Agilent ICP-MS

ISIS 3 (Integrated Sample Introduction System)

Agilent Technologies

Notices

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Contents

1 Introduction

Overview 6

Specifications 7

- Dimensions 7
- Environment 7

2 Set Up

Hardware Setup 10

- Discrete Sampling 10

Software Setup 13

3 Operations for the Discrete Sampling

Introduction 16

Operation Procedure 17

- 1. Preparing the Hardware 17
- 2. Starting up the Instrument 18
- 3. Optimizing the ISIS Parameters 20

Example of ISIS-DS Setup 24

- ISIS-DS Initial Configuration 24
- Check List for ISIS-DS Optimization 25
- Edit Method 31
- Verify ISIS Operation Configurations 32

4 Maintenance

Replacing the Valve Rotor and Stator for the ISIS Valve 34

- Disassembly 35

Reassembly 36

Replacing the Lip Seals of the ISIS Pump 37

1 Introduction

Overview 6
Specifications 7
Dimensions 7
Environment 7

The ISIS 3 (Integrated Sample Introduction System) is a very flexible sample introduction system for the Agilent 7800/7900/8900 ICP-MS.

Overview

The fully integrated ISIS 3 system: ISIS valve; ISIS pump; and Tune/ISTD valve is pictured below.

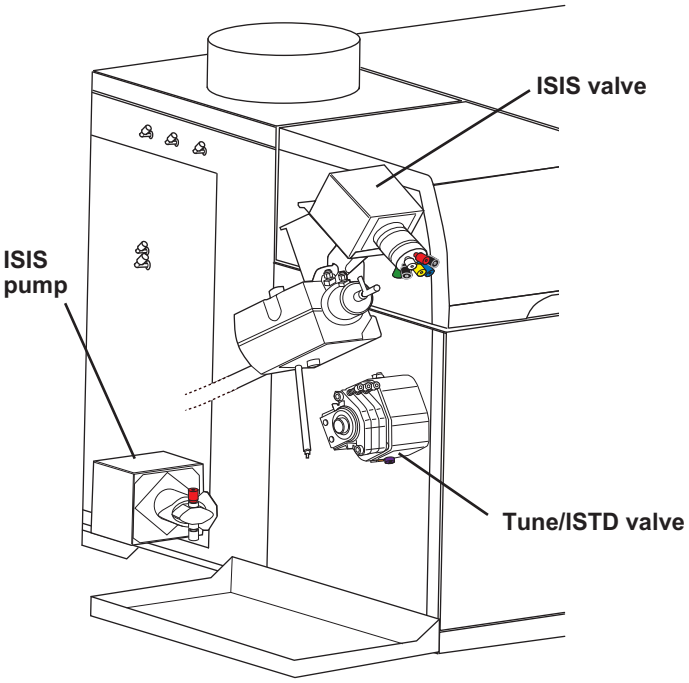

Figure 1 ISIS 3 System Sketch

Specifications

Dimensions

Size: 90(W) x 215(D) x 470(H) mm

Environment

ISIS 3 should be used under the installation conditions described below in the same manner as ICP-MS.

Operation condition:

Ambient temperature: 15 to 30 °C
Relative humidity: 20 to 80 % (non-condensing)

Storage condition:

Ambient temperature: -35 to 65 °C
Relative humidity: 15 to 85 % (non-condensing)

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2 Set Up

Hardware Setup 10
Discrete Sampling 10
Software Setup 13

Hardware Setup

Discrete Sampling

Connecting the Tubing

The following figure shows the discrete sampling tubing.

