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METALLURGY—RAW MATERIALS

UNITED STATES ATOMIC ENERGY COMMISSION

LABORATORY INVESTIGATION OF DAKOTA LIGNITES

By
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April 1, 1957

Raw Materials Development Laboratory
National Lead Company, Inc.
Winchester, Massachusetts

Technical Information Service Extension, Oak Ridge, Tenn.

UNCLASSIFIED

II

Printed in USA, Price $1.00. Available from the Office of Technical Services, Department of Commerce, Washington 25, D. C.

AEC Technical Information Service Extension
Oak Ridge, Tennessee

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TOPICAL REPORT WIN-54

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Contract AT(49-6)-924

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ABSTRACT

This report deals with the laboratory investigation into methods of recovery of uranium from Dakota Lignites. A flowsheet is recommended for pilot plant testing.

INTRODUCTION

Earlier investigations into the extraction of uranium from Dakota lignites have been conducted at the Battelle Memorial Institute[1] and at the Oak Ridge National Laboratory[2]. The uranium content of the earlier samples ranged from less than 0.01 to about 0.05 per cent. Although the uranium was soluble in acid or alkaline carbonate systems, the consumption of leaching reagent was high on a per ton basis, and resulted in excessive costs per pound of product extracted.

Later prospecting in the lignite areas of North and South Dakota disclosed deposits of much higher uranium grade, and a testing program was initiated at the Raw Materials Development Laboratory in November 1954. The progress of the lignite investigation has been reported monthly and several interim reports have been issued and made available to the Atomic Energy Commission for distribution.

Since this is the first topical report issued on the subject, it covers a period of about two years of extensive, and, at times, intensive investigation. The scope of the work includes acid and alkaline leaching of raw lignites and a wide variety of furnace products.

During the course of the investigation, nearly 50 individual lignite samples and specially prepared char products were investigated in the laboratory. Early in the test program, it became apparent that the processing costs for lignite treatment would be high in comparison with those for other uranium ores in the Western United States. Consequently, much of the investigation was of the nature of process development rather than straightforward amenability testing. Emphasis on process development also appeared to be advisable since few of the samples were representative of any significant tonnage.

Two-stage countercurrent acid leaching was studied on both raw lignite and lignite ash. Flotation of carbon from incompletely burned ash was investigated as a means of improving the overall efficiency of roasting and leaching. Various modifications of resin-in-pulp methods were tried in both acid and carbonate slurries. Solvent extraction studies included testing of organic phosphate and amine extractants as well as development

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1/ Ewing, R. A., et al, "Recovery of Uranium from North Dakota Lignites", BMI-237, July 31, 1950

2/ Brown, K. B., et al, "Recovery of Uranium from Lignites", Progress Report ORNL 1569, June 1, 1955 of equipment to combat the emulsion problem. Ion exchange testing was conducted on liquors after purification by activated charcoal and after partial neutralization, and the direct application of ion exchange to liquors produced by two-stage leaching was also investigated.

The Coal Technology Section of the U. S. Bureau of Mines cooperated with the Winchester group in conducting a series of tests in their commercial-scale equipment aimed at production of char for carbonate leaching. Similar work was conducted at Montana State College at Bozeman.

Although various methods of producing lignite ash were tried, this very important phase of lignite processing has not yet been fully explored.

SUMMARY

The uranium bearing lignites from the Dakotas, as represented by the samples studied, are not true lignites in the sense that they might represent a significant source of fuel. The ash content is high and quite variable with a majority of the samples containing from 30 to 60 per cent ash as determined by an oxidizing muffle roast of dry lignite for two to three hours at 450°C. In fact, samples from the different deposits, and sometimes from the same deposit, varied to extremes in almost all categories pertinent to uranium processing. Uranium assays ranged from 0.06 to 1.5 per cent U_3O_8 on the dry basis. Moisture contents ran as high as 45 per cent with a median reportedly at about 33 per cent. In some samples a host of common uranium minerals were readily identifiable while in others essentially all of the uranium was apparently present as an adsorbed phase. Pulps ground in water ranged from strongly alkaline to weakly acidic in reaction and the resulting liquors were, in many cases, rendered an opaque black coloration by water soluble organics.

Several approaches were explored in attempting to develop a satisfactory process for treatment of raw lignites. Acid leaching, followed by solvent extraction with EHPA, holds some promise, although the extraction step was successfully demonstrated only on the samples containing the minimum of acid soluble organic compounds. A rather expensive method of purifying raw acid leach liquor, with respect to both organics and molybdenum by treatment with finely divided activated carbon, was demonstrated on a cyclic basis. This, or a comparable liquor pretreatment step, would probably be required in any plant employing acid leaching of raw lignite.

Attempts to recover uranium from raw pulps by various modifications of resin-in-pulp methods were not attractive. Organic fouling and molybdenum poisoning were encountered and uranium loadings on the resin were low.

Although carbonate solutions extract uranium from raw lignites, a practical means of making a solid-liquid separation of the resulting highly viscous pulps was not found. Resin-in-pulp methods were less attractive in the carbonate than in the acid system.

A continuous retorting method[1/] of producing liquid fuel and char was investigated as a method of preparing uranium leach feed. Initial laboratory tests indicated that a narrow temperature range around 400°C would produce chars that could be

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[1/] Parry, V. F., et al, "Drying and Carbonizing Fine Coal in Entrained and Fluidized State", U. S. Bureau of Mines, Rept. Inv. 4954, 1953 leached in carbonate solutions and filtered. Although higher retort temperatures improved filtration, the extraction dropped off sharply with increased temperature. Several tests in the commercial size equipment at the Coal Technology Station of the U. S. Bureau of Mines in Denver and at Montana State College in Bozeman were unsuccessful.

Roasting under variable conditions to oxidize all or part of the organic matter resulted in products yielding poor leachability, poor filterability, or both in the carbonate system.

Uranium extraction by sulfuric acid leaching from either retort char or partly burned products was inferior to that obtained from raw lignites.

Complete oxidation of the organic matter, followed by acid leaching of the ash, was found to be the most universally applicable process to the many samples studied and forms the basis of the flowsheet presented at the end of this summary (Figure 1).

In comparing results of leaching raw and roasted products from individual samples it was found that treatment of dead burned ash yielded higher uranium extraction with less acid provided the furnace temperature was not much above 450°C.

Two-stage countercurrent leaching tests in the laboratory indicated several advantages and this feature is included in the flowsheet. Three of the most important advantages over single-stage leaching are:

1. Reduction in acid consumption
2. Production of a cleaner pregnant solution with respect to interfering salts.
3. Reduction in amount of alkali needed for tails neutralization.

Flotation of unburned carbon is also included in the proposed flowsheet for pilot testing. The laboratory testing indicates that the size of a roaster unit for a given lignite throughput can be cut nearly in half if flotation is properly employed. The amount of carbon recycled through the roaster is not excessive because a high percentage of the carbon is decomposed in less than 50 per cent of the time required for dead burning in a single pass. Flotation can be positioned either before or after leaching. Laboratory cyclic tests indicate better uranium recovery in the "after leach" position but the burden on leaching and filtration facilities is higher and a little more acid was apparently consumed.

The method of recovery of the uranium from solution selected for initial pilot plant testing is to solvent extract with an amine. Continuous laboratory tests indicated that pregnant liquors from lignite ash could be satisfactorily treated in internal mixer settlers. Stripping of the solvent with chloride, followed by carbonate stripping, separates uranium and molybdenum, and the molybdenum is readily recoverable as a by-product.

The neutralizing stage of the two-stage leach can be operated under conditions that will hydrolyze nearly all of the molybdenum and ferric iron and the resulting liquor can be satisfactorily treated by ion exchange. Loadings of better than five pounds per cubic foot of IRA-400 resin were obtained. The possibilities of ion exchange should definitely not be overlooked.

Investigation of roasting methods is incomplete. The problem yet unsolved is to burn the volatiles and fixed carbon at a high rate without producing excessive temperatures. Flotation, combined with deliberate under-roasting in the fluosolids reactor is recommended for pilot plant study.

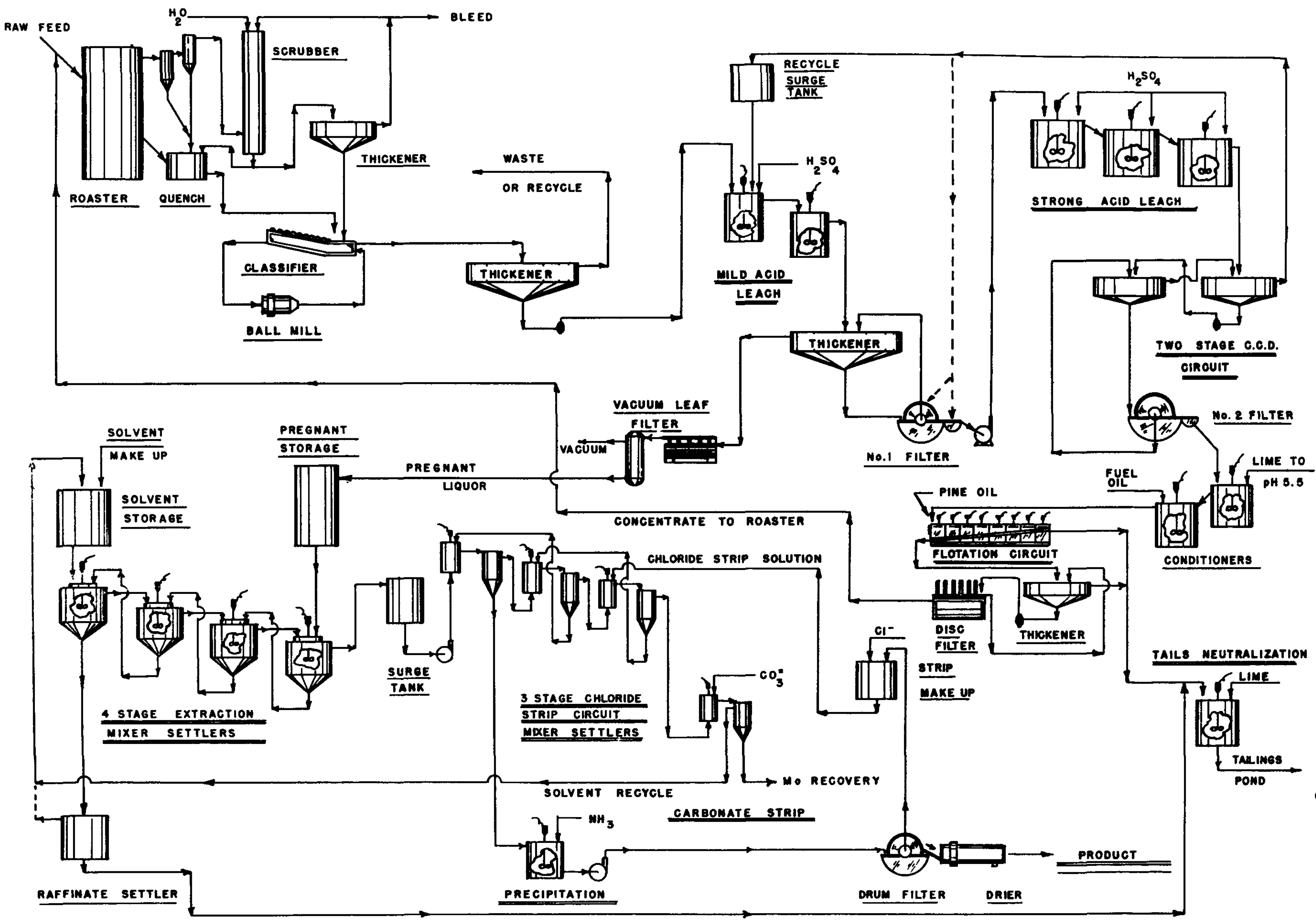


FIGURE - I, ACID LEACH — SOLVENT EXTRACTION CIRCUIT FOR URANIFEROUS LIGNITE ORES

ORIGIN AND DESCRIPTION OF SAMPLES

A. Source of Samples

The samples of lignite which are the subject of this report were obtained from Harding County, in the northwest corner of South Dakota and in Bowman, Slope, and Billings Counties in adjacent North Dakota. A location map of the principal mining areas is shown in Figure 2 and pertinent information on the individual samples is presented in Appendix Table A-1.

B. Preparation of Samples

The samples received ranged in weight from about 10 pounds to two tons and in particle size from minus 100-mesh to six inch lumps. Most of the samples were wet, the moisture content ranging from about 15 to 50 per cent.

In general, the samples weighing less than approximately 50 pounds were prepared for testing by drying in an oven at 100°C and then crushing to minus 10-mesh with a jaw crusher and steel rolls in closed circuit with a 10-mesh vibrating screen. The dried ore was then split and weighed into 500-gram and one-kilogram test charges. The larger samples were air dried, sometimes with the aid of infra-red lamps, to a low enough moisture content to permit handling and were then screened on a one-half inch screen. The oversize was crushed, and the sample was coned and quartered to a convenient size. The final sample was then oven dried and prepared for testing by crushing to minus 10-mesh, as described above. In several instances, quantities of lignite for larger scale leaching and roasting tests were prepared from air dried ore by crushing in a hammer mill.

C. Chemical Analyses

Chemical analyses of the samples are presented in Appendix Table A-2. Spectrographic analyses of each of the samples showed that, in addition to the constituents reported in the chemical analyses, zirconium, titanium, and barium occur in concentrations ranging from 0.1 to 1.0 per cent. Nickel, manganese, strontium, chromium, copper, and lithium are usually present in amounts ranging from a trace to 0.1 per cent.

In the chemical analyses, the content of combustible and volatile matter is indicated by the total carbon content and ignition loss. Appendix Table A-3 presents the results of a precision laboratory distillation analysis on Bryco Sample 185-4. Coal analyses were not warranted on the individual samples which are the subject of this report. However, coal analyses on samples from a 2000 ton shipment of uraniferous lignites to the Grand Junction Pilot Plant are presented in Appendix Tables A-4, A-5, A-6, and A-7 for the purposes of general information.

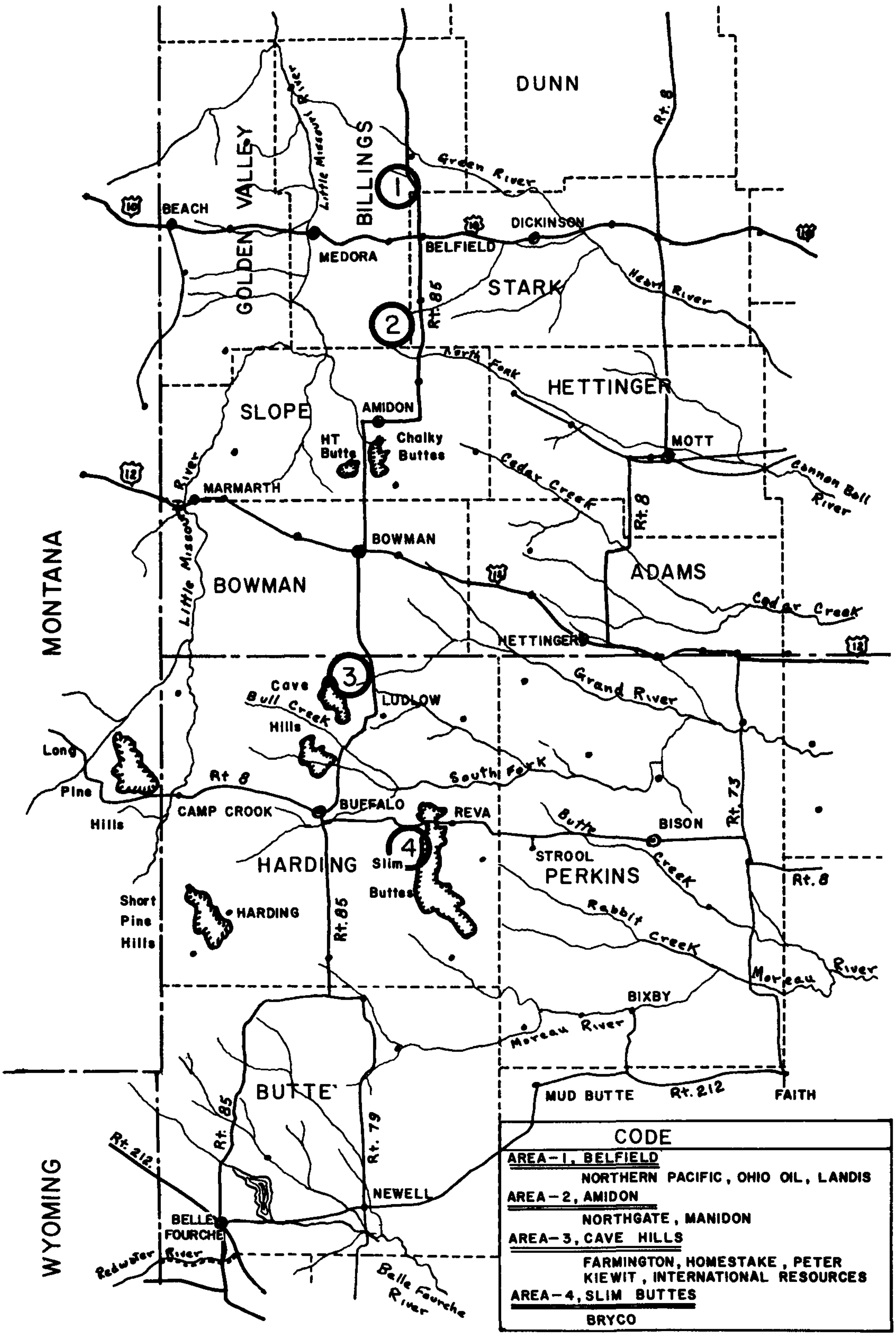


Figure 2

LOCATION OF PRINCIPAL LIGNITE AREAS

D. Mineralogy and Petrography

1. Bulk Ore Textures, Colors, and Lusters

The Dakota Lignites, received and examined at the Winchester Laboratory exhibited wide variations in texture, color, mineral compositions, ash content, and uranium assay. The ores were examined as received and then subjected to a series of physical, chemical, and microscopic determinations.

The samples were received in varying degrees of fragmentation, ranging from coarse chunks about six inches in diameter to fine, powdery material. Some of the samples were extremely wet and others appeared dry. The textural nature of the lignites ranges from dense, hard, massive and sometimes spongy material to soft, friable material which crumbles readily to powder in the hand. Many of the samples slack on drying. Parting along bedding surfaces is very common and some of the material exhibits checking or cross-jointing which permits ready fragmentation. Wood fragments are present in some samples while others exhibit more of a spongy, peat-like texture. Others consist of granular and powdery material.

The organic material ranges in color from jet black to yellowish brown, with all variations in between such as dark brown, red brown, and light chocolate brown. Luster of the organic material ranges from bright to dull. These variations in color, luster, and texture may be observed within one ore, and even within one specimen in the form of interbedded layers. There is an interbedding of vitrain, durain, and fusain in layers ranging from a fraction of an inch to over six inches in thickness.

2. Inorganic Impurities

The lignite ores carry small amounts of sand, ranging from coarse to fine-grained in texture and occurring as interbedded lenses or small nodules. Clay is a common impurity, in the form of interbedded layers or as clay balls up to six inches thick. The sand and clay are major constituents of the ash content, there being a direct relationship between those ores which were observed to have high sand-clay content and those which yielded the highest ash values. Occasional dense, rounded pebbles and cobbles occur in the lignite and cause some crushing difficulties.

Jarosite occurs in the bulk ore as yellow, powdery films along joints and bedding surfaces and as soft, powdery nodules within the lignite itself.

Gypsum is present in many of the samples as thin seams and fracture fillings.

Secondary calcite is observed as white encrustations along joints and bedding surfaces and as small nodules within the lignitic material.

Some thin, dark shale fragments are found in some of the lignite ores but this is not found as a quantitatively significant impurity.

3. Ash Content

Ash content, in the various samples examined, ranges from a low of 18 per cent to a high of 86 per cent, with the majority of samples falling into the range of 30 to 60 per cent ash content. Interbedded sands and clays constitute the major components of the ash.

4. Uranium Content

In 55 samples examined, the uranium values ranged from a low of 0.013 per cent U_3O_8 to a high of 1.49 per cent U_3O_8. The average grade of these samples is 0.376 per cent U_3O_8.

5. Mineralogy

The lignite consists predominantly of the carbonized remains of the various components of plant material; cellular textures of wood fibres are distinguishable under the microscope. Most of the uranium in the lignites is associated with organic material in an adsorbed form. Part of the uranium also occurs as inorganic material in the form of epigenetic minerals found concentrated along fracture and bedding surfaces. These minerals in decreasing order of abundance, are: autunite $CaO\cdot 2UO_3\cdot P_2O_5\cdot 8H_2O$, meta-tyuyamunite $CaO\cdot UO_3\cdot V_2O_5\cdot nH_2O$, uranophane $CaO\cdot 2UO_3\cdot 2SiO_2\cdot 6H_2O$, carnotite $K_2O\cdot 2UO_3\cdot V_2O_5\cdot 3H_2O$, and zippeite $2UO_3\cdot SO_3\cdot nH_2O$. The relative amounts of uranium carried by organic and by inorganic constituents vary with each ore.

The non-uraniferous minerals, in decreasing order of abundance, are: quartz, clay minerals (undifferentiated), jarosite $K_2Fe_6(OH)_{12}(SO_4)_4$, gypsum $CaSO_4\cdot 2H_2O$, analcite $NaAlSi_2O_6\cdot H_2O$, calcite $CaCO_3$, limonite $Fe_2O_3\cdot nH_2O$, hematite Fe_2O_3, pyrite FeS_2, barite $BaSO_4$, hornblende $CaMg_2(Al,Fe)_2(SiO_4)_3$, zircon $ZrSiO_4$, and opal $SiO_2\cdot nH_2O$. Of this group, the minerals of syngenetic origin appear to be quartz, pyrite, hornblende, and barite.

Though no molybdenum minerals were observed, chemical assays show a range from 0.042 to 0.60 per cent molybdenum content.

There is a direct relationship between high molybdenum values and high uranium values and it appears that, like the uranium, much or all of the molybdenum is held by adsorption on the organic material.

SECTION I

ACID LEACHING OF LIGNITE ASH

A. Batch Leaching Tests

1. Determination of Standard Roasting Conditions

After the initial preparation of drying and crushing to minus 10-mesh, the lignite samples were roasted in a laboratory electric muffle furnace to determine standard roasting conditions for leaching of lignite ash. Satisfactory roasting was accomplished by burning the lignite at a nominal muffle temperature of 450°C. An ore bed of 1/2 inch was used, and the bed was rabbled at 20 minute intervals. Complete combustion of carbon was obtained for most of the samples within two hours. Complete carbon removal by roasting and rabbling at a furnace temperature of 450°C is the method used for determining loss on ignition (LOI) in all the lignite testing. This method, rather than the standard LOI procedure employed in coal technology, is used because the latter does not give a true measure of the ash produced under roasting conditions that are optimum for leaching.

A time and temperature study of the roasting conditions had indicated that:

1. Complete burning of the carbon was necessary for efficient acid leaching.
2. Roasting temperatures much higher than 450°C tended to cause insolubilization of uranium by acid leaching.
3. Baking the ash for longer periods of time than required for complete carbon removal did not significantly affect leaching efficiency.

The effect of varying the roasting time and temperature, over and above the time required for complete carbon removal, is indicated by the test results listed in Table 1. These tests on Sample 185-18 indicate a serious insolubilization of uranium when the roasting temperature was increased to 650°C and only a slight increase in extraction by increasing the roasting time from two to four hours.

Table 1

Effect of Roasting Time and Temperature on Acid Leaching Efficiency

Sample 185-18
Peter Kiewit

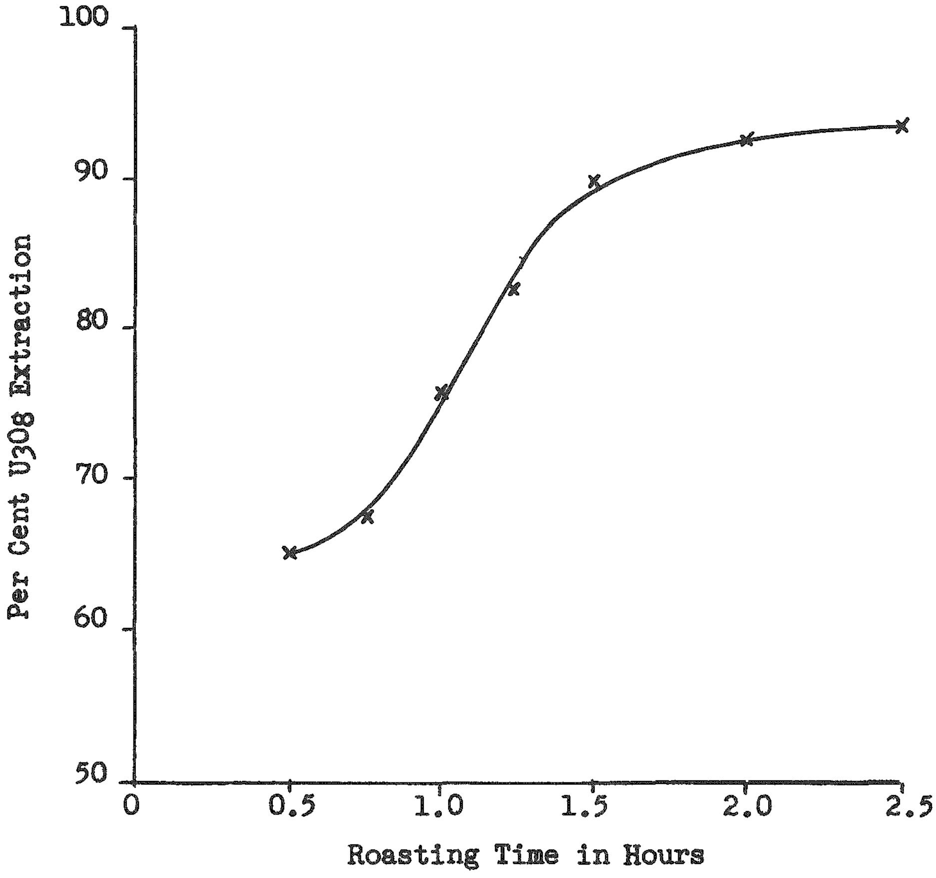
Roast Time, Hours	Muffle Temperature °C	Residue Assay % U_3O_8	% U_3O_8 Extraction
2	450	0.015	97.1
2	550	0.023	96.0
2	650	0.054	89.2
4	450	0.012	97.8
4	550	0.020	96.4
4	650	0.051	91.5

The effect of less than complete removal of carbon is indicated by Figure 3 for Samples 185-30, 31, and 33 (composite sample of Ohio Oil Company) and shows that substantially complete combustion of carbon is required for optimum uranium extraction.

Further discussions of the effect of roasting variables on U_3O_8 extraction by acid leaching are included in Sections III and IV.

2. Batch Leaching Procedure

Samples of ash produced by muffle roasting lignite at 450°C were ground in water in a laboratory rod mill to a nominal minus 35-mesh maximum particle size. The grinding water was removed by filtration, and the ground ash mixed with water at 30 to 50 per cent solids. In most cases, acceptable agitation was obtained at 40 per cent solids. Concentrated H_2SO_4 was added to the pulp to maintain a desired pH, and further additions were made as the acid was consumed during leaching. The pulp was mechanically agitated by a motor driven stainless steel turbine impeller, and the leaching temperature maintained either by a gas burner or electric hot plate. Samples of leach pulp were cooled to 25°C before pH measurements were made. At the conclusion of the leaching time, the pulp was diluted to 20 to 30 per cent solids, as required, and Dow Separan was added as 0.5 gram per liter stock solution to flocculate the pulp. Addition of the Separan-2610 to the dilution water was found to be the most effective method. The residue was filtered by vacuum on a Buchner funnel and the cake washed with hot water or hot dilute H_2SO_4 to a final solution to feed ratio of about four to one. When water was used for washing, the residue was washed by repulping prior to assay. The uranium extraction was based on the amount of uranium remaining in the residue. Acid requirements are expressed as pounds of 100 per cent H_2SO_4 per ton of leach feed.



<u>Figure-3. Effect of Roasting Time on Leaching</u>

3. Experimental Results

Results of single-stage acid leaching tests of ash produced by completely burning the various lignite samples at 450°C in a muffle furnace are included in Appendix Tables B-1 and B-2. These tests included investigation of the effect of the leaching variables on extraction efficiency as well as routine testing of the later samples under relatively fixed conditions.

Investigation of the effects of grinding, leaching time and temperature, acid concentration, and oxidant addition on uranium extraction indicated that optimum leaching efficiency in a single leaching stage was generally obtained under the following conditions:

1. Grind to minus 35-mesh and dewater
2. Leach for six hours at 75°C
3. Maintain 0.5 pH and 40 per cent solids.

In some cases lower acid concentrations produced satisfactory extraction of uranium, and , therefore, acid concentration was maintained a variable in routine testing.

a. Grinding and Dewatering

Lignite ash is extremely friable, and minus 10-mesh material disintegrates to a powder during leaching. Consequently, fine grinding is not necessary to liberate uranium from coarse calcine. However, appreciable quantities of acid consuming alkali may be washed out in a wet grinding circuit. Also, up to about 30 per cent of the molybdenum is removed in the grinding water. In the case of Sample 185-18 (Peter Kiewit), test results indicate that an acid saving of the order of 300 pounds per ton of ash was achieved by grinding the highly alkaline (pH 11.5) ash in water and filtering prior to acid leaching. Dewatering after grinding provided subsequent improvements.in thickening and filtration. In addition, approximately 12 per cent of the Mo was extracted during grinding. Many of the samples gave alkaline reactions in water, indicating that grinding of ash should be an integral part of a lignite processing flowsheet. Satisfactory grinding was noted at 40 per cent solids in most cases, with only a few ash samples (Manidon Samples 185-7, 20, and 22) requiring grinding at higher dilutions. Several of the ash samples produced an acid reaction in water, and would require a pH adjustment in the grinding circuit to prevent corrosion as well as possible uranium solubilization. The extreme friability of the ash allowed very short grinding time for size reduction from minus 10-mesh to minus 35-mesh. Grinding pH values for individual ash samples are included in

Appendix B, Table B-3, which summarizes the results of acid leaching tests of the various lignite samples under approximately optimum leaching conditions. Detailed results are included in Appendix B, Tables B-1 and B-2.

b. Effect of Leaching Time

The effect of leaching time on uranium extraction was studied for several ash samples, using a leaching temperature of 75° to 80°C. Typical results are shown in Figure 4. The tests indicate that very short leaching times were sufficient at the temperature and acid concentrations used. Little advantage was obtained by leaching for longer than six hours, and satisfactory extraction was obtained in shorter times for some samples.

In these tests, all acid was added at the start of the leaching. Some decrease in uranium extraction was noted for longer leaching times, probably because of hydrolysis which occurred as the leaching pH was allowed to rise.

c. Effect of Leaching Temperature

Because of the large quantities of acid required for leaching of many of the lignite ash samples, the heat of reaction, when the acid is added, provides initial leaching temperatures up to 80°C. On large scale leaching operations, much of this heat would be retained by the leach pulp for several hours. Therefore, little emphasis was placed on low temperature acid leaching, and most of the testing was performed at 75°C or more. Investigation of the effect of leaching temperature on uranium extraction in six hours indicated an increased uranium extraction with temperature in one case, and no effect in two cases, as shown in Figure 5. Cooling of the pulp was required to obtain the 25°C leaching temperature.

d. Effect of Acid Concentration

Although most of the lignite ash samples required high acid concentrations (terminal leaching pH zero to one) for efficient uranium extraction, it was noted that satisfactory extraction of uranium was obtained in some cases with low free acid concentrations at the end of leaching. Therefore, bench scale testing generally included leaching at two or more acid concentrations. The effect of acid addition on extraction of uranium and on terminal leaching pH is presented, for a few of the samples, in Figure 6. Detailed test results are reported in Appendix B, Tables B-1 and B-2. The acid concentration required for each sample is included in Table 2 for near optimum uranium extraction. A sharp increase in the extractions of molybdenum and iron was noted for the higher acid additions.

