# thermo scientific

**APPLICATION NOTE 43444** 

High matrix tolerance of the Thermo Scientific iCAP 7000 Plus Series ICP-OES with the radial Ceramic D-Torch and the sheath gas adaptor

#### **Authors**

Nora Bartsch, Application Specialist, Thermo Fisher Scientific, Bremen, Germany

#### **Keywords**

Ceramic D-Torch, high matrix, NaCl, robustness, salt, sheath gas adaptor

### Goal

This application note describes how routine analysis of high matrix samples like 25% NaCl can be performed with the Thermo Scientific iCAP 7400 ICP-OES Radial fitted with the additional gas accessory in combination with the Ceramic D-Torch and the sheath gas adaptor.

#### Introduction

The sample introduction system of an ICP-OES has a large influence on the analytical performance of the spectrometer and is the main area of the hardware that users interface with. One key component of this system is the ICP torch. The ICP torch is a relatively high cost consumable item, which can require regular maintenance and replacement when performing more demanding applications. Currently, the majority of ICP torches are made from quartz, which is a crystalline form of silicon dioxide (silica). When a quartz ICP torch is heated (by the plasma) it can undergo a process known as devitrification (which means becoming less glasslike). The process of devitrification can decrease the expected lifetime of the ICP torch and is commonly seen when samples are analyzed that contain high concentrations (greater than 1000 mg·L<sup>-1</sup>) of group I or group II elements (Figure 1).



To overcome these problems the Ceramic D-Torch was developed (Figure 2). The outer tube of the Ceramic D-Torch is made from sialon, which is a ceramic material derived from silicon nitride. The intermediate tube is made from alumina, which again has excellent properties for chemical and temperature resistance and has been proven as a material for ICP torches as it is commonly used for center tubes. When analyzing samples that contain elements that promote the devitrification process, the Ceramic D-Torch does not suffer from effects of devitrification, this is demonstrated by the analyses of 25% NaCl solution.

When measuring high solid samples the major problem is that salts can crystallize at the tip of the torch injector tube and restrict sample transport to the plasma. Any change in the sample transport efficiency to the plasma will result in a change of signal and cause analytical drift. To prevent crystallization of the sample in the injector tube, a sheath gas can be used. A sheath gas envelopes the sample aerosol tangentially, preventing contact with the injector tube, reducing the deposition of the sample on the injector tube. As little or no sample deposition is taking place, the need for extended washing phases between samples is negated. As the use of a sheath gas enables a higher tolerance for total dissolved solids (TDS), less sample dilution is required, hence the application of the sheath gas adaptor improves method detection limits. The sheath gas is introduced between the spray chamber and the torch (Figure 3) using the sheath gas adaptor, which must be supplied with a constant gas flow of argon via a mass flow controller.



Figure 2. The radial D-Torch ceramic outer tube (A) and the radial D-Torch, with a quartz outer tube to show the geometry of the intermediate tube (B).



Figure 3. Radial sheath gas adaptor.



Figure 1. Examples of devitrification (A) and premature failure (B) of quartz ICP torches.

#### Instrumentation

The analysis of salt-containing samples is a challenge for ICP-OES. For this analysis the iCAP 7400 ICP-OES Radial fitted with the additional gas accessory was used with a sample introduction kit consisting of the components listed in Table 2. The radial system was chosen because of its high matrix tolerance and its ability to optimize the radial viewing height. The optimization of the radial viewing height helps to reduce spectral interferences. This is critical as interferences from carbon based emissions can be reduced especially in the visible region of the spectrum. Salt tends to be deposited in the nebulizer tip, changing the nebulization efficiency, resulting in signal drift and high RSDs. Therefore a Burgener Mira Mist® Nebulizer was used to minimize salt deposition in the nebulizer tip. To allow efficient analysis of salt-containing samples as well as to overcome any challenges associated with such analysis a sheath gas adaptor was used, which prevents salt deposition forming on the inner walls of the center tube. A Teledyne CETAC ASX-560 Autosampler was used to transfer the sample to the introduction system of the ICP-OES.

#### Sample preparation

High matrix blank, calibration standards and stability solutions were prepared from single element solutions (1000 mg·L<sup>-1</sup>, SPEX CertiPrep Group, Metuchen, US), according to the concentrations in Table 1. The individual solutions were made with 18 M $\Omega$  ultra-pure water and trace metal grade nitric acid (67-69%, Fisher Chemical, Loughborough, UK) as well as common table salt, to a final concentration of 25% NaCl as well as 0.2% HNO<sub>3</sub>. All solutions were spiked with 10 mg·L<sup>-1</sup> yttrium (1000 mg·L<sup>-1</sup>, SPEX CertiPrep).

Table 1. Calibration standards and sample concentrations (µ	g∙L⁻	1)
---	------	----

Element and wavelength (nm)	Blank	Standard 1 (μg⋅L⁻¹)	Standard 2 (μg⋅L⁻¹)	Standard 3 (µg⋅L⁻¹)	Stability solution (µg·L⁻¹)
AI 167.079	0	100	250	500	500
Ba 455.403	0	100	250	500	500
Co 228.616	0	100	250	500	500
Cr 205.560	0	100	250	500	500
Cu 324.754	0	100	250	500	500
Fe 259.940	0	100	250	500	500
Mg 279.553	0	100	250	500	500
Mn 257.610	0	100	250	500	500
Ni 221.647	0	100	250	500	500
Sr 216.596	0	100	250	500	500
Zn 202.548	0	100	250	500	500

#### Method development and analysis

A LabBook was created using the Thermo Scientific™ Qtegra<sup>™</sup> Intelligent Scientific Data Solution<sup>™</sup> (ISDS) Software. For each element, wavelengths were selected using the intuitive wavelength selection tool of the Qtegra ISDS Software. The wavelengths used for analysis are shown in Table 1, these were selected as they were free from interferences and provided the sensitivity to quantify the elements of interest in the expected concentration range. Optionally, these steps can be simplified by using the Qtegra ISDS Software Element Finder plug-in. The plasma was ignited and the instrument allowed to warm up for a period of 30 minutes. Various instrument parameters were applied to reach optimal plasma conditions. The parameters used for the method can be found in Table 2. A torch alignment and a spectrometer optimization were performed directly before analysis and an AutoPeak was run within the LabBook prior to analysis. A detection limit study was carried out by analyzing the calibration blank three times with ten replicates and multiplying the standard deviation of this analysis by three. To ensure freedom from interferences, the subarray plots were examined and background correction points were set appropriately.

