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General Operating Procedure for:

NexION 350x ICP-MS

Department of Health and Environmental Sciences

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Contents

1. Purpose

The purpose of this GOP is to regulate the operation, usage and maintenance of ICPMS.

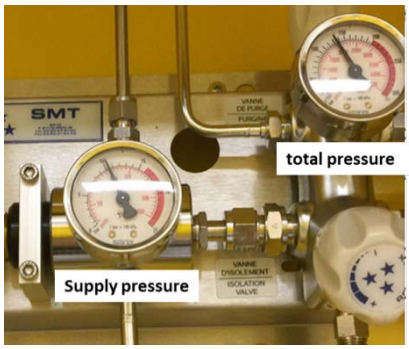
2. Scope

This GOP applies to all personnel using and managing ICPMS.

3. Procedure

3.1 Preparation Before ignite the plasma

3.1.1 Check gas pressure and the previous records in logbook. Record the current pressure in the log book. Make sure the gas is open (3 cylinders each time). And:



- Ar: Total pressure: >5MPa. Gas supply pressure: 90~110 psig.
- Turn the push rod to proper side. (Up means left side and down means right side). Keep an eye on the gas during your experiment; remember to turn the push rod to another side when the gas you are using is less than 2 Mpa.
- Turn the tag on the gas cylinder to the right status. (full, empty or in use.)
- If the gas is not enough for your experiment, please contact Yili Cheng (158-9558-5536).

If using KED/DRC mode, make sure collision gas (He) or reaction gas is connected to the machine. Open the valve of gas cylinder and check the gas supply pressure, it should be 10-20psi, if not, report to Yili Cheng.

3.1.2 Check the ventilation system, obvious shaking of the vent tube should be seen.

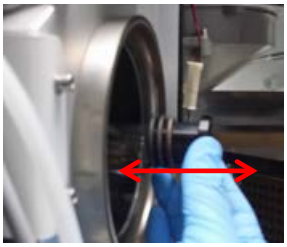
3.1.3 The room temperature should be under 25^{O}C and humidity 30% to 70%. If not, contact Yili.

3.1.4 Check the torch and cones.

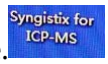
3.1.4.1 Open the cap of ICP-MS and set up the support. 

Press on the side panel and Open the torch box.

3.1.4.2 Check the torch and take out the cones.



3.1.4.3 If the cones are dirty (**covered by obvious carbon or salt deposits**), wash it with the cone washing solution (first 2% HNO3 and then ultrapure water). Make sure they are clean, dry and correctly installed. **(It is suggested that you check and wash the cones a day before your experiment to make sure they are totally dried.)** Then close the torch-box and the cap.

3.1.5 Open the software. 

3.1.6 Vacuum check: less than 2*10^{-6} Torr and show “ready”. If not, stop use it and report to Yili.

3.1.7 Turn on the cooling water.

Make sure water volume is more than 70-80% of the container volume, Temperature is 18±2^{o}C and pressure is 45-65 psi.

3.1.8 Correctly install and tighten the tubes of peristaltic pumps.

Click Devices → peristaltic →fast, observe the tube connection. Make sure the liquid in and out are right. The rotation of the pump is anticlockwise.

If using auto sampler, correctly install the tubes for auto sampler. The rotation of the pump is also anticlockwise.

3.2 Creating method and analyzing

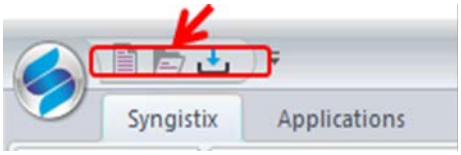
3.2.1 Stop the peristaltic pumps then ignite the plasma: click “start” under plasma on the instrument page.

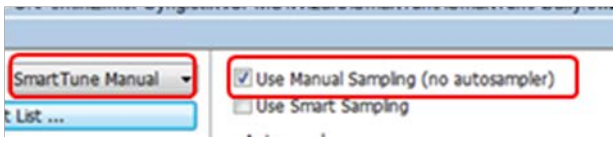
3.2.2 Wash with 2% HNO_3 for 15 min. (you can use this time to create the analysis method).