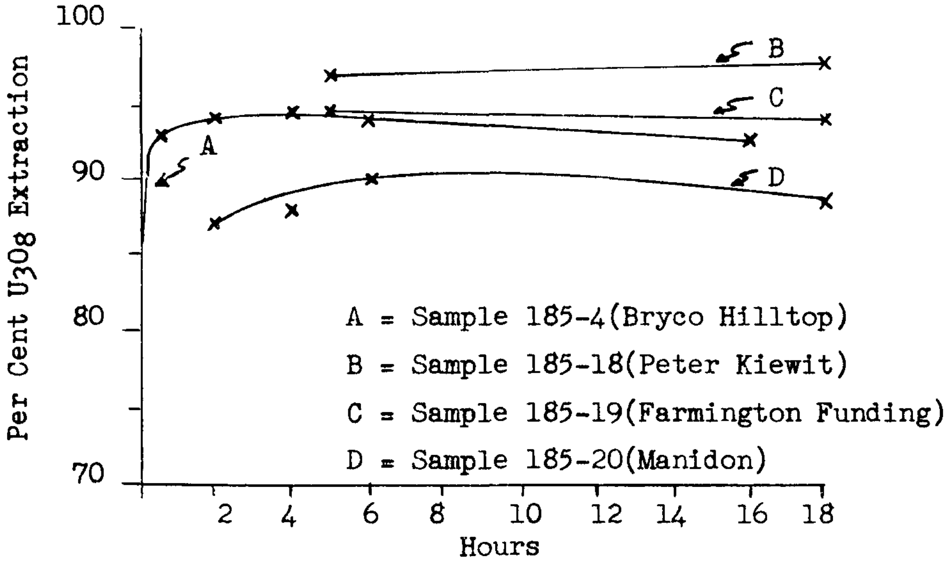


Figure-4, Effect of Leaching Time on Uranium Extraction

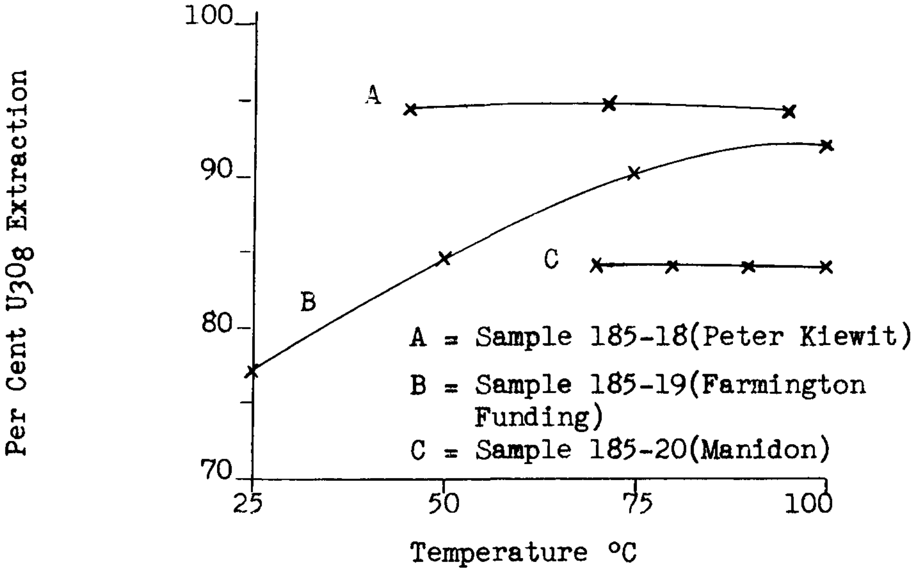


Figure-5, Effect of Leaching Temperature

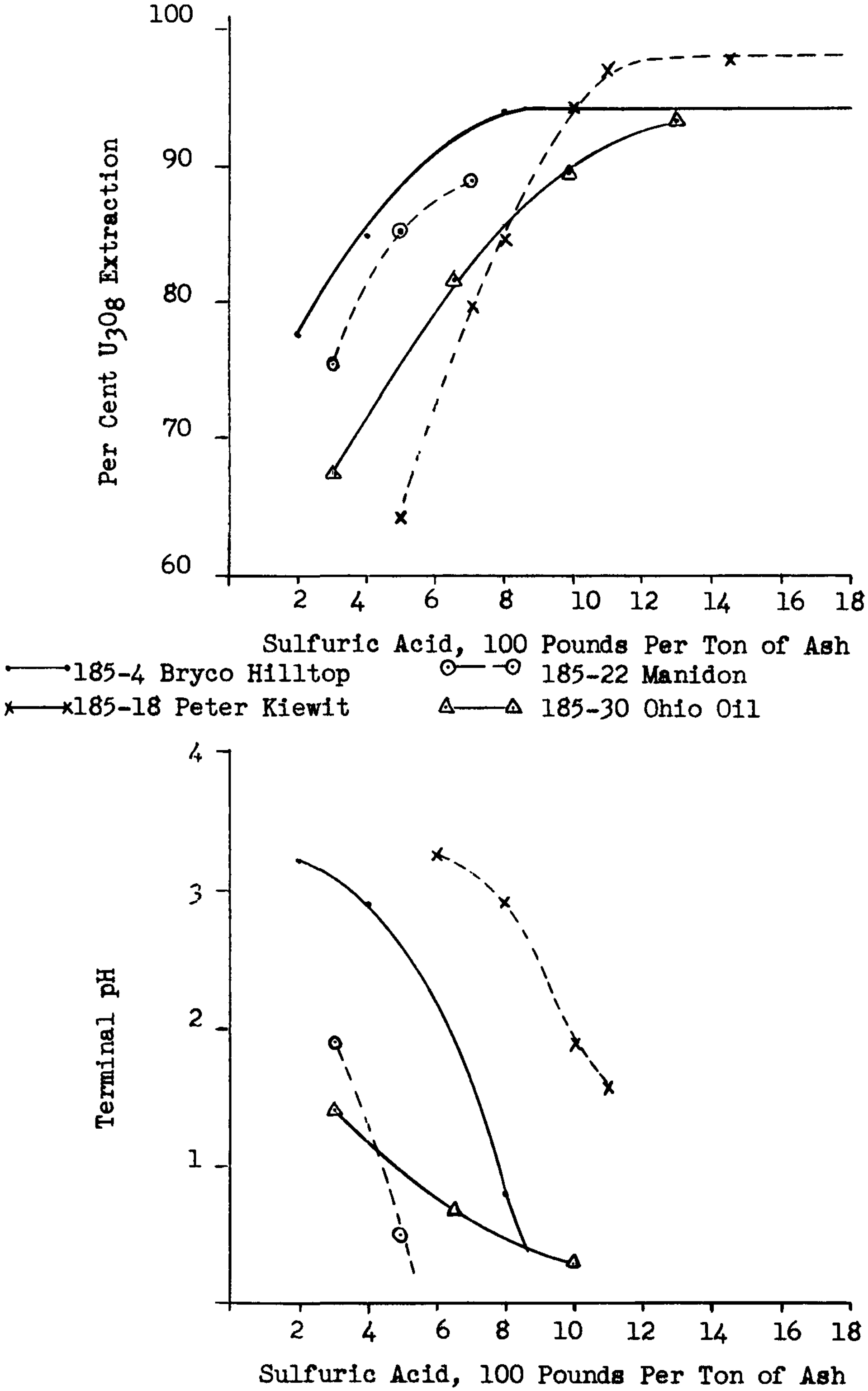


Figure-6, Effect of Acid

Table 2
Acid Leaching Tests of Lignite Ash - 450°C Muffle Roast

Sample Number	Ash % U_3O_8	Grind pH	H_2SO_4, Lb/Ton		Terminal pH	% U_3O_8 Ext'n
			Ash	Lignite		
185-1	0.47	--	875	510	--	95
185-2	0.21	--	625	175	--	70
185-3	0.52	--	425	135	--	70
185-4	0.60	--	800	500	0.8	94
185-5	0.41	--	600	350	--	92
185-6	0.48	--	700	420	0.5	97
185-7	0.90	--	900	160	0.7	89
185-8	0.52	11.0	1500	750	0.5	97
185-16	0.55	5.0	615	370	0.5	90
185-18	0.49	11.5	1100	530	2.1	95
185-19	0.50	6.0	700	400	1.1	91
185-20	0.50	4.5	700	225	0.0	90
185-21	0.18	3.9	700	370	0.2	92
185-22	0.50	4.0	500	150	0.5	85
185-25	0.66	--	800	270	1.4	95
185-26	0.35	7.5	900	485	1.1	95
185-27	0.45	4.5	500	160	0.5	86
185-28	0.16	4.2	700	380	0.6	93
185-29	1.18	8.2	1200	590	0.05	94
185-30	0.66	4.8	975	370	0.3	90
185-31	0.82	10.6	1200	515	1.0	97
185-33	0.29	10.8	820	270	1.2	92
185-34	0.42	4.4	700	280	0.4	88
185-35	1.33	11.0	1400	715	0.4	98
185-37	1.04	7.8	1100	365	0.8	93
185-38	0.39	10.3	1000	450	1.0	96
185-39	0.88	10.5	1030	410	1.0	98
185-40	0.40	8.6	790	310	0.8	86
185-41	0.57	10.2	1070	380	0.5	93
185-42	0.16	9.1	840	305	1.0	85
185-43	0.105	6.4	300	260	1.0	94
185-44	1.21	6.9	575	235	0.5	92
185-45	0.55	4.0	440	210	0.5	89
185-46	0.36	10.8	1030	600	0.5	98
185-47	0.23	4.0	505	315	0.5	92
185-48	0.30	10.5	1210	410	1.0	97

Note: Minus 10- or minus 35-mesh ash leached for six hours at 33 to 50 per cent solids and 75° to 80°C.

When leaching was carried out at 40 per cent solids and pH 0.5, the terminal free acid concentration was about 25 grams per liter, or 75 pounds of H_2SO_4 per ton of ash. This would appear to be about average terminal acid concentration for single-stage leaching of muffle roasted lignite.

e. Effect of Oxidant

In the few tests where MnO_2 was added to acid leach pulp, no significant effect on uranium extraction was noted. As the leaching solution often contained very high concentrations of iron, very little variation in oxidation potential was achieved by addition of relatively large quantitites of MnO_2. Satisfactory oxidation potentials (emf numerically higher than -400 millivolts) were generally obtained by acid leaching of muffle ash without addition of MnO_2.

f. Analyses of Leach Liquors

The concentrations of uranium, molybdenum, iron, and sulfate in acid leach liquors from batch leaching tests of lignite ash samples are recorded in Appendix B, Tables B-1 and B-2. Partial analyses of a few typical leach liquors, at a four to one solution to ash ratio, are presented in Table 3, below. Other major constituents are aluminum, magnesium, and the alkali metals. The leaching conditions were about optimum for uranium extraction.

Table 3

Partial Analyses of Typical Leach Liquors

Sample Number	Source	Grams/Liter U_3O_8	Mo	Fe	$SO_4^=$
185-6	Bryco Hilltop	1.24	0.69	4.2	92
185-18	Peter Kiewit	1.0	0.4	15.0	150
185-19	Farmington	1.18	1.0	17.0	123
185-27	Northgate	1.01	0.68	6.2	92
185-28	Ohio Oil	1.48	1.09	23.6	122
185-30	Homestake	3.22	0.10	6.5	101
185-35	Int'l. Resources	0.94	0.06	11.4	101
185-40	Ohio Oil	0.86	1.0	15.0	99
185-47	Whitman	0.53	0.59	5.7	75

B. Two-Stage Leaching Tests

In single-stage acid leaching procedures, lignite ash was generally leached at 75°C with enough acid to give a terminal pH of about 0.5 for efficient dissolution of the uranium. The resultant leach liquor usually contained sufficient molybdenum to complicate the recovery of uranium from solution. A typical leach solution contained 1.0 gram U_3O_8, 0.5 to 0.6 gram Mo, and 15 to 20 gram total Fe per liter.

A method for purifying leach liquors by partial neutralization at elevated temperature has been reported by P. J. Magno[1] and will be discussed further in Section VI of this report. However, the leach liquors obtained by single-stage leaching of lignite ash under optimum conditions for uranium extraction require excessive alkali to neutralize the free acid and pH buffering salts.

In order to take advantage of hydrolysis, a modified two-stage leach flowsheet was tested in which the excess acid from the second stage was neutralized on fresh ash in the first stage. This procedure permits maximum utilization of acid while giving a pregnant solution of high pH. At the same time, sufficient acid may be used in the second stage to obtain maximum uranium extraction. The high leaching temperature causes iron and molybdenum to be hydrolyzed out of solution, at pH values low enough to hold most of the uranium in solution, producing a pregnant solution which is more suitable for feed to a conventional uranium recovery circuit. Table 4 compares the analyses of leach liquors from single-stage and two-stage leaching of Sample 185-18 (Peter Kiewit).

Table 4

Comparison of Leach Liquors

Constituent	Assay, Grams/Liter Single Stage	 Two-Stage
U_3O_8	1.0	1.0
Mo	0.4	0.020
Fe (Total)	15	1.0
$SO_4^=$	150	80
pH	1.0	2.7

The flowsheet employed is shown in Figure 7. Roasted ore was ground (minus 35-mesh) at 30 per cent solids for three minutes and filtered. This was done to simulate a wet grinding circuit. During our test work, the filtrate was discarded but, in actual plant operation, at least part of the grinding circuit water might be recycled.

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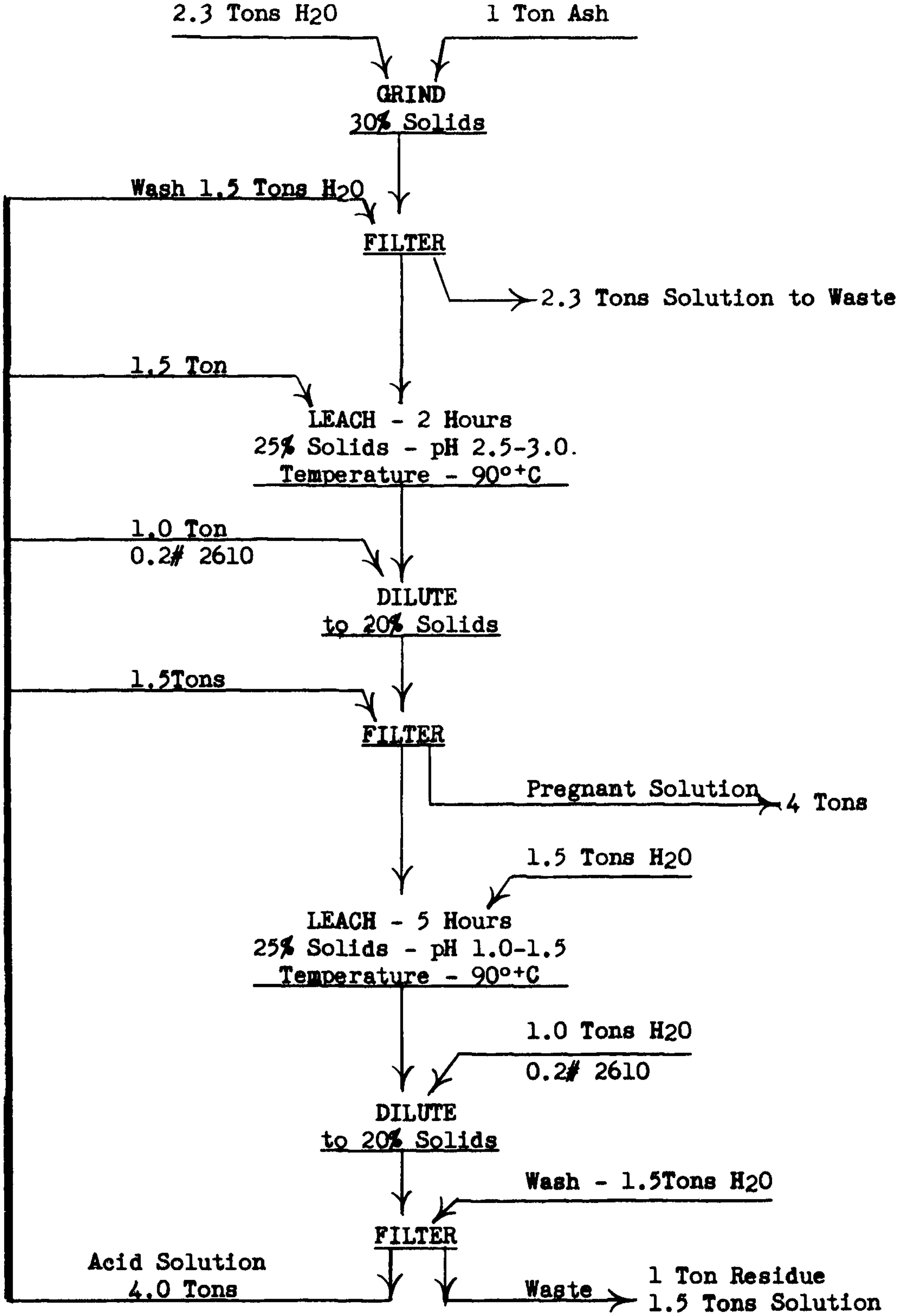
[1] Magno, P.J., "Precipitation of Molybdenum from Ion Exchange Feeds by Neutralization in the Presence of Iron", AEC Research and Development Report, WIN-48, 11/15/56



Flowsheet - Two Stage Leaching Test of Peter Kiewit Lignite Ash
Sample 185-18

Figure 7

The filtered ore from the grinding circuit was repulped at 25 per cent solids with strong acid leach liquor from the previous cycle and enough H_2SO_4 was added to give a terminal pH of 2.5 to 3.0 after leaching for 2.5 hours at 95°C. The pulp was then diluted to 20 per cent solids with recycled solution to which was added 0.2 pound Separan per ton of ore and then filtered and washed with the balance of the recycled solution.

The filter cake from the neutralizing leach was repulped at 25 per cent solids with H_2O and leached for five hours at 95°C with enough H_2SO_4 to give a terminal pH of 1.0 to 1.5. The pulp was then diluted to 20 per cent solids with H_2O containing enough Separan to give a concentration of 0.2 pound per ton of feed. The pulp was then filtered and washed. The residue was discarded and the solution advanced to the neutralizing leach of the next charge.

Eleven cycles were completed employing the flowsheet as shown in Figure 7. Uranium extractions of 95 per cent were realized using 1,085 pounds of H_2SO_4 per ton of lignite ash or 124 pounds of H_2SO_4 per pound of U_3O_8 extracted. To obtain comparable uranium extractions employing single-stage leaching, it was necessary to use approximately 1,400 pounds of H_2SO_4 per ton of ash or 158 pounds of H_2SO_4 per pound of U_3O_8 extracted.

Two-stage leaching of ashed 185-27 Manidon lignite was also studied on a cyclic basis. An average of the results obtained on eight cycles, after the system was in balance, indicated an acid consumption of 364 pounds of H_2SO_4 per ton of ash or 120 pounds per ton of lignite and an uranium extraction of 92 per cent. Data from single-stage leaching of this ash indicates that 525 pounds of H_2SO_4 per ton of ash or 175 pounds per ton of lignite was necessary to obtain 90 per cent extraction of the uranium.

The flowsheet employed with this ore is primarily the same as that employed for Sample 185-18 except the overall dilution for leaching and washing was lower, thereby giving a higher grade liquor. The detailed flowsheet for two-stage leaching of Sample 185-27 ash is included as Appendix Figure B-1.

Further countercurrent two-stage leaching tests were conducted on roasted lignite from other samples. The detailed results for two-stage leaching at 90° to 95°C are included in Appendix B, Tables B-3 through B-6. Two-stage tests at a leaching temperature of 70°C instead of 90°C resulted in lower uranium extractions and higher molybdenum concentrations in the pregnant solutions. The results are presented in Appendix B, Tables B-7 and B-8. The soluble losses indicated in the Appendix tables were determined by repulping the leached filter cakes and analyzing the resulting liquors.

The acid requirements of two-stage leaching tests of ash, compared with other methods of leaching both raw and ashed lignite, are summarized in Table 5. It is noted that two-stage countercurrent leaching of lignite ash provided substantially lower acid consumptions for equivalent uranium extraction. All of these two-stage tests were made at leaching temperatures of over 90°C.

Table 5

Acid Requirements of Lignite Ores Employing Various Leaching Procedures

Sample Number	Source	Type	Leaching Procedure	Lb H_2SO_4/Ton Raw Ore	% U_3O_8 Ext'n
185-18	Peter Kiewit	Raw	Single-Stage	1000	93.0
		Raw	Two-Stage	740	94.0
		Ash	Single-Stage	700	95.0
		Ash	Two-Stage	550	95.0
185-27	Manidon	Raw	Single-Stage	400	87.9
		Ash	Single-Stage	175	90.0
		Ash	Two-Stage	120	92.0
185-28	Northgate	Raw	Single-Stage	400	89.9
		Ash	Single-Stage	195	86.0
		Ash	Two-Stage	195	94.0
Composite 185-30,31 and 33	Ohio Oil	Raw	Single-Stage	530	90.0
		Ash	Single-Stage	300	91.0
		Ash	Two-Stage	240	90.0

The resultant leach liquor from two-stage leaching of lignite ash appears to be amenable to ion exchange or solvent extraction for recovery of uranium. The analysis of typical liquors produced by two-stage leaching of the samples tested is given in Table 6.

Table 6

Two-Stage Leach Liquor from Ashed Lignite

Sample Number	Grams/Liter					Sol'n:Feed Ratio	pH
	U_3O_8	Mo	Fe^{++}	Fe^{+++}	SO_4		
185-18	1.04	0.020	0.4	0.7	80	4.0:1	2.9
185-27	1.70	0.090	2.2	0.5	75	1.5:1	3.1
185-28	0.80	0.005	1.5	Nil	70	1.5:1	2.6
Composite[1] 185-30,31,33	2.50	0.015	2.0	Nil	80	1.5:1	2.5

[1] Equal weight.

Extraction efficiencies in each stage of the two-stage leaching tests on lignite ash are presented in Table 7. In all cases, a substantial proportion of the uranium was extracted in the first, or neutralizing, stage. As a consequence, washing of the final residue need not be as extensive as for single-stage leaching.

Soluble losses for one stage of filtration with one displacement wash after the strong acid leach ranged from 0.8 to 2.8 per cent of the total uranium extracted.

Table 7

Stage Efficiencies in Two-Stage Leaching Tests

Sample Number	Source	Neutralizing Stage pH	Neutralizing Stage % U_3O_8 Extraction	Strong Acid Stage pH	Strong Acid Stage % U_3O_8 Extraction
185-18	Peter Kiewit	2.7	83	1.1	12
185-27	Manidon	2.5	57	0	35
185-28	Northgate	2.5	58	0.1	36
185-30,31,33 1/	Ohio Oil	2.3	38	0	52

1/ Composite of Chrisofilli, Obrigewitch, and Chrusch samples.

C. Thickening and Filtration

1. Unleached Pulps

Filtration and thickening rates of unleached ground pulps of lignite ash were generally satisfactory with little or no addition of flocculant. The results of thickening tests on several ash samples are summarized in Table 9 and reported more completely in Appendix B, Table B-9.

2. Acid Leached Pulps

a. Thickening

Thickening tests on lignite ash after acid leaching under conditions previously determined as satisfactory for uranium extraction were performed using the Coe-Clevenger procedure. It was noted that the most satisfactory flocculation was obtained by addition of Separan as a dilute solution, or to a dilute pulp. The pulp appeared to reach the compression zone at a relatively low density, and flocculation without some dilution was not effective. The results of thickening tests are summarized in

Table 9, and detailed results are listed in Table B-10 of Appendix B. The majority of ash samples required thickener areas of less than 10 square feet per ton of solids per day, when 0.2 pound or less of Separan per ton of ash was added, but a few samples were not amenable to thickening regardless of Separan addition. The acid leached pulp of Sample 185-16 Bryco ash was tested in two stages of CCD with 0.25 pound of Separan per ton of ash added to the first stage. Cold water was used for dilution. The results are shown in Table 8.

Table 8

Thickening Tests of Bryco Ash Sample 185-16

Feed Dilution Lb Soln/Lb Solids	Area, Sq Ft/Ton/Day Stage 1	Area, Sq Ft/Ton/Day Stage 2	Terminal % Solids
4.0	0.93	1.50	53
3.0	1.02	1.77	53

Sample 185-16 was one of the more amenable samples tested and could obviously be processed in conventional CCD washing circuits.

Thickening tests on acid leached pulps prepared from the ash of Sample 185-8 (Peter Kiewit) have shown that this sample is extremely difficult to settle. This appears to be due to the high salt concentration which results from the excessive amount of acid (1200 to 1500 pounds per ton) required for leaching. This salt concentration is reduced somewhat by grinding in water before acid leaching. Without grinding to reduce the soluble salts, the thickener area requirement at a dilution of 10:1 was about 300 square feet per ton per day using two pounds of Separan per ton of ash.

Table 9 summarizes thickening tests on various neutral and acid leached lignite ash samples. Detailed results are included in Appendix B, Tables B-9 and B-10. The leached samples were ground to minus 35-mesh, the grinding water removed by filtration and the ash leached at 40 per cent solids for six hours at 75°C.

Table 9

Summarized Thickening Test Results - Lignite Ash

Sample Number	Source	Description	H_2SO_4 Lb/Ton Ash	Pulp pH	Separan Lb/Ton Ash	Terminal Density % Solids	Area Sq Ft/Ton/ 24 Hours
185-29[1]	Int'l Resources	Unleached	--	8.1	0.05	50	0.5
		Leached	1200	1.2	0.05	41	6.5
185-30,[2] 31,33	Ohio Oil	Unleached	--	8.3	0.05	46	1.4
		Leached	900	1.7	0.2	39	10.9
185-34	Ohio Oil	Unleached	--	4.1	0.05	42	0.7
		Leached	700	1.6	0.2	42	9.7
185-35	Homestake	Unleached	--	10.4	None	30	3.0
		Leached	1100	1.8	0.8	22	14.3
185-37	Int'l Resources	Unleached	--	7.5	0.05	43	0.6
		Leached	1100	1.9	0.7	42	29.0
185-38	Int'l Resources	Unleached	--	9.0	None	48	3.9
		Leached	1000	1.7	0.2	34	4.4

[1] Composite of Samples 185-29A through I
Equal weight composite

b. Filtration

Effective filtration of pulps from single-stage acid leached lignite ash required a prior stage of dilution and thickening. Filtration of the leach pulp directly from the leaching circuit was generally impractical. The filtration tests were made with a standard 0.1 square foot Oliver test leaf, using a coarse weave Nylon cloth[1] and 25 per cent submergence. It was noted that the best results were obtained when less than full vacuum (about 10 inches of Hg) was used on cake pickup, and full vacuum (25 inches) for the drain and wash portions of the filtration cycle. The high moisture retention of ash cakes resulted in high soluble losses of U_3O_8 in a single stage of filtration, and indicated that at least two stages of filtration, or additional preliminary washing by CCD, would be required for efficient recovery. The test results shown in Table 10 were obtained for acid leached Sample 185-16 (Bryco) after two stages of thickening, using 0.25 pound Separan per ton of ash.

Table 10
Filter Tests - After CCD on Bryco Ash - Sample 185-16

Cycle Time, Minutes	Cake Thickness Inches	% Moisture	Displacement Washes	Lb Dry Cake/ Sq.Ft.Filter Area/Day
1	1/2	46.5	1.72	2518
2	5/8	43.2	1.59	1960
4	1	43.2	1.10	1668

The thickener underflow was filtered at 50 per cent solids, using a vacuum of eight inches of Hg. The results indicate very high capacities and a moderate moisture content. The soluble loss was less than one per cent of the uranium extracted.

The results of filtration tests on other ash samples are summarized in Table 11. Detailed test results are included in Appendix B, Table B-11.

A few of the results reported in Table 11 are average values for identical tests. The individual tests appear in the Appendix B, Table B-11. It is obvious from the test results that wide variations in filter characteristics occur between individual ash samples. Adequate filtration rates were noted for most samples when 0.2 pound of Separan was used to flocculate the pulp in one stage of thickening, and the underflow filtered under moderate vacuum.

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[1] National Filter Media, Nylon, Style No.N.S. 1201

Table 11

Summarized Filter Test Data - Acid Leached Lignite

Sample Number	Source	Thickener, % Solids		Separan	Capacity	%	Soluble[1] Loss
		Feed	Discharge	Lb/Ton	Lb/Ft^2/Day	Moisture	%
185-19	Farmington Funding	32	35	0.25	1300	58	23
185-22	Manidon	32	45	0.15	800	40	9.6
185-27	Manidon	22	46	0.10	1800	43	7.2
185-28	Northgate	32	44	0.05	1800	46	15
185-29[2]	Int'l Resources	22	43	0.10	750	46	0.6
185-30,31,33[2]	Ohio Oil	27	43	0.15	2400	48	18
185-34	Ohio Oil	20	46	0.20	850	45	1.3
185-35	Homestake-Riley	26[3]	26	0.30	140	63	9.6
185-37	Int'l Resources	21	39	0.30	150	48	0.8
185-38	Int'l Resources	21	35	0.20	600	52	1.4

1/ Based on dissolved uranium in the thickener feed.

2/ Composite samples.

3/ No thickening occurred.

SECTION II

SOLVENT EXTRACTION OF ACID LIQUORS

A. Roasted Lignite

Because of the extremely high concentrations of molybdenum, ferric iron, and bisulfate present in the leach liquors prepared from most ashed lignite samples, solvent extraction offers certain advantages over other methods of uranium recovery and purification. For these reasons, an extensive investigation into the solvent extraction of uranium from lignite leach liquors was undertaken.

The solvent extraction of uranium from sulfuric acid leach liquors has been intensively investigated.[1,2,3,4] Of the many compounds which have exhibited promise as uranium extractants, only five are commercially available in production or pilot plant quantities. These are monododecylphosphoric acid (DDPA), di(2-ethylhexyl)phosphoric acid (EHPA), tri(isooctyl)amine (TIOA) a tertiary amine, and two secondary amines, Amine S-24 and Amine 9D-178. Of these, DDPA appeared least promising because of significant concentrations of titanium and zirconium and an unusually high concentration of aluminum in most lignite leach liquors. Therefore, no investigations into the use of DDPA were conducted.

The use of EHPA as an uranium extractant has been studied by Blake[1] et al. EHPA exhibits moderate selectivity towards uranium with molybdenum, zirconium, titanium, and ferric iron being the principal interfering elements found in lignite solutions. Ferric iron extractions can be minimized through its reduction to the ferrous state. The extraction of the other elements, however, must be accepted.

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[1] Blake, C.A., Brown, K.B., and Coleman, C.F., "The Extraction and Recovery of Uranium (and Vanadium) from Acid Liquors with Di(2-ethylhexyl)phosphoric Acid and Some Other Organophosphorous Acids", ORNL, ORNL-1903, May 13, 1955.

[2] Crouse, D.J. and Brown, K.B., "Amine Extraction Processes for Uranium Recovery from Sulfate Liquors, Volume I", ORNL, ORNL-1959, May 27, 1954.

[3] Crouse, D.J, Brown, K.B., Arnold, W.D., Moore, J.B., Lowrie, R.S., "Progress Report on Uranium Extraction with Organonitrogen Compounds", ORNL, ORNL-2099, May 14, 1956.

[4] Grinstead, R.R., Shaw, K.G., Long, R.S., "Solvent Extraction of Uranium from Acid Leach Slurries and Solutions", Proceedings of the International Conference on the Peaceful Uses of Atomic Energy, Volume 8, 71-76 (1956).

The re-extraction or stripping of uranium and contaminants from EHPA can be efficiently performed with aqueous sodium carbonate solution. Since the sodium salt of EHPA which is formed during the stripping operation has a low solubility in kerosene, it is necessary to modify the kerosene diluent with a few volume per cent of tributyl phosphate (TBP) or some long chain alcohol such as primary decyl alcohol (PDA). Once the kerosene diluent has been modified with the TBP or PDA, the solubility of the sodium salt of EHPA is greatly increased, and it becomes possible to strip uranium from EHPA in a continuous process.

Sulfuric acid leach solution prepared from various lignites were solvent extracted with a 0.1$\underline{M}$ EHPA - 0.1$\underline{M}$ TBP solution according to the flowsheet shown in Figure 8. The solvent extraction was performed in external mixer-settlers of the design depicted in Figure 9. Severe emulsions resulted with most lignite ash liquors, and operation of the system for a period greater than a few minutes was impossible.

