Micro Volume Sample Introduction System for ICP-MS

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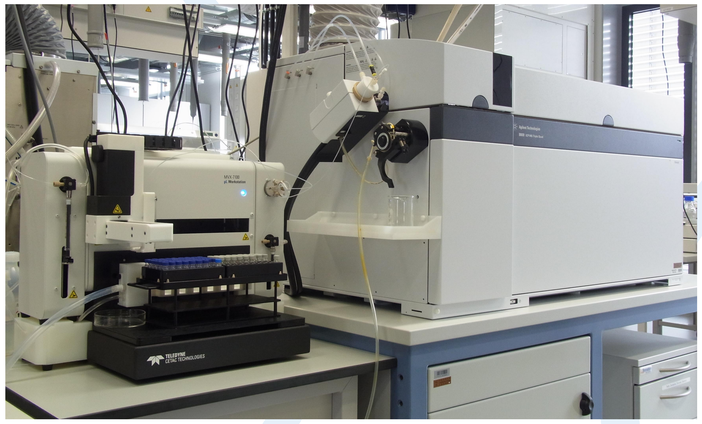
Introduction

There is a growing demand for the sampling and introduction of small liquid sample volumes to ICP-MS instrumentation. Numerous publications have noted that a limiting factor for some ICP-MS measurements is sample volume. Other limiting sample introduction factors include working with toxic or volatile samples that would be best handled in sealed vials and the chemical compatibility of the liquid facing flow path between the sample source and the ICP instrumentation. In this work we describe a micro-volume workstation that meets these requirements.

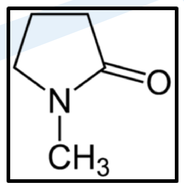


MVX-7100 µL Workstation.

- **Injection range (5µL to 1mL)**
- **Septum piercing**
- **Syringe driven**
- **Temperature controlled**

Liquid Crystal/Semiconductor

The solvent N-Methyl-2-Pyrrolidone (NMP) is a sample type that is difficult to work with, analytically, in its concentrated form – one of the uses of NMP is the breakdown of polymer materials so standard automation using peristaltic pump tubing does not work for batch analysis. NMP is also a costly reagent to work with so it is preferable to work with low volumes. NMP is a solvent used in liquid crystal material manufacturing as well as in the semiconductor industry, it also has applications in the pharmaceutical and petrochemical industries. For such applications it is critical that the trace element composition of NMP is as low as possible, therefore it is subject to quality control testing using ICP-MS.



N-Methyl-2-Pyrrolidone (NMP).

Method

To prove the concept of routine ICP-MS analysis for quality control of NMP the MVX-7100 µL workstation was used as part of analysis of trace element spiked NMP samples which were calibrated against NMP based calibration standards. NMP calibration standards were prepared by hand and then the MVX-7100 was used to introduce samples, standards and blank solutions to an Agilent 8800 ICP-QQQ-MS for measurement. 200 µL sample aliquots were taken up for introduction from septum sealed vials and introduced via a 50 µL min^{-1} nebulizer. This arrangement allowed approximately 3 minutes of stable analysis time for the measurement of 16 isotopes against 2 internal standard isotopes via 3 different ICP-MS measurement modes.

Table 1: Element calibration data with QQQ method listed.

Element	Mass	Method	R^2
Boron	10	Helium	0.9989
Phosphorus	31	Helium	0.9992
Sulfur	34	Helium	0.9993
Chlorine	35	Helium	0.9997
Chlorine	35	Hydrogen 35→37	1.000
Iron	56	Helium	0.9991
Copper	63	Helium	0.9999
Iron	56	Oxygen 56→72	0.9992
Bromine	79	Hydrogen	0.9998
Bromine	79	Oxygen	0.9999
Palladium	105	Helium	0.9999
Iodine	127	Helium	0.9999

Table 1 shows that the calibration linearity of the NMP analysis was of the order of 0.999 or better for the elements of interest. The 8800 ICP-MS was optimized with different collision/reaction gas modes for the attenuation of interfering polyatomic species. Quantified data from trace element spiked NMP for this testing is shown in Table 3. As part of this testing we also showed that the MVX-7100 with its metal free liquid facing flow path delivers low blank signal compared with similar automation systems (with metallic components) for HPLC analysis (see Table 2).

Table 2: Comparison for Iron contamination between the MVX and an HPLC autosampler.

Element / Isotope	Measured m/z	Measurement mode	Blank (CPS) MVX	Blank (CPS) HPLC A/S
^{56}Fe	56	Collision Cell (Helium)	2831.60	11954.97
^{56}Fe	72	Collision Cell (Oxygen)	853.62	2429.58

Results

Table 3: Recovery data for a spiked sample of NMP.

Element / Isotope	Measured m/z ratio	Measurement mode	RSD (%)	Target Concentration (ppb)	Measured (ppb)	Recovery (%)
^{10}B	10	Helium	6.12	300	314.55	104.85
^{31}P	31	Helium	1.70	300	312.56	104.19
^{35}Cl	35	Helium	3.31	1500	1481.56	98.77
^{31}P	47	Oxygen	0.60	300	304.62	101.54
^{32}S	48	Oxygen	1.00	3000	3101.37	103.38
^{35}Cl	37	Hydrogen	1.26	1500	1481.43	98.76
^{56}Fe	56	Helium	2.03	30	19.72	65.73
^{63}Cu	63	Helium	1.12	30	28.22	94.07
^{56}Fe	72	Oxygen	1.95	30	19.84	66.13
^{79}Br	79	Helium	1.31	300	302.64	100.88
^{79}Br	80	Hydrogen	0.39	300	307.95	102.65
^{79}Br	95	Oxygen	0.74	300	310.68	103.56
^{105}Pd	105	Helium	0.82	30	29.85	99.50
^{127}I	127	Helium	0.91	30	29.41	98.03

As shown in Table 3, all elements were quantified to within 6 % of the target concentration, depending on the QQQ detection method, except for iron. The discrepancy with iron was found to be carryover from the previous analysis that was washing out.

Conclusions

The MVX-7100 used in conjunction with ICP-MS instrumentation was shown to be fit for purpose for routine analysis of low volumes (in this case volumes of the order of 200 µL) of a difficult matrix. The sample injection flexibility of the MVX-7100 workstation paired with the detection capability of the Agilent 8800 ICP-QQQ-MS produces an analytical tool for element detection in specialty applications as well as for challenging sample volumes without analytical compromise.