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LYOMETALLURGICAL TESTS ON MARYSVALE URANIUM ORES

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Topical Report

LYOMETALLURGICAL TESTS ON MARYSVALE URANIUM ORES

By

F. W. Bloecher, Jr.

November 30, 1950

ABSTRACT

Lyometallurgical leach tests have been run on samples of uranium ore from Marysvale, Utah. Uranium extractions range from 80 per cent to 92 per cent either by leaching raw ore with 10 per cent nitrated ethyl ether or by leaching sulfuric acid-digested material with 5 per cent nitrated ethyl ether

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Approved for A. M. Gaudin, Director

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By

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I. INTRODUCTION

Three samples of uranium ore from the Marysvale district, Piute County, Utah were received by the MIT Mineral Engineering Laboratory, and designated Q1, Q2, and Q3. Q1 and Q2, received in January 1950, contained secondary uranium minerals, chiefly autunite and torbernite, while Q3, received in May, was somewhat less oxidized and contained some primary uraninite. Table 1 summarizes pertinent information concerning the three samples.

Table 1. Description of Marysvale Uranium Ore Samples

Sample	Head Assay, % U_3O_8	Source	Shipper
Q1	0.113	Bullion-Monarch Mine	U.S. Bur. Mines, Salt Lake City
Q2	0.160	Prospector Lease (Vanadium Corp. of America)	U.S. Bur. Mines, Salt Lake City
Q3	0.200	Vanadium Corp. of America	AEC Sampling Plant, Grand Junction, Colorado

Topical Report MITG-253 contains a mineralogical description of these ores as well as a description of a series of hydrometallurgical leaching tests carried out on them. Two informative articles on the Marysvale uranium district have recently appeared in mining magazines[1,2]*.

Solvent leaching tests on a variety of uranium-bearing materials, both raw ores and plant products, had indicated that the process might be economically feasible for treating low-grade materials using nitrated ether to leach either the raw material or a sulfuric acid-digested product[3,4,5,6,7]. This report describes a number of preliminary lyometallurgical** leach tests carried

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*References are listed in the Bibliography.

**A new term for the solvent leaching process, LYOMETALLURGY, has been coined by the MIT Mineral Engineering Laboratory. It is, of course, synonymous with the term "solvent leaching" used in earlier reports. Lyo is a combining form from the Greek word, lyein, meaning to loose or dissolve. Lyometallurgy is a more euphoneous term than either "anhydrometallurgy", or the contradictory term, "non-aqueous hydrometallurgy", which were also suggested as appropriate names for the solvent leaching process.

out on the Marysvale ores, Q1, Q2, and Q3, during the period May through August, 1950.

Leaching was tried on the raw ores, roasted ores, and sulfuric acid-digested ores using a batch agitation method with the all-glass leaching apparatus described in MITG-246. In addition, a number of static leaches were run on the raw ores, and several percolation leaches were tried on raw Q3 ore. One large-scale batch test was run on -4 mesh Q3 ore. A solution of nitric acid in ethyl ether was the leach solution used in most of the tests.

II. SUMMARY

Three samples of uranium ore from the Marysvale, Utah district were tested to determine if uranium could successfully be extracted by lyometallurgical leaching.

From the more oxidized Q1 and Q2 ores extractions of the order of 70 per cent were effected, while from Q3 ore over 90 per cent uranium extraction was achieved by leaching with nitrated ethyl ether*, 10 per cent nitric acid by volume. Roasting the ores did not appreciably affect uranium extraction from Q2 and Q3, but did raise the extraction from Q1 about 5 per cent. Nitrated isopropyl ether solution was not as effective a lixiviant as nitrated ethyl ether.

Sulfuric acid digestion of Q1 and Q2 ores at 50 per cent solids for 24 hours at room temperature, followed by drying and leaching with 5 per cent nitrated ethyl ether raised uranium extractions to over 80 per cent and cut down the nitric acid requirements. Additional sulfation tests have been reported in MITG-246.

Percolation leaching of Q3 ore with 10 per cent nitrated ethyl ether effected uranium extractions of over 90 per cent at several flow rates, both on one-inch-deep beds of approximately 100 mesh ore, and on an 11-inch bed of -20 mesh ore. Reagent requirements were of the same order of magnitude as those in the batch leaching tests. Only a few inches of head were needed to get adequate flow rates of solvent through the bed of undeslimed ore.

Uranium extractions from these three ores compare favorably with those achieved by hydrometallurgical technique. Although lyometallurgical leaching utilizes more expensive reagents, it completely eliminates the filtration and thickening difficulties experienced in the wet (water) process. For instance, filtration in the lyometallurgical process is 270 times faster than in the sulfuric acid process.

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*The term "nitrated ether" in this report refers to a solution of ether and nitric acid and does not necessarily imply attachment of the nitrate groups to the carbon atoms of the ether.

III. AGITATION LEACHING

Preliminary Batch Leaching

Each of the Marysvale ore samples was ground in a disc pulverizer to approximately 70 to 90 per cent -100 mesh, and then leached with nitrated ethyl and isopropyl ethers under an arbitrary set of conditions, chosen on the basis of previous experience with low-grade ores. Fifty-gram charges of ore were leached with 75 ml of leach solution, 10 per cent nitric acid by volume, for 1 hour in the standard leaching apparatus described in MITG-246. The pregnant liquor was then withdrawn to the level of the solid residue, and the cake given two displacement washes with an amount of plain, non-nitrated ether equal to that held up in the cake. Water was then added to the pregnant and wash solutions and the ether evaporated on a steam bath. The resultant aqueous solution was made up to a standard volume (usually 100 ml) and assayed fluorimetrically* for uranium. The leach residue was dried overnight at about 80°C, weighed and also assayed fluorimetrically for uranium.*

The addition of water to the ether solutions was, of course, merely a laboratory convenience. In a plant operation the uranium would be precipitated directly from the ether with ammonia gas, the precipitate separated by filtration, and the ether reused.

Table 2 summarizes the results of the preliminary tests run on Q1, Q2 and Q3 ores using nitrated ethyl and isopropyl ethers, 10 per cent nitric acid by volume. Also included in Table 2 are data on roasted ores. Raw Q3 ore was quite amenable to leaching with ethyl ether solution, uranium extraction being of the order of 92 per cent. Uranium extractions from Q1 and Q2 ores were 70 per cent and 65 per cent, respectively, using nitrated ethyl ether as the leach solution.

Leaching of Roasted Ores

Samples of each of the Marysvale ores were roasted prior to lyometallurgical leaching to see if uranium extraction could be improved. Charges of 100 mesh** ore were placed in a thin layer in stainless steel pans and roasted at 400°C in a muffle furnace for 5 hours. The ore was rabbled occasionally during the roast. Weight losses (as per cent of initial charge weight) were:

Q1	4.2%
Q2	1.4%
Q3	2.2%

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*All uranium assays in the tests reported here, both of solutions and solids, were made fluorimetrically.

**In this report 100 mesh ore denotes ore that has been pulverized to 70 to 90 per cent -100 mesh.

Table 2. Leaching of Raw and Roasted Ores

Material	Test No.	Ether[1]	Wash Ether Added, ml/g Ore[2]	Per Cent U_3O_8 Calc. Head	Per Cent U_3O_8 Extraction[3]
Q1, Raw	Q149	Ethyl	1.72	0.11	70.4
Q1, Raw	Q148	Isopropyl	1.46	0.12	67.0
Q2, Raw	Q249	Ethyl	1.66	0.18	65.9
Q2, Raw	Q248	Isopropyl	1.72	0.16	53.3
Q3, Raw	Q316-1	Ethyl	1.52	0.23	91.8
Q3, Raw	Q316-2	Ethyl[6]	1.32	0.21	73.9
Q1, Roast[4]	Q155	Ethyl	1.16	0.12	75.0
Q1, Roast	Q160	Isopropyl	1.24	0.11	65.0
Q2, Roast	Q255	Ethyl	1.38	0.16	64.3
Q2, Roast	Q260	Isopropyl	1.24	0.14	36.4
Q3, Roast	Q319-1	Ethyl	1.00	0.21	88.0
Q3, Roast	Q319-2	Ethyl[6,7]	1.15	0.21	63.9
Q1, Roast	Q112-2	Ethyl[5]	1.23	0.13	77.1

Note: Fifty-gram charges 100 mesh material were leached for 1 hour with 75 ml leach solution in glass chamber.

