

APPLICATION

AAS

1

Zn-MEASUREMENTS IN METALLURGICAL WASTE WATER SAMPLES USING HIGH SPEED SELF REVERSIBLE BACKGROUND CORRECTION

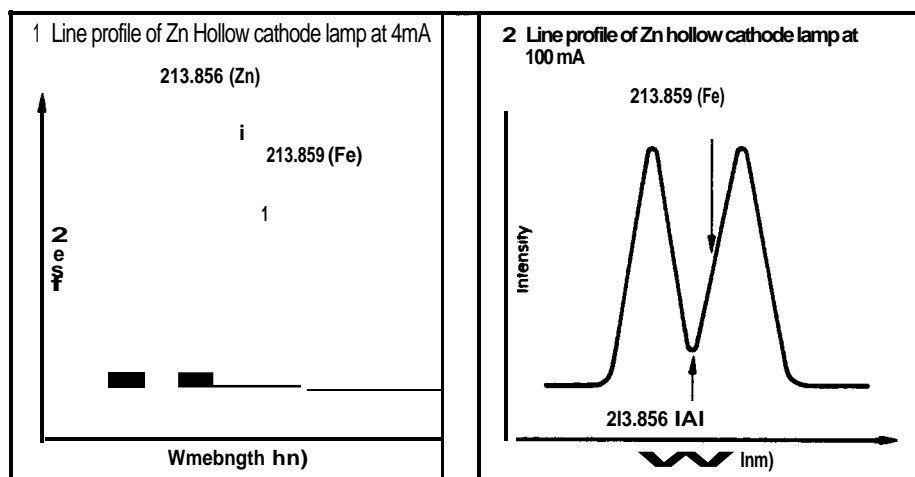
U. Oppermann, Shimadzu Europa

Spectral interferences in Atomic Absorption Spectroscopy (AAS) are mainly caused by molecular absorption, particulate caused scattering and absorption line overlapping. In comparison to a conventional background correction system (Deuterium lamp as a continuous source) which fails in correcting interferences caused by absorption line overlapping, the high speed self reversible method is able to overcome such effects over the entire wavelength range.

The high speed self reversible background correction method is based on the fact, that the absorbance of a selected element decreases when the hollow cathode lamp is operated at progressively higher currents, because the absorption line at the specified wavelength is broadened and becomes self-reversed.

Figure 1 shows the absorption line of a Zn-hollow cathode lamp at 213.856 nm under normal operating current. The absorption line of Fe at 213.859 nm is very closely positioned to the primary Zn wavelength. That means measuring a sample containing Zn and Fe under normal lamp current (4 mA) would cause a signal corresponding to both elements.

Figure 2 shows the absorption line profile of the Zn-hollow cathode lamp at the same wavelength of 213.856 nm, but now obtained with a lamp current of 100 mA. The line profile becomes broadened and self-reversed, that



means the absorption caused by the Zn-atoms decreases to a minimum and mainly the Fe-atoms are responsible for the absorption. Subtraction of the absorption at high current from the value obtained under normal lamp current provides a high degree of freedom from background caused by spectral interferences.

This sophisticated technique was applied to samples, which present the problem described above: Zn to be determined besides a large excess of iron. Such samples are a common problem in metallurgical waste waters.

All measurements were done on the Shimadzu atomic absorption spectrometer AA-660. Table 1 shows the parameters for all determinations. For the high speed self reversible method a high current of 100 mA was used in addition. A calibration curve for zinc

was set up with three standards of aqueous solutions in a concentration range from 0.05 to 0.20 mg/l. For checking the influence of spectral interference by iron due to absorption line overlapping, the samples were measured with an iron concentration up to 1000 mg/l.

In Table 2 different background correction methods are compared for these samples. Erroneous high Zn-values, in samples with a large excess of Fe, were obtained without background correction, and with the D2 correction method as well. Only the self reversible correction method gave correct values.

The great advantage of this technique is a very good precision and a high sample throughput, because special pretreatment of samples or standard addition method is not necessary.



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APPLICATION

HC Lamp Current	4/100 mA*	Flame	Air-C ₂ H ₂
Slit width	0.5 nm	C ₂ H ₂ flow rate	1.5 l/min
Wavelength	213.9 nm	Air flow rate	8 l/min
Pre spray time	3 s	Burner slot	10 cm
Integration time	5 s	Burner height	8 cm

* See text

BGC	Fe	Zn (0.050 Standard)		Zn (0.100 Standard)		Zn (0.200 Standard)	
		Found	Deviation (%)	Found	Deviation (%)	Found	Deviation (%)
-	-	0.049	- 2	0.099	- 1	0.198	- 1
-	100	0.059	+ 18	0.105	+ 5	0.211	+ 6
-	1000	0.101	+ 102	0.149	+ 49	0.247	+ 24
D ₂	1000	0.096	+ 92	0.143	+ 43	0.238	+ 19
SR	1000	0.049	- 2	0.096	- 4	0.193	- 2

BGC background correction
 D2 deuterium lamp method
 SR high speed self reversible method

All amounts in mg/l

Instrumentation

Atom-absorptions-spectrometer
 Autoamplifier

AA-660
 ASC-60F

 **SHIMADZU**

Shimadzu Europa GmbH
 Albert-Hahn-Straße 6 - 10, D-47269 Duisburg
 Telefon: (0203) 7687-0
 Fax: (0203) 766625, Telex: 855220 shim