Since emulsion formation with lignite leach solutions appeared to be the rule, means of avoiding this difficulty were investigated. Operation of the external mixer-settler unit at an elevated temperature greatly alleviated the problem. Maintaining the system at 45° to 50°C allowed emulsion-free operation. This, however, was not a particularly attractive solution to the problem since 45°C is near the flash point of kerosene.

Laboratory experiments indicated a second possible solution to the emulsion problem. It was found that if the organic-to-aqueous ratio in the mixer was maintained at about one to one, phase disengagement was excellent. To maintain such a high organic-to-aqueous ratio in the mixer would involve an organic recycle in each individual mixer-settler unit, since, in most operations, the organic-to-aqueous ratio is seldom greater than one to three. In order to provide this organic recycle automatically without the need of extra pumping equipment, an internal mixer-settler, shown in Figure 10, was designed. Through the use of this type of equipment, emulsion free performance was possible.

Lignite 185-8 from the Peter Kiewit property in the Rocky Ridge district was one of several lignite samples treated according to the flowsheet in Figure 8. The ashed lignite was sulfuric acid leached at 75°C using sufficient acid to give a terminal pH of 0.6 after a six hour leach period. The ore was filtered and washed to give a solution of the compositions shown in Table 12.

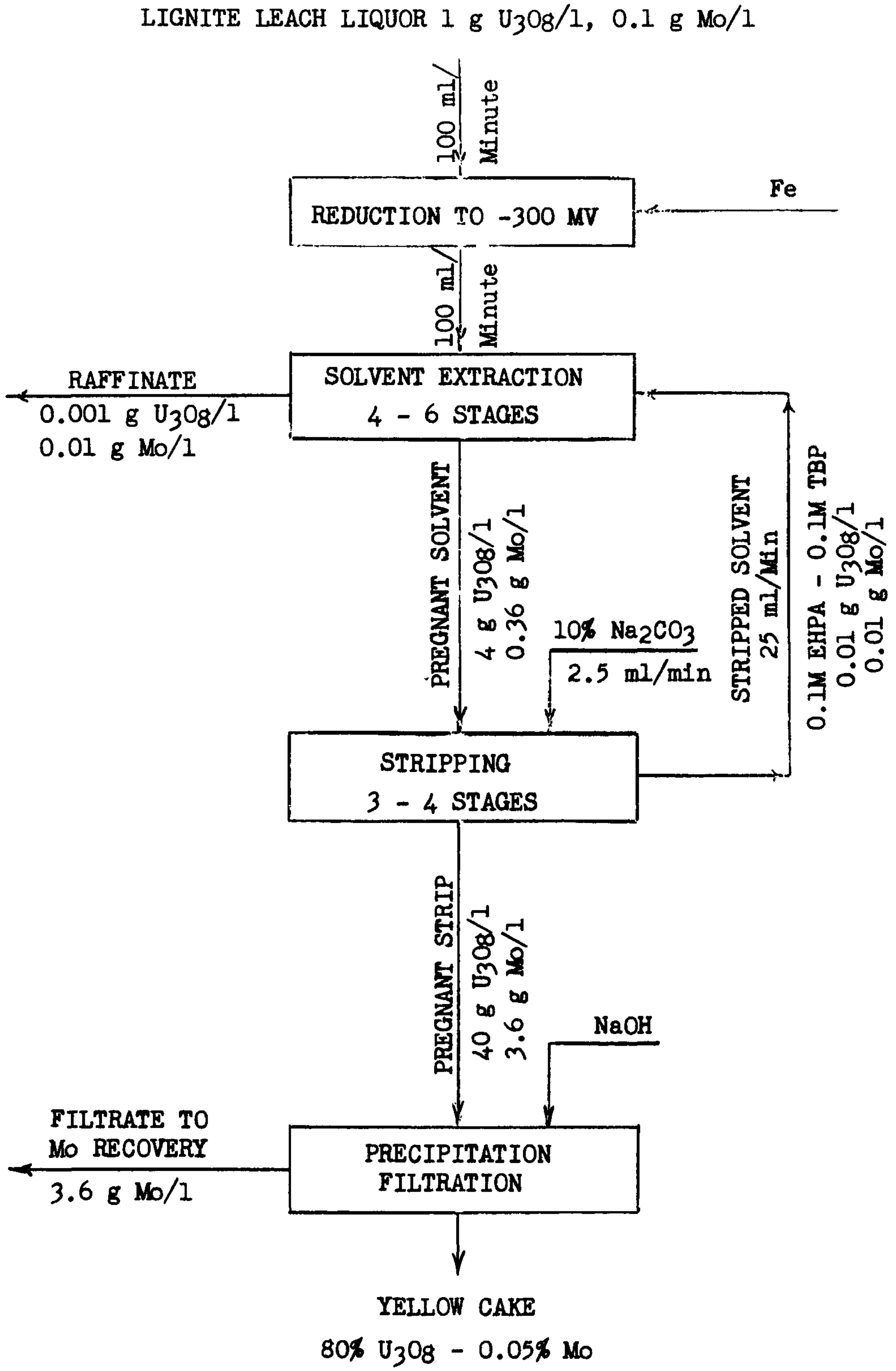


FIGURE 8

SOLVENT EXTRACTION OF LIGNITE LEACH LIQUOR WITH 0.1M EHPA - 0.1M TBP

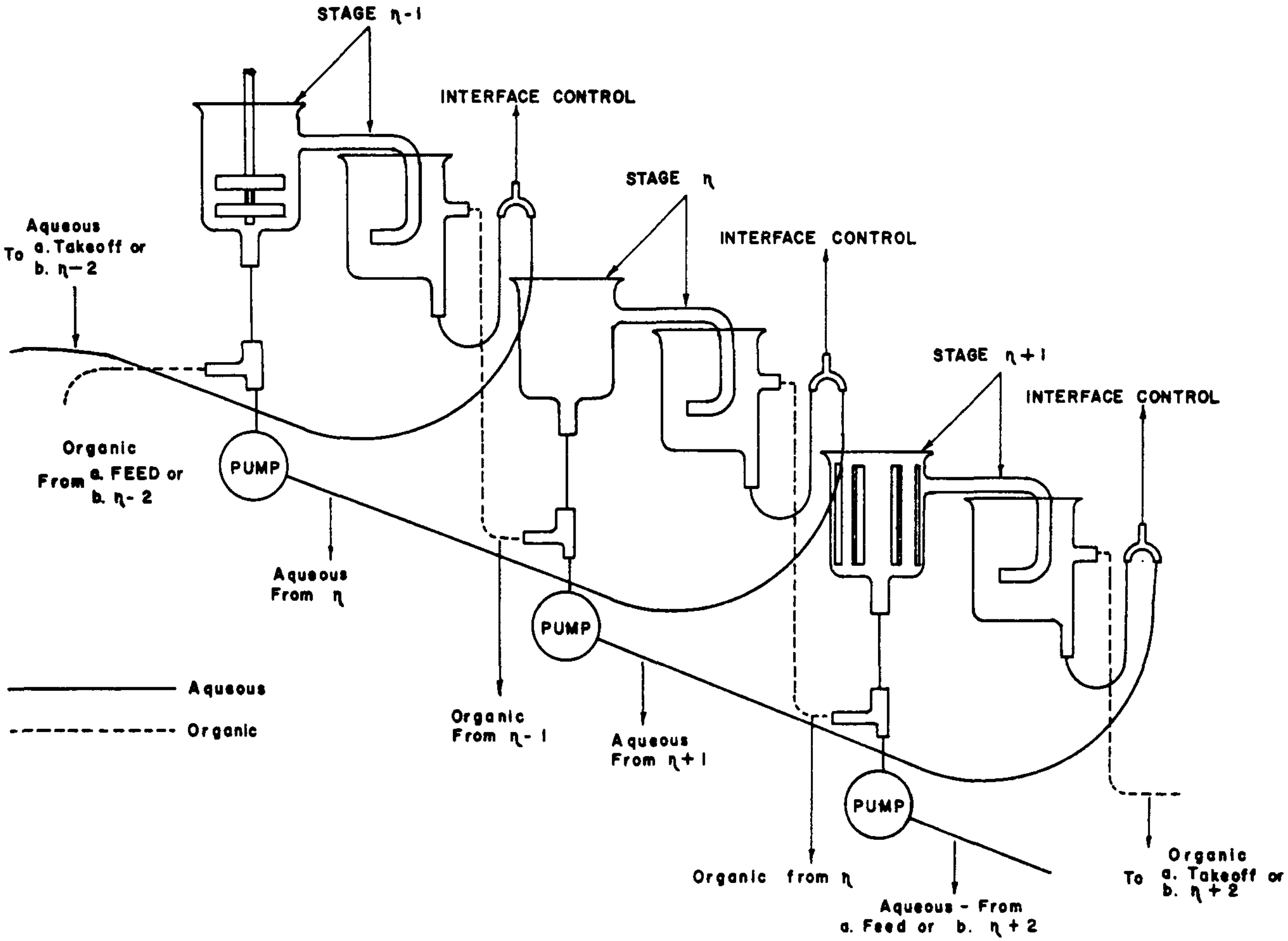
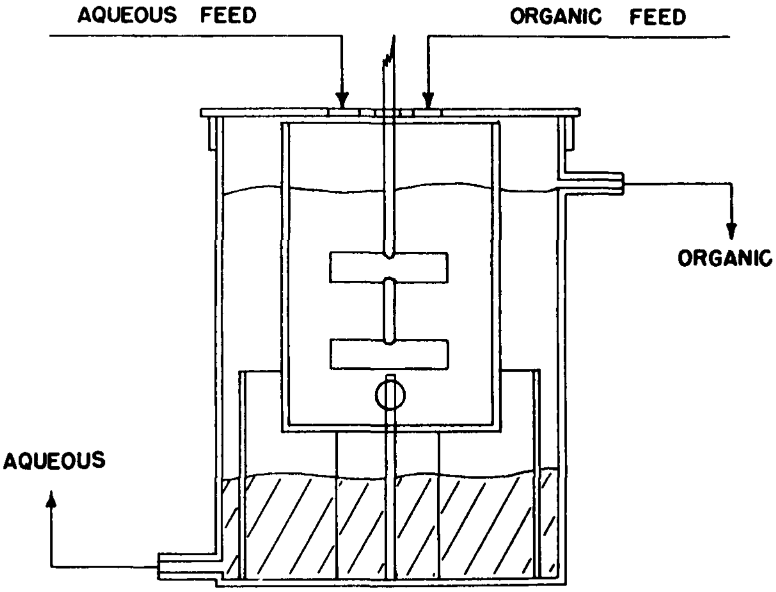


FIGURE - 9, EXTERNAL MIXER SETTLER SYSTEM



WINCHESTER INTERNAL MIXER SETTLER

FIGURE 20

Table 12

Solution Composition
Sample 185-8

Constituent	Grams/Liter
U_3O_8	1.1 - 1.2
Mo	0.1 - 0.15
Fe^{+++}	10 - 14
$SO_4^=$	140 - 180
Al_2O_3	20 - 30
pH	1.1

Prior to solvent extraction, the ferric iron was reduced with powdered iron metal to minus 290 millivolts (S.C.E.). The reduced solution assayed 21.0 grams Fe^{++} per liter and 0.2 gram Fe^{+++} per liter. In this case, iron consumption was about six pounds per pound of U_3O_8.

The reduced liquor was solvent extracted in six stages of internal mixer-settlers with 0.1M EHPA - 0.1M TBP in kerosene as the solvent. Using an organic-to-aqueous ratio of about one to four, uranium extraction was 99.6 per cent and molybdenum extraction 90 per cent.

The pregnant organic, assaying 4.0 grams U_3O_8 per liter and 0.4 to 0.5 gram Mo per liter was stripped in four stages with 10 per cent aqueous sodium carbonate solution at an organic-to-strip ratio of 10 to one. Under these conditions, the stripping of extracted uranium, molybdenum, titanium, zirconium, and iron was essentially complete. The stripped iron, titanium and zirconium precipitated in the stripping and concentrated in the aqueous phase. The movement of the stripping solution swept most of this precipitate out of the system, so that continuous operation was possible.

Two different techniques were used to recover uranium from the pregnant strip solution. One method involved the destruction of the sodium carbonate with sulfuric acid and the subsequent precipitation of uranium with ammonia. This yielded a product assaying 79.4 per cent U_3O_8 and five to 10 per cent molybdenum. This product does not meet the yellow cake molybdenum specification. It was found, however, that a low molybdenum yellow cake could be produced by precipitating the uranium directly from the carbonate strip with sodium hydroxide. This yellow cake product assayed 84.0 per cent U_3O_8 and 0.05 per cent molybdenum. The bulk of the molybdenum remained in the filtrate from which it can be recovered, if so desired.

Chemical cost data, as determined in the laboratory for an EHPA-TBP extraction of a solution assaying 1.0 gram U_3O_8 per liter and 12 grams Fe^{+++} per liter are shown in Table 13.

Table 13

Reagent Costs Per Pound U_3O_8 for EHPA-TBP Extraction Process

Reagent	Lb/Lb U_3O_8	Cost/Lb U_3O_8
Fe	6	$0.30
Na_2CO_3	2	$0.05
H_2SO_4	1	$0.01
NaOH	0.75	$0.04
EHPA	0.05	$0.04
TBP	0.05	$0.03
Total		$0.47/Lb U_3O_8

The recovery of uranium from lignite leach liquors with EHPA suffers from several disadvantages. The iron reduction step is costly. The precipitation of iron, titanium, and zirconium in the stripping unit, while not particularly troublesome in the laboratory, could be very troublesome in a mill. The molybdenum contamination problem, although it can be circumvented through the use of a caustic precipitation, is an additional expense. For these reasons, the use of EHPA was at least temporarily abandoned in order to investigate the use of amine extractants.

The use of amine extractants has several advantages over the use of EHPA. The amines, 9D-178, TIOA, or S-24, are very selective towards uranium with respect to ferric iron, zirconium, titanium, aluminum, and vanadium; and it is not necessary to reduce the solution prior to solvent extraction. The amines will extract molybdenum nearly quantitatively, but a very clean uranium-molybdenum separation can be affected through the use of a sodium chloride stripping solution. Chloride ion will quantitatively re-extract uranium while leaving the bulk of molybdenum in the solvent. The molybdenum can then be easily removed with an alkaline wash using either aqueous sodium carbonate or ammonia solution. There is little precipitation in the stripping unit since any extracted iron is removed from the solvent in the chloride stripping process and therefore is not present during the alkaline wash.

Lignite solutions have been processed with amine extractants according to the flowsheet shown in Figure 11; three different amines were used, TIOA, Amine S-24, and Amine 9D-178. In all cases, a five-volume per cent amine solution in kerosene containing 2.5 volume per cent PDA was the solvent. The purpose of the PDA is to increase phase disengagement rates and prevent third phase formation in the extraction circuit.

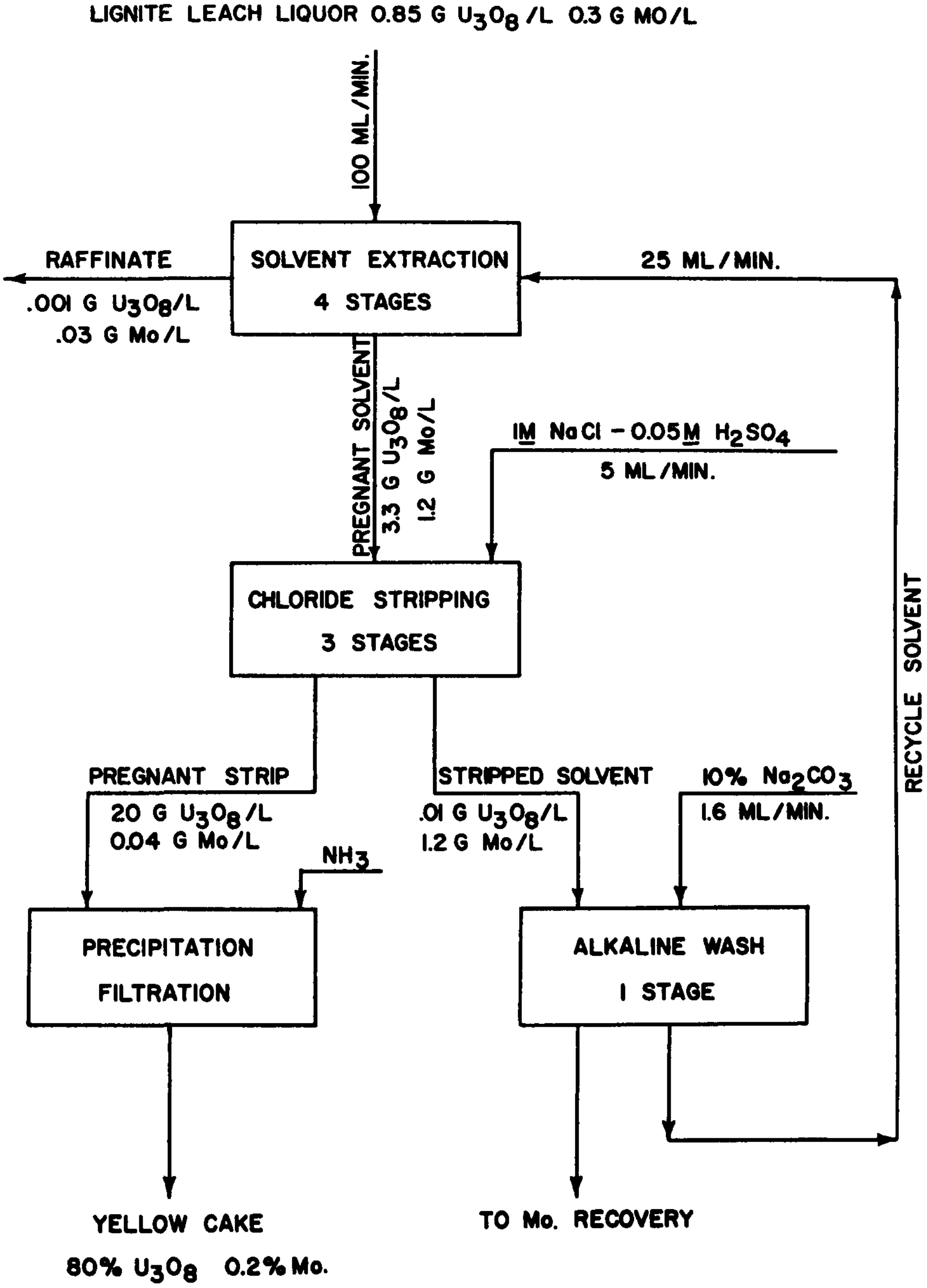


Figure 11. Flowsheet for Solvent Extraction Circuit

The composition of the leach liquor prepared from ashed lignite 185-8 is shown in Table 14.

Table 14

Leach Liquor Composition
Sample 185-8

Constituent	Grams/Liter
U_3O_8	0.85
Mo	0.37
Fe	8.8
Fe^{+++}	6.0
SO_4	130

This solution was solvent extracted to recover uranium using the flowsheet indicated in Figure 11. Amine S-24 was the extractant. The extraction circuit consisted of four stages of internal mixer-settlers. At an organic-to-aqueous ratio of four to one, uranium extraction was 99.9 per cent complete while about 95 per cent of the molybdenum was also extracted. The pregnant solvent, assaying 3.3 grams U_3O_8 per liter and 1.2 grams Mo per liter, was stripped in three stages of external mixer-settlers with a 1M NaCl 0.05M H_2SO_4 solution. The organic-to-stripping solution ratio was five to one. Uranium stripping was in excess of 99 per cent while molybdenum stripping was negligible.

The molybdenum was removed from the uranium-free solvent by washing in one stage with 10 per cent aqueous sodium carbonate at an aqueous-to-organic ratio of about 15 to one. The washed solvent was nearly free of molybdenum while the carbonate washing solution assayed 20 grams of Mo per liter.

Uranium was precipitated from the hot (60°C) pregnant chloride stripping solution with ammonia gas, and the yellow cake filtered, washed, and dried at 100°C. The final product assayed 78.5 per cent U_3O_8, 0.33 per cent Fe, 0.05 per cent V_2O_5, 0.2 per cent Mo, and 0.009 per cent Cl.

Molybdenum was recovered from the carbonate wash solution. The acidity of the solution was adjusted to two volume per cent free sulfuric acid and MoS_3 precipitated with H_2S. The sulfide cake, after washing and drying, assayed 90 per cent MoS_3.

Reagent costs for the solvent extraction of a leach liquor by Amine S-24 are listed in Table 15. It is assumed that the leach liquor contains 1.0 gram U_3O_8 per liter and 0.3 to 0.4 gram Mo per liter. For a liquor prepared by two-stage leaching, the molybdenum concentration would be expected to be much less than the concentration shown here for single-stage leaching. Results for the extraction of uranium from a Northgate calcine (185-28), with Amine S-24, are shown in Appendix C, Table C-1.

Table 15

Reagent Costs for the Solvent Extraction of Uranium Using Amine S-24

Reagent	Lb/Lb U_3O_8	Cost/Lb U_3O_8
NaCl	3.5	$0.035
NH_3	0.25	$0.015
Amine	0.07	$0.07
PDA	0.02	$0.006
Total		$0.126

No emulsion formation was encountered as long as the internal mixer-settlers were used. As with EHPA, severe emulsion problems were encountered when operation in an external mixer-settler system was attempted. Once again, however, operation at 45°C eliminated emulsion formation. In general, emulsion formation was more severe when using EHPA than when operating with an amine solvent.

B. Raw Lignite

The recovery of uranium from leach liquors prepared from the acid leaching of raw lignite was investigated. The treatment of raw lignite presents most of the problems encountered when treating calcined material. In addition, the presence of high concentrations of water-soluble organic matter further complicates the treatment of acid leach liquors prepared from raw lignite.

The use of an amine as an extractant for uranium from raw lignite leach solutions was not successful. Many lignite samples, and in particular Northgate and Peter Kiewit samples, gave precipitation during the extraction step. In addition, large quantities of organic material were extracted into the amine solvent, thereby lowering the capacity for uranium. In all cases, stripping of the pregnant amine with either acidified sodium chloride or ammonium nitrate gave emulsions and precipitation. It was possible to strip the amine of all extracted uranium, molybdenum, and organic material without precipitation or emulsion formation by using aqueous sodium carbonate. The resulting strip solution, a black viscous solution, could not be completely freed of its uranium values by precipitation or hydrogen reduction.

The solvent extraction of uranium from raw lignite with EHPA met with more success, since the solvent extracted little organic material from the liquor. However, a gelatinous precipitate was formed with most lignites, Bryco and Manidon lignites being exceptions, which interfered with operation. Also, the cost of reducing ferric iron was extremely high. While the use of EHPA as a solvent for uranium from raw lignite solutions is possible, it is, at best, an unattractive process.

SECTION III

FLOTATION OF UNBURNED CARBON TO IMPROVE ROASTING AND LEACHING EFFICIENCY

In order to produce leach liquor which is amenable to solvent extraction or ion exchange, it is desirable to roast the lignite ore. Uranium extraction is considerably better for a dead roasted lignite than from ore containing remnants of unburned carbon. A Grand Junction Pilot Plant fluosolids calcine, for instance, was found to yield only 83 per cent uranium extraction (residue assay - 0.050 per cent U_3O_8) when leached with the optimum amount of acid for muffle roasted ash. However, when the sample was dead burned in a muffle prior to leaching, the extraction increased to 94.4 per cent (residue assay - 0.017 per cent U_3O_8) when leached under the same conditions with respect to time, temperature, and acid addition.

In practice, it is difficult and expensive to produce a dead-burned ash. From Figure 12 it may be noted that two and one-half hours of muffle roasting at 450°C, with rabbling at 15 minute intervals, were required to produce a 100 per cent dead-burned roast. However, 92.6 per cent of dead roast weight was reached in one and one-quarter hours or 50 per cent of the dead roasting time. As a consequence of the high roasting efficiency during the early stages of burning, flotation was considered as a tool to remove unburned carbon, in order to avoid the necessity of dead roasting lignite ores in a single pass.

When carbon was removed from fluosolids calcine leached residue by a standard coal flotation method, the residue assay was reduced from 0.050 per cent U_3O_8 to 0.018 per cent U_3O_8, yielding a 94.4 per cent recovery for the leach-flotation process. The carbon concentrate, representing 8.1 per cent of the residue weight, assayed 0.41 per cent U_3O_8. The details of the test are shown in Appendix Table D-1.

A series of tests were made in which minus 10-mesh charges of an equal weight composite of Ohio Oil Samples 185-30, 31, and 33 were roasted for varying fractions of dead roast time (two and one-half hours) at 450°C in a muffle. The charges were rabbled at 15 minute intervals. Each calcine was ground for two and one-half minutes in a laboratory rod mill. One group of calcine charges was leached with H_2SO_4 to a terminal pH of 0.5 to 0.6 for six hours at 40 per cent solids and 75°C. The leached residue was filtered, washed, neutralized with lime, conditioned with fuel oil at 40 per cent solids, and floated

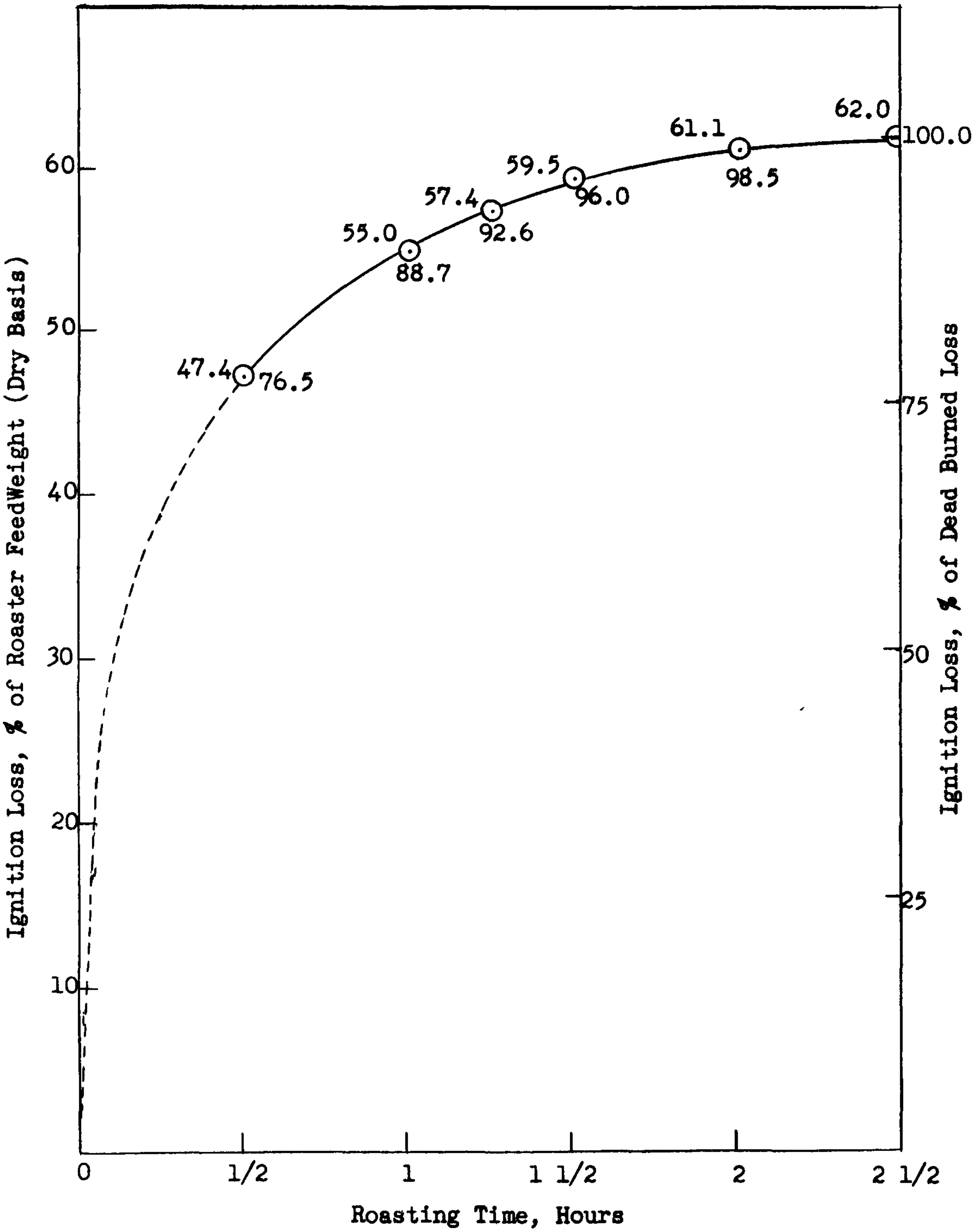


Figure 12. The Effect of Roasting Time on Ignition Loss of Equal Weight Composite Sample 185-30, 31, 33

in a Fagergren flotation machine at about 25 per cent solids using pine oil frother. The data and results, as well as a comparison with a dead roasted leached lignite, are shown in Appendix Table D-2. Another group of ground calcine charges was conditioned at approximately 40 per cent solids with fuel oil and floated at 25 per cent solids to remove a carbon concentrate. The flotation tailing was then leached under conditionssimilar to those described above. The data and results of these tests are shown in Appendix Table D-3.

In addition, a third group of charges, which had been roasted for 50 per cent of dead roast time, was treated by cyclic leach-flotation and flotation-leach tests to determine the effect of recycling flotation concentrate on the roaster. The flowsheets for these tests, which are similar to the batch tests, are shown in Appendix D, Figures D-1 and D-2. The data and results are shown in Appendix D, Tables D-4 and D-5. A summary of the important differences between the methods have been included in Table 16.

Aside from the obvious reduction in the size of the roaster needed to treat a given tonnage of raw lignite, which is nearly the same for either process, the outstanding difference between the leach-flotation and flotation-leach procedures is the consistently better U_3O_8 extraction obtained by leaching ahead of flotation. This is consistent with the results of the cyclic tests. The flotation tailing assays for leach-flotation, presented in Table D-4, tend to decrease with each cycle; whereas, the residue assays for the flotation-leach in Table D-5 show the reverse trend.

Three characteristics of the leach-flotation circuit account for the better extractions. First, between 25 and 50 per cent of the U_3O_8 associated with the carbon is extracted during the leach; thus, the carbon concentrate, which is recycled to roasting, is lower grade than that produced in the flotation-leach process. Second, the carbon is continuously circulated through the roasting and leaching circuits until it is completely ashed. In the flotation-leach circuit, the calcine does not reach the leaching circuit until it has been dead burned; thus, the calcine is leached only once. Third, the carbon appears to float better after leaching than it does before leaching.

Table 16

Leach-Flotation (L-F) and Flotation-Leach (F-L) Data on Composite Lignite Sample 185-30, 31, 33 Roasted for Various Fractions of Dead Roast Time

	Per Cent Dead Roast Time														
	1/2 Hour 20 Per Cent		3/4 Hour 30 Per Cent		1 Hour 40 Per Cent		1 1/4 Hour 50 Per Cent		1 1/4 Hour 50 Per Cent Cyclic Test		1 1/2 Hour 60 Per Cent		2 Hours 80 Per Cent		2 1/2 Hours 100 Per Cent Leach Only
	L-F	F-L	L-F	F-L	L-F	F-L	L-F	F-L	L-F	F-L	L-F	F-L	L-F	F-L	
Roaster Capacity Requirement (% of Dead Roast Capacity)	30	29	29		47	46	56	54	56	54.3	62	62	81	81	100
Unit Feed Weights (% of Dry[1/] Lignite Feed Weight)															
Roast	149	145	131		117	115	112	107	112.0	108.6	104	103	101	101	100
Leach	83	34	68		55	38	50	39	47.2	38.4	42	39	40	38	40
Residue Filtration	71	24	58		43	27	39	27	38.5	27	31	27	30	27	30
Flotation	71	76	58		43	52	39	46	38.5	47	31	42	30	39	0
Carbon Concentrate Filtration	49	42	32		18	14	12	7	12.0	8.6	4	3	1	1	0
Reagent Consumption, Lb/Ton of[2/] Raw Lignite Ore															
H_2SO_4 (Terminal pH = 0.5 to 0.6)	407	186	421		418	262	431	350	448	391	453	291	461	307	480
No. 2 Fuel Oil	25	50	10		5	10	2.5	5	2.5	2.5	1.5	3.0	1.0	2.0	None
Pine Oil	0.58	--	0.33		0.25	--	0.2	0.5	0.2	0.2	0.15	0.3	0.15	0.25	None
Lime (Neutralization after leach)	11.6	None	9.9		6.6	None	2.5	None	9.0	None	1.5	None	1.0	None	None
Final Waste Product Assay, % U_3O_8 (Flotation Tail or Residue)	0.007	0.040	0.032		0.030	0.058	0.039	0.055	0.041	0.070	0.046	0.072	0.049	0.079	0.051
U_3O_8 Loss in Waste Product, % of U_3O_8 in Lignite Feed	0.5	3.1	2.7		2.7	6.6	4.1	6.6	5.2	7.9	5.5	9.6	6.5	10.6	6.5

[1/] With the exception of the cyclic tests, all weights are based on batch test results and include a calculated weight buildup throughout the circuit resulting from recycling the carbon flotation concentrate to the roasting step.

