



Testing Cert. #2797.01

X-RAY FLUORESCENCE (XRF)
ANALYSIS REPORT
08 Dec 2015

JOB NUMBER C0FSD762 PO NUMBER Credit Card

for

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XRF ANALYSIS REPORT

Requester: Joshua Vojtisek Job Number: C0FSD762 Analysis Date: 08 Dec 2015

Purpose:

The purpose of this analysis was to determine the composition of a white solid suspended in water.

Summary:

The results are summarized in <u>Table 1</u>. The white solid is primarily zinc carbonate, ZnCO₃, along with trace levels of additional species.

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 10mm diameter

The sample was centrifuged to concentrate the solid, after which most of the water was removed. The remaining wet sample was dried in an oven. The dried powder was pressed into a pellet for analysis.

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 $\sim 20\%$ for major elements.

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.

The relative C, O and Zn concentrations are in very good agreement with ZnCO₃.

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

Element	Conc.	Element	Conc.
С	7.0	K	0.030
N	0.52	Ca	0.17
0	39.69	Mn	0.005
Mg	0.28	Fe	0.071
Al	0.11	Ni	0.006
Si	0.28	Cu	0.012
P	0.008	Zn	51.6
S	0.060	Zr	0.004
CI	0.17		

The results are normalized to 100% of the measured and detected elements Note: 1.0 wt%=10,000ppm

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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.

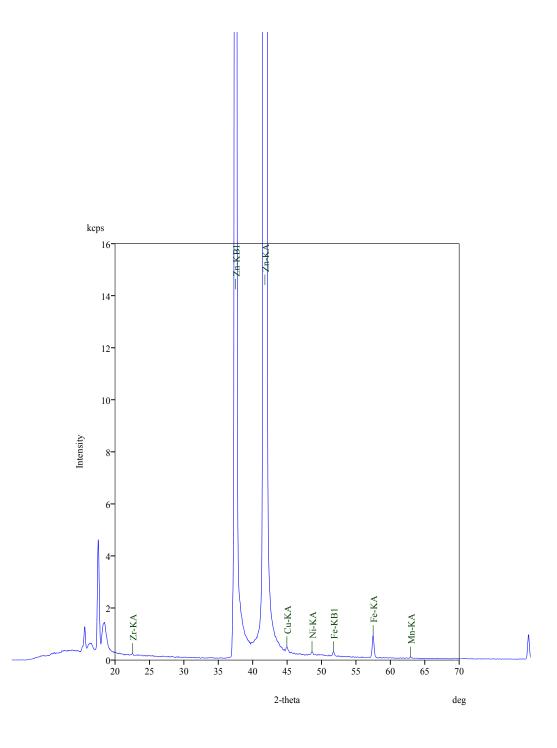


Figure 1

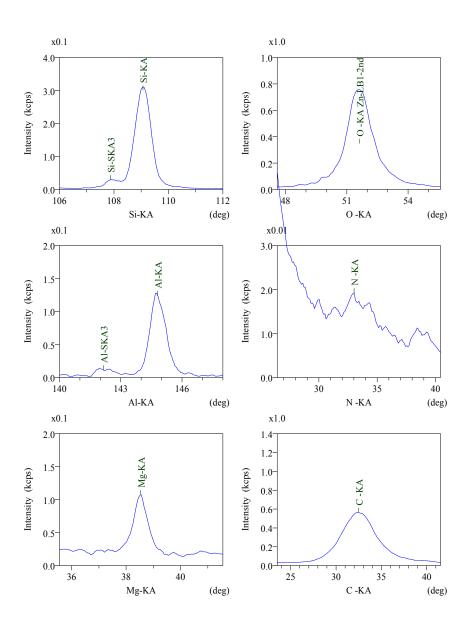


Figure 2

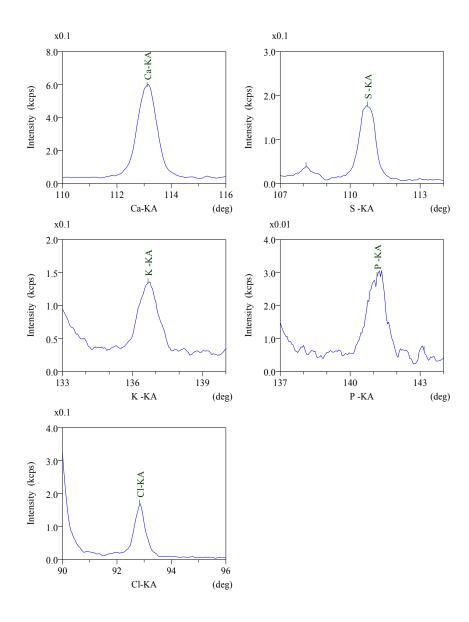


Figure 3