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NIES Certified Reference Material, Pond Sediment

The National Institute for Environmental Studies (NIES) announces the availability of NIES Certified Reference Material No.2, Pond Sediment.

Although a large number of sediment analyses have been carried out all over the world, there are many analytical problems associated with sample handling and analysis. These situations stress the importance and need for sediment CRM in environmental analysis.

The National Bureau of Standards (U.S.A.) has issued River Sediment Standard Reference Material. The International Atomic Energy Agency (IAEA) is distributing Lake Sediment reference material. Despite the efforts to prepare and certify sediment reference materials at various national and international organizations, the number of sediment CRMs has been limited so far. Considering the importance and great demand for a new type of sediment CRM in environmental analysis, NIES has recently prepared and certified Pond Sediment reference material.

The material was prepared from the sediment collected from the bottom surface of Sanshiro Pond within the grounds of the University of Tokyo. The sediment was air-dried, ground, sieved to pass a 200-mesh (71 μm) screen, blended and finally sterilized by Co-60 radiation. The bottles contain about 20 grams of material.

Certified values are provided for Al, Fe, Ca, K, Na, Zn, Cu, Pb, Cr, Ni, Co, As, Cd, while reference values are reported for Si, Ti, P, Mn, V, Sr, Rb, Sc, La, Br, Sb and Hg. The elemental composition of this reference material may be considered as a typical sediment sample found in the vicinity of a large city.

For further information on the availability of environmental reference materials write to Dr.M.Morita at the National Institute for Environmental Studies, Yatabe-machi, Tsukuba, Ibaraki, 305, Japan.

Preparation of Material

The sediment used for this reference material was collected in May 1977 from the bottom surface of Sanshiro Pond within the grounds of the University of Tokyo (about 500 kg, fresh wt). The sediment was sieved through a nylon sieve (2 mm) to remove gravel and leaves, filtered by means of suction with a Buchner funnel (30 cmϕ) to remove interstitial water and air-dried on a filter paper at room temperature for about 2 weeks. The air-dried sediment was ground for about 1 hr in an alumina ball-mill, which had previously been used for grinding a small portion of the sediment to minimize contamination.

The pulverized samples were placed on a set of the nylon sieves arranged as follows: a 50-mesh (297 μm) sieve (top), a 100-mesh (151 μm) one (second), a 200-mesh (71 μm) one (third), and a reservoir made of polyvinylchloride (bottom). The apparatus was then vibrated mechanically for 15 min.

The sediment powder (about 40 kg) that passed through a 200-mesh sieve was homogenized by passing through a riffle sampler 11 times. This homogenized sample, which constituted the reference material, was packaged in 2,000 acid-washed glass bottles (about 20 g each). Finally, the bottled samples were sterilized by Co-60 radiation (2M rad) at the Japan Atomic Energy Research Institute (Takasaki) to minimize deterioration due to biological activity.

Homogeneity Assessment

In order to estimate homogeneity of the material, the variation of elemental content in several bottles was examined by acid-dissolution followed by AAS analysis. Eleven bottles were randomly selected from the lot of 2,000 bottles and 3 aliquots (about 1 g dry wt) were taken from each bottle.

The homogeneity of Pond Sediment reference material was estimated using the analysis of variance. For the elements Zn, Cu, Pb, Ni and Co, variations due to sample variability were estimated to be less than 1 % (as relative standard deviation), indicating that the prepared Pond Sediment satisfies the homogeneity criteria for a reference material.

Certified Values

The certified values are based on results of determinations by at least three independent analytical techniques. The uncertainties of the certified values were estimated based on consideration of 2 times the standard deviation of the mean of the acceptable values, and of the 95 % confidence intervals for the mean of individual methods.

Instruction for Drying

The material should be dried in an air-oven at 110 ℃ for 4 hrs before use. The mean moisture loss was about 11 %. For the determination of volatile elements such as Hg and Se, drying should be done on samples separate to those for analysis.

Sample Size

A minimum sample weight of 300 mg of the dry material should be used.

Storage

The material should be kept tightly closed in its original bottle and stored in a desiccator at room temperature.

Additional Information

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 through the use of a mixture of nitric/perchloric/hydrofluoric acids.

Certificate For
NIES Certified Reference Material,
No. 2 "POND SEDIMENT"

Certified Values

Element	Content*
Major and Minor Constituents	(Wt. Percent)
Aluminium[c,f,h,i,j]	10.6 ± 0.5
Iron[a,c,d,f,g,h,j]	6.53 ± 0.35
Calcium[a,c,d,f,g,i,j]	0.81 ± 0.06
Potassium[a,b,c,d,f]	0.68 ± 0.06
Sodium[a,b,f,g]	0.57 ± 0.04
Trace Constituents	(μg/g)
Zinc[a,c,d,f]	343 ± 17
Copper[a,c,d,e,f]	210 ± 12
Lead[a,c,d,e,g]	105 ± 6
Chromium[a,c,f,h]	75 ± 5
Nickel[a,c,d,g]	40 ± 3
Cobalt[a,c,f,g]	27 ± 3
Arsenic[a,f,g,h]	12 ± 2
Cadmium[a,c,e]	0.82 ± 0.06

a: atomic absorption spectrometry, b: flame emission spectrometry,
c: inductively coupled plasma emission spectrometry,
d: x-ray fluorescence spectrometry, e: isotope dilution mass spectrometry,
f: neutron activation analysis, g: photon activation analysis,
h: spectrophotometry, i: gravimetry, j: volumetric analysis

Reference Values

	(Wt. Percent)
Silicon	21
Titanium	0.64
Phosphorus	0.14
	(μg/g)
Manganese	770
Vanadium	250
Strontium	110
Rubidium	42
Scandium	28
Lanthanum	17
Bromine	17
Antimony	2.0
Mercury	1.3

*Based on dry weight: The material should be dried in an air-oven at 110°C for 4 hrs (mean moisture content, approximately 11%). A minimum sample size of 300 mg of the dry material should be used.

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National Institute
for Environmental Studies
Environment Agency of Japan