Challenge
Determination of the elements Ag, Al, As, Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, Pb, Sb, Se, Tl, and Zn in accordance with ISO 15586

Solution
Quantification of elements using the ZEEnit series in graphite furnace mode

Intended audience
Government and commercial environmental laboratories

Quantification of Elements In Accordance with ISO 15586 in Soil, Sediment, and Water Using LS-AAS

Introduction

The steady rise in industrialization and anthropogenic influences have led to an increasing burden of heavy metals in the environment, raising serious concerns about environmental and health risks. In the face of this challenge, accurate analysis, and monitoring of heavy metal concentrations in various environmental media such as water, soil, and sediment become crucial.

In this context, atomic absorption spectrometry (AAS) has taken on an important role in environmental analysis due to its high robustness, precision, and flexibility. Graphite furnace AAS (GF-AAS) in particular is a powerful technique that enables even the lowest concentrations of heavy metals to be reliably determined with minimal sample consumption. The aim of this application note is to demonstrate the applicability of GF-AAS for the analysis of heavy metals in water, soil, and sediment.

This application note describes the determination of the elements aluminum, antimony, arsenic, cadmium, chromium, cobalt, copper, iron, lead, manganese, molybdenum, nickel, selenium, silver, tellurium, and zinc by graphite furnace atomic absorption spectrometry using the ZEEnit 650P.

The ZEEnit 650P atomic absorption spectrometer is equipped with a lamp turret with eight positions for hollow cathode lamps (HCL). The third-generation graphite furnace with Zeeman background correction with its variable magnetic field up to a magnetic flux density of one tesla is the ideal instrument for the quantification of metals in the low µg/L concentration range and below. The powerful Zeeman background correction makes it possible to reliably separate even low atomic absorption signals from the background absorption without mathematical models on a physical basis. This enables an easy routine measurement

of even difficult matrices. Due to the variable magnetic field and the evaluation in 3-field mode, very sensitive detectable elements such as zinc can be determined conveniently under standard laboratory conditions by attenuated measurement signal intensity.

The AS-GF autosampler can carry out fully automatic dilutions before the actual measurement and an intelligent dilution when the highest calibration standard is exceeded. This autosampler can also be used to automatically prepare the solutions required for the calibration from a stock standard solution. Further functions of the AS-GF are the fully automatic preparation of the solutions for standard addition method, automatic dosing of modifier solution and spiking of the sample with a known standard concentration

Materials and Methods

Samples and reagents

Reference material:

- 1640a natural water (NIST, National Institute of Standards & Technology)
- BCR-143R sewage sludge with enriched soil (European Commission, Institute for Reference Materials and Measurements)
- BCR-146R sewage sludge of industrial origin (European Commission, Institute for Reference Materials and Measurements)
- BAM-U110 contaminated soil (BAM, Bundesanstalt für Materialforschung und -prüfung, 2006)
- PACS-2 marine sediment (National Research Council of Canada)
- IAEA-457 marine sediment (IAEA International Atomic Energy Agency)

Reagents:

- Concentrated HNO_3 (approx. 60%, purified via subboiling distillation)
- Mg matrix modifier (10 g/L)
- Pd matrix modifier (10 g/L)
- Ascorbic acid (p.a.)
- Certified single element standards for aluminum, antimony, arsenic, cadmium, chromium, cobalt, copper, iron, lead, manganese, molybdenum, nickel, selenium, silver, tellurium, and zinc (analyte concentration 1000 mg/L each)

Sample preparation

The samples can be digested in accordance with ISO 54321. In this application example, the solid samples were treated with microwave-assisted digestion with the use of aqua regia. The weight of the sample material was approx. 0.3 g and the filling volume was 50 mL. The supernatant in the sample containers was used for the measurement. Further dilutions were carried out using 0.5% (v/v) HNO_3 as a diluent. For an effective stabilization of dissolved silver over a longer period of time, a dilution solution with 2-5% (v/v) HNO_3 is preferable, alternatively 10% (w/w) HCl can also be used. For the analyte antimony, a diluent with HCl (5% HCl (v/v)) should be used if a longer stability of the dissolved antimony content is to be ensured. Alternatively, traces of HF or tartaric acid can be added to solutions containing HNO_3 to improve the long-term stability of antimony content.

