



ISO 17025

Testing Cert. #2797.01

**X-RAY FLUORESCENCE (XRF)
ANALYSIS REPORT
22 Mar 2013**

**JOB NUMBER C0DHZ309
PO NUMBER**

for

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XRF ANALYSIS REPORT

Requester: Joe Cerniglia
Job Number: C0DHZ309
Analysis Date: 22 Mar 2013

Purpose:

The purpose of this analysis was to determine if a glass shard contains high levels of fluorine, which would identify it as a fluoride glass.

Summary:

The glass composition is summarized in [Table 1](#). Fluorine was not detected. Based on the estimated detection limit of 0.2 wt% for F, the sample is not consistent with a fluoride glass. The composition is consistent with a soda lime or possibly a borosilicate glass.

Experimental:

X-ray Fluorescence (XRF) is a non-destructive technique that can identify and quantify the elemental constituents of a sample using the secondary fluorescence signal produced by irradiation with high energy x-rays. This analysis utilized a wavelength dispersive spectrometer (WDXRF) that is capable of detecting elements from atomic number (Z) 4 (beryllium) through atomic number 92 (uranium) at concentrations from the low parts per million (ppm) range up to 100% by weight.

Analytical Parameters

| | |
|----------------------|------------------------|
| Instrument | Rigaku Primus II WDXRF |
| X-ray source | Rhodium x-ray tube |
| Atmosphere | Vacuum |
| Analysis area | 1mm diameter |

Quantification was performed using the Fundamental Parameters (FP) standardless quantification software associated with the system. The fundamental parameters approach uses x-ray physics coupled with established sensitivity factors for pure elements. Relative accuracy by this method usually ranges from better than 5% up to ~20% for major elements.

Data were collected in both scanned and fixed angle modes. In normal scanning mode analysis, the detector is rotated through the full range of dispersion to collect the diffracted x-ray signals coming from the sample and spectra are generated. In a fixed angle analysis the detector dwells at a specific angle for an extended period while collecting a signal only from the peak maximum and from the adjacent background. Fixed angle analysis significantly improves sensitivities. Since a fixed angle analysis collects intensities from only two points (peak maximum and baseline) no spectra are created.

Results and Interpretations:

Spectra are included in the attached figures. Sample or area names are provided on the spectra. The results are summarized in [Table 1](#).

Fluorine was not detected. Based on the estimated detection limit of 0.2 wt% for F, the sample is not consistent with a fluoride glass.

The high levels of both Na (Na₂O) and Ca (CaO) are most consistent with a soda lime glass; although a considerable amount of boron (B₂O₃) was also tentatively identified. The presence of boron at high levels *may* indicate a sodium borosilicate glass. However, it must be noted that detection and quantification of boron under these analysis conditions (1mm small spot XRF) is very difficult.

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<http://www.eaglabs.com/main-survey.html?job=C0DHZ309>

Table 1. Glass Composition (in Wt%)^a

| Element | Conc. | | Element | Conc. |
|--------------------------------|------------------|--|--------------------------------|-------|
| B ₂ O ₃ | 3.5 ^b | | CaO | 5.7 |
| F | - ^c | | Cr ₂ O ₃ | 0.12 |
| Na ₂ O | 10.5 | | Fe ₂ O ₃ | 0.27 |
| MgO | 2.7 | | NiO | 0.032 |
| Al ₂ O ₃ | 1.0 | | SrO | 0.019 |
| SiO ₂ | 74.6 | | ZrO ₂ | 0.016 |
| K ₂ O | 0.45 | | BaO | 1.0 |

^a The results are normalized to 100% of the measured and detected elements and converted to oxides

^b Boron is difficult to detect under these analytical conditions. Therefore its presence even at the reported levels must be considered tentative.

^c The estimated detection limit for fluorine in this analysis is 0.2 wt%.

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XRF Analysis ReportPage 4 of 8
Job Number C0DHZ30922 Mar 2013
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Wavelength Dispersive X-ray Fluorescence Spectroscopy (WDXRF) Description Appendix

In XRF photons from an x-ray tube irradiate a sample causing the ejection of inner shell electrons from the excitation volume of the sample, creating inner shell vacancies. In order to reestablish a stable electron configuration, electrons from outer shells fill the inner shell vacancies. In this process fluorescent photons are produced to balance the energy difference between the outer and inner shells. These fluorescent x-rays are the source of the signal in x-ray fluorescence spectroscopy, and their energies are characteristic of the atoms from which they originate. Therefore the fluorescent signal can determine the elements present in the sample matrix and, from the relative intensities, the concentrations. By using an appropriate elemental and matrix reference standard, or fundamental parameter algorithms when standards are unavailable, accurate quantification of the elemental make-up of the sample can be obtained. With appropriate standards accuracies can be better than 1% relative; while using the Fundamental Parameters method typically yields accuracies of better than 5% to ~20% relative for major elements. Long term measurement reproducibility is ~2% at the 95% confidence limit.

