

WU/C/211

FUEL RESEARCH INSTITUTE OF SOUTH AFRICA

TECHNICAL MEMORANDUM NO. 12 OF 1965

INVESTIGATION OF MINERALS IN COAL, SHALES AND
MAGNETITES BY THE X-RAY DIFFRACTION METHOD

by

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and

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A. COAL SAMPLES:

Because of the effect of inorganic impurities on the utilization of coal, it was decided to carry out a survey on the minerals present in production samples from different mines.

The minerals were separated in a centrifuge from the bulk of the coal by means of the float-and-sink method, using 20-gram portions of minus 20 mesh coal and carbon tetrachloride (s. g. 1.60) as medium. The sink portions were then ground to pass a 270 mesh (53 μ) sieve and used as such for the mineral identification.

To assist in the identification, a technique which consisted of completely oxidizing the coal in a current of air at 350°C was also employed. The sink portions were all heated in this manner, in some cases also the unseparated coal, and in one instance the float portion.

Where some uncertainty existed about the presence of small amounts of kaolinite, the sample was heated at 600°C for one hour, resulting in the disintegration of kaolinite and the removal of its diffraction pattern, which served to confirm the presence of kaolinite. This technique was also employed to assist in the identification of other minerals, whereby the number of lines was reduced by the removal of kaolinite lines, but this involved alterations of some of the minerals present, e. g. pyrites. In some cases, the samples were treated with dilute hydrochloric acid to remove lines of calcite and dolomite.

The samples were then scanned at half a degree per minute, using cobalt radiation.

The/...

The results obtained are tabulated in Table 1, with a rough estimate of the amounts of minerals present.

In the Kendal sample, lines at 3.24 \AA and 3.49 \AA appeared in the sink portion, heated at 350°C , and even stronger in the unseparated sample heated in this manner, but were not observed in the unheated sample. They persisted after heating at 600°C for one hour but disappeared on treatment with hydrochloric acid. The mineral or minerals having these lines could not be identified as there were too few lines present. This sample also showed a broad reflection at 9.9 to 10.5 \AA , which could perhaps be that of the basal spacing of a small amount of mica.

In the Utrecht sample there again appeared a line in the vicinity of 10 \AA , which could indicate a small amount of mica, but there were also some other weak lines which could not be accounted for.

The weak 2.79 \AA line in the New Clydesdale sample could indicate a small amount of siderite.

It is being planned to do quantitative determinations of the elements present in the coal ash by means of vacuum x-ray fluorescence methods. The results will then be combined with the identification of the minerals in the coal.

A map indicating the location of the mines where samples were taken is included in this report.

B. SHALE SAMPLES FOR MINING RESEARCH:

A number of shale samples in drill core form was received. The main object was to identify the minerals. In some cases the α -quartz content was determined, as some experience was gained previously with the determination of this mineral.

Sample No. 64m/1 from Tweefontein Mine consisted of carbonaceous shale with small pieces of rocky material.

Sample/...

Sample No. 64m/3, also from Tweefontein Mine, was divided into three parts, viz.

- 1) Consisting only of carbonaceous shale,
- 2) Consisting of carbonaceous shale and bands of coal, and
- 3) Consisting of a small piece of pyrites-rich shale.

These samples were ground to pass a 325 mesh (43 microns) sieve and were used as such for the mineral identification by means of x-ray diffraction, and also for the quantitative determination of α -quartz, using the internal standard method (calcium fluoride as internal standard). These determinations were repeated with the -325 mesh samples heated to constant weight at 350°C in a current of air. At the same time, the moisture content was determined so that the percentage of carbonaceous material could be found by difference. The results are tabulated in Table 2.

In each case the kaolinite was of the poorly crystallised variety.

A difference of only up to 2% was found between the direct quartz determination of the unheated sample and the indirect one using the values obtained from the heated sample. Where only α -quartz and kaolinite were present, the kaolinite content was found by difference.

At a later stage, two samples, 64m/37 from Tweefontein Mine and 64m/65 from Douglas Mine, were received.

Sample 64m/37 consisted only of α -quartz and kaolinite. The α -quartz content was not determined.

Sample 64m/65 consisted of α -quartz, a small amount of kaolinite and a clay mineral with a broad reflection at 11 Å. However, after removal of the carbonaceous material by heating at 350°C, the reflection became sharp and shifted to 10 Å. The lines of illite could clearly be distinguished. Perhaps the presence of carbonaceous material has an influence on the basal spacings.

According/...

According to disintegration tests in water done on these two samples by the Mining Research Section, a much higher disintegration took place with this sample than in the case of sample 64m/37, which contained kaolinite.

C. MAGNETITE SAMPLES:

Quantitative determinations of the minerals by means of x-ray diffraction methods were done on magnetite samples from different locations in Australia and compared with those of South African magnetites and Alanwood magnetite. The results are tabulated in Table 3.

It is difficult to differentiate between maghemite and magnetite when both are present at the same time. Most of the strong maghemite lines coincide with or lie very near to those of magnetite, so that when there is a small amount of maghemite, its lines will be obscured.