[1] Ten per cent nitric acid by volume, except where noted.
[2] As two displacement washes of plain ether, in amounts equal to the holdup in the bed.
[3] Based on calculated head assay.
[4] All roasting was done at 400°C for five hours.
[5] Two-hour leach with 40 grams calcine, 60 ml 10 per cent nitrated ether.
[6] Five per cent nitric acid by volume.
[7] Charge was only 20 grams calcine, 30 ml 5 per cent nitrated ether.

The calcines were leached by the same procedure used on the raw ores in the tests outlined in Table 2. The calcine leach data are also included in Table 2 for ease of comparison with the raw-ore data.

Roasting under the conditions outlined seemed to improve uranium extraction slightly in the leaching of Q1 calcine, did not affect uranium extraction in the case of Q2, and decreased uranium extraction from Q3 calcine as compared to the raw ore.

The results of these preliminary batch tests on raw and roasted ores indicated that nitrated ethyl ether was a better lixiviant than nitrated isopropyl ether, judging from uranium extractions with each. Therefore, in all further tests only nitrated ethyl ether was used.

Effect of Leach Time

A series of tests was run on Q1 ore varying the leach time from 1 to 8 hours. Leach conditions were the same as specified in Table 2, 10 per cent nitrated ethyl ether being used as the leach solution. The data collected are presented graphically in Figure 1 where per cent uranium extraction is plotted against leach time. Also shown on Figure 1 are several tests on raw Q3 ore, and on roasted (5 hours at 400°C) Q1 ore.

Uranium extraction from Q1 ore increases with time, going from 70 per cent to 78 per cent with an increase of leach time from 1 to 8 hours. The roasted Q1 ore illustrates the same trend. Uranium extraction from Q3 ore, however, seems to have reached a maximum after only 1 hour of leaching. Uranium extractions are based on calculated head assays.

Effect of Nitrated Ether Strength

A number of additional 1-hour tests were run with 2 per cent and 5 per cent nitrated ethyl ether to learn how uranium extraction varied with acidity of the leach solution. Leach conditions were the same as those in the preliminary tests on raw and roasted ores, with from 1.2 to 1.7 ml/g of plain ether being used in the displacement washing. Figure 2 is a graphical summary of the data.

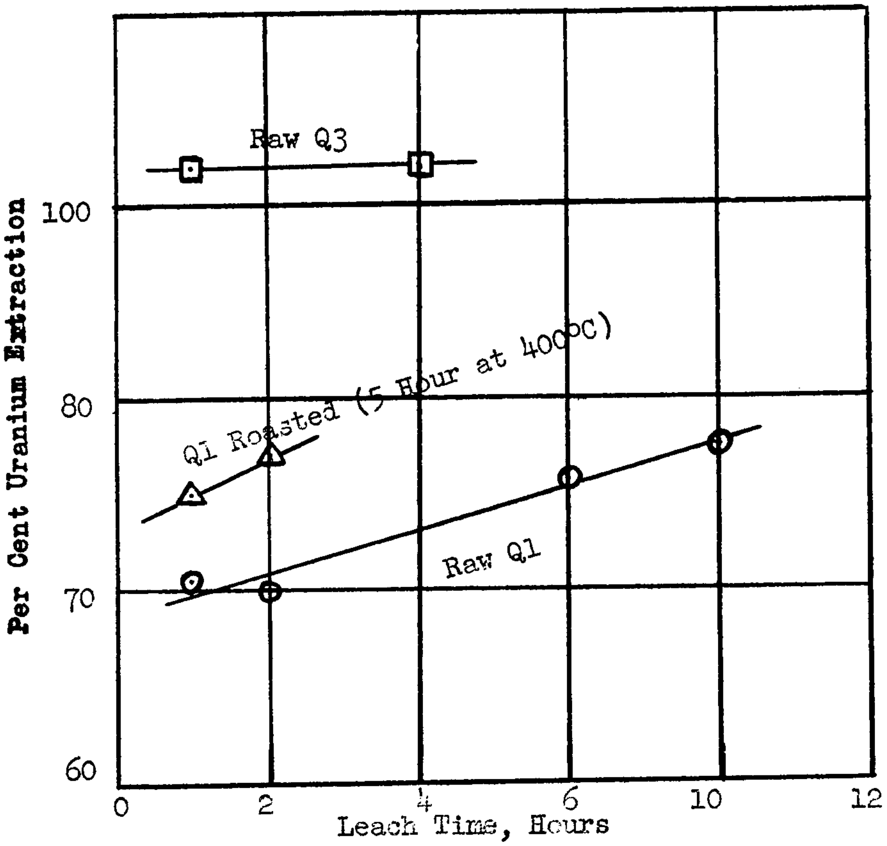
Uranium extraction from Q2 and Q3 ore fell off linearly with decreasing acid strength, but extraction of uranium from Q1 ore seemed to level off above 5 per cent nitric acid strength. Uranium extractions are based on calculated head assays.

Two-Stage Leaching of Q1 and Q3 Ores

Two-stage leaches were tried on Q1 and Q3 ores using two 1-hour leaching periods. Q1 was leached with 10 per cent nitrated ether (1.5 ml/g ore) for 1 hour, the pregnant solution withdrawn and the cake washed with 10 per cent ether. It was then releached for 1 hour with 1.5 ml/g of 10 per cent nitrated ether, the second pregnant withdrawn and the cake washed with plain ether. Q3 was similarly treated except that 5 per cent nitrated ether was used and the displacement wash at the end of the first leach was made with plain ether. The test results are summarized in Table 3.

The two-stage leach on Q1 ore gave a total of 75.6 per cent uranium extraction, 58.9 per cent in the first stage and 16.7 per cent in the second stage. A single leach on this material, with two displacement washes, had given 70.4 per cent uranium extraction. A two-stage leach of this type on Q1 ore probably would not be economically justified by the small increase in uranium extraction effected.

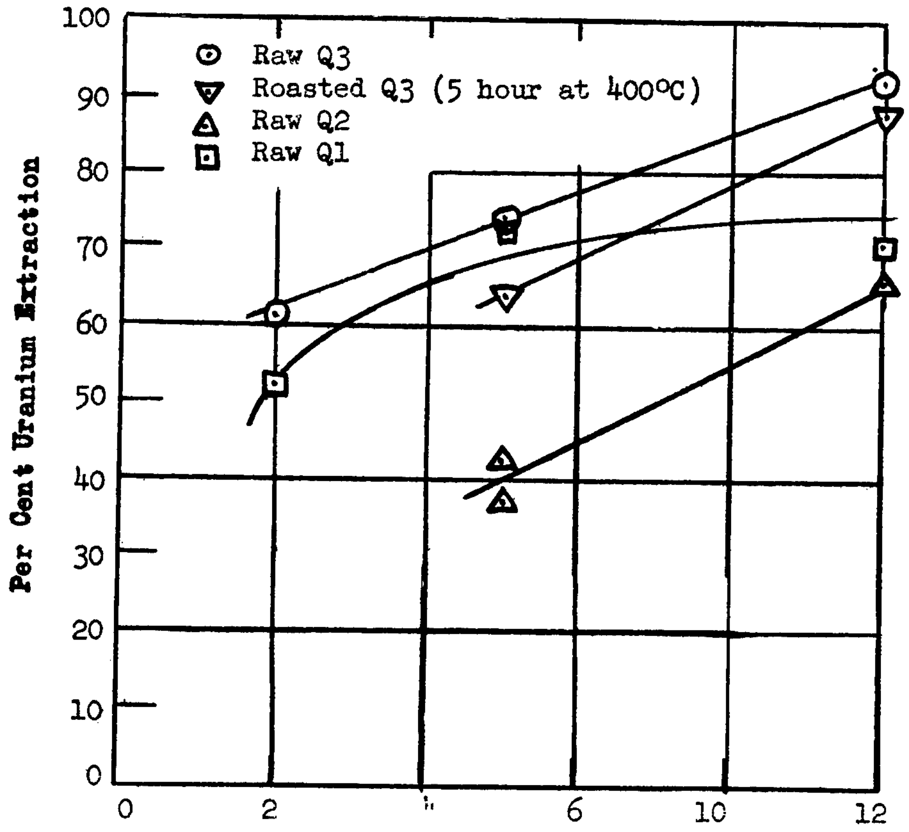
The two-stage leach on Q3 ore was tried to see if two 1-hour leaches with 5 per cent nitrated ether gave the same uranium extraction as a single 1-hour leach with 10 per cent nitrated ether. They did not, the extraction being only 82.2 per cent, as compared to 91.8 per cent in a single 1-hour leach with 10 per cent nitrated ether.



