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.

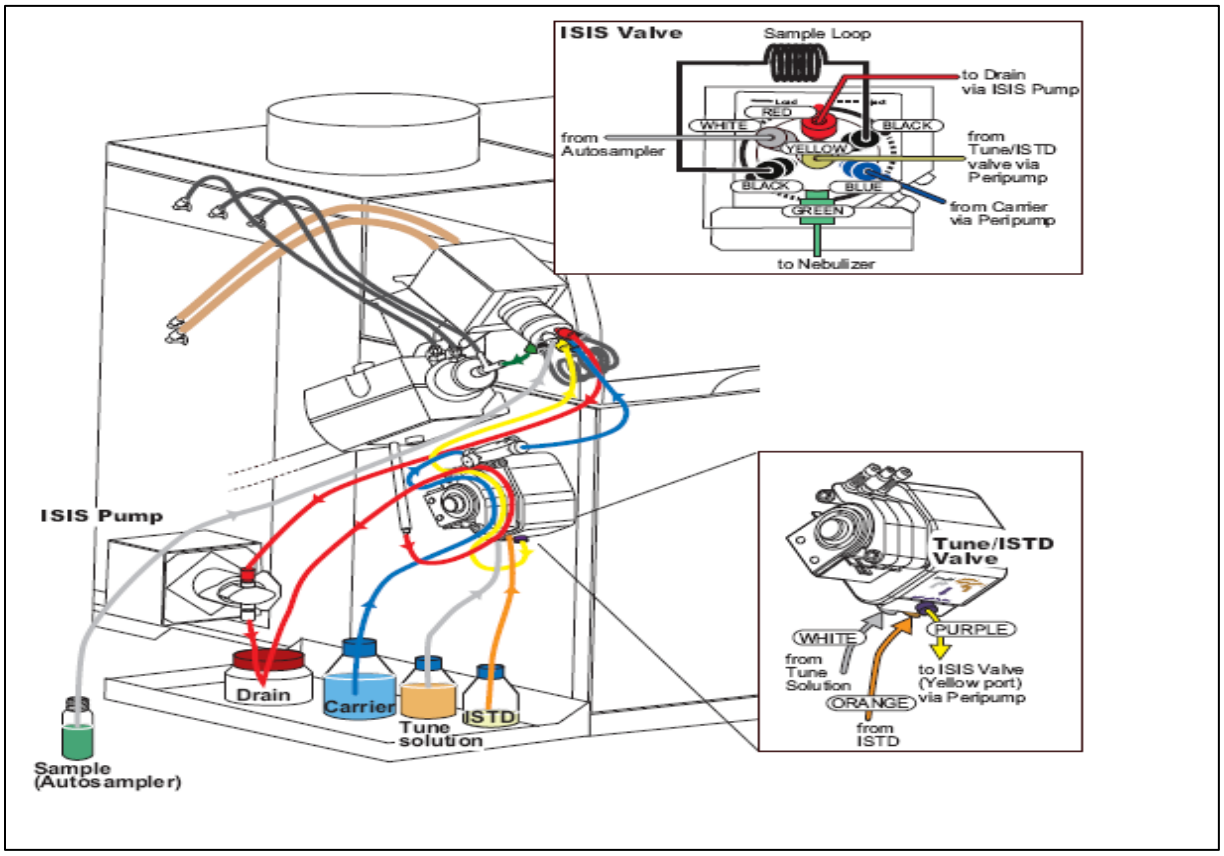

Figure 1

Optimizing Procedure

