RMO 2619

THE LEAD VANADATE PRECIPITATION
METHOD FOR THE RECOVERY OF VANADIUM FROM
CARBONATE LEACH SOLUTIONS

ARTHUR D. LITTLE, INC.

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RMO 2619
Metallurgy - Raw Materials

Topical Report

THE LEAD VANADATE PRECIPITATION METHOD

FOR THE RECOVERY OF VANADIUM

FROM CARBONATE LEACH SOLUTIONS

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I. ABSTRACT

A process for the removal (and recovery) of vanadium from carbonate leach solution has been developed. PbSO_4 is used to precipitate vanadium and is regenerated by treating the lead vanadate precipitate with H_2SO_4 . Vanadium concentration can be reduced to any desired level, or removed completely, with essentially no carbonate or uranium losses. H_2SO_4 is the only reagent consumed making the process superior to FeSO_4 precipitation of vanadium on an economic as well as efficiency basis. The removal of vanadium from carbonate leach solutions improves the efficiency and economics of subsequent uranium recovery process. An economic evaluation compares the lead vanadate-caustic process with other possible processes for recovery of uranium and vanadium from carbonate leach solutions.

II. INTRODUCTION

In our work on uranium recovery from carbonate solutions it soon became evident that virtually every process considered was more efficient and economic in the absence of vanadium. For example, in any reduction process, if both vanadium and uranium are present, the vanadium is reduced before the uranium. In typical carbonate leach solutions, which contain considerably more vanadium than uranium on a mole basis, this necessitates the use of far more reducing agent than would be required for uranium reduction alone. As another illustration, diuranate precipitation is less effective in the presence of vanadium, apparently due to the existence of a weak, soluble, uranyl vanadate complex. For this reason it is advantageous to have an economic method of selectively removing vanadium from the leach solution prior to treatment for uranium recovery.

A very successful method was found in the course of investigating uranium recovery processes. It consists of the addition of lead sulfate to the leach solution, resulting in the formation of a precipitate of the more insoluble lead vanadate. This precipitate is then separated from the leach solution and treated with sulfuric acid, resulting in the formation of insoluble lead sulfate which is recycled, and an unstable solution of vanadium which precipitates V_2O_5 on heating.

III. SUMMARY

Quantitative removal of vanadium from carbonate leach solutions can be achieved by the addition of 3.33 PbSO_4 per V_2O_5 . If complete vanadium removal is not required, a lower ratio will suffice approaching a minimum value of 2.0 PbSO_4 per V_2O_5 precipitated when the concentration of vanadium remaining in solution is sufficiently high and the pH sufficiently low.

Sulfate is given up to the solution in this process but both calculation and experiment show that sulfate concentration can never build up to a point where it might interfere with the conversion of PbSO_4 to lead vanadate.

The lead vanadate precipitate ($3.33 \text{PbO} \cdot \text{V}_2\text{O}_5 - 2.0 \text{PbO} \cdot \text{V}_2\text{O}_5$) formed can be treated with H_2SO_4 to regenerate PbSO_4 and form an unstable vanadium solution. Optimum conditions for this step appear to be 1 H_2SO_4 per PbO plus 1 H_2SO_4 per V_2O_5 . The PbSO_4 is recycled and the unstable vanadium solution heated to 90°C to precipitate V_2O_5 . After V_2O_5 precipitation, the acid leach can be treated with sufficient concentrated H_2SO_4 to replace the acid consumed by PbO , and then recycled. It is seen that the only reagent consumption involved in this process is $3.33 - 2.0 \text{H}_2\text{SO}_4$ per V_2O_5 recovered.

Uranium losses are negligible. Only about .2% of the uranium was precipitated with over 95% of the vanadium from a carbonate leach solution containing 3.5 g/l U_3O_8 and 4.5 g/l V_2O_5 . Even what uranium is precipitated concentrates in the H_2SO_4 leach and can be recovered when desired.

Direct comparison in this process with the ferrous vanadate process proposed by Battelle Memorial Institute ^{1/} for the alkaline leach pilot plant at Grand Junction shows the lead vanadate process to be far superior from the standpoint of efficiency, and somewhat superior from the standpoint of cost. Calculations give cost estimates of \$4.24 PTS for the BMI ferrous vanadate-caustic process and \$3.97 PTS for a comparable lead vanadate-caustic process (based on maximum H_2SO_4 consumption, i.e. - 3.33 H_2SO_4 per V_2O_5). The cost advantage becomes much more pronounced as the amount of

^{1/} Numbers refer to reference in Appendix page 42.

vanadium removed per cycle is increased.

In comparison with other processes for the recovery of uranium and vanadium from carbonate leach solutions the lead vanadate-caustic procedure shows additional advantages in its simplicity of operation and low capital investment requirements.

IV. EXPERIMENTAL DETAILS

Synthetic solutions were prepared from "Baker Analyzed" reagent grade $\text{Na}_2\text{CO}_3 \cdot \text{H}_2\text{O}$, NaHCO_3 , $\text{Pb}(\text{NO}_3)_2$ and $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, DuPont C.P. reagent H_2SO_4 , V_2O_5 prepared by thermal decomposition under a stream of oxygen or Fisher Scientific Co. purified NH_4VO_3 , and distilled water. Actual carbonate leach solutions used were obtained from Monticello mill of the U. S. AEC.

Most experiments were done in test tubes with agitation provided by motor driven stirrers. Precipitates were usually separated from supernatant liquor by centrifuging, decanting the liquor and replacing it with distilled water, mixing the precipitate and water thoroughly, and again centrifuging and decanting. Since precipitate volumes were generally about 10% of total volume, this washing procedure resulted in about 1% of the original liquor being retained with the precipitate. The error introduced by this is negligible. In those cases in which the precipitate was analyzed for minor constituents - i.e. CO_2 or uranium - the precipitate was filtered and washed more thoroughly. Unless otherwise stated, lead was added to the vanadium solution as lead nitrate solution rather than lead sulfate solid. This was simply a matter of convenience and had no bearing on the results, as shown by quite comparable results obtained when lead sulfate was used.

