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TECHNOLOGY—FEED MATERIALS

0
PROGRESS REPORT

on

REFINING OF MgX AND OTHER URANIUM-BEARING MATERIALS

by

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ABSTRACT

The effects of acidity on extraction of vanadium and molybdenum by diethyl ether are described. Solubility data for several vanadium compounds in nitrate process solutions are presented. Preliminary results on the extraction of uranium from process slurries by tributyl phosphate are reported, including data on the contaminants extracted with the uranium.

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2 INTRODUCTION

Considerable attention was devoted during the month to the problem of vanadium in the refining of MgX. It now appears reasonably certain that vanadium boil-down precipitates (obtained during the concentration of nitric acid digest liquors) will present no problem with respect to possible losses of uranium. The problem of vanadium transfer to the ether extract is a real one, however, and is being studied intensively. Control of acidities during extraction appears to offer promise as one method of greatly reducing the transfer of both vanadium and molybdenum.

A preliminary examination of the use of tributyl phosphate (diluted with a refined kerosene) for the solvent extraction of uranium process slurries has indicated that this solvent has two important advantages over diethyl ether: (1) uranium distribution coefficients are greater than those for ether, by factors of up to ten-fold, and (2) distribution coefficients for other components are less than those encountered in acid-ether extractions. By utilization of these two factors, it might be possible to produce specification uranium by only one extraction.

SUMMARY

Further investigation of the effects of acidity on the transfer of vanadium to the ether extract concurrently with the uranium has shown the desirability of low acid levels. When process solutions, containing less than 10 grams of excess acid/liter, were extracted with ether 0.25N in HNO₃, or lower, vanadium transfer was of the order of only 2 to 4.

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per cent. The vanadium content of the ether extract under these conditions was only 150 to 250 p.p.m., on a uranium basis. Uranium extraction did not appear to be adversely affected by the conditions used. Under the same conditions, the transfer of molybdenum was even further reduced. In one test, only one per cent of the molybdenum appeared in the extract; 99 per cent remained in the raffinate. On a uranium basis, the molybdenum content of the extract was only 135 p.p.m.

These results were obtained on batch extractions, with no scrub. The additional decontamination attainable by a water scrub remains to be investigated. Additional data, particularly on continuous extractions, are desirable to confirm the preliminary hand shakeout tests.

One series of tests indicated that boiling down of MgX slurry resulted in unusually high vanadium transfer, even when the extractions were conducted under conditions of low acidity. These extracts were highly colored and it has been hypothesized that boiling down in contact with the siliceous solid phase promoted the formation of an ether-soluble vanadate complex. When the solid phase was removed before boiling down, the extracts were low in vanadium and were not highly colored. This phenomenon is to be investigated further, but on the basis of the work to date, it appears that boiling down of MgX slurries should be avoided in the plant.

Study of the effects of acidity on the solubility of vanadium pentoxide, uranyl vanadate, and sodium uranyl vanadate in process solution showed that the solubility of the salts increased slightly (from 1 to 5 grams of vanadium/liter) with increasing acidity. Over the same range

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of acidity (0-120 grams of excess acid/liter), the solubility of vanadium pentoxide was essentially constant, at 1.6 grams of vanadium/liter.

Lengthening the calming sections of the Scheibel continuous-extraction column, and reducing the number of stages, so that it more nearly corresponded to the similar Scheibel column at Plant 6, produced no appreciable changes in operating characteristics or results.

Tributyl phosphate diluted with a refined kerosene was subjected to a preliminary investigation as a replacement for diethyl ether in the solvent extraction of uranium from process slurries prepared from MgX and pitchblende. Batch extractions showed that high uranium distribution coefficients were attainable and that the distribution coefficients for many of the other components of process solutions were quite low. The important distribution ratio, uranium to contaminants, appears to be greater than with diethyl ether, so that a higher level of decontamination should be attainable. On the basis of preliminary results, there is some reason to believe that it may be possible to produce specification uranyl nitrate by a one-step extraction with thorough scrubbing of the organic phase.

