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ROASTING OF MOLYBDENUM SULPHIDE CONCENTRATE FOR GEO-MET REACTORS LIMITED, OTTAWA

by

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EXTRACTION METALLURGY DIVISION

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SUMMARY OF RESULTS

Concentrate containing 18.35% molybdenum and 21.2% sulphur was roasted under oxidizing conditions in an electrically heated rotating retort. This retort was open at one end to permit air to enter and react with the charge. The object was to produce a calcine containing less than 0.5% sulphide sulphur. The amount of sulphate sulphur that was present in the calcines produced was not objectionable.

When the concentrate was charged into the retort which was at 625° C and then cylinder oxygen admitted, the calcine produced contained 1.6% total sulphur of which 0.11% was present as sulphide sulphur. When the concentrate was charged into the retort at 600° C and cylinder oxygen was not admitted, the calcine contained 3.0% total sulphur.

In the experiment performed to obtain an estimate of the time required for roasting, it was found that when the concentrate was charged into the retort which was at 625° C, the roasting was essentially completed within two hours.

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INTRODUCTION

A request to roast some molybdenum sulphide concentrate was received from Dr. W.A. Morgan, President and Managing Director, Geo-Met Reactors Limited, Ottawa, in a letter dated May 28, 1964. The details of the request were discussed further with Mr. M. Campbell of Geo-Met Reactors Limited.

The object of this work was to produce a calcine with less than 0.5% sulphide sulphur. This calcine would be used by Geo-Met Reactors Limited to determine the leaching parameters. It was understood that some sulphate sulphur in the calcine would not be objectionable in the leaching. After this objective had been achieved Mr. Campbell requested that an experiment be performed to obtain an estimate of the time required for roasting.

It was agreed that the chemical analysis required for the investigation would be performed by Geo-Met Reactors Limited. On June 19, 1964, about 100 lb of flotation concentrate was received for this work.

METHODS OF TREATING MOLYBDENUM CONCENTRATES

Molybdenum sulphide concentrate may be treated by two processes (1). In one, the concentrate is roasted at 1000 to 1050° C, the volatile oxide, molybdenum trioxide (MoO_3), passes through a metal flue and is collected in a bag filter as a fine light concentrate. The second treatment involves roasting at 600° C with a large excess of air. Temperatures must be closely controlled because volatilization losses are excessive at 700° C and the oxide melts at 800° C. The molybdenum in the calcine is present as molybdenum trioxide and its extraction and purification can be accomplished by leaching. In the present work the second method was used.

EQUIPMENT AND PROCEDURES

Roasting was performed in an open-end rotating retort 6 ft long x 16 in. ID which was electrically heated from the outside to give a hot zone 4 ft long. The thermocouple used to control the temperature in the furnace was located

between the retort and the furnace wall. Another thermocouple was placed in the hot end of the retort to measure the temperature in the charge. This thermocouple was connected to a recorder and the temperature was continuously recorded.

In all of the experiments the retort was left open to permit air to come in contact with the charge. Roasting was then accomplished by the reaction of sulphur in the charge and oxygen from the surrounding atmosphere. In one experiment oxygen in the atmosphere was supplemented with cylinder oxygen. This was accomplished by admitting the oxygen into the hot end of the retort through a 1/4 in. pipe. The stream of oxygen was directed at the surface of the charge.

An exhaust hood was located just above the open end of the retort and this provided a slight draught to remove the sulphur-bearing gases.

During each experiment samples of the calcine were taken with a long scoop. At the end of each experiment, when the calcine was removed and allowed to cool, a head sample was obtained by riffling.

In Experiment 1, the furnace was heated to the operating temperature, then 20 lb of concentrate was introduced. Three times during the experiment an addition of 5 lb of concentrate was made. It was thought that if all of the charge (35 lb) was added at once, the temperature in the charge might become excessive and the calcine would fuse. However later (in Experiment 3) it was found that 35 lb could be charged at the start of the experiment.

The procedure used in Experiment 2 was similar to that used in Experiment 1, except that shortly after the 20 lb of concentrate was introduced, cylinder oxygen was admitted into the retort through a 1/4 in. pipe.

