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THE PREPARATION OF HIGH-PURITY AMMONIUM METAVANADATE FROM IMPURE VANADIUM PENTOXIDE BY PRECIPITATION WITH AMMONIUM CHLORIDE

by

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

ABSTRACT

High-purity ammonium metavanadate can be obtained as a precipitate when the filtered solutions from the leaching of crude vanadium pentoxide are treated with ammonium chloride. The effect of using an excess of sodium carbonate, as a leaching agent, on the rate of leaching of vanadium pentoxide has been qualitatively investigated, and the effects of excess sodium carbonate, and of the concentration of vanadium pentoxide in the leach liquor, on the efficiency of precipitation with ammonium chloride have been quantitatively determined. Data are given showing the results obtained when both pure and crude vanadium pentoxides are treated in this way. Conditions are specified which result in the dissolution of 97% of the vanadium from crude vanadium pentoxide, and in the precipitation of 99% of the dissolved vanadium as ammonium metavanadate of high purity.

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LA PRÉPARATION DE MÉTAVANADATE D'AMMONIUM DE HAUTE PURETÉ PAR PRÉCIPITATION, AU CHLORURE D'AMMONIUM, À PARTIR D'ANHYDRIDE VANADIQUE IMPUR

par

J.A. Vézina* et W.A. Gow**

RÉSUMÉ

On peut obtenir du métavanadate d'ammonium très pur sous forme de précipité lorsque les solutions filtrées provenant du lessivage de l'anhydride vanadique brut sont traitées au chlorure d'ammonium. On a effectué des recherches qualitatives sur l'effet que peut avoir l'addition d'un excès de l'agent de lessivage. le carbonate de sodium, sur le pourcentage de l'anhydride vanadique dissous. On a déterminé quantitativement les effets que peuvent avoir l'excès de carbonate de sodium et la concentration de l'anhydride vanadique dans la liqueur de lessivage, sur l'efficacité de la précipitation au chlorure d'ammonium. Les données présentées montrent les résultats obtenus lorsque l'on traite de cette façon des anhydrides vanadiques soit purs, soit bruts. Les auteurs posent les conditions expérimentales qui permettent de dissoudre 97 p. 100 du vanadium contenu dans l'anhydride vanadique brut et de précipiter 99 p. 100 du vanadium dissous sous forme de métavanadate d'ammonium de haute pureté.

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INTRODUCTION

In a search for a method for purifying impure vanadium pentoxide, references were found indicating that sodium hexavanadate, when freshly precipitated, is readily dissolved by alkaline solutions from which it may be precipitated as ammonium metavanadate upon the addition of an excess of ammonium chloride, and that when this precipitate is washed, dried, and calcined, a vanadium pentoxide of very high purity results (1)(2) These references, however, did not provide quantitative data relevant to this method of purifying impure vanadium pentoxide by leaching in sodium carbonate solution. Our work was therefore conceived specifically, first, to determine economic conditions for the preparation of ammonium metavanadate with high yields, from solutions of pure vanadium pentoxide and sodium carbonate by precipitation with ammonium chloride; and secondly, to apply these conditions to the treatment of impure vanadium pentoxide

Theoretically, V_2O_5 and Na_2CO_3 react in aqueous solution in the proportion 1.72 g V_2O_5 /g Na_2CO_3 to produce $NaVO_3$. In turn, $NaVO_3$ and NH_4C1 react in the proportion 0.59 g NH_4C1 /g V_2O_5 to produce NH_4VO_3 , which is almost insoluble in an excess of NH_4C1 .

A series of tests, using reagent-grade chemicals, was conducted to examine the effects, on the precipitation efficiency, of varying the concentrations of the components of the solution. The following ranges were considered: V_2O_5 from 10 to 100 g/1, V_2O_5/Na_2CO_3 w/w from 0.2 to 1.5, and NH_4Cl/V_2O_5 w/w from 0.4 to 10. These tests made it possible to establish a set of conditions that were efficient and economical for producing ammonium metavanadate. A second series of tests was then carried out in which these conditions for the production of high-purity ammonium metavanadate were applied to three types of crude vanadium pentoxide.

PROCEDURE

The synthetic aqueous solutions of sodium vanadate and sodium carbonate were prepared by leaching vanadium pentoxide with an excess of sodium carbonate in glass beakers; a 100 ml volume of solution was used for each test. At concentrations up to 50 g V_{2O_5}/l , the dissolution of the vanadium took place readily at room temperature, but at a concentration of 100 g V_{2O_5}/l it was necessary to heat the solution to boiling temperature and agitate for about one hour. In all cases, the dissolution was complete and the solutions were clear and pale-yellow in colour.