Calibration

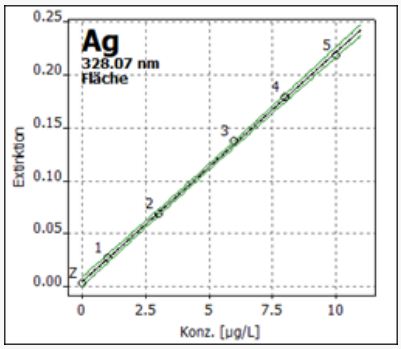
In accordance with the ISO 15586 standard, the calibration standards were prepared in a solution with 0.5% (v/v) HNO_3. A solution of 0.5% (v/v) HNO_3 was used as the blank value.

Table 1: Concentrations used for calibration according to ISO 15586

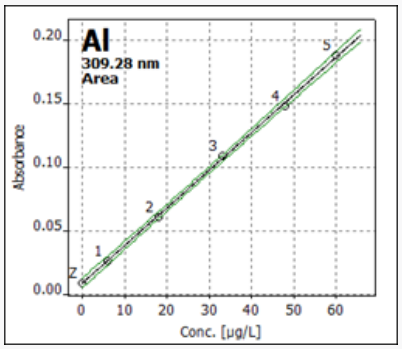
Standard	Concentration [µg L^{-1}]									
	Ag	Al	As	Cd	Co	Cr	Cu	Fe	Mn	Mo
Cal. 0	0	0	0	0	0	0	0	0	0	0
Std. 1	1	6	10	0.4	6	2	3	3	1.5	6
Std. 2	3	18	30	1.2	18	6	9	9	4.5	18
Std. 3	6	33	50	2.0	30	10	15	16.5	7.5	33
Std. 4	8	48	70	2.8	42	14	21	24	10.5	48
Std. 5	10	60	100	4.0	60	20	30	30	15	60

Table 1 continued: Concentrations used for calibration according to ISO 15586

Standard	Concentration [µg L^{-1}]					
	Ni	Pb	Sb	Se	Tl	Zn
Cal. 0	0	0	0	0	0	0
Std. 1	7	10	10	15	6	0.5
Std. 2	21	35	30	45	21	1.5
Std. 3	35	55	30	82.5	33	2.75
Std. 4	49	75	42	120	48	4
Std. 5	70	100	100	150	60	5



$R^2_{(adj.)}$ 0.9990 (linear), Modifier Pd/Mg



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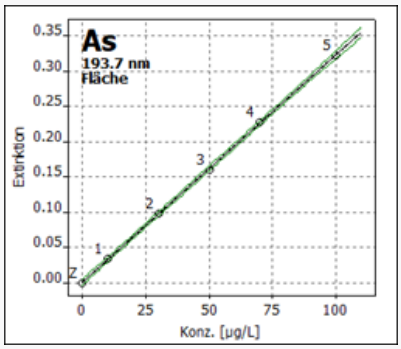
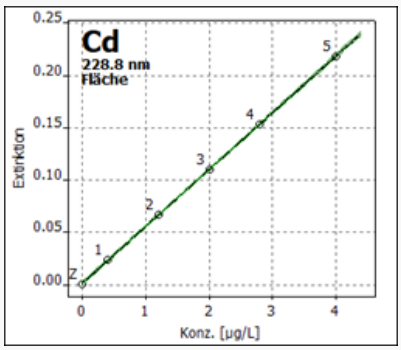
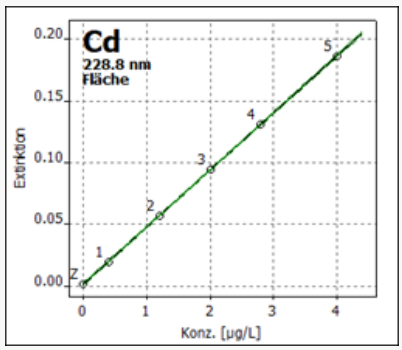


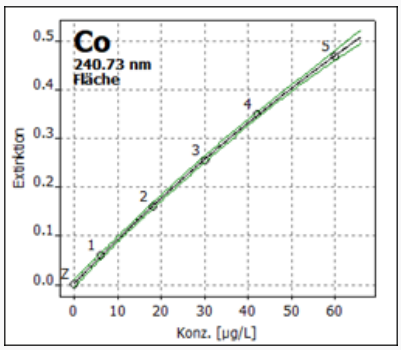
$R^2_{(adj.)}$ 0.9997 (linear), Modifier Pd/Mg



