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URANIUM RECOVERY PROCESS

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This invention relates to a process of recovering uranium from very low grade uranium containing ores and ore residues, and more particularly it relates to the use of sodium carbonate in the selective extraction of uranium from the low grade uranium containing hydroxide precipitates which are obtained by alkali neutralization of sulfuric acid leach liquors derived from the aforesaid low grade uranium containing ores and ore residues followed by precipitation of uranium from the sodium carbonate extracts derived from the low grade uranium containing hydroxide precipitates. These extraction and precipitation steps serve to upgrade the low grade uranium containing hydroxide precipitates and to convert them into high grade uranium concentrates.

The very low grade uranium containing ores and ore residues with which this invention is concerned contain silicates and aluminum and iron containing minerals, in addition to minute amounts of uranium and therefore the sulfuric acid leach liquors derived from these ores and ore residues contain large amounts of dissolved silica, iron and aluminum in addition to uranium. The addition of an alkali such as caustic calcined magnesia or dolomite to these sulfuric acid leach liquors, therefore, results in the formation of precipitates which contain large amounts of silica, iron, and aluminum, in addition to a small amount of uranium. This leads to the problem of upgrading these precipitates in order to eliminate the silica, aluminum, and iron, and thereby to secure high grade uranium concentrates or solutions.

This invention has as an object to provide a process for obtaining concentrated uranium containing solutions from very low grade uranium containing ores and ore residues. A further object is to provide a process for upgrading the low grade uranium containing hydroxide precipitates which are obtained by alkali neutralization of sulfuric acid leach liquors derived from the aforesaid low grade uranium containing ores and ore residues. A still further object is to provide a process for selectively extracting uranium from these low grade uranium containing hydroxide precipitates. Other objects will appear hereinafter.

These objects are accomplished by the following invention in accordance with which low grade uranium containing hydroxide precipitates which also contain large amounts of hydrated silica and iron and aluminum hydroxides are given a preliminary treatment with water and then subjected to multiple leachings with aqueous solutions of sodium carbonate at a pH of at least 9. This leaching serves to selectively extract the uranium from the precipitate, but to leave the greater part of the silica, iron, and aluminum with the solid leached residue. The uranium is then precipitated from the leach liquor by the addition of an acid to expel CO_2 followed by the addition of ammonia to precipitate uranium. These extraction and precipitation steps constitute the most important features of applicants' novel process of upgrading low grade uranium containing hydroxide precipitates to obtain a high grade uranium concentrate therefrom.

This invention is generally applicable for the upgrading of low grade uranium containing hydroxide precipitates derived from a wide variety of very low grade uranium containing ores, and has been found to be especially useful for selectively upgrading the uranium content of low grade hydroxide precipitates which are derived from sulfuric acid leach liquors obtained from ores that assay less than 0.2% in U_3O_8. The ores to which this invention is particularly applicable, and on which the greater part of the experimental work described in this specification has been performed, are all mineralogically similar being composed of quartz pebbles surrounded by a quartzitic matrix in which the valuable minerals occur. The ores typically contain about 10 percent of foliated silicate minerals, which may be either sericite or pyrophyllite or both. In addition they contain pyrite, uraninite, carbonaceous material, gold, and very small quantities of heavy minerals such as ilmenite, rutile, chromite, magnetite, zircon, and tourmaline. The pyrite content of these ores varies from about 1 percent to about 3 percent. The pyrite is rich in gold, but does not have much uraninite associated with it. The carbonaceous material in these ores is a fragile hydrocarbon mineral of low specific gravity which is rich in both gold and uraninite. Uraninite is the only uranium mineral of importance in these ores. It occurs free and as locked particles with other mineral constituents of the ores, especially hydrocarbon. The gold content of these ores may vary from about 0.005 oz. to about 3.4 oz. per ton. The U_3O_8 content of these ores may vary from about 0.001% to about 0.17% depending upon richness of vein and selectivity of mining.