Several continuous extractions were run on the Scheibel column, using tributyl phosphate diluted with a petroleum solvent. The uranium contents of the raffinates were as low as 0.02 to 0.09 gram of uranium per liter. These values were much lower than those previously obtained with diethyl ether, even in those tests with the aqueous phase continuous. Spectrographic analyses of the extracts from these tests showed them to be of reasonably high purity.

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X-ray-diffraction studies of several ignited samples of actual and synthetic MgX disclosed the presence of a major phase common to all. However, it has been impossible to identify this unknown phase from available diffraction data.

EXPERIMENTAL DETAILS AND DISCUSSION OF RESULTS

Ether-Extraction Studies

Most of the batch ether-extraction studies of the month were concerned with the problems of vanadium and molybdenum transfer, although some interesting uranium distribution coefficients were obtained in several tests in which MgX was extracted alone. Some continuous extractions were conducted using the Scheibel column, although this unit was utilized more extensively for the preliminary investigation of tributyl phosphate extraction of uranium from process slurries.

Extraction of Vanadium and Molybdenum

The previous report (BMI-247) noted that the transference of vanadium to the ether phase appeared to be appreciably affected by acidity. Further investigation has confirmed the earlier observation that the extraction of vanadium can be substantially reduced by controlling acidities.

In one series of tests, the effects of varying acidities of the process solution feed and of the ether were examined, in the low acid range, since this appeared to produce the optimum results. A filtered

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pitchblende-MgX process solution was used, which contained about 1.2-1.5 grams of vanadium/liter. This vanadium concentration appears to be comparable to that which can be expected in the proposed plant feed. The results of these tests, shown in Table 36, indicated that only about 2-4 per cent of the vanadium was extracted with the uranium. This compares with the 20 per cent, or more, extracted when a similar solution (containing 40 grams/liter of excess acid) was extracted with 1N ether. On a uranium basis, the extracts in the present series contained 150-250 p.p.m. of vanadium.

In another series of tests, the effect of the presence of a boil-down precipitate during the extraction was examined. In these tests, MgX slurry (P-1) was filtered, and the filtrate was concentrated by boiling until the characteristic reddish boil-down precipitate was observed. This occurred at a specific gravity of about 1.8 (measured hot). The samples were diluted with water to about 56° Bé and extracted, with the results shown in Table 37. Vanadium extraction was approximately 2-3 per cent. The solids remaining in the raffinate were filtered off and analyzed for uranium and vanadium. Confirming earlier analyses of boil-down precipitates, these analyses showed that the precipitates, although high in vanadium, were virtually barren of uranium.

One striking feature of these MgX-extraction tests was the high uranium distribution coefficients, which ranged from about 1.5 to 7.0. These are the equal of those obtained on simple uranium nitrate-magnesium nitrate systems. On mixed pitchblende-MgX feeds of comparable concentration and specific gravity, the usual uranium distribution coefficient has only been of the order of 0.3 to 2.0. Insufficient data

TABLE 36. EFFECT OF ACIDITY ON EXTRACTION OF VANADIUM AND MOLYBDENUM FROM FILTERED PROCESS SOLUTION(1)

Excess Acid		Vanadium			Uranium			Uranium
Feed	Raff.	Ext.	Raff.	Total mg.	Ext.	Raff.	Total mg.	Extract V, p.p.m.(2)
<u>Neutral Ether</u>								
9.5 g./l.	pH 0.9	2.4	97.6	69.7	98.3	1.7	11,650	150
pH 0	pH 1.0	2.5	97.5	64.6	95.9	4.1	11,440	145
pH 0.5	pH 1.5	3.3	96.7	53.8	90.1	9.9	11,350	175
pH 1.1	pH 2.0	3.1	96.9	76.4	81.0	19.0	12,050	-
<u>0.1N Ether</u>								
9.5 g./l.	pH 0.3	3.2	96.8	71.3	98.4	1.6	11,090	210
pH 0	pH 0.2	2.9	97.1	57.7	98.7	1.3	10,380	165
pH 0.5	pH 0.2	3.8	96.2	47.8	98.2	1.8	11,780	155
pH 1.1	pH 1.7	2.9	97.1	72.1	84.9	4.1	11,840	-
<u>0.25N Ether</u>								
9.5 g./l.	pH 0	4.4	95.6	68.0	98.8	1.2	11,830	255
pH 0	pH 0	3.0	97.0	59.8	99.0	1.0	11,360	160(3)
pH 0.5	pH 0	3.5	96.5	62.2	99.0	1.0	11,470	195
pH 1.1	pH 0.5	3.7	96.3	80.0	97.0	3.0	12,290	250

