

Robust and sensitive analysis of acetonitrile using ICP-MS

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Keywords

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Goal

To assess and demonstrate the performance of the Thermo Scientific[™] iCAP[™] RQ ICP-MS equipped with the PLUS Torch for the analysis of organic solvents such as acetonitrile.

Introduction

Acetonitrile is an important solvent that is used in a variety of applications in different industries. Because of its miscibility with both water and numerous organic solvents, it is widely used as a mid-polar solvent in various laboratory processes. Due to its dipole moment and a high dielectric constant, acetonitrile dissolves a wide range of ionic and nonpolar compounds; for example, it is used to solubilize organic carbonates commonly used as electrolytes in modern Li-ion batteries. For similar reasons, it is a popular solvent in cyclic voltammetry as well. Acetonitrile is transparent in the ultraviolet range, and together with its low viscosity and low chemical reactivity, it is a popular choice for high-performance liquid chromatography (HPLC) and HPLC-mass spectrometry (MS) applications. Acetonitrile plays a significant role as a solvent used in the manufacturing of DNA oligonucleotides from monomers.

On an industrial scale, acetonitrile is produced as a byproduct in the production of acrylonitrile and is used in the production of 1,3-butadiene. In addition, it is used as a solvent for the manufacturing of pharmaceuticals and photographic film. In some cases, acetonitrile is used as solvent for standards and sample preparation while performing analysis of active pharmaceutical ingredients (API) in accordance with USP Chapters <232> and <233>. Due to its wide range of applications, analysis of acetonitrile for elemental contaminants at trace level is crucial.

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Direct analysis of volatile organic solvents, such as acetonitrile but also methanol or other alcohols, using ICP-MS is challenging mainly due to their high vapor pressure, causing plasma destabilization and carbon deposition on interface cones due to incomplete combustion.

Another challenge associated with this type of analysis is the need for more frequent inspection, maintenance, and exchange of components in the sample introduction system, like injector tubes and torches. Direct aspiration of organic solvents enhances plasma temperature considerably, mostly due to the more intense UV emission. Conventional torches made of high purity quartz may deteriorate and crack quickly as they suffer from devitrification of the torch material, affecting overall analytical performance and productivity of ICP-MS analysis.

To overcome these challenges, the novel PLUS Torch, compatible with all Thermo Scientific[™] iCAP[™] Qnova[™] Series ICP-MS, has been developed. This torch is made of a high purity and high-performance ceramic material, providing significantly improved measurement stability and increased lifetime with less frequent maintenance requirements.

Ceramic torches are widely used with ICP-OES instruments and are a proven solution for the analysis of challenging matrices, such as samples containing large amounts of dissolved solids, high concentrations of aggressive acids, and organic solvents. The PLUS Torch offers another key benefit over conventional quartz torches in reducing backgrounds observed for some analytes, especially silicon (Si), hence enabling more accurate trace level analysis of this element. This enhanced performance is also beneficial for the determination of impurities in metals and metallurgical products, chemicals, crude oil and refinery products, pharmaceutical analysis, and environmental analysis.

Experimental

Instrument parameters and experimental conditions

A Thermo Scientific[™] iCAP[™] RQ ICP-MS equipped with a 250 mL·min⁻¹ additional mass flow controller was used in this study. To allow for unattended operation, the system was operated in conjunction with a Teledyne[™] CETAC[™] ASX-560 autosampler (Teledyne CETAC Technologies, Omaha, NE, USA). The sample introduction system was configured for the analysis of organic samples using the components summarized in Table 1. A constant flow of pure oxygen (equivalent to 20% of the specified output of the mass flow controller) was introduced into the spray chamber elbow to ensure complete combustion of carbon, thereby preventing carbon deposition on the interface cones.

Table 1. Instrument configuration and typical operating parameters

Parameter	Value
Nebulizer	PFA microflow nebulizer (100 μL·min ⁻¹)
Interface cones	Pt – tipped sample and skimmer
Skimmer cone insert	High matrix
Spray chamber	Cyclonic quartz
Injector	Quartz, 1.0 mm i.d.
Torch	PLUS Torch
Auxiliary gas flow (L·min-1)	0.8
Cool gas flow (L-min-1)	14
Nebulizer gas flow (L·min-1)	0.57
Additional gas flow $(\%)^*$	20
RF power (W)	1,550
Sampling depth (mm)	6
Number of replicates	3
Spray chamber temp. (°C)	2.7
KED settings	Helium, 4.8 mL·min ⁻¹ ,
(Flow rate in mL·min-1)	3 V energy barrier
Number of sweeps	10
Dwell time (s)	0.05

* Relative setting of the total capacity of the mass flow controller used. Pure oxygen was used as additional gas to aid in the combustion of carbon.

Sample and standard preparation

To determine analytical figures of merit, such as detection sensitivity and instrumental detection limits (IDLs), a calibration curve was generated for 29 analytes using five linearity standards and a calibration blank. Linearity standards were prepared using an aqueous multi-element stock solution (10 mg·L⁻¹, SPEX[™] CertiPrep[™], Metuchen, NJ, USA). The aqueous standard was then diluted gravimetrically using acetonitrile as a diluent to result in the concentrations specified in Table 2. An internal standard solution containing 10 µg·L⁻¹ of Sc, Ge, Y, Te, Tb, and Th was added on-line during the entire duration of the measurement.

