

ANALYSIS OF INORGANIC MAJOR AND MINOR COMPOUNDS IN UNASHED COAL SAMPLES PREPARED AS PRESSED POWDER



Introduction

This application note shows that the Epsilon 4 – a high performance energy dispersive X-ray fluorescence spectrometer – is capable of analyzing Na₂O, MgO, Al₂O₃, SiO₂, P₂O₅, S, K₂O, CaO, TiO₂, MnO, Fe₂O₃, SrO and BaO in unashed coal samples.

Application background

Coal is classified and valued according to its sulfur, phosphorous, volatile and ash content. The composition of ash can effect coal processing, handling and final usage.

For example, coking coal used in the steel making industry requires coal with low sulfur and phosphorous content otherwise the quality of steel produced is adversely affected. In case of burning coal for the production of electricity, sulfur can be a major atmospheric pollutant. In addition to elemental sulfur (S), other elements routinely determined and expressed as oxides, include Na₂O, MgO, Al₂O₃, SiO₂, P₂O₅, K₂O, CaO, TiO₂, MnO, Fe₂O₃ and SrO.

Instrumentation

Measurements were performed using an Epsilon 4 EDXRF spectrometer, equipped with a 10 W, 50 kV silver anode X-ray tube, 6 filters, a helium purge facility, a high-resolution silicon drift detector, a spinner and a 10-position removable sample changer.

Sample preparation

The coal samples were dried at 105 °C, ground in a tungsten carbide swing mill for 60 s, mixed with wax (8 g sample + 2 g wax) and pressed into pellets under 20 tons for 30 s. The pellet samples were put in standard sample holders and loaded into the Epsilon 4.

Measurement procedure

A number of commercially available coal standards, RMs and CRMs from Alpha Resources and from the SA Bureau of Standards were used to setup the calibrations. The details of the measurement conditions are given in **Table 1**. Sodium was analyzed using a region of interest (ROI). **Figures 1** and **2** show the spectra obtained for the elements in a typical coal standard.

Table 1. Measurement conditions

Conditions	Elements	kV	μA	Measurement time (s)	Medium	Filter
1	Sr	50	200	30	Air	Ag
2	K, Ca, Ti, Fe, Ba	12	800	30	Air	Al thin
3	Na, Mg, Al, Si, P, S	5	2000	60	Helium	none

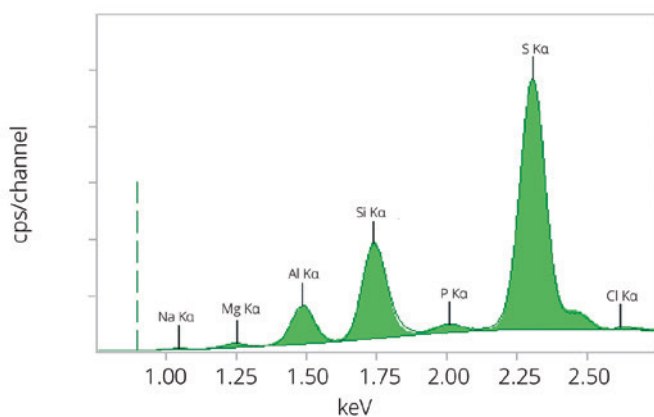


Figure 1. Spectrum obtained using measurement condition 3 (Table 1) and showing the excellent resolution of the detector

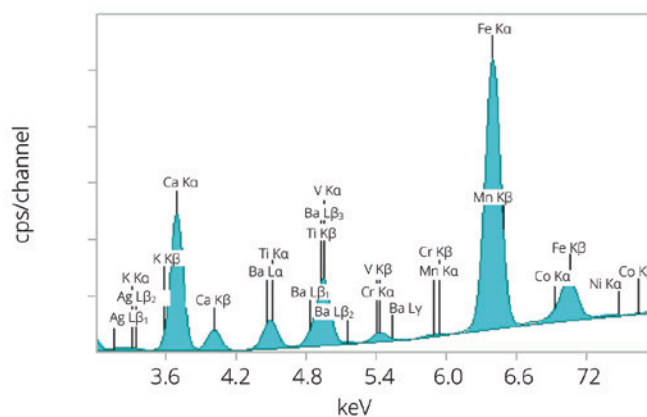


Figure 2. Spectrum obtained using measurement condition 2 (Table 1)

