



### Challenge

Efficient multi-element analysis of waste leachate and eluates of varying matrix composition

### Solution

The PlasmaQuant MS Q enables robust, sensitive, and accurate multi-element analysis of semi-metals and heavy metals in waste eluates and leachates permitting fast routine analysis for evaluating hazardousness

## Determination of Trace Elements in Waste Eluates and Leachates in Accordance with DIN EN ISO 17294-2 Using ICP-MS

### Introduction

Waste that cannot be recycled or thermally recovered is disposed of in landfills. In Germany, there is a classification of the hazardousness of waste for the environment and the resulting precautions that are necessary for storage. For mineral residues and waste in the lowest landfill class, DK 0, there is also the possibility of material recycling in soil-like applications (e.g., backfilling), technical structures (e.g., noise barriers and road embankments) or as landfill substitute construction materials. In Germany, the Landfill Ordinance (DepV)<sup>[1]</sup> regulates the allocation values for the landfill classes and the area of use of landfill substitute construction materials, as well as the parameters and analytical methods to determine them. The technical rules for the recycling of mineral residues and wastes are described in Communication 20 of the German Federal/State Working Group on Waste (LAGA)<sup>[2]</sup>. The LAGA is a working body of the Conference of Environment Ministers, in which representatives of all federal and state environment

ministries sit and strive to achieve uniform regulations in waste legislation.

For the assessment of the environmental hazard of waste to be landfilled or recycled, the fraction of pollutants that can be washed out by precipitation and enter the environment or groundwater is particularly relevant. In order to be able to estimate this, eluates of the waste must be produced according to methods specified in the DepV, which are then examined for pollutants such as toxic semimetals and heavy metals. In addition, there are limit values for the natural leachate, which – if exceeded – require the leachate to be treated before being discharged into receiving waters. The handling of landfill waste leachate and the corresponding limit values are regulated in the German Wastewater Ordinance (AbwV)<sup>[3]</sup>.

Due to the different types of waste, eluates and leachates are characterized by a high variability of matrix composition. Therefore, in order to comply with the ever lower regulatory

limits, analytical systems with high sensitivity and robustness are required. The ICP-MS technique meets this requirement by offering the possibility of fast, robust, and sensitive multi-element analysis. This work uses eluate and leachate samples to demonstrate the robust and sensitive determination of toxic semi-metals and heavy metals as required by DepV and AbwV in accordance with DIN EN ISO 17294-2<sup>[4]</sup> using the PlasmaQuant MS Q and its suitability for routine analysis of waste eluates and leachates.

In other countries, legislation and regulations can be different. In the U.S., waste is classified as hazardous

or non-hazardous waste with regard to the toxicity characteristic based on the maximum concentration of contaminants in the waste eluate, which is specified in 40 CFR Part 261 § 261.24<sup>[5]</sup>. Additionally, it has to be ensured that landfill waste leachate does not contaminate ground water by monitoring the ground water from wells around the landfill. Samples taken from the ground water should not exceed the maximum contaminant levels specified in 40 CFR Part 264 §264.94<sup>[6]</sup>.

## Materials and Methods

### Samples and reagents

The samples were five filtered (0.45 µm) waste eluate samples prepared according to DepV and one waste leachate sample also filtered over 0.45 µm, all of which were stored in the refrigerator until measurement. All samples were diluted 100- and 10-fold by 1% nitric acid (HNO<sub>3</sub>, ultra-pure, distilled), prior to measurement, except for the leachate sample (100- and 1000-fold). The eluate samples 1, 2, and 5 were additionally also diluted 1000-fold to remain within the calibration range for individual elements.

For validating the calibration, the surface water reference materials NIST 1640a (10-fold dilution, NIST, U.S.) and NIST 1643f (100-fold dilution, NIST, U.S.) were diluted with 1% HNO<sub>3</sub> and measured. For validating the results of the samples, two waste eluate samples and the waste leachate sample were spiked with a multi-element standard.

