National Institute for Environmental Studies, Japan

Date

No.

National Institute for Environmental Studies

Certificate of Analysis

NIES CRM No. 31 Lake Sediment

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 lake sediment and in materials of similar matrix. This CRM supersedes NIES CRM No. 2 Pond Sediment. Certified values and reference values are given for the whole material as supplied, and for the HCl/HNO_3/$HClO_4$ extract, determined according to "The Methods for Sediment Surveillance" of the Ministry of the Environment, Japan (August, 2012).

Certified Values of NIES CRM No.31 in the whole material as supplied

Element	Mass fraction			Analytical method *
	Unit	Certified value	Uncertainty	
Aluminum (Al)	%	9.17	0.86	AAS, ICP-MS, ICP-OES, INAA, XRF
Calcium (Ca)	%	1.25	0.12	ICP-MS, ICP-OES, XRF
Iron (Fe)	%	5.38	0.55	ICP-MS, ICP-OES, INAA, XRF
Sodium (Na)	%	0.882	0.063	AAS, ICP-OES, INAA, XRF
Titanium (Ti)	%	0.442	0.044	ICP-MS, ICP-OES, XRF
Chromium (Cr)	mg/kg	43.3	3.0	ICP-MS, ICP-OES, INAA, XRF
Copper (Cu)	mg/kg	53.1	4.6	ICP-MS, ICP-OES, INAA, XRF
Lead (Pb)	mg/kg	25.1	2.5	ICP-MS, ICP-OES
Manganese (Mn)	mg/kg	978	92	ICP-MS, ICP-OES, INAA, XRF
Nickel (Ni)	mg/kg	25.3	2.8	ICP-MS, ICP-OES
Phosphorus (P)	mg/kg	925	60	AAS, ICP-OES, XRF
Vanadium (V)	mg/kg	154	11	ICP-MS, ICP-OES, INAA
Zinc (Zn)	mg/kg	121	13	ICP-MS, ICP-OES, INAA, XRF

* AAS, atomic absorption spectroscopy
ICP-OES, inductively coupled plasma-optical emission spectrometry
ICP-MS, inductively coupled plasma-mass spectrometry
ID-ICP-MS, isotope dilution-inductively coupled plasma-mass spectrometry
INAA, instrumental neutron activation analysis

XRF, X-ray fluorescence spectroscopy

Reference Values of NIES CRM No.31 in the whole material as supplied

Element	Mass fraction			Analytical method *
	Unit	Reference value	Uncertainty	
Magnesium (Mg)	%	0.836	0.068	AAS, ICP-MS, ICP-OES, INAA, XRF
Potassium (K)	%	0.991	0.134	AAS, ICP-MS, ICP-OES, INAA, XRF
Arsenic (As)	mg/kg	13.9	1.5	ICP-MS, INAA
Barium (Ba)	mg/kg	338	49	ICP-MS, ICP-OES, INAA, XRF
Cadmium (Cd)	mg/kg	0.342	0.043	ICP-MS, ID-ICP-MS
Cobalt (Co)	mg/kg	18.1	1.9	ICP-MS, ICP-OES, INAA
Lanthanum (La)	mg/kg	20.4	2.2	ICP-MS, ICP-OES, INAA
Scandium (Sc)	mg/kg	19.1	3.4	ICP-MS, ICP-OES, INAA
Strontium (Sr)	mg/kg	125	14	ICP-MS, ICP-OES, XRF

* AAS, atomic absorption spectroscopy
ICP-OES, inductively coupled plasma-optical emission spectrometry
ICP-MS, inductively coupled plasma-mass spectrometry
ID-ICP-MS, isotope dilution-inductively coupled plasma-mass spectrometry
INAA, instrumental neutron activation analysis
XRF, X-ray fluorescence spectroscopy

Certified Values of NIES CRM No.31 in the $HCl/HNO_3/HClO_4$ extract

Element	Mass fraction			Analytical method *
	Unit	Certified value	Uncertainty	
Copper (Cu)	mg/kg	50.6	8.1	ICP-MS, ICP-OES
Lead (Pb)	mg/kg	22.0	3.0	ICP-MS, ICP-OES
Manganese (Mn)	mg/kg	881	85	AAS, ICP-MS, ICP-OES
Nickel (Ni)	mg/kg	22.2	2.3	ICP-MS, ICP-OES
Vanadium (V)	mg/kg	133	23	ICP-MS, ICP-OES
Zinc (Zn)	mg/kg	110	13	AAS, ICP-MS, ICP-OES

* AAS, atomic absorption spectroscopy
ICP-OES, inductively coupled plasma-optical emission spectrometry
ICP-MS, inductively coupled plasma-mass spectrometry

Reference Values of NIES CRM No.31 in the $HCl/HNO_3/HClO_4$ extract

Element	Mass fraction			Analytical method *
	Unit	Reference value	Uncertainty	
Cadmium (Cd)	mg/kg	0.285	0.067	ICP-MS, ICP-OES
Chromium (Cr)	mg/kg	33.7	6.1	ICP-MS, ICP-OES

* ICP-OES, inductively coupled plasma-optical emission spectrometry
ICP-MS, inductively coupled plasma-mass spectrometry

Characterization

The property values of the material were statistically determined based on chemical analyses by 13 organizations (including 20 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 ten, and
3) the number of analytical methods contributing to the mean of the laboratory means was at least two.

