Application note | 41971

X-Ray Fluorescence

EDXRF analysis of coal fly ash as loose powders

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Introduction

All the waste that remains after coal is burned, often in a coal-fired power station, is referred to as coal ash. Coal ash may contain lead, mercury, arsenic and many other heavy metals. Fly ash and bottom ash are two subcategories of coal ash, typically separated based on particle size.

More than half of coal leftovers are fly ash, the most abundant and well-known component. Particles of fly ash, the lightest type of coal ash, “fly” up into a power plant’s exhaust stacks because they are so light. About 99 % of the ash are captured by filters inside the stacks, which draw it in with opposing electrical charges. Fly ash can be recycled. The fine particles are a perfect material for wallboard and concrete because they bond together and solidify, especially when combined with water.

But fly ash from coal is also a dangerous industrial solid waste. The natural environment and human health are seriously threatened by its excessive worldwide production, inappropriate disposal methods, underutilization, and lack of knowledge about its inherent risks. Therefore, a method that is quick, precise, and accurate, is required to quantify coal fly ash. Many companies use X-ray fluorescence spectrometry (XRF) as a preferred technology for process and quality control since it is a proven analytical approach that can accurately assess the chemical composition of materials with limited sample preparation.

Instrumentation

The Thermo Scientific™ ARL™ QUANT’X EDXRF Spectrometer is equipped with a Silicon Drift Detector (SDD) and a 50 Watt Rh or Ag target X-ray tube which is air cooled with a maximum excitation v The Thermo Scientific™ ARL QUANT’X™ EDXRF Spectrometer is equipped with the following features: and radiation of heat in our atmosphere; N_2 and O_2 which are present in much higher concentrations do not have this same characteristic effect. Atmospheric CO_2 levels increase the temperature of the surface of our planet, simultaneously excess CO_2 is dissolved into the oceans gradually lowering the water’s pH, thus making the seas more acidic.

- Silicon drift detector (SDD): The SDD remains the performance benchmark for all energy dispersive detectors. The large active area of 30 mm^2 enables effective capture of characteristic element X-rays emitted by the sample.
- 50 W Rh or Ag target X-ray tube: Air-cooled with a maximum excitation voltage of 50 kV.
- Nine primary beam filters: Designed to optimize the peak-to-background signals for all elements from F to Am.
- 10-Position auto-sampler with spinner: Allows for unattended analysis of multiple samples.

Excitation conditions

In EDXRF, sensitivity and precision are achieved by targeted excitation of the sample to fluoresce only the elements of interest. The ARL QUANT'X EDXRF Spectrometer offers many combinations of excitation voltages (4-50 kV) and multiple primary beam filters for optimal background control.

As shown in Table 1, spectra were collected on each coal fly ash sample for a total live time of 2 minutes. Measurement time can be further fine-tuned according to specific applications. Analysis is conducted in vacuum without spinning.

Table 1. Analytical conditions.

Condition	Filter	Voltage (kV)	Current (mA)	Auto Current	Atmosphere	Live Time	Analytes
Low Za	No Filter	4	2	Yes	Vacuum	80	Na,Mg,Al, Si,P,S,Rh
Mid Za	Pd Thin	16	2	Yes	Vacuum	40	Ti,Fe,Mn,K,Ca

Sample Preparation

Calibration was done using 10 calibration samples. Samples were prepared as loose powders. About 1 gram of sample was poured into plastic cups with vented caps. The cups are closed on bottom side with a 4-micron polypropylene film as shown in Figure 1. This set-up permits to measure the fly ash powders in vacuum. Table 2 shows the concentration ranges of the different oxides covered by the calibration as well as the R^2 and RMSE (root mean square error) values obtained for the different compounds.

Figure 1. Picture of a coal fly ash sample prepared in as loose powder in liquid cup.

Table 2. Concentration ranges and calibration parameter values for the analysis of coal fly ash.