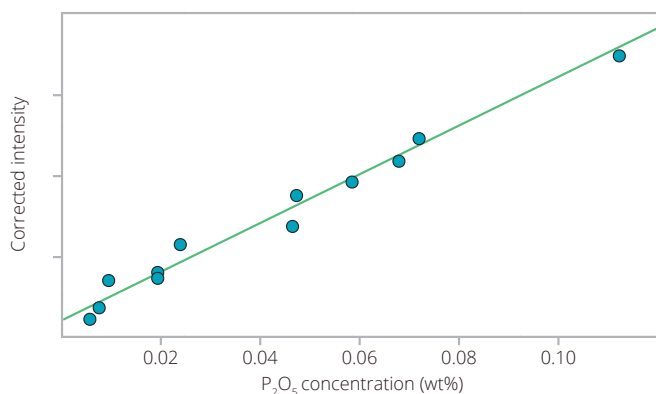


Figure 3. Calibration graph of P₂O₅

Accurate calibration results

The calibration plots shown in **Figures 3 to 4** demonstrate the strong correlation between the given concentrations and the measured intensities. **Table 2** presents the RMS values of the calibrations, and the achievable detection limits.

Table 2. The concentration range and RMS values of the calibration

Compound	Concentration range (wt%)	RMS* (wt%)	LLD (ppm) application time
Na ₂ O	0.014 - 0.29	0.0131	150
MgO	0.025 - 0.43	0.0111	100
Al ₂ O ₃	0.86 - 11.27	0.1480	55
SiO ₂	1.6 - 17.66	0.2810	45
P ₂ O ₅	0.007 - 0.14	0.0067	40
S	0.51 - 3.58	0.0764	10
K ₂ O	0.029 - 0.215	0.0060	12
CaO	0.044 - 1.87	0.0232	8
TiO ₂	0.06 - 0.63	0.0055	7
MnO	0 - 0.02	0.0006	7
Fe ₂ O ₃	0.147 - 3.815	0.0395	6
SrO	0.001 - 0.033	0.0010	2
BaO	0.004 - 0.042	0.0017	20

* RMS (Root Mean Square). The more accurate calibrations have the smaller RMS values.

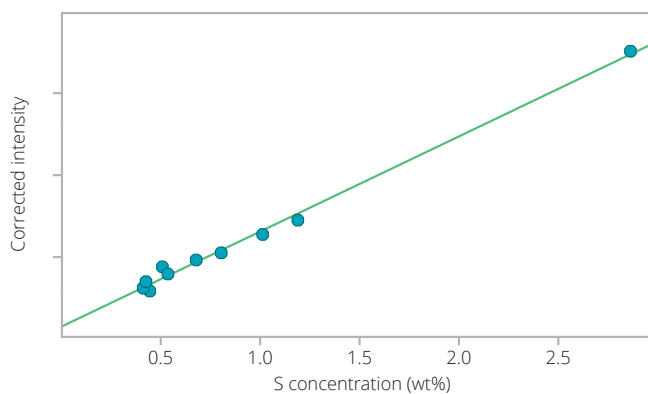


Figure 4. Calibration graph of S

Precision

One sample was measured 20 times consecutively. The averages and the standard deviations (1 sigma) are presented in **Table 3**.

Table 3. Results of the repeatability test

Compound	Average concentration (wt%)	Standard deviation (wt%)
Na ₂ O	0.26	0.009
MgO	0.23	0.007
Al ₂ O ₃	8.05	0.014
SiO ₂	15.12	0.044
P ₂ O ₅	0.042	0.004
S	1.40	0.003
K ₂ O	0.24	0.002
CaO	1.43	0.012
TiO ₂	0.35	0.001
MnO	0.021	0.0003
Fe ₂ O ₃	1.75	0.004
SrO	0.014	0.0002
BaO	0.036	0.001



Conclusions: This work clearly demonstrates the suitability of the Epsilon 4 EDXRF spectrometer for the quantitative analysis of P₂O₅, S, Na₂O, MgO, Al₂O₃, SiO₂, K₂O, CaO, TiO₂, MnO, Fe₂O₃, SrO and BaO in unashed coal samples. Accurate and precise results can be obtained in just 2 minutes.



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