3.2.3 Daily performance check

Click SmartTune. 

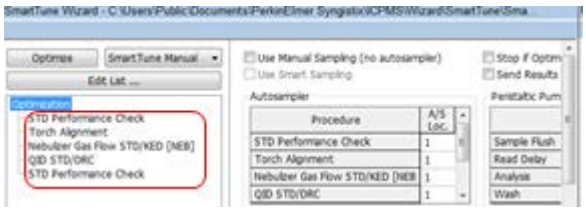
Click the File open button, and choose “Smart tune daily. swz ”. The route: “C: \PerkinElmer Syngistix \ ICPMS \Wizard \Smart Tune\ Smart Tune Daily.swz [read only]”.





Pump in the setup solution (NO: N8145051), click “optimize” to one click optimize. It will show “passed” if it is OK. Record the result in the ICP-MS LOGBOOK.

If failed, it will run the item below orderly to optimize separately.

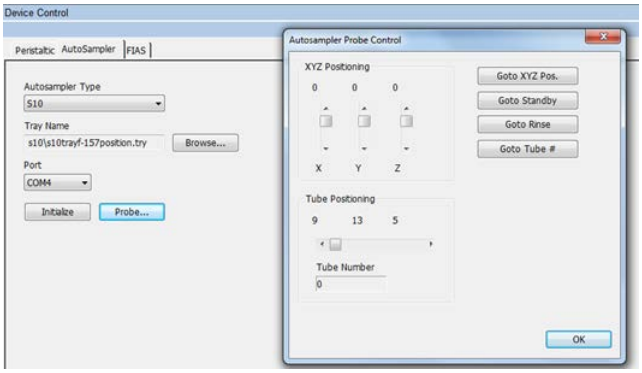


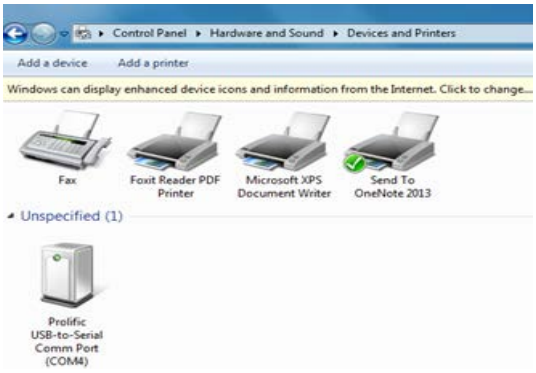
If still failed, report to Yili Cheng.

3.2.4 Check the auto sampler (if use auto sampling)

Power on the auto sampler and click 

, **AutoSampler**. Click initialize to initialize the auto sampler. If failed, check the port through “control panel> hardware and sound> devices and printers.”



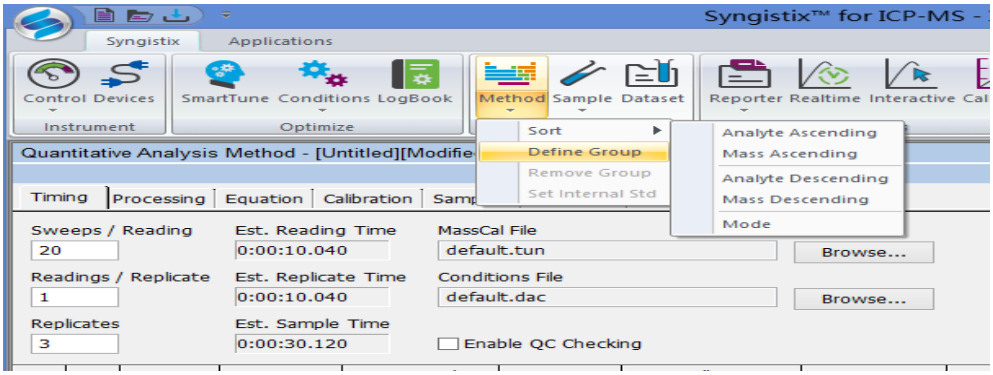


3.2.5 Establishment of analysis method

- Click 

→new method → quantitative analysis.
- **Timing** → Sweeps/Reading 20, Reading/ Replicates 1， Replicates 3.
- Set internal standard: Choose the analyte and internal standards elements, click 

→define group. Then choose the internal standard element and set internal std. (You may need to sort the analyte by mass ascending or mode before define group).



