

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1875

Aluminum–Magnesium–Phosphate Glasses for Microanalysis

K-496, K-497, and K-1013

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 1875 consists of three different aluminum-magnesium-phosphate glasses in rod-form, approximately 2 x 2 x 20 mm, which can be divided into several specimens for microanalysis.

Aluminum, magnesium, and phosphorus are certified in all three glasses. These major constituents show no serious heterogeneity on the micrometer scale. For the dopant elements, the values in parentheses are from electron probe microanalyses and the values in brackets are nominal values calculated from the weighed amounts of oxides added to the melts. The oxygen values were calculated by difference because the stoichiometry of the phosphorus oxides in the glasses is unpredictable. The values in parentheses and brackets are provided for information only and are *not certified*.

Table 1. Compositions in Weight Percent

Element	Glass		
	K-496	K-497	K-1013
Al	6.47 ± 0.20	5.97 ± 0.22	6.08 ± 0.21
Mg	6.65 ± 0.17	6.49 ± 0.17	5.86 ± 0.26
P	32.98 ± 0.55	31.59 ± 0.58	32.26 ± 0.56
O	(53.90)*	(52.46)*	(53.05)*
Pb		(0.86) [0.92]	
Si		(0.13) [0.13]	
B		[0.05]	
Ti		(0.22) [0.21]	(0.21) [0.22]
Cr			(0.14) [0.23]
Fe		(0.26) [0.24]	
Li		[0.0005]	
Ni			(0.31) [0.26]
Ge			(0.34) [0.33]
Zr		(0.32) [0.40]	(0.45) [0.41]
Ba			(0.52) [0.61]
Ce		(0.94) [0.62]	
Eu			(0.53) [0.67]
Ta		(0.71) [0.88]**	
Th			(0.10) [0.11]
U			(0.15) [0.13]
Total	(100.00)	(100.00)	(100.00)

* Oxygen values were calculated by difference.

** Neutron activation: Ta = (0.83).

Aluminum was determined in all three glasses by EPMA and neutron activation, and in K-496 by wet chemical analysis. Phosphorus and magnesium were determined in all three glasses by EPMA and wet chemical analysis. The certified values were calculated as the weighted average of the two or three different methods of analysis.¹ The quantitative error, $\pm 2s$, for each element is two times the pooled standard deviation of the certified value for all three glasses. 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 oxides in compositions of 2 percent or less in glasses K-497 and K-1013, 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. Tantalum in glass K-497 was 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. A small inhomogeneity error for a single measurement was obtained from these tests. This error is included (with the quantitative error) in the uncertainty (two standard deviations) for each of the certified compositions. For phosphorus, the inhomogeneity error is the major contribution to the uncertainty; while for aluminum and magnesium, 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.

¹Paule, R.C. and Mandel, J., J. Res. of Nat. Bur. Stds., 87, No. 5, p. 377 (1982)

²Henoc, J. Heinrich, K.F.J., and Myklebust, R.L., Nat. Bur. Stds. Tech. Note 769, 1973.