample** only.

   b Depending on the method you started with, you may have to use the **Add->** and **<-Remove** buttons to create the desired list.

   c Click **OK**.

5 On the Interference Equations dialog box:

   a Display interference correction equations in one of the following ways:

   - Mark **Show All Masses** to display all interference correction equations.
   - From the pull-down menu at the bottom right, select Interference Equation Library, to display interference correction equations in the selected library.

   b To edit the interference equation for a mass:

   - Click on the desired mass number in the list.
   - Click the **Edit** button to open the Edit Equation dialog box.
   - Edit the equation as described in online help and click **OK** to close the Edit Equation dialog box.

   c Repeat Step 5b for all masses you want to edit.

   d (*optional*) Click the **Save** button on the Interference Equation dialog box to save the edited equations for use with other methods.

   e When finished, click **OK**.

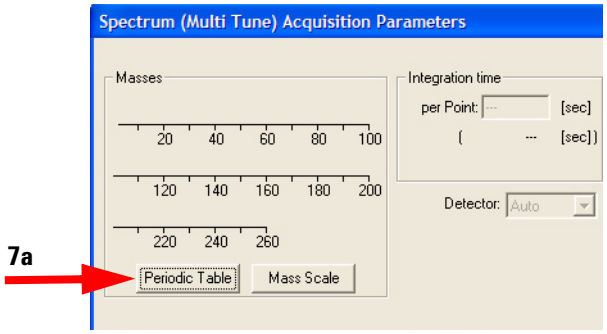
6 On the Acquisition Mode dialog box:

   a Check the **Spectrum (Multi Tune)** option.

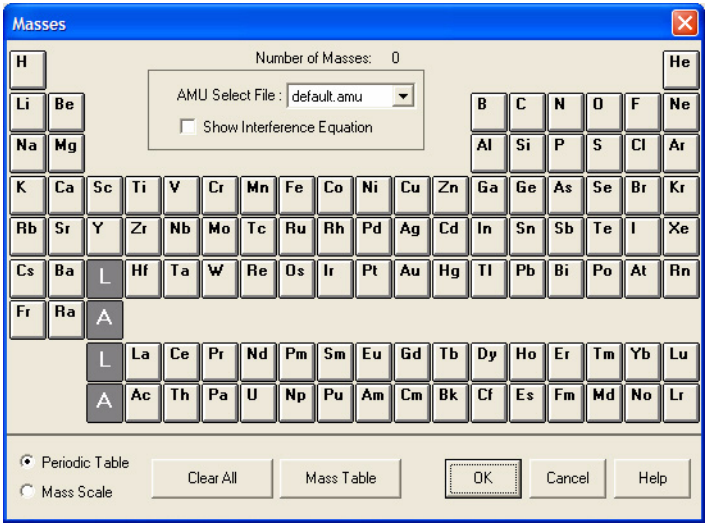
   b Click **OK**. Multi Tune allows you to use different tuning modes (e.g. Hydrogen and Helium modes) for different elements in a single run.

7 Specify the masses and element to analyze as follows:

a On the Spectrum (Multi Tune) Acquisition Parameters dialog box, click the **Periodic Table** button as shown below:



This opens the Masses dialog box shown below:



b Using Table 4 on page 18 as a guide, click to select the masses to monitor for each element. Selected masses appear in red in the dialog box.
Double-click on a element to open the Select Isotopes dialog box. Click the buttons for the isotopes you want to monitor. You can select one or more isotopes per element. Red check marks indicate the selected isotopes. To clear an isotope, click the right mouse button. Click **OK** when you are finished selecting isotopes for that mass.

c Repeat Step 7b to set up each mass as shown in Table 4.

d When you are finished, click the **Mass Table** button. The Mass Table dialog box opens to display the masses to be monitored for each element. Check that the masses listed in the Mass Table dialog box match those shown in Table 4 and adjust them if necessary. This is the list of masses that will be monitored in the method.

e When finished setting up all the required elements and masses, click **OK** on the Mass Table and Masses dialog boxes.

8 Set the **Tune File** and **Stabilization time** for each step in the upper right area of the Spectrum (Multi Tune) Acquisition Parameters dialog box:

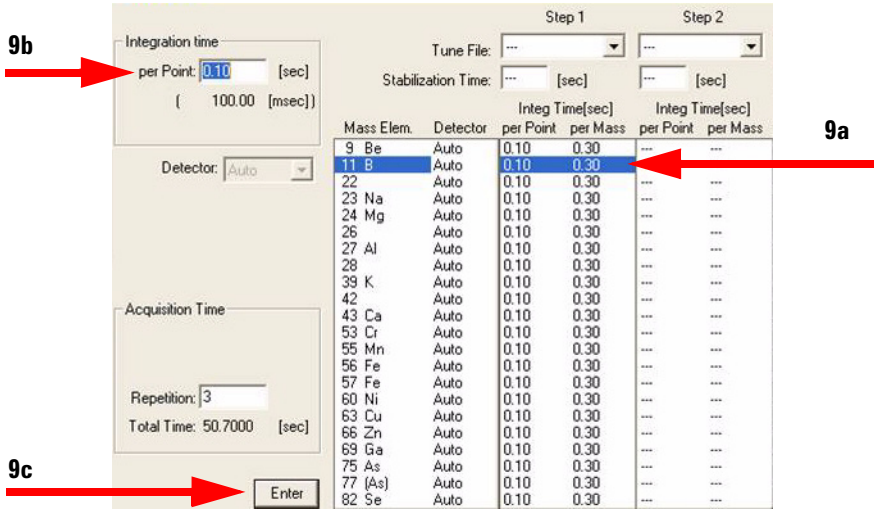
Step	Tune File	Stabilization Time
1	Hydrogen mode	30 sec
2	Helium mode	30-50 sec

9 Set the **Integration time per Point** for each mass:

a Click on the row for the mass in the column for the appropriate step (i.e. **Step 1** for Hydrogen mode, **Step 2** for Helium mode) as specified in the **Reaction Gas** column in the Table 4 on page 18. You can select multiple masses by **<Ctrl>+clicking** on them.

b Enter the **Integration time per Point** as specified in Table 4. The integration times in the table are generally relatively short, and can be extended as necessary.

c Click the **Enter** button. The **per Point** integration time and calculated **per Mass** integration time will be updated in the table.

