# Agilent ICP-MS Interactive Troubleshooting tool for Low sensitivity

Rev. B 2017, Janyary, 20

#### **Preconditions**

- 7900, 7800, 7700, or 8800 ICP-MS is used.
- Sample introduction type is "PeriPump". If you are using a special configuration such as LC/GC integration or ISIS, setup the instrument with standard configuration.
- You have the "Hardware Maintenance Manual" for your instrument. Some details of the operations are not in this tool. You should refer to the manual.

# Legends

Note

Notes or tips that help troubleshoot.

Reference

You need to refer to another document for detail of the steps.

Good

Good example of situation.

Bad

Bad example of situation.

Did the ion lens test pass? ←Questions are displayed in blue you need to click

buttons such as

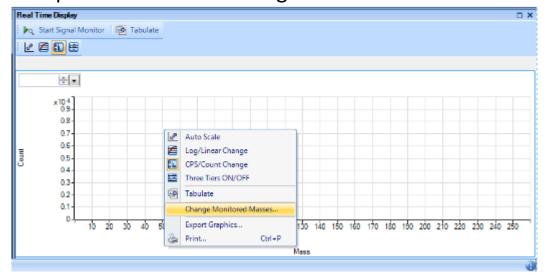
Based on your answer the tool will navigate you to next step.

or

<Previous

## 1.1.1 Signal monitor using mass 12, 56, and 80

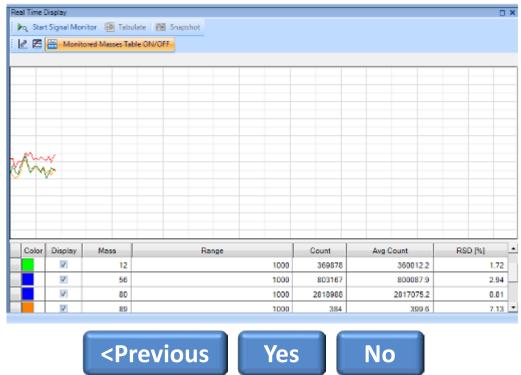
- (1) Ignite plasma
- (2) Introduce pure water
- (3) Go to "Queue" Pane→"Real Time Display"→"Start Signal Monitor"
- (4) Right click on the pane and select "Change Monitored Masses..."





## 1.1.2 Signal monitor using mass 12, 56, and 80

- (5) Add measured mass 12, 56, and 80.
- (6) Click "Signal Monitor" and check counts of 12, 56, and 80.
- (7) Are all counts of 12, 56, and 80 zero?



## 1.2.1 Adjust res/axis using mass 12, 56, and 80

(1) Go to "Startup" Pane and set the following. "Select Custom Setting" = OFF.

"Monitored Masses" = 12, 56, 80 (Just type "12 56 80" in the box).

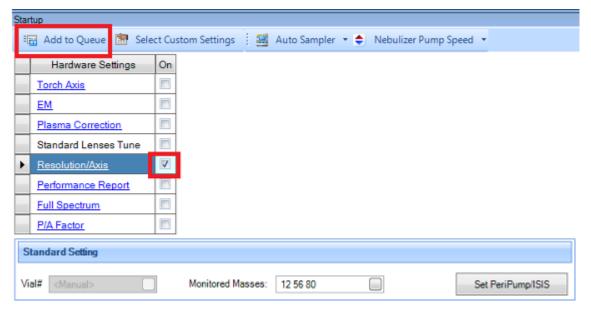
Sta	rtup										
	Add to Queue	🛅 Selec	t Cus	tom Settings	**	Auto Samp	er 🕶 💠	Neb	ulizer Pum	p Speed	•
	Hardware Set	tings	On								
	Torch Axis		7								
	<u>EM</u>		<b>V</b>								
٠	Plasma Correction		V								
	Standard Lenses Tune										
	Resolution/Axis		<b>V</b>								
	Performance Rep	oort									
	Full Spectrum										
	P/A Factor										
Standard Setting											
Vi	al# <manual></manual>	Monitored Ma	sses	12 56 80				5	Set PeriPump/ISIS		



## 1.2.2 Adjust res/axis using mass 12, 56, and 80

(2) Check "On" for "Resolution Axis". Uncheck the others. Click "Add to Queue" to Execute

startup.

