Title: Agilent 7700x Inductively Coupled Plasma Mass Spectrometer operation, data acquisition, processing and reporting		Copy No: ##
SOP No: 6.22/1.0/S	Effective Date: May 13, 2013	Location: ###

QSM Approval: _____

Agilent 7700x Inductively Coupled Plasma Mass Spectrometer operation, data acquisition, processing and reporting

1 INTRODUCTION AND SCOPE

1.1 This Standard Operating Procedure (SOP) provides procedures for the operation of the Agilent 7700x Inductively Coupled Plasma Mass Spectrometer (ICP-MS), and for data acquisition, processing and reporting using the MassHunter software.

1.2 Agilent 7700x ICP-MS system is restricted for use by, or under supervision of experienced and properly trained personnel.

2 INSTRUMENT START-UP

2.1 Shield torch must be used all the time when using ICP-MS. For installation of the Shield Torch, refer to the *Agilent 7700 Series ICP-MS Hardware Manual*.

2.2 The autosampler (ASX-500 Series) should be properly installed and configured for automatic control using MassHunter software (refer to the *(Agilent 7700/7500 Series ICP-MS, ASX-500 Series Autosampler, manual)*. Turn the autosampler **ON.**

2.3 Start the ICP-MS MassHunter Workstation software by clicking the ***ICP-MS Control*** button on the Windows desktop ( ). Select ***Hardware*** at the Task Bar (Figure 1).

2.4 Select the ***Autosampler Type*** (ASX 520) and set the ***Autosampler Rack*** configuration. Usually, the 60 positions racks are used for 10-mL tubes and the 21 position racks are used for the 40-ml tubes.

2.5 Start rinsing the sample probe by clicking ***Instrument >> ALS Rinse port***. The Rinse port should be filled with rinse solution and drained properly into the waste bottle.

2.6 Fill ***Bottle 1*** with fresh double deionised water (DDW), ***Bottle 2*** with fresh 1% HNO_3[1] and ***Bottle 3*** with tuning solution (1 µg/L Li, Y, Tl and Ce, in 2% HNO_3). Click the arrow at button and go to ***Bottle 1.***

[1] Acid concentrations in this document are expressed as % (v/v)

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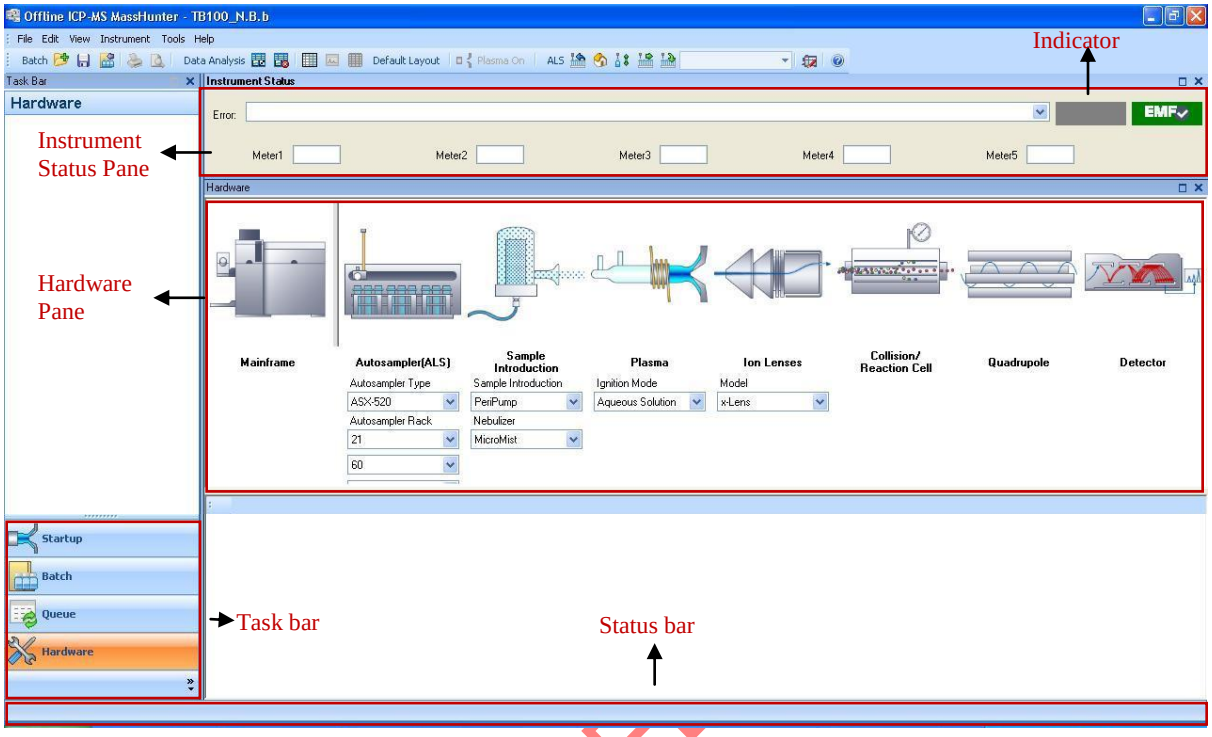