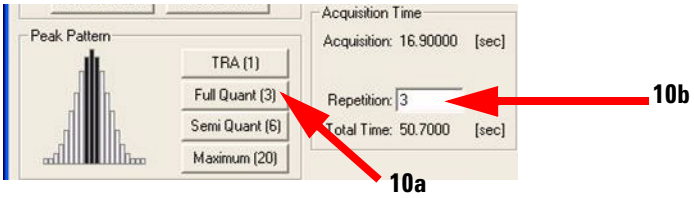


d Repeat for each mass in the table. For masses such as 71 that are monitored in both steps, remember to enter integration times for both.

10 Make the following additional selections on the Spectrum (Multi Tune) Acquisition Parameters dialog box:

a Select **Full Quant (3)** for the **Peak Pattern**.

b Set the **Repetition** count to **3**.



c Mark the **Return to First Tune Step** option at the bottom of the screen, and click **OK**.

11 On the Peristaltic Pump Program dialog box, set the following parameters, then click **OK**. See online help for more information on these parameters and on the optional features: Intelligent Rinse and Execute Pre-emptive Rinse.

- **Before Acquisition**
- **After Acquisition (Rinse Port)**
- **After Acquisition (Rinse Vial)**

12 (*optional*) Set up pre-emptive rinsing as follows:

a Select **Set PeriPump Program** from the PeriPump menu in the Acquisition window.

b On the Peristaltic Pump Program dialog box, mark the **Execute Pre-emptive Rinse** option at the bottom of the screen.

c Enter an initial time of **15 seconds**, then click **OK**.

d Adjust the time so that the analysis is completed 5 seconds before the rinse solution reaches the nebulizer.

13 To save the method:

a On the Method Save Options dialog box, mark all items, and click **OK**.

b On the Save Method As dialog box, enter a method name, and click **OK**.

Table 4 Set up information for elements and masses for Environmental Analysis

Element	Mass	Confirmation Mass[1]	Mode	Internal Standard[2]	Approx. Integration Time (sec/Point)
Be	9		No gas	^{6}Li	0.3
B	11		No gas	^{6}Li	0.1
Na	23		He	^{45}Sc	0.05
Mg	24		He	^{45}Sc	0.05
Al	27		He	^{45}Sc	0.3
P	31		No Gas	^{6}Li (^{72}Ge, ^{45}Sc)[3]	0.1
K	39		He	9 or 71	0.05
Ca	44	43	He	9 or 71	0.1
V	51		He	^{45}Sc	0.5
Cr	52	53	He	^{45}Sc	1
Mn	55		He	^{45}Sc	0.1
Fe	56	57	He	^{45}Sc	0.1
Co	59		He	^{45}Sc	0.1
Ni	60		He	^{45}Sc	1
Cu	63	65	He	^{45}Sc	0.1
Zn	66	64	He	^{72}Ge	0.1
As	75		He	^{72}Ge	1
Se	78		He or (H_2)[4]	^{72}Ge	5
Br	79		No Gas	^{72}Ge	0.1
Mo	95	98	He	^{103}Rh	0.1
Ag	107	109	No Gas	^{115}In	0.1
Cd	111	114	No Gas	^{115}In, (^{209}Bi)	1
Sn	118		No Gas	^{103}Rh (^{115}In, ^{209}Bi)	0.1
Sb	121		No Gas	^{115}In, (^{209}Bi)	0.1
I	127		No Gas	^{103}Rh (^{115}In, ^{209}Bi)	0.1
Ba	137	135	No Gas	^{115}In, (^{209}Bi)	0.1
Hg	201	202	No Gas	^{209}Bi	1
Tl	205		No Gas	^{175}Lu, (^{209}Bi)	0.1
Pb	208		No Gas	^{175}Lu, (^{209}Bi)	0.1
Th	232		No Gas	^{175}Lu, (^{209}Bi)	0.1
U	238		No Gas	^{175}Lu, (^{209}Bi)	1

1) Can be subject to interferences.
2) ISTD solution P/N is 5188-6525. See page 23 for more information.
3) Alternate internal standards are shown in parentheses.
4) When H_2 option is installed (then better MDL can be achieved).

Access Method parameters directly

You can access some method panels directly, using the following menu items:

Method parameters	Window	Menu	Menu item
Method Information	ICP-MS Top	Methods	Edit Method Information
Select Sample Items, Select QC Items (Intelligent Sequence Software is installed)	ICP-MS Top	Methods	Select QC Items
Interference Equation	ICP-MS Top	Methods	Edit Interference Equation
Acquisition Mode	Acquisition	EditParameters	Set Mode
Spectrum Acquisition Parameters	Acquisition	EditParameters	Set Parameters
Peristaltic Pump Program	Acquisition	PeriPump	Set Peripump Program
Method Save Options	ICP-MS Top	Methods	Save As

Use the Method Wizard to create a method

This way of creating a method makes it easy to create methods for environmental analyses, since it supplies many of the parameters that are appropriate for various sample types and regulations.

1 Select **Run Method Wizard** from the Methods menu in ICP-MS Top.

General Items

2 Enter name for the method and click the **Next>** button.

Sample

3 Select the sample as follows:

- **a** Select **Environmental** as the **Application Type**.
- **b** Select a **Sample**, such as **Drinking Water** or **High TDS**. A description of the current selection is displayed in the lower part of the window.
- **c** Select a **Regulation**, such as EPA200.8 or EPA6020. Only the guidelines that apply to the selected sample are displayed.
- **d** Click the **Next>** button.

Elements

4 The list of masses and elements that will be monitored is displayed. The information in this list is based on the sample and regulation selected above. Review the element information and make any changes as follows:

a Right-click on the element of interest in the Element List Information.

b Select one of the following options from the shortcut menu:

- **Add** - *see Step 4c below*
- **Delete** - removes the selected mass from the list
- **Edit** - *see Step 4c below*

c If you are adding or editing an element, enter the following information on the **Add** (or **Edit**) **Elements** dialog box, then click **OK**.

- **Mass**
- **Element Name**
- **Integration time (sec/Point)**
- **Tune step number**

d (*optional*) Click the **Edit/View Equation** button to view or edit an interference correction equation. See online help for more information.

e Click the **Next>** button.

Finish

5 Review the method information, then click the **Finish** button.

Tip

You can edit the method later by selecting **Edit Entire Method** from the Methods menu and marking the parts of the method to edit, or from the menus items shown in the previous section.

Running the Samples

The following topics will help you prepare a sequence, and run your samples with the Agilent 7700 Series ICP-MS MassHunter Workstation.