#### Table 2. Instrument parameters.

Parameter	Setting
Pump Tubing (Standard Pump)	Sample Tygon <sup>®</sup> orange/white Drain Tygon <sup>®</sup> white/white
Spray Chamber	Baffled cyclonic
Nebulizer	Burgener Mira Mist
Center Tube	2.0 mm (ceramic)
Torch	Ceramic D-Torch
Pump Speed	50 rpm
Flush Pump Speed	100 rpm
Pump Stabilization Time	10 s
Wash Time	30 s
Nebulizer Gas Flow	0.55 L∙min⁻¹
Auxiliary Gas Flow	0.5 L·min <sup>-1</sup>
Coolant Gas Flow	12 L∙min <sup>-1</sup>
Additional Gas Flow	0.1 L·min <sup>-1</sup>
RF Power	1400 W
Radial Viewing Height	10 mm
Exposure Time	UV 15 s, Vis 15 s

### Results

The linearity of the calibration curves is very good with correlation coefficients greater than 0.999 for each wavelength. Exemplary calibration curves for Ba 455.403 nm and Al 167.079 nm are shown in Figures 6 and 7. Recoveries of the spiked samples are within a range of 97-104% (Table 3). The 10 hour long-term stability data also show recoveries within the range of 95-105% (Figures 4 and 5). The method detection limits (MDL) for most elements are in the single digit  $\mu$ g·L<sup>-1</sup> range or lower (Table 3).

Table 3. Analytical wavelength, correlation coefficient R<sup>2</sup> and internal standard used for the analysis, as well as spike recoveries and method detection limit (MDL) for each element.

Element and wavelength (nm)	R²	Internal standard wavelength (nm)	Known spike concentration (μg·L <sup>-1</sup> )	Measured spike concentration (μg·L <sup>-1</sup> )	Recovery (%)	MDL (µg∙L⁻¹)
AI 167.079	0.9997	Y 224.306	500	497.2	97.2	4.1
Ba 455.403	1.0000	Y 317.030	500	499.9	99.9	0.9
Co 228.616	0.9999	Y 224.306	500	500.6	100.6	10.6
Cr 205.560	0.9999	Y 224.307	500	500.5	100.5	4.5
Cu 324.754	0.9999	Y 324.228	500	501.7	101.7	13.0
Fe 238.204	0.9995	Y 320.332	500	503.2	103.6	8.8
Mg 279.553	0.9999	Y 320.332	500	501.0	101.0	0.5
Mn 257.610	0.9999	Y 320.332	500	501.1	101.1	1.7
Ni 221.647	0.9999	Y 224.306	500	500.8	100.8	8.0
Sr 216.596	0.9999	Y 224.306	500	501.3	101.3	10.2
Zn 202.548	1.0000	Y 224.306	500	499.9	99.9	2.0



Figure 4. Results obtained for the 10 hour stability analysis (wavelengths > 230 nm).



Figure 5. Results obtained for the 10 hour stability analysis (wavelengths < 230 nm).



Figure 6. Calibration curve for Ba 455.403 nm.



Figure 7. Calibration curve for Al 167.079 nm.

#### Conclusion

The Thermo Scientific iCAP 7400 ICP-OES Radial fitted with the additional gas accessory used in conjunction with the Ceramic D-Torch, baffled spray chamber, the Burgener Mira Mist Nebulizer and the sheath gas adaptor allows for ultimate high solids tolerance. Moreover, very good spike recoveries indicate that an internal standard is capable of correcting for physical interferences induced by this complex sample matrix. The sheath gas adaptor isolates the sample stream, which reduces sample deposition on the center tube and torch surfaces. It is easy to use and maintain with the iCAP 7400 and iCAP 7600 ICP-OES instruments, enabling improved long-term stability when high TDS samples like 25% NaCl are analyzed. This is demonstated in Figure 8, which shows the results of the measurement under the same conditions without sheath gas. In addition, the Ceramic D-Torch improves the robust instrument performance and the powerful software platform Qtegra ISDS Software simplifies method development and makes postprocessing of the sample data an easy operation.



Figure 8. Results obtained for less than 2 hour stability analysis without sheath gas.

## Find out more at thermofisher.com/ICP-OES

For Research Use Only. Not for use in diagnostic procedures. ©2017 Thermo Fisher Scientific Inc. All rights reserved. Burgener Mira Mist is a trademark of John Burgener of Burgener Research Inc. Tygon is a trademark of Saint-Gobain Corporation. SPEX CertiPrep is a trademark of the SPEX CertiPrep Group LLC. CETAC is a trademark of Teledyne CETAC Technologies INC. All other trademarks are the property of Thermo Fisher Scientific and its subsidiaries. This information is presented as an example of the capabilities of Thermo Fisher Scientific products. It is not intended to encourage use of these products in any manner that might infringe the intellectual property rights of others. Specifications, terms and pricing are subject to change. Not all products are available in all countries. Please consult your local sales representative for details. AM3444-EN 0417