V. DISCUSSION OF TEST WORK

A. General: The possibility of using lead to remove vanadium selectively from carbonate leach solutions was indicated in the Textbook of Inorganic Chemistry edited by Friend 2: "A general method for the separation of V from As, Mo, P, Cr, U, W, and Si consists in precipitating these metals as their respective lead salts and digesting the precipitate with potassium carbonate, whereupon all the lead salts are decomposed with the exception of lead vanadate".

Various cations, including lead, were tried as precipitants for vanadium in $\text{CO}_3^{=}\text{-HCO}_3^{-}$ solution (See Table I) and lead (+2) definitely appeared the most promising on the basis of cost and efficiency. However, it was readily apparent that even lead would have to be recycled to keep costs within reasonable limits. Ideally, this might be done by selectively dissolving out the lead, which is then recycled in solution, and leaving a solid V_2O_5 product behind. This was tried with acetic acid, acetic acid-acetate, and nitric acid with negative results.

Another possibility is to dissolve the lead vanadate completely in nitric acid and then separate the lead and vanadium by ion exchange or solvent extraction. This could quite possibly be developed into a workable process, but it has not been investigated here due to the efficiency and simplicity of the alternative process investigated.

The process developed is based on the third alternative which is to leach out the vanadium as a soluble species leaving the lead as a solid. This can be done using any strong acid that forms an insoluble lead salt (e.g. HCl , or H_2SO_4) since vanadate is converted over to a soluble positive charged vanadium species in acid. It might also be done by treating the precipitate with a salt solution the anion of which forms a compound with lead even more insoluble than lead vanadate, thereby leaching vanadate as such. This was tried with various salts but only Na_2S proved effective (Table II). It is, of course, obvious that the PbS formed would have to be converted to another salt before the lead would be available for

vanadium precipitation from carbonate leach solutions.

Various insoluble lead salts were tried and it was found that several would precipitate vanadium from $\text{CO}_3^{=}\text{-HCO}_3^-$ solution (Table III). The use of any of the carbonate or hydroxide salts of lead would avoid the addition of a foreign ion to the carbonate leach solution but they have the disadvantage that they cannot be formed from lead vanadate in a single step. PbSO_4 can be formed in a single step from lead vanadate by treatment with H_2SO_4 . While treatment with PbSO_4 does add a foreign ion to the carbonate leach solution it is found by both experiment (Table II - attempted leach of lead vanadate with Na_2SO_4) and calculation (solubility product $\frac{3}{\text{PbCO}_3 = 1.5 \times 10^{-13}}$, $\text{PbSO}_4 = 1.8 \times 10^{-8}$ - but $\text{CO}_3^{=}$ does not interfere with lead vanadate precipitation (Table IV)) that sulfate concentration can never build up to the point where it would interfere with vanadate precipitation.

Everything considered, it appeared that treating the lead vanadate with sulfuric acid to leach out vanadium and recycling the lead as lead sulfate had the most promise, and this process will be considered in detail.

B. Precipitation of lead vanadate: It was found that the amount of vanadium precipitated for a given amount of lead decreased with increasing pH (Table V), and increased with an increase in the ratio of initial vanadium to initial lead concentration (or, probably more accurately, with an increase in final vanadium concentration) (Table VI). Carbonate does not appear to interfere. Analyses of supernatant liquid for CO_2 were erratic but supported the view that little if any carbonate was precipitated. Analysis of the solid precipitate also supports this view, CO_2 present appearing to be due to Na_2CO_3 and Na HCO_3 not thoroughly washed out (Table VII). In all experiments the mole ratio of vanadium to lead approached a minimum value of .60 and a maximum value 1.0. Complete (>99%) precipitation of vanadium in the $\text{CO}_3^{=}\text{-HCO}_3^-$ pH range requires 3.33 moles of PbSO_4 per mole of V_2O_5 - i.e. the ratio of vanadium to lead in the precipitate is .60. However, over much of this pH range, a ratio in the precipitate of 1.0 can be approached by use of a countercurrent flow method - i.e. contacting the PbSO_4 with successive batches

of carbonate leach solution, resulting in the final solid being in equilibrium with the initial concentration of vanadium, and contacting the carbonate leach with successive batches of PbSO_4 , resulting in complete vanadium removal from the final solution. How close to 1.0/^{the}ratio of vanadium to lead in the precipitate comes is determined by the vanadium concentration and pH of the carbonate leach solution feed.

These facts can be explained, at least qualitatively, by assumption of the equilibrium $\text{V}_2\text{O}_7^{-4} + 2\text{OH}^- = 2\text{VO}_4^{-3} + \text{H}_2\text{O}$

$$\frac{(\text{VO}_4^{-3})^2}{(\text{V}_2\text{O}_7^{-4})(\text{OH}^-)^2} = K$$

and the precipitating species

1) $\text{Pb}_2\text{V}_2\text{O}_7(\text{H}_2\text{O})_x$, (i.e. $2\text{PbO} \cdot \text{V}_2\text{O}_5$); and 2) $\text{Pb}_{10}(\text{VO}_4)_6\text{O}(\text{H}_2\text{O})_x$, (i.e. $3.33\text{PbO} \cdot \text{V}_2\text{O}_5$)

It is apparent that high pH and low vanadium concentration shift the assumed equilibrium to the right and favor the second precipitating species. Some support for this picture can be found in the document ACCO 49 4/. $\text{V}_2\text{O}_7^{-4}$ and VO_4^{-3} are stated to be in equilibrium in the range pH 10.6 to 12 (pg. 9). This range is included in the $\text{CO}_3^{--} = \text{HCO}_3^-$ pH range in which our work was done. There is also a compound, $3.33\text{SrO} \cdot \text{V}_2\text{O}_5$, reported (pg. 18) which is analogous to our assumed compound $3.33\text{PbO} \cdot \text{V}_2\text{O}_5$. However the reported (pg. 18) precipitation of $3\text{PbO} \cdot \text{V}_2\text{O}_5$ at pH 4.2-4.6 and $2\text{PbO} \cdot \text{V}_2\text{O}_5$ at pH 1.8-2.4 does not agree with our data, which indicate these are formed at much higher pH values (the existence of $3\text{PbO} \cdot \text{V}_2\text{O}_5$ is neither confirmed nor denied by our data).