In Experiment 3, the object was to obtain an estimate of the time required for roasting. The complete charge of 35 lb of concentrate was introduced at the start of the experiment when the temperature in the furnace had reached 625° C. This large a starting charge did not cause excessive heating. Samples of the calcine were taken every 15 min for the first hour, and every half hour for the next four hours after which the calcine was removed from the furnace.

EXPERIMENTAL WORK

The raw materials used in this work were the molybdenum concentrate and commercial cylinder oxygen. A head sample of the molybdenum concentrate was obtained by riffling and the chemical analysis is shown in Table 1.

TABLE 1

Chemical Analysis of the Molybdenum Concentrate

Constituent	(%)
Mo	18.35
Cu	6.05
Total S	21.2
Moisture	5.0

Three experiments were performed in this investigation. The product from the first two experiments was used by Geo-Met Reactors Limited to determine the leaching parameters. The third experiment was performed to obtain some estimate of the time required for roasting. The details of the experiments performed and the results obtained are shown in Table 2.

The results in Table 2 show that oxygen aids in roasting. When oxygen was used, the total sulphur content of the calcine was 1.6% and most of this was present as sulphate sulphur. When oxygen was not used the total sulphur content was 3.0%.

In Experiment 3 the results showed that roasting was essentially completed in less than two hours.

TABLE 2

Details of Experiments 1, 2 and 3 and the Chemical Analyses of the Products

Exp. No.	Time (hr)	Temp in the Furnace before charging (°C)	Temp in the charge (°C)	Oxygen Flow Rate (l/m)	Wt of Conc Charged (lb)	Sample No.	Wt of Product (lb)	Analysis of the Product (%)					
								Mo	Cu	Fe	Sulphur		
											Total	Sulphate	Sulphide**
1	0	600			20								
	0.5												
	1		625		5								
	1.5		595		5								
	2		580		5								
	3		510			1		19.25	5.83		3.2		
	3.5		570										
	4.25		600										
	4.5		585			2		19.30	6.05		2.8		
	5.5		530										
						3*	30.2	19.1	5.83	13.2	3.0		
2	0	625			20								
	0.25		590										
	0.5		630	13.7	5								
	1		655		5								
	1.5		635		5								
	2.5		625			1					1.5		
	3.5		630			2					1.6		
	4.5		630			3					1.6		
						4*	29.4				1.6	1.49	0.11
3	0	625			35.2								
	0.25		455			1					22		
	0.5		580			2					20.7		
	0.75		640			3					18.5		
	1.0		670			4					17.1		
	1.5		695			5					12.5		
	2		660			6					2.4		
	2.5		600			7					2.2		
	3.0		545			8					1.8		
	3.5		537			9					2.0		
	4.0		545			10					2.0		
	4.5		545			11					2.0		
	5.0		545			12					2.0		
						13*	30.3				2.2		

* Head sample of the calcine

** By difference

DISCUSSION

The product from Experiments 1 and 2 were combined by and used by Geo-Met Reactors Limited to determine the leaching parameters. The chemical analysis of the composite calcine is shown in Table 3.

TABLE 3

Chemical Analysis of the Composite Calcine from Experiments 1 and 2

Constituent	(%)
Mo	22.3
Oxidized Mo	21.0
Fe	13.0
Cu	6.0
S	2.1

The parameter, "Oxidized Molybdenum" shown in Table 3 is not a definite quantity but is indicative of the maximum amount of molybdenum which could be leached under ideal conditions. The actual amount of recoverable molybdenum will be about 90% of this figure.

CONCLUSION

1. Sufficient calcine suitable to determine the leaching parameters was produced by roasting the molybdenum sulphide concentrate with and without cylinder oxygen. The use of cylinder oxygen aided in lowering the total sulphur content of the calcine.

2. The roasting of the concentrate was essentially completed in less than two hours when the conditions of Experiment 3 were used.

REFERENCES

1. C.A. Hampel, "Rare Metals Handbook", Reinhold Publishing Corporation, New York, 271-289, (1954).

GVS/dm