



DEPARTMENT OF
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OTTAWA

*IMPROVEMENT OF A CONTINUOUS
PROCESS FOR THE PRODUCTION OF
HIGH-PURITY TUNGSTIC TRIOXIDE
FROM SCHEELITE: PRECIPITATION
AND REMOVAL OF IRON WITH
AMMONIUM AND MAGNESIUM CHLORIDES*

J. A. VEZINA AND W. A. GOW

EXTRACTION METALLURGY DIVISION

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HIGH-PURITY TUNGSTIC TRIOXIDE FROM SCHEELITE;
PRECIPITATION AND REMOVAL OF IRON WITH AMMONIUM
AND MAGNESIUM CHLORIDES

by

J. A. Vezina* and W. A. Gow**

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ABSTRACT

Test work was carried out to remove iron and other impurities from a sodium tungstate solution. This constituted an improvement to the process previously developed at the Mines Branch for refining Canadian scheelite concentrates of grades as low as 41 per cent WO_3 . In this test work, by making the intermediate sodium tungstate solution ammoniacal and adding specific quantities of magnesium chloride and ammonium chloride, the iron concentration was decreased from 0.33 to 0.007 gram Fe per litre.

When the basic process was modified to include this iron-removal step, it was capable of producing ammonium paratungstate of a chemical purity suitable for the production of tungsten metal.

The additional cost involved in the precipitation of the iron from the process circuit was \$0.023 per pound of tungsten obtained.

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Direction des mines

PERFECTIONNEMENT D'UN PROCÉDÉ DE PRODUCTION CONTINUE
DE TRIOXYDE DE TUNGSTÈNE TRÈS PUR À PARTIR DE LA
SCHEELITE PAR LA PRÉCIPITATION ET L'ÉLIMINATION DU
FER AU MOYEN DES CHLORURES D'AMMONIUM ET DE MAGNÉSIUM

par

J. A. Vezina* et W. A. Gow**

RÉSUMÉ

Divers essais ont été effectués afin d'éliminer le fer et les autres impuretés d'une solution de tungstate de sodium. On a ainsi amélioré le procédé mis au point antérieurement à la Direction des mines pour l'affinage des concentrés de scheelite canadienne titrant aussi peu que 41 p. 100 de WO_3 . Au cours de ces essais, en rendant ammoniacale la solution intermédiaire de tungstate de sodium et en y ajoutant des quantités déterminées de chlorure de magnésium et de chlorure d'ammonium, il a été possible de réduire la concentration en fer de 0.33 à 0.007 gramme par litre.

Après que le procédé de base eut été modifié de façon à inclure cette étape de l'élimination du fer, on a été en mesure de produire du paratungstate d'ammonium d'une pureté chimique convenant à la production de tungstène métal.

Le coût additionnel que représente ce procédé de précipitation du fer est de \$0.023 par livre de tungstène produite.

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INTRODUCTION

In a previous study⁽¹⁾ a general procedure was developed for processing Canadian scheelite concentrates, of grades between 41 and 64 per cent WO_3 , for the production of tungstic trioxide and ammonium paratungstate. This procedure, which can be used for both batch and continuous operations, consisted of decomposing the scheelite mineral with hydrochloric acid, dissolving the crude tungstic acid with sodium hydroxide, and converting the sodium tungstate solution to ammonium tungstate by means of a cation-exchanging resin. The ammonium tungstate was recovered from the solution by two methods: in the first, the solution was evaporated until crystals appeared, and the tungsten was precipitated by neutralizing the solution with nitric acid; in the second, the solution was evaporated until 78 per cent of the tungsten had crystallized. The product of the first method contained about 0.5 per cent impurities, and the ammonium paratungstate produced by the second contained less than 0.05 per cent impurities. The analysis of this latter product, reproduced from previous work⁽¹⁾, is shown in Table 1. The mother liquor resulting from the partial crystallization of tungsten in the second method, and containing 22 per cent of the tungsten processed, may be treated with calcium chloride by a well-known method⁽²⁾ to produce artificial scheelite, a material widely accepted for producing ferro-tungsten.

