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Standard Reference Materials:

Glasses for Microanalysis: SRM's 1871–1875

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Glasses for Microanalysis: SRM's 1871–1875

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Preface

Standard Reference Materials (SRM's) as defined by the National Institute of Standards and Technology (NIST) are well-characterized materials, produced in quantity and certified for one or more physical or chemical properties. They are used to assure the accuracy and compatibility of measurements throughout the Nation. SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. They are also used extensively in the fields of environmental and clinical analysis. In many applications, traceability of quality control and measurement processes to the national measurement system is carried out through the mechanism and use of SRM's. For many of the Nation's scientists and technologists, it is therefore of more than passing interest to know the details of the measurements made at NIST in arriving at the certified values of the SRM's produced. The NIST Special Publication 260 Series is a series of papers reserved for this purpose.

The 260 Series is dedicated to the dissemination of information on different phases of the preparation, measurement, certification, and use of NIST SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing greatest care and accuracy. These papers also should provide sufficient additional information so SRM's can be utilized in new applications in diverse fields not foreseen at the time the SRM was originally issued.

Inquiries concerning the technical content of this paper should be directed to the author(s). Other questions concerned with the availability, delivery, price, and so forth, will receive prompt attention from:

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Gaithersburg, MD 20899

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Office of Standard Reference Materials

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GLASSES FOR MICROANALYSIS: SRM'S 1871-1875

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INTRODUCTION

Glass is an ideal material for multielement microanalytical standards. Over 60 elements can be used in making glasses and as many as 20 or more elements can be incorporated into a single glass. Oxide glasses generally exhibit excellent chemical and physical stability as well as structureless homogeneity. A glass matrix was therefore chosen for making this group of microanalytical standards which spans a wide range of average atomic numbers with many other oxides as minor constituents.

The preparation, homogeneity testing, and quantitative analyses of the Glasses for Microanalysis, Standard Reference Materials (SRM's) 1871 - 1875 are described. Each SRM represents a different glass matrix; these are lead-silicate (SRM 1871), lead-germanate (SRM 1872), barium-zinc-silicate (SRM 1873), lithium-aluminum-borate (SRM 1874), and aluminum-magnesium-phosphate (SRM 1875). There are three glasses in each SRM, one composed only of the matrix oxides and the other two having small additions (less than two percent) of several other elements in oxide form. The certified compositions, information values (non-certified compositions), and nominal values (amount of weighed material added to the melt during preparation) are listed in tables 1-5.

PREPARATION

The glasses were made at the National Institute of Standards and Technology (NIST) by D. H. Blackburn and D. A. Kauffman, Inorganic Materials Division, Center for Materials Science and Engineering. The chemicals used were reagent grade materials or compounds of equal or greater purity. In most preparations, stable oxides were used, but where this was not possible, carbonates, nitrates, and phosphates, which decompose to the appropriate oxides in the glass-melting process, were used. The batch materials were well-mixed as dry powders prior to melting. Each glass was melted in a 300 mL platinum crucible and stirred with a motor-driven platinum-rhodium alloy propeller-type stirrer. Melting and stirring operations were performed in electrically heated furnaces in an oxidizing atmosphere (air). Temperatures ranged from below 1000 °C to above 1500 °C depending upon the glass being prepared. All glasses were stirred 4 to 6 hours to insure homogeneity. After stirring, the molten glass was cast into a rectangular block and annealed for half an hour to remove residual strain. The mold had an aluminum bottom and stainless steel sides. Details about the preparation of each glass matrix are discussed below.

Lead-Silicate Glasses, K-456, K-493, and K-523: Silicon dioxide makes an excellent glass, but without modifications, the glass has a high melting temperature and the molten glass is extremely viscous. Lead oxide was selected as a modifier because it lowers both the melting temperature and the viscosity without sacrificing glass stability. These glasses were melted, stirred, and poured in the 1290-1320 °C temperature range and annealed at 480 °C.

Lead-Germanate Glasses, K-453, K-491, and K-968: Germanium dioxide also makes a glass when cooled from a melt, but even at 1600 °C is extremely viscous. The resulting glass is also not stable in the presence of water vapor. Lead oxide lowers the viscosity and improves the moisture stability. The glasses were processed in the 980-1010 °C temperature range and annealed at 400 °C.

Barium-Zinc-Silicate Glasses, K-458, K-489, and K-963: Barium oxide lowers the melting temperature and viscosity of the silicon dioxide, but barium-silicate glasses exhibit sub-liquidus phase separation due to metastable immiscibility. Small additions of zinc oxide prevent the immiscibility and resulting inhomogeneity. Processing temperatures for these glasses were from 1500-1540 °C, and the glasses were annealed at 680 °C.

Lithium-Aluminum-Borate Glasses, K-495, K-490, and K-546: Boron oxide, without modification, makes a glass which is not stable in the presence of water vapor. The addition of lithium oxide serves a dual purpose. It improves moisture stability and increases the solubility of other oxides in the resulting glass. Aluminum oxide additions further improve durability against moisture. The glasses were melted, stirred, and poured between 1050 and 1100 °C and annealed at 525 °C.

Aluminum-Magnesium-Phosphate Glasses, K-496, K-497, and K-1013: Phosphorus pentoxide is a glass-former, but under normal pressure sublimes rather than melts when heated. It is also water-soluble and extremely hygroscopic. Aluminum metaphosphate, $Al(PO_3)_3$, however, makes a very stable glass that is not affected by moisture even though it contains a high percentage of phosphorus pentoxide. Additions of magnesium phosphate, $Mg(PO_3)_2$ were made to lower both the melting temperature and viscosity without sacrificing moisture resistance. The glasses were processed in the 1380-1420 °C temperature range and were annealed at 620 °C.

The SRM specimens were cut from each glass block using a precision wafering saw with a 0.07 × 15.2 cm (0.028 × 6") 100-grit diamond wheel and a petroleum-based solvent. Each bar was cut into several hundred rod-like pieces approximately 2 × 2 × 20 mm. From the final lot, specimens were randomly selected for wet chemical analysis. Each rod was broken in half, one half to be submitted for the wet chemical analysis and the remaining half to be retained at NIST for future reference or for additional analyses if needed. Three grams of sample per element determination were submitted for quantitative analysis. This quantity provided enough sample for triplicate determination of each oxide. Five or six additional rods were randomly selected from the lot and mounted with standards in silver epoxy in a titanium sample mount for x-ray microanalysis. The sample mounts were polished with diamond polishing compound and cerium oxide and then carbon-coated for microprobe analysis.

HOMOGENEITY

The micro- and macrohomogeneity (between-specimen homogeneity) of the major constituents in each glass were determined by R. Marinenko with the electron microprobe according to the procedures described in references (1) and (2). The transverse microhomogeneity was observed with periodic integrator traces (1) such as those in figures 1 to 4. Here the specimen was moved in 1-µm steps under a 1-µm diameter electron beam. In all cases these traces gave signals that were within the ±3s limits delineated by the double-headed arrow to the extreme right of each trace, where s is $\sqrt{N}$ and N is the average number of counts per 10-second counting period for the trace. Also each trace of the moving specimen appeared to be the same as the time-resolved trace to the left in each figure which was recorded with a 20-μm electron beam on a stationary specimen. Experience with statistical analyses of traces such as these has shown that when the visual criteria cited above are met in a periodic integrator trace, there are no inhomogeneities of practical significance (1,2).

Periodic integrator traces of aluminum in the glasses in SRM 1874 exhibited a drift, regardless of the current used. This is not necessarily because aluminum is inhomogeneous in these glasses; the homogeneity of aluminum will be demonstrated below in another test. Rather, the drift may be due in part to a loss of components caused by electron beam damage to the specimen. These glasses do not hold up well under the electron beam - obvious damage can be seen even with a defocussed beam at the lowest currents. When the beam is moved randomly over large distances from one spot to another as was done in the following experiment, these glasses are subject to less damage than in the preparation of periodic integrator traces where each point is adjacent to the previous one. Also, the presence of lithium, which is known to migrate under the electron beam, could contribute to the difficulty in analyzing these glasses. Since these glasses were originally intended for use in Secondary Ion Mass Spectrometry (SIMS) analysis and not electron probe microanalysis (EPMA), such problems were not pursued further.

In the homogeneity tests and quantitative analysis of SRM 1874 which will be described below, EPMA was used successfully. These analyses were conducted very carefully in an effort to obtain as much information as possible about these glasses for certification purposes. Use of these standards in routine quantitative EPMA work is discouraged. Because of the low atomic number matrices, these standards are far better suited as standards for SIMS analyses.

A second homogeneity testing procedure (2) was used to evaluate statistically both the inter-specimen macrohomogeneity as well as the within-specimen microhomogeneity. Duplicate readings were taken on six randomly selected points of five specimens and each specimen was randomly sampled twice in the experiment. This results in a total of 120 readings per experiment, two on each of 60 different points.

Another sampling procedure was later adopted. It improves the randomness of the inter- and intra- specimen sampling, minimizing the effects of beam current drift during the experiment. In the second sampling procedure, only two points (with duplicate readings on each) are sampled per specimen before moving on to the next specimen. The five specimens are thus sampled in random order six times, again giving a total of 120 readings, two on each of 60 points. In tables 6-10 where the results of these homogeneity tests are tabulated, experiments K-453D and K-491G and H were done with the second sampling procedure. But in experiments K-496C, K-497C, and K-1013F only one point was read twice before proceeding to the next specimen.

For all homogeneity tests, three crystal spectrometers were used. When there were only two major components in a glass, duplicate data were taken for one of the elements with the third spectrometer. The excitation potential chosen depended upon the element being tested. The beam current was within the range of 30-40 nA. The beam current regulation mode was used to minimize current drift during each experiment, which lasted about 1 1/2 hours. At least duplicate tests were run on each glass. These were prepared on different days and, when possible, by different operators. Counting periods were 20 s. A point beam (less than 1 μm diameter) was used for SRM's 1871-1873. The glasses in SRM's 1874 and 1875 were more difficult to analyze because of beam damage to the specimens. Several experiments were run to check for damage to these glasses at different beam sizes, beam currents, and counting times. A 10 × 10 μm scanning raster minimized the beam damage to the specimens within the 20 s counting period at the selected beam current. Adequate counting statistics without drift during the counting time could be obtained under these conditions. In addition this beam size was not so large as to cause defocusing outside the optimum detection range of the spectrometers. The use of the same beam size is recommended when using these specimens as standards in WDS analyses. With the lower currents used in EDS analyses, a point beam may be used.

Three different error contributions can be estimated from this testing procedure. They are s_S^2, the variance between specimens, s_B^2, the variance within specimens on the micrometer scale, and s_E^2', the variance of a single measurement error. These errors are described in detail in reference 2. They are tabulated for each experiment in tables 6 to 10. The errors for the glasses in SRM's 1871-3 are generally lower than the errors for the glasses in SRM's 1874-5. This does not necessarily mean that the former are more homogeneous. The difference is attributable to the greater difficulty in testing the latter, where high counting statistics were not practical because of specimen damage at high currents.

With very few exceptions, the error between points within a given specimen, s_B, is below one percent as shown in the tables. This information in combination with the periodic integrator traces confirms that these glasses show no practical within-specimen inhomogeneity on the micrometer scale.

The experimental standard deviation, s_E, is estimated from the net number of counts per counting period; therefore, the higher the number of integrated counts, the lower will be this error. Only for zinc in SRM 1873 and for aluminum in SRM 1874 is this error as high as one percent relative. For each element, the integrated number of counts per 20 s was only 10,000.

The between-specimens standard deviation, s_S, is generally well below one percent; in the experiments where it does exceed one percent, the error is not consistent from one experiment to another on the same glass. Only for magnesium in K-1013 of SRM 1875 is there a consistently large between-specimen error. With this one exception, no between-specimen inhomogeneity of any practical significance can be inferred.

The standard deviation of a single measurement, s_P, is defined below according to the equation

$$s_P^2 = s_E^2 + s_B^2 + s_S^2.$$

The values for s_P are listed in the extreme right column of each table. For the glasses in SRM's 1871-3, these errors are small. The major elements in these glasses show no inhomogeneity of any practical significance. These errors for SRM's 1874 and 1875 are slightly larger. Therefore an inhomogeneity error was calculated and combined with the quantitative analysis error to obtain the final uncertainties quoted in the certificates.

The inhomogeneity error, $2s_I$ was calculated for a single point. For each element in each experiment the between-points and between-specimen errors were combined according to the expression

$$2s_I^2 = s_B^2 + s_S^2.$$

For each element an average value for $2s_I$ is calculated from the different experiments on each glass. This average value is then combined in quadrature with the quantitative analysis error (two standard deviations from the average) to obtain the uncertainty listed with the certified value.

