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Application News

Quantitative Analysis of Leaching of Heavy Metals in Soil Using ICP-MS

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User Benefits

- High-sensitivity analysis can be performed at levels below 1/100 of the soil leaching standards for heavy metals specified by the Japanese Soil Contamination Countermeasures Act.
- Interference from the matrix in soil leachates is minimal, enabling stable measurements even when analyzing multiple samples.
- The mini-torch system reduces argon gas consumption and significantly reduces running costs.

■ Introduction

When soil is polluted by human activities such as industrial operations and waste disposal, harmful substances can be absorbed into the human body. This may have an adverse effect on health. In Japan, the Soil Contamination Countermeasures Act stipulates soil survey methods and appropriate management methods to prevent adverse health effects.

The health risks due to soil contamination can be divided into two categories : those caused by leaching of harmful substances contained in soil into groundwater and ingestion of groundwater containing such harmful substances (ingestion from groundwater), and those caused by direct ingestion of harmful substances contained in soil through the mouth or skin (direct ingestion). Under the Soil Contamination Countermeasures Act, soil leaching standards are set to address the risk of ingestion from groundwater, and soil content standards are set to address the risk of direct ingestion. These standards are expected to become stricter in the future in line with other environmental regulations. Thus, it is important to select a highly sensitive analytical method in advance.

In this application, the results of analyzing the concentrations of heavy metals leached from five types of soil samples using ICP-MS, which is currently recognized as the most sensitive method for elemental analysis in soil, will be introduced.

■ Samples

Soil A, Soil B, Soil C, Soil D, Soil E

■ Sample Pretreatment

The following describes the sample pretreatment method. The method for preparing the test solution for the soil leaching test is described in the appendix of Environment Agency Notification No. 46, "Environmental Standards for Soil Contamination"[1]. The analytical method for each measurement target is described in the attached table of the Environmental Agency Notification No. 18, "Determination of Measurement Methods for Soil Elution test"[2]. In this study , the additional pretreatment described in item 4 below was performed.

In the soil leaching standard, Cr is specified as Cr (VI), but in this case, the measurement was performed as total Cr.

1. Mix soil with ultrapure water at a weight-to-volume ratio of 10 %.
2. Set the shaker to approximately 200 shakes per minute and to shake horizontally for 6 hours.
3. Let the sample solution settle, centrifuge it, and then filter through a membrane filter with a pore size of 0.45 µm and a diameter of 90 mm.
4. Take 40 mL of the filtrate, add 2 mL of nitric acid, boil for 10 minutes, then dilute with pure water to 50 mL.

* For the samples to be added in the spike test, a concentration equivalent to 1/2 of the soil leaching standard was added.

■ Calibration Curve Samples

Calibration curve samples were prepared by mixing single-element standard solutions. The calibration curve samples were prepared in a concentration range from the soil leaching standard value to 1/100 of that value, taking into consideration the dilution factor of 1.25 during the pretreatment.

Calibration curve sample concentrations are shown in Table 1.

Table 1 Calibration Sample Concentrations (µg/L)

Elem.	STD1	STD2	STD3	STD4	STD5	STD6	STD7
As	0	0.08	0.16	0.32	0.8	1.6	8
B	0	8	16	32	80	160	800
Cd	0	0.024	0.048	0.096	0.24	0.48	2.4
Cr	0	0.4	0.8	1.6	4	8	40
Pb	0	0.08	0.16	0.32	0.8	1.6	8
Se	0	0.08	0.16	0.32	0.8	1.6	8
HNO_3	4 % (v/v)						

■ Internal Standard Solution

An Internal standard solution (Be 200 µg/L, Co, In, Tl 100 µg/L, Ga 500 µg/L) was prepared with 4 % (v/v) nitric acid. The internal standard solution was added using an internal standard automatic addition kit , and measurements were performed while mixing the internal standard solution with the sample at a ratio of 1:9.

■ Instrument Configurations and Analysis Conditions

Table 2 ICP-MS System Configurations

Instrument	:	ICPMS-2050
Nebulizer	:	Nebulizer DC04
Torch	:	Mini Torch
Chamber	:	Cyclone Chamber
Sampling Cone	:	Nickel
Skimmer Cone	:	Nickel
Internal Standard Elements	:	Internal Standard Automatic Addition Kit

Table 3 ICP-MS Analysis Conditions

RF Power	:		1.2 kW	
Sampling Depth	:		5 mm	
Plasma Gas Flowrate	:		9.0 L/min	
Auxiliary Gas Flowrate	:		1.1 L/min	
Carrier Gas Flowrate	:		0.85 L/min	
Dilution Gas Flowrate	:		0 L/min	
Cell Gas	:	No Gas	He	H_2
Plasma Gas Flowrate	:	-	5 mL/min	7 mL/min
Cell Voltage	:	-	-30 V	-21 V
Energy Filter	:	-	7 V	7 V