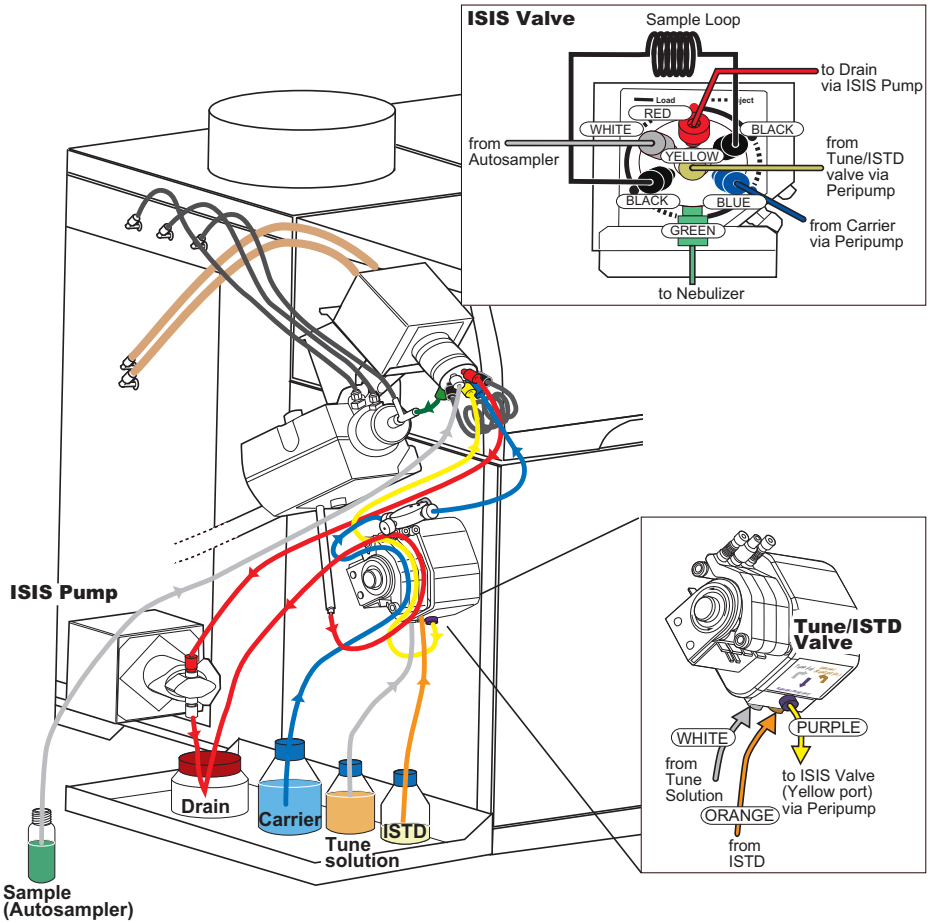


Figure 2 Tubing Sketch for Discrete Sampling

Tubing Sketch

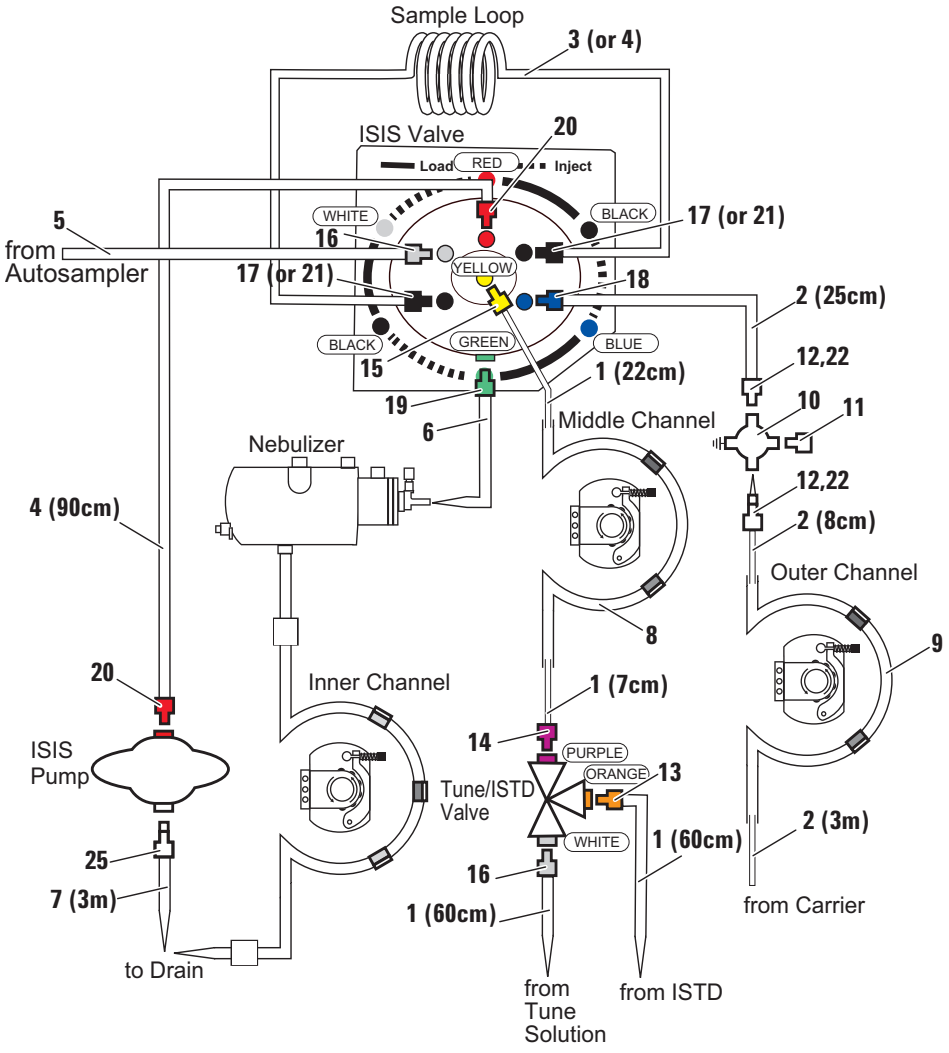


Figure 3

Table 1 Parts List for Discrete Sampling

No.	P/N	Part name	Qty
1	G1820-65478	Sample tubing 0.3 mm ID, 1/16" OD	3 m
2	G1820-65105	Sample tubing 0.5 mm ID, 1/16" OD	5 m
3	G8411-67117	Sample tubing 1.0 mm ID, 1/16" OD	5 m
4	G8411-67118	Sample tubing 2.18 mm ID, 1/8" OD	5 m
5	G3286-80119	Sample probe, carbon fibre 1.0 mm ID ASX	1
6	G3266-80013	UniFit sample tube for MicroMist neb. 0.25 mm ID	10
7	G3280-60077	Tubing 1/8" ID, 1/4" OD	3 m
8	G3280-67047	Peri-pump tubing, 0.25 mm ID for ISTD, flared end	12
9	G1833-65569	Peri-pump tubing, 1.02 mm ID for carrier	12
10	G3280-67062	T-Joint with earth block	1
11	G3138-65129	Plug	3
12	5064-8023	PTFE nut for 1/16" OD tubing	10
13	5023-1519	1/16" tube end PP nut and CTFE ferrule set, orange	5
14	5023-1520	1/16" tube end PP nut and CTFE ferrule set, purple	5
15	5023-1521	1/16" tube end PP nut and CTFE ferrule set, yellow	5
16	5023-1522	1/16" tube end PP nut and CTFE ferrule set, white	5
17	5023-1523	1/16" tube end PP nut and CTFE ferrule set, black	5
18	5023-1524	1/16" tube end PP nut and CTFE ferrule set, blue	5
19	5023-1525	1/16" tube end PP nut and CTFE ferrule set, green	5
20	5023-1526	1/8" tube end PP nut and CTFE ferrule set, red	5
21	5023-1527	1/8" tube end PP nut and CTFE ferrule set, black	5
22	5064-8024	Front and back ferrule for 1/16" OD tubing	10
23	5043-0054	1/16" tube end CTFE ferrule 10/pk (spare for item# 13 to 19)	10
24	5043-0055	1/8" tube end CTFE ferrule 10/pk (spare for item# 20 and 21)	10
25	5023-1517	Barb fitting, 1/4-28UNF-ID3.2	1

Note that the contents and part numbers indicated in the parts list are subject to change without notice.

Software Setup

Configure the software settings to operate ISIS 3 in the MassHunter Workstation. Follow the steps below to configure the software settings to perform Discreet Sampling.

NOTE The term "ALS" has been changed to "Autosampler" in MassHunter 4.4.

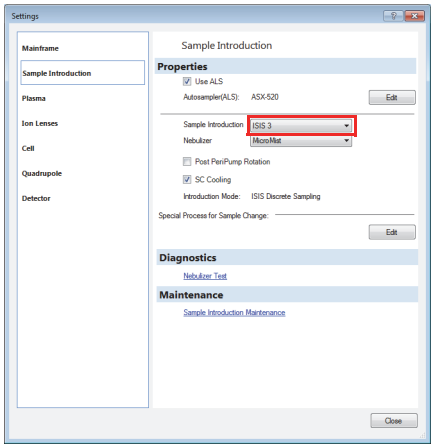
1 **Launch MassHunter, and then click the *Settings* gadget on the dashboard.**
The Settings dialog box appears.

2 **On MassHunter 4.2 or later, click *System*, and then click ⊕ at the side of *Hardware*.**
On MassHunter 4.1, skip this step.