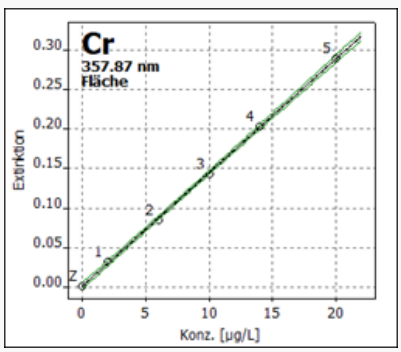
$R^2_{(adj.)}$ 0.99990 (linear), Modifier $NH_4H_2PO_4$



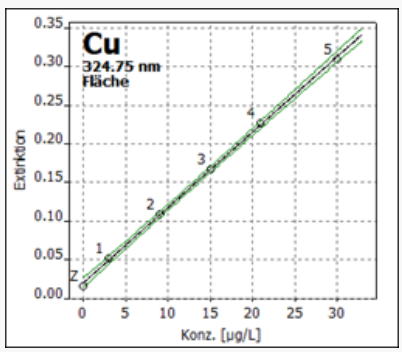
$R^2_{(adj.)}$ 0.99993 (linear), Modifier Pd/Mg



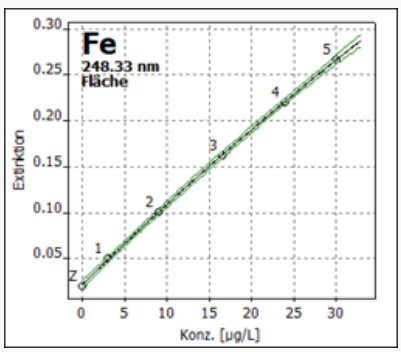
$R^2_{(adj.)}$ 0.9995 (non-linear), Modifier Pd/Mg



$R^2_{(adj.)}$ 0.9995 (linear), Modifier Mg

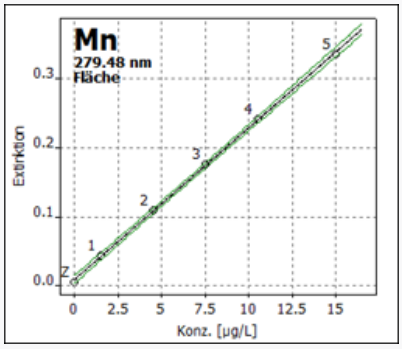


$R^2_{(adj.)}$ 0.9990 (linear), Modifier Pd/Mg

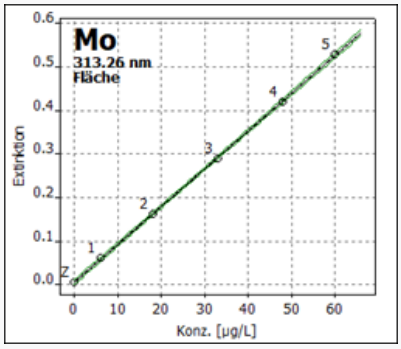


$R^2_{(adj.)}$ 0.9996 (non-linear), Modifier Mg

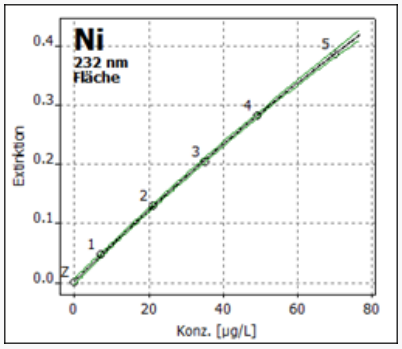
Figure 1: Typical calibration functions according to ISO 15586



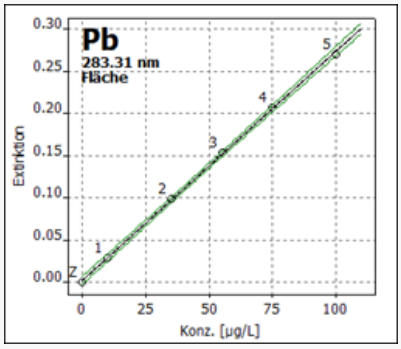
$R^2_{(adj.)}$ 0.9991 (linear), Modifier Pd/Mg