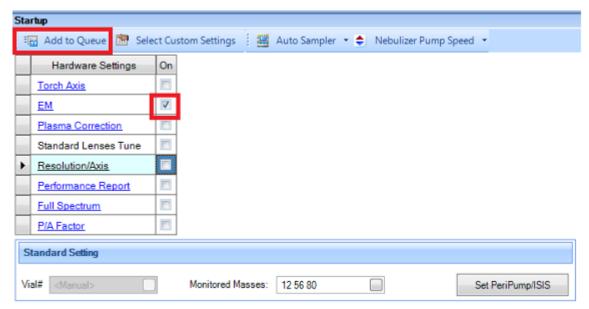


(3) Did the startup task end without error?



## 1.3.1 Adjust EM voltage

(1) In the same manner, check "On" for "EM". Uncheck the others. Click "Add to Queue" to Execute startup.

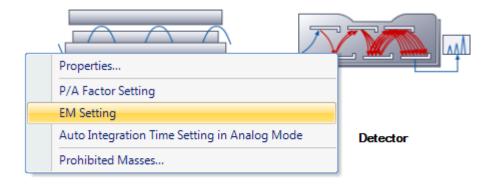


(2) Did the startup task end without error?



## 1.4.1 Check EM voltage

(1) Go to "Hardware" Pane. Right click on "Detector" and select "EM Setting" from the menu.





## 1.4.2 Check EM voltage

(2) Confirm optimized value of Analog HV, Pulse HV

_			Auto Sampler		
	Parameter	Current Value	Unit		
٠	Discriminator	3.0	mV		
	Analog HV	2350	V		
	Pulse HV	950	V		

(3) Do any exceed the values below?

Analog HV: 3300 V

Pulse HV: 1800 V

If exceeded, your EM has come to the end of it's life. You need to replace.

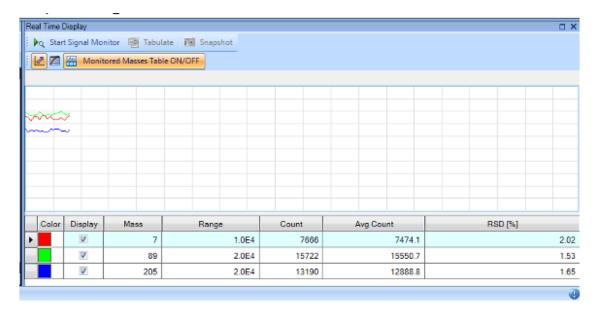


#### 1.5.1 Signal monitor using tune solution

(1) Introduce tune solution.

(2) Go to "Queue" Pane  $\rightarrow$  "Real Time Display"  $\rightarrow$  "Start Signal Monitor" and acquire signal of

mass 7, 89, 205.



(3) Are all counts of 7, 89, and 205 high enough?



1.6.1

(1)Do you use gas modes?

#### 1.7.1 Check sensitivity for gas modes

- (1) Introduce tune solution.
- (2) Open the batch in which you intend to use the gas modes.
- (3) Go to "Batch" pane  $\rightarrow$  "Tune" tab.
- (4) Run "Start Signal Monitor" for all gas modes.
- (5) Are all counts of 59, 89, and 205 high enough?

#### 1.8.1 Check aerosol

#### (1) Can you see aerosol from spray chamber?



Good You can see aerosol flowing.



Bad You can see no aerosol.

Note The aerosol is subtle, especially when micro flow nebulizer is in use. If it is not clear, regard it as "No".

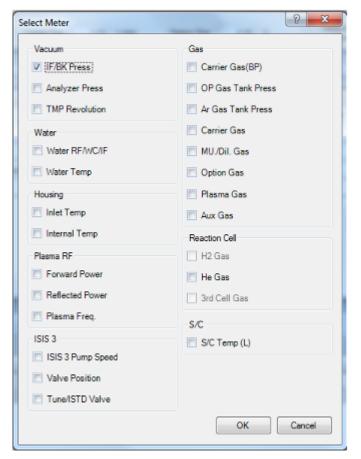
Yes

No or not clear

## 1.9.1 Check IF/BK pressure

(1) Select "View" → "Meters" in main menu of ICP-MS Instrument Control.

