

Analysis of Metals Contained in Soil

■ Introduction

In Japan, the soil contamination with heavy metals etc. has been actualized and become problems recently because of the increase of the redevelopment of the vacant lots of former factory. In response to this situation, the "Soil Contamination Countermeasures Law" was proclaimed on May 29, 2002 and has been in effect since February 15, 2003. The purposes are to protect the health of citizens by comprehending the contamination levels and to prevent damage to people's health. This law identifies nine items (such as heavy metals) that may potentially accumulate in

surface soils over long periods of time at high concentrations as specific harmful substances. The law also prescribes 25 items as targets of leaching standards in the environmental quality standards for soil, which are established considering the intake of underground water.

This Application News presents analysis examples of harmful metal content in soil using AA, ICP and ICP-MS, which are widely used for analyzing inorganic elements.

■ Target Elements and Measurement Methods

	Soil Elution Standard (mg/L)	Secondary Elution Standard (mg/L)	Soil Content Level Standard (mg/kg)	Measurement Methods
Cd	<0.01	<0.3	<150	FAA, FLAA, ICP, ICPMS
Cr ⁶⁺	<0.05	<1.5	<250	UV, FAA, FLAA ICP, ICPMS
Total Hg	<0.0005	<0.005	<15	Reduction Vapor AA
Se	<0.01	<0.3	<150	Hydride Generation FAA, ICP
Pb	<0.01	<0.3	<150	FAA, FLAA, ICP, ICPMS
As	<0.01	<0.3	<150	UV, Hydride Generation FAA, ICP
B	<1	<30	<4000	UV, ICP, ICPMS

* FAA refers to flame atomic absorption spectrometry and FLAA refers to flameless atomic absorption spectrometry.

■ Sample Preparation

(1) Sample Solution Preparation Method for Soil Elution Standards

(Ministry of the Environment, Notice No. 18, March 6, 2003)

Target Substances: Alkyl mercury, mercury, cadmium, lead, hexavalent chromium, arsenic, selenium, cyanide and boron

Store the soil in a glass container or a container that will not absorb nor elute the target substances. After drying the soil in air, obtain a 50g or larger weight of sample of uniform particle size by using a 2-mm non-metallic sieve. Prepare the pH 5.8 - 6.3 for the solvent with diluted hydrochloric acid. After mixing the solvent and sample to a liquid-to-solid ratio of 10/1, perform elution for six hours at room temperature at the rate of approximately 200rpm with 4 - 5cm shaking widths. Then centrifuge for 20minutes at 3000rpm and filter the liquid through a 0.45μm membrane filter. This is the sample solution.

(2) Sample Solution Preparation Method for Soil Content Level Standards

(Ministry of the Environment, Notice No. 19, March 6, 2003)

Target Substances: Hg, Cd, Pb, Cr⁶⁺, As, Se, B and F
Store the soil in a polyethylene container or a container that will not absorb nor leach the target substances. After drying the soil in air, obtain at least 6g of sample of uniform particle size by using a 2-mm non-metallic sieve. Use 1M(mol/L) hydrochloric acid for the solvent. (For Cr⁶⁺, use a 5mM Na₂CO₃ + 10mM NaHCO₃ alkaline buffer solution). After mixing the solvent and sample to a liquid-to-solid ratio of 100/3, perform elution for two hours at room temperature at the rate of 200rpm with 4 - 5cm shaking width. Then filter the liquid through a 0.45μm membrane filter. This is the sample solution.

■ Measurement Examples

Standard soil sample JSAC0411 and SRM2711, and standard pond sediment sample NIES No. 2 were analyzed by the test methods for soil content standards. The contents of metals that are specified in the Soil Contamination Countermeasures Law for each standard sample are shown below. (Units: mg/kg)

	JSAC0411	SRM2711	NIES No.2
As	11.3±0.5	105±8	12
Cd	0.274±0.02	41.7±0.25	0.82
Cr	23.5±1.8	47	75
Pb	18.9±2.6	1162±31	105
Se	1.32±0.27	1.52±0.14	Not Included

Fig. 1 and 2 show the measurement results for Cd and Pb, and Fig. 3 shows ICP profile examples.

Cd(mg/kg)	JSAC041	SRM2711	NIES No.2
AA	0.2	30.2	0.6
ICP	0.2	36.5	0.6
ICP-MS	0.2	37.4	0.6

Fig.1 Cd Measurement Results

Pb(mg/kg)	JSAC041	SRM2711	NIES No.2
AA	10.5	1046	67.6
ICP	11.8	1070	67.5
ICP-MS	12.0	1040	69.2

Fig.2 Pb Measurement Results

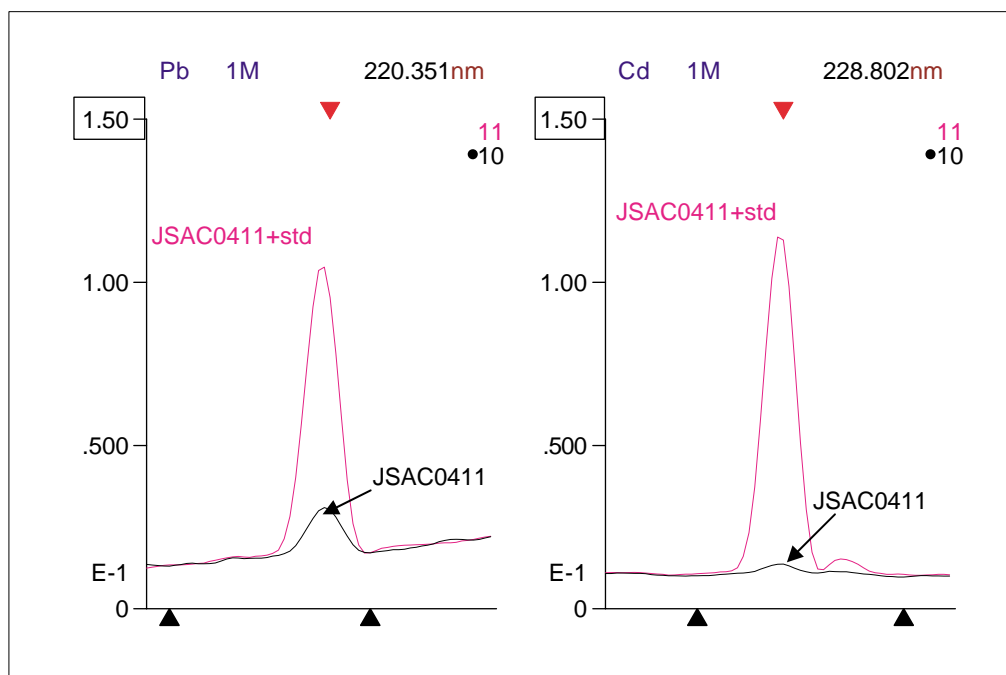


Fig.3 ICP Peak Profile

■ Conclusion

When preparing samples according to the soil content standards, 1 mol/L hydrochloric acid is used for elution, but generally speaking, samples will not completely dissolve under those conditions. Therefore, when standard substances are analyzed, the obtained content levels will normally be lower than the guaranteed values.

There are two methods to measure the contents of heavy metals in soil; measuring the contents in the soil and measuring the amounts eluted from the soil. Typically, elution amount measurements present lower values than contents measurements. Therefore, elution level analysis requires more sensitive measurement methods.



SHIMADZU CORPORATION. International Marketing Division

3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan Phone: 81(3)3219-5641 Fax: 81(3)3219-5710
Cable Add.: SHIMADZU TOKYO

Printed in Japan 3100-06401-10A-1K