For stabilizing Hg, 200 µg/L Au (Certipur®, 1000 mg/L) was added to the diluted samples. In order to correct for long-term signal drifts and matrix effects, a solution of 20 µg/L of Sc, Y, Tb, and Ir (Sc, Tb: TraceCert®, 1000 mg/L; Y: TraceCert®, 10000 mg/L; In, Ir: High-Purity Standards, 1000 mg/L) was added as an internal standard online via a Y-piece. For sample introduction via the peristaltic pump, black/black PVC tubing (0.76 mm ID) for samples and orange/green PVC tubing (0.38 mm ID) for the internal standard solution were used. For blank and wash samples, carrier and rinsing solution, 1% HNO<sub>3</sub> was used, and 200 µg/L Au was added to the blank and wash samples to stabilize Hg and avoid memory effects. Sample preparation and measurements did not take place under cleanroom conditions.

### Calibration

Table 1: Concentration levels for calibration standards of the elements

Element	STD1 [µg/L]	STD2 [µg/L]	STD3 [µg/L]	STD4 [µg/L]	STD5 [µg/L]	STD6 [µg/L]	STD7 [µg/L]	STD8 [µg/L]	STD9 [µg/L]	STD10 [µg/L]
As	0.1	0.2	0.5	1	2	5	10	-	-	-
Ba	0.1	0.2	0.5	1	2	5	10	20	50	-
Cd	0.001	0.002	0.005	0.01	0.02	0.05	0.1	0.2	-	-
Cr	0.1	0.2	0.5	1	2	5	10	-	-	-
Cu	0.1	0.2	0.5	1	2	5	10	20	50	-
Hg	0.001	0.002	0.005	0.01	0.02	0.05	0.1	0.2	0.5	1
Mo	0.1	0.2	0.5	1	2	5	10	-	-	-
Ni	0.1	0.2	0.5	1	2	5	10	20	50	100
Pb	0.01	0.02	0.05	0.1	0.2	0.5	1	2	5	-
Sb	0.01	0.02	0.05	0.1	0.2	0.5	1	-	-	-

Table 1 (continued): Concentration levels for calibration standards of the elements

Element	STD1 [µg/L]	STD2 [µg/L]	STD3 [µg/L]	STD4 [µg/L]	STD5 [µg/L]	STD6 [µg/L]	STD7 [µg/L]	STD8 [µg/L]	STD9 [µg/L]	STD10 [µg/L]
Se	-	0.02	0.05	0.1	0.2	0.5	1	2	5	-
Tl	0.001	0.002	0.005	0.01	0.02	0.05	0.1	0.2	-	-
Zn	0.1	0.2	0.5	1	2	5	10	20	50	-

The calibration solutions were prepared in 1% HNO<sub>3</sub>. For stabilizing Hg, 200 µg/L Au was added. Single element standards (As, Cd, Cu, Hg, Pb, Tl: Certipur®, 1000 mg/L; Ba, Cr, Mo, Ni: TraceCert®, 1000 mg/L; Sb: TraceCert®, 1000 mg/L; Zn: TraceCert®, 10000 mg/L; Se: High-Purity Standards, 1000 mg/L) were used to prepare the multi-element standards for calibration. The concentration levels used are shown in Table 1.

### Instrument parameters

For the analysis, the PlasmaQuant MS Q was equipped with an autosampler (ASX-560, CETAC) with cover and HEPA filter, Seaspray nebulizer (0.4 mL/min), Scott double-pass spray chamber, Fassel-torch with 2.4 mm injector, and Ni sampler and skimmer cones. For higher sample throughput, the ASXPress Plus (CETAC) rapid sample introduction system was used.

### Method parameters

Table 3: Method parameters

Parameter	Specification
Plasma gas flow	9.0 L/min
Auxiliary gas flow	1.20 L/min
Sheath gas flow	0.00 L/min
Nebulizer gas flow	0.97 L/min
RF power	1.35 kW
Sampling depth	5.0 mm
Pump speed	20 rpm
Stabilization time	25 s (H <sub>2</sub> ); 15 s (H <sub>2</sub> B); 15 s (nG); 20 s (He)*
Spray chamber temperature	3 °C
Skimmer voltage (BOOST)	10 V (H <sub>2</sub> B)
Points per peak	1 (peak hopping)
Scans per replicate	12
Replicates	5
Dwell time	200 ms (Hg); 20 ms (all other isotopes)

\* Switching times of < 5 s between measurement modes can be selected. In favor of the best possible measurement precision, however, higher stabilization times were used here, achieving an RSD of < 2% on average.

