REMOVAL OF IRON FROM SYNERGISTIC DI-2-ETHYLHEXYL PHOSPHORIC ACID AND TRI BUTYL PHOSPHATE ORGANIC SOLVENT LOADED WITH URANIUM FROM SULFATE LEACH LIQUOR

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ABSTRACT

Uranium leach liquor was obtained by uranium leaching of a technological sample of Gattar GV area, eastern desert, Egypt. Ore was subjected to uranium extraction using the synergistic organic solvent technique; the solvent used was mixture of di-2-ethylhexyl phosphoric acid and tributyl phosphate. The effect of sulfuric acid concentration, effect of leaching time, effect of leaching temperature, and liquid/solid phase ratio were investigated to produce sulfate leach liquor from GV. Synergistic 0.5 M D2EHPA and 0.5 M TBP organic solvent agitated with leach liquor solution of GV (Org./Aq. 2:1) for 5 min at room temperature were the optimum conditions for uranium extraction of 96%. Iron co-extracted into the loaded organic phase with uranium was removed by using oxalic acid (scrubbing process). In this concern, different parameters were investigated such as, effect of scrubbing time, effect of oxalic acid concentration, effect of temperature, and effect of organic to aqueous phase ratio.

INTRODUCTION

Uranyl leach liquor obtained by uranium leaching of a technological sample of Gattar GV ore was subjected to uranium extraction using solvent extraction technique. The solvent extraction process in its simplest form involves two steps; extraction and stripping (Johnd, 1992). In the extraction step, the organic solvent is in contact with the liquor to be treated and the chosen element is transferred to the solvent. In the stripping step, the loaded solvent is brought in contact with a suitable aqueous solution and the chosen element is transferred from the organic solvent back to the aqueous phase (Murthy et al. 1970). Recently (Singh et al.,2005) have developed a novel process based on the synergistic mixture of 1.5 M D2EHPA (di-2-ethylhexyl phosphoric acid) +0.2 M TBP (tri butyl phosphate) in paraffin for treating wet process phosphoric acid for the production of uranium peroxide (UO$_2$2H$_2$O) yellow cake. The attractive features of this process are the relatively lower cost of TBP compared to TOPO (tri-octyl phosphine oxide) and also its ability to co-extract rare earth elements along with uranium. The process is comprised of two cycles of solvent extraction. The first cycle consists of eight stages of uranium extraction from wet process phosphoric acid and eight stages stripping of uranium from loaded extract with concentrated and reduced phosphoric acid (55% P$_2$O$_5$). In the second cycle, uranium is extracted in eight stages from the stripped solution of first cycle after dilution and oxidation and then iron and rare earths are scrubbed with 30% H$_2$SO$_4$ prior to the recovery of uranium by ammonium carbonate solution. It has been
observed that during the second cycle operation of the solvent extraction process an appreciable concentration of iron (1.1 g/L) remains in the extract and creates problems due to hydroxide precipitation during subsequent carbonate stripping. A new process based on the use of steel scrap as a reducing agent directly in the organic phase in the galvanic stripping of iron from D2EHPA has been reported (Sun and O’Keefe, 2002). In case of a solvent extraction process, difficulty in stripping iron from the organic phase of D2EHPA may possibly be due to the conditions under which it is extracted. It forms different complexes with D2EHPA (H2A2) such as Fe(OH)A2(HA)2 and Fe(HA)3 when it is extracted from aqueous phase having different acidities (Shuqiu and Chen, 1989). When iron is extracted from a highly acidic and loaded on the D2EHPA phase, its stripping from it is difficult even with concentrated H2SO4 acid. In view of the previous difficulty, it is therefore desirable to examine the feasibility of using other reagents such as oxalic acid, which has been used on a large scale for the removal of iron stains from kaoline, quartz, and bauxite (Bonney, 1994; Veglio et al. 1996; Ambikadevi and M. Lalithambika, 2000). The use of oxalic acid for removal of iron from alkyl pyrophosphoric acid has also been reported (Worthington and A. Magdics, 1984). Iron was removed from the D2EHPA+TBP extract produced during uranium recovery from phosphoric acid by oxalic acid (Singh et al. 2006). The results revealed that a four stage counter-current stripping of iron has been carried out from an extract comprised of 1.5 M D2EHPA+0.2 M TBP+1.12 g/L Fe+14.8 g/L U3O8 with 7.5 wt. % oxalic acid using an organic to aqueous phase ratio (O/A) of 3 at 50 °C. This yields an extract with only traces of iron. From the resulting extract, a yellow cake exceeding the specification of commercial grade has been produced. The uranium extraction from Gattar GV sulfate leach liquor using aliquat-336 in a liquid emulsion membrane process was investigated (Maysa, 2003), it was possible to determine suitable operating parameters. These include 0.02 M Aliquat 336 in kerosene (5 wt. % Span 80) and a sulfuric acid concentration of 15–30 g cm−3 in the external feed phase. A maximum volume ratio of 1:1:80 for a uranium concentration in the feed liquor 1.2 g cm−3 with a molarity of the internal phase slightly over 1 M Na2CO3 and for about 25 min mixing time at 180 rpm would lead to complete uranium transfer. The present study aims to the removal of iron from loaded synergistic D2EHPA and TBP organic solvent produced from extraction of uranium from Gattar GV to produce pure uranium stripped solution and finally production of iron free yellow cake.