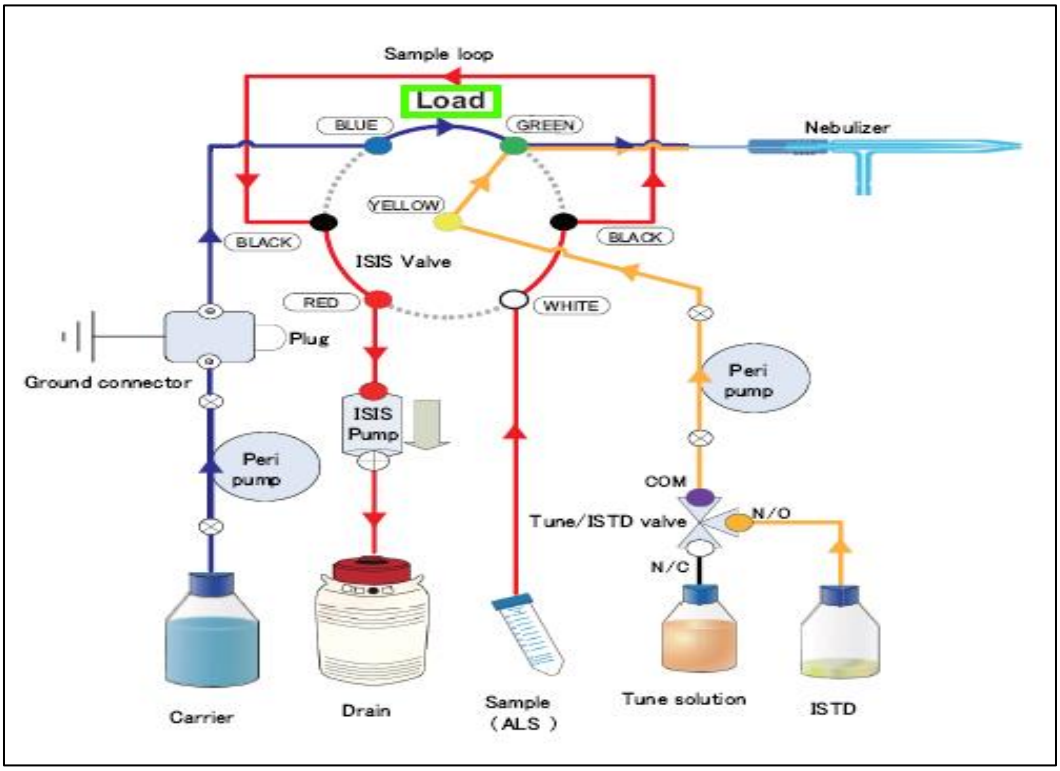

Figure 2

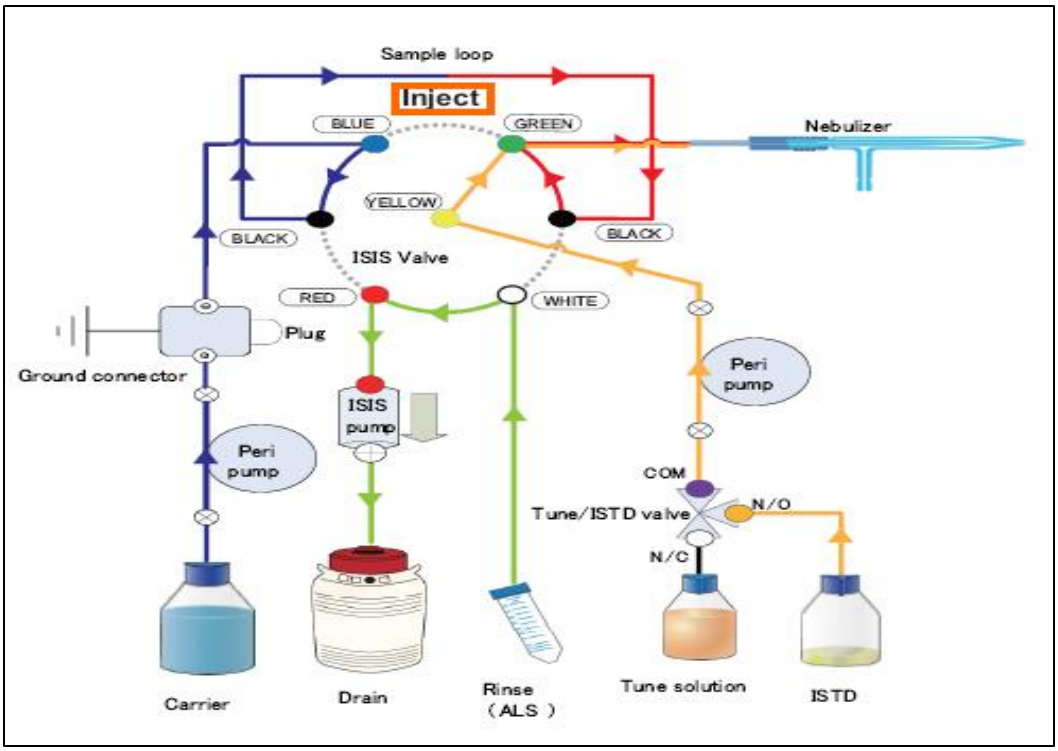

Figure 3

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.