(2) Check "IF/BK Pressure"





## 1.9.2 Check IF/BK pressure

(3) Display "Instrument Status" using "View"  $\rightarrow$  "Instrument Status".



(4) Does the IF/BK pressure satisfy the criteria below?

7700, 7800, 8800: Below 270Pa

7900: Below 170Pa

Note If you are using s-lens, check the pressure in Hot mode.



#### 1.10.1 Check cones (without high IF/BK pressure)

- (1) Turnoff plasma
- (2) Remove sampling cone and skimmer cone. For details, refer to Hardware Maintenance manual.

Reference Hardware Maintenance Manual  $\rightarrow$  "Maintenance" "Interface"  $\rightarrow$  "Sampling Cone and Skimmer Cone"  $\rightarrow$  "Removal"

## 1.10.2 Check cones (without high IF/BK pressure)

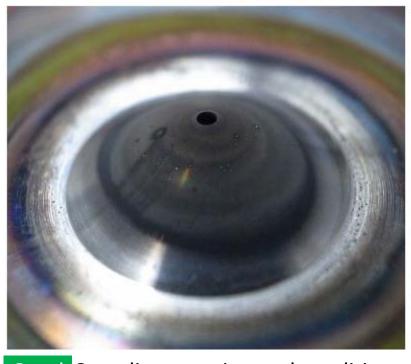
(3) Check if there are any faults in the orifice, such as clogging or distortion.



Good Skimmer cone in good condition. Skimmer cone with distorted orifice.

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# 1.10.3 Check cones (without high IF/BK pressure)



Good Sampling cone in good condition.



Bad Sampling cone with distorted orifice.

<Previous

## 1.10.4 Check cones (without high IF/BK pressure)



Bad Dirty skimmer cone whose orifice is nearly clogged.



Bad Dirty sampling cone whose orifice is nearly clogged.

<Previous

## 1.10.5 Check cones (without high IF/BK pressure)



Bad Completely clogged sampling cone due to carbon formation caused by poor option gas supply in organic solvent analysis.

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1.10.6 Check cones (without high IF/BK pressure)

(4) Any fault regarding skimmer cone or sampling cone orifice found?

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance" "Interface"  $\rightarrow$  "Sampling Cone and Skimmer Cone"  $\rightarrow$  "Check the orifice"

Note Distortions of cones are subtle, especially with skimmer cone. If it is not clear, regard it as "Yes".

<Previous Yes or not clear

#### 1.11.1 Maintain cones

(1) If distortion was found or not clear, replace it.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance" "Interface"  $\rightarrow$  "Sampling Cone and Skimmer Cone"  $\rightarrow$  "Installation"

(2) If clogging was found, clean it.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance" "Interface"  $\rightarrow$  "Sampling Cone and Skimmer Cone"  $\rightarrow$  "Cleaning the Cones"

- (3) Ignite plasma.
- (4) Introduce tune solution
- (5) Are all counts of 7, 89, and 205 high enough?

#### 1.12.1 Check cones (with high IF/BK pressure)

- (1) Turnoff plasma
- (2) Remove sampling cone and skimmer cone. For details, refer to Hardware Maintenance manual.

Reference Hardware Maintenance Manual  $\rightarrow$  "Maintenance" "Interface"  $\rightarrow$  "Sampling Cone and Skimmer Cone"  $\rightarrow$  "Removal"

## 1.12.2 Check cones (with high IF/BK pressure)

(3) Check if there are any faults in the orifice, such as clogging or distortion.

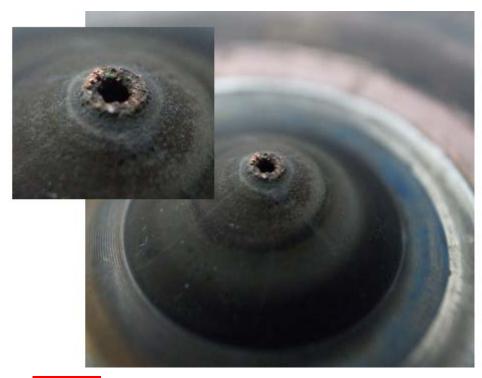


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# 1.12.3 Check cones (with high IF/BK pressure)



Good Sampling cone in good condition.