Figure 1

2.7 Ignite the plasma.

2.7.1 Open the liquid argon (Ar) gas valve and the hydrogen (H_2) and/or helium (He) gas cylinders valves. Make sure that the outlet pressure of liquid Ar Dewar is more than 100 psi (or Dewar is more than 30% full) and the gas cylinders pressures are not below 500 psi. **NOTE**: If the liquid Ar outlet pressure is lower than 100 psi, turn on the pressure-building valve on the Dewar; it may have to be kept open during the analysis.

2.7.2 Check the gas delivery pressures from the switchover gas line system. The Ar gas delivery pressure should be between 110 -120 psi. Adjust the knob on the switchover system and check the reading of the Ar pressure from the MassHunter until it is between 700 – 730 kPa. The H_2 or He gas outlet pressure should be 5 psi.

2.7.3 Make sure that the chiller is **ON**. The water temperature should be 12 to 15^{o}C and the water delivery pressure not less than 50 psi.

2.7.4 Check the drain vessels of autosampler and instrument, and empty if necessary.

2.7.5 If the instrument is in **STANDBY** mode, the LED on the top right side of the top cover and the indicator in the ***Instrument Status Pane*** displays an orange light. Go to Step 2.7.6

2.7.5.1 If the instrument is in **SHUTDOWN** mode, turn on the instrument and the computer.

2.7.5.2 Start the MassHunter software by double clicking ***ICP-MS Instrument Control*** icon on the desktop; click ***Hardware*** icon on the Task Bar. The ***Instrument Control*** window with the diagram of the instrument status will appear.

2.7.5.3 Right-click the ***Mainframe*** icon and select ***Vacuum ON***. Click ***Yes*** at the dialog box to confirm. It usually takes about 40 minutes for the vacuum chamber to attain its correct pressure of 5 x 10^{-4} Pa, depending on how long the vacuum chamber has been open to the atmosphere. The LED on the top right side of the top cover and the indicator in the ***Instrument Status Pane*** will be flashing until proper vacuum is achieved.

2.7.6 Check the condition of the peristaltic pump tubes and replace if necessary. Ensure that they are correctly clamped into the peristaltic pump.

2.7.7 Ensure the autosampler needle is in the ***Bottle 1***

2.7.8 Complete the "Standby Mode" section of the logbook. The typical values for Ar delivery pressure, backing pressure and analyser pressure are shown in Appendix A (Table A1). NOTE: Meter readings are displayed in the ***Instrument Status Pane***. Click ***View >> Meters…*** from the top panel and check the boxes of meters you want to display.

2.7.9 Select ***Instrument >> Plasma ON***. Click ***No*** to confirmation dialog box: *Run Startup after plasma ignition?* NOTE: Select ***Yes*** ONLY if an automated optimization of hardware components is deemed necessary.

2.7.10 When changing to the **ANALYSIS** mode is completed, the LED on the top right side of the top cover and the indicator in the ***Instrument Status Pane*** will display a green light. Check and ensure the drain is flowing.

2.7.11 Wait for at least 45 minutes for the system to stabilize. Then record in the "Analysis Mode" section of the logbook the following meter readings from the MassHunter: Ar gas tank pressure, forward and reflected power, interface and backing pressure (IF/BK pressure), analyzer pressure, cooling water flow rate at the interface and RF generator (RF/WC/IF). The typical values for these parameters are shown in Appendix A (Table A2).

3 CREATING A BATCH

3.1 In the ***ICP-MS Instrument Control*** window click ***Batch*** icon on the Task Bar. The acquisition method (including tuning, acquisition parameters and peripump program), data analysis method and sample list for a batch are created and stored in a single batch folder.

Create a new batch template before starting an acquisition. If an appropriate Batch Template is already created go to 3.11.

3.2 Click and select ***Preset methods*** from the ***Create from*** list. Click ***Select… >> Generic Method >> General Purpose (7700x)***. Enter the new batch name (e.g. save as 20130123_NT_N a batch template created on January 23rd 2013 for analysis of near-total metals (Method 6.11/*.*/M) in no-gas mode). Click ***Create***. NOTE: **It is strongly recommended that ONLY the person with EXTENSIVE EXPERIENCE on ICP-MS create or modify the Batch Templates**.

