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CANADA

DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 61-2

DETERMINATION OF THE CHEMICALLY COMBINED IRON IN SPHALERITE FROM NORANDA MINES LIMITED

by

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MINERAL SCIENCES DIVISION

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COPY NO. 16

JANUARY 11, 1961

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SUMMARY OF RESULTS

The amounts of iron chemically combined in the sphalerite in two samples of zinc concentrate from Noranda Mines Limited has been investigated by three methods. The agreement of the results is regarded as very satisfactory within the limits of accuracy of the methods. The average of the determinations for the chemically combined iron in the sphalerite is of the order of 6 per cent, and there was no evidence of any wide or even significant variation in the iron content of the sphalerite. Some observations concerning the minerals and their associations in the two concentrates are being given.

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INTRODUCTION

Two samples of zinc concentrate from the testing laboratory of Noranda Mines Limited at Noranda, Quebec, were received by Mr. L.E. Djingheuzian, Chief of the Mineral Processing Division, Mines Branch, on October 27, 1960. They were re-directed to the Mineral Sciences Division together with a covering letter from Mr. W. Hrynewich, Metallurgist, who requested that the amount of iron chemically combined in the sphalerite in each sample be established.

METHODS OF INVESTIGATION

Three methods of establishing the amount of iron chemically combined in sphalerite were used.

Microscopical Investigation

Polished sections were prepared and Rosiwal analyses were made under the microscope to determine the percentages of the minerals by volume. Using normal physical and chemical properties of the minerals, the percentages by weight and the distribution of the elements were calculated. The iron and zinc contents were determined chemically, and the microscopical data were recalculated on the basis of the chemical analyses.

X-ray Diffraction Analysis

The cell dimensions of the sphalerite were determined by measuring X-ray diffraction patterns. The iron contents were then

calculated according to the method described by Skinner, Barton and Kullerud (1). The accuracy of this method is regarded as of the order of plus or minus one per cent iron.

Determination by Magnetic Susceptibility

The magnetic susceptibility of the sphalerite was determined on the Frantz isodynamic separator. The amount of chemically combined iron affects the magnetic susceptibility of sphalerite, and the figure is obtained by referring to a prepared standard curve. The accuracy with this method is also regarded as about plus or minus one per cent iron. Careful preparation of the sphalerite for magnetic separation is necessary to ensure that sphalerite is substantially free from contaminating minerals. After screening, the minus 150 - plus 200 fraction was examined under the binocular microscope; it was immediately apparent that all of the grains were thickly coated with very fine dust. The dust was removed by agitation in alcohol in a super-sonic generator.

RESULTS OF INVESTIGATION

The results obtained by the three different methods are in remarkably close agreement, considering the accuracy expected. It is to be noted that J.M. Stewart* performed the X-ray work, R. Pinard* made the magnetic susceptibility tests, and the writer performed the Rosiwal analyses. The results are thus based on the work of three independent sets of observations.

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*Senior Technician and *Assistant Technician respectively in the Mineralogy Section.

Microscopical Analyses

Examination of the polished sections reveals that both samples consist essentially of sphalerite accompanied by pyrite, pyrrhotite, chalcopyrite and small quantities of gangue. Magnetite was not positively identified, but tests with a strong magnet indicate the presence of a very small fraction which appears to be too strongly magnetic for normal pyrrhotite.

The data resulting from calculations based on the Rosiwal analyses are arranged in Tables 1 and 2. The chemical analyses used in the calculations are shown in Table 3.

TABLE 1

Rosiwal Analysis and Calculated Iron Distribution
in Zinc Concentrate Sample No. 1

Mineral	Per cent by volume	Per cent by weight	Data used in calculations		Calculated iron distribution based on total Fe = 12.42 per cent	
			S.G.	Iron content		
Sphalerite	85.4	84.7	4			6.13 (by difference)
Pyrite	7.3	9.1	5	46.6	4.24	
Pyrrhotite	3.2	3.2	4	60.4	1.93	
Chalcopyrite	0.4	0.4	4.2	30.5	0.12	
Gangue	3.7	2.6	2.8	?	?	
Totals	100.0	100.0	---	----	12.42	

TABLE 2

Rosiwal Analysis and Calculated Iron Distribution in Zinc Concentrate Sample No. 2

Mineral	Per cent by volume	Per cent by weight	Data used in calculations		Calculated iron distribution based on total Fe = 9.27 per cent
			S.G.	Iron content	
Sphalerite	94.2	94.1	4		6.80 (by difference)
Pyrite	1.9	2.4	5	46.6	1.11
Pyrrhotite	2.1	2.1	4	60.4	1.27
Chalcopyrite	0.3	0.3	4.2	30.5	0.09
Gangue	1.5	1.1	2.8	?	?
Totals	100.0	100.0	---	----	9.27

The figures for the distribution of the iron in the mineral sphalerite in Samples No. 1 and No. 2 as shown in the Tables (1 and 2) are on the basis of 84.7 and 94.1 per cent sphalerite respectively. Calculations of the iron content based on 100 per cent sphalerite yield the following results:

Sample No. 1 7.2 per cent

Sample No. 2 7.2 per cent

TABLE 3

Chemical Analyses of Two Zinc Concentrates
Performed by R. McAdam, Analytical Sub-division

Sample	Total Fe per cent	Total Zn per cent
No. 1	12.42	47.84
No. 2	9.27	53.25

X-ray Diffraction Determinations

Table 4 shows the results of the X-ray determinations.

