Application Note: 41661

Analysis of Coal Fly Ash

Thermo Scientific ARL PERFORM'X Series Advanced X-Ray Fluorescence Spectrometers

Key Words

- ARL PERFORM'X 4200 W
- Coal fly ash
- XRF
- X-ray fluorescence

Introduction

One of the most common source of energy production is coal fire power plants. These plants use millions of tons of coal each year. During the coal burning process, one of the waste products is fly ash. Fly ash is a fine grained siliceous or alumino-siliceous material, making it ideal for use in cement/concrete, road base material, and in asphalt paving.

One of the primary uses for coal fly ash is in the cement industry because fly ash can easily combine with calcium hydroxide to form needed compounds in the cement process. Fly ash is commonly used as replacement for clay, sand, limestone and gravel as a cheaper substitute and energy saver.

For use in Portland cement, fly ash must meet the requirements of ASTM C618, which separates the material into two classes (F and C). These classes differ in the amount of calcium (Ca), silica (Si), aluminum (Al) and iron (Fe) content.

Cement companies must monitor the composition to insure high quality control of their final product. For this control, many companies use wavelength dispersive X-ray fluorescence (WDXRF). WDXRF analysis is a stable and fast analytical technique which requires little sample preparation. Samples in WDXRF simply can be analyzed as loose powder, pressed pellet or lithium borate fusion.

Instrument

Thermo Scientific ARL PERFORM'X series spectrometer used in this analysis was a 4200 watt system. This system is configured with 6 primary beam filters, 4 collimators, up to nine crystals, two detectors, helium purge and our 5GN+ Rh X-ray tube for best performance from ultra-light to heaviest elements thanks to its 50 micron Be window. This new X-ray tube fitted with a low current filament ensures an unequalled analytical stability month after month.

The ARL PERFORM'X offers the ultimate in performance and sample analysis safety. Its unique LoadSafe design includes a series of features that prevent any trouble during sample pumping and loading.

Liquid cassette recognition prevents any liquid sample to be exposed to vacuum by mistake. Over exposure safety automatically ejects a liquid sample if X-ray exposure time is too long.

The Secutainer system protects the primary chamber by vacuum collecting any loose powders in a specially designed container, easily removed and cleaned by any operator. For spectral chamber protection, the ARL PERFORM'X uses a helium shutter designed for absolute protection of your goniometer during liquid analysis under helium operation. In the "LoadSafe Ultra" configuration, a special X-ray tube shield provides total protection against sample breakage or liquid cell rupture.

Calibration

Analyzing the fly ash samples accurately and precisely requires not only the detection of low X-ray intensities at the trace elemental concentrations, but also at very high concentrations. For optimal analysis, the conditions and parameters used were set to maximize the excitation of each element while keeping the background as low as possible.

A set of certified reference standards were used in the creation of elemental regression plots. These graphs are correlations of the known concentration plotted against the measured intensities. The linearity of these curves depends upon the stability of the instrument, inter-elemental correction capabilities of the software, the accuracy of the standard and the quality of the sample preparation.

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Results

The calibration ranges depend on the calibration standards used for this report. These ranges can be increased by the addition of standards containing the newly desired concentration. The standard deviations (SD) and relative standard deviations (RSD) are the typical deviation from the standard values achieved from the linear regression at 20 seconds counting times.

Elements	Calibration Range (%)	Fused Bead % RSD	Pressed Pellet % RSD
SiO_2	35.1 - 71.5	0.125	2.185
CaO	0.95 - 12.1	0.062	0.177
Al_2O_3	16.67 - 37.21	0.320	0.734
Fe_2O_3	1.55 - 12.1	0.063	0.160
MgO	0.70 - 3.21	0.022	0.071
SO_3	0.21 - 6.30	0.062	0.202
P_2O_5	0.10 - 2.75	0.011	0.011
K_2O	0.60 - 2.35	0.014	0.022
TiO_2	0.60 - 5.85	0.014	0.032
Na_2O	0.10 - 1.28	0.003	0.001

Table 1: Typical standard results.

Table 2 represents a 12-hour stability of a fused bead sample. The analysis time was reduced to only 10 seconds per element. The results illustrate the ARL PERFORM'X outstanding stability and precision. The precision and accuracy are achieved by providing unmatched power stability and environmental conditions inside the spectral and primary tanks.

Elements	Sample Conc. (%)	St.Dev. (%)	RSD %	Count Time
SiO_2	52.1	0.068	0.111	10 Sec
CaO	2.31	0.386	0.045	10 Sec
Al_2O_3	27.56	0.065	0.180	10 Sec
Fe_2O_3	13.011	0.119	0.086	10 Sec
MgO	0.652	0.046	0.316	10 Sec
SO_3	0.673	0.086	0.098	10 Sec
P_2O_5	0.324	0.047	0.284	10 Sec
K_2O	1.984	0.045	0.226	10 Sec
TiO_2	1.211	0.022	0.476	10 Sec
Na_2O	0.175	0.017	0.746	10 Sec

Table 2: Precision test results over 12 hours.

Fuse bead sample preparation is the most accurate method of presenting a sample for XRF analysis. Fusing oxide materials is the best way of completely removing both grain size and mineralogical effects. Essentially, the procedure consists of heating a mixture of sample and a borate flux, namely lithium tetraborate and/or lithium metaborate at high temperature (1000°-1200°) so that the flux melts and dissolves the sample. The overall composition and cooling conditions must be such that the end product after cooling is a single phase glass.

Conclusion

The results show that coal fly analysis can easily be performed with the ARL PERFORM'X sequential XRF spectrometer. The precision and accuracy are shown to be very high in this matrix type. Of course the precision can easily be increased by extended the elemental counting times. This would allow for much better SD and %RSD at all concentration ranges.

Furthermore, operation is made easy through the newest and most advanced state-of-the-art Thermo Scientific OXSAS WDXRF software which operates with the latest Microsoft Windows® 7 packages.

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