3.3 Tune Tab

3.3.1 Right-click any of the tune tabs, choose ***Configure*** and delete the tuning modes that do not apply to intended analysis (Preset Method have tuning parameters for all modes). Start tuning in no gas mode.

3.3.2 Make sure that the internal standard line is in DDW and the autosampler needle is in ***Bottle 3*** containing tuning solution (1 µg/L Li, Y, Tl and Ce in 2% HNO_3). Wait for about 5 minutes for the uptake of the solution to the nebulizer and stabilization of the signal. Peristaltic pump speed should be the same as that of analysis (e.g., 0.10 rps).

3.3.3 Right-click anywhere in the tuning window and select ***Tune >> Configure Tune Way***. Select ***Custom Tune*** and check the box ***Override Hardware Settings***. NOTE: ***Auto Tune*** can be selected when the instrument has not been used in a long time, after Shutdown and/or after major changes and repairs of the hardware. However, the ***Custom Tune*** has to be performed every time for fine tuning before saving the batch template.

3.3.4 Right-click anywhere in the tuning window and select ***Tune >> Acq. Parameters >> Set Acq. Parameters for Manual Sensitivity Tune*** (or click on the toolbar).

- Select masses 7, 89 and 205 to adjust sensitivity and obtain high stable signal over the whole mass range. Refer to the *Agilent 7700 Series ICP-MS MassHunter Workstation User Guide* (page 48) and Appendix B (Table B1) for typical values of tuning parameters.
- Select 156/140 and 70/140 ratios to monitor the oxides and doubly charged ions ratio, respectively.

3.3.5 Click the to start ***Signal Monitor*** for selected masses, and view the numerical values in the real-time display. When a parameter is modified, click Send To ICP-MS for the changes to take place. Click to stop the signal monitor.

3.3.6 The typical values of sensitivity are shown in Appendix B (Table B2). The RSD should not exceed 5% and the background counts should be less than 10 counts per

second (CPS) for each mass. The 140/156 and 70/140 ratios should be below 1.5% and 2%, respectively. The resolution (W-10%) should be 0.65 - 0.85 AMU, and the axis should be within ± 0.05 of the selected mass. If not, the following actions may be carried out:

- Increase the sampling depth
- Decrease the carrier gas and make up gas flows (sum should be ~1 L/min)
- Increase the RF power

3.3.7 After tuning is complete, right-click the ***Tune*** tab and rename the tuning file (for example Jan23_2013N if tuning in no gas mode is done on January 23rd, 2013).

3.3.8 Click 

to generate the tuning report. Check all the boxes of the dialog box and click ***Generate***. Print and keep the report as a record. Save the electronic copy of the tuning report in the appropriate directory.

3.3.9 Record Sensitivities, Oxide and Doubly Charged Ions percentages in the ICP-MS Instrument Performance charts/tables in the designated folder.

3.4 Tune the instrument in reaction/collision cell mode using He or H_2 gas, if needed. For details refer to Refer to the *Agilent 7700 Series ICP-MS MassHunter Workstation User Guide* (page 48) and Appendix B (Table B1) for typical values of tuning parameters.

3.4.1 It is recommended to tune in He or H_2 mode after tuning in no gas mode. Autotune cannot be used in this mode. However, after using Autotune in no gas mode, edit the tune values and change only values necessary for He or H_2 mode (Table B1).

3.4.2 Go to ***Cell*** >> ***Use Gas.*** Check the ***He flow*** or ***H2 flow*** box and set the value 4.5 mL/min or 5.0 mL/min for He or H_2 gas flow rate, respectively.

3.4.3 Modify the other parameters according to Table B1.

3.4.4 Maximize the sensitivity of ^{89}Y by adjusting the Cell Entrance, QP focus and Cell Exit.

3.4.5 Aspirate fresh DDW and measure the background for mass 51 AMU (He mode) and 56 AMU (H_2 mode). If the counts of these masses are too high, increase the flow rate of He or H_2 gas (up to 6.0 mL/min); then repeat steps 3.3.5 and 3.3.6 to obtain the best sensitivity (Table B2).

3.4.6 Record the analyzer pressure in the "Analysis Mode" section of the logbook.

3.5 Tune the instrument in the high matrix introduction (HMI) mode if analyses of lanthanoids (La to Lu) are required.

- Fill ***Bottle 4*** with the tuning solution for lanthanoids (5 µg/L Ba, Pr, Dy, Tm, Lu in 2% HNO_3). Set plasma conditions for the HMI mode (Table B1) and follow the steps 3.3.3 to 3.3.9.
- The sensitivity should be within limits specified in Appendix B (Table B2). The RSD should not exceed 5%; the oxide ratio should range between 0.5 and 1% and doubly charged ratio should be less than 2%.