Instead of processing the ores as they are mined and crushed, it is frequently preferred to apply the invention to ore residues which remain after the ores have been leached with a cyanide solution to extract the gold from the ore. These cyanided residues frequently contain about 1% of metallic iron, weathered aluminum silicates, and some lime derived from the cyanide leaching in addition to the minerals mentioned in the preceding paragraph. These cyanided ore residues generally assay from 0.015% to 0.030% in U_3O_8, from 2% to 3% in Fe, from 85% to 88% in SiO_2, from 7% to 9% in Al_2O_3, from 1% to 4% in CaO, from 1% to 2% in MgO.

This invention is generally applicable for upgrading the low grade uranium containing hydroxide precipitates which are made by treating sulfuric acid leach liquors of the previously described ores and ore residues with a precipitating agent, such as caustic calcined magnesia or caustic calcined dolomite. These precipitates usually analyze from 1% to 10% in U_3O_8, from 1% to 15% in Fe, from 10% to 30% in Al_2O_3, from 5% to 30% in SiO_2, from 5% to 20% in MgO, and from 5% to 20% in CaO.

The invention is illustrated but not limited by the following examples.

Example 1

A very low grade uranium containing ore residue of the type described above from which gold had been removed by cyanide leaching and which assayed 0.020% in U_3O_8, 2.69% in Fe, 85.5% in SiO_2, 7.4% in Al_2O_3, 1.9% in CaO and 1.73% in MgO was diluted with an equal weight of water. To the ore pulp there was added 5 pounds of MnO_2 and 40 pounds of H_2SO_4 (sp. gr. 1.84) per ton of ore residue. After the addition of these reagents the leaching reactions were allowed to continue for about 20 hours. The leach liquor was separated from the leached ore residue by filtration, and partially neutralized to a pH of about 3.5 by being agitated with another batch of ore residue which was to be subsequently leached with sulfuric acid. As a result of the leach liquor

being partially neutralized to a pH of about 3.5, the ferric iron dissolved therein precipitated upon the second batch of ore residue. The leach liquor was separated from the second batch of ore residue by filtration, acidified, and oxidized with sufficient MnO_2 to convert the ferrous iron dissolved therein to the ferric state. The oxidized leach liquor was partially neutralized with caustic calcined magnesia to a pH of about 2.7, and the ferric iron which precipitated as a result of this partial neutralization step was removed by filtration. The filtered and oxidized leach liquor was then further neutralized to a pH of 7 by the addition of an aqueous slurry of caustic calcined magnesia. This latter neutralization step resulted in the precipitation of an aluminum, uranium, and silicon containing precipitate which after drying assayed approximately 4% in U_3O_8, 2% in Fe, 30% in SiO_2, 30% in Al_2O_3, 5% in CaO, 20% in MgO and 1% in Mn.

A wet filter cake which analyzed 0.686% in U_3O_8 and contained about 20% of solids having the dry assay given in the last sentence of the preceding paragraph was pulped with an equal weight of water, and to this pulp there was added 57 grams of Na_2CO_3 for each kilogram of wet filter cake being treated. The wet pulp with the added Na_2CO_3 was heated at 85–95° C. for 5 minutes and then filtered and washed. For each gram of the wet filter cake there was obtained 4.40 milliliters of a leach solution containing 0.79 gram of U_3O_8 per liter. This leach solution contained 51% of the uranium values that were originally present in the filter cake that was being treated. The filter cake that remained after this first leaching with Na_2CO_3 was repulped with an equal weight of water and to this pulp there was added the same amount of Na_2CO_3 that had been used in the first Na_2CO_3 treatment step. The pulp with the added Na_2CO_3 was heated for 10 minutes at 85–95° C. and then filtered and washed. For each gram of the original wet filter cake that was treated there was obtained 3.93 ml. of leach solution containing 0.57 g. of U_3O_8 per liter. This second leach solution contained 32% of the uranium values that were originally present in the filter cake that was being treated. The two sodium carbonate extractions together removed 83% of the uranium values that were originally present in the filter cake that was being treated.