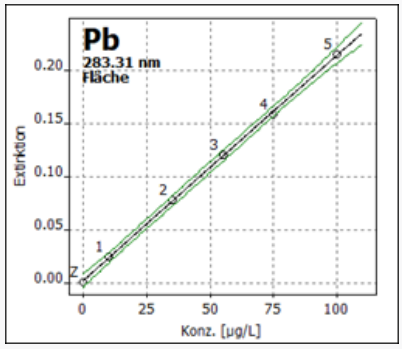
$R^2_{(adj.)}$ 0.9997 (linear), Modifier Mg



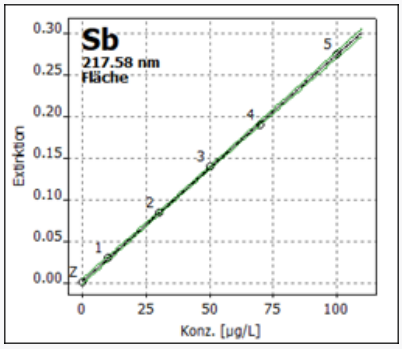
$R^2_{(adj.)}$ 0.9997 (non-linear), Modifier Mg



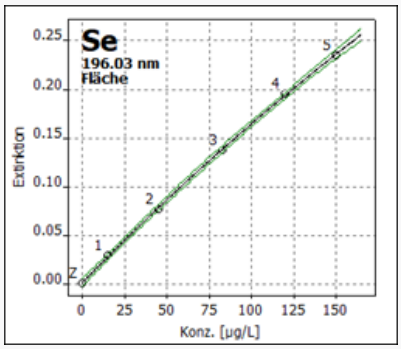
$R^2_{(adj.)}$ 0.9992 (linear), Modifier $NH_4H_2PO_4$



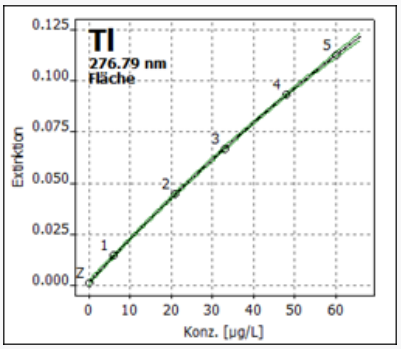
$R^2_{(adj.)}$ 0.9990 (linear), Modifier Pd/Mg



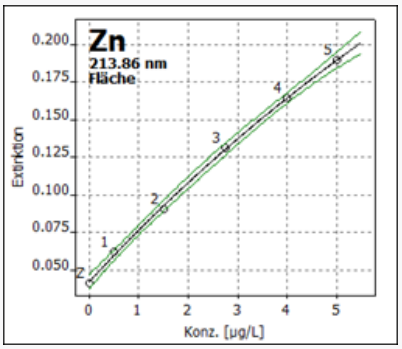
$R^2_{(adj.)}$ 0.9996 (linear), Modifier Pd/Mg



$R^2_{(adj.)}$ 0.9996 (non-linear), Modifier Pd/Mg



$R^2_{(adj.)}$ 0.9998 (non-linear), Modifier Pd/Mg



$R^2_{(adj.)}$ 0.9990 (non-linear), Modifier Pd/Mg

Figure 1 continued: Typical calibration functions according to ISO 15586

Instrument settings

The ZEEnit 650P atomic absorption spectrometer was used to determine the content of the analytes in the tested samples (soil, sediment, and water) in accordance with ISO 15586. The AS-GF autosampler can automatically perform variable sample dilutions or preparation of calibration solutions, standard addition method, and spiking of samples. This analysis technique also offers the option of injecting samples into the tube several times and drying them between the steps. This allows samples and standards to be automatically enriched prior to measurement.
The device specifications and measurement parameters used are listed in table 2 and 3.

Magnesium nitrate with a concentration of 0.5 g L^{-1} Mg and a mixture of palladium and magnesium nitrate (concentration: 1 g L^{-1} Pd and 0.5 g L^{-1} Mg) were used as matrix modifiers. Another variant of the matrix modifier for the analytes Cd and Pb is ammonium dihydrogen phosphate. A concentration of 10 g L^{-1} $NH_4H_2PO_4$ was used in the series of measurements presented. The used modifier solutions for the quantification of the analytes is shown in table 2.
In sample digestions with HCl or aqua regia highly volatile chlorine compounds of the analytes can lead to lower signal intensities and thus to lower results in the calibration with HNO_3-stabilized standards. By reducing the palladium

modifier with ascorbic acid (concentration of ascorbic acid solution: 1 g L^{-1}), this interference can be minimized or even eliminated. To prevent the palladium(II) nitrate from precipitating in the pipetting tube, it is recommended to separate the modifier solution from the reducing solution. This option can be selected in the user software.