3.6 Acq. Parameters Tab

3.6.1 Click the ***Acq Parameters*** tab and select parameters as follows:

Acq Mode:	***Spectrum Mode Options***:	Peak Pattern: 3 points
Spectrum		Replicates: 3
		Sweeps/Replicate: 100

3.6.2 Click  to select the elements and the masses that will be acquired (see Table B3).

3.6.3 In the ***Selected Elements*** window, click 

>> ***Add*** and fill out the dialog box as follows:

Corrected element	*Mass*	*Equations*	*Mass*	*Multiplier*
Pb	208		208	1
			207	1
			206	1

3.7 PeriPump/ISIS Tab

3.7.1 Click the ***PeriPump/ISIS*** tab and set the parameters as shown in Appendix B (Table B4). **NOTE:** When samples or standards are sequentially analyzed, memory effect or carry-over may occur if large concentration differences are present. The extent of memory effect is affected by sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer. The rinse period between samples must be long enough to eliminate significant memory effects.

3.8 Data Analysis Method Tab

3.8.1 Select the ***Basic Information*** tab and check the ***FullQuant***[2] analysis box.

3.8.2 Select the ***Analytes tab***. Click  to load the list of analytes that were specified in the acquisition method. Specify Y, In and Ho as internal standards elements (ISTD).

3.8.3 Select the ***FullQuant*** tab and choose the parameters as follows:

[2] The SemiQuant analysis can be also performed. However this document refers to FullQuant analysis only. Refer to MassHunter Workstation user guide for details on SemiQuant method

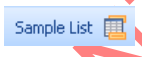
Basic Calibration Parameters			
Calibration method:		*Virtual ISTD correction:*	*VIS Interpolation fit:*
External calibration		Check box	Point to Point
Analyte			
Curve fit:	*ISTD*:	*Min Conc*:	*Unit*:
Linear	VIS	0	ppb
Level			
Type the concentrations of the calibration standards, in ppb. To add more levels right-click anywhere and select ***Add levels***			
ISTD			
Check the ***VIS Flag*** box			

NOTE: These parameters can be modified after analysis. Different curve fit equations, handling of the origin of calibration curves and internal standard correction methods may be used in order to improve the recoveries of the CRMs.

3.9 Sample List Tab

3.9.1 Click ***Acquisition Order*** >> ***Sequence Flow*** >> ***Calibration Standards*** and fill out the table related to it. The ***Sample Type*** should be either CalBlk or CalStd for the calibration blank and calibration standards, respectively. Assign Level 1 to the calibration blank. The levels of the standards assigned here should match the levels chosen at the ***Data Analysis*** set up.

3.9.2 Click ***Acquisition Order*** >> ***Sequence Flow*** >> ***Unknown Samples*** and fill out the table related to it. The ***Sample Type*** should be either Sample or QC for the unknown and QC samples, respectively.

3.9.3 Click 

and print the sequence. Load the autosampler with vials containing samples, blanks and standards. **NOTE**: Using cleaned plastic forceps, remove the filters from the vials.

3.10 Click  to ***Configure the Batch Acquisition***. Check ONLY the boxes of ***P/A factor adjustment*** and ***Rinse After Batch***. ***Rinse After Batch*** is designed to rinse the system for 5 minutes with DDW at pump speed 0.1 rps. Longer rinse times should be used if high matrix samples were analyzed.

3.11 Click 

to validate the method. If there are no errors or warning found, click OK and go to 3.12. Otherwise check the ***Method Error List*** at the bottom of the screen and make the necessary corrections.

3.12 Click 

to save the batch in the ***C:\Agilent\ICPMH\1\Batch Templates*** folder The template is now ready and should be used for further analysis.

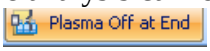
3.13 If an appropriate Batch Template was already created, select ***File*** >> ***New Batch Folder*** >> ***Create From*** >> ***Existing Batch*** >> ***Select.*** Choose the appropriate batch from ***C:\Agilent\ICPMH\1\Batch Templates*** folder, click ***Open,*** check the boxes for the items to be copied, such as ***Acq Method***, ***Tune Mode*** and ***DA Method,*** and click ***OK***. Name the new batch and click ***Create***. Follow steps 3.3 to 3.5 to check tuning and generate the Tune Report.

4 EXCECUTING THE QUEUE

4.1 Add the batch to the queue by clicking 

. Click ***Yes*** at the dialog box: *Save changes to the batch and add it to the queue*? When the acquisition starts, the ***ICP-MS Data Analysis*** window is opened automatically.

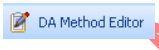
4.2 Click ***Queue*** in the task bar. The current progress status of the automatic acquisition is displayed in the status bar at the bottom right of the screen. For more details on executing the queue, refer to the *Agilent 7700 Series ICP-MS MassHunter Workstation User's Guide* (page 38).

