Challenge

Determination of Cd, Pb, Ni, Cu, Zn and Cr in sewage sludge.

Solution

Reliable routine analysis using flame AAS on the novAA® 800 F.

Determination of Toxic and Eco-toxic Elements in Sewage Sludge

Introduction

A global boom of industrialization and an increasing demand for advanced materials and products is leading to environmental release of harmful and even toxic compounds in many regions of the world.

Toxic metals, such as cadmium, lead or chromium as well as high concentrations of other potentially harmful elements, e.g., nickel or copper, often pass into eco systems through sewage sludge from industrial sites, as well as from weathering or wearing of pipes, reactors and other industrial facilities. Hence, a close monitoring of sewage sludge is the key to meeting statutory limits, and to allow targeted intervention in case of potential hazards.

This application note describes a straight-forward and robust flame AAS method for routine analysis of cadmium, lead, nickel, copper, zinc and chromium in sewage sludge for industrial QC labs with moderate sample loads.

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Materials and Methods

Samples and Reagents

- Sewage sludge
- Reference material for sewage sludge, BCR 146R

A reference material for sewage sludge with known analyte concentrations was analyzed for method validation.

Sample Preparation

The sample was prepared according to ISO 15587-1 (1 g in 100 mL) using 12 mL aqua regia as digestion agent and the microwave system TOPwave® (vessel type PL100). Approximately 0.5 g of the reference sample was digested accordingly and transferred into a graduated flask and filled up to 50 mL with deionized water.
Please note, that ISO 15587-2 (using nitric acid as digestion agent) can be followed, too; when extraction of potential fumes can not be ensured. Besides, sample digestion in a beaker (on a hot plate) may be an alternative to the microwave-assisted digestion. However, silicons or some organic content of the sewage sludge may not be completely dissolved.

Calibration

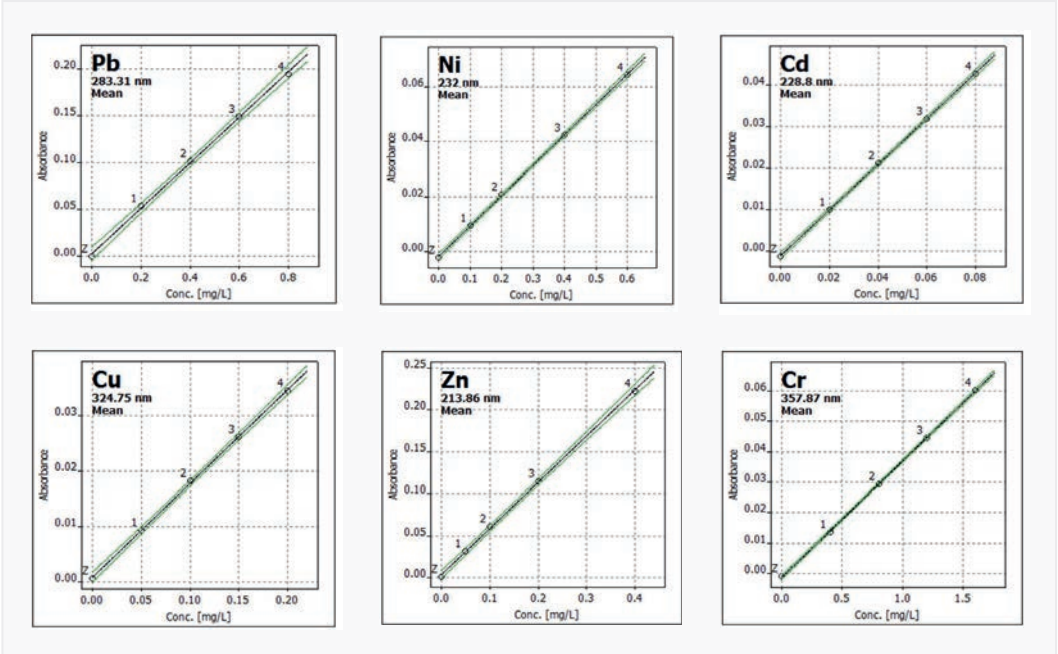
A standard calibration was applied and standards were prepared manually using 1 % HCl and 0.1 % CsCl/$LaCl_3$.