Set up the Sequence

Set up a sequence for your analysis as follows:

1. Select **Edit Sample Log Table** from the Sequence menu in the ICP-MS Top window. The samples you enter will be injected in the order they are listed in the table.
2. Enter the following information for the first sample (Row 1) of the table:
   - **a** **Method**: Double-click in the **Method** column of Row 1 to open the Select Method dialog box. Select the desired method and click **OK**.
   - **b** **Type**: Click in the **Type** column of Row 1 to open a list of types.
     - Select **CalBlk** for calibration blanks.
     - Select **CalStd** for calibration standards.
     - Select **Sample** for unknown samples.
   - **c** **Vial**: Click in the **Vial** column of Row 1 and enter the vial number of the first sample or standard.
     - The large bottles for cleaning solutions are positioned at 1, 2, and 3.
     - The small vials on the rack are positioned at 1001, 1002, 1003... etc.
   - **d** **Data File**: Leave this cell blank to assign a file name automatically at run time.
   - **e** **Sample**: Enter a name for the sample in the **Sample** column.
   - **f** **Comment**: Enter a comment for the sample in the **Comment** column.
   - **g** **Dil/Lvl**: Specify a Dilution factor or Level.
     - **For standard solutions**: click in the **Dil/Lvl** column of Row 1 and select the desired level from the list.
     - **For samples**: click in the **Dil/Lvl** column of Row 1 and type in the dilution factor (1 for undiluted), or double-click to open the Calculate Dilution Factor dialog box. See online help for more information.
   - **h** **ISTD Conc**: Double-click in the **ISTD Conc** column of Row 1 to open the Set ISTD Conc dialog box. Select the desired calibration level or set a ISTD Conc value, then click **OK**.

3 Fill in values for the rest of your samples as follows:

   a Select the first row and the subsequent rows by dragging the mouse.

   b Right-click in the highlighted rows and select **Fill Down** from the shortcut menu.

   c The values for **Method**, **Type**, **Dil/LVL** and **ISTD Conc** are copied from Row 1 to the selected blank rows. Sequential **Vial** numbers are assigned.

4 Modify the information for the sample rows added in Step 3 to match your analyses.

5 (*optional*) To turn off the plasma automatically at the end of the sequence, append the following row to the end of the sequence:

   - **Type**: **Keyword**
   - **Data File**: **StandBy**
   - Leave all other cells blank

6 (*optional*) Select the **Import DA Method from Existing Batch** check box to import an existing data analysis method from the specified data batch directory. You can import the **DA Method Only** or the **DA method and Std Data**. You can also apply a data analysis method *after* data acquisition.

7 Click **OK** to close the Edit Sample Log Table dialog box.

8 If errors are found in the table, a message appears.

   a Click **OK** and the offending cell will be highlighted in the table.

   b Fix the problem in the highlighted cell.

9 Repeat Steps 7 & 8 until no errors are detected.

10 (*optional*) Select **Save** from the Sequence menu in the ICP-MS Top window. Enter a name for the sequence you just created, and click **OK**. You don't need to save a sequence to run it, but doing so allows you to open it later and use it as a starting point for creating a new sequence more quickly.

Analyze the samples

1 Prepare the following standards, samples, and each solutions to use for analysis. The solutions should contain 0.5% HCl as well as nitric acid.

- Make up the following calibration standards by diluting SPEX XSTC-760A with nitric acid to approx. 1%. The concentration of the undiluted solution is 100x that of drinking water quality standard.

Calibration curve ranges	SPEX XSTC-760A	Na (ppm)	Mg, K, Ca (ppm)
Level 1	Blank HNO3	0	0
Level 2	1/1000 dilution (1/10 of standard)	10	5
Level 3	1/200 dilution (1/2 of standard)	50	25
Level 4	1/100 dilution (standard concentration)	100	50

For high-concentration Na, Mg, K, and Ca, it is safer to prepare their mixed standard solutions separately instead of mixing with the SPEX mixed standard solution, so as to prevent contamination with the reagent, particularly if the required concentration level is 1/100 of the standard.

- Dilute samples with nitric acid to approx. 1%
- Dilute Internal standard solution (*P/N 5188-6525*) 200 times (for on-line internal standard)
- Use of nitric acid (1 to 5%) is recommended for the cleaning solution, as it will clean the introduction system better than pure water. If pure water is used, residual memory may be washed out while introducing acidic samples, affecting the quantitative measurement.

2 Select **Run** from the Sequence menu or click the  icon on the toolbar to open the Start Sequence dialog box.

3 Set the parameters to the desired values.

4 The **Data Batch Directory** is set to a unique name based on the current date and time. The format is year, month, day, hour, and sequential numbers. For example, **08H14K01.B** is the second batch directory created between 10AM and 11AM on August 14, 2008. The parts of the name are derived as follows:

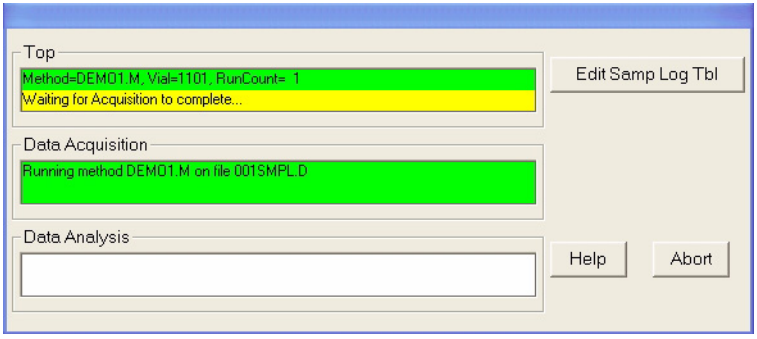
08 =	2008
H =	August
	(month - A through L corresponds to the 12 months, in order, so A = January, etc.)
14=	the day of the month
K=	the hour of the day
	(A - X correspond to 0 - 23)
01=	a sequential number for sequences run within the same hour, i.e. 00, 01, 02, and so on
.B=	the file extension for a batch file

If you wish, you can type in a different directory name instead. The name can be up to eight characters long. The batch folder will be created in **C:\ICPMH\1\DATA**.

5 (*optional*) Select the **Import DA Method from Existing Batch** check box to import an existing data analysis method from the specified data batch directory. You can import the DA Method Only or the DA method and Std. Data.