Two other batches of this same wet filter cake having the dry assay set forth in the last sentence of the first paragraph of this example were subjected to a two step sodium carbonate leaching process substantially as indicated in the second paragraph of this example with the exception that the initial treatments of the filter cake with Na_2CO_3 at 85–95° C. were carried on for 10 minutes and 20 minutes respectively instead of for 5 minutes. In the case of the batch of filter cake which was initially treated with Na_2CO_3 for 10 minutes, 44% of the uranium values were removed from the filter cake in the first extraction step and 38% of the uranium values were removed in the second extraction step to give a total extraction of 82%. In the case of the batch of filter cake which was initially treated with Na_2CO_3 for 20 minutes, 32% of the uranium values were removed from the filter cake in the first extraction step and 46% of the uranium values were removed in the second extraction step to give a total extraction of 78%. These additional results show that only low, inconsistent, and non-reproducible results can be obtained in attempting to extract uranium from these filter cakes by a one step Na_2CO_3 extraction process whereas when the extraction process is carried out in two steps the total amount of uranium extracted from the filter cake becomes fairly consistent, and fairly reproducible extractions can be obtained. Increasing the initial extraction time from 5 minutes to 20 minutes appears not to cause any appreciable variation in the total amount of uranium extracted in a two step Na_2CO_3 extraction process.

A fourth batch of this same wet filter cake having the dry assay set forth in the last sentence of the first paragraph of this example was digested with hot (85–95° C.) water for 10 minutes. The water, which contained substantially no uranium, was filtered off. Then the water leached filter cake was subjected to a two step sodium carbonate leaching process substantially as indicated in the second paragraph of this example with the exception that the initial treatment of the filter cake with Na_2CO_3 at 85–95° C. was carried on for 40 minutes instead of 5 minutes. As a result of the first Na_2CO_3 extraction step 74% of the uranium values were removed from the water leached filter cake, while 18% of the uranium values were removed from the filter cake in the second Na_2CO_3 extraction step to give a total extraction of 92%. This experiment showed that a previous water leach greatly enhanced the recovery of uranium from a uranium containing precipitate by a two step sodium carbonate extraction process.

Example 2

A wet filter cake was obtained in the manner set forth in the first paragraph of Example 1. This precipitate after drying assayed approximately 5.5% in U_3O_8, 2% in Fe, 30% in SiO_2, 30% in Al_2O_3, 5% in CaO, 20% in MgO, and 1% in Mn. This wet filter cake contained about 20% of solids and in its wet state it analyzed 1.13% in U_3O_8.

A 35 gram charge of this wet filter cake was diluted with 105 grams of water and agitated therewith at 85–90° C. for one hour. The water, which contained no significant amount of uranium, was filtered off. The wet filter cake was diluted with an equal weight of water, 2 grams of Na_2CO_3 (57 grams of Na_2CO_3 per kilogram of wet cake) was added, and the filter cake was digested with the added Na_2CO_3 for 30 minutes at 85–95° C. For each gram of the wet filter cake employed at the beginning of the run there was obtained 4.59 ml. of a leach solution which analyzed 1.83 grams of U_3O_8 per liter. This leach solution contained 73.7% of the uranium values that were originally present in the filter cake that was being treated. Two additional digestions of this filter cake with two additional 2 gram portions of Na_2CO_3 were carried out just like the first digestion for 30 minutes at 85–95° C. These two additional digestions raised the total extraction of uranium values from the starting filter cake to 95.7%. During these extractions the pH of the leach liquor ranged between 9.0 and 10.6. The residue left after the completion of the three Na_2CO_3 extractions assayed 0.35% in U_3O_8 after being dried.

Another 35 gram charge of this same wet filter cake was diluted with 105 grams of water and agitated therewith at 85–95° C. for one hour. The water, which contained no significant amount of uranium, was filtered off. The wet filter cake was diluted with an equal weight of water, 2 grams of Na_2CO_3 was added, and the filter cake was digested with the added Na_2CO_3 for 24 hours at 20–30° C. For each gram of the wet filter cake employed at the beginning of the run, there was obtained 3.84 ml. of a leach solution which analyzed 1.1 grams of U_3O_8 per liter. This leach solution contained 55.7% of the uranium values that were originally present in the filter cake that was being treated. Two additional digestions of this filter cake with two additional 2 gram portions of Na_2CO_3 were carried out just like the first digestion for 24 hours at 20–30° C. These two additional digestions raised the total extraction of uranium values from the starting filter cake to 95.4%. The residue left after the completion of the three Na_2CO_3 extractions assayed 0.235% in U_3O_8 after being dried.

