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The Beneficiation of a Low-Grade Uranium Ore

Frank P. Howald

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THE BENEFICIATION OF A LOW-GRADE URANIUM ORE

by

Frank P. Howald

A Thesis
Submitted to the Department of Mineral Dressing
In Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Mineral Dressing

MONTANA SCHOOL OF MINES
Butte, Montana
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ABSTRACT

The uranium in the Sponsor's Ore was proven amenable to the standard hydrometallurgical processes -- the acid leach and the carbonate leach. From an economical standpoint, the results of the heap-leaching and flotation studies were not favorable because of low recoveries. The possibility of employing an emulsion-induced selective flocculation flotation procedure still exists.
INTRODUCTION

The increasing importance of uranium in atomic research and the recent discoveries of extensive deposits of low-grade uranium ores in the Colorado Plateau have stimulated interest in the beneficiation of marginal ores. The purpose of this investigation was to determine if a commercial concentrate could be obtained from an ore received from the Juniper Oil and Mining Company.

Location:

The area where this deposit is located includes most of Township 17 South and the northerly part of Township 18 South, Range 73 West (6th P. M.) in Fremont County, Colorado. The area is about 16 miles northwest of Canon City and about 22 miles southwest of Cripple Creek, on the northwest trend of the Wet Mountains (see Figures I and II).

Geography:

The area is drained by the north, middle, and south branches of Tallahassee Creek and their tributaries, which join southeast of the map area and drain into the Arkansas River. Relief is commonly 500 to 1000 feet. The land surface is used exclusively for ranching and lumbering. Much of the area is timbered, in particular north slopes, which are covered with ponderosa pine. Pinion pine, scrub oak, and juniper are also common. Access to the area is by a network of graded and ungraded unsurfaced roads, most of which are shown on the map.

Geological Formations:

The oldest rocks in the area are a Pre-Cambrian igneous and metamorphic complex, including principally fine to medium grained biotite granites and gneisses, abundantly intruded by pegmatites and lesser quartz veinings. Commonly, the
RECONNAISSANCE GEOLOGIC MAP OF THE TALLAHASSEE CREEK MINING DISTRICT FREMONT COUNTY COLORADO

FIGURE I. TALLAHASSEE CREEK MINING DISTRICT
FIGURE II. SNOOPER GROUP
structural elements in the Pre-Cambrian rocks are steeply inclined. The granitic surface on which Tertiary deposits of the area rest is irregular, strongly suggesting an ancient topography having several hundreds of feet of fairly gentle relief.

The oldest Tertiary formation is an ancient arkosic alluvium which apparently occurs locally in old drainage courses on the granitic surface. This alluvium is semi-consolidated to lithified and consists of fine to course arkose sand interbedded with cobble and boulder conglomerates, having clasts up to 18 inches in diameter. These clasts are smooth and well-rounded and they include a high percentage of Pre-Cambrian rocks, though cobbles of volcanic rocks and tuffaceous debris are also abundant. This unit is locally at least several scores of feet thick, but in many places it is absent.

Onto a surface of Pre-Cambrian granitics and Tertiary arkose and conglomerate poured the earliest of the Tertiary volcanic rocks of this area: a sanidine-bearing rhyolite flow having numerous vesicles up to 3 or 4 inches long. The rock is tan, grading locally to various purplish and reddish shades of tan. The flow consists of massive beds up to 8 feet thick and the unit as a whole ranges in thickness from about 250 feet to a feather edge. In general, it is thicker to the north. Its upper surface is gently undulating.

A thick layer of andesite breccia overlies the rhyolite and locally, where the rhyolite is absent, it lies on the arkose or directly on Pre-Cambrian rocks where both the rhyolite and arkose are missing. The breccia weathers to a dark reddish brown color, and it is composed of angular fragments, usually less than 4 inches long in dimension. The unit ranges in thickness from a few score feet to a few hundred feet, and, like the underlying unit, it is generally thicker toward the north. The upper part of this andesite formation includes several score
feet of massive very dark brown basalt, vesicular chocolate brown basalt, and rhyolitic tuffs.

The fourth and uppermost formations of the Tertiary volcanic pile in this area consists of rhyolitic flows and tuffs aggregating several hundreds of feet in thickness. The base of this acidic sequence is a discontinuous layer of cliff-forming felsite. Above the felsite, rhyolitic breccias, welded tuffs, massive flows, and dark-colored scoria are all common. Most of the higher parts of the sequence are present only in the southern part of this map area.

The Quarternary deposits in this area are recent alluvium along the modern drainage lines, and landslide debris. The alluvium is usually less than 10 feet thick, and contains highly angular clasts in a poorly sorted matrix.

Landslide debris is fairly common in the area. In all cases the material that has slid is dominantly the upper rhyolite formation. Possibly this is because permeable tuffs are present near the lower contact.

Uranium Occurrence:

The geologic formations outcropping on the Juniper Oil and Mining Company properties are the same as those in the surrounding area. They are discussed in the general geology section of this report.

The host rocks of uranium mineralization are (1) Tertiary arkose and conglomerate, (2) Tertiary andesite flow breccia, and (3) possibly Pre-Cambrian gneiss. The arkose and conglomerate formation is probably the most extensive host rock for uranium minerals in this area. On the basis of field examination, the radioactive minerals are autunite and a mineral identified as uraninite by the Atomic Energy Commission. In the Smaller Brothers mine and numerous outcrops, the autunite and uraninite are concentrated in the more carbonaceous portions of the arkose formation.
A natural affinity of uranium for carbon is a well established geologic condition in many radioactive rocks throughout the world.

