



National Bureau of Standards

Certificate of Analysis

Standard Reference Material 97b

Flint Clay

This Standard Reference Material (SRM) is intended for use in the determination of constituent elements in clay or material of similar matrix. SRM 97b is powdered clay that was air-dried, ball-milled, and blended to ensure homogeneity.

The certified constituent elements of SRM 97b are given below in Table 1. The certified values are based on measurements made using two or more independent reliable methods or techniques. Non-certified values for constituent elements are given in Table 2 as additional information on the composition. The non-certified values should not be used for calibration or quality control. All values are based on samples that were dried for 2 hours in a conventional oven at 140 °C and a minimum sample size of 250 mg.

Table 1

Certified Values for Constituent Elements

<u>Element</u> ¹	<u>Content, Wt. %</u> ²	<u>Element</u>	<u>Content, Wt. %</u>
Aluminum ^{c,d,g}	20.76 ± 0.15	Manganese ^{b,g}	0.0047 ± 0.0005
Calcium ^{b,d,f}	0.0249 ± 0.0026	Potassium ^{b,c,f,g,i}	0.513 ± 0.023
Chromium ^{c,g}	0.0227 ± 0.0012	Silicon ^{e,i}	19.81 ± 0.04
Iron ^{c,g}	0.831 ± 0.008	Sodium ^{b,d,g}	0.0492 ± 0.0023
Lithium ^{d,f}	0.055 ± 0.001	Strontium ^{d,f,g}	0.0084 ± 0.0002
Magnesium ^{b,c}	0.113 ± 0.002	Titanium ^{b,g,h,i}	1.43 ± 0.04

¹ Methods / Techniques

- a Colorimetry (o-phenanthroline)
- b DC Plasma Spectrometry
- c Flame Atomic Absorption Spectrometry
- d Flame Emission Spectrometry
- e Gravimetry

- f Isotope Dilution Mass Spectrometry
- g Instrumental Neutron Activation Analysis
- h Spectrophotometry
- i X-ray Fluorescence

²The certified value is a weighted mean of results from two or more analytical techniques. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel (NBS Journal of Research 87, 1982, pp. 377-385). The uncertainty is the sum, in quadrature, of the half-width of a 95% expected tolerance interval and an allowance for systematic error among the methods used. The interval whose endpoints are the certified value minus and plus the uncertainty, respectively, will cover the concentration in a minimum sample size of 250 mg of this SRM for at least 95% of the samples with 95% confidence.

Gaithersburg, MD 20899
April 21, 1988

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Table 2

Non-certified Values for Constituent Elements

<u>Element</u>	<u>Content, Wt. %</u>	<u>Element</u>	<u>Content, Wt %</u>
Barium	(0.018)	Zirconium	(0.05)
Phosphorus	(0.02)		
<u>Element</u>	<u>Content, $\mu\text{g/g}$</u>	<u>Element</u>	<u>Content, $\mu\text{g/g}$</u>
Antimony	(2.2)	Rubidium	(33)
Cesium	(3.4)	Scandium	(22)
Cobalt	(3.8)	Thorium	(36)
Europium	(0.84)	Zinc	(87)
Hafnium	(13)		
		Loss on Ignition	(13.3 wt%)

Loss on ignition was obtained by igniting sample for 2 hours at 1100 °C after sample was dried for two hours at 140 °C.

Source and Preparation

The flint clay for SRM 97b was donated to NBS by F.J. Flanagan and J.W. Hasterman of the United States Geological Survey, Reston, Virginia. Approximately 220 kg of flint clay was collected from a stockpile near the Harbison-Walker Refractories Co. Mine on Anderson Creek, Pike Township, Clearfield County, PA. The collected clay was air-dried and processed by the same method used to prepare USGS rock standards (USGS Bulletin 1582, Flanagan, 1986). After processing, the sample was delivered to NBS, where it was again mixed in a 0.3 cubic meter "V" blender for approximately 45 minutes. After blending the clay was placed in polyethylene lined aluminum pails and subsequently bottled.

Homogeneity testing was performed using x-ray fluorescence and instrumental activation analysis on randomly selected samples from cans of bulk material. There were no significant differences among samples for any of the measured elements.

Chemical analyses were performed in the following laboratories:

- National Bureau of Standards, Center for Analytical Chemistry, E.S. Beary, D.A. Becker, W.A. Bowman III, T.A. Butler, K.A. Brletic, J.W. Gramlich, D. Mo, J.R. Moody, and T.C. Rains.
- Mineral Constitution Laboratory, Pennsylvania State University, University Park, Pennsylvania, J.B. Bodkin.
- Engelhard Corporation, Specialty Chemical Division, Edison, New Jersey, B.P. Scibek.
- Construction Technology Laboratories, Inc., Skokie, Illinois, H.M. Kanare.

The statistical analysis and evaluation of the data for certification was performed by K.R. Eberhardt and S.B. Schiller of the Statistical Engineering Division and R.L. Watters, Jr. of the Inorganic Analytical Research Division.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills.