

77-51



Energy, Mines and
Resources Canada

Énergie, Mines et
Ressources Canada

CANMET

Canada Centre
for Mineral
and Energy
Technology

Centre canadien
de la technologie
des minéraux
et de l'énergie

COMPARATIVE ANALYSES OF FIVE SAMPLES
OF SHALE BY X-RAY FLUORESCENCE
ENERGY RESEARCH LABORATORIES, OTTAWA
CANADA CEMENT LAFARGE LTD., BELLEVILLE

W.J. MONTGOMERY

SOLID FUELS ANALYSIS & STANDARDIZATION LABORATORY
ANALYTICAL & EVALUATION SERVICES GROUP

ENERGY RESEARCH PROGRAM

ENERGY RESEARCH LABORATORIES
REPORT ERP/ERL 77-51 (TR)

ERP/ERL 77-51 (TR)

COMPARATIVE ANALYSES OF FIVE SAMPLES
OF SHALE BY X-RAY FLUORESCENCE
ENERGY RESEARCH LABORATORIES, OTTAWA
CANADA CEMENT LAFARGE LTD. - BELLEVILLE

by

W.J. Montgomery

INTRODUCTION

Discussions of the use of X-Ray fluorescence to analyse both coal ash and raw materials used in the manufacture of cement have been held over the past few years between Peter S. Grindrod of Canada Cement Lafarge Ltd., and W.J. Montgomery of E.R.L. As both laboratories have had experience in this type of analysis, it was decided that an exchange of samples and ideas could prove mutually beneficial. Mr. Grindrod submitted five samples of shale from their plant at Exshaw Alberta, and provided the analyses for comparison. The samples were received in April, 1977.

Experimental

In the analysis of coal ash it is mandatory that the coal be ashed in an oxidizing atmosphere to constant weight at 750°C. In cement analysis the temperature is 1050°C.

A portion of each sample was ashed at each of the two temperatures for comparative purposes and the loss on ignition recorded. A lithium tetra-borate pellet was prepared at a 4.5:1 ratio of lithium tetra-borate to ash and each analysed 5 times by XRF for silica, alumina, iron, titanium, phosphorus, calcium oxide and magnesium oxide. Sulphur was determined by the Leco IR 33 and sodium and potassium by the flame photometer.

Table 1 records the data obtained for comparison with the analytical values provided by Mr. Grindrod. Mr. Grindrod informed us that they feel that much of the sulphur in these shales exists in the elemental form and is reported as such. The sulphur values were carried out by Canada Cement Lafarge on the unignited sample in each case.

The bracketed values opposite SO_3 in the CCL column are in fact elemental S. The bracketed total in the CCL column includes the elemental sulphur, the other value without inclusion of sulphur.

Discussion

As might be expected the loss on ignition is higher at 1050°C than at 750°C . There is obviously some loss of sulphur at the higher temperature. The high totals for the Canada Cement Lafarge samples could be attributed to this high temperature loss. There is no apparent loss of sodium or potassium up to 1050°C in samples of this composition. For some reason the TiO_2 values reported by CCF are about one half those reported by E.R.L. and the P_2O_5 values double.

TABLE I

XRF ASH ANALYSIS REPORT

OXIDE	2419-77			2420-77		
	* ERL PELLET A	* ERL PELLET B	CCL	ERL PELLET A	ERL PELLET B	CCL
SiO ₂	65.81	66.01	66.73	65.24	65.75	66.00
Al ₂ O ₃	12.51	12.34	12.55	14.62	14.76	13.82
Fe ₂ O ₃	4.17	4.12	4.00	4.78	4.52	4.50
TiO ₂	0.54	0.54	0.26	0.60	0.67	0.26
P ₂ O ₅	0.17	0.16	0.32	0.16	0.15	0.32
CaO	2.49	2.56	2.65	1.26	1.25	1.47
MgO	2.52	2.57	2.70	1.81	1.80	2.00
SO ₃	1.34	0.58	(1.50)	1.25	0.46	(1.40)
Na ₂ O	0.55	0.54	0.55	0.56	0.56	0.62
K ₂ O	2.21	2.21	2.29	2.65	2.67	2.85
LOSS ON IGNITION	6.61	7.74	7.94	5.84	7.04	7.22
TOTAL	98.92	99.37	99.99 (101.49)	98.77	99.63	99.06 (100.46)

PELLET A - PREPARED FROM SHALE IGNITED AT 750°C

PELLET B - PREPARED FROM SHALE IGNITED AT 1050°C

TABLE I (cont'd)

XRF ASH ANALYSIS REPORT2421-77
EXSHAW SHALE #122422-77
EXSHAW SHALE #13

OXIDE	* ERL PELLET A	* ERL PELLET B	CCL			ERL PELLET A	ERL PELLET B	CCL
SiO ₂	62.87	63.54	64.42			61.97	62.04	63.99
Al ₂ O ₃	13.17	13.35	12.91			12.62	12.60	11.66
Fe ₂ O ₃	4.43	4.29	4.30			4.32	4.30	4.13
TiO ₂	0.58	0.58	0.25			0.56	0.54	0.21
P ₂ O ₅	0.18	0.18	0.32			0.20	0.19	0.33
CaO	3.01	2.93	3.20			4.05	4.02	4.10
MgO	2.70	2.59	2.65			2.78	2.61	2.72
SO ₃	1.74	0.91	(1.62)			1.69	0.84	(1.67)
Na ₂ O	0.60	0.59	0.60			0.60	0.59	0.50
K ₂ O	2.35	2.37	2.52			2.24	2.27	2.22
LOSS ON IGNITION	7.23	8.50	9.17			8.14	9.19	10.00
TOTAL	98.86	99.83	100.34 (101.96)			99.17	99.19	99.83 (101.50)

TABLE I (cont'd)

XRF ASH ANALYSIS REPORT

SAMPLE NO 2423-77

MARKED EXSHAW SHALE # 14

OXIDE	* ERL PELLET A	* ERL PELLET B	CCL			ERL PELLET A	ERL PELLET B	CCL
SiO ₂	59.25	59.87	61.64					
Al ₂ O ₃	12.28	12.32	11.25					
Fe ₂ O ₃	4.22	4.23	3.96					
TiO ₂	0.56	0.57	0.21					
P ₂ O ₅	0.26	0.20	0.35					
CaO	5.51	5.56	5.70					
MgO	3.12	3.12	3.22					
SO ₃	2.39	0.95	(1.64)					
Na ₂ O	0.56	0.55	0.50					
K ₂ O	2.16	2.16	2.36					
LOSS ON IGNITION	9.14	10.77	11.66					
TOTAL	100.01	100.30	100.85 (102.49)					