Table 2: General instrument parameters

Parameter	Specification
Device	ZEEnit 650P
Tube type	PIN platform wall tube for Mo
Injection volume	20 µL
Modifier	Injection volume 5 µL Pd/Mg: 1 g L^{-1} Pd, 0.5 g L^{-1} Mg Mg: 0.5 g L^{-1} Mg $NH_4H_2PO_4$: 10 g L^{-1}
Background correction	Zeeman, 2-field-mode Cu 1 T, other elements 0.8 T
Rinsing solution	1% (v/v) HNO_3, 0.05 % (w/w) Tergitol™ 9-S-15

Table 3: Applied lamp and spectrometer parameters

Element	Wavelength [nm]	Slit width [nm]	HCL current [mA]
Ag	328.1	0.8	2.5
Al	309.3	0.8	4
As	193.7	0.8	5.5
Cd	228.8	0.8	2
Co	240.7	0.2	5
Cr	357.9	0.2	4
Cu	324.7	0.8	2
Fe	248.3	0.2	5
Mn	279.5	0.2	5
Mo	313.3	0.8	5
Ni	232.0	0.2	4
Pb	283.3	0.8	3
Sb	217.6	0.2	6
Se	196.0	1.2	5
Tl	276.8	0.5	4
Zn	213.8	0.8	2

Table 4: Recommended temperature-time programs for the analytes

Element

Temperature-time program

Ag

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	650	300	15	16.0	Max	Stop
6		AZ*	650	0	6	6.0	Stop	Stop
7		Atomize	1800	1450	4	4.8	Stop	Stop
8		Clean	2450	500	4	5.3	Max	Stop

Al

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1000	300	10	12.2	Max	Stop
6		AZ*	1000	0	6	6.0	Stop	Stop
7		Atomize	2500	1450	4	5.0	Stop	Stop
8		Clean	2550	500	4	4.1	Max	Stop

As

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1150	500	15	16.6	Max	Stop
6		AZ*	1150	0	6	6.0	Stop	Stop
7		Atomize	2100	1450	4	4.7	Stop	Stop
8		Clean	2450	500	4	4.7	Max	Stop

Cd

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	550	300	15	15.7	Max	Stop
6		AZ*	550	0	6	6.0	Stop	Stop
7		Atomize	1450	1400	3	3.6	Stop	Stop
8		Clean	2450	500	4	6.0	Max	Stop

Modifier: $NH_4H_2PO_4$

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	600	300	15	15.8	Max	Stop
6		AZ*	600	0	6	6.0	Stop	Stop
7		Atomize	1650	1400	3	3.8	Stop	Stop
8		Clean	2450	500	4	5.6	Max	Stop

Modifier: Pd/Mg

Co

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1000	300	15	17.2	Max	Stop
6		AZ*	1000	0	6	6.0	Stop	Stop
7		Atomize	2250	1450	5	5.9	Stop	Stop
8		Clean	2450	500	4	4.4	Max	Stop

Table 4 continued: Recommended temperature-time programs for the analytes

Element | **Temperature-time program**

Cr

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1050	300	10	12.3	Max	Stop
6		AZ*	1050	0	6	6.0	Stop	Stop
7		Atomize	2450	1500	5	5.9	Stop	Stop
8		Clean	2550	500	4	4.2	Max	Stop

Cu

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	0	1.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1000	300	10	12.2	Max	Stop
6		AZ*	1000	0	6	6.0	Stop	Stop
7		Atomize	2100	1500	4	4.7	Stop	Stop
8		Clean	2450	500	4	4.7	Max	Stop

Fe

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1000	300	10	12.2	Max	Stop
6		AZ*	1000	0	6	6.0	Stop	Stop
7		Atomize	2200	1450	4	4.8	Stop	Stop
8		Clean	2450	500	4	4.5	Max	Stop