A comparison of the results given in the two preceding paragraphs of this example shows that the total extractions obtained with three 24 hour cold (20–30° C.) leaches and with three 30 minute hot (85–95° C.) leaches are comparable. However, it has been found that in the cold leaching tests the extraction during the first two leaches was lower than in the corresponding hot leaches.

The low grade uranium containing precipitates which were made in accordance with the general procedure outlined in the first paragraph of Example 1 wherein caustic calcined magnesia was used as the reagent to cause the precipitation both of the ferric iron precipitate and the aluminum, uranium and silicon containing precipitate from the oxidised leach liquor generally analyzed from 4% to 6% in U_3O_8 on a dry basis. When calcined dolomite was substituted for calcined magnesia only for causing the ferric iron to precipitate but calcined magnesia was used to bring down the aluminum, uranium, and silicon containing precipitate, the U_3O_8 content of the latter precipitate on a dry basis still ranged from 4% to 6%. However, if calcined dolomite was used as a precipitant both to bring down the ferric iron precipitate and the aluminum, uranium, and silicon containing precipitate, then the uranium content of the latter precipitate was decreased because the latter precipitate contained a large amount of calcium sulfate. The uranium analysis of an aluminum, silicon, and uranium containing precipitate obtained using calcined dolomite as a precipitant generally varied from 2% to 3.5% in U_3O_8 on a dry basis. These uranium containing precipitates obtained by the use of calcined dolomite as a precipitant have also been successfully processed in accordance with the present invention to extract the uranium therefrom by a preliminary hot water treatment followed by three or four Na_2CO_3 leaches. However, these low grade uranium containing precipitates obtained by the use of calcined dolomite have not had their uranium as economically extracted by means of sodium carbonate as the precipitates obtained using calcined magnesia as a precipitant for uranium. The uranium containing precipitates obtained using calcined dolomite as a precipitant required an increased consumption of sodium carbonate in order to secure a uranium extraction comparable to that obtained from uranium containing precipitates obtained using calcined magnesia as a precipitant. It was found that it was often necessary to use NaOH along with Na_2CO_3 in the first Na_2CO_3 leach of these low grade uranium containing precipitates obtained with the use of calcined dolomite as a precipitant in order to keep the pH of the leach liquor above 9, which is essential for good leaching of uranium.

Low grade uranium containing precipitates having a higher iron assay than the precipitate mentioned in the last sentence of the first paragraph of Example 1 were made by following the procedure set forth in said paragraph but omitting the steps of oxidizing the filtered leach liquor with MnO_2 and of partially neutralizing the oxidized leach liquor to a pH of about 2.7 with caustic calcined magnesia to obtain a ferric iron precipitate. A typical precipitate obtained in accordance with this simplified procedure analyzed when dry 1.62% in U_3O_8, 9.1% in Fe, 6.3% in SiO_2, 17.1% in Al_2O_3, 16.7% in CaO, 13.1% in MgO, and 1.14% in Mn. Uranium was readily extracted from precipitates having an analysis similar to that set forth in the preceding sentence by a preliminary water digestion followed by several Na_2CO_3 leachings. These precipitates, which contained more iron than the precipitates mentioned in Examples 1 and 2, required the use of more Na_2CO_3 in order to secure a comparable uranium extraction. In processing these precipitates, it was also often necessary to add NaOH to the first Na_2CO_3 leach liquor so that this leach liquor would have a pH above 9.