(1) Process solution 5269-81, prepared so that 2/3 of uranium derived from pitchblende Blend A-1 and 1/3 derived from MgX blend B-1. Sp. gr. approximately 56° Bé in all tests. Acidity adjusted with MgO. 50-ml. samples, given 6 shakeouts with equal volumes of ether, using the normal time cycle.

(2) Uranium basis.

(3) Mo content of extract was 135 p.p.m. (uranium basis). Mo extraction = 1 per cent.

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TABLE 37. EFFECT OF ACIDITY ON THE EXTRACTION OF VANADIUM FROM MgX FEED SOLUTIONS CONTAINING BOIL-DOWN PRECIPITATES⁽¹⁾

Ether Normality (HNO ₃)	Excess Acid in Feed, g./l.	Vanadium			Total mg. Found	Uranium			Total mg. Found
		Ether Ext.	Per Cent in Raffinate Ppt.	in Filt.		Ether Ext.	Per Cent in Raffinate Ppt.	in Filt.	
Neutral	14	3.0	89.4	7.6	156.7	99.99	<0.002	0.009	10,440
0.1N	16	1.9	80.8	17.3	149.9	99.99	<0.001	0.003	10,320
0.25N	13	3.0	63.0	34.0	176.3	99.98	0.002	0.022	9,950

(1) 100-ml. samples of P-1 MgX-digest filtrate were boiled down until a boil-down precipitate was formed. Solutions were then adjusted to 56° Bé, and extracted by the usual procedure.

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are available for a definite explanation of the difference. However, one hypothesis, based on the assumption that salting strengths were comparable, is that the interfering components (such as sulfate and phosphate) are satisfactorily complexed in MgX; as opposed to pitchblende. This may be a reasonable assumption, since MgX was originally precipitated from solution.

Additional tests were run on mixtures of pitchblende and MgX solutions, boiled down together, and boiled down separately. When the two were boiled down together, no boil-down precipitate was formed. The samples were diluted to 56° Bé, and extracted, with the results shown in Table 38. The indications were that vanadium transfer was nearly constant whether the vanadium was partially tied up as a boil-down precipitate or not (i.e., it was independent of the quantity of vanadium in solution). The extraction of vanadium was slightly greater than in some of the other tests described, presumably due to the fact that the acidity of the feed was high. In these tests, the vanadium in the extract varied from 300-500 p.p.m. on a uranium basis.

The results of the above tests confirm earlier indications that the boil-down precipitate is not a problem, with respect to uranium loss to the precipitate. However, they indicate also that deliberate formation of a boil-down precipitate will be of slight assistance in reducing the carry-over of vanadium to the extract.

All of the preceding tests were made using MgX filtrate, so that boil-down and other precipitates could be followed visually. Several recent tests were performed to observe the behavior of slurries when

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TABLE 38. EFFECT OF ACIDITY AND BOIL-DOWN PRECIPITATE ON EXTRACTION OF VANADIUM FROM PROCESS SOLUTIONS

Ether Normality (HNO ₃)	<u>Vanadium Distribution</u>				<u>Uranium Distribution</u>			
	<u>Per Cent in</u>			Total mg. Found	<u>Per Cent in</u>			Total mg. Found
	Ether Ext.	<u>Raffinate</u>			Ether Ext.	<u>Raffinate</u>		
		Ppt.	Filt.			Ppt.	Filt.	
<u>(A) Solutions Boiled Down Separately⁽¹⁾</u>								
Neutral	5.4	59.1	35.5	84.6	96.3	0.1	3.6	15,050
0.1N	5.6	30.0	64.4	93.2	98.1	0.02	1.9	14,510
0.25N	5.4	30.9	63.7	100.4	98.3	0.02	1.7	15,360
<u>(B) Solutions Boiled Down Together⁽²⁾</u>								
Neutral	4.0	-	96.0	110.4	96.6	-	3.4	15,460
0.1N	7.6	-	92.4	79.0	98.1	-	1.9	14,990
0.25N	5.7	-	94.3	89.1	98.6	-	1.4	15,660