TABLE 1 (1)

Analysis of Ammonium Paratungstate Produced
from Low-Grade Scheelite Concentrate

		<u>Per Cent</u>		
}	Fe	-	0.014	
	P	-	0.003	
	* Si	-	0.007	
	Mn	-	<0.005	
	Na	-	<0.005	
**	Mo	-	0.013	
	{Others	-	<0.02	total, but each less than 0.01 per cent.

*Reproduced from Table 8, reference 1.

**Reproduced from Table 7, reference 1.

Ammonium paratungstate of the composition shown in Table 1 is probably of chemical quality suitable for the preparation of tungsten metal of high purity, but producers of tungsten metal probably would prefer to start with a material containing less than 0.05 per cent total impurities, with no single impurity being of a concentration exceeding 0.01 per cent. The product the analysis of which is shown in Table 1 did not meet these specifications, since the iron and molybdenum concentrations were greater than 0.01 per cent.

Methods for eliminating molybdenum from tungstate solutions are known to tungsten processors⁽³⁾, and no additional work was carried out in that direction. The work reported here was thus undertaken to find a method, which was both economical and incorporatable into our process⁽¹⁾, to eliminate sufficient iron from our process circuit so that ammonium paratungstate containing less than 0.01 per cent Fe could be produced.

In the course of our previous work with tungsten concentrates, it was observed that the alkaline solutions of sodium tungstate prepared by our general procedure contained iron and phosphorus in ratios that appeared reasonably constant. This observation led us to assume that the iron and phosphorus in the solutions were combined in the form of a soluble complex ion. This assumption appeared reasonable to us because soluble complex ions of iron and phosphorus are known to exist in alkaline solutions.

In searching the literature for a method to eliminate iron from sodium tungstate solutions, a reference was found to a procedure, used for the production of artificial scheelite, which appeared suitable for our purpose. This method was based on the fact that the phosphates of ammonium and of some metals such as magnesium and manganese are almost insoluble in ammoniacal solutions⁽²⁾⁽⁴⁾. Mention was also made of the fact that when the solutions contained small quantities of phosphorus, the precipitation of the double phosphates was hastened by stirring vigorously and cooling⁽⁵⁾. This information led the authors to speculate that the iron-phosphorus complex ion in the sodium tungstate solutions might be broken by deliberately adding ammonium chloride and magnesium chloride. If the complex could be broken, the phosphorus would be precipitated by the ammonia, magnesium and manganese, while the iron would precipitate as the hydroxide. Of course, adding magnesium to the solution meant that an additional impurity was being introduced into the process circuit, but this was not thought to be an objection, because it appeared probable that the magnesium would be removed from the solution along with the sodium during the conversion of the solution to ammonium tungstate in the operation that followed⁽¹⁾.

This possible approach was attractive for three reasons. First, it held the hope of removing the iron, phosphorus and manganese from the process circuit. Secondly, the possibility existed that the silica would also be removed, because silica has a tendency to be carried with the double phosphate precipitate⁽²⁾. Thirdly, it appeared to be both economical and suitable for easy integration into the process circuit.

Our work was thus planned to find conditions for the efficient and economical elimination of the iron from a sodium tungstate solution which had been prepared by our general procedure from a low-grade Canadian scheelite concentrate by precipitation of the double phosphate of ammonium and magnesium and iron hydroxide.

EXPERIMENTAL PROCEDURE

The sodium tungstate solution used in this investigation was prepared from a low-grade scheelite concentrate supplied by Canada Tungsten Mining Corporation, Limited. The procedure used for preparing the sodium tungstate was described in a previous report⁽¹⁾. The analyses of the scheelite concentrate and of the sodium tungstate solution are reported in Tables 2 and 3.

