Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1871

Lead-Silicate Glasses for Microanalysis

K-456, K-493, and K-523

This Standard Reference Material (SRM) is intended primarily for the analysis of glasses, ceramics, and minerals by microanalytical techniques. It consists of three different lead-silicate glasses in rod-form approximately 2 x 2 x 20 mm, which can be divided into several specimens for microanalysis. These glasses were specifically fabricated for use in microanalytical techniques such as electron probe microanalysis (EPMA) and secondary ion mass spectrometry (SIMS).

The major constituents of these glasses show no heterogeneity of any practical significance on the micrometer scale. Lead is certified in all three glasses, while silicon is certified in glass K-456 only. For the dopant elements, the values in parentheses are electron microprobe analyses and the values in brackets are nominal values calculated from the weighed amounts of the oxides added to the melts. The oxygen values were calculated from the stoichiometry of the oxides. The values in parentheses and brackets are provided as information only and are *not certified.*

Table 1. Compositions in Weight Percent

Element	Glass		
	K-456	K-493	K-523
Pb	65.67 ± 0.26	63.28 ± 0.26	63.10 ± 0.26
Si	13.37 ± .24	(13.09 ± .24)	(12.94 ± .24)
O	(20.35)	(20.58)	(20.80)
Mg			(0.12) [0.10]
Al		(0.13) [0.11]	
P			(.24) [.25]
Ti		(.20) [.19]	(.21) [.19]
Cr			(.20) [.21]
Fe		(.25) [.22]	
Ni			(.25) [.24]
Ge			(.20) [.29]
Zr		(.38) [.36]	(.33) [.36]
Ba			(.61) [.55]
Ce		(.53) [.55]	
Eu			(.73) [.60]
Ta		(.64) [.72]*	
Th			(.08) [.10]
U			(.23) [.11]
Total	(99.38)	(99.08)	(100.19)

*Neutron activation: Ta = (0.74)

Washington, DC 20234
May 1, 1984

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Lead and silicon values were determined by wet chemical analysis and EPMA. In addition, inductively coupled plasma spectrometry was used to determine silicon in glass K-456. The certified values and the nominal values for silicon were determined from the weighted average of the two or three different methods of analysis.[1] A standard deviation of the certified value was calculated from the variances within as well as between the different analytical procedures. A pooled standard deviation was then obtained for each element by combining the standard deviations from all three glasses. The error cited is two times the pooled standard deviation of the certified value.

The dopant elements, present as oxides in concentrations of 2 percent or less in glasses K-493 and K-523, were determined with the electron microprobe, and are compared to the nominal values in brackets. These dopant elements are not certified for either composition or homogeneity. Tantalum in glass K-493 was also determined by neutron activation analysis.

The glasses were tested for microhomogeneity using periodic integrator traces and random sampling techniques. Inter-specimen homogeneity was also tested. No inhomogeneity of any practical significance was observed.

The glasses were prepared by D.H. Blackburn and D.A. Kauffman, NBS Center for Materials Research.

Quantitative wet chemical analyses were performed by J.B. Bodkin under the direction of N.H. Suhr, Pennsylvania State University, University Park, Pa.

The inductively coupled plasma spectrometry was performed by R.L. Watters, NBS Center for Analytical Chemistry.

Quantitative EPMA and homogeneity testing were performed by R.B. Marinenko, NBS Center for Analytical Chemistry. Data reduction for the quantitative analysis, was done with the NBS correction procedure, COR.[2]

Neutron activation analysis was done by G.J. Lutz, NBS Center for Analytical Chemistry.

The technical measurements leading to certification were coordinated by R.B. Marinenko under the direction of H. Rook, NBS Center for Analytical Chemistry.

Statistical consultation was provided by R.C. Paule, NBS National Measurement Laboratory.

The support aspects involved in the certification and development of this Standard Reference Material was coordinated through the Office of Standard Reference Materials by R.W. Seward.

[1]Paule, R.C. and Mandel, J., J. Res. of Nat. Bur. Stds., 87, No. 5, p. 377 (1982).
[2]Henoc, J., Heinrich, K.F.J., and Myklebust, R.L., Nat. Bur. Stds. Technical Note 769, (1973).