6 Click the **Edit DA Method** button in the lower left of the dialog box, then create a Data Analysis method as described on page 28.

7 Click the **Run Sequence** button to start the run. The Run Sequence Status dialog box appears.



8 If desired, you can monitor acquisition as described in online help.

9 When the sequence is complete, analyze the data as described in the following section.

Quantitative Data Analysis

The following topics will help you perform data analysis on your samples with the Agilent 7700 Series ICP-MS MassHunter Workstation.

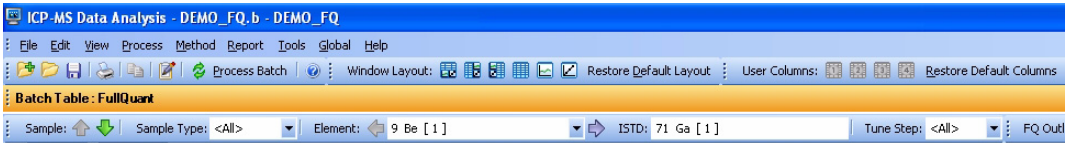
Typical scenarios for Data Analysis

	Description
To import an existing method	The data was acquired with reference to a Data Analysis (DA) method. (E.g. the "Edit DA Method" button was clicked or the "Import DA Method from Existing Batch" option was selected when setting up the sequence). In this scenario, the ICP-MS Data Analysis window opens automatically after acquiring the data. Proceed in *one* of the following ways: • If no changes are required to the DA method, then proceed to "View analysis results" on page 33 and to "Generate a report" on page 34. • If you wish to modify the existing DA method for this acquired data, then proceed to "Create the Data Analysis (DA) method for Quantitative data analysis" on page 28 to open the Method Editor and make changes to the DA method.
To create a method after completing data acquisition	The data was acquired *without* reference to a DA method. In this scenario, the ICP-MS Data Analysis window opens automatically after acquiring the data, but only the file name is displayed, since no analysis method has yet been applied. Proceed as follows: **a** Open the method editor as described in "Create the Data Analysis (DA) method for Quantitative data analysis" on page 28. **b** Proceed to create a DA method for Quantitative data analysis.
To create a method before performing data acquisition	You want to create a DA method without reference to any acquired data. **a** Follow the steps in "Open an existing batch for data analysis" on page 26 to create a new batch folder, since a data batch does not already exist. **b** If desired, import samples as described in Step 3. **c** Proceed to "Create the Data Analysis (DA) method for Quantitative data analysis" on page 28.

Open an existing batch for data analysis

If the window is not already open, open the ICP-MS Data Analysis window in *either* of the following ways:

- Select **Main Panel** from the DataAnalysis menu in the ICP-MS Top window, *or*
- Click the  button on the ICP-MS Top toolbar.



Tip To open an existing batch:

a Select **Open Analysis File** from the File menu to open the Open Batch dialog box.

b Select the file of interest and click **Open**. This opens an Analysis file with data that has been previously acquired.

c Then proceed to "Create the Data Analysis (DA) method for Quantitative data analysis" on page 28 to create a Data Analysis (DA) method (if a DA method was not applied at time of data acquisition) or to modify the method, if needed (if the DA method was already applied).

Creating a new batch for data acquisition

1 Create a new Batch Folder, which will be used to contain your data analysis results:

a Open the New Batch Folder dialog box in *either* of the following ways:

- Click the New Batch Folder toolbar button  , *or*
- Select **New Batch Folder** from the File menu.

b Type in a name for the new folder (you may select a different location if desired).

c Click the **Create** button.

2 Import the samples as follows:

a Select **Import Samples** from the File menu.

b Navigate to the data file folder where the data files of interest are located and click the **Open** button. The data files in that folder are displayed on the Import Samples dialog box.

c To import samples from another folder, click the **Browse** button.

d Repeat Steps b and c until all samples of interest are displayed on the Import Samples dialog box.

e Select the samples to import:

- **<Ctrl>+click** to select multiple samples from the list.
- Click the **Select All** button to select all samples in the list.

f Click **OK**.

Create the Data Analysis (DA) method for Quantitative data analysis

1 Open the Method Editor window in *either* of the following ways:

- Click the **Edit Method** toolbar button , *or*
- Select **Edit** from the Method menu.

Tip To import existing DA method, click **Import Method only** or **Import Method and Standard Data** and select an analysis file to import.

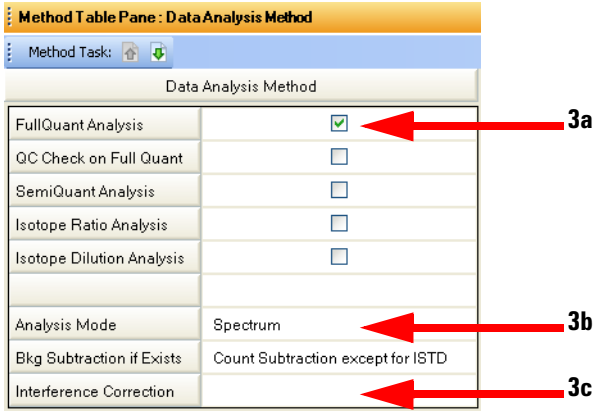
2 Select **Data Analysis Method** from Method Tasks section 2 (on the left side of the screen) to display the Data Analysis Method pane

3 Set the options as follows:

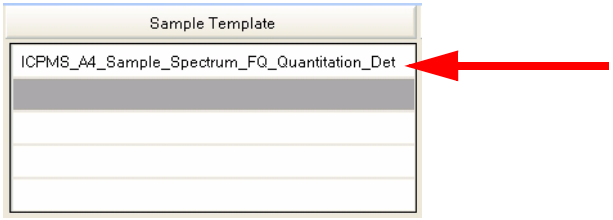
a Mark the check box for **FullQuant Analysis**.

b Set the **Analysis Mode** to **Spectrum**.

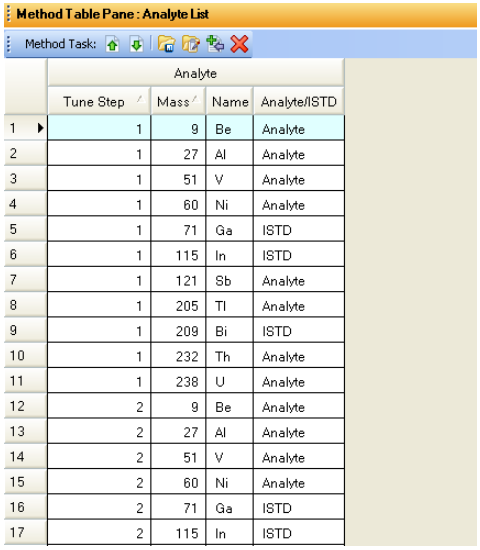
c Select the correction method from the **Interference Correction** list.