TABLE 4

Chemically Combined Iron in the Sphalerite in Two Samples of Zinc Concentrates as Determined by X-ray Diffraction Method

Sample	Fe per cent
No. 1	5.6
No. 2	5.7

Magnetic Susceptibility Determinations

Table 5 gives the results of magnetic susceptibility determinations.

TABLE 5

Chemically Combined Iron in the Sphalerite in Two Samples of Zinc Concentrates as Determined from their Magnetic Susceptibilities

Sample	Fe per cent
No. 1	6.2
No. 2	7.0

SUMMARY AND CONCLUSION

A comparison of the results of three different methods of determining the quantities of chemically combined iron in the sphalerite of two concentrates from Noranda Mines Limited may be made from the summary of results shown in Table 6.

TABLE 6

Summary of Results of Determinations of Chemically Combined Iron in the Sphalerite in Two Samples from Noranda Mines Limited

Method of determination	Sample No. 1 Fe per cent in sphalerite	Sample No. 2 Fe per cent in sphalerite
Microscopical (adjusted to agree with chemical analysis)	7.2	7.2
X-ray diffraction (cell edge)	5.6	5.7
Magnetic susceptibility	6.2	7.0

It will be noted that the results obtained by the Rosiwal method are higher than the average of the results obtained by X-ray and magnetic susceptibility methods. In calculating the iron contents of the sphalerites from microscopic data, the iron contributed by gangue and possibly also by a small amount of magnetite (or even steel from the ball mills) was not known and hence could not be taken into consideration. This iron was therefore included with that calculated as occurring in chemical combination in the sphalerite. This would mean that the figures obtained by the microscopical method

should be regarded as maxima, and should probably be reduced by an unknown (but possibly quite small) amount.

It is probable also that there is no very significant variation in the iron contents of the sphalerites, either from sample to sample or within the samples themselves. No such variation is indicated either by the X-ray diffraction patterns, which would tend to be diffuse if the variations were significant, or the microscopical observations which would reveal a wide variation in the colour of the sphalerite (refer to observations in Appendix).

It would seem therefore, that a reasonable interpretation of the results would give between 6 and $6\frac{1}{2}$ per cent as the order of of magnitude for the iron chemically combined in the sphalerite. All of the determined values lie within the limits of error of the X-ray and magnetic susceptibility methods which are regarded as about plus or minus one per cent iron.

APPENDIX

Some Observations Respecting the Character of the Two Zinc Concentrates from Noranda Mines Limited

While performing the Rosiwal analyses, certain features of the samples were observed in polished sections.

The distribution of the minerals is shown in Table 7.

TABLE 7

Distribution of the Minerals in Two Samples of Zinc Concentrate from Noranda Mines Limited. Based on Microscopical Analyses

Minerals	Percentages calculated to weight	
	Sample No. 1	Sample No. 2
Sphalerite	84.7	94.1
Pyrite	9.1	2.4
Pyrrhotite	3.2	2.1
Chalcopyrite	0.4	0.3+
Gangue	2.6	1.1
Totals	100.0	100.0

It will be noted that the percentage of sphalerite is higher in Sample No. 2. Sample No. 2 is considerably finer than No. 1; the sphalerite of Sample No. 2 shows considerably more freedom from included sulphides than does Sample No. 1 (93% compared to 87%), and contains much less pyrite than Sample No. 1. The higher iron content of Sample No. 1 is due mostly to the higher pyrite content. The degree of freedom of the sulphides is also higher in Sample No. 2 than in Sample No. 1, as is shown in Table 8.

TABLE 8

Estimated Degrees of Freedom of the Minerals in Two Zinc Concentrates from Noranda Mines Limited

Minerals	Percentages (by weight) free from combination with other sulphides	
	Sample No. 1	Sample No. 2
Pyrite	50	90
Pyrrhotite	78	85
Chalcopyrite	66	30
Sphalerite	87	93

Most of the sulphides in Sample No. 2 are very finely divided.

It was observed that the sphalerite in the polished sections did not show significant variation in the colour of the internal reflections, and this was interpreted as indicating no wide variations in the iron content of the mineral. The screened, cleaned and magnetically split fractions, when viewed under the binocular microscope did, however, show a small amount of sphalerite which is very light amber in colour, and which obviously contains only a very small amount of iron. The amount is so small that it can be neglected in practical applications.

REFERENCE

(1) B.J. Skinner, P.B. Barton, and G. Kullerud, Economic Geology, 54, pp. 1040-1046 (1959).

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