

# A Discrete Sampling Accessory for ICP-AES and ICP-MS

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## Abstract:

Conventional liquid sample introduction for ICP-AES and ICP-MS uses a peristaltic pump and autosampler to deliver blank, standard, and sample solutions. The time segments required for sample delivery, stabilization, and washout are significant parts of the total analysis time. Reducing the duration of these time segments is of particular interest for high-throughput laboratories.

A discrete sampling accessory will be described which can reduce the above listed time segments, allowing more rapid sample analysis. Advantages include less sample and rinse solution waste, lower argon consumption, less exposure of the ICP-AES and /or ICP-MS hardware to the sample matrix, and elimination of any cumulative sample memory effects from peristaltic pump tubing.

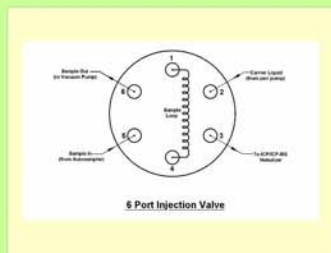
System performance and capabilities will be compared to a conventional sample introduction setup.

## DSA-7 Discrete Sampling Accessory



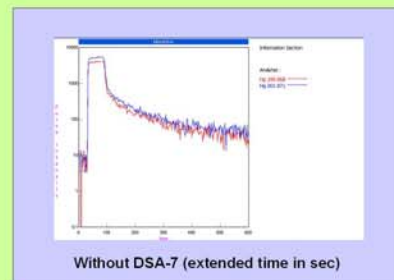
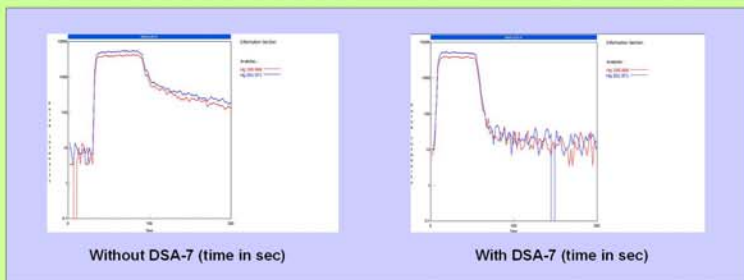
### DSA-7 Features:

- > Inert, high-speed vacuum pump
- > 6-port injection valve
- > Inject/Load indicators
- > Solenoid valve to prevent liquid siphoning
- > Dedicated software interface
- > Connect directly to CETAC autosampler
- > Small footprint (7.1 cm x 10.5 cm) and size (10.5 cm W x 20 cm D x 21.5 cm H)



## Hg Washout for ICP-MS

Hg washout for ICP-MS was tested with and without the DSA-7. The ICP-MS used was a PerkinElmer ELAN DRc equipped with a standard glass cyclonic spray chamber and a Conical glass concentric nebulizer (Glass Expansion, Pocasset, MA USA). The test solution was 1 µg/L Hg in 3% (v/v) hydrochloric acid that was loaded into a 2-mL sample loop.

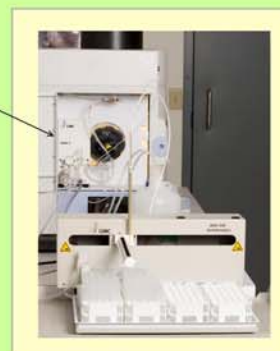


The wash solution used was 2% HNO<sub>3</sub>, pumped at 1.6 ml/min. After more than 2 minutes there is still significant Hg signal with conventional sample introduction; with the DSA-7 the Hg signal is reduced to the original background in ~ 30 sec.

Extending the time scan (left) shows the Hg is still not completely washed out with conventional sample introduction even after 8 minutes.

## Sample Throughput for ICP-AES

Sample throughput for ICP-AES was compared using a normal speed autosampler with and without the DSA-7 accessory. The autosampler used was the CETAC ASX-520 coupled to a PerkinElmer Optima 5300DV ICP-AES instrument. The on-board ICP-AES peristaltic pump was used with the standard nebulizer (Meinhard glass concentric) and spray chamber (glass cyclonic).



### DSA-7 Setup

The picture on left shows the DSA-7 placed next to the standard nebulizer/spray chamber of the ICP-AES instrument. The sample uptake line from the ASX-520 autosampler is attached directly to the 6-port injection valve.

Note that the vacuum pump used for sample loading is built into the DSA-7 module, just to the right of the injection valve.

A communication cable from the bottom front of the DSA-7 connects to an auxiliary communications port on the back of the autosampler.

A simple software program, installed into the host ICP-AES computer, is used to set the sample loading and probe wash times.

## Comparison of Experimental Parameters

Two runs of 120 samples (2 x 60-position racks) each were performed with and without the DSA-7. After calibration with a 1% HNO<sub>3</sub> blank and a 200 µg/L 20-element standard, the same blank and standard solutions were placed in alternate sample vials in the 60-position racks. Five replicate measurements with a 2-second integration time were performed for each sample, loaded into a 2-mL sample loop. Sample introduction timing values are listed below:

Setup	Pump Rate (mL/min)	Flush Time (s)	Wash Time (s)	Loop Load Time (s)	Read Delay (s)
Without DSA-7	1.50/3.00	38	30	NA	8
With DSA-7	1.50	5	0	6	8

Note during the flush cycle without the DSA-7, the peristaltic pump rate is increased to 3.0 mL/min. Without the DSA-7, the total sampling time was **3 hours and 55 minutes**. With the DSA-7, the total time was only **1 hour 30 minutes**, a reduction in analysis time of over 60%.

Time savings is provided by the very rapid loading (via the vacuum pump) of the fixed volume sample loop, the rinsing of the autosampler probe (also via the vacuum pump) during the sample measurement phase, and the very short distance between the injection valve and the nebulizer that enables faster sample washout.