Element	N	Min (%)	Max (%)	R2	RMS
Na_2O	7	0.060	2.240	0.9907	0.078
MgO	10	0.540	6.700	0.9867	0.240
Al_2O_3	10	6.790	32.440	0.9883	1.152
SiO_2	10	35.800	58.750	0.9912	1.736
P_2O_5	10	0.059	0.586	0.9758	0.027
SO_3	10	0.018	4.975	0.9917	0.145
K_2O	10	0.510	1.960	0.9827	0.086
CaO	10	1.490	20.910	0.9995	0.149
TiO_2	10	0.350	1.900	0.9877	0.070
Mn_3O_4	10	0.043	0.318	0.9932	0.008
Fe_2O_3	10	4.430	17.870	0.9894	0.566

Calibration

Calibration curves have been derived relating element characteristic X-ray intensity to oxide concentration. X-ray fluorescence measures elements, but the results can be related directly to the oxide forms of these elements when only one single form of oxide is present in the sample. Figures 2 to 12 show the calculated versus given concentration plots obtained for Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, SO_3, K_2O, CaO, TiO_2, Mn_3O_4, and Fe_2O_3.

Calibration summary

Na_2O

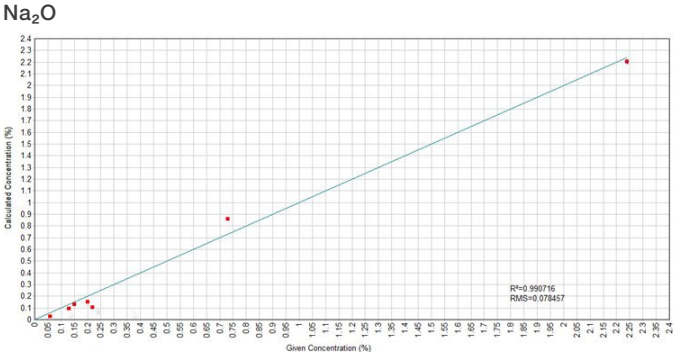


Figure 2. Na_2O calculated versus given concentrations.

MgO

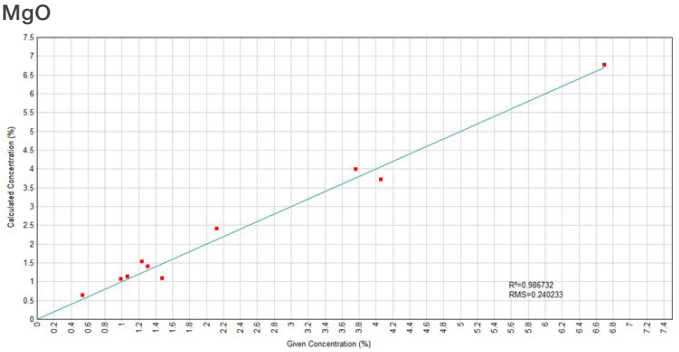


Figure 3. MgO calculated versus given concentrations.

Al_2O_3

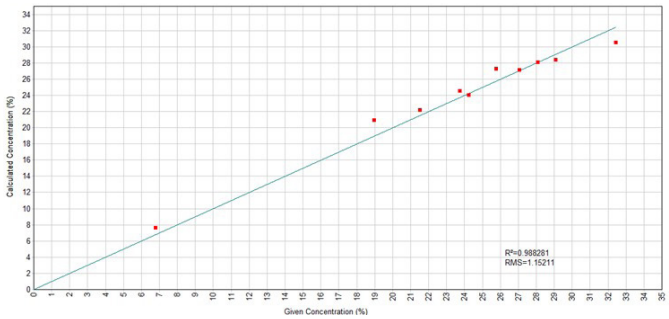


Figure 4. Al_2O_3 calculated versus given concentrations.

SiO_2

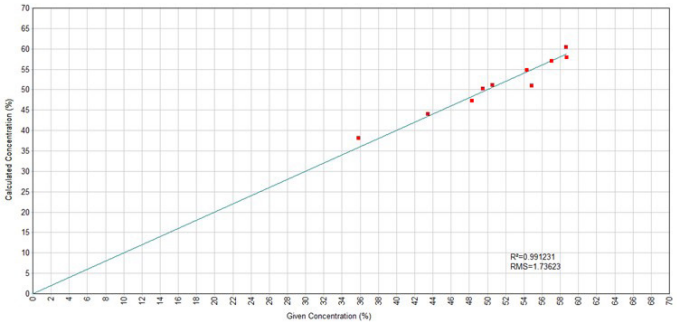


Figure 5. SiO_2 calculated versus given concentrations.