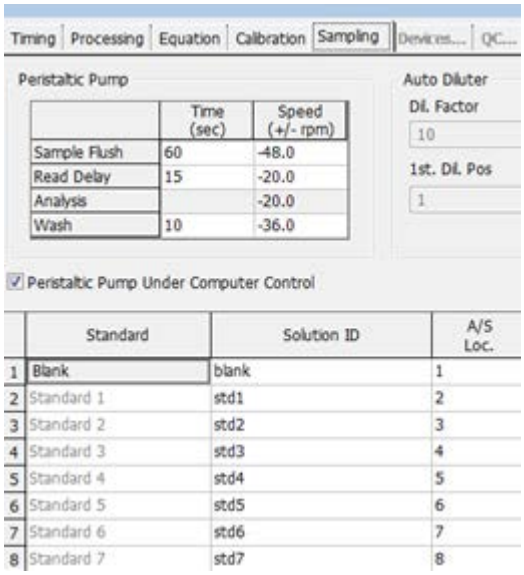
- Different testing mode. STD/KED or STD/DRC could be used together. For KED mode, cell gas A will be auto filled; for DRC mode, cell gas A and

	Int Std	Analyte	Mass (amu)	Scan Mode (*)	MCA Channels	Dwell Time per AMU (ms)	Integration Time (ms)	Corrections	Mode (*)	Cell Gas A	Cell Gas B	RP a	RP q
1		Be	9.0122	Peak Hopping	1	50	1000		Standard	0	0	0	0.25
2		Sc	44.9559	Peak Hopping	1	50	1000		Standard	0	0	0	0.25
3		Mg	23.985	Peak Hopping	1	50	1000		KED	4.5	0	0	0.25
4		K	38.9637	Peak Hopping	1	50	1000		KED	4.5	0	0	0.25
5		Fe	56.9354	Peak Hopping	1	50	1000		KED	4.5	0	0	0.25
6		Sc-1	44.9559	Peak Hopping	1	50	1000		KED	3	0	0	0.25
7		Al	26.9815	Peak Hopping	1	50	1000		KED	3	0	0	0.25
8		Ti	46.9518	Peak Hopping	1	50	1000		KED	3	0	0	0.25
9		V	50.944	Peak Hopping	1	50	1000		KED	3	0	0	0.25

RPq will be auto filled after you have send the parameter to method; you can also edit these parameter by your own. Please refer to appendix for more information.

- **Equation** some of the corrections should be deleted when using KED mod; all corrections should be deleted when using DRC mod.
- **Calibration** Choose calibration type, set the curve type and std concentration. (don't set the concentration for internal standard element)
- **Sampling** Sample flush 35ms, read delay 15ms and wash 10ms. This need be changed when you changed the length of sampling tubes.

  If use auto sampler, fill in the location of your blank and stands.



- **Save method.**

3.2.6 Click 

, click  , build new file to save your data.

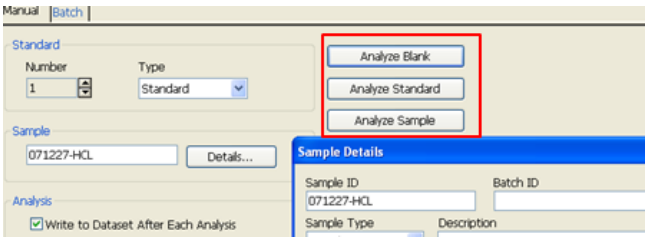
3.2.7 Separate the two sampling tube, and put one into internal standard solution, another one into sample container.

