CANADA

DEPARTMENT OF ENERGY, MINES AND RESOURCES

MINES BRANCH

OTTAWA

Mines Branch Investigation Report IR 70-32

DETERMINATION OF TUNGSTEN IN SAMPLES FROM CHEMEX LABORATORIES LIMITED

by

R. Kobus

NOTE: This report relates essentially to the samples as received. The report and any correspondence connected therewith shall not be used in full or in part as publicity or advertising matter.

Copy No.

June 3, 1970

Mines Branch Investigation Report IR 70-32

DETERMINATION OF TUNGSTEN IN SAMPLES FROM CHEMEX LABORATORIES LIMITED

by

R. Kobus*

- - -

ABSTRACT

An accepted colourimetric method for the determination of tungsten was applied to four samples of tungsten ore submitted by Chemex Laboratories Limited of Vancouver, B. C., who have been experiencing difficulty in obtaining acceptable tungsten analyses. This report discusses possible causes of the difficulty, and gives tungsten analyses for the four samples.

---

*Technician, Analytical Chemistry Section, Mineral Sciences Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

INTRODUCTION

During a visit by Mr. G. H. Faye of the Mineral Sciences Division, Chemex Laboratories Limited of Vancouver requested the advice of the Mines Branch on a difficulty experienced in the determination of tungsten by their laboratories. Chemex Labs is using the colourimetric thiocyanate method described in Mines Branch Technical Bulletin TB 37, decomposing the sample with a pyrosulphate fusion. They have found that tungsten values for some samples tend to be low and erratic.

Four samples from Chemex were submitted to the Analytical Chemistry Section, marked #14, #14, #17 and #50. The first two samples were re-marked #14-A and #14-B, and the four samples were assigned laboratory numbers 463, 464, 465 and 466, respectively. This report describes an investigation of the application of the tungsten method to these four samples.

PRELIMINARY TESTS

The four samples were carried through the procedure described in Technical Bulletin TB 37, using either the acid attack or the pyrosulphate fusion. In all cases, the reduction proceeded for one hour in a boiling water bath, and no extractions were performed. It was noted that the acid attack decomposed all four samples completely. The results are shown in Table 1.

TABLE 1

Sample	% WO_3	
	Acid Attack	Fusion
463	0.23, 0.21	0.23
464	0.79, 0.78	0.70, 0.68
465	0.65	0.62
466	0.38	0.40

It is seen that no difficulty has been experienced for samples #463, 465 and 466. The results for sample #464 show an unacceptable difference between the two methods of decomposition.

ANALYSIS OF SAMPLE #464

Further analyses of sample #464 were carried out using the method described, to discover the magnitude of the variations of the results. The results are shown in Table 2.

TABLE 2

% WO_3	
Acid Attack	Fusion
0.79	0.62, 0.69
0.78	0.55, 0.47
0.78	0.68

The results from the fusion vary over a wide range, whereas the results from the acid attack are quite reproducible.

In order to determine whether the variations are due to incomplete reduction of the tungsten, an additional sample was decomposed by fusion, and two aliquots taken through the balance of the procedure. One aliquot was reduced for one hour as usual, and the other for a period of two hours. In both cases, the results were identical at 0.58% WO_3. This indicates that the reduction step as described in the method is satisfactory.

EXAMINATION OF SAMPLES #464 and #465

Further evaluation was requested in order to obtain more information on sample #464. Sample #465, which exhibited normal analytical behaviour, was evaluated at the same time for comparison.

The two samples were examined by semi-quantitative spectrographic analysis (1). No significant differences were observed between the two samples.

The small quantities of the samples remaining from the above tests were subjected to a heavy liquid separation to concentrate the heavy minerals away from the bulk of the gangue material. The gangue was discarded and the concentrates examined by X-ray diffraction (2). The only tungsten mineral identified was scheelite. There were no differences of mineralogy between the two samples that could cause difficulties during the determination of tungsten.

SIZE DISTRIBUTION OF SAMPLE #464

The heavy minerals concentrate obtained for the mineralogical examination of sample #464 was passed through screens of 100 mesh and 200 mesh. The distribution is shown in Table 3.

TABLE 3

Mesh Size	Weight Distribution
+100 m	approx. 5%
-100+200 m	approx. 30%
-200 m	approx. 65%

This suggests that the sample may be too coarse to permit complete dissolution by pyrosulphate fusion. The coarseness did not affect the acid attack, as it was observed that the sample was completely dissolved.

The three size fractions mentioned in Table 3 were ground to -200 mesh by hand in an agate mortar and combined. Tungsten was determined in this ground concentrate, with the results shown in Table 4.

TABLE 4

Sample	$\%WO_3$	
	Acid Attack	Fusion
#464 concentrated and ground to -200 m	1.09	1.05
	1.11	1.06

These results show much better agreement for the two decomposition procedures, and indicate that the difficulty in analysis has two possible causes:

1. Presence of material in the gangue that prevents complete decomposition of the tungsten by pyrosulphate fusion.
2. Particle size is too coarse for effective fusion.

None of the original sample #464 was available for further tests. However, of the two causes, the second is judged to be the more likely.

USE OF TEFLON DISH

As the platinum dishes used in the acid attack are expensive (although they last for many years), it was decided to evaluate a Teflon dish as a possible replacement.

A Teflon dish was used for the acid attack of sample #464. After being used twice, the bottom of the dish was greatly distorted, leaving only a small portion of the dish in contact with the hotplate, and thus greatly increasing the time necessary to bring the contents to a paste of phosphoric acid. In addition, the inside of the dish was coated with a black substance that could not be removed with a policeman, using both concentrated hydrochloric acid and hot water.

Accordingly the use of a Teflon dish at the temperature necessary for this procedure is not recommended.

RECOMMENDATIONS

It is recommended that samples similar to the four examined in this report be decomposed in platinum dishes by the acid attack for the determination of tungsten as described in Mines Branch Technical Bulletin TB37.

If the acid attack is not possible, the samples should be pulverized at least to -200 mesh before attempting to dissolve them by pyrosulphate fusion.

ACKNOWLEDGEMENTS

The assistance of Mr. D. P. Palombo of the Spectrochemistry Section, and Mr. J. M. Stewart of the Crystal Structure Group, is gratefully acknowledged.

REFERENCES

1. Mineral Sciences Division Internal Report MS-AC-70-71 containing Spectrochemical Report SL 70-58, by D. P. Palombo, April 13, 1970.

2. Mineral Sciences Division Test Report CS-70-80, by J. M. Stewart, May 22, 1970.

= = =