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(54) URANIUM EXTRACTION FROM UNDERGROUND DEPOSITS

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URANIUM EXTRACTION FROM UNDERGROUND DEPOSITS

ABSTRACT OF THE DISCLOSURE

Uranium is extracted from underground deposits by passing an aqueous oxidizing solution of carbon dioxide over the uranium in the presence of calcium ions. Complex uranium carbonate or bicarbonate ions are formed which enter the solution. The solution is forced to the surface and the uranium removed from it.

BACKGROUND OF THE INVENTION

Underground deposits of uranium have been extracted

10   with solutions of ammonium bicarbonate, usually also contain-

ing hydrogen peroxide to oxidize any uranium in the +4 state

to the +6 state necessary for the formation of soluble

complex uranium carbonate and bicarbonate ions. While

ammonium bicarbonate solutions were successful in extracting

uranium, they were difficult to work with because ion ex-

changes occurred between the ammonium ions and calcium ions

in calcite deposits, leaving ammonia underground which

polluted water sources and added calcium to the solution.

As the solution was brought to the surface the decrease in

20   hydrostatic pressure resulted in the release of carbon

dioxide which raised the pH of the solution ($CO_2 \uparrow + 2H_2O \rightleftharpoons$

$H_3O^+ + HCO_3^-$) precipitating calcium carbonate ($CaCO_3 \downarrow + H_3O^+$

$+ HCO_3^- \rightleftharpoons Ca^{++} + 2HCO_3^- + H_2O$) which clogged the pipes. Also,

the use of hydrogen peroxide and ammonium bicarbonate added

to the costs of the extraction process, both in the purchase

of the chemicals and in metering them into the solution and

monitoring their concentrations. Alkali metal carbonates

and bicarbonates have also been used, but they also present

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problems by polluting underground aquafers.

PRIOR ART

U.S. Patent 2,992,887 uses low pressure CO_2 to maintain the integrity of Na_2CO_3 in a solution used to leach underground deposits of uranium.

U.S. Patent 3,130,960 uses a solution of CO_2 to leach uranium.

SUMMARY OF THE INVENTION

I have found that the use of ammonium and alkali
10   metal carbonates and bicarbonates to extract uranium can be •
entirely eliminated. Instead, I can use only carbon dioxide
and air. Not only are carbon dioxide and air very inexpen-
sive to use, but they present virtually no pollution problems.

I have found that solutions of carbon dioxide and
air extract almost as much uranium as do solutions of ammon-
ium carbonate. Also, since ammonium ions are not present to
exchange with calcium ions, precipitation of calcium carbon-
ate is less of a problem. I have also found that hydrogen
peroxide may precipitate uranium and decrease the amount
20   recovered. By using the oxygen in air, I am able to elimin-
ate the use of hydrogen peroxide except for deposits con-
taining predominantly uranium in the +4 state and for
recovery of traces of uranium after extraction without
hydrogen peroxide is complete.

DESCRIPTION OF THE INVENTION

An oxidizing aqueous solution of carbon dioxide is
prepared free of ammonium and alkali metal carbonates and
bicarbonates. The solution must have a bicarbonate ion
concentration of less than 5 grams/l as greater concentra-
30   tion can result in the precipitation of $CaCO_3$ which can clog

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the pipes. If no $CaCO_3$ is leached into the solution the solution will have a bicarbonate concentration of less than 1 gram/l. The solution must contain oxygen or its equivalent (e.g., hydrogen peroxide) at a concentration equivalent to at least one atmosphere of air, and preferably equivalent to at least one atmosphere of oxygen. These high oxygen concentrations are necessary because uranium in the +4 state is not solubilized by bicarbonate ions and will remain underground if it is not oxidized to the +6 state. The

10   solution is preferably prepared by injecting air and carbon dioxide, as a gas or as dry ice, into water as it is forced underground. Saturation with a blanket of carbon dioxide at atmospheric pressure can also be used but oxygen must then be sprayed in because the carbon dioxide drives out dissolved oxygen. The addition of chemicals to the solution which release carbon dioxide, such as calcium carbonate and hydrochloric acid, is not as desirable since the acid anion can create other problems. The use of oxygen gas may be desirable if the uranium in the underground deposit is

20   largely in the +4 state, but usually the more-costly use of oxygen gas will not be necessary. Hydrogen peroxide can also be used but is even less desirable as it is expensive, it promotes the precipitation of calcium carbonate, and it may be conveniently objectionable due to the dissolution of other oxidizable species. However, it may be useful in areas of predominately +4 state uranium or to recover uranium traces after remaining after extraction without hydrogen peroxide.

