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Application Note

Reduction of Solvent-Based Interferences Using Ultrasonic Nebulization/Membrane Desolvation for Measurement of Trace Elements in Isopropyl Alcohol with Quadrupole ICP-MS Detection

Introduction

Determination of trace elements in organic solvents with quadrupole ICP-MS detection is problematic for several reasons: plasma instability and/or failure, carbon buildup on the ICP-MS sampling cones, and solvent-based (carbon-containing) mass spectral interferences. A specialized sample introduction accessory can reduce solvent-based interferences and enhance trace element detection using a combination of ultrasonic nebulization, membrane desolvation, and (if required) kinetic energy dissociation (KED) gas addition.

The Teledyne CETAC U6000AT+ Ultrasonic Nebulizer / Membrane Desolvator removes much of the sample solvent during analyte transport to the host ICP-MS, allowing stable plasma operation. For this ICP-MS application, the Teledyne CETAC BGX-100 Blend Gas Accessory is also used to add a low flow of oxygen between the U6000AT+ and the ICP-MS torch. This oxygen gas addition prevents buildup of carbon on the ICP-MS torch and sampler/skimmer cones. Helium KED gas addition completes the method development for improved detection of selected trace elements in isopropyl alcohol (IPA) using quadrupole ICP-MS. Isopropyl alcohol is of interest as this solvent is used in silicon wafer (semiconductor) processing, an application which requires very low reagent blank levels to minimize contamination and possible wafer failure.

Equipment Setup

A picture of the U6000AT+ Ultrasonic Nebulizer / Membrane Desolvator is given in Figure 1; the quadrupole ICP-MS used for trace element detection was the PerkinElmer NexION 300D. A schematic diagram of setup connections is given in Figure 2.

Figure 1. Teledyne CETAC U6000AT+ and BGX-100

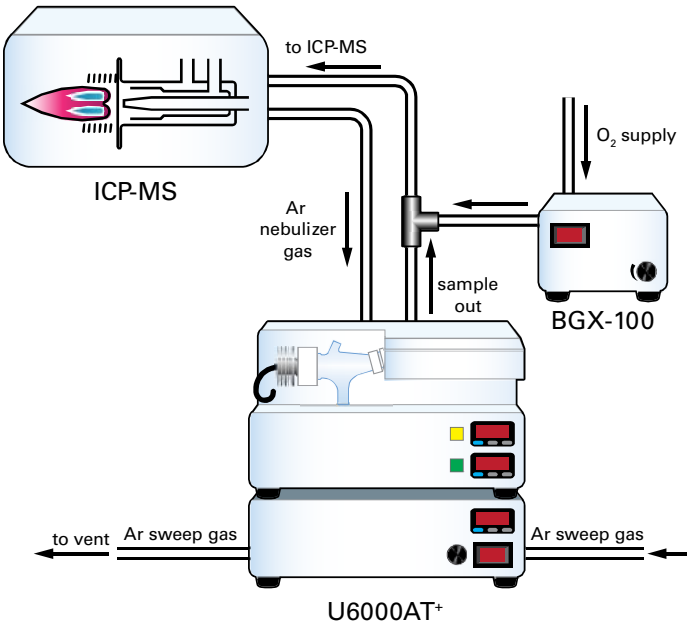

Figure 2. Schematic Diagram of ICP-MS Setup w. U6000AT+ and BGX-100

Operating Conditions

Operating conditions for the NexION 300D ICP-MS after Syngistix™ software optimization are given in Table 1, including the He KED gas flow setting. Operating conditions for the U6000AT+ and BGX-100 combination are given in Table 2.

Table 1. PerkinElmer NexION 300D ICP-MS Operating Conditions

Parameter	Setting
RF Power	1200 W
Plasma Gas	14.0 L/min
Nebulizer Gas	0.40 L/min
Auxiliary Gas	0.88 L/min
Helium (for KED)	5.0 mL/min
Torch injector	2.0 mm Quartz
Sampler and Skimmer Cones	Nickel
Sweeps/replicate	50
Number of replicates	3
Integration time	2.5 s