Example 3

A very low grade uranium containing ore residue of the type described above from which gold had been removed by cyanide leaching and which assayed 0.022% in U_3O_8, 2.4% in Fe, 85.4% in SiO_2, 7.7% in Al_2O_3, 0.6% in CaO, and 1.02% in MgO was pulped with water to form a slurry having a 60% solids content. To this slurry there was added 50 pounds of H_2SO_4 (sp. gr. 1.84) and 15 pounds of MnO_2 per ton of ore residue. The slurry was agitated for 16 hours with these added leaching reagents. The amount of MnO_2 used for leaching was sufficient to convert all iron dissolved from the ore residue to the ferric state. The amount of H_2SO_4 employed for leaching was sufficient to give the leach slurry a pH at least as low as 1.5. After 16 hours of leaching sufficient limestone was added to the leach slurry to cause it to attain a pH of 3.5. The leach slurry with the added limestone was agitated for about 4 hours. This partial neutralization of the leach slurry with limestone to a pH of 3.5 caused the ferric iron which was dissolved in the leach liquor to precipitate upon the leach residue. The leach liquor was then separated from the leach residue by filtration. Caustic calcined dolomite was added to the filtered leach liquor until it attained a pH of 6.2. This addition of calcined dolomite caused the formation of a precipitate which on a dry basis analyzed 6.55% in U_3O_8, 17.4% in Al_2O_3, 1.24% in Fe, 11.5% in SiO_2, 11.5% in CaO, 2.8% in Mn, 21.6% in SO_4 and 3% in MgO.

One kilogram of a wet precipitate containing about 20% of solids having the dry analysis indicated in the preceding sentence and containing 1.28% of U_3O_8 on a wet basis was diluted with 6.04 liters of water. This slurry was held at 85–90° C. for one hour with constant agitation. The slurry was filtered, and the filtrate therefrom was found to contain only 0.1% of the uranium that was present in the wet precipitate which was being treated. The water treated precipitate was converted to a 5% solids slurry by the addition of water thereto, and 65 grams of Na_2CO_3 was added to the slurry. The slurry with the added Na_2CO_3 was heated for 30 minutes at 85–90° C. and then filtered. 3.48 liters of extract liquor were thus obtained which contained 3.39 grams of U_3O_8 per liter. This first carbonate extract liquor contained 85.6% of the uranium values that were originally present in the precipitate that was treated. The precipitate was again converted to a 5% solids slurry by the addition of water thereto and 65 grams of Na_2CO_3 was added to the slurry. The slurry with the added Na_2CO_3 was heated for 30 minutes at 85–90° C. and then filtered. 4.32 liters of extract liquor were thus obtained which contained 0.35 gram of U_3O_8 per liter. This second carbonate extract liquor contained 10.9% of the uranium values that were originally present in the precipitate that was treated. The two carbonate extract liquors together contained 96.5% of the uranium values that were originally present in the precipitate that was treated. The residue left after the completion of the two Na_2CO_3 extractions assayed 0.45% in U_3O_8. In accordance with the procedure set forth in this example 9.5 grams of Na_2CO_3 are used per gram of U_3O_8 extracted from the wet precipitate.

It has been found that if the wet low grade uranium containing filter cakes are agitated for about one hour with cold (20–30° C.) or hot (85–95° C.) water the amount of uranium extracted therefrom in two or three subsequent Na_2CO_3 leaches is enhanced to about the same extent. The increase in extraction during the first Na_2CO_3 leach is reflected in the second Na_2CO_3 leach, but by the third or fourth Na_2CO_3 leach the difference in total uranium extraction becomes negligible whether the filter cake is given a preliminary hot or cold water treatment or no water treatment at all. It thus appears that a preliminary hot or cold water digestion of the filter cake can replace either one hot or one cold leach with sodium carbonate thereby reducing the reagent cost in the present process of extracting uranium from these filter cakes.

When precipitates were given a preliminary hot water treatment, the precipitate was usually diluted with water to form a slurry containing 2% of solids, and the slurry was held at 85 to 90° C. for one hour with constant agitation.

Precipitates obtained by the use of calcined dolomite as a precipitant, such as the one disclosed in Example 3, have a high calcium and a high sulfate content, which signifies that from 30 to 50% of the precipitate weight on a dry basis is calcium sulfate. By leaching these wet gypsum containing precipitates for 16 hours at 20–30° with 150 times their weight of water, the uranium content of the precipitates can be almost doubled and the quantity of Na_2CO_3 required per pound of uranium halved. When uranium containing precipitates obtained by the use of calcined dolomite as a precipitant are to be extracted with Na_2CO_3 solutions, it is frequently desirable to use large quantities of water in the preliminary water treatments in order to upgrade these precipitates by dissolving the calcium sulfate contained therein. Water leaching of the gypsum contained in these precipitates decreases the amount of Na_2CO_3 needed to extract the uranium therefrom.