The existence of the mineral vanadinite, $\text{Pb}_{10}(\text{VO}_4)_6\text{Cl}_2$ 5/, is further support for existence of the analogous $\text{Pb}_{10}(\text{VO}_4)_6(\text{OH})_2$ - i.e. $3.33\text{PbO} \cdot \text{V}_2\text{O}_5 \cdot .33\text{H}_2\text{O}$.

It was found that 10 minutes or even less is sufficient for complete conversion of freshly prepared lead sulfate to lead vanadate (Table VIII).

C. Regeneration of PbSO_4 and separation of V_2O_5 The experiments were based on treatment of $3.33\text{PbO} \cdot \text{V}_2\text{O}_5$ prepared by adding the calculated amount

of $\text{Pb}(\text{NO}_3)_2$ or PbSO_4 to the carbonate leach solution. This precipitate was chosen since it is the least favorable that would occur, from a reagent consumption standpoint, and also is the species that would result from complete vanadium precipitation in a single stage contacting process. Conclusions drawn from these experiments apply equally to $2.0 \text{ PbO} \cdot \text{V}_2\text{O}_5$ except that correspondingly less acid per vanadium recovered would be required.

It was found that complete dissolution of the vanadium required 1 H_2SO_4 per PbO and between 2 and 3 H_2SO_4 per V_2O_5 - i.e. 5.33 - 6.33 H_2SO_4 per $3.33 \text{ PbO} \cdot \text{V}_2\text{O}_5$ (Table IX). The same acid equivalent requirement was found with HNO_3 or HCl . Using smaller quantities of acid resulted in unstable solutions which precipitated V_2O_5 with time and, more efficiently, heat (Tables IX, X, XI).

The most efficient process consists of leaching with 4.33 H_2SO_4 per $3.33 \text{ PbO} \cdot \text{V}_2\text{O}_5$ (Table IX) for 5 minutes (Table XII). Acid concentration used is not critical below 0.5M, but efficiency falls off above that concentration (Table XIII). The vast majority of the unstable vanadium will precipitate out in 5 minutes at 100°C but some additional precipitate may form on an hour's heating (Table XIV). That the acid leach solutions should be unstable is shown by consideration of the equation given by Latimer ^{6/}.

$$\text{Thus } \log K = \frac{2500}{-1364} = -1.84 \text{ and } \frac{(\text{V}(\text{OH})_4^+)^2}{(\text{H}^+)^2} = 10^{-1.84}$$

or $\text{H}^+ = 10^{.92} \times \text{V}(\text{OH})_4^+ = 8.3 \text{ V}(\text{OH})_4^+$. Since these calculations indicate that approximately ten times as much hydrogen ion as vanadium ion is required for a stable solution, even those solutions prepared with 2H^+ per Pb and 3H^+ per V should have been acid deficient at the concentrations used. (They did not precipitate V_2O_5 though even on several days standing and they were not heated.) The final vanadium concentrations in these heated solutions agree quite well (Table X) with those calculated using the equilibrium expression and constant derived in the preceding paragraph. (It is, of course, true that the + 1 vanadium species could be VO_2^+ , or even any singly charged polymeric vanadium

species, rather than $V(OH)_4^+$ but the species is definitely singly charged since the equilibrium expression $\frac{VpOq^{+y}}{(H^+)^y} \rightleftharpoons K$ will fit our data only when $y = 1$, or a fractional value close to it.)

The average charge per vanadium atom, not necessarily species, in the unstable acid leach solution was determined by measuring the change in pH during precipitation, thereby permitting calculation of the hydrogen ion required to replace a known amount of vanadium in solution (Table XI). This calculation, as well as calculation based on the pH, SO_4^{2-} and V concentrations in the unstable acid leach prior to precipitation, gives a value of + .4 for the average charge per vanadium atom. This indicates that the vanadium is polymerized in these unstable solutions, although it could be that there is present a mixture of monomeric singly charged vanadium species and colloidal V_2O_5 . The weight of precipitate obtained, compared with the weight expected on the basis of V_2O_5 , strongly indicates that the precipitate is indeed V_2O_5 (Table X).

D. Demonstration of process on leach solutions: The entire process was tested on an actual carbonate leach solution from the Monticello Mill of the U. S. AEC. Results (Table XV) were slightly less favorable than anticipated from synthetic solutions but still quite satisfactory. Overall vanadium recovery was 71% and uranium losses were .2%.

However the process is actually even better than this due to the possibility of recycling various reagents. The lead sulfate is, of course, **recycled** and with it any vanadium not removed in the acid leach. This vanadium (amounting to 13.5% of heads in this experiment) is therefore not lost but simply requires a somewhat higher lead inventory.

In addition, the acid leach solution can be recycled after the addition of concentrated H_2SO_4 to replace that used to form $PbSO_4$. The vanadium remaining in this acid leach (13.4% of heads in this experiment) is therefore not lost either but builds up to a steady state where all the vanadium leached is precipitated. In an experiment run to demonstrate this, the steady state was reached as early as the second cycle (Table XVI). It is seen that about 75% of the vanadium was leached and precipitated in each cycle. Since vanadium precipitation from carbonate solution by lead is quantitative, we can achieve complete vanadium removal from the carbonate leach solution and 100% V_2O_5 recovery simply by using $\frac{100}{75} = 133\%$ of the **theoretical** minimum amount of lead - i.e.

133% of that amount based on 100% acid leaching of vanadium. Since we added 1 H_2SO_4 per lead per cycle this might make it appear that 4.44 H_2SO_4 rather than 3.33 H_2SO_4 per recovered V_2O_5 is necessary. We do not believe this is the case as H_2SO_4 was building up in the acid leach, being equivalent to 1.45 H_2SO_4 after the fourth cycle rather than 1.00. Also, in order that more than 3.33 H_2SO_4 per recovered V_2O_5 be consumed, it is necessary that V_2O_5 or vanadyl sulfate be retained as such with the $PbSO_4$. This may have occurred in this small scale experiment as the precipitates were not washed due to the difficulty of using a reasonable ratio of wash liquid to leach solutions. However it is not likely to present a serious problem in large scale operations since vanadyl sulfate should be easily washed out with water and V_2O_5 could, if necessary, be washed out with a little H_2O_2 which dissolves V_2O_5 readily and then gradually decomposes to reprecipitate it ✓.

