S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
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National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1874

Lithium-Aluminum-Borate Glasses for Microanalysis

K-495, K-490, and K-546

This Standard Reference Material (SRM) was fabricated primarily for use in the analysis of glasses, ceramics, and minerals by microanalytical techniques such as electron probe microanalysis (EPMA) and secondary ion mass spectrometry (SIMS). SRM 1874 consists of three different lithium-aluminum-borate glasses in rod-form, approximately 2 x 2 x 20 mm, which can be divided into several specimens for microanalysis.

The aluminum concentration in glass K-495 is the only value certified for SRM 1874. For lithium and boron, information values from wet chemistry analysis are in parentheses, and for aluminum in glasses K-490 and K-546, information values obtained by EPMA are in parentheses. No error limits are given for information values because they were obtained by only one method. The homogeneity for aluminum was examined by EPMA. No serious inhomogeneities were observed. For the dopant elements, the values in parentheses were obtained by microprobe analysis and the values in brackets are nominal values calculated from the weighed amounts of oxides added to the melts. The oxygen values were calculated from the stoichiometry of the oxides. The values in parentheses and brackets are provided for information only and are *not certified.*

Table 1. Compositions in Weight Percent

Element	Glass		
	K-495	K-490	K-546
Li	(2.3)	(2.2)	(2.2)
Al	10.89 ± 0.23	(10.2)	(10.1)
B	(23.0)	(21.5)	(21.6)
O	(63.49)	(60.74)	(61.36)
Mg			(0.17) [0.17]
P			(0.42) [0.43]
Ti		(0.31) [0.35]	(0.39) [0.34]
Cr			(0.14) [0.14]
Fe		(0.38) [0.38]	
Ni			(0.39) [0.41]
Ge			(0.50) [0.51]
Zr		(0.53) [0.63]	(0.52) [0.64]
Ba			(0.99) [0.96]
Ce		(1.46) [0.97]	
Eu			(1.21) [1.06]
Ta		(1.02) [1.25]	
Th			(0.16) [0.16]
U			(0.24) [0.20]
Pb		(1.47) [1.44]	
Si		(0.19) [0.20]	
Total	(99.68)	(100.01)	(100.39)

Gaithersburg, MD 20899
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Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Aluminum in K-495 and lithium and boron in all three glasses were determined by wet chemistry. EPMA was used to determine aluminum in all glasses. The certified value for aluminum in K-495 was determined based on the weighted average of the two different methods of analysis.[1] The quantitative error, ±2s, is two times the pooled standard deviation of the certified value for aluminum determined in three glasses of a related SRM (SRM 1875) plus glass K-495 in SRM 1874. One standard deviation of the certified value was calculated from the variances within, as well as between, the different analytical procedures.

The dopant elements, present as oxide constituents of 2 percent or less in glasses K-490 and K-546, were determined with the electron microprobe, and are compared to the nominal values in brackets. These dopant elements are not certified either for composition or for homogeneity.

Aluminum in all three glasses was tested for microhomogeneity using periodic integrator traces and random sampling techniques. Interspecimen homogeneity was also tested. A small inhomogeneity error for a single measurement was determined from the within and between specimen errors. This inhomogeneity error is included with the quantitative error in the uncertainty (two standard deviations) for the certified composition. The variability between the different analytical methods (the quantitative error) is the predominant contribution to the uncertainty.

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.

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 R.M. Lindstrom and 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.L. 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 were coordinated through the Office of Standard Reference Materials by R.W. Seward.

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[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. Tech. Note 769, 1973.