QUANTITATIVE ANALYSIS

Most of the major constituents of each glass, with the exception of oxygen, were quantitatively determined by wet chemical analysis and EPMA. Silicon in glasses K-456 and K-453 was determined by emission spectrometry; inductively coupled plasma (ICP) spectrometry was used for K-456, and ICP and direct current plasma (DCP) spectrometry were used for K-453. Zinc in glass K-458 of SRM 1873 and aluminum in all three glasses of SRM 1875 were determined by neutron activation. Aluminum in glasses K-490 and K-546 of SRM 1874 and in K-497 and K-1013 of SRM 1875 was not determined by wet chemistry because several of the dopant elements in these glasses interfere with aluminum determinations. Lithium and boron in the glasses of SRM 1874 were not determined with

EPMA because accurate quantitative results for these elements could not be obtained with our present instrumentation. The individual results from each analytical procedure are listed in tables 11-15. These results are plotted for each element in figures 5-9.

The certified value for each major constituent (tables 1-5 and 11-15) was determined from the weighted average of the results from the two or three different analytical procedures. This weighted average was calculated according to the method described by Paule and Mandel (3). The error cited is ±2s, which for all elements but barium in glass K-963 is two times the pooled standard deviation of the certified value. The pooled value was determined from all three glasses in each SRM, and for aluminum it was taken from the three glasses in SRM 1875 plus K-495 in SRM 1874. For barium in SRM 1873 this error was calculated differently because the standard deviation for barium in glass K-963 was more than twice that of the other two glasses in this SRM. Therefore, a pooled value (±.20) was used for K-458 and K-489 while a single value (±.48) was used for K-963. For glasses K-493 and K-523 in SRM 1871 (table 1) and K-489 and K-963 in SRM 1873 (table 2) the silicon values (in parentheses) are not certified and are provided for information only. Also, only information values (in parentheses) are provided for aluminum in glasses K-490 and K-546 of SRM 1874 as well as for lithium and boron in all three glasses of this SRM. These information values have no errors reported because they were calculated from the average of repeated analyses by only one method. These elements will have to be determined by a second analytical procedure for final certification.

Most of the minor elements were determined with EPMA, and tantalum in four glasses was determined with neutron activation. Lithium and boron also were not determined with EPMA, so only nominal values are reported. The results are also listed in tables 1-5 and are compared to the nominal values in brackets.

ELECTRON MICROPROBE

The major and minor constituents were quantitatively determined by R. Marinenko with wavelength dispersive spectrometry (WDS). The voltages and standards used in the determination of each element are listed in table 16. For most glasses, a point beam (less than 1 μm) was used, but when beam damage on the specimen was a problem, a 10 × 10 μm scanning raster was used. Five or six randomly selected points were sampled on the standards, the glasses, and the background specimens. Results were averaged and background-corrected, then k-ratios were calculated for data reduction with either FRAME (4) or COR (5).

In all quantitative analyses the beam current was monitored intermittently throughout each experiment with a Faraday cup. With the current regulator, the beam current showed little or no drift (usually much less than one percent). For the major constituents each glass was analyzed in three different experiments. As previously mentioned the results of the analyses are listed in tables 11-15 and the individual values are plotted in figures 5-9. The EPMA values for the minor components are in most cases in good agreement with the nominal values; but since these elements occur in amounts so close to the lower limit of detection of the electron microprobe, a significant error is associated with these determinations. Therefore, the EPMA values are being provided for information only.

The combination of matrix elements in each of these SRM's had not been determined in our laboratory before these studies. There were some limitations on the excitation potential and current that could safely be used in the analyses of these SRM's without affecting the composition of the glass specimens. Below is a brief description of the problems or special considerations associated with the analysis of each SRM.

SRM 1871, Lead-silicate glasses. Some errors were observed in the silicon determinations using both FRAME and COR data reduction procedures. When quartz (SiO_2) and the mineral glass K-411 (SRM 470) were used as standards, the calculated silicon concentration was consistently several percent higher than expected. Also the analytical total exceeded 100 percent, confirming that at least one of the elements in the glasses was being calculated too high. These two standards have an average atomic number of 11 or below while the average atomic number of the lead-silicate glasses is about 56. Benitoite ($BaTiSi_3O_9$) with an average atomic number of 27, turned out to be a better standard, although the silicon results were still somewhat high. There is probably an atomic number effect from lead which is not entirely accounted for in our data reduction procedures. The best standard proved to be another NIST lead-silicate glass, K-227, which we have used successfully in our laboratory for many years. The nominal values, wet chemistry results, and electron microprobe results for this glass are shown in table 17. Because of the good agreement between the electron microprobe and the wet chemistry analyses, the average value from the latter analyses was used for this standard in the data reduction calculations. An excitation potential of 15 kV was used.

SRM 1872, Lead-germanate glasses. This combination of elements was also new to us. Analysis is straightforward as long as COR is used for matrix corrections. The germanium K line is excited by the lead L lines. For simplicity, the correction for this fluorescence, which is usually very small or insignificant, is not present in the FRAME program. Without the fluorescence correction, the calculated germanium concentration was as much as three percent above the expected concentration. The standards used were K-227 (Pb) and germanium. A 15 kV excitation potential was used.

SRM 1873, Barium-zinc-silicate glasses. The analysis of these glasses was straightforward. Both FRAME and COR gave the same results. The standards were Benitoite (Ba), K-227 (Si), and zinc. The excitation potential was 15 kV. One of the minor constituents was not determined. This was cerium in glass K-489. The barium La peak, which is close to the cerium La peak, contributes a considerable background under the cerium peak. Because of the low cerium concentration, the La peak cannot be resolved above the barium background.

SRM 1874, Lithium-aluminum-borate glasses. Because of the low average atomic number of these glasses, the current had to be maintained low enough (below 15 nA) to avoid damage to the specimen during the counting time. A 10 × 10 mm scanning raster was also used to minimize specimen damage. An optimum current with this beam size which would give the best possible counting statistics for aluminum without showing any change in the count rate during the 40-s counting period was experimentally determined. COR was used for data reduction; lithium and boron were treated as knowns, using concentrations for these elements that had been determined by wet chemistry. The standard was aluminum oxide.

SRM 1875, Aluminum-magnesium-phosphate glasses. The low average atomic number of these glasses presented the same problem cited above in the analysis of SRM 1874. A 10 × 10 mm scanning raster again was used to minimize specimen damage, and the current was carefully selected (50 nA or below) to prevent count rate drift during analyses. The excitation potential was 10 kV. The standard for aluminum and magnesium was glass K-412 (SRM 470), and for phosphorus, apatite was used. COR was used for data reduction.

WET CHEMISTRY

The quantitative wet chemical analyses were done in the Mineral Constitution Laboratories at The Pennsylvania State University by Joseph B. Bodkin and co-workers under the direction of Dr. Norman H. Suhr. Only the major constituents were determined for certification. The procedures used are listed in table 18. The results are listed in tables 11-15 and plotted in figures 5-9.

EMISSION SPECTROMETRY

Emission spectrometry analyses were done in the Inorganic Analytical Research Division, NIST. The ICP spectrometry was done by R. L. Watters and the DCP spectrometry by M. S. Epstein. For ICP analysis, duplicate specimens were fused with sodium carbonate, dissolved in water and brought to volume. Lead precipitation from the K-456 sample was prevented by the addition of about 2 g/L tartaric acid. Two silicon standards were prepared to match the sodium carbonate content of the samples. The concentration value reported for each specimen is the average of four ICP integrations.

For DCP spectrometry the specimens were fused with lithium borate, dissolved in dilute hydrochloric acid, and brought to volume. Standards were prepared with the same lithium concentration as the specimens. At least four 10-s integrations were taken per specimen. These were averaged for the final value reported. Data are listed in tables 11 and 13 and plotted in figures 5 and 7.

NEUTRON ACTIVATION

Neutron Activation Analysis was done by R. M. Lindstrom and G. J. Lutz of the Inorganic Analytical Research Division, NIST. The determination of aluminum in the SRM 1875 glasses was done at the NIST reactor facility. To avoid interference from the fast-neutron reaction on phosphorus, the samples were irradiated in the thermal-column facility RT5 at a flux of 1.6×10^{11} n/cm^{2}s. Five samples of each glass, standards of pure aluminum wire, check standards, and blanks were irradiated singly for 2 minutes and counted on the ATF detector at 20-cm geometry. The PEAK program was used to integrate the Al-28 peak. Corrections were made for decay, dead time, and pulse pileup.

Irradiation of K_2HPO_4 showed that the fast-neutron reaction contributed an apparent aluminum concentration of less than 0.002 grams of aluminum per gram of glass containing 75 percent P_2O_5, at the 95 percent confidence level. Analysis of SRM 120a Phosphate Rock (34.4 percent P_2O_5) for aluminum confirmed the absence of phosphorus interferences; SRM 1633a Fly Ash was analyzed to verify the procedure. Three determinations of aluminum in SRM 1633a, Fly Ash, gave 14.4±0.3 percent aluminum. A previous determination of aluminum in SRM 1633a gave 14.1±0.1 percent.

The determinations of tantalum and zinc were done at the University of Missouri Research Reactor. Specimens were irradiated for 5 minutes at a flux of 5×10^{13}n/cm^2-s. Because of a 15 percent flux gradient from side to side in the rabbit at this facility, a zinc flux monitor was taped to each packaged specimen. A solution of tantalum was used as a tantalum standard, and high purity zinc metal was used as a standard in the zinc determination.

The nuclear reactions and gamma-rays used in the analysis were:

^{181}Ta(n,g)^{182}Ta	1.121 Mev
^{64}Zn(n,g) ^{65}Zn	1.115 Mev.

Tantalum was determined in glasses K-493, K-491, K-489, and K-497. Zinc was determined in glass K-458. Attempts to determine Zn in glass K-489 were unsuccessful because of interferences from the 1.113 and 1.121 Mev lines of ^{182}Ta. Efforts to strip the tantalum component from the spectrum of this glass were also unsuccessful.

NEUTRON DEPTH PROFILING

Boron depletion is known to occur at the surface of boron- containing glasses when exposed to an aqueous environment (6). This depletion occurs because of the formation and dissolution of boric acid when the specimen comes into contact with water. All of these SRM glass specimens had been in an aqueous environment during cutting and polishing. Neutron depth profiling (NDP) was therefore done on specimens K-490, K-495, and K-546 to determine the extent of depletion. The analysis was done by R. G. Downing (7) of the Inorganic Analytical Research Division, NIST.

For each glass, a region about 1 cm in diameter was studied. Observation of the emission of 1472.3 keV alpha particles from the ^{10}B(n,a)^{7}Li reaction showed that there is indeed boron depletion near the surface. Relative to the bulk concentration observed at 1.5 mm in depth, the specimens were found to be 20-30 percent boron- depleted at 0.12 mm with depletion dropping to 3-7 percent at 0.5 mm; no depletion was observed beyond 1.3 mm.

A Monte Carlo calculation (8) was run to determine the depth from which x rays are generated in a specimen with the composition of glass K-495 at an excitation potential of 10 kV. This type of calculation is used to theoretically predict what physical interactions occur in a specimen of given composition when excited by an electron beam. Each electron event is calculated individually. For these studies, 10,000 trajectories were calculated.

The results of these calculations showed that aluminum x-rays are generated from a depth of up to 1.3 mm. If boron depletion effectively led to aluminum enrichment, the amount of generated aluminum x-rays would increase by one-third. One would expect from these results that electron microprobe analysis of K-495 would yield a much higher concentration of aluminum than was determined by wet chemistry. This, in fact, does not occur as can be seen in table 14, where wet chemistry and the EPMA results are in very good agreement. It appears, therefore, that the loss of boron has not meant that aluminum has, in part, filled the void. Probably another replacement, such as water, has occurred or the voids have remained unfilled.

Because of this boron depletion on the surface as well as the presence of lithium, which is known to migrate under the electron beam, the use of these glasses (SRM 1874) for quantitative EPMA is not recommended. This SRM is most useful in SIMS analysis where surface depletion is less of a problem because of the possibility of using ion beam erosion to remove the altered layer. A freshly cleaved surface should be used to avoid as much as possible the loss of boron from the surface.

CONCLUSIONS

These SRM's are unique microanalytical standards. The matrices are unusual combinations of oxides not found in presently available standards for microanalysis. The major components show no serious inhomogeneities on the micrometer scale. And the additional low-concentration oxides (dopants), though not certified for either microhomogeneity or composition, are useful when a measurement reference at these concentration levels are needed.