In the second series of tests, three types of impure V_2O_5 were tested, two of them being market products designated as "commercial grade V_2O_5 ", and the third being a low-grade, off-specification V_2O_5 product prepared at the Mines Branch laboratories in the course of another study (3). The analyses of these products are reported in Table 1. None of these feed materials dissolved readily in the sodium carbonate solution, and it was necessary to agitate the slurries for up to 6 hours at boiling temperature. When leaching Type 2 (refer to Table 1), it was necessary to add a small quantity of sodium chlorate to obtain a clear filtrate pale-yellow in colour. In all cases, the dissolution was not complete and the slurries were filtered under vacuum. The leach efficiencies were calculated from the analyses of either the filtrates or the residues.

The precipitation of the vanadium was carried out by adding crystalline ammonium chloride to the stirred V_2O_5 -Na₂CO₃ solutions at room temperature. The slurries produced were stirred for 15 minutes and allowed to settle for 24 hours. The precipitates were vacuum filtered and the barren solutions were analysed for their vanadium content to determine the recoveries. The precipitates were washed with ethyl alcohol and analysed by chemical, spectrographic, and x-ray diffraction methods.

RESULTS

Precipitation of Ammonium Metavanadate from Pure Solutions

The results of the first series of tests, using reagent-grade chemicals, are reported in Figures 1, 2, 3, 4 and 5. Each figure presents a set of curves plotted from the results of experiments carried out at a single vanadium concentration. The five concentrations chosen for these experiments were, respectively; 10, 20, 30, 50 and 100 g V $_2O_5/1$.

For each of the three sets of tests reported in Figures 1, 2 and 3 and for which the vanadium concentrations were 10, 20 and 30 g $V_2O_5/1$ respectively, five levels of sodium carbonate additions were used for leaching the vanadium pentoxide. The sodium carbonate additions, expressed as a weight ratio of V_2O_5 to Na_2CO_3 , were 0.2, 0.33, 0.50, 0.66 and 1.0. The quantities of ammonium chloride used for precipitating the vanadium, expressed as a weight ratio of NH_4C1 to V_2O_5 , ranged from 0.5 to 5.0. The results from these three sets of tests (Figure 1, 2 and 3) showed clearly that increasing the V_2O_5/Na_2CO_3 weight ratio used in leaching from 0.20 to 1.0 brought about a corresponding increase in the yield of ammonium metavanadate for each of the various levels of ammonium chloride used. These results also showed that the amount of ammonium chloride required to produce a satisfactory yield of ammonium metavanadate decreased with an increase in the V_2O_5 concentration of the leach liquor.

TABLE 1

Analyses of the Low-Grade V₂O₅ Products Tested (Percent)

<u></u>	Commercial-Grade Off-Specification,							
	V 2		Low-Grade V2O5					
	<u>Type 1</u>	Type 2						
v ₂ o ₅	85.6	87.0	75.9					
Na	4.9	4.9	0.10					
Fe	0.02		4.64					
Ni	Tr		0.01					
Si	Tr?	0.03	0.02					
Al	0.01	0.09	0.17					
SO ₄			1.68					
NH3			3.06					
Mn	0.05	0.03						
Zr Mg	0.2	0.11 0.10						
Ca	0.3	0.31						
Ċu	Tr?	0.02						
Ti	Tr?							
Мо	0.09	0.11						
Р			<0.02					
L.O.I.(600°C)	1.2	1.0	16.5					

Note: The values reported under "commercial-grade V₂O₅" were obtained as follows: V₂O₅, Na by chemical methods, all the others from a general semi-quantitative spectrographic analysis. The values reported under "off-specification, low-grade V₂O₅" were obtained by chemical methods.