P_2O_5

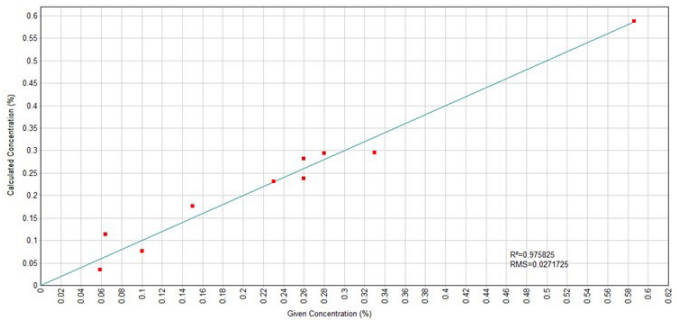


Figure 6. P_2O_5 calculated versus given concentrations.

SO_3

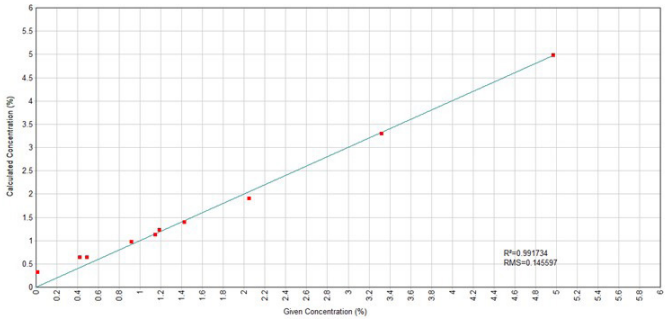


Figure 7. SO_3 calculated versus given concentrations.

K_2O

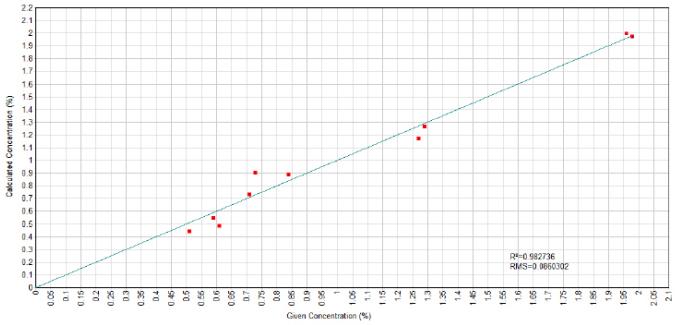


Figure 8. K_2O calculated versus given concentrations.

CaO

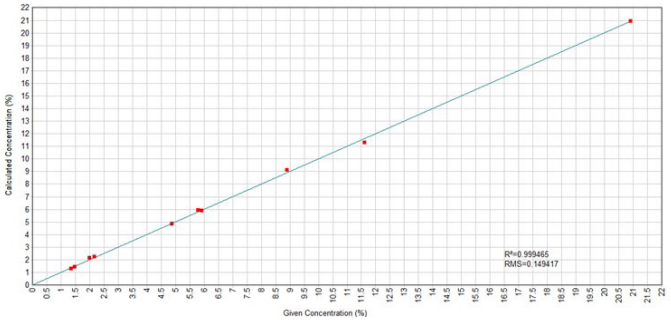


Figure 9. CaO calculated versus given concentrations.

TiO_2

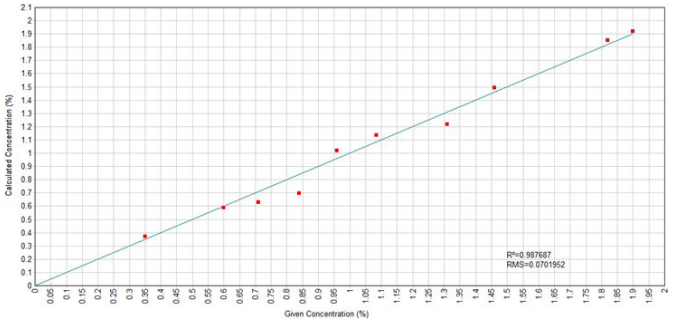


Figure 10. TiO_2 calculated versus given concentrations.

Mn_3O_4

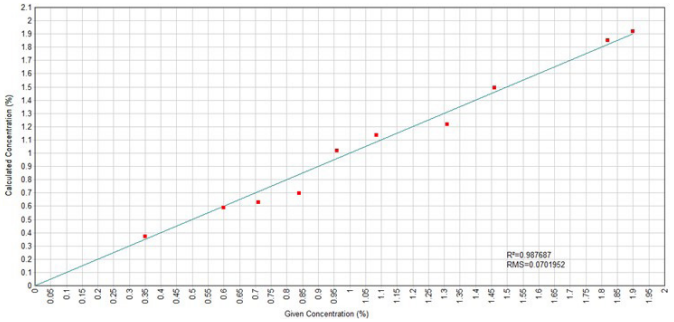


Figure 11. Mn_3O_4 calculated versus given concentrations.