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Table 1. SRM 1871, Lead-Silicate Glasses
Concentrations 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 ± 0.24	(13.09 ± 0.24)	(12.94 ± 0.24)
O	(20.35)	(20.58)	(20.80)
Li		[0.0005]	
B		[0.04]	
Mg			(0.12)[0.10]
Al		(0.13)[0.11]	
P			(0.24)[0.25]
Ti		(0.20)[0.19]	(0.21)[0.19]
Cr			(0.20)[0.21]
Fe		(0.25)[0.22]	
Ni			(0.25)[0.24]
Ge			(0.20)[0.29]
Zr		(0.38)[0.36]	(0.33)[0.36]
Ba			(0.61)[0.55]
Ce		(0.53)[0.55]	
Eu			(0.73)[0.60]
Ta		(0.64)[0.72]*	
Th			(0.08)[0.10]
U			(0.23)[0.11]
Total	(99.38)	(99.12)	(100.19)

Values in parentheses are not certified and are provided for Information Only; values in brackets are nominal values and are not certified.

*Concentration by neutron activation analysis for Ta = (0.74)

Table 2. SRM 1872, Lead-Germanate Glasses
Concentrations in Weight Percent

Element		Glass	
	K-453	K-491	K-968
Pb	54.21 ± 0.26	54.69 ± 0.26	54.74 ± 0.26
Ge	28.43 ± 0.34	26.10 ± 0.34	25.93 ± 0.34
O	(16.73)	(16.45)	(16.67)
Li		[0.0005]	
B		[0.03]	
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)[0.09]
Total	(99.37)	(99.16)	(100.07)

Values in parentheses are not certified and are provided for Information Only; values in brackets are nominal values and are not certified.

*Concentration by neutron activation analysis for Ta = (0.58)

Table 3. SRM 1873, Barium-Zinc-Silicate Glasses

Concentrations in Weight Percent

Element		Glasses	
	K-458	K-489	K-963
Ba	41.79 ± 0.20	39.53 ± 0.20	39.21 ± 0.48
Zn	3.01 ± 0.06	2.93 ± 0.06	2.95 ± 0.06
Si	23.05 ± 0.34	(22.23 ± 0.34)	(21.96 ± 0.34)
O	(31.86)	(31.70)	(32.00)
Li		[0.0009]	
B		[0.06]	
Mg			(0.34)[0.14]
Al		(0.11)[0.15]	
P		(0.33)[0.36]	
Ti		(0.27)[0.28]	(0.32)[0.36]
Cr			(0.31)[0.30]
Fe		(0.35)[0.32]	
Ni			(0.33)[0.34]
Ge			(0.47)[0.42]
Zr		(0.40)[0.52]	(0.61)[0.53]
Ce		[0.80]	
Eu			(0.95)[0.88]
Ta		(0.95)[1.03]*	
Pb		(1.32)[1.19]	
Th			(0.06)[0.13]
U			(0.16)[0.17]
Total	(99.71)	(100.65)	(100.00)

Values in parentheses are not certified and are provided for Information Only; values in brackets are nominal values and are not certified.

*Concentration by neutron activation analysis for Ta = (1.03)

Table 4. SRM 1874, Lithium-Aluminum-Borate Glasses

Concentrations in Weight Percent

Element		Glass	
	K-495	K-490	K-546
Li	(2.33)	(2.22)	(2.16)
Al	10.89 ± 0.23	(10.15)	(10.06)
B	(22.97	(21.48)	(21.62)
O	(63.39)	(60.74)	(60.75)
Mg			(0.17)[0.17]
Si		(0.19)[0.20]	
P			(0.42)[0.43]
Ti		(0.31)[0.33]	(0.39)[0.34]
Cr			(0.15)[0.16]
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]	
Pb		(1.47)[1.44]	
Th			(0.16)[0.16]
U			(0.24)[0.20]
Total	(99.58)	(99.49)	(99.52)

Values in parentheses are not certified and are provided for Information Only; values in brackets are nominal values and are not certified.

Table 5. SRM 1875, Aluminum-Magnesium Phosphate Glasses
Concentrations 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)*	(51.30)*	(52.37)*
Li		[0.0005]	
B		[0.05]	
Si		(0.13)[0.13]	
Ti		(0.22)[0.21]	(0.21)[0.22]
Cr			(0.14)[0.23]
Fe		(0.26)[0.24]	
Ni			(0.31)[0.26]
Ge			(0.25)[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)[0.80]**	
Pb		(0.86)[0.92]	
Th			(0.10)[0.11]
U			(0.15)[0.13]

Values in parentheses are not certified and are provided for Information Only; values in brackets are nominal values and are not certified.

*Oxygen values calculated by difference because the stoichiometry of the phosphorus oxides in these glasses is unpredictable

**Concentration by neutron activation analysis for Ta = (0.83)

Table 6. Homogeneity Evaluation of Glasses K-456, K-493, and K-523[a]

				Standard Deviations in Weight Percent[c]			
Glass	Exp	Element	Wt. %[b]	s_E	s_B	s_S	s_P
K-456	A	Si	13.37	0.04(0.30)	0.06(0.45)	0.02(0.10)	0.07(0.52)
		Pb	65.67	0.32(0.49)	0.68(1.03)	0.19(0.29)	0.77(1.17)
		Si	13.37	0.04(0.30)	0.05(0.26)	0.02(0.10)	0.06(0.44)
	B	Si	13.37	0.04(0.30)	0.03(0.22)	0.03(0.22)	0.06(0.45)
		Pb	65.67	0.32(0.49)	0.10(0.15)	0.0	0.34(0.52)
		Si	13.37	0.04(0.30)	0.02(0.15)	0.01(0.07)	0.05(0.37)
K-493	B	Si	[13.09]	0.03(0.23)	0.03(0.23)	0.01(0.07)	0.04(0.31)
		Pb	63.28	0.32(0.50)	0.17(0.27)	0.06(0.09)	0.36(0.57)
		Si	[13.09]	0.03(0.23)	0.03(0.23)	0.0	0.09(0.33)
	C	Si	[13.09]	0.04(0.31)	0.03(0.22)	0.03(0.22)	0.06(0.45)
		Pb	63.28	0.32(0.51)	0.39(0.62)	0.24(0.38)	0.56(0.88)
		Si	[13.09]	0.04(0.31)	0.04(0.31)	0.02(0.15)	0.06(0.45)
K-523	A	Si	[12.94]	0.03(0.23)	0.01(0.08)	0.01(0.08)	0.04(0.31)
		Pb	63.10	0.32(0.50)	0.0	0.09(0.15)	0.32(0.51)
		Si	[12.94]	0.03(0.23)	0.03(0.22)	0.05(0.18)	0.05(0.39)
	B	Si	[12.94]	0.04(0.31)	0.02(0.15)	0.01(0.08)	0.04(0.31)
		Pb	63.10	0.32(0.51)	0.29(0.46)	0.07(0.11)	0.44(0.70)
		Si	[12.94]	0.04(0.31)	0.06(0.46)	0.01(0.08)	0.07(0.54)

---

[a] 15 kV excitation potential and five specimens of each glass.

[b] Certified concentrations; numbers in brackets are not certified and are provided for Information Only.

[c] Values in parentheses are relative errors in percent.

Table 7. Homogeneity Evaluation of Glasses K-453, K-491, and K-968[a]

				Standard Deviations in Weight Percent[c]			
Glass	Exp	Element	Wt. %[b]	s_E	s_B	s_S	s_P
K-453	A	Pb	54.21	0.35(0.64)	0.34(0.63)	0.0	0.49(0.90)
		Pb	54.21	0.24(0.44)	0.29(0.53)	0.0	0.37(0.69)
		Ge	28.43	0.15(0.53)	0.03(0.12)	0.12(0.44)	0.19(0.67)
	D	Pb	54.21	0.33(0.61)	0.10(0.18)	0.0	0.34(0.64)
		Pb	54.21	0.24(0.44)	0.13(0.24)	0.03(0.06)	0.27(0.51)
		Ge	28.43	0.14(0.49)	0.01(0.04)	0.03(0.11)	0.14(0.50)
K-491	G	Pb	54.69	0.33(0.60)	0.0	0.08(0.14)	0.34(0.62)
		Pb	54.69	0.24(0.44)	0.08(0.15)	0.06(0.11)	0.26(0.48)
		Ge	26.10	0.13(0.50)	0.0	0.0	0.13(0.50)
	H	Pb	54.69	0.33(0.60)	0.13(0.24)	0.0	0.35(0.64)
		Pb	54.69	0.24(0.44)	0.16(0.29)	0.03(0.05)	0.29(0.55)
		Ge	26.10	0.13(0.50)	0.02(0.08)	0.06(0.23)	0.14(0.54)
K-968	A	Pb	54.74	0.35(0.64)	0.19(0.35)	0.10(0.18)	0.42(0.76)
		Pb	54.74	0.24(0.44)	0.18(0.33)	0.11(0.18)	0.32(0.58)
		Ge	25.93	0.20(0.54)	0.08(0.31)	0.04(0.15)	0.17(0.66)
	B	Pb	54.74	0.35(0.64)	0.21(0.39)	0.21(0.39)	0.46(0.85)
		Pb	54.74	0.25(0.46)	0.29(0.53)	0.21(0.39)	0.44(0.80)
		Ge	25.93	0.14(0.54)	0.09(0.35)	0.07(0.27)	0.18(0.69)

---

[a] 25 kV excitation potential and five specimens of each glass

[b] Certified concentrations.

[c] Values in parentheses are relative errors in percent.

Table 8. Homogeneity Evaluation of Glasses K-458, K-489, and K-963[a]

				Standard Deviations in Weight Percent[c]			
Glass	Exp	Element	Wt. %[b]	s_E	s_B	s_S	s_P
K-458	A	Si	23.05	0.04(0.17)	0.07(0.30)	0.05(0.22)	0.10(0.43)
		Zn	3.01	0.03(1.00)	0.02(0.66)	0.01(0.33)	0.04(1.33)
		Ba	41.79	0.25(0.59)	0.16(0.38)	0.0	0.30(0.70)
	B	Si	23.05	0.04(0.17)	0.07(0.30)	0.03(0.13)	0.09(0.39)
		Zn	3.01	0.03(1.00)	0.01(0.33)	0.0	0.03(1.00)
		Ba	41.79	0.26(0.62)	0.09(0.21)	0.07(0.17)	0.29(0.68)
K-489	C	Si	[22.23]	0.04(0.18)	0.06(0.27)	0.04(0.18)	0.08(0.36)
		Zn	2.93	0.03(1.02)	0.02(0.68)	0.01(0.34)	0.04(1.37)
		Ba	39.53	0.24(0.61)	0.10(0.25)	0.0	0.26(0.66)
	D	Si	[22.23]	0.04(0.18)	0.06(0.27)	0.03(0.13)	0.15(0.67)
		Zn	2.93	0.03(1.02)	0.02(0.68)	0.02(0.68)	0.04(1.37)
		Ba	39.53	0.25(0.63)	0.19(0.48)	0.18(0.46)	0.36(0.91)
K-963	A	Si	[21.96]	0.04(0.18)	0.03(0.14)	0.05(0.23)	0.07(0.32)
		Zn	2.95	0.03(1.02)	0.02(0.68)	0.0	0.04(1.36)
		Ba	39.21	0.25(0.64)	0.13(0.33)	0.0	0.27(0.69)
	B	Si	[21.96]	0.04(0.18)	0.06(0.27)	0.01(0.02)	0.07(0.32)
		Zn	2.95	0.03(1.02)	0.01(0.34)	0.02(0.68)	0.04(1.36)
		Ba	39.21	0.25(0.64)	0.0	0.01(0.03)	0.25(0.64)

---

[a] 15 kV excitation potential and five specimens of each glass.

[b] Certified concentrations; numbers in brackets are not certified and are provided for Information Only.

[c] Values in parentheses are relative errors in percent.