Calibration curves

Table 1: Concentration of calibration standards

Standard	Concentration [mg/L]					
	Cd	Pb	Ni	Cu	Zn	Cr
Cal. 0	0	0	0	0	0	0
Cal. Std. 1	0.02	0.2	0.1	0.05	0.05	0.2
Cal. Std. 2	0.04	0.4	0.2	0.1	0.1	0.4
Cal. Std. 3	0.06	0.6	0.4	0.15	0.2	0.6
Cal. Std. 4	0.08	0.8	0.6	0.2	0.4	0.8

Instrumentation



The measurements were performed using a novAA® 800 F for flame AAS, equipped with injection switch SFS 6.0 and an autosampler with automatic dilution function. The analysis was carried out using a 100 mm burner head for air/acetylene flame.
The use of an automatic burner head cleaner, the Scraper, allows automated removal of deposits from the burner slit at regular intervals when using the nitrous oxide flame.

Instrument Settings and Method Parameters

Results and Discussion

Table 2: Instrument settings and method parameters

Element	Wavelength [nm]	Slit [nm]	Lamp current [mA]	Burner with [mm]	Burner angle [°]	Burner height [mm]	Flame type	Fuel gas flow [L/h]
Cd	228.8	1.2	2.0	100	0	6	C_2H_2/air	40
Pb	283.3	1.2	4.0	100	0	6	C_2H_2/air	65
Ni	232.0	0.5	5.0	100	0	5	C_2H_2/air	65
Cu	324.7	1.2	3.0	100	0	5	C_2H_2/air	50
Zn	213.9	0.8	2.0	100	0	6	C_2H_2/air	60
Cr	359.3	0.8	4.0	50	0	5	C_2H_2/N_2O*	185

* Cr as a refractory metal requires higher atomization temperatures, hence a C_2H_2/N_2O gas mixture and a 50 mm burner head may benefit the Cr analysis

Table 3 shows the measurement results for the samples as well as matrix-specific limits of quantification. Besides, a spike recovery test was conducted to test method robustness. Hence, the sample was spiked with a defined analyte concentration ("QC spike") in order to check for non-spectral interferences.

Conclusion

Table 3: Measurement results

Sample	Element	DF	Concentration [mg/kg]	Spike Concentration Increase [mg/L]	Recovery rate [%]	LOQ* (mg/L)
Sewage sludge	Cd	1	0.87 ± 0.19	0.04	92.1	0.003
	Pb	1	46.1 ± 1.70	0.4	95.3	0.024
	Ni	1	28.8 ± 0.53	0.2	92.6	0.09
	Cu	50	360 ± 21.5	0.1	102	0.012
	Zn	50	1219 ± 51.9	0.1	104	0.003
	Cr	1	43.1 ± 0.67	0.4	95.5	0.153

DF: Dilution factor
*Limit of Quantification (LOQ): LOD *3
LOD method specific detection limits from 11 times standard deviation of matrix blank

Table 4: Analysis of the reference material

Sample	Element	Dilution Factor	Concentration [mg/kg]	Certified value / expected value [mg/kg]	RSD [%]
BCR 146 R Sewage sludge (industrial origin)	Cd	1	18.4 ± 0.45	18.4 ± 0.4	0.8
	Pb	10	554 ± 17.6	583 ± 17	0.4
	Ni	10	62.1 ± 3.9	65.0 ± 3	0.9
	Cu	100	787 ± 39.9	831 ± 16	1.4
	Zn	100	2740 ± 95.3	3040 ± 60	0.4
	Cr	1	170 ± 3.2	174 ± 7	0.7

The novAA® 800 F allows fast, simple and highly precise determination of cadmium, lead, nickel, copper, zinc and chromium in pre-digested sewage sludge samples. A QC spike recovery test showing recovery rates of 92-104 % demonstrates the high method robustness in high matrix samples. Besides, good agreement of results for a certified reference material and RSD values about or well-below 1 % proof validity of the procedure including the microwave-assisted digestion.

The SFS 6.0 injection switch with continuous rinsing function and segmented sample injection ensures reduced carryover in case of high salt and matrix content while the automatic cleaning of the burner head using the Scraper provides stable analysis conditions for highly reproducible results. Using the autosampler with integrated dilution function enables a high sample throughput even for high matrix samples.

Headquarters
Analytik Jena AG
Konrad-Zuse-Strasse 1
07745 Jena · Germany

Phone +49 3641 77 70
Fax +49 3641 77 9279

info@analytik-jena.com
www.analytik-jena.com

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