H<sub>2</sub> – Hydrogen mode, H<sub>2</sub>B – Hydrogen boost mode, nG – No gas mode, He – Helium mode

Table 2: Instrument configuration

Parameter	Specification
Nebulizer	Seaspray (0.4 mL/min)
Spray chamber	Scott double-pass
Torch	Fassel-Torch with 2.4 mm injector
Cones	Nickel sampler and skimmer
iCRC gas, flow	H <sub>2</sub> – 80 mL/min; H <sub>2</sub> – 200 mL/min (BOOST); He – 120 mL/min
Autosampler	ASX-560 (CETAC) with cover, HEPA filter and ASX-press Plus (CETAC) rapid sample introduction system

### Acquisition parameters

For Pb, the sum of the three main isotopes ( $^{206}\text{Pb}$ ,  $^{207}\text{Pb}$ ,  $^{208}\text{Pb}$ ) was formed to account for possible variations in the relative isotope abundances in samples and standards.

For Hg, the sum of the four main isotopes ( $^{199}\text{Hg}$ ,  $^{200}\text{Hg}$ ,  $^{201}\text{Hg}$ ,  $^{202}\text{Hg}$ ) was formed to achieve higher sensitivity. For correction by the internal standard, the isotopes  $^{45}\text{Sc}$ ,  $^{89}\text{Y}$ ,  $^{115}\text{In}$ ,  $^{159}\text{Tb}$ , and  $^{193}\text{Ir}$  were selected.

For the removal of matrix and/or plasma based polyatomic interferences, helium was used as the collision gas and hydrogen as the reaction gas via Analytik Jena's patented integrated collision and reaction cell (iCRC). To achieve the highest sensitivity and lowest detection limits for elements

measured in reaction gas mode, the patented BOOST technology was used. In BOOST mode, a positive voltage is applied to the skimmer cone. This enables compensating for the loss of sensitivity when using high reaction gas flows. Isotopes without polyatomic interference were measured in no gas mode. Thus, four measurement modes were used within one measurement:  $\text{H}_2$ ,  $\text{H}_2\text{Boost}$ , no-gas, and He mode.

For automated and optimized sample introduction, the ASXpress Plus rapid sample introduction system with an ASX-560 autosampler (both CETAC) was used, allowing a measurement time of approx. 3 minutes per sample.

Table 4: Expected interferences on the chosen isotopes and corresponding modes applied for measurements

Isotope	Expected polyatomic interference	Applied iCRC mode	Correction equation
$^{52}\text{Cr}$	$^{35}\text{Cl}^{16}\text{O}^{1}\text{H}^+$ , $^{34}\text{S}^{16}\text{O}^+$ , $^{36}\text{Ar}^{16}\text{O}^+$ , $^{40}\text{Ar}^{12}\text{C}^+$	He	
$^{60}\text{Ni}$	$^{44}\text{Ca}^{16}\text{O}^+$ , $^{23}\text{Na}^{37}\text{Cl}^+$	He	
$^{65}\text{Cu}$	$^{40}\text{Ar}^{25}\text{Mg}^+$	$\text{H}_2\text{B}$	
$^{66}\text{Zn}$	$^{31}\text{P}^{35}\text{Cl}^+$ , $^{32}\text{S}^{34}\text{S}^+$	He	
$^{75}\text{As}$	$^{40}\text{Ar}^{35}\text{Cl}^+$	$\text{H}_2$	
$^{78}\text{Se}$	$^{40}\text{Ar}^{38}\text{Ar}^+$ , $^{38}\text{Ar}^{40}\text{Ca}^+$	$\text{H}_2$	$- 0.03043 * ^{83}\text{Kr}$
$^{95}\text{Mo}$	-	nG	
$^{114}\text{Cd}$	-	nG	$- 0.0268 * ^{118}\text{Sn}$
$^{121}\text{Sb}$	-	nG	
$^{137}\text{Ba}$	-	nG	
$^{199-202}\text{Hg}$	-	nG	
$^{205}\text{Tl}$	-	nG	
$^{206-208}\text{Pb}$	-	nG	

