A New Tool for Environmental Analysis
USING ICP SPECTROMETRY

ENVIRONMENTAL
Analysis

Author Details

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Inductively Coupled Plasma Optical (or Atomic) Emission Spectroscopy (ICP-OES/AES) is an efficient analytical process for the determination of inorganic pollutants in environmentally relevant matrices. With the establishment of EPA Method 200.7, ISO 11885: 1997 and DIN 38 406, Part 22 the application of this technique has been selected as the standard method of analysis for water, wastewater and sludge. Customer applications may vary from one lab to the next: monitoring toxic elements in effluents or sludges, leachates from wastes, and even air particulates in the working atmosphere. In this paper we will present data on the analysis of certified samples of water and sludge to validate the new ACTIVA™ ICP Spectrometer featuring Advanced CCD Technology with simultaneous SimShot acquisition.

Instrument Description

ACTIVA incorporates a new innovative design featuring a 0.64 m Czerny-Turner optical system. This dispersive system integrates an Achromatic Entrance Imager that was designed to optimize photon collection and minimize optical aberrations. Compared to the use of a single lens for focusing, the Achromatic Entrance Imager uses 2 mirrors: one concave and one plane. The concave mirror is inherently free of chromatic aberrations across the spectrum, rather than at one wavelength, and the plane mirror directs the light into the optical system. The Achromatic Imager uses a MgF_2 window. This Imager has been designed for imaging the best analytical zone of the plasma onto the 6 mm entrance slit height. It allows radial viewing of the entire 10 mm Normal Analytical Zone of the plasma. This entrance slit is imaged through the spectrometer onto the full 6 mm height of the 2048 wide x 512 high pixel detector. This entrance optic associated with large Czerny-Turner optical components featuring 80 mm x 110 mm dual gratings, provides the highest optical luminosity from the far UV to the near infrared.

ACTIVA operates with several nm wide spectral windows called Wavelength Analytical Views (WAV). The width is given by the reciprocal linear dispersion (RLD) of the grating multiplied by the detector pixel size and the number of pixels. The aberration free, JY holographic gratings combined with the 2048 pixel wide CCD detector result in two WAV sizes: up to 8 and 16 nanometers. The two gratings are used back-to-back to provide first order light from 165 to 800 nm. The use of first order light eliminates order hopping resulting from overlapping orders typically seen in echelle grating spectrometers and results in a constant intensity across the WAV. The ACTIVA CCD is at the super flat field position of the Czerny-Turner spectrometer allowing an excellent constant resolution and a constant intensity within a WAV.

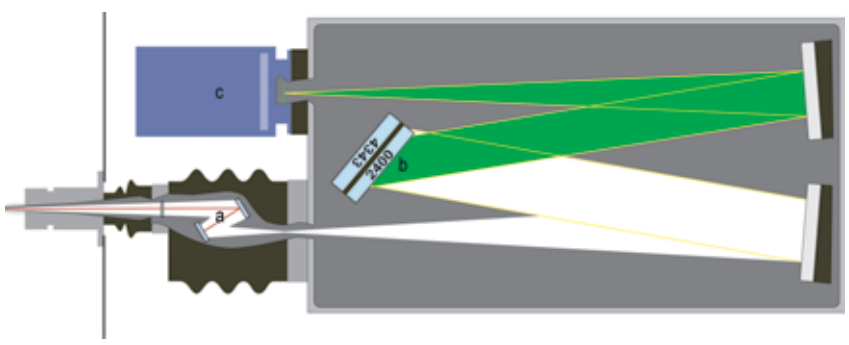

Figure 1: Unique high resolution optical design of ACTIVA with (a) achromatic entrance imager, (b) dual holographic gratings and (c) CCD detection system.

Water Analysis

A strong advantage of the ACTIVA ICP Spectrometer is the ability to perform rapid, simultaneous multi-line analysis. This leads to a great improvement in accuracy with no penalty to the analysis time. Results shown in Figure 2 for NIST SRM 1640 show theoretical and experimental concentrations in µg/L. A simple two point calibration curve was used between 0 and 5 mg/L. Because experimental concentrations were significantly above the upper point, accuracy of the results provided evidence of a good linearity. The analysis was performed on 35 elements at over 57 elemental lines with a total analysis time of 3.5 minutes. The sample was analyzed with the conditions of analysis shown in Table 1.