In a wavelength dispersive XRF spectrometer (WDXRF) the fluorescence signals from the sample are collimated, after which they impinge upon one or more crystals. Each signal is diffracted at a specific angle based on the lattice spacing of the crystal and the fluorescent photon energy, following Bragg's law. Wavelength dispersive systems are generally operated by sequentially scanning the detectors over the full dispersion range of one or more crystals to collect the elemental signals. The relative intensities of the signals are a function of the concentration of the element, matrix effects, and factors attributable to the primary x-ray radiation. The system used in this analysis is capable of detecting elements of atomic number (Z) 4 (beryllium) through atomic number 92 (uranium) at concentrations from the low parts per million (ppm) range to 100% by weight.

The excitation volume for XRF is both element and matrix dependent. It can range from the micrometer range for light elements in dense metallic materials to a centimeter or more for heavier elements in light element matrices such as polymers.

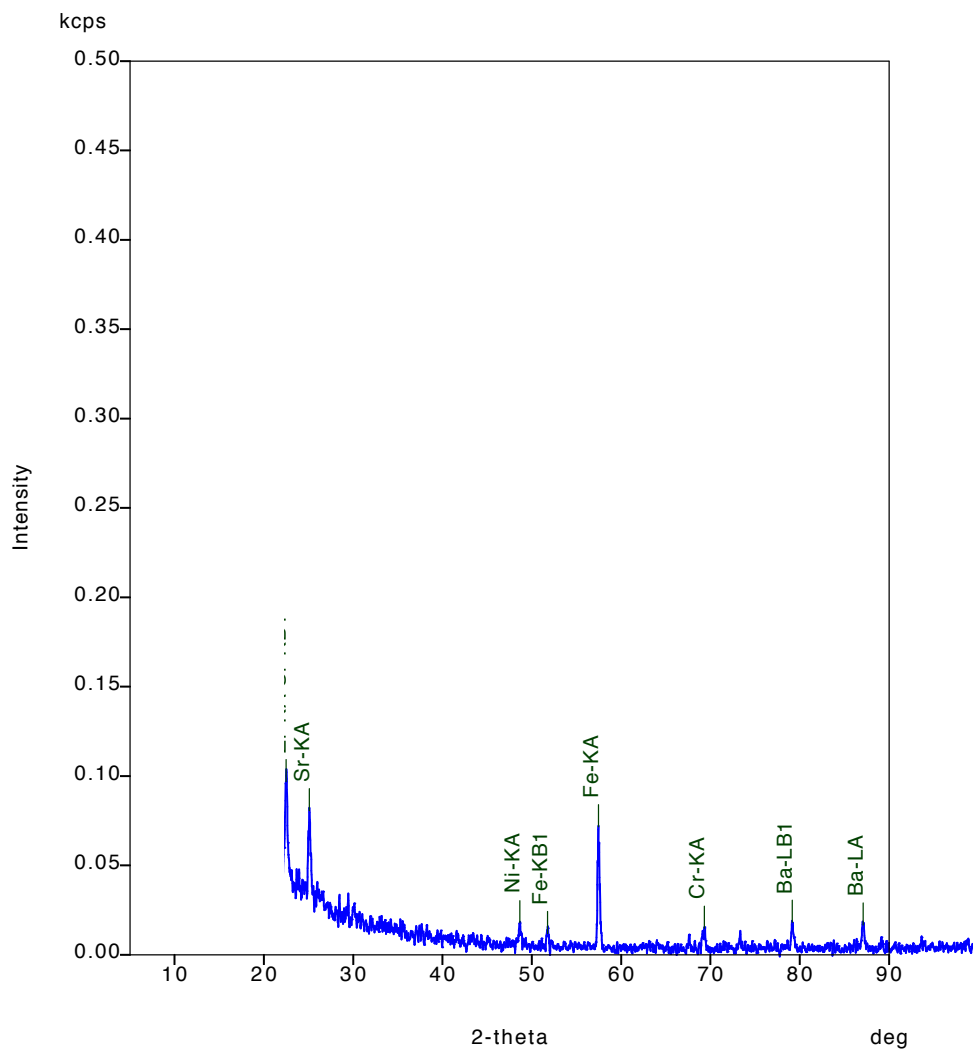


Figure 1

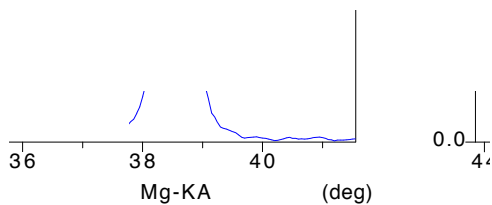


Figure 2

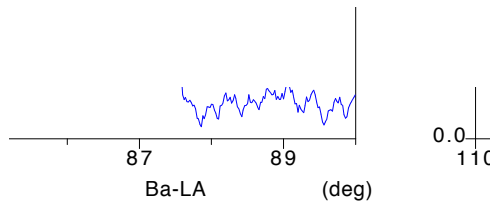
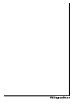


Figure 3