Another advantage of recycling the acid leach is that what little uranium (.2%) is precipitated with lead vanadate (Table XV), concentrates in this acid solution and therefore could be allowed to build up until treatment of the acid leach for uranium recovery was desirable. In order to be sure that no uranium was remaining with the solid PbSO_4 , an experiment was run in which the lead vanadate precipitate was completely dissolved in nitric acid, and again .2% of the uranium was found in the acid solution.

It is therefore seen that the net process consists simply of replacing vanadate and some hydroxide by sulfate in the carbonate leach solution. A maximum of 3.33 H_2SO_4 per V_2O_5 is required and a minimum of 2.0 H_2SO_4 per V_2O_5 could be approached with a countercurrent process. The chemical cost, aside from losses and inventory, is just the cost of this H_2SO_4 .

A patent^{8/} was issued in 1913 for a process containing some of the same principles used here - i.e. precipitation of lead vanadate by lead sulfate and subsequent leaching of the vanadium by H_2SO_4 and recycling of the lead sulfate. The precipitation was done in dilute acid rather than carbonate solution, and the subsequent H_2SO_4 leaching resulted in recovery of an "acid solution of vanadium" rather than the unstable solution, which precipitates V_2O_5 on heating that we obtain by using a deficient amount of H_2SO_4 .

E. Effect of vanadium removal on subsequent sodium diuranate precipitation:

It is seen that the pH is somewhat lowered in lead vanadate precipitation (Table XV), as is to be expected from the formula of the precipitate. One might therefore expect that additional base would be required for subsequent diuranate precipitation. This was checked by comparing diuranate precipitation from two portions of Monticello carbonate leach solution, one portion of which had been previously treated with PbSO_4 to remove vanadium (Table XV). It was found that more base was indeed required for the same amount of uranium precipitation in the PbSO_4 treated leach until the point was reached at which the tails were .23 g/l U_3O_8 . At this point both treated and untreated leach required the same amount of base. Beyond this point the same amount of base gives lower tails with the treated leach, even though the pH is lower than with untreated leach (Table XVII). This is presumably due to vanadate complexing of uranium in the untreated leach.

Furthermore, dissolution of the diuranate precipitates shows that the treated leach gives a substantially purer product, containing 1 part by weight V_2O_5 for 81 parts U_3O_8 while untreated leach gave 1 part V_2O_5 for only 14 parts U_3O_8 .

These results, while illustrating that lead vanadate precipitation not only doesn't interfere with subsequent diuranate precipitation, but actually improves it, are certainly not the optimum possible with this process. A countercurrent lead vanadate process would result in less pH lowering since the precipitate formula would change from $3.33 \text{ PbO} \cdot \text{V}_2\text{O}_5$ toward $2 \text{ PbO} \cdot \text{V}_2\text{O}_5$ which, of course, ties up less hydroxide in the precipitate. Also, more complete vanadium precipitation is easily obtainable with either a batch or countercurrent process, the only reason for the relatively high vanadium tails (.11 g/l V_2O_5 Table XV) in our example was PbSO_4 losses in handling. Decreasing the vanadium concentration even further would, of course, further improve the purity of the diuranate precipitate.

F. Comparison with ferrous vanadate process: Cost calculations for a lead vanadate-sodium diuranate process analogous to the BMI ferrous vanadate-diuranate process ^{1/} have been made and are presented in section VI of this report.

Attempts to duplicate the specified BMI ferrous vanadate precipitation in this laboratory have been unsuccessful (Table XVIII), as was an attempt to improve ferrous vanadate precipitation by lowering the pH with CO_2 prior to adding the ferrous sulfate (Table XIX). Lead, in concentration specified herein, precipitated vanadium quantitatively from these solutions and was far superior to an equal amount of ferrous, both at this concentration and at the lesser concentration of precipitant specified by BMI for ferrous vanadate precipitation (Tables XVIII, XIX).

The data of Halpern ^{2/} on ferrous vanadate precipitation, on which the BMI specifications are supposedly based, actually support our view that the BMI flowsheet will not work. The BMI specifications call for a leach solution containing 50 gm/l Na_2CO_3 and 20 gm/l NaHCO_3 , but according to Halpern ^{10/} "The efficiency of the precipitation falls off very markedly as the concentration of carbonate or bicarbonate in the solution is increased. At 50 gm/l of Na_2CO_3 or NaHCO_3 only 75% of the vanadium is precipitated with the same (110% stoichiometric) amount of FeSO_4 . Above 50 gm/l of Na_2CO_3 precipitation is even less efficient, and the method becomes unsuitable." (Reference to the data confirms that Na_2CO_3 in the last sentence is used in the sense of $\text{Na}_2\text{CO}_3 + \text{NaHCO}_3$). Furthermore the above statement was made in connection with experiments based on an initial V_2O_5 concentration of 9.8 gm/l.

Examination of the U.B.C. data ^{11/} (Table XX) shows the efficiency of precipitation to fall off as the concentration of vanadium is decreased below 5 gm/l. It is seen that above this concentration, precipitation is essentially stoichiometric (on a 1 Fe per V basis) but if we consider a process of going from their final solution in experiment number:

UVS 1-4 ($\text{V}_2\text{O}_5 = 4.82$ gm/l; $\text{Na}_2\text{CO}_3 = 17.4$ gm/l; $\text{NaHCO}_3 = 24.3$ gm/l) to that in UVS 1-5 ($\text{V}_2\text{O}_5 = 1.29$ gm/l; $\text{Na}_2\text{CO}_3 = 6.6$ gm/l; $\text{NaHCO}_3 = 31.5$ gm/l) by the addition of 15 gm/l $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (as in their experiments), it is seen that .0540 moles/l Fe^{+2} removes .0388 moles/l V, or only 72% of the amount expected

on a 1 to 1 basis, and in addition .036 moles/l of total CO_2 (or 3.8 gm/l as Na_2CO_3) are lost. Even the above results are much more favorable than can be expected from the proposed BMI flowsheet which specifies lower initial and final vanadium concentrations and higher total CO_2 concentration, all of which tend to make the process less efficient, as easily seen from the UBC data.