Table 9. Homogeneity Evaluation of Glasses K-495, K-490, and K-546[a]

				Standard Deviations in Weight Percent[c]			
Glass	Exp	Element	Wt. %[b]	s_E	s_B	s_S	s_P
K-495	A	Al	10.89	0.10(0.92)	0.0	0.04(0.37)	0.11(1.01)
		Al	10.89	0.11(1.01)	0.03(0.28)	0.06(0.55)	0.13(1.19)
	B	Al	10.89	0.10(0.92)	0.05(0.46)	0.07(0.64)	0.13(1.19)
		Al	10.89	0.11(1.01)	0.0	0.09(0.83)	0.14(1.29)
	C	Al	10.89	0.10(0.92)	0.05(0.46)	0.04(0.37)	0.13(1.19)
		Al	10.89	0.11(1.01)	0.04(0.37)	0.11(1.01)	0.16(1.47)
K-490	A	Al	[10.15]	0.10(0.98)	0.0	0.0	0.10(0.98)
		Al	[10.15]	0.11(1.08)	0.02(0.20)	0.0	0.11(1.08)
	B	Al	[10.15]	0.10(0.98)	0.02(0.20)	0.0	0.10(0.98)
		Al	[10.15]	0.11(1.08)	0.04(0.39)	0.07(0.69)	0.14(1.38)
K-546	A	Al	[10.06]	0.10(0.99)	0.01(0.10)	0.02(0.20)	0.10(0.99)
		Al	[10.06]	0.11(1.09)	0.0	0.03(0.30)	0.11(1.09)
	B	Al	[10.06]	0.10(0.99)	0.05(0.50)	0.12(1.19)	0.16(1.59)
		Al	[10.06]	0.11(1.09)	0.07(0.70)	0.15(1.49)	0.19(1.89)
	D	Al	[10.06]	0.10(0.99)	0.04(0.40)	0.0	0.11(1.09)
		Al	[10.06]	0.10(0.99)	0.04(0.40)	0.03(0.30)	0.11(1.09)

---

[a] 15 kV excitation potential and five specimens of each glass.

[b] Certified concentrations; numbers in brackets are not certified and are provided for Information Only.

[c] Values in parentheses are relative errors in percent.

Table 10. Homogeneity Evaluation of Glasses K-496, K-497, and K-1013[a]

Glass	Exp	Element	Wt. %[b]	Standard Deviations in Weight Percent[c]			
				s_E	s_B	s_S	s_P
K-496	A	Mg	6.65	0.03(0.45)	0.04(0.60)	0.01(0.15)	0.05(0.75)
		P	32.98	0.12(0.36)	0.22(0.67)	0.10(0.30)	0.27(0.82)
		Al	6.47	0.03(0.46)	0.08(1.24)	0.04(0.62)	0.09(1.39)
	B	Mg	6.65	0.03(0.45)	0.03(0.45)	0.0	0.04(0.60)
		P	32.98	0.12(0.36)	0.08(0.24)	0.10(0.30)	0.17(0.52)
		Al	6.47	0.03(0.46)	0.02(0.31)	0.01(0.15)	0.03(0.46)
	C	Mg	6.65	0.04(0.60)	0.01(0.15)	0.01(0.15)	0.04(0.60)
		P	32.98	0.24(0.73)	0.25(0.76)	0.0	0.34(1.03)
		Al	6.47	0.03(0.46)	0.0	0.03(0.46)	0.04(0.62)
K-497	A	Mg	6.49	0.03(0.46)	0.05(0.77)	0.01(0.15)	0.05(0.77)
		P	31.59	0.12(0.38)	0.24(0.76)	0.12(0.38)	0.30(0.95)
		Al	5.97	0.03(0.50)	0.09(1.51)	0.04(0.67)	0.11(1.84)
	B	Mg	6.49	0.03(0.46)	0.04(0.62)	0.02(0.31)	0.05(0.77)
		P	31.59	0.12(0.38)	0.12(0.38)	0.22(0.70)	0.27(0.85)
		Al	5.97	0.03(0.50)	0.05(0.84)	0.01(0.17)	0.06(1.01)
	C	Mg	6.49	0.04(0.62)	0.01(0.15)	0.01(0.15)	0.04(0.62)
		P	1.59	0.21(0.66)	0.17(0.54)	0.0	0.27(0.85)
		Al	5.97	0.03(0.50)	0.03(0.50)	0.01(0.17)	0.04(0.67)
K-1013	A	Mg	5.86	0.02(0.34)	0.05(0.85)	0.08(1.37)	0.10(1.71)
		P	32.26	0.13(0.40)	0.15(0.46)	0.05(0.15)	0.21(0.65)
		Al	6.08	0.03(0.49)	0.02(0.33)	0.03(0.49)	0.05(0.82)
	B	Mg	5.86	0.02(0.34)	0.07(1.19)	0.07(1.19)	0.10(1.71)
		P	32.26	0.12(0.37)	0.30(0.93)	0.07(0.22)	0.19(0.59)
		Al	6.08	0.03(0.49)	0.03(0.49)	0.04(0.66)	0.05(0.82)
	F	Mg	5.86	0.03(0.51)	0.05(0.85)	0.10(1.71)	0.11(1.88)
		P	32.26	0.22(0.68)	0.17(0.53)	0.04(0.12)	0.28(0.87)
		Al	6.08	0.03(0.49)	0.05(0.82)	0.04(0.66)	0.07(1.15)

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[a] 15 kV excitation potential and five specimens of each glass.
[b] Certified concentrations.
[c] Values in parentheses are relative errors in percent.

Table 11. Quantitative Analysis Of The Lead-Silicate Glasses, SRM 1781
Concentrations in Weight Percent

Glass	Element	Nominal	Wet Chem.	EPMA	ICP	Certified Value*
K-456	Si	13.45	13.28	13.48	12.89	13.37 ± 10.24
			13.29	13.58	13.54	
			13.32	13.64		
	Pb	66.12	65.71	65.18		65.67±10.26
			65.77	65.85		
			65.77	65.74		
K-493	Si	13.04	12.99	12.96		(13.09 ± 10.24)
			13.01	13.28		
			13.01	13.29		
	Pb	64.13	63.37	62.84		63.28 ± 10.26
			63.20	63.46		
			63.27	63.56		
K-523	Si	12.90	12.81	13.13		(12.94 ± 10.24)
			12.83	13.06		
			12.77	13.05		
	Pb	63.32	63.27	62.32		63.10 ± 10.26
			63.30	63.38		
			63.17	63.18		

*Numbers in parentheses are not certified and are provided for Information Only.

Table 12. Quantitative Analysis of the Lead-Germanate Glasses, SRM 1782
Concentrations in Weight Percent

Glass	Element	Nominal	Wet Chem.	EPMA	Certified Value*
K-453	Ge	28.65	28.64 28.55 28.60	28.35 28.37 28.05	28.43 ± 10.34
	Pb	54.51	54.20 54.10 54.15	54.38 54.40 54.03	54.21 ± 10.26
K-491	Ge	26.36	26.14 26.23 26.44	26.32 25.91 25.55	26.10 ± 10.34
	Pb	55.10	54.82 55.01 54.74	54.58 54.53 54.45	54.69 ± 10.26
K-968	Ge	26.27	25.97 26.27 26.06	25.94 25.86 25.48	25.93 ± 10.34
	Pb	54.91	54.89 54.85 54.92	54.78 54.66 54.36	54.74 ± 10.26

Table 13. Quantitative Analysis of Barium-Zinc-Silicate Glasses, SRM 1873
Concentrations in Weight Percent

Glass	Element	Nominal	Wet Chem.	EPMA	ICP	DCP	Certified Value*
K-458	Si	23.08	22.65	23.42	23.12	22.98	23.05 ± 10.34
			22.71	23.64	22.74	23.02	
					22.68	23.64	
					Neutron Activation		
	Zn	3.07	3.04	2.89	3.10		3.01 ± 10.06
			3.05	2.92	2.99		
			3.04	3.02	3.00		
			3.02	3.04			
				3.03			
				2.99			
				2.95			
	Ba	41.92	41.85	42.00			41.79 ± 10.20
			41.71	41.41			
			41.95	41.82			
K-489	Si	21.86	22.08	22.32			(22.23 ± 10.34)
			22.08	22.47			
			22.04	22.38			
	Zn	2.99	2.96	2.83			2.93 ± 10.06
			2.97	2.94			
			2.96	2.87			
				2.90			
				2.91			
				2.92			
				2.88			
	Ba	39.30	39.45	39.36			39.53 ± 10.20
			39.56	39.60			
			39.26	39.95			
K-963	Si	21.99	21.93	21.24			21.96 ± 10.34)
			21.97	22.27			
			21.93	22.43			
	Zn	3.00	3.00	2.95			2.95 ± 10.06
			3.00	2.96			
			2.99	2.84			
				2.94			
				2.90			
				2.91			
				2.89			
	Ba	39.52	39.59	39.32			39.21 ± 10.48
			39.40	38.58			
			39.36	38.99			

*Numbers in parentheses are not certified and are provided for Information Only.

Table 14. Quantitative Analysis of the Lithium-Aluminum-Borate Glasses, SRM 1874
Concentrations in Weight Percent

Glass	Element	Nominal	Wet Chem.	EPMA	Certified Value*
K-495	Li	2.32	2.31		(2.33)
			2.35		
			2.32		
			2.35		
	Al	0.58	10.91	11.13	10.89 ± 0.23
			10.88	10.87	
			10.88	10.85	
				10.67	
				10.75	
	B	23.29	22.99	(22.97)	
			22.99		
			22.93		
K-490	Li	2.17	2.20		(2.22)
			2.22		
			2.22		
			2.22		
	Al	9.89		9.75	(10.15)
				10.30	
				10.14	
				10.24	
	B	21.74	21.49		(21.48)
			21.47		
			21.47		
K-546	Li	2.16	2.16		(2.16)
			2.14		
			2.17		
	Al	9.86	9.91		(10.06)
			10.18		
			10.18		
			9.97		
			10.11		
	B	1.68	21.58		(21.62)
			21.64		
			21.64		

*Numbers in parentheses are not certified and are provided for Information Only.

Table 15. Quantitative Analysis of the Aluminum-Magnesium Phosphate Glasses, SRM 1875
Concentrations in Weight Percent

Glass	Element	Nominal	Wet Chem.	EPMA	Neut. Act.	Certified Value
K-496	Al	6.05	6.48	6.44	6.33	6.47 ± 0.20
			6.57	6.45	6.33	
			6.48	6.41	6.36	
					6.43	
					6.33	
	Mg	5.44	6.73	6.56		6.65 ± 0.17
			6.71	6.61		
			6.73	6.52		
	P	34.71	32.74	33.58		32.98 ± 0.55
			32.77	33.07		
			32.78	33.11		
K-497	Al	5.78		6.03	5.85	5.97 ± 0.22
				6.04	5.94	
				6.01	5.95	
					5.94	
					5.84	
	Mg	5.21	6.56	6.40		6.49 ± 0.17
			6.58	6.42		
			6.59	6.38		
	P	33.81	31.59	31.90		31.59 ± 0.58
			31.61	31.51		
			31.57	31.53		
K-1013	Al	5.82		6.26	5.95	6.08 ± 0.21
				6.29	5.82	
				6.19	5.83	
					5.84	
					6.01	
	Mg	5.24	5.80	5.93		5.86 ± 0.26
			5.80	5.94		
			5.79	5.91		
	P	33.40	32.40	32.17		32.26 ± 0.56
			32.52	32.05		
			32.46	31.95		

Table 16. Voltages and Standards Used in Wavelength Dispersive Analysis

Element/Line	Voltage (kV)	Standard(s)
MgKα	15	MgO
	10	K-412 glass-SRM 470
AlKα	15	Al_2O_3
	10	K-412 glass-SRM 470
SiKα	15	K-227*,SiO_2
PKα	10	Apatite
TiKα	10,15	Ti
CrKα	15	Cr
FeKα	15	Fe
NiKα	20	Ni
ZnKα	20	Zn
GeKα	20	Ge
ZrLα	10,15	Zr
BaLα	15	Benitoite
CeLα	15	K-521
EuLα	15	$Eu(PO_3)_3$*
TaMα	15	Ta
PbMαa	15	K-227*,PbTe
ThMα	10	ThF_4
UMα	10	U

*These glasses were fabricated at NIST by the Inorganic Materials Division, Center for Materials Science and Engineering. $Eu(PO_3)_3$ is a stoichiometric compound. The composition and wet chemistry values used for K-227 are listed in table 17. The nominal values were used for glass K-521; these are 68 wt. percent B_2O_3, 25 wt. percent CeO_2 and 7 wt. percent LiO_2.

Table 17. Quantitative Analysis of Standard Glass K-227

Element	Concentrations in Weight Percent		
	Nominal	Wet Chem.	EPMA (standards)
Silicon	9.34	9.58	9.54 (Benitoite)
Lead	74.26	73.86	73.31 (PbS)
Oxygen*	16.40	16.64	16.58
Total	100.00	100.08	99.45

*Calculated from stoichiometry

Table 18. Wet Chemistry Procedures Used in the Quantitative Analyses of SRM's 1871-1875

SRM 1871, Lead-Silicate Glasses

SiO_2 - Gravimetric determination by single or double dehydration with any silica remaining in solution determined by DC plasma spectrometry.

PbO - Separation by extraction with sodium diethyldithiocarbamate in chloroform followed by precipitation as $PbCrO_4$.

