



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1872

Lead-Germanate Glasses for Microanalysis
K -453, K-491, and K -968

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-germanate glasses in rod-form approximately 2 mm × 2 mm × 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).

Expiration of Certification: The certification of SRM 1872 is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). However, the certification will be nullified if the SRM is damaged or contaminated.

Maintenance of Certification: NIST will monitor this SRM lot over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Stability: This material is considered to be stable during the period of certification.

The glasses were prepared in the NIST Materials Science and Engineering Lab. Quantitative wet chemical analyses were performed at Pennsylvania State University, University Park, PA.

Quantitative EPMA and homogeneity testing were performed by R.B. Marinenko, NIST Chemical Science and Technology Laboratory. Data reduction for the quantitative analysis was done with the correction procedure, COR, described in Henoc et al. [1].

Neutron activation analysis was also performed in the NIST Chemical Science and Technology Laboratory.

The technical measurements leading to certification were coordinated by R.B. Marinenko.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

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The major constituents of these glasses show no heterogeneity of any practical significance on the micrometer scale. Lead and germanium are certified in all three glasses. 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-453	K-491	K-968
Ge	28.43 ± 0.34	26.10 ± 0.34	25.93 ± 0.34
Pb	54.21 ± 0.26	54.69 ± 0.26	54.74 ± 0.26
O	(16.73)	(16.45)	(16.67)
Mg			(0.22) [0.08]
Al		(0.10) [0.09]	
Si		(0.11) [0.09]	
P			(0.21) [0.20]
Ti		(0.14) (0.16)	(0.16) [0.16]
Cr			(0.19) [0.17]
Fe		(0.17) [0.18]	
Ni			(0.20) [0.19]
Zr		(0.26) [0.30]	(0.48) [0.30]
Ba			(0.46) [0.45]
Ce		(0.59) [0.46]	
Eu			(0.64) [0.49]
Ta		(0.52) [0.59]*	
Th			(0.12) [0.08]
U			(0.05) [0.091]
Total	(99.37)	(99.13)	(100.07)

*Neutron activation: Ta = (0.58)

INSTRUCTIONS FOR USE

Lead and germanium values were determined by wet chemical analysis and EPMA. The certified values were determined from the weighted average of the two different methods of analysis [2]. 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 deviation 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 % or less in glasses K-491 and K-968, 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-491 was also determined by neutron activation analysis.

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

REFERENCES

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

Certificate Revision History: 18 December 2007 (Editorial and technical changes); 01 May 1984 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.