- (1) 50-ml. samples of P-1 MgX-digest filtrate were boiled down until a boil-down precipitate was formed. 59-ml. samples of A-1 pitchblende digest liquor were boiled to sp. gr. of 2.0; no precipitate formed. Solutions were mixed, adjusted to 56° Bé, and 30 g./l. excess acid, and extracted by usual procedure.
- (2) 50-ml. samples of P-1 MgX-digest filtrate were combined with 59-ml. samples of A-1 pitchblende digest filtrate (giving a uranium ratio of 1:2) and boiled to a sp. gr. of 2.0; no precipitate formed. Solutions were adjusted to 56° Bé, and 30 g./l. excess acid, and extracted by usual procedure.

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similarly treated. In these tests, P-1 MgX slurry was boiled down; during this operation, the slurry became almost a paste, and some boil-down precipitate appeared to have been formed. The slurry was diluted to approximately 56° Bé and extracted. A moderate to strong reddish color was observed in all extracts after the first or second. Although the vanadium metal balances were somewhat high, and the tests require confirmation, the indications were that vanadium transfer was high, in the neighborhood of 30-40 per cent. This was unexpected, since the conditions of acidity used were those which had, in previous tests, permitted only about 3 per cent of the vanadium to be extracted. One hypothesis suggests that the prolonged digestion in contact with a rather reactive form of silica (possessing a tremendous surface area) promoted the formation of an ether-soluble vanadate complex. Further tests are planned in which the effects of digestion of MgX slurries on vanadium transfer, and on the ease of removing vanadium from extracts by an aqueous scrub, will be studied. If the preliminary results are confirmed, boiling of MgX slurries would not appear to be advisable.

Recent results lead toward the conclusion that the species of the vanadium in feed solutions may have more effect on the transfer of vanadium than the actual concentration of vanadium in the solutions. It has not been possible to develop any relationship between vanadium solubility and vanadium transfer.

Column-Extraction Tests

In an attempt to obtain longer contact times between the organic and aqueous phases than were obtainable in the earlier studies reported

in BMI-247, several tests were run with the Scheibel column in which the aqueous phase was continuous. Results of these tests are compared with similar ones, in which the organic phase was continuous, in Table 39. Phase separation was observed at several sections of the column. This separation was probably a result of the viscous nature of the feed, which prevented the uniform flow of ether through the column. Under these conditions, it is doubtful if the laboratory column can be relied on to reach a state of equilibrium. Future tests will, therefore, be made with the organic phase continuous.

It was suggested that longer calming sections would improve the operation of the column. For two of the tests shown in Table 39, the column was modified so that the calming sections were increased about 50 per cent in length. This necessitated reducing the number of calming sections from 15 to 11, and the number of mixing sections from 14 to 10. Differences in results were negligible, and the column subsequently was changed back to the original design.

Calculations have indicated that for standard operating conditions⁽¹⁾, using the column with 14 mixing sections, the column efficiency was about 40 per cent. No figures are available for the column with the modified 11-mixing-section arrangement.

Tributyl Phosphate Extraction Studies

During a meeting at Mallinckrodt Chemical Works on October 2, 1950, it was decided that solvents other than ether for the extraction of

(1) 1000 ml. feed/hr.; ether:feed ratio of 1.6:1; ether phase continuous; 3000 r.p.m. stirrer speed; standard 56° Bé slurry.

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TABLE 39. CONTINUOUS-EXTRACTION TESTS ON PROCESS SLURRIES IN A SCHEIBEL ONE-INCH EXTRACTION COLUMN

Column Operating Conditions

Slurry acidity = 60 g. excess acid/liter.
 Ether acidity = 60 g. HNO₃/liter.
 Ether temperature = 0-10°C.
 Feed temperature = 20-25°C.
 Stirrer speed = 2500 r.p.m.