The results shown in Figures 1 to 3 suggested that to increase the yield of ammonium metavanadate and decrease the ammonium chloride consumption, concentrations of vanadium of over 30 g/l (the maximum used in these tests), at V₂O₅/Na₂CO₃ weight ratios of at least 0.5, should be considered for precipitation feed. Consequently, another series of tests was done using V_{2O_5}/Na_2CO_3 weight ratios of 0.5 and 1.0 with a common V_2O_5 concentration in the precipitation feed liquor of 50 g/1. The results of these tests are given in Figure 4 and, when considered along with the results shown in Figures 1 to 3, show clearly that the efficient precipitation of the vanadium, using an excess of ammonium chloride, was related to the concentrations of both the vanadium and sodium carbonate in the feed solution; and that the highest yield of ammonium metavanadate at the lowest V₂O₅/NH₄Cl weight ratio was obtained when the vanadium concentration was the highest (50 g $V_2O_5/1$) and the V_2O/Na_2CO_3 weight ratio was the highest (1.0). From the results of the tests reported in Figure 4, it is suggested that the optimum conditions for efficiency and economy of the precipitation of the vanadium were not reached, and that even higher vanadium concentrations and higher V2O5/Na2CO3 weight ratios would provide still greater precipitation efficiency.

This suggestion was confirmed by the results of a fifth set of tests, carried out using solutions containing 100 g $V_2O_5/1$ and a V_2O_5/Na_2CO_3 weight ratio of 1.5, a value representing 0.08 g $Na_2CO_3/g V_2O_5$ above the theoretical requirement for NaVO₃ production (Figure 5). To obtain these concentrations, it was necessary to boil the solution for one hour. A comparison of these results with those of the top curve of Figure 4 shows that the increases in the vanadium concentration from 50 to 100 g $V_2O_5/1$ and the V_2O_5/Na_2CO_3 ratio from 1.0 to 1.5 resulted in a further reduction in the NH_4Cl/V_2O_5 ratio required to effect the complete precipitation of the vanadium. Because of the observed difficulty in dissolving the V_2O_5 when making a solution of 100 g $V_2O_5/1$, higher concentrations of V_2O_5 in the precipitation feed solutions were not investigated.

Further reference to Figure 5, and to Figure 6 which shows an assembly of the best results from Figures 1 to 4, shows that the most economical reagent consumption was obtained when the leach solution (precipitation feed) contained 100 g V₂O₅/l and a weight ratio of V₂O₅/Na₂CO₃ of 1.5. With this solution, the weight ratio of NH₄Cl/V₂O₅ required for complete precipitation was 1.0, as against the theoretically required NH₄Cl/V₂O₅ ratio of 0.59.

Leaching of Crude V_2O_5

The results of the second series of tests, carried out with impure vanadium pentoxides containing from 75.9 to 87% V₂O₅, are reported in Table 2. Low-grade vanadium pentoxide prepared at the Mines Branch laboratories was used in Tests 1 and 2, and market types of commercialgrade V₂O₅ were used for Tests 3 and 4. The analyses of these products are reported in Table 1. The reason for testing two types of commercialgrade V_2O_5 was that, under the same conditions, their leaching characteristics were quite different. The dissolution of the low-grade vanadium pentoxide and commercial-grade V₂O₅, Type 1 in sodium carbonate solution at boiling temperature produced filtrates that were clear and pale-yellow in colour. whereas, under the same conditions, commercial-grade V₂O₅, Type 2 produced an opaque, dark-blue filtrate that did not clarify even with a filter aid. However, when sodium chlorate was added during the leaching, the filtrate was clear and pale-yellow as with the others. In all cases, the dissolution of the vanadium was incomplete and was not improved by adding sodium chlorate during the leaching. The V₂O₅ extraction in leaching these low-grade products ranged from 90.2 to 98%.

Test 1 was carried out with quantities of low-grade vanadium pentoxide and sodium carbonate to produce a solution containing 50 g/1 each of V_2O_5 and Na₂CO₃. The excess sodium carbonate used was 0.42 g Na₂CO₃/g V₂O₅ above that required theoretically to combine the vanadium as NaVO₃. This resulted in the dissolution of 97.4% of the vanadium. In Test 2, amounts to produce a solution containing 100 g V₂O₅/1 and 66 g Na₂CO₃/1 were used, the excess Na₂CO₃ being 0.08 g Na₂CO₃/g V₂O₅. The vanadium extraction was 90.2%, that is, considerably lower than that obtained in Test 1. This would suggest that with impure V₂O₅ products, an excess of Na₂CO₃ is required in the leaching step to produce efficient leaching of the V₂O₅.

In Tests 3 and 4, with commercial-grade V_2O_5 , the leaching conditions were those used for Test 2, except that the leaching time was decreased to 2 hours, and 0.015 g NaClO₃/g V_2O_5 was added in Test 4. The vanadium extraction was satisfactory in both tests, although it was somewhat higher with commercial-grade Type 1; the reason for this difference is not known. It may be pointed out that the extractions in Tests 3 and 4 were higher than that obtained in Test 2, probably because of the relatively larger sodium content of the commercial-grade V_2O_5 used in Tests 3 and 4.