EXPERIMENTAL

Material

Leaching experiments

A number of experiments was performed using 50 g portions of the powdered sample (-5:+60 mish size) in a glass beaker then adding the desired amount of sulfuric acid (1 to 40 %) with mass/volume ratio ranged from 1/1 to 1/4 the reaction carried out at different times from 0.5 up to 7 hr at different temperatures from room temperature (about 298 K) up to 333 K and the obtained reaction slurry was thoroughly mixed. After leaching experiments were ended it subjected to the filtration and then the leaching efficiencies were calculated.

Solutions and reagents

Stock solution of uranium (1000 ppm) was supplied from AccuStandard, USA. All chemicals and reagents were of A.R. grade and used without further purification. Fe was determined by atomic adsorption spectrometer (GBC 932- AAS).

Analytical procedures

Uranium was analyzed in the corresponding aqueous phases using ArsenazoIII reagent under different conditions (Marczenko, 1976) for this purpose, a Lambada UV/VIS spectro-
photometer (Perkin-Elmer, USA) was used. In addition, uranium was also analyzed by an oxidimetric titration method against ammonium metavanadate in the presence of diphenylamine sulfonate indicator prior to titration; proper reduction of uranium was performed using ferrous sulfate (Ritcey and Ashbrook, 1984). The representative sample of Gattar V composite sample used in this study was obtained from Gattar GV by Nuclear Materials Authority (NMA).

**Extraction process**

The experiments were carried out by batch technique. Uranium extraction experiments were carried out in mechanically agitated beakers containing 50 mL of leach liquor solution of (1.1 g/l) uranium with diethylhexyl phosphoric acid (D2EHPA) and tributylphosphate (TBP) synergistic organic solvent to aqueous phase (O/A) ranged from 0.2 to 3, the vessel was immersed in a water bath controlled at different temperatures. The content was agitated with a constant stirring rate of 400 rpm, at the preset times, after stirring the organic phase was separated from the aqueous phase, and the concentration was analyzed. The concentration of extracted uranium was determined from the difference between the initial and final concentrations of uranium in aqueous solutions.

**Scrubbing process**

The organic extract may still have high concentration of undesirable iron (4685 ppm). Scrubbing of the organic extract is conventionally carried out using an aqueous solution of oxalic acid. Because the scrubbing process is an important step towards getting high grade of yellow cake product, the optimum conditions should be determined by studying the effect of the factors controlling the efficiency of this process such as the oxalic acid concentration, scrubbing temperature and scrubbing stirring time. In this concern, 50 mL of loaded organic solvent containing 1.056 g/l uranium and 4685 ppm iron of was agitated with different concentrations of oxalic acid ranged from 1 to 20 g/100ml; the vessel was immersed in a water bath controlled at different temperatures (298-333 K). The content was agitated with a constant stirring rate of 400 rpm. The preset times after stirring, the organic phase was separated from the aqueous phase, and the concentration was analyzed. The concentration of removed iron was determined from the difference between the initial and final concentrations of iron in aqueous solutions.