Table 1: Conditions of operation

Parameter	Condition
RF Generator Power	1000W
Plasma gas flowrate	12 L/min
Aux gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer flowrate	0.7 L/min
Sample uptake	1 mL/min
Type of nebulizer	Pneumatic, K3
Type of spray chamber	Cyclonic

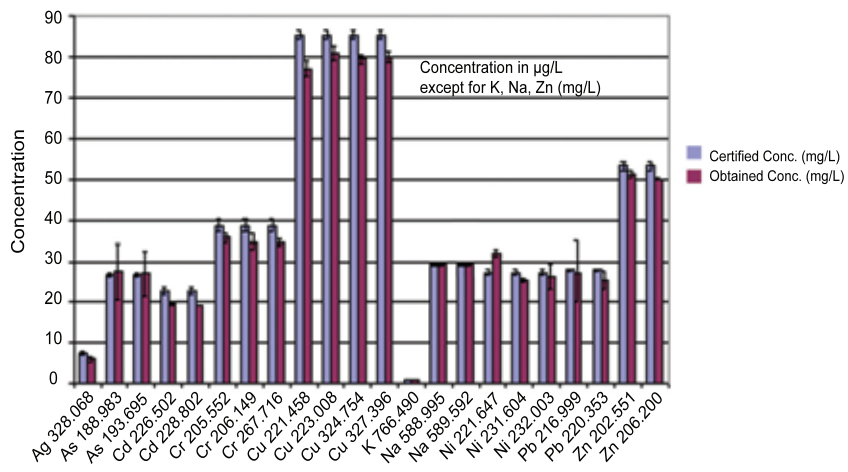

Figure 2: Certified and experimental concentrations on NIST SRM 1640

Sludge Analysis

Similarly, a quantitative method was developed for multi-line analysis of sludge. The same plasma conditions were used as for water analysis shown above in Table 1 with a power increase to 1200 W. The good recoveries for the certified sludge shown in Figure 3 demonstrate the advantages of Total Plasma View using a radial plasma for fewer matrix effects (e.g. no influence of Ca present) and a high dynamic range from the CCD.

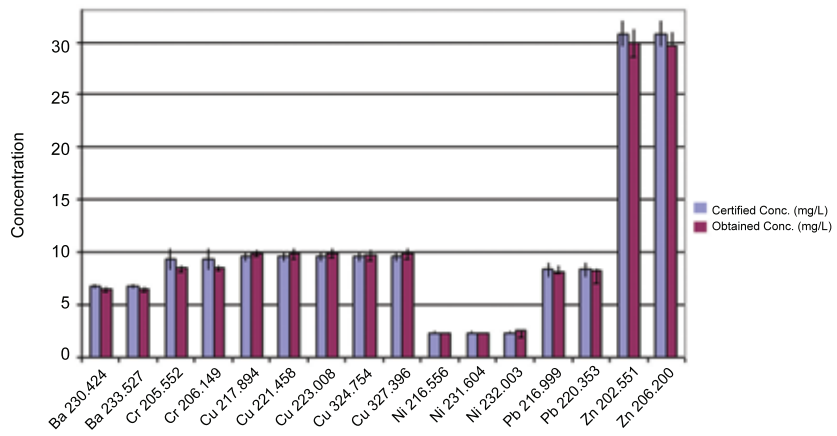

Figure 3: Certified and experimental concentrations on certified sludge

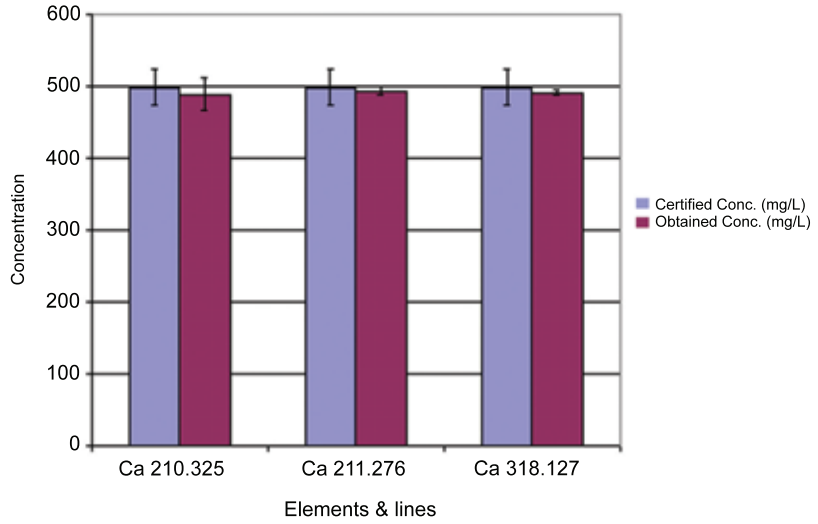

Figure 4: Multi-line analysis of Calcium in sludge shows excellent agreement of results.