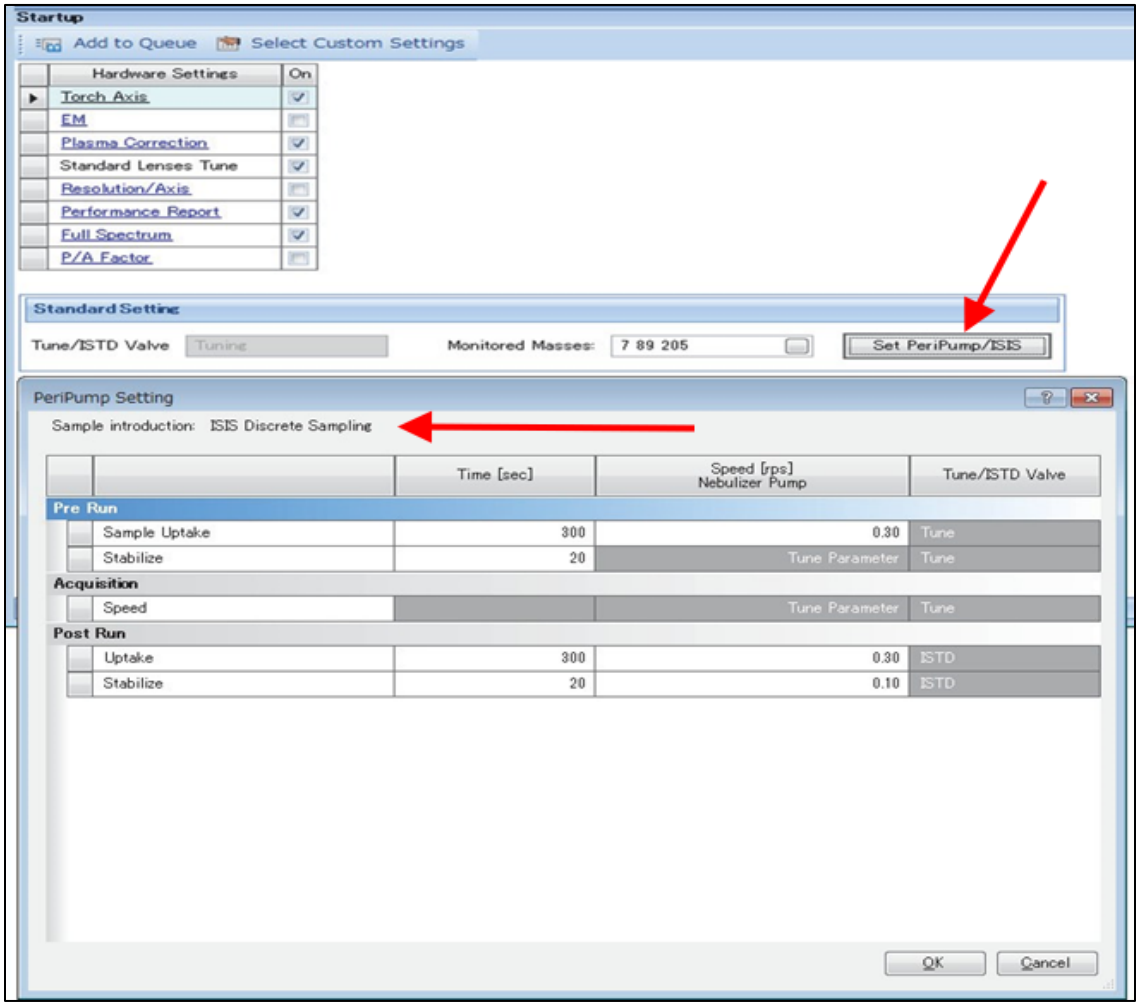

Figure 4

Optimizing Procedure

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.