Leach Conditions

50 Grams 100 Mesh Ore; 1.5 ml/g of 10%
Nitrated Ethyl Ether; 1.5 to 2.2 ml/g of Plain
Wash Ether Used

Figure 1. Effect of Leach Time Upon Uranium Extraction



Leach Conditions

50 Grams 100 Mesh Ore; 1.5 ml/g of Nitrated Ethyl Ether; 1.2 to 1.7 ml/g of Plain Wash Ether Used; Leach Time: 1 Hour

Figure 2. Effect of Nitrated Ether Strength Upon Uranium Extraction

Table 3. Two-Stage Leaching of Q1 and Q3 Ores

Ore	Test No.	Wash Ether Added, ml/g Ore		% Uranium Extraction[1]			Calc. Head, % U_3O_8
		Stage 1	Stage 2	Stage 1	Stage 2	Total	
Q1	Q18-2	0.78[2]	1.88[3]	58.9	16.7	75.6	0.11
Q3	Q331	0.62[4]	1.60[3]	76.9	5.3	82.2	0.20

Note: Leach conditions were: 1.5 ml/g of nitrated ether for each of two 1-hour periods, ten per cent strength in Q1 test, and five per cent strength in Q3 test. Fifty-gram charges of 100 mesh ore were used.

[1] Based on calculated head assay.
[2] As one displacement wash with 10 per cent nitrated ether (an amount equal to holdup in the bed).
[3] As two displacement washes of plain ether.
[4] As one displacement wash of plain ether.

Batch Leach of -4 Mesh Q3 Ore

Uranium extraction by a percolation leach of -20 mesh Q3 ore (See Section IV) compared so favorably with results of batch and percolation leaches of 100 mesh ore that a batch leach was tried on -4 mesh Q3 ore. A 400-gram charge of -4 mesh Q3 ore was leached with 1.5 ml/g of 10 per cent nitrated ethyl ether for 4 hours by rolling in a 2-liter bottle. The bottle was fitted with a condenser attached through a packing gland. At the end of the leach the pregnant liquor was filtered off and the cake given two displacement washes with plain ether, totaling 1.25 ml/g. The data are summarized in Table 4. The total uranium extraction was 78.3 per cent as compared to 92 per cent on the -20 mesh and 100 mesh ores.

A 4-hour sulfuric acid leach was also tried on the -4 mesh ore using 160 lb H_2SO_4 per ton of ore.* A 400-gram charge of ore was pulped in a bottle with distilled water at 50 per cent solids by weight, the sulfuric acid added and the bottle rolled for 4 hours. The pregnant solution was filtered off and the cake given two displacement washes with an amount of water totaling 1.25 ml/g, or 2500 lb/ton of ore. These data are also summarized in Table 4. The total uranium extraction was 83.2 per cent.

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*Hydrometallurgical tests had indicated that 160 lb H_2SO_4 per ton of ore would give maximum uranium extraction; see MITG-253.

Table 4. Leaching of -4 Mesh Q3 Ore

Test No.	Type of Leach	Per Cent Uranium Extraction[1/] Preg.	Wash 1	Wash 2	Total	% U_3O_8 in Tails	% Wt. Loss
Q355-1	Ether[2/]	72.4	5.1	0.8	78.3	0.049	0
Q355-2	Sulfuric Acid[3/]	42.6	20.4	20.2	83.2	0.049	3.5

[1/] Based on calculated head assay. Ether leach calculated head: 0.23 per cent, sulfuric acid leach calculated head: 0.28 per cent.

[2/] Nitrated ethyl ether, 10 per cent nitric acid by volume, was used; 1.5 ml/g of ore; leach time was 4 hours.

[3/] 160 lb H_2SO_4 per ton ore added to pulp at 50 per cent solids; final pH was 0.3; leach time was 4 hours.

Uranium assay of the tails from both leaches was the same, 0.049 per cent U_3O_8. The difference in uranium extraction in the two leaches is therefore not too significant, the 5 per cent increment being reflected in the difference in calculated heads, the sulfuric acid leach head being 0.28 per cent U_3O_8, rather high compared to the assay head. However, it is evident from the weight losses observed after leaching that the aqueous sulfuric acid attack is not as selective as the ether-nitric acid attack upon the ore. The more selective the leaching, of course, the higher the grade of the product will be.

IV. FIXED-BED LEACHING OF Q3 ORE

Static Leaching

The feasibility of percolation leaching of Q3 ore had been indicated by the ease with which the ether solutions filtered through the leach cake (as with all Q ores), and by the readiness with which the uranium was extracted from Q3 ore. Prior to running percolation leaches, however, a series of static leaches was run using 40-gram charges of ore with 60 ml of 10 per cent nitrated ethyl ether solution in glass-stoppered erlenmeyer flasks. At the end of the leach the liquor was filtered off on a fritted-glass filter, and the cake washed by displacement with 75 ml of plain ether.

Figure 3 is a graphical summary of the leach data. The peak uranium extraction from the 100 mesh ore was 92 per cent, reached after 8 hours of leaching. The uranium extraction from the -20 mesh ore did not reach 90 per cent even after 48 hours of leaching.

Percolation Leaching

Three percolation leaches were run on Q3 ore, two on 100 mesh material in a one-inch bed, and one on -20 mesh ore in a bed 11 inches deep. The leach solution, 10 per cent nitrated ethyl ether, was percolated through the bed from above, the flow rate being controlled by the depth of the pool above the bed. This method of flow rate control was necessary because as the leach solution passed through the ore, the bed picked up moisture and gradually increased its resistance to fluid flow. Consequently, as the percolation leach progressed, the pool depth had to be increased in order to maintain a constant flow rate. Addition of a small amount of acetone to the leach solution might prevent the increase in resistance to flow which was observed. This has not yet been checked, however.

The one-inch bed of 100 mesh ore was contained in an ordinary, coarse-frit leach chamber, the effluent being discharged into the standard receiver used with the leach chambers. The -20 mesh ore was held in a 22-mm diameter glass tube with a coarse fritted-glass filter sealed to the bottom, together with a stopcock and joint which fit into the standard receiver. The ore was first pulped in plain ether in a beaker and poured into place. The ether was drawn off to the level of the top of the ore, and the flow of nitrated ether leach solution started. The leach solution was fed by gravity from a constant head reservoir, its flow rate being controlled by a hose clamp.

During the leach, increments of effluent were drawn off from the receiver, water added, and the ether evaporated on a steam bath. The resultant aqueous solutions were made up to 100 ml and assayed for uranium. At the end of the leach percolation was halted, and the pool of solution above the bed and as much holdup liquor as could be drawn off by a slight vacuum were collected and assayed. In the test with the 11-inch, -20 mesh bed, the solution in the pool was decanted; vacuum filtration would have evaporated a considerable amount of the ether. The residues were dried

