

Date ______No.

National Institute for Environmental Studies Certificate of Analysis NIES CRM No. 27 Typical Japanese Diet

This certified reference material (CRM) is intended for use in quality assurance of analyses of minor and trace elements in the Japanese diet and in similar food matrices. One unit of this CRM consists of approximately 18 g of the material in an amber glass bottle. It was produced by the National Institute for Environmental Studies (NIES), Ibaraki, Japan, and the National Institute of Radiological Sciences (NIRS), Chiba, Japan, and is distributed by NIES.

Certified Values				
	Mass fraction			
Element	Unit	Certified value	Uncertainty	Analytical method *
Calcium (Ca)	%	0.125	0.004	AAS, ICP-MS, ICP-OES, ID-ICP-MS, INAA, PIXE
Potassium (K)	%	0.550	0.015	AAS, FES, ICP-MS, ICP-OES, INAA, PGA, PIXE
Sodium (Na)	%	1.00	0.04	AAS, FES, ICP-MS, ICP-OES, INAA, PGA
Arsenic (As)	mg/kg	0.60	0.04	AAS, ICP-MS, INAA
Barium (Ba)	mg/kg	1.1	0.1	ICP-MS, ICP-OES, INAA
Cadmium (Cd)	mg/kg	0.069	0.009	AAS, ICP-MS, ID-ICP-MS, INAA
Copper (Cu)	mg/kg	2.8	0.1	AAS, ICP-MS, ICP-OES, ID-ICP-MS, PIXE
Magnesium (Mg)	mg/kg	576	12	AAS, ICP-MS, ICP-OES
Manganese (Mn)	mg/kg	8.9	0.2	AAS, ICP-MS, ICP-OES, INAA, PIXE
Selenium (Se)	mg/kg	0.25	0.02	AAS, ID-GC-MS, INAA
Strontium (Sr)	mg/kg	4.9	0.2	AAS, ICP-MS, ICP-OES, ID-ICP-MS, INAA, RNAA
Tin (Sn)	mg/kg	1.6	0.1	AAS, ICP-MS, ID-ICP-MS, INAA
Zinc (Zn)	mg/kg	20.9	0.9	AAS, ICP-MS, ICP-OES, ID-ICP-MS, INAA
Uranium (U)	mg/kg	0.0029	0.0004	ICP-MS, INAA, RNAA

All certified values were determined based on dry mass. 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 %.

* AAS, atomic absorption spectrometry

FES, flame emission spectrometry

ICP-MS, inductively coupled plasma-mass spectrometry

ICP-OES, inductively coupled plasma-optical emission spectrometry

ID-GC-MS, isotope dilution-gas chromatography-mass spectrometry

ID-ICP-MS, isotope dilution-inductively coupled plasma-mass spectrometry

INAA, instrumental neutron activation analysis

RNAA, radiochemical neutron activation analysis

PGA, prompt gamma ray analysis

PIXE, particle induced X-ray emission spectrometry

Reference Values

Element —	Mass fraction		
Element —	Unit	Reference value	
Chlorine (Cl)	%	1.5	
Phosphorus (P)	%	0.26	
Bromine (Br)	mg/kg	24	
Cesium (Cs)	mg/kg	0.020	
Cobalt (Co)	mg/kg	0.022	
Iodine (I)	mg/kg	1.9	
Iron (Fe)	mg/kg	18	
Lead (Pb)	mg/kg	0.62	
Molybdenum (Mo)	mg/kg	0.43	
Nickel (Ni)	mg/kg	0.39	
Rubidium (Rb)	mg/kg	4.7	
Thorium (Th)	mg/kg	0.002	

All reference values were determined based on dry mass.