The base and lower portion of the andesite flow breccia are mineralized at several localities along altered and fractured zones. The uranium minerals in the andesite are autunite and uraninite. Moderately radioactive gneiss in a highly fractured, altered and oxidized condition is exposed about 2,000 feet northeast of the Snooper claims. The radioactive mineral present in the gneiss was not determined.

Uranium mineralization appears to be controlled by:

1. fracturing and faulting and primary porosity giving access to mineralizing solutions, and
2. concentrations of carbonaceous matter.

The source of the uranium in the area appears to be the basal rhyolite and closely associated (in space and time) volcanic rocks. This conclusion is based on evidence summarized as follows:

1. Some of the mineralized zones occur in the arkose sand and conglomerate immediately underlying the rhyolite.
2. Others occur in the andesite breccia immediately overlying the rhyolite.
3. Still others seem to lie in volcanic and arkosic rocks rich in rhyolitic ash and breccia, and
4. The basal rhyolite flow itself is about twice as radioactive as other rocks in the area.

Thus, uranium-bearing solutions and vapors probably originated in the rhyolite magma. They probably percolated from the rhyolitic rocks into the underlying arkose and overlying andesite, precipitating uranium minerals in carbonaceous
zones and in favorable fractures.

As suggested by the radioactive gneiss mentioned above, some mineralizing solutions and vapors may have moved toward the surface from the deep sources of volcanism directly through the Pre-Cambrian into overlying rocks. Ground water may have later effected secondary uranium redistribution and mineralization (autunite).

The age of uranium mineralization is believed to be contemporaneous with and closely following the rhyolite extrusion, which is tentatively assigned to the Miocene. The secondary uranium minerals may have formed at a much later date.

Marketing Requirements: 7

Uranium ores are marketed on the basis of uranous oxide (U₃O₈) content. The specifications that the uranium ore has to meet are those established by the Atomic Energy Commission (AEC). Ores as low as 0.1 per cent U₃O₈ are purchased depending upon the characteristics of the ore. Due to government regulation of U₃O₈ content of mill feeds, many low grade ores are purchased for mixing with high grade ores to meet the government's specifications.

The government's basic price of $16.00 per pound depends upon the percentage of thorium and vanadium present in mill concentrate, which normally runs 75-83 per cent U₃O₈. With increasing percentages of thorium and vanadium above 0.1 per cent of the Atomic Energy Commission's specifications, penalties become very heavy.
ORE PREPARATION

The ore tested was a low-grade uranium ore from Cripple Creek, Colorado. The sample was submitted by the Juniper Oil and Mining Company of Denver, Colorado.

Receiving the Sample:

Three sample sacks of the Sponsor's ore were received on November 12th, 1956. They weighed a total of 280 pounds and consisted of pieces approximately 1/2 to 1 inch in diameter, with a large percentage of fines. The method of sampling is unknown, but all of the sample came from the same pit -- the Dickson-Snooper number 11 ore body -- about 30 feet from the surface. Apparently the sample was fresh, because there was no visual evidence of weathering.

The average specific gravity of the ore was determined by use of the Taggert formula\(^{12}\) for specific gravity and was recorded as 2.02. The moisture content was 11.3 per cent.

Comminution of the Sample:

One sack of ore was prepared for grinding studies, amenability tests, and flotation studies. The remainder of the ore was retained for heap-leaching studies.

The ore was crushed until a product of all minus 8 mesh was obtained. Figure III shows the crushing circuit. The clay minerals and the moisture in the ore caused clogging during crushing and screening.
FIGURE III. CRUSHING CIRCUIT
Sampling and Chemical Analysis:

After comminution to minus 8 mesh, the ore sample was thoroughly mixed and passed through a Jones riffle until an adequate sample was obtained for assay. This sample was reduced to all minus 65 mesh in a Braun pulverizer, thoroughly mixed, and analyzed by Mr. Clem J. Bartzen, analyst for the Montana Bureau of Mines and Geology. The method employed by Mr. Bartzen was a gravimetric analysis. This method is extremely difficult in that it requires seven separate precipitations and filtrations and is insufficiently sensitive for low-grade ores. For example, the head assay reported by the Sponsor was 0.10 to 0.15 per cent U$_3$O$_8$ and the results obtained here were from 0.04 to 0.05 per cent U$_3$O$_8$. The Sponsor was informed of this low assay. Further written correspondence revealed that the Sponsor's assays were only approximations and the results obtained by Mr. Bartzen were probably more nearly correct.

Mineralogical Examinations:

The various size fractions obtained by screen analysis of the grinding circuit product were examined with a binocular microscope. Orthoclase, plagioclase (albite), calcite, quartz and uraninite, in order of abundance, were the only minerals which could be identified as being present in any quantity. Very minor amounts of pyrite and magnetite were also observed.

Grinding Studies:

The Sponsor desired that the product for testing have the following specifications: all minus 60 mesh, 70 per cent minus 200 mesh, and 50 per cent minus 325 mesh, since this size distribution is desirable for carbonate leaching, in that it allows adequate particle surface for sufficient leaching without the presence of considerable slimes.
Four grinding tests were conducted in a Galigher laboratory rod mill at 40 per cent solids with a full rod load (see Appendix I). A time of 2½ minutes was found to be satisfactory. This grind was employed for all the carbonate leach tests.
AMENABILITY TESTS

Amenability tests are designed to evaluate the two major methods of leaching uranium selectively away from the gangue matrix of the uranium minerals — the carbonate leach and the acid leach. To obtain a knowledge of the characteristics of the Sponsor's ore, the standard amenability procedures used by the Anaconda Company at Grants, New Mexico, were employed. Such data apply to heap leaching and have a bearing on the contemplated flotation studies, in terms of possible activation procedures.