Table 2. Teledyne CETAC U6000AT+ and BGX-100 Operating Conditions

Parameter	Setting
Ultrasonic Nebulizer Heater Temperature	140°C
Ultrasonic Nebulizer Cooler Temperature	0°C
Membrane Oven Temperature	160°C
Ar Sweep Gas Flow	1.80 L/min
Sample Uptake Rate	1.0 mL/min
O_2 Gas Flow Rate (BGX-100)	10 mL/min

Calibration

The ICP-MS was calibrated by introducing a blank and four multielement standards at 20, 50, 100, and 200 ng/L spiked in ACS reagent grade isopropyl alcohol (IPA); all standards were prepared by weight (using density) in low density polyethylene (LDPE) bottles. Calibration curves must be 0.995 or better to be considered valid. Note that calibration curves for Fe, Mn, and Sn begin at 50 ng/L and include 2 additional standards at 100 ng/L and 200 ng/L.

The blank and standards were pumped to the U6000AT+ transducer using PVC peristaltic pump tubing; no internal standards were used for calibration.

Detection Limits

Instrument detection limits (IDLs) were determined for the elements listed in Table 3 by measuring an isopropyl alcohol blank ten times after calibration; the IDLs are calculated as 3x the standard deviation of the blank concentration. As oxygen addition was used during analysis, KED mode with He gas addition was necessary to decrease oxide levels for some analytes. Note that these IDLs were obtained under non-cleanroom conditions.

Table 3. Instrument Detection Limits

Element	m/z	Calibration Range (ng/L)	IDL (ng/L)	Mode
Ag	107	20-200	1.4	Standard
Al	27	20-200	2.5	Standard
Ba	137	20-200	0.20	Standard
Cd	111	20-200	1.0	Standard
Co	59	20-200	0.89	KED
Cr	52	20-200	6.6	KED
Cu	63	20-200	4.2	Standard
Fe	57	50-200	12.9	KED
Li	7	20-200	2.4	Standard
Mg	24	20-200	9.1	Standard
Mn	55	50-200	12.6	KED
Mo	98	20-200	0.94	Standard
Ni	60	20-200	1.2	KED
Pb	208	20-200	1.1	Standard
Sb	121	20-200	1.7	Standard
Sn	120	50-200	6.9	Standard
Ti	48	20-200	7.5	Standard
V	51	20-200	0.36	Standard

Reduction of Interferences

Several elements had severe carbon-based interferences at highest % abundant isotope masses: ^{24}Mg, ^{27}Al, and ^{52}Cr. Using the NexION 300D ICP-MS with the U6000AT+ and BGX-100, background at these masses were reduced so that sub-ppb calibration was possible. Detailed information including calibration coefficients and background equivalent concentrations (BECs) are given in Table 4; full calibration curves are depicted in Figures 3a, 3b, and 3c.

Table 4. Background and Calibration Information for Mg, Al, Cr

Analyte	m/z	Blank (cps)	BEC (ng/L)	Calibration Corr.
Mg	24	2343	18.8	0.9999
Al	27	420	2.7	0.9992
Cr	52	8	7.4	0.9996