Bad Sampling cone with distorted orifice.

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## 1.12.4 Check cones (with high IF/BK pressure)

(4) Any fault regarding skimmer cone or sampling cone orifice found?

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance" "Interface"  $\rightarrow$  "Sampling Cone and Skimmer Cone"  $\rightarrow$  "Check the orifice"

Note Distortions of cones are subtle, especially with skimmer cone. If it is not clear, regard it as "Yes".

<Previous Yes or not clear

#### 1.13.1 Maintain cones

(1) If distortion was found or not clear, replace it.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance" "Interface"  $\rightarrow$  "Sampling Cone and Skimmer Cone"  $\rightarrow$  "Installation"

- (2) Ignite plasma.
- (3) Display "IF/BK Pressure" meter.
- (4) Does the pressure satisfy the criteria below?

7700, 7800, 8800: Below 270Pa

7900: Below 170Pa

Note If you are using s-lens, check the pressure in Hot mode.



#### 1.14.1

- (1) Introduce tune solution.
- (2) Are all counts of 7, 89, and 205 high enough?

#### 1.15.1

- (1) Ignite plasma
- (2) "Display "IF/BK Pressure" meter.
- (3) Does the pressure satisfy the criteria below?

7700, 7800, 8800: Below 270Pa

7900: Below 170Pa

Note If you are using s-lens, check the pressure in Hot mode.

#### 1.16.1 Check torch box

- (1) If plasma is ON, turn off.
- (2) Open torch box. Remove torch.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Torch Area"

(3) Check if torch, shield plate, shield contact are OK (from the next page).

#### 1.16.2 Check torch box

Check if shield plate is installed.



Good With shield plate.



Bad Without shield plate.

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#### 1.16.3 Check torch box

Check if shield plate is damaged.



Good Good shield plate.



Bad Damaged shield plate.

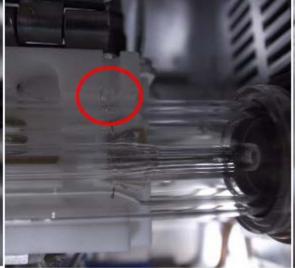
<Previous

#### 1.16.4 Check torch box

Check if shield/torch is correctly assembled.



Good Set the projection on torch in the hole of the shield plate.



Good Set the projection on torch in the notch of the holder.

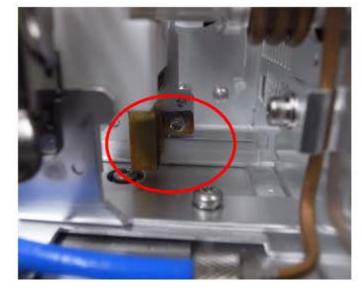


Good Good contact between shield plate and shield contact.

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#### 1.16.5 Check torch box

#### Check shield contact.



Good Shield contact in good condition.



Bad Damaged shield contact.

<Previous

#### 1.16.6 Check torch box

Check torch

Bad Clogging (No image)

Bad Significant distortion (No image)

### 1.16.7 Check torch box

(4) Is there any failure of torch, shield plate, shield contact?

## 1.17.1

(1) Is the shield contact damaged?

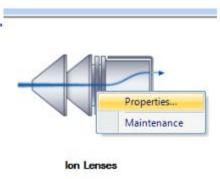


#### 1.18.1 Maintain torch box

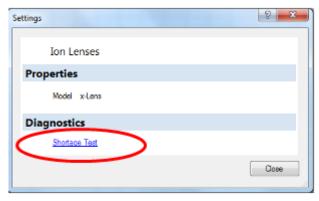
- (1) If shield plate is missing set a shield plate.
- (2) If shield plate is damaged, replace.
- (3) If the torch is clogged, clean or replace.
- (4) Ignite plasma.
- (5) Introduce tune solution.
- (6) Are all counts of 7, 89, and 205 high enough?

