

# National Institute for Environmental Studies

## Certificate of Analysis

### Certified Reference Material 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.

#### *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- $\mu\text{m}$  nylon screen, blended and bottled. These procedures were compliant with ISO GUIDE 34. Then the material was sterilized by  $^{60}\text{Co}$  irradiation (2.5 Mrad).

#### *Homogeneity*

The homogeneity of this CRM was assessed at NIES by measuring, by inductively coupled plasma atomic 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  $\text{HNO}_3/\text{HClO}_4/\text{HF}$  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: the resulting Certified Values are listed in Table 1. 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.

Table 2 lists Reference Values for this CRM. 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.

## ***Expiration 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

## ***Reference***

Yoshinaga J., Morita M., Yukawa M., Shiraishi K. and Kawamura H.:  
Certified Reference Material for Analytical Quality Assurance of Minor and Trace Elements in Food and Related Matrixes Based on a Typical Japanese Diet: Interlaboratory Study  
Journal of AOAC INTERNATIONAL, 84(4), 1202-1208(2001)

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Table 1. Certified values for NIES/NIRS Typical Japanese Diet CRM

	Unit	Certified Value <sup>1,2</sup>	Analytical method <sup>3</sup>
Ca	%	0.125±0.004	AAS ICP ICPMS IDICPMS INAA PIXE
K	%	0.550±0.015	AAS FES ICP ICPMS INAA PGA PIXE
Na	%	1.00±0.04	AAS FES ICP ICPMS INAA PGA
As	mg/kg	0.60±0.04	AAS ICPMS INAA
Ba	mg/kg	1.1±0.1	ICP ICPMS INAA
Cd	mg/kg	0.069±0.009	AAS ICPMS IDICPMS INAA
Cu	mg/kg	2.8±0.1	AAS ICP ICPMS IDICPMS PIXE
Mg	mg/kg	576±12	AAS ICP ICPMS
Mn	mg/kg	8.9±0.2	AAS ICP ICPMS INAA PIXE
Se	mg/kg	0.25±0.02	AAS INAA IDGCMS
Sn	mg/kg	1.6±0.1	AAS ICPMS IDICPMS INAA
Sr	mg/kg	4.9±0.2	AAS ICP ICPMS IDICPMS INAA RNAA
Zn	mg/kg	20.9±0.9	AAS ICP ICPMS IDICPMS INAA
U	mg/kg	0.0029±0.0004	ICPMS INAA RNAA

1. Expressed on a dry weight basis. Analytical values should be corrected for the moisture content. See text for the method for moisture content measurement.
2. Certified Values are the means of the acceptable mean values obtained in the collaborative analysis. Uncertainty is represented by the 95% confidence interval of the mean. The minimum sample on which the Certified Values are based was 500 mg.
3. Abbreviations: AAS, atomic absorption spectrometry; FES, flame emission spectrometry; ICP, inductively coupled plasma atomic emission spectrometry; ICPMS, ICP mass spectrometry; IDICPMS, isotope dilution ICPMS; INAA, instrumental neutron activation analysis; RNAA, radiochemical neutron activation analysis; PGA, prompt gamma ray analysis; PIXE, particle induced X-ray emission spectrometry; IDGCMS, isotope dilution gas chromatography-mass spectrometry.

Table 2. Reference values for NIES/NIRS Typical Japanese Diet CRM

	Unit	Reference Value <sup>1,2</sup>		Unit	Reference Value <sup>1,2</sup>
Br	mg/kg	24	Mo	mg/kg	0.43
Cl	%	1.5	Ni	mg/kg	0.39
Co	mg/kg	0.022	P	%	0.26
Cs	mg/kg	0.020	Pb	mg/kg	0.62
Fe	mg/kg	18	Rb	mg/kg	4.7
I	mg/kg	1.9	Th	mg/kg	0.002

1. Expressed on a dry weight basis. Analytical values should be corrected for the moisture content. See text for the method for moisture content measurement.
2. Recommended minimum sample weight is 500 mg.