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 based on dry mass.

Description of the Material

The CRM is supplied as fine powder in an amber glass bottle. It is dark grayish-yellow in color.

Preparation of the CRM

The origin of this material is sediment collected at Lake Kasumigaura, Japan in February 1999. The raw sediment was dried in the sun at NIES and the clods that formed were crushed, sieved through a 2 mm sieve, and dried at 105 ºC overnight. This dry material was then ground in an alumina ball mill, and passed first through a 212 µm sieve and then through a 75 µm sieve. The sieved material was homogenized with a V-blender to yield 12.2 kg of powder suitable for use as the CRM. The final material was placed in amber glass bottles (20 g in each bottle, 560 bottles), and sterilized by ^{60}Co irradiation (20 kGy). All procedures complied with ISO Guide 34.

Homogeneity

Mass fractions of multi-elements, including those for which certified values are given, were determined by XRF in material taken from 10 bottles selected from the total 560 bottles by stratified random sampling. 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 1 %. The material, therefore, is 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 ºC).
3. Prior to weighing portions for analysis, the contents of the bottle should be shaken gently.
4. It is recommended that a sample intake of 0.05 g is the minimum for convenient handling.
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 a dry mass basis. This CRM, as received, has less than 1 % water as measured in NIES by drying for 4 h at 105 ºC. Correction to dry mass should be determined by drying a separate sub-sample.

Expiry Date of Certification

The expiry date for the certified values of this CRM is December 2034 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.

Collaborating Laboratories in Analysis

The certified and reference values for this CRM were based on analytical values from the following 13 participating organizations:

National Institute for Environmental Studies; China Institute of Atomic Energy; Elemental Analysis, Inc.; Environmental Control Center Co., Ltd.; Environmental Research Center Co., Ltd.; IDEA Consultants, Inc.; Japan Environment Sanitation Center; Japan Food Research Laboratories; Kanagawa Industrial Technology Center; Murata Keisokuki Service Co., Ltd.; Naitoh Environmental Science Co., Ltd.; Nippon Steel & Sumikin Technology Co., Ltd.; Shimadzu Techno-Research Inc.

Technical Information

Technical information and the latest research reports regarding this material can be obtained from the website.
http://www.nies.go.jp/labo/crm-e/index.html

December 24, 2014
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Original certificate date: December 24, 2014
Certificate revision date: April 1, 2021 (Editorial changes)
Certificate revision date: December 23, 2024 (Update of expiry date)
Certificate revision date: June 2, 2025 (Addition of Hg isotopes)