[2/] Batch test reagent consumption with the exception of cyclic tests.

The improved U_3O_8 extraction in the leach-flotation process is not without its price. The acid consumption in this process is substantially higher. The cyclic tests shown in Table 16 indicate that extraction is improved 2.7 per cent at the expense of 75 pounds of H_2SO_4 per ton of raw lignite. To this must be added the expense of leaching and filtering a higher tonnage of calcine. The leaching circuit, during leach-flotation, carries a 23 per cent greater load than the same circuit in the flotation-leach process. Similarly, the residue filtration circuit carries a 30 per cent greater load. These factors weigh heavily on the side of taking the higher residue loss resulting from the flotation-leach process.

In addition to the tests made on Grand Junction Pilot Plant fluosolids calcine and composite Sample 185-30, 31, and 33, other tests were made on lignite samples of varying U_3O_8 and hydrocarbon content. In these tests, the leaching of a dead-roasted lignite sample was compared with leach-flotation and flotation-leach procedures on charges roasted at approximately 50 per cent of dead roast time. The conditions and results are shown in Appendix D, Table D-6.

With the exception of high ash Sample 185-43 (Landis-Rodakowski), on which no roasting was done prior to leach-flotation or flotation-leach, the recoveries were generally comparable to those obtained on Sample 185-30, 31, and 33 and the roaster capacity requirement at 50 per cent of dead roast time was 55 to 60 per cent of dead-roast capacity. The flotation technique for each ore is somewhat different and additional tests are needed to establish optimum conditions.

Since Sample 185-43 contained only three per cent total carbon, an attempt was made to float the carbon without roasting. Although the results were successful, there seems little justification for additional processing since the ore leached well without roasting.

SECTION IV

ROASTING INVESTIGATIONS

Almost at the start of the lignite test program it became apparent that filtration or thickening of raw lignite was impossible after carbonate leaching and difficult after acid leaching. In addition, recovery of uranium by solvent extraction or ion exchange, from solutions produced from raw lignite, was practical only after expensive purification with activated charcoal. As a consequence, a major effort was devoted to the investigation of different methods of producing a calcine or char which would be amenable to acid and/or alkaline leaching and yield a pulp which could be settled or filtered and a pregnant solution containing no significant concentrations of soluble organic compounds.

Test work on removal of interfering organic constituents were conducted with two objectives in mind:

1. Roasting or burning to eliminate all organic material, and
2. Charring or coking to destroy volatile and soluble organic materials but not fixed carbon.

In the processing of uranium ores, roasting is frequently practiced; however, the temperature must be closely controlled since, at temperatures above 600°C, the extraction of uranium is sharply decreased. The lignites are no exception. Figure 13 shows the effect of roasting temperature on the subsequent extraction of uranium by acid leaching. In this series of tests, charges of dry lignite (Sample 185-18) were roasted in the presence of excess oxygen for two hours, at the indicated temperature, in an electrically heated muffle or assay furnace. The roasted ore was then cooled and leached for six hours at 75°C with 700 pounds of H_2SO_4 per ton of original raw lignite. As will be noted, maximum extraction was obtained from the calcine roasted at 450°C. As indicated on the chart, the second curve represents the leaching results of ore from the Belfield area, North Dakota, which was roasted in the multiple hearth roaster. The temperatures shown are the average temperatures of the hottest hearth during the period the sample was collected. Similar tests on other samples yielded comparable results, and it was apparent that a maximum furnace temperature of the order of 450° to 550°C was desirable. Actual bed temperatures were probably more nearly 600° to 650°C, as estimated from the color of the burning carbon particles.

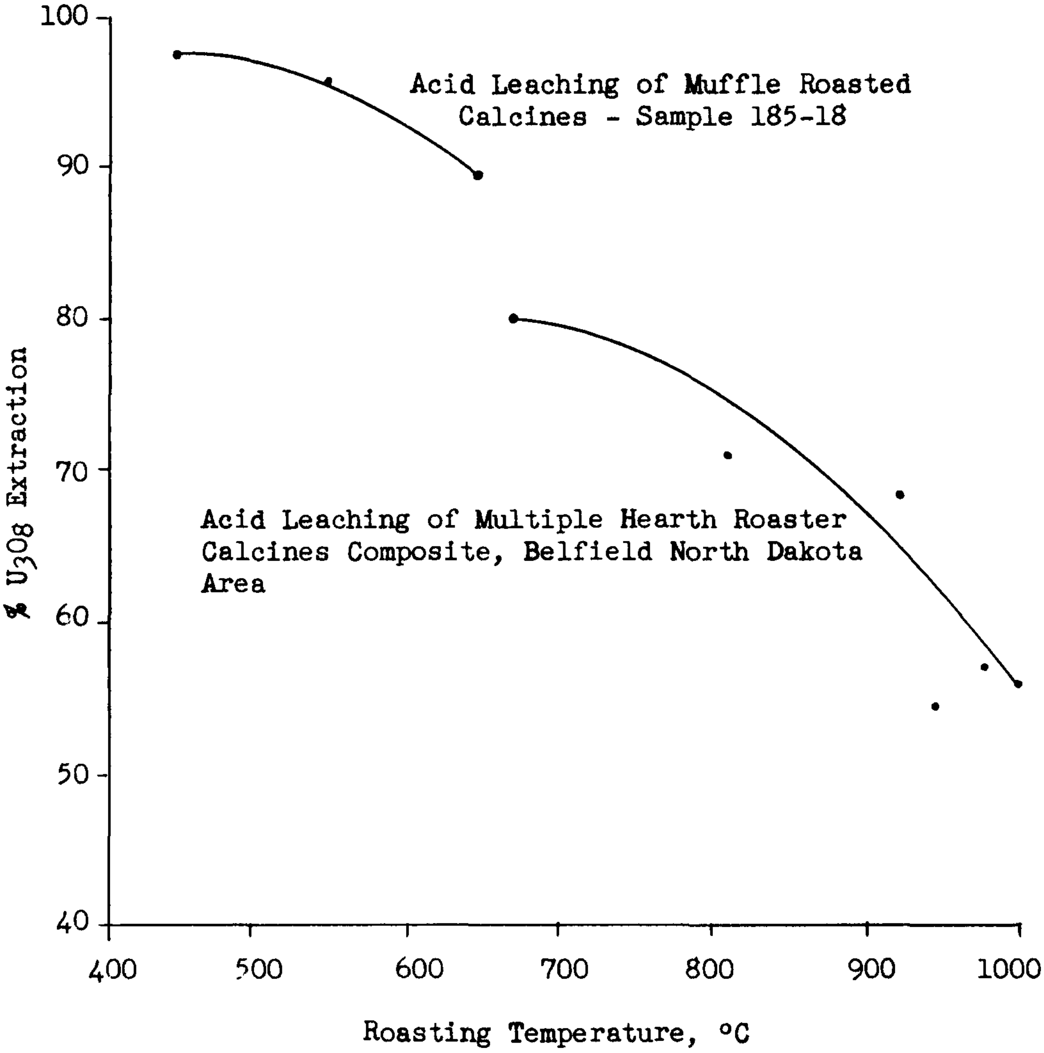


Figure 13. The Relationship of Roasting Temperature to U_3O_8 Extraction During Acid Leaching

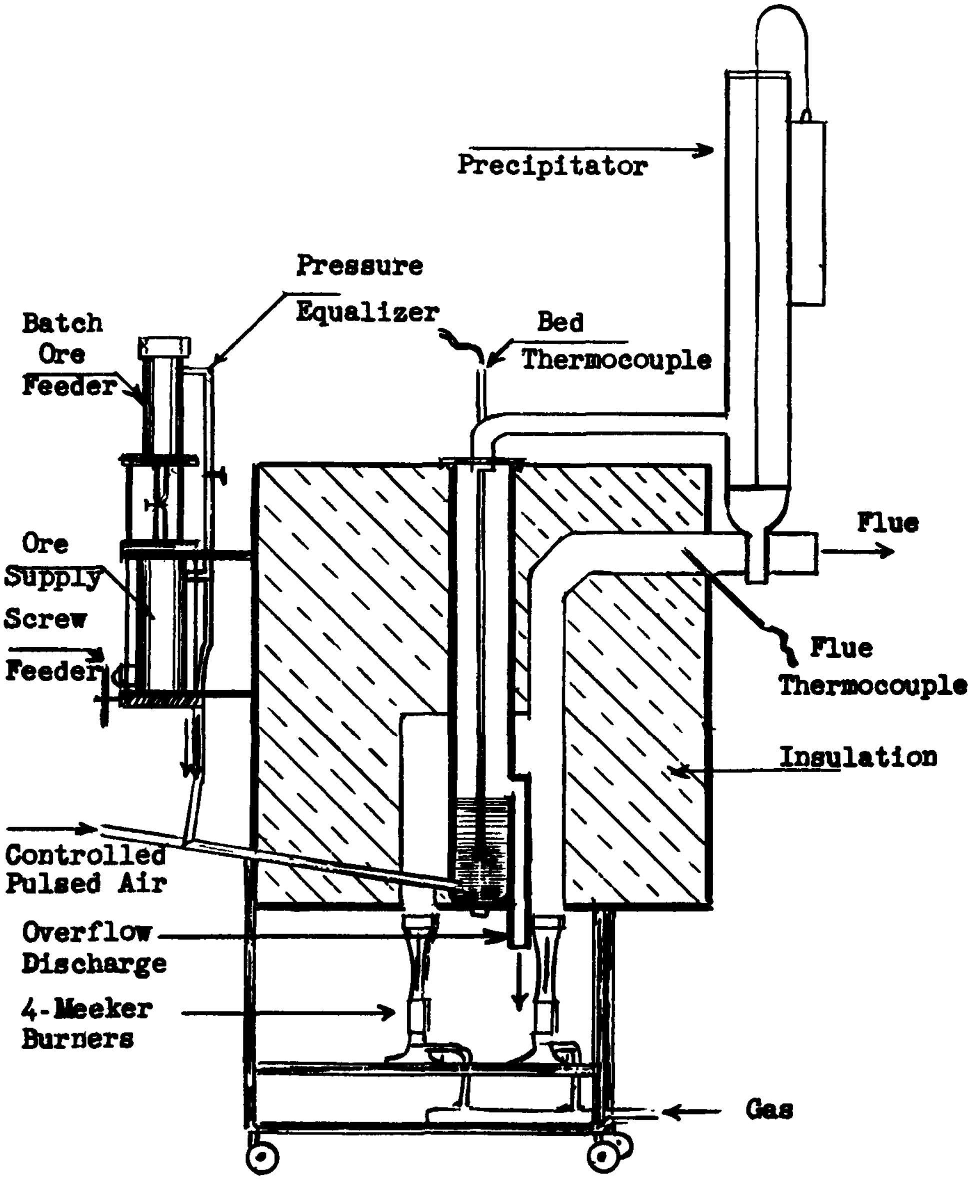
Control of the burning temperature on a larger scale, however, presents serious heat dissipation problems. The lignites have a fuel value ranging from about 3000 to 5000 BTU, and it is obvious that burning rate and heat dissipation are closely related. The principal objective, therefore, was to determine what type of roasting procedure would yield a leachable calcine at minimum roasting cost. Four types of roasting were investigated:

1. Fluosolids Roasting
2. Multiple Hearth Roasting
3. Moving Grate Roasting
4. Flash Roasting

A. Fluosolids Roasting

Fluosolids roasting was conducted in a laboratory size fluosolids roaster, constructed at the Winchester Laboratory, and shown diagramatically in Figure 14. The roasting chamber consists of a 4 1/4" by 34" stainless steel tube enclosed in a gas fired muffle. Heat is supplied by four Meker burners. Feed is introduced into the bottom of the roasting chamber by entrainment in the air or gas used for fluidization of the bed and is fed into the air stream by a screw feeder. The calcine overflows at the side eight inches above the bottom of the bed, and the dust laden gases pass on through the top to an electrostatic precipitator. The power supply for the precipitator consists of a radio frequency oscillator circuit, transformer, and rectifier similar to that used in the high voltage section of a television set.

Laboratory fluosolids roasting tests were made on Sample 185-4, 185-19, 185-20, and 185-21 representing, respectively, Bryco, Farmington, Manidon, and Northgate lignites. At a retention time of approximately two hours, equivalent to a feed rate of 320 to 430 pounds of dry lignite per square foot of cross-sectional area per 24 hours, satisfactory roasting of Samples 185-4, 185-19, and 185-20 was obtained, and no difficulty was experienced in holding the bed temperature in the range of 400 to 500°C. Sample 185-20, containing 45 per cent carbon, presented a roasting problem in that the bed temperature could be held below 600°C only by blending the sample with other feeds of lower carbon content, or by diluting the fluidizing air with an inert gas, such as nitrogen, and drastically decreasing the feed rate. In other tests, water was fed into the bed with negative results.



Fluosolids Roaster

Figure 14

In those tests in which satisfactory roasting was obtained, the flow of fluidizing air was 0.92 cubic feet per minute. This is equivalent to a space rate, under standard conditions, of approximately 0.16 and was nearly the minimum required to fluidize the bed. In addition to the fluidizing air, a small flow of air was introduced just above the surface of the bed since gas analyses indicated a deficiency of oxygen in the freeboard.

Results of acid leaching tests on calcines from the laboratory fluosolids roasting tests are presented in Table 17 and show that the uranium extractions from Samples 185-4, 185-19, and 185-21 ranged from 85 to 90 per cent at terminal leaching acidities of approximately pH 0.5. This is five to 10 per cent lower than the extractions obtained, at comparable acid concentrations, using ash prepared by careful muffle roasting. The substandard extractions were the result of over-roasting of the calcines and under-roasting of the dust fractions.

In addition to the laboratory fluosolids roasting studies, larger scale roasting tests were conducted with the three-foot diameter fluosolids roaster, by personnel of the Grand Junction Pilot Plant. Feeds used in these tests were lignite from Stockpile No. 5 at Edgemont and from the Northgate properties in North Dakota. Detailed information on these roasting tests will be presented in a later report by personnel of the Pilot Plant. During the course of the tests, however, samples of calcine and dust from the test on Stockpile No. 5 lignite were tested at Winchester for amenability to acid leaching.

The samples of calcine and dust were representative of a test run at a feed rate of 350 pounds of dry lignite (16 per cent moisture) per hour. The roaster bed temperature was 650°C, and the freeboard temperature was 732°C during the period in which the samples were obtained.

Acid leaching tests, as reported in Table 18, indicated an overall uranium extraction of 84 per cent using 518 pounds of H_2SO_4 per ton of composite roaster product (60 per cent calcine - 40 per cent dust) when leached for six hours at 50 per cent solids and 75°C. Under the same conditions, the extraction was increased to 87 per cent by addition of 10 pounds of $NaClO_3$.

The calcine and dust received from Grand Junction contained visible amounts of unburned carbon and had an overall grey-brown color, indicative of reducing conditions. Tests were therefore conducted to investigate the effect of reburning, under oxidizing conditions, on the subsequent extraction of uranium.

Table 17

Acid Leaching Tests of Laboratory Fluosolids Roaster Products

Sample Number	Source	% U_3O_8	Description	Roasting Conditions Time[1] Hours	Roasting Conditions Temp °C	Leaching Conditions[2] Temp °C	Leaching Conditions[2] Weight %	Leaching Conditions[2] H_2SO_4 Lb/Ton[3]	Terminal Conditions pH	Terminal Conditions Emf(mv)	Residue Assay % U_3O_8	% U_3O_8 Ext'n
185-4	Bryco	0.39	Calcine	2	400	40	81	490	0.65	-435	0.059	85.0
		1.1	Dust	2	400	40	19	770	0.45	-440	0.14	89.5
		1.1	Dust	2	400	40	19	1050	0.0	-450	0.077	94.6
			Composite	2	427	80	100	151	3.0	-300	0.30	44.7
			Composite	2	427	80	100	302	3.15	-315	0.16	71.3
			Composite	2	427	80	100	453	1.25	-385	0.10	84.1
			Composite	2	427	80	100	604	0.6	-410	0.089	85.9
185-19	Farmington	0.82	Dust	2	400	75	28	980	0.7	-425	0.087	90.6
		0.33	Calcine	2	400	75	72	470	0.75	-445	0.035	88.9
		0.74	Dust	2	500	75	30	980	0.6	-425	0.083	89.5
		0.31	Calcine	2	500	75	70	470	0.6	-410	0.032	88.7
185-21	Northgate	0.35	Dust	2	480	75	32	666	0.55	-440	0.066	81.4
		0.096	Calcine	2	480	75	68	446	0.6	-460	0.014	86.7
		0.32	Dust	2	650	75	34	633	0.4	-440	0.11	67.1
		0.10	Calcine	2	650	75	66	515	0.4	-465	0.026	76.0

[1] Burning rate on ores tested varied between 320 and 430 pounds of dry lignite per square foot of cross-sectional area per day.

[2] All tests leached for six hours on unground roaster products. Thirty-three per cent solids for dust; 40 per cent solids for calcine.

[3] Based on leach feed.

Table 18

Acid Leaching of Fluosolids Products from Grand Junction Pilot Plant

Sample 185-36

		Leaching Conditions[3]			Terminal Conditions		Residue Assay	
Description	% U_3O_8	Time, Hours	Lb/Ton H_2SO_4	Lb/Ton $NaClO_3$	pH	Emf(mv)	% U_3O_8	% U_3O_8 Ext'n
Calcine	0.157	6	475	0	0.55	-430	0.027	83.8
Dust	0.433	6	438	0	0.8	-425	0.093	79.8
Dust	0.433	6	584	0	0.3	-450	0.073	84.7
Dust	0.433	24	1300	0	0.0[4]	-470	0.053	89.7
Composite[1]	0.27	6	518	0	0.65	-435	0.048	83.7
Composite[1]	0.27	6	518	10	0.75	-675	0.036	87.6
Calcine[2]	0.16	6	475	0	0.3	-465	0.014	91.7
Dust[2]	0.45	6	584	0	0.25	-495	0.019	96.1

[1] Composite: 60 per cent calcine; 40 per cent dust.

[2] Re-roasted for two hours at 450°C prior to leaching.

[3] 75°C at 50 per cent solids.

[4] Free acidity approximately 300 pounds per ton.

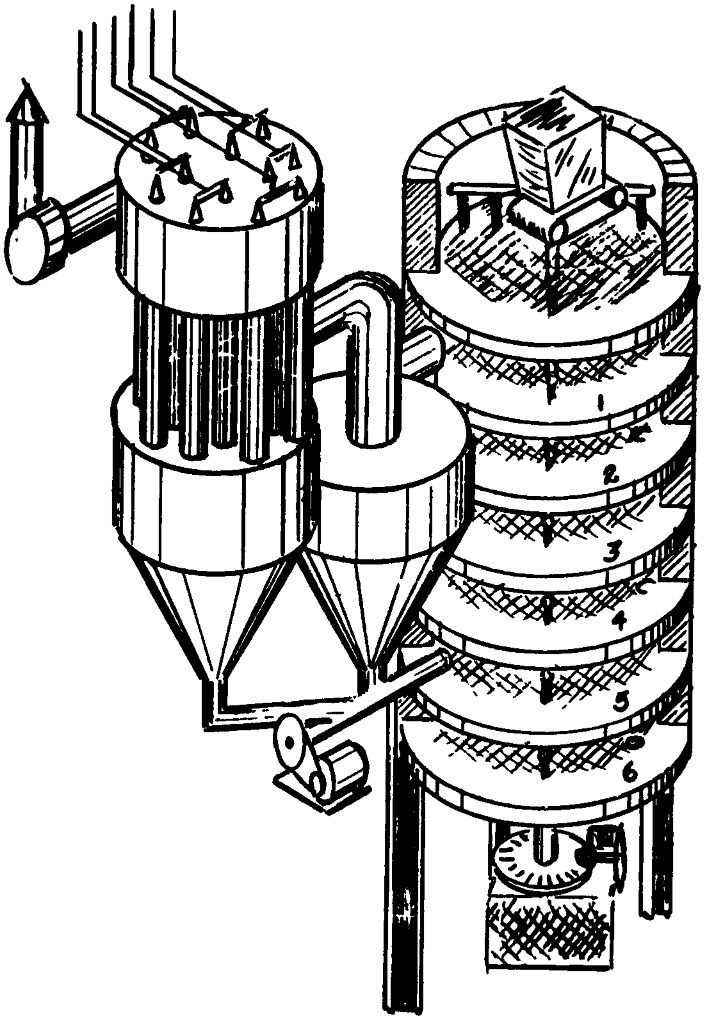
Samples of calcine and dust were muffle roasted for two hours at 450°C. This resulted in a weight loss of 0.8 per cent from the calcine and 2.8 per cent for the dust fraction. Both products were brick red in color and yielded an overall uranium extraction of 94 per cent when leached under the same conditions which previously had resulted in an overall 84 per cent uranium extraction from the as-received calcine and dust. Comparable improvement in uranium extraction was obtained by floating the unburned carbon and leaching the carbon free residue. This indicated that the poor leaching extractions were primarily the result of the poor leachability of the residual carbonaceous material.

B. Multiple Hearth Roasting

The favorable leaching characteristics of calcines produced by roasting and rabbling a shallow bed of lignite in a muffle indicate that a multiple hearth roaster might be applicable to the burning of lignites. Testing was delayed, however, pending the construction of a three-foot diameter, six hearth, gas fired unit. The roaster constructed is of conventional design and its basic features are shown in Figure 15. Raw lignite is fed by a belt to the top or drying hearth. Exit gases and dust are removed at the Number 1 hearth and passed through two cyclones and an electrostatic precipitator. The recovered dust is returned to the fifth hearth by means of a screw conveyor. The finished calcine is discharged from the sixth hearth into a sealed container.

Three tests have been completed; the first test was in the nature of a shakedown. Sufficient lignite was not available for an extended run; however, it was possible to operate for approximately 16 hours at a feed rate of 140 pounds of wet lignite, 77 pounds of dry lignite per hour, or 44 pounds per square foot of total hearth area per day. During the first part of the run,Sample 185-23 (Peter Kiewit) was used. During the latter part of the run, the feed was Sample 185-18 (also Peter Kiewit). At no time, except during the initial heat-up period, was it necessary to use the gas burners.

The calcines produced during the shakedown test were collected in five separate portions. These are indicated as Barrels 1 through 5 and are identified in the metallurgical balance shown in Table 19. Barrel 1 was partially roasted material collected before the roaster came to equilibrium. Barrel 2 was material collected during the period when the operating conditions listed in Table 20 were maintained. During this period of operation, grab samples were taken from each hearth to determine the degree of roasting. Each of the grab samples was then re-roasted in a



Multiple Hearth Roaster

Figure 15

muffle furnace for two hours at 450°C. Figure 16 is a plot of the ignition loss, expressed as per cent carbon removed, versus the hearth number or roasting time. As will be noted, about 85 per cent of the roasting is accomplished on the first three hearths, or in half the total roasting time. Barrels 3, 4, and 5 were produced under conditions of excess air in an attempt to increase the roasting rate. This resulted in excessive temperatures, the temperature of the Number 3 hearth exceeding 950°C.

Table 19

Hearth Roasting Test - Metallurgical Balance

Sample 185-18

Barrel Number	Wet Pounds	% Moisture	Dry Pounds	% U_3O_8	Pounds U_3O_8	% Dist'n
			Feed			
	2217	43.4	1225	0.218	2.736	100.0
			Products[1/]			
1	126	Nil	126	0.343	0.432	15.8
2	94	Nil	94	0.373	0.351	12.8
3	148	Nil	148	0.407	0.602	22.0
4	139	Nil	139	0.381	0.530	19.4
5	144	Nil	144	0.474	0.683	25.0
Unaccounted For					0.138	5.0
					2.736	100.0

1/ Calcine collected in five drums which were weighed and assayed separately.

Table 20

Temperatures and Gas Analyses

Hearth Number	Temp °C	Hearth Atmosphere % CO_2	 % O_2
1	210	8.1	12.9
2	450	13.6	8.0
3	700	12.7	5.3
4	720	8.3	11.5
5	600	6.0	13.7
6	560	6.0	13.7

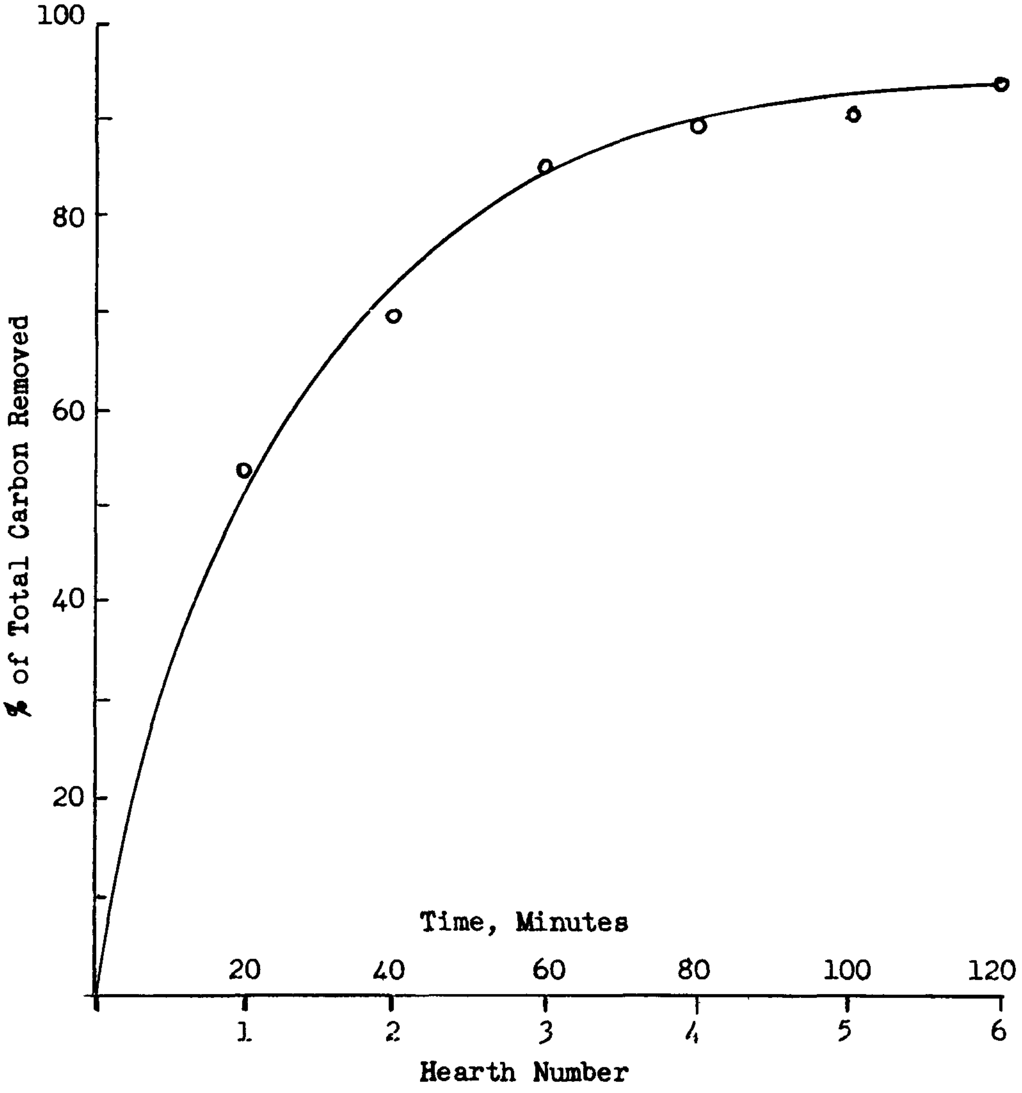


Figure 16. Effect of Time Versus Per Cent Carbon Removal

At the end of the roasting tests, samples of calcine, representative of each of the barrels produced, were acid leached at terminal acidities of approximately pH 1.0 and 0.5. The results of these tests are presented in Table 21 and show that 90 to 93 per cent of the uranium was extracted from the calcines from Barrels 1 and 2. Barrels 3, 4, and 5 were more refractory due to the excessive roasting temperatures.

Table 21

Acid Leaching Tests

Multiple Hearth Roaster Products
Sample 185-18

Barrel Number	% U_3O_8	H_2SO_4 Lb/Ton Ash	Terminal pH	Terminal Emf(mv)	Residue Assay % U_3O_8	% U_3O_8 Ext'n
1	0.343	1260	1.0	-425	0.026	93.2
2	0.373	1260	1.0	-410	0.043	89.5
3	0.407	1230	1.1	-435	0.074	83.1
4	0.381	1090	1.0	-415	0.116	71.0
5	0.474	1100	1.0	-420	0.116	76.5
1	0.343	1680	0.5	-420	0.028	92.6
2	0.373	1680	0.5	-430	0.034	91.7
3	0.407	1570	0.5	-450	0.061	86.3
4	0.381	1400	0.6	-435	0.088	78.9
5	0.474	1540	0.5	-445	0.120	77.0

Leaching Conditions: Six hours, 40 per cent solids, 75°C on ore ground to minus 35-mesh.

Following the shakedown run, a more extended test was made using a composite of 45 drums of lignite from the Belfield area of North Dakota. These are identified in Appendix A as Samples 185-39 through 185-48. A total of 6473 pounds of wet lignite (4040 pounds, dry weight) was roasted in the multiple hearth roaster at a feed rate of about 100 pounds of wet lignite or 62.4 pounds of dry lignite per hour. The calcine was discharged into 14 drums (average 125 pounds), each representing about five hours of roaster operation. The average moisture content of the feed was 37.6 per cent, and the overall ash content , on a dry ore basis, was 43.0 per cent. The multiple hearth roaster was operated at a burning rate of 35.7 pounds of dry lignite per square foot of hearth area per day.

Acid leaching tests were made on samples of ash from the 14 individual drums of calcine. The average U_3O_8 assay of the calcine was 0.365 per cent, and the average loss on ignition was 1.9 per cent. The ash was ground to minus 35-mesh, the water removed by filtration, and the ground calcine leached for six hours at 75°C, 40 per cent solids, and a pH of 0.5. The average U_3O_8 extraction was 61 per cent with an acid requirement of 610 pounds of H_2SO_4 per ton of calcine (262 pounds of H_2SO_4 per ton of raw lignite). No significant improvement in extraction of U_3O_8 was obtained by addition of an oxidant to the leach, or by removal of the small amount of residual carbon by flotation or reburning. Filtration of leach pulps was excellent with as little as 0.1 pound of Separanper ton of ash.

An attempt was made during roasting to hold the temperature down by nearly total restriction of air. Despite this action, the maximum roasting temperature could not be maintained below about 950°C. A reduction of the feed rate from 105 to 80 pounds per hour did not significantly affect the temperature. Gas analyses during the roasting test indicated a reducing atmosphere for at least part of the time. The roasting conditions apparently caused insolubilization of the uranium, as the leach extractions were much lower than predicted by previous amenability tests on the 10 individual samples from the Belfield area. Examination of the ash indicated extensive reconsolidation of the ash by incipient fusion. The calcine was nearly all magnetic, and uranium was fairly evenly distributed between light and heavy minerals. The maximum roaster temperature, feed rate, leach extraction, and carbon content of calcine (as loss on ignition) is indicated for each barrel of calcine in Figure 17.

