

National Institute for Environmental Studies

Certificate of Analysis

NIES CRM No. 33 Landfill Cover Soil

This environmental certified reference material (CRM) was developed and certified by the National Institute for Environmental Studies (NIES) for the determination of multi-elements in soil and materials of similar matrix following extraction with 1 mol/L hydrochloric acid. The method of analysis is based on “The method of compliance test of the Ministry of Environment Japan, Notification 19 (March, 2003)”.

Certified Values (after HCl extraction)

Element	Mass fraction		Analytical method **	
	Unit	Certified value		Uncertainty
Aluminum (Al)	%	0.207	0.019	ICP-MS, ICP-MS/MS, ICP-OES
Cadmium (Cd)	mg/kg	0.240	0.039	ICP-MS, ICP-MS/MS, ICP-OES
Chromium (Cr) *	mg/kg	1.65	0.29	ICP-MS, ICP-MS/MS, ICP-OES
Cobalt (Co)	mg/kg	2.77	0.27	ICP-MS, ICP-MS/MS, ICP-OES
Copper (Cu)	mg/kg	59.3	4.2	AAS, ICP-MS, ICP-MS/MS, ICP-OES
Lead (Pb)	mg/kg	37.1	4.4	AAS, ICP-MS, ICP-MS/MS, ICP-OES
Manganese (Mn)	mg/kg	193	28	AAS, ICP-MS, ICP-MS/MS, ICP-OES
Nickel (Ni)	mg/kg	2.15	0.31	ICP-MS, ICP-MS/MS, ICP-OES
Vanadium (V)	mg/kg	4.80	0.58	ICP-MS, ICP-MS/MS, ICP-OES
Zinc (Zn)	mg/kg	85.7	8.5	AAS, ICP-MS, ICP-MS/MS, ICP-OES

All certified values were determined on an “as received” basis, without drying the material.

The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor $k = 2$, corresponding to the half-width of a confidence interval of approximately 95 %.

* The value for Cr is for total extractable chromium (not hexavalent chromium).

** AAS, atomic absorption spectrometry

ICP-MS, inductively coupled plasma-mass spectrometry

ICP-MS/MS, inductively coupled plasma-mass spectrometry/mass spectrometry

ICP-OES, inductively coupled plasma-optical emission spectrometry

Reference Values (after HCl extraction)

Element	Mass fraction		Analytical method *
	Unit	Reference value	
Iron (Fe)	%	0.18	AAS, ICP-MS, ICP-MS/MS, ICP-OES
Arsenic (As)	mg/kg	1.3	HG-AAS, HG-ICP-OES, ICP-MS/MS
Mercury (Hg)	mg/kg	0.089	CVAAS, ICP-MS/MS

All reference values were determined on an “as received” basis, without drying the material.

*AAS, atomic absorption spectrometry

CVAAS, cold vapour atomic absorption spectrometry

HG-AAS, hydride generation-atomic absorption spectrometry

HG-ICP-OES, hydride generation-inductively coupled plasma-optical emission spectrometry

ICP-MS, inductively coupled plasma-mass spectrometry

ICP-MS/MS, inductively coupled plasma-mass spectrometry/mass spectrometry

ICP-OES, inductively coupled plasma-optical emission spectrometry

Characterization

The property values of the material were determined statistically based on chemical analyses by 7 organizations (10 laboratories) using a wide range of methods. A property value satisfying the following conditions was accepted as a certified value:

- 1) the relative standard deviation associated with the mean of the laboratory means was 10 % or less,
- 2) the number of laboratories contributing to the mean of the laboratory means was at least eight, and
- 3) the number of analytical methods contributing to the mean of the laboratory means was at least three.

The uncertainty attached to the certified values is the expanded uncertainty using a coverage factor $k = 2$, corresponding to the half-width of a confidence interval of approximately 95 %. A property value failing to satisfy one or two of the NIES criteria for certification but supplying valuable additional information about the material is given as a reference value. All certified and reference values were determined on an “as received” basis, that is, the values were determined without drying the material.

Description of the Material

The CRM is supplied as fine powder in an amber glass bottle. The powder is beige in color. One unit of NIES CRM No. 33 consists of a two-bottle set, each bottle containing 30 g of landfill cover soil.

Preparation of the CRM

The CRM was prepared from natural soil. The soil was collected at a landfill site in Japan and air-dried in the sun in 1998. Thereafter this material was sieved (106 μm), homogenized by a rotary blender, bottled (30 g each, 404 bottles) in amber glass bottles and finally sterilized by ^{60}Co irradiation (21 kGy). All procedures complied with ISO Guide 34.

