

# Studies on uranium recovery from a uranium-bearing phosphatic sandstone by a combined heap leaching–liquid–gel extraction process. 1—Heap leaching

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## ABSTRACT

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In the mineralization under investigation uranium does not form a definite mineral but it is hosted by apatites. The batch used in the present work assays between 0.045 and 0.048% uranium. Heap leaching was first tried with a  $\text{Fe}_2(\text{SO}_4)_3/\text{H}_2\text{SO}_4$  solution of pH 1.7 but the results were discouraging. Leaching with  $\text{H}_2\text{SO}_4$  solutions of moderate acidity containing a low concentration of  $\text{HNO}_3$  was next tried. The process was found to proceed in two distinct stages, dissolution and a step called rinsing. The factors that affect uranium recovery and acid consumption during the dissolution stage were investigated and the optimal conditions for uranium recovery were determined.

## INTRODUCTION

Interest in low-grade uranium resources has increased markedly due to increasing energy requirements and the rapid exhaustion of the known conventional uranium ores. However, there are factors which limit the beneficiation of low-grade materials, which include production cost and environmental constraints on the mining and milling of large amounts of ore. Therefore, intensive studies are needed to find methods for economic beneficiation of low-grade materials.

Of the low-grade uranium materials, phosphates have received the greatest attention. In Egypt the phosphate reserves present at the Qatrany area, south-

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west of Cairo, have a relatively high uranium assay. Therefore, efforts have been mainly directed towards the beneficiation of these reserves.

Previous work [1,2] has shown that the Qatrany phosphate sandstone (hereafter abbreviated to QPS) is mainly formed of quartz grains cemented together with a siliceous matrix. Small apatite grains were found to be present at the borders of, and sometimes as inclusions in, the quartz grains. Mineralogical studies complemented by chemical, X-ray and emulsion plate investigations have revealed the complete absence of a definite uranium mineral, suggesting that the uranium-bearing minerals are in the form of carbonate-apatite species. The chemical analysis of a representative homogenized sample of the QPS used in the present work (Table 1) indicates that it is mainly composed of silica and calcium phosphate. The uranium assay is low, between 0.045 and 0.048%.

In sedimentary phosphates uranium is mainly present in the tetravalent state, the remainder being in the hexavalent form [3]. The tetravalent uranium is believed to be in isomorphous substitution with  $\text{Ca}^{2+}$  ions in the apatite crystals. Hexavalent uranium is apparently adsorbed on the surface. Since most of the uranium is present in the crystal lattice of the apatite, it is not possible to separate a uranium concentrate from the QPS by physical means. Therefore, separation of the uranium-bearing minerals from the siliceous gangue was tried [4–7] but the ordinary physical methods appear, so far, to offer little chance of successful application. On the other hand, no chemical process could be found which would selectively extract uranium from QPS. Moreover, leaching by the conventional methods leads to difficulties in the solid–liquid separation and high operating costs. For this reason, and also because the relatively small reserves of the QPS do not justify either the cost of its transport to distant processing centres or the erection of a conventional plant in the area, studies have been directed to develop a relatively simple process for uranium extraction and separation at the mines. Among the apparently suitable solutions to this problem, heap leaching combined with the liquid–gel extraction process [24] seemed to be worth considering.

TABLE 1

Chemical assay of the major constituents in the QPS sample under investigation

Constituent	Content (%)	Constituent	Content (%)
$\text{SiO}_2$	83.5	$\text{K}_2\text{O}$	0.09
$\text{CaO}$	6.8	$\text{Fe}_2\text{O}_3$	0.36
$\text{P}_2\text{O}_5$	5.01	$\text{TiO}_2$	0.18
$\text{Al}_2\text{O}_3$	1.4	$\text{SO}_3$	0.17
$\text{CO}_2$	0.60	$\text{MnO}_2$	0.13
$\text{Na}_2\text{O}$	0.46	Loss on ignition	0.90

The heap leaching process [8] simply consists in constructing heaps of the ore over a collection system. The lixiviants, usually very dilute sulphuric acid, are distributed over the top surface of the heap and allowed to pass downward through the ore bed at very slow rates. The bed may be run-of-mine ore or coarse-crushed material.