The BMI assumption of 1 V removed for every Fe added is definitely incorrect but, simply for the sake of comparison, it was assumed that the BMI process would work as specified in making the cost analyses that follow.

VI. ECONOMIC EVALUATION

The experimental work discussed in the preceding sections of this report indicated that the lead sulfate - caustic method for recovering uranium and vanadium from carbonate leach solutions is a feasible process. In order to evaluate the process further it was decided to make a preliminary cost estimate for this process and compare this figure with the costs of other recovery methods.

As one condition for study the solution illustrated in Battelle's report ^{1/} on the alkaline leach pilot plant was selected, using 100 T/day of solution. This solution assayed 2.23 g/l V_2O_5 and 2.15 g/l U_3O_8 and the tails were 1.14 g/l V_2O_5 and 0.2 g/l U_3O_8 .

Only the costs directly connected with the precipitation section were considered. No building, general facilities, or operating capital costs are included since it is thought that these figures will be roughly equivalent for all processes.

The estimate for the lead sulfate - caustic precipitation is based on the flow-sheet shown in Figure 1. This flowsheet assumes that the lead vanadate cake may be leached in the filter press and the cake, as lead sulfate, returned to the initial agitator. Make up lead sulfate in the amount of 1% of the total is assumed. Plant investment and operating costs are tabulated in Tables XXI and XXII. The plant cost was found to be approximately \$75,000 and the operating cost was estimated at \$3.97 PTS.

An estimate was also made on the ferrous sulfate - caustic precipitation process for solution feed rate of 100 T/day. The equipment specified in the Battelle report was scaled up to handle the flow and the cost of this scaled up equipment was estimated, together with operating cost. The investment and operating cost are \$84,000 and \$4.24 PTS respectively.

The discussion of section V-F should be recalled at this point. It must be recognized that the ferrous sulfate process will not work as outlined and that actual costs to achieve the desired vanadium recovery by the process will be considerably higher than these estimates indicate.

In addition to the ferrous sulfate method, estimates were also prepared on other methods for recovery of U and V from carbonate solutions. The processes included in this study are acidification 12/, electrolytic reduction 13/, and sodium amalgam reduction 14/. The investment and operating costs for the various processes are tabulated in Table XXIII.

Vanadium in the leach solution has an appreciable effect on the recovery process. When present in relatively large amounts it has a deleterious action on the uranium precipitation, and it also increases the costs in many processes by consuming reagents or power. Since this is the case, estimates were made for two other solutions containing larger amounts of vanadium. The concentrations in these solutions were 14 g/l V_2O_5 , 3 g/l U_3O_8 and 25 g/l V_2O_5 , 3 g/l U_3O_8 . These concentrations correspond to two samples of actual leach solution obtained from the Monticello Mill for use in some of our prior investigations. The estimates of the cost for treating these solutions is shown in Table XXIII and curves showing the operating costs as a function of vanadium recovery are given in Figure 2.

It is apparent from the curves in Figure 2 that on the basis of operating cost alone the lead sulfate-caustic, amalgam, and electrolytic are all competitive processes if large amounts of vanadium are present. However, from the standpoint of operating simplicity and investment required the lead sulfate process has the advantage since no relatively complex equipment is required.

TABLE ISURVEY OF POSSIBLE PRECIPITANTS FOR +5V FROM CO_3^{2-} - HCO_3^- SOLUTION

<u>Test No.</u>	<u>Solution</u>	<u>Precipitant</u>	<u>Percent Vanadium Precipitated on Three Days Agitation</u>
20.53	90ml of .05M V_2O_5 .5M Na_2CO_3 , 5M NaHCO_3	.01 mole ZnCl_2	0
"	"	" $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$	1
"	"	" $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	2
"	"	" $\text{Ba}(\text{NO}_3)_2$	2
"	"	" HgCl_2	17
"	"	" $\text{CrCl}_3 \cdot 6\text{H}_2\text{O}$	26
"	"	" $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	30
"	"	" FeSO_4	34
20.57	"	" Hg_2Cl_2	52
20.53	"	" $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$	72
"	"	.02 mole AgNO_3	74
"	"	.011 mole $\text{Pb}(\text{NO}_3)_2$	75
"	"	" $\text{Pb}(\text{NO}_3)_2 + .0023$ mole NaCl^*	75

* Attempting to form vanadinite $3\text{Pb}(\text{VO}_4)_3 \cdot \text{PbCl}_2$

TABLE II

NON-ACIDIC LEACHING OF LEAD VANADATE

<u>Test No.</u>	<u>Leach Solution</u>	<u>Moles base/mole 3.33 PbO·V₂O₅</u>	<u>%V Leached</u>
20.131	.651M Na ₂ S	6.51	100.
"	.325 "	3.25	88.9
20.130	.325 "	3.25	84.7
"	.163 "	1.63	39.7
"	4.0 M NaOH	40.0	25.1
"	.80 "	8.0	1.7
20.94	.182M Na ₂ oxalate	4.0	0
"	.455M Na ₃ tartrate	10.0	0
20.89	1.0M Na ₂ SO ₄	20.0	0
"	.167 "	3.33	0
20.85	.17M Na ₄ P ₂ O ₇	1.00	0
"	.53M NaH ₂ PO ₄	2.22	0

TABLE III

EFFICIENCY OF VARIOUS LEAD SALTS AS PRECIPITATING AGENTS

<u>Test No.</u>	<u>Solution</u>	<u>Pb Salt*</u>	<u>% V Precipitated</u>	
			<u>Agitation</u> <u>1 day</u>	<u>4 day</u>
20.65	100ml .05M V_2O_5 , .5M Na_2CO_3 , .5M $NaHCO_3$	.01 mole $PbSO_4$	71.2	80.0
		.033 mole $Pb_3(OH)_2(CO_3)_2$	63.8	63.8
		.01 mole $PbCO_3$	61.9	62.0
		" $Pb(OH)_2$	57.8	58.2
		" PbO (Baker and Adamson)	26.0	52.3
		" $NaHPbO_2$	28.3	41.3

* All salts were freshly prepared except Baker and Adamson PbO . The more basic salts quite possibly appear somewhat inferior due to the pH effect (Table V).