$\text{H}_2$  – Hydrogen mode

$\text{H}_2\text{B}$  – Hydrogen boost mode

nG – No gas mode

He – Helium mode

## Results and Discussion

### Validation

The calibration was validated by measuring certified reference materials (surface water). These were diluted 10- and 100-fold to cover the same concentration range as the samples. As shown in Table 5, recoveries in the range of 90% to 108% were obtained, with most results between 95% and 106%.

Table 5: Measured and certified element concentrations and recoveries of the surface water reference materials (measured in 10-fold and 100-fold dilution)

Isotope [Mode]	NIST 1640a (10-fold dilution)			NIST 1643f (100-fold dilution)		
	Concentration [µg/L]	Reference value [µg/L]	Recovery [%]	Concentration [µg/L]	Reference value [µg/L]	Recovery [%]
<sup>52</sup> Cr [He]	42.4	40.54 ± 0.30	105	19.6	18.50 ± 0.10	106
<sup>60</sup> Ni [He]	26.6	25.32 ± 0.14	105	61.6	59.8 ± 1.4	103
<sup>65</sup> Cu [H <sub>2</sub> B]	87.3	85.75 ± 0.51	102	19.8	21.66 ± 0.71	91
<sup>66</sup> Zn [He]	51.6	55.64 ± 0.35	93	66.9	74.4 ± 1.7	90
<sup>75</sup> As [H <sub>2</sub> ]	8.04	8.075 ± 0.070	100	54.5	57.42 ± 0.38	95
<sup>78</sup> Se [H <sub>2</sub> ]	20.1	20.13 ± 0.17	100	12.6	11.700 ± 0.081	108
<sup>95</sup> Mo [nG]	45.4	45.60 ± 0.61	100	112	115.3 ± 1.7	97
<sup>114</sup> Cd [nG]	3.95	3.992 ± 0.074	99	5.80	5.89 ± 0.13	98
<sup>121</sup> Sb [nG]	5.22	5.105 ± 0.046	102	58.8	55.45 ± 0.40	106
<sup>137</sup> Ba [nG]	160	151.80 ± 0.83	106	494	518.2 ± 7.3	104
<sup>199-202</sup> Hg [nG]	-	n. s.	-	-	n. s.	-
<sup>205</sup> Tl [nG]	1.72	1.619 ± 0.016	106	7.06	6.892 ± 0.035	102
<sup>206-208</sup> Pb [nG]	12.4	12.101 ± 0.050	102	18.1	18.488 ± 0.084	98

H<sub>2</sub> – Hydrogen mode

H<sub>2</sub>B – Hydrogen boost mode

nG – No gas mode

He – Helium mode

n. s. – not specified

### Elemental concentrations

The analyte concentrations determined in the waste eluate samples are shown in Table 6.

Table 6: Elemental concentrations in waste eluate samples

Isotope [Mode]	Concentration				
	Sample 1 [µg/L]	Sample 2 [µg/L]	Sample 3 [µg/L]	Sample 4 [µg/L]	Sample 5 [µg/L]
<sup>52</sup> Cr [He]	0.49	0.57	49.5	1.44	13.9
<sup>60</sup> Ni [He]	3632	6937	8.88	0.89	16.5
<sup>65</sup> Cu [H <sub>2</sub> B]	11.3	4.93	127	3.29	722
<sup>66</sup> Zn [He]	148	201	< 1.1	2.83	377
<sup>75</sup> As [H <sub>2</sub> ]	2.67	2.74	0.65	8.83	< 0.57