SRM 1872, Lead-Germanate Glasses

GeO_2 - Fusion of 230 mg. samples with a mixture of Na_2O_2 and NaOH. Germanium distilled as $GeCl_4$ followed by titration with standard KIO_3.

PbO - Samples were dissolved in HCL to remove germanium. For K-453 and K-491 the lead was first isolated as $PbSO_4$ and then dissolved in excess standard Mg-EDTA solution. The equivalent amount of released magnesium was titrated with standard CDTA. On sample K-968, the lead was determined gravimetrically as $PbCrO_4$.

SRM 1873, Barium-zinc-silicate Glasses

SiO_2 - Same as silica in SRM 1871 above.

ZnO - Complexometric determination after ion exchange separation following removal of barium.

BaO - Gravimetric determination after HF, $HClO_4$ dissolution and ion exchange separation to remove interferences. Barium still remaining in solution determined by DC plasma spectrometry.

SRM 1874, Lithium-aluminum-borate Glasses

Li_2O - Solution of sample with HF and H_2SO_4. Interfering elements removed by the addition of CaO. Li_2O determined with atomic absorption spectrophotometry after the removal of excess CaO.

Al_2O_3 - CDTA titration following fusion of the sample with K_2SO_7.

B_2O_3 - Titration with sodium hydroxide following fusion with potassium pyrosulfate and complexation of interfering elements with CDTA.

SRM 1875, Aluminum-magnesium-phosphate Glasses

Al_2O_3 - After fusion of K-496 with and an equimolar mixture of Na_2CO_3 and $Na_2B_4O_7$ and separation from P_2O_5 with a cation exchanger, a CDTA titration with a zinc back titration was used.

MgO - After fusion with and an equimolar mixture of Na_2CO_3 and K_2CO_3 and removal of interferences, MgO in K-496 was determined by EDTA titration and in K-497 and K-1013 by gravimetry as $Mg_2P_2O_7$.

P_2O_5 - After the above-described fusion (MgO), dissolution with HCl, and removal of interferences with a cation exchanger, P_2O_5 was determined gravimetrically as $Mg_2P_2O_7$.

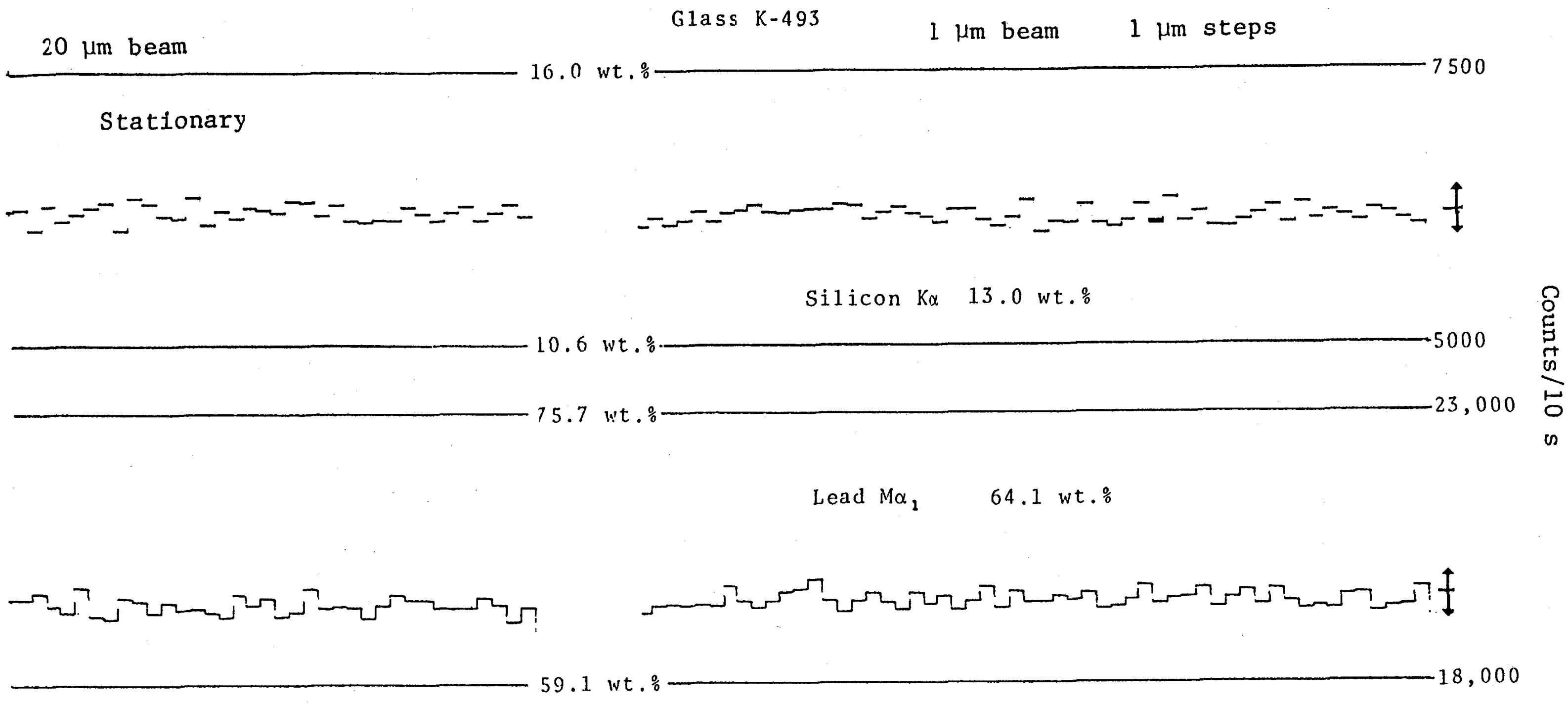

Figure 1. Periodic integrator homogeneity traces of lead and silicon simultaneoulsy recorded from Glass K-493 (15 Kv, 7.5 × 10^{-8} A beam current). In the traces on the right, the specimen was advanced 1-µm under a 1-µm diameter electron beam after each 10-second counting period. To the left is a time-resolved trace taken from repeated 10-second counting periods on a stationary specimen with a 20 × 20 µm scanning raster. The double-headed arrows to the right represent a range of $\pm 3\sqrt{N}$ around the average number of counts per 10 seconds, N, for the entire trace.

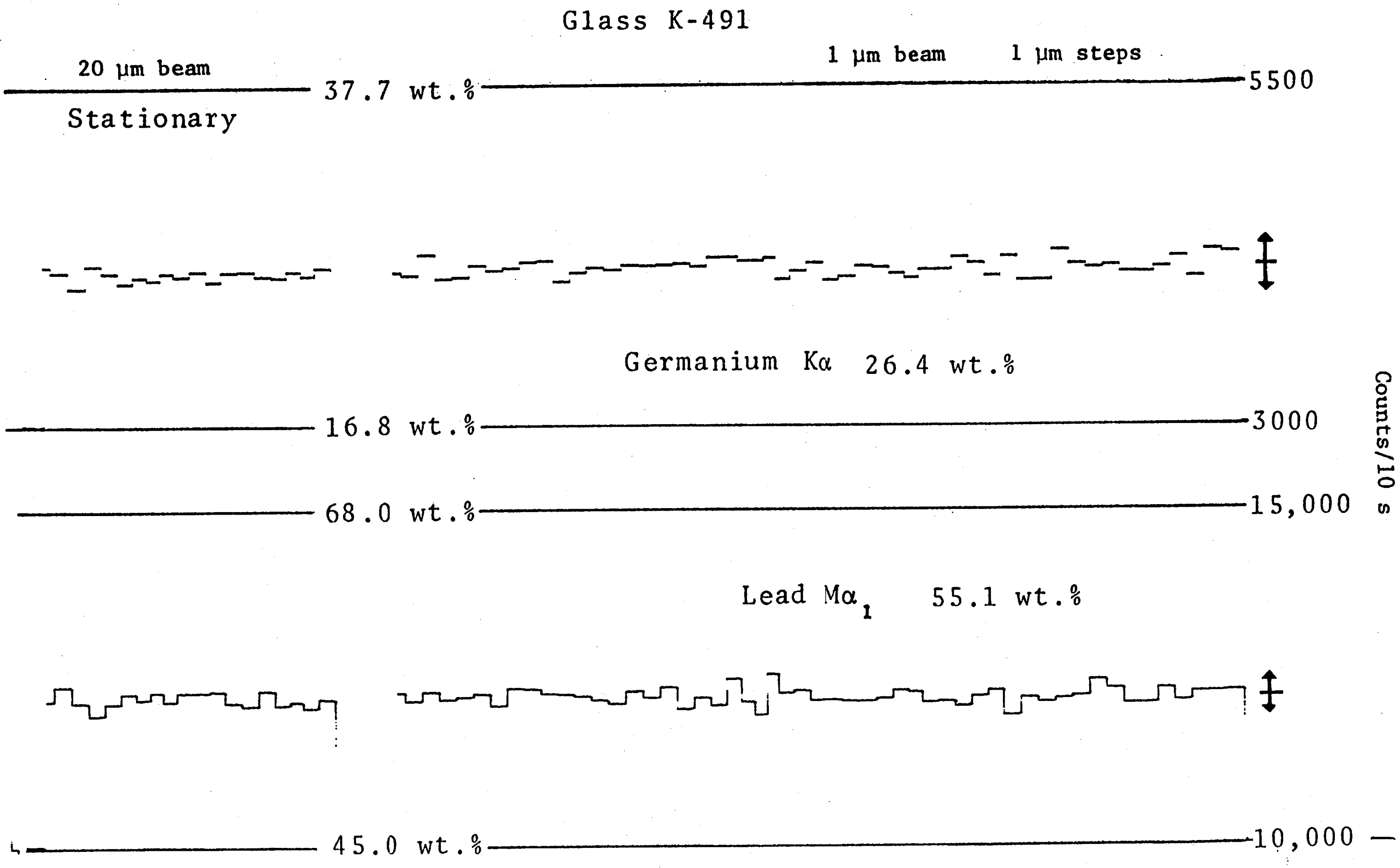

Figure 2. Periodic integrator homogeneity traces of germanium and lead simultaneoulsy recorded from Glass K-491 (15 Kv, 7.5 × 10^{-8} A beam current). In the traces on the right, the specimen was advanced 1-μm under a 1-μm diameter electron beam after each 10-second counting period. To the left is a time-resolved trace taken from repeated 10-second counting periods on a stationary specimen with a 20 × 20 μm scanning raster. The double-headed arrows to the right represent a range of $\pm 3\sqrt{N}$ around the average number of counts per 10 seconds, N, for the entire trace.

