

focus on

Mass Spectrometry & Spectroscopy

New Advances in High-Resolution ICP-Optical Emission Spectrometry

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Since the introduction of ICP-optical emission spectrometry, both the spectral resolution and the analytical stability of the plasma have been main concerns with respect to accuracy and sensitivity.

The wealth of emission lines from the plasma frequently leads to overlapping between lines of the analyte and that of accompanying elements. In many sample matrices such spectral interferences impair the recognition of the analyte signal and thus restrict the free choice of analytical lines. Hence, less sensitive alternative lines are frequently used that exhibit poorer detection limits.

Similarly, the detection power of ICP-optical emission spectrometry is hugely affected by the ability of the plasma to tolerate high sample loads. For matrix-rich samples like brine, crude oil or high-alloyed steel effective detection limits will then be best when excellent plasma and signal stability are achieved, while sample dilution is minimal. Otherwise, already small analyte signals of traces and ultra-traces will become undetectable.

A New Dimension of Spectral Resolution

For an increasing number of sample matrices in elemental analysis the high spectral resolution of the PlasmaQuant® PQ 9000 by Analytik Jena will deliver a new kind of effectiveness in data acquisition. With a spectral resolution of about 2 pm at 200 nm, its High-Resolution Optics can resolve much more spectral interference than any other ICP- OES, which is best-illustrated by the Cadmium and Arsenic line pair at about 228.8 nm (Figure 1).

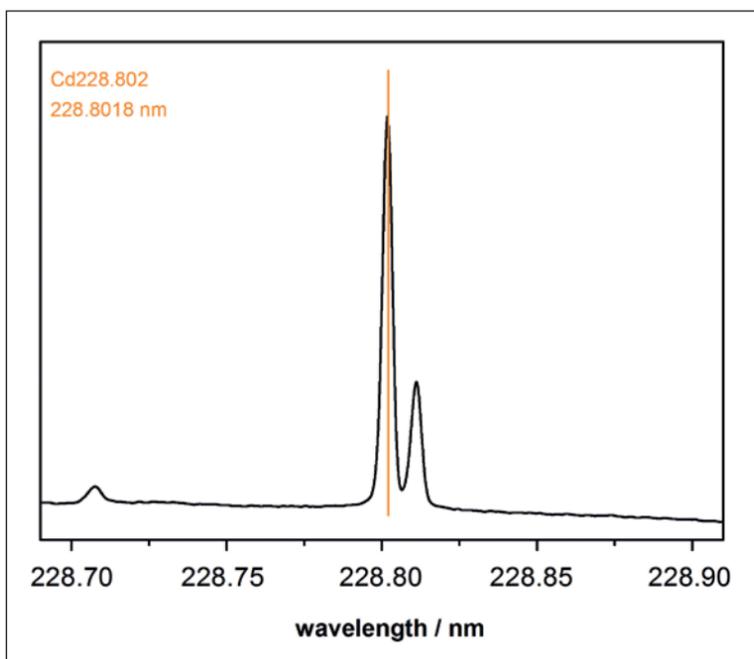


Figure 1. HR ICP-optical emission spectrum of a soil sample showing the Cd 228.802 nm line (orange line) in close vicinity to the As 228.812 nm line.

Originating from a soil sample containing trace levels of Cadmium ($\leq 5 \mu\text{g/L}$) and Arsenic ($\leq 50 \mu\text{g/L}$), this spectrum shows that the Cd 228.802 nm and the As 228.812 nm line are well-separated, while their FWHM (full width on half maximum) values are 3.4 and 3.0 pm, respectively. Highly resolved spectral presentations such as this translate into ultimate analytical precision as overestimation of Cadmium due to false contribution of an Arsenic signal to the adjacent Cadmium line can be ruled out. Moreover, the increased power of detection for the As 228.812 nm line will make it an attractive candidate in a number of applications, among which is the analysis of pharmaceuticals as a detection limit of $2 \mu\text{g/L}$ is achieved for the Arsenic line at 228.812 nm.



Figure 2. PlasmaQuant® PQ 9000 by Analytik Jena.

Plasma Performance for Heavy Matrices

Sustaining long-term stability for high sample loads and, hence, good signal stability with low signal-to-noise levels are important prerequisites for a wide applicability of ICP-optical emission spectrometry. Intensity deviations smaller than ± 5 to 10% over an average working day are typically accepted when analysing e.g. brine samples or fusion products.