Table 6 (continued): Elemental concentrations in waste eluate samples

Isotope [Mode]	Concentration				
	Sample 1 [ $\mu\text{g/L}$ ]	Sample 2 [ $\mu\text{g/L}$ ]	Sample 3 [ $\mu\text{g/L}$ ]	Sample 4 [ $\mu\text{g/L}$ ]	Sample 5 [ $\mu\text{g/L}$ ]
$^{95}\text{Mo}$ [nG]	7.43	13.9	22.5	3.51	174
$^{114}\text{Cd}$ [nG]	0.69	0.27	0.027	0.014	0.20
$^{121}\text{Sb}$ [nG]	5.04	5.56	2.42	2.87	1.58
$^{137}\text{Ba}$ [nG]	46.0	23.2	44.6	10.2	968
$^{199-202}\text{Hg}$ [nG]	0.032	0.023	0.015	0.010	0.013
$^{205}\text{Tl}$ [nG]	0.044	< 0.030	1.67	0.080	0.29
$^{206-208}\text{Pb}$ [nG]	1.09	0.37	0.034	1.68	3507

 $\text{H}_2$  – Hydrogen mode $\text{H}_2\text{B}$  – Hydrogen boost mode

nG – No gas mode

He – Helium mode

As an example, two of the eluate samples were spiked with a multi-element standard and the recovery of the spiked concentrations was checked. As shown in Table 7, recoveries between 91% and 118% were achieved, which underlines the validity of the results.

Table 7: Recoveries of added elemental concentration in two selected waste eluate samples

Isotope [Mode]	Eluate sample 1			Eluate sample 2		
	Original concentration [ $\mu\text{g/L}$ ]	Spiked concentration [ $\mu\text{g/L}$ ]	Recovery [%]	Original concentration [ $\mu\text{g/L}$ ]	Spiked concentration [ $\mu\text{g/L}$ ]	Recovery [%]
$^{52}\text{Cr}$ [He]	0.49	3	108	0.57	2	103
$^{60}\text{Ni}$ [He]	3632	5000	107	6937	5000	103
$^{65}\text{Cu}$ [ $\text{H}_2\text{B}$ ]	11.3	10	118	4.93	2	108
$^{66}\text{Zn}$ [He]	148	75	102	201	75	108
$^{75}\text{As}$ [ $\text{H}_2$ ]	2.67	3	97	2.74	2	117
$^{78}\text{Se}$ [ $\text{H}_2$ ]	< 0.47	2	100	< 0.47	2	102
$^{95}\text{Mo}$ [nG]	7.43	3	96	13.9	2	104
$^{114}\text{Cd}$ [nG]	0.69	0.75	110	0.27	0.7	98
$^{121}\text{Sb}$ [nG]	5.04	7.5	94	5.56	7	92
$^{137}\text{Ba}$ [nG]	46.0	75	91	23.2	70	94
$^{199-202}\text{Hg}$ [nG]	0.044	0.15	93	< 0.030	0.15	100
$^{205}\text{Tl}$ [nG]	0.032	0.03	104	0.023	0.02	103
$^{206-208}\text{Pb}$ [nG]	1.09	7.5	99	0.37	0.2	99

 $\text{H}_2$  – Hydrogen mode $\text{H}_2\text{B}$  – Hydrogen boost mode

nG – No gas mode

He – Helium mode

The element concentrations of the waste leachate sample are shown in Table 8. This sample was also spiked with a multi-element standard. The recoveries ranged from 85% to 117%.

Table 8: Elemental concentrations and recoveries of spiked concentrations in the waste leachate sample

Isotope [Mode]	Leachate sample		
	Original concentration [ $\mu\text{g/L}$ ]	Spiked concentration [ $\mu\text{g/L}$ ]	Recovery [%]
$^{52}\text{Cr}$ [He]	232	200	107
$^{60}\text{Ni}$ [He]	929	750	101
$^{65}\text{Cu}$ [ $\text{H}_2\text{B}$ ]	790	750	117
$^{66}\text{Zn}$ [He]	1081	750	103
$^{75}\text{As}$ [ $\text{H}_2$ ]	42.6	10	101
$^{78}\text{Se}$ [ $\text{H}_2$ ]	< 4.7	20	101
$^{95}\text{Mo}$ [nG]	217	200	92
$^{114}\text{Cd}$ [nG]	2.92	2	107
$^{121}\text{Sb}$ [nG]	19.1	20	99
$^{137}\text{Ba}$ [nG]	1008	200	98
$^{199-202}\text{Hg}$ [nG]	1.51	1.5	107
$^{205}\text{Tl}$ [nG]	0.20	0.1	90
$^{206-208}\text{Pb}$ [nG]	10.4	20	85