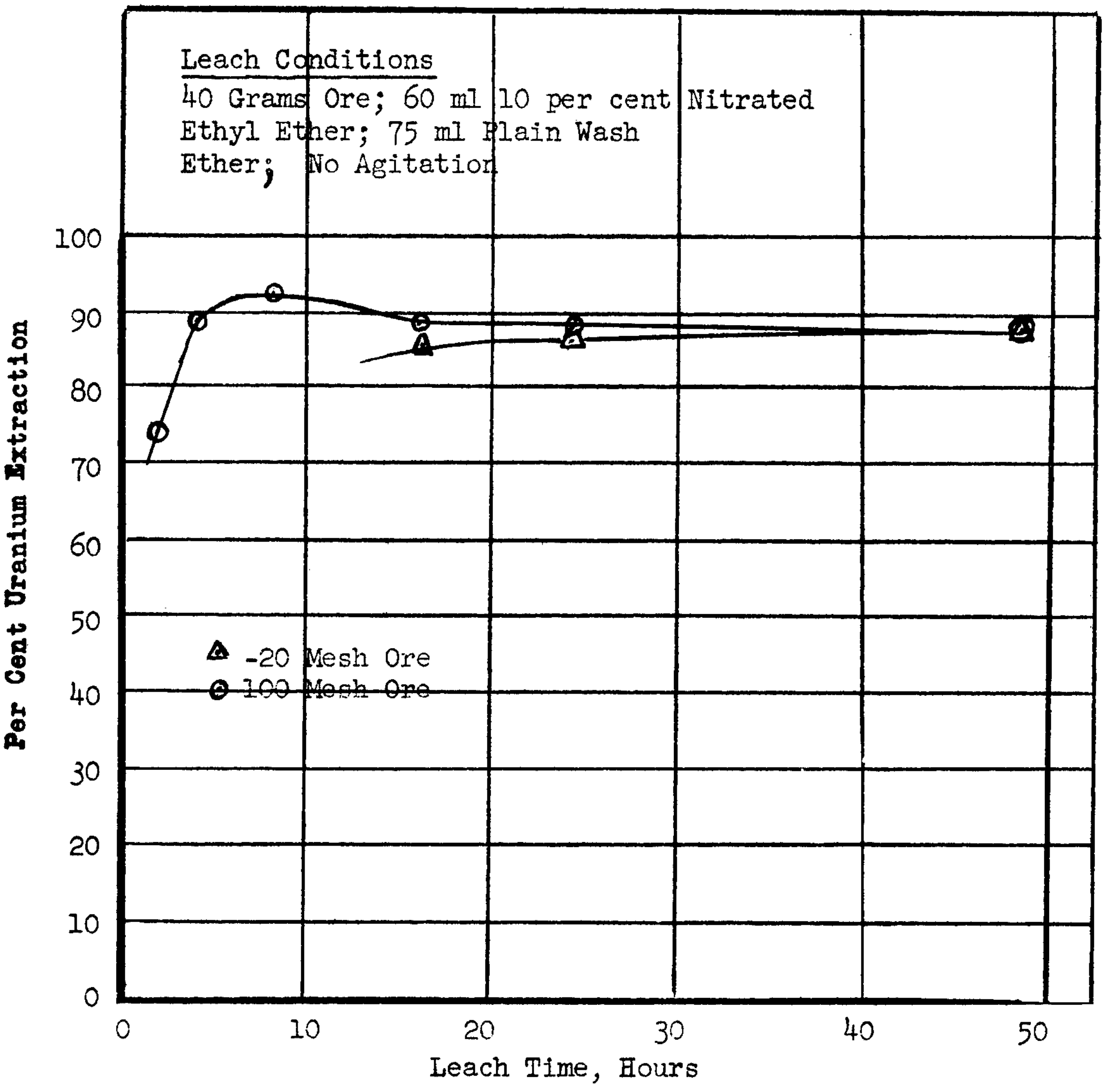

Figure 3. Static Leaching of Q3 Ore

overnight at 80°C, weighed and assayed for uranium. The data for the three percolation leaches are presented in detail in Appendix A. Table 5 is a summary of the data from the three tests.

Table 5. Percolation Leaching of Q3 Ore

Test No.	Ore Size, Mesh	Average Flow Rate, ml/min/g Ore	Total ml thru Bed	ml per g Ore	Per Cent U_3O_8 Calc. Head	Per Cent U_3O_8 Extraction[1]
Q345	100	0.017	393[2]	7.86	0.21	92.0
Q348	100	0.0036	95[2]	1.90	0.22	90.0
Q350	-20	0.0047	503[3]	3.35	0.21	92.9

Note: In tests Q345 and Q348, 50-gram charges of 100 mesh ore made a bed one inch deep. In test Q350, 150 grams of -20 mesh ore made a bed 11 inches deep. Leach solution used was 10 per cent nitrated ethyl ether.

[1] Based on calculated head assay, and including uranium contained in pool and holdup solution recovered at end of test.

[2] Includes 21 ml of plain holdup ether in bed at start of leach.

[3] Includes 120 ml of plain holdup ether in bed at start of leach.

The average flow rates during the leaching of the 100 mesh ore were 0.0036 and 0.017 ml/min/g ore. At the slower rate uranium extraction had reached 89.5 per cent after passage of 1.6 ml/g ore (including 0.42 ml/g ore of plain holdup ether in the bed at the start of the leach) through the bed in 7 hours, 28 minutes. At the faster flow rate, 91.6 per cent uranium extraction was reached after passage of 5.32 ml/g ore (including the plain ether in the bed) through the bed in 5 hours, 28 minutes. The flow rate through the 11-inch bed of -20 mesh ore averaged 0.0047 ml/min/g ore. It fell off towards the end of the leach, finishing at 0.0035 ml/min/g ore, because after the solution level had reached the top of the glass tube containing the bed, the increase in flow resistance could no longer be compensated for by further increase in pool depth. Uranium extraction had reached 91.5 per cent after passage of 1.97 ml/g ore (including 0.80 ml/g of plain ether in the bed at the start of the leach) through the bed in 6 hours, 55 minutes.

Figures 4, 5 and 6 show the relationship between cumulative uranium extraction and total amount of solution passed through the bed, together with the distribution of the uranium in the effluent during the leach for each percolation test.