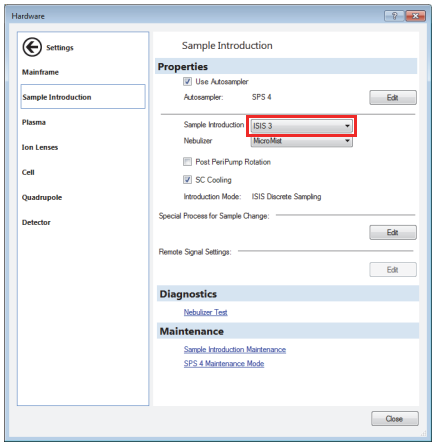
3 **Click *Sample Introduction*, and then select *Use Autosampler* in *Properties*.**

4 **Click *Edit* in *Properties*, and then configure the Autosampler in the *Configure Autosampler* dialog box.**
For how to configure the Autosampler, refer to the respective Autosampler manual.

5 **After configuring the Autosampler, select *ISIS 3* from the *Sample Introduction* list box.**



MassHunter 4.1



MassHunter 4.2 or later

Figure 4 *Settings* dialog box

6 Click *Close* to close the *Settings* dialog box.

3
Operations for the Discrete Sampling

Introduction 16
Operation Procedure 17
1. Preparing the Hardware 17
2. Starting up the Instrument 18
3. Optimizing the ISIS Parameters 20
Example of ISIS-DS Setup 24
ISIS-DS Initial Configuration 24
Check List for ISIS-DS Optimization 25
Edit Method 31
Verify ISIS Operation Configurations 32

Introduction

Discrete Sampling is a method of introducing a fixed, small-volume sample. This is also known as "flow injection" or "micro sampling".

The carrier solution and the ISTD are pumped into the nebulizer by the peristaltic pump. The sample is pumped into the sample loop by the ISIS pump. When the valve turns from the LOAD position to the INJECT position, the carrier solution carries the sample to the nebulizer and rinse solution is pumped to wash autosampler probe by the ISIS pump.

The flow injection acquisition mode is comprised of a TRA (time analysis) mode and spectrum mode. We will refer to "spectrum mode" as ISIS-DS. Its main features are a high sample throughput and robustness against high matrix samples.

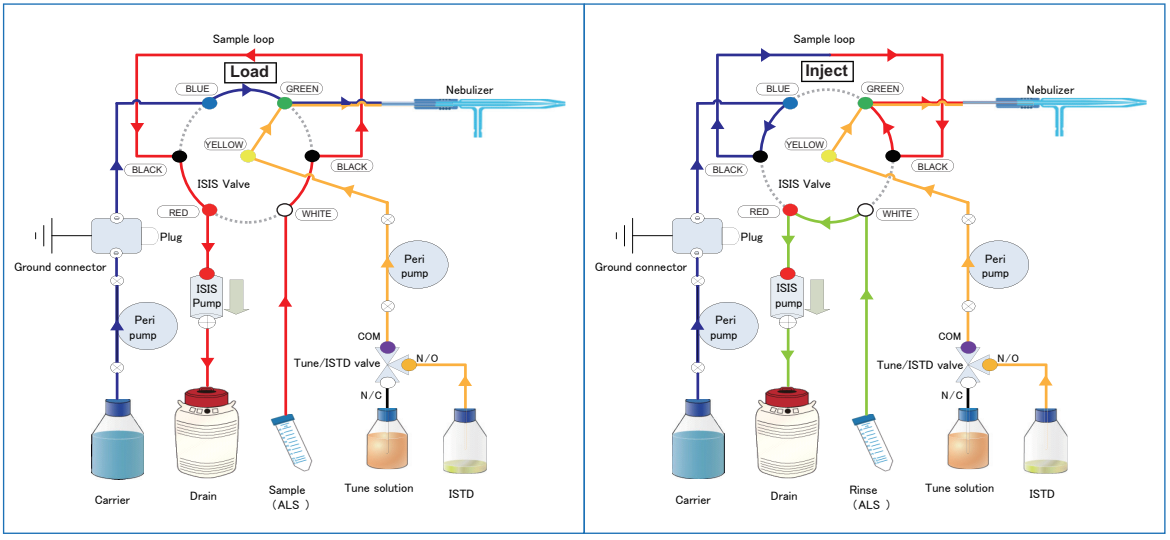


Figure 5

Operation Procedure

The standard settings and operations for ISIS are shown below. Other settings and operations are the same as when ISIS is not being used.

1. Preparing the Hardware

Check the following items. For detailed information, refer to Chapter 2, "Set Up".

- Sample introduction components and Autosampler connection
- Standard solution, internal standard solution for online internal standard addition, rinse solution, samples, etc.
- Remaining drain tank capacity

2. Starting up the Instrument

Before igniting the plasma, configure **Set PeriPump/ISIS** from the Startup pane.

In the ***PeriPump Setting*** dialog box, select **ISIS Discrete Sampling** from the ***Sample Introduction*** list box.

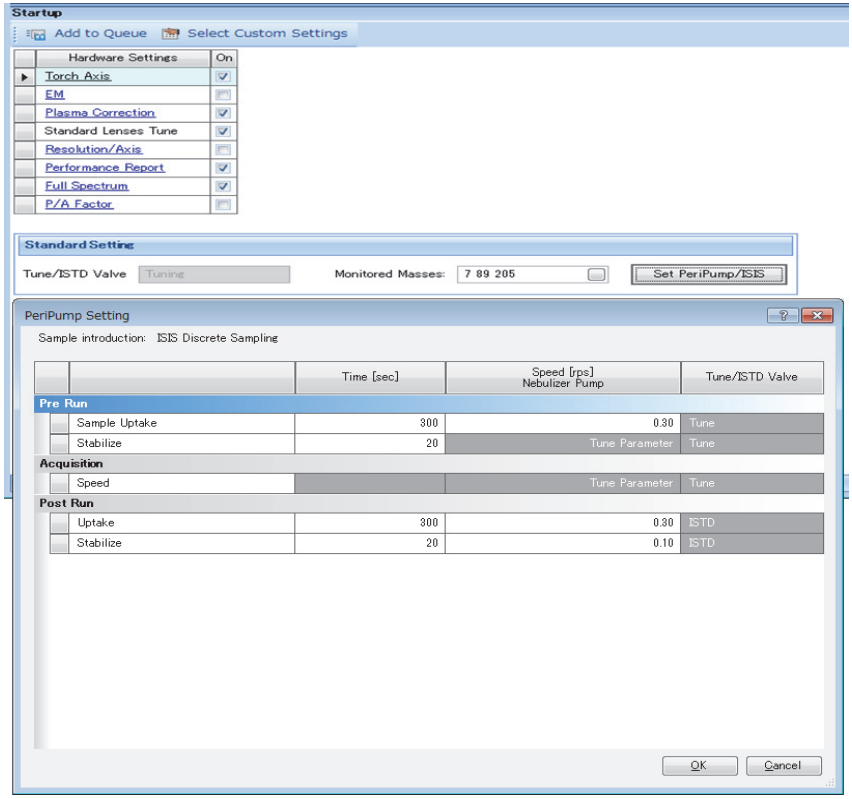


Figure 6 [PeriPump/ISIS] (ISIS Discrete Sampling)

Pre Run starts just after the instrument transitions to analysis mode. During **Pre Run**, the Tune/ISTD valve is switched to introduce the tuning solution.