In the first series of tests, 100-ml volumes of the sodium tungstate solution were used. Ammonium chloride and magnesium chloride were added in the crystalline form. For close temperature control, the tests were carried out in open Teflon beakers resting in a water bath. Teflon was used to prevent contamination of the test products with silica. Each solution was agitated vigorously for half an hour (the duration of each test) at the specified temperature and then settled and filtered, using a bed of cellulose powder and Büchner funnel.

In the second and third series of tests, concentrated ammonium hydroxide was added to the solutions before adding the ammonium and magnesium chlorides. Otherwise, the procedure described for the first series of tests was followed.

Finally, the conditions which were found most efficient for the elimination of the impurities from the tungstate solution in the three series of tests, were used on a fresh batch of the solution. The purified solution from this work was then passed through a 500-ml column of Dowex 50W x 8 and the ammonium tungstate solution thus produced was evaporated, but not to completion, to crystallize ammonium paratungstate. The crystals of ammonium paratungstate were washed with water and dried at 100°C.

TABLE 2

Analysis of Scheelite Concentrate

<u>By Chemical Methods</u>		<u>By Spectrographic Method</u>	
	<u>Per Cent</u>		<u>Per Cent</u>
WO ₃	- 41.3	Al	- 0.35
CaO	- 17.0	Mn	- 0.19
Mo	- 0.002	Mg	- 0.25
SiO ₂	- 15.8	Ti	- 0.03
Fe	- 11.7		
P	- 0.07		
S	- 3.2		
As	- <0.01		
Bi	- 0.04		

TABLE 3

Analysis of Stock Sodium Tungstate Solution

<u>Grams/litre</u>	<u>Grams/litre</u>
W - 73.4	Fe - 0.33
Na- 22.6	Mn - 0.009
P - 0.062	SiO ₂ - 0.70

The experiments were carried out with C.P.-grade reagents. The quantities of magnesium chloride and ammonium chloride added to the sodium tungstate solution were varied as required, but always in the proportion expressed by the formula $\text{MgCl}_2 \cdot \text{NH}_4\text{Cl} \cdot 6\text{H}_2\text{O}$.

EXPERIMENTAL RESULTS

In the first series of tests, which are reported in Table 4, ammonium and magnesium chlorides were added to the sodium tungstate stock solution in amounts ranging between 1 and 100 grams $\text{MgCl}_2 \cdot \text{NH}_4\text{Cl} \cdot 6\text{H}_2\text{O}$ per litre of solution, and the tests were carried out at temperatures ranging between 10 and 40°C. The results of these tests showed that adding ammonium and magnesium chlorides to the sodium tungstate solution caused the precipitation of some iron, but that, even when a large excess of the reagents was added, the quantities of iron precipitated were far short of the results desired. It did not appear that varying the temperature of the solution from 10 to 40°C had had any significant effect.

In the second series of twelve tests, which are reported in Table 5, the sodium tungstate stock solution was made ammoniacal by adding 25 grams NH_4OH per litre of solution, and was then treated with ammonium and magnesium chlorides at various temperatures as in the first series of tests. The results of these tests showed that, in all cases, the iron concentration in the solution was decreased drastically. The significance of varying the additions of ammonium and magnesium chlorides between 1 and 100 grams $\text{MgCl}_2 \cdot \text{NH}_4\text{Cl} \cdot 6\text{H}_2\text{O}$ per litre of solution to the ammoniacal sodium tungstate solution is shown in Table 6. These results show that when carried out at 10°C, the iron precipitation achieved with 10 grams of magnesium-ammonium chlorides per litre was practically the same as that obtained when 100 grams of magnesium-ammonium chlorides per litre was used.

The results of Table 5 show that, within each of the three groups of tests, the temperature was not a critical factor, provided it was 30°C or less. This point has been brought up because, at the industrial scale, the cooling of large volumes of solution may be expensive and, unless imperatively required, should be avoided. This second series of tests has served to show conclusively that the sodium tungstate solution must be made ammoniacal prior to treating with ammonium and magnesium chlorides, if a reasonable precipitation of the iron is to be obtained.