        Since the concentration of bicarbonate ion is very

30   small (i.e., less than 1 gm/l) unless calcium ions are

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present, it is necessary to add calcium ions to the solution
if the deposit does not contain enough calcium, which is
often present in the ground as calcite, $CaCO_3$. If the
deposit is deficient in calcium, it can be added to the
solution by pumping it over a bed of limestone ($CaCO_3$),
dolomite ($CaCO_3 \cdot MgCO_3$), or lime (CaO), until the solution
has picked up enough calcium ion or has become saturated.
When calcium is present, at unit activity, the bicarbonate
concentration is about 0.014 molar at 1 atmosphere of CO_2,
10   which is about the same bicarbonate concentration that would
result from using 1 gm/l of NH_4HCO_3.

The solution, which has a pH of about 3 to about 7
before entering the ground and about 5.5 to about 7.5 immedi-
ately after returning, preferably does not contain any
additive, in addition to those described. It is pumped into
the uranium deposit through one or more injection wells.
Due to the weight of the water and the pump pressure, the
CO_2 pressure at the bottom is considered to be at least 2
atmospheres which further increases the bicarbonate ion
20   concentration and therefore the amount of uranium extracted.
Some time later, the solution is pumped up again through one
or more recovery wells. Upon reaching the surface, the
uranium is removed from the solution by any suitable method.
A particularly useful method is to pass the solution through
ion exchange columns which extract the uranium onto the
column. The uranium is then washed off the column and is
precipitated, for example, as ammonium duranate (ADU). The
ADU can then be calcined to produce U_3O_8. The following
examples further illustrate this invention.

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EXAMPLES

Initially, the following compositions were prepared.

	# 1	# 2
A	50 g Lamprecht Uranium Ore (L.O.) 100 ml H_2O Air (1 atm) 0.8 g CO_2	50 g L.O. 100 ml 0.02 M – $CaCl_2$ Air (1 atm) 0.8 g CO_2
10 B	Same as above except solution contained 1 g/l H_2O_2 and 0.1% NH_4HCO_3	Same as above except solution contained 1 g/l H_2O_2 and 0.1% NH_4HCO_3

Each of the above formulations was to be contained
in polypropylene liners inside a Parr bomb with a volume of
approximately 325 ml. Assuming a free volume of 200 ml
after all components were placed in the bomb, the partial
pressure of CO_2 was approximately 2 atm. and the partial
pressure of O_2 was approximately 0.2 atm. Each of the

20 solutions from the above experiments were filtered and
analyzed for U and Ca. The duration of contact between the
solutions and the ore was noted carefully and no agitation
was provided during the experiment.

Several hundred grams of Lamprecht uranium ore
were obtained which reportedly contained approximately 0.1%
uranium. When it was discovered that a cold dilute H_2SO_4
leach of the ore recovered approximately three times the
amount of uranium reported to be present in the ore, a more
accurate analysis of the ore was initiated. Three samples

30 of Lamprecht ore from different bottles, obtained at different
times were analyzed by the differential pulse polarographic
(DPP) technique. All of these samples passed a 20 mesh
screen but were analyzed without further homogenization.
These ores contained 0.32, 0.34, and 0.36% uranium by the

DPP method. Spectrophotometric analyses showed some serious disagreement but x-ray fluorescence analysis of the ore confirmed the DPP results. The analysis for calcium was performed by an atomic absorption procedure involving use of synthetic standards and appropriate dilutions of the samples.

The results of the various leaching experiments are contained in the table which follows. While examining this data it is important to note both the uranium content and the calcium content as a function of leach composition.

10 The calcium analyses were performed several days after the leaching experiments were conducted. The importance of this point will become obvious in the discussion of the fate of uranium leached by solutions containing peroxide.

The pH of the solutions measured within a half-hour after filtering from the ore was consistently around 6.5. After several hours the pH's of the various solutions rose to values between 7.2 to 7.8. This behavior is similar to data reported from field tests of mining solutions and can be attributed to loss of CO_2 from the solutions.

