

Agilent ISIS-3 Integrated Sample Introduction System Discrete Sampler Optimization Procedure

Introduction

Discrete Sampling is a method of introducing a fixed, small- volume sample. This is also known as "flow injection" or "micro sampling". The ISIS-3 is a fully integrated system with the ISIS valve; ISIS pump; and Tune/ISTD valves for introduction into the flow path.

Purpose

This paper will discuss the optimization of the ISIS-3 for sample introduction, tuning and cleaning.

Scope

The ISIS-3 optimization is for advanced users and will detail the settings and explaintions of the setting for the parameters to set the optimal condition for acquisition.

Referenced Documents

- G8411-90023 ISIS-3
- G8411-90024 ISIS-3 User manual

Responsibilities

1. Users

- 1.1. All Users must obtain training with TRACES Staff prior to system operation. It is the responsibility of the User to ensure they have a good understanding of the instrument and all operation protocols.
- 1.2. If additional training sessions are needed it is the responsibility of the User to schedule these with TRACES Staff.

Equipment

- Agilent 7900 ICP-MS
- Agilent ISIS-3
- Agilent SPS-4



Safety

IMPORTANT:

- All the organic solvents are flammable
 - Take the appropriate precautions
- Seek assistance from Teaching or TRACES Staff if you have any concerns

ISIS-3 Operation Overview

- 1. The ISIS-3 system operates by having the carrier solution and the ISTD or Tuning solution pumped into the nebulizer by the peristaltic pump. Figure 1 details the setup in comparison to the ICP-MS.
- 2. The sample is pumped into the sample loop by the ISIS pump in the Load position (Figure 2).
- 3. When the valve turns from the LOAD position to the INJECT position (Figure 2), the carrier solution carries the sample to the nebulizer and rinse solution is pumped to wash autosampler probe by the ISIS pump.
- 4. The cleaning regime is then initiated during the acquisition run and post-run.











Hardware Preparation

- 5. Check the following items
 - a. Sample introduction components and SPS-4 Autosampler connection
 - b. Standard Tuning solution (for signal monitoring)
 - c. Internal standard solution
 - d. Rinse solution
 - e. Remaining drain tank capacity
 - f.

Instrument Start-up

- 1. Before igniting the plasma, configure Set **PeriPump/ISIS** from the Startup pane.
- 2. In the **PeriPump** Setting dialog box, select **ISIS Discrete Sampling** from **the Sample Introduction** list box. Ensure the default values in Figure 4 are populated correctly.

Torch Axis Image: Correction EM Image: Correction Standard Lenses Tune Image: Correction Resolution/Axis Image: Correction Performance Report Image: Correction Full Spectrum Image: Correction P/A Factor Image: Correction andard Setting Sample introduction: ISIS Discrete S /re Run	Monitored Masses	7 89 205 Set 1	PeriPump/ISIS
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Full Spectrum Image: Comparison of the spectrum	Monitored Masses	7 89 205	PeriPump/ISIS
P/A Factor.	Monitored Masses	7 89 205 Set	PeriPump/ISIS
andard Setting re/ISTD Valve Tuning Pump Setting Sample introduction: ISIS Discrete S	Monitored Masses	7 89 205	PeriPump/ISIS
Pre Run	Compring		
Pre Run	Time [sec]	Speed [rps] Nebulizer Pump	Tune/ISTD Valve
Sample Uptake	300	0.30	Tune
Stabilize	20	Tune Parameter	Tune
Acquisition			
Speed		Tune Parameter	Tune
Yost Run			
Uptake	300	0.30	ISTD
Stabilize	20	0.10	ISTD
Stabilize	20	0.10	ISTD



- 3. **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.
- 4. Acquisition starts just after the instrument has completed its Startup procedure.
- 5. During **Acquisition**, hardware optimization is performed.
- 6. **Post Run** starts just after **Acquisition** is finished. During **Post Run**, the Tune/ISTD valve is switched to introduce the ISTD solution.
- 7. 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 Figure 5 below. The label attached on the PeriPump block indicates which port should be connected to the tuning solution and the ISTD.
- 8. 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.
- 9. Ignite the plasma and wait until the instrument is in analysis mode.





ISIS-3 Optimization

- 10. 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.
 - i. Sweep width
 - ii. Relaxation delay
 - iii. Autosampler Queue Setup Spectral Center(sic)
- 11. ISIS Parameters for Discrete Sampling
 - a. ISIS parameters can be configured by selecting ISIS Discrete Sampling from the Sample Introduction list box on the PeriPump/ISIS tab (Figure 6).
 - b. ISIS settings are comprised of three sections;
 - i. PreRun
 - ii. Acquisition
 - iii. PostRun
 - c. 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 <u>must</u> be configured.

Parameters PeriPumpri515 Tu	ne	2							
ple Introduction: ISIS Discrete Sar	mpling 🔻								
Sample Acquisition				Auto tune					
	Time [sec]	Speed [rps] Nebulizer Pump	Speed [%] ISIS Uptake Pump	Vial#	Valve		Time [sec]	Speed [rps] Nebulizer Pump	Valve
mmon						Pre Run			
Carrier Speed						Sample Uptake	300	0.30	
Run				_		Stabilize	20	Tune Parameter	Tune
Sample Load		Tune Parameter		Sample	Load	Acquisition			
Stabilize		Tune Parameter		Rinse Port	Inject	Speed		Tune Parameter	Tune
uisition						PostRun			
Probe Rinse (Sample)		Tune Parameter		Rinse Port	Inject	Uptake	300	0.30	ISTD
Probe Rinse (Std)				Rinse Port	Inject	Stabilize	20	0.10	ISTD
Rinse 1		Tune Parameter			Inject				
Probe Rinse 1				Rinse Port	Inject				
Rinse 2		Tune Parameter		(Inject	This pump p	rogram ta	able is used v	when
Probe Rinse 2				Rinse Port		the AutoTun	e hefore l	natch functio	n is a
Rinse 3		Tune Parameter			Inject				iniou
Probe Rinse 3				Rinse Port		Usage is sar	ne at tha	t in startup.	
tRun									
Ont Loop Prohe Wash	0	Tune Parameter		Rinse Port	Load				



PeriPump/ISIS Setting Outline

Setting	Explanation
Time [sec]	Execution time in [sec] for each setting.
Speed [rps] Nebulizer Pum p	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 sam ple loop with the sam ple.				
	The valve is in the load position and the Autosam pler probe moves to the vial of the acquired sample.				
	During this step, the ISIS Pump increases in speed <u>in order to</u> hasten sample uptake into the sample loop.				
	The nebulizer pum p rotates at the speed determ ined in the tune parameter for all steps.				
Stabilize	The step for delivering the sam ple inside the sam ple loop to the nebulizer.				
	The valve switches to the "Inject" position. During this step, the Autosampler probe moves to the rinse port for rinsing.				

For the Acquisition:

Acquisition settings.

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 (Sam ple)	Rinses the probe with the rinse port during acquisition. Rinse tim e can be configured individually for sam ples and standard solution.			
Probe Rinse (Std)				
Rinse 1	Perform s additional rinse with the specified rinse bottle.			
Probe Rinse 1	Perform s additional rinse with the rinse port. It is possible to configure additional rinse stages Rinse 2 and Rinse 3 as necessary. <u>Leave</u> 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 (norm ally about 80 %) in the <u>PreRun</u> setting.
Loop Wash	The step for rinsing the sam ple 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 (norm ally about 80 %) in the <u>PreRun</u> setting.

12. 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.

*The TRACES Manager will provide full details during hands-on training.