TABLE 4

Effects of Adding Magnesium Chloride and Ammonium Chloride to the Sodium Tungstate Solution in First Series of Tests

Test No.	Temp. (°C)	MgCl ₂ .6H ₂ O added (g/l)	NH ₄ Cl added (g/l)	Analyses of Solutions (g/l)					
				Fe	Mn	P	SiO ₂	Total	Mg
<u>Stock Sodium Tungstate Solution</u>									
				0.33	0.009	0.062	0.70	1.101	-
<u>Purified Sodium Tungstate Solutions</u>									
1	10	0.79	0.21	0.26	0.006	0.067	0.53	0.863	0.08
2	20	0.79	0.21	0.29	0.007	0.080	0.67	1.047	0.10
3	30	0.79	0.21	0.29	0.007	0.082	0.69	1.069	0.11
4	40	0.79	0.21	0.29	0.007	0.078	0.70	1.075	0.11
5	10	7.9	2.1	0.25	0.007	0.040	0.58	0.877	0.83
6	20	7.9	2.1	0.25	0.007	0.068	0.60	0.925	0.83
7	30	7.9	2.1	0.25	0.007	0.066	0.62	0.943	0.92
8	40	7.9	2.1	0.26	0.007	0.058	0.55	0.875	0.88
9	10	79	21	0.14	0.002	0.018	0.19	0.350	6.73
10	20	79	21	0.16	0.002	0.033	0.23	0.425	7.59
11	30	79	21	0.09	0.002	0.029	0.35	0.471	8.13
12	40	79	21	0.16	0.003	0.014	0.33	0.507	8.72

TABLE 5

Effects of Adding Magnesium Chloride and Ammonium Chloride to the Ammoniacal Sodium Tungstate Solution in the Second Series of Tests

Test No.	Temp (°C)	MgCl ₂ .6H ₂ O added (g/l)	NH ₄ Cl added (g/l)	NH ₄ OH added (g/l)	Analyses of the Solutions (g/l)					
					Fe	Mn	P	SiO ₂	Total	Mg
<u>Stock Sodium Tungstate Solution</u>										
					0.33	0.009	0.062	0.70	1.101	-
<u>Purified Sodium Tungstate Solutions</u>										
13	10	0.79	0.21	25	0.064	0.004	0.056	0.28	0.404	0.044
14	20	0.79	0.21	25	0.064	0.004	0.069	0.28	0.417	0.053
15	30	0.79	0.21	25	0.070	0.004	0.075	0.14	0.284	0.053
16	40	0.79	0.21	25	0.150	0.005	0.071	0.56	0.786	0.062
17	10	7.9	2.1	25	0.013	0.002	0.008	0.04	0.063	0.39
18	20	7.9	2.1	25	0.021	0.002	0.011	0.06	0.094	0.42
19	30	7.9	2.1	25	0.034	0.003	0.014	0.10	0.151	0.43
20	40	7.9	2.1	25	0.023	0.002	0.009	0.07	0.104	0.48
21	10	79	21	25	0.010	0.001	0.007	0.04	0.058	7.10
22	20	79	21	25	0.017	0.001	0.007	0.06	0.085	7.68
23	30	79	21	25	0.012	0.001	0.008	0.04	0.061	7.58
24	40	79	21	25	0.058	0.003	0.012	0.13	0.203	8.30

TABLE 6

The Significance of Adding Magnesium Chloride and Ammonium Chloride to the Ammoniacal Sodium Tungstate Solution in the Second Series of Tests

Test No.	Temp. (°C)	MgCl ₂ .NH ₄ Cl.6H ₂ O added (g/l)	NH ₄ OH added (g/l)	Fe Precipitated (%)
13	10	1	25	80.6
17	10	10	25	96.1
21	10	100	25	97.0

The 25 grams/litre proportion of ammonium hydroxide, used to make the sodium tungstate solution ammoniacal in the second series of tests, had been chosen arbitrarily and therefore, in a third series of tests (which are reported in Table 7), the effects of increasing the additions to 50 and 75 grams of NH₄OH per litre were sought. As in the two previous series of tests, the ammonium and magnesium chlorides additions were varied between 1 and 100 grams of MgCl₂.NH₄Cl.6H₂O per litre. In this series, the precipitation temperature was maintained at 30°C for all of the tests.