## EXPERIMENTAL

### *Outline of the leaching procedure*

The QPS was crushed to  $-1/2$  inch ( $-12$  mm), well homogenized, sampled and analyzed. Samples, each weighing 4 kg, were placed in pyrex glass columns of 9.2 cm internal diameter. The depth of the QPS bed was about 66 cm. Each column was closed at one end to form a flat bottom drawn at the center into a 2 cm diameter discharge tube. At the bottom of each column was placed a thick layer of glass wool. The surface of the bed was covered with a thick layer of filter paper for the homogeneous distribution of the lixiviant solution. Leaching was started by just saturating the bed with water to establish circulation and prevent channelling. The leaching solution was then allowed to drop on to the surface of the bed at the specified rate. The effluent solutions (leach liquors) were collected in fractions at the end of certain leaching periods (usually 24 h). The volume of each fraction was measured and its pH value determined. Aliquots were subjected to chemical analyses for the determination of U(VI), Fe(III) and Fe(II), calcium and  $P_2O_5$ .

### *Analysis*

Depending on its concentration, U(VI) was determined either polarographically using the catalytic nitrate wave [9] after purification on a strong anion-exchange resin [10] or fluorometrically after a purification step involving the extraction of U(VI) by ethyl acetate [11]. Fluorometric measurements were run on five or more samples and the average taken. The percent uranium leached was calculated using the formula:

$$\% \text{ extraction} = \frac{\text{uranium leached}}{\text{residual uranium in the bed} + \text{total uranium leached}} \times 100$$

Fe(II) was determined spectrophotometrically [12], while the total iron was determined either spectrophotometrically [12] or by atomic absorption spectrophotometry. However, no Fe(II) could be detected in the leach liquors probably due to prevailing oxidation conditions in the bed or oxidation of Fe(II) by air during the long periods of collection. Calcium in low concentrations was determined by flame emission spectrophotometry. Higher amounts were determined by EDTA titration [13].

$P_2O_5$  was determined by reduction of the yellow phosphomolybdate complex and measuring the transmittance of the solution [14]. In the presence of excessive amounts of Fe(III), the yellow phosphomolybdate complex was extracted with isobutyl alcohol before reduction.

## RESULTS

Leaching experiments were run with either a ferric sulphate/sulphuric acid solution of pH 1.7, or sulphuric acid solutions of moderate acidities.

### *Leaching with a ferric sulphate/sulphuric acid solution of pH 1.7 containing 2 g Fe(III)/l*

This solution was allowed to pass downwards through the QPS bed at a flow rate of  $\sim 30$  ml/kg QPS bed per 24 h. Conditions of the experiment and the results obtained for the leaching of uranium, iron, calcium and  $P_2O_5$  over a leaching period of more than 500 days are summarized in Fig. 1.

### *Leaching with sulphuric acid solutions of moderate acidities*

#### *Effect of the rate of sprinkling*

The effect of the sprinkling rate on both the pH of the effluent solution and the rate of uranium, iron, calcium and  $P_2O_5$  leaching has been investigated at two levels of sulphuric acid concentration, namely 16.1 g  $H_2SO_4$ /l and 48.3 g  $H_2SO_4$ /l of tap water and sprinkling rates of about 0.4, 1.2, 2 and 4 l/4 kg

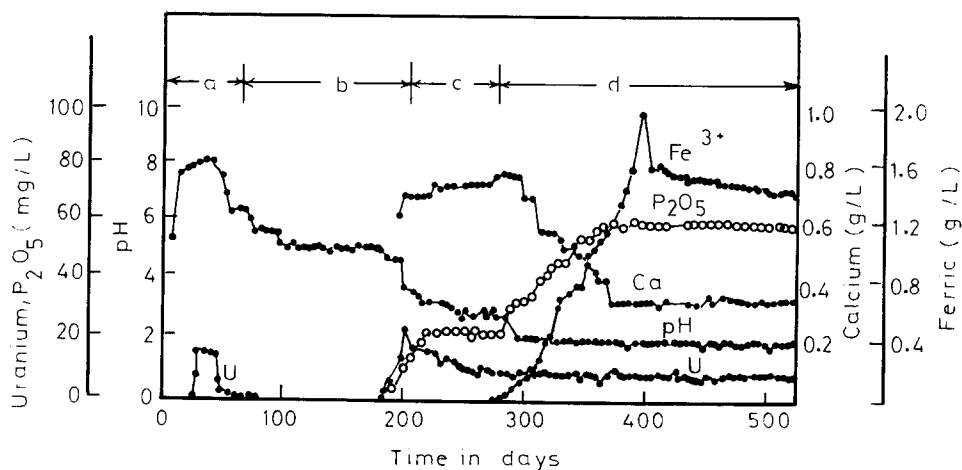


Fig. 1. Heap leaching with  $Fe_2(SO_4)_3/H_2SO_4$  solutions of pH 1.7. Conditions: charge of column 6 kg QPS assaying 0.046% uranium; rate 180 ml/24 h.