Precipitation of Ammonium Metavanadate from Impure Solutions

The precipitation of the vanadium was 99.2% in Test 1, Table 2, when $1.44 \text{ g NH}_4\text{Cl/g V}_2\text{O}_5$ was used; this value compares favourably with the results, shown in Figure 4, previously obtained with reagent-grade V₂O₅. The yields of ammonium metavanadate for Test 2, 3 and 4 are shown in Table 2 and have been reproduced in plot form in Figure 7. As a means of comparing these results with those obtained previously with reagent-grade V₂O₅, the curve shown in Figure 5 has been transposed in Figure 7. The effect of the vanadium concentration on the yield of ammonium metavanadate is clearly shown in Figure 7 and follows a pattern that was determined previously with reagent-grade V₂O₅ in Figure 6; that is, the best yields were produced with the minimum ammonium chloride when the concentration of V_2O_5 was the highest. It is indicated in Figure 7 that greater than 99% of the vanadium was precipitated from solutions of impure V2O5 with an ammonium chloride consumption of about 1.2 g NH4Cl/g V2O5, as compared with 1.0g NH4Cl/g V_2O_5 when reagent-grade V_2O_5 was used to prepare the precipitation feed solution.

The precipitates of vanadium produced in Tests 1, 2, 3 and 4, Table 2, were analysed by spectrographic, chemical and x-ray diffraction methods; the results of these analyses are reported in Table 3. They show that the precipitates were of high purity. For comparison purposes, ammonium metavanadate theoretically contains 77.78% V_2O_5 , 14.53% NH₃, and 7.69% H₂O (crystallization). The precipitates from Tests 1 and 3 were identified by x-ray diffraction as ammonium metavanadate.

DISCUSSION OF RESULTS

The series of tests with reagent grade-chemicals established the fact that to achieve high yields of ammonium metavanadate from solutions of vanadium in sodium carbonate with the least consumption of ammonium chloride, it was necessary to prepare solutions containing high concentrations of vanadium with only a slight excess of sodium carbonate.

For practical purposes, an excess of sodium carbonate above that theoretically required must be used. In fact, to produce a solution containing 100 g V₂O₅/1 from vanadium pentoxide with the equivalent of 66 g Na₂CO₃/1, i.e., an amount slightly in excess of the theoretical requirement, required agitation at boiling temperature for two hours. The excess NaCO₃ in this case was 13%. It is reasonable to assume that solutions containing vanadium in concentration larger than 100 g V₂O₅/1 may be prepared from vanadium pentoxide using an excess of sodium carbonate less than 13%, but it is foreseen that this would probably necessitate more persuasion than a one-hour leach at boiling temperature. TABLE 2

Results of Tests for Determining the Efficiency of Precipitation of Ammonium Metavanadate from Solutions of Impure Vanadium Pentoxide with Ammonium Chloride

	Leaching Conditions												Ammoniu						
Test No.	1 I		est Source of V2O5 V2O5 Temp. Time Vanadium Percent Vanadium Precipita																
		(0/ /	(wt ratio)			(percent)	0.67	0.77	0.87	0.94	1.02	1.05	1.11	1.22	1.33	1.44	1.53	1,59	1.66
1	Off-Specification, Low-Grade V ₂ O ₅	48.7	1	100	6	97.4										99.2			
2	Off-Specification, Low-Grade V ₂ O ₅	90.2	1.4	100	6	90.2	60.3		78.5	86.7			96.0	99.9	99.9	99.9	99.9		99.9
3	Commercial-Grade V ₂ O ₅ , Type 1	98.0	1.5	100	2	98.0		87.3	94.2		98.1						99.9		
4*	Commercial-Grade V ₂ O ₅ , Type 2	94.4	1.4	100	2	94.4	64.4	. 82.3		90.3		97.2						99.6	

* 0.015 g NaClO₃/g V₂O₅ added.