Increasing the ammonium hydroxide from 25 to 50 grams of NH₄OH per litre appeared to be advantageous in one test only (Test 29), in that when the solution was treated with 100 grams of MgCl₂.NH₄Cl.6H₂O per litre, the iron precipitation was increased from 96.4 to 97.9 per cent; however, increasing the ammonium hydroxide addition from 50 to 75 grams NH₄OH per litre did not improve the iron precipitation further. In the first two groups of tests reported in Table 7, in which the chlorides additions were 1 and 10 grams of MgCl₂.NH₄Cl.6H₂O per litre, increasing the ammonium hydroxide addition from 25 to 50 to 75 grams NH₄OH per litre did not improve the iron precipitation efficiency.

TABLE 7

Effects of Adding Magnesium Chloride and Ammonium Chloride to the Ammoniacal Sodium Tungstate Solution in the Third Series of Tests

Test No.	Temp (°C)	MgCl ₂ .6H ₂ O added (g/l)	NH ₄ Cl added (g/l)	NH ₄ OH added (g/l)	Analyses of the Solution (g/l)					
					Fe	Mn	P	SiO ₂	Total	Mg
<u>Stock Sodium Tungstate Solution</u>										
					0.33	0.009	0.062	0.70	1.101	-
<u>Purified Sodium Tungstate Solutions</u>										
15	30	0.79	0.21	25	0.070	0.004	0.075	0.14	0.284	0.05
25	30	0.79	0.21	50	0.15	0.002	0.064	0.25	0.466	0.03
26	30	0.79	0.21	75	0.12	0.002	0.068	0.24	0.430	0.03
19	30	7.9	2.1	25	0.034	0.003	0.014	0.10	0.151	0.43
27	30	7.9	2.1	50	0.039	0.001	0.009	0.05	0.099	0.22
28	30	7.9	2.1	75	0.033	<0.001	0.013	0.04	0.087	0.12
23	30	79	21	25	0.012	0.001	0.008	0.04	0.061	7.58
29	30	79	21	50	0.007	<0.001	0.005	0.01	0.022	5.80
30	30	79	21	75	0.007	<0.001	0.006	0.03	0.043	5.29

The results of the three series of tests reported in Tables 4, 5 and 7 have established conditions for decreasing the iron in the sodium tungstate solution from 0.33 to 0.007 gram Fe per litre. These conditions were those used in Test 29, Table 7. However, the large quantity of magnesium chloride used to achieve this result presented a serious disadvantage in that the iron-purified solution was contaminated with 5.80 grams Mg per litre. Quantitative tests showed that passing the solution through a column of Dowex 50W x 8 to convert the solution to ammonium tungstate failed to remove the magnesium completely from the solution, and that the converted solution still contained about 0.04 part of Mg per 100 parts of WO_3 . This work showed that the use of 100 grams of $MgCl_2 \cdot NH_4Cl \cdot 6H_2O$ per litre to precipitate about 98 per cent of the iron from the solution resulted in an ammonium paratungstate product that had an unacceptably high magnesium content.

In the second group of tests of Table 7, in which 10 grams of $MgCl_2 \cdot NH_4Cl \cdot 6H_2O$ per litre was added, increasing the ammonium hydroxide addition from 25 to 50 g NH_4OH per litre did not improve the iron precipitation further, but it did have desirable effects in that, in addition to precipitating 88 per cent of the iron it achieved the removal of about 90 per cent of the manganese, 87 per cent of the phosphorus and 93 per cent of the silica from the solution. Of course, the tungsten could not be precipitated completely as ammonium paratungstate of high purity from the solution because the total-impurities level was in excess of the desired 0.05 per cent. However, the conditions used to achieve these results were attractive because of the moderate quantities of reagents used and the small quantity of magnesium retained in the purified solution, which would perhaps permit the partial crystallization of ammonium paratungstate of desired purity from the converted solution of ammonium tungstate.

