Telephone: 0298-51-6111 Cable: KOGAIKENTSUKUBA

P.O. 16-2 Onogawa, Tsukuba, Ibaraki 305 Japan

October, 1980

NIES Certified Reference Material, Pepperbush

The National Institute for Environmental Studies (NIES) announces the availability of NIES Certified Reference Material No.1, Pepperbush.

Pepperbush tree (Clethra barbinervis Sieb et Zucc) has two anomalous properties which characterize this tree from other normal plant species. First, the ability of the tree to accumulate certain heavy metals in the leaves has been established. Further, it is known that pepperbush tree grows well in areas of volcanic activity and also near ore refineries, suggesting that pepperbush plant is tolerant to exposure to sulfur compounds.

The elemental composition of pepperbush leaves is significantly different from those of NBS botanical SRMs, therefore, the development of Pepperbush reference material has been undertaken.

The material was prepared from the leaves of pepperbush collected from Mikouchi in the Ashio district, Japan. The leaves were washed with deionized water, dried at 80 $^{\circ}$ C, ground, sieved to pass a 80-mesh (177 μ m) screen, blended and bottled. The bottles contain about 14g of material.

Certified values are provided for K, Ca, Mg, Mn, Zn, Fe, Ba, Na, Rb, Sr, Co, Cu, Ni, Cd, Pb, As, while reference values are reported for P, Cr, Cs, Tl and Hg. The most typical characteristics of Pepperbush relative to the botanical SRMs issued by NBS are the high concentrations of Mn, Zn, Co, Ni and Cd.

Prepartion of Material

The leaves of pepperbush used for this reference material were collected in September, 1975, at Mikouchi in the Ashio district where large amounts of the plant are readily available. The leaves, free from stems, were washed with deionized water and dried in an air oven at 80 °C overnight. About 30 kg of the dry leaves were used in this work.

The dry leaves (about 700 g) were ground for about 1 hr in a ball-mill (95 % Al₂O₃, 7 liters) which had been previously ground well with the leaves to minimize contamination. The pulverized samples were placed on a set of sieves, a 50-mesh (297 μ m) nylon sieve (top), a 80-mesh (177 μ m) nylon sieve (middle) and a reservoir made of vinylchloride (bottom), and then vibrated mechanically for 15 min.

The powder having passed through a 80-mesh sieve was divided into two parts with a riffle sampler. The powder was piled up in two layers and again divided by passing through the riffle sampler. Pepperbush samples were homogenized by repeating this procedure ten times and packaged into 1,150 glass bottles (about 14 g, each).

Homogeneity Assessment

In order to check the homogeneity of the pepperbush samples, 38 samples were taken from each of 30 bottles, dry-ashed and analyzed. Homogeneity was estimated from the relative standard deviations of Zn, Fe and Mn determinations of the pepperbush samples. The variation of the metal contents of the pepperbush samples were almost the same as the corresponding results for the standard solutions, indicating that the prepared Pepperbush samples are homogeneous and practically useful as 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 (1) in an air-oven at 85 °C for 4 hrs or (2) in a desiccator over silica gel for 10 days, before use. The mean moisture loss was about 8 %. 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.

Certificate For NIES Certified Reference Material No. 1 "PEPPERBUSH"

Certified Values	
Element	Content*
Major and Minor Constituents	(Wt. Percent)
Patassium ^{a,b,c,d,e,f}	1.51 ± 0.06
Calcium ^{a,b,c,d,e,f,g,i}	1.38 ± 0.07
Magnesium ^{a,c,e,f,g}	0.408 ± 0.020
Manganese ^{a,c,d,f,g}	0.203 ± 0.017
Trace Constituents	(μg/g)
Zinc ^{a,c,d,f,g}	340 ± 20
Iron ^{a,c,d,f,h}	205 ± 17
Barium ^{c,e,f,g}	165 ± 10
Sodium ^{a,b,c,f,g}	106 ± 13
Rubidium ^{a,b,d,e,f,g}	75 ± 4
Strontium ^{a,b,c,d,e,g}	36 ± 4
Cobalt ^{a,c,f,h}	23 ± 3
Copper ^{a,c,d,e,f}	12 + 1
Nickel ^{a,c,d,g,h}	8.7 ± 0.6
Cadmium ^{a,c,d,e,f}	6.7 ± 0.5
Lead ^a ,c,d,e	5.5 ± 0.8
Arsenic ^{a,c,d,f,g}	2.3 ± 0.3
a: atomic absorption spectromety,	b: flame emission spectrometry
e: inductively coupled plasma emission specto	rometry,
d: x-ray fluorescence spectrometry,	e: isotope dilution mass spectrometry
f: neutron activation analysis,	g: photon activation analysis
h: spectrophotometry,	i: gravimetry
Reference	Values
	(Wt. Percent)
Phosphorus	0.11
	(μg/g)
Chromium	1.3
Ces <mark>ium</mark> Thallium	1.2
Mercury	0.13 0.056

*Based on dry weight: The material should be dried, (1) in an air-oven at 85°C for 4 hrs or (2) in a desiccator over silica gel for 10 days, before use (mean moisture content, approximately 8.0 %). A minimum sample size of 300 mg of the dry material should be used.

16-2 Onogawa, Tsukuba Ibaraki, 305, Japan October, 1980

National Institute for Environmental Studies Environment Agency of Japan