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Report of Investigation

Glasses for Microanalysis RM 30
Glass Fibers for Microanalysis RM 31

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Five types of glasses are available as research materials (RM) in the form of bulk samples, 2x2x20 mm, and as fibers ranging from 10-100 μm in diameter and 50-60 mm long.

The major constituents of the glasses were selected so that a variety of matrices of differing average atomic numbers would be represented. For each of the five matrices, there are two glasses – one with and one without the low-concentration additives.

Applications

These Reference Materials were developed for use in microanalysis techniques such as electron probe microanalysis (EPMA) and secondary ion mass spectrometry (SIMS). These homogeneous vitreous solids containing known low-concentration additions of several elements should be useful in the analysis of complicated unknowns. The fibers were drawn for use in particulate analyses for which there presently are no well-characterized standards of complex composition and simple shape.

Preparation of the Glasses

The compounds employed in the manufacture of the glasses were reagent-grade materials or compounds of equal or greater purity. Usually the oxides were used, but where this was not possible, the carbonates, nitrates, or phosphates were used. After weighing, the batch materials were mixed thoroughly as dry powders prior to melting.

The glass components were melted in 300 ml platinum crucibles in electrically heated furnaces in an oxidizing atmosphere (air). To ensure homogeneity, the glasses were stirred from four to six hours with a motor-driven, double-bladed, propeller-type stirrer made of a platinum-rhodium-alloy. Melting and stirring temperatures ranged from 900-1400 °C depending upon the glass being prepared. After completion of the melting and stirring operations, the glass was cast into a rectangular block and annealed to remove residual strain.

Composition

The compositions in weight percent of the oxides in the glasses are listed in the attached table. These are nominal values calculated from the weight of the component added to the melt during the manufacture of the glass. Preliminary analyses with the electron probe indicate that these nominal values are accurate to five percent relative or better for the major constituents, and to 20 percent relative for the low-concentration additions.

A thorough analysis and homogeneity study of these glasses by the electron probe is now in progress. Other analytical methods are also being used when necessary. This report will be revised when the analytical results are available.

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Preparation of Bulk Samples

The bulk samples, approximately 2x2x20 mm, were cut from the large annealed rectangular glass block. A precision wafering saw was used with a 0.071x15.2 cm, (0.028 x 6") 100 grit diamond wheel and a petroleum-based coolant. An acetone-soluble adhesive was used to mount the glass block and slices onto the cutting plate. Several 2 mm slices were cut from near the middle of the glass block. These slices were in turn cut at 2 mm intervals. The glass slices were removed from the cutting plate and rinsed with acetone several times to remove all traces of adhesive.

Preparation of Fibers

Preliminary to drawing fibers, a glass fragment was melted in a platinum boat that was heated directly by an electric current. The fiber was formed by drawing a thread of molten glass through an orifice in the bottom of the platinum boat. The continuous glass fiber was wound onto a 25.4 cm (10 inch) diameter metal drum driven by a variable speed electric motor (1000-4000 rpm). Different orifice sizes and drum speeds were used in drawing the fibers, depending upon the viscosity and surface tension of each glass. This procedure requires a minimum amount of working time and low temperatures, therefore minimizing compositional changes due to vaproization.

1. Fiori, C. E.; Heinrich, K. F. J.; Marinenko, R. B.; Darr, M. M.; Blackburn, D. H.; Newbury, D. E.; and Small, J. A., "An Overview of the Glass Standards Program for Microanalysis at the National Bureau of Standards," paper to be presented at the Joint Meeting of Electron Microscopy Society of America and Microbeam Analysis Society, Miami Beach, Fla., August 1976.

2. Marinenko, R. B.; Heinrich, K. F. J.; Fiori, C. E.; Darr, M. M., Blackburn, D. H.; Newbury, D. E.; and Small, J. A., "Glass Standards for Microanalysis of Particles," paper to be presented at Federation of the Analytical Chemistry and Spectroscopy Societies 3rd Annual Meeting, Philadelphia, Pa., November, 1976.

Glasses for Microanalysis

Melt No.	K-456	K-493	K-453	K-491	K-458	K-489	K-495	K-490	K-496	K-497
					Composition in Weight Percent					
SiO_2	28.77	27.89	---	0.19	49.38	46.76	---	0.42	---	0.27
PbO	71.23	69.08	58.72	59.35	---	1.28	---	1.55	---	.99
GeO_2	---	---	41.28	37.98	---	---	---	---	---	---
BaO	---	---	---	---	46.80	43.88	---	---	---	---
ZnO	---	---	---	---	3.82	3.72	---	---	---	---
P_2O_5	---	---	---	---	---	---	---	---	79.54	76.03
MgO	---	---	---	---	---	---	---	---	9.03	8.64
Al_2O_3	---	0.20	---	0.16	---	0.29	20.00	18.68	11.43	10.92
B_2O_3	---	.14	---	.11	---	.20	75.00	70.00	---	0.15
ZrO_2	---	.49	---	.40	---	.70	---	0.85	---	.54
TiO_2	---	.32	---	.26	---	.46	---	.55	---	.35
CeO_2	---	.68	---	.56	---	.98	---	1.19	---	.76
Ta_2O_5	---	.88	---	.72	---	1.26	---	1.53	---	.98
Fe_2O_3	---	.32	---	.26	---	0.46	---	0.55	---	.35
Li_2O	---	.001	---	.001	---	.002	5.00	4.67	---	.001