Acquisition starts just after instrument warm up is finished. During **Acquisition**, hardware optimization is performed.

Post Run starts just after **Acquisition** is finished. During **Post Run**, the Tune/ISTD valve is switched so that to introduce the ISTD solution.

NOTE Before igniting the plasma, confirm that 10 ppb tuning solution is correctly set in the Tune/ISTD valve.

The Tune/ISTD valve switches the tuning solution and the ISTD. Set the tuning solution correctly in the downstream of the Tune/ISTD valve as shown in the figure below. The label attached on the PeriPump block indicates which port should be connected to the tuning solution and the ISTD.

Tuning solution is introduced through the ISTD line, and is diluted about 15 times by the carrier solution. 10 ppb tuning solution is necessary for startup to acquire enough signal intensity.

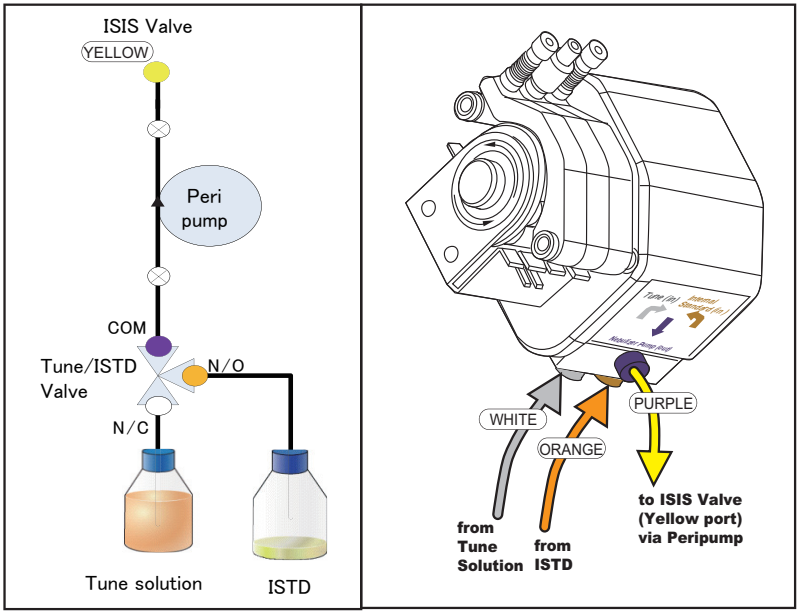


Figure 7 Tune/ISTD valve

Ignite the plasma and wait until the instrument is in analysis mode.

3. Optimizing the ISIS Parameters

Before performing analysis with the ISIS, it is necessary to optimize the ISIS parameters as well as batch normal settings. Parameters can be configured from the ***PeriPump/ISIS*** tab in Batch.

ISIS Parameters for Discrete Sampling

ISIS parameters can be configured by selecting **ISIS Discrete Sampling** from the ***Sample Introduction*** list box on the ***PeriPump/ISIS*** tab (Figure 8).

ISIS settings are comprised of three sections; **PreRun**, **Acquisition**, and **PostRun**.

Boxes that are grayed out will not be used or are referring to values entered elsewhere and do not need to be configured. White boxes are parameters that must be configured.

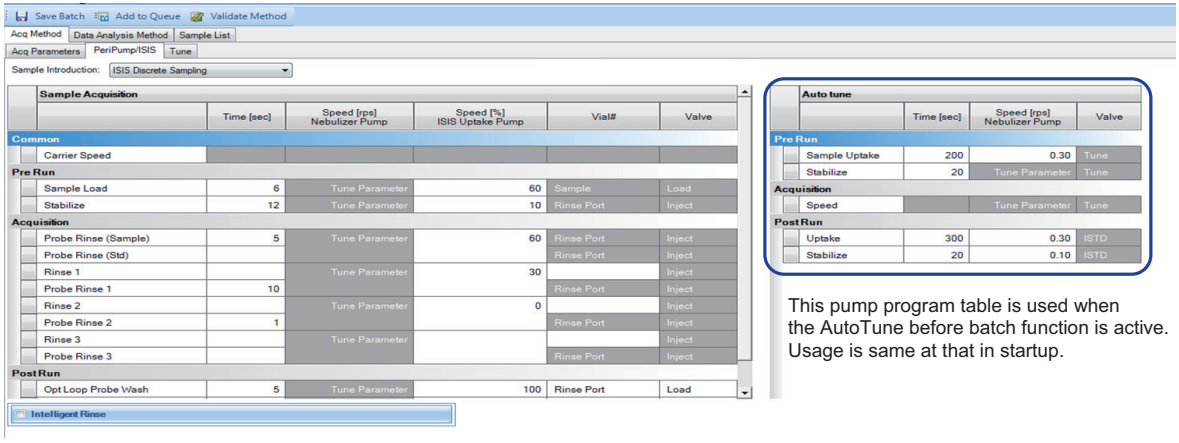


Figure 8 [PeriPump/ISIS] tab in Batch (ISIS Discrete Sampling)

PeriPump / ISIS Settings Outline [Each Pump Speed and Vial Position]

Setting	Explanation
Time [sec]	Execution time in [sec] for each setting.
Speed [rps] Nebulizer Pump	Nebulizer pump rotation speed in [rps] executed for each setting.
Speed [%] ISIS Uptake Pump	ISIS pump rotation speed in [%] executed for each setting.
Vial#	Autosampler valve position executed for each setting. Moves to the rinse port if "Rinse Port" is selected or moves to the vial position indicated in the batch sample list if "Sample" is selected.
Valve	ISIS valve (Load or Inject) settings executed for each setting.

PreRun:

The sample introduction time and stabilization time are configured in PreRun (before acquisition).

Setting	Explanation
Sample Load	The step for filling the sample loop with the sample. The valve is in the load position and the Autosampler probe moves to the vial of the acquired sample. During this step, the ISIS Pump increases in speed in order to hasten sample uptake into the sample loop. The nebulizer pump rotates at the speed determined in the tune parameter for all steps.
Stabilize	The step for delivering the sample inside the sample loop to the nebulizer. The valve switches to the "Inject" position. During this step, the Autosampler probe moves to the rinse port for rinsing.

Acquisition:

Acquisition settings.