Preparation of the CRM

The starting material for this CRM was a composite stock of menus served in 29 households in Japan. The households were asked to duplicate all meals for 3 consecutive days in November 1997-January 1998 and for another 3 days in March-June 1998. The meals were typical of Japanese households and consisted primarily of traditional Japanese foodstuffs with some Western dishes. The meals were frozen in the households and transported frozen to NIES. The food materials were then homogenized, freeze-dried, pulverized to pass a 250-µm nylon screen, blended and bottled. These procedures were compliant with ISO Guide 34. Then the material was sterilized by ⁶⁰Co irradiation (25 kGy).

Homogeneity

The homogeneity of this CRM was assessed at NIES by measuring, by inductively coupled plasma optical emission spectrometry, the concentrations of Na, Mg, P, K, Ca, Mn, Fe and Zn in four 500-mg-subsamples from each of 6 randomly selected bottles after HNO₃/HClO₄/HF digestion. No significant between-bottle variation was detected for these elements. However, a small but significant within-bottle variation was found for Fe, which was excluded from certification.

Certified and Reference Values

Certified Values were determined for Na, Mg, K, Ca, Mn, Cu, Zn, As, Se, Sr, Cd, Sn, Ba and U based on a collaborative analysis involving NIES, NIRS and 20 other laboratories. Means of the acceptable mean values from the collaborating laboratories were assigned as Certified Values (with their 95 % confidence intervals representing the uncertainty ranges) only when they were from 3 independent analytical methods based on different analytical principles, pursuant to ISO Guide 35.

Reference Values, rather than Certified Values, are reported when the collaborative analytical results were consistent among the laboratories but when within-bottle inhomogeneity was detected (Fe), when contamination during the preparation was suspected (Pb), or when the values resulted from only 2 independent analytical methods. For the above reasons, the Reference Values may include bias. Certified and Reference Values are expressed on a dry weight basis. Analytical values should be corrected for the measured moisture content to relate to the Certified and Reference Values. See below for the drying procedure.

Instructions for Use

Storage

The bottle should be tightly capped and stored at 4 °C or below. Avoid excess exposure to light and UV.

Use

Allow the bottle to come to room temperature and gently shake it before opening to ensure homogeneity. A minimum sample weight of 500 mg is recommended for analysis.

Drying

Approximately 1 g of the material is to be accurately weighed into a dry glass or metal vessel of known weight. The vessel with the material should then stand under vacuum below room temperature for 24 h; the resulting weight loss is to be assigned as moisture content. The moisture content of the material in freshly opened bottles, measured at NIES by this method, was 0.6 %. However, moisture content of the material may vary with storage conditions. All analytical values should be corrected for moisture content measured immediately prior to analysis. Drying at a higher temperature (e.g., oven drying) will result in a greater weight loss, probably due to evaporation of volatile constituents, and thus should be avoided. The subsample used to determine the moisture content should not be used for element analysis.

Warning

This CRM is for laboratory use only and is not for human consumption.

Expiry Date of Certification

The certification of this CRM is valid until July 2025, provided that storage is under the appropriate conditions specified above.

Collaborating Laboratories

The certified values for this CRM (No. 27) have been determined with substantial contributions of the following participating laboratories:

University of Massachusetts; Bhabha Atomic Research Centre; International Atomic Energy Agency, Shimadzu Co.; Japan Radioisotope Association; Musashi Institute of Technology; Hokkaido Institute of Environmental Studies; Seiko Instruments Inc.; National Institute of Radiological Sciences; Chiba Institute of Technology; Kagoshima University; Iwate Medical College; National Food Research Institute; Kyoto University; Konan University; Tokyo Metropolitan Industrial Technology Research Institute; Beltsville Human Nutrition Research Center; Tohoku National Agricultural Experiment Station; Japan Atomic Energy Research Institute; National Institute for Environmental Studies

Technical Information

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

> July 2005 Makoto M. Watanabe

Director

Laboratory of Intellectual Fundamentals for Environmental Studies National Institute for Environmental Studies

Health and Environmental Risk Division, National Institute for Environmental Studies, 16-2 Onogawa, Tsukuba, Ibaraki 305-8506 Japan FAX: +81-29-850-2900, Email: nies.crm@nies.go.jp

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