20 The leach solutions containing Ca^{+2} were prepared by adding 2.222 g of $CaCl_2$ to one liter of water. This should have given a solution 0.02 M in $CaCl_2$ or about 800 ppm Ca^{+2}. The actual analysis of the solution indicated that it contained 775 ppm Ca^{+2}. The leach solution utilized in Experiment #6 was prepared by adding 0.5 ml of 10% $(NH)_4CO_3$ to 500 ml of H_2O containing 775 ppm Ca^{+2} and 0.1% H_2O_2. A precipitate of $CaCO_3$ formed immediately, thus removing some of the Ca^{+2} from solution. The resulting Ca^{+2} concentration was determined to be 445 ppm Ca^{+2}.

30 The data in the table which follows indicates

relationships, or lack thereof, between calcite solubility and the leaching of uranium.

Experiments #1 and #2 were conducted simultaneously and were intended to show the effect of high Ca^{+2} content on the leaching of uranium. Unfortunately, the reaction vessel was not sealed properly in #2 and CO_2 gradually leaked out during the experiment. Nonetheless, it is interesting to note that the solution containing 775 ppm Ca^{+2} leached approximately 67% as much uranium as did the solution which

10   retained CO_2 pressure and contained no Ca^{+2} originally. Furthermore, it is very significant that the Ca^{+2} content in Experiment #2 did not change even though 247 ppm of uranium were taken into solution. Since such high levels of uranium were recovered in these experiments in 16 hours, many subsequent experiments were conducted for only 70 minutes in order to get more data in a shorter period of time.

Experiments #3 and #4 were identical to #1 and #2 except that contact time was reduced from 16 hours to 70 minutes. Again, the solution containing no Ca^{+2} originally

20   leached more uranium than the one containing 775 ppm Ca^{+2} originally. This time, however, the solution containing Ca^{+2} leached more than 85% as much uranium as did the solution containing no Ca^{+2}. Also, note that the Ca^{+2} concentration actually declined slightly to 750 ppm in Experiment #4.

It is also interesting to compare Experiment #3 with #1. The 16 hour experiment leached 216 ppm more of uranium than did the 70 minute experiment but only leached 18 ppm more of calcium.

30   In order to determine why the presence of 775 ppm

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of Ca^{+2} apparently retards, albeit only slightly, the leach-
ing of uranium if the extraction of uranium does not depend
upon the dissolution of calcite another experiment (#13) was
devised to show the effect of the presence of a different
salt upon the leaching of uranium. The experiment was
devised so that the ionic strength given by a 0.02 M solution
of $CaCl_2$ would be matched by a NaCl solution. In order to
have a NaCl solution whose ionic strength is equivalent to
that of a 0.02 M $CaCl_2$ solution, the NaCl solution must be

10   0.06 M. The results of Experiment #13 show an even smaller
amount of uranium leached (113 ppm versus 129) by a 0.06 M
NaCl solution than was leached by the 0.02 M $CaCl_2$ solution.
In addition, the NaCl solution caused the dissolution of 175
ppm Ca^{+2} versus 118 ppm leached in Experiment #3 where no
salt was added. The latter result is not surprising since
it is well known that calcite is many times more soluble in
sea water than in fresh water. This bodes ill for the
operations in the solution mining program which introduce
chloride into the leach solution.

20   Having conducted Experiments #1, #2, #3, and #4,
it was desired to determine the effectiveness of the reagents
currently being used in the field and compare these results
to Experiments #3 and #4. The leach solutions in Experiments
#5 and #6 are identical to those in Experiments #3 and #4
except that they contain 0.1% $(NH_4)_2 CO_3$ and 0.1% H_2O_2.
The results of Experiments #5 and #6 were, for a while,
quite perplexing. The uranium content of these solutions
when first measured were less than 70 ppm in Experiment #5
and less than 40 ppm in Experiment #6. These analyses were

30   quoted as "less than" because the excess H_2O_2 gave a huge

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polarographic reduction wave which interfered with the measurement of the uranium (VI) peak. The uranium was obviously present but its concentration declined after several hours and declined further after several days until only a few ppm of uranium were left in solution. Concurrently, the appearance of a yellow-green sludge was noted in the bottom on the sample container. Clear liquid decanted from this sludge would begin to precipitate more of the material after standing for a few hours. After filtering these solutions the residue remaining on a 0.45 Millipore filter had a bright yellow appearance. The partial characterization of this deposit is described in some detail below, but suffice it to say that the residue was found to contain major amounts of uranium and calcium. Note that the solution which had no calcium added was very low in calcium and that the solution which originally contained 445 ppm Ca^{+2} increased in calcium content by only 40 ppm.