Carbonate Leach:

The carbonate leach is best suited for ores that are high in calcite (CaCO₃), dolomite (Mg, CaCO₃), and other acid consumers. The basis for this method is the fact that secondary uranium minerals are readily soluble in a hot carbonate solution. The reaction forms a soluble uranyl-carbonate complex.

Procedure:

A leaching solution was prepared and was found to contain 71.5 gm per liter Na₂CO₃ and 22.0 gm per liter NaHCO₃. This solution was added to two 1000-gm samples to prepare pulps of 40 per cent solids for grinding. Five tenths of a pound of KMnO₄, 10 pounds of MnO₂, and 0.5 pound of MgO per ton of dry solids were added to both pulps upon removal from the rod mills. One sample was placed in a laboratory autoclave (see Figure IV) to be leached at 107°C and 30 psi for 6½ hours. The remaining sample was leached at atmospheric conditions for 6½ hours. A laboratory agitator was employed to leach at these conditions. After leaching, an addition of 0.1 of a pound of Dow's Reagent 2610 (Separan) was added for the purpose of flocculation. After proper flocculation, each pulp was filtered and

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FIGURE IV. LABORATORY AUTOCLAVE
then washed several times with water.

Complete assays were made of the "pregnant solution" and the tailing, and necessary calculations were made.

Results:

The results of the carbonate leaching were very satisfactory, indicating that the Sponsor's ore was amenable to carbonate leaching. From Appendix II, attention is drawn to test number two. Atmospheric conditions alone resulted in 63.9 per cent recovery. This is unusually high, the average recovery from these conditions being from 35 to 40 per cent. Test number three, the autoclave leach, achieved 91.6 per cent recovery. This can be regarded as an average expectable recovery under the given conditions.

Acid Leach:

The acid leach is best suited for uranium ores low in acid consumers, such as calcite (CaCO₃) and dolomite (Mg, CaCO₃). The basis for this method is the fact that secondary uranium minerals are very soluble in a sulfuric acid solution, forming soluble uranyl and uranous sulfates.

Procedure:

A 500-gm sample of ore all minus 65 mesh in size, and 500 gm of water were placed in a beaker and agitated until all the particles were in suspension. Sulfuric acid was added as an oxidant by means of a burette. A one-milliter portion was added at 2 to 5 minute intervals at the start of the leach until a stable pH of 1.1 was obtained. The aim was to neutralize substantially all the carbonates at a pH of 2.0 or higher, so that the gypsum was formed before any uranium went into solution. Sulfuric acid was added as required to maintain the
pH from 1.05 to 1.10 throughout the leaching process. Ten pounds of MnO₂ and 0.5 pound of KMnO₄ per ton of dry solids were added during the leaching period in increment amounts. Complete leaching required 9½ hours.

At the completion of the leaching, Dow Reagent 2610 (Separan) was added for the purpose of flocculation. After proper flocculation, the pulp was filtered and then washed several times. Assays were made of the pregnant solution and tailing so that the necessary calculations could be made.

Results:

The Sponsor's ore is amenable to an acid leach. The recovery of 92.9 per cent was an average expectable recovery, but the sulfuric acid consumption was rather high, 292 pounds per ton, compared to other ores requiring from 190 to 220 pounds of sulfuric acid per ton.²

Conclusion:

A comparison of the results shows a definite advantage of the carbonate leach over the acid leach on the Sponsor's ore. The carbonate leach had a lower reagent consumption (see Appendix II) than the acid leach with only a one per cent difference in recovery.

Even with the satisfactory recoveries, the economic factors in application would depend largely upon the grade of the Sponsor's ore. The purpose of the projects that followed were to collect data that would permit a better evaluation of these factors.

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HEAP LEACHING STUDIES

A survey of the literature was conducted to obtain a more thorough knowledge of heap leaching processes now in use. The Phelps Dodge heap-leaching process at Bisbee, Arizona seemed to be the most efficient process in use at the present time.

Field Heap Leaching: 6

In initiating a heap-leach process, ground with a slight slope of 3-5 degrees is selected. It is cleared of any growths and is then rolled and packed with clay or slimes to make it as nearly waterproof as possible. Large boulders of ore are selected for building culverts and cross culverts for drainage and ventilation purposes, and the drainage is directed to a common point. The ore, without crushing or other preparation, is then carefully piled on this prepared area. In some cases, it has been found helpful to classify material to the extent of placing the coarsest material on the bottom and the finest on top as an aid to both ventilation and solution circulation. The top of the ore pile is provided with distributing trenches. Solution containing the proper solvent is directed over the ore pile. The solution from the bottom of the pile is directed to a pump, from which it is pumped to the precipitation tank. After precipitation, the barren solution is recycled to the leach pile.

Laboratory Heap Leach:

The problem of developing a laboratory method of heap leaching was to simulate actual field conditions. To maintain these conditions, the following system was constructed and employed:
Nonpermeable Bed: Lucite was chosen for the nonpermeable bed because it is:

1. Chemically inert with the chosen solvents,
2. Nonpermeable by the chosen solvents, and
3. Strong.

The leaching table of lucite was constructed with a slight slope. A three degree slope was deemed adequate to allow free drainage of the leaching solution from the ore pile. By means of guides, the leaching solution was directed to a reservoir to be pumped back to the leaching table (see Figure V).

Pumping System: The pumping system was designed to prevent an introduction of foreign ions into the system. The pump was stainless steel and rubber tubing was employed to circulate the solutions from the reservoir to the leaching table.