TABLE IVEFFECT OF $\text{CO}_3^{=}$ CONCENTRATION ON PRECIPITATION OF +5V by +2Pb

<u>Test No.</u>	<u>$\text{CO}_3^{=}$</u>	<u>Vanadium Concentration</u>		<u>V/Pb mole ratio</u>		<u>Final pH</u>
		<u>Initial</u>	<u>Final</u>	<u>Initial</u>	<u>Precipitate</u>	
20.77	.429 m/l	.0429 m/l	.0166 m/l	1.0	.613	13.1
"	0	"	.0166	"	.613	13.2
"	.429	.0857	.0323	"	.623	12.9
"	0	"	.0324	"	.622	12.9

TABLE V

EFFECT OF pH ON PRECIPITATION OF +5V by +2Pb

<u>Test No.</u>	<u>$\text{CO}_3^{=}$ + HCO_3^{-}</u>	<u>Vanadium Concentration</u>		<u>V/Pb mole ratio</u>		<u>Final pH</u>
		<u>Initial</u>	<u>Final</u>	<u>Initial</u>	<u>Precipitate</u>	
20.72	.857 m/l	.0857	.00171	1.0	.980	8.8
20.71	"	"	.0222	"	.743	9.4
20.72	"	"	.0307	"	.641	9.65

TABLE VI

EFFECT OF INITIAL V/Pb RATIO ON PRECIPITATION OF +5V by +2Pb

<u>Test No.</u>	<u>CO₃⁼ + HCO₃⁻ Concentration</u>	<u>Vanadium Concentration</u>		<u>V/Pb mole ratio</u>		<u>Final pH</u>
		<u>Initial</u>	<u>Final</u>	<u>Initial</u>	<u>Precipitate</u>	
20.71	.857 m/l	.0857 m/l	.00083 m/l	.60	.594	9.2
"	"	"	.0120	.75	.645	9.3
"	"	"	.0222	1.0	.743	9.4
"	"	"	.0612	3.0	.962	9.6
20.66	"	"	.0887	10.0	1.05	10.4

TABLE VII

COMPOSITION OF LEAD VANADATE SOLID

Test No.	$\text{CO}_3^{=} + \text{HCO}_3^-$ Concentration (m/l)	Vanadium Concentration		V/Pb mole ratio		pH	Precipitate Analysis				V/Pb mole ratio
		Initial	Final	Initial	Precipitate		V_2O_5	Pb	CO_2	Na	
		(m/l)	(m/l)				(%)	(%)	(%)	(%)	
20.62	.870	.0870	.0076	.67	.61	10	18.8	68.1	2.0	1.22	.63
"	"	"	.0552	2.0	.72	"	21.2	68.5	.89	.56	.71
							Weight of Precipitate from 25 ml of slurry* Dried 20 Dried 45 Washed and hours minutes sucked dry at 110°C at 500°C				
20.71	.857	.0857	.00083	.60	.594	"	2.239gm	.9773gm	.9576gm		
	"	"	"	"	"	"	2.297	1.0149	.9942		

* Calculated weight on the basis of $3.33 \text{ PbO} \cdot \text{V}_2\text{O}_5$ is .995gm

TABLE VIIIRATE OF PRECIPITATION OF +5V by RECENTLY PRECIPITATED PbSO_4

<u>Test No.</u>	$\text{CO}_3^{=}$ + HCO_3^{-} <u>Concentration</u>	<u>Vanadium</u> <u>Concentration</u>		<u>Initial V/Pb</u> <u>mole ratio</u>	<u>pH</u>	<u>Agitation</u> <u>Time</u>
		<u>Initial</u>	<u>Final</u>			
20.78	.857 m/l	.0857 m/l	.0001 m/l	.60	10	10Min.
"	"	"	.0028	"	"	50
"	"	"	.0027	"	"	250
"	"	"	.0025	"	"	350

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TABLE IXEFFECT OF VARIATION IN THE AMOUNT OF H_2SO_4 ON THE LEACHING OF $3.33 PbO \cdot V_2O_5$

Test No.	Acid Concentration	Moles acid/ mole $3.33 PbO \cdot V_2O_5$	% Vanadium in Leach Solution			
20.96	.075 m/l	1.67	26, 30m agitation			
"	.150	3.33	68	"	plus 17 hrs. at R.T.	
"	.195	4.33	81	"	18	
"	.240	5.33	92	"	52	
20.86	.285	6.33	100	"	--	
						plus 1 hr. at 100°C
20.115.1	.319	3.83	84, 5m agitation	13	4	
" .2	.361	4.33	87	"	20	6
" .3	.402	4.83	89	"	38	13
" .4	.444	5.33	93	"	55	18

TABLE X

COMPARISON OF CALCULATED AND EXPERIMENTAL +5V CONCENTRATION
IN DILUTE ACID

<u>Solution</u>		<u>Final V</u> <u>Concentration</u>	<u>Final pH</u>	<u>Calculated</u> <u>V Concentration</u>
20.115.1	(See Table IX)	$.69 \times 10^{-2}$ m/l	1.40	$10^{-.92-1.40} = 10^{-2.32} = .48 \times 10^{-2}$ m/l
.2	"	1.0 "	1.05	$10^{-1.97} = 1.1$ "
.3	"	2.1 "	.65	$10^{-1.57} = 2.7$ "
.4	"	3.0 "	.65	$10^{-1.57} = 2.7$ "

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TABLE XI

AVERAGE CHARGE PER V IN THE UNSTABLE ACID LEACH SOLUTION

<u>Initial Solution</u>		<u>Final Solution</u> (after 2 hrs. at 100°C)
pH:	1.19	.99
V:	.101 m/l	.0164 m/l
$\text{SO}_4^{=}$ + HSO_4^- :	.091 m/l	.095 m/l
$\text{SO}_4^{=}$:	.014 m/l	.010 m/l
HSO_4^- :	.077 m/l	.085 m/l
H^+ :	.065 m/l	.102 m/l
Average charge per V + .40 $2 \times \text{SO}_4^{=} + \text{HSO}_4^- - \text{H}^+$		+ .2 (inaccurate due to small difference of large quantities)
$\frac{\text{H}_f^+ - \text{H}_i^+}{\text{V}_i - \text{V}_f} = +.4$		