Fe_2O_3

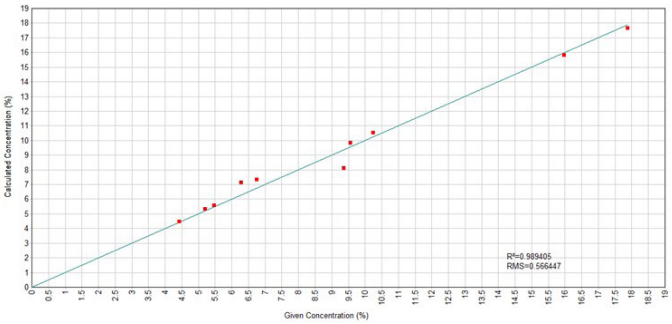


Figure 12. Fe_2O_3 calculated versus given concentrations.

Validation

Coal fly ash reference material, ARM 013, was used to validate the calibration. Table 3 shows the analysis results for the reference materials. Reference values were compared with an average of 3 replicates on the same prepared sample of the two standards while Table 4 shows the repeatability results for each CRM.

Table 3. Accuracy of results for standards ARM 013 using ARL QUANT'X spectrometer.

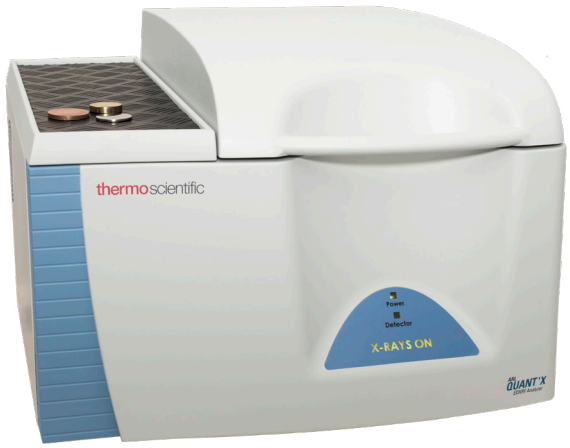
Sample Name	Na2O %	MgO %	Al2O3 %	SiO2 %	P2O5 %	SO3 %	K2O %	CaO %	TiO2 %	Mn3O4 %	Fe2O3 %
ARM 013 Reference	0.060	0.540	32.440	54.890	0.330	2.050	0.840	2.160	1.820	0.041	4.430
ARM 013 Analysis	0.028	0.708	32.827	54.276	0.325	2.062	0.900	2.308	1.914	0.051	4.602

Table 4. Repeatability results for standards ARM013 using ARL QUANT'X spectrometer.

Sample Name	Na2O %	MgO %	Al2O3 %	SiO2 %	P2O5 %	SO3 %	K2O %	CaO %	TiO2 %	Mn3O4 %	Fe2O3 %
Repeats 1	0.031	0.706	32.844	54.252	0.325	2.061	0.902	2.296	1.921	0.052	4.612
Repeats 2	0.027	0.710	32.837	54.238	0.327	2.063	0.894	2.327	1.919	0.050	4.608
Repeats 3	0.025	0.708	32.802	54.336	0.323	2.062	0.905	2.300	1.904	0.050	4.585
Average	0.028	0.708	32.827	54.276	0.325	2.062	0.900	2.308	1.914	0.051	4.602
1-Sigma	0.003	0.002	0.023	0.053	0.002	0.001	0.006	0.017	0.009	0.001	0.015
% RSD	11.9	0.3	0.1	0.1	0.5	0.0	0.6	0.7	0.5	1.3	0.3

Conclusion

This application note shows the suitability of the **ARL QUANT'X EDXRF Spectrometer** for the analysis of coal fly ash samples. This compact instrument allows for reliable and fast analysis of coal fly ash. Accuracy and repeatability results show that satisfactory results are obtainable for an analysis of loose powder in vacuum. This is a significant advantage for simple sample preparation while providing good accuracy & precision.



References

1. https://www.nrdc.org/stories/coal-ash-fly-ash-bottom-ash-and-boiler-slag

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