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An upgrading procedure for the uranium concentrate product of Gattar pilot plant via ammonium carbonate

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ABSTRACT

An impure crude uranium concentrate is produced at Gattar pilot plant in which the ore material is processed by acid heap leaching followed by ion-exchange resin. Before proper refining of such a concentrate by the well developed solvent extraction techniques, it was found necessary to apply a prior upgrading procedure to diminish the associated impurities. Ammonium carbonate is used for this purpose and the two relevant factors that would minimize the latter are studied, namely the $(NH_4)_2CO_3$ concentration and the required time for effective settling of the precipitated impurities. Accordingly, it has been possible to increase the uranium assay in the working concentrate from about 36 up to 68% while that of Fe and V for example have been decreased from 1.22 and 1.53 % down to only 0.01and 0.029 % respectively. In refining of the upgraded product by solvent extraction, such impurities and the other remaining elements would be easily decreased to the permissible limits.

KEYWORDS

Uranium;
Ammonium carbonate;
Purification;
Yellow cake;
Precipitation.

INTRODUCTION

One of the most important steps in the manufacture of the nuclear fuel cycle is the uranium refining and conversion which goes from the yellow cake to three different uranium products, namely uranium dioxide (UO_2), natural metallic uranium (U) and uranium tetrafluoride (UF_4). The refining step is indeed a process of minimizing the concentration of the harmful impurities which always defect nuclear fuel fabrication and performance. Uranium refining is currently carried out through two main technological routes. The first route is a dry refining process which employs fractional distillation of impure uranium hexafluoride for the production of reactor grade UF_6[1]. The second refining route is a wet process in which solvent extraction technology is applied to produce nuclear grade products; namely uranium trioxide (orange oxide; UO_3) or else the ammonium uranyl tricarbonate[2]. The latter products would then be converted to UO_2 and metallic uranium. The applied solvents include trialkyl phosphates, trialkyl phosphine oxides, dialkyl phosphoric acid as well as high-molecular weight long-chain tertiary amines.

Uranium precipitated from its bearing solution over a wide pH range, acid or alkaline, depending upon the solution type and precipitant used[3-6]. In the production of yellow cake by precipitation techniques, the most commonly used method was ammonium hydroxide precipitation to form ammonium diuranate[7]. Although the yellow cake produced either by NH_4OH or by MgO/

$Mg(OH)_2$ was of high quality, hydrogen peroxide was used to precipitate uranium as uranium trioxide[8-13]. The pH of the solution, temperature and duration of precipitation as peroxide are of vital importance in the production of pure uranium peroxide[14]. In all the wet refining processes solvent extraction technique are applied using different types of organic solvents with special emphasis on TBP and tertiary amines[15,16].

In Egypt, several promising uranium mineralizations have been discovered in both the Eastern and western Deserts as well as in Sinai and which are either associated with igneous or sedimentary host rocks. Among these, that of Gabal Gattar in North eastern Desert represents an interesting uranium prospect that is mainly hosted in younger granites (G-I to G-XIII) except the GV occurrence which is located in El-Hammamat sedimentary rocks. In this mineralization, uranium is mainly found as secondary minerals which are essentially represented by uranophane and beta uranophane[17,18].

After successful laboratory scale studies for uranium recovery from the G. Gattar mineralized younger granite occurrences, it was decided to construct an experimental pilot plant at the site for testing the studied flowsheet. In this pilot plant, a crude uranium concentrate (yellow cake) is currently produced through acid heap leaching followed by concentration and purification through anion-exchange recovery unit. In the heap leaching of the ore material, uranium is leached by 20-40 g/l H_2SO_4 acid and from the obtained leach liquor uranium is then recovered through its adsorption by a special anion exchange resin. Uranium is afterwards eluted from the loaded resin with 1M NaCl solution acidified with about 0.05M sulfuric acid solution followed by precipitation with NaOH solution as sodium diuranate. However, analysis of the latter has revealed that it is actually a highly impure low grade product ($\approx$ 36 % U) besides the presence of several harmful elements (metals and nonmetals). This low quality concentrate can be attributed to the difficult working conditions as well as to the relatively poor quality of the adopted resin. For example it has been ascertained that vanadium which assays up to the extent of 1.53% in the obtained concentrate is considered a serious highly undesired contaminant. This is due to the fact that it would render the subsequent conversion of the concentrate into uranium metal or other uranium compounds such as the hexa-flouride much more difficult besides exerting a penalty if its content expressed as V_2O_5 exceeds 2 weight percent of the concentrate[19]. Vanadium can actually be removed by addition of ammonium carbonate where it would be converted to the yellow ammonium metavanadate precipitate at a pH value that ranges from 5 to 10; viz:

$$2NaVO_3 + (NH_4)_2CO_3 \rightarrow 2NH_4VO_3 + Na_2CO_3$$

Choice of $(NH_4)_2CO_3$ versus Na_2CO_3 has actually behind its efficiency in precipitating vanadium. In the meantime, metathesis (double replacement reaction) with a concentrated solution of ammonium sulphate can also be occasionally used to lower the sodium content, a matter which allows for partial conversion of the sodium diuranate into ammonium diuranate, viz:

$$Na_2U_2O_7 + (NH_4)_2SO_4 \rightarrow (NH_4)_2U_2O_7 + 2Na^+ + SO_4^{2-}$$

On the other hand, the ferric sulphate, in the leach solution could substantially react with all the phosphate in solution to form ferric phosphate ($FePO_4$) precipitate as well as the applied carbonate to precipitate ferric hydroxide at pH 10 according to the following reaction:

$$Fe_2(SO_4)_3 + 6(NH_4)_2CO_3 + 6H_2O \rightarrow 3(NH_4)_2SO_4 + 6(NH_4)HCO_3 + 2Fe(OH)_3$$

Concerning, the other associated impurities including calcium, magnesium, aluminum, zinc, etc. they could actually be precipitated to a large extent by increasing the pH up to 10 during application of the suggested procedure of ammonium carbonate leaching.

From the above giving, it was found greatly interesting to apply a suggested procedure for upgrading G. Gattar crude yellow cake via its proper treatment with ammonium carbonate. The latter would indeed upgrade the working concentrate to a level suitable for the required final refining and conversion techniques. In this regard, the two relevant factors in this treatment and that would minimize the different associated impurities would involve the $(NH_4)_2CO_3$ concentration and in turn the leach solution pH besides the required time for effective settling of the precipitated impurities. Accordingly, it has been possible to minimize the latter and increase the uranium assay in the obtained uranium concentrate by this treatment from about 36 up to 68%. The present work thus provides indeed a relatively simple and inexpensive process for a considerable decrease of the impurities associated with uranium in the prepared G. Gattar pilot plant crude yellow cake with ammonium carbonate solution.

EXPERIMENTAL

Material characterization

The highly impure working yellow cake (sodium diuranate) which is produced at G. Gattar pilot plant has first been chemically analyzed using both qualitative and quantitative analytical procedures. The latter have involved several analytical wet and instrumental techniques.

For the upgrading procedure suggested in the present work, a proper 1L sulphate solution has been prepared by dissolution of 10 g of the crude concentrate in 30 g/l sulphuric acid solution. During dissolution the gross amount of silica and other insolubles have been removed as insoluble residue which was then filtered and washed until having 1 litre clear solution of the working crude uranium concentrate. The latter has also been subjected to complete chemical analysis.

$$\mathrm{Na_2U_2O_7 + 3H_2SO_4 \rightarrow 2UO_2SO_4 + Na_2SO_4 + 3H_2O}$$

$$\mathrm{UO_2SO_4 + SO_4^{2-} \rightarrow UO_2(SO_4)_2^{2-}}$$

$$\mathrm{UO_2(SO_4)_2^{2-} + SO_4^{2-} \rightarrow UO_2(SO_4)_3^{4-}}$$

Experimental procedures

Several upgrading experiments were performed to optimize the mentioned two relevant factors. In these experiments, 25 ml samples of the prepared crude uranium concentrate solution were progressively treated with an increasingly amount of $\mathrm{(NH_4)_2CO_3}$ ranging from 0.375 up to 4.125 gm (equivalent pH 3 to 10). During these experiments, the different impurities would gradually precipitate while uranium would also gradually precipitate as $\mathrm{(NH_4)_2U_2O_7}$ until a pH of about 7 behind which it would selectively dissolve again in the form of the uranyl carbonate complex; viz:

$$2Na_4UO_2(SO_4)_3 + 3(NH_4)_2CO_3 \xrightarrow{pH\,7} (NH_4)_2U_2O_7 + 4Na_2SO_4 + 2(NH_4)_2SO_4 + 3CO_2 \uparrow$$

$$(NH_4)_2U_2O_7 + 6(NH_4)_2CO_3 + 3H_2O \xrightarrow{pH\,>7} 2(NH_4)_4UO_2(CO_3)_3 + 6NH_4OH$$