QPS bed per 24 h. The leaching curves obtained for the different ions under the various experimental conditions tested were found to have similar patterns. Therefore only the curves obtained for leaching with 48.3 g  $\text{H}_2\text{SO}_4/\text{l}$  at a sprinkling rate of about 1.2 l/4 kg QPS bed per 24 h are presented (Figs. 2 and 3) for illustration.

As shown in Fig. 2, with the increase of the effluent volume the rate of uranium leaching increases sharply (dissolution stage) and then tends to level off to form what is termed the rinsing stage. The results obtained for the effect

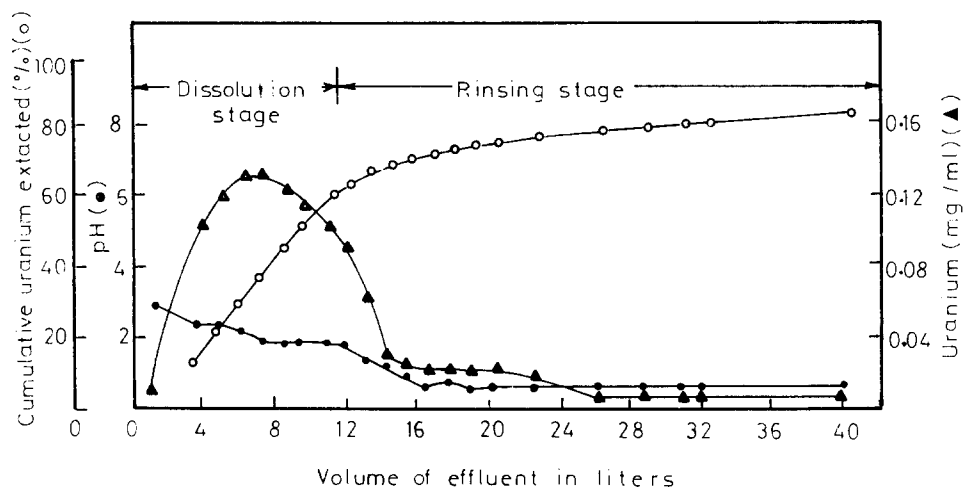


Fig. 2. Heap leaching with 48.3 g of  $\text{H}_2\text{SO}_4/\text{l}$  (pH and uranium extraction). Conditions: charge of column 4 kg QPS assaying 0.047% uranium; rate 1.2 l/24 h.

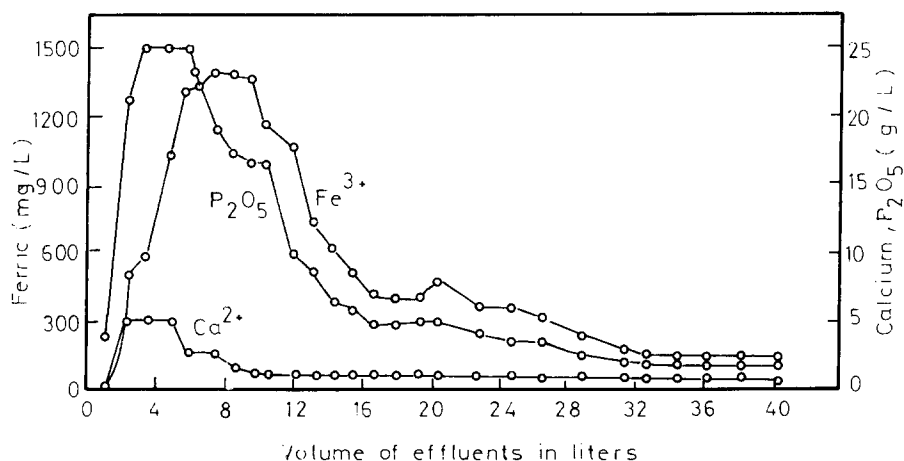


Fig. 3. Heap leaching with 48.3 g of  $\text{H}_2\text{SO}_4/\text{l}$  (ferric,  $\text{P}_2\text{O}_5$  and calcium extraction). Conditions: charge of column 4 kg QPS assaying 0.047% uranium; rate 1.2 l/24 h.

of the rate of sprinkling on the percentage cumulative uranium dissolved during the dissolution stage are summarized in Table 2 which also presents the amounts of  $\text{H}_2\text{SO}_4$  required to extract 1 kg of uranium under the different leaching conditions tested. As shown in the table, both the percentage cumulative uranium recovery and the  $\text{H}_2\text{SO}_4$  requirements increase as the rate of sprinkling is increased.