## 1.19.1 Do lens shortage test

(1) Go to "Hardware" pane. Right click on "Ion Lens" and select "Properties..." from the menu.



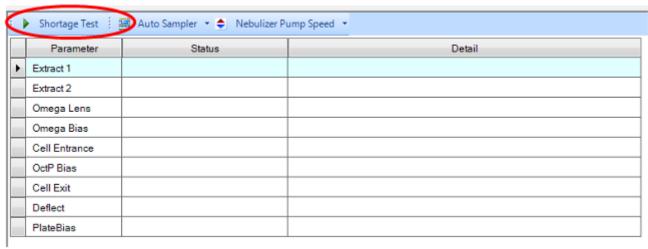
(2) Click "Shortage test" test.





# 1.19.2 Do lens shortage test

(3) Click Shortage test.



(4) Did the test pass?



### 1.20.1 Check ion lens contact

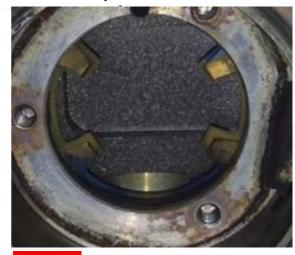
(1) Remove ion lens.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Extraction Lens-Omega Lens Assembly"  $\rightarrow$  "Removal"

(2) Check lens contacts on on the Interface assy.



Good Clean lens contacts.



Bad Dirty lens contacts.

Yes

No

### 1.21.1 Check ion lens

(1) Check ion lenses.

Bad

(2) Are they clean?

Dirty ion lenses (7500 ICP-MS).

Yes

### 1.22.1 Maintain ion lens

(1) Polish ion lenses

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Extraction Lens-Omega Lens Assembly"  $\rightarrow$  "Dissembly" and "Cleaning"

(2) Assemble ion lenses and set in the instrument.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Extraction Lens-Omega Lens Assembly"  $\rightarrow$  "Assembly"

- (3) Set skimmer cone and sampling cone to instrument.
- (4) Ignite plasma.
- (5) Introduce tune solution.
- (6) Are all counts of 7, 89, and 205 high enough?

## 1.23.1 Do lens shortage test without ion lens

(1) Remove ion lens.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Extraction Lens-Omega Lens Assembly"  $\rightarrow$  "Removal"

- (2) Do the ion lens test again.
- (3) Did the test pass?

### 1.24.1 Check ion lens assembly

(1) Disassemble ion lens to check if they are assembled correctly.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Extraction Lens-Omega Lens Assembly"  $\rightarrow$  "Disassembly"

(2) Correct?

### 1.25.1

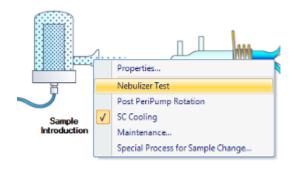
(1) Assemble ion lenses and set in the instrument.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Extraction Lens-Omega Lens Assembly"  $\rightarrow$  "Assembly"

- (2) Run ion lens test again
- (3) Did the test pass?

### 2.1.1 Check aerosol

- (1) Turn off plasma.
- (2) Go to "Hardware" pane. Right click on "Sample Introduction" and select "Nebulizer Test" from the menu.



- (3) Click "Nebulizer Test" in the toolbar.
- (4) If the test passed, go to (5), otherwise go to (11).



### 2.1.2 Check aerosol

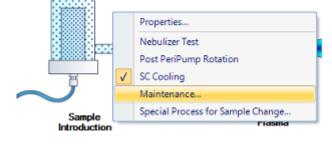
(5) Introduce pure water.

(6) Pull out the nebulizer from the spray chamber, but keep the carrier gas line and sample line connected.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Nebulizer and Spray Chamber"  $\rightarrow$  "Nebulizer"  $\rightarrow$  "Removal"

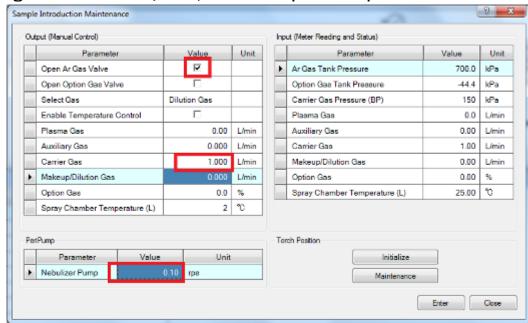
(7) Go to "Hardware" pane. Right click on "Sample Introduction" and select "Maintenance..."

from the menu.