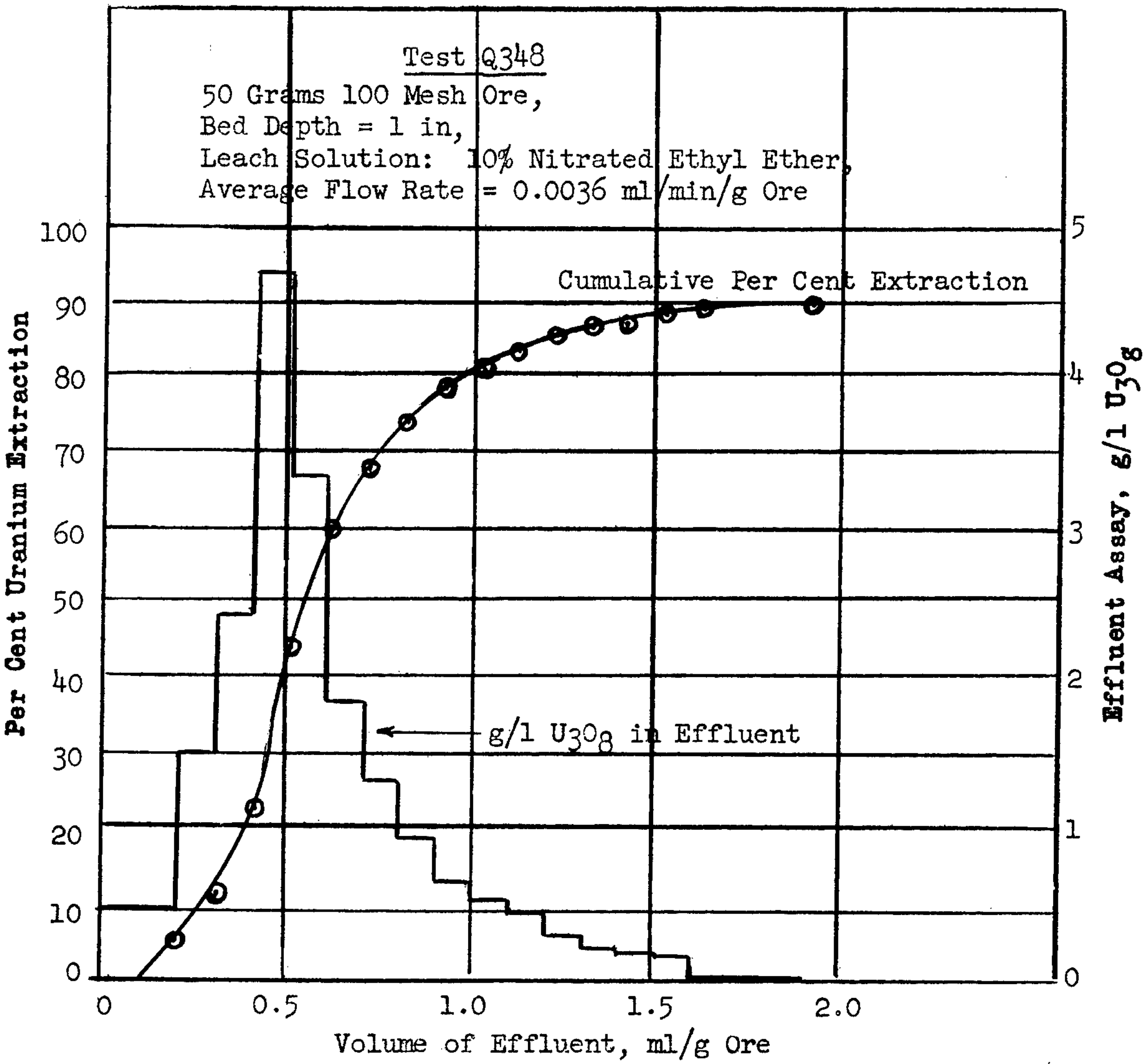

Figure 4. Percolation Leach of 100 Mesh Q3 Ore, Slow Flow Rate

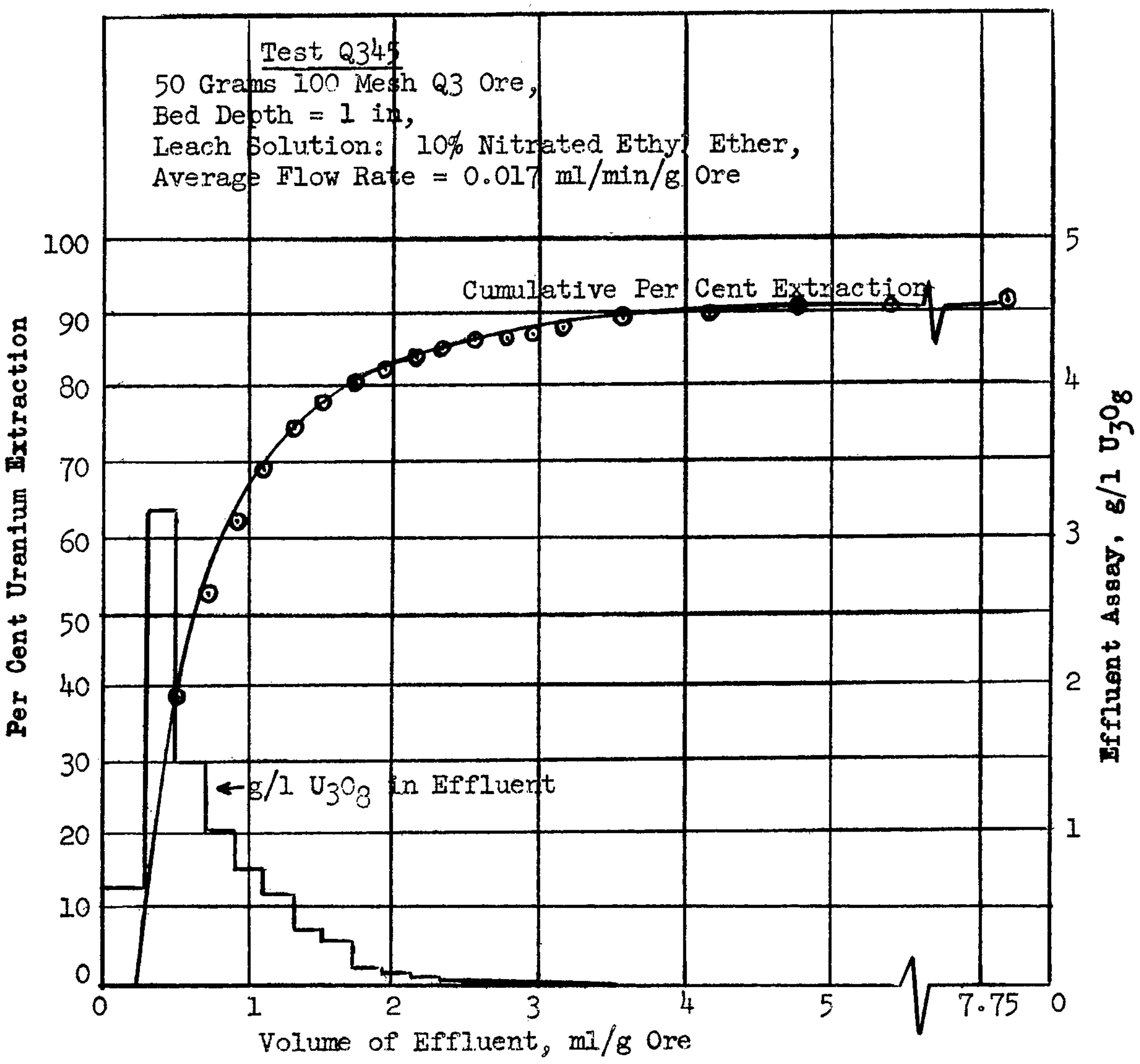

Figure 5. Percolation Leach of 100 Mesh Q3 Ore, Fast Flow Rate

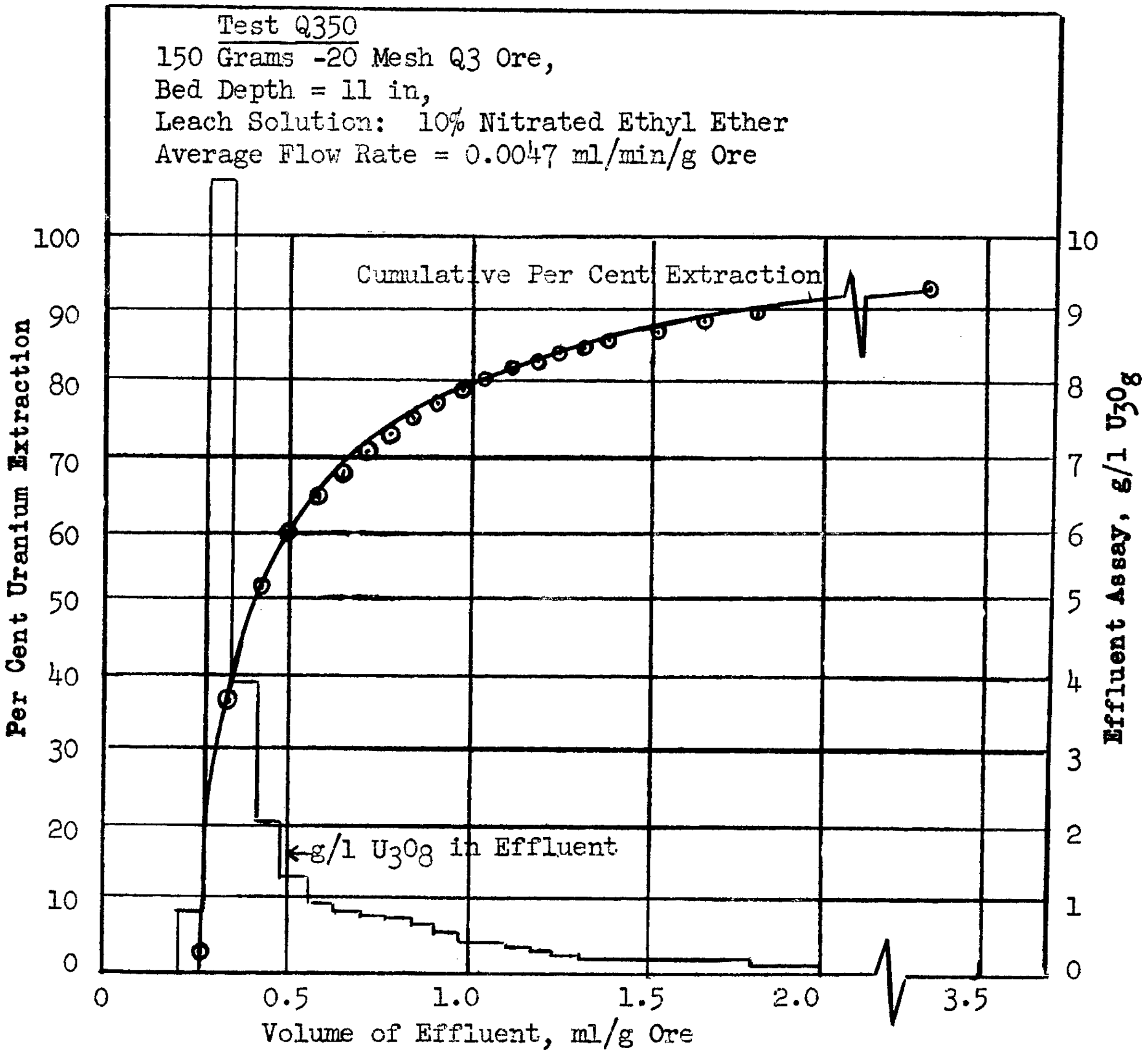

Figure 6. Percolation Leach of -20 Mesh Q3 Ore

V. SULFATION OF Q1 AND Q2 ORES PRIOR TO LEACHING

A number of lyometallurgical leach tests was run on Q1 and Q2 ores that had first been digested for 24 hours with sulfuric acid at room temperature. Sulfuric acid leaching had effected uranium extractions (MITG-253) higher than those obtained by direct lyometallurgical leaching of these ores and it was hoped that sulfuric acid digestion might solubilize additional uranium that could be extracted by the nitrated ether leach as it had on the carnotites (MITG-246).

Charges of 100 mesh ore were pulped in distilled water, Q1 at 43 per cent solids by weight and Q2 at 50 per cent solids by weight, the acid added and the slurry mixed in an open bottle on rolls for 24 hours. The final pH of the slurry was measured and the pulp dried at 80°C. Twenty-gram charges of the dried, digested material were then leached in a chamber with 1.5 ml/g of 5 per cent or 10 per cent nitrated ethyl ether for 1 hour, the pregnant liquor withdrawn and the cake given 2 displacement washes. The data are given in detail in Appendix B, and are presented graphically in Figures 7 and 8 where total uranium extraction is plotted against weight of H_2SO_4 used per ton of ore.