A third extended test was completed in the multiple hearth roaster, treating International Resources lignite, Sample 185-54 and Ohio Oil lignite, Sample 185-55. The samples had the following U_3O_8 assays, dead-burned calcine weights, and moistures:

	Sample 185-54	Sample 185-55
U_3O_8 Assay, % of dry lignite ore	0.119	0.190
Calcine Weight, % of dry lignite ore	50.5-51.8	48.3-54.2
Moisture, % of wet lignite ore	28.1*	33.8

*Moisture, as received, was 33 per cent. Some drying took place during handling and storage.

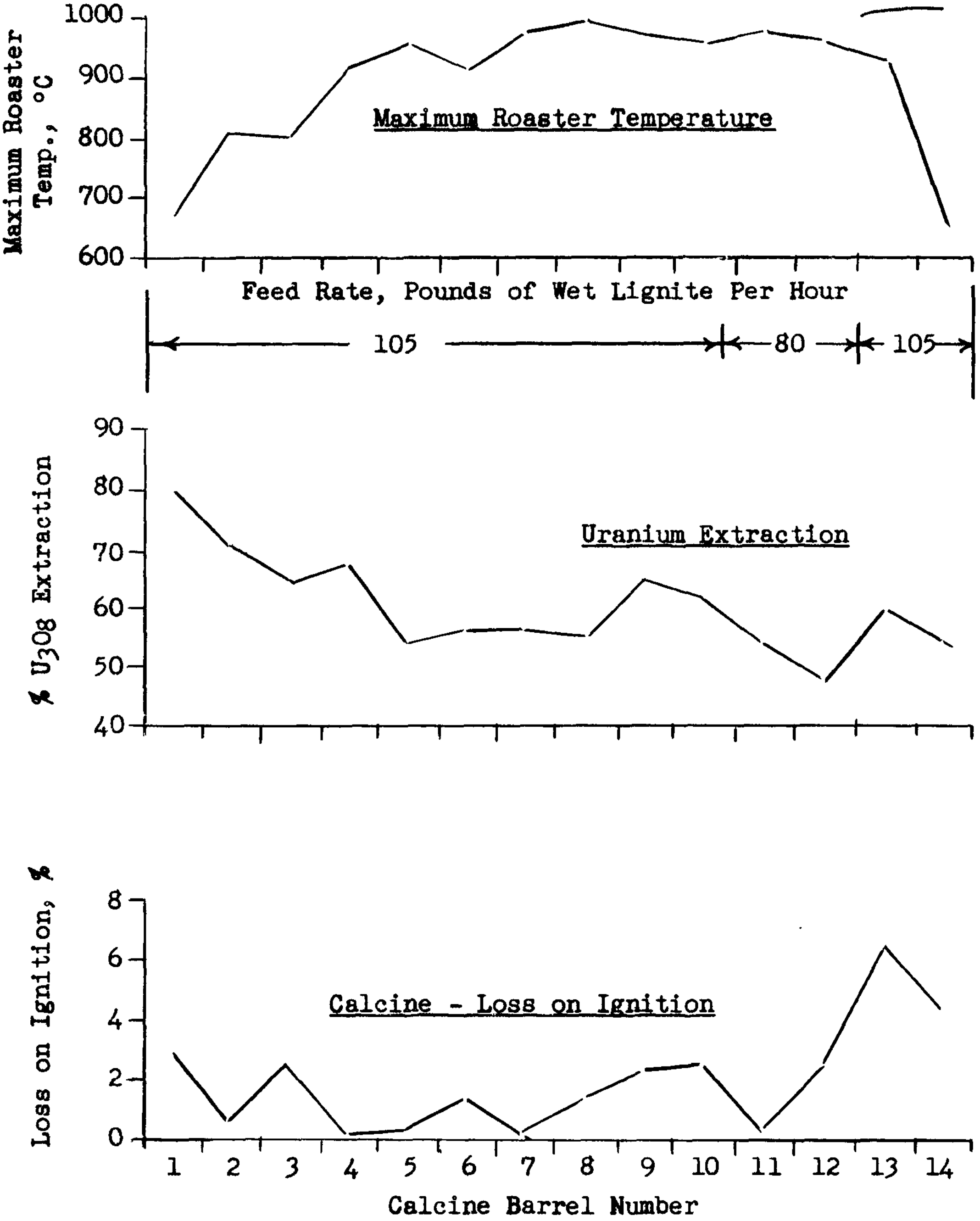


Figure 17. Multiple Hearth Roasting and Leaching of Composite Lignite from Belfield, North Dakota

During the entire run, drying and burning of the lignite were completed on four hearths. The dust from the collecting system was returned to the fifth hearth where no burning was accomplished. The fifth hearth burners were not lit since it was feared that the additional heat would raise the temperatures in the upper hearths above the desirable maximum.

The roaster was fed at a rate of 50 pounds of wet lignite per hour, equivalent to 36.0 pounds of dry lignite Sample 185-54 and 33.1 pounds of dry lignite Sample 185-55 per hour. Since burning was complete on the fourth hearth, a hearth area of 26.4 square feet (6.6 square feet per hearth) was required. Thus, the burning rate during this test lay between 32.7 and 30.1 pounds of dry lignite per square foot of hearth area per day.

A more detailed tabulation of the test, including leaching results, is shown in Table 22. Five hundred gram samples of calcine were leached at 45 to 50 per cent solids at 75°C for six hours with varying amounts of acid. In addition to the above acid leaching tests, each of the samples was re-roasted in an electric muffle and leached under similar conditions.

C. Grate Roasting

A series of batch tests were run in the six inch diameter pot roaster, shown in Figure 18, to investigate the feasibility of continuous down draft grate roasting. Lignite ore containing 20 per cent moisture was screened through four-mesh into the pot. The lignite was ignited with gas flame and air drawn through the bed at a known rate in order to control the temperature. The ash was acid leached at 40 per cent solids for six hours. The results of the tests are shown in Table 23.

Burning rates in the pot ranged from 33.7 to 176 pounds of dry ore per square foot per day on Sample 185-18. The rate increased not only with temperature, as expected, but also appeared to increase with decreasing bed depth. The test results were sufficiently favorable to warrant construction and testing of a laboratory continuous grate roaster.

In the continuous grate roaster, shown in Figure 19, the raw wet lignite was conveyed on a four-inch wide stainless steel 35-mesh screen from a hopper, under an ignition chamber, and over a series of 10 hoppers through which air was drawn. The pressure differential on each hopper was measured in the pipe connecting the hopper with a vacuum manifold.

Table 22

Results of Multiple Hearth Roasting Tests

Fraction	% of[1] Dead Burned Roast	Sampling Period Hours	Feed Weight Lb Wet Lignite Per Hour	Temperature, °C Average Maximum	Temperature, °C Absolute Maximum	% of Time at Absolute Maximum Temp	H_2SO_4 Lb/Ton of Calcine	Term. pH	% U_3O_8 Assay Head (Calcine)	% U_3O_8 Leach Residue	% U_3O_8 Calc. Head Assay (Calcine)	% U_3O_8 Ext'n[2]
Sample 185-54												
1	84.0	32	50	603	725	1.5	735		0.248	0.044	0.217	81.9
							910	0.8		0.034	0.218	86.2
	100.0						910	1.1		0.035	0.243	87.2
2	85.2	24	50	580	625	20.0	910	0.2	0.204	0.022	0.192	89.9
	100.0						910	1.3		0.017	0.206	92.7
3	81.0	24	50	600	675	27.0	910	0.55	0.198	0.024	0.213	90.0
	100.0						910	1.45		0.017	0.222	93.2
4	82.4	16	50	635	675	22.0	910	1.05	0.229	0.032	0.219	86.1
	100.0						910	1.40		0.024	0.239	90.3
Sample 185-55												
5	70.6	29	40	479	545	33.0	490	0.3	0.262	0.057	0.245	79.2
	100.0						525	0.85		0.057	0.298	82.6
6	90.6	25	50	551	625	37.0	525	0.5	0.311	0.093	0.320	72.9
	100.0						525	0.85		0.073	0.318	78.7
7	96.8	20.5	50	510	625	12.0	525	0.5	0.355	0.101	0.372	72.2
	100.0						525	0.95		0.079	0.365	80.1
8	93.2	24	50	615	700	14.0	525	0.2	0.395	0.108	0.358	69.5
	93.2[3]						525	1.2		0.096	0.382	77.0
	100.0						525	0.9		0.079	0.364	80.0

[1] Each sample from the multiple hearth roaster was reroasted in an electric muffle. Sample 185-54 was reroasted for two hours at 450°C. Sample 185-55 was reroasted for four hours at 450°C. Leaching of reroasted products was done in order to determine whether refractory uranium resulted from overheating or from unburned carbon.

[2] Extractions are based on U_3O_8 distribution in leached products.

[3] Calcine ground to minus 35-mesh before leaching.

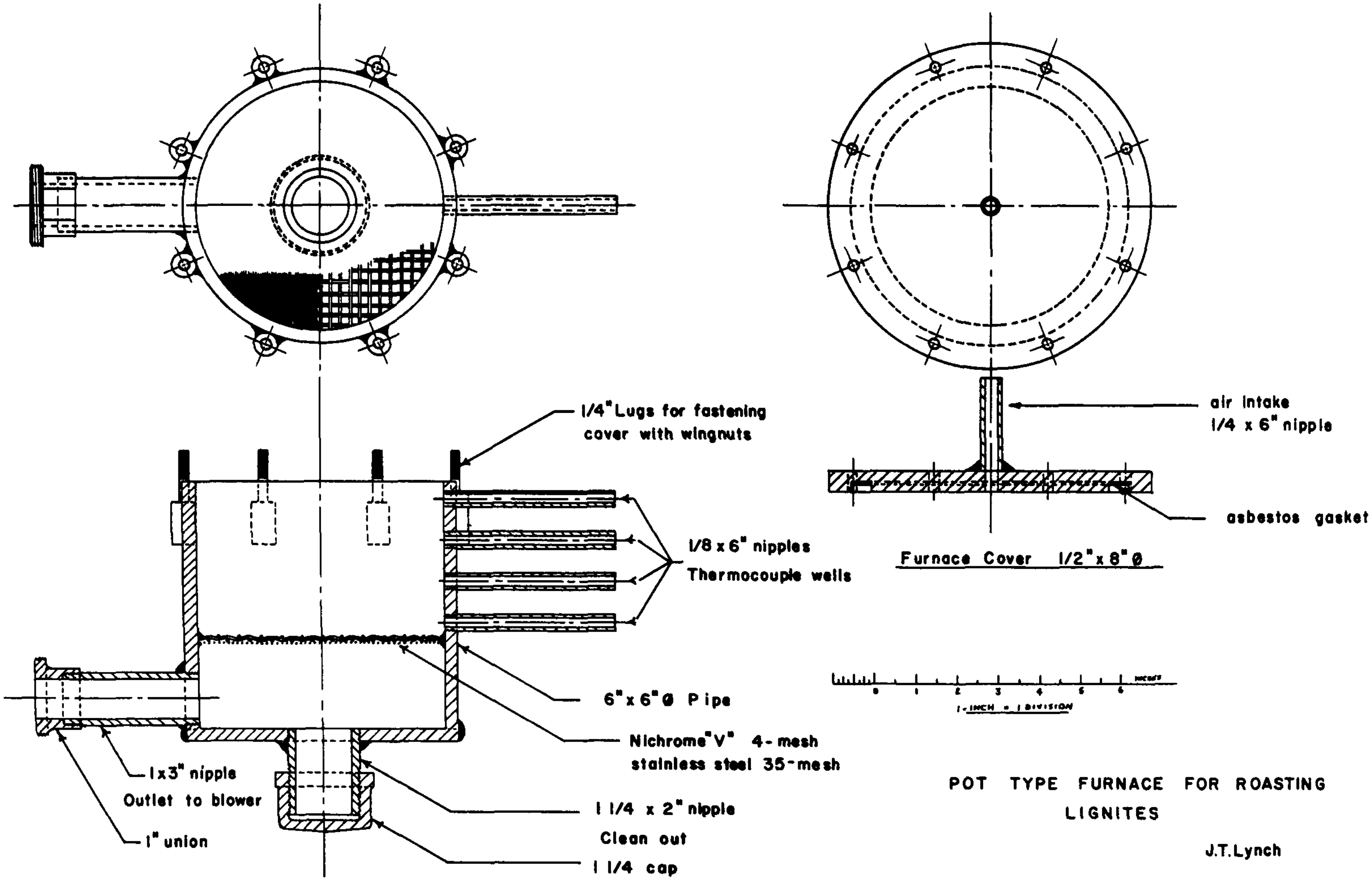

1/4" Lugs for fastening
cover with wingnuts
1/8 x 6" nipples
Thermocouple wells
6" x 6" Ø Pipe
Nichrome "V" 4-mesh
stainless steel 35-mesh
1 1/4 x 2" nipple
Clean out
1 1/4 cap
1 x 3" nipple
Outlet to blower
1" union
air intake
1/4 x 6" nipple
asbestos gasket
Furnace Cover 1/2" x 8" Ø
0 1 2 3 4 5 6 inches
1-inch = 1 division
POT TYPE FURNACE FOR ROASTING
LIGNITES
J.T.Lynch

Figure 18

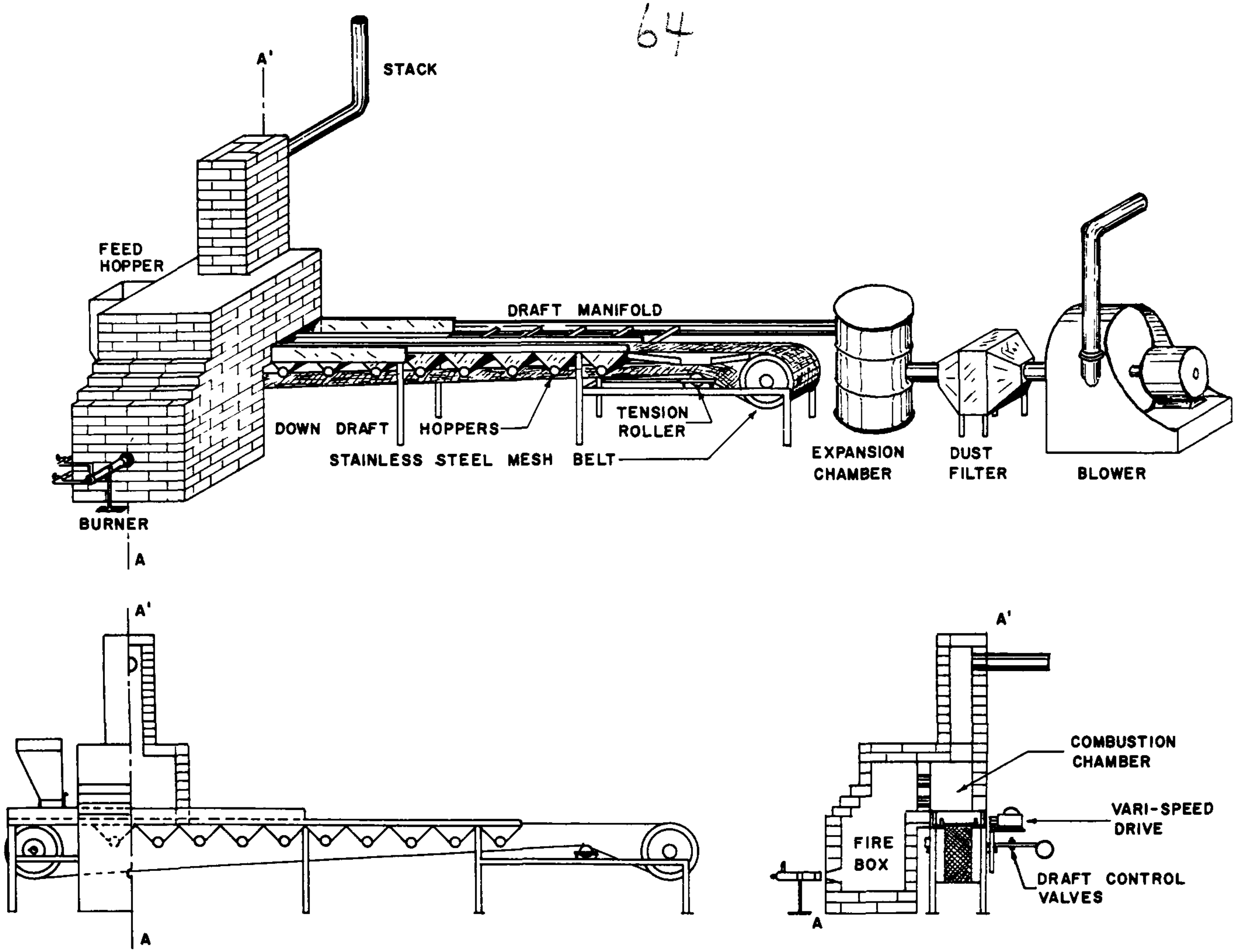
Table 23

Acid Leaching of Batch Grate Roasted Lignite Ash

	Roasting								Leaching		
Sample Number	Bed Depth Inches	Air Rate CFH	Max. Temp °C	Ignition Time Minutes	Burning Time Minutes	Dry Weight Charge Grams	Loss on Ignition %	Burning Rate Lb Dry Ore/ Sq Ft/Day	H_2SO_4 Lb/Ton Ash	Residue Assay % U_3O_8	% U_3O_8 Ext'n
185-18	1	-	1/	2	38	-	--	--	1400	0.060	91.3
	2 1/2	-	1/	2	48	-	--	--	1400	0.085	83.3
	2 1/2	4	730	1	210	450	48	33.7	1400	0.042	90.4
	1 1/2	2-4	600	2	90	360	62	62.0	1400	0.046	91.3
	5/8	2-4	800	2	7	100	46	176	1400	0.055	88.5
	5/8	2-4	2/	1	15	100	46	99	1400	0.042	91.7
	5/8	2-4	2/	2	--	-	--	--	1400	0.047	89.1
185-28	3	-	2/	4	90	400	56	67.3	700	0.011	94.9
185-27	3	2-4	2/	2	150	400	63	41.8	600	0.13	62.6

1/ No thermocouple, scoping tests.

2/ Thermocouple destroyed by heat.



GRATE ROASTER FOR BURNING LIGNITE ORES

<u>Figure 19</u>

An extended test of the grate roaster was made on International Resources lignite, Sample 185-54, assaying 0.119 per cent U_3O_8. The ore contained 33 per cent moisture on a wet basis and was approximately 51 per cent ash on a dry basis. The ore was fed to the roaster at a rate of approximately 11 pounds (wet) per hour or 7.4 pounds (dry) per hour. Assuming a total grate area (0.33 feet wide by 10 feet long) of 3.3 square feet, the average burning rate during the entire test was 53.8 pounds of dry lignite per square foot of grate area per day. Samples removed during the test were leached with varying quantities of acid for six hours at 75°C and 40 per cent solids. The U_3O_8 extractions on all samples were higher than 90 per cent when the acid addition was 1000 pounds of H_2SO_4 per ton of calcine. By comparison, a sample roasted in an electric muffle for two hours at 450°C and leached under similar conditions, yielded a 96.1 per cent U_3O_8 extraction. The details of the continuous grate roasting test are shown in Table 24.

D. Charring

Very early in the testing program, the U. S. Bureau of Mines, Coal Technology Laboratory, at Denver, conducted small-scale retorting tests with the aim of producing a char which would be free of soluble organic constituents and amenable to carbonate leaching. Encouraging results were obtained in the early experiments, and the testing was extended to acid leaching of retorted lignite.

The details of the retort roasting studies are discussed in Section VII (Carbonate Leaching). Appendix E, Table E-1, presents the results of acid leaching tests on various retort products. In general, the extractions of uranium were not improved over those obtained by acid leaching of raw lignite. Filterability and elimination of soluble organics in the leach liquors increased with increasing retort temperature, but as in the case of carbonate leaching, uranium extractions decreased sharply when the retort temperature exceeded about 400°C. Subsequent pilot testing by the Bureau of Mines showed that it was not possible to continuously retort lignite in the narrow temperature range required to insure high uranium extractions and elimination of soluble organics. Consequently, further studies on acid leaching of char products were discontinued.

E. Sulfate Roasting

The effect of roasting lignite ash with pyrite on the extraction of U_3O_8 by water leaching was studied. Muffle ash of Samples 185-27 and 185-28 was mixed with varying amounts of

Table 24

Results of Continuous Grate Roasting Test

Sample 185-54

		Temperature, °C					% U_3O_8			
		Average Maximum								
Sample Number	Sampling Period Hours	Under Firebox	Along Belt Including Firebox	Absolute Maximum	H_2SO_4 Lb/Ton Calcine	Terminal pH	Assay Head (Calcine)	Leached Residue	Calculated Head (Calcine)	% U_3O_8[1] Ext'n
1	4	503	578	700	1000	0.6	0.225	0.019	0.207	91.8
					750	1.2	0.225	0.021	0.203	90.6
2	7	643	678	720	1000	-	0.212	0.016	0.203	93.4
					750	1.45	0.212	0.019	0.206	91.5
3[2]	6.5	340	619	680	1000	0.6	0.208	0.012	0.203	95.0
					750	1.45	0.208	0.017	0.218	92.7
4	1	571	-	615	1000	0.35	0.237	0.017	0.215	93.3
					750	1.4	0.237	0.020	0.207	91.0
5[3]	15	485	616	850	1000	1.0	0.211	0.025	0.209	90.0
					750	1.4	0.211	0.026	0.211	88.5
6	8	512	-	720	1000	0.6	0.216	0.022	0.226	91.4
					750	1.3	0.216	0.024	0.207	89.3
7	4	491	736	800	1000	0.3	0.204	0.021	0.196	91.0
					750	1.3	0.204	0.027	0.200	87.5
8	11	401	522	720	1000	1.15	0.205	0.022	0.212	90.3
					750	1.1	0.205	0.025	0.193	88.2
9	8	431	565	740	1000	1.1	0.208	0.014	0.188	93.5
					750	1.3	0.208	0.017	0.207	92.6
10	12	501	604	830	1000	1.0	0.210	0.020	0.213	91.7
					750	1.35	0.210	0.024	0.209	89.3
11	3	451	514	740	1000	0.9	0.191	0.020	0.219	92.1
					750	1.25	0.191	0.023	0.205	89.7
Muffle Roast				450	1000	0.8		0.009	0.207	96.1

[1] Extractions are based on U_3O_8 distribution in leach products.

[2] Auto-ignition - during sampling period no heat was added at the firebox.

[3] High temperatures during run caused fusion of ash.

pyrite and roasted at 450°C in a muffle furnace. The exhaust blower was turned off to maintain the SO_2 atmosphere in the roaster. The ash bed depth was one-half inch. After roasting, the ash was leached in water for four hours at 40 per cent solids and room temperature. Uranium extractions of 53 to 68 per cent were obtained with varying roasting time and pyrite additions. Rabbling of the bed did not significantly affect U_3O_8 extraction. The natural pH of the muffle ash in water was 5.4 for Sample 185-27 (1.3 per cent U_3O_8 extraction) and 4.2 for Sample 185-28 (21 per cent U_3O_8 extraction). Roasting the ash with 200 pounds of pyrite per ton of ash produced enough acid to bring the leaching pH down to 3.1 for Sample 185-27 and to 2.5 for Sample 185-28.

A charge of raw lignite (Sample 185-28) was roasted for two hours at 450°C in the muffle furnace after addition of 400 pounds of pyrite per ton of lignite (215 pounds of pyrite per ton of ash). The water leaching pH (3.1 to 3.5) was higher than tests using similar pyrite additions to roasted ore, and the U_3O_8 extraction (46.5 per cent) was lower. Results of the test work are reported in Table 25.

F. Flash Roasting Studies

Although it was realized that flash roasting results in excessive particle temperatures, it was thought that in view of the very short burning time, a time-temperature relationship might exist which would permit the use of flash roasting techniques. Testing was conducted in a 10 foot high gas fired "Chimney" at various temperatures and concentrations of oxygen. Feed was blown into the top of the furnace. The coarse ash was recovered from an ash pit at the base, and the air-borne dust was recovered with cyclones and an electrostatic precipitator.

In all cases complete burning of the carbon was not attained without excessive fusing of the ash and insolubilization of the uranium. Consideration was also given to roasting of lignites by burning with a powdered coal burner, but no tests were conducted since it was felt that this type of burning would be analogous to that encountered in flash roasting.

Table 25

Roasting of Lignite Ash with Pyrite

Sample Number	Ash Assay, % U_3O_8	Pyrite Lb/Ton Ash	Roast[1] Time, Hours	Leach[2] pH Range	Final Emf(mv)	Residue Assay % U_3O_8	% U_3O_8[3] Ext'n
185-27	0.45	200	2	2.8-3.3	-355	0.21	52.6
		400	2	2.6-3.0	-420	0.18	57.2
185-28	0.16	100	2	2.7	-425	0.060	63.3
		200	2	2.5-2.7	-435	0.063	60.0
		400	2	2.3-2.5	-435	0.062	58.3
		800	2	2.0-2.3	-400	0.043	68.0
		200	1/2	2.3-2.6	-400	0.068	57.3
		200	1	2.2-2.4	-430	0.059	62.7
		200	2	2.5-2.7	-435	0.063	60.0
		200	4	2.3-2.5	-440	0.060	62.1
		200	2[4]	2.1-2.4	-420	0.066	58.7
	0.087	215	2[5]	3.1-3.5	-265	0.062	46.5

1/ Muffle roast at 450°C - 1/2 inch bed, no rabble.

2/ Ground to minus 35-mesh, leached four hours, 40 per cent solids, 25°C.

3/ Based on U_3O_8 assay of residue.

4/ Rabbled at 30-minute intervals.

5/ Raw ore mixed with pyrite, 1/4 inch bed, roasted at 450°C with rabbling at 20-minute intervals.

SECTION V

ACID TREATMENT OF RAW LIGNITE

A. Batch Leaching Tests

The lignite ore, as received, was dried, crushed to minus 10-mesh, and split into representative charges for testing. Acid leaching tests were made on the raw lignite following procedures similar to the testing of lignite ash as described in Section 1. It was found that extraction of uranium from raw lignite is favored by elevated leaching temperature and a large excess of sulfuric acid. The majority of tests were made after grinding the lignite to about minus 35-mesh. The grindability of raw lignites varied widely, as indicated by screen analyses included in Appendix F, Table F-1. The variation appeared to be caused by differences in the quantity and nature of the carbon in the ore. Grinding of the lignite was included in the testing procedure, as some of the samples contained water soluble organic material and molybdenum (especially Peter Kiewit Samples 185-8 and 185-18).

Investigations of the effect of leaching time and temperature on raw lignite (Bryco Sample 185-4) indicated satisfactory extraction in four to six hours at 80°C. Consequently, most of the leaching tests involved varying acid concentrations under standard leaching conditions of six hours at about 75°C. Results obtained under these conditions are summarized in Table 26. The addition of a chemical oxidant did not benefit the uranium extraction of raw Sample 185-20 (Manidon), although some increase in the oxidation potential was noted. Adequate mixing of the pulp was generally obtained at 40 per cent solids. Thickening of the pulp upon addition of acid prevented leaching at a higher density. The detailed test results are included in Appendix F, Table F-2. The optimum leaching efficiency appeared to require a terminal leaching pH below 1.0 and in some cases below 0.5. Figure 20 indicates the effect of varying acid concentration under otherwise constant leaching conditions for several raw lignite samples.

B. Acid Resin-in-Pulp

In a letter to Dr. G. G. Marvin, dated June 28, 1955, Dr. M.D. Hassialis of Columbia University presented data which indicated that 94.5 per cent of the uranium may be removed from a weakly acidic slurry of raw lignite by a single contact resin-in-pulp process.

Table 26

Acid Leaching of Raw Lignite

Sample Number	Source	% U_3O_8	Grind pH	H_2SO_4 Lb/Ton	Terminal pH	% U_3O_8 Ext'n
185-4	Bryco	0.37	-	400	0.5	83.6
				800	0.1	89.4
185-16	Bryco	0.35	5	360	0.4	71.5
185-18	Peter Kiewit	0.22	8	500	1.9	88.6
				750	1.2	96.8
185-19	Farmington	0.28	5.5	400	0.8	88.3
				600	0.2	94.6
185-20	Manidon	0.16	4.8	300	0.2	83.6
				450	-	88.5
185-21	Northgate,Smith #1	0.10	3.8	300	0.2	86.2
185-22	Manidon	0.15	4.8	200	0.8	74.5
				300	0.4	81.1
185-25	Northgate, #6	0.23	-	300	0.8	78.5
				400	0.4	85.3
185-26	Northgate, #7	0.18	7.5	500	1.4	75.7
				600	1.2	81.5
				700	0.8	91.1
185-27	Manidon	0.145	4.5	300	0.2	79.4
				400	0	87.9
185-28	Northgate,Smith #1	0.09	4.3	200	0.8	80.0
185-29	Int'l Resources	0.61	-	550	0.6	90.5
185-30	Ohio Oil #8	0.27	4.2	300	1.0	85.0
				450	0.4	90.1
185-31	Ohio Oil #9	0.36	5.6	450	1.1	76.6
				600	0.6	89.1
185-33	Ohio Oil #10	0.10	5.0	350	0.8	76.5
185-34	Ohil Oil #11	0.18	4.2	300	1.0	81.9
				400	0.5	88.0
185-35	Homestake #4	0.68	8	600	1.0	85.9
185-37	Int'l Resources #1	0.35	5.8	500	0.6	82.7
185-38	Int'l Resources #2	0.18	5.3	500	0.8	90.3
185-39	Northern Pacific	0.35	6.1	650	0.4	92.1
185-40	Ohio-Talkington	0.16	5.0	515	0.4	90.8
185-41	Landis-Hecker #2	0.20	5.4	500	0.5	86.3
185-44	Landis-Rodakowski	0.50	4.5	380	0.4	90.0
185-45	Gardner-Smith #2	0.26	3.9	390	0.5	93.3
185-46	Ohio-Obrigewitch	0.13	8.1	700	0.5	79.0
185-47	Whitman-Schwartz	0.14	4.6	350	0.3	88.8
185-48	Ohio-Chrusch	0.12	6.7	600	0.4	91.2

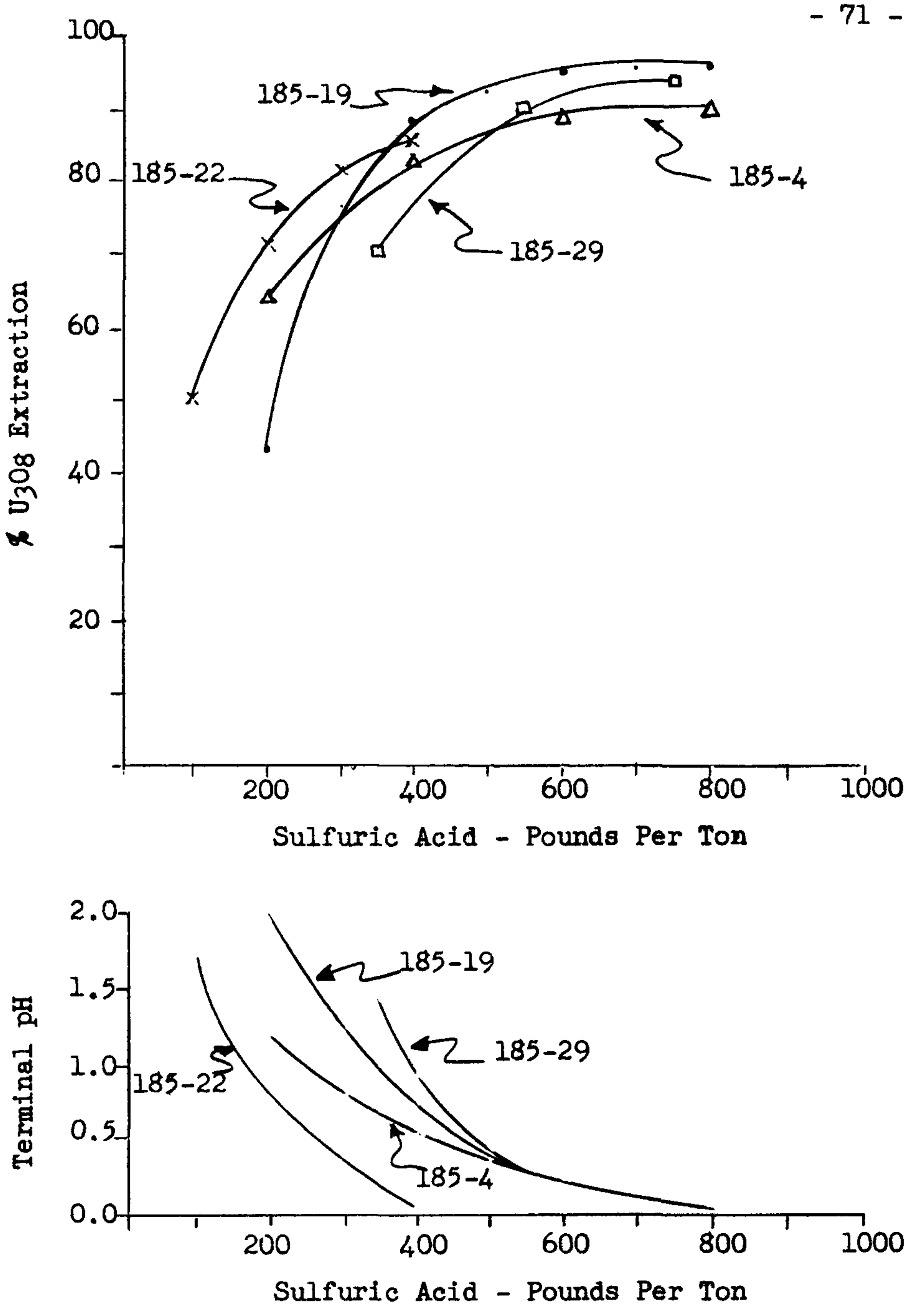


Figure 20. Effect of Acid on U_3O_8 Extraction

The effect of pH of the lignite slurry on uranium adsorption on ion exchange resin was studied by contacting 50 gram samples of pulverized Sample 185-6 (Bryco) at 25 per cent solids for 64 hours with 200 ml of XE-123 resin (sulfate form) and varying amounts of sulfuric acid. The results of the study are presented in Figure 21 and in Appendix F, Table F-3 and show that over 90 per cent of the uranium was extracted using 250 to 600 pounds of H_2SO_4 per ton. The single contact resin-in-pulp process is substantially simpler than any other of the extraction processes studied for lignite treatment. It should be noted, however, that the maximum loading obtainable under the above conditions was 0.725 gram U_3O_8 per liter of wet settled resin or approximately 0.05 pound of U_3O_8 per cubic foot.