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TABLE 3

Results of the Analyses Done on the Precipitates Prepared in Tests 1, 2, 3 and 4 Reported in Table 2

(Percent)

	By Chemi	ical Method	з,	By General Semi-Quantitative Spectrographic Method								
v ₂ 05	NH3	50 ₄	C1.	Si	· A1	Mg	Fe	Cu	Na.	Mo		
76.6	14.6	<0.003	0.25	0.02	0.05	0.002	0.05	0.002	ND	ND		
				0.02	0.07	0.005	0.02	0.016	ND	ND		
76.5	1,4.2	<0.003	0.60	0.06	ND	0.02	ND	0.003	ND	ND		
				0.04	ND	0.002	ND	0,004	0.07	0.006		
	V ₂ O ₅ 76.6	V ₂ O ₅ NH ₃ 76.6 14.6	$\begin{array}{c c c c c c c c c c c c c c c c c c c $	76.6 14.6 <0.003 0.25	V_2O_5 NH ₃ SO_4 C1. Si 76.6 14.6 <0.003	V_2O_5 NH ₃ SO_4 Cl. Si Al 76.6 14.6 <0.003	V_2O_5 NH_3 SO_4 $C1$ Si $A1$ Mg 76.6 14.6 <0.003	V2O5 NH3 SO4 C1 Si A1 Mg Fe 76.6 14.6 <0.003	V_2O_5 NH ₃ SO ₄ C1. Si A1 Mg Fe Cu 76.6 14.6 <0.003	V2O5 NH3 SO4 C1 Si A1 Mg Fe Cu Na 76.6 14.6 <0.003		

It was known that an excess of ammonium chloride is necessary to produce high yields of ammonium metavanadate from solutions of sodium vanadate (NaVO₃) (1) (2). The results of the first series of tests, reported in Figures 1 to 5, showed that an excess is indeed required and that it is quantitatively related to the concentrations of the vanadium and sodium carbonate in the solution. Under conditions favouring the economical use of ammonium chloride--that is, with solutions containing a large V_2O_5 concentration (100 g/1) and a small excess of sodium carbonate above that theoretically required to dissolve the vanadium--the theoretical quantity of NH₄Cl necessary to combine with all vanadium (0.59 g NH₄Cl/g V_2O_5) precipitated only 81% of the vanadium, but when an excess of 70% NH₄Cl was used the yield was increased to 99.3%.

Solutions containing vanadium concentrations larger than 100 g $V_{2O_5}/1$ were not investigated, but from the results shown in Figure 5 it may be assumed that they would improve the yield of ammonium metavanadate and the economy of use of ammonium chloride.

The leaching of the low grades of vanadium pentoxide was not as efficient as that observed with reagent-grade V_2O_5 . It would appear, from the results obtained with the three types of impure V_2O_5 considered, that an excess of sodium carbonate is required for the satisfactory leaching of the vanadium and that this excess is dependent on some characteristics of the products which were not determined. It was shown, however, that extractions greater than 90% were obtained with an excess of 0.08 g Na₂CO₃/g V_2O_5 . In one instance, it was necessary to add a small quantity (0.015 g/g V₂O₅) of sodium chlorate to obtain a solution suitable for the precipitation of the vanadium.

The precipitation of the vanadium from solutions prepared with the lowgrade V₂O₅ products was efficient and economical under the conditions determined previously with reagent-grade V₂O₅. It has been shown, in Figure 7, that with solutions of impure V₂O₅ containing 94 and 98 g V₂O₅/1, the precipitation of the vanadium had been 99% and more when about 1.1 g NH₄Cl/g V₂O₅ was used, but that about 1.2 g NH₄Cl/g V₂O₅ was required to precipitate the vanadium to the same extent with solutions containing only 90.2 g V₂O₅/1. These values compare reasonably well with a consumption of 1.0 g NH₄Cl/g V₂O₅ observed previously with reagent-grade V₂O₅.

The ammonium metavanadate produced from impure V_2O_5 was of high purity, as evidenced by the analyses reported in Table 3, and its composition was near that of pure ammonium metavanadate.

CONCLUSIONS

Ammonium metavanadate can be precipitated from pure solutions of vanadium pentoxide and sodium carbonate containing 100 g $V_2O_5/1$ and 66 g $Na_2CO_3/1$ by adding 1 g $NH_4Cl/g V_2O_5$ in crystalline form. The yield under these conditions is greater than 99%, and the ammonium chloride consumption is in excess of that theoretically required by 0.40 g $NH_4Cl/g V_2O_5$. Decreases in the vanadium concentration and in the $V_2O_5/NaCO_3$ ratio of the leach solutions result in lower yields of ammonium metavanadate and increased consumption of ammonium chloride.

The conditions determined with pure solutions apply reasonably well for the treatment of solutions obtained from the leaching of crude vanadium pentoxide products. Solutions produced from impure V_2O_5 and containing 94 g $V_2O_5/1$ and 66 g Na₂CO₃/1, and to which 1.1 g NH₄Cl/g V_2O_5 was added, produced a precipitation yield greater than 99%.