4.3 In case the analysis cannot be completed before the end of the working hours, make sure to click the 

button (it should be highlighted in yellow) so that the plasma will turn off automatically.

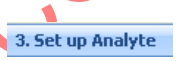
5 DATA PROCESSING FOR QUANTITATIVE ANALYSIS OF TRACE ELEMENTS

5.1 The analysis results can be checked on each pane in the ***ICP-MS Data Analysis*** window. For details refer to the *Agilent 7700 Series ICP-MS MassHunter Workstation User's Guide* (page 41)

5.2 Click 

on the toolbar and review the data analysis parameters.

- Click 

and make sure that the ***FullQuant Analysis*** check box is selected
- Click 

and make sure that the analytes and ISTD elements are properly specified
- Under the 

tab, select ***FullQuant Outliers*** and enable the following outlier settings:

Count RSD%	Less than 10%
ISTD Recovery % (compared with CalBlk)	80 to 120%
Calibration curve fir R	0.98
Out of Calibration Curve Concentration Range	Check box

Results that do not meet these criteria will be flagged.

- Select Return to Batch-at-a-Glance. Click ***Yes*** at the dialog box: Update *Data Analysis Method?*
- Click Process Batch on the ***ICP-MS Data Analysis*** toolbar

5.3 Make sure that the internal standard signal is stable during the run (there is no trend or points that exceed the outlier criteria).

5.4 Click to display the calibration curve and to display the curve fit information. Visually observe the calibration curves for each element; check if the correlation coefficients R are 0.998 or better and the calculated concentrations for each standard are within 20% of the assigned values. If not, optimize the calibration parameters (such as curve fit equation, handling of origin and ISTD correction equation) or remove any obvious outliers.

5.5 Reprocess the batch and click to save the analysis file.

5.6 On the ICP-MS Data Analysis toolbar select ***View*** >> ***Column Setting*** >> ***Add/Remove Columns…***Choose the parameters as follows:

Select Columns From	***Show these columns in the following order:***
Sample:	Data file Acq. Date-Time Type Level Sample name
Analyte:	Concentration
ISTD	CPS CPS RSD

5.7 On the ***ICP-MS Data Analysis*** toolbar select ***File*** >> ***Export*** >> ***Export Table.*** The entire table will be exported in Excel file format. Save the Excel file to the C drive using the same name as the corresponding batch.

5.8 Save the Excel spreadsheet to ICPMSBackup$ drive in the yyyy Excel folder as "yyyymmdd_description of batch". The data in the spreadsheet can now be accessed by the analyst at his/her office computer for reporting.

5.9 Transfer raw data to the corresponding sub-directories in the ICPMSBackup$ shared drive immediately after the analysis is completed and before any reprocessing. The data are then copied to the analyst's computer for further reprocessing and reporting.

5.10 Remove the data from the local disk drive (C drive) and save them in the ICPMSBackup$ shared drive or the external hard drive every six months to ensure that the C drive has enough memory to perform all the steps of analysis.

6 DATA REPROCESSING FOR LEAD ISOTOPES ANALYSIS

6.1 Click DA Method Editor on the toolbar. Check ONLY the ***Isotopes Ratio Analysis*** checkbox; select ***None*** at the ***Interference Correction*** drop box.

6.2 Go to 4. Set up Analysis Parameters >> ***Isotope Ratio***. Click on the ***DA Method Task*** toolbar. Select Pb.

6.3 Select mass 206 at the drop down box of ***Numerator*** and masses 207 and 208 as Mass 1 and Mass 2 for ***Denominator,*** respectively**.** Check the certificate of the IR standard and type the correct values here. The isotopic composition of NIST SRM 981 is ^{206}Pb = 24.1442%, ^{207}Pb = 22.0833% and ^{208}Pb = 52.3470%

6.4 Select Return to Batch-at-a-Glance. Click ***Yes*** to the dialog box: *Update Data Analysis Method?* Click Process Batch on the ***ICP-MS Data Analysis*** toolbar.

6.5 On the ***ICP-MS Data Analysis*** toolbar select ***File*** >> ***Save as*** and save the analysis file using the same name as the batch file, adding IR. For example, name the TMB100 batch run in no gas mode on January 23, 2013, as: 20130123_TMB100_IR

6.6 On the ICP-MS Data Analysis toolbar select ***View*** >> ***Column Setting*** >> ***Add/Remove Columns…***Choose the parameters as follows:

Select Columns From	***Show these columns in the following order:***
Sample:	Data file
	Acq. Date-Time
	Type
	Level
	Sample name
Numerator:	CPS
	CPS RSD
Denominator	MBCC
	CPS
	IR
	IR RSD

6.7 On the ***ICP-MS Data Analysis*** toolbar select ***File*** >> ***Export*** >> ***Export Table.*** The entire table will be exported in Excel file format. Save the Excel file to the C drive using the same name as the corresponding batch. Follow steps 5.8 to 5.10.