4 Select a **Sample Template** from the list.



5 Select **Analyte List** from Method Tasks section 3 to display the Analyte List pane.



Method Table Pane : Analyte List

Method Task:

	Analyte			
	Tune Step	Mass	Name	Analyte/ISTD
1	1	9	Be	Analyte
2	1	27	Al	Analyte
3	1	51	V	Analyte
4	1	60	Ni	Analyte
5	1	71	Ga	ISTD
6	1	115	In	ISTD
7	1	121	Sb	Analyte
8	1	205	Tl	Analyte
9	1	209	Bi	ISTD
10	1	232	Th	Analyte
11	1	238	U	Analyte
12	2	9	Be	Analyte
13	2	27	Al	Analyte
14	2	51	V	Analyte
15	2	60	Ni	Analyte
16	2	71	Ga	ISTD
17	2	115	In	ISTD

To open the analyte list from the existing data:

- Click the button, *or*
- Right-click in the pane and select **Load list from acquired data**.

Tip You can also create an analyte list in *either* of the following ways:

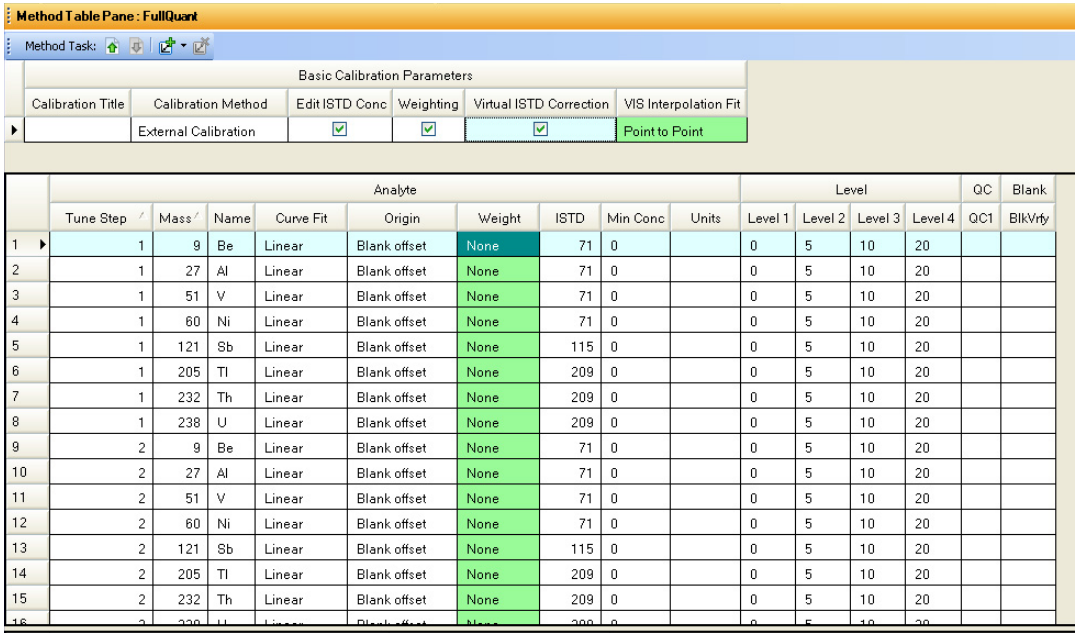
- To load an analyte list from an acquisition method that has already been developed, click the button.
- To add new analyte lines, click the button, then designate the Tune Step, Mass, Name and Analyte/ISTD for each new line as described in Step 6.

6 Enter the following information for *each analyte*:

a Click the button at the right end of the **Tune Step** column, and select a tune step number.

b Click the button at the right end of the **Analyte/ISTD** column and select either analyte or ISTD.

7 Select **Full Quant** from Method Tasks section 4 to display the FullQuant Method Table pane:



Method Table Pane : FullQuant

Method Task:

Basic Calibration Parameters					
Calibration Title	Calibration Method	Edit ISTD Conc	Weighting	Virtual ISTD Correction	VIS Interpolation Fit
	External Calibration	☑	☑	☑	Point to Point

	Analyte									Level				QC	Blank
	Tune Step	Mass	Name	Curve Fit	Origin	Weight	ISTD	Min Conc	Units	Level 1	Level 2	Level 3	Level 4	QC1	BlkVrfy
1	1	9	Be	Linear	Blank offset	None	71	0		0	5	10	20		
2	1	27	Al	Linear	Blank offset	None	71	0		0	5	10	20		
3	1	51	V	Linear	Blank offset	None	71	0		0	5	10	20		
4	1	60	Ni	Linear	Blank offset	None	71	0		0	5	10	20		
5	1	121	Sb	Linear	Blank offset	None	115	0		0	5	10	20		
6	1	205	Tl	Linear	Blank offset	None	209	0		0	5	10	20		
7	1	232	Th	Linear	Blank offset	None	209	0		0	5	10	20		
8	1	238	U	Linear	Blank offset	None	209	0		0	5	10	20		
9	2	9	Be	Linear	Blank offset	None	71	0		0	5	10	20		
10	2	27	Al	Linear	Blank offset	None	71	0		0	5	10	20		
11	2	51	V	Linear	Blank offset	None	71	0		0	5	10	20		
12	2	60	Ni	Linear	Blank offset	None	71	0		0	5	10	20		
13	2	121	Sb	Linear	Blank offset	None	115	0		0	5	10	20		
14	2	205	Tl	Linear	Blank offset	None	209	0		0	5	10	20		
15	2	232	Th	Linear	Blank offset	None	209	0		0	5	10	20		

8 Set the following calibration curve parameters:

- The calibration curve method (External Calibration / Standard Addition)
- Type of VIS correction for ISTD elements
- Curve fit type (Linear / Quadratic / Excluded)
- Handling of the origin (Ignore / Force / Blank offset)
- Calibration curve weight
- ISTD elements used for correction
- Concentration of the calibration curve level (Min Conc). For most applications, enter **0**.
- Concentration unit
- Concentration of the blank