In each experiment, the obtained precipitate was left to settle down after which the slurry was subjected to filtration and the precipitate was thoroughly washed while the obtained filtrate and washing were completed to a known volume before being directed for the analysis of both uranium and impurities. On the other hand, the final filtrate obtained at pH 10 has then been subjected to adjust its pH to 12 using 30% sodium hydroxide to form the upgraded sodium diuranate precipitate as follows:

$$\mathrm{2(NH_4)_4UO_2(CO_3)_3 + 14NaOH \xrightarrow{pH\,12} Na_2U_2O_7 + 6Na_2CO_3 + 8NH_4OH + 3H_2O}$$

Analytical procedures

Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) and X-ray fluorescence techniques were used to qualitatively identify the composition of both the working uranium ore concentrate and the obtained purified yellow cake (sodium diuranate). However, the quantitative analysis was then used for the determination of the impurities like Si, Al, P and Ni using Unicam UV2-100 UV/Vis Spectrometer according to standard methods of analysis[20]. The concentrations of the other elements have been determined through the ICP-OES technique using the proper standards.

Concerning the uranium concentration whether in the crude or purified yellow cake as well as in the increasingly purified solutions have all the time being measured by the ICP-OES technique and confirmed by an oxidimetric titration using ammonium metavanadate[21].

Chemicals and reagents

Sulfuric acid (98%, prolabo), ammonia solution (25-30% prolabo), sodium hydroxide (99%, prolabo), ammonium carbonate (99%, E-Merck purified product), yellow cake (sodium diuranate) from G. Gattar plant and other analytical reagents are used. All chemicals used are of analytical reagent grades.

RESULTS AND DISCUSSION

Material characteristics

As mentioned above the working G. Gattar pilot plant crude uranium concentrate has completely been analyzed using the mentioned procedures and the obtained results are shown in TABLE 1. From these results it is clearly evident that besides Ca (2.12%) and Fe (1.22%), both V and REEs (Ce, Sm, Tb, Ho and Tm) assay up to 1.53 and 1.09%.

On the other hand, the prepared solution of the working crude uranium concentrate (pH 0.85) has also been completely analyzed and the obtained results are given in the first column of TABLE 3.

TABLE 1 : Chemical analysis of uranium and associated major impurities in G.Gatter pilot plant crude yellow cake (sodium diuranate)

Element	Wt. %	Element	Wt. %
U (%)	35.8000	Na	4.2794
Ag	0.0668	Ni	0.0266
Al	0.0886	P	0.0742
Ca	2.1222	Si	0.0120
Cd	0.0046	Sr	0.0490
Co	0.0086	V	1.5270
Cr	0.0094	Zn	0.0095
Cs	0.0320	Ce	0.0202
Cu	0.0164	Sm	0.6708
Fe	1.2234	Tb	0.1888
K	0.2612	Ho	0.1080
Mg	0.4377	Tm	0.1024
Mn	0.0174		

Results of the upgrading procedure

Given the highly impurities of G. Gattar pilot plant uranium concentrate (sodium diuranate) as shown in TABLE 2 in comparison with Swedish products[22], it was decided to subject the former to an efficient upgrading procedures.

Choice of $(NH_4)_2CO_3$ as an upgrading means for the former has actually been based upon economic reasons. Two relevant factors have indeed been chosen; namely $(NH_4)_2CO_3$ amount and in turn the pH besides the time allowed for proper settling of the precipitated impurities.

(a) Effect of ammonium carbonate amount/pH

A series of 25 ml samples of the prepared clarified solution (pH 0.85) has been used to study the influence of the progressive addition of equal quantities of ammonium carbonate powder (0.375 g) and in turn the progressive increase of the pH of each solution. The studied sample solutions were left each time for one

TABLE 2 : Chemical composition of G.Gattar pilot plant crude yellow cake (sodium diuranate) compared to swedish products (sodium diuranate)