Analysis of a Waste Treatment Stream

Typical waste treatment streams are analyzed for the periodic table. With Multi-WAV acquisition and WAV-Explorer, ACTIVA allows the immediate qualitative analysis and automatic identification of any and all elements in this type of sample. The sample shown at the top in Figure 5 represents a permanent fingerprint of the sample showing over 50 elements in solution. The zoom shown in the middle in Figure 5 represents an 8 nm WAV at 213 nm. A further zoom, shown on the bottom in Figure 5, shows the Pb 220 nm line with no interferences and excellent resolution.

Instrument Detection Limits

ACTIVA provides excellent limits of detection as shown in Table 2 below including those below 190 nm. Total Plasma View extends the analysis of trace elements to a variety of difficult environmental matrices, including organics and high dissolved solids.

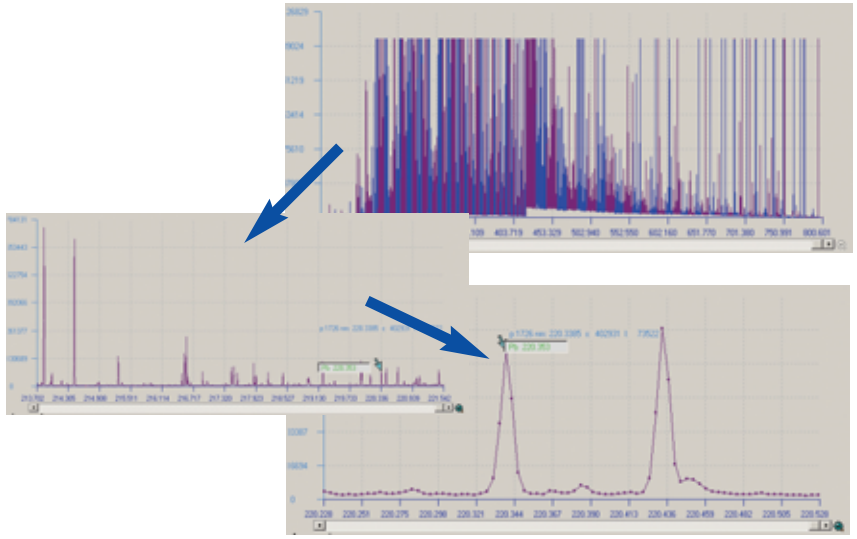

Figure 5: (top) Full spectrum acquisition of Waste Treatment Stream sample; (middle) 8 nm WAV zoom at 213 nm; (bottom) further zoom to Pb at 220.353 nm.

Table 2: Typical detection limits of ACTIVA at 3 sigma in µg/L

Element	Wavelength (nm)	Detection Limit (µg/L)
Al	167.020	0.7
As	189.042	5
As	193.695	5
B	208.959	1.5
Cd	228.802	0.3
Co	228.616	0.4
Cr	267.716	0.4
Cu	324.754	0.4
Fe	259.940	0.4
Li	670.784	0.05
Mg	279.553	0.02
Mn	257.610	0.06
Mo	202.030	0.6
Ni	221.647	0.5
P	178.229	9
Pb	220.353	2.5
Sb	206.833	4
Se	196.026	4
Zn	213.856	0.3

Conclusion

The combination of new CCD technology with optimized optical design leads to limits of detection and fast analysis perfectly suited for a wide variety of environmental samples. The powerful WAV-Explorer allows any user to rapidly and easily identify any element of the periodic chart and select the best wavelength(s) for analysis by referencing the innovative ACTIVA element database. The benefits of high resolution, linear calibration over a large dynamic range and radial plasma viewing add up to make the ACTIVA an excellent tool for environmental analysis.