<u>Slurry Feed (1)</u>			Continuous Phase	Ratio, Ether: Feed	<u>Uranium</u>		Metal Balance, Per Cent
Slurry No.	U, g./l.	ml./hr.			In Raffinate, g./l.	Extracted, Per Cent (2)	
4(3)	262	433	Aqueous	1.8:1	3.3	98.7	124
6(3)	270	938	Ether	1.7:1	0.3	99.2	108
7(4)	234	1000	Ether	2.0:1	3.2	98.8	105
7(4)	234	900	Aqueous	2.1:1	<0.1	>99.9	101

(1) Slurry feeds prepared from pitchblende Blend A-1 and MgX Blend B-1 so that uranium ratio was 2:1. All slurries approximately 55-56° Bé.

(2) Based on raffinate analyses.

(3) Column contained 14 mixing and 15 calming sections.

(4) Column contained 10 mixing and 11 calming sections.

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uranium, particularly tributyl phosphate, warranted consideration. Earlier work at Battelle Memorial Institute on the extraction of thorium and uranium from monazite sand⁽¹⁾ and more recent work at other installations have shown that tributyl phosphate in the presence of a hydrocarbon diluent is an extremely effective solvent, and a preliminary investigation of its application to the problem of refining MgX and pitchblende was initiated. The scope of the investigation included only the application of tributyl phosphate to the contemplated direct slurry-extraction process at Plant 6; the application of tributyl phosphate to the so-called lyometallurgical extraction of uranium is being investigated elsewhere.

Batch-Extraction Tests

Several batch extractions, using equal volumes of solvent and aqueous phase, were conducted on typical samples of pitchblende-MgX process slurry. The solvent was a 50:50 volume mixture of tributyl phosphate and a refined kerosene (Gulfspray Naphtha). The results of these tests, shown in Table 40, indicated that three shakeouts effectively removed over 99 per cent of the uranium. The extraction of uranium appeared to be a function of both acid concentration and uranium concentration. This confirms data obtained during the monazite investigation⁽²⁾. The extremely high distribution coefficients obtained in the second shakeouts of Tests Nos. 1 and 2 are worthy of note.

(1) BMI-JDS-151, November 15, 1948.

(2) See Figures 6 and 7, BMI-JDS-225, February 15, 1950.

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TABLE 40. EXTRACTION OF URANIUM FROM PROCESS
SLURRY WITH TRIBUTYL PHOSPHATE

Tributyl phosphate - Gulfspray Naphtha⁽¹⁾ (50:50 by volume).
 Equal volumes of organic and aqueous.
 Agitation time = 1 minute.
 Phase separation time = 1-1/2 minutes (approximately).
 Temperature = 25 ±2°C.

Uranium in Feed, g./l.	Shakeout	Normality of HNO ₃		Uranium Distribution g./l.			Ext., Per Cent (2)	Metal Balance, Per Cent
		Org.	Aq.	Org.	Aq.	E _A		
<u>Test No. 1</u>								
276	1	0.7	0.7	172	71	2.4	99.8	86
	2	0.3	0.3	63	1.1	58		
	3	0.3	-	1.1	0.56	2.0		
<u>Test No. 2</u>								
213	1	0.6	2.7	152	46	3.3	99.7	98
	2	0.2	1.7	56	1.1	51		
	3	0.9	0.7	0.84	0.56	1.5		
<u>Test No. 3</u>								
208	1	0.7	3.7	156	56	2.8	99.8	93
	2	0.2	2.5	38	10.2(3)	3.7		
	3	1.2	1.3	0.32	0.48	0.7		

(1) Gulfspray Naphtha is a highly refined kerosene fraction obtained from the Gulf Oil Company.

(2) Based on raffinate and feed analyses.

(3) This value appears quite questionable.

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Spectrographic analyses of the extracts from the above tests showed the uranium to be of high purity, as illustrated by Table 41. The data indicate that maintaining total nitric acid concentration above 0.5N minimized the extraction of molybdenum.

Column-Extraction Tests

The promising results obtained on the preliminary batch extractions were confirmed by several continuous-extraction tests made with the Scheibel column. The results of these tests, tabulated in Table 42, showed that uranium concentrations in the raffinate had been reduced to 20-90 milligrams/liter.

