# Progress Report for the Month of December, 1947

Contract No. W-35-094-eng-27



Prepared by: E. J. Center
H. A. Pray
A. C. Richardson
J. D. Sullivan

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## ORE DRESSING AND PYROMETALLURGICAL STUDIES

(A. C. Richardson, Supervisor)

Mr. J. D. Sullivan visited the smelter of the Consolidated Mining and Smelting Company, Ltd. at Trail, B. C., the latter part of November. While at Trail, he obtained a small grab sample of the phosphate rock from which they make fertilizer. This rock is mined near Garrison, Montana, and the small grab sample assayed 0.012 per cent of uranium. In the analytical procedure the Garrison sample behaved in a manner similar to the sample of Idaho phosphate rock, G-2090-8.

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## CHEMICAL WORK ON PHOSPHATES

(H. A. Frey, Supervisor)

The work during December has been directed to nitric acid leaching of superphosphate and leaching the residue from a water leach of superphosphate with various reagents in an effort to increase the extraction. The work in recovery was a study of the water leach solutions from all the available superphosphates and attempts to improve the uranium recovery by cooling the leach solutions, not washing the amine precipitate, using larger amounts of amine, etc. Some new organic reagents were also tried. Work in progress includes further treatment of superphosphate residues, roasting the residues and phosphate rocks prior to leaching, leaching of defluorinated phosphate rock and the precipitation of uranium with amines from roasted phosphate rock leach solutions.

### Superphosphate

A series of tests was made to determine the effect of nitric acid ($HNO_3$) leaching on uranium removal from superphosphate. Florida superphosphate 1090-20, a product of International Minerals and Chemical Company, was used for the tests. The results are shown in Table 82 along with the conditions.

The data indicated some improvement over water leaching, but the results were not better than those obtained with sulfuric acid under similar conditions. (See September report.)

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TABLE 82. THE EFFECT OF DILUTE NITRIC ACID SOLUTIONS ON LEACHING URANIUM FROM FLORIDA SUPERPHOSPHATE (1090-20)

Florida Superphosphate 1090-20. Product of International Minerals and Chemical Company

Basis: 190 g. superphosphate (equivalent to 100 g. of phosphate rock).
Uranium content: 11 mg.

Treatment: 190-g.-sample was leached with 500 g. of nitric acid solution of the concentration shown. Washed with 50 ml. of water.

| Sample No. | Nitric Acid Concentration, % | Uranium Extracted mg. | % |
|---|---|---|---|
| 2940-39-1 | 1 | 5.4 | 49 |
| 2940-52-1 | 5 | 6.0 | 55 |
| 2940-52-2 | 10 | 6.6 | 60 |
| 2940-52-3 | 15 | 6.8 | 62 |

Note: A water leach removes 4.0 mg. or 33% of the uranium.

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Superphosphate Residue

A bulk supply of residue was made by a water leach of Florida superphosphate, 1080-20, and a representative sample analyzed for uranium. The samples used for the following work were taken from this supply of residue.

The following reagents were used in the tests reported on this month: 96 per cent sulfuric acid, ammonium sulfate, sodium thiosulfate, hot dilute hydrochloric acid, glycerol, roast at 850° C. with sulfur, roast at 850° C. with carbon (both roasts were followed by dilute hydrochloric acid leaches), and treatment with ion-exchange resins — Deucil and Amberlite XE-3.

The results and conditions of these tests are shown in Table 83. The only results that stood out were those for dilute hydrochloric acid and the two roasts followed by hydrochloric acid leaches.

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TABLE 83. URANIUM EXTRACTION FROM FLORIDA SUPERPHOSPHATE RESIDUE FORMED BY WATER LEACHING

Florida Superphosphate 1090-20. Product of International Minerals and Chemical Company

Basis: Residue formed by leaching 190 g. of superphosphate with 500 ml. of water.
Uranium content of superphosphate: 11 mg.
Uranium content of residue: 7 mg.

| Sample No. | Reagent(1) | Method of Application | Uranium Extracted From Residue. mg. | % | Total. mg. | % |
|---|---|---|---|---|---|---|
| 2940-58-1 | 96% $H_2SO_4$ | Leach | 2.1 | 30 | 6.1 | 55 |
| 2940-58-2 | $(NH_4)_2 SO_4$ | Leach | 1.9 | 27 | 5.9 | 54 |
| 2940-58-3 | $Na_2S_2O_3 \cdot 5H_2O$ | Leach | Nil | - | 4.0 | 36 |
| 2940-58-4 | Hot 1:5 HCl | Leach | 4.4 | 63 | 8.4 | 76 |
| 2940-58-5 | Cycerol | Leach | Nil | - | 4.0 | 36 |
| 2940-58-6 | Sulphur | Roast + HCl leach | 5.1 | 73 | 9.1 | 83 |
| 2940-58-7 | Carbon | Roast + HCl leach | 5.8 | 83 | 9.8 | 89 |
| Ion Exchange Resins | | | | | | |
| 2940-59-1 | Domcil | (2) | 1.9 | 27 | 5.9 | 54 |
| 2940-59-2 | Amberlite, XE-3 | (2) | 2.3 | 33 | 6.3 | 57 |

(1) Quantity of reagent equal to number of moles of $CaSO_4$ in residue. Water used about 500 ml.

(2) Twice as much resin as residue by weight was slurried for 1 hour. The resin was screened to separate it from the residue. The uranium was removed from the resin by a dilute HCl wash. This process was repeated using the same resin and residue.

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Work in Progress

Two different phosphate rocks have been calcined and treated with different reagents in an attempt to remove the uranium.

Some of the residue referred to in the previous section was roasted and separate batches treated with different acids.

