ADVANCING PEFORMANCE OF AN AXIALLY VIEWED INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER FOR SAMPLES HIGH DISSOLVED SOLIDS BY INNOVATIVE TORCH DESIGN

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Abstract

ICP-OES with axially viewed plasma typically offers an order of magnitude improvement in detection limit performance compared to an ICP-OES with radially viewed plasma. However, for samples with total dissolved solids (TDS) greater than 3% an axially viewed plasma traditionally been more prone to blockages. Through improvements in the torch geometry and optimization of sample introduction components, the tolerance axially viewed plasma to samples with very high TDS has improved markedly. Further improvements in torch design ensure the tolerance of axially viewed plasma ICP-OES for samples with very high dissolved solids are now comparable to systems with a radially viewed plasma with the benefit of improved detection limit performance. This work will evaluate detection limits, short term precision and long term stability of 20 elements in 20% NaCl brine solution.

KeyWords : Inductively coupled plasma, optical spectroscopy, axial viewing, heavy metals, sheath gas torch

Experimental

The Agilent axially viewed simultaneous for OES 720 ES has been used for analysis. The instrument features an innovative axial design with Cooled Cone Interface (CCI) to deflect the plasma tail and reduces background. The patented CCD detector together with the optimized echelle optical design (fig 1) allows meeting the analysis requirements :

- Fast, multi-element technique
- Able to measure down to trace levels of heavy metals
- Able to analyse samples with HDS levels up to 10% w/v using conventional torch design.

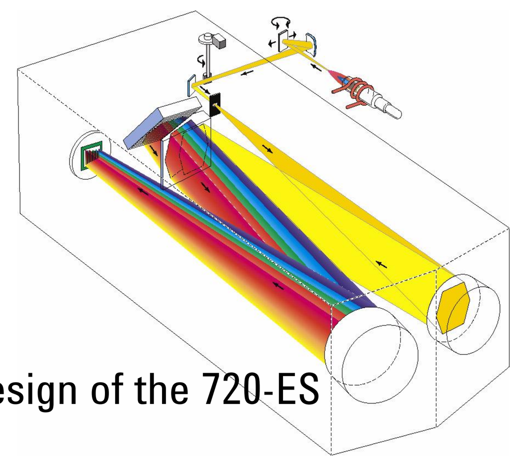


Fig 1. Optical design of the 720-ES

Sample Introduction plays an important role in the analytical performance of HDS samples :

Larger aerosol droplets have greater tendency to collide with torch walls,

Solvent is evaporated leaving salt deposit,

Deposit exacerbates further salt accumulation,

Larger droplets are not effectively broken down in plasma; they don't contribute to analyte signal, only background.

Sample uptake rate was found to be critical to rate of salt accumulation in torch injector. Uptake rate of 0.5mL/min is recommended

The recommended sample introduction system uses :

-High efficiency double pass Sturman Master's spray chamber with a greater efficiency at removing larger aspirated droplets compared to smaller volume glass versions

- High solids capable, v-groove nebulizer offering the ability to aspirate slurries and solutions >30% HDS

- An argon saturator to humidify the nebulizer gas and avoid the blockage.

-Reagents : Suprapur NaCl (99.99%) provided by Merck, Germany, was used for sample preparation. The multi-element standard solution S28 was provided by SCP Science, France.

Table1 below summarizes the instrument parameters used for the analysis. Reduced uptake rate settings have been applied for greater nebulization efficiency and reduced salt load.

Instrument Configuration	Axial
Plasma Power	1.2 kW
Plasma Gas Flow	15 L/min
Auxiliary Gas Flow	2.25 L/min
Nebulizer Gas Flow	0.7 L/min
Sheath Gas Flow	0.13 L/min
Pump Rate	12 rpm
Pump Tubing	0.03" I.D. (black/black)
Sample Uptake Rate	0.5 mL/min
Read Time	20 seconds

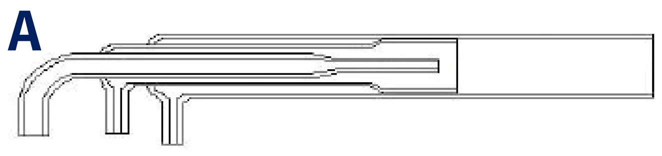
Table 1. 720-ES Instrument parameters

New Torches design

Agilent introduced the High Dissolved Solids (HDS) torch for axial ICP in 2002. Figure 2 shows both conventional axially-viewed torch **(A)** and the HDS torch **(B)**. (International publication number WO 03/005780 A1). This latter features :

- A modified and patented gradually tapered injector tube
- A reduced torch length to minimize devitrification

A



B

HDS torch (B) provides more laminar pathway for the aerosol reducing salt build-up in injector at a specific point.