Precipitation System: If a heap leach could be applied to the Sponsor's ore, a precipitation system employing a suitable resin could be used. Resin placed upon a wire screen of a mesh to retain it would adsorb the soluble uranium present in the leaching solution. The use of a resin would allow a continual flow of the leaching solution with only minor adjustments of reagent concentration.

Procedure:

For each test, a five-pound sample of as-received ore was used. The coarse material was placed on the bottom to build culverts for drainage and ventilation, and the finest placed on top. The leaching solution was circulated continuously through the ore for a pre-determined length of time depending upon the extraction power of the solvent used. At 5 or 10 hour intervals, samples were taken, the flow rate determined, and the volume of the leaching solution measured.

At the completion of the leach, the tailing was removed, dried, and sampled for assay.
FIGURE V. HEAP-LEACH APPARATUS
The following solutions were chosen as possible solvents in heap leaching:

1. Water
2. Normal sodium carbonate and 0.25 N sodium bicarbonate solution with oxidants (MnO₂ and K₂MnO₄).
3. Sulfuric acid solution (0.705 N) with oxidants (MnO₂ and K₂MnO₄).

The bases for choice were the factors of economy and the information obtained from the amenability tests. Water would be the cheapest solvent available if applicable, and a carbonate solution and a sulfuric acid solution were known solvents for the uranium present in the Sponsor's ore.

Results: The results are presented in Table I.

*TABLE I. HEAP LEACHING RESULTS*

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Recovery</th>
<th>Time</th>
<th>Flow Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>%U₃O₈</td>
<td>hr/5lb</td>
<td>yr/ton</td>
</tr>
<tr>
<td>Water</td>
<td>0.875</td>
<td>50</td>
<td>2-1/4</td>
</tr>
<tr>
<td>NaCO₃</td>
<td>6.45</td>
<td>40</td>
<td>1-3/4</td>
</tr>
<tr>
<td>H₂SO₄</td>
<td>11.05</td>
<td>40</td>
<td>1-3/4</td>
</tr>
</tbody>
</table>

* see Appendix III for the complete results.

Discussion:

Attention is drawn to Figure VI. The carbonate solution required 20 hours before reacting with the U₃O₈ in the Sponsor's ore, but once the reaction began, it proceeded at approximately the same rate as sulfuric acid, which reacted
immediately. From the sudden rise and leveling off of the curves for the carbonate and the acid solutions, there seems to be a certain amount of $\text{U}_3\text{O}_8$ available on or near the particle surface.

The following factors could have caused the low recoveries obtained:

1. Swelling of the clay.
2. Flocculation of the ore.
3. Solvents employed.

When wetted by the solvent, the clay in the ore swelled approximately four times in volume and the top of the heap became slippery. The swelling could have prevented a satisfactory percolation of the solvent by lowering the permeability of the ore.

Within 2 to 3 minutes, flocculation of the finer material present in the ore occurred. The flocs increased in size very rapidly until they were approximately $1/8 - 1/4$ of an inch in diameter. With flocculation, the total surface area in immediate contact with the solvent would be lowered, probably resulting in lower recoveries.

With advancements in the knowledge of the hydrometallurgy of uranium being obtained through the continuous research by the Atomic Energy Commission, a more suitable solvent may be found which could be applied to a heap-leach process.

**Conclusion:**

Considering the length of time required and the low recoveries obtained, it is concluded that this method of leaching the Sponsor's ore would not be economical with the solvents employed.
The flotation studies concern an emulsion-induced selective flocculation procedure. This approach is based upon experience gained in the Mineral Dressing Laboratories on metallic oxides. This application of emulsion techniques for up-grading uranium-bearing minerals is not without precedent, for a limited amount of work is mentioned in the literature.

**Effect of pH:**

To initiate the study, an emulsion was chosen that had given excellent results in manganese dioxide flotation. The following is the composition of the emulsion chosen:

- 70 per cent diesel oil
- 20 per cent saponified Aliphat 44E
- 10 per cent Oronite

The quantity of emulsion used was 50 pound per ton. Four tests were conducted with this emulsion to establish the effect of pH on flotation results. The range of pH to be studied was limited by the results obtained by B. C. Mariacher, who employed a similar emulsion. He established that a pH of 6.0 resulted in the best recovery. A lower pH resulted in improving concentrate grade, but was not suitable because of a lower $U_3O_8$ recovery.

A pH range from 5.0 to 6.5 was chosen. Hydrofluoric acid was used to regulate the pH because of results obtained with this acid in manganese dioxide flotation and because of the action of fluorides in depressing quartz. One test was conducted using $Na_2CO_3$ to adjust the pH to 8.0 because of the possibilities of $Na_2CO_3$ as an activator under the given conditions.
Effect of Emulsion Composition:

The emulsion compositions studied are given in Table II. A pH of 6.0 was used for the three flotations because the results of the pH study were not available and the results obtained by Mariacher at a pH of 6.0 were the most favorable.

<table>
<thead>
<tr>
<th>Test No.</th>
<th>lb/ton</th>
<th>Diesel Oil, %</th>
<th>44E, %</th>
<th>Cronite, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>200</td>
<td>50</td>
<td>90.0</td>
<td>5.0</td>
<td>5.0</td>
</tr>
<tr>
<td>201</td>
<td>50</td>
<td>85.0</td>
<td>10.0</td>
<td>5.0</td>
</tr>
<tr>
<td>216*</td>
<td>50</td>
<td>42.9</td>
<td>42.9</td>
<td>14.2</td>
</tr>
</tbody>
</table>

*Emulsion composition employed by Mariacher

Contact Angle Studies:

During a survey of the present literature concerning uranium, information was found in regard to the impurities often occluded in metallic uranium. Physical metallurgists employed by the Atomic Energy Commission established that nitrogen was the most common impurity occluded in metallic uranium. This occlusion of nitrogen is probably due to its affinity for uranium. With this thought in mind and with the cooperation of the National Science Foundation, a study of some of the nitriles (RCN) on prepared uranninite samples was conducted.