NOTE

The total rinse time for this setting should be shorter than the data acquisition time set in **Acq Parameters**. Otherwise, **Post Run** does not start even if the data acquisition is finished, and the sample throughput could be compromised.

Setting	Explanation
Probe Rinse (Sample)	Rinses the probe with the rinse port during acquisition. Rinse time can be configured individually for samples and standard solution.
Probe Rinse (Std)	
Rinse 1	Performs additional rinse with the specified rinse bottle.
Probe Rinse 1	Performs additional rinse with the rinse port. It is possible to configure additional rinse stages Rinse 2 and Rinse 3 as necessary. Leave blank if unnecessary.

PostRun:

Settings for loop and probe rinse after acquisition.

Setting	Explanation
Loop/Probe Wash	The step for rinsing the sample loop and probe with the rinse port. The valve switches to “Load” and the probe moves to the rinse port. ISIS Pump speeds up to the speed configured in the sample introduction field (normally about 80 %) in the PreRun setting.
Loop Wash	The step for rinsing the sample loop and probe with the rinse bottle rinse solution. Moves the probe to the specified rinse bottle. ISIS Pump speeds up to the speed configured in the sample introduction field (normally about 80 %) in the PreRun setting.

NOTE

In Time Resolved Analysis, the repetition number must be 1.

NOTE Before performing analysis, it is recommended that the valve operation in manual operation is checked by opening the ***ISIS Manual Operation*** dialog box from **ISIS** in the ***Tune*** tab.

NOTE You cannot switch the ISIS valve within 4 seconds after the latest ISIS valve switching.

CAUTION After analysis, rinse the sample introduction system including the ISIS prior to turning OFF the plasma. To wash the sample introduction system, move the probe of the ASX-500 Series Autosampler to the rinse solution and rinse the system. If the ASX-500 Series Autosampler is not being used, introduce the rinse solution manually. To clean the valve, alternately click **Load** and **Inject** for the valve in the ***ISIS Manual Operation*** dialog box. Turn OFF the plasma, open the clamps of all the peristaltic pumps and remove the pump tubing from the hooks of the pump.

CAUTION Do not run the ISIS Pump if no liquid has been delivered for an extended period of time because liquid delivered by this pump acts as coolant and lubricant. If the ISIS Pump is used in this condition, it could be damaged.

Example of ISIS-DS Setup

In the following description of setting up ISIS-DS, we will look at an example of setting up the 7900 for the highest possible throughput and minimized matrix exposure using discrete sampling in spectrum acquisition mode. Typical run times in He mode for ~30 elements with 40 seconds total data acquisition time will be about 1 minute sample to sample.

ISIS-DS Initial Configuration

Configure ISIS for discrete sampling according to the ISIS manual and the tubing guide shown in Figure 2.

NOTE All tubing lengths including the autosampler probe tube should be as short as possible in order to minimize the volume and time to fill as well as minimize carryover.

Check List for ISIS-DS Optimization

You will need to check that the loop volume as well as sample, carrier and internal standard flows are correct and determine the exact time needed for each step of the ISIS program.

- Fill the carrier bottle with 2 % HNO_3/0.5 % HCl (or whatever your typical rinse solution is) and place the carrier uptake tube in the bottle.
- Fill the ISTD bottle with your normal ISTD solution (typically 1 ppm of ISTD mix).
- Fill the tuning bottle with tuning solution. (typically 10 ppb of tuning mix)

  Tuning solution is introduced to the nebulizer by way of an ISTD line whose pump tube id is 0.25 mm. So the tuning solution is diluted by carrier solution by about 15 times. That is why we need 10 ppb tuning solution instead of 1 ppb.
- Make sure the ISTD solution is online and has reached the nebulizer.
- Open the ***Tune*** tab, change the Tune/ISTD valve position into “tuning”, and check the sensitivity of the signal monitor.
- Set the nebulizer pump as normal in tune (normally ~0.1 rps).
- Set acquired masses for at least 1 tune solution element (3 are shown in Figure 14) that is not an ISTD element such as Co and one internal standard element (2 are shown in Figure 14) that is not also a tune element, such as In. Set both to display.

1 Determine the loop size needed.

The size of the sample loop needed will depend on the acquisition time for your method which depends on many things including number of elements and associated integration times, number of points per peak, number of tune modes used and number of replicates. After determining the shortest acquisition conditions which meet your data quality objectives, you will need to provide a loop which will provide constant delivery of sample to the nebulizer for the entire acquisition.

The required loop volume can be calculated from the nebulizer flow and total acquisition time,

[neb flow (µL/min) * acq time (min) = minimum loop volume (µL)].

The calculated value should be increased by ~20 % to insure sufficient volume under actual operating conditions.

NOTE

You can choose either 1.0 mm id or 2.18 mm id tubing for the sample loop. Wider bore (2.18 mm id) tubing is recommended if the data acquisition time is long. For narrower bore (1.0 mm id) tubing for a large volume sample loop, the tubing should be longer. This generates a significant flow restriction lengthening the sample load time mentioned later, and generating air bubbles in the loop which cause analytical problems.

Make the loop according to the formula below.

Sample loop 300-500 µL made from 1.0mm ID tubing. Volume can be calculated as follows:

$V = \pi(r)^{2*} L$

- Where V is volume in µL
- r is internal radius in mm
- L is length in mm

  38 cm (380 mm) of 1.0 mm id tubing will result in an approximately 300 µL loop.

The following table shows typical sample loop volume v.s. required data acquisition time.

Total acq time [s]	Typical loop tubing length [mm]	
	1.0 mm id	2.18 mm id
10	260	
30	540	110
50	830	170
80		260

2 Install the loop between two “Black” ports on the ISIS valve, and optimize the loop.

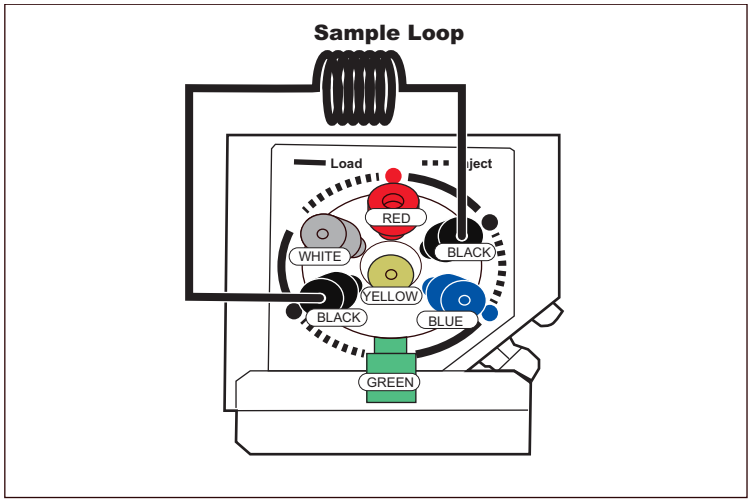


Figure 9

a Open "PeriPump/ISIS setting" in Batch and set enough time to fill the loop with tune solution in the "Pre Run" "Sample Load" section. The normal time and speed is about 10 seconds and 60 %.