7 PLASMA TURN-OFF

7.1 After completing the analysis, rinse the system with 2% HNO_3 for at least 5 minutes, followed by rinsing with DDW for 5 minutes.

7.2 Select ***Plasma OFF*** from the top panel of ***Instrument Control***. A dialogue box will appear confirming if you wish to turn off the plasma; click "***Yes***". Release the peristaltic pump tubing.

7.3 Close all gas line valves.

7.4 Turn off the chiller if the instrument will not be used for 3 days or longer.

8 AUTOSAMPLER TURN-OFF

8.1 Turn off the autosampler at the end of the analysis by turning off the power switch located at the back of the autosampler.

8.2 Release the Tygon tubing at the autosampler's peristaltic pump.

9 INSTRUMENT SHUT DOWN FOR MAINTENANCE

9.1 Shut down the instrument when maintenance inside the vacuum chamber is to be performed, or when the instrument will not be used for a prolonged period of time, e.g. 2 months and longer.

9.2 To shut down the instrument, THE VACUUM MUST BE TURNED OFF FIRST and THE ARGON SUPPLY MUST BE ON.

9.3 When the LED on the top right side of the top cover of the ICP-MS stops flashing (usually takes a few minutes), turn off the power by pushing the power switch located at lower right of the instrument. Unplug the power supply if necessary.

10 APPLICABLE DOCUMENTS

6.10/*.*/M "Determination of Trace Elements in Aqueous Extracts of Airborne Particulate Matter and Other Aqueous Solutions by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)"

6.11/*.*/M "Determination of Near-Total Trace Elements in Airborne Particulate Matter by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)"

6.13/*.*/M "Determination of Trace Elements and Lanthanoids in Airborne Particulate Matter by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS)

SWP-001/*.*: Safe Working Procedures and Policies

11 REVISIONS

May 2013: Author, Valbona Celo. New document SOP 6.22/1.0/S

REFERENCES

Agilent Technologies, *Agilent 7700 Series ICP-MS MassHunter Workstation User Guide*, Rev.A, October 2011

Agilent Technologies, *Agilent 7700/7500 Series ICP-MS ASX-500 Series Autosampler*, Rev. A, June 2009

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Approved by: Ewa Dabek

Title: Head, Particulate Characterization Unit

Appendix A

Table A1. Typical Range for 7700x instrument parameters at Standby Mode

Meter	Typical Range	Recommended range
Ar Gas Delivery Pressure	720 - 730 kPa	500 to 700 kPa
Backing Pressure	1 to 2 Pa	0.3 to 5 Pa
Analyzer Pressure	1×10^{-5} to 5×10^{-5} Pa	1×10^{-5} to 6×10^{-4} Pa

Table A2. Typical Range for 7700x instrument parameters at Analysis Mode

Meter	Typical Range	Recommended Range
Ar Gas Delivery Pressure	720 - 730 kPa	500 to 700 kPa
Forward power	1400 to 1600 W	700 to 1600 W
Reflected power	< 5 W	< 20 W
Cooling water flow rate (RF/WC/IF)	1.0 to 2.0 L/min	1.0 to 2.0 L/min
Interface/Backing pressure (IF/BK)	250 to 300 Pa	250 to 490 Pa
Analyzer Pressure in no gas mode	1×10^{-4} to 2×10^{-3} Pa	1×10^{-4} to 2×10^{-3} Pa
Analyzer Pressure in gas mode	5×10^{-4} to 1×10^{-3} Pa	NA

Appendix B

Table B1. Typical Values of Tuning Parameters

Parameter	Typical Values (no gas mode)
RF Power (W)	1450 to 1550 (1600 for HMI mode)
Sampling Depth (mm)	6.5 to 7.5 (10 for HMI mode)
Carrier Gas (L/min)	0.8 to 0.9 (0.35 for HMI mode)
Dilution Gas (L/min)	0.2 to 0.4 (Makeup gas 0.60 for HMI mode)
Nebulizer pump (rps)	0.10
S/C Temp (°C)	2
Extract 1 (V)	0 to 2
Extract 2 (V)	-200 to -195
Omega Bias (V)	-90 to -80
Omega Lens (V)	8 to 11
Cell Entrance (V)	-30 to -40
Cell Exit (V)	-45 to -35
Deflect (V)	12 to 13 (-0.8 in cell gas mode)
Plate bias (V)	-55 to -50 (-53 in cell gas mode)
OctP RF (V)	150 to 155
OctP Bias (V)	-6 (-18 in cell gas mode)
QP Bias (V)	-3 (-15 in cell gas mode)