9 The following **Advanced Info** method tasks are described in online help:

- FullQuant Outlier
- Edit Report Templates

10 Select **Validate** from Method Tasks section 5. Correct any method errors before proceeding.

11 Select **Return to Batch-at-a-Glance** from Method Tasks section 5. Click **Yes** to save the Data Analysis method.

Process the batch

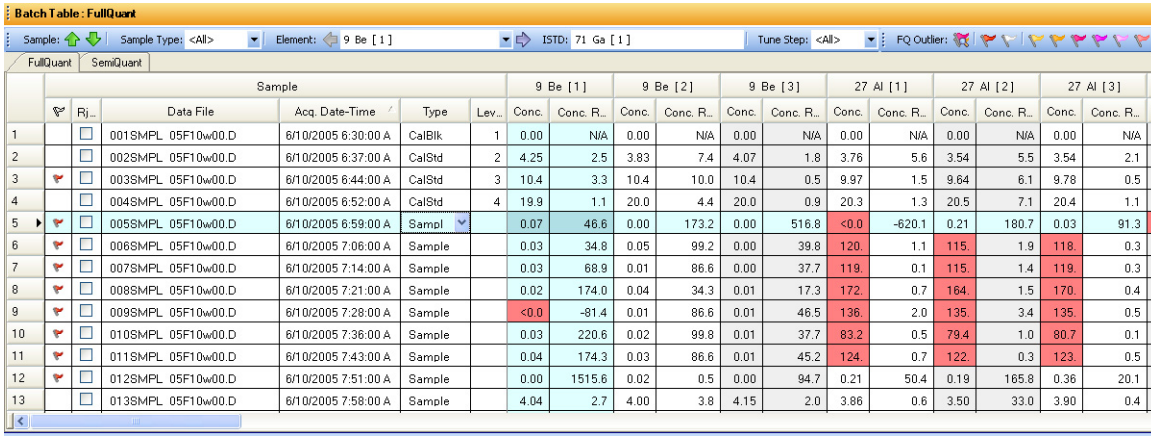
1 Initiate batch processing in *either* of the following ways:

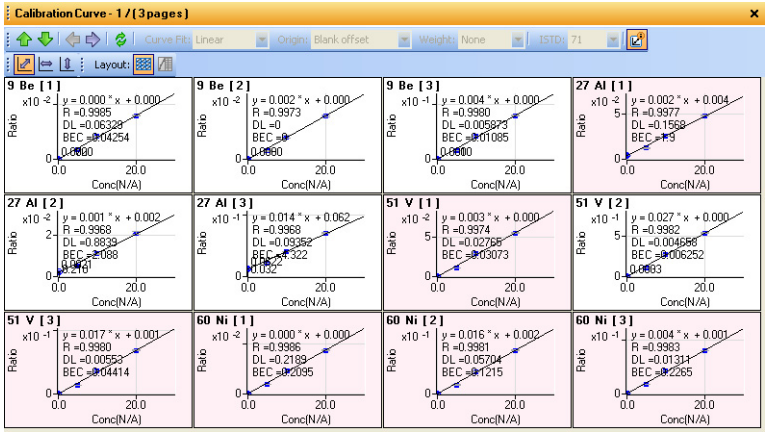
- Click the Process Batch toolbar button 

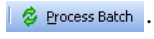
, *or*
- Select **Process Batch** from the Process menu.

Analysis is performed on the samples you added to the batch in the previous topic, using parameters set in the DA method. See “Create the Data Analysis (DA) method for Quantitative data analysis” on page 28..

2 Review the analysis results that are displayed in the Batch Table and Calibration Curve panes, as shown in the following examples.





Note The Process Batch button changes to black after processing: 

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View analysis results

Batch Table pane

1 View the concentration and count for each element in the samples in the Batch Table pane.

- Concentration and count for problematic data (Outlier) are displayed with a color of each Outlier background.
- See online help for information if you want to change the way the data is displayed, such as sorting or customizing the columns
- Also see online help for information on changing the layout or doing auto review.

Spectrum pane

2 View mass spectra in the Spectrum pane. You can zoom in to identify elements by right-clicking and dragging the mouse cursor around the desired mass number. See online help for information on other ways to display or process data in the Spectrum pane, such as:

- Switching between log and linear
- Switching between one-row and three-row
- Adding comments
- Identifying unknown spectra against an element database
- Subtracting background spectra
- Overlaying multiple spectra
- Tabulating the spectral information

Calibration Curve pane

3 View calibration curves for each element in the samples in the Calibration Curve pane.

- Problematic calibration curves (Outliers) are displayed with a pink background.
- See online help for information on other changes you can make to the calibration curve, such as excluding a calibration curve level, changing the concentration of a calibration curve level, changing the calibration curve type, changing the handling of the origin, or changing the weighting of calibration curve.

ISTD Stability Graph pane

4 View the percent recovery of each ISTD element in the ISTD Stability Graph pane. See online help for more information.

Save analysis results

Be sure to *always save the analysis results* in one of the following ways:

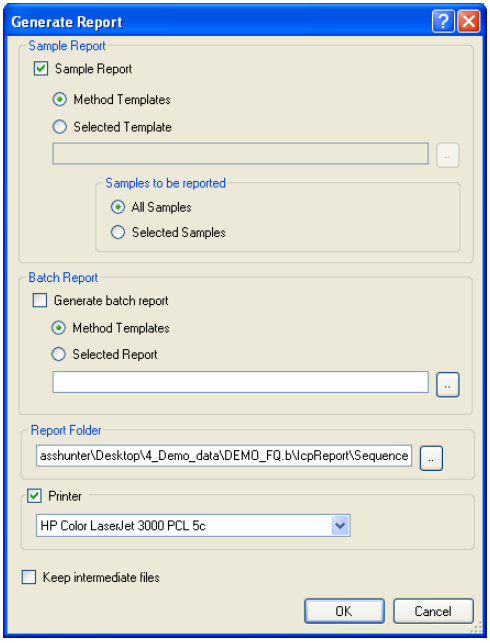
As a new file: Select **Save Analysis File As** from the File menu.

To overwrite an existing results file with the same name:

- Click the Save toolbar button , *or*
- Select **Save** from the File menu.

Generate a report

1. Select **Generate** from the Report menu in the MassHunter ICP-MS Analysis window.
2. Mark the desired report options and click **OK** (see example below).