3.2.8 Get the calibration curve of given elements.

- Click calibview→ clear calibration and blank.
- Open the method which you have just saved.

  If using manual injection.

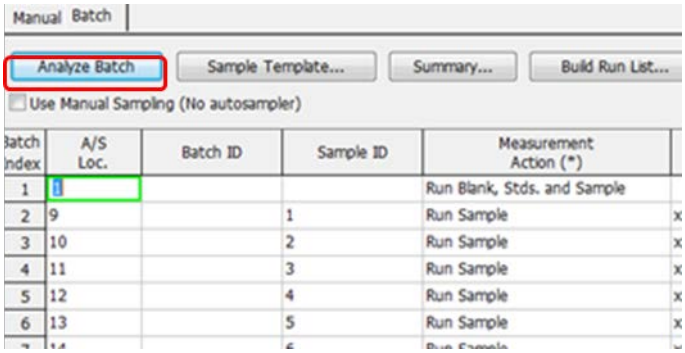
- Choose “write to dataset after each analysis”.
- First analyze blank, Click “Analyze Blank”
- Analyze standard solution in each concentration according to previous set. Change the “Number” then Click “Analyze Standard”. Get the calibration curves and examine them.



3.2.9 Sample analyze: Input the label name of each sample, click “Analyze Sample”

Wash with 2% HNO_3 after each sample.

If using auto sampler, fill in the table as follow, edit the wash override according to your samples. Choose the samples waiting for analysis, and click “analyze batch”.



Batch Index	A/S Loc.	Batch ID	Sample ID	Measurement Action (*)	
1	8			Run Blank, Stds. and Sample	
2	9		1	Run Sample	x
3	10		2	Run Sample	x
4	11		3	Run Sample	x
5	12		4	Run Sample	x
6	13		5	Run Sample	x

Click reporter or realtime to see your data.

Caution:

- ***The RSD for most elements should be less than 5%. (Elements with small intensity could be higher.) If not, change the flush time. If still not, report to Yili Cheng.***
- ***Test a standard (as a sample) after 15 samples.***
- ***Stop to test your sample if the gas pressure is less than 2.5 MPa and ask Yili to order new gas.***
- ***Make sure there is enough internal standard solution during measurement.***
- ***Check the stability of intensity of internal standard elements.***

3.2.10 After all measurement, you can save your data by clicking “export data” in the item of “Report”

3.3 Turning off the machine

3.3.1 Wash with 2% HNO_3 for 5 min and ultrapure water for 10 min, take the pump tube out of solution to totally pipe the liquid out the machine.

3.3.2 Click “stop” under plasma on the instrument page to stop the plasma.

3.3.3 Release the pump tubes and the regulating knob. Then put the two tubes into ultrapure water. (If using auto sampler, also release those for auto sampler.)

3.3.4 Record the end time and Ar pressure.

3.3.5 Turn off the water chiller (**5 min** after the plasma has been stopped).

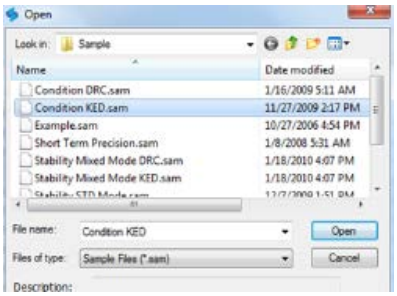
3.3.6 Turn off the computer’s monitor.

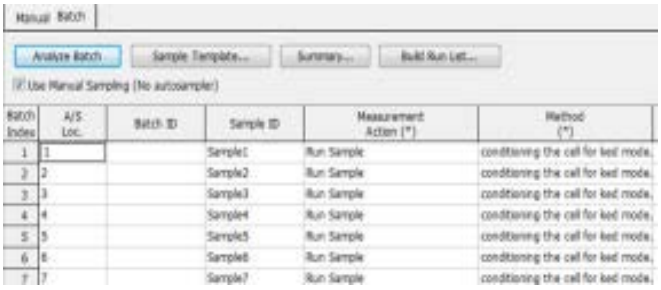
4. Appendix

4.1 Conditioning the cell for KED/DRC mode

(This must be done after change the cell gas or restart ICPMS).