Mn

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	950	300	10	12.0	Max	Stop
6		AZ*	950	0	6	6.0	Stop	Stop
7		Atomize	2100	1500	4	4.8	Stop	Stop
8		Clean	2450	500	4	4.7	Max	Stop

Mo

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1600	1100	10	11.1	Max	Stop
6		AZ*	1600	0	6	6.0	Stop	Stop
7		Atomize	2600	1450	4	4.7	Stop	Stop
8		Clean	2650	500	4	4.1	Max	Stop

Ni

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1000	300	10	12.2	Max	Stop
6		AZ*	1000	0	6	6.0	Stop	Stop
7		Atomize	2450	1500	5	6.0	Stop	Stop
8		Clean	2550	500	4	4.2	Max	Stop

Table 4 continued: Recommended temperature-time programs for the analytes

Element | **Temperature-time program**

Pb

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	750	300	15	16.3	Max	Stop
6		AZ*	750	0	6	6.0	Stop	Stop
7		Atomize	1600	1400	3	3.6	Stop	Stop
8		Clean	2450	500	4	5.7	Max	Stop

Modifier: $NH_4H_2PO_4$

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	900	300	15	16.8	Max	Stop
6		AZ*	900	0	6	6.0	Stop	Stop
7		Atomize	2150	1400	3	3.9	Stop	Stop
8		Clean	2450	500	4	4.6	Max	Stop

Modifier: Pd/Mg

Sb

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1050	500	15	16.4	Max	Stop
6		AZ*	1050	0	6	6.0	Stop	Stop
7		Atomize	2050	1450	4	4.7	Stop	Stop
8		Clean	2450	500	4	4.8	Max	Stop

Se

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	50	5	9.8	Max	Stop
5		Pyrolysis	1000	300	10	12.2	Max	Stop
6		AZ*	1000	0	6	6.0	Stop	Stop
7		Atomize	2200	1450	3	3.8	Stop	Stop
8		Clean	2450	500	4	4.5	Max	Stop

Tl

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	160	50	5	6.0	Max	Stop
5		AZ*	160	0	6	6.0	Stop	Stop
6		Atomize	1850	1450	3	4.2	Stop	Stop
7		Clean	2450	500	4	5.2	Max	Stop

Zn

Step	*	Name	Temp. [°C]	Ramp [°C/s]	Hold s	Time s	Gas int.	Gas Add.
1		Drying	85	6	20	29.2	Max	Stop
2		Drying	90	3	20	21.7	Max	Stop
3		Drying	110	5	10	14.0	Max	Stop
4		Pyrolysis	350	150	5	6.6	Max	Stop
5		Pyrolysis	750	500	10	10.8	Max	Stop
6		AZ*	750	0	6	6.0	Stop	Stop
7		Atomize	1950	1450	2	2.8	Stop	Stop
8		Clean	2450	500	4	5.0	Max	Stop

Results and Discussion

Table 5 lists the typically achievable detection and quantification limits of the device. The limits were determined using the blank value method. An 11-fold blank value measurement was carried out and the 3σ or 9σ criterion of the standard deviation was used.

The elements aluminum, antimony, arsenic, cadmium, chromium, cobalt, copper, iron, lead, manganese, molybdenum, nickel, selenium, silver, tellurium, and zinc were determined in solids and in natural water in accordance with ISO 15586. The results of the series of measurements are shown in table 6 and compared with the expected value of the reference materials. The certificates of the materials BAM U110, BCR 143R and BCR 146R indicate values for the total content and the aqua regia soluble content of the analytes (reference to table 7, 1st value total content, 2nd value content of aqua regia extract). The measurement uncertainty of the AAS determination is based on the standard deviation of the three replicates.

Table 5: Achievable limits of detection (LOD) and limits of quantification (LOQ) of the presented method according to the 3σ and 9σ criterion

Element	Wavelength [nm]	LOD [µg L^{-1}]	LOQ [µg L^{-1}]
Ag	328	0.033	0.098
Al	309	0.5	1.5
As	193	1.1	3.3
Cd	228	0.010	0.030
Co	240	0.24	0.71
Cr	357	0.070	0.21
Cu	324	0.11	0.33
Fe	248	0.9	2.7
Mn	279	0.044	0.13
Mo	313	0.17	0.51
Ni	232	0.41	1.2
Pb	283	0.33	1.0
Sb	217	1.2	3.6
Se	196	1.5	4.5
Tl	276	0.68 0.11*	2.0 0.33*
Zn	213	0.037	0.11