Element	Crude yellow cake as sodium diuranate in G.Gattar pilot plant (%)	Production period (1965-1968) Sodium diuranate[22] (%)	Pilot plant Sodium diuranate[22] (%)
U	35.80	72.00	70.70
Ag	0.0668	-	-
Al	0.0886	-	-
As	-	0.001 0	<0.000 5
B	-	0.000 05	0.000 05
Br	-	<0.000 5	<0.000 5
Ca	2.1222	0.15	0.03
Cd	0.0046	-	-
Cl	-	0.0005	0.0005
Co	0.0086	-	-
Cr	0.0094	-	-
Cs	0.0320	-	-
Cu	0.0164	-	-
F	-	0.000 7	<0.000 5
Fe	1.2234	0.05	0.02
I	-	<0.005	<0.001
K	0.2612	0.03	0.01
Mg	0.4377	-	-
Mn	0.0174	-	-
Mo	-	0.015	0.03
Na	4.2794	7.4	7.6
Ni	0.0266	-	-
P	0.0742	0.010	0.12
S	-	0.03	0.10
Si	0.0120	0.02	0.02
Sr	0.0490	-	-
Th	-	<0.005	<0.005
V	1.5270	<0.000 5	<0.005
Zn	0.0095	-	-
Ce	0.0202	-	-
Sm	0.6708	-	-
Tb	0.1888	-	-
Ho	0.1080	-	-
Tm	0.1024	-	-

(-) not detected

day to ensure complete settling down of the obtained precipitates before their filtration and proper washing. All the obtained solutions were then subjected to the analysis of both uranium and the impurities that have not been precipitated and the obtained results are shown in TABLE 3.

TABLE 3 : Analytical results and pH values of uranium (g/l) and impurities (mg/l) in the prepared crude uranium cake solutions after progressive treatment with ammonium carbonate

Amount NH_4CO_3 (g)	0	0.375	0.750	1.125	1.500	1.875	2.250	2.625	3.000	3.375	3.750	4.125
pH	**0.85**	**3**	**4**	**4.5**	**6**	**6.6**	**7.3**	**8**	**8.7**	**9**	**10**	**10**
Element												
U (g/l)	3.58	3.34	3.15	3.12	2.75	0.008	0.001	0.009	1.87	3.44	3.56	3.56
Ag	668	661	665	661	663	654	662	660	661	662	660	660
Al	886	870	856	845	800	810	781	777	712	432	321	321
Ca	21222	20276	19873	18932	17876	15235	13651	11876	9362	3517	910	910
Cd	46	45	45	43	43	37	35	30	29	25	22	22
Co	86	80	78	77	75	76	75	71	73	70	71	71
Cr	94	83	79	73	71	70	69	67	55	50	40	40
Cs	320	315	300	299	286	277	265	259	251	195	165	165
Cu	164	155	144	142	140	137	134	133	120	115	111	111
Fe	12234	11244	10677	9781	8563	7673	6925	5452	1342	652	160	160
K	2612	2563	2427	2364	1986	1854	1813	1767	1754	1674	1612	1612
Mg	4377	3751	3522	3628	2970	2457	2142	1934	1851	1833	1802	1802
Mn	174	155	152	141	134	125	119	108	97	88	82	82
Na	42794	40235	3809	3608	28951	18515	15946	13086	9100	6321	5202	5202
Ni	266	260	256	247	236	242	231	199	156	119	104	104
P	742	700	688	665	642	632	624	420	333	212	124	124
Si	120	70	68	67	70	50	52	64	51	51	51	51
Sr	490	447	423	388	367	361	251	252	201	182	188	188
V	15270	13112	12234	11732	10162	9025	6861	4996	3432	1012	979	979
Zn	95	90	88	85	82	74	70	66	49	36	21	21
Ce	202	192	169	162	156	145	130	100	90	50	26	26
Sm	6708	6018	5813	5409	5215	4202	3937	2781	2256	1450	1050	1050
Tb	1888	1810	1665	1525	1451	1378	1266	1165	966	499	267	267
Ho	1080	992	834	731	635	567	467	356	305	192	129	129
Tm	1024	980	928	864	768	674	559	459	348	189	112	112

From the obtained results, it is clearly evident that beside of increasing the pH value, the concentration of most of the impurities in the working crude uranium cake solutions has decreased with increasing the amount of ammonium carbonate. The minimum concentration of these impurities has actually been obtained after addition of up to 3.75 g of the ammonium carbonate powder and where the pH has amounted to 10. Further addition of $(NH_4)_2CO_3$ has not affected the pH which remained at 10 and also there has been no further precipitation of impurities. It is interesting in this regard to indicate that uranium has almost been completely precipitated at pH 7.3 and started to re-dissolve at pH 8 until pH 9 where it has almost been completely re-dissolved at pH 9. Concerning increasing the latter to pH 10 has however led to further precipitation of Ca from 3.52 down to 0.91 g/l, of Fe from 652 to 160 ppm, of P from 212 to only 124 ppm and of REEs from 2.38 to 1.58 g/l. It can thus be concluded that under the working conditions, 1 L of the prepared sulphate solution of G. Gattar pilot plant crude uranium concentrate (3.58 g U/l) would require 150 g $(NH_4)_2CO_3$ to increase its pH to 10 and where an adequate decrease in the assay of the associated impurities could be realized. Further addition of 15 g $(NH_4)_2CO_3$ has neither increased the pH nor realized any decrease of the uranium associated impurities.