Isonitrile $\text{C}_{12}$ and Duomeen 12 gave the most favorable results (see Appendix V). High contact angles of 75 degrees were encountered at pH's of 9 and 11 for isonitrile $\text{C}_{12}$ and at pH's of 7 and 11 for Duomeen 12. This demonstrates that
these two reagents have a high degree of surface reactivity with uraninite at the stated pH values. The other nitriles studied, isonitrile C₈ and C₁₀ and n-valeronitrile, did not prove too favorable, although isonitrile C₁₀ had a contact angle of 60 degrees at a pH of 11 (see Appendix V). The possibilities of isonitrile C₁₂ and Duomeen 12 as collector reagents in an emulsion system are very favorable.

Procedure:

For each test, a five minute grind was employed on a 400-gm sample of ore, eight mesh in size. (See Appendix I, Test I). The emulsions were prepared by weighing the reagents and passing them through an emulsifier. The seven flotation tests were run employing a standard flotation procedure, Figure VII.
pH Modifier  →  Pulp Conditioned  →  Emulsion

7½ min at 2200 rpm → Flotation

at 1200 rpm → Rough Tailing

Rough Concentrate

Cleaning Operation

2 cleanings at 1200 rpm → Middling

Cleaned Concentrate

FIGURE VII. STANDARD FLOTATION PROCEDURE
Results:

For a rough determination of the results, the radioactivity of each weighed product was measured. This method failed in obtaining an estimate of the U$_3$O$_8$ content, but showed a difference between the radioactivity of the concentrates and tailings. The count of the concentrates was 140–200 and the tailings from 100–120. The background count was 80.

A microscopic examination was made of the concentrates. Orthoclase, pyrite, magnetite, quartz and uraninite, in order of abundance, were the only minerals identified as being present in any quantity. There was a definite concentration of pyrite and magnetite, but uraninite was in doubt, because of the difficulty in identification.

Test number 201, which employed an emulsion with a high percentage of diesel oil (85 per cent) showed the best results. A concentrate assaying 0.26 per cent U$_3$O$_8$ was obtained with a recovery of 30.5 per cent (See Appendix IV).

In the pH study, test number 100 was the most favorable at a pH of 6.5. A concentrate assaying 0.27 per cent U$_3$O$_8$ was obtained with a recovery of 26.1 per cent.

Discussion:

In each test, during the cleaning operations, the froth was characteristic of a sulfide float; it was bronze in color and very heavy. The end-point of each operation in every test was very definite with the appearance of a white froth.

In test number 100, flocculation occurred in the roughing operation at a pH of 7.0 (See Appendix IV). In the other six flotation tests where the pH of the rougher was not at 7.0, flocculation did not occur until the cleaning operations.
The pH of the cleaning operations of several tests was measured, and proved to be from 6.9 to 7.0. When flocculation did occur, the flocs became very large in size, approximately 1/4-1/3 of an inch in diameter.

The results of the pH study agreed with Mariacher's conclusion, except better results were obtained at a pH of 6.5 rather than 6.0.

From an economical standpoint, the results from this study were not favorable because of the low recoveries. The grade of the concentrates obtained from the flotation tests definitely establishes the possibility of an emulsion-induced selective flocculation procedure on the Sponsor's ore, if a more surface reactive and selective collector is employed. Isonitrile C\textsubscript{12} and Duomeen 12 could possibly be the collectors required.

Conclusion:

The following are conclusions concerning an emulsion-induced selective flocculation procedure on the Sponsor's ore:

1. Flocculation occurred near a pH of 7.0 with the emulsions used.
2. A pH of 6.5 resulted in the highest recovery in the pH range studied.
3. Economically, the employed emulsion system is not favorable because of low recoveries.
4. The grade of the concentrates indicates the possibility of this procedure.
5. The possibilities of isonitrile C\textsubscript{12} and Duomeen 12 as collector reagents in an emulsion system are very favorable.
ACKNOWLEDGMENTS

The author wishes to acknowledge the kind help and assistance received from Professor Donald W. McGlashan and Mr. Robert R. Beebe, of the Mineral Dressing Department; Mr. Clem J. Bartzen, analyst for the Montana Bureau of Mines and Geology; Mr. John E. Blixt, President, Juniper Oil and Mining Company; and fellow student, Au-Ngoc-Lieu.
BIBLIOGRAPHY


5. Grose, L. T., Geology of Tallahassee Creek Mining District, Fremont County, Colorado, Juniper Oil and Mining Company, October, 1955.


11. National Science Research Foundation Grant, Montana School of Mines, unpublished.

APPENDICES

Appendix I.  Grading Analysis
Appendix II. Amenability Tests
Appendix III. Heap-Leach Studies
Appendix IV. Flotation Tests
Appendix V. Contact Angle Studies
A standard load of a Galigher Laboratory Rod Mill was employed, Figure VIII.

The load was as follows:

- 8 - 3/4" rods
- 8 - 5/8" rods
- 8 - 1/2" rods

Total weight of 8586 gms.