New HDS torch with sheath gas option has been developed **(C)**

Uses patented torch injector

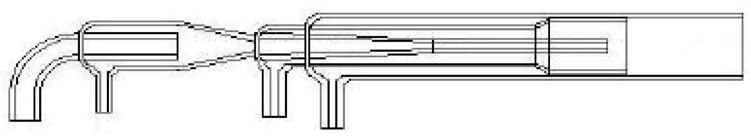
Reduced torch length

Additional Sheath gas option

- •Reduces impact of droplets on injector walls
- •Significantly reduces salt build up in injector
- •Improves long term stability and precision
- •Improves detection limits

This is in comparison to the previous HDS torch

C



Results and Discussion

Stability test

Comparison of the stability test using the HDS torch (B) and the sheath gas HDS torch (C) is shown. The standard conventional torch would not provide more than a couple of hours of reasonable stable performance and results were not included for comparison. Figure 2 shows the signal measured over 4 hours using the HDS torch (B). Reproducibility over 4 hours as found to be less than 5% RSD.

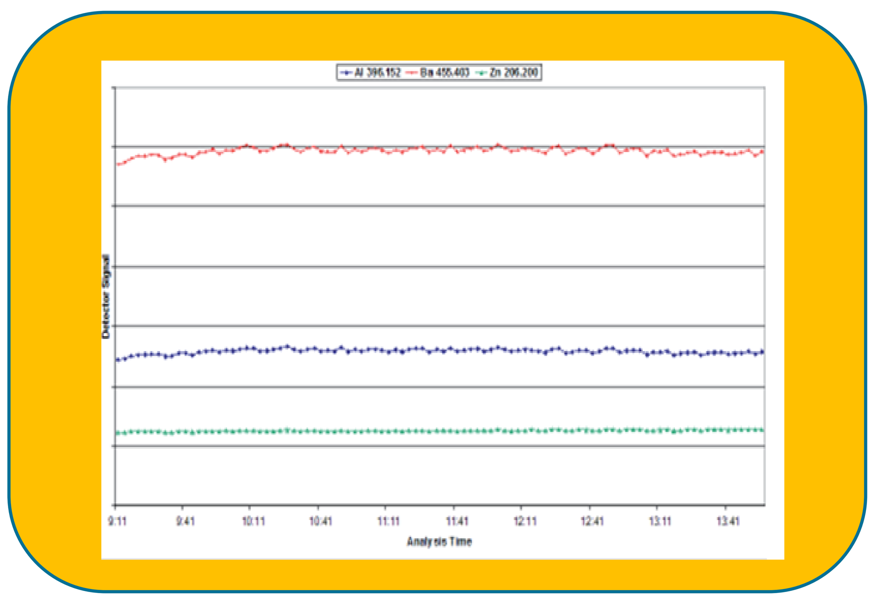


Fig 2. Stability test over 4 hours for the S28 solution using the HDS torch (B)

A long term stability test (LTS) was repeated using the sheath gas HDS torch over a 24 hours period. A 20% w/v NaCl solution spiked with of a multi-element S28 standard was used for the measurements. The signal of various element wavelengths was monitored. The elements and wavelengths used are those recommended by Prof J. M. Mermet and E. Poussel in the evaluation of the long term stability and the identification of the possible origins of drift in ICP-OES.