In the case of the two Grafton samples, weak lines were noticed at 1.818 \AA . According to A.S.T.M. Card No. 13-458, maghemite has a strong line in this position, whereas magnetite has no line at 1.818 \AA . It is thus possible that there are small amounts of maghemite.

Weak lines at 3.34 \AA in the Grafton and Gladstone samples could be responsible for small amounts of α -quartz. The same was the case with the 3.03 \AA line in the Gladstone sample, indicating calcite.

The Bon Accord sample consisted only of maghemite with lines coinciding with those on A.S.T.M. Card No. 4-0755, with no line at 1.818 \AA .

The only difference which occurred in the diffraction patterns of the Alanwood, Ermelo and Foskor samples was in the intensities of the magnetite lines.

As/...

As there is, perhaps, a connection between the hematite content and the coercive force of the Australian magnetite samples, it was decided to determine the hematite quantitatively.

The method used was that of adding a small amount of hematite to the sample and, after mixing well, measuring the intensities of the $2.70 \overset{\circ}{\text{Å}}$ line before and after addition. Although the method is not very accurate, it is quick and will give an indication of the amounts of hematite. As a check, a synthetic mixture of Alanwood magnetite and hematite was also analysed. The results are tabulated in Table 4.

J. H. COPEMAN
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and

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Principal Research Officer.

PRETORIA,
13th April, 1965.

OvR.

TABLE 1

Type of Coal	Percentage Sinks												
Grootvlei South	30.5	M	H	M	M	L	H	H	H	H	VL	H	L
Cornelia Betty Shaft	39.4	H	H	M	M	H	H	H	H	H	H	VL	H
Coalbrook No. 2	45.8	M	H	M	M	H	H	H	H	H	VL	H	L
Coalbrook No. 3	48.9	M	H	M	M	L	M	H	H	H	VL	H	L
Utrecht	18.5	L	H	M	M	L	M	H	H	H	VL	H	L
Tshoba	9.1	H	H	M	M	L	M	H	H	H	VL	H	L
Natal Steam	23.4	VL	H	VL	H	H	H	H	H	H	M	H	L
New Clydesdale	9.4	H	VL	H	H	H	H	H	H	H	M	H	L
Bellevue	11.3	L	H	M	M	M	M	M	M	M	VL	M	L
Blinkpan	26.6	M	H	M	M	L	M	H	H	H	VL	M	L
Spitzkop (Peas)	11.4	M	H	M	M	M	M	M	M	M	VL	M	L
Spitzkop (Duff)	12.9	M	H	M	M	M	M	M	M	M	VL	M	L
New Middelburg	19.1	H	L	M	M	M	M	M	M	M	VL	M	L
Kendal	19.4	L	H	H	H	H	H	H	H	H	VL	M	L

The letters refer to concentration as follows:

H = High
 M = Medium
 L = Low
 VL = Very Low.

TABLE 2

Sample No.	Part No.	Minerals	Moisture %	Percentage Composition			
				Carbonaceous material dry basis	α -Quartz	Kaolinite by difference	Kaolinite and pyrites by difference
64m/1	-	1) Kaolinite 2) α -Quartz	1.1	18	40	42	-
	1	1) Kaolinite 2) α -Quartz 3) Very weak line at 3.25A possibly FeSO ₄	1.7	27	23	50	-
	2	1) Kaolinite 2) α -Quartz 3) Pyrites	1.8	41	16	-	43
64m/3	3	1) Kaolinite 2) α -Quartz 3) Pyrites 4) Very weak line at 3.25A possibly FeSO ₄	0.9	7	32	-	61

TABLE 3

Sample	Grafton	Lense 300 yds. from Grafton	Gladstone	Bon Accord	Alanwood	Ermelo Nos. 14, 15, 16	Foskor No. 8
Grain Size	-43 μ	Unsifted	-43 μ	-44 μ	-44 μ	-44 μ	-44 μ
Minerals present	Magnetite Hematite Maghemite α -Quartz	Magnetite Hematite Maghemite α -Quartz	Magnetite Hematite α -Quartz Calcite	Maghemite	Magnetite	Magnetite	Magnetite

TABLE 4

Sample No.	Grafton	Lense 300 yds. from Grafton	Gladstone	9.1 % Hematite in Alanwood
% Hematite	11	14	3	10.6

TRANSVAAL.

● PRETORIA

● MIDDELBURG
■ NEW MIDDELBURG
● WITBANK

■ KENDAL
■ BLINKPAN
■ NEW CLYDESDALE

● BETHAL
■ SPITZKOP
■ BELLEVUE
● ERMELO

● HEIDELBERG
■ GROOTVLEI SOUTH

● VEREENIGING
■ CORNELIA BETTY
● SASOLBURG
COAL BROOK
■ 2
■ 3

● POTCHEFSTROOM

● KLERKSDORP

● VOLKSRUST

■ UTRECHT
● UTRECHT

● NEWCASTLE

● VRYHEID ■ TSHOBA

● DUNDEE

■ NATAL STEAM

● Ladysmith

NATAL.

O. F. S.

■ mines