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NIES Certified Reference Material No. 10 " Rice Flour - Unpolished"

The National Institute for Environmental Studies (NIES) announces the availability of NIES Certified Reference Material No. 10, " Rice Flour - Unpolished". It consists of three samples, each containing different levels of cadmium.

NIES No. 10 "Rice Flour - Unpolished "

No. 10-a	Low	Level	0.023 ± 0.003 μg/g Cd
No. 10-b	Medium	Level	0.32 ± 0.02 μg/g Cd
No. 10-c	High	Level	1.82 ± 0.06 μg/g Cd

Environmental pollution by Cd has become one of the major health issues or problems in Japan. For example, "Itai-Itai " (Ouch-Ouch) disease was caused mainly by consumptiom of rice and drinking water which was contaminated with Cd derived from a river basin. On the basis of investigations on Cd levels in rice, which is the main Cd source for the Japanese, the following legal critical values have been set for Cd in rice: "have been set " implies that considerations other than scientific aspects have been taken into account.

(1) The surveillance level of Cd in unpolished rice is 0.4 μg/g.
(2) The maximum permissible level of Cd in unpolished rice is 1.0 μg/g.

It has therefore been required to accurately determine a range of concentrations of Cd and other heavy metals in unpolished rice and NIES has recently undertaken the development of "Rice Flour - Unpolished " for use primaily in the validation of analytical methods and in the calibration of analytical instruments covering 3 orders of magnitude of Cd levels.

The above reference materials were each prepared in an identical manner from unpolished rice collected from three different locations in Japan. The unpolished rice was pulverized with a rotor speed-mill, sieved to pass a 0.5 mm screen, blended and bottled. The Rice Flour-Unpolished reference material is available in a set of three bottles (60 g each) from NIES.

Certified values are provided for Ca, Cd, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Rb and Zn, while reference values are reported for Al, As, Br, Cl, Co, Cr, Hg, Se and Sr. The elemental compositions (except Cd) in the three unpolished rice flour samples are very similar and are considered typical of Japanese unpolished rice.

Preparation of Material

This reference material was prepared from unpolished rice collected from three different locations in Japan.

	Cd Level	Variety		Location
No. 10-a	Low	Koshihikari	60 kg	Tsukuba, Ibaraki Prefecture
No. 10-b	Medium	Koshihikari	60 kg	Naruto, Chiba Prefecture
No. 10-c	High+	Toyonishiki	60 kg	Jumonji, Akita Prefecture

+ Produced in a Cd-contaminated paddy field

The following procedure was used for the preparation of each of the three unpolished rice flour samples. A batch of unpolished rice (about 1.5 kg) was pulverized with a rotor speed-mill(20,000rpm, Fritsch Pulverisette 14), sieved to pass a 50 - mesh screen and dried at 80℃ for 4 hr in an air-oven. After repeating this procedure for the remaining batches, the unpolished rice flour (60 kg) was mixed in a V-blender (150 l) for 2 hr. The homogenized powder was packaged into 1,000 acid-washed glass bottles (60 g, each).

Homogeneity Assessment

In order to determine the homogeneity of the Rice Flour-Unpolished reference material, the variation of elemental content in several bottles was determined by atomic absorption analysis after acid dissolution of the samples. For each of the three reference materials, five bottles were randomly selected from the lot of 1,000 bottles and five aliquots (approximately 400 mg dry mass) were taken from each bottle (total 25 samples).

The homogeneity of this reference material was determined using one-way analysis of variance. Variations due to sample variability were calculated to be less than 1 % for Cd, Mn, Zn, Fe and Cu, indicating that the prepared Rice Flour Unpolished satisfies the homogeneity criteria for a reference material.

Instruction for Drying

The material should be dried in an air-oven at 85 ℃ for 4hr before use. The mean moisture loss will be about 5 %. For the determination of volatile elements such as Hg and Se, drying should be done on samples separate from those for analysis.

Sample Size

A minimum sample weight of 400 mg of the dry material should be used.

Additional Information

①This reference material contains a small quantity of siliceous material, which is an integral part of the sample. The analytical values in Table 1 are based on analyses performed on the entire sample. Therefore, decomposition procedures should be designed to achieve complete dissolution of the material such as by the use of a mixture of nitric/perchloric/hydrofluoric acids.
②Each of the three reference materials contains about 1 μg/g of Pb, which was estimated to be less than 0.01 μg/g in the original materials, showing Pb contamination from the rotor speed-mill was significant. Although efforts were made to prevent Pb contamination, the use of the rotor-mill was necessary to pulverize the large quantity of relatively hard unpolished rice grains.

Analytical Values for NIES Certified Reference Material
No. 10 " Rice Flour - Unpolished "

Certified Values

	Low Level (Cd)	Medium Level (Cd)	High Level (Cd)
Minor Constituents		Wt. Percent †	
Phosphorus[a,c,h]	0.340 ± 0.007	0.315 ± 0.006	0.335 ± 0.008
Potassium [a,b,c,f]	0.280 ± 0.008	0.245 ± 0.010	0.275 ± 0.010
Magnesium [a,c,f]	0.134 ± 0.008	0.131 ± 0.006	0.125 ± 0.008
Trace Constituents		μg/g †	
Calcium [a,c,f]	93 ± 3	78 ± 3	95 ± 2
Manganese [a,c,f]	34.7 ± 1.8	31.5 ± 1.6	40.1 ± 2.0
Zinc[a,c,d,e,f,g]	25.2 ± 0.8	22.3 ± 0.9	23.1 ± 0.8
Iron[a,c,f,g]	12.7 ± 0.7	13.4 ± 0.9	11.4 ± 0.8
Sodium[a,b,c,f]	10.2 ± 0.3	17.8 ± 0.4	14.0 ± 0.4
Rubidium[b,f,g]	4.5 ± 0.3	3.3 ± 0.3	5.7 ± 0.3
Copper[a,c,d,e,f,g,h]	3.5 ± 0.3	3.3 ± 0.2	4.1 ± 0.3
Molybdenum[c,e,f]	0.35 ± 0.05	0.42 ± 0.05	1.6 ± 0.1
Nickel[a,c,d,g]	0.19 ± 0.03	0.39 ± 0.04	0.30 ± 0.03
Cadmium [a,c,d,e,f,h]	0.023 ± 0.003	0.32 ± 0.02	1.82 ± 0.06

Reference Values

	Low Level (Cd)	Medium Level (Cd)	High Level (Cd)
	μg/g †	μg/g †	μg/g †
Chlorine	260	310	230
Aluminium	3	2	1.5
Bromine	0.3	0.5	0.5
Strontium	0.3	0.3	0.2
Arsenic	0.17	0.11	0.15
Chromium	0.07	0.22	0.08
Selenium	0.06	0.02	0.07
Cobalt	0.02	0.02	0.007
Mercury	0.004	0.003	0.005

† On a dry weight basis (see "Instructions for Use").
Analytical techniques used: [a] atomic absorption spectrometry, [b] flame emission spectrometry, [c] inductively coupled plasma emisssion spectrometry, [d] isotope dilution mass spectrometry, thermal ionization, [e] isotope dilution mass spectrometry, ICP-MS, [f] neutron activation analysis, [g] proton induced X-ray emission analysis, [h] spectrophotometry.