**Stripping process**

Stripping of the extracted uranium from the organic layer was successfully performed using different concentrations of sodium carbonate (1-20 g/100ml). The stripping investigations were carried out to choose the best conditions that can successfully strip uranium from the organic phase. The effect of stirring time from 0.5 to 30 min. and temperature from 298 to 333 K were studied.

**Calculations**

The percent yield (Y%) and the distribution coefficient D were calculated from the equations:

\[
\text{Yield (Y %)} = \frac{[\text{U}]_{\text{feed solution}} - [\text{U}]_{\text{raffinate}}}{[\text{U}]_{\text{feed solution}}} \times 100
\]

The distribution ratio (D) was calculated from the equation:

\[
D = \frac{\text{Total concentration of uranium in organic phase}}{\text{Total concentration of uranium in aqueous phase}}
\]

**RESULTS AND DISCUSSION**

**Leaching of Gattar GV Ore Using Sulfuric Acid**

**Effect of acid concentration**

In order to study the effect of acid concentration upon the leaching efficiency of Gattar GV composite sample, series of leach-
ing experiments were performed using different acid concentrations ranging from 1% up to 40%. The experiments were carried out under fixed conditions of 298 K leaching temperature for 5 hr., and 3/1 volume/mass ratio. The obtained data were plotted on Fig. (1). From this figure, it is clearly obvious that 40% acid is the economically preferable specially other leaching factors are taken into.

**Effect of liquid/solid ratio**

In order to study the effect of liquid/solid (v/m) ratio upon the leaching efficiency of Gattar GV composite sample, series of leaching experiments were performed using different liquid/solid ratio ranging from 1/1 up to 4/1. The experiments were carried out under fixed conditions of 298 K leaching temperature for 5 hr., and 40% sulfuric acid concentration. The obtained data are plotted on Fig. 2. From this figure, it is clearly obvious that leach efficiency increasing as the acid amount increases until the experiment number three (L/S equal 3/1). Later experiments did not show any leaching efficiency improvements.

**Effect of the leaching temperature**

To investigate the effect temperature upon leaching efficiency of uranium from Gattar GV composite sample, series of leaching experiments were performed using different temperatures ranging from 298 to 333 K. The experiments were carried out under fixed conditions of 1/2 S/L ratio, reaction time of 5 hr, and acid concentration 40%. The obtained data are plotted on Fig. 3. From this figure, it is clearly obvious that leach efficiency increase from about 80.6 to 95.0% at 298 and 333 K respectively. From economical point of view, 298 K was chosen to be the reaction temperature.
ing experiments were performed at different time ranging from 0.5 hr to 7 hr. The experiments were carried out under fixed other conditions of 3/1 v/m ratio, temperature of 298 K, and acid concentration 40%. The obtained data were plotted on Fig. 4. From this figure, it is clear that the leaching efficiency is increasing as the reaction time increases from 0.5 hr to 4 hr from about 12.4% to about 91.2% respectively, and no high leaching efficiency increase by increasing the stirring time more than 5 hr; so that the optimum time is 5 hr.

**Uranium Extraction Process**

**Effect of organic to aqueous phase ratio**

The extraction of uranium from Gattar GV leach liquor sulfate solution was studied at room temperature. The extraction of uranium with synergistic organic solvent (0.5 M D2EHPA and 0.5 M TBP) was investigated by varying the organic to aqueous phase ratio from 0.2 to 3.0. The extraction was performed at room temperature (298 K) during stirring for 5 min. The results are represented graphically on Fig. 5 as a relation between organic/aqueous phase ratio and uranium extraction efficiency percent. The extraction percent increases with the increase of organic phase to aqueous phase to reach 97.7% for org./aq. 3:1, while it reached to 82.9% in the case of org./aq. 1:3.

**Effect of D2EHPA/TBP molar ratio**

The extraction of uranium from leach liquor sulfate solution using (0.5 M D2EHPA and 0.5 M TBP) mixture solvent was achieved under room temperature and organic to aqueous phase ratio 2:1. Different molar concentrations of D2EHPA and TBP in the range of 0.33 to 3.00 were used and stirring time of 5 min. The results are graphically represented on Fig. 6. The results indicate that the uranium extraction efficiency percent increases with the increase of D2EHPA concentration while it decrease with the increase of TBP concentration. The uranium extraction efficiency percent reached to 98.3% by using D2EHPA/
TBP equal 3:1 while the extraction efficiency decreased to 82.6 % by using D2EHPA/TBP equal 1:3.