Phosphate rock has been treated with varying amounts and concentrations of nitric acid in an effort to determine more about the form of existence of the uranium compounds in the rock. More specifically it was hoped that these tests would establish whether or not there was a definite relationship between the fluorine and the uranium.

More work along this line will be done by studying how defluorinated rock responds to sulfuric acid treatment.

Uranium Recovery

The only superphosphates available for leaching prior to a couple of months ago were: (1) those prepared at Battelle from the various phosphate rock samples, (2) Sacco, a commercial product manufactured by Smith Agricultural Chemical Company and purchased on the open market, and (3) a sample (1090-20) secured by the U.S. Geological Survey from International Minerals and Chemical Corporation. The leach solutions from these superphosphates varied considerably as did the amount of uranium recovered from them. There are now available four more superphosphates 1090-28, 29, 55

and 56. They have been described in previous reports. Bulk supplies of leach solutions were prepared and precipitation tests were made. Table 84 gives a summary of the tests. Superphosphates 1090-20, 28, 55 and 56 are apparently similar and give similar leach solutions and recoveries. There is no way of knowing the history of the Sacco superphosphates but 1090-29 is a granular material of large particle size and very hard. No difference could be noted between the material as received and when the large particles were ground to pass 20 mesh before extraction. The extraction of uranium either in the ground or the as received condition being very low. There seems to be a relation between the amount of uranium and the amount of fluorine in solution; when the fluorine content is high the uranium content is correspondingly higher. Further tests are underway to determine the effect of fluorine on uranium recovery with amines.

Previous experiments had shown that heating a leach solution during precipitation with AM coco B was detrimental. Cooling the solutions in an ice bath did not increase the uranium recovery.

The amine precipitates had always been washed with distilled water after filtering from the leach solution. If the removal of uranium from the leach solution is an adsorption phenomena, this washing may cause a low yield. The yield was not increased by not washing the amine precipitate.

TABLE 84. LEACHING VARIOUS SUPERPHOSPHATES AND THE PRECIPITATION OF URANIUM FROM THE LEACH SOLUTION.

400 cc. of leach solution from 190 gr. of superphosphate.

500 mg. of Armeen C D used to precipitate the uranium

| Super-phosphate | Fluorine in 400 cc. leach solution | Uranium | | | | | |
|---|---|---|---|---|---|---|---|
| | | In Super-phosphate | In 400 c.c. leach solution | | Recovered by Armeen | | Left in Solution |
| | G. | Mg. | Mg. | % | Mg. | % | mg/l |
| Sacco | 0.08 | 15.2 | 2.0 | 13 | 1.6<br>1.5 | 75 | 1.3 |
| 1090-20 | 0.12 | 11.4 | 4.5 | 39 | 1.5<br>2.0 | 36 | 7.2 |
| 1090-28 | 0.09 | 15.2 | 4.0 | 26 | 1.6<br>1.6 | 40 | 6.0 |
| 1090-29(a) | 0.04 | 17.2 | 1.3 | 7.5 | 0.6<br>0.5 | 46 | 0.7 |
| 1090-29(b) | 0.04 | 17.2 | 1.3<br>1.2 | 7.5 | No test | | |
| 1090-55 | 0.17 | 13.2 | 4.6 | 35 | 1.3<br>1.2 | 28 | 8.2 |
| 1090-56 | No test | 13.2 | 4.6 | 35 | 1.3<br>1.2 | 28 | 8.2 |

(a) Granular as received

(b) Ground to -20 mesh

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There is no advantage in reprecipitating, with an additional 250 mg of AM cocc B, the filtrate from the first 250 mg precipitation over a single 500 mg precipitation. The sum of the two 250-mg yields of uranium were the same as a single 500-mg yield.

None of the fractions from fractionally distilled AM cocc B showed any appreciable advantage over the other as uranium precipitants from superphosphate leach solutions.

A report on the use of larger amounts of amine will be given as soon as analytical results are available.

Three new organic reagents were tested as possible uranium precipitants. They were: 1, 3 diaminobutane, Ethylene-diaminetetracetic acid and quinaldinic acid. All three gave precipitates in neutral or slightly acid uranyl nitrate solutions but only quinaldinic acid gave a precipitate in a superphosphate leach solution. Upon analysis the quinaldinic acid precipitate showed less than 0.05-mg of uranium from a leach solution containing 4.5 mg.

Phosphate rock (1090-18) has been roasted to remove the organic matter and tests are being made to prepare superphosphate from the roasted rock which will give a leach solution comparable in acidity to that from superphosphate 1090-20. It is hoped the removal of the organic matter will permit a better recovery in the amine precipitation.

Tests were also conducted using aluminum salts to tie up the fluorine in the superphosphate leach solution prior to the amine precipitation. Analytical results are not yet available.

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## ANALYTICAL WORK

(E. J. Center, Assistant Supervisor)

The fluorophotometer for the determination of micro amounts of uranium has been received from the Argonne National Laboratories. The instrument was not ready for operation since no wiring had been done and certain essential parts including lenses, reflector, and filters were missing.

At this writing missing parts have been obtained or original members reassembled to complete the optics of the fluorophotometer. Also a constant voltage source for the photo-multiplier tube has been constructed and all the electrical wiring finished. After aligning the optical system, five photo-multiplier tubes were checked for dark current. The preliminary tests showed them all to be satisfactory.

A die for the special type platinum dishes has been completed and a number of dishes prepared. Various grades of sodium fluoride are now being tested for uranium content. When suitable flux (NaF) is obtained standardization of the instrument will follow immediately.

Installation of the "Electro-Airmat" (dry-type electronic precipitator) in the photometer room is complete and the equipment is in operation.

Chemical analyses are continuing at an increasing rate by the modified ether extraction procedure.

EJR/HAP/ACR/JDS:fm
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