*Effect of the acidity of the lixiviant solution on the uranium recovery*

The percentage cumulative uranium recovery obtained at various constant sprinkling rates and different acidities are given in Table 3. This table indicates that under comparable experimental conditions, both the uranium recovery and the  $\text{H}_2\text{SO}_4$  requirement markedly increase on increasing the acidity of the leaching liquor. However, from an economic point of view the optimal acidity and sprinkling rate seem to be about 16.1 g  $\text{H}_2\text{SO}_4/\text{l}$  and 4 l/4 kg QPS bed per 24 h (i.e. 1 l/kg of QPS per 24 h), respectively.

*Effect of the QPS bed depth*

The results obtained for the recovery of uranium by leaching 8 and 4 kg QPS beds under the optimal conditions of acidity and of sprinkling rate are presented in Table 4. As shown in the table, on increasing the QPS bed depth the uranium recovery decreases and the acid corresponding consumption increases.

*Effect of nitric acid addition to the lixiviant solution on uranium recovery*

The effect of the replacement of 10% of the  $\text{H}_2\text{SO}_4$  amount used in the leaching process by an equivalent quantity of  $\text{HNO}_3$  was investigated using a leaching solution of total acidity equivalent to 16.1 g  $\text{H}_2\text{SO}_4/\text{l}$ , 8 kg QPS bed and a sprinkling rate of 1 l/kg of QPS per 24 h. On comparing the results

TABLE 2

Uranium recoveries and the corresponding acid consumptions obtained over the dissolution stage using lixiviants of two acidity levels and different sprinkling rates

Factor	Acidity of lixiviant solution							
	16.1 g $\text{H}_2\text{SO}_4/\text{l}$				48.3 g $\text{H}_2\text{SO}_4/\text{l}$			
Rate of effluent (l/ore bed per 24 h)	0.4	1.2	2.0	4.0	0.4	1.2	2.0	4.0
Uranium recovery (%)	35	50	58	68	50	60	68	70
$\text{H}_2\text{SO}_4$ passed (kg/t ore)	60.4	82.8	104.4	130.1	92.4	132.8	167.3	248.4
kg $\text{H}_2\text{SO}_4/\text{kg}$ uranium recovered	271.12	360.0	391.4	416.3	401.7	470.9	529.0	763.1

TABLE 3

Uranium recoveries and the corresponding acid consumptions obtained over the dissolution stage using lixiviants of different acidities and various constant sprinkling rates

Factor	Sprinkling rate (l/4 kg ore bed per 24 h)									
	0.4		1.2		2		4			
Acid strength of the lixiviant (g H <sub>2</sub> SO <sub>4</sub> /l)	16.1	32.2	48.4	16.1	48.3	16.1	32.2	48.3	16.1	48.3
Uranium recovery (%)	35	42	50	50	60	58	63.5	68	68	70
H <sub>2</sub> SO <sub>4</sub> passed through the bed (kg/t ore)	60.4	73.1	92.6	82.8	132.8	104.4	127.1	167.3	130.1	248.4
kg H <sub>2</sub> SO <sub>4</sub> /kg uranium recovered	271.1	317.8	401.7	360	470.9	391.4	430.4	529	416.3	763.1

TABLE 4

Effect of ore bed depth and HNO<sub>3</sub> addition on the uranium recovery and the acid requirements obtained at the end of the dissolution stage using lixiviants of constant total acidity (16.1 g H<sub>2</sub>SO<sub>4</sub>/l) and different flow rates

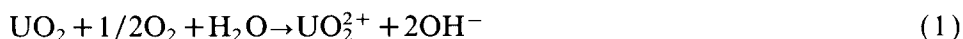
	Acid used			
	H <sub>2</sub> SO <sub>4</sub>		H <sub>2</sub> SO <sub>4</sub> :HNO <sub>3</sub> 9:1 (eq:eq)	
Weight of ore bed (kg)	4	8	8	8
Rate of effluent (l/kg ore/24 h)	1	1	1	0.75
Uranium recovery (%)	68	57	79	76
H <sub>2</sub> SO <sub>4</sub> passed (kg/t ore)	130.1	142.7	129.7	115.3
kg H <sub>2</sub> SO <sub>4</sub> passed/kg uranium recovered	407.7	538.4	355.4	326.2