Acq Method | Data Analysis Method | Sample List

Acq Parameters | PeriPump/ISIS | Tune

Sample Introduction: ISIS Discrete Sampling

Sample Acquisition	Time [sec]	Speed [rps] Nebulizer Pump	Speed [%] ISIS Uptake Pump	Vial#	Valve
Common					
Carrier Speed					
Pre Run					
Sample Load	10	Tune Parameter	60	Sample	Load
Stabilize	9	Tune Parameter	30	Rinse Port	Inject
Acquisition					
Probe Rinse (Sample)	20	Tune Parameter	80	Rinse Port	Inject
Probe Rinse (Std)	0			Rinse Port	Inject
Rinse 1		Tune Parameter	50		Inject
Probe Rinse 1	1			Rinse Port	Inject
Rinse 2	0	Tune Parameter			Inject
Probe Rinse 2	0			Rinse Port	Inject
Rinse 3	0	Tune Parameter			Inject
Probe Rinse 3	0			Rinse Port	Inject
Post Run					
Opt Loop Probe Wash	4	Tune Parameter	80	Rinse Port	Load
Opt Loop Wash		Tune Parameter			Load

Intelligent Rinse

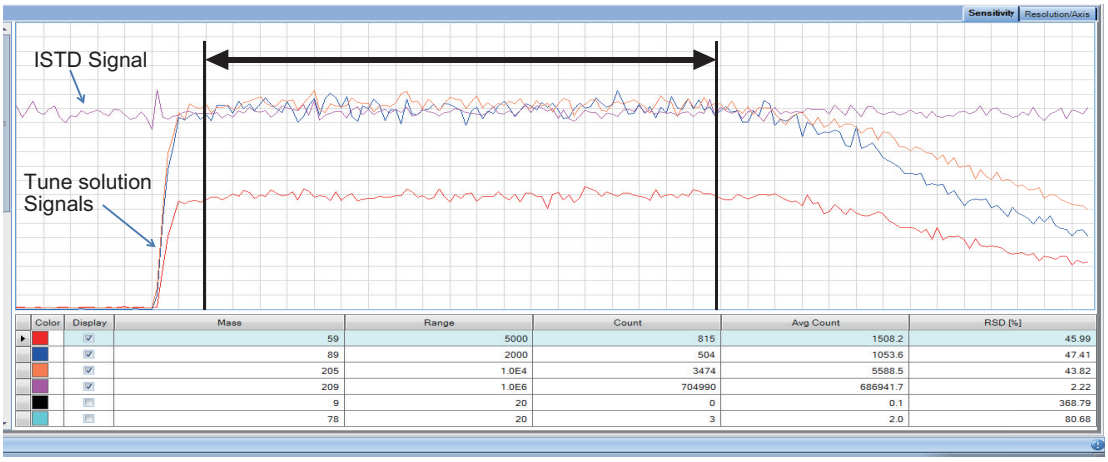
Figure 10 *PeriPump/ISIS* tab in Batch (ISIS Discrete Sampling)

b Open the tune window. Start the signal monitor and check the ISTD signal is stable and tune solution signal is low enough.

c Change the autosampler probe position into tuning solution.

d Start the "Pre Run". This operation automatically fills the loop with tune solution first and after that injects the tune solution to the nebulizer. Confirm that the tune solution has filled the loop.

e You will see a signal like the one shown below. Start the stopwatch when the tuning signal become stable and stop the stopwatch when the tuning signal starts to drop. Optimize the loop size to match the data acquisition time based on your requirements.



Color	Display	Mass	Range	Count	Avg Count	RSD [%]
	☑	59	5000	815	1508.2	45.99
	☑	89	2000	504	1053.6	47.41
	☑	205	1.0E4	3474	5588.5	43.82
	☑	209	1.0E6	704990	686941.7	2.22
	☐	9	20	0	0.1	368.79
	☐	78	20	3	2.0	80.68

Figure 11

3 Determine the sample load time necessary to flush and fill the loop.

- Position the autosampler probe in the home position using Autosampler Manual Control.
- Set the ISIS pump speed to be about 20 % using ISIS manual operation dialog box and fill the loop with air. To open this dialog box, right-click in ***Tune*** tab to display the context menu, open **ISIS > ISIS Manual Operation**.
- Stop the ISIS pump and position the autosampler probe in tune solution.
- Start the "Pre Run" and your stopwatch at the same time. Stop your stopwatch when the tune solution is filled completely within the sample loop and no bubbles remain.

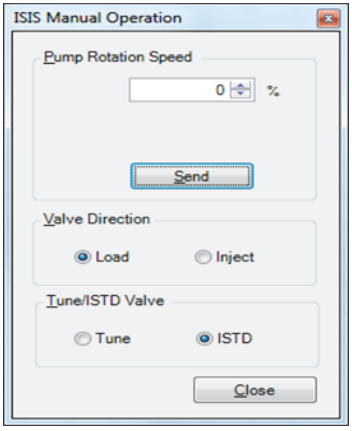


Figure 12 *ISIS Manual Operation* dialog box

- Typical uptake time is normally ~5 seconds. Enter this time as the sample load time.