FIGURE VIII. GALIGHER ROD MILL
### TEST NO. 1

**Conditions:** 40% solids, standard load

**Time:** 5 minutes

<table>
<thead>
<tr>
<th>Tyler Mesh Size</th>
<th>Wt Between Sieves</th>
<th>% Between Sieves</th>
<th>Cum % on each sieve</th>
<th>% Passing</th>
</tr>
</thead>
<tbody>
<tr>
<td>48</td>
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<td>0.0</td>
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### TEST NO. 2

**Conditions:** 40% solids, standard load

**Time:** 4 minutes

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<th>Wt Between Sieves</th>
<th>% Between Sieves</th>
<th>Cum % on each sieve</th>
<th>% Passing</th>
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<td>17.7</td>
<td>37.9</td>
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</tr>
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<td>62.1</td>
<td>100.0</td>
<td>0.0</td>
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<td>426.9</td>
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TEST NO. 3

Conditions: 40% solids, standard load

Time: 3 minutes

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<th>Tyler Mesh Size</th>
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<th>% Between Sieves</th>
<th>Cum % on each sieve</th>
<th>% Passing</th>
</tr>
</thead>
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<td>0.0</td>
<td>100.0</td>
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</tbody>
</table>

TEST NO. 4

Conditions: 40% solids, standard load

Time: 2½ minutes

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<th>% Between Sieves</th>
<th>Cum % on each sieve</th>
<th>% Passing</th>
</tr>
</thead>
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<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
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<tr>
<td>100</td>
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<td>86.2</td>
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<td>-325</td>
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4-A
<table>
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<tr>
<th>Test No.</th>
<th>Description</th>
<th>Wet or Vol. % Assay</th>
<th>Moisture Content</th>
<th>Recovery</th>
<th>Tp/lun</th>
</tr>
</thead>
<tbody>
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<td>60-60 1000</td>
<td>0.50 0.50 0.40</td>
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<td>90</td>
<td>16.4</td>
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<td>16.4</td>
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**Material Recovery Tests**

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<th>Sample</th>
<th>Water Mesh</th>
<th>KNO3</th>
<th>N2O2 (AIR)</th>
<th>Sample</th>
<th>Water Mesh</th>
<th>KNO3</th>
<th>N2O2 (AIR)</th>
<th>Sample</th>
<th>Water Mesh</th>
<th>KNO3</th>
<th>N2O2 (AIR)</th>
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</thead>
<tbody>
<tr>
<td>Test No.</td>
<td>Description</td>
<td>Wt or Vol</td>
<td>Weight (mesh)</td>
<td>Oxidants (lb/ton)</td>
<td>Solids %</td>
<td>Time</td>
<td>Temperature</td>
<td>Pressure</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>---------</td>
<td>-------------</td>
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<td>---------------</td>
<td>------------------</td>
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<td>-------------</td>
<td>----------</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>A-1</td>
<td>Free Solution residue</td>
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<td>Atmospheric</td>
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<td></td>
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<td></td>
<td>Mt or Vol</td>
<td>2.024</td>
<td>0.033 g/l</td>
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<td></td>
<td></td>
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<td></td>
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<td>Assay</td>
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<td>0.0520</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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<td></td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>U3O8 content</td>
<td>0.05820</td>
<td>0.0320</td>
<td></td>
<td></td>
<td></td>
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<td>Recovery</td>
<td>0.92.9</td>
<td>0.71.1</td>
<td></td>
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<td></td>
<td></td>
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<td></td>
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<tr>
<td></td>
<td>Reagent Consumption</td>
<td>292 g</td>
<td>H2SO4 (lb/ton)</td>
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<td></td>
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<td></td>
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</table>
APPENDIX III

HEAP-LEACHING STUDIES
Test number 1 - Water Leach

Leaching Solution:

Water

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Time (hr)</th>
<th>Rate of Flow (ml/min)</th>
<th>Volume (ml)</th>
<th>Assay $U_3O_8$ content (gm)</th>
<th>Recovery %</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
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<td>present</td>
<td>added</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>5</td>
<td>250</td>
<td>6000</td>
<td>1000</td>
<td>7.5</td>
</tr>
<tr>
<td>2</td>
<td>10</td>
<td>245</td>
<td>6800</td>
<td>2000</td>
<td>7.5</td>
</tr>
<tr>
<td>3</td>
<td>15</td>
<td>275</td>
<td>7109</td>
<td>1000</td>
<td>7.5</td>
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<tr>
<td>4</td>
<td>20</td>
<td>270</td>
<td>7218</td>
<td>1000</td>
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</tr>
<tr>
<td>5</td>
<td>25</td>
<td>275</td>
<td>7327</td>
<td>2000</td>
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<tr>
<td>6</td>
<td>30</td>
<td>280</td>
<td>7436</td>
<td>-</td>
<td>8.5</td>
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<td>7</td>
<td>35</td>
<td>240</td>
<td>7327</td>
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<td>8</td>
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<td>350</td>
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<td>-</td>
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<td>45</td>
<td>290</td>
<td>5040</td>
<td>1000</td>
<td>20.0</td>
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<tr>
<td>10</td>
<td>50</td>
<td>270</td>
<td>5540</td>
<td>-</td>
<td>20.0</td>
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</tbody>
</table>

Tailing weighed 4515 gm

Tailing assayed 0.28% $U_3O_8$

Tailing $U_3O_8$ content 12.65 gm

Average Rate of Flow:
274.5 ml/min or 30.25 gal/ton/min

Total Time:
50 hr/5 lb or 2.28 yr/ton

Reagent Consumption:
None

2-C
Test number 2 - Carbonate Leach

**Leaching Solution:**

- $\text{Na}_2\text{CO}_3 - 63.0 \text{ gm - 55.5#/ton}$
- $\text{NaHCO}_3 - 22.7 \text{ gm - 20.0#/ton}$
- $\text{KMnO}_4 - 12.6 \text{ gm - 11.10#/ton}$
- $\text{MgO - 4.2 gm - 3.7#/ton}$

