Dilution of Metal Samples for Spectroscopic and Spectrometric Analysis using the Microlab 600 Diluter

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Introduction

Atomic Absorption (AA) as well as Inductively Coupled Plasma-Optical Emission Spectroscopy or Mass Spectrometry (ICP-OES or ICP-MS) are frequently used for determination and speciation of metals and metalloids in environmental samples or consumer products.

The AA technique is based on the absorption of specific wavelengths of light by ground state metals after ionization in a flame or graphite furnace. In the other techniques the metals are ionized in a plasma stream by heating of an inert gas stream in an electrical field of high frequency. In ICP-OES the high temperature of the plasma stimulates the sample atoms and element specific radiation is emitted at specific wavelength. A detector identifies the wavelengths of the absorbed or emitted light. In ICP-MS the detector is a mass spectrometer which separates and detects the ionized species according to the mass/charge ratio.

For all of these techniques, accurate sample preparation is essential for proper determination of the metal content. It is common to use highly concentrated acids for extraction and dissolution of the metal ions. The Microlab 600 technology provides outstanding accuracy and precision as well as user-independent handling to ensure excellent analysis results. Since the species of interest are often contained in soil samples an extraction with concentrated acids is necessary very frequently. The Microlab 600 contains a fully inert flow path with PTFE, PE or glass as wetted materials. For this reason the instrument is perfectly suited for handling of corrosive solutions. Moreover, the direct contact between operator and the sample solution is avoided which highly improves the safety at work.

Material & Methods

The Microlab 625 Advanced Dual Syringe Diluter (p/n ML625-DIL) was used for preparing the following dilutions (Dilution factor sample/deionized water):

- 1/10 – (175 dilutions/day)
- 1/20 – (25 dilutions/day)
- 1/50 – (25 dilutions/day)
- 1/100 – (25 dilutions/day)

About 250 samples are processed every day.
The most frequent diluted samples are :

- Nitrohydrochloric acid digestions
- Microwave extractions
- Environmental water samples
- Waste water samples
- Standards (e.g. Mercury (Hg))

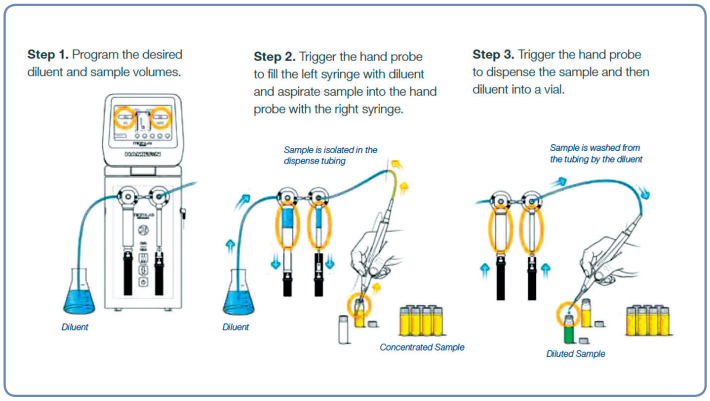

Figure 1: Schematic illustration of the dual syringe dilutor method

The following instruments are used for analysis:

- ICP-MS, Agilent 7500, 7700
- ICP-OEM Varian VistaMPX radial & axial
- AAS Perkin Elmer FIMS 100 & 400

Since the techniques vary in their detection limit, the use of the most suitable equipment depends on the sample and the requirements of the laboratory performing the analysis.

Results and Discussion

The Advanced Microlab Diluter (p/n ML625-DIL) was used for preparation of the dilutions as described above. The Microlab 600 combines the advantages of semi-automation with the flexibility of a manual process. The biggest advantage of using this instrument is the higher number of samples may be analyzed in a very short time with excellent accuracy and precision, providing high reproducibility and less errors compared to manual liquid handling.

In Figures 2 and 3 a mercury standard was used and was diluted to a certain concentration for two times with the Microlab 600. These data demonstrate that the sensitive AA analysis indicate that the Microlab 600 shows excellent accuracy and precision of the diluted sample. The number of processed samples was increased from below 100 samples a day to 250 and more. This illustrates how the Microlab 600 saves time by allowing for faster sample preparation of even high dilutions in one step. Moreover, this positive volume displacement system provides superior accuracy and precision of handling solutions regardless of physical parameters of the sample and diluent solutions.

	Std. conc. µg/L		Blank µg/L	
	Figure 2	Figure 3	Figure 2	Figure 3
Mean	5.633	0.981	0.0754	0.0131
SD	0.026	0.005	0.0004	0.0001
% RSD	0.467	0.492	0.470	0.490

Table 1: Results and standard deviations for measured values and blanks calculated from data shown in Fig. 2 and 3

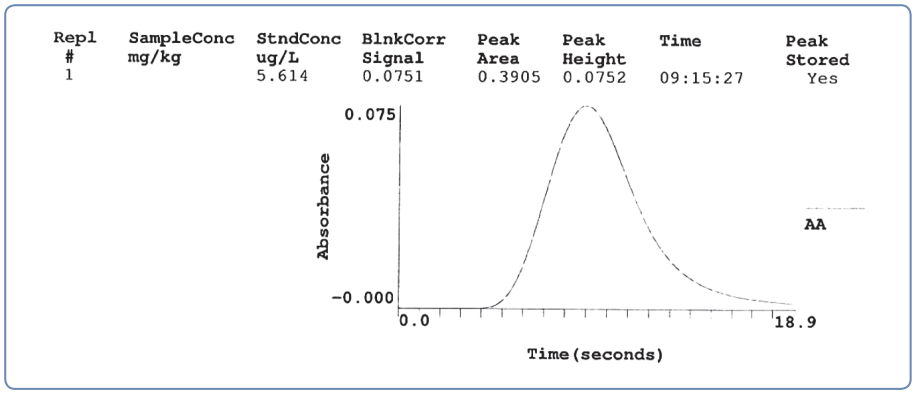

Figure 2: AA plot of high concentrated Hg-standard

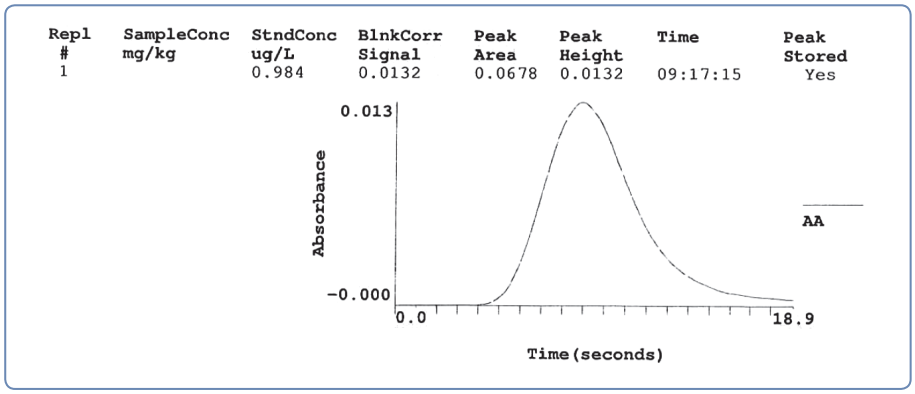

Figure 3: AA plot of low concentrated Hg-standard

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