Uranium extraction from Q1 ore reached 82 per cent upon leaching for 1 hour with 5 per cent nitrated ethyl ether after digestion with 120 lb H_2SO_4 per ton of ore. This compares to a sulfuric acid leach extraction of 75 to 82 per cent achieved with 145 to 160 lb H_2SO_4 per ton of -10 mesh ore, leached at 40 to 50 per cent solids for six hours (MITG-253), and an extraction of 70 per cent by direct leaching of raw ore with 10 per cent nitrated ethyl ether.

Extraction of uranium from sulfated Q2 ore reached a maximum of 81 per cent upon leaching for 1 hour with 5 per cent nitrated ethyl ether, and 85 per cent with 10 per cent ether, after digestion with 50 to 65 lb H_2SO_4 per ton of ore. A sulfuric acid leach of -10 mesh Q2 ore with 60 to 70 lb H_2SO_4 per ton, at 50 per cent solids for 24 hours, leached 88 to 90 per cent of the uranium (MITG-253). Direct leaching with 10 per cent nitrated ethyl ether extracted 66 per cent of the uranium in 1 hour.

The curves of Figures 7 and 8 show that excess sulfuric acid seems to have an inhibiting effect on subsequent lyometallurgical leaching of the sulfated ore. This effect has also been observed in tests with Chatanooga shale[6] and carnotite ores. In the carnotite sulfation-leaching tests it was found that if the excess sulfuric acid were neutralized with calcium salts, the inhibiting effect was less marked and the uranium could be leached out in a shorter time.[7]

A modified sulfation technique worked out by Galvanek for carnotites[7] involves acid digestion at a high per cent solids followed by leaching of the damp product with an acetone-ether or alcohol-ether solution together with calcium nitrate as a nitrate source. This new technique has not yet been tested on these Marysvale ores, however.

