

## TECHNICAL NOTES

### SOLVENT EXTRACTION PROCEDURE FOR THE DETERMINATION OF TUNGSTEN IN ORES

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#### INTRODUCTION

Atomic absorption methods have not been widely used for the determination of tungsten in ores due to its low sensitivity in aqueous solutions (1). A method has now been developed for solvent extraction of tungsten, making rapid determination of tungsten at low concentrations possible.

It was found that tungstates, when converted to phospho-tungstates, can be effectively extracted into di-isobutyl ketone (2-6 dimethyl-4-heptanone) (DIBK) containing Aliquat 336 (methyl tricapryl ammonium chloride from General Mills). This system was effectively used for the extraction of gold from cyanide solutions (2). Even in aqueous solutions, phospho-tungstates give greater sensitivity (37  $\mu\text{g}/\text{ml}$  for 1% absorption) compared to simple tungstates (63  $\mu\text{g}/\text{ml}$  for 1% absorption). Standard tungsten solutions for extraction studies were prepared by converting aqueous solutions of sodium tungstate to sodium phospho-tungstate by boiling with ortho phosphoric acid.

A Perkin-Elmer Model 303 atomic absorption spectrophotometer was used with a nitrous oxide-acetylene flame at a wavelength of 4008.75 Å.

#### PROCEDURE

Weigh 0.2 g of sample and 1 g anhydrous lithium metaborate, mix thoroughly and fuse in preignited graphite fusion crucibles (3). Pour the melt into a 50-ml test tube containing approximately 40 ml of 5-10% phosphoric acid. Bring the solution to boil on a magnetic stirrer hot plate using aluminum blocks drilled to hold the test tubes. With a preheated hot plate, complete dissolution can be made in a few minutes. Any precipitates formed will not interfere

with the analysis. When cool, add 10 ml di-isobutyl ketone containing 3% Aliquat 336. Shake vigorously for three minutes, centrifuge and read the atomic absorption against standards prepared from tungstic acid fusions as described above, extracted similarly with di-isobutyl ketone containing 3% Aliquat. The concentration of phosphoric acid and lithium metaborate should be the same for the aqueous sample and standard solutions. The procedure is adequate for ores containing more than 0.5% tungsten, which will give 16% absorption at 1x scale expansion. A sacrifice of precision will permit analysis of samples containing lower tungsten.

#### ANALYSIS OF TUNGSTEN ORES

Samples of silicate tungsten ores were analyzed by this technique. Table I shows a comparison of chemical and the solvent extraction techniques. The results are in good agreement showing the effectiveness of the suggested technique.

TABLE I  
 Analysis of Tungsten Ores

Sample No.	U.S.B.M. No.	% WO <sub>3</sub> Chemical by U.S.B.M.	% WO <sub>3</sub> Solvent Extraction		
1	6-10	5.3	5.3	5.4	5.5
2	6-95	2.0	2.1	2.0	2.1
3	5-2742	0.42	0.45	0.44	0.44

#### EXPERIMENTAL

Table II shows the effect of Aliquat concentration in

DIBK on the extraction of tungsten. One percent Aliquat concentration is adequate for extraction of aqueous solutions, however, with tungsten ores, 3% Aliquat was found to be necessary for complete extraction. Increase in Aliquat concentration, however, reduces sensitivity due to increased viscosity of the organic layer.

**TABLE II**  
Effect of Aliquat 336 Concentration  
On Extraction of Tungsten

% Aliquat 336 in DIBK	Absorbance (1000 $\mu\text{g}/\text{ml W}$ )
1	0.39
2	0.38
3	0.38
5	0.36

Table III shows the effect of phosphoric acid concentration. The extraction efficiency was improved up to 5%

**TABLE III**  
Effect of Phosphoric Acid Concentration  
On Extraction of Tungsten

$\text{H}_3\text{PO}_4$ (v/v)	Absorbance (1000 $\mu\text{g}/\text{ml W}$ )
0.5	0.15
1	0.21
3	0.15
5	0.36
10	0.36
20	0.33

phosphoric acid after which further increase did not have any appreciable effect.

To test the efficiency of extraction, 25 ml of 10,000  $\mu\text{g}/\text{ml}$  tungsten standard was extracted with 25 ml of DIBK containing 1% Aliquat 336. Ten ml of the organic layer was evaporated in a weighed platinum crucible and ignited at 750°C. The  $\text{WO}_3$  residue was weighed and recovery of tungsten in the organic layer was calculated. Extraction was also determined for other systems and Table IV summarizes the results. The best extraction of 91% was obtained with the DIBK-Aliquat 336 system.

**TABLE IV**  
Recovery of Tungsten

Extraction System	Recovery, %
DIBK alone	76
DIBK + 1% Aliquat 336	91
MIBK alone	30
MIBK + 1% Aliquat	50

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