In order to study the feasibility of countercurrent resin-in-pulp, an eight stage system was set up in which resin was moved counter to a raw lignite slurry. No leaching was done prior to RIP since much of the uranium was believed to be held by the lignite as an adsorbed phase. Agitation took place in pachucas and the resin-slurry separation was done on a 40-mesh screen. Initially, each stage contained 10 ml of wet settled XE-123 resin, in the neutral sulfate form, and 100 grams (dry basis) of Sample 185-6 ground to minus 100-mesh. The retention time per stage was one-half hour. The flowsheet is shown in Figure 22.

The circuit was operated, originally, at the natural pH of 4.3 until it was established that the extraction was nil. Two hundred and fifty pounds of H_2SO_4 per ton were then added to the slurry in Stage One giving an initial pH of 0.8 and a final pH in Stage Eight of 1.25. The uranium recovery on the resin was 64 per cent and the soluble loss was 5.6 per cent.

Reducing the amount of acid added to Stage One resulted in an increase in the initial and final pH to 1.1 and 1.4, respectively. It was felt that increasing the pH in Stage One would raise the loading in that stage and produce better overall extractions, but the extraction was not improved.

The quantity of resin fed to the system was doubled (20 ml of wet settled resin per stage), thus reducing the theoretical loading from 30 grams U_3O_8 per liter of wet settled resin to 15 grams U_3O_8 per liter of wet settled resin. The additional resin improved the recovery to 75 per cent and reduced the soluble loss from 5.6 per cent to 1.3 per cent.

The acid addition to Stage One was reduced to between 100 and 125 pounds H_2SO_4 per ton, permitting the pH to rise to between 1.5 to 1.6. Acid was added to Stage Seven to maintain the pH of Stages Seven and Eight at 1.0 to 1.1 (175 to 225 pounds per ton). The soluble loss remained about 1.3 per cent, and the U_3O_8 recovery on the resin was 81 per cent.

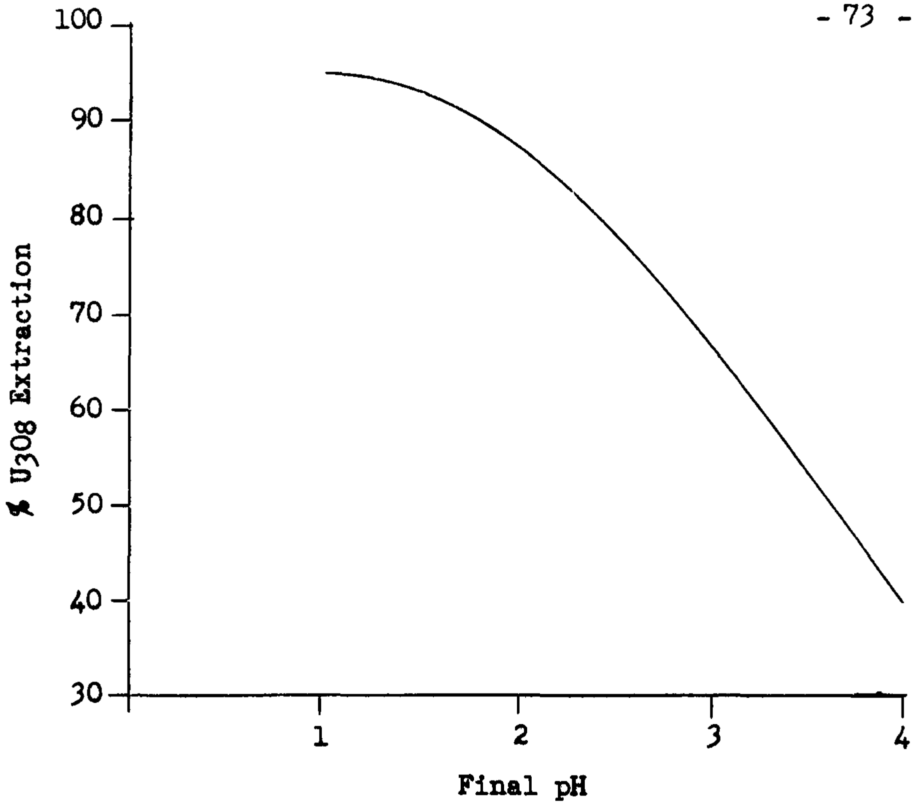


Figure 21. Effect of pH on Single Batch Contact R.I.P.

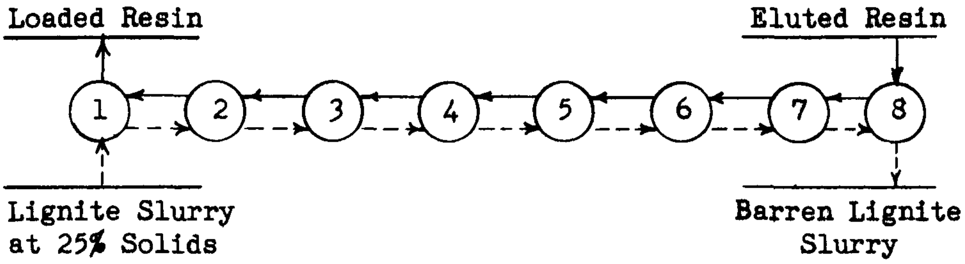


Figure 22. Countercurrent Resin-In-Pulp

The retention time per stage was increased to three and one-half hours giving a total resin and slurry retention time of 28 hours. The pH of the entire circuit was held at 1.0 to 1.1. The recovery of uranium on the resin was 89.1 per cent and the soluble loss was 0.8 per cent. The acid consumption during this treatment was about 350 to 400 pounds of H_2SO_4 per ton of raw lignite.

It should be noted that, as a result of screening and washing pulp from resin, the dilution decreased the pulp density from 25 per cent solids in Stage One to about five per cent solids in Stage Eight. This resulted in an acid consumption which was 100 to 150 pounds per ton higher than that anticipated in practice.

Further work on the resin-in-pulp process was discontinued due to the low resin loadings and fouling of the resin by molybdenum and organic material.

C. Water Leaching of Raw Lignite

During the course of investigations conducted on Peter Kiewit Sample 185-18, it was observed that when the raw lignite is given a preliminary water leach, such as would be the case if ground in water and then thickened and filtered, up to 25 per cent of the molybdenum as well as appreciable amounts of other soluble salts and organic matter were extracted. As a consequence, the water-leached residue was more readily filtered after acid leaching, and extractions of uranium were somewhat higher, possibly as a result of improved filtration and washing.

Water leaching studies were made to determine the conditions favoring the solubilization of the molybdenum. Leaching at various per cent solids and various times indicated that maximum solubilization of the molybdenum was favored by a low per cent solids during leaching and a contact time of eight to 16 hours. The test results are presented in Figure 23.

Figure 24 shows that 10 successive one-half hour contacts with fresh water, at 25 per cent solids and a temperature of 30°C, solubilized 28 per cent of the molybdenum. Under the same conditions at 75°C, 50 per cent of the molybdenum was solubilized. In both series of tests, less than 0.5 per cent of the uranium was dissolved.

The testing of molybdenum removal by water washing was discontinued when it was determined that many of the other raw lignite samples did not contain as much water soluble molybdenum or organic material.

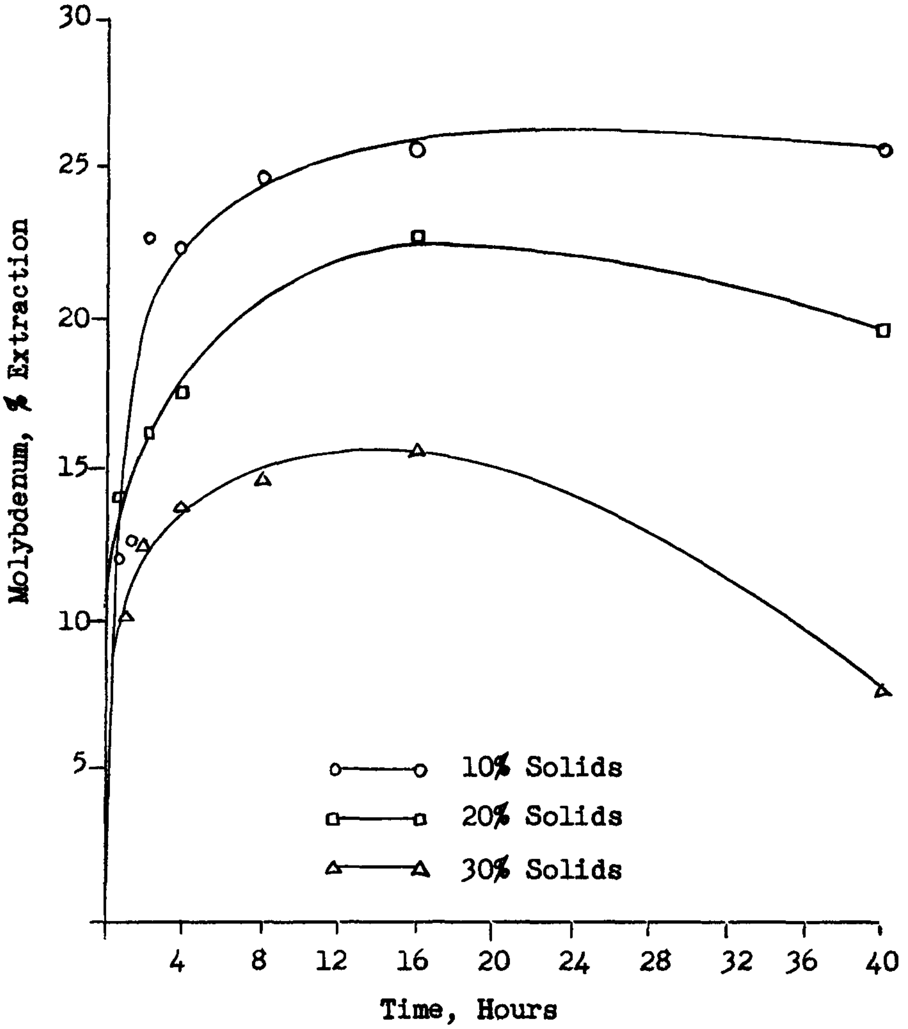


Figure 23. Molybdenum Extraction by a Water Leach at Room Temperature on Raw Peter Kiewit Lignite, Sample 185-18

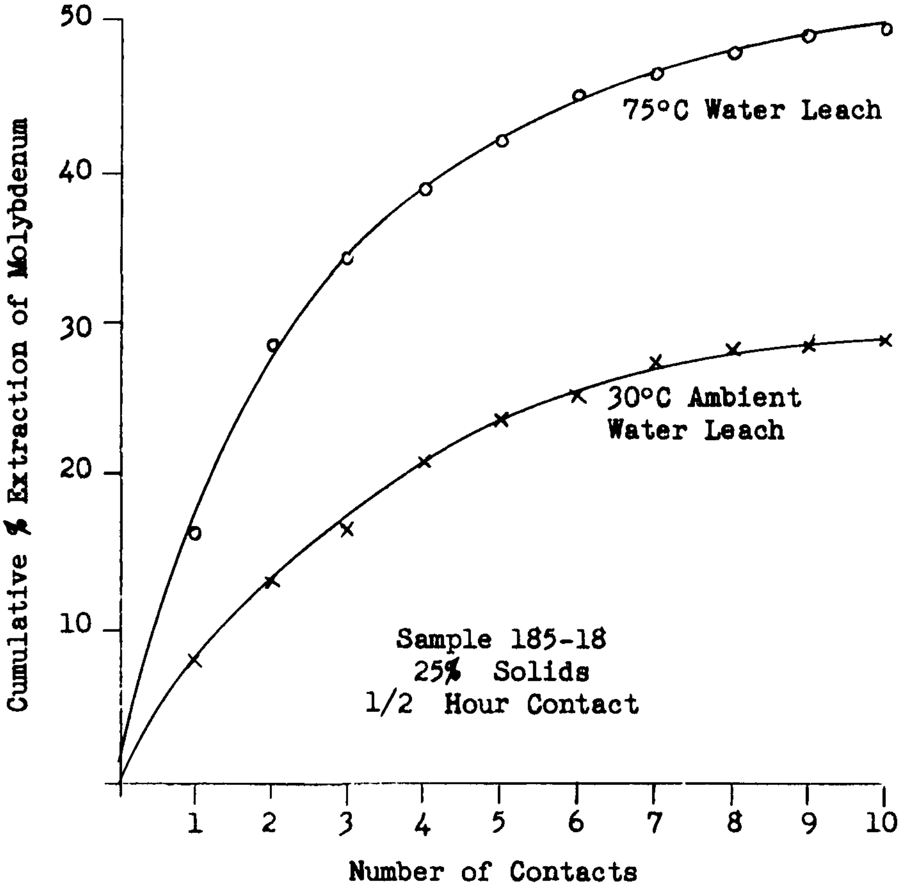


Figure 24. Extraction of Molybdenum by Water Leaching Raw Peter Kiewit Lignite (Sample 185-18)

D. Thickening and Filtration

1. Thickening Tests of Unleached and Acid Leached Raw Lignite

Settling tests were made on raw lignite pulps after grinding in water. Adequate settling rates were obtained by the use of sufficient Separan, but the terminal densities were less than 45 per cent solids in all but one case tested.

Raw lignites were acid leached under approximately optimum leaching conditions and the acid pulps settled. The pulps appeared to go into compression at rather high dilution, and therefore usually only one feed dilution was tested. The terminal densities after acid leaching were generally less than 40 per cent solids. The results of thickening tests on unleached and acid leached pulps are summarized in Table 27 , and detailed test results are presented in Appendix F, Table F-4.

Table 27

Thickening Tests on Raw Lignite

Sample Number	Description	Separan Lb/Ton	Area Requirement Sq.Ft/Ton/Day	Terminal Density, % Solids
185-18	Unleached	0.50	0.7	30
	Leached	0.50	10.2	24
185-19	Unleached	0.05	2.5	47
	Leached	0.20	5.2	36
185-20	Unleached	0.10	3.1	42
	Leached	0.10	1.2	39
185-21	Unleached	0.15	2.9	42
	Leached	0.20	28.2	41

2. Filtration

The filtration characteristics of unleached lignite, after grinding in water, were observed to be satisfactory after addition of a small amount of Separan.

Filtration tests were made on acid leached raw lignites using a standard 0.1 square foot Oliver test leaf. The test procedure was similar to that described for filtration tests on lignite ash and, as was the case with the ash, effective filtration required a prior stage of dilution and thickening.

Filtration test results for the samples tested are summarized in Table 28. The filter capacity was high in many cases, when enough Separan was used. The high soluble losses indicate that at least two stages of filtration are required for adequate washing of uranium. The test results are detailed in Appendix F, Table F-5.

Table 28

Filter Tests of Raw Lignite Acid Leached

Sample Number	Source	Separan Lb/Ton	Capacity Lb/Sq Ft/Day	% Moisture	%2/ Soluble Loss
185-18	Peter Kiewit	0.50	1000+	60	--
185-19	Farmington	0.25	830	51	18.2
185-22	Manidon	0.10	3600	48	11.7
185-27	Manidon	0.15	3840	47	8.6
185-28	Northgate	0.30	635	48	19.3
185-30 31,331/	Ohio Oil	0.60	450	51	11.0

1/ Equal weight composite
2/ Includes one stage of thickening.

E. Purification of Raw Leach Liquor by Activated Charcoal

An investigation was made to determine the possibility of removing molybdenum and organic coloring material from raw lignite leach liquors by means of powdered, activated charcoal. The charcoal was added directly to the leach liquor, agitated for the desired time, then filtered off. The test work was performed using a three-stage countercurrent system with filters between stages. The carbon concentration in each stage was 80 grams per liter of solution and a 20 minute contact was used at each stage. The feed liquor was obtained by an acid leach of raw Peter Kiewit lignite (Sample 185-18), and contained 0.47 gram U_3O_8 per liter and 0.17 gram Mo per liter. After three contacts, the charcoal was eluted with six per cent NaOH solution. This procedure gave over 95 per cent removal of the molybdenum, and visual examination of treated liquor indicated complete removal of the organic material.

A cyclic test using this procedure was made to determine the number of times the charcoal could be recycled without loss of efficiency. Sixteen cycles, or 48 contacts, were completed with no significant change in adsorption characteristics. The charcoal concentration was 50 grams per liter of solution. Typical results of char performance are shown in Table 29.

Table 29

Molybdenum Removal by Activated Charcoal
Single-Stage Leach Liquor
Sample 185-18

	g Mo/1			
Cycle	Stage 1	Stage 2	Stage 3	% Mo Removal
4	0.096	0.043	0.020	88.2
6	0.093	0.043	0.016	90.6
11	0.090	0.035	0.011	93.5
13	0.105	0.050	0.011	93.5
14	0.068	0.028	0.016	90.6
15	0.090	0.033	0.012	92.9
16	0.100	0.040	0.011	93.5

Uranium losses in the system appeared to be in the range of 0.3 to 0.5 per cent. This figure was arrived at by analysis of the caustic soda eluting solution after contact with the charcoal. The results indicate that, initially, there was an adsorption of a portion of the uranium from the liquor; however, this uranium was not completely desorbed by the eluting solution. After two complete cycles, or six contacts, the liquor from the reactors contained the same uranium concentration as the initial feed. This would indicate an equilibrium between the charcoal and the feed solution.

The charcoal treated leach liquor was adjusted with powdered iron and $CaCO_3$ to an emf of minus 280 mv and a pH of 2.0, and tested on a single column of IRA-400 ion exchange resin. The ion exchange feed analysis was as follows:

Table 30

Ion Exchange Feed Analysis
Sample 185-18

Constituent	Grams/Liter
U_3O_8	0.43
Mo	0.005
Fe	7.18
Fe^{+++}	0.12
$SO_4^{=}$	87.7

Using an exhaustion retention time of three minutes, the saturation loading was 65 grams of U_3O_8 per liter of wet settled resin.

F. Two-Stage Countercurrent Acid Leaching and Charcoal Purification

Two-stage countercurrent acid leaching was studied for raw lignite using a flowsheet similar to that used in two-stage leaching of ash (Figure 7). The study was made to determine the effect of two-stage leaching of raw lignite on the concentrations of organic matter and molybdenum in acid liquors. It was felt that a leach liquor might be produced which could be easily purified by activated charcoal and treated by ion exchange.

A significant reduction in Mo concentration and some decrease in organic matter was achieved by the two-stage leach as compared with single-stage leaching. Moreover, the acid requirement was generally less for an equivalent uranium extraction. The results of the two-stage tests of Sample 185-18, and tests using a similar flowsheet for Samples 185-19, 20, and 21 are included in Appendix F, Table F-6.

The leach solution prepared by the two-stage leaching of raw lignite from Samples 185-18 was tested in the countercurrent charcoal purification system. The pregnant liquor, containing 0.046 gram Mo per liter, was contacted in a three-stage system with 50 grams of charcoal per liter of solution. Complete organic removal was obtained in two countercurrent contacts and the molybdenum concentration of the final liquor was, in most instances, less than 0.001 gram Mo per liter. Typical results of charcoal treatment are shown in Table 31.

Table 31

Molybdenum Removal by Activated Charcoal
Two-Stage Leach Liquor
Sample 185-18

Cycle	g Mo/l Stage 1	 Stage 2	 Stage 3	% Mo Removal
8	0.008	0.001	0.001	98^+
11	0.008	0.001	0.001	98^+
12	0.005	0.001	0.001	98^+

SECTION VI

PURIFICATION AND ION EXCHANGE OF ACID LIQUORS PRODUCED FROM LIGNITE ASH

Ion exchange of solutions obtained by single-stage acid leaching of lignite ash encountered early breakthrough and comparatively low uranium loadings. This was attributable to molybdenum poisoning and the high ferric iron and sulfate content of the liquors. Consequently, studies were made of the purification of pregnant liquors prior to ion exchange. The results of these studies showed that the interfering ions could be reduced to an acceptable level by hydrolysis and partial neutralization of the pregnant liquor. This can be accomplished after a single stage acid leach by addition of calcium carbonate or limestone or preferably by precipitating the interfering ions in the neutralizing stage of a two-stage countercurrent leach, thus eliminating the relatively large amounts of lime required for neutralization. It was further found that residual molybdenum left in the purified pregnant liquors could be effectively adsorbed on activated charcoal.

This section of the report presents the data on tne ion exchange recovery of uranium from untreated and purified liquors. The method and results of liquor purification by neutralizing with lime are presented as is also the use of activated charcoal. Details on the results of purification by two-stage leaching are presented in Section I of this report.

A. Ion Exchange of Untreated Leach Liquor

Initial ion exchange testing was conducted on solution prepared by acid leaching of ash from Sample 185-1 (Edgemont Stockpile). Ash was prepared by calcining the lignite in a laboratory assay furnace for two hours at 450°C and leaching the ash at 75°C for six hours at 50 per cent solids with 865 pounds of H_2SO_4 per ton of ash. The pregnant liquor was diluted to a final solution to ash ratio of five to one and a portion of the solution was reduced with powdered iron to decrease the ferric iron content. Analyses of the solutions are shown in Table 32.

Table 32

Chemical Analyses

		Solution Assay, Grams/Liter							
Emf(mv)	pH	U_3O_8	V_2O_5	Fe^{++}	Fe^{+++}	P_2O_5	SiO_2	$SO_4^=$	Mo
-310	1.25	0.83	0.07	8.65	0.90	0.45	2.73	77.5	0.10
-425	1.25	0.80	0.07	1.85	4.55	0.45	2.64	77.7	0.10

Ion exchange tests were made on both oxidized and reduced liquors using IRA-400 resin. The retention times were three minutes for exhaustion and 10 minutes for elution. The eluting solution was 1.0M NaCl, 0.1N H_2SO_4, 100 grams $SO_4^=$ per liter as $MgSO_4$. Results of the tests are presented in Table 33.

Table 33

Ion Exchange Testing of Dakota Lignite Acid Liquor
Sample 185-1

Emf(mv)	Cycle	Bed Volumes To: Cutoff	Bed Volumes To: Saturation	Bed Volumes To: Elution	Saturation Loading g U_3O_8/1 WSR
-310	1	27	120	20	50.3
-310	2	24	124	20	42.0
-310	3	14	124	20	40.7
-310	4[1]	33	132	19	47.0
-425	1	24	102	18	45.1
-425	2	20	100	18	42.0
-425	3	15	100	19	42.7

1/ After regeneration with 12 bed volumes of six per cent NaOH using a 10-minute retention time.

The data show a sharp drop in adsorption rate of uranium with each successive cycle and an accompanying drop in the uranium loading. This drop in loading is the result of molybdenum adsorption. After the third cycle, a portion of the resin loaded from the reduced liquor was analyzed and found to contain 22 grams of molybdenum per liter of wet settled resin. The resin was then regenerated with 12 column volumes of six per cent NaOH which removed 69 per cent of the adsorbed molybdenum. Subsequent loading of the resin showed that its U_3O_8 capacity had been restored to almost its initial value of 50 grams per liter.

B. Partial Neutralization and Ion Exchange

It had been found in studying acid leach liquors from other uranium ores that nearly complete removal of molybdenum may be accomplished by precipitation in the presence of ferric iron. This method of solution purification is described in WIN-48[1] and in essence consists of adding an alkali, preferably lime or limestone, and raising the pH to about 3.3 to 3.8.

1/ Magno, P., "Precipitation of Molybdenum From Ion Exchange Feeds by Neutralization In the Presence of Iron", WIN-48, 11/15/56

Precipitation of molybdenum is favored by the presence of a molar ratio of ferric iron to molybdenum of at least two to one and the coprecipitation of uranium is reduced at elevated temperatures.

In the first test on purifying lignite liquors, a solution produced from muffle roasted Bryco lignite, Sample 185-16, was used. The liquor was first oxidized with potassium permanganate to an emf of minus 540 mv, to insure the presence of adequate ferric iron. The solution was then heated to 50°C, neutralized with calcium carbonate to pH 3.5, agitated for a short period, filtered and the precipitate washed. The calcium carbonate requirement was equivalent to approximately 400 pounds per ton of ash.

A second test was made in almost identical manner using a liquor obtained from Sample 185-18 ash (Peter Kiewit). Consumption of calcium carbonate was 410 pounds per ton of ash.

Analyses of both solutions are given in Table 34.

Table 34

Analyses of Solutions Before and After Partial Neutralization

Samples 185-16 and 185-18

Sample Number	pH	Emf (mv)	Grams/Liter U_3O_8	Mo	Fe^{+++}	Fe^{++}	SO_4	Al_2O_3
185-16-Before	1.5	-420	1.10	0.52	2.66	1.29	77.7	19.0
185-16-After	3.7	-540	1.04	0.012	0.01	0.07	28.0	7.5
185-18-Before	1.5	-460	0.98	0.42	7.70	0.77	90.0	18.5
185-18-After	3.9	-360	0.86	0.01	0.02	0.10	39.0	8.8

As will be noted, in each case 98 per cent or more of the molybdenum and about 60 per cent of the sulfate was precipitated. The loss of uranium was approximately six per cent for Sample 185-16 and 12 per cent for Sample 185-18. Subsequent repulping of the precipitates with sulfuric acid at a pH of 3.0 (approximately 100 pounds per ton) reduced the loss of uranium from Sample 185-16 to 1.2 per cent and to 2.5 per cent for Sample 185-18. In each instance about 10 per cent of the molybdenum and iron were redissolved.

Ion exchange testing was conducted on the purified solutions, shown in Table 35, in a three column ion exchange system, each column containing 25 ml of wet settled IRA-400 resin. In these tests, two columns, in series, were used for exhaustion while the third column was being eluted. Retention time on exhaustion was three minutes, and the loading was stopped when the effluent from the second column reached an assay of approximately 0.01 gram U_3O_8 per liter. Elution was conducted at a retention time of 10 minutes using a one molar solution of NaCl acidified to pH 1.1 with either H_2SO_4 or HCl.

Prior to ion exchange testing, the pregnant liquors were passed through a 25 ml column of "Columbia" activated charcoal. This removed most of the residual molybdenum and resulted in a feed to ion exchange containing less than 0.005 gram of Mo per liter.

Results of the ion exchange tests, comprising four complete resin cycles on Sample 185-16 liquor and three cycles on Sample 185-18, are shown in Tables 35 and 36.

Table 35

Cyclic Ion Exchange of Treated Lignite Liquor Sample 185-16

Column Number	Cycle	Breakthru on 2nd Column	Elution	Loading[1] g U_3O_8/l WSR
1	1	174[2]	13	88.7
2	1	99	12	88.1
3	1	93	13	92.2
1	2	107	13	95.7
2	2	85	13	94.2
3	2	94	13	90.5
1	3	98	13	96.8
2	3	89	13	89.9
3	3	92	13	89.6
1	4	98	10[3]	94.4
2	4	85	10	95.8
3	4	96	10	91.4

1/ Loadings calculated from assays of the eluates

2/ Initial startup. Two fresh columns on exhaustion

3/ Eluate cycle 4 was 1.0<u>M</u> NaCl acidified to pH 1.0 with HCl.

Table 36

Cyclic Ion Exchange of Treated Lignite Liquor

Sample 185-18

Column	Cycle	Solution Throughput Bed Volumes For		Loading g U_3O_8/l WSR
		Breakthru on 2nd Column	Elution 1/	
1	1	196	10	92.5
2	1	110	10	91.3
3	1	110	10	86.3
1	2	110	10	87.8
2	2	110	10	85.6
3	2	110	10	87.1
1	3	-	10	91.5
2	3	-	10	--
3	3	130	10	--

1/ Eluate was 1.0M NaCl acidified to pH 1.0 with HCl.

The data show that the purified pregnant liquors had excellent ion exchange properties. The average uranium loadings were in excess of 5.5 pounds of U_3O_8 per cubic foot of wet settled resin and there was no evidence of resin poisoning.

As indicated earlier, charcoal columns were used ahead of the ion exchange columns to remove residual molybdenum from the ion exchange feed. In the case of Sample 185-16, 20 liters or 800 column volumes of solution were passed through the charcoal bed. The final fraction assayed 0.004 gram of Mo per liter. For Sample 185-18, 26.6 liters, or 1064 column volumes were passed through the column. The final effluent assayed 0.003 gram of Mo per liter.

C. Effect of Recycling Repulp Solution

In the preceding tests, the uranium recovered by repulping the purification sludge, or iron-molybdenum precipitate, with acid was not recycled. Consequently, a series of cyclic tests was conducted on Sample 185-16 in which the repulp solution was advanced to the next leach to determine the effect of this recycle solution on the buildup of impurities.

The results of this test are summarized in Appendix G, Table G-1, and show that while there was no buildup of iron or molybdenum over four cycles, the alumina increased from 19.5 grams per liter to 53 grams per liter. As a result of this excessive buildup, the acid required for repulping increased from 158 pounds to 310 pounds per ton and the calcium carbonate for neutralizing increased from 380 to 650 pounds per ton It is therefore obvious that some other means of handling the repulp solution will be required if the process is to be incorporated in a plant flowsheet.

D. Ion Exchange of Acid Liquors Produced by Two-Stage Leaching of Lignite Ash

As an alternative to the neutralization and precipitation of molybdenum and ferric iron with lime, the precipitation of molybdenum and ferric iron may be accomplished in the neutralizing stage of a two-stage countercurrent leach, thus eliminating the lime or limestone required for neutralization and the acid required for recovering the coprecipitated uranium. Details of the two-stage leaching procedure are discussed in Section I, and Appendix B, Tables B-3 through B-8, show the results of cyclic leaching tests on Samples 185-18, 27, and 28 and on a composite of Samples 185-30, 31, and 33. The results reported show that there was no increase in acid consumption or buildup of alumina or other constituents in the pregnant solutions using the two-stage leach process for solution purification.

Ion exchange testing of pregnant solutions produced by two-stage leaching of ash has been limited to Sample 185-18. The solution used was obtained from Cycle 8, described in Appendix B, Table B-3, and had the following composition.