The precipitates from the solutions of impure V_2O_5 were identified as ammonium metavanadate and, except for a small residual ammonium chloride, were of high purity.

ACKNOWLEDGEMENTS

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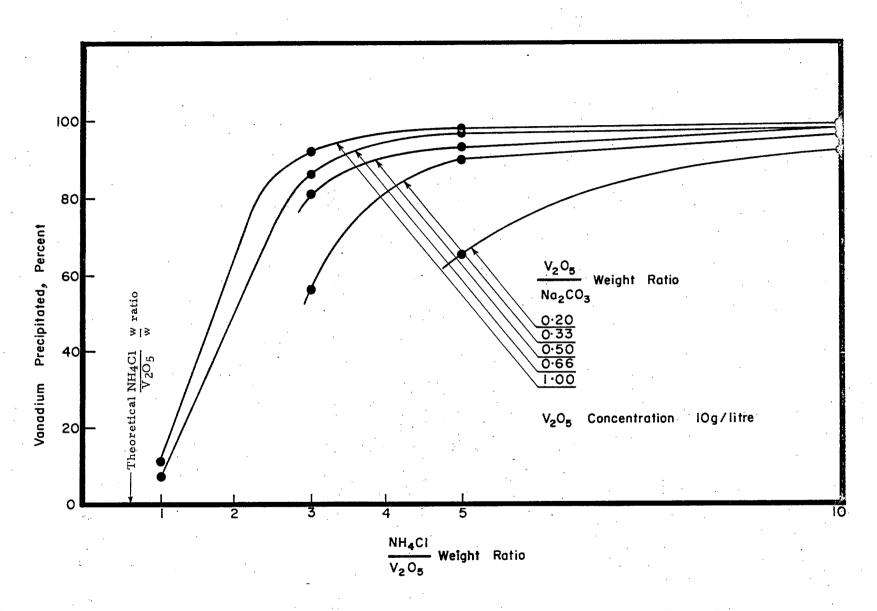
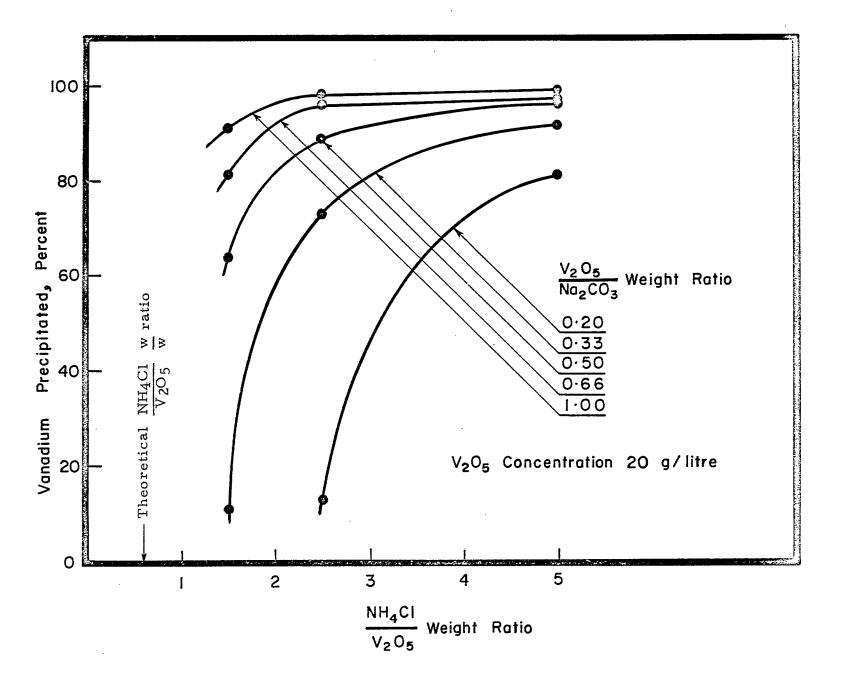
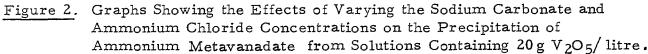


Figure 1. Graphs Showing the Effects of Varying the Sodium Carbonate and Ammonium Chloride Concentrations on the Precipitation of Ammonium Metavanadate from Solutions Containing 10g V₂O₅/litre