In Test No. 1, tributyl phosphate, diluted with an equal volume of naphtha, was the solvent. In another run, under the same conditions, attempts were made to scrub the extract with 0.5N nitric acid. Due to the slight difference in density between the scrub and the organic extract, plus other effects apparently due to low flow rates, the scrub rose to the top of the column. In order to overcome this difficulty, the solvent composition for the remainder of the tests was changed to a 30:70 ratio of tributyl phosphate to naphtha. This lower density solvent mixture had good operating characteristics, and permitted the use of a scrub.

From the data in Table 42, it is apparent that control of total acidity and inclusion of a scrub of the extract are important factors in reducing the concentration of contaminants in the uranium extract. Additional study will be required to define attainable purity limits and to establish the optimum operating conditions, but the preliminary results are encouraging.

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TABLE 41. SPECTROGRAPHIC ANALYSES OF URANIUM FROM BATCH
EXTRACTIONS WITH TRIBUTYL PHOSPHATE

Test No.	Shakeout No.	Uranium Basis, p.p.m.							
		Mo	V	Pb	Cu	Ni	Fe	Si	Mn
1(1)	1	20-100	<20	<40	<10	<50	<20	8-40	<5
	2	>500	<20	<40	<10	<50	<20	8-40	<5
2	1	20-100	<20	<40	<10	<50	<20	40-200	<5
3	1	20-100	<20	<40	<10	<50	<20	8-40	<5

(1) See Table 40 for other data.

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TABLE 42. CONTINUOUS-EXTRACTION TESTS ON PROCESS SLURRIES WITH A TRIBUTYL PHOSPHATE SOLVENT

Operating Conditions

Feed = 200-250 grams of uranium/liter.

Solvent = 30 per cent tributyl phosphate - 70 per cent Gulfspray

Naphtha, except in Test No. 1.

Temperature = 25 $\pm$ 2°C.

Stirrer speed = 2500 r.p.m.

Point of entrance to column: scrub - top,
feed - 5 mechanical stages below top.

Organic phase continuous.

Test No.	Ratio, Feed:Scrub: Organic	Feed, ml./hr.	HNO ₃ Normality			Raff. (1)	Raffinate U Conc., g./l.	Uranium	
			Feed (1)	Organic	Scrub			Ext., % (2)	Meta Bal. %
1(3)	1.0:-:1.1	960	4.1	0.0	None	3.5	0.02	>99.9	93
2	1.0:0.25:2.3	757	4.3	0.0	0.5	3.4	0.09	99.9	92
3	1.0:0.26:2.4	625	1.3	0.0	0.0(4)	1.0	0.49	99.8	100
4	1.0:0.21:2.4	840	1.3	0.34	0.0(4)	1.4	4.9	97.9	84

Test No.	Spectrographic Analyses											
	Uranium Basis, p.p.m.											
	Ni	V	P	Cr	Co	Ni	Pb	B	Fe	Si	Cd	Cu
1	30(5)	90(5)	-	-	-	-	-	-	-	-	-	-
2	20-100	<20	N.D.	N.D.	N.D.	<50	<40	<1	<20	10-50	N.D.	<10
3	95	<20	<50	40	<20	<20	<10	<1	<20	25	<0.5	10
4	50	<20	<50	40	<20	<20	<10	<1	<20	25	<0.5	10

- (1) Excess HNO₃, expressed as normality.
 (2) Based on raffinate and feed analyses.
 (3) Solvent composed of tributyl phosphate and Gulfspray Naphtha in ratio of 50:50 by volume in Test No. 1.
 (4) Water scrub.
 (5) Chemical analysis.

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19Vanadium-Solubility Studies

The effects of varying acidity on the solubility of vanadium pentoxide, uranyl vanadate, and sodium uranyl vanadate in process solution have been studied, over the range from zero to 120 grams of excess acid per liter. As shown by Table 43, over this range, the solubility of vanadium pentoxide was very nearly constant, at about 1.6 grams of vanadium/liter. This was in marked contrast to its solubility in nitric acid (described in BMI-247). In the latter case, the solubility ranged from about 10 to 18 grams of vanadium/liter, over the same range of acidity. It may be that the presence in the feed solution of such cations as iron was the determining factor in reducing the solubility.

