ACIDIC DILUTIONS FOR ICP-MS ANALYSIS

AUTOMATION OF SAMPLE DILUTIONS WITH A MODIFIED GX-271/VERITY® 4120 SYSTEM USING ICP-MS RACKS AND ACCESSORIES RESISTANT TO STRONG ACIDS

TECHNICAL NOTE TN227

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INTRODUCTION

ICP-MS analysis is a technique of choice in many laboratories to quantify trace elements in many matrices.

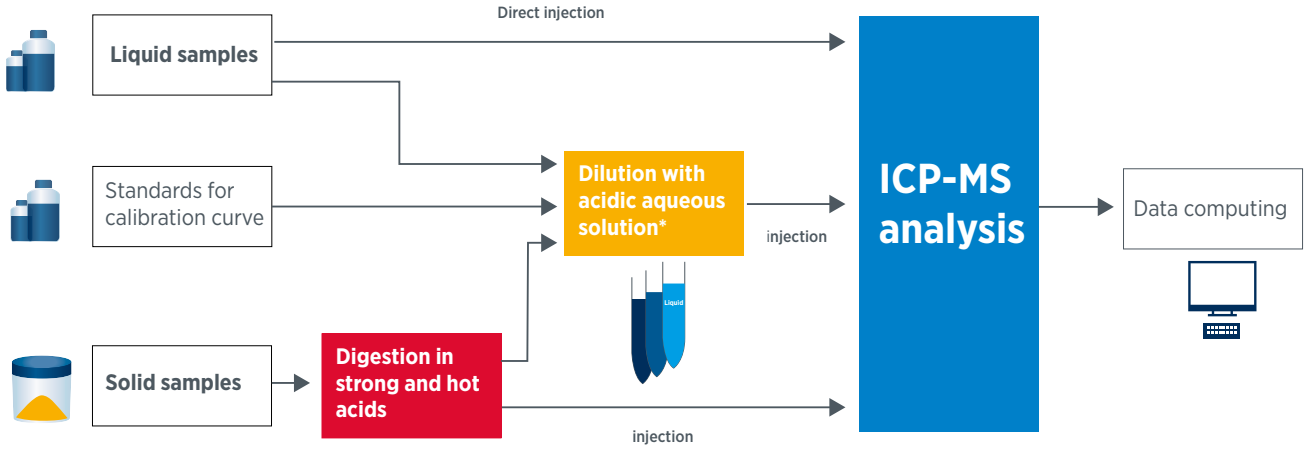
Prior to analysis, the sample is treated with strong acids to dissolve solid material. All samples must be accurately diluted into a concentration range that is inside the specified range for the ICP-MS spectrometer in use.

This technical note shows how the use of a Gilson liquid handler and TRILUTION® LH Software makes it possible to automate those acidic dilutions prior to analysis by ICP-MS.

Automating this process reduces manual handling errors and provides traceability where manual preparation has none.

ICP-MS analysis is a very common technique in environmental laboratories, soil analysis companies, food analysis laboratories, research labs in agronomic/environment, cosmetic industry etc. These methods require the use of liquefied samples; thus acid digestion of solid materials is often required.

ICP-MS WORKFLOW



* Step where automation with GX-271 Liquid Handler could take place

SAMPLE PREPARATION BEFORE ICP-MS

Sample preparation for ICP-MS typically requires acid digestion. This is accomplished by adding concentrated inorganic acids like nitric, sulfuric, or aqua regia, and heating, if necessary. This requires the user to take proper precautions e.g. working in a fume hood and wearing appropriate Personal Protective Equipment (PPE). When it is necessary to destroy a matrix to release an analyte, acid digestion is a common and effective technique. After this digestion, the samples are completely dissolved in the acid solution. Those samples can be directly injected on the ICP-MS detector and may need an additional dilution.

AUTOMATION OF THE ICP-MS ANALYSIS:

The ICP-MS spectrometer is usually coupled with a simple injector, such as a Teledyne CETAC™ using a peristaltic pump for injection, and Bel-Art racks for sample tubes. These autosamplers and injectors are incapable of performing the sample dilution due to their inability to accurately and precisely aspirate and dispense small volumes.

When the number of samples to be processed by ICP-MS analysis increases and generates a bottleneck, automation of sample preparation before injection becomes necessary. The Gilson liquid handlers, especially the GX-271, is a convenient system for this purpose:

- Syringe pump for accurate and repeatable volumes.
- Small footprint of the instrument means it can be installed in a hood.
- Configurations specifically designed to be compatible with strong acids.
- Dedicated configuration to prepare the samples in the racks that are used on the ICP-MS injector.
- User-friendly software for easy programming and running methods.