For such samples PlasmaQuant® PQ 9000 shows great potentials as can be seen exemplarily for a sample taken from an electrolysis bath with a nominal NaCl content of 30 g/L (Figure 3). Thanks to its redesigned, free-running High-Frequency Generator the normalised intensities for a 1 mg/L spike of Lead, Iron, Aluminum and Barium exhibit only minor deviations over a period of two hours. Please note that no internal standard was used here and that the detection limits achieved in this matrix were $1.0 \mu\text{g/L}$ for Pb, $0.34 \mu\text{g/L}$ for Fe, $0.42 \mu\text{g/L}$ for Al as well as $0.01 \mu\text{g/L}$ for Ba.

High Sensitivity in Heavy Matrices

The trace and ultra-trace analysis of high-alloyed steel samples poses one of the most complex problems for conventional ICP-optical emission spectrometry. With more than 3,300 registered Iron emission lines, their high Iron contents give rise to severe spectral interferences. The emission spectra of such steel samples are often further complicated by significant amounts of refractory additives like Molybdenum, Vanadium or Tungsten that possess numerous emission lines, too.

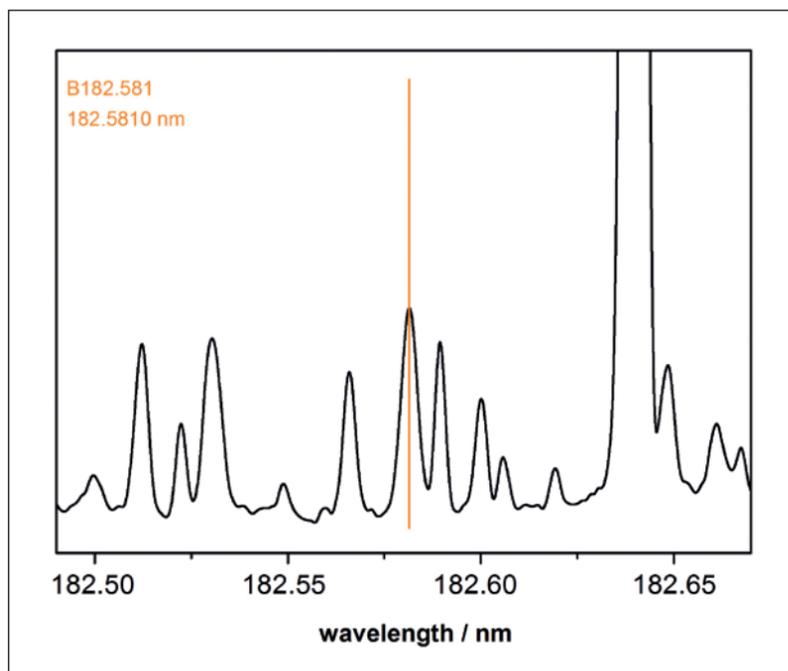


Figure 3. Plot of normalised intensities for four emission lines collected from a 30 g/L NaCl solution.

When it matters to detect the emission line of a trace element like Boron, sample dilution is usually the way to suppress matrix-related spectral interferences in ICP techniques. However, significant sample dilution can result in the signal of a trace element to vanish, particularly, for conventional ICP-optical emission spectrometry with ordinary sensitivity. The high spectral resolution and sensitivity of PlasmaQuant® PQ 9000 offers an alternative approach.

For the Boron line at 182.581 nm a well-separated individual line is typically observed (Figure 4), which is embedded within a complex spectral pattern of emission lines, most of which originating from Iron and Molybdenum. While the B 182.581 nm line is not the most sensitive of the Boron lines, it certainly is interference-free and offers excellent sensitivity given a detection limit of 0.64 mg/kg.

The robustness of PlasmaQuant® PQ 9000 for routine trace analysis of high alloyed steel samples becomes clear when comparing the measured Boron contents in a certified reference material (CRM). A mean value of 24.8 mg/kg was in good agreement with the reported value of 26 ± 2 mg/kg for the respective CRM (Table 1). Besides, good sensitivity and method robustness was achieved for other trace elements like Phosphorus or Aluminium as well as for major elements like Chromium or Copper.