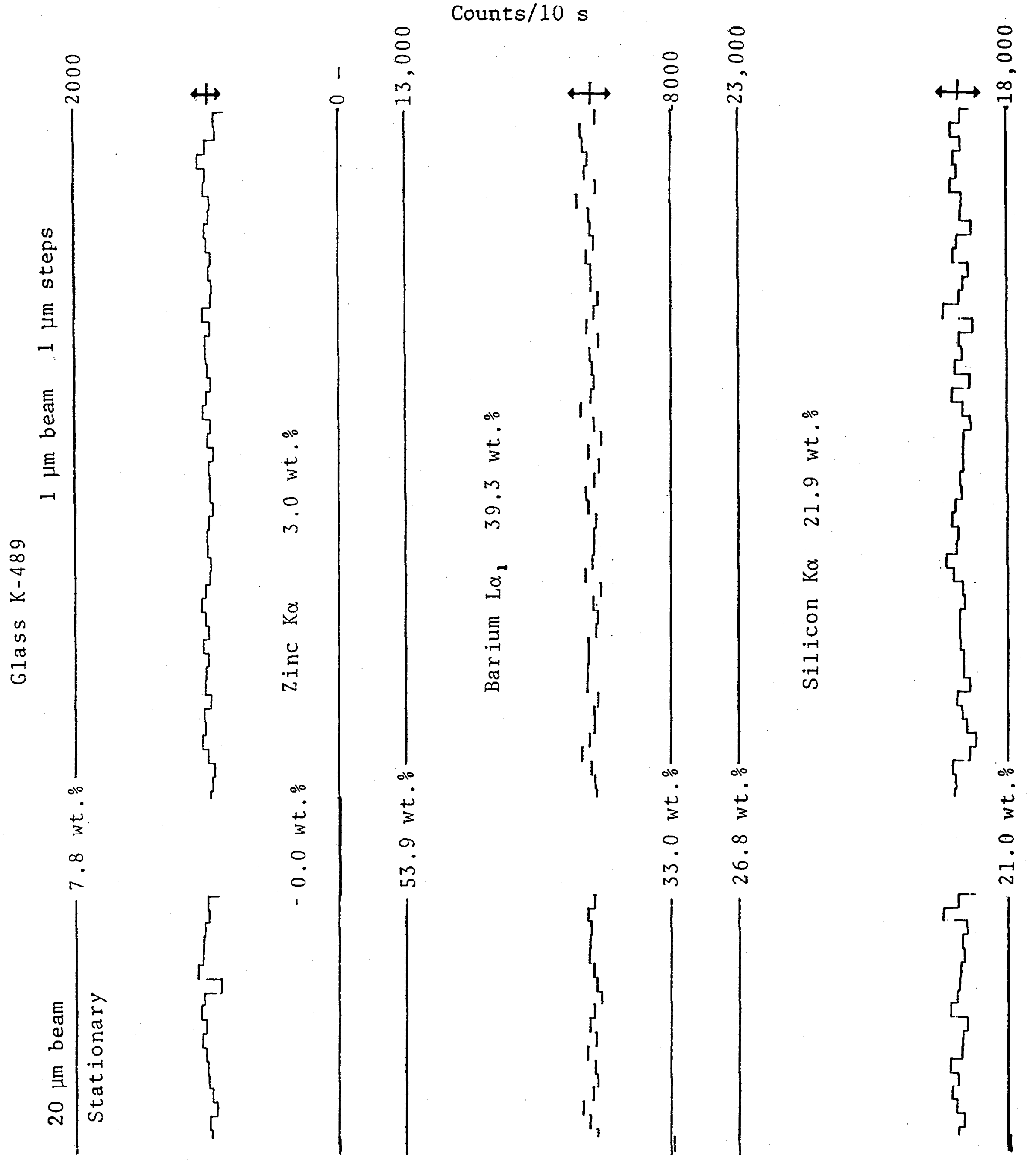

Figure 3. Periodic integrator homogeneity traces of silicon, barium and zinc simultaneoulsy recorded from Glass K-489 (15 Kv, 7.5 × 10^{-8} A beam current). In the traces on the right, the specimen was advanced 1-µm under a 1-µm diameter electron beam after each 10-second counting period. To the left is a time-resolved trace taken from repeated 10-second counting periods on a stationary specimen with a 20 × 20 µm scanning raster. The double-headed arrows to the right represent a range of $\pm 3\sqrt{N}$ around the average number of counts per 10 seconds, N, for the entire trace.

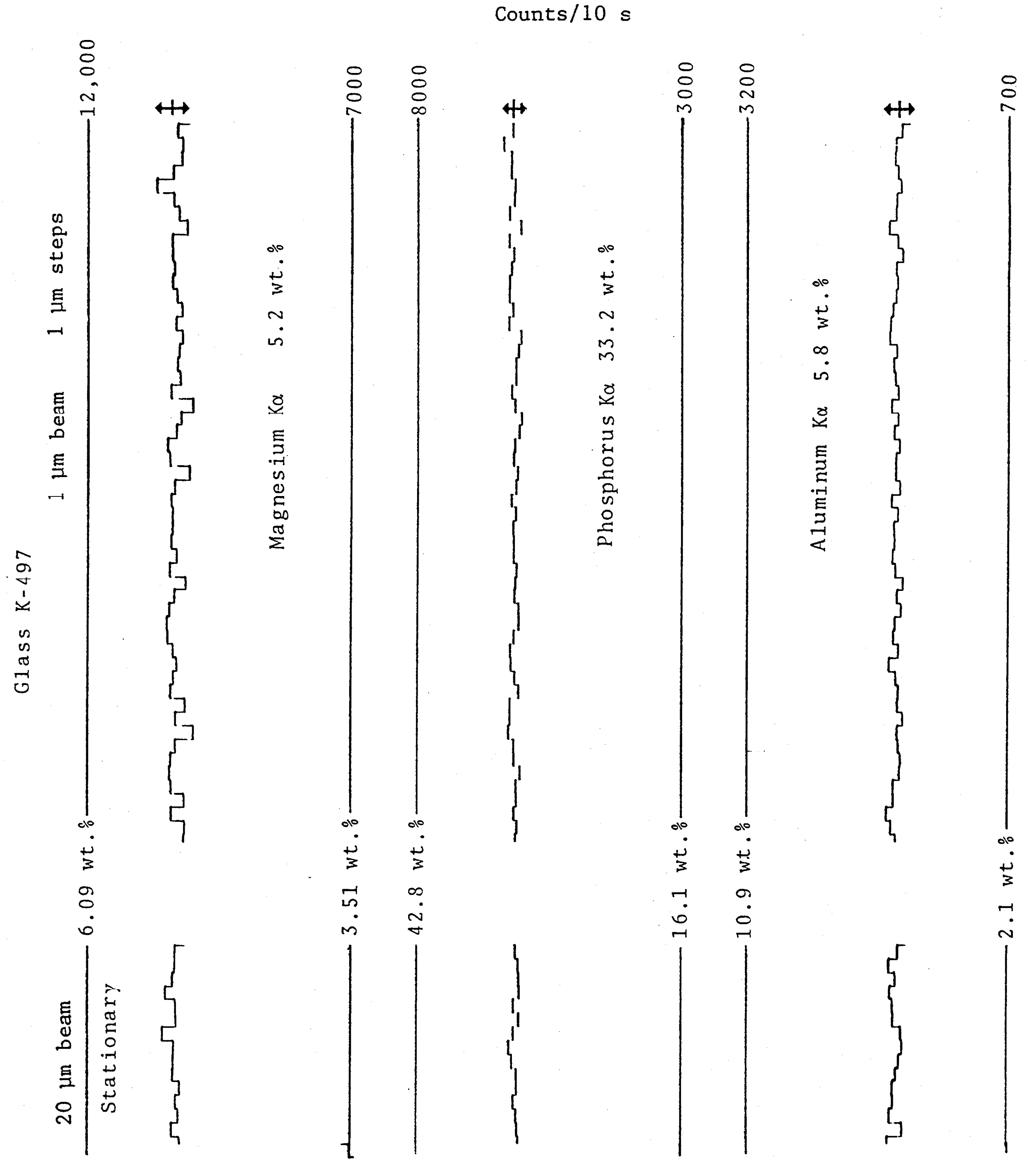

Figure 4. Periodic integrator homogeneity traces of magnesium, phosphorous and aluminum simultaneoulsy recorded from Glass K-497 (15 Kv, 7.5×10^{-8} A beam current). In the traces on the right, the specimen was advanced 1-µm under a 1-µm diameter electron beam after each 10-second counting period. To the left is a time-resolved trace taken from repeated 10-second counting periods on a stationary specimen with a 20 × 20 µm scanning raster. The double-headed arrows to the right represent a range of $\pm 3\sqrt{N}$ around the average number of counts per 10 seconds, N, for the entire trace.

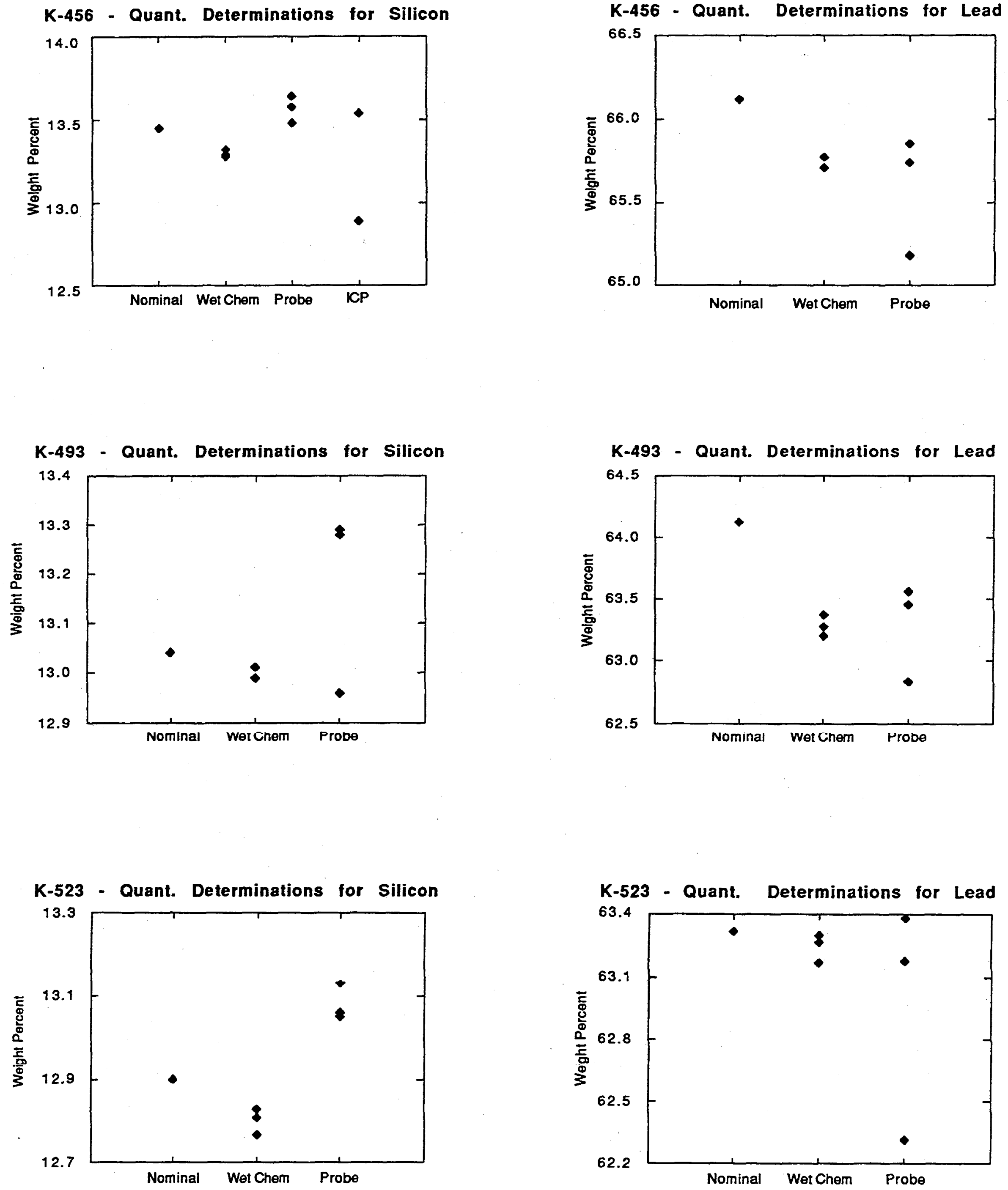

Figure 5. SRM 1871. Distribution plots of the results of wet chemistry, electron microprobe, and inductively coupled plasma spectrometry analyses compared to the nominal compositions.