The wet filter cake which contains precipitated uranium comprises about 20% of solids and 80% of water. Before the wet cake is digested with sodium carbonate, it has been found desirable to dilute it with at least an equal weight of water so that the sodium carbonate extractions are carried out on pulps that contain no more than about 10% of solids. If desired, the wet filter cake can be diluted to a 2.5%, 5%, or 7.5% solids pulp before the addition of the extraction reagent, but no very significant advantage has been noted in diluting the pulp of wet filter cake to contain less than 10% of solids. Experimental work has shown that variation in percent solids from 2.5% to 7.5% has very little effect on uranium extraction. The carbonate extraction from the various uranium containing precipitates mentioned herein has been found to be nearly constant per unit weight of precipitate, and hence the amount of Na_2CO_3 required per unit weight of uranium extracted varies inversely with the uranium content of the precipitate. If the leaching is done countercurrently, the amount of Na_2CO_3 required is decreased. Experience has shown that the pH of the Na_2CO_3 solution should be at least 9 to obtain good extraction of uranium. The requirement that the Na_2CO_3 leach liquor should have a pH of at least 9 is a deciding factor in determining the minimum amounts of Na_2CO_3 that must be placed in these leach liquors.

A given amount of carbonate per unit weight of precipitate gives the same percentage extraction on either high or low grade precipitates, and therefore the Na_2CO_3 required per pound of uranium extracted varies inversely with the precipitate grade. The same quantity of Na_2CO_3 per unit weight of precipitates of similar type will produce the same uranium extraction on a percentage basis irrespective of grade. Therefore, although the carbonate consumption per unit weight of wet precipitate for a given percentage recovery of U_3O_8 may be relatively constant, the carbonate consumption per unit weight of uranium recovered varies inversely with the precipitate grade.

If NaOH is used in conjunction with Na_2CO_3 in extracting uranium from these low grade uranium containing wet filter cakes, the amount of uranium extracted after the first digestion is somewhat increased, but after three digestions the total amount of uranium extracted from the filter cakes is about the same whether NaOH is used along with Na_2CO_3 in the digestions or Na_2CO_3 is used alone. Good uranium extraction with Na_2CO_3 is not obtained if the pH is less than 9. It is, therefore, sometimes desirable to add NaOH to a Na_2CO_3 leach liquor in order to raise its pH above 9.

During the Na_2CO_3 extraction operation, it is important that the Na_2CO_3 liquors contain sufficient of Na_2CO_3 or of Na_2CO_3 plus NaOH so that the pH of these liquors does not fall below 9 in the extraction operation. The pH of these Na_2CO_3 extraction liquors should be kept below 11 to avoid any precipitation of uranium.

In order to conserve water and Na_2CO_3 in the present process of leaching uranium from low grade uranium containing precipitates, it has been found desirable to employ a system of countercurrent leaching to extract the uranium from successive charges of these precipitates. To simplify the explanation of this system of countercurrent leaching, the successive charges of precipitates are designated by numbers 1, 2, 3, 4, 5, etc., and the various Na_2CO_3 leach solutions are designated as A, B, C, D, E, etc., in the order of their use. Solution A was used for the first leach of charge 1 only. Solution B was used for the second leach of charge 1 and the first leach of charge 2. Solution C was used for the third leach of charge 1, the second leach of charge 2, and the first leach of charge 3. Solution D was used for the fourth leach of charge 1, the third leach of charge 2, the second leach of charge 3, and the first leach of charge 4. Solution E was used for the fourth leach of charge 2, the third leach of charge 3, the second leach of charge 4, and the first leach of charge 5. Other consecutive charges of precipitate are in turn leached countercurrently four times by separate Na_2CO_3 leach solutions in accordance with this general pattern. By this countercurrent scheme of leaching the leaching capacity of Na_2CO_3 leach solutions was fully utilized by using them successively in the fourth, third, second and first leachings of successive charges of precipitates. To conserve water wash water was used to dissolve the Na_2CO_3 that was used in the various leach solutions A, B, C, etc. Instead of subjecting each new charge of precipitate to four successive Na_2CO_3 leachings in countercurrent fashion it has been found adequate in many instances to subject each charge of precipitate to a two or three stage countercurrent leaching system using sufficient Na_2CO_3 to maintain a pH of 9 or above in all stages. The countercurrent leaching with Na_2CO_3 was always preceded by a preliminary hot water treatment of the precipitate. It was frequently found necessary to add more Na_2CO_3 to a partially spent Na_2CO_3 leach solution before it was used for the first extraction of a fresh charge of precipitate in order that said extraction could be carried out at a pH of at least 9. Experience has shown that the pH of the Na_2CO_3 leach solutions had to be above 9 to obtain good extraction of uranium. Uranium containing precipitates, such as those described in Examples 1 to 3 have been subjected to a four stage countercurrent leaching with Na_2CO_3 leach solutions, and more than 95% of the uranium has thus been extracted from these precipitates, and about 12 pounds of Na_2CO_3 has been used per pound of uranium recovered.