The two vanadium salts both exhibited slight increases in solubility with increasing acidity. Both showed higher solubilities than did the oxide, although neither exceeded 5 grams/liter.

Qualitative tests have indicated that sodium uranyl vanadate (artificial carnotite) breaks down when digested in concentrated nitric acid. One product of the degradation appears visually to be vanadium pentoxide. Although it appears to have no application to the problem of vanadium in current feeds, this approach may be of interest with respect to the handling of high-vanadium feeds.

X-Ray-Diffraction Studies

A series of samples of actual and synthetic MgX precipitates has been examined by means of X-ray diffraction, with the results shown in Table 44. The ignited samples of synthetic MgX had the same major phase,

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TABLE 43. EFFECT OF SOLUTION ACIDITY ON THE SOLUBILITY OF VANADIUM COMPOUNDS IN PROVESS SOLUTION(1)

Procedure: Excess solid phase was equilibrated with process solution by shaking mechanically for 140 hours at room temperature (20-30°C.).

Orig. XS Acid, g./l.	No Sulfate Removed				Pitchblende Sulfate Removed(2)			
	g./l.				g./l.			
	XS Acid	U	V	V, as % of U	XS Acid	U	V	V, as % of U
<u>Vanadium Pentoxide</u>								
0	2	214	1.45	0.68	3	215	1.54	0.72
30	34	214	1.49	0.70	28	226	1.59	0.70
60	50	207	1.64	0.79	62	236	1.61	0.68
120	121	206	1.60	0.78	118	226	1.69	0.74
<u>Sodium Uranyl Vanadate(3)</u>								
0	0	224	1.21	0.54	3	210	0.38	0.18
30	19	218	4.17	1.91	9	238	1.57	0.66
60	40	220	4.28	1.95	46	240	4.18	1.74
120	100	234	5.00	2.14	97	224	4.92	2.20
<u>Uranyl Vanadate(3)</u>								
0	5	204	1.26	0.62	1	222	1.28	0.58
30	22	208	1.67	0.80	24	218	1.73	0.79
60	51	208	2.39	1.15	58	221	2.44	1.10
120	103	206	4.18	2.03	102	236	4.46	1.89

- (1) Process solution prepared with 2/3 of U derived from pitchblende Blend A-1 and 1/3 derived from MgX Blend B-1.
- (2) Sulfate precipitated from pitchblende digest liquor before combining with MgX solution.
- (3) Preparation and analysis of these compounds described on pages 75-76 of BMI-246.

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TABLE 44. PHASE IDENTIFICATION OF MgX SAMPLES FROM
X-RAY-DIFFRACTION DATA

Sample	Description	Phases Present
EC-10-1	Synthetic MgX precipitate	Amorphous
EC-10-2	Ditto	Amorphous, trace $Mg(CH)_2$
EC-10-3	"	Amorphous, trace $Mg(CH)_2$
EC-10-1 ignited (1)	"	X(2), trace Fe_3O_4
EC-10-2 ditto	"	X(2), trace Fe_3O_4 , MgO
EC-10-3 "	"	X(2), trace fused $(Cu)_3(PO_4)_2$, MgO
P-88	MgX precipitate	Amorphous
P-92	Ditto	Amorphous
P-88 ignited	"	X(2)
P-92 ditto	"	X(2)
P-1 "	"	Crystalline(3)
5269-92A	Synthetic uranyl vanadate	Crystalline(3)
5269-92B	Ditto	Ditto
5597-2A (ignited)	Synthetic uranyl phosphate	"
5597-2B (ignited)	Ditto	"
4268-741	Fe-V boildown precipitate	"

(1) Ignited at 800°C.

(2) X phase is an unidentified crystalline phase.

(3) Phase is not identifiable by available diffraction data.

designated as "X", as did ignited samples of P-88 and P-92. The remaining samples were all crystalline, with unidentified diffraction patterns, none of which matched that of the unidentified "X" phase. The pattern of P-1 was unlike that of P-88 and P-92, although also unidentifiable by available diffraction data.

Notebook References

The original data for the work covered by this report are recorded in the following Laboratory Record Books:

<u>Notebook No.</u>	<u>Pages</u>
5268	80-89
5269	95-100
5531	11-26
5583	1-18

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