$\text{H}_2$  – Hydrogen mode

$\text{H}_2\text{B}$  – Hydrogen boost mode

nG – No gas mode

He – Helium mode

### Sensitivity

The instrumental limits of detection (iLOD) and quantification (iLOQ) of the calibration were determined using the blank method according to DIN 32645<sup>[7]</sup> and are shown in Table 9. The method detection (mLOD) and quantification (mLOQ) limits were calculated taking the dilution factor into account.

Table 9: Limits of detection and quantification according to DIN EN 32645 compared with assignment values for waste eluates and limit concentrations for wastewater and leachate

Isotope [Mode]	iLOD [ $\mu\text{g/L}$ ]	iLOQ [ $\mu\text{g/L}$ ]	10-fold dilution		100-fold dilution		Lowest assignment value <sup>1</sup> [ $\mu\text{g/L}$ ]	Limit waste water <sup>2</sup> [ $\mu\text{g/L}$ ]
			mLOD [ $\mu\text{g/L}$ ]	mLOQ [ $\mu\text{g/L}$ ]	mLOD [ $\mu\text{g/L}$ ]	mLOQ [ $\mu\text{g/L}$ ]		
$^{52}\text{Cr}$ [He]	0.0082	0.0273	0.082	0.273	0.82	2.73	15 (LAGA Z 0)	500
$^{60}\text{Ni}$ [He]	0.0019	0.0063	0.019	0.063	0.19	0.63	40 (LAGA Z 0)	1000
$^{65}\text{Cu}$ [ $\text{H}_2\text{B}$ ]	0.0103	0.0343	0.103	0.343	1.03	3.43	50 (LAGA Z 0)	500
$^{66}\text{Zn}$ [He]	0.0334	0.1112	0.334	1.112	3.34	11.12	100 (LAGA Z 0)	2000
$^{75}\text{As}$ [ $\text{H}_2$ ]	0.0017	0.0058	0.017	0.058	0.17	0.58	10 (LAGA Z 0)	100
$^{78}\text{Se}$ [ $\text{H}_2$ ]	0.0141	0.0468	0.141	0.468	1.41	4.68	10 (DepV DK 0)	-
$^{95}\text{Mo}$ [nG]	0.0003	0.0010	0.003	0.010	0.03	0.10	50 (DepV DK 0)	-

Table 9 (continued): Limits of detection and quantification according to DIN EN 32645 compared with assignment values for waste eluates and limit concentrations for waste water and leachate

Isotope [Mode]	iLOD [µg/L]	iLOQ [µg/L]	10-fold dilution		100-fold dilution		Lowest assign- ment value <sup>1</sup> [µg/L]	Limit waste water <sup>2</sup> [µg/L]
			mLOD [µg/L]	mLOQ [µg/L]	mLOD [µg/L]	mLOQ [µg/L]		
<sup>114</sup> Cd [nG]	0.0001	0.0005	0.001	0.005	0.01	0.05	2 (LAGA Z 0)	100
<sup>121</sup> Sb [nG]	0.0002	0.0007	0.002	0.007	0.02	0.07	6 (DepV DK 0)	-
<sup>137</sup> Ba [nG]	0.0012	0.0039	0.012	0.039	0.12	0.39	2000 (DepV DK 0)	-
<sup>199-202</sup> Hg [nG]	0.0009	0.0030	0.009	0.030	0.09	0.30	0.2 (LAGA Z 0)	50
<sup>205</sup> Tl [nG]	0.0001	0.0003	0.001	0.003	0.01	0.03	1 (LAGA Z 0)	-
<sup>206-208</sup> Pb [nG]	0.0005	0.0017	0.005	0.017	0.05	0.17	20 (LAGA Z 0)	500

<sup>1</sup> According to DepV, annex 3 (DK – landfill class) or LAGA communication 20 (Z – assignment value)