In order to recover uranium from the carbonate leach solutions, it has generally been found preferable to acidify the filtered leach solutions with H_2SO_4 to a pH of 3. Vacuum was applied or the leach solution was boiled to help eliminate the CO_2 which was given off. Ammonia was then run into the leach solution to precipitate the uranium, which was filtered off and dried.

Resort may be had to such modifications and variations as fall within the spirit of the invention and the scope of the appended claims.

We claim:

1. A process for selectively recovering a high grade uranium concentrate from a low grade uranium containing hydroxide precipitate that has been obtained by the reaction of an alkaline earth metal hydroxide upon a sulfuric acid leach liquor containing uranium, said precipitate also containing large amounts of hydrated silica and iron and aluminum hydroxides, which comprises digesting said precipitate with hot water at 85–95° C., filtering, then leaching said precipitate at least twice at a pH between 9 and 11 with aqueous Na_2CO_3 solutions, separating the leach liquors from the leached solid residue, adding acid to the leach liquor to cause the evolution of carbon dioxide, and then adding ammonia to the leach liquor to cause the precipitation of uranium values therefrom.

2. A process for selectively recovering a high grade uranium concentrate from a low grade uranium contain-

ing hydroxide precipitate that has been obtained by the reaction of an alkaline earth metal hydroxide upon a sulfuric acid leach liquor containing uranium, said precipitate also containing large amounts of hydrated silica and iron and aluminum hydroxides, which comprises digesting said precipitate with water at 85–95° C., filtering then leaching said precipitate in a 2.5% to 10% solids slurry at least twice at a pH between 9 and 11 and at a temperature of 85–95° C. with aqueous solutions of sodium carbonate, separating the leach liquor from the leached solid residue, adding acid to the leach liquor to cause the evolution of carbon dioxide, and then adding ammonia to the leach liquor to cause the precipitation of uranium values therefrom.

3. A process for selectively extracting the uranium values from a low grade uranium containing hydroxide precipitate that has been obtained by the reaction of an alkaline earth metal hydroxide upon a sulfuric acid leach liquor containing uranium, said precipitate also containing large amounts of hydrated silica and iron and aluminum hydroxides, which comprises digesting said precipitate with water at a temperature of about 90° C. for at least one hour, filtering, then leaching said precipitate in a 2.5% to 10% solids slurry at least twice at a pH between 9 and 11 and at a temperature of 85–95° C. with aqueous solutions of Na_2CO_3, separating the uranium containing digest liquor from the digested solid residue which contains the greater part of the silica, iron and aluminum that were originally associated with the precipitate that was digested.

References Cited in the file of this patent

UNITED STATES PATENTS

1,001,480 Ulzer et al. ---------- Aug. 22, 1911

OTHER REFERENCES

Murray et al.: U. S. Atomic Energy Comm. unclassified paper No. MITG–A49, June 21, 1948, 21 pages; available from Office of Technical Services, Dept. of Commerce.

Michal: U. S. Atomic Energy Comm. unclassified paper No. MITG–A51, September 30, 1948.