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THE USE OF THE X-RAY DIFFRACTOMETER FOR CLASSIFYING IRON ORES OF THE KNOB LAKE AREA

by

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MINERAL SCIENCES DIVISION

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CLASSIFYING IRON ORES OF THE KNOB LAKE AREA

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SUMMARY OF RESULTS

A study of samples of iron ore from operating mines of the
Knob Lake area was undertaken to determine whether the X-ray diffracto-
meter could be used for the rapid classification of the ores being mined.
The investigation indicated quite conclusively that the method is not
suitable for this purpose.

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INTRODUCTION

The study of 13 specially selected samples of iron ore from the mines of the Knob Lake area was undertaken on behalf of the Ore Testing and Research Laboratories of the Iron Ore Company of Canada at Schefferville, Quebec. Current practice requires that the ores be categorized into several types, depending chiefly upon their colour and composition. The determination of the composition is by chemical means which entails a lengthy procedure, a faster method is highly desirable. It was suggested that the X-ray diffractometer might provide this means and a series of tests was therefore performed to ascertain the suitability of this method.

These tests were made in the period October 22, 1963 to November 16, 1963 and in all cases the results were forwarded to the company within two weeks of receipt of the samples; the present report is prepared at this time primarily for record purposes.

APPARATUS USED

The apparatus used in this series of tests was a Philips basic X-ray diffraction unit fitted with a wide-angle goniometer, a Geiger counter as a detector, a scaling circuit and a strip-chart recorder. Iron-filtered cobalt radiation was used because it is less strongly absorbed by the iron-bearing minerals than other radiations such as copper. The unit was operated at 50 kV and 10 mA.

Two types of sample holders were used, as follows:

1) A flat aluminium holder having a sample cavity 21 mm by 10 mm by 1.5 mm in depth.

2) A rotating aluminium holder having a round sample cavity 22 mm in diameter and a depth of 1.5 mm.

EXPERIMENTAL PROCEDURE

A representative fraction of about 3 grams was prepared from each of the 13 samples by successive passes through a riffle-type sample splitter. Each fraction was then pulverized in a mechanical mortar for one hour.

Six mounts were made from each of the fractions, 3 in the rectangular aluminium type, 2 in the round aluminium type, and 1 in the shallow copper type. The constituents were first identified from strip charts by the rapid scanning of the samples in one of the rectangular aluminium sample holders. Next, each of the 3 rectangular mounts and the

2 rotating mounts were slowly scanned at $\frac{1}{4}$ degree per 2 θ per minute from 20° 2 θ to 42° 2 θ. The principal peaks of hematite, goethite, and quartz occur within this range. The minerals were also identified by the Debye-Scherrer method.

The quantities of hematite, goethite and quartz were determined by measuring the peak heights minus background, and comparing this value with the peak heights of the same minerals in a standard sample. The standard sample used in these tests was Number 14792 High Grade Blue, with a total Fe content of 63.95% and known to consist substantially of only hematite, goethite and quartz. The percentage of goethite was calculated from the 3.41% loss on ignition reported in the chemical analysis.* The remainder of the sample was assumed to be hematite and quartz.

Usually a correction is made for the mass absorption co-efficient of the minerals in a mixture. With cobalt radiation, however, the mass coefficient of hematite, goethite and quartz are so close to one another that this correction is unnecessary.

The peaks chosen were as follows:

Hematite 2 θ = 41.60, d = 2.51A (this peak was chosen instead of the higher intensity of d = 2.69A because a goethite peak also occurs at this position.)

Goethite 2 θ = 24.80, d = 4.16A

Quartz 2 θ = 31.00, d = 3.34A.

The results obtained by the method described were checked by two other procedures as follow:

1) An internal standard procedure with 25% Al_2O_3 added as the internal standard.

2) A procedure using a shallow copper mount which makes possible the use of the copper mount itself as the internal standard.

The Mn red ore could not be determined with Co radiation because of the interference due to fluorescence of the manganese minerals. The constituents were identified by the Debye-Scherrer method using Mn-filtered Fe radiation.