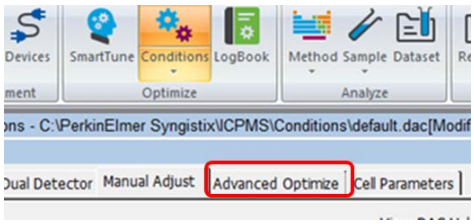
- Click “sample”, click the file open button, and choose “condition KED.sam” or “condition DRC. sam”.
- Use ultrapure water as sample, choose “use manual sampling” and click “analyze batch”. 2 samples are enough for conditioning the cell. (It costs about 40 minutes for each sample)



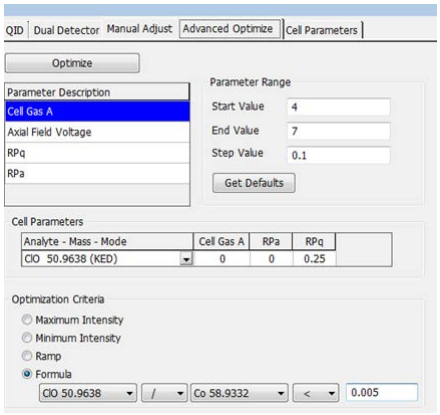


4.2 KED optimization

- Click “method”, click the file open button, and choose “KED optimize. mth”.
- Click “conditions”, click “advanced optimize”.



- Choose cell gas A, start value 4, end value 7, step value 0.1. Optimization criteria use “formula”, ClO /Co < 0.005.



- Pump in KED optimize solution (No: N8145052). Click “optimize ”.
- Save the condition. Then it will autofill the parameter for KED mode when create the method.

4.3 DRC optimization

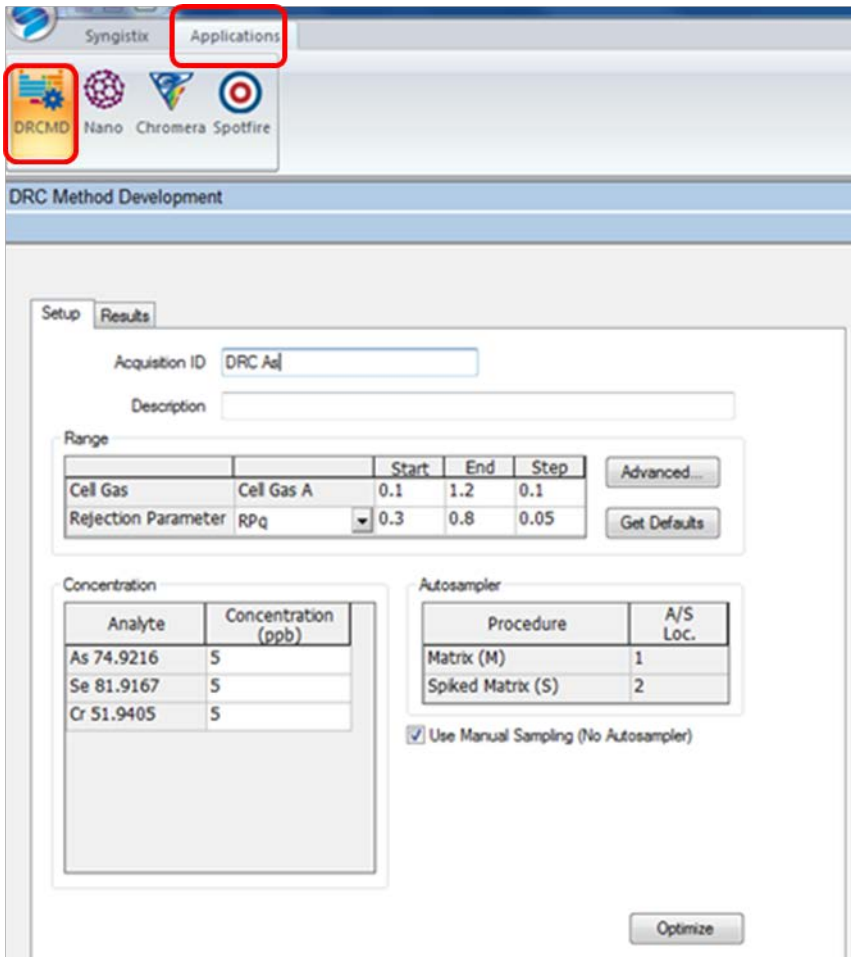
- Click method, new, create a data only method. Sweeps/Reading 20, Reading/ Replicates 1，Replicates 3.