#### 2.1.3 Check aerosol

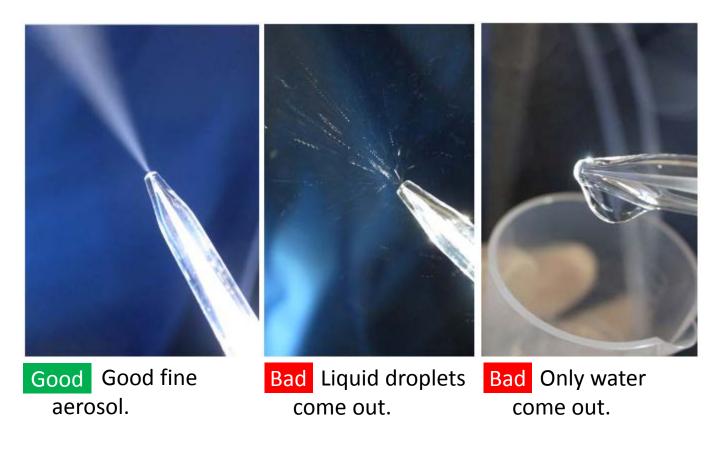
(8) Set the following. Ar valve = On, Carrier gas flow = 1.0 L/min, PeriPump = 0.1 rps.



(9) Observe aerosol (the next page).



### 2.1.4 Check aerosol



Bad Only carrier gas come out (No image).

Bad Nothing come out (No image).

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### 2.1.5 Check aerosol

(10) Set the following. Carrier gas flow = 0.0 L/min, PeriPump = 0.0 rps.

(11) Was the nebulizer test and aerosol OK?

## 2.2.1 Check peripump tubing

(1) Is there any fault on PeriPump tubing?

①[Front] From Sample

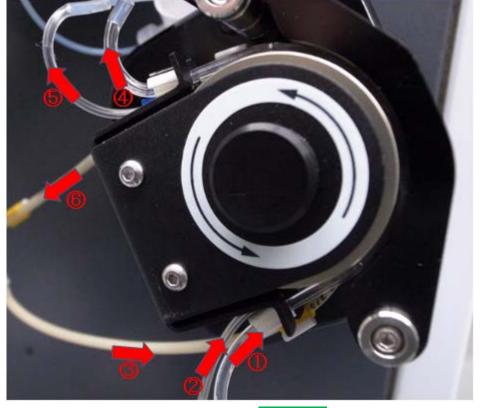
2[Middle] From ISTD

3[Back] From spray chamber

4[Front] To 3 way connector\*1

**⑤**[Middle] To 3 way connector\*1

**6**[Back] To drain



Correct tubing. Good



Yes

No

2.3.1

(1) Fix the PeriPump tubing.



## 2.4.1 Check carrier gas connection

#### (1) Check carrier gas connection.



Push carrier gas line into connector.



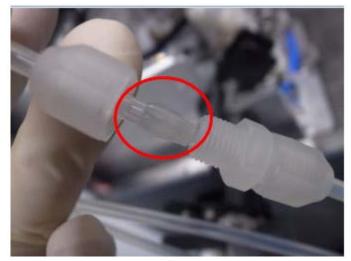
Push carrier gas line into connector (In case glass nebulizer).



Push connector toward nebulizer (In case glass nebulizer).



## 2.4.2 Check carrier gas connection



Check if ferrule is correctly installed (In case of PFA nebulizer).



Bad loose nut.

Tighten the nut, if it is loose.

(In case of PFA nebulizer).

- (2) Check the aerosol again.
- (3) Is the aerosol OK?



#### 2.5.1 Wash the nebulizer

(1) Wash the nebulizer.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Nebulizer and Spray Chamber"  $\rightarrow$  "Nebulizer"  $\rightarrow$  "Cleaning"

- (2) Check the aerosol again.
- (3) Is the aerosol OK?