<table>
<thead>
<tr>
<th>Sample No</th>
<th>Rate of Flow ( \text{ml/min} )</th>
<th>Volume ( \text{ml} )</th>
<th>Assay $\text{U}_3\text{O}_8$ content ( \text{gm} )</th>
<th>Recovery %</th>
</tr>
</thead>
<tbody>
<tr>
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<td>added</td>
<td>mg/l</td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>270</td>
<td>5975</td>
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<tr>
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<td>2000</td>
<td>nil</td>
</tr>
<tr>
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<td>225</td>
<td>5925</td>
<td>nil</td>
<td></td>
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<tr>
<td>4</td>
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<td>4900</td>
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<td>nil</td>
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<td>7</td>
<td>275</td>
<td>3050</td>
<td>105.0</td>
<td>.320</td>
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</table>

Tailing weighed 4510 gm

Tailing assayed .11% $\text{U}_3\text{O}_8$

Tailing $\text{U}_3\text{O}_8$ content 4.96 gm

**Average Rate of Flow:**

247 ml/min or 27.2 gal/ton/min

**Total Time:**

40 hr/51b or 1.82yr/ton

**Reagent Consumption:**

- $\text{Na}_2\text{CO}_3 - 7.1 \text{ lb/ton}$
- $\text{NaHCO}_3 - 1.3 \text{ lb/ton}$
Test number 3 - Acid Leach

**Leaching Solution:**

- $H_2SO_4$ - 0.705 N - 30#/ton/liter
- $KMnO_4$ - 4.2 gm - 3.7#/ton
- $MnO_2$ - 11.5 gm - 10.5#/ton

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Time/hr</th>
<th>Rate of Flow/ml/min</th>
<th>Volume (ml) present</th>
<th>added</th>
<th>Assay $U_3O_8$ mg/l</th>
<th>$U_3O_8$ content gm</th>
<th>Recovery %</th>
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</thead>
<tbody>
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<td>3800</td>
<td>1000</td>
<td>245</td>
<td>0.930</td>
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<td>-</td>
<td>255</td>
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<td>1000</td>
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<td>7000</td>
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<td>7750</td>
<td>-</td>
<td>193</td>
<td>1.495</td>
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</table>

Tailing weighed 4512 gm

Tailing assayed 0.30% $U_3O_8$

Tailing $U_3O_8$ content 13.57 gm

**Average Rate of Flow:**

266 ml/min or 29.2 gal/ton/min

**Average Time:**

40 hr/5lb or 1.82 yr/ton

**Reagent Consumption:**

$H_2SO_4$ - 23.21b/ton
APPENDIX IV
FLOTATION TESTS
TEST NO. 100

Grind: Galigher Rod Mill
Mesh: 80% - 200

Ore: J.O.M. high-grade
Time: 5 minutes

Reagents

Emulsion Composition

50 lb/ton

70% Diesel Oil (7gm)-----------------------------35 lb/ton
20% Aliphat 44E (20ml of a 10% sol.) ----------10 lb/ton
10% Oronite (2.15gm, 43% active)--------------5 lb/ton

HF to adjust pH

Products

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight (gm)</th>
<th>Weight %</th>
<th>Assay</th>
<th>Distribution %</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>(U_{2}O_{8})</td>
<td>(V_{2}O_{5})</td>
</tr>
<tr>
<td>Cl. Concentrate</td>
<td>64.5</td>
<td>14.9</td>
<td>.27</td>
<td>.06</td>
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<tr>
<td>Middling</td>
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<td>.17</td>
<td>.03</td>
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<td>Rough Tailing</td>
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<td>.13</td>
<td>.02</td>
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<td></td>
<td>434.6</td>
<td>100.0</td>
<td>.15</td>
<td>.03</td>
</tr>
</tbody>
</table>

Remarks

Initial pH: 7.0
Adjusted pH: 6.5

Emulsion pH
Start: 7.2
Final: 7.0

Breaking Time: 1½ min
Conditioning Time: 7½ min
TEST NO. 101

Grind: Galigher Rod Mill
Mesh: 80%-200

Reagents

**Emulsion Composition**

**50 lb/ton**

- **70% Diesel Oil** (7gm)-------------------------35lb/ton
- **20% Aliphat 44E** (20ml of a 10% sol.)---------10lb/ton
- **10% Oronite** (2.15gm, 43% active)------------5 lb/ton

**HF to adjust pH**

**Products**

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight (gm)</th>
<th>Weight %</th>
<th>Assay U₃O₈</th>
<th>Assay V₂O₅</th>
<th>Distribution U₃O₈</th>
<th>Distribution V₂O₅</th>
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<tbody>
<tr>
<td>Cl. Concentrate</td>
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<td>0.26</td>
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<td>Middling</td>
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<td>0.03</td>
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**Remarks**

- **Emulsion pH**
  - Start: 6.8
  - Final: 6.6

**Initial pH**: 6.9
**Adjusted pH**: 6.0

**Breaking Time**: 3 minutes
**Conditioning Time**: 7½ minutes
TEST NO. 102

Ore: J.O.M. high-grade
Time: 5 minutes

Grind: Galigher Rod Mill
Mesh: 80% - 200

Reagents

Emulsion Composition

50 lb/ton

70% Diesel Oil (7gm)------------------35lb/ton
20% Aliphat 44E (20ml of a 10% sol.)--------10lb/ton
10% Oronite (2.15gm, 43% active)---------- 5 lb/ton