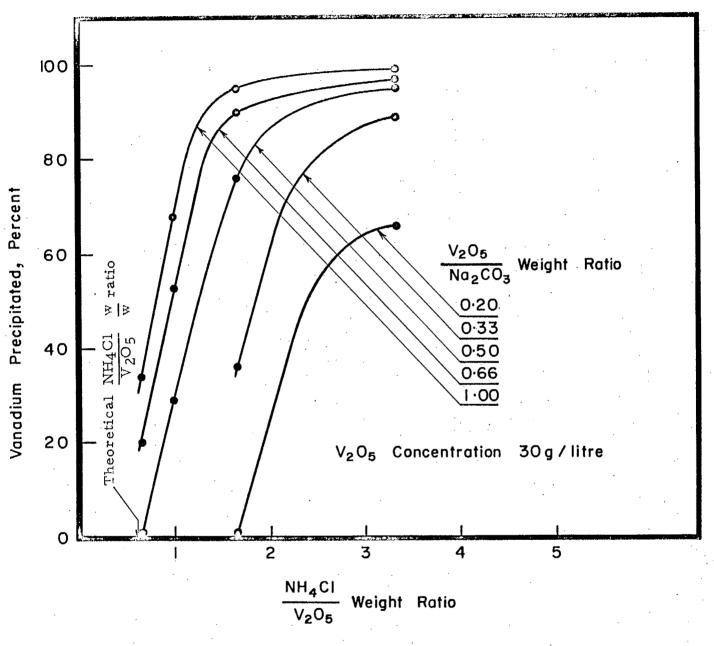
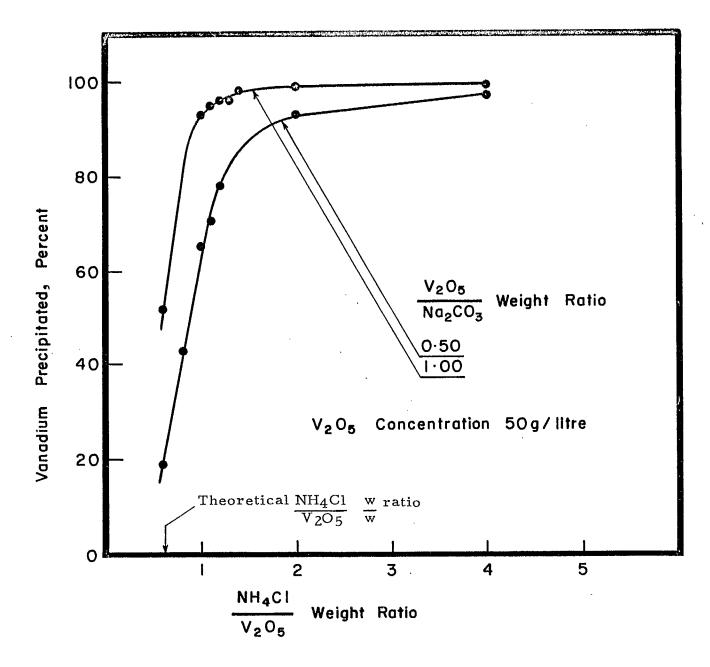
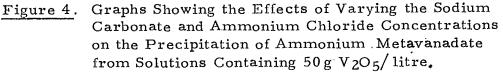
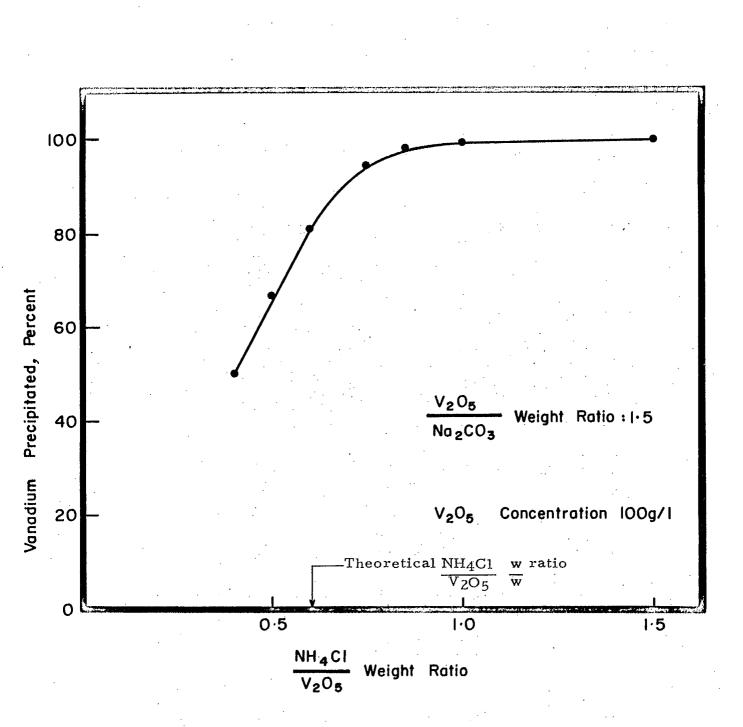
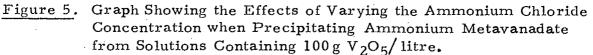