### 2.6.1 Replace the nebulizer

(1) Replace the nebulizer.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$ "Nebulizer and Spray Chamber"  $\rightarrow$  "Nebulizer"  $\rightarrow$  "Removal"

- (2) Check the aerosol again.
- (3) Is the aerosol OK?

## 2.7.1

### (1) Is your nebulizer type MicroMist?

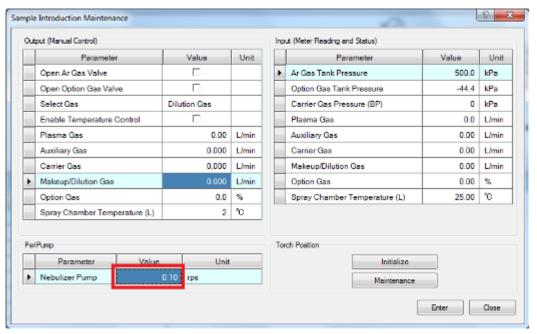


Yes

No

### 2.8.1 Check air bubble intake

- (1) Go to Sample Introduction maintenance.
- (2) Set PeriPump: 0.1 rps.



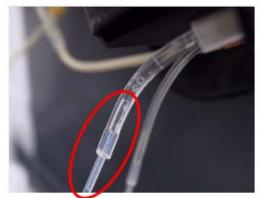


### 2.8.2 Check air bubble intake

(3) Pull out sample tube from the pure water to introduce air bubbles to the sample tube.



(4) Can you observe an air bubble is intaken and it moves toward the nebulizer?



<Previous Yes No

2.9.1

(1) Is there any leak of pure water between sample and nebulizer?

2.10.1

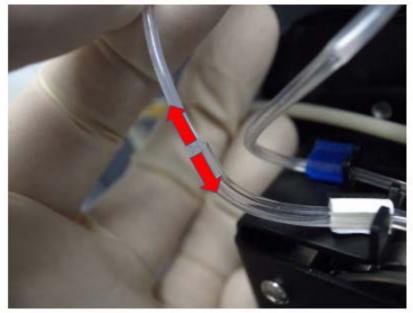
(1) Fix the leak.



# 2.11.1 Check clogging in sample tube

(1) Pull out tube between PeriPump tube and 3 way connector.

(2) Does water come out from PeriPump tube?





2.12.1

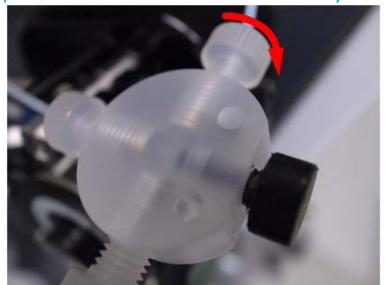
(1) Change sample line between PeriPump tube and sample.



## 2.13.1 Check clogging in 3 way connector

- (1) Connect again tubing between PeriPump tube and 3 way connector.
- (2) Loosen screw on 3 way connecter and take off the line to nebulizer.

(3) Does water come out from 3 way connector?





2.14.1

(1) Change the 3 way connector.



### 3.1.1 Check tune parameters

(1) Refer to User Guide to confirm current tune parameters are typical ones. If not, set to typical one.

Reference MassHunter Workstation User Guide  $\rightarrow$  "Appendix"  $\rightarrow$  "Recommended Values for Tuning Parameters"

(2) Is the problem solved?

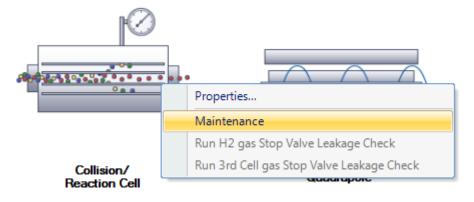
## 3.2.1 Purge cell gas line

Purge cell gas line with the problem, following these steps.

(1) Turn off plasma.

(2) Go to "Hardware" pane. Right click on "Collision/Reaction Cell" and select "Maintenance"

from the menu.





## 3.2.2 Purge cell gas line

(3) Set the flow rate maximum for the cell gas with the sensitivity problem.