**Effect of temperature**

The extraction of uranium from the leach liquor sulfate solution at different temperature was investigated. The extraction experiments were carried out by contacting the leach liquor sulfate solution with (0.5 M D2EHPA and 0.5 M TBP) for 5 min. while the org./aq. ratio was fixed at 2:1, the temperature of extraction was varied from 298 to 333 K. The results on Fig. 7 shows that the extraction percent is slightly increased from 96.0 to 97.0 % only with the rise of the temperature from 298 to 333K. So that, the extraction process was preferable at room temperature from economic point of view.

**Effect of oxidation state**

The effect of the oxidation state on uranium extraction from Gattar V leach liquor sulfate solution was investigated at org. / aq. phase ratio equal to 2:1. All experiments were performed using a mixture of 0.5 M D2EHPA and 0.5 M TBP during stirring time for 5 min. at room temperature. Figure (8) shows clearly that the extraction of uranium increases with the increase of the oxidation state 97% at 700 mv while the uranium extraction efficiency decreases by reducing the leach liquor solution with iron to reach its minimum value of 50% at 330 mv. The oxidation state of the Gattar V leach liquor sulfate solution was measured as 520 mv, at this oxidation state the extraction efficiency was 96% and by oxidizing the solution using hydrogen peroxide to 700 mv, the uranium extraction efficiency increase with small value from 93 % to 97%. So that the preferred extraction oxidation state is 520 mv.

**Effect of stirring time**

Stirring time is an important factor in determining the efficiency of uranium extraction processes which involve mass transfer between two liquids. The effect of stirring time was investigated between 0.5 and 30.0 min, at extraction temperature 298 k. The results represented on Fig. 9 reveal that (i) within half minute; the extraction efficiency reached 70.6% indicating that the extraction is very rapid. (ii) Stirring time has a positive effect on the extraction efficiency which increased to 96 % within 5 min. Beyond 5 min. stirring, the increase of extraction efficiency was too small to be neglected. Based on these foundations, the effect of other parameters on the extraction of uranium with a mixture of 0.5 M D2EHPA and 0.5 M TBP was studied keeping the stirring time constant at 5 min.
McCabe Thiele diagram for extraction of uranium

Uranium extraction equilibrium curve (Fig. 10), was used to calculate the number of counter current stages required to achieve about 98.3% extraction yield at room temperature. The operating line was drawn according to the uranium concentration of the feed solution. The number of stages to obtain a yield of 98.3% according to the method of McCabe Thiele was found to be 3.00 stages.

Scrubbing Process Using Oxalic Acid

During the uranium extraction process from the Gattar V leaching liquor sulfate solution, iron was extracted with the uranium and loaded on the organic phase, so the produced yellow cake will include a high concentration of iron. So that, scrubbing or (stripping of iron) before stripping and precipitation of the uranium from organic phase is a necessary process. Many trials have been studied in the previous works for scrubbing process (Singh et al., 2006). Table (1) summarizes the effect of various reagents studied for the stripping of iron from the organic phase of 1.5 MD2EHPA+0.2 M TBP loaded with 10 g/L Fe at (Org/Aq.) ratio equal to 1 and 27 °C (Singh et al. 2006), the contact time is maintained at 20 min for all the test runs. The stripping efficiency of the studied reagent is found to follow the order: oxalic acid, ammonium oxalate, hydrochloric acid, sulphuric acid, sodium oxalate and citric acid. Hirato et al., 1992 have observed 83% stripping iron in a single stage with 2 M HCl from the organic phase comprised of 0.3 M D2EHPA+0.6 M TBP+2.4 g/L Fe, the poor stripping of iron (7.53%) obtained as showing in (Table 1) is most likely due to difference in the concentration of extractant and mole ratio of D2EHPA and TBP, it is of interest to examine the stripping behaviour of oxalic acid for uranium, accordingly in further experiments, organic phase of 1.5 M D2EHPA+0.2 M TBP loaded with 14.80 g/L U₃O₈, 1.0 and 5.0 g/L

Table 1: Effect of various reagents on removal of iron (Singh et al., 2006)