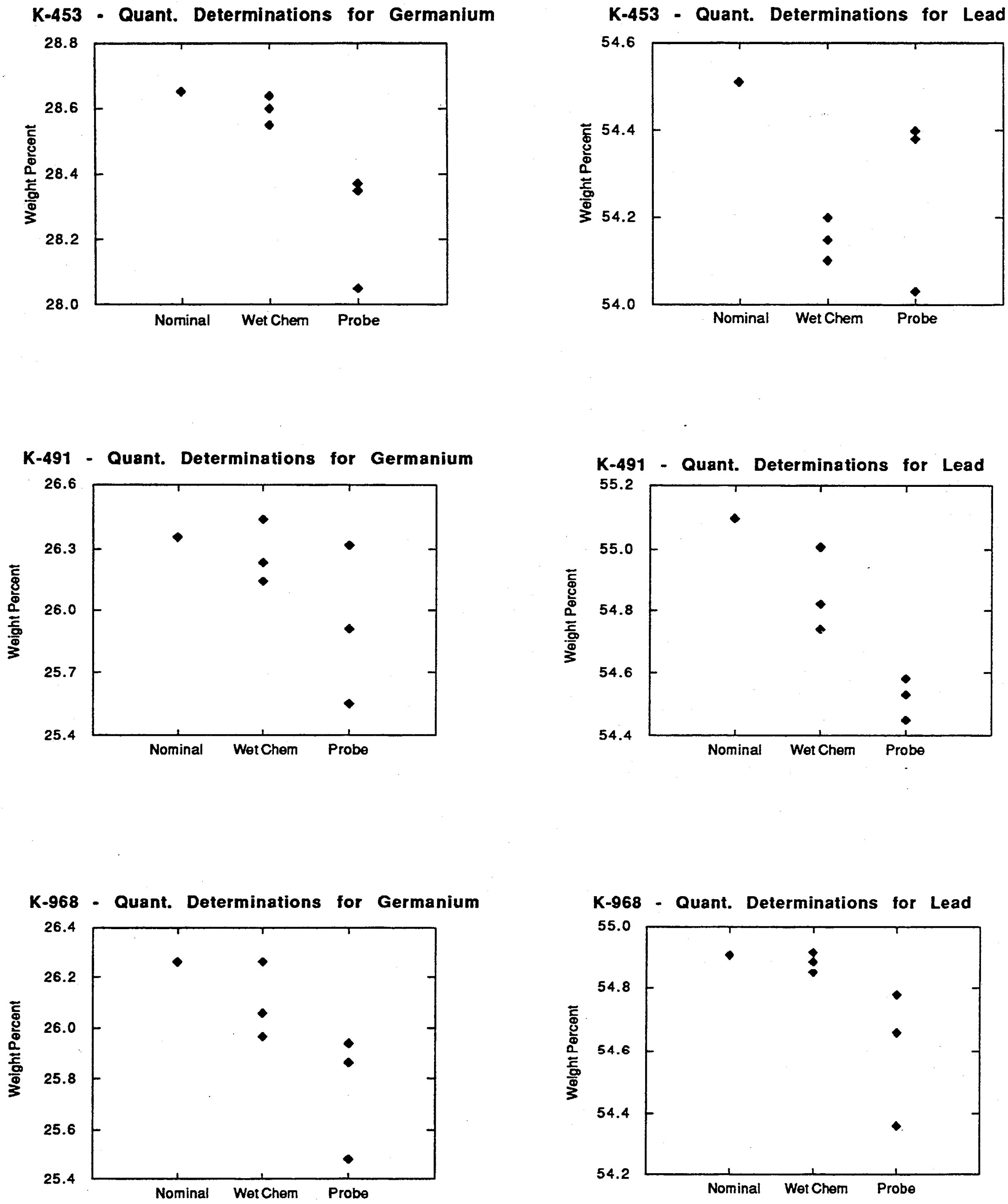

Figure 6. SRM 1872. Distribution plots of the results of wet chemistry and electron microprobe analyses compared to the nominal compositions.

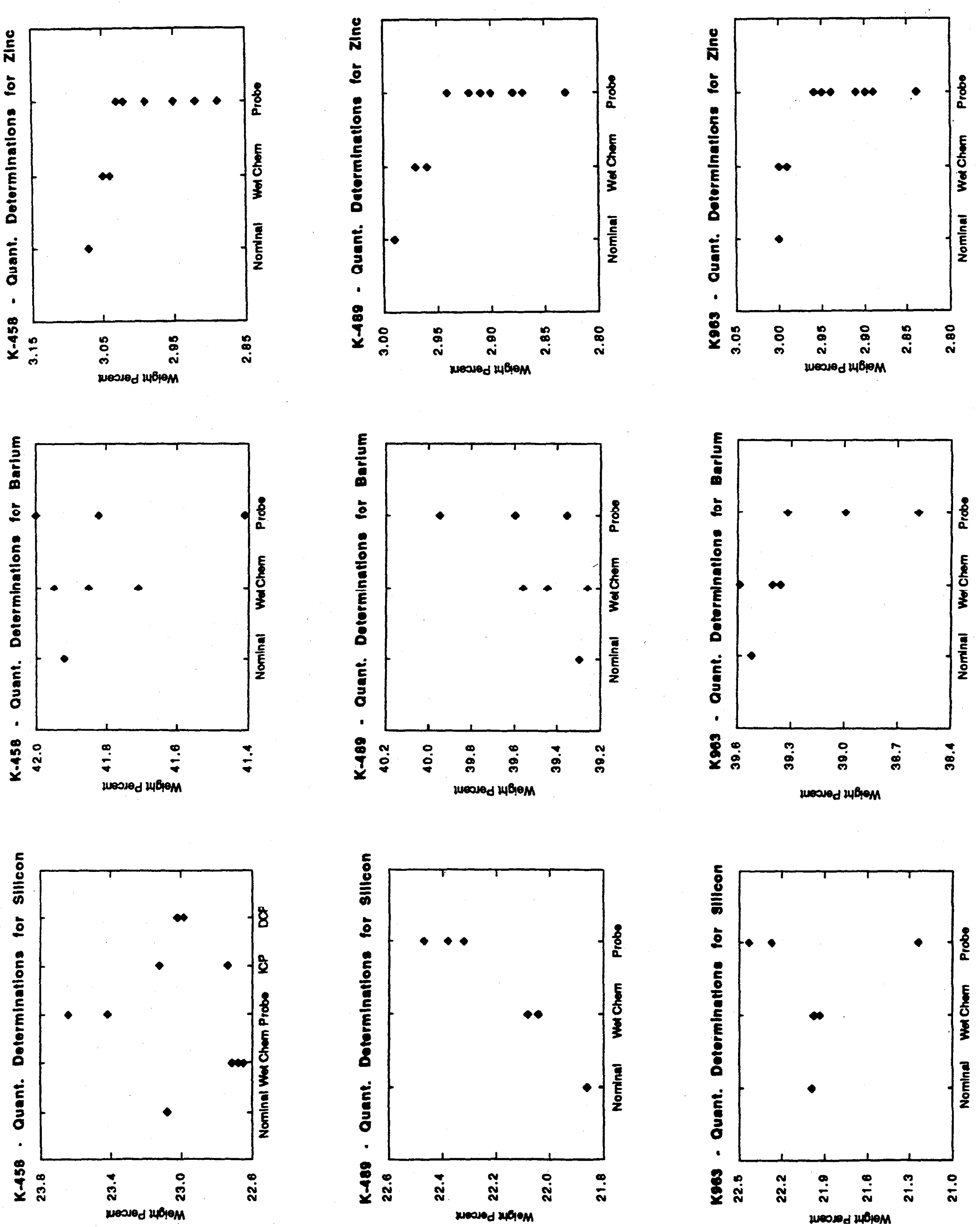

Figure 7. SRM 1873. Distribution plots of the results of wet chemistry, electron microprobe, inductively coupled and direct current plasma spectrometry analyses compared to the nominal compositions.

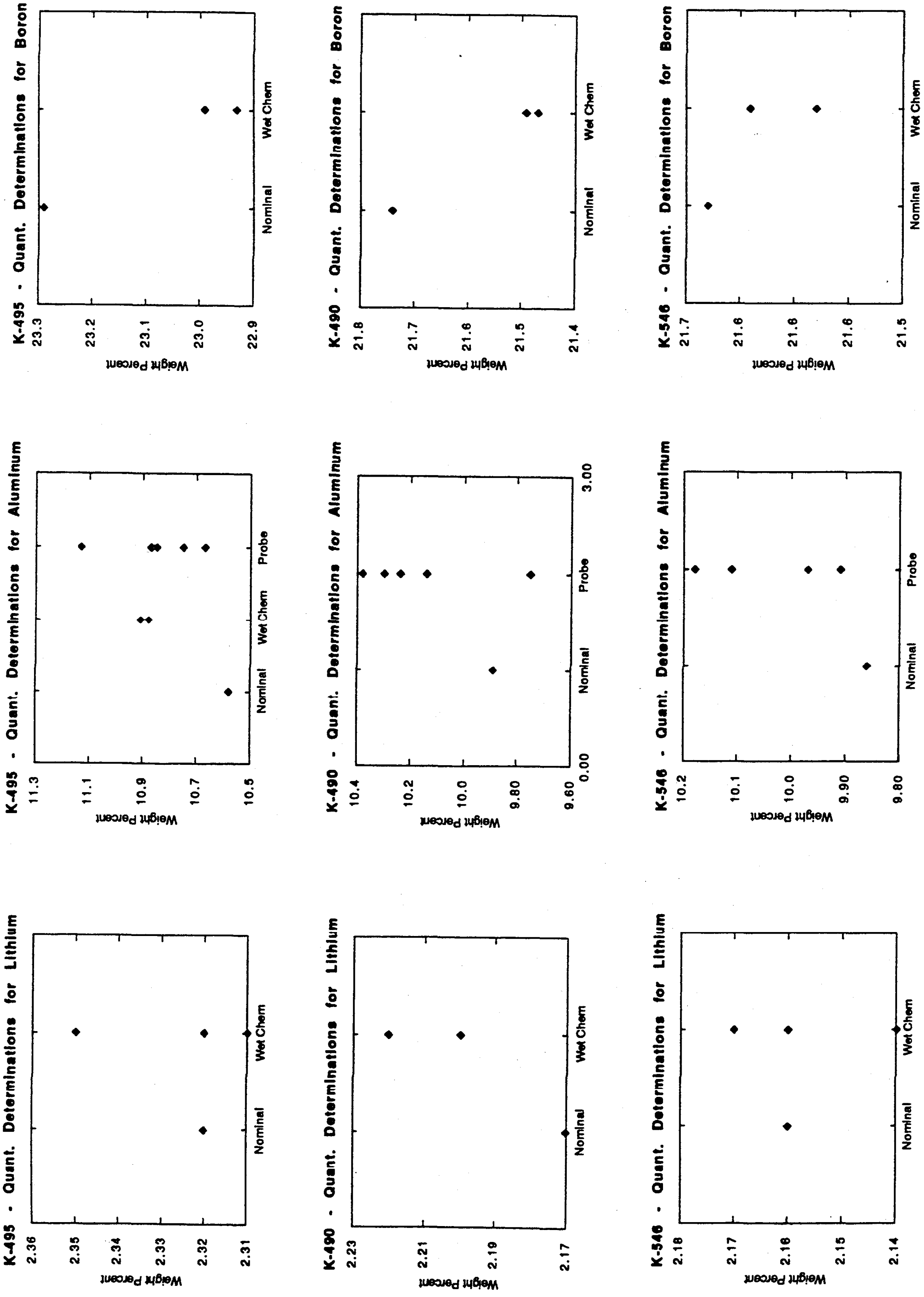

Figure 8. SRM 1874. Distribution plots of the results of wet chemistry and electron microprobe analyses compared to the nominal compositions.

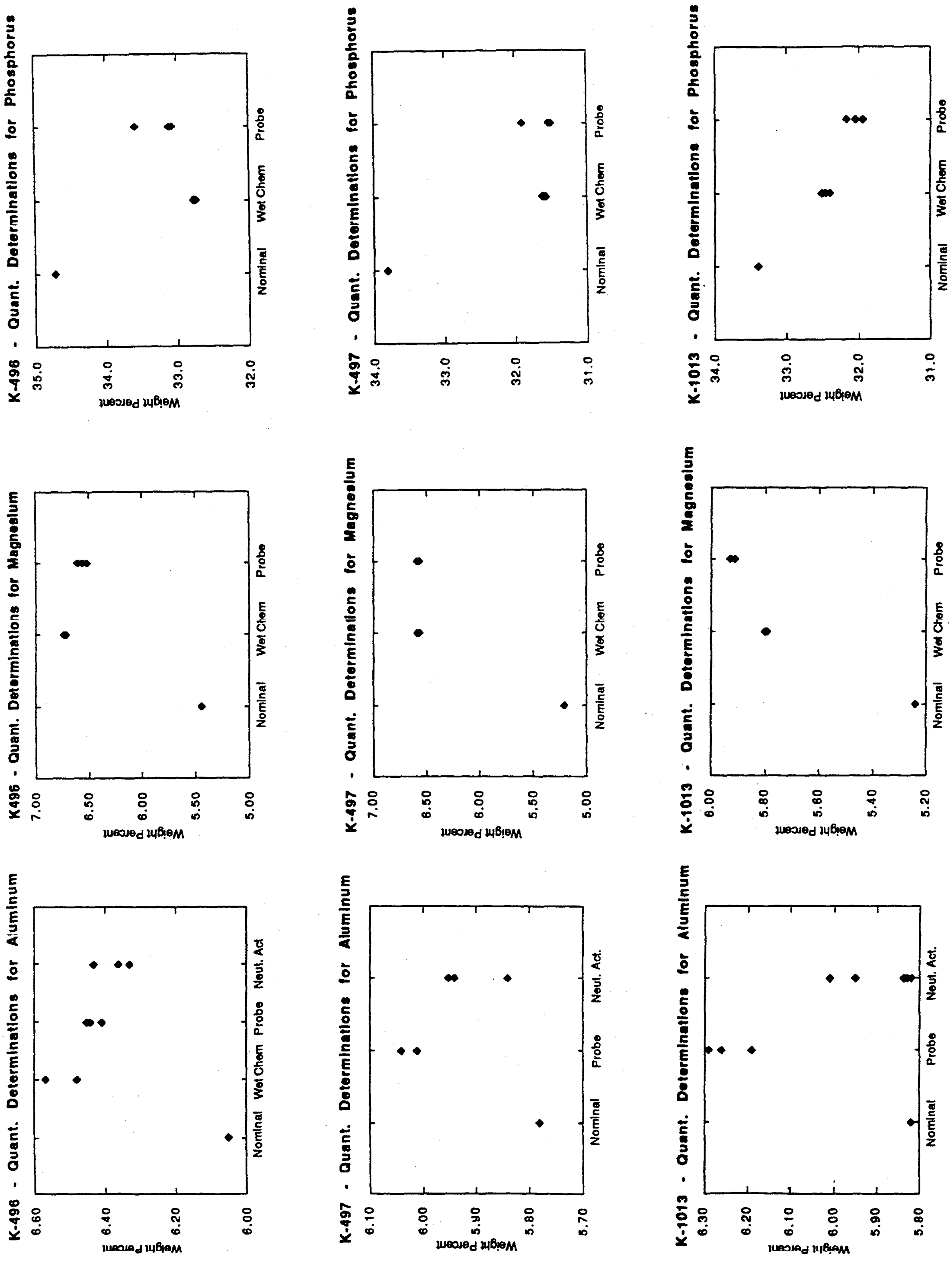

Figure 9. SRM 1875. Distribution plots of the results of wet chemistry, electron microprobe, and neutron activation analyses compared to the nominal compositions.