🍲 Open Bypass Valve 😝 OctPole Matching 🚆 Auto Sampler 🔻 🖨 Nebulizer Pump Speed 🔻				
	Parameter	Output (Manual Control)	Input (Meter Reading and Status)	Unit
	H2 Gas Flow Rate	0.00	0.00	mL/min
	He Gas Flow Rate	12.00	12.00	mL/min
٠	3rd Cell Gas Flow Rate			%
	IF/BK Pressure		4.60E+0	Pa
	Analyzer Pressure		8.50E-5	Pa
	TMP Rotation		100.0	%
	TMP Current		0.000	Α

- (5) Wait 30 minutes.
- (4) Ignite plasma.
- (5)Go to "Batch" pane→"Tune" tab
- (6) Are all counts of 59, 89, and 205 high enough with the gas mode?



3.3.1

(1) Is the sensitivity better than before purging?

## 3.4.1 Check cell gas purity

(1) Check specification of cell gas to confirm if it meets the criteria below.

(2) Is it OK?

He: ≧99.999%

 $H_2$ :  $\geq 99.999\%$ 

Xe : ≥99.999%

NH<sub>3</sub>/He 10%/90% :99.999%

O<sub>2</sub>/Ar 20%/80% :99.999%

3.5.1

(1) Replace with correct gas.



### 3.6.1 Check Gas Clean Filter

(1) Does the indicator of the Gas Clean Filter System indicate end of life?



Yes No or No Gas Clean Filter in use

3.7.1 Maintain Gas Clean Filter

(1) Replace the Gas Clean Filter System

### 3.8.1 Do leak test for cell gas line

Perform a leak test, following the steps below.

- (1) Turn off plasma (if it is ON)
- (2) Set the pressure of the reaction gas with the sensitivity problem as below. You can do it by adjusting regulator on gas lines.

to 110kPa (16psi) for He

to 40kPa (5.8psi) for H<sub>2</sub>, 3rd, and 4th cell gas.

- (3) Close gas cylinder main valve.
- (4) Go to "Hardware" pane. Right click on "Collision/Reaction Cell" and select "Maintenance" from the menu.

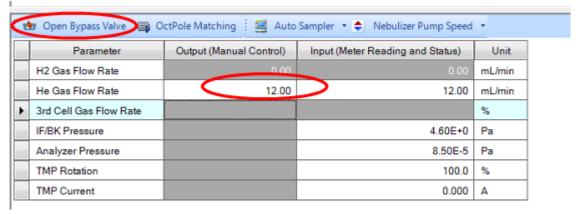


Properties...

Run H2 gas Stop Valve Leakage Check Run 3rd Cell gas Stop Valve Leakage Check

## 3.8.2 Do leak test for cell gas line

- (5) Click the open "Bypass Valve" icon on the Task Setting bar.
- (6) Confirm that the flow setting of the cell gas with the sensitivity problem is 0.
- (7) Set the flow rate maximum for the cell gas with the sensitivity problem.



## 3.8.3 Do leak test for cell gas line

- (8) Monitor the actual gas flow. Wait for the actual flow to reach less than 0.1ml/min (or less than 1%), this will take several hours depending on the length of tubing, the size of two stage regulator, and the in- line filter. If the flow becomes constant above 0.1ml/min (or above 1%) the test failed.
- (9) Set the flow to 0ml/min, and then open the gas cylinder main valve.
- (10) Set the cell gas flow to 0 and close the reaction gas screen.
- (11) Did it pass? In other words, did the actual flow reach less than 0.1ml/min in step 8?

- (1) Confirm the gas tubing. Tighten nuts if required.
- (2) Perform leak test again (same as previous).
- (3) Does it pass?

## 4.1.1 Check rotary pump

(1) Check and Maintain rotary pump.

Reference Hardware Maintenance manual  $\rightarrow$  "Maintenance"  $\rightarrow$  "Maintaining Other Parts"

# 5.1 End of procedure

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## 5.2 End of procedure

To solve the problem an engineer from Agilent is needed. Please contact Agilent using the link below.

http://www.agilent.com/en-us/contact-us/page

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