Weight of V precipitate = .233 gm

" " " " calculated as V_2O_5 = .217 gm

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TABLE XIIIEFFECT OF TIME ON THE LEACHING OF $3.33 \text{ PbO} \cdot \text{V}_2\text{O}_5$ BY H_2SO_4

<u>Test No.</u>	<u>Acid Conc.</u>	Moles H_2SO_4 / mole 3.33 $\text{PbO} \cdot \text{V}_2\text{O}_5$	<u>Agitation Time</u>	<u>% Vanadium in Leach Solution</u>		
				<u>After Agitation Time</u>	<u>Plus Three Days at R.T.</u>	<u>Plus 30Min. at 100°C</u>
20,99	.195 m/l	4.33	5 Min .	94	25	12
"	"	"	15	88	20	9
"	"	"	30	87	28	14
"	"	"	180	54	29	13

TABLE XIIIEFFECT OF ACID CONCENTRATION ON THE LEACHING OF $3.33 \text{ PbO} \cdot \text{V}_2\text{O}_5$

<u>Test No.</u>	<u>Acid Conc.</u>	Moles H_2SO_4 / mole $3.33 \text{ PbO} \cdot \text{V}_2\text{O}_5$	<u>% Vanadium in Leach Solution</u>		
			<u>After 5 Min.</u> <u>Agitation</u>	<u>Plus</u> <u>17 Hrs. at</u> <u>R.T.</u>	<u>Plus</u> <u>1 Hr. at</u> <u>100° C</u>
20.117	.685 m/l	4.33	79	43	22
"	.513	"	82	39	33
"	.342	"	84	53	20
"	.171	"	91	54	16
20.115	.361	"	87	20	6
20.102	.374		88	-	8
				<u>Plus Three</u> <u>Days at</u> <u>R.T.</u>	<u>Plus 30Min.</u> <u>at 100° C</u>
20.99	.195		94	25	12

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TABLE XIV

EFFECT OF BOILING TIME ON THE UNSTABLE ACID LEACH SOLUTION

<u>Test No.</u>	<u>Acid Conc.</u>	Moles H_2SO_4 / mole 3.33 $PbO \cdot V_2O_5$	<u>Agitation Time</u>	<u>% Vanadium in Leach Solution</u>		
				<u>Initial</u>	<u>Final</u>	<u>pH</u>
20.102	.374 m/l	4.33	5Min.	90 - 1 Hr. at R.T. →	88	1.36
"	"	"	"	88 - 1 Hr. at 100°C →	8	1.20
20.103	"	"	10	88 - 5 Min. at 100°C →	12	1.21
"	"	"	"	85 - 1 Hr. at 100°C →	10	1.21

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TABLE XV

LEAD VANADATE PROCESS DEMONSTRATION ON

(Test No. 20.120)

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TABLE XVI

EFFECT OF RECYCLING THE H_2SO_4 LEACH

(Test No. 20,258)

Flowsheet: Experimental Data:

Cycle	Vanadium Content			
	PbSO_4 Precipitate	Acid V Sol'n.	Recycled H_2SO_4	V_2O_5 Precipitate
1	13%	7.89 g/l	-	87% of Feed
2	27	6.69	-	74
3	24	6.88	-	75
4	25	6.88	-	75
5	-	-	.648 g/l	-

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TABLE XVII

EFFECT OF LEAD VANADATE PRECIPITATION FROM MONTICELLO LEACH SOLUTION
ON SUBSEQUENT DIURANATE PRECIPITATION

(Test No. 20,123)

<u>NaOH Added</u>	<u>Initial pH</u>		<u>U₃O₈ After Five Days Agitation</u>		<u>Final pH</u>	
	<u>L-2*</u>	<u>Filtrate**</u>	<u>L-2</u>	<u>Filtrate</u>	<u>L-2</u>	<u>Filtrate</u>
0 gm/l	9.83	9.64	3.41 gm/l	3.34 gm/l	-	-
8.0	11.30	10.63	3.31	3.06	11.21	10.50
10.2	12.25	11.71	3.43	3.40	12.11	11.50
11.5	12.43	12.32	1.63	2.97	12.24	12.19
12.9	12.58	12.50	.49	.87	12.38	12.28
14.5	12.70	12.61	.20	.17	12.53	12.45
16.5	12.80	12.72	.12	.082	12.70	12.61
19.0	12.90	12.86	.097 <u>1/</u>	.055 <u>2/</u>	12.72	12.69
22.2	12.99	12.95	.094	.035	12.82	12.80

1/ Dissolution of the precipitate showed V₂O₅ to be 1/14 the U₃O₈ by weight.

2/ V₂O₅ = 1/81 U₃O₈ by weight.

* Leach solution without vanadium removed.

** See Table 15.

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TABLE XVIII

COMPARISON OF +2 Pb AND +2 Fe AS PRECIPITANTS FOR +5V IN "BMI" SOLUTION

BMI Solution: U_3O_8 .21 gm/l (Used equivalent amount of $UO_2(NO_3)_2$)
 V_2O_5 2.23 (Measured value = 2.17)
 Na_2SO_4 49.2
 Na_2CO_3 65.7
 $Na OH$ 4.92 pH 13.20 (Measured)

<u>Test No.</u>	<u>Added</u>	<u>Tails</u> <u>(1 Hr. Shaking)</u>	<u>pH</u>
20.211.1	1.82 g/l $FeSO_4$ (BMI specification)	2.16 gm/l V_2O_5	13.05
2	12.0 m/l $Pb(NO_3)_2$ (Equivalent Amount)	1.52 ($\frac{V}{Pb} = .595$)	13.11
3	41.0 " " (3.33 Pb/ V_2O_5)	0.00 ($\frac{V}{Pb} = .60$)	12.87
4	6.24 g/l $FeSO_4$ (3.33 Fe/ V_2O_5)	2.12	12.25

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TABLE XIX

COMPARISON AS IN TABLE XVIII WITH MODIFIED "BMI" SOLUTION

"BMI" solution with the pH lowered to 10.18 with CO_2 .