Table 4 Measurement Results - Concentration in Soil Eluate (µg/L)

Element		^{75}As	^{11}B	^{111}Cd	^{52}Cr*	^{208}Pb	^{78}Se
Gas Mode		He	No Gas	He	He	He	H_2
Internal Standard Element		^{71}Ga	^{9}Be	^{115}In	^{59}Co	^{205}Tl	^{71}Ga
Lower Limit of Detection 3σ		0.0008	0.03	0.0002	0.003	0.0004	0.001
Lower Limit of Quantification 10σ		0.003	0.09	0.0006	0.009	0.001	0.004
Soil Leaching Standard		10	1000	3	50	10	10
Samples	Soil A	0.246	23.9	0.0030	0.286	0.118	0.154
	Soil B	0.925	15.9	0.0014	0.249	0.0030	0.044
	Soil C	0.926	4.14	0.0008	0.276	0.0395	0.031
	Soil D	0.611	11.6	0.0049	0.189	0.0120	0.040
	Soil E	2.65	31.0	0.0053	0.116	0.245	0.246
Spike Samples	Spike Conc.	5	500	1.5	25	5	5
	Soil A	5.49	531	1.54	25.8	5.13	5.35
	Soil B	6.16	525	1.53	25.9	4.99	5.28
	Soil C	6.10	514	1.54	25.9	5.06	5.16
	Soil D	5.80	523	1.54	26.0	4.98	5.25
	Soil E	7.79	550	1.54	24.8	5.16	5.48
Recovery	Soil A	105 %	101 %	102 %	102 %	100 %	104 %
	Soil B	105 %	102 %	102 %	103 %	100 %	105 %
	Soil C	103 %	102 %	102 %	102 %	100 %	103 %
	Soil D	104 %	102 %	102 %	103 %	99 %	104 %
	Soil E	103 %	104 %	102 %	99 %	98 %	105 %

* In the soil elution standard, the standard value is set as Cr (VI), but the analytical value is the concentration of total Cr.

■ Quantitative Analysis

Table 4 shows the measurement results converted to soil leachate concentrations.

The lower limit of detection and lower limit of quantification were calculated from the standard deviation σ of repeated measurements of a blank sample (STD1) in the calibration curve. These values were well below the soil eluate standard, and it was confirmed that the measurement sensitivity was sufficient in the soil leaching test.

In addition, the spike recoveries of the five soil eluates were 98 to 105 %, which showed that the interference of the matrix in the soil eluate was small.

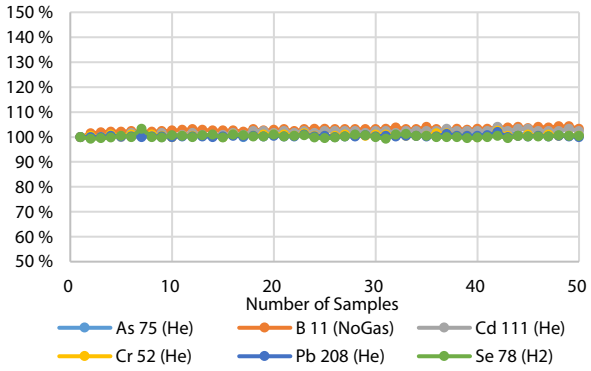

Fig. 1 Variation in Quantitative Values of Measured Elements

■ Confirmation of Long-Term Stability

The soil eluates with concentrations equivalent to 1/2 of the soil leaching standards for each element added were measured 50 times continuously, and variations in quantitative values were confirmed. The variation of the quantitative value based on the first measurement result is shown in Fig. 1. The variation of the quantitative value over 50 consecutive measurements was in the range of 99 to 104 %, indicating high reproducibility and stability.

The intensity fluctuation of the internal standard element in this long-time measurement was in the range of 89 to 105 %, and no significant variation in the measurement sensitivity was observed.

■ Conclusion

Leaching analysis of heavy metals in soil was conducted using the ICPMS-2050. The measurement sensitivity of the system was sufficient to meet the soil leaching standard, and the spike recovery test showed that the influence of the matrix on this measurement was small.

By adopting a unique mini-torch system, ICPMS-2050 can perform high-sensitivity and low-interference measurements at about 2/3 (about 11 L/min) of the argon gas flow rate of conventional ICP-MS. In addition, good results in a long-term stability test, where 50 samples were measured continuously, demonstrated its performance (high sensitivity, low interference) and cost savings, especially in the routine analysis of multiple samples.

<References>

1) Environment Agency Notification No. 46 “Environmental Standards for Soil Contamination”
2) Environment Notification No. 18 “Determination of Measurement Methods for Soil Elution Test”

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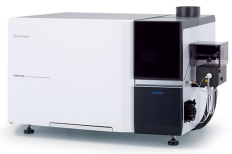

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