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* Supplied by Iron Ore Company of Canada, Schefferville, Quebec.

RESULTS OF INVESTIGATION

The chief minerals present in the samples were hematite, goethite and quartz. Clay and feldspar were identified in the "lean red ores" and pyrolusite was found in the "Mn red ore".

The results of diffractometer determinations for hematite, goethite and quartz are given in Table I. The rectangular aluminium sample holder was used. Similar results were obtained using the round rotating sample holder, the internal standard method, and the shallow copper holder.

The total Fe contents calculated from the diffractometer results for hematite and goethite do not agree with the chemically analysed values reported by the Iron Ore Company of Canada. Comparisons of the results are shown in Table 2.

TABLE 1

Classification	Sample No.	Diffractometer Determinations		
		Quartz%	Hematite%	Goethite%
High Grade Blue	T-14792	5.4	61.2	30.2
	T-14786	9.0	40.79	11.32
	H-16507	10.0	81.60	2.51
Lean Blue	T-14772	20.0	36.72	65.43
	T-14774	14.5	44.88	16.34
High Grade Red	T-14790	3.3	44.88	17.61
Lean Red Blend	T-14745 T-14757	9.6	22.44	20.13
Lean Red	T-14742	4.1	20.34	11.33
High Grade Yellow	T-14787	2.0	16.32	33.97
	H-16506	1.0	18.36	46.56
Lean Yellow	T-14700	16.6	4.08	41.52
	T-14758	14.1	4.08	33.97
Mn Red Ore	H-16504	nil	nil	nil

TABLE 2

Comparison of Diffractometer and Chemical Analysis

Classification	Sample No.	SiO$_2$%		Fe%	
		Chemical*	Diffractometer**	Chemical*	Diffractometer**
High Grade Blue	T-14792	4.88	5.4	63.95	63.95***
	T-14786	9.26	9.6	58.03	35.65
	H-16507	6.88	10.0	61.30	58.64
Lean Blue	T-14772	21.10	20.0	50.52	66.84
	T-14774	18.01	14.5	53.63	41.67
High Grade Red	T-14790	5.07	3.3	61.31	42.47
Lean Red Blend	T-14745 T-14745	12.22	9.6	52.63	34.65
Lean Red	T-14742	17.73	4.1	43.44	21.36
High Grade Yellow	T-14787	3.63	2.0	56.98	32.78
	H-16506	2.91	1.0	58.99	42.12
Lean Yellow	T-14700	16.00	16.6	52.23	28.97
	T-14758	15.61	14.1	50.94	24.22
Mn Red Ore	H-16504	4.41	nil	nil	nil

*   Chemical analyses supplied by Iron Ore Company of Canada.

**   Calculated from the hematite and goethite values in Table 1.

*** Used as standard (See page 2).

DISCUSSION OF RESULTS

The peak heights of goethite are not reproducible within any one of the samples. This is true also of the peak heights of the hematite in some of the samples. For this reason the X-ray diffractometer results are subject to error, and this is borne out by the comparison with the chemical values. This behaviour is ascribed to variations in crystallinity of the minerals and to the presence of amorphous goethite.

Quartz determinations are shown in Table 1. A comparison of the X-ray diffractometer and chemical determinations (see Table 2) shows that some of the results are in reasonable agreement. The divergence in the quartz value obtained for the "lean red ores" can be attributed to the considerable amounts of clay and feldspar present, a fact which has already been established by the high Al_2O_3 content and the loss on ignition, as reported in the chemical analysis.

An attempt to classify these ores by the diffractometer is shown in Table 3.

TABLE 3

Colour Type	SiO_2 Content	Hematite/Goethite Ratio
High Grade Blue	low	high
Lean Blue	high	variable
High Grade Red	low	high
Lean Red Blend	high	approx. equal
Lean Red	low	high
High Grade Yellow	low	low
Lean Yellow	high	low

CONCLUSION

From the results obtained it is evident that the use of the diffractometer as a rapid and accurate means of classifying iron ores of the Knob Lake area is of limited usefulness.

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