Table B2. Typical Values of Sensitivity (CPS)[(a)]

No gas mode		H_2/He mode		HMI mode	
^{7}Li	10 to 15 × 10^3	^{56}Fe/^{51}V	< 1000	^{138}Ba	5 to 10 × 10^3
^{89}Y	20 to 40 × 10^3	^{89}Y	10 to 20 × 10^3	^{162}Dy	3 to 5 × 10^3
^{205}Tl	10 to 20 × 10^3			^{175}Lu	8 to 15 × 10^3

[(a)]Signal of a solution containing 1 µg/L of each analyte and integration time 0.1 sec

Table B3. Example of Data Acquisition parameters

Isotope	Integration Time (sec/mass)			Interference Equation & Notes
	No gas mode	H_2 Mode	He Mode	
^{9}Be	0.30	--	--	Interference Equation: $^{208}Pb = {}^{206}Pb + {}^{207}Pb + {}^{208}Pb$ Notes: (a) If no interference is suspected, Cr can be determined in no gas mode, where ^{52}Cr or ^{53}Cr is acquired. (b) Lead isotopes ^{206}Pb and ^{207}Pb are acquired for the use of interference equation. ^{208}Pb is reported for quantitative determination of Pb (c) Bi can be determined if not added to the mixed internal standard solution (d) Lanthanoids (La to Lu) are analyzed in HMI mode with an Integration time 0.30 sec/mass
^{27}Al	0.15	--	--	
^{47}Ti	0.30	--	--	
^{51}V	0.15	--	0.30	
^{52}Cr	0.15[(a)]	0.30	0.30	
^{53}Cr	0.30[(a)]	--	0.30	
^{55}Mn	0.15	--	--	
^{56}Fe	--	0.15	0.15	
^{59}Co	0.09	--	--	
^{60}Ni	0.15	--	--	
^{65}Cu	0.15	--	--	
^{66}Zn	0.15	--	--	
^{69}Ga	0.30	--	--	
^{72}Ge	0.30	--	--	
^{75}As	0.30	--	0.30	
^{78}Se	--	0.60	--	
^{85}Rb	0.30	--	--	
^{88}Sr	0.15	--	--	
^{89}Y (ISTD)	0.09	0.15	0.15	
^{90}Zr	0.30	--		
^{93}Nb	0.30	--	--	
^{95}Mo	0.15	--	--	
^{107}Ag	0.15	--	--	
^{111}Cd	0.15	--	--	
^{115}In (ISTD)	0.09	0.15	0.15	
^{118}Sn	0.30	--	--	
^{121}Sb	0.30	--	--	
^{137}Ba	0.30	--	--	
^{139}La	0.30	--	--	
^{140}Ce	0.30	--	--	
^{165}Ho (ISTD)	0.09	0.15	0.15	
^{182}W	0.30	--	--	
^{205}Tl	0.15	--	--	
^{206}Pb	0.30 [(b)]	--	--	
^{207}Pb	0.30 [(b)]	--	--	
^{208}Pb	0.30 [(b)]	--	--	
^{209}Bi	0.15[(c)]	--	--	
^{238}U	0.30	--	--	

Table B4. Proposed program for the nebulizer peristaltic pump operation

	Time (sec)	Speed (rps) Nebulizer pump	Vial#
Pre Run			
Sample uptake	50	0.3	Sample
Stabilizing	25	Tune parameter	Sample
Acquisition			
Speed		Tune parameter	Sample
Post Run			
Probe Rinse (Sample)	25	0.3	Rinse Port
Probe Rinse (std)	25		Rinse Port
Rinse 1	30	0.1	2

Appendix C

Table C1. Proposed sequence for the analysis of water-soluble metals in PM using the FullQuant method (Method 6.10/*.*/M)

Sample Type	Sample Name	Vial#	Level	Dilution
CalBlk	Calblk	1101	Level 1	1
CalStd	0.1ppb	1102	Level 2	1
CalStd	0.5ppb	1103	Level 3	1
CalStd	1ppb	1104	Level 4	1
CalStd	2ppb	1105	Level 5	1
CalStd	5ppb	1106	Level 6	1
CalStd	10ppb	1107	Level 7	1
CalStd	20ppb	1201	Level 8	1
CalStd	50ppb	1202	Level 9	1
CalStd	100ppb	1203	Level 10	1
Sample	Blank1	2		1
Sample	Blank2	2		1
Sample	LCS_WS	1204		1
Sample	CS_WS	1205		1
Sample	Blank3	2		1
Sample	ICPblk1	2101		1
Sample	ICblk-R1	2102		1
Sample	ICblk-R2	2103		1
Sample	ICblk-M1	2104		1
Sample	ICblk-M2	2105		1
Sample	Samples 1-5	2106-2110		1
Sample	LTMxxx-1	1206		1
Sample	Samples 6 - 10	2111-2203		1
Sample	Duplicate 1	2106		1
Sample	Samples 11 - 16	2204-2208		1
Sample	MTMxxx-1	1207		1.07
Sample	Blank4	2		1
Sample	Samples 16-25	2209-2306		1
Sample	Spiked sample 1	2307		1
Sample	Blank5	2		1
Sample	Samples 26-30	2308-2311		1
Sample	ICPspk-1	2312		1
Sample	ICspk1-1	2401		1
Sample	ICspk2-1	2402		1
Sample	MDL-Spk	2403		1
Sample	HTMxxx-1	1208		1.07
Sample	Blank6	2		1
Sample	VS_1ppb	1104		1
Sample	VS_10ppb	1107		1
Sample	VS_100ppb	1203		1
Sample	Blank7	2		1