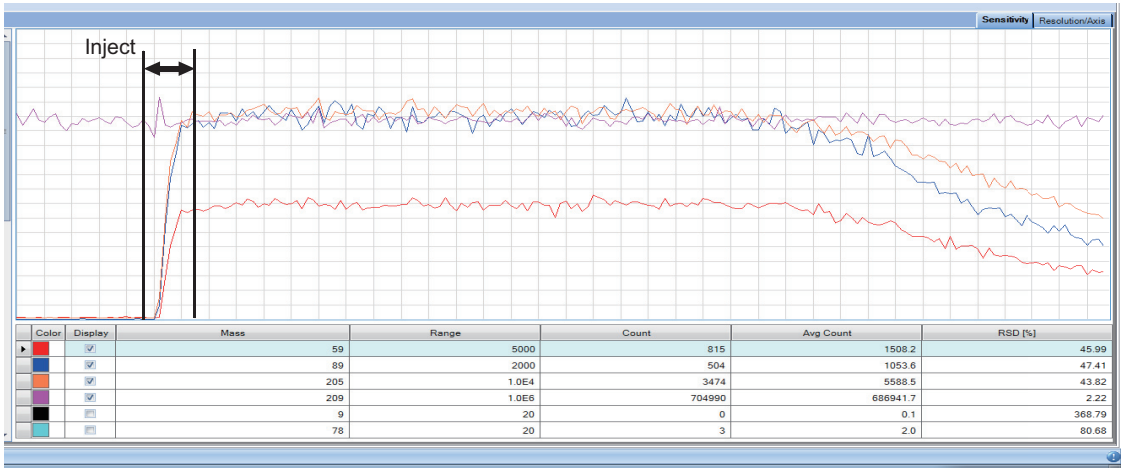


Figure 13

4 Determine the delay and acquisition times.

- Position the autosampler probe in the tuning solution using Autosampler manual control.
- Start the signal monitor on the tuning window.
- Start the "Pre Run" program and start your stopwatch when the ISIS valve position switches into inject position after the tuning solution is filled.
- Stop your stopwatch when the signal of tuning solution becomes stable.
- Typical stabilize time is ~8 seconds. Enter this time as the stabilize time.

5 Make a note of the ISIS parameters based on your measurements in Steps 3 and 4.
Save Tune and exit.

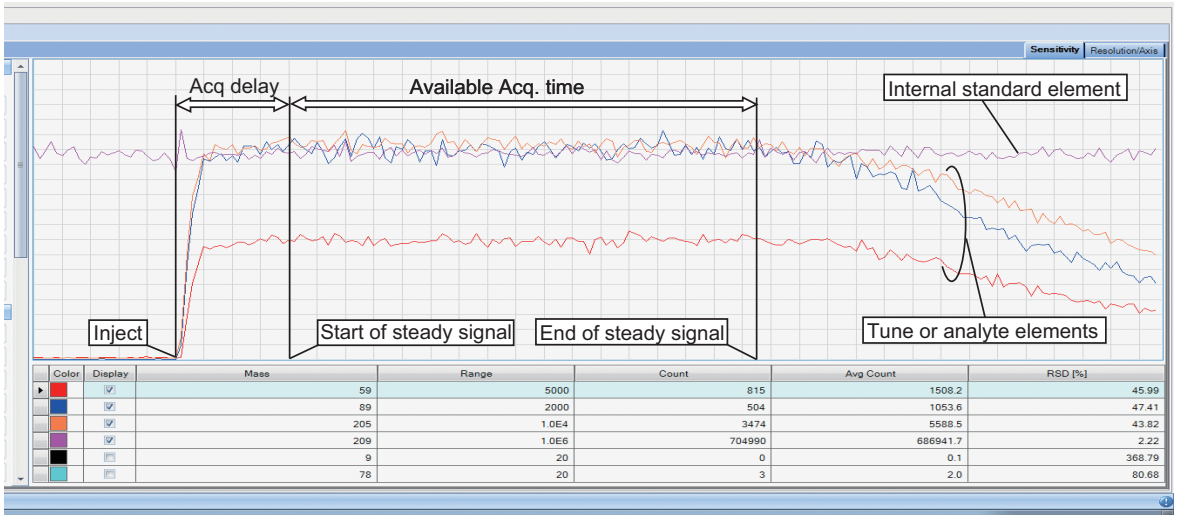


Figure 14

Edit Method

Check your method acquisition parameters to be sure that your total acquisition time is shorter than the available acquisition time as determined by the loop size (above). You should allow at least 5-10 seconds of margin between the available time and the actual time.

Minimize acquisition time as much as possible by doing the following

- Use only He mode if possible
- Use one point for peak pattern
- Use short integration times for high response elements and elements which do not require ultimate sensitivity (0.05-0.1 sec), use somewhat longer integration times as needed for low response elements such as Be, Se etc.

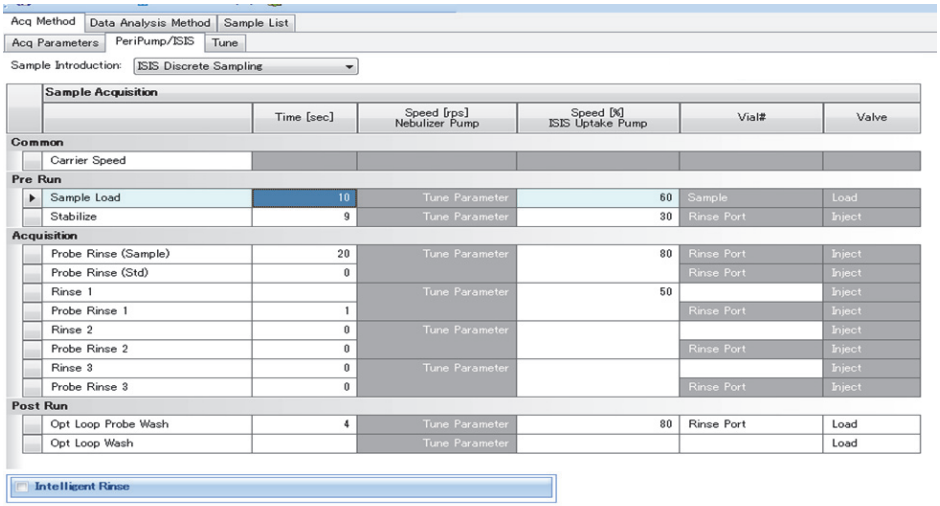


Figure 15 *PeriPump/ISIS* tab in Batch

In the ISIS-DS, a large volume of rinse solution is required for use with the Autosampler rinse port. The flow of the ASX-500 Series Autosampler pump rinse solution is 9 mL/min. The volume of required rinse solution can be derived by multiplying 9 mL/min x rinse time for per sample x number of samples.

NOTE Refer to pages 20 to 23 for more details about each setting.

Verify ISIS Operation Configurations

Prepare two samples, create a batch, and execute a performance check. Prepare a solution containing all elements to be acquired and a blank solution.

Execute the batch and examine the %RSD for both the internal standard elements and analyte elements. If the acquisition delay time and available acquisition times are sufficient, you should see low %RSD values (< 5 %) for both internal standard and analyte elements. If the ISTD %RSDs are low enough, but analyte %RSDs are high, this suggests the acquisition delay time is too short, or the stabilization time is insufficient. Make any necessary adjustments in the ***PeriPump/ISIS*** tab.

Next, look at the blank counts. Verify that all analyte elements were adequately rinsed. Typically, there is a 3-4 orders of magnitude difference between the sample and the blank. If excessive carryover is apparent, increase the rinse time to rinse the sample introduction system and lengthen the loop uptake time.