<table>
<thead>
<tr>
<th>Reagent</th>
<th>Concentration, M</th>
<th>D</th>
<th>Removal of iron, %</th>
</tr>
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<tbody>
<tr>
<td>Citric acid</td>
<td>0.5</td>
<td>20.8</td>
<td>4.8</td>
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<tr>
<td>Oxalic acid</td>
<td>1.3</td>
<td>0.4</td>
<td>70.0</td>
</tr>
<tr>
<td>HCl</td>
<td>2.0</td>
<td>13.3</td>
<td>7.5</td>
</tr>
<tr>
<td>H₂SO₄</td>
<td>5.4</td>
<td>15.6</td>
<td>6.9</td>
</tr>
<tr>
<td>Sodium oxalate</td>
<td>0.2</td>
<td>57.2</td>
<td>1.7</td>
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Organic extract: 1.5 M D2EHPA + 0.2 M TBP + 10 g/L Fe, phase ratio org/aq/ = 1 and 27 °C
Y(III) separately (representing the concentration of these metal ions in the extract) have been equilibrated with 10% oxalic acid solution using Org/Aq. = 1 at 27 and 55°C respectively, the stripping of uranium and yttrium are found to be 0.2% and 90%, respectively, at both the temperatures. Similar observation has been reported by Hung et al., 1990 for stripping of rare earths loaded in D2EHPA by oxalic acid. Since oxalic acid gives satisfactory results for effective stripping of iron and rare earths with insignificant loss of uranium, the rest of the work in the present investigation has been carried out only with oxalic acid.

**Effect of scrubbing time**

The effect of stirring time was studied in the range of 0.5 to 30.0 min. The stripping percentage of iron from the organic phase comprised of 0.5 M D2EHPA and 0.5 M TBP loaded with 4.685 g/L iron obtained by stripping with 15% oxalic acid as a function of contact time at room temperature (298 k) is shown on Fig.11. Equilibrium is attained after 10 min stirring of phases, irrespective of the concentration of iron in the loaded organic phases. Similar behaviour has been reported for iron stripping from an alkyl pyrophosphoric acid extractant (Worthington and Magdics, 1984).

**Effect of oxalic acid concentration**

The effect of oxalic acid concentration on iron stripping from the organic phase loaded with 4.685 g/L Fe has been studied at room temperature and stirring time for 10 min. The results are given on Fig.12. It is seen that stripping percentage of iron increases with increasing oxalic acid concentration. The optimum condition for effective stripping is found at 15% oxalic acid solution as stripping agent at 298 k for 10 min. by using these conditions, the iron stripping efficiency in 15% oxalic acid phase was observed 88%.

**Effect of iron removal temperature**

The stripping of iron from the loaded 0.5 M D2EHPA and 0.5 M TBP organic phase was investigated at different temperatures. The stripping experiments were carried out by stirring the loaded organic phase with 15% oxalic acid solution for 10 min. while the org./aq. ratio was fixed at 1:2, the temperature of extraction was varied from 298 to 333 k. The results on Fig. 13 shows that the iron stripping efficiency percent increased from 88.0 to 97.0% with the rise of the temperature from
Effect of organic to aqueous phase ratio

A series of experiments was performed with different organic phase to oxalic acid ratios ranging from 0.25 to 4.0. The experiments were conducted using 15% oxalic acid as stripping solution and stirring time 10 min under heating at 333 K. From the results represented graphically on Fig. 14, the iron stripping efficiency was increased by increasing the Org/Aq phase ratio reached 97 % at Org/Aq phase ratio equal to 1:2.

Stripping of Uranium From Extract Organic Phase

The iron free extract organic phase is then subjected to uranium stripping to produce highly pure grade yellow cake by stripping process using sodium carbonate.

Effect of sodium carbonate concentration

The stripping process of uranium from the synergistic 0.5 M D2EHPA + 0.5 M TBP mixtures at room temperature and org / aq phase ratio is equal 1:4 by using 15% sodium carbonate (wt/v) was investigated. From Fig. (15), it is noticed that the uranium stripping efficiency percent is increased by increasing the concentration of sodium carbonate to reache 95 % using 15.0 % (wt/v) solution of sodium carbonate.