Appendix

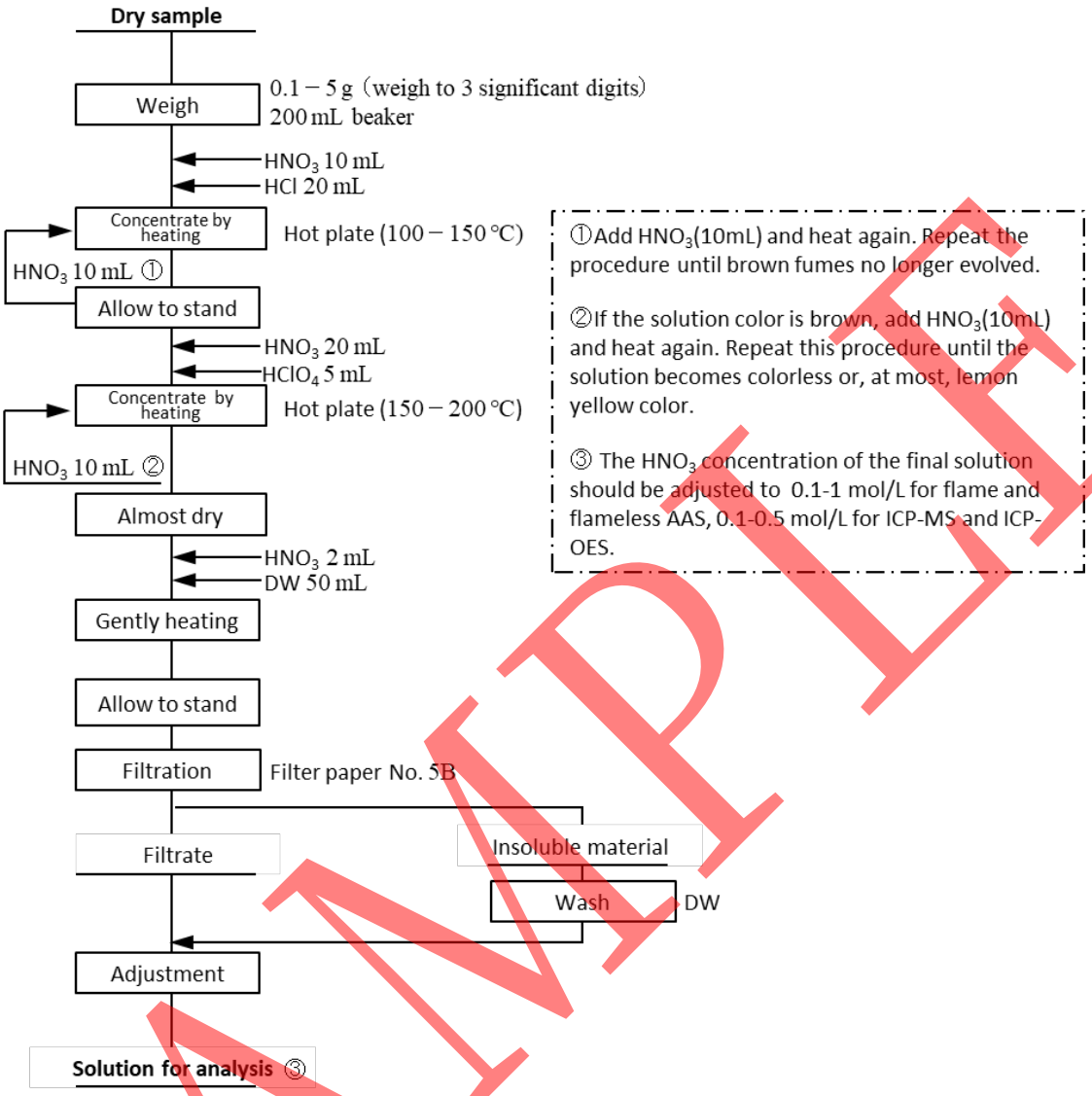

Fig. A1: Schematic flow-chart of the HCl/HNO_3/$HClO_4$ extraction method according to "The Methods for Sediment Surveillance" (in Japanese) of the Ministry of the Environment, Japan (public notice, August 2012).

Table A1: Information values of Mass fractions of C, N, Si and S in NIES CRM No.31 Lake Sediment

Element	Mass fraction		Analytical Method *
	Unit	Content	
Carbon (C)	%	3.22	EA
Nitrogen (N)	%	0.373	EA
Silicon (Si)	%	25.1	Gravimetry, XRF
Sulfur (S)	%	0.391	ICP-OES

* EA, elemental analysis
XRF, X-ray fluorescence spectroscopy
ICP-OES, inductively coupled plasma-optical emission spectrometry

Isotopic composition of Hg in NIES CRM No. 31 Lake Sediment

The isotopic composition of Hg in NIES CRM No. 31 Lake Sediment was measured using a multi-collector inductively coupled plasma mass spectrometer (MC-ICP-MS; Nu Plasma II, Nu Instruments, UK) at NIES (Table A2). A Hot Dry Bath (HDB-2N, AS ONE Corp., Japan) and double-furnace combustion system (ARF-16K, Asahi-Rika, Japan) were employed as pretreatment methods for isotope analysis. It was confirmed that there was no difference in the Hg isotopic composition of this CRM among the different types of decomposition methods. This CRM can be used to confirm analytical results and for the precision management of analytical data.

Table A2: Hg isotopic composition of NIES CRM No. 31 Lake Sediment

	δ^{199}Hg	δ^{200}Hg	δ^{201}Hg	δ^{202}Hg	Δ^{199}Hg	Δ^{200}Hg	Δ^{201}Hg
(n=35)	‰	‰	‰	‰	‰	‰	‰
Mean	−0.18	−0.43	−0.64	−0.86	0.03	0.01	0.00
2SD	0.11	0.12	0.19	0.20	0.08	0.05	0.07

＜Supplemental Information＞

Isotopic compositions are reported in the delta (δ) notation relative to NIST SRM 3133:

$\delta^{***}Hg\ (‰) = ([(^{***}Hg/^{198}Hg)_{sample}/(^{***}Hg/^{198}Hg)_{NIST\ SRM\ 3133}] - 1) \times 1000$

(*** : mass of the Hg isotopes: 199, 200, 201, 202)

Mass-independent fractionation (MIF) is reported in capital delta (Δ) notation as the difference between the measured and the theoretical δ^{***}Hg value:

$\Delta^{***}Hg\ (‰) = \delta^{***}Hg - (\beta \times \delta^{202}Hg)$,

(β : the kinetic or equilibrium fractionation factor appropriate for the particular isotope: $\delta^{199}Hg/\delta^{202}Hg = 0.252$, $\delta^{200}Hg/\delta^{202}Hg = 0.502$, $\delta^{201}Hg/\delta^{202}Hg = 0.752$ (Bergquist and Blum, 2007)).

＜References＞

B. A. Bergquist, J. D. Blum : Science, 318, 417(2007).

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