As soon as it was discovered that the uranium had been precipitating from the solutions from Experiments #5 and #6 another experiment (#9) was begun using the same leach solution but with a contact time of four hours. The uranium content of this solution proved to be less than the amount found from Experiment #5. At first glance one is tempted to dismiss this information as entirely predictable since the four hour contact time gave sufficient time for more uranium to be removed from solution by precipitation. This observation is true but it also means that the precipitation took place in the simulated underground condition and was not a result of bringing the solution to atmospheric pressure with a concurrent loss of CO_2 and rise in pH. This

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implies that uranium, although oxidized underground, may also be precipitated as an insoluble calcium salt underground. The calcium level in this solution from Experiment #9 was very low, as it should be if it is being precipitated along with the uranium. The appearance of the yellow precipitate in the bottom of the sample container was obvious.

Up to this point, no precipitate had been observed from any experiment except those containing $(NH_4)_2CO_3$ and H_2O_2. Experiments #7 and #8 had already been planned which would help pin down the conditions necessary to form the precipitate. If the CO_2 partial pressure or HCO_3^- concentration were factors in producing the precipitate then we should be able to produce the precipitate with $(NH_4)_2 CO_3$ (Experiment #8) or by raising the partial pressure of CO_2 (Experiment #7) which would have the effect of raising the HCO_3^- concentration and would also enhance calcite solubility.

As shown in the table which follows, the uranium content of the solution from Experiment #7 is within 10% of that obtained from Experiment #3, the calcium content is about a factor of 2 higher, and no precipitate was observed even after several days. The solution from Experiment #8 contained essentially the same level of uranium as did Experiment #3 and the calcium content was 25 ppm lower than Experiment #3. Again, no precipitate was observed even after several days.

Upon completion of these experiments it was obvious that the only time precipitation of uranium occurred was when H_2O_2 was present in solution.

Two more experiments seemed desirable in order to answer two persistent questions. "Will the uranium content

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of the solutions continue to rise with longer contact times?"
and "Will water and air alone effectively leach uranium from
the ore?"

Experiments #10 and #11 were designed to more
closely approximate contact times in the field although the
66 hours may still be considerably less than the field
condition. Experiment #11 which was conducted with 0.1%
$(NH_4)_2 CO_3$ extracted 580 ppm which is approximately 10% more
uranium and about 60 ppm less calcium than did the solution

10   in Experiment #10 which contained just CO_2, air, and H_2O.

In order to answer the second question asked
above, Experiment #12 was conducted utilizing only water and
air. Both uranium and calcium concentrations in this exper-
iment were very low, suggesting that carbon dioxide is
indeed needed for effective leaching of uranium.

Experiment #	Weight Ore (g)	Contact Time	H_2O_2	$(NH_4)_2 CO_3$	CO_2 (atm)	Air (atm)	Other	Analyses (ppm) U	Analyses (ppm) Ca^{+2}	Precipitate ?
1	50.28	16 hr.	0	0	2	1	None	367	136	no
2	50.40	16 hr.	0	0	2	1	0.02 M $CaCl_2$	247	775	no
3	50.26	70 min.	0	0	2	1	None	151	118	no
4	50.33	70 min.	0	0	2	1	0.02 M $CaCl_2$	129	750	no
5	45.09	70 min.	0.1%	0.1%	2	1	None	<70+	15.5	yes
6	45.19	70 min.	0.1%	0.1%	2	1	0.02 M $CaCl_2$ (precipitated $CaCO_3$)	<40+	485	yes
7	50.78	70 min.	0	0	4	1	None	138	255	no
8	50.07	70 min.	0	0.1%	2	1	None	149	93	no
9	51.46	4 hr.	0.1%	0.1%	2	1	None	<60+	35	yes
10	50.75	66 hr.	0	0	2	1	None	524	313	no
11	49.95	66 hr.	0	0.1%	2	1	None	580	253	no
12	50.24	70 min.	0	0	0	1	None	26.6	49	no
13	49.83	70 min.	0	0	2	1	0.06 M NaCl (3500 ppm NaCl)	113	175	no

0.02 M $CaCl_2$ leach solution 775

0.1% $(NH_4)_2 CO_3$ + 0.02 M $CaCl_2$ leach solution ($CaCO_3$ precipitated) 445

After careful examination of the data in the above

Table, the following conclusions can be made:

(1) Uranium recoveries from leach solutions containing H_2O_2 are poor due to the precipitation of an insoluble calcium-uranium salt.