	Analyte	Begin Mass (amu)	End Mass (amu)	Scan Mode (*)	MCA Channels	Dwell Time per AMU (ms)	Integration Time (ms)	Corrections	Mode (*)	Cell Gas A
1	As	74.9216		Peak Hopping	1	50	1000		DRC	
2	Se	81.9167		Peak Hopping	1	50	1000		DRC	
3	Cr	51.9405		Peak Hopping	1	50	1000		DRC	
4										
5										

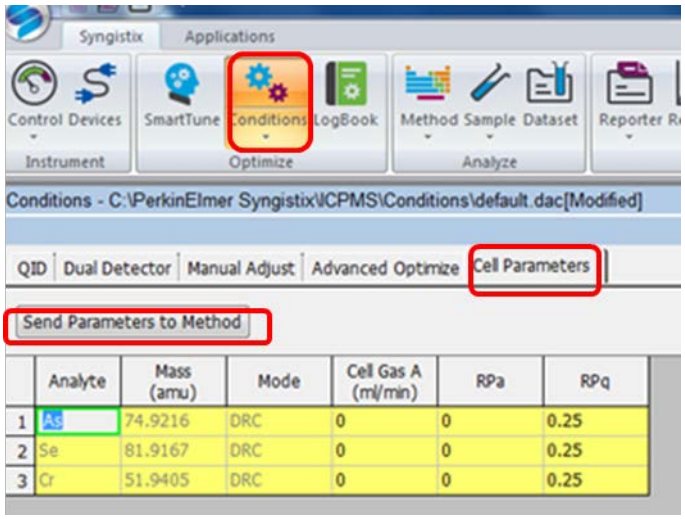
- Applications > DRCMD

  Edit the acquisition ID. Set the range for cell gas and RPq as follow. Set the concentration for spiked matrix. Choose use manual sampling if so.

- Prepare the matrix and the spiked matrix. Click “optimize” and analyze these samples following the subsequent prompts.



- Conditions> cell parameters, click save.



- Open your testing method.
- Go back to conditions and click “send parameters to method”.

4.4 Recommend test mode

sample	element	Mass	mode	gas A
Environmental samples. (water, soil, sludge)	K	39	STD/KED	He
	Ca	43	STD/KED	He
	V	51	STD/KED	He
	Cr	52	STD/KED	He
	Mn	55	STD/KED	He
	Fe	56	STD/KED	He
	Ni	60	STD/KED	He
	Co	59	STD/KED	He
	Cu	63	STD/KED	He
	Zn	66	STD/KED	He
	As	75	STD/DRC/KED	CH4/He
	Se	80/82	STD/DRC/KED	CH4/He
Food	K	39	STD	
	Ca	43/44	STD	
	V	51	DRC	NH3
	Cr	52	DRC	NH3
	Mn	55	DRC	NH3
	Fe	56	DRC	NH3
	Ni	60	STD	
	Co	59	STD	
	Cu	63	STD	
	Zn	66	STD	
	Se	78	DRC	NH3
	AsO	91	DRC	O2

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