(b) Effect of the settling time of the precipitates

For studying the effect of settling time required for efficient collection of precipitated impurities from the prepared solution of the working crude yellow cake, the pH of a series of 25 ml samples therefore was adjusted at pH 10 using 3.750 g ammonium carbonate. The obtained precipitates were then left to settle down for different time period ranging from only 1 hr up to 3 days. After proper filtration and washing of the precipitated impurities the obtained filtrates were then analyzed for both the uranium and the still dissolved impurities in the filtrate using ICP-OES and the other required techniques and the results are shown in TABLE 4.

TABLE 4: Effect of settle time upon the separation efficiency of the impurities precipitated at pH 10 upon the assay of U (g/l) and the associated impurities (mg/l) of G.Gattar pilot plant purified uranium concentrate solution

Time	0	1 hr	4 hrs	1 day	2 days	3 days
Element						
U (g/l)	3.58	2.56	2.97	3.57	3.56	3.56
Ag	668	660	660	660	660	660
Al	886	350	341	322	322	322
Ca	21222	1200	1076	912	912	912
Cd	46	40	34	22	22	22
Co	86	82	81	71	71	71
Cr	94	60	53	41	41	41
Cs	320	202	188	167	167	167
Cu	164	121	119	109	110	110
Fe	12234	259	187	160	160	160
K	2612	2613	2022	1612	1612	1612
Mg	4377	2132	1970	1801	1801	1801
Mn	174	109	102	82	82	82
Na	42794	15202	15032	5202	5202	5202
Ni	266	124	114	104	104	104
P	742	162	141	123	123	123
Si	120	60	52	50	51	50
Sr	490	213	216	188	188	188
V	15270	1329	1174	979	979	979
Zn	95	45	40	21	21	21
Ce	202	67	43	26	26	26
Sm	6708	1955	1765	1050	1050	1050
Tb	1888	365	347	267	267	267
Ho	1080	444	388	129	129	129
Tm	1024	365	327	112	112	112

From the obtained data, it is clearly obvious that the concentration of most of the impurities has gradually decreased to their minimum values with increasing the settling time of the obtained precipitates from 1hr to 1day. Thereafter, the concentration of both uranium and associated impurities has almost been kept constant up to 3 days. Therefore, it can be concluded that one day is quite enough for complete settling of the precipitated impurities.

Sodic decomposition

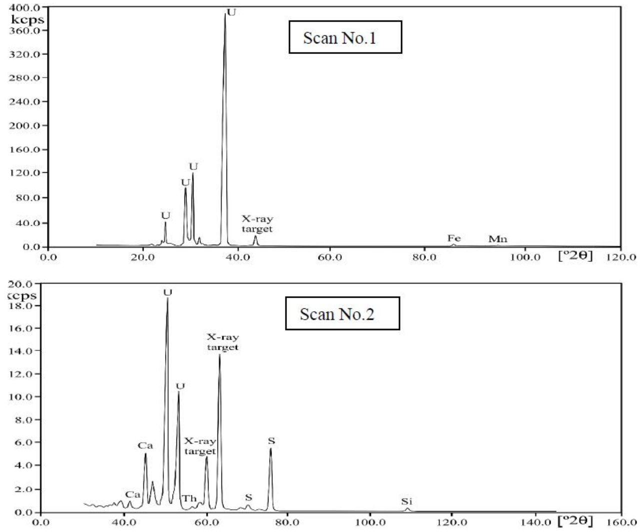
A sample weighing, 20 g of G. Gattar pilot plant crude uranium concentrate was suspended in 100 ml of distilled water followed by adding 50 g of sulphuric acid

TABLE 5: Chemical composition of the upgraded purified uranium concentrate in comparison with G.Gattar pilot plant crude concentrate