* 5-fold enrichment of sample/standard

Table 6: Measurement results of the determination of analytes in surface water, sediment, sewage sludge and soil

Sample	Element	Dilution factor	Recovery [%]	Measured value		Target value	
				[µg kg^{-1}]			
SRM 1640a	Ag	1	101	8.127	± 0.051	8.017	± 0.042
	Al	1	100	52.7	± 1.4	52.6	± 1.8
	As	1	105	8.41	± 0.44	8.010	± 0.067
	Cd	2	100	3.948	± 0.047	3.961	± 0.072
	Co	1	97	19.57	± 0.25	20.08	± 0.24
	Cr	3	102	41.02	± 1.2	40.22	± 0.28
	Cu	3	98	83.56	± 3.1	85.07	± 0.48
	Fe	2	95	34.8	± 0.68	36.5	± 1.7
	Mn	4	100	40.07	± 0.40	40.07	± 0.35
	Mo	1	98	44.5	± 0.15	45.24	± 0.59
	Ni	1	100	25.17	± 0.42	25.12	± 0.12
	Pb	1	99	11.9	± 0.36	12.005	± 0.040
	Sb	2-fold enriched	98	4.950	± 0.022	5.064	± 0.045
	Se	1	99	19.8	± 0.65	19.97	± 0.16
	Tl	4-fold enriched	87	1.40	± 0.13	1.606	± 0.015
	Zn	2	105	57.7	± 3.1	55.20	± 0.32
Sample	**Element**	**Dilution factor**	**Recovery [%]**	**Measured value**		**Target value**	
				[mg kg^{-1}]			
BAM U110	As	2	84 102	13.28	± 0.11	15.8 13.0*	± 1.4 ± 1.1
	Cd	20	96 100	7.0	± 0.23	7.3 7.0*	± 0.6 ± 0.4
	Co	2	85 95	13.8	± 0.36	16.2 14.5*	± 1.6 ± 0.8
	Cr	100	98 119	226	± 4.7	230 190*	± 13 ± 9
	Cu	100	98 98	258	± 2.7	263 262*	± 12 ± 9
	Mn	300	97 103	600.1	± 1.4	621 580*	± 20 ± 19
	Ni	20	90 95	91.2	± 0.1	101 95.6*	± 5 ± 4.0
	Pb	20	99	195	± 3.2	197	± 14

Table 6 continued: Measurement results of the determination of analytes in surface water, sediment, sewage sludge and soil

Sample	Element	Dilution factor	Recovery [%]	Measured value		Target value	
				[µg kg^{-1}]			
BCR 143R	Cd	200	98 98	70.52	± 2.8	71.8 72.0*	± 1.2 ± 1.8
	Co	2	94	11.6	± 0.19	12.3	± 0.3
	Cu	30	101	132	± 2.0	130.6	± 1.5
	Mn	400	101 106	912	± 12	904 858*	± 13 ± 11
	Ni	30	98 99	292.4	± 5.4	299 296*	± 5 ± 4
	Pb	20	100 103	180	± 1.4	179.7 174*	± 2.1 ± 5
BCR 146R	Cd	30	103 105	19.3	± 0.21	18.8 18.4*	± 0.5 ± 0.4
	Co	1	91 103	6.7	± 0.18	7.39 6.5*	± 0.27 ± 0.4
	Cr	100	99 111	194	± 1.8	196 174*	± 7 ± 7
	Cu	200	99 99	826	± 2.3	838 831*	± 16 ± 16
	Mn	200	98 106	317	± 1.7	323 298*	± 7 ± 9
	Ni	10	86 93	60.2	± 1.0	70 65.0*	± 5 ± 3.0
	Pb	50	98 102	593.8	± 5.0	609 583*	± 14 ± 17
PACS 2	Ag	1	108	1.321	± 0.062	1.22	± 0.14
	As	2	101	26.4	± 0.53	26.2	± 1.5
	Cd	4	104	2.2	± 0.19	2.11	± 0.15
	Co	2	95	10.9	± 0.22	11.5	± 0.3
	Cr	30	94	85.2	± 4.6	90.7	± 4.6
	Cu	100	99	308	± 2.4	310	± 12
	Mn	200	101	443	± 3.7	440	± 19
	Mo	1	98	5.32	± 0.13	5.43	± 0.28
	Ni	5	95	37.35	± 0.32	39.5	± 2.3
	Pb	20	103	189.1	± 1.1	183	± 8
	Sb	1	95	10.73	± 0.29	11.3	± 2.6
	Se	1	88	0.813	± 0.072	0.92	± 0.22