<sup>2</sup> According to AbwV, annex 51<sup>[9]</sup>

H<sub>2</sub> – Hydrogen mode  
H<sub>2</sub>B – Hydrogen boost mode  
nG – No gas mode  
He – Helium mode

All method quantification limits are below the assignment values listed in DepV, annex 3<sup>[8]</sup> and LAGA communication 20<sup>[2]</sup>, and limit concentrations for waste leachate listed in AbwV, annex 51<sup>[9]</sup> by at least a factor of 5. However, this is only true for Se and Hg in waste eluate samples diluted 10-fold. All other elements, as well as Hg in waste leachate, can also be determined in samples diluted 100-fold. As shown in Table 10, this also applies to samples analyzed under EPA 40 CFR regulations<sup>[5,6]</sup>. All elements can be determined in samples diluted 100-fold with exception of Se in ground water, for which the sample should be diluted only 10-fold. Ag was not determined within this work because it is not specified in German regulations, but further information on the determination of Ag in wastewater can be found in the respective application note "Wastewater analysis following U.S. EPA 200.8 using ICP-MS"<sup>[10]</sup>.

Table 10: Limits of detection and quantification according to DIN EN 32645 compared with EPA 40 CFR maximum contaminant concentration in waste extracts/eluates and ground water potentially mixed with waste leachates

Isotope [Mode]	iLOD [µg/L]	iLOQ [µg/L]	10-fold dilution		100-fold dilution		Extract concentration <sup>1</sup> [µg/L]	MCL <sup>2</sup> [µg/L]
			mLOD [µg/L]	mLOQ [µg/L]	mLOD [µg/L]	mLOQ [µg/L]		
<sup>52</sup> Cr [He]	0.0082	0.0273	0.082	0.273	0.82	2.73	5000	50
<sup>75</sup> As [H <sup>2</sup> ]	0.0017	0.0058	0.017	0.058	0.17	0.58	5000	50
<sup>78</sup> Se [H <sup>2</sup> ]	0.0141	0.0468	0.141	0.468	1.41	4.68	1000	10
<sup>114</sup> Cd [nG]	0.0001	0.0005	0.001	0.005	0.01	0.05	1000	10
<sup>137</sup> Ba [nG]	0.0012	0.0039	0.012	0.039	0.12	0.39	100000	1000
<sup>199-202</sup> Hg [nG]	0.0009	0.0030	0.009	0.030	0.09	0.30	200	2
<sup>206-208</sup> Pb [nG]	0.0005	0.0017	0.005	0.017	0.05	0.17	5000	50
Ag	n. d. <sup>3</sup>	n. d. <sup>3</sup>	n. d. <sup>3</sup>	n. d. <sup>3</sup>	n. d. <sup>3</sup>	n. d. <sup>3</sup>	5000	50

<sup>1</sup> Maximum contaminant concentration in waste extracts/eluates according to EPA 40 CFR Part 261 § 261.24

<sup>2</sup> Maximum contaminant level in ground water potentially mixed with waste leachate according to EPA 40 CFR Part 264 § 264.94

<sup>3</sup> Not determined because Ag is not specified in German regulations of waste eluates and leachates

H<sub>2</sub> – Hydrogen mode  
H<sub>2</sub>B – Hydrogen boost mode  
nG – No gas mode  
He – Helium mode

### Long-term stability

The waste eluate sample 1 was diluted 100-fold in 1% HNO<sub>3</sub> and spiked with 5 µg/L of each analyte. To monitor the long-term stability of the system, the diluted sample was measured repeatedly over a period of eight hours. The result of this measurement is shown in Figure 1, and the intensities of the analytes were normalized to the first data point in each case. The signal stability shown over the entire measurement period of eight hours and the relative standard deviation of the individual signals, ranging from 1.1% to 2.8%, confirm the good precision and robustness of the PlasmaQuant MS.