The GX-271 accommodates the Bel-Art rack on the bed and performs the dilutions directly in the tubes held by the rack. After dilution, the entire rack can be moved to the autosampler used for injection on to the ICP-MS spectrometer (CETAC™ for example).

GILSON SYSTEM DESCRIPTION

Configuration of the system: GX-271 Liquid Handler with a VERITY 4120 Syringe Pump with 10 mL and 1 mL syringes (Figure 2), controlled by TRILUTION® LH. For syringe specifications see Table 1.

Figure 2

GX-271 Liquid Handler with a VERITY® 4120 Dual With Tee Syringe Pump

Table 1

Syringe Specifications

SYRINGE SPECIFICATIONS			
SYRINGE SIZE		**1ML**	**10ML**
10% volume	Accuracy	±1.5%	±1.2%
	Precision	≤0.5%	≤0.5%
50% volume	Accuracy	±0.8%	±0.8%
	Precision	≤0.25%	≤0.25%
100% volume	Accuracy	±0.6%	±0.6%
	Precision	≤0.15%	≤0.15%

PROTOCOLS:

Dilutions: From 1/2 to 1/100 dilution, final volume 10ml (for example). If larger dilution factor is needed, it is done in two steps.

Diluent: It is a very strong acid solution. For example: aqua regia = hydrochloric acid + nitric acid.

The samples are in tubes placed on a Bel-Art rack. Empty tubes for the diluted samples are placed on another Bel-Art rack.

The specials “SPL-832A-HDW coated adapter for one single Bel-Art rack (Figure 3) (vertical)” & “SPL-2263-HDW coated adapter for three Bel-Art racks (horizontal)” are adapters placed on the GX-271 bed layout to allow four Bel-Art racks (Figure 4) to be positioned. Those Bel Art racks are commonly used on the CETAC™ injector ICP-MS analyser.

Figure 3

Bel-Art Racks on the GX-271 Liquid Handler

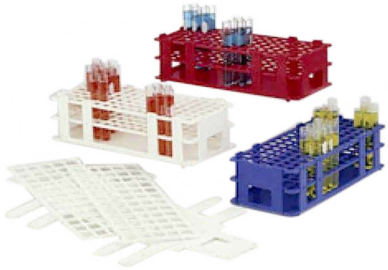


Figure 4

Bel-Art Racks

The diluent is placed on the “reservoir side” aspirated by the 10mL syringe from the inlet tubing and dispensed to the probe side.

The software, TRILUTION® LH, allows you to define the dilution factor at each run, and calculates automatically the volume of the diluent and the volume of the sample. The diluent will be dispensed by the 10mL syringe, the sample will be dispensed by the proper syringe (1mL or 10mL) depending on the sample volume to be dispensed (Figure 5).

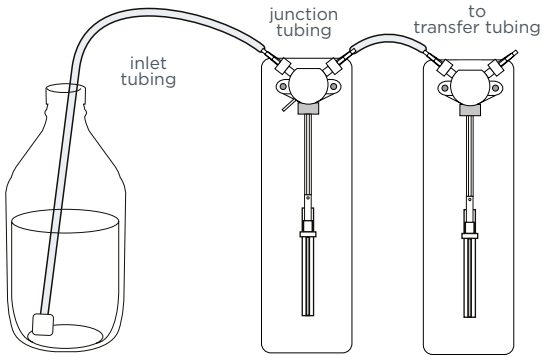


Figure 5

Tubing Connections: Verity® 4120 Dual Tee Syringe Pump

The method step sequence is as follows:

- At the beginning of the run, all the tubing and syringes are primed with the diluent.
- Aspiration of the sample. The volume of the sample is inside the transfer tubing between the probe and the syringe pump.
- Aspiration of the diluent by the 10mL syringe from the reservoir.
- Dispense the sample and the diluent volume into an empty result tube.
- Finally, rinse the outside of the probe with the diluent, in the rinse station.

CONCLUSION

The Gilson GX-271 Liquid Handler with its ICP-MS configuration is a very robust system with very good accuracy and repeatability. The automated dilutions with the GX-271 are not faster than the manual method, but automation will prevent human errors and free technicians to do other tasks during the run. This system is very easy to handle due to the fact that it is using the same racks as the ICP-MS injectors. When the dilutions are finished, the racks are just moved from the dilutor onto the injector. There is no risk from moving the sample tube itself.