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**PROBLEMS OF LEACHING AND DIGESTION
OF URANIFEROUS SLAGS**

Progress Report No. 1, April–May 1951

By
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THE VIBRO MANUFACTURING COMPANY

PROBLEMS OF LEACHING AND DIGESTION OF URANIFEROUS SLAGS

Period: April - May 1951

Contract: AT(30-1)-1134

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1. Scope of Program

As of May 31, 1951, the research program consisted of the following projects:

Digestion

Investigate the applicability of various procedures for digesting C-slag materials, placing emphasis on procedures that are adaptable to the Canonsburg plant with a minimum of change in equipment.

Refining

Investigate the changes that can be made in the present refining operation to produce a cheaper, less pure product sufficiently low in fluoride, sulfate and phosphate for use in later refining steps without blending.

Liquid Waste Treatment

Investigate the recovery of uranium from certain liquid waste streams produced in present refining operations.

2. Previous Report

None

3. Project Status

Digestion

The results of laboratory and plant tests on the digestion of roasted C-slag materials in hydrochloric acid solution at a PH of 0 with sodium chlorate oxidation, indicate that a satisfactory hot digestion can be made in three hours at a temperature of 90°C and a satisfactory cold digestion can be made in six hours if the heat of neutralization of the lime is sufficient to maintain the digest slurry temperature above 80°C for the first two hours.

The results of laboratory and plant tests on the digestion of non-roasted C-slag materials, in hydrochloric acid solution at a PH of 0 and with sodium chlorate oxidation, indicate that a satisfactory hot digestion can be made in about nine hours at a temperature of 90° C if the material is ground fine enough to pass through a 100 mesh screen. This digestion is hazardous since the hydrogen evolved rapidly enough to ignite spontaneously at this temperature.

The results of laboratory tests on the so called cold digestion indicate that a satisfactory digestion can be made in nine hours if enough heat is added to simulate the temperature curve of the cold roasted digestion. The plant test on the digestion of slacked non-roasted C-scrap did not attain satisfactory temperature levels.

Refining

The results of laboratory tests indicate that fluoride cannot be satisfactorily removed by (1) clarification of the digest liquor, (2) complete washing of the crude ammonium uranate precipitate or (3) by reprecipitation of the ammonium uranate after digestion in dilute sulfuric acid.

A satisfactory product can be obtained in the form of sodium uranate by performing a sodium carbonate separation on the crude ammonium uranate and precipitating the sodium uranate with sodium hydroxide.

Liquid Waste Treatment

The project was inactive during the present report period.

DIGESTION

Laboratory digestions were made at 90°C on 100 gram charges of roasted C-scrap in hydrochloric acid solution with sodium chlorate oxidation. The uranium was found to be essentially removed, (acid insoluble residues less than 0.025% U3O8), after three hour digestions at a PH of 0 and only traces of uranium were found in the acid insoluble residue after five hour digestions at a PH of 0. Digestions at a PH of 1.0 for three hours left twice as much uranium in the insoluble residue as the ones at a PH of 0.0 and were discontinued.

A plant digest of a 5,000 pound charge of roasted C-scrap was made at 90°C for 10 hours with hourly samples being taken and analyzed and plotted as in Table I. The acid insoluble press cake contained only traces of U3O8.

Laboratory digestions were made on 100 grams charges of roasted C-scrap with 1.0 gram of sodium chlorate, sufficient hydrochloric acid to maintain a PH of 0.0 and no added heat. The results were not consistent but the fluctuations were probably caused by the differences in the temperatures of the solutions. It was decided to run plant tests in order to determine the

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temperature levels that might be expected under plant conditions. The results of these digestions are plotted in table II and indicate that the digest slurries must reach a temperature of 80° C for an efficient extraction.

Three hours laboratory digestions of non-roasted C-scrap at 90°C and 0.0 PH left acid insoluble residues analyzing about 0.04% U3O8 but these residues sometimes contained small particles of metallic magnesium. The digestions at 0.0 PH without added heat left acid insoluble residues analyzing from 0.18 to 0.51% U3O8. When the speed of acid addition was controlled and the slurry heated in a water bath in order to simulate the slow fall in temperature obtained in the cold plant digest of roasted C-scrap, (85°C to 39°C in six hours), six hour digestions left acid insoluble residues averaging 0.04% U3O8.