The test solution was continuously aspirated for 24 hours with no rinsing between samples. Internal standard correction used. Figure 3 displays the normalized sensitivity over the test period. Typical Performance observed :

< 1.5 %RSD after 1 hour

< 2 %RSD after 6 hours

< 3 %RSD after 12 hours

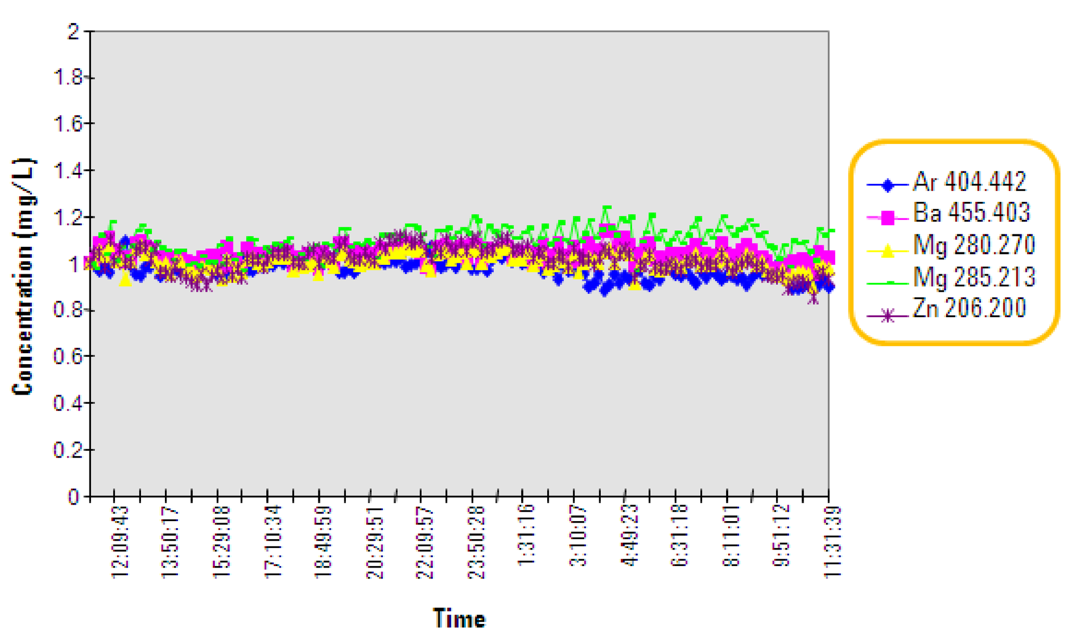


Fig 3. Stability in 20% NaCl using the Agilent sheath gas torch

Method Detection limits were calculated using the equation LD = 3xσ (where σ is the standard deviation for10 measuremetns of the blank matrix NaCl 20% w/v. The list of analytes correspond to the most important elements usually measured in NaCl.

Table 2. Method Detection limits in **µg/L** measured in 20% w/v NaCl matrix using a high solid torch with and without sheath gas

Element	High Solids Torch	Sheath Gas Torch
Al	1.5	0.5
Ba	0.4	0.2
Ca	0.2	0.2
Cu	3	1.5
Fe	2	1.5
Mg	0.2	0.2
Mn	0.2	0.1
Ni	5.7	2
Pb	11	4
Sr	0.3	0.1
Ti	0.9	0.5
Zn	1	0.5
Be	0.3	0.07
Cd	0.9	0.4
Co	2	1
Cr	1	0.6
K	2	1
Li	1.5	0.4
Mo	3.6	2
Sb	11	5
Se	32	10
Tl	8	5
V	1.5	1

Comparison of results shows improvement of the method detection limits by a factor of 2 – 3 when using the sheath gas torch as compared to the High solids torch. The addition of argon through the sheath gas line helps diluting sample aerosol in the plasma improving thus the desolvation efficiency and plasma robustness. A better accuracy on the blank measurements has also been observed leading to lower detection limits.

Conclusions

Innovative designs for torches have been used for determination of metals in brines using ICP-OES with axial-viewing.

The HDS Torch with Sheath Gas shows excellent performances when running solutions up to 20% w/v HDS. Sheath gas torch provides better LTS and DL's compared to the HDS torch and significantly better performance compared to the standard torch which would not be capable of handling this application. Only a negligible flow of 0.13L/min of sheath gas is required.

Increasing tolerance of axial ICP when using the sheath gas HDS toch offers many advantages :

- Achieve even lower detection limits
- Greater long term stability during analysis
    - Improved accuracy and precision
    - Greater productivity
    - Less cleaning and maintenance
- Reduce sample preparation
    - Eliminate dilution of concentrated brines
    - Avoid contamination

-Make analysis simple and routine

- As per radial viewed ICP-OES