(2) Carbon dioxide, air, and water will effectively leach uranium from the ore, but water and air will not.

(3) $(NH_4)_2 CO_3$ solutions may provide some slight advantage over CO_2, air, and water but this has not been conclusively proven, and furthermore calcite precipitation is introduced in the injection wells when $(NH_4)_2 CO_3$ is used.

(4) The amount of uranium in the various leach solutions does not appear to bear any relationship to the amount of calcite that has been solubilized and is affected only slightly by the amount of calcium in the leach solution, though some calcium must be present.

Characterization of the Calcium-Uranium Precipitate

When the precipitate was first noted, a small piece of filter paper containing a thin layer of the residue was dissolved in HNO_3 and H_2SO_4 and the solution was analyzed for uranium. The equivalent of 3.5 mg of uranium was found. Although this was a qualitative check for uranium, it was obvious that uranium was a major constituent of the residue.

An x-ray diffraction analysis of the residue was performed and showed strong patterns suggesting $CaCO_3$ and other compounds which obviously were due to some uranium compound, but it could not be positively identified.

An emission spectrographic analysis of the residue

was performed and showed that the major constituents were Ca
and uranium with lesser amounts of silicon, aluminum, mag-
nesium, and titanium.

The Use of NH_4HCO_3

The equilibrium concentration value for HCO_3^-
in equilibrium with $CaCO_3$ under a CO_2 atmosphere is $[HCO_3^-] =$
1.41×10^{-2} M. This conclusion is reached by utilizing the
first acid dissociation constant for carbonic acid, assuming
a partial pressure of $CO_2 = 1$ atm, a pH of 6, and substi-
10   tuting these values into the appropriate equilibrium expression
which is

$$\frac{[H^+] [HCO_3^-]}{[CO_2]} = K_{a_1} = 4.4 \times 10^{-7}$$

The molar solubility of CO_2 under 1 atm of CO_2 is
3.4×10^{-2} Moles/l. Substituting all of these values into
the above expression:

$$[HCO_3^-] = 1.41 \times 10^{-2} \text{ M}.$$

If 1 g/l of NH_4HCO_3 is added to the leach solution
the bicarbonate concentration would be approximately 1.3 x
10^{-2} M bicarbonate. This is essentially the same value that
20   would be obtained by allowing water to come to equilibrium
with calcite under 1 atm of CO_2. If the partial pressure of
CO_2 is greater than 1 atm then the bicarbonate ion concen-
tration would be even higher than 1.41×10^{-2} M.

The point to be made here is that the particular
ore tested is essentially an infinite source of bicarbonate
and that it is not necessary to add to it. In addition, the
NH_4^+ may exchange with Mg^{+2} and Ca^{+2} in certain "ion exchange"
clays in the ore, thus raising their levels in solution and

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creating a more serious calcite precipitation problem.

The Use of H_2O_2

The experiments show that uranium is precipitated very slowly from leaching solutions containing excess peroxide. Although this precipitate has not been quantitatively characterized it is known to contain high amounts of uranium. The use of hydrogen peroxide may very well be causing uranium oxidation in the ore body, but followed by uranium precipitation in the ore body. Some of the above-ground precipitates

10 that occur in the surge tanks and silting in the ion-exchange columns may also be uranium-bearing compounds. It is popular to claim that H_2O_2 will decompose upon contact with the ore and therefore, underground precipitation of uranium is not a factor. However, the experiments showed that after four hours of contact with the ore that the peroxide level was still very high. The quantitative precipitation of uranium can also occur in solutions of pH < 3.5 in the presence of H_2O_2, so incomplete precipitation of uranium at pH $5.5 - 6.0$ underground would not be surprising.

20   ### Calcite Precipitation

The reason that there is a calcite precipitation problem on the injection side of the process is that the pH of the leach solution has been raised by the addition of NH_3 or NH_4HCO_3 to such a level that calcite has only limited solubility. The raising of the pH is an unnecessary and futile exercise since the solution eventually equilibrates with the ore to produce a solution whose pH is governed largely by the CO_2-H_2O-calcite equilibria.