Element	Purified yellow cake (%)	Crude yellow cake (%)
U	67.96	35.8
Ag	0.0131	0.0668
Al	0.0120	0.0886
Ca	0.0800	2.1222
Cd	0.0020	0.0046
Co	0.0054	0.086
Cr	0.0030	0.094
Cs	0.0111	0.0320
Cu	0.0078	0.0164
Fe	0.0100	1.2234
K	0.00320	0.2612
Mg	0.0812	0.4377
Mn	0.0081	0.0174
Na	6.3678	4.2794
Ni	0.0066	0.0266
P	0.0012	0.0742
Si	0.0030	0.0120
Sr	0.0132	0.0490
V	0.0288	1.5270
Zn	0.0021	0.0095
Ce	0.0031	0.0202
Sm	0.1060	0.6708
Tb	0.0272	0.1888
Ho	0.0131	0.1080
Tm	0.0113	0.1024

(98%) and the volume was then completed to 1 litre with distilled water. The insoluble matter of the latter was then filtered to clarify the turbid solution. Afterwards; the insoluble matter has been dried and its weight was found to attain 1.5 g. To the obtained clarified solution 150 g of ammonium carbonate was added to adjust its pH up to 10. From the prepared ammonium uranyl tricarbonate solution most of the associated impurities have been precipitated and which were filtered off after being left for one day for efficient settling down. The latter was then dried at 105 °C and where its weight was found to attain up to 8.5 g.

Recovery of uranium from the ammonium uranyl tricarbonate filtrate was achieved through sodic decomposition by the addition of 30 % sodium hydroxide solution where uranium in the form of pure sodium diuranate has been precipitated at pH 12. After proper drying of the latter, its weight has attained 9.55 g. To determine the uranium associated impurities, the obtained purified cake was subjected to qualitative analysis using X-ray fluorescence technique as shown in Figure 1. On the other hand, a complete quantitative analysis by ICP-OES has been performed for both uranium and the associated impurities in comparison with the G. Gattar pilot plant crude uranium concentrate (TABLE 5). From the obtained results, it can be concluded that the precipitated $Na_2U_2O_7$ attains a purity of 90.5%. On the other hand, it is clearly evident that a noticeable drop in the co-associated impurities has indeed been obtained.



Condition of scan No. 1 Rh-target tube, 70 kV, 15 mA, LiF-220 analyzing crystal; Condition of scan No. 2 Rh-target tube, 30 kV, 60 mA, PE-001 analyzing crystal

Figure 1: X-ray fluorescence qualitative analysis of the prepared purified yellow cake from the crude concentrate of G. Gattar pilot plant by two scan

A proposed flowsheet for the upgrading of G. Gattar pilot plant crude uranium concentrate using ammonium carbonate has been formulated and is shown in Figure 2.

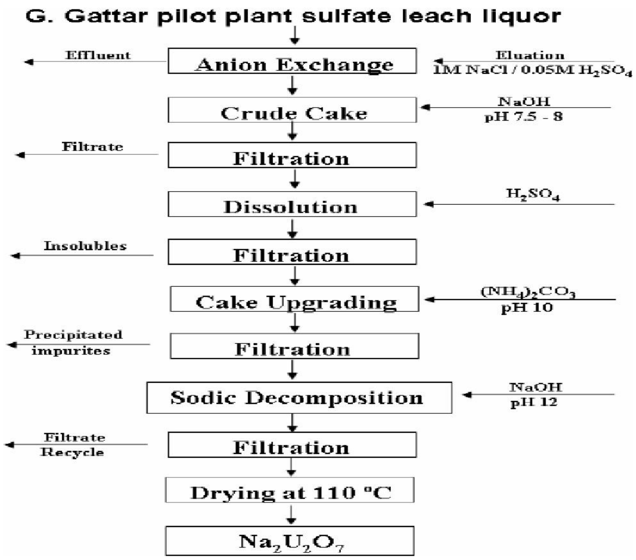


Figure 2 : Formulated flowsheet for upgrading G. Gattar crude uranium concentrate into highly purified sodium diuranate

CONCLUSION

An upgrading procedure using ammonium carbonate is presented for the highly impure G. Gattar pilot plant sodium diuranate product (35.8 % U). The two relevant factors studied involved adjustment of the sulphate solution of crude cake by ammonium carbonate at pH 10 and allowing a settling time of one day for efficient precipitation of the impurities.

A highly purified final product of sodium diuranate with 90.5 % purity and assaying up to 67.96 % U has been obtained by sodic decomposition of the prepared highly pure ammonium uranyl tricarbonate solution at pH 12. Using ICP-OES for analysis of the purified product reveals a high purity product of quite low amounts of associated impurities. A flowsheet summarizing the worked procedure has been formulated.

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