In a final test, the iron-purified solution of Test 27 was converted to ammonium tungstate, using a column of Dowex 50W x 8. The solution was then evaporated until 82 per cent of the tungsten had been crystallized as ammonium paratungstate. The crystals were washed with distilled water, dried, and analysed. The analysis of this product is shown in Table 8.

TABLE 8

Analysis of Ammonium Paratungstate

	<u>Per Cent</u>		<u>Per Cent</u>
W	- 69.9	Mg	- 0.002
Ca	- 0.004	Mn	- <0.001
Fe	- 0.002	Cl	- <0.01
P	- 0.002	Na	- 0.001
Si	- <0.006		

The ammonium paratungstate produced was of a chemical quality that satisfied our objective in that it contained less than 0.05 per cent total impurities and (except for molybdenum, which we did not attempt to eliminate for reasons previously mentioned) less than 0.01 per cent of any single impurity.

DISCUSSION OF RESULTS

This investigation has resulted in a means for improving the previously described basic process⁽¹⁾ for the continuous production of ammonium paratungstate from scheelite concentrates of grades as low as 41 per cent WO_3 . The improvement involves the precipitation of iron and phosphorus, which are both present as impurities - presumably iron hydroxide and ammonium magnesium phosphate - in the sodium tungstate solution produced in the basic process. The application of this improvement permits recovery of 82 per cent of the tungsten from the sodium tungstate solution in the form of high-purity ammonium paratungstate, and the remaining 18 per cent as artificial scheelite. Thus the improved process permits of recovering all the tungsten extracted from the low-grade scheelite concentrate as marketable products.

Therefore, assuming that the process can extract 98 per cent of the tungsten from the low-grade concentrate and that, of this tungsten, 82 per cent can be marketed as high-purity ammonium paratungstate at \$3.00 per pound of contained tungsten and 18 per cent as artificial scheelite at \$2.00 per pound of contained tungsten, then the following appraisal can be made:

1. <u>Revenue from Sale of Products</u> (per pound of tungsten in concentrate)	
Ammonium paratungstate	$0.98 \times 0.82 \times \$3.00 = \$2.42$
Artificial scheelite	$0.98 \times 0.18 \times \$2.00 = 0.35$
	<u>\$2.77</u>
2. <u>Reagent Cost</u> (per pound of tungsten in concentrate)	
Basic process (1)	\$0.306
Iron precipitation	
Ammonia	\$0.017
Magnesium chloride	0.003
Ammonium chloride	0.003
	<u>\$0.329</u>
3. <u>Net Return on Basis of Reagents Only</u> (per pound of tungsten in concentrate) ...	<u>\$2.44</u>

Of course, a process cannot be evaluated on the basis of reagent cost alone, but these calculations show that the improved process has the potential of providing an important advantage to a tungsten concentrate producer--it permits producing marketable products from low-grade scheelite concentrates, thus giving him an alternative to marketing high-grade concentrates with strict specifications. The worth of the process can be fully evaluated only by a concentrate producer who is in a position to equate the costs he would incur in refining a low-grade concentrate to market products, with the tungsten recovery loss and costs he must bear in upgrading a low-grade concentrate to a grade acceptable to buyers.

CONCLUSIONS

This work has provided a method for purifying sodium tungstate solution which, when incorporated in the basic process (previously described⁽¹⁾) for refining low-grade scheelite concentrates,

permits of producing marketable products with high tungsten recovery at a reagent cost of \$0.329 per pound of tungsten. The improved process presents a tungsten concentrate producer with an alternative to the production of high-grade concentrates which must meet a variety of strict specifications.

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