4 Maintenance

Replacing the Valve Rotor and Stator for the ISIS Valve 34
Replacing the Lip Seals of the ISIS Pump 37

CAUTION Before performing any maintenance, clean the flow path inside the pump and valve with de-ionized water to remove any acids or other hazardous liquids.

Replacing the Valve Rotor and Stator for the ISIS Valve

Cleaning a valve can often be accomplished by flushing all of the lines with appropriate solvents.

The valve rotor and/or stator should be replaced under the following circumstances:

- when the valve is clogged
- when the valve is leaking and it is not visible from the connector parts
- when the signals become unstable because of internal leakage

Do not disassemble the valve unless the system malfunction is definitely isolated to the valve.

WARNING **When changing the valve rotor or stator, manually turn off the power switch before disassembling.**

CAUTION Failure to observe proper sanitary procedures during the installation of the valve voids the manufacturer's warranty.

To replace the valve rotor or stator, follow the steps below.

NOTE Before disassembling the valve, ensure that port "1" on the stator is situated in the red dot position as shown in Figure 16.

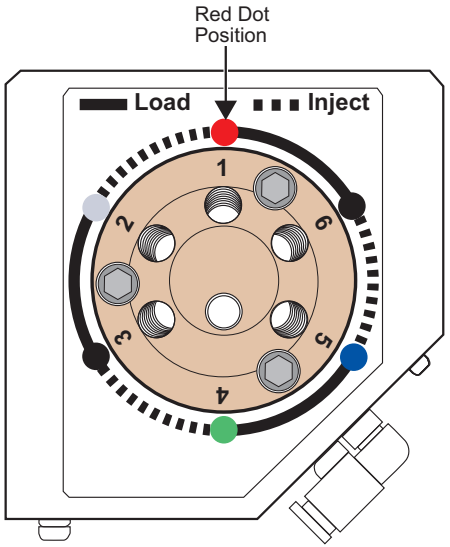


Figure 16

Disassembly

1 **Use a 7/64” hex driver to remove the socket head screws that secure the stator to the valve.**

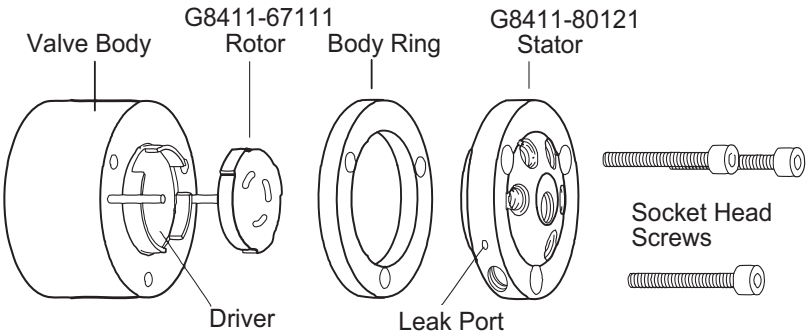


Figure 17

2 To ensure that the sealing surface of the stator does not get damaged, place it with the outer surface facing down. Or, if the tubing is still connected, leave it suspended by the tubing.

3 With your fingers or a small tool, gently pry the rotor away from the driver.

4 Examine the rotor sealing surface for scratches. If scratches are visible to the naked eye, the rotor must be replaced. If no scratches are visible, clean all of the parts thoroughly with an appropriate solvent, taking care that no surfaces get scratched. (A common problem is the formation of buffer crystals, which are usually water-soluble.) You do not need to dry the rotor.

Reassembly

1 Replace the rotor in the driver, making sure that the rotor sealing surface with its engraved flow passages is facing out. The pattern is asymmetrical to prevent improper placement.

2 Replace the stator. At this time, orient it so that the port "1" on the stator is in the red dot position as shown in Figure 16. Insert the three socket head screws and tighten them gently until both are snug. Do not over-tighten them – the screws simply hold the assembly together and do not affect the sealing force, which is automatically set as the screws close the cap against the valve body.

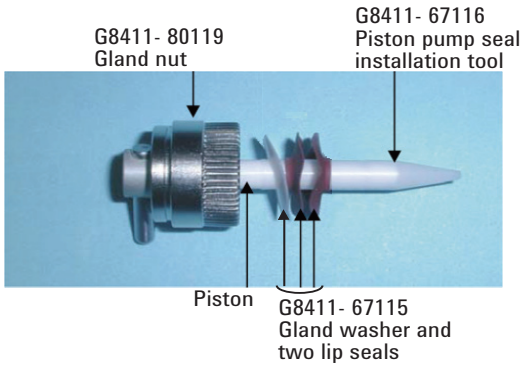
3 Test the valve by pressurizing the system. If it does not hold pressure, the whole ISIS valve assembly should be replaced. The part number is G8411-67113.

Replacing the Lip Seals of the ISIS Pump

Seals in the ISIS pump are consumable parts. If any leakage is visible on the ISIS pump, replace the seals with new ones.

Agilent supplies the following consumable parts for the ISIS pump.

- G8411-67115: Gland washer and two lip seals
- G8411-80119: Gland nut
- G8411-67116: Piston pump seal installation tool



To replace the seals in the ISIS pump, follow the steps below.

1 **Remove the two screws that secure the black top of the ISIS pump.**

2 **Remove the black top.**

3 **Carefully pull out the cylinder assembly.**

4 **Loosen the gland nut on the cylinder assembly, and then open the assembly.**

TIP: If the gland nut is made of PEEK plastic (not pictured) you may find it slippery or difficult to loosen. Use a rubber glove to help establish a firm grip.

You will see a gland nut, a gland washer, and two lip seals on the piston assembly.

5 **Slide off the used gland washer and lip seals.**

6 **Slide a new gland washer onto the piston.**

7 **Insert the lip seal installation tool into the end of the piston.**

8 **Carefully place one lip seal onto the tool lip side last while carefully rotating the seal to avoid damaging the lip. This process will form the lip of the lip seal around the tool. Next, remove the seal from the tool, reverse the lip seal direction, and reinstall the seal lip side first onto the tool and slide on to the piston.**

9 **Carefully place the other lip seal onto the tool with the lip side facing out, and while rotating the seal to the tool to avoid damaging the lip slide the seal onto the piston, lips side last.**

10 **Slide the tool off the piston assembly.**

11 **Insert the piston into the cylinder assembly, and then tighten the gland nut to a torque of 22 ±10 % inch·Lbs. (2.49 N·m ±10 %).**

12 **Rotate the piston by hand after reassembly to ensure free movement in the cylinder.**

CAUTION Never withdraw the piston more than 3/4"(19 mm) otherwise seals could be damaged as the piston flattens.

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In This Book

The *ISIS 3 (Integrated Sample Introduction System) Manual* explains the information necessary for performing analysis using the ISIS 3.

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