Challenge

Determination of Cd, Pb, Ni, Cu, Zn, and Cr in industrial effluents.

Solution

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

Determination of Toxic and Eco-toxic Elements in Industrial Effluents

Introduction

Industrialization and an ever-growing population with rising standards of living produce huge volumes of wastewater, while at the same time the demand for drinking water is increasing. Hence, water recycling and continuous monitoring of industrial/communal wastewater is becoming more and more important globally.

This application note describes a method for fast routine analysis of several toxic key elements, such as cadmium, lead, nickel, copper, zinc, and chromium, in industrial effluents.

These metals may pass into ecosystems through wastewater from industrial or municipal sites. Water treatment plants help to filter and purify toxic elements and other contaminants out of wastewater before it is released into the environment.

Thus, tight monitoring of industrial effluents is key for meeting statutory limits (as, for example, regulated in national drinking water directives) and for allowing targeted intervention in case of potential hazards. This is crucial for guaranteeing a safe supply of healthy water to everyone.

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Materials and Methods

Samples and Reagents

An intra-laboratory reference materials for wastewater, RV Q3D2 with typical analyte concentrations of industrial effluents was tested for proof of principle.
In addition, method specific detection limits were obtained to compare performance of flame AAS to requirements of the German drinking water directive.

Sample Preparation

The wastewater sample was pretreated with a mixture of HNO_3 and H_2O_2 in a beaker. Sample dilutions were prepared with 1% HCl and 0.1% CsCl/$LaCl_3$.

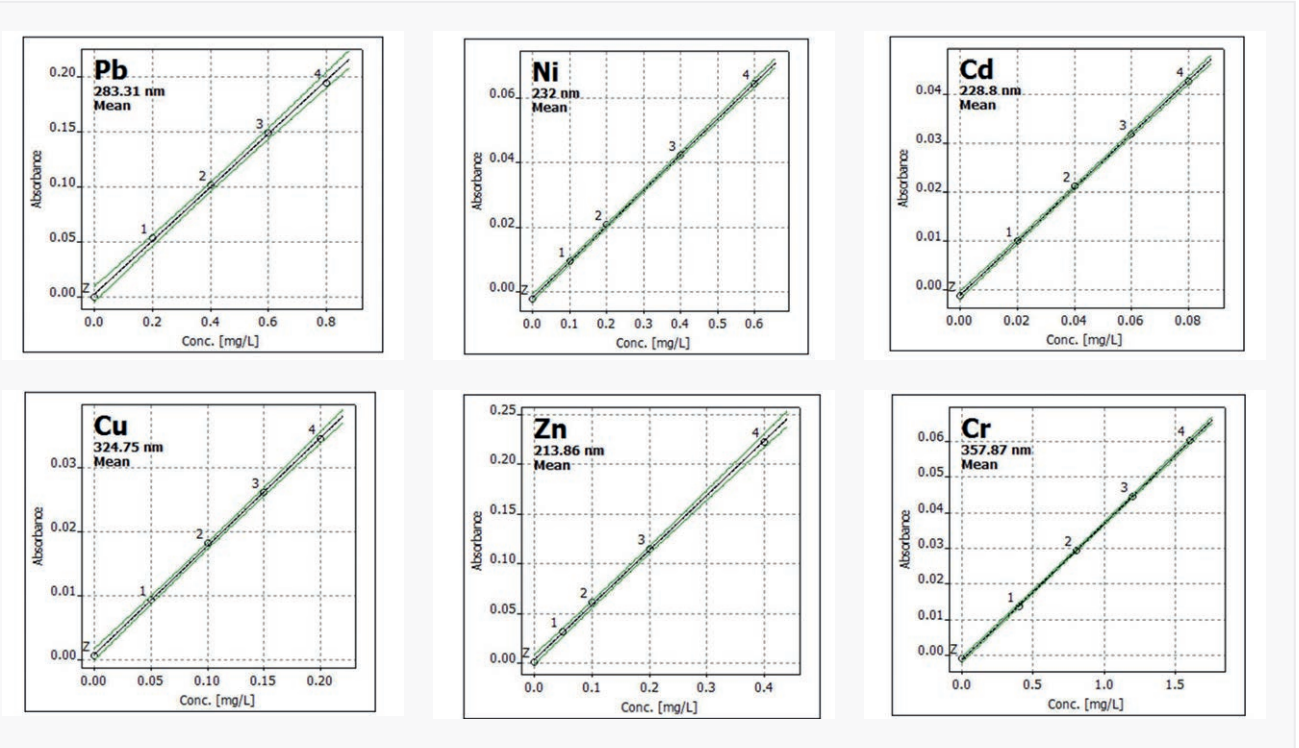
Calibration

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

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

Calibration curves



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 determination of Cr the use of a nitrous oxide flame is recommended, since matrix interferences can be avoided through the higher flame temperature. 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

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

Results and Discussion

Table 3 shows both the results for the certified waste water sample and the respective limits of the German drinking water directive, which are binding for the treatment of waste water.

Table 3: Analysis of quality control samples

Sample	Element	DF	Concentration [µg/L]	Certified value / expected value [µg/L]	Recovery rate [%]	LOQ[2] [mg/L]
RV Q 3D2 Waste water	Cd	1	< DL[1]	4.0 ± 0.41	-	0.003
	Pb	1	160.3 ± 19.7	157.4 ± 8.5	102	0.024
	Ni	1	374.0 ± 3.7	360.0 ± 23.76	104	0.09
	Cu	1	282.1 ± 8.6	289.6 ± 17.38	97.4	0.012
	Zn	1	277.1 ± 10.2	297.3 ± 14.87	93.2	0.003
	Cr	1	455.6 ± 7.0	457.5 ± 24.25	99.6	0.153

DF: Dilution factor
1 Limit of Quantification (LOQ): Detection Limit*3
2 Limit of Detection (DL): 11 consecutive measurements of matrix blank

Close comparison of determined and certified values show good agreement indicating good method robustness. Besides, method-specific detection limits for Cu, Zn and Cr were found to be well-below requirements of the German drinking water directive; while for Cd, Pb and Ni the detection limits of the flame AAS are not suffieciently low enough. Here, graphite furnace AAS or ICP routine would be the technique of choice. However, flame AAS is often used for analysis of all these elements, when their concentration is significantly higher as e.g. in industrial effluents.

Conclusion

This application note demonstrates a simple and highly precise flame AAS method for the determination of Cd, Pb, Ni, Cu, Zn, and Cr in industrial effluents using the novAA® 800 F. Recoveries for a wastewater reference material (RV Q 3D2) were well within the certified concentration range.

A range of performance-enhancing accessories are available to improve ease of use and productivity. Specifically, the Segmented Flow Star (SFS 6.0) injection switch with continuous rinsing and segmented sample introduction ensures reduced carryover in case of high salt and matrix content. The automatic cleaning of the burner head using the scraper provides stable analysis conditions for highly reproducible results. Moreover, using the autosampler AS-FD with integrated dilution function makes it possible to determine all elements in one run without time-consuming predilution.

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