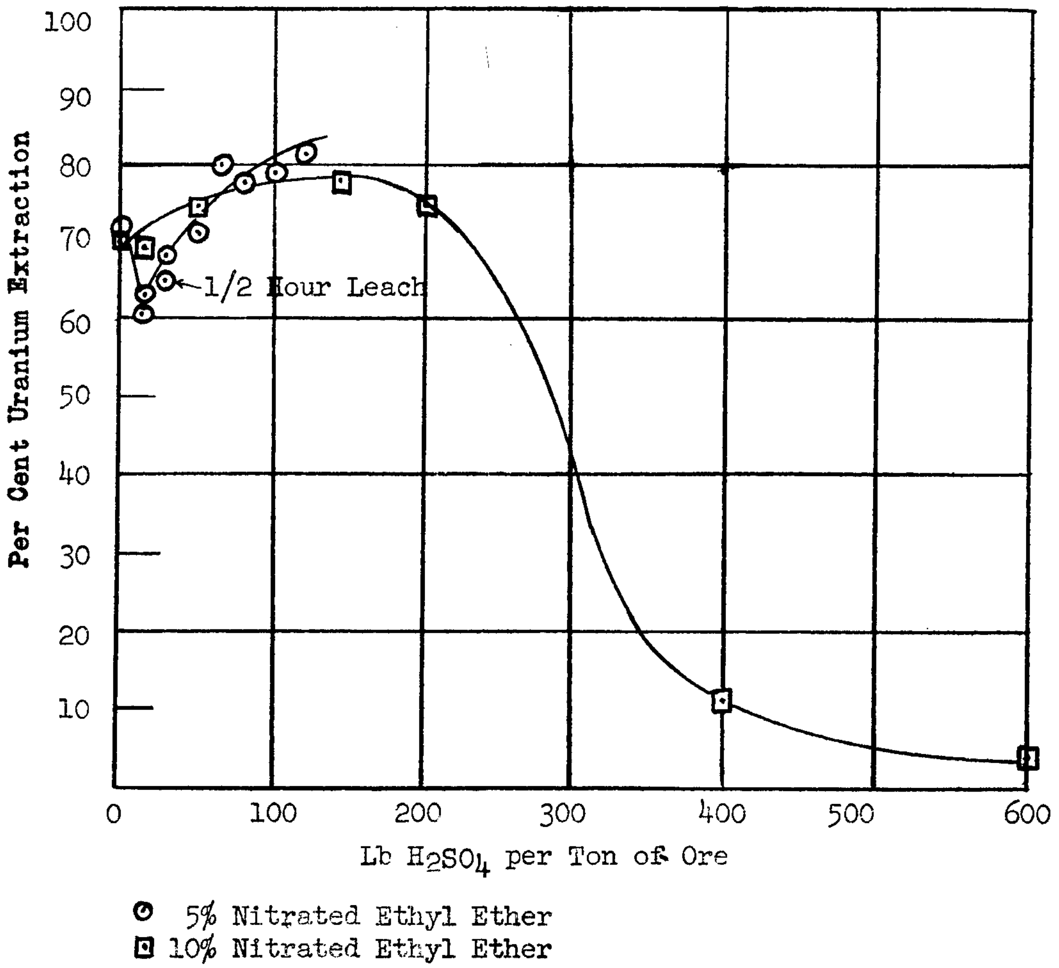

Figure 7. Leaching of Sulfuric Acid Digested Q1 Ore

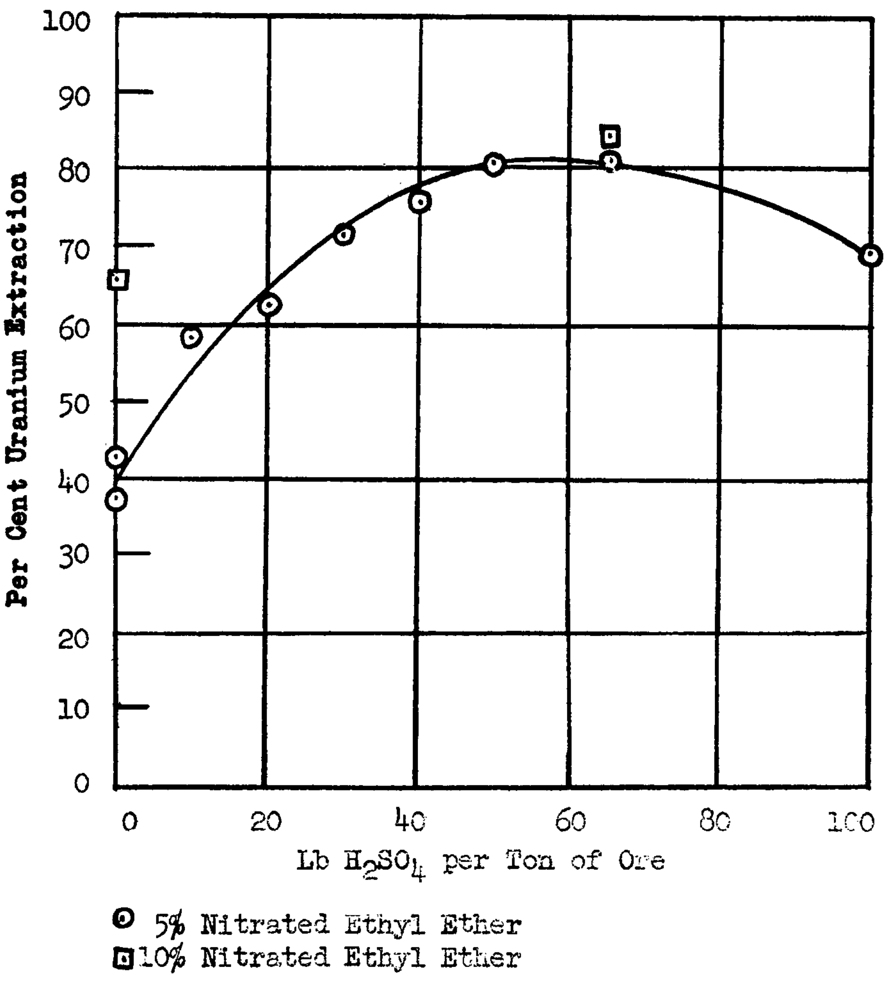

Figure 8. Leaching of Sulfuric Acid Digested Q2 Ore

VI. REAGENT CONSUMPTIONS

In order to cut down the costs of a lyometallurgical leaching process it will be necessary to recover a large amount of the relatively expensive ether and nitric acid which the process uses. Some preliminary tests to determine what reagent recovery could be expected had been run in connection with some earlier lyometallurgical test work[3/]. It was found that a minimum of 99 per cent of the ether and 90 per cent of the nitric acid could be recovered for recycling. The ether would be recovered from the leach residues and ammonia precipitate by vacuum flashing after filtration. The nitric acid would be recovered from the ammonia precipitate by heat decomposing the nitrates that had been washed out with water and collecting the evolved oxides of nitrogen in water. Heat decomposing of nitrates in the leach residues may or may not be economic.

Using those recovery figures, reagent-consumption data have been calculated for some of the better leach tests on each of the three ores. Table 6 is a summary of that data listed as pounds of reagent consumed per pound of U_3O_8 extracted. Included are consumption figures for sulfuric acid, where it was used, assumed to be economically unrecoverable for re-use. The cheapest uranium would, of course, be that produced from the higher-grade Q3 ore, which also gives the highest uranium extraction. Nitrate-consumption figures for the Q3 tests were based on the assumption that the nitrate appearing in the tails (5 per cent of the total) would not be recovered. Therefore, only 0.9 x 95 per cent, or 85.5 per cent of the total nitrate was considered recoverable for re-use. Nitrate determinations on the tails from the other tests were not made. Therefore, it was assumed for those tests that 90 per cent of the total nitrate was recoverable. That would mean, of course, heating the leach residues to decompose nitrates.

It must be remembered that these reagent requirements are calculated from the results of rather sketchy preliminary tests. They therefore do not necessarily represent optimum conditions.

Table 6. Reagent Consumptions per Pound of U_3O_8 Extracted

		Uranium Extraction		Reagent Consumption, lb/lb U_3O_8[1]		
Ore	Test No.	Per Cent	Lb U_3O_8 per Ton of Ore	Ether	Nitric Acid	Sulfuric Acid
Q1	Q121	82.0	1.80	21	12[2]	67
Q1	Q113-2	77.6	1.86	27	23	None Used
Q2	Q224	80.8	2.75	15	7.7[2]	18
Q2	Q220	81.1	2.43	17	8.8[2]	27
Q2	Q210	84.6	2.54	15	17	25
Q3	Q316-1	91.8	4.23	9.7	11[3]	None Used
Q3	Q327	92.0	4.05	11	11[3]	None Used

Note: Except where noted (footnote 2/) 10 per cent nitrated ethyl ether was used in the leaching.

[1] Assuming 99 per cent recovery of ether, 90 per cent recovery of nitric acid and no recovery of sulfuric acid for re-use.

[2] Five per cent nitrated ethyl ether used in these tests.

[3] Nitrate analysis on the leach tails from these tests showed that 5 per cent of total nitric acid used remained in tails. Consumption is based on recovery of 90 per cent of nitric acid appearing in pregnant and wash solutions.

VII. FILTRATION OF Q3 LEACH LIQUORS

A signal advantage of lyometallurgical leaching is the remarkable ease with which the organic solvent can be separated from the ore by filtration. In order to quantify filtration rates in lyometallurgical and hydrometallurgical processes so that they could be compared, leaches were run by each method on 50-gram charges of -20 mesh Q3 ore, and filtration rates measured under the same conditions of vacuum and cake thickness.

The filtration was carried out in a coarse-frit leach chamber, the filtrate being discharged into a standard volumetric receiver. The amount of ore used made a 1-inch filter cake, rather thick for hydrometallurgical filtration, but typical of laboratory scale lyometallurgical leach cakes. The vacuum was kept at 20 inches of mercury by means of an air bleeder and a five-gallon glass vacuum reservoir connected to a vacuum line. The vacuum was measured with a mercury manometer, and was easily controlled to within ± 1/8 inch. During the ether filtrations 3 ether-dry ice traps were placed in series in the vacuum line adjacent to the filtrate receiver to prevent passage of ether into the vacuum line.

The sulfuric acid leach was carried out for 12 hours in a rolling bottle at 50 per cent solids with 160 lb H_2SO_4 per ton of ore. The pulp was quantitatively transferred to the coarse frit chamber at the end of the leach, the pregnant liquor withdrawn until the top of the filter cake was just dry, and the cake then displacement washed 3 times with plain water. The lyometallurgical leach was carried out for 1 hour by agitation of 50 grams of -20 mesh ore with 1.5 ml/g of 10 per cent nitrated ethyl ether in a coarse-frit leach chamber. The pregnant liquor was withdrawn and the cake given 2 displacement washes with plain ether.

The data are summarized in Table 7. For comparison with the ether filtration only the first 2 washes of the sulfuric acid leach cake have been considered. The total time required for the ether filtration and washing was only 33.6 seconds as compared to 97.4 minutes in the sulfuric acid leach. The ether filtration rate, in terms of volume of filtrate per unit time, was 273 times faster than the hydrometallurgical leach rate. The final moisture content of the sulfuric acid leach cake was 25 per cent (wet basis). The 1-hour direct solvent leach on -20 mesh Q3 ore does not afford sufficient time to effect a 92 per cent uranium extraction as achieved on 100 mesh ore. A longer leach time would have raised uranium extraction above 90 per cent.

Table 7. Comparative Q3 Filtration Rates

Test No.	Type of Leach	% U_3O_8 Extraction[1]	Total Wash ml/g Ore[2]	Overall Filtration Rates[3]	
				lb/sq.ft/24 hr.	gal/sq.ft/24 hr
Q358	H_2SO_4[4]	92.3	0.80	100	17.4
Q360	Ether[5]	74.4	1.08	17300	4750
	Ratio of ether rate to H_2SO_4 rate:			173/1	273/1

[1] Based on calculated head assay.

[2] As 2 displacement washes of plain ether or water.

[3] Overall rates include time required for displacement washing of cake. Units are: lb of ore per sq.ft of filter per 24 hr, and gal of filtrate per sq.ft of filter per 24 hr.
Vacuum was 20" of mercury, and cake was 1 inch thick.

[4] 12-hr leach of -20 mesh ore at 50 per cent solids with 160 lb H_2SO_4 per ton; final pH was 0.77.

[5] One hour leach of -20 mesh ore with 1.5 ml/g of 10 per cent nitrated ethyl ether.