Homogeneity

A stratified random sampling plan was devised to test for homogeneity across the lot of bottles and the homogeneity assessment was made based on analyses of the 1 mol/L hydrochloric acid extracts, in accordance with “The method of

compliance test of the Ministry of Environment Japan, Notification 19 (March, 2003)". The between-bottle variation evaluated by a one-way analysis of variance (ANOVA) showed the relative standard deviations between bottles for the analytes to be less than 2 %. The material, therefore, was judged to be sufficiently homogeneous for its intended use as a reference material.

Instructions for Use

1. Care should be taken to avoid contamination when opening the bottles. It is desirable to use up the contents as quickly as possible after opening.
2. This CRM should be kept tightly closed in its original bottle and stored in a desiccator at room temperature (≤ 30 °C).
3. Prior to weighing portions for analysis, the contents of the bottle should be shaken gently.
4. It is recommended that a sample mass of 6 g is the minimum to be used for analysis, in accordance with "The method of compliance test of the Ministry of Environment Japan, Notification 19 (March, 2003)".
5. Precautions should be taken to avoid inhalation of the material.
6. This CRM should not be used for purposes other than research. When disposing of the material, local laws concerning processing and disposal of waste materials should be strictly adhered to.
7. The mass fractions of elements in this CRM are reported on an "as received" basis without drying. This CRM, as received, has an average moisture content of approximately 1 % as measured in NIES by drying separate sub-samples for 4 h at 105 °C.
8. In the case of measurement of As by ICP-MS, the possibility of spectral interference by Nd and Sm should be checked.

Expiry Date of Certification

The expiry date for the certified values of this CRM is January 2029 assuming that the recommended storage conditions are adhered to. NIES will notify via its website if any changes in the contents are recognized within the term of validity.
<http://www.nies.go.jp/labo/crm-e/index.html>

Collaborating Laboratories

The certified and reference values for this CRM were based on analytical values from the following seven participating organizations:

National Institute for Environmental Studies; Environmental Control Center Co., Ltd.; IDEA Consultants, Inc.; MURATA Keisokuki Service Co., Ltd.; NIPPON STEEL & SUMIKIN TECHNOLOGY Co., Ltd.; Research Institute of Environment, Agriculture and Fisheries, Osaka Prefecture; Tokyo University of Science

Technical Information

"The method of compliance test of the Ministry of Environment Japan, Notification 19 (March, 2003)" (in Japanese) is available at <https://www.env.go.jp/hourei/06/000029.html>.

Technical information and the latest reports regarding this material can be obtained from the website.

<http://www.nies.go.jp/labo/crm-e/index.html>

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Original certificate date: January 29, 2019

Certificate revision date: December 9, 2019 (The uncertainties of some certified values were reassigned.)

Certificate revision date: April 1, 2021 (Editorial changes)

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Appendix

Information values may be useful for handling this material, though the values are not certified. These values are for “total element content” and not for acid-extractable content.

Information Values for total elemental content

Element	Mass fraction		Analytical method **
	Unit	Content *	
Aluminum (Al)	%	8.3	ICP-OES, XRF
Iron (Fe)	%	2.2	ICP-MS, ICP-OES, XRF
Manganese (Mn)	%	0.037	ICP-MS, ICP-OES, XRF
Zinc (Zn)	%	0.019	ICP-MS, ICP-OES
Arsenic (As)	mg/kg	6.9	HG-AAS, HG-ICP-OES, ICP-MS/MS
Cadmium (Cd)	mg/kg	0.34	ICP-MS
Chromium (Cr)	mg/kg	21	ICP-MS, ICP-OES
Cobalt (Co)	mg/kg	6.0	ICP-MS
Copper (Cu)	mg/kg	87	ICP-MS, ICP-OES
Lead (Pb)	mg/kg	53	ICP-MS, ICP-OES
Mercury (Hg)	mg/kg	0.31	AFS, CVAAS, Thermal decomposition-AAS
Nickel (Ni)	mg/kg	8.7	ICP-MS
Vanadium (V)	mg/kg	45	ICP-MS, ICP-OES

* These values were determined based on the analytical results of three organizations.

** AFS, atomic fluorescence spectrometry

CVAAS, cold vapour atomic absorption spectrometry

HG-AAS, hydride generation-atomic absorption spectrometry

HG-ICP-OES, hydride generation-inductively coupled plasma-optical emission spectrometry

ICP-MS, inductively coupled plasma-mass spectrometry

ICP-MS/MS, inductively coupled plasma-mass spectrometry/mass spectrometry

ICP-OES, inductively coupled plasma-optical emission spectrometry

Thermal decomposition-AAS, thermal decomposition-gold amalgamation-atomic absorption spectrometry

XRF, X-ray fluorescence spectrometry