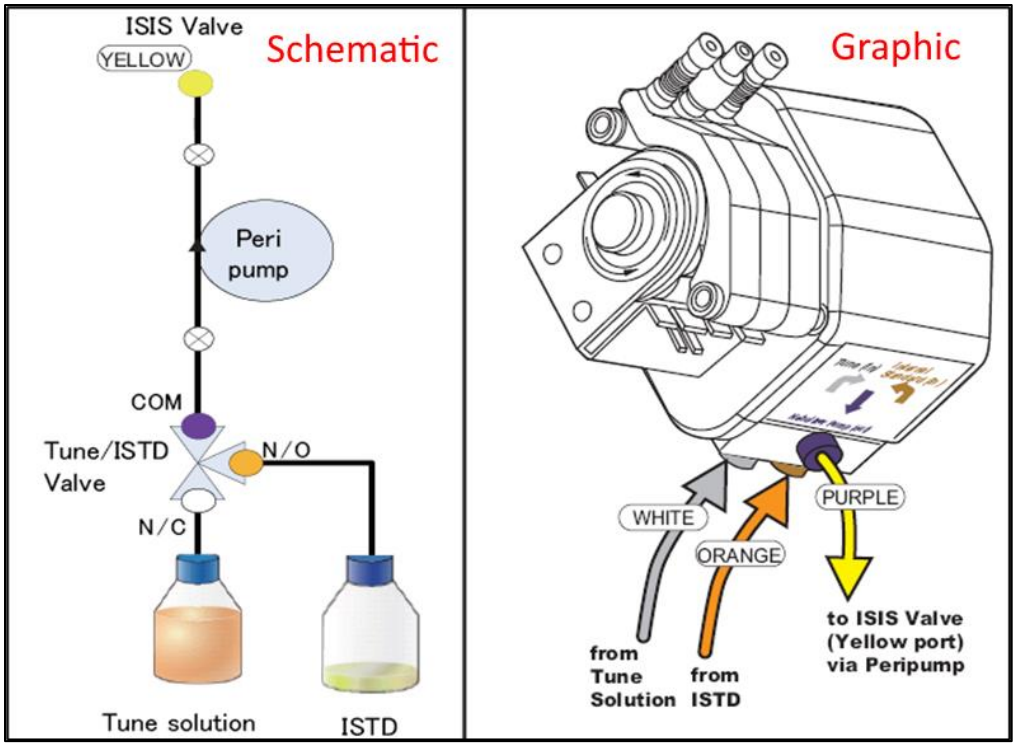

Figure 5

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 must be configured.

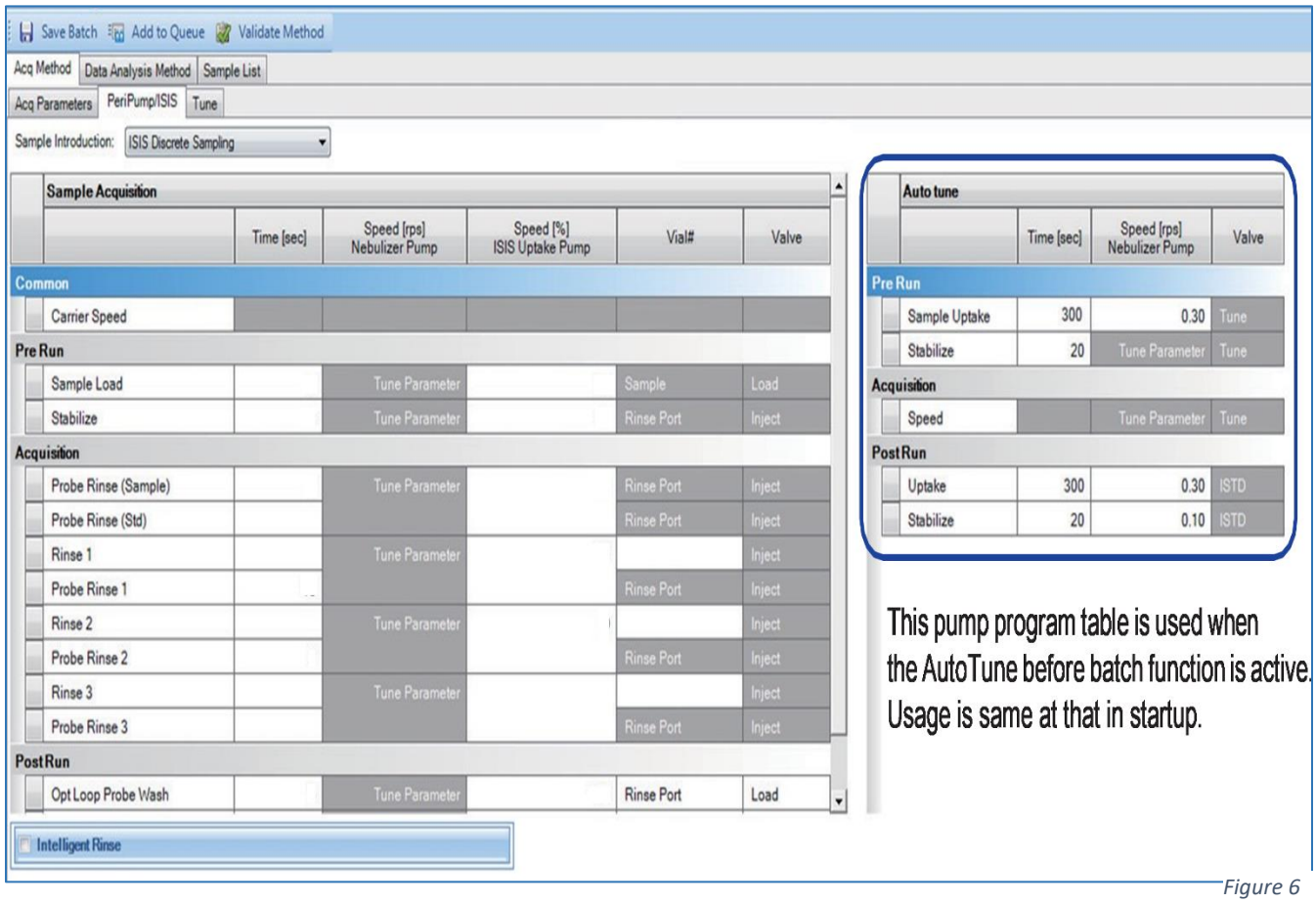

Figure 6

PeriPump/ISIS Setting Outline

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.

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 (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.

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.