Effect of uranium stripping stirring time

The effect of stirring time on the stripping of uranium was studied in the range of 1.0 – 30.0 min. From Fig. (16), it is noticed that the uranium stripping efficiency percent is increased by increasing the stirring time to reache 95.0 % at 10 min. Uranium was stripped.
from the loaded organic phase with 15% wt/v sodium carbonate solution.

**Effect of aqueous to organic phase ratio**

In order to study the effect of the aqueous/organic phase ratio upon uranium stripping efficiency from the uranium-loaded solvent sample of GV composite sample, a series of stripping experiments were performed using Org./Aq. ratios ranging from 1/1 down to 1/5. In these experiments, 15% wt/v sodium carbonate solution was used and the time was fixed at 10 min. The obtained data given in Fig. 17 revealed that, the uranium stripping efficiency increased by increasing the aqueous to organic phase ratio to reach 95% at aq./org. ratio equal 4:1.

**CONCLUSION**

From the obtained results, the uranium leaching efficiency increased to reach 91.2% by stirring 40% sulfuric acid for 5 hr at room temperature and by using aqueous to mass phase ratio equal to 3:1.

For uranium extraction process; a synergistic 0.5 M D2EHPA and 0.5 M TBP organic solvent was used at room temperature and mixed with the GV sulfate leach liquor solution by org/aq. phase ratio equal 2:1 and stirring for 5 min. by applying these conditions, 96% of the uranium in the GV sulfate leach liquor was successfully extracted. A process for the removal of iron with oxalic acid from the extract synergistic 0.5 M D2EHPA and 0.5 M TBP organic solvent is obtained. It is concluded that, 15% oxalic acid was suitable for removal of 88.0% of the iron content in the loaded organic phase with uranium, while by increasing the temperature the iron removal efficiency increased to reach 97.0% by applying the same conditions under org/aq. phase ratio equal 1:2 and stirring time for 10 min. The uranium was stripped from the iron free extract organic phase by using 15% sodium carbonate solution for 10 min stirring time, these conditions strip 95% of the loaded uranium. The stripped uranium solution was used to precipitate the uranium and filtered as a peroxide hydtrate, washed, dried, and calcined at 400°C to obtain UO$_3$ powder.

**REFERENCES**


REMOVAL OF IRON FROM SYNERGISTIC DI-2-ETHYLHEXYL


حمض كبريتيك لمدة 5 ساعات في درجة حرارة الغرفة بنسبة محلول حمض الكبريتيك إلى الخامس.

تمت دراسة العوامل المختلفة لعملية استخلاص اليورانيوم باستخدام حلول المذيب العضوي التأريري
ثنائي ايثيل هيكسيل حمض الفوسفوريك وثلاثي بيوتيل الفوسفات مثل نسبة المذيب إلىحلول. زمن التقليل. تركز المذيب. تركيز ثنائي ايثيل هيكسيل حمض الفوسفوريك إلى ثلاثي بيوتيل الفوسفات.
ودرجة الحرارة. وكانت أفضل الظروف لاستخلاص حوالي 96% من اليورانيوم كالآتي: عمل حلوط من
0.5 مولار ثنائي ايثيل هيكسيل حمض الفوسفوريك و0.5 مولار ثلاثي بيوتيل الفوسفات والتقليل لمدة 5
دقائق مع المحول في درجة حرارة الغرفة بنسبة مذيب عضوي إلى المحول 1:1. ثم بعد ذلك استخدام
محلول من حمض الأوكساليك تركيز 15% لعمل ازالة للحديد في المحلول من اليورانيوم في المذيب العضوي
ودراسة العوامل المختلفة لانجاز تقليل الحديد قبل عملية استرجاع اليورانيوم. ولانجاز هذا الهدف تم
التقليل المذيب العضوي المحمل باليورانيوم وال الحديد مع محلول الأوكساليك لمدة 10 دقائق وبنسبة 2:1
وكانت نسبة ازالة الحديد وصلت إلى 88٪ عند التطبيق في درجة حرارة الغرفة بينما تزيد هذه النسبة
تصل إلى 97٪ بالتسخين عند 60 درجة مئوية. بعد اجراء عملية ازالة الحديد تم استرجاع اليورانيوم من
المذيب العضوي باستخدام محلول من كربونات الصوديوم ثم ترسيب اليورانيوم.