Figure 3. Graphs Showing the Effects of Varying the Sodium Carbonate and Ammonium Chloride Concentrations on the Precipitation of Ammonium Metavanadate from Solutions Containing $30 \text{ g V}_2O_5/$ litre.









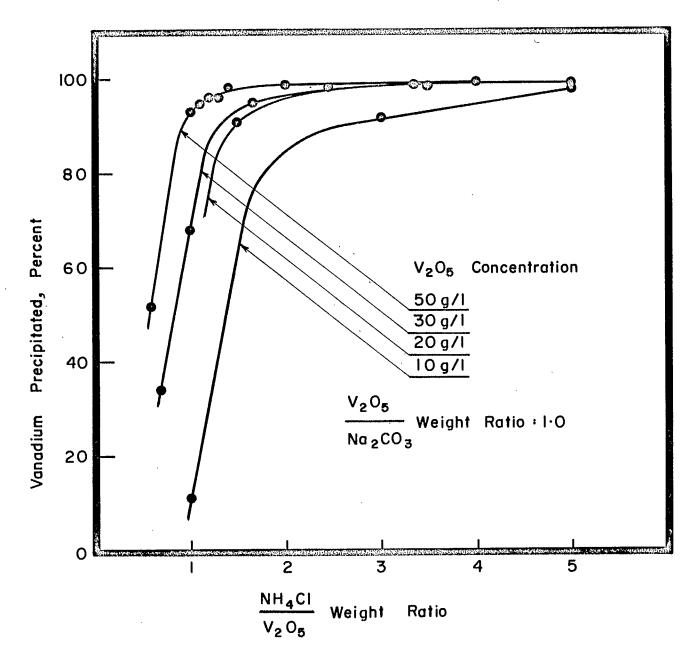


Figure 6. Graphs Showing the Effects of Varying the Vanadium and Ammonium Chloride Concentrations on the Precipitation of Ammonium Metavanadate from Solutions Containing a Ratio V_2O_5/Na_2CO_3 of 1.

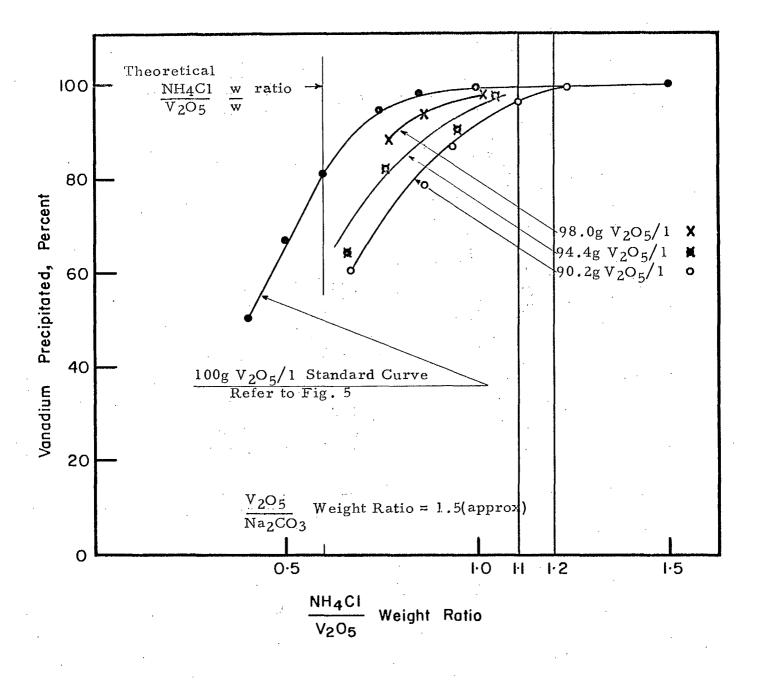


Figure 7. Graphs Showing the Effects of Varying the Ammonium Chloride and the Vanadium Concentrations when Precipitating Ammonium Metavanadate from Solutions of Impure V₂O₅.

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