3. Click **OK** to generate the report.

Semi-Quantitative Analysis

Semi-quantitative analysis allows you to quickly scan and obtain information about the concentrations of all elements that are present in an unknown sample. It is useful when you want to obtain concentration information for a large number of elements (>70) without external calibration, such as screening prior to quantitative analysis on target elements. Typically, semi-quantitative analysis is accurate to +/- 30% or better on completely unknown samples.

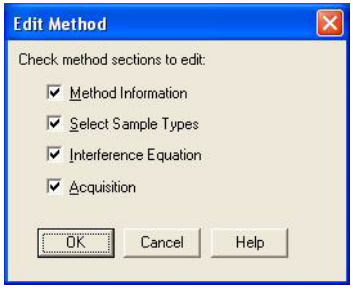
Prepare for SemiQuant Analysis

Prepare the following samples and solutions for analysis:

- Samples (dilute with nitric acid to approx. 1%)
- Tuning solution (Li, Co, Y, Ce, Tl, 1ppb each, with approx. 1% nitric acid)
- Cleaning solution (1 to 5% nitric acid)

Create a SemiQuant acquisition method

1. Select **Edit Entire Method** from the Methods menu. This initiates a series of dialog boxes, so you can set the parameters for your acquisition method. See the online help for a full description of the items on these dialog boxes.
2. On the Edit Method dialog box, mark the following options, then click **OK**.



3. On the Method Information dialog box, set the following parameters, then click **OK**:
   a. Enter a comment
   b. Be sure that the **Data Analysis** check box is checked; this is required to run the data analysis automatically after data acquisition.

4 On the Select Sample Types (or Select QC Items, if the Intelligent Sequence software is installed) dialog box, set the following parameters, then click **OK**:

   a Configure the list of **Selected QC Items** on the right to contain **Sample**, **SQStd**, **SQBlk**, and **SQISTD**.

   b Depending on the method you started with, you may have to use the **Add->** and **<-Remove** buttons to create the desired list.

5 Set the Data Acquisition method parameters:

   a On the Acquisition Mode dialog box, mark the **Spectrum** option and click **OK**.

   b On the Spectrum Acquisition Parameters dialog box, click the **Mass Scale** button.

   c On the Masses dialog box:

   - Click the **Clear All** button.
   - Select all mass numbers by double-clicking on the top, middle, and bottom rows, then click **OK**.

   d Make the following additional selections on the Spectrum Acquisition Parameters dialog box, then click **OK**:

   - Select **Semi Quant (6)** for the **Peak Pattern**.
   - *Clear* the **Set every Mass** option, then set the **per Point Integration time** to 0.05 or 0.1 sec.
   - Set the **Repetition** count to **1**.

   e On the Peristaltic Pump Program dialog box, set the following parameters, then click **OK**.

   - **Before Acquisition**: Uptake Speed: 0 rps, Uptake Time: 0 sec, Stabilization Time: 70 sec
   - **After Acquisition (Rinse Port)**: Rinse time: 30 sec
   - **After Acquisition (Rinse Vial)**

   See online help for more information on these parameters and on the optional features: Intelligent Rinse and Execute Pre-emptive Rinse.

6 To save the method:

   a On the Method Save Options dialog box, mark all items, and click **OK**.

   b On the Save Method As dialog box, enter a name for the method, and click **OK**.

Analyze the samples

1. In the sequence table, set up a sequence with samples in the following order:
   a. Tuning solution (1 ppb Li, Co, Y, Ce, Tl) - Sample Type is “SQStd”
   b. Blank (i.e. nitric acid) - Sample Type is “SQBlk”
   c. Sample 1 -> Sample 2 -> Sample 3 ... - Sample Type is “Sample”
2. Run the sequence:
   a. Select **Run** from the Sequence menu or click the  icon on the toolbar to open the Start Sequence dialog box.
   b. Click the **Run Sequence** button to start the run. The Run Sequence Status dialog box appears.
   c. If desired, you can monitor acquisition as described in online help.

NOTE The same scenarios apply for offline Semi-quantitative data analysis as described in “Typical scenarios for Data Analysis” on page 25.

Create a new batch or open a new batch for SQ data analysis

Refer to “Open an existing batch for data analysis” on page 26 and “Creating a new batch for data acquisition” on page 26.

Create the Data Analysis (DA) method for SemiQuant data analysis

1 Open the Method Editor window in *either* of the following ways:

- Click the Edit Method toolbar button , *or*
- Select **Edit** from the Method menu.

Tip To import existing DA method, click **Import Method only** or **Import Method and Standard Data** and select an analysis file to import.

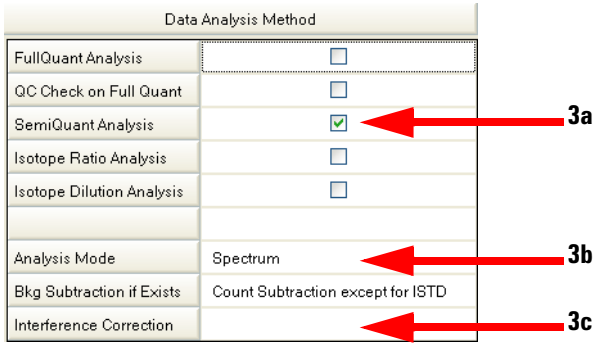
2 Select **Data Analysis Method** from Method Tasks section 2 (on the left side of the screen) to display the Data Analysis Method pane.

3 Set the options as shown below:

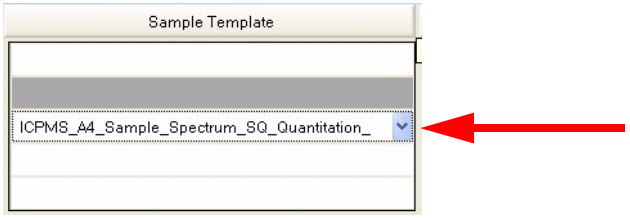
a Mark the check box for **SemiQuant Analysis**.

b Set the **Analysis Mode** to **Spectrum**.

c Select the correction method from the **Interference Correction** list.