If the pH of the leach solution is not raised but

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is instead lowered, calcite precipitation will not be a
problem. This implies, of course, that the leach solution
would be recirculated without removing the calcium and begs
the question, "Can the uranium be effectively recovered
without removing the calcium?" It is believed that the
answer to this question is "Yes, it can," although the
experimentation to date does not conclusively prove that
point. Consider what the results of these experiments with
regard to calcium concentration and uranium recovery have

10   shown. The experiment with 800 ppm Ca^{+2} showed that the
recovery of uranium was approximately 85% of the amount
recovered with no calcium present. This is not a drastic
reduction and may be a higher absolute recovery than is
presently obtained with the current process.

The calcite removal system is costly and causes
increasing levels of Cl^- in the leach solutions which leads
to an increased solubility of calcite in the ore, creating a
"vicious circle" which can be avoided by eliminating the
calcite removal system, (i.e., the use of NH_4HCO_3).

20   If CO_2, air, and H_2O is added to the solution as
it comes off the uranium recovery ion exchange column and is
returned to the field, the pH of the solution will be lowered
so that no calcium will precipitate. The calcium levels in
the mining solutions will remain fairly constant with a
slight upward drift due to some gradual increase in ionic
strength caused in part by the increase in Cl^- levels as a
result of exchange of Cl^- for uranium species on the ion
exchange columns. If it were not for the increase in ionic
strength, the calcium content would be expected to rise to

30   an equilibrium level somewhere between 100 ppm and 1000 ppm

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at the recovery well and remain constant. It would remain

constant because the leach solution would already have been

in equilibrium with solid calcite in the ore body.

Some calcite precipitation as the solution is

brought above ground due to the rise in pH and escape of CO_2

can be expected. However, less precipitation should occur

under this invention since calcium levels should be less

than they are using the NH_4HCO_3 process which induces greater

calcite solubility underground, and therefore, more precipi-

10   tated calcite above ground.

What I claim is:

1.   1. A method of extracting uranium from under-

2. ground deposits comprising:

3.   (1) forming a solution of carbon dioxide which has

4. a ā bicarbonate concentration of less than 5 gms/l, which is

5. free of alkali metal and ammonium carbonates and bicarbonates,

6. and which contains oxygen or its equivalent at a concentration

7. equivalent to at least one atmosphere of air;

8.   (2) contacting said underground deposits with said

9. solution in the presence of calcium ions; and

10.   (3) forcing said solution to the surface.

1.   2. A method according to Claim 1 wherein said

2. solution is saturated with carbon dioxide.

1.   3. A method according to Claim 1 wherein air is

2. added to said solution to render it oxidizing.

1.   4. A method according to Claim 1 wherein said

2. calcium ions result from the dissolution of underground

3. deposits of calcite into said solution.

1.   5. A method according to Claim 1 wherein calcium

2. ions are introduced into said solution by passing said

3. solution over a calcium compound before said solution.

1.   6. A method according to Claim 5 wherein said

2. calcium compound is $CaCO_3$.

1.   7. A method according to Claim 5 wherein said

2. calcium compound is CaO.

1.   8. A method according to Claim 1 wherein hydrogen

2. peroxide is added to said solution.

1.   9. A method according to Claim 8 wherein the

2. amount of hydrogen peroxide is about 0.5 to about 1 gm. per

3. liter of solution.

1   10. A method according to Claim 1 wherein after

2 said method is applied to an underground uranium deposit,

3 said method is repeated using a solution to which hydrogen

4 peroxide has been added.

1   11. A method according to Claim 1 including the

2 additional last steps of passing said solution through an

3 ion exchange column whereby the uranium in said solution is

4 collected on said column.

1   12. A method according to Claim 11 including the

2 additional last steps of washing uranium off said column and

3 precipitating it from the wash solution.

1   13. A method according to Claim 12 wherein said

2 uranium is precipitated and calcined to produce U_3O_8.

1   14. A method according to Claim 1 wherein said

2 solution contains oxygen or its equivalent at a concentration

3 equivalent to at least one atmosphere of oxygen.

1   15. A method according to Claim 1 wherein said

2 solution is formed by carbon dioxide and air are injected

3 into water.

SUBSTITUTE

REMPLACEMENT

SECTION is not Present

Cette Section est Absente