VIII. BIBLIOGRAPHY

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APPENDIX A

Table A1. Percolation Leach Data, Test Q345

Effluent Fraction	Effluent Vol., ml	Flow Rate, ml/min/g Ore	Total Vol. Effluent, ml/g Ore	Effluent Assay, g/l U_3O_8	Uranium Extraction, Cum. %[1]
1	15	0.0316	0.30	0.633	8.9
2	10	0.0174	0.50	3.18	38.6
3	10	0.0154	0.70	1.48	52.5
4	10	0.0126	0.90	1.04	62.1
5	10	0.0120	1.10	0.76	69.2
6	10	0.0145	1.30	0.60	74.9
7	10	0.0186	1.50	0.36	78.2
8	10	0.0158	1.70	0.26	80.6
9	10	0.0145	1.90	0.22	82.7
10	10	0.0160	2.10	0.18	84.4
11	11	0.0206	2.30	0.11	85.4
12	10	0.0206	2.52	0.088	86.2
13	10	0.0186	2.72	0.073	86.9
14	10	0.0166	2.92	0.072	87.6
15	10	0.0154	3.12	0.070	88.2
16	20	0.0142	3.52	0.013	89.4
17	30	0.0176	4.12	0.011	90.5
18	30	0.0188	4.72	0.0075	91.2
19	30	0.0198	5.32	0.0044	91.6
20	127	Pool plus Holdup	7.86	0.0040	92.0

Tails: 50.6 grams at 0.017 per cent U_3O_8; Calc. Head = 0.214 per cent.

Note: Bed was 1 inch deep, composed of 50 grams of 100 mesh Q3 ore; leach solution was 10 per cent nitrated ethyl ether. Total time of leach, 313 minutes. Leach was started with 21 ml (0.42 ml/g) of plain ether in voids of bed.

[1] Based on calculated head assay.

APPENDIX A

Table A2. Percolation Leach Data, Test Q348

Effluent Fraction	Effluent Vol., ml	Flow Rate, ml/min/g Ore	Total Vol. Effluent, ml/g Ore	Effluent Assay, g/l U_3O_8	Uranium Extraction, Cum. %[1]
1	10	0.00606	0.20	0.44	4.1
2	5	0.00364	0.30	1.48	11.0
3	5	0.00326	0.40	2.40	22.1
4	5	0.00334	0.50	4.68	43.9
5	5	0.00318	0.60	3.32	59.4
6	5	0.00370	0.70	1.84	67.8
7	5	0.00352	0.80	1.30	74.0
8	5	0.00330	0.90	0.92	78.2
9	5	0.00318	1.0	0.64	81.2
10	5	0.00334	1.1	0.52	83.6
11	5	0.00334	1.2	0.44	85.6
12	5	0.00434	1.3	0.28	87.0
13	5	0.00328	1.4	0.20	87.9
14	5	0.00322	1.5	0.18	88.8
15	5	0.00332	1.6	0.16	89.5
16	15	Pool plus Holdup	1.9	0.03	90.0

Tails: 51.7 grams at 0.021 per cent U_3O_8; calc. head = 0.215 per cent.

Note: Bed was 1 inch deep, composed of 50 grams of 100 mesh Q3 ore; leach solution was 10 per cent nitrated ethyl ether; total time of leach, 448 minutes. Leach was started with 21 ml (0.42 ml/g) of plain ether in voids of bed.

[1] Based on calculated head assay.

APPENDIX A

Table A3. Percolation Leach Data, Test Q350

Effluent Fraction	Effluent Vol., ml	Flow Rate, ml/min/g Ore	Total Vol. Effluent, ml/g Ore	Effluent Assay, g/l U_3O_8	Uranium Extraction, Cum. %1/
1	10	0.00754	0.067	---	---
2	10	0.00638	0.133	---	---
3	10	0.00580	0.200	---	---
4	10	0.00535	0.267	0.82	2.6
5	10	0.00519	0.333	10.7	37.1
6	12	0.00612	0.413	3.90	52.2
7	11	0.00608	0.486	2.05	59.4
8	12	0.00574	0.566	1.28	64.4
9	10	0.00543	0.634	0.92	67.4
10	10	0.00512	0.700	0.83	70.2
11	10	0.00515	0.767	0.78	72.5
12	10	0.00512	0.834	0.76	75.0
13	10	0.00518	0.900	0.62	77.1
14	10	0.00471	0.967	0.54	78.8
15	10	0.00387	1.03	0.44	80.2
16	10	0.00436	1.10	0.43	81.6
17	10	0.00504	1.17	0.39	82.8
18	10	0.00494	1.23	0.34	83.9
19	10	0.00489	1.30	0.29	84.8
20	10	0.00472	1.37	0.26	85.7
21	20	0.00448	1.50	0.22	87.2
22	20	0.00411	1.63	0.22	88.6
23	20	0.00383	1.77	0.205	89.9
24	30	0.00347	1.97	0.170	91.5
25	208	Pool plus Holdup	3.35	0.0021	92.9

Tails: 150.8 grams at 0.020 per cent U_3O_8; calculated head = 0.207 per cent.

Note: Bed was 11 inches deep, composed of 150 grams of -20 mesh Q3 ore; leach solution was 10 per cent nitrated ethyl ether; total time of leach, 415 minutes. Leach was started with 120 ml (0.80 ml/g) of plain ether in voids of bed.

1/ Based on calculated head assay.

APPENDIX B

Table B1. Sulfation - Leaching of Q1 and Q2 Ore

Ore	Test No.	Lb H_2SO_4 Per Ton Ore	Final pH	Weight Increase, Per Cent	Ether, % Nitric Acid by Volume	Wash Ether, ml/g Ore[1]	Per Cent U_3O_8	
							Calc. Head	Extraction[2]
Q1	Q121	15	4.7	--	5	1.15	0.11	63.8
Q1	Q123-1	15	4.7	--	5	1.35	0.12	61.9
Q1	Q121	30	4.1	--	5	1.85	0.11	69.0
Q1	Q123-2	30	4.1	--	5	1.40	0.12	65.7
Q1	Q115-1	50[3]	3.3	--	5	1.50	0.10	71.8
Q1	Q121	65	2.8	--	5	1.50	0.11	80.4
Q1	Q121	80	2.5	1.1	5	1.40	0.10	78.9
Q1	Q121	100	2.3	2.3	5	1.50	0.11	79.8
Q1	Q121	120	2.0	4.9	5	1.20	0.11	82.0
Q1	Q122-2	15	4.7	--	10	1.35	0.12	69.8
Q1	Q121	50[3]	3.3	--	10	1.90	0.12	75.0
Q1	Q110	145[4]	1.7	6.8	10	1.28	0.10	78.6
Q1	Q121	200[3]	1.1	7.8	10	1.55	0.094	75.3
Q1	Q121	400[3]	0.2	19	10	1.75	0.086	11.3
Q1	Q122-1	600[3]	0	33	10	1.60	0.076	4.2
Q2	Q224	10	4.9	--	5	1.35	0.15	58.3
Q2	Q224	20	4.3	--	5	1.25	0.15	62.9
Q2	Q224	30	3.3	--	5	1.50	0.16	71.9
Q2	Q224	40	2.4	--	5	1.20	0.17	75.9
Q2	Q224	50	2.1	--	5	1.45	0.17	80.8
Q2	Q220	65	2.3	3.2	5	1.46	0.15	81.1
Q2	Q210	65	2.3	3.2	10	1.34	0.15	84.6
Q2	Q224	100	1.1	1.5	5	1.50	0.16	69.2

Note: Except where noted sulfuric acid digestion was carried out on 65-gram charges 100 mesh Q1 ore at 43 per cent solids by weight, and 65-gram charges 100 mesh Q2 ore at 50 per cent solids, for 24 hours on rolls in open bottles at room temperature. Slurries were dried at 80°C, and 20-gram charges leached for one hour with 1.5 ml/gram of nitrated ether.

[1] As two displacement washes of plain ether, in amounts equal to holdup in the bed.

[2] Based on calculated head assay.

[3] Sulfation carried out on 60-gram charge.

[4] Sulfation carried out on 220-gram charge; ether leach done on 50-gram charge sulfated ore using 75 ml leach solution.