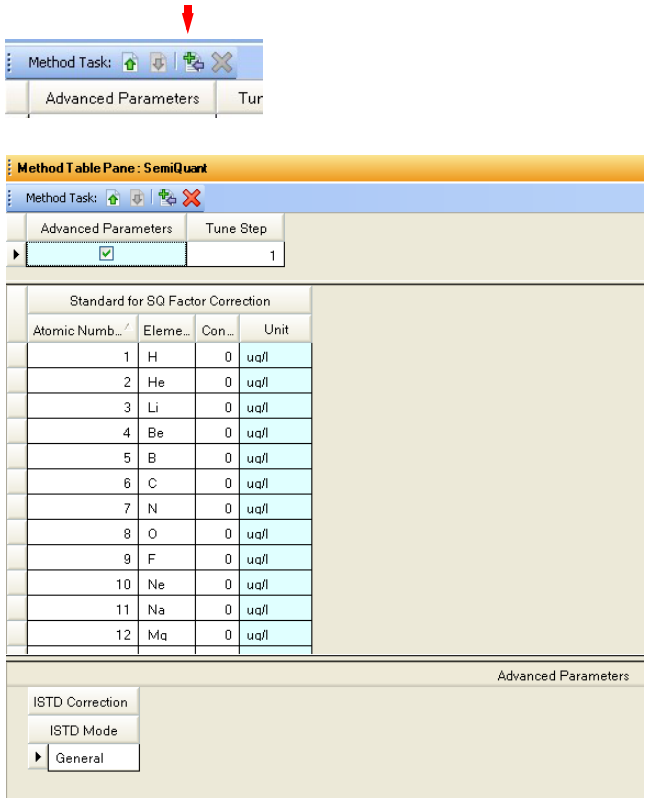


4 Select a **Sample Template** from the list.



5 Select **SemiQuant** from Method Tasks section 3 to display the SemiQuant pane.

Tip You can select elements to show in the pane by clicking **Add/Remove Standard Element**.



6 On the SemiQuant Method Table pane:

a Mark the check box for **Advanced Parameters**.

b Set the concentration of the elements in the standard sample being used for the SemiQuant factor correction.

c Select the ISTD correction method.

For setting details, refer to “SemiQuant Pane” in the “Commands Reference” section of online help.

7 Select **Validate** from Method Tasks section 5. Correct any method errors before proceeding.

8 Select **Return to Batch-at-a-Glance** from Method Tasks section 5. Click **Yes** to save the Data Analysis method.

Process the batch

1 Initiate batch processing in *either* of the following ways:

- Click the Process Batch toolbar button 

, *or*
- Select **Process Batch** from the Process menu.

Analysis is performed on the samples you added to the batch in the previous topic, using parameters set in the Data Analysis method, as described in "Create the Data Analysis (DA) method for SemiQuant data analysis" on page 38.

2 View/check the analysis results as follows:

a **Batch Table pane**--Displays the concentration and count for each element in the samples.

b **Spectrum pane**--Displays mass spectra. You can zoom in to identify elements by right-clicking and dragging the mouse cursor around the desired mass number.

c **Semiquant Factor pane**--Displays the SemiQuant factor graph for each sample. Place the cursor near a point on a graph to display element names and SemiQuant factor correction details. Use the arrow keys to move between samples. If necessary, the factors can be changed as described in "Change the SemiQuant Factors" on page 42.
If the SemiQuant Factor pane is not currently displayed, select **Calibration Curve** from the View menu.

d **ISTD Stability Graph pane**--Displays the percent recovery of each ISTD element in the samples. See online help for more information.

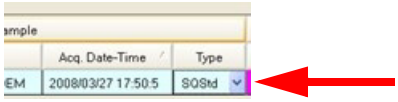
3 Save results using **Save Analysis File As** or **Save** from the File menu.

4 Select **Generate** from the Report menu, select report options, and click **OK**.

Correcting the Semiquant Factors

On MassHunter Workstation, the SemiQuant factors are predefined for each element. The greater the SemiQuant factor, the more efficiently that element is ionized in the plasma, yielding a higher sensitivity. Use the following procedure to correct the SemiQuant factors, as necessary.

1. Select and measure standard sample containing three or four elements whose concentration are known. Make sure that the concentrations of the elements in the standard sample cover the low, medium, and high ranges of the mass spectrum, or use the tuning solution that contains Li, Co, Y, Ce and TI as the standard sample.
2. In the ICM- MS Data Analysis window, load the data for the standard sample into the batch folder.
3. Set the sample type of the loaded Standard Sample to **SQStd** as shown in the example below:



4. Process the batch in *either* of the following ways:
   - Click the Process Batch toolbar button 

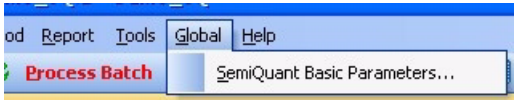
, *or*
   - Select **Process Batch** from the Process menu.

   The SemiQuant factor for *all elements* will be corrected based on the elements of known concentration in the standard sample (SQStd).

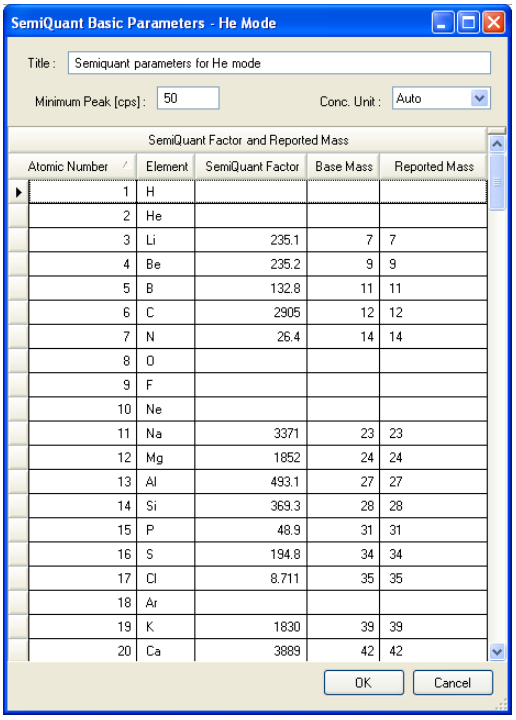
Change the SemiQuant Factors

You can configure the SemiQuant factor, Base mass, and Reported mass (isotopes).

1 Select **SemiQuant Basic Parameters** from the **Global** drop-down menu in the ICP-Analysis window:



The SemiQuant Basic Parameters dialog box opens.



2 Make the change by either:

- Right-clicking within the table and selecting from the context menu; or
- Selecting a cell and directly editing its content.

Refer to the Help for more details.

Notices

Manual Part Number

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In this Book

This *Quick Start Guide* presents first steps to using the Agilent 7700 Series ICP-MS MassHunter Workstation.

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