Table C2. Proposed sequence for the analysis of near-total metals and lead isotopes in PM using the FullQuant method (Method 6.11/*.*/M and 6.13/*.*/M)

Sample Type	Sample Name	Level	Vial #	Dilution
CalBlk	Calblk	1	1101	1
CalStd	05ppb	2	1102	1
CalStd	1ppb	3	1103	1
CalStd	2ppb	4	1104	1
CalStd	5ppb	5	1105	1
CalStd	10ppb	6	1106	1
CalStd	20ppb	7	1107	1
CalStd	50ppb	8	1201	1
CalStd	100ppb	9	1202	1
CalStd	200ppb	10	1203	1
Sample	981-1		1306	1
Sample	Blank2		2	1
Sample	Blank3		3	1
Sample	LCS-NT		1204	1
Sample	Blank4		2	1
Sample	Vialblk1		2101	1
Sample	Reagblk1		2102	1
Sample	Methblk1		2103	1
Sample	982-1		1307	1
Sample	Sample 1 to 5		2104 to 2201	1
Sample	LTMxxx		1205	1.04
Sample	Sample 6 to 10		2201 to 2205	1
Sample	Sample 1duplicate		2104	1
Sample	981-2		1306	1
Sample	Sample 11 to 16		2206 to 2304	1
Sample	982-2		1307	1
Sample	MTMxxx		1206	1.04
Sample	Sample 17 to 22		2305-3102	1
Sample	981-3		1306	1
Sample	Sample 22 to 27		3103 to 3107	1
Sample	Sample 4 spiked		4102	1
Sample	HTMxx		1207	1.04
Sample	982-3		1307	1
Sample	Blank6		2	1
Sample	Sample 27 to 32		3201 to 3205	1
Sample	Blank7		2	1
Sample	MDLspk		3206	1
Sample	Spike2		3207	1
Sample	981-5		1306	1
Sample	VS1 ppb		1103	1
Sample	VS100 ppb		1202	1
Sample	Blank9		2	1

Table C3. Proposed sequence for analysis of lanthanoids in PM using the FullQuant method (Method 6.13/*.*/M)

Type	Sample Name	Vial #	Level	Dilution
CalBlk	Calblk	1101	1	1
CalStd	0.5ppb	1102	2	1
CalStd	1ppb	1103	3	1
CalStd	2ppb	1104	4	1
CalStd	5ppb	1105	5	1
CalStd	10ppb	1106	6	1
CalStd	20ppb	1107	7	1
CalStd	50ppb	1201	8	1
CalStd	100ppb	1202	9	1
CalStd	200ppb	1203	10	1
Sample	Blank2	2		1
Sample	LCS-LA-1	1204		1
Sample	Blank3	2		1
Sample	Vialblk1	2101		1
Sample	Reagblk1	2102		1
Sample	Methblk1	2103		1
Sample	Sample 1 to 5	2104 to		1
Sample	CS-LA-1	1205		1
Sample	Sample 6 to 10	2201 to		1
Sample	Sample 1d	2104		1
Sample	Sample 11 to 16	2206 to		1
Sample	LCS-LA1	1204		1
Sample	Sample 17 to 22	2305-		1
Sample	Sample 22 to 27	3103 to		1
Sample	Sample 4 spiked	4102		1
Sample	CS-LA-1	1205		1
Sample	Blank4	2		1
Sample	Sample 27 to 32	3201 to		1
Sample	Blank5	2		1
Sample	MDLspk	3206		1
Sample	Spike2	3207		1
Sample	Sample 33 to 35	3306 to		1
Sample	NIST1648-1	4102		10
Sample	BCR677-1	4103		10
Sample	Blank6	2		1
Sample	VS1 ppb	1103		1
Sample	VS10 ppb	1106		1
Sample	VS100 ppb	1202		1
Sample	Blank7	2		1