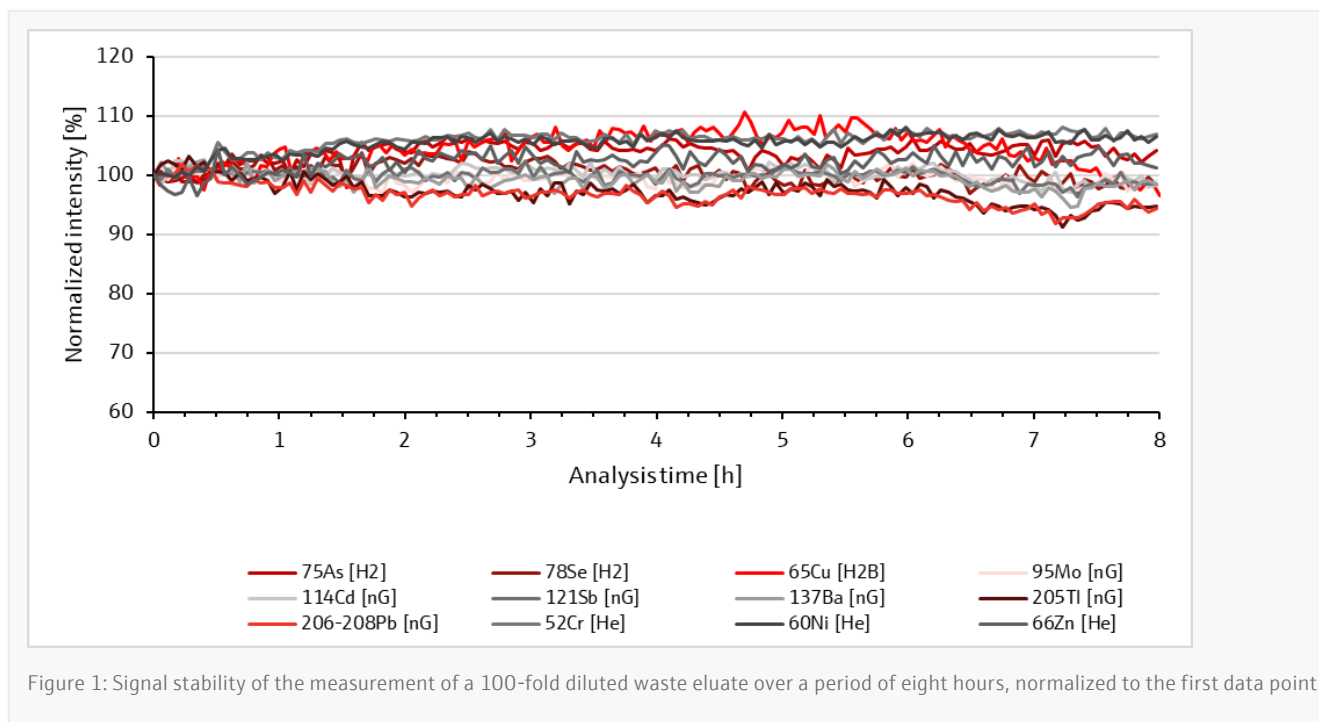


Figure 1: Signal stability of the measurement of a 100-fold diluted waste eluate over a period of eight hours, normalized to the first data point

## Conclusion

ICP-MS enables precise, sensitive, and cost-efficient routine analysis of eluates and leachates. For environmental laboratories, this is the appropriate method for routine analysis of the elutable fraction of elemental contents of waste according to DepV and DIN EN ISO 17294-2. The PlasmaQuant MS Q incorporates several patented technologies (Eco Plasma, ReflexION ion mirror, iCRC – integrated Collision Reaction Cell, pre-quadrupole, fully digital detector) which offer several advantages for the analysis of waste eluates and leachate: The high sensitivity enables the achievement of lowest detection limits over the entire mass range for reliably evaluating the lowest required assignment values. The advantage is that matrix-matched calibration can be easily replaced by sample dilution to avoid matrix effects. This contributes to high flexibility and time savings as different matrices can be analyzed in the same run without the need for separate matrix adjustment. The excellent plasma robustness enables long-term stable routine measurements and lowest operating costs due to a total Ar consumption of only 11.17 L/min (9.5 L/min plasma gas, 1.2 L/min auxiliary gas, 0.97 L/min nebulizer gas). The iCRC combines collision and reaction modes within one measurement run to remove polyatomic interferences for accurate quantification.



Figure 2: PlasmaQuant MS Q

## References

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