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NIES Certified Reference Material No. 9 " Sargasso "

The National Institute for Environmental Studies (NIES) announces the availability of NIES Certified Reference Material No. 9 "Sargasso".

Trace element analysis of a variety of marine samples has been carried out throughout the world in the fields of marine and environmental sciences. In 1984 NIES issued Mussel certified reference material (CRM) as the NIES CRM No. 6 and this reference material, which has an elemental composition close to background levels, has been used in many domestic and overseas laboratories. Following the Mussel CRM, NIES has recently undertaken the development of a new type of biological reference material of marine origin, Sargasso.

The material was prepared from sargasso seaweed (Sargassum fulvellum) obtained from Shimoda Bay, Shizuoka Prefecture, Japan. The sargasso was freeze-dried, ground, sieved to pass a 80-mesh screen, blended and bottled. The bottles contain about 10 grams of material. This reference material contains high concentrations of alkali, alkaline earth elements and arsenic, together with low levels of heavy metals.

Certified values are provided for Ag, As, Ca, Cd, Co, Cu, Fe, K, Mg, Mn, Na, Pb, Rb, Sr, V and Zn, while reference values are reported for Al, Br, Cl, Cr, Cs, Hg, I, P, S, Sb, Sc, Se, Ti and U. The elemental composition of NIES Sargasso reference material is considered typical of marine brown algae.

Preparation of Material

This reference material was prepared from sargasso (Sargassum fulvellum) obtained from Shimoda Bay, Shizuoka Prefecture, Japan. The sargasso (about 110 kg fresh weight) was collected in late June, 1984, and transported to the laboratory. The samples were washed with deionized water and then freeze-dried in one lot (about 20 kg dry weight).

A batch (about 500 g) of the dry seaweed was ground for 1 hr in an alumina ball-mill (7 L, 95% alumina), which had been precleaned by a previous grinding of sargasso. The pluverized samples were placed on a set of screens, a 50-mesh (297 um) nylon screen (top), a 80-mesh (177 um) nylon screen (middle) and a reservoir made of polyvinylvhloride (bottom), and then vibrated mechanically for 15 min.

After repeating this procedure for the remaining batches, the powder having passed through the 80-mesh screen (about 12kg) was mixed in a V-blender for 2hr. The homogenized powder was packaged into about 900 glass bottles (13 g, each).

At this stage, a homogeneity test using several bottles indicated that the prepared material was not sufficiently homogeneous for certain elements, probably due to difference in elemental distribution in the original materials. Therefore re-grinding of the material was carried out: all samples were ground again in an agate ball-mill (planetary type) for 15 min and the powder was sieved through the screens mentioned above. The powder having passed through the 80-mesh screen was mixed in the V-blender for 2 hr. The mixed powder (about 8 kg) was packaged again into about 800 glass bottles (10 g, each).

Homogeneity Assessment

In order to determine the homogeneity of the Sargasso reference material, the variation of elemental content in several bottles were determined by inductively coupled plasma emission and atomic absorption analysis after acid dissolution of the samples. Six bottles were randomly selected from the lot of 800 bottles and five aliquots (approximately 300 mg dry mass) were taken from each bottle (total 30 samples).

The homogeneity of this reference material was determined using one-way analysis of variance. Variations due to sample variability were insignificant ($p<0.05$) and calculated to be less than 1% for K, Mg, Sr, Fe, Mn, Zn and Cu, indicating that the prepared Sargasso reference material satisfies the homogeneity criteria for a reference material.

Instruction for Drying

The material should be dried in an air-oven at 85℃ for 4 hr before use. The mean moisture loss will be about 6 %. 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

This reference material contains siliceous material, which is an integral part of the sample. The certified and reference values 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.

Analytical Values for NIES Certified Reference Material
No. 9 " Sargasso "

Certified Values

Element	Content*	Element	Content*
Major and Minor Constituents		Trace Constituents	
	Wt. Percent		μg/g
Potassium[a,b,c,f]	6.10 ± 0.20	Iron[a,c,f]	187 ± 6
Sodium [a,b,c,f]	1.70 ± 0.08	Arsenic [a,c,f]	115 ± 9
Calcium[a,c,f]	1.34 ± 0.05	Rubidium[a,b,f]	24 ± 2
Magnesium[a,c,f]	0.65 ± 0.03	Manganese [a,c,f]	21.2 ± 1.0
Strontium[a,c,f]	0.100 ± 0.003	Zinc[a,c,d,f,g]	15.6 ± 1.2
		Copper[a,c,d,g]	4.9 ± 0.2
		Lead[a,d,e]	1.35 ± 0.05
		Vanadium[a,f,g]	1.0 ± 0.1
		Silver[a,d,f]	0.31 ± 0.02
		Cadmium [a,d,g,f]	0.15 ± 0.02
		Cobalt[a,f,g]	0.12 ± 0.01

Analytical techniques used: [a] atomic absorption spectrometry, [b] flame emission spectrometry, [c] inductively coupled plasma emission spectrometry, [d] isotope dilution mass spectrometry, thermal ionization, [e] isotope dilution mass spectrometry, ICP-MS, [f] instrumental neutron activation analysis, [g] spectrophotometry

Reference Values

	Wt. Percent		μg/g
Chlorine	5.1	Uranium	0.4
Sulphur	1.2	Chromium	0.2
Phosphorus	0.26	Scandium	0.09
	μg/g	Selenium	0.05
Iodine	520	Antimony	0.04
Bromine	270	Cesium	0.04
Aluminium	215	Mercury	0.04
Titanium	9		

* On a dry weight basis (see " Instructions for Use ").

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