A 3,000 pound charge of raw C-scrap was slacked and ball milled in two charges for fourteen hours. The material attained a temperature of 56°C during slacking and the mix was 48°C at the end of the ball milling. Screen tests are listed in Table III.

The ground slacked C-scrap was pumped through a 16 mesh screen and digested with hydrochloric acid and sodium chlorate for nine hours. The maximum temperature was 60°C and the acid insoluble residue after nine hours analyzed 0.065% U3O8. The results of the samples are plotted in table IV.

A similar charge of slacked non-roasted C-scrap was digested at 90°C and 0.0 PH. The metallic particles reacted violently enough to envelope the surface of the solution in flames for a short period of time. The acid insoluble residue at the end of six hours analyzed 0.10% U3O8 but it was found that the PH had dropped to 0.5. The PH was adjusted to 0.2 and after decanting the solution, the final acid insoluble cake analyzed .028% U3O8. The results of this test are plotted in Table V.

Samples of raw C-scrap and raw C-liner were screened and fired in a muffle furnace. Visual observation indicated that the plus 10 mesh fractions contained more metallics than the minus 10 mesh fractions. Quantitative methods of determining the difference are being investigated.

The partial neutralization of roasted C-scrap to a PH of 7.0 was investigated on 750 gram charges. It was determined that only 45% of the soluble portion of the C-scrap was removed by this method. This work was discontinued since the reduction in bulk did not seem to be enough to compensate for the additional volumes of effluent solutions which mean longer uranium losses at this point.

REFINING

The crude ammonium uranate from the plant contains from 8% to 20% fluoride. Laboratory precipitations of the ammonium uranate at PH levels of 6.2, 7.0 and 8.5 indicated no change in the fluoride content. Washing of

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the precipitate by repulping it with a 1% solution of ammonium hydroxide or ammonium sulfate removed about half of the fluoride.

Filtration of the decanted digest liquor before the ammonium uranate precipitation has a large effect on its fluoride content. Laboratory precipitations with washing of the ammonium uranate until the filtrate has a chloride content equal to tap water gives a product which has a fluoride content of from 0.40 to 0.70 when fired at 1300°F. The fluoride content sometimes rises to a 4%, however, when a single repulp wash is substituted for the complete washing. The results indicate that fluoride contents of 1% to 2% can be expected if the digest liquor is clarified and the plant washing of the ammonium uranate improved.

Digestion of the crude ammonium uranate at 1.0 PH with sulfuric acid followed by a filtration and reprecipitation of the ammonium uranate was also tried but no consistent improvement was obtained.

The ammonium uranate press cake was leached both hot and cold with 2½% and 5% solutions of sodium carbonate. These experiments were discontinued as the residues were still rich in uranium after three leachings.

New sodium uranates were prepared by a sodium carbonate separation of the crude ammonium uranates, but examination of the analytical data on previous sodium uranates made from high fluoride feed indicates that the fluoride content will be well below the 0.5% limit.

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Table I--Hot Digestion of Roasted C-Scrap. Percent U_3O_8 in Acid Insoluble Residue vs. Time of Digestion.

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Table II--Cold Digestion of Roasted C-Scrap. Percent U_3O_8 in Acid Insoluble Residue vs. Time of Digestion.

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TABLE IIICLASSIFICATION OF BALL MILLED SLACKED C-SCRAP

	<u>Charge I</u> <u>3 hours</u>	<u>Charge I</u> <u>6 hours</u>	<u>Charge I</u> <u>9 hours</u>	<u>Charge I</u> <u>12 hours</u>	<u>Charge II</u> <u>12 hours</u>	<u>Remarks</u>
Plus 40 mesh	17.0%	8.0%	2.0%	5.0%	0.5%	Mostly Metallic
Minus 40 Plus 60	8.0%	9.0%	4.0%	2.0%	5.0%	Mostly Metallic
Minus 60 Plus 100	9.0%	11.0%	10.0%	9.0%	12.5%	
Minus 100 Plus 200	12.0%	12.0%	16.0%	16.0%	10.0%	
Minus 200 mesh	54.0%	60.0%	68.0%	68.0%	72.0%	

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Table IV--Cold Digestion of Raw C-Scrap.

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Table V--Hot Digestion of Raw C-Scrap.

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