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BARIUM AND STRONTIUM IN COAL ASH - SUPPLEMENTARY ANALYSES NECESSARY TO INCREASE THE ACCURACY OF MAJOR ELEMENT DETERMINATIONS CARRIED OUT BY ATOMIC ABSORPTION AND X-RAY FLUORESCENCE

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INTRODUCTION

When analysing coal ash for the 10 major constituents, one normally expects the total percentage of the oxides to be 98 to 101% ignoring trace amounts of other elements; to the analyst these totals are acceptable, however, when the total is less than 98, possibly as low as 95, the question arises as to which of the determined values is low. If on examination the values are confirmed, the analyst must determine what the other constituents are.

Western Canadian coals are known to contain more than trace amounts of barium and strontium and can account for several percent of the low totals encountered, eg. Ba can range from 0.2 to 2.0 per cent and Sr from 0.15 to 0.5 per cent. In x-ray fluorescence the presence of Ba (a heavy absorber) effects the determined values of all 10 major elements and for accurate analysis must be taken into consideration, and known prior to the analyses being made by x-ray fluorescence so that the appropriate inter-element corrections may be applied to the 10 major elements being determined. Strontium although not as important from the point of view of inter-element corrections should also be determined prior to analysis.

Until recently the only indication of the presence of significant amounts of barium and strontium was the low total oxides obtained by x-ray fluorescence, necessitating the chemical determination of barium and strontium in the sample, and re-running the x-ray fluorescence analyses and applying the appropriate corrections. To overcome this difficulty a preliminary scan is made of all lithium tetraborate pellets to determine the presence or absence of barium and strontium.

As the energy-dispersive x-ray fluorescence unit used in these laboratories cannot determine elements below (12) in the periodic table, sodium (11) is determined on a seperate portion of the coal ash, by flame photometry or atomic absorption on a solution prepared by fusing the coal ash with lithium tetraborate, and dissolving in hydrochloric acid. The determination of barium, strontium, nickel, chromium, zinc, molybdenum, cobalt, titanium, vanadium and iron are normally determined on the same solution in accordance with the ISO/TC 27 W6 13 (Sec-27) - draft proposal.

In the analyses of a large number of western Canadian coals and fluidized bed residues and deposits using these coals, low totals were encountered, and as a result it was decided that barium and strontium should be determined in addition to the 10 major elements.

METHODS OF ANALYSIS

Preparation of Solution

0.1 to 0.2 g of coal ash prepared in accordance with ISO 1170 and ground to pass a #200 sieve (74 um) is fused with 1 g of lithium tetraborate and fused at 1000°C for 15 minutes in a non-wetting platinum crucible, cooled and dissolved in 150 ml of 5% HCl on a stirring hot-plate with magnatic stirring bar. The time required is about ½ hr. The solution is transferred to a 200 ml volumetric flask and brought to volume with 5% HCl.

Using barium and strontium hollow cathode lamps, with the monochrometer set at 553.6 nm and 460.7 nm respectively, the aforementioned elements are determined.

EXPERIMENTAL

Four samples of fluid bed residues resulting from the combustion of Saskatchewan lignite (Bienfact) were analysed by x-ray fluorescence, Run No. 1, Table 1 presents data obtained assuming that no major interferences were present. Run No. 2, Table II includes chemically determined values for barium and strontium and loss on fusion, all values have been corrected for the presence of barium and strontium by the computer program.

CONCLUSIONS

Table I shows low total oxides, indicating the need to determine barium and strontium. Table II which includes the chemically determined values for barium and strontium has also been recalculated to take into consideration the absorption effects due to the presence of barium and strontium. These effects are particularly significant for SiO_2, Al_2O_3, Fe_2O_3 and TiO_2.

As a result of this work, the determination of barium and strontium will be determined routinely if a preliminary scan indicates their presence.

TABLE 1

RUN # 1

Raw Values Assuming Barium and Strontion Absent

Sample No.	2555-79	2556-79	2557-79	2558-79
Component	%	%	%	%
SiO_2	46.69	43.29	41.29	42.04
Al_2O_3	22.45	20.70	20.27	20.92
Fe_2O_3	4.42	4.71	4.73	5.17
TiO_2	0.96	0.81	0.81	0.94
P_2O_5	0.31	0.34	0.40	0.42
CaO	12.79	11.69	15.07	15.28
MgO	2.24	2.36	3.08	3.26
SO_3	1.77	6.72	4.33	3.66
Na_2O	3.82	3.95	3.94	4.25
K_2O	0.41	0.39	0.31	0.33
SrO	-	-	-	-
BaO	-	-	-	-
L.O.F.	0.00	0.27	0.21	0.00
Total	95.86	95.23	94.65	96.27

TABLE II

RUN # 2

Corrected Values Including Chemically Detm'd BaO and SrO

Sample No.	2555-79	2556-79	2557-79	2558-79
Component	%	%	%	%
SiO_2	47.07	43.80	41.68	42.39
Al_2O_3	22.64	20.95	20.47	21.10
Fe_2O_3	4.58	4.96	4.92	5.35
TiO_2	0.98	0.84	0.80	0.94
P_2O_5	0.31	0.34	0.40	0.42
CaO	12.95	11.89	15.28	15.47
MgO	2.24	2.36	3.08	3.26
SO_3	1.77	6.72	4.33	3.66
Na_2O	3.82	3.95	3.94	4.25
K_2O	0.41	0.39	0.31	0.33
SrO	0.39	0.38	0.50	0.49
BaO	1.57	2.08	1.59	1.64
L.O.F.	0.00	0.27	0.21	0.00
Total	98.73	98.93	97.51	99.31