All 35 analytes (including the internal standards) were measured using kinetic energy discrimination (KED) mode, with pure helium used as a collision gas. Pure acetonitrile was used as a technical replicate during a long-term analytical sequence of 28 hours of continuous analysis. A total of 450 samples were analyzed during this robustness study, including 400 unknowns.

Table 2. List of target elements and concentrations in calibration standards in µg· L-1

Elements	Standard 1	Standard 2	Standard 3	Standard 4	Standard 5
Li, Be, Na, Mg, K, Ca, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, As, Se, Rb, Sr, Ag, Cd, In, Cs, Ba, Hg, Tl, Pb, Bi ,U	0.1	0.5	1	5	10

Linearity and instrument detection limits

Instrumental detection limits for all analytes are summarized in Table 3. Excellent detection limits were achieved for most of the analytes, except for typical major elements like Na, Mg, K, Ca, and Zn. These elements were found to be present in high concentrations in the acetonitrile used in this study, hence limiting the achievable detection limits due to an elevated background. As there is no upfront dilution or sample preparation involved before analysis, instrument detection limits readily equate method detection limits (MDLs).

Method accuracy

To assess accuracy of the entire analytical set-up, a QC standard containing 1 μ g·L⁻¹ of all analytes investigated in this study was prepared independently using the same stock solution and analyzed every 10 samples throughout the entire analytical run. Figure 1 shows the average recovery over all readings of the QC standard (n=43). The average percent recovery observed for all analytes was found to be in the range of 90% to 110%, with a relative standard deviation of less than 10%.

System robustness

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During the entire analytical sequence, the response of all internal standards was monitored to assess the robustness of the proposed method and the instrumental set-up. Figure 2 shows the response of the internal standards obtained throughout an analytical run. As noted above, pure acetonitrile was used as unknown samples, and a QC standard was measured every 10 samples. The response of all internal standards read back in the range between 90% and 115% of their initial values, indicating that the proposed analytical set-up is suitable for the analysis of longer batches containing such types of challenging matrices.

Table 3. List of analytes, m/z, measurement mode, correlation coefficients, and instrumental detection limits (all results expressed as μ g-L⁻¹)

Element	m/z	Mode	R ²	IDL
Li	7	KED	0.9997	0.007
Be	9	KED	0.9998	0.008
Na	23	KED	0.9996	0.211
Mg	24	KED	0.9994	0.785
К	39	KED	0.9981	0.143
Ca	44	KED	0.9981	0.453
V	51	KED	0.9992	0.006
Cr	52	KED	>0.9999	0.007
Mn	55	KED	>0.9999	0.011
Fe	57	KED	0.9997	0.08
Со	59	KED	>0.9999	0.002
Ni	60	KED	>0.9999	0.01
Cu	63	KED	>0.9999	0.012
Zn	66	KED	>0.9999	0.321
Ga	71	KED	>0.9999	0.004
As	75	KED	>0.9999	0.003
Se	78	KED	0.9987	0.04
Rb	85	KED	>0.9999	0.006
Sr	88	KED	>0.9999	0.002
Ag	109	KED	>0.9999	0.003
Cd	111	KED	>0.9999	0.002
In	115	KED	0.9995	0.002
Cs	133	KED	>0.9999	0.001
Ва	137	KED	0.9994	0.003
Hg	202	KED	>0.9999	0.002
TI	205	KED	>0.9999	0.001
Pb	208	KED	>0.9999	0.002
Bi	209	KED	>0.9999	0.0006
11	238	KED		0.0005



Figure 1. Average % recovery of 1 µg·L⁻¹ QC standard over 28 hours (n=43)





At the end of the run, all components of the sample introduction system were removed and examined carefully. No visible signs of deposition, deterioration, or damage were observed, and the analysis could have been continued further with no maintenance required. Figure 3 shows an image of the PLUS Torch used for this analysis after completion of the run.



Figure 3. PLUS Torch after continuous measurements of acetonitrile samples for 28 hours

Conclusions

- The quality of the analytical data and the robustness observed in the study suggests that the iCAP RQ ICP-MS in conjunction with the PLUS Torch is a highly robust and reliable solution for the analysis of challenging sample types like acetonitrile.
- Instrument detection limits and consistent recovery of the QC standard indicate excellent robustness and stability without compromising analytical sensitivity or affecting attainable detection limits.
- The novel PLUS Torch minimizes the need for cleaning and potential frequent replacement of the torch, reducing downtime and increasing overall productivity of the laboratory operations.
- The proposed analytical set-up can be easily transferred to, for example, the Thermo Scientific[™] iCAP[™] TQ ICP-MS, offering superior interference removal and improved detection limits for key elements, such as sulfur or phosphorous³.

References

- 1. https://www.sciencedirect.com/topics/chemistry/acetonitrile
- 2. Thermo Scientific Specification Sheet 44485: <u>PLUS Torch for iCAP Qnova Series</u> ICP-MS
- 3. Thermo Scientific Application Note 43446: <u>Addressing the challenges of routine</u> <u>determination of elemental impurities in refinery products using a robust</u> <u>ICP-MS approach</u>

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