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ADDENDUM

The glasses were analyzed by R. L. Korotev, Senior Research Scientist, at Washington University, St. Louis, Missouri, using neutron activation analysis. The results of his analyses for several of the elements present in these glasses are listed in the table below. Trace amounts of other elements were also observed, but are not reported here. The complete report of these analyses can be obtained from R. Marinenko at NIST (Bldg.222 Rm. A113, Gaithersburg, MD. 20899).

Neutron Activation Analysis of Glasses for Microanalysis
(Washington University in St. Louis)
Concentration in weight percent

GLASS

Ele.	K-458	K-493	K-491	K-489	K-490*	K-497
Fe		0.25±0.10	<0.34	<0.77	<0.32	
Zn	3.00±.04			3.56±0.07		
Zr		0.37±0.08	0.24±0.06	0.47±0.10	0.19±0.07	0.43±0.12
Ba	40.1±0.60			3.79±1.1		
Ce		0.344±0.006	0.259±0.005	0.663±0.012	0.326±.005	0.602±0.011
Ta		0.78±0.04	0.63±0.03	1.13±0.05	0.71±0.03	0.89±0.04

GLASS

Ele.	K-523	K-968	K-963	K-546*	K-1013
Cr	0.184±0.006	0.154±0.005	0.296±0.009	0.0508±0.0017	0.219±0.007
Zn			2.92±0.06		
Ba	0.61±0.15	0.33±0.11	36.40±1.80	0.50±0.09	0.68±0.25
Eu	0.563±0.016	0.444±0.012	0.82±0.02	0.347±0.007	0.652±0.018
Th	0.078±0.003	0.0621±0.002	0.125±0.004	0.077±0.0017	0.097±0.003
U	0.103±0.003	0.082±0.003	0.175±0.007	0.153±0.003	0.134±0.004

Uncertainties are one standard deviation estimates of analytical precision (counting statistics). Single samples were analyzed and specimen sizes were 12-36 mg.

* Because of flux loss resulting from the high boron concentrations in these samples, the listed values are low by a factor of up to 2-3; this has been confirmed[a] by neutron self-shielding calculations[b] which predicted a flux loss of 50 percent.

[a] R. F. Fleming and R. M. Lindstrom, private communication

[b] R. F. Fleming, Neutron Self-shielding Factors for Simple Geometries, Int. J. Appl. Radiat. Isot. Vol. 33, pp 1263-8, 1982.

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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

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. Interspecimen 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.

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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. Technical Note 769, (1973).

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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 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 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 ± .26	54.69 ± .26	54.74 ± .26
O	(16.73)	(16.45)	(16.67)
Mg			(0.22) [0.08]
Al		(0.10) [0.09]	
Si		(.11) [.09]	
P			(.21) [.20]
Ti		(.14) [.16]	(.16) [.16]
Cr			(.19) [.17]
Fe		(.17) [.18]	
Ni			(.20) [.19]
Zr		(.26) [.30]	(.48) [.30]
Ba			(.46) [.45]
Ce		(.59) [.46]	
Eu			(.64) [.49]
Ta		(.52) [.59]*	
Th			(.12) [.08]
U			(.05) [.09]
Total	(99.37)	(99.13)	(100.07)

*Neutron activation: Ta = (0.58)

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May 1, 1984

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Office of Standard Reference Materials

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.[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 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 percent 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 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 and B. Takano 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 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.

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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. Technical Note 769, (1973).

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Standard Reference Material 1873

Barium-Zinc-Silicate Glasses for Microanalysis

K-458, K-489, and K-963

This Standard Reference Material (SRM) is intended primarily for the analysis of glasses, ceramics, and minerals by microanalytical techniques. It consists of three different barium-zinc-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. Barium and zinc are certified in all three glasses, while silicon is certified in glass K-458 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-458	K-489	K-963
Ba	41.79 ± 0.20	39.53 ± 0.20	39.21 ± 0.48
Zn	3.01 ± .06	2.93 ± .06	2.95 ± .06
Si	23.05 ± .34	(22.23 ± .34)	(21.96 ± .34)
O	(31.86)	(31.70)	(32.00)
Mg			(0.34) [0.14]
Al		(0.11) [0.15]	
P			(.33) [.36]
Ti		(.27) [.28]	(.32) [.36]
Cr			(.31) [.30]
Fe		(.35) [.32]	
Ni			(.33) [.34]
Ge			(.47) [.42]
Zr		(.40) [.52]	(.61) [.53]
Ce		[.80]	
Eu			(.95) [.88]
Ta		(.95) [1.03]*	
Pb		(1.32) [1.19]	
Th			(.06) [.13]
U			(.16) [.17]
Total	(99.71)	(100.59)	(100.00)

*Neutron activation: Ta = (1.03)

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May 1, 1984

Stanley D. Rasberry, Chief
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Barium, zinc, and silicon values were determined by wet chemical analysis and EPMA. In addition, plasma emission spectrometry (inductively coupled and direct current plasma spectrometry) were used to determine silicon in glass K-458. The certified values and the silicon information values 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. For zinc and silicon, a pooled standard deviation was then obtained for each element by combining the standard deviations from all three glasses. For barium, a pooled error was determined from glasses K-458 and K-489, as the error for K-963 was quite different from the other two glasses. The error cited in the table is two times the pooled (or individual) standard deviation of the certified value.

The dopant elements, present as oxides in concentrations of 2 percent or less in glasses K-489 and K-963, 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-489 was also determined by neutron activation analysis.

The glasses were tested for microhomogeneity using periodic integrator traces and random sampling techniques. Interspecimen 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 and B. Takano under the direction of N.H. Suhr, Pennsylvania State University, University Park, Pa.

The inductively coupled and direct plasma spectrometry was performed by R.L. Watters and M.S. Epstein, respectively, 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.

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

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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
December 28, 1984

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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.

U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

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).

Gaithersburg, MD 20899
December 28, 1984

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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, ±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.

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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.

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U.S. DEPARTMENT OF COMMERCE
National Bureau of Standards

Standard Reference Materials 1871–1875

Glasses for Microchemical Analysis

Fall 1984

The NBS Office of Standard Reference Materials announces a new series of Standard Reference Materials (SRM's) for use in microanalytical techniques such as electron-probe microanalysis and secondary-ion mass spectrometry. These SRM's, 1871-1875, are glasses that show no serious inhomogeneities on the micrometer scale for the certified constituents. The individual glasses are in rod form, approximately 2 x 2 x 20 mm. Each SRM represents a different glass matrix, and in each SRM there is a base-composition glass plus two glasses of the same base composition to which several dopant elements have been added. The different matrices and dopants make these glasses useful in the analysis of complex unknowns.

The compositions of the fifteen glasses in this SRM series are listed in the attached table. The values for the currently certified elements are not enclosed in either parentheses or brackets. Those enclosed in parentheses are informational values,while those in brackets are nominal values calculated from the weighed material added to the glass melt during preparation.

These SRM's may be purchased for $196.00 per set from:

Office of Standard Reference Materials
Room B311 Chemistry Building
National Bureau of Standards
Gaithersburg, MD 20899
Telephone: (301) 921-2045

1084

	SRM 1871			SRM 1872			SRM 1873			SRM 1874			SRM 1875		
	Glass			Glass			Glass			Glass			Glass		
	K-456	K-493	K-523	K-453	K-491	K-968	K-458	K-489	K-963	K-495	K-490	K-546	K-496	K-497	K-1013
Elem.	(Wt. %)			(Wt. %)			(Wt. %)			(Wt. %)			(Wt. %)		
Pb	65.67	63.28	63.10	54.21	54.69	54.74	-------	(1.32)	-------	-------	(1.47)	-------	-------	(0.86)	-------
Si	13.37	(13.09)	(12.94)	-------	(0.11)	-------	23.05	(22.23)	(21.96)	-------	(0.19)	-------	-------	(0.13)	-------
Ge	-------	-------	(0.24)	28.43	26.10	25.93	-------	-------	(0.39)	-------	-------	(0.50)	-------	-------	(0.34)
Ba	-------	-------	(0.61)	-------	-------	(0.46)	41.79	39.53	39.21	-------	-------	(0.99)	-------	-------	(0.52)
Zn	-------	-------	-------	-------	-------	-------	3.01	2.93	2.95	-------	-------	-------	-------	-------	-------
P	-------	-------	(0.24)	-------	-------	(0.21)	-------	-------	(0.33)	-------	-------	(0.42)	32.98	31.59	32.26
Mg	-------	-------	(0.12)	-------	-------	(0.22)	-------	-------	(0.34)	-------	-------	(0.17)	6.65	6.49	5.86
Al	-------	(0.13)	-------	-------	(0.10)	-------	-------	(0.11)	-------	10.89	(10.2)	(10.1)	6.47	5.97	6.08
B	-------	[0.04]	-------	-------	[0.03]	-------	-------	[0.06]	-------	(23.0)	(21.5)	(21.6)	-------	[0.05]	-------
Zr	-------	(0.38)	(0.33)	-------	(0.26)	(0.48)	-------	(0.40)	(0.61)	-------	(0.53)	(0.52)	-------	(0.32)	(0.45)
Ti	-------	(0.20)	(0.21)	-------	(0.14)	(0.16)	-------	(0.27)	(0.32)	-------	(0.31)	(0.39)	-------	(0.22)	(0.21)
Ce	-------	(0.53)	-------	-------	(0.59)	-------	-------	[0.80]	-------	-------	(1.46)	-------	-------	(0.94)	-------
Ta	-------	(0.64)	-------	-------	(0.52)	-------	-------	(0.95)	-------	-------	(1.02)	-------	-------	(0.71)	-------
Fe	-------	(0.25)	-------	-------	(0.17)	-------	-------	(0.35)	-------	-------	(0.38)	-------	-------	(0.26)	-------
Li	-------	[0.0005]	-------	-------	[0.0005]	-------	-------	[0.0009]	-------	(2.3)	(2.2)	(2.2)	-------	[0.0005]	-------
Ni	-------	-------	(0.25)	-------	-------	(0.20)	-------	-------	(0.33)	-------	-------	(0.39)	-------	-------	(0.31)
Eu	-------	-------	(0.73)	-------	-------	(0.64)	-------	-------	(0.95)	-------	-------	(1.21)	-------	-------	(0.53)
U	-------	-------	(0.23)	-------	-------	(0.05)	-------	-------	(0.16)	-------	-------	(0.24)	-------	-------	(0.15)
Th	-------	-------	(0.08)	-------	-------	(0.12)	-------	-------	(0.06)	-------	-------	(0.16)	-------	-------	(0.10)
Cr	-------	-------	(0.20)	-------	-------	(0.19)	-------	-------	(0.31)	-------	-------	(0.14)	-------	-------	(0.14)
O	(20.35)	(20.58)	(20.82)	(16.73)	(16.45)	(16.67)	(31.86)	(31.84)	(31.96)	(63.49)	(60.75)	(61.36)	(53.90)*	(52.46)*	(53.05)*
Total	(99.39)	(99.12)	(100.10)	(99.37)	(99.16)	(100.07)	(99.71)	(100.79)	(99.88)	(99.68)	(100.01)	(100.39)	(100.00)	(100.00)	(100.00)

Values in parentheses are for information only, they are *not certified.*

Values in brackets were calculated from the weight of material added to the melt, they are *not certified.*

*Oxygen values in SRM 1875 were calculated by difference, not by the stoichiometry of the oxides as was done for the other glasses.