HF to adjust pH

Products

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight(gm)</th>
<th>Weight %</th>
<th>Assay</th>
<th>Distribution %</th>
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<td>(U_3O_8)</td>
<td>(V_2O_5)</td>
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<td>.05</td>
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<td>.15</td>
<td>.03</td>
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</tbody>
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Remarks

Initial pH: 7.0
Adjusted pH: 5.5

Emulsion pH

Start: 6.1
Final: 6.0

Breaking Time: 5 minutes
Conditioning Time: 7½ minutes
TEST NO. 103

Ore: J.O.M. high-grade

Time: 5 minutes

Grind: Galigher Rod Mill

Mesh: 80% - 200

Reagents

Emulsion Composition

50 lb/ton

70% Diesel Oil (7gm)----------------------35lb/ton
20% Aliphat 44E (20ml of a 10% sol.)---------10lb/ton
10% Oronite (2.15 gm, 43% active)---------- 5lb/ton

Na₂CO₃ to adjust pH

Products

<table>
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<tr>
<th>Description</th>
<th>Weight (gm)</th>
<th>Weight %</th>
<th>Assay</th>
<th>Distribution %</th>
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<td>.03</td>
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</table>

Remarks

Initial pH: 8.0

Adjusted pH: 8.0

Emulsion pH

Start: 8.3

Final: 8.2

Breaking Time: ½ minute

Conditioning Time: 7½ minutes

5-D
TEST NO. 200

Ore: J.O.M. high-grade
Time: 5 minutes

Grind: Galigher Rod Mill
Mesh: 80% - 200

Reagents

Emulsion Composition

50 lb/ton

90% Diesel Oil (9gm)------------------------45 lb/ton
5% Aliphat 44E (5ml of a 10% sol.)---------2.5 lb/ton
5% Oronite (1.16gm, 43% active)--------2.5 lb/ton

HF to adjust pH

Products

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight (gm)</th>
<th>Weight %</th>
<th>Assay</th>
<th>Distribution %</th>
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</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>( U_{3O8} )</td>
<td>( V_{2O5} )</td>
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<td>Cl. Concentrate</td>
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<tr>
<td>Middling</td>
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<td>14.6</td>
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<td>321.6</td>
<td>74.5</td>
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<td>0.02</td>
</tr>
<tr>
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<td>0.02</td>
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</table>

Remarks

Initial pH: 7.4
Adjusted pH: 6.0

Emulsion pH
Start: 6.6
Final: 6.0

Breaking Time: 2 \( \frac{1}{2} \) minutes
Conditioning Time: 7 \( \frac{1}{2} \) minutes

6-D
TEST NO. 201

Dore: J.O.M. high-grade
Time: 5 minutes

Grind: Galigher Rod Mill
Mesh: 80% - 200

Reagents

Emulsion Composition

50 lb/ton

85% Diesel Oil (8.5 gm) — 42.5 lb/ton
10% Aliphat 44E (10 ml of a 10% sol.) — 5.0 lb/ton
5% Oronite (1.16 gm, 43% active) — 2.5 lb/ton
HF to adjust pH

Products

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight (gm)</th>
<th>Weight %</th>
<th>Assay U₂O₅</th>
<th>Distribution %</th>
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<td>Cl. Concentrate</td>
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<tr>
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<td>.14</td>
<td>12.6</td>
</tr>
<tr>
<td>Rough Tailing</td>
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<td>69.6</td>
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<td>56.9</td>
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<tr>
<td>Comp. (calc)</td>
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<td>100.0</td>
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</table>

Remarks

Initial pH: 6.9
Adjusted pH: 6.0

Emulsion pH
Start: 7.0
Final: 6.7

Breaking Time: 11½ minutes
Conditioning Time: 15 minutes

7-D
### TEST NO. 216

**Ore:** J.O.M. high-grade

**Time:** 5 minutes

**Grind:** Galigher Rod Mill

**Mesh:** 80% - 200

**Reagents**

**Emulsion Composition**

50 lb/ton

- 42.9% Diesel Oil (4.3 gm) ——— 21.4 lb/ton
- 42.9% Aliphat 44E (42.9 ml of a 10% sol.) ——— 21.4 lb/ton
- 14.2% Oronite (3.45 gm, 43% active) ——— 7.2 lb/ton

HF to adjust pH

### Products

<table>
<thead>
<tr>
<th>Description</th>
<th>Weight (gm)</th>
<th>Weight %</th>
<th>Assay</th>
<th>Distribution %</th>
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<td>Cl. Concentrate</td>
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<td>.20</td>
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<tr>
<td>Middling</td>
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<td>13.0</td>
<td>.22</td>
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<tr>
<td>Rough Tailing</td>
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<tr>
<td>Comp. (calc)</td>
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### Remarks

- **Initial pH:** 7.1
- **Adjusted pH:**

<table>
<thead>
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<th>Remarks</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>Emulsion pH</td>
</tr>
<tr>
<td></td>
<td>Start: 6.7</td>
</tr>
<tr>
<td></td>
<td>Final: 6.5</td>
</tr>
</tbody>
</table>

- **Breaking Time:** 3\(\frac{1}{2}\) minutes
- **Conditioning Time:** 7\(\frac{1}{2}\) minutes

8-D
APPENDIX V

CONTACT ANGLE STUDIES
C_{10}ISONITRILE