(Table 4 column 3) with those achieved in the absence of HNO<sub>3</sub> (Table 4 column 2) it is clearly seen that the addition of HNO<sub>3</sub> results in both an increase in the uranium yield and a reduction in the acid consumption. Table 4

(columns 3 and 4) also shows that more acid savings with only slight losses in the uranium recovery can be attained by reducing the sprinkling rate.

## DISCUSSION

In the mineralization under test, uranium is found mainly in the tetravalent state. This state is known to be insoluble in dilute sulphuric acid and must first be oxidized [15]. Oxidation can be accomplished with oxygen of the air:



Molecular oxygen is a relatively inefficient oxidant [16,17], therefore, ferrous iron was used to oxidize the tetravalent uranium as indicated below [18]:

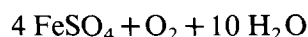
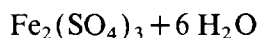


This reaction proceeds rapidly with solutions containing as little as 0.2 g of  $\text{Fe}^{3+}$ /l provided that the ratio of  $\text{Fe}^{3+}/\text{Fe}^{2+}$  in the solution is kept at a sufficiently high value to maintain the redox potential above that of the U(IV)/U(VI) system [19,20]. The acidity does not play any important role in this reaction [21]. Ferric iron has the additional benefit that it can complex the phosphate anion and hence prevent or minimize the precipitating effect of phosphates on dissolved uranium.

As shown in Fig. 1, during the first ten weeks of leaching the pH of the effluent was about 8 and only uranium could be detected. Its concentration was about 13.5 mg/l. Since calculations using the hydrolysis data of U(VI) [22] indicate that uranium at a concentration of 13.5 mg/l starts to precipitate at about pH 6 and is completely precipitated as the hydrous oxide near pH 8, the relatively high uranium concentration (13.5 mg/l) found at about pH 8 cannot be attributed to the reaction of the phosphate minerals with sulphuric acid and the consequent liberation of U(VI). This relatively high uranium concentration should be attributed to the fact that the lixiviant reacts with the carbonate minerals in the QPS and the liberated  $\text{CO}_3^{2-}$  and  $\text{HCO}_3^-$  dissolve U(VI) mostly adsorbed on the surface of the minerals present in the QPS forming the soluble complexes  $\text{UO}_2(\text{CO}_3)_2^{2-}$  and  $\text{UO}_2(\text{CO}_3)_3^{4-}$ . This conclusion is supported by the finding that during the initial 6 month leaching period neither  $\text{Ca}^{2+}$  nor  $\text{P}_2\text{O}_5$  could be detected in the effluents suggesting that the phosphate minerals were not appreciably dissolved.

Iron could not be detected in the solutions leaving the column during the initial 10 months where the pH was above 2. This indicates that precipitation of iron through the QPS bed takes place most probably according to the following reactions:





The sulphuric acid liberated according to the last two equations plays an important role in the leaching process. This conclusion agrees with the observation that when a solution of only sulphuric acid of pH 1.7 was used to leach the QPS [23] under conditions exactly similar to the present ones, the pH of the effluent solution did not fall below about pH 4.5 even after a 16 month leaching period.

At pH values of about 4.5, dissolution of the phosphate minerals starts as shown by the increased concentration of  $\text{P}_2\text{O}_5$  in the effluent (Fig. 1) and hence besides being precipitated according to eqs. (3) and (4), Fe(III) may also be precipitated as phosphate. As shown in the figure, the uranium concentration in the leach liquor is very low, the liquor is severely contaminated with iron and very long leaching periods would be required to attain successful uranium recoveries. Therefore, leaching of the uranium with more acidic solutions was next tried in an attempt to find conditions which can provide rapid uranium recovery but avoid contamination of the effluent with high amounts of iron. In these attempts the method of heap leaching was generally adopted but the sprinkling rate and lixiviant acidity were increased. The results obtained (Figs. 2 and 3) indicate that when the QPS is being treated, the pH of the effluent falls gradually and the calcium and phosphorus contents of the effluent rise rapidly to maxima indicating dissolution of the phosphate minerals of the QPS. This leaching stage may be termed the dissolution stage.

