Application Note #AA3001A

BUCK Scientific

Sample Preparation of Glass and Ceramic Materials for Atomic Absorption Analysis

The analysis of silicate-based glass and ceramics has always been a moderately difficult job, since the components that comprise the glass are usually not stable together in solution. For example, most typical acidic digestions that solubilize silicon will cause calcium fluoride to precipitate. If no fluoride is present, the silicon will not be fully decomposed, and if the solution is heated, boron can be lost, etc.

There are several industry standard procedures that are used for specific elements, thereby requiring at least two (2) preparations to be used for the complete, reproducible analysis of each sample. If only certain components are important, then perhaps a single preparation can be employed. Listed below are some methods developed at Buck Scientific for use on the Model 210VGP Atomic Absorption Spectrophotometers.

[1] Fuse 0.2-1.0 grams of powdered glass with 5x the amount of a mixed lithium carbonate/zinc oxide flux (2:1) for 10-15 minutes at 900-950^{o}C in an Ni or Pt crucible. Cool and dissolve the melt in hydrochloric acid (1:1) with warming. The resultant solution is quantitatively diluted, filtered to remove insoluble silica, and analyzed for Ca, Mg, Al, B, Na, K, Fe, Cu, and Mn. If Cr, V, Ti or Mo are to be determined, the flux should contain ~1/2 part sodium peroxide, or the "melt" can be dissolved in 25% hydrochloric acid and 10% hydrogen peroxide with satisfactory results.

[2] Fuse 0.2-1.0 grams of powdered glass with ~7x the amount of either sodium hydroxide or potassium hydroxide for 10-30 minutes at 400-500^{o}C in an Ni or Pt crucible. The melt is cooled, dissolved in water, and promptly analyzed for Si, B, Mo, Ti, V, Zn, halogens, P, and Al.

[3] Moisten 0.1-0.5 grams of powdered glass with 1 ml of alcohol in a Pt or PtFe dish, add ~5 ml water, 1 ml perchloric acid, 1 ml sulfuric acid, and ~10 ml hydrofluoric acid. Digest on a steam bath or hot plate until fumes are evolved. Add 1 ml sulfuric acid, 10 ml hydrofluoric acid, and 2-10 ml methyl alcohol. Take to near dryness (sulfuric acid minimizes undesirable reactions from perchloric acid). Add 5-20 ml of hydrochloric acid, dilute, and analyze. This procedure allows the accurate determination of almost all elements except Si and B. Adding 0.1-0.2% lanthanum or strontium will improve the response for Al, Ca, and Mg.

The Buck 210VGP series can accurately determine these major and minor elements in ranges from 0.005% to over 20%, depending on the dilution used, with typical precision of 1.5% or better if using bracketed standards. The entire procedure, from preparation of sample to final analysis, can be standardized to a simple format so that a novice lab technician can perform the test easily.

Analyst: Gerald J. DeMenna

SIC: 321, 322, 3253, 326

Analytical Results for Standard Glass Samples

Samples: Standard Reference Materials from NIST/NBS, ASTM and BCS
Preparation: Method #2 for Si, Al, Zn & B; #3 for the others
Calibration: Buck Certified Atomic Absorption Standards
Instrument: The Buck 210VGP Atomic Absorption Spectrophotometer
Conditions: Air/Acetylene flame, N_2O/Acetylene flame, Integrate mode, Normal parameters
[Potassium (K) and Sodium (Na) are done in **emission** mode]

NOTE:All values listed below are in **Weight Percent** (%wt) in the **original** glass sample; **[K]**=known assay, **[M]**=measured data.

	Soda-Lime (green)		Borosilicate (hard, Hi-B)		Lead "Crystal" (tinted)	
Element	**[K]**	**[M]**	**[K]**	**[M]**	**[K]**	**[M]**
[#2 prep]						
Si	34.2	34.0	37.8	37.2	30.6	30.9
Al	0.17	0.15	1.21	1.24	0.09	0.09 *
Zn	0.18	0.19	.011	.009	1.11	1.09 #
B	1.42	1.38	3.93	3.90	0.05	0.04
[#3 prep]						
Na	10.0	10.3	2.97	3.08	4.22	4.25
K	0.49	0.48	.012	.011	6.97	7.03
Zn	0.16	0.18	.006	.007	1.07	1.04 #
Ca	6.15	5.97	.007	.009	0.28	0.30
Mg	2.35	2.40	.008	.008	0.05	0.04
Mn	.015	.016	.004	.005	0.11	0.12
Ba	0.17	0.15	4.77	4.61	1.38	1.42
Al	0.15	0.14	1.22	1.20	0.08	0.07 *
Pb	<0.01	<0.01	<0.01	<0.01	16.3	16.1
Cu	0.08	0.09	.002	.002	0.16	0.15
Fe	4.08	3.95	.024	.022	0.41	0.42

The above data shows the powerful flexibility and stability of the Buck 210VGP system for the wide-ranging requirements of the glass and ceramics industry. The excellent correlation between the known and measured values ranges from 0.6% to 3.0% (average RSD = 1.6%) demonstrates the precision of the instrument. The fact that two different preparations gave superb reproducibility on two difficult elements, Zn (#) and Al (*), reveals the high accuracy of the chemistry.

Basic System: $12,950.00

Turnkey System: $18,160.00 Includes: All recommended lamps, standards, and accessories for normal operation.

For detailed configuration, refer to Quote #AA4001A

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BUCK Scientific

Application Note
#AA3001B

BUCK Scientific

Sample Preparation of Glass and Ceramic Materials for Atomic Absorption Analysis

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[1] Fuse 0.2-1.0 grams of powdered glass with 5x the amount of a mixed lithium carbonate/zinc oxide flux (2:1) for 10-15 minutes at 900-950^{o}C in an Ni or Pt crucible. Cool and dissolve the melt in hydrochloric acid (1:1) with warming. The resultant solution is quantitatively diluted, filtered to remove insoluble silica, and analyzed for Ca, Mg, Al, B, Na, K, Fe, Cu, and Mn. If Cr, V, Ti or Mo are to be determined, the flux should contain ~1/2 part sodium peroxide, or the "melt" can be dissolved in 25% hydrochloric acid and 10% hydrogen peroxide with satisfactory results.

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