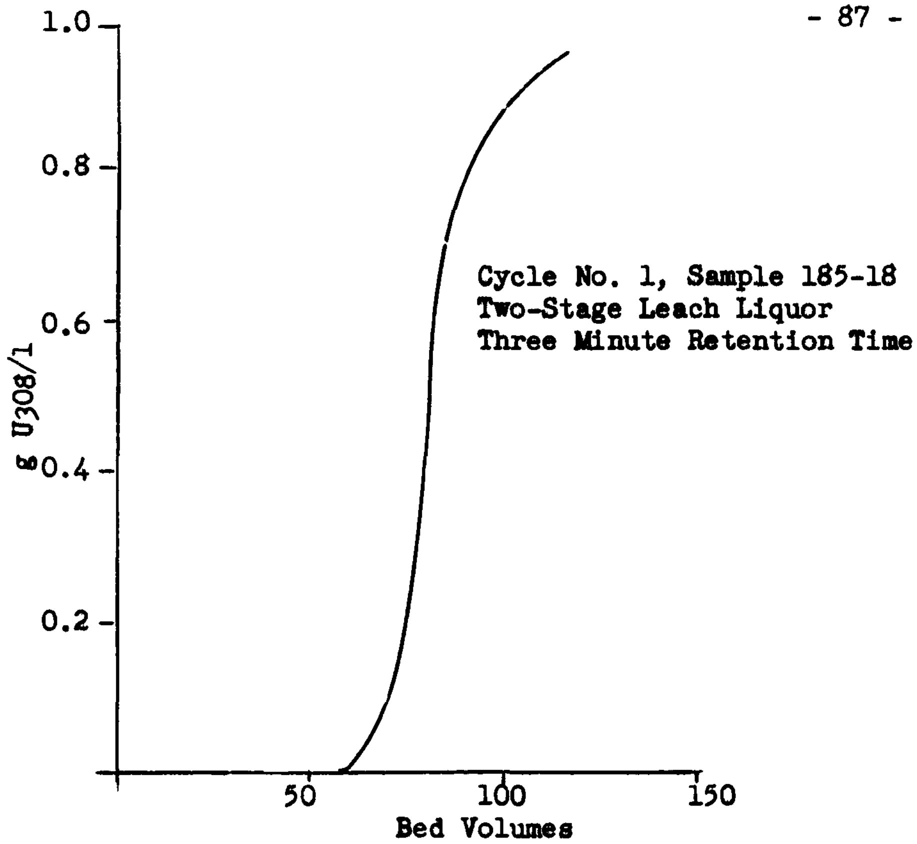
Table 37

Two-Stage Leach Liquor - Sample 185-18

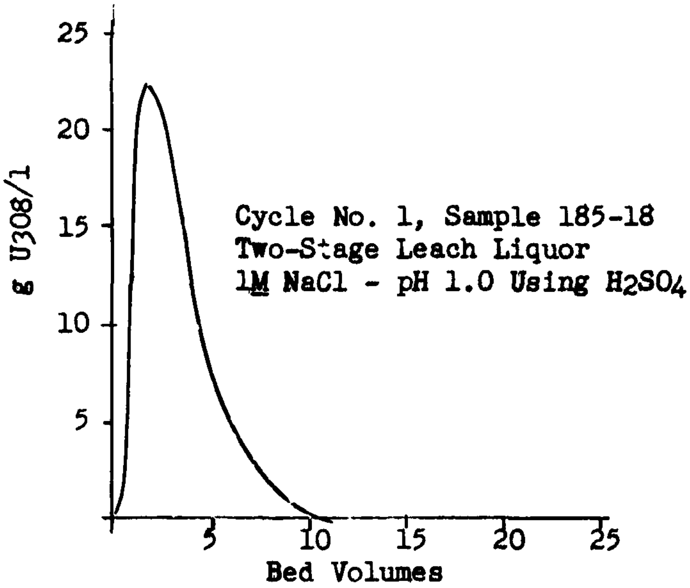
Grams/Liter					
U_3O_8	Fe^{++}	Fe^{+++}	Mo	SO_4	pH
0.99	1.22	0.81	0.019	83.4	2.8

The amenability of the liquor to ion exchange was tested using a single 20 ml column of IRA-400 and determining the shape of the adsorption curve. Prior to ion exchange the pregnant liquor was passed through a charcoal column to remove the residual molybdenum.

The results of the ion exchange tests are plotted graphically in Figure 25 and indicate a cutoff loading of approximately 65 grams of U_3O_8 per liter of wet settled resin. The saturation loading, based upon the elution data, was 80 grams of U_3O_8 per liter of wet settled resin.



Loading Curve IRA-400



Elution Curve IRA-400

Figure 25

SECTION VII

CARBONATE LEACHING STUDIES

Carbonate leaching tests were made on several samples of lignite during the period December 1954 through April 1956. Although exceptionally high U_3O_8 extractions were obtained by sodium carbonate leaching of raw lignites, the pulp presented a nearly impossible solid-liquid separation problem. The best results in the laboratory were obtained on lignite samples retorted at 400°C to remove volatiles. A 96 per cent U_3O_8 extraction was obtained and the residue filtered reasonably well. Unfortunately, the retorting temperature was extremely critical and good filtration and U_3O_8 extraction were not obtained simultaneously in the larger scale tests that were carried out at the Coal Technology Station of the U. S. Bureau of Mines at Denver, Colorado.

A. Treatment of Raw Lignites

In spite of the high U_3O_8 extractions indicated by the test results shown in Table 38, many difficulties were encountered during the carbonate leaching of raw lignites. In the presence of sodium bicarbonate, serious foaming occurred. Air blown through the pulp for oxidation also caused foaming. During leaching the pulp became gelatinous and agitation of the pulp was impractical except in the range of about 20 per cent solids. The consumption of sodium carbonate was high due to the presence of soluble sulfates and organic material. The pulp had the appearance of an oil sludge and was unfilterable. Because of this, it was necessary to centrifuge the pulp to obtain a small amount of clear liquor upon which extractions could be based.

Table 38

Carbonate Leaching of Raw Lignite
Sample 185-4

% Solids	Mesh	Leaching Conditions Time, Hours	 Temp °C	 Na_2CO_3 g/l	% U_3O_8[1/] Ext'n
20	-10	24	Amb.	100	92.8
20	-10	24	80	100	96.4
10	-10	24	Amb.	50	92.0

1/ Unfilterable pulp - extractions based on liquor assay obtained by centrifuging the pulp.

In one series of tests, countercurrent resin-in-pulp was used to extract the uranium from carbonate leached raw lignite. Minus 65-mesh Sample 185-6 (Bryco) was leached for 24 hours at room temperature and 25 per cent solids with 400 pounds of Na_2CO_3 per ton. The pulp was then contacted in five stages with 20 grams of fresh XE-123 resin in the bicarbonate form for each 100 grams of ore. The retention time per stage was 1.5 hours. Although the theoretical resin loading had been calculated at 15 grams of U_3O_8 per liter of wet settled resin, the actual loading obtained was 12.9 grams per liter. This loading was equivalent to a 91 per cent U_3O_8 recovery. In addition to the low initial capacity, difficulty was experienced in eluting U_3O_8 from the resin. Complete elution was not achieved with 1*M* NaCl, 1*M* HCl, or 1*M* $NaHCO_3$.

Two samples of resin that had been through five stages of loading were eluted in a column and recycled to the RIP circuit. The resin loading on the second pass dropped to 10.4 grams of U_3O_8 per liter of wet settled resin. It was noted that the black color on the resin, acquired during loading, remained after elution.

Sample 185-6 was leached using ammonium carbonate under pressure. The conditions were as follows: six hours at 20 per cent solids and 110°C using a solution containing 100 grams $(NH_4)_2CO_3$ and 20 grams NH_4HCO_3 per liter, with a pressure of either 60 or 100 psi using ammonia, or 700 psi using oxygen. The leach pulp was unfilterable, but uranium extractions of over 95 per cent were indicated by centrifuging leach liquor or by adsorption on ion exchange resin.

B. Treatment of Charred Lignites

A number of small-scale retort roasting tests were carried out over a temperature range of 200°C to 600°C on minus 10-mesh Bryco lignite, Sample 185-4, by the U. S. Bureau of Mines, Coal Technology Branch, Denver, Colorado.

The tests were carried out in their precision laboratory distillation-assay apparatus. This equipment consists of a small steel retort, housed in a precision controlled electric furnace. When the retort is heated, volatile hydrocarbons, water, and gases evolved are trapped separately in a laboratory gas train. In carbonate leaching tests the retort products were leached at 20 per cent solids and 80°C for 24 hours with 100 grams of Na_2CO_3 and 50 grams of $NaHCO_3$ per liter. The results of these tests are shown in Figure 26. The data show a high uranium extraction at retort temperatures below 400°C. Extractions were sharply reduced at temperatures above 400°C. The carbonate leached residues were unfilterable at temperatures below 400°C Above 400°C, the filtration characteristics improved with increasing temperature.

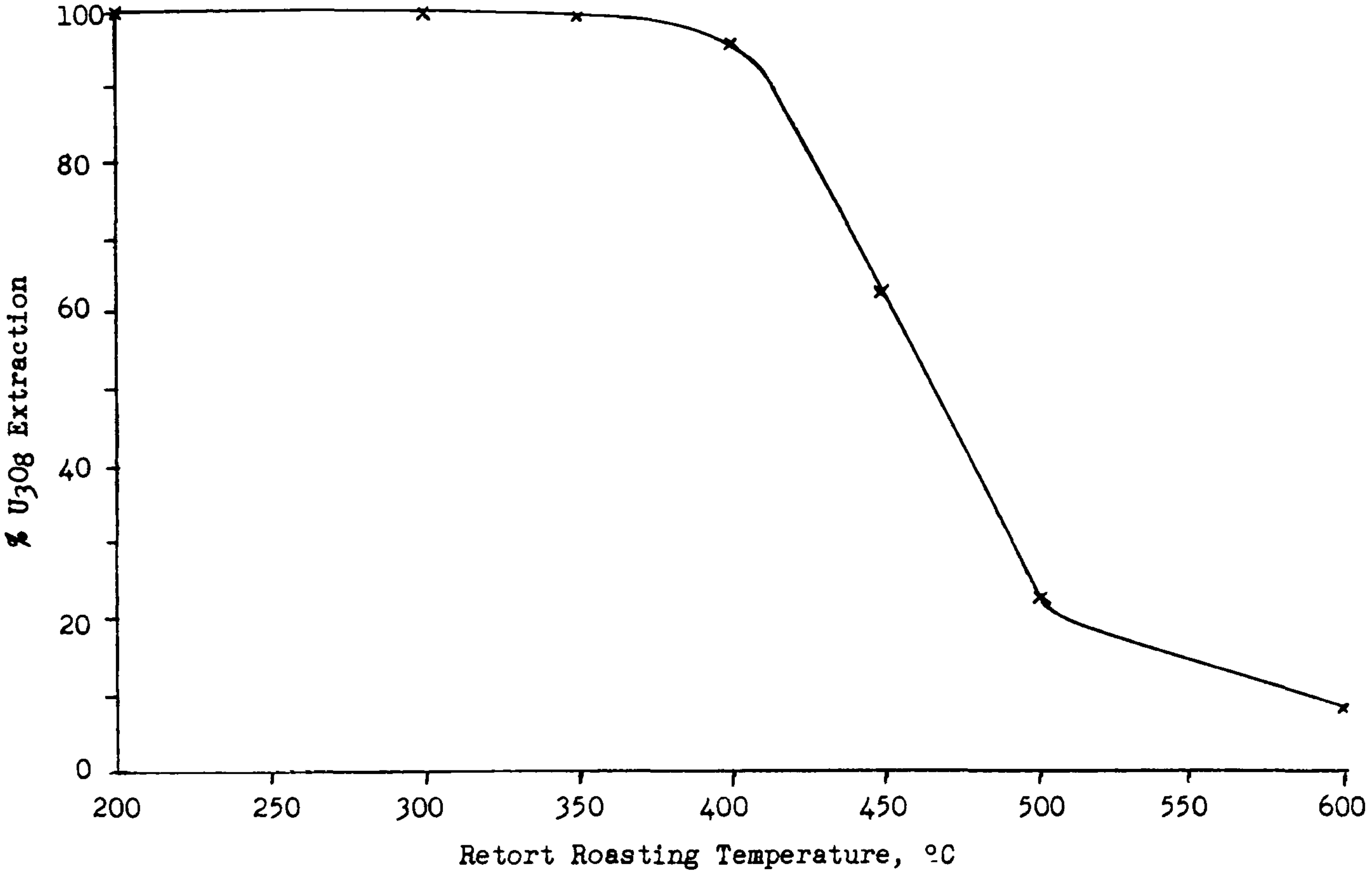


Figure 26. The Relationship of Retort Roasting Temperature to U_3O_8 Extraction by Carbonate Leaching

A retorting test was made on Sample 185-4 at Winchester, using a one-hour retort contact time and a temperature of 400°C. The char was leached for 24 hours at 80°C and 20 or 40 per cent solids with a solution containing 100 grams of Na_2CO_3 and 50 grams of $NaHCO_3$ per liter. The uranium extraction was 89 per cent at 20 per cent solids, and it was 83 per cent at 40 per cent solids. The filtration was slow but not prohibitive. Carbonate consumptions for these tests were about 200 pounds of Na_2CO_3 per ton of leach feed.

Further testing of Bryco and Peter Kiewit lignites on a larger scale was carried out in the Parry Dryer by the Denver, USBM Laboratory. This treatment consists of drying the lignite and carbonizing the dried material in an indirect fired reactor. In both operations the lignite is transported through the furnace turbulently entrained in gaseous products of combusion from the heating gas. Additional details of this test work are shown in Part I of Appendix H.

Retorting tests on Bryco lignite were also run in the Porter-Dillon-Pringle retort at Bozeman, Montana. This equipment consists of an annular-shaped vertical retort vessel which is heated on both the inner and outer walls by hot combustion gases. The raw wet lignite was fed by gravity to the top of the retort, and char was removed from the bottom by means of motor-driven augers. Gases and tars are drawn off from the vessel with a slight fan suction into a dust and by-product collection system. Further information on the Bozeman tests are shown in Part II of Appendix H.

The tests on Bryco lignites at Denver and Bozeman indicated that the temperature control required to produce a char amenable to both leaching and filtration was not within the limits of commercial retort equipment. The tests on Peter Kiewit lignite, Sample 185-9, at Denver indicated 80 to 90 per cent extraction of uranium by carbonate leaching when the retort temperature was at 400°C, but filtration was slow. The filtration was satisfactory on leached products from a 500°C retorting test, but the uranium extraction was poor.

C. Treatment of Muffle Roasted Ash

Sodium carbonate leaching tests were made on several lignite samples which had been completely ashed at 450°C in a muffle furnace. The filtration characteristics of leached pulps were generally satisfactory. Tests indicated that 75 to 85 per cent extraction of uranium is possible in about 24 hours at 80 to 90°C with a reagent consumption of about 200 pounds of Na_2CO_3 per ton of ash. All tests employed air oxidation. The use of $KMnO_4$ as a chemical oxidant was not beneficial. Test data are reported in Table 39.

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Table 39

Carbonate Leaching of Muffle Ash

		Leaching Conditions				Grams/Liter			Residue	
Sample Number	Ash	Feed % U_3O_8	Time, Hours	Temp °C	% Solids	Na_2CO_3	$NaHCO_3$	$KMnO_4$ Lb/Ton	Assay % U_3O_8	% U_3O_8 Ext'n
185-1	60	0.44	16	90	33	100	100	0	0.30	32
			16	90	33	100	100	10	0.25	43
185-4[1]	57	0.56	6	90	40	100	50	0	0.14	75.4
			24					0	0.10	82.5
			48					0	0.099	82.6
			72					0	0.12	79.0
			96					0	0.11	80.7
			24	90	33	50	50	10	0.135	77.0
			48					0	0.14	76.0
			24					20	0.16	72.0
			48					0	0.13	78.0
185-5	50	0.48	24	90	33	50	50	10	0.081	83.0
			24	90	33	50	50	20	0.098	80.0
185-20[2]	32.5	0.50	24	80	20	100	50	0	0.078	84.5

1/ Sample 185-4 consumption: 212.5 pounds of Na_2CO_3 per ton of ash.

2/ Sample 185-20 consumption: 186 pounds of Na_2CO_3 per ton of ash.

D. Treatment of Fluosolids Ash

Coarse calcine and dust products from a laboratory fluosolids reactor were tested for amenability to carbonate leaching. It was found that the roasting conditions which were required for efficient extraction of uranium were detrimental to the filtration characteristics of the leached pulp. When the roaster products exhibited satisfactory filtration characteristics, extraction suffered. Samples 185-5 and 185-6 were roasted in a nitrogen atmosphere with a two hour retention time and roasting temperatures ranging from 316° to 427°C. The lower temperatures provided high extraction of uranium and poor filtration, and the reverse was true for the higher temperatures. An attempt was made to improve filtration and extraction by a longer roast in a nitrogen atmosphere, by roasting in air, or by roasting in an air-N_2 mixture. The results did not indicate that high extraction and good filtration could be obtained simultaneously. The results of carbonate leaching tests of fluosolids products are reported in Table 40. The leaching conditions were as follows: calcine (roaster discharge) and dust products leached for 24 hours at 80°C and 20 per cent solids with a solution containing 100 grams of Na_2CO_3 and 50 grams of $NaHCO_3$ per liter. The comparative filtration rates represent hours to filter and wash a 250-gram charge on a 24 cm Buchner funnel. The reagent consumption was 160 to 216 pounds of Na_2CO_3 per ton of ash.

E. Treatment of Rotary Kiln Products

Sample 185-6 was roasted in a electrically heated laboratory rotary kiln, and the ash tested for amenability to carbonate leaching. The U_3O_8 extraction was 95 to 98 per cent when the kiln temperature was 300°C, but the filtration characteristics of the leach pulp were unsatisfactory. Improved filtration was noted when roasting was done at 400°C, but the U_3O_8 extraction was reduced to 73 to 75 per cent. High U_3O_8 extraction and poor filtration were again observed at 350° or 400°C when the air supply to the roaster was limited. The carbonate consumption appeared to be about 230 pounds of Na_2CO_3 per ton of ash when a two hour retention time was used. The results of the carbonate leaching of rotary kiln ash (Sample 185-6) are reported in Table 41.

Table 40

Carbonate Leaching of Fluosolids Ash

Sample 185-6

Roasting Conditions			% Extraction[1]		Na_2CO_3 Consumption	Filtration Hours	
Temp °C	Time, Hours	Atmosphere	Calcine	Dust	Lb/Ton Ash	Calcine	Dust
316-343	2	N_2	94.5	Unfilterable	---	48	--
343-371	2	N_2	90.5	95.7	200	24	--
371-399	2	N_2	64.8	87.2	160-195	1.0	--
399-427	2	N_2	51.0	79.3	---	0.25	--
399-427[2]	2	N_2	63.0	--	---	0.25	--
371-399[3]	2	N_2	68.0	81.5	166-216	1.5	--
316-343	4	N_2	90.9	89.0	214	3.0	72
343-371	4	N_2	87.5	93.0	208	2.5	0.1
371-399	4	N_2	73.0	90.0	196	0.5	0.1
399-427	4	N_2	52.0	86.0	183-213	0.25	0.1
371-399	2	Air	71.0	54.1	171-181	0.5	0.1
427-454	2	Air	53.7	51.7	177	0.25	--
371-399	2	Air+N_2	73.7	89.1	185	0.5	48

[1] Leached 24 hours at 80°C and 20 per cent solids with a solution of 100 grams Na_2CO_3 and 50 grams $NaHCO_3$ per liter. Air oxidation used in all tests.

[2] Fifty pounds of $KMnO_4$ per ton also used.

[3] Sample 185-5.

Table 41

Carbonate Leaching of Rotary Kiln Ash
Sample 185-6

Kiln Temp °C	Retention Time, Hours	% U_3O_8[1] Extraction	Na_2CO_3 Consumption Lb/Ton Ash	Filtration[2] Rate Hours
300	2	95.5	243	5.5
300	1	97.5	-	5.5
400	2	75.3	238	0.5
400	1	73.1	-	0.5
350[3]	2	97.1	241	5.5
400[3]	2	96.5	229	3.0

[1] Ash leached for 24 hours at 80°C and 20 per cent solids with a solution containing 100 grams of Na_2CO_3 and 50 grams of $NaHCO_3$ per liter.

[2] Comparative filtration rates - time for filtration and wash of 250 grams on a 24 cm Buchner funnel.

[3] Air supply to roast was limited.

SECTION VIII

PHYSICAL BENEFICIATION STUDIES

Gravity tabling and magnetic separation tests were made on lignite ores and ash in an attempt to separate a barren waste product. In over half the ores which were tested, either a large proportion of the gangue was too fine to respond to gravity or magnetic separation or the gangue was intimately associated with the carbon and did not yield a rejectable barren waste product.

In addition, difficulties were encountered in avoiding overgrinding of the raw lignite, in effecting an efficient classification, except at low pulp densities and in dewatering the test products by filtration. Work on both methods of physical separation was therefore discontinued because the process had only limited application.

A. Gravity Table Concentration

In all lignites a part of the gangue, which upon roasting becomes ash, is intimately associated with the carbon. In some ores, however, much of the gangue is free of carbon and uranium and fairly coarse. It is possible to treat these raw lignites by gravity tabling and to separate a waste product of very low uranium content. Such treatment could conceivably remove a small load from the roasting step and substantially reduce the quantity of material treated in the leaching circuit.

Batch tabling tests on Peter Kiewit ore, Sample 185-18, rejected 13.9 per cent of the raw lignite feed weight in a product which assayed 0.032 per cent U_3O_8. The calcine from the whole ore was equivalent to 48 per cent of the raw lignite roaster feed. After tabling, the calcine from the carbon concentrate was equivalent to 30.2 per cent of the raw lignite feed weight which represented a reduction of 37 per cent in the weight of feed to the leaching circuit. In addition, the removal of the waste product effected a saving of 74 pounds of H_2SO_4 per ton of raw lignite during leaching. More detailed results of the test are shown in Appendix Table J-1.

Similar results were obtained on Northgate, Sample 185-21. A reject or tailing was made which assayed 0.013 per cent U_3O_8 and represented 10.6 per cent of the raw lignite feed weight. This resulted in a reduction of 55 per cent in the weight of feed to leaching. The saving in acid, however, was only 20 pounds of H_2SO_4 per ton of raw lignite.

The same technique was not successful on Manidon ore, Sample 185-7. Although this ore contained 46 per cent gangue, most of the gangue was intimately associated with the carbon and could not be separated on the table.

In addition to the batch tabling tests, three continuous tests were made. The test on Northgate Smith No. 1 ore, Sample 185-28, is described here. A flowsheet of the test is shown in Figure 27 and the metallurgical results, including roasting and leaching data, for batch leaching testing of products, are presented in Table 42.

The results of the test were not as favorable as had been hoped. In previous tests on Northgate and Peter Kiewit ores removal of ash by tabling had resulted in a filter cake containing only 25 to 30 per cent ash; whereas, in this test, tabling reduced the ash content from 52 per cent to only 44 per cent. The work, however, did point out potential problems in the physical handling of raw lignite ores.

During the first hour, the lignite ore at minus one inch was fed over a vibrating feeder to an agitator ahead of the rod mill. The moisture content of the lignite was 34 per cent. As a result, the feed was sticky and the feed rate extremely slow. Therefore, hand feeding was adopted for the remainder of the run.

The rod mill, with a light charge of only eight rods, was operated, initially, at about 10 per cent solids. When hand feeding commenced, the pulp density was increased to between 25 and 35 per cent solids. In spite of variations in pulp density and feed rate, it was not possible to obtain a substantial amount of plus 10-mesh product for recycle on the Sweco screen. Also, in spite of making this run at a maximum size of 10-mesh, rather than the 20-mesh of the previous tests, the classifier did not rake sands except at low pulp density. The classifier was operated at an average pulp density of 12.3 per cent solids, 50 per cent higher than the density of the previous test. The classifier feed and overflow were 32 per cent and 56 per cent minus 200-mesh respectively. The pulp density of the overflow was approximately four per cent solids.

The classifier sands were fed to a rougher table where a reject, middling and carbon product were removed. The entire reject was collected and the carbon product was pumped to the thickener. The middling was retabled. The resulting reject and middling were combined with the rougher reject. The reject prepresented 11.1 per cent of the feed weight, assayed 0.012 per cent U_3O_8, and contained 1.21 per cent of the U_3O_8 in the feed. The middling carbon product, although it was collected in this test, would normally be pumped to the thickener.

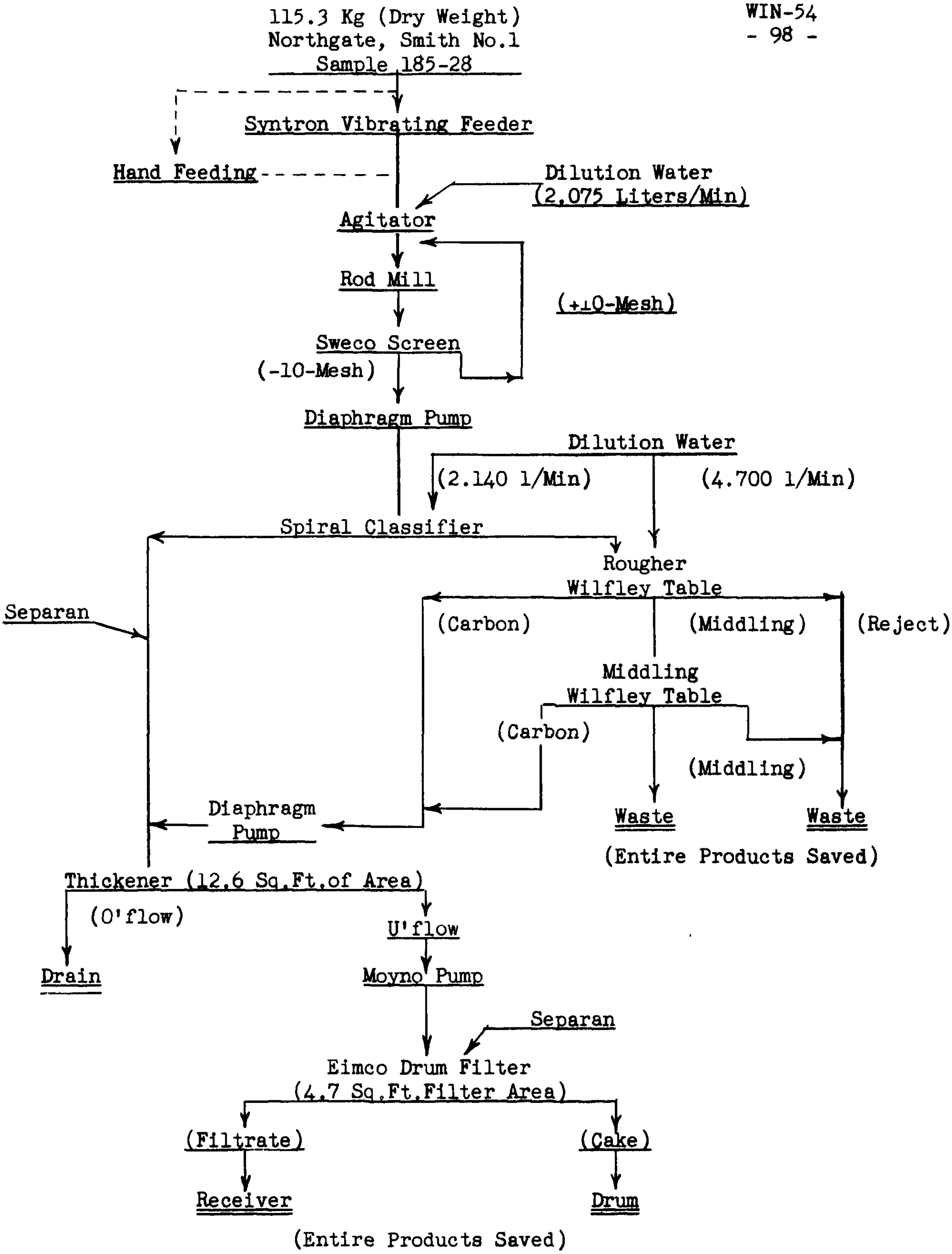

Figure 27. Flowsheet for Physical Beneficiation Testing

Table 42

Metallurgical Results of Physical Beneficiation Test

Sample 185-28

			U_3O_8				
			Assay				
Product	Weight (Kg)	Volume (L)	%	g/l	% Distribution	LOI 1/	% Ash
Ball Mill Feed	115.3		0.095		100.00	47.75	52.2
Table Reject	11.118		0.012		1.21	5.1	94.9
Middling Carbon Product	5.260		0.062		2.98	32.1	67.9
Thickener O'flow (Solution)		1270		0.0002	0.23		
Thickener O'flow (Solids)	9.9		0.094		8.50		
Filtrate		92		0.0015	1.26		
Filter Cake	89.022		0.106		85.82	55.95	44.0

1/ Determined by oxidizing roast for two hours at 450°C.

The thickener, with a total area of 12.6 square feet, was operated with a settling area of 13.9 square feet per ton per day. Separan was used in an attempt to selectively flocculate the carbon (approximately 0.15 pound per ton). It was intended that barren slimes should overflow the thickener. Due either to insufficient Separan or to excessive turbulence in the thickener, the U_3O_8 loss resulting from the carbon in the overflow was higher than anticipated. The assay of the unclarified slime overflow was 0.0075 gram U_3O_8 per liter, which is substantially higher than the 0.001 gram U_3O_8 per liter previously obtained in batch tabling tests on a similar feed and represented 8.7 per cent of the uranium in the feed. The U_3O_8 assay of the solids in the overflow was 0.094 per cent and represented 8.5 per cent of the feed U_3O_8. The solution in the overflow assayed 0.0002 gram U_3O_8 per liter and represented 0.2 per cent of the feed U_3O_8. The thickener overflow was discharged at 37 per cent solids and filtered.

Approximately 0.1 pound of Separan per ton of mill feed was added at the feed box of the filter. The filter was operated on a 2 1/2 minute cycle (the shortest cycle for this filter). Longer cycles resulted in slower filtration. Initially, the vacuum on the filter was between 10 and 15 inches of mercury. The filter area requirement and moisture were 10.4 square feet per ton per day and 70 per cent, respectively. The vacuum gradually built up to a range of 20 to 25 inches of mercury, presumably as a result of cloth blinding. At the end of 1 1/2 hours, the filter area requirement increased to 18.7 square feet per ton per day, and the moisture decreased to 50 per cent. After 2 3/4 hours, the area and moisture had changed to 26.1 square feet per ton per day and 44 per cent. A sample of the filter cake, coned and quartered from the entire product, assayed 0.1 per cent U_3O_8 and 55.9 per cent loss on ignition (two hours at 450°C) and contained 57 per cent moisture.

B. Magnetic Separation

Magnetic separation tests were made on the ash of Samples 185-18, 185-19, and 185-20. For this test work, a Carpco Engineering Corp. Model 102 induced roll separator was used. Only the plus 48-mesh fraction of the sample was passed through the separator.

On Sample 185-18, a split could be made with a field strength of 1.0 ampere to give a non-magnetic reject representing 23.2 per cent of the weight and containing one per cent of the uranium. On Samples 185-19 and 185-20, a satisfactory separation could not be obtained, the non-magnetic fractions assaying 0.12 and 0.19 per cent U_3O_8, respectively. On the lignite ashes of high iron content, a magnetic separation may be practicable, but the procedure does not appear to be applicable to the majority of the lignite ores.