<u>Test No.</u>	<u>Added</u>	<u>Tails (1 Hr. shaking)</u>
20.219.1	1.82 g/l FeSO_4	2.07 g/l V_2O_5
2	12.0 ml/l $\text{Pb}(\text{NO}_3)_2$	1.35 ($\frac{\text{V}}{\text{Pb}} = .684$)
3	41.0 ml/l "	.03 ($\frac{\text{V}}{\text{Pb}} = .592$)
4	6.24 g/l FeSO_4	1.44

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TABLE XX

PRECIPITATION OF VANADIUM FROM CARBONATE SOLUTIONS
WITH FERROUS SULPHATE

Initial Solution: V_2O_5 - 19.5 gm/l (0.214 mV/l)
 U_3O_8 - 3.5 gm/l (0.0125 m U/l)
 Na_2CO_3 44.6 gm/l (0.421 m/l)
 $NaHCO_3$ 8.65 gm/l (0.103 m/l)

Procedure: Mixture shaken in stoppered flask for 1 hour.

Experiment No.	$FeSO_4$ gm/l	$7 H_2O$ m Fe mV	Final Concentration (gm/l)			
			V_2O_5	U_3O_8	Na_2CO_3	$NaHCO_3$
UVS- 1-1	0	0	19.5	3.5	44.6	8.65
UVS- 1-2	15.0	0.252	14.9	-	33.3	16.8
UVS- 1-3	30.0	0.504	9.5	-	24.6	20.5
UVS- 1-4	45.0	0.756	4.82	-	17.4	24.3
UVS- 1-5	60.0	1.008	1.29	3.3	6.6	31.5
UVS- 1-6	75.0	1.260	0.45	3.3	1.9	33.8

(Data taken from University of British Columbia Progress Report No. 1
December 1, 1951 to March 31, 1952 by J. Halpern)

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TABLE XXIPLANT INVESTMENT PbSO_4 - CAUSTIC PRECIPITATION

100 TONS SOLUTION/DAY

	Cost
Lead Vanadate Precipitation Tank	\$ 775.
Slurry Pump	200.
Lead Vanadate Filter	10,920.
Reagent Feeder (PbSO_4)	425.
Red Cake Precipitation Tank	1,880.
Red Cake Slurry Pump	300.
Red Cake Filter	4,440.
Surge Tank	435.
Pump	300.
NaOH Mixing Tank	550.
Diuranate Precipitation Tank	1,225.
Diuranate Filter	5,460.
Diurante Slurry Pump	200.
	<hr/>
Total	\$ 27,110.
Installation @ 50%	13,555.
Installed Equipment Cost (IEC)	\$ 40,665.
Piping @ 10% IEC	4,066.
Instrumentation @ 3% IEC	1,219.
Wiring	700.
Painting & Scaffolding @ 2% IEC	812.
Footings & Foundations @ 5% IEC	2,033.
Frts. & Handling	4,096.
	<hr/>
Installed Plant Cost (IPC)	\$ 53,591.
Engineering & Field Indirects 10% IPC	5,359.
Contractors' Fee 10% IPC	5,359.
Contingency 20% IPC	10,718.
	<hr/>
Total Plant Investment	\$ 75,027.

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Table XXIIOPERATING COST - PbSO_4 - CAUSTIC PRECIPITATION

100 TONS SOLUTION/DAY

	Cost \$PTS
(1) Operating Labor 0.32 Man Hr. @ \$2.00	\$ 0.64
(2) Maintenance Labor @ 4% Investment	<u>0.08</u>
	\$ 0.72
(3) Supervision & Clerical @ 15% Labor	<u>0.11</u>
	\$ 0.83
(4) Payroll OH @ 12.6%	.10
(5) Maintenance Supplies 5% Investment	<u>.10</u>
	\$ 1.03
(6) Power 2.15 Kwhr. @ 1.1¢	.02
(7) Steam 77 lb. @ 2.00 /M	.08
(8) Reagents: PbSO_4 0.12 lb. @ .22¢	.03
NaOH 20.1b. @ 7.16 ¢	1.43
H_2SO_4 3.87 lb. @ 1.74 ¢	<u>.07</u>
	\$ 2.66
(9) General Plant OH @ 40% (1-5)	.41
(10) Administration OH @ 10% (1-5)	.10
(11) Taxes & Insurance @ $1\frac{1}{2}\%$ Investment	.03
(12) Amortization (5 yrs.)	<u>.41</u>
	\$ 3.61
Contingency 10%	<u>.36</u>
	\$ 3.97

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TABLE XXIII

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SUMMARY OF COSTS - VARIOUS METHODS FOR RECOVERY OF U & V FROM CARBONATE

LEACH SOLUTIONS 100 TONS SOLUTION/DAY

Process		V ₂ O ₅ g/l	U ₃ O ₈ g/l	V ₂ O ₅ g/l	U ₃ O ₈ g/l	V ₂ O ₅ g/l	U ₃ O ₈ g/l
		Heads	Tails	Heads	Tails	Heads	Tails
		2.23	1.14	2.15	0.2	3	< 0.5
		25	4	14	4	25	< 0.5
		Investment	Op. Cost	Investment	Op. Cost	Investment	Op. Cost
		\$	\$ PTS	\$	\$ PTS	\$	\$ PTS
Lead Sulfate-Caustic		75,000	3.97	75,000	4.37	75,000	5.28
Ferrous Sulfate-Caustic		84,000	4.24	85,000	16.09	85,000	30.17
Sodium Amalgam *		52,830	3.66	172,360	4.99	247,460	6.63
Electrolytic *		238,600	4.92	298,000	5.73	377,200	6.71
Acidification		84,000	8.40	84,000	8.40	84,000	8.40

* Cost includes mercury inventory.

Operating cost includes 5 year amortization.

Investment does not include building, general facilities or operating capital.

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FIGURE 1

Lead Vanadate Precipitation Flowsheet

FIGURE 2
Recovery Costs
100 T/Day

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- (10) Op. Cit. Progress Report #2.
- (11) Op. Cit. " " #1 Table 18 p73.
- (12) Eng. Min. J 152 56-62 (1951).
- (13) Arthur D. Little Report RMO 2618 (March 1955).
- (14) Merrill Company Report RMO 2609 (November 1952).

Arthur D. Little, Inc.