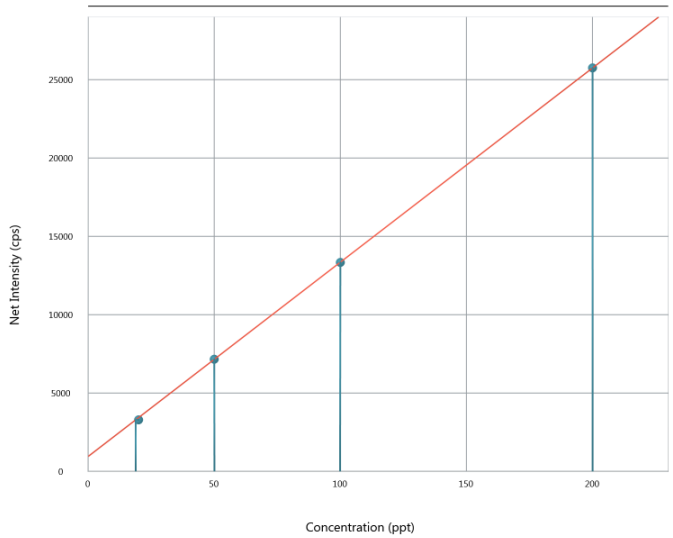

Figure 3a. Calibration Curve for ^{24}Mg

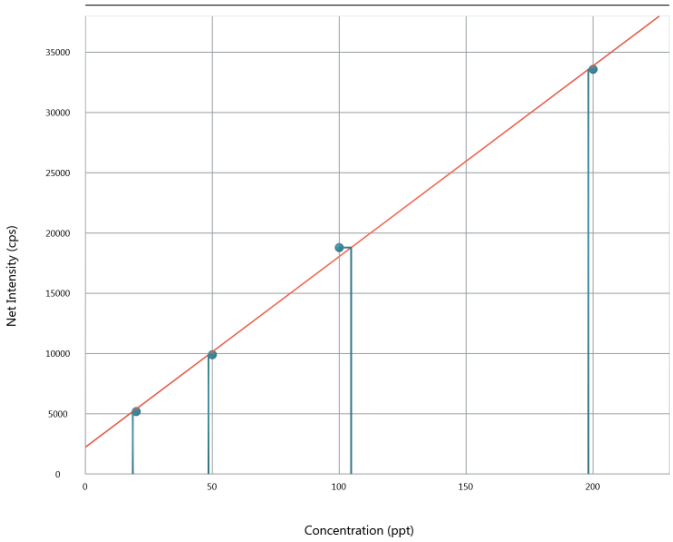

Figure 3b. Calibration Curve for ^{27}Al

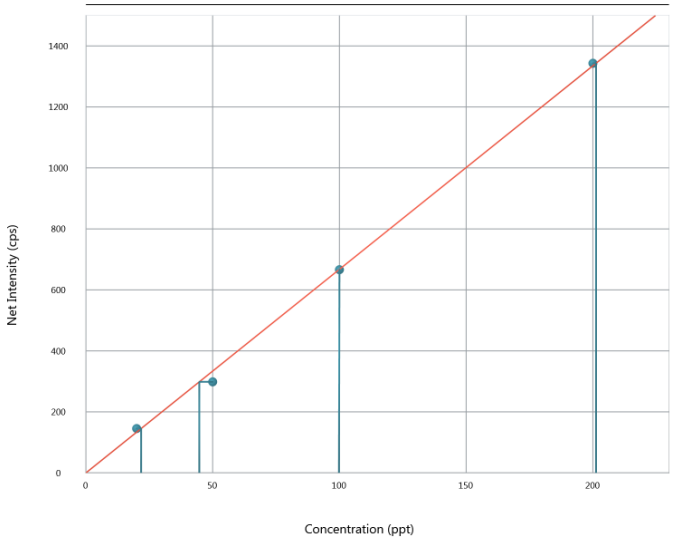

Figure 3c. Calibration Curve for ^{52}Cr

Spike Recovery

An isopropyl alcohol aliquot was spiked at the lowest concentration of 20 ng/L and introduced to the NexION 300D ICP-MS using the U6000AT+ and BGX-100 combination. Spike recovery results are in given in Table 5; Mn, Sn, and Fe are not included as the lowest standard of their curve is 50 ng/L. All recoveries are within 85% to 115%.

Table 5. 20 ng/L Spike Recoveries in Isopropyl Alcohol

Element	m/z	Measured (ng/L)	% Recovery
Ag	107	22.3	112
Al	27	19.5	97
Ba	137	20.9	105
Cd	111	21.1	106
Co	59	17.2	86
Cr	52	22.4	112
Cu	63	19.6	98
Li	7	17.5	88
Mg	24	23.0	115
Mo	98	18.1	90
Ni	60	19.1	95
Pb	208	21.1	106
Sb	121	18.5	93
Ti	48	21.0	105
V	51	21.9	109

Conclusion

The Teledyne CETAC U6000AT+ Ultrasonic Nebulizer/Membrane Desolvator with the BGX-100 Blend Gas Accessory alleviates inherent problems associated with direct analysis of reagent grade isopropyl alcohol using quadrupole ICP-MS detection. With a simple, rapid setup and straightforward method development, carbon-based spectral interferences can be reduced such that IDLs in a range of 0.2 to 15 ng/L can be achieved for 18 trace elements of interest under non-cleanroom conditions.

Note

If the ICP-MS is equipped with an oxygen addition capability, then this oxygen supply can be used in place of the Teledyne CETAC BGX-100.

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