

March, 1985

NIES Certified Reference Material, Human Hair

The National Institute for Environmental Studies (NIES) announces the availability of NIES Certified Reference Material No.5, Human Hair.

Trace element analysis of human hair has been carried out in laboratories throughout the world for the purpose of assessing the nutritional and toxicological status of the individual. In order to assist the validation of analytical procedures, a hair reference material certified for elemental composition has been prepared. The material was prepared from human scalp hair (Japanese male, black in colour) collected from barber shops in Tsukuba and Tokyo. The scalp hair was washed in a nonionic detergent solution in an ultrasonic cleaner, dried, ground in an agate ball-mill (planetary type), sieved through a polyethylene net, blended, bottled and finally sterilized by Co-60 radiation. The bottles contain about 2 grams of material.

Certified values are provided for Ca, Cd, Cr, Cu, Fe, Hg, K, Mg, Mn, Na, Ni, Sr and Zn, while reference values are reported for Al, Ba, Br, Cl, Co, P, Pb, Rb, Sb, Sc, Se and Ti. The elemental composition of this reference material is considered similar to "normal values" of the Japanese male population.

For further information on the availability of NIES environmental reference materials, write to Dr. M. Morita at the National Institute for Environmental Studies, 16-2 Onogawa, Tsukuba, Ibaraki, 305 Japan.

Preparation of Material

The scalp hair (about 20 kg) for this reference material was male scalp hair and was collected from three barber shops in Tsukuba and Tokyo. After removal of visible contaminants, the hair was washed by ultrasonic cleaning (20 min) in a 0.3 % nonionic detergent (polyethylene glycol mono-lauryl ether) solution, and then the detergent was removed by copious rinse with distilled water. The cleaned hair was dried in an air-oven at 80 °C overnight.

For pulverizing the hair sample, several apparatus were tested from the viewpoints of grinding efficiency and contamination, since hair is a most difficult material to be pulverized. As a result, an agate ball-mill (planetary type) was selected as technically the best method, though contamination with small portion of agate debris was unavoidable. About 60 g of the hair was ground initially in the agate ball-mill (250 ml x 4) and this procedure was repeated for about 2 kg of the cleaned hair. The pulverized powder was sieved through a polyethylene net to remove undestroyed fibrous hair. The sieved powder was mixed in a V-blender (150 L) for 2 hrs and packaged into acid-washed vials (1100 vials, 2 g each). The bottles were sterilized by Co-60 radiation (2 M rad) at the Japan Atomic Energy Research Institute (Takasaki).

Homogeneity Assessment

In order to estimate homogeneity of the material, the variation of elemental content in several bottles was examined by acid-digestion followed by inductively coupled plasma atomic emission spectrometry and atomic absorption spectrometry analysis. Five bottles were randomly selected from the lot of 1,100 bottles and 5 aliquots (about 200 mg dry weight) were taken from each bottle (total 25 samples).

The homogeneity of the hair reference material was estimated using the analysis of variance. For the elements Ca, Fe, Mg, Zn, Na, Cu, Mn and Sr, variations due to sample variability were estimated to be less than 1 % (as relative standard deviation), indicating that the prepared Hair 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.

Sample Size

A minimum sample weight of 200 mg of the dry material should be used. A homogeneity test varying the sample size from 20 mg to 200 mg showed the best homogeneity for the 200 mg sample.

The material readily adheres to the wall of glass and Teflon vessels by electrostatic attraction and care should be taken in transfer operations.

Instruction for Drying

The material should be dried in an air-oven at 85 °C for 4 hrs before use. The moisture loss should be determined for each bottle (ranging from 6 % to 9 %). For the determination of volatile elements such as Hg and Se, drying should be done on samples separate to those for analysis.

Storage

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

Additional Information

This reference material contains debris of an agate ball-mill, namely SiO₂, used for pulverization of the material. 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.

It is recommended to remove from the sample being analysed fibrous hair which, occasionally, may be present in the vial.

Analytical Values
NIES Reference Material No.5
"Human HAIR"

Certified Values	
Element	Content* ($\mu\text{g/g}$)
Calcium ^{a,c,d,e,f}	728 \pm 30
Iron ^{a,c,e,f}	225 \pm 9
Magnesium ^{a,c,e}	208 \pm 10
Zinc ^{a,c,d,e}	169 \pm 10
Potassium ^{a,b,c,d,e}	34 \pm 3
Sodium ^{a,b,c,e}	26 \pm 1
Copper ^{a,c,d,e}	16.3 \pm 1.2
Manganese ^{a,c,e}	5.2 \pm 0.3
Mercury ^{a,e,g,h}	4.4 \pm 0.4
Strontium ^{c,d,e}	2.3 \pm 0.2
Nickel ^{a,d,e}	1.8 \pm 0.1
Chromium ^{a,c,e}	1.4 \pm 0.2
Cadmium ^{a,d,e}	0.20 \pm 0.03

a: atomic absorption spectrometry, b: flame emission spectrometry,
 c: inductively coupled plasma emission spectrometry,
 d: isotope dilution mass spectrometry, thermal ionization,
 e: neutron activation analysis, f: spectrophotometry,
 g: atomic fluorescence spectrometry,
 h: microwave induced plasma emission spectrometry,

Reference Values			
Content* ($\mu\text{g/g}$)			
Chlorine	250	Barium	3.2
Aluminum	240	Selenium	1.4
Phosphorus	165	Rubidium	0.19
Bromine	90	Cobalt	0.10
Titanium	22	Antimony	0.07
Lead	6.0	Scandium	0.05

*Based on dry weight; The material should be dried in an air-oven at 85°C for 4 hrs before use (moisture content 6% - 9%). A minimum sample size of 200 mg of the dry material should be used.

16-2 Onogawa, Tsukuba,
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National Institute
for Environmental Studies
Environment Agency of Japan