In the presence of phosphate, uranium is precipitated from sulphate media at relatively low pH values and it is, therefore, to be expected that a fraction of the uranium dissolved at the upper layers of the QPS bed would be precipitated as phosphate at lower bed layers by reason of the increasing pH. The precipitated uranium would be expected to redissolve and appear at still lower pH values. Therefore, further spraying of the bed, after the dissolution stage is over, seems to be necessary to redissolve the precipitated uranium and to wash the reactant products out of the column. This stage of leaching may be termed the rinsing stage (Fig. 2).

At the end of the rinsing stage a relatively high uranium recovery can be achieved, but a very large volume of the lixiviant would be required and hence the acid consumption would be extremely high. Therefore, from an economic point of view it becomes advisable to either recycle the low pH effluents obtained at the rinsing stage or to sacrifice the recovery obtained at this stage

and stop the leaching process at the end of the dissolution stage. Another possibility is to apply the calculated acid requirement as a concentrated solution containing say 20%  $\text{H}_2\text{SO}_4$  followed by water or by recycling the dilute effluent. Intermittent leaching may also be profitable. However, the aim of the present work was only to find the best conditions that can lead to maximum uranium recovery and minimum acid consumption during the dissolution stage.

Leaching of uranium during the dissolution stage is mainly influenced by:

(1) factors affecting the rate of dissolution which in turn is mainly affected by the ionic strength;

(2) factors affecting the rate of diffusion of the solution within the rock bed and rock particles; and

(3) the rate of oxidation of  $\text{U(IV)}$  to  $\text{U(VI)}$ .

Obvious, the diffusion rates are controlled by the particle size of the QPS and the rate of sprinkling, while the rate of oxidation of uranium is influenced by the depth and the porosity of the QPS bed and the presence of oxidizing agents.

As shown in Table 4, on increasing the QPS bed depth the acid required to obtain a certain uranium recovery increases. Under identical conditions the pH of the effluent solution was found to be unaffected by the QPS bed depth and hence the reduction in the uranium recovery cannot be attributed to fall in acidity through the bed but may be related to prevailing reducing conditions at the lower parts of the column by reason of little aeration at the lower depths. In support of this conclusion is that analysis indicated that at the end of the dissolution stage uranium was found to be more concentrated at the lower layers of the bed. However, the deleterious effect of the increase of the QPS bed depth could be overcome by replacing 10% of the  $\text{H}_2\text{SO}_4$  used during leaching by an equivalent amount of  $\text{HNO}_3$ . It seems that in addition to increasing the solubility of  $\text{U(IV)}$  by oxidizing it, nitric acid can also minimize the uranium losses in the gypsum residues perhaps by its easier penetration through the gypsum coating.

From the results obtained, the optimal conditions for uranium recovery from the QPS sample under test can be summarized as follows: grind/  $-1/2$  inch ( $-12$  mm) acid/ $\text{HNO}_3/\text{H}_2\text{SO}_4$  mixture 1/9 eq/eq, total acid strength/ equivalent to 16.1 g  $\text{H}_2\text{SO}_4/\text{l}$  and a sprinkling rate of 0.75–1 l/kg of QPS per 24 h. Under these conditions laboratory experiments indicate that about 76–79% of the uranium present in a 4 kg QPS bed of 9.2 cm diameter can be recovered in 9 days (the dissolution stage). The corresponding total acid consumption is equivalent to 115–130 kg  $\text{H}_2\text{SO}_4/\text{t}$  QPS. However, although this recovery is lower than that achieved by agitation leaching (about 93% uranium recovery and about 130 kg  $\text{H}_2\text{SO}_4/\text{t}$  QPS acid consumption [4,23] there are many advantages in favour of the suggested heap leaching procedure. The advantages include:

(1) Clear effluent liquors are produced which are suitable for subsequent treatment. This would eliminate the usual liquid–solid separation step which represents some special difficulties with this particular mineralization.

(2) Grinding costs would be eliminated because only coarse crushing required.

(3) The installations are simple. Consequently the initial capital investment and the operating costs are comparatively low.

(4) The operation is simple, requires only a minimum of skill and can be run in the mine thus eliminating the transportation costs.

(5) The suggested method does not suffer from the very long leaching periods are normally required when the conventional heap leaching method is used.

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