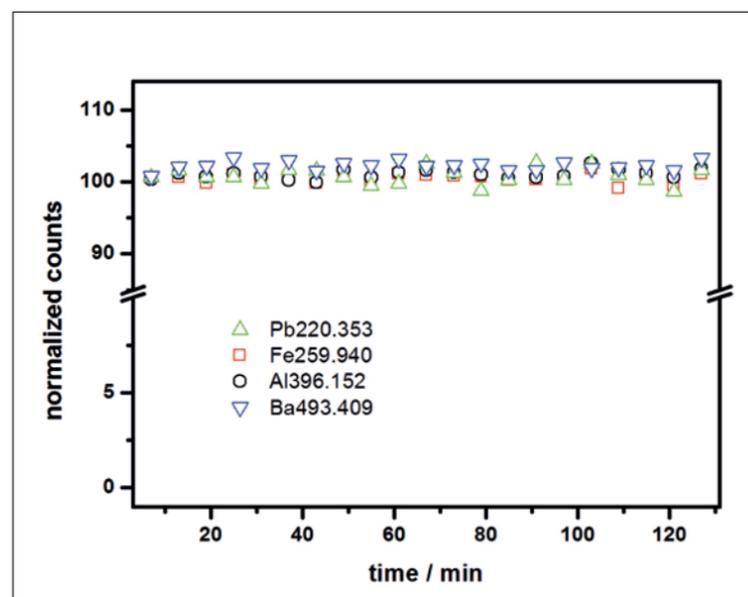


Figure 4. HR ICP-optical emission spectrum of the B182.581 nm line in high-alloyed steel.

Table 1. Results from measurement of a EURONORM standard reference material.

| Element | CRM No. 284-2 [mg/kg] | Mean [mg/kg] | Recovery [%] | Detection limit [mg/kg] |
|------------|-----------------------|--------------|--------------|-------------------------|
| Cr 267.716 | 168,110 | 173,460 | 103 | 0.14 |
| Ni 341.476 | 107,200 | 102,400 | 95.5 | 0.22 |
| Mo 202.030 | 21,110 | 20,980 | 99.4 | 0.52 |
| Cu 327.396 | 1,831 | 1,865 | 102 | 0.24 |
| Co 228.615 | 530 | 498 | 94.0 | 0.76 |
| V 290.881 | 425 | 400 | 94.1 | 0.20 |
| P 178.224 | 258 | 248 | 96.1 | 9.78 |
| Al 394.401 | 27 | 28.5 | 105 | 0.64 |
| B 182.581 | 26 | 24.8 | 95.4 | 0.64 |

Concerning the advances in spectral resolution and sensitivity of ICP-optical emission spectrometry as well as in matrix tolerance of the plasma presented here, the PlasmaQuant® PQ 9000 by Analytik Jena brings clarity, simplicity and confidence even to the most delicate analytical routine.



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New Compact SIMS for Mass Spectrometry in Solid Material



The **Hiden** Compact SIMS tool is designed for fast and easy characterisation of layer structures, surface contamination and impurities with sensitive detection of positive ions being assisted by the oxygen primary ion beam and provides isotopic sensitivity across the entire periodic table. The ion gun geometry is optimised to be ideal for nanometre depth resolution and near surface analysis.

A rotary carousel enables 10 samples to be simultaneously loaded for measurement into the dry-pumped vacuum chamber. The instrument has a small footprint and is exceptionally easy to use, it boasts the same control software and ion gun system as the fully featured Hiden SIMS Workstation family, providing depth profiles, 3D and 2D images and mass spectral data. The MAXIM-600P detector is based around the highly reliable Hiden 6mm triple quadrupole mass filter with pulse ion detection. An electron gun option is available for analysis of insulating samples.

In addition to SIMS, the Compact SIMS has an SNMS facility that is useful for quantification of high concentration elements, such as alloys.

It is suitable for the following applications: solar cells; glass coatings; metallic thin films.

Features include: small footprint; easy user friendly layout; positive SIMS and SNMS; depth profiling; 3D characterisation and imaging; mass spectra; isotopic analysis; analysis on the nanometre scale.

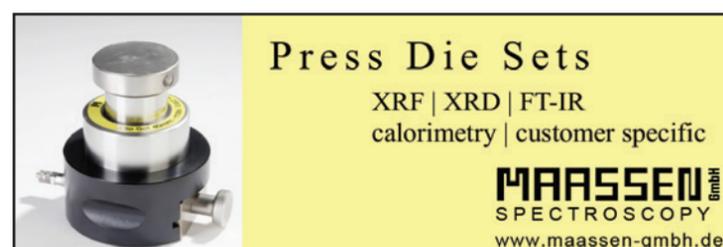
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