Table 6 continued: Measurement results of the determination of analytes in surface water, sediment, sewage sludge and soil

Sample	Element	Dilution factor	Recovery [%]	Measured value		Target value	
				[mg kg^{-1}]			
IAEA 457	Ag	2	109	2.020	± 0.015	1.85	± 0.39
	As	1	105	10.70	± 0.075	10.2	± 1.0
	Cd	2	98	1.07	± 0.015	1.09	± 0.08
	Co	2	95	13.9	± 0.11	14.7	± 1.0
	Cr	50	92	132	± 1.2	144	± 8
	Cu	100	101	368	± 1.9	365	± 19

* Aqua regia soluble content

Summary

Cost-effective analysis of the elements aluminum, antimony, arsenic, cadmium, chromium, cobalt, copper, iron, lead, manganese, molybdenum, nickel, selenium, silver, tellurium, and zinc in solid and water samples in accordance with ISO 15586 is easy and user-friendly with the ZEEnit 650P electrothermal atomic absorption spectrometer. The third-generation Zeeman furnace not only enables precise measurement of the analytes even with a high background signal, it is also possible to significantly extend the calibration range by physically attenuating the signal (3-field mode). Convenient and automatic sample handling is guaranteed by the AS-GF autosampler. Variable, automatic, and predefined sample dilutions, sample spiking, standard addition procedures and sample enrichment in the graphite tube can be implemented easily and software-controlled with this autosampler.

Figure 2: ZEEnit 650P

Recommended device configuration

Table 7: Overview of devices, accessories, and consumables

Item	Part number	Description
ZEEnit 650P	813-0650P-2-K	ZEEnit 650P - Graphite Furnace AAS with Zeeman background correction
Chiller, 50 Hz	810-60053-0	Software-controlled cooling system, (50 Hz power supply)
Chiller, 60 Hz	810-60052-0	Software-controlled cooling system, (60 Hz power supply)
Graphite tube platform	407-152.314	Z-graphite tube PIN-platform -pyrolytically coated (10 pcs.)
Graphite tube wall	407-152.315	Z-standard graphite tube - pyrolytically coated (10 pcs.)
Ag-HCL	480-450.051C	Coded Hollow Cathode Lamp Silver (Ag)
Al-HCL	480-450.001C	Coded Hollow Cathode Lamp Aluminum (Al)
As-HCL	480-450.003C	Coded Hollow Cathode Lamp Arsenic (As)
Cd-HCL	480-450.008C	Coded Hollow Cathode Lamp Cadmium (Cd)
Co-HCL	480-450.013C	Coded Hollow Cathode Lamp Cobalt (Co)
Cr-HCL	480-450.012C	Coded Hollow Cathode Lamp Chromium (Cr)
Cu-HCL	480-450.014C	Coded Hollow Cathode Lamp Cupper (Co)
Fe-HCL	480-450.026C	Coded Hollow Cathode Lamp Iron (Fe)
Mn-HCL	480-450.032C	Coded Hollow Cathode Lamp Manganese (Mn)
Mo-HCL	480-450.034C	Coded Hollow Cathode Lamp Molybdenum (Mo)
Ni-HCL	480-450.036C	Coded Hollow Cathode Lamp Nickel (Ni)
Pb-HCL	480-450.028C	Coded Hollow Cathode Lamp Lead (Pb)
Sb-HCL	480-450.002C	Coded Hollow Cathode Lamp Antimony (Sb)
Se-HCL	480-450.049C	Coded Hollow Cathode Lamp Selenium (Se)
Tl-HCL	480-450.057C	Coded Hollow Cathode Lamp Thallium (Tl)
Zn-HCL	480-450.067C	Coded Hollow Cathode Lamp Zinc (Zn)

References

[1] EN ISO 15586: Water quality – Determination of trace elements using atomic absorption spectrometry with graphite furnace

[2] EN ISO 54321:2021: Soil, treated biowaste, sludge and waste – Digestion of aqua regia soluble fractions of elements

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