APPENDIX A

Table A-1

Source of Samples

Sample 185-1

66 lbs; received 11/18/54 from A. S. & R. Edgemont
References: Tonry to Woody, 10/20/54
Youngberg to A.S. & R. Edgemont, 10/22/54
Woody to Marvin, 10/26/54
Marvin to Woody, 11/18/54
Enclosure: Tonry to Marvin 11/10/54

Sample 185-2

21.5 lbs; received 1/17/55
Peter Kiewit Company, Rapid City, South Dakota
References: None

Sample 185-3

30 lbs; received 1/17/55
Peter Kiewit
References: None

Sample 185-4

560 lbs; received 4/1/55
Tagged as originated from Bryco Mining Company
Hilltop No. 1 Claim
Note: We shipped 70 lbs of this to Hassialis
References: Youngberg to A.S. & R., 2/14/55
Marvin to Wimpfen, 2/15/55
Van Blarcom to Breitenstein, 2/16/55
Tonry to Marvin, 3/17/55
Woody to A.S. & R., 4/1/55

Sample 185-5

569 lbs; received 4/6/55
Edgemont Stockpile No. 5 (tag in container)
References: Woody to A.S. & R., 4/6/55
Carlson to Van Blarcom, 7/21/55

Sample 185-6

607 lbs; received 5/25/55 from Battelle
Designated as Bryco Hilltop Lignite but may be Stockpile 5
References: Marvin to Breitenstein, 4/19/55
Enclosure: Tonry to Sullivan, BMI 4/15/55
Van Blarcom to Breitenstein with 3 enclosures 5/19/55

Table A-1 (Continued)

Sources of Samples

Sample 185-7

Manidon Property, Billings Co., N.D., NE 1/4 of SW 1/2
Sec 34, T 137 N, R 100 W, top 1/3 of 22 inch bed under
10 to 12 feet of overburden.
510 lbs; received 6/29/55
Manidon, from Rocky Ridge Area, N. Dakota
Shipped by AEC Hot Springs Sub Office
References: King to Breitenstein, 6/27/55

Sample 185-8

6000 lbs; received 7/7/55
Peter Kiewit Jim 1
Drum markings. Shipped by A. S. & R.

Sample 185-9 through 15

Products from Denver
Bureau of Mines Tests
Peter Kiewit Ore

Sample 185-16

Bryco Mining Company; Center of Bryco Hilltop No. 1 Claim
SW 1/4, Sec. 12, T 16 N, R 8 E, Harding Co., South Dakota
14 inch bed under 30 feet of overburden.
4000 lbs gross; received 11/8/55
Shipped from Hot Springs
References: None

Sample 185-17

4225 lbs gross; received 11/14/55
Peter Kiewit
Shipped from Montanta State Collge
Retort Roast Products

Sample 185-18

Peter Kiewit Sons' Company; Jim I - South side of Jim I
Claim, SE 1/4 of NE 1/4 of SE 1/4 of Sec. 22, T 22N,
R 5 E in Harding Co, South Dakota; 16 inch bed from stripped
area. 182 lbs; received 1/16/55; Bbl. marked Peter Kiewit
4000 lbs; received 12/20/55; Shipped by A.S. & R,Hot Springs
References: Letter Upchurch to Breitenstein 12/7/55
Enclosure teletype Simmons to Upchurch 12/6/55

Table A-1 (Continued)

Sources of Samples

Sample 185-19

218 lbs; received 2/24/56
Farmington Lignite Sect. 27, T 22 N, R 5 E, Harding Co.
South Dakota, "E" bed of Tongue River Formation.
Shipped by Farming Funding Corp.
References: Marvin to Chase, 1/25/56
Roscoe to Breitenstein, 2/11/56

Sample 185-20

180 lbs; received 2/29/56
Manidon, Church Lease, Billings Co., North Dakota
Shipped by Manidon Mining Company

Sample 185-21

160 lbs; received 3/26/56
Northgate from Smith No. 1; Northgate Uranium & Oil Co.
Shipped by Lucius Pitkin (Sample request No. 11)
Reference: Rasor to Lucius Pitking, Inc., 2/20/56

Sample 185-22

181 lbs; received 3/26/56
Manidon, Church Lease
Shipper - Lucius Pitkin

Sample 185-23

4000 lbs; received 3/30/56
Peter Kiewit Lignite
Shipper - Lucius Pitkin
References: Sample request No. 1 Rasor to Gaines, 2/15/56
Letter, Pierce to George, 3/21/56

Sample 185-24

4000 lbs; received 3/30/56
Bryco Hilltop from Lucius Pitkin
Reference: Same as 185-23

Sample 185-25

218 lbs; received 4/2/56
Northgate, Ohio Oil Sample No. 6, SW 1/4, Sec. 35, T 141 N,
R 99 W, Billings Co., North Dakota
Reference: Letter Klopp to Breitenstein, 3/23/56

Table A-1 (Continued)

Sources of Samples

Sample 185-26

311 lbs; received 4/2/56
Northgate, Ohio Oil Sample
No. 7, NE 1/4, S 35, T 141 N, R 99 W
Reference: Same as 185-25

Sample 185-27

1000 lbs; received 4/2/56
Manidon, Church Lease
Shipped by Lucius Pitkin
Reference: Sample Request No. 21
Baker to Gaines 3/27/56

Sample 185-28

1000 lbs; received 4/2/56
Northgate Smith No. 1
Northgate Uranium & Oil Co.
Shipper: Licius Pitkin
References: Sample Request No. 21
Baker to Clucius Pitkin, Inc. 3/27/56

Sample 185-29 (A through I) Received 4/2/56

These samples were composited by weight as follows:

Sample	Bed Inches	Depth % of Total	Assay % U_3O_8
A	18	11.75	1.34
B	26	17.0	0.319
C	16	10.4	1.49
D	14	9.2	0.33
E	12	7.9	0.013
F	16	10.4	0.701
G	18	11.75	0.118
H	20	13.1	0.83
I	13	8.5	0.193
Composite	153	100.0	0.61

A series of samples from International Resources, North Cave Hills Area, Harding County, South Dakota, submitted by Mr. Eyrich and described as follows in a letter to Marvin from Eyrich dated March 12, 1956.

Table A-1 (Continued)

Sources of Samples

Sample 185-29A - Can #1 and Can #2

79 lbs, Sample "E"-1, lignite ore, bottom section "E" bed. Woody mateiral-18" thick - from trench in ore in strip pit #1 on claim Lucky Strike #1.

Sample 185-29B - Can #3

37 lbs, Sample "E"-2, lignite ore, top section "E" bed. Unconsolidated material - 26" thick - from trench in ore in strip pit #1 on claim Lucky Strike #1.

Sample 185-29C - Can #4

37.5 lbs, Sample "E"-3, lignite ore, full section "E" bed. Unconsolidated material - 16" thick - from trench in ore in strip pit #4 on claim Last Chance #1.

Sample 185-29D - Can #5

41 lbs, Sample "E"-4, lignite ore, full section "E" bed face sample - 14" thick - from dozer cut 20 feet from original outcrop line on claim Last Chance #1.

Sample 185-29E - Can #6

52.5 lbs, Sample "E"-5, lignite ore, full section "E" bed-12" ore - from trench cut in ore in #2 strip pit on claim Lucky Seven #5.

Sample 185-29F - Can #7

51 lbs, Sample "D"-1, lignite ore. Full section top bench "D" bed - 16" thick - cut from trench in ore in strip pit #3 on claim Hoop and Holler #3.

Sample 185-29G - Can #8

47.5 lbs, Sample "D"-2, lignite ore. Full section top bench "D" bed face sample - 18" thick - from dozer cut head of Knutson Creek on claim Lucky Strike #2.

Sample 185-29H - Can #9

43 lbs, Sample "D"-3, lignite ore. Full section top bench "D" bed - 20" thick - cut from trench in ore in strip pit #5 on claim Hoop and Holler #2.

Sample 185-29I - Can #10

41 lbs, Sample "E"-6, lignite ore. Highly oxidized material. "E" bed face sample - 13" thick - from dozer cuts on claim Last Chance #1. This type material represents a very small percentage of the total, but it is different and probably has resulted from burning of the lignite bed.

Table A-1 (Continued)

Source of Samples

Sample 185-30

281.5 lbs; received 4/10/56
Ohio Oil Co., Sample No. 8
Saddle Buttes Area, Billings Co., North Dakota, Ohio-Pew
N.E. 1/4, Sect 9, T 140 N, R 100 W
Reference: Letter Klopp to Breitenstein 4/23/56

Sample 185-31

274 lbs; received 4/16/56
Ohio Oil Co., Sample No. 9, Saddle Buttes Area
Ohio-Pew Obrigewitch
SE 1/4, Sec. 23, T 142 N, R 99 W
References: Letter Klopp to Breitenstein 4/23/56

Sample 185-32

Products from roasting studies at Coal Technology
Pilot Plant, U. S. Bureau of Mines, Denver, Colorado
References: Letter Landers to Thorpe, 4/17/56

Sample 185-33

294.5 lbs; received 4/24/56
Ohio Oil Company, Sample No. 10
Saddle Buttes Area, Ohio-Pew
Church Lease, SE 1/4, Sec. 29, T 142 N, R 99 W
References: See Sample 185-30

Sample 185-34

271 lbs; received 4/25/56
Ohio Oil Company, Sample No. 11
South Belfield Area, Billings Co., North Dakota,
Uranco Properties
SE 1/4, Sec. 5, T 136 N, R 100 W
References: Same as above

Sample 185-35

211 lbs; received 5/2/56
Homestake Riley 4, Bowman, North Dakota
References: None

Sample 185-36A

11.5 lbs; received 3/28/56
Sample of fluosolids roaster discharge from Grand Junction
Pilot Plant

Table A-1 (Continued)

Source of Samples

Sample 185-36B

13.5 lbs; received 3/28/56 - Sample of No. 1 cyclone dust from Grand Junction Pilot Plant

Sample 185-37

62.5 lbs; received 6/15/56 from Grand Junction Pilot Plant
International Resources Corp., Mine Run E-Bed #1 -
Strip Belt - Lot #1, Bowman, North Dakota
References: None

Sample 185-38

76.0 lbs; received 6/15/56 from Grand Junction Pilot Plant
International Resources Corp., Mine Run D-Bed #5 -
Strip Belt - Lot #2, Bowman, North Dakota
References: None

Sample 185-39

567 lbs in drums 1-3; received 8/6/56
Norther Pacific Railway Company
NE 1/4, Sec. 15, T 140 N, R 100 W, Billings Co. N. Dakota
References: Curtiss to Woody, 7/26/56
Curtiss to Foran, et al, 7/30/56

Sample 185-40

1712 lbs in drums 4-13; received 8/6/56
Ohil Oil Co. (H.R. Talkington and reaquired Federal land)
3 bbls, Pit No. 1, NE 1/4, Sec 9, T 140 N, R 100 W, Billings Co., N. Dakota
2 bbls, Pit No. 2, NE 1/4, Sec 10, T 140 N, R 100 W, Billings Co.,N. Dakota
3 bbls, Pit No. 3, NE 1/4, Sec 10, T 140 N, R 100 W, Billings Co.,N. Dakota
2 bbls, Pit No. 4, NE 1/4, Sec 10, T 140 N, R 100 W, Billings Co., N. Dakota
References: Same as 185-39

Sample 185-41

507 lbs in drums 14-16, received 8/6/56
T. L. Landis, Lessee, (Louis Hecker, owner)
Pit No. 1, East end of ore block #2, SW 1/4, Sec 35, T 141 N, R 99 W, Billings Co., N. Dakota
Reference: Same as 185-39

Table A-1 (Continued)

Source of Samples

Sample 185-42

680 lbs in drums 17-21, received 8/6/56 from USAEC,
Dickinson, North Dakota T. L. Landis (Louis Hecker)
1 bbl from Pit No. 1 (ore block #1) NE 1/4, Sec 35,
T 141 N, R 99 W, Billings Co., North Dakota
2 bbls from Pit No. 2 (ore block #1)
References: Sample as 185-39

Sample 185-43

602 lbs in drums 21-23, received 8/6/56 from USAEC,
Dickinson, North Dakota, T. L. Landis, Lessee,
(Emil Rodakowski, Owner),
NE 1/4, Sec 36, T 141 N, R 99 W, Billings Co.,North Dakota
References: Same as 185-39

Sample 185-44

545 lbs in drums 24-26, received 8/6/56 from USAEC,
Dickinson, North Dakota, T. L. Landis, Lessee,
(Anton Rodakowski, Owner)
SE 1/4 NE 1/4, Sec 34, T 141 N, R 99 W, Billings Co.,
North Dakota
References: Same as 185-39

Sample 185-45

797 lbs in drums 27-30, received 8/6/56 from USAEC,
Dickinson, North Dakota, Leo Gardner, Lessee,
(L. E. Smith, Owner No. 2 Lease)
2 bbls from Pit No. 1, 4600 feet north and 1900 feet west
of the SE corner of Sec. 6 in the N 1/2 N 1/2, Sec 6,
T 137 N, R 100 W, Billings Co., North Dakota
2 bbls from Pit No. 2, 4600 feet north and 1000 feet west
of the SE corner of Sec 6 in the N 1/2 N 1/2, Sec 6,
T 137 N, R 100 W, Billings Co., North Dakota
References: Same as 185-39

Sample 185-46

509 lbs in drums 31-33, received 8/6/56 from USAEC,
Dickinson, North Dakota, Ohio Oil Co., Lessee
(Joseph Obrigewitch Owner)
SE 1/4, Sec 23, T 142 N, R 99 W, Billings Co., North Dakota
References: Same as 185-39

Table A-1 (Continued)

Source of Samples

Sample 185-47

2262 lbs in drums 34-44, received 8/6/56
G. Whitman, Lessee (A. Schwartz, Owner)
2 bbls from Pit No. 1, NW 1/4, Sec 2, T 137 N, R 100 W, Billings Co., North Dakota
2 bbls from Pit No. 1, NW 1/4, Sec 3, T 137 N, R 100 W, Billings Co., North Dakota
2 bbls from Pit No. 2, NW 1/4, Sec 3, T 137 N, R 100 W, Billings Co., North Dakota
3 bbls from Pit No. 1, NW 1/4, Sec 11, T 137 N, R 100 W, Billings Co., North Dakota
2 bbls from Pit No. 2, NW 1/4, Sec 11, T 137 N, R 100 W, Billings Co., North Dakota
References: Same as 185-39

Sample 185-48

144 lbs in one drum, received 8/6/56
Ohio Oil Company, Lessee (J. Chrusch, Owner)
SE 1/4, Sec 29, T 142 N, R 99 W, Billings Co., North Dakota
References: Same as 185-39

Sample 185-54

International Resources Corporation, North Cave Hills, South Dakota, Last Chance No. 142, Sec 21, TWP 22 N, R 5 E. From stockpile at Grand Junction Pilot Plant.
Received at Winchester 12/6/56 - 6000 pounds
Reference: Letter from W. L. Lennemann to J. F. Foran 11/29/56

Sample 185-55

Ohio Oil Company - Northgate Uranium and Oil Company, Billings Co., North Dakota. Johnson Mine, Sec 3, TWP 140 N, R 100 W; Klyn Mine, Sec 26, TWP 142 N, R 99 W;
Received at Winchester 12/6/56 - 6000 pounds
Reference: Same as 185-54

Table A-2

Analyses of Samples

Sample Number	U_3O_8	Mo	Total Carbon	LOI 1/	Fe	Al_2O_3	CaO	MgO	Na_2O + K_2O	Total S as $SO_4^=$	Sol. $SO_4^=$	CO_2	SiO_2	P_2O_5	V_2O_5
185-1	0.275	0.33	18.6	41.7	6.0	--	2.48	<0.1	--	1.42	--	--	32.7	1.45	0.04
185-2	0.160	0.085	39.8	72.1	5.05	3.9	0.97	0.37	2.75	3.2	--	--	9.03	0.63	0.043
185-3	0.166	0.14	29.4	68	6.06	3.0	1.72	0.36	3.31	7.63	--	--	10.6	0.90	0.027
185-4	0.317	0.31	20.2	38	2.61	9.0	1.67	0.99	--	4.93	--	--	32.5	0.154	0.012
185-5	0.241	0.34	23.9	42	2.76	9.4	1.65	1.05	--	5.10	--	--	30.6	0.256	<0.001
185-6	0.286	0.29	20.9	40	2.78	10.5	2.55	0.98	--	6.11	--	0.24	30.8	0.175	0.03
185-7	0.156	0.12	53.6	82	1.93	2.1	1.45	0.48	--	8.21	--		5.65	0.15	0.02
185-8	0.29			46											
185-9	0.410	--	--	--	2.80	10.3	10.2	0.84	--	11.7	--	0.38	28.2	0.14	0.03
185-10	0.491	--	--	--	3.28	12.1	2.89	1.24	--	8.24	--	0.81	32.4	0.13	0.03
185-11	0.431	--	--	--	--	--	--	--	--	--	--	--	--	--	--
185-12	0.415	--	--	--	2.97	11.7	2.40	1.02	--	7.26	--	0.93	29.4	0.24	0.03
185-13	0.402	--	--	--	2.89	13.0	2.42	1.09	--	6.45	--	0.71	31.4	0.10	0.03
185-14	0.455	--	--	--	2.91	13.0	2.35	1.01	--	6.73	--	0.86	31.8	0.08	0.03
185-15	0.503	--	--	--	3.00	11.8	2.41	1.16	--	6.97	--	0.88	30.7	0.10	0.03
185-16	0.353	--	--	40	2.82	9.9	2.10	0.82	2.42	6.22	--	--	31.5	0.13	0.03
185-17															
185-18	0.205	--	--	52	5.73	7.4	3.89	1.25	3.04	1.66	--	0.75	18.7	0.11	<0.01
185-19	0.283	0.60	20.3	43	10.1	6.3	3.60	0.70	1.25	6.20	--	0.28	21.3	0.33	0.03
185-20	0.162	0.14	45.5	68	4.03	3.3	1.25	0.42	--	10.9	--	0.62	15.8	0.18	0.02
185-21	0.10	0.11	24.7	47	6.6	8.2	0.43	0.47	1.50	5.02	0.27	0.83	28.5	0.15	0.02
185-22	0.154	0.15	--	69	3.66	3.1	1.25	0.40	0.70	10.3	--	0.48	15.6	0.18	0.012
185-23															
185-24															
185-25	0.233	0.19	42.8	66	3.78	2.4	4.36	0.51	0.15	2.46	--	0.23	8.60	0.10	0.004

Table A-2 (Continued)

Analyses of Samples

Sample Number	U_3O_8	Mo	Total Carbon	LOI [1]	Fe	Al_2O_3	CaO	MgO	Na_2O + K_2O	Total S as $SO_4^=$	Sol. $SO_4^=$	CO_2	SiO_2	P_2O_5	V_2O_5
185-26	0.136	0.114	18.8	46	10.3	5.5	6.02	0.95	0.68	2.66	--	1.15	20.5	0.22	0.016
185-27	0.145			32											
185-28	0.087	0.093		46											
185-29A	1.34	0.30	45.8	71.4											
185-29B	0.319	0.16	25.5	51.9											
185-29C	1.49	0.12	31.1	58.9											
185-29D	0.33	0.096	37.5	61.7											
185-29E	0.013	0.069	7.64	20.5											
185-29F	0.701	0.087	20.5	42.1											
185-29G	0.118	0.019	25.3	44.7											
185-29H	0.83	0.042	36.5	63.7											
185-29I	0.193	0.15	11.8	31.1											
185-29 (A-I)[2]	0.593	0.045	--	51	6.20	5.7	2.97	0.86	1.82	7.01	--	0.44	19.7	--	0.031
185-30	0.267	0.24	31.9	62	7.94	3.9	3.76	0.43	1.53	9.30	--	0.34	9.32	0.21	0.18
185-31	0.359	0.063	30.1	57	9.37	3.3	6.77	1.48	0.64	7.50	--	0.30	9.50	0.26	0.029
185-32	No assays - Miscellaneous products for U.S.B.M., Denver, Colorado														
185-33	0.103	0.096	38.2	67	5.49	2.4	4.82	2.0	--	6.69	--	0.26	7.15	0.11	0.017
185-34	0.181	0.11	31.9	60	7.91	3.1	1.56	0.25	--	6.08	--	0.25	16.9	0.17	0.017
185-35	0.680	0.15	--	49	7.96	6.2	3.73	0.85	2.5	2.12	--	0.28	20.0	0.19	0.051
185-36A	0.157	0.18	1.05	Roaster Cyclone											
185-36B	0.433	0.37	2.87	Roaster Cyclone											
185-37	0.347	0.088	--	67	2.79	5.2	1.92	0.66	1.83	5.97	--	0.27	12.2	0.09	0.009
185-38	0.176	0.049	--	55	5.26	3.4	5.48	1.22	1.27	7.09	--	0.29	17.3	0.15	0.022
185-39	0.347	0.213	24.6	60	5.54	--	4.24	3.2	0.84	--	2.3	0.47	12.5	0.20	0.012

Table A-2 (Continued)

Analyses of Samples

Sample Number	U_3O_8	Mo	Total Carbon	LOI[1/]	Fe	Al_2O_3	CaO	MgO	Na_2O + K_2O	Total S as $SO_4^=$	Sol. $SO_4^=$	CO_2	SiO_2	P_2O_5	V_2O_5
185-40	0.157	0.434	28.0	61	6.60	--	5.41	0.89	1.60	--	3.11	0.32	10.1	0.19	0.014
185-41	0.202	0.223	27.8	64	7.66	--	3.40	--	0.3	--	0.13	0.32	12.3	0.13	0.017
185-42	0.058	0.081	29.3	64	--	3.7	3.45	1.10	0.78	--	2.27	0.25	10.9	0.12	0.017
185-43	0.090	0.080	3.02	14	5.39	14.3	3.06	1.41	--	--	2.32	1.31	46.1	0.24	0.045
185-44	0.560	1.31	28.5	59	3.96	3.8	2.27	0.74	--	--	0.35	0.43	22.1	0.42	0.013
185-45	0.260	0.328	23.5	52	6.92	6.9	0.65	0.37	--	--	0.38	0.71	23.5	0.23	0.030
185-46	0.131	0.11	29.0	63	4.32	2.9	6.48	2.4	--	--	0.38	1.51	11.9	0.10	0.015
185-47	0.143	0.290	19.4	37	5.72	9.6	0.54	0.45	--	--	0.47	0.22	36.1	0.26	0.029
185-48	0.119	0.095	27.9	66	4.66	4.3	4.77	2.2	0.97	--	1.72	0.72	14.0	0.13	0.014

1/ LOI = Loss on Ignition determined by roasting in an electric muffle furnace at 450°C for two to three hours - Sample hand rabbled at 20 minute intervals.

2/ See makeup of composite in Appendix A, Table A-1

Table A-3

Precision laboratory distillation assays and analyses. Temperature study on uraniferous lignite, AEC sample No.185-4.(Denver Lab.No.1042)3/

Temperature of distillation,°C	200	200	300	300	350	350	400	400	450	450	500	500	600	600
Assay No.	529	530	527	528	538	539	519	521	540	541	516	517	518	520
Carbonization yields, MAF, pct.														
Char	98.6	98.8	85.8	86.6	78.9	78.8	72.0	72.3	66.8	66.5	60.0	59.7	48.0	49.7
Water formed	0.0	0.0	4.3	4.6	6.0	6.0	5.3	5.3	7.7	7.6	9.1	9.1	13.5	12.1
Tar, dry	0.0	0.0	0.0	0.0	0.0	0.3	0.0	0.0	0.3	0.0	0.6	0.9	1.2	1.5
Light oil	0.6	0.6	1.7	1.7	1.7	2.0	2.6	2.1	2.0	2.6	2.6	2.9	2.9	2.6
Gas	0.8	0.8	8.0	8.4	13.2	13.1	20.3	20.6	22.6	23.0	27.6	27.3	33.8	34.1
Hydrogen sulfide	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.3	0.3	0.3	0.3	0.3	0.3
Total	100.0	100.0	99.8	100.3	100.0	100.2	100.2	100.3	99.7	100.0	100.2	100.2	99.7	100.3
Composition of gas, pct. 1/														
CO_2	84.1	90.7	88.9	89.3	90.2	89.2	92.8	92.0	89.6	92.1	85.6	88.2	75.8	76.1
Illuminants	0.0	0.0	0.4	0.3	0.1	0.2	0.2	0.3	0.2	0.1	0.4	0.2	0.2	0.2
CO	1.4	0.9	4.5	4.3	4.6	5.8	3.8	4.4	4.3	1.7	4.8	4.2	6.2	6.2
H_2	8.7	6.2	3.1	2.9	2.4	1.5	2.0	2.1	3.2	3.8	5.7	4.1	11.8	11.1
CH_4	2.9	0.9	1.4	1.3	1.3	2.7	0.9	0.9	1.8	2.2	2.1	2.7	5.9	6.3
C_2H_6	2.9	1.3	1.7	1.9	1.4	0.6	0.3	0.3	0.9	0.1	1.4	0.6	0.1	0.1
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Net gas yield, MAF, cu.ft./lb.	0.096	0.051	0.732	0.677	1.177	1.200	1.808	1.838	2.071	2.106	2.622	2.542	3.545	3.535
Heat in gas, MAF, Btu/lb.	10.9	2.8	57	52	74	79	69	77	130	127	233	175	443	446
Heating value, Btu/cu.ft.calc.	114	55	78	77	63	66	38	42	63	60	89	69	125	126
Sp. gr.,60°/30 in.,dry, calc.	1.342	1.410	1.430	1.435	1.443	1.434	1.464	1.455	1.428	1.434	1.385	1.410	1.242	1.260
Prox. analysis of coal, MAF														
Volatile matter	68.9	68.9	68.9	68.9	68.9	68.9	68.9	68.9	68.9	68.9	68.9	68.9	68.9	68.9
Fixed carbon	31.1	31.1	31.1	31.1	31.1	31.1	31.1	31.1	31.1	31.1	31.1	31.1	31.1	31.1
Prox.analysis of assay char,MF.														
Volatile matter	27.5	27.5	23.7	23.7	21.2	21.2	18.2	18.2	16.6	16.6	14.2	14.2	9.7	9.7
Fixed carbon	11.9	11.9	11.8	11.8	13.6	13.6	14.1	14.1	14.1	14.1	15.8	15.8	17.8	17.8
Ash	60.6	60.6	64.5	64.5	65.2	65.2	67.7	67.7	69.3	69.3	70.0	70.0	72.5	72.5
Caking characteristic of char2/	1	1	1	1	1	1	1	1	1	1	1	1	1	1

1/ Gas analyses shown on oxygen- and nitrogen-free basis.
2/ Char caking: (1) None (2) Weakly (3) Strong.

3/ Base sample prepared by Atomic Energy Commission (Nat. Lead Co.) Dakota Lignite, Cavehill Mining Co., No.1 claim, Harding Co.,So.Dakota.(Seam 18"-24"). Chem.Anal.not requested.

Table A-4

6-139
(September 1943)

UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF MINES

G—COAL-ANALYSIS REPORT

Test No. .... Lab. No. F-31603

Sample of Coal - NAa Can No. ....

Operator .... Mine ....

State .... County .... Bed ....

~~xxxxx~~ Submitted by National Lead Co., Winchester, Mass.
International Resources Corp., Lignite from Grd. Jct.
~~xxxxxxxxxxx~~ P. P. Rep. Cut 4.5 lbs. net.

Method of sampling .... Gross weight, lbs. .... Net weight, grams 2052.5

Date of sampling .... Date of Lab. sampling 12/12/56 Date of analysis ....

B. of M. o~~xxxxxx~~ section Referee Collector ....

	Air-dry Loss	Coal (Air dried)	Coal (As received)	Coal (Moisture free)	Coal (Moisture and ash free)
Proximate Analysis	Moisture		20.2		
	Volatile matter *		27.7	34.7	
	Fixed carbon		14.2	17.8	
	Ash		37.9	47.5	
			100.0	100.0	
Ultimate Analysis	Hydrogen		3.8	1.9	
	Carbon		24.3	30.4	
	Nitrogen		.5	.6	
	Oxygen		32.3	18.1	
	Sulphur		1.2	1.5	
	Ash		37.9	47.5	
			100.0	100.0	
	British thermal units		3570	4470	

Fusibility of Ash, °F.	Initial deformation temperature	
	Softening temperature	
	Fluid temperature	

*Determined by modified method

Date December 31, 1956 (Signed) Roy F. Abernethy *Chemist.*

BBK

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U. S. GOVERNMENT PRINTING OFFICE 16—35304-1

Table A-5

6-189
(September 1943)

UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF MINES

Test No. ....

G—COAL-ANALYSIS REPORT

Lab. No. F-31604

Sample of Coal - NAa

Can No. ....

Operator .... Mine ....

State .... County .... Bed ....

XXXX Submitted by National Lead Co., Winchester, Mass.
Ohio Oil Co. Lignite from Grd. Jct. P. P. 4.5 lbs. net
XXXXXXXXXXXX Rep. Cut. as rec'd.

Method of sampling .... Gross weight, lbs. .... Net weight, grams 2030.5

Date of sampling .... Date of Lab. sampling 12/12/56 Date of analysis ....

B. of M. XXXXXXXX. section Referee Collector ....

	Air-dry Loss	Coal (Air dried)	Coal (As received)	Coal (Moisture free)	Coal (Moisture and ash free)
Proximate Analysis	Moisture		28.8		
	Volatile matter *		25.3	35.5	
	Fixed carbon		12.1	17.0	
	Ash		33.8	47.5	
			100.0	100.0	
Ultimate Analysis	Hydrogen		4.7	2.1	
	Carbon		21.0	29.5	
	Nitrogen		.4	.6	
	Oxygen		39.5	19.5	
	Sulphur		.6	.8	
	Ash		33.8	47.5	
			100.0	100.0	
	British thermal units		3090	4340	

Fusibility of Ash, °F.
Initial deformation temperature ....
Softening temperature ....
Fluid temperature ....

*Determined by modified method

Date December 31, 1956
BBK

(Signed) Roy F. Abernethy
Chemist.

U. S. GOVERNMENT PRINTING OFFICE 10—35304-1

Table A-6

6-139
(September 1943)

UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF MINES

G—COAL-ANALYSIS REPORT

Test No. ....

Lab. No. F-31605

Sample of Coal - NAa

Can No. ....

Operator ....

Mine ....

State .... County .... Bed ....

~~Town~~ Submitted by National Lead Co., Winchester, Mass.

~~Location in mine~~ Edgmont S. P. 5 Lignite from Grd. Jct. P. P. 3.25 lbs. net (1/2 cut as rec'd)

Method of sampling .... Gross weight, lbs. .... Net weight, grams 1556.5

Date of sampling .... Date of Lab. sampling 12/12/56 Date of analysis ....

B. of M. or ~~U.S.G.S.~~ section Referee Collector ....

	Air-dry Loss	Coal (Air dried)	Coal (As received)	Coal (Moisture free)	Coal (Moisture and ash free)
Proximate Analysis	Moisture		9.7		
	Volatile matter*		22.5	24.9	
	Fixed carbon		11.0	12.2	
	Ash		56.8	62.9	
			100.0	100.0	
Ultimate Analysis	Hydrogen		2.3	1.3	
	Carbon		19.8	21.9	
	Nitrogen		.4	.4	
	Oxygen		20.1	12.8	
	Sulphur		.6	.7	
	Ash		56.8	62.9	
			100.0	100.0	
	British thermal units		2870	3180	

Fusibility of Ash, °F.		
	Initial deformation temperature	
	Softening temperature	
	Fluid temperature	

*Determined by modified method

Date December 31, 1956 (Signed) Roy F. Abernethy
Chemist.

BBK

U. S. GOVERNMENT PRINTING OFFICE 16—[illegible]

Table A-7

6-189
(September 1948)

UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF MINES

G—COAL-ANALYSIS REPORT

Test No.

Lab. No. F-31606

Sample of Coal - NAa

Can No.

Operator

Mine

State

County

Bed

XXXX Submitted by National Lead Co., Winchester, Mass.
North Gate Lignite from Grd. Jct. P. P., net wt. 2.5

XXXXXXXXXXXX lbs. as rec'd

Method of sampling

Gross weight, lbs.

Net weight, grams 1200.0

Date of sampling

Date of Lab. sampling 12/12/56

Date of analysis

B. of M. oXXXXXXXX section Referee

Collector

	Air-dry Loss	Coal (Air dried)	Coal (As received)	Coal (Moisture free)	Coal (Moisture and ash free)
Proximate Analysis	Moisture		6.2		
	Volatile matter *		26.1	27.9	
	Fixed carbon		9.7	10.3	
	Ash		58.0	61.8	
			100.0	100.0	
Ultimate Analysis	Hydrogen		2.1	1.5	
	Carbon		20.0	21.3	
	Nitrogen		.4	.5	
	Oxygen		18.7	14.0	
	Sulphur		.8	.9	
	Ash		58.0	61.8	
			100.0	100.0	
	British thermal units		2910	3100	

Fusibility of Ash, °F.
- Initial deformation temperature
- Softening temperature
- Fluid temperature

*Determined by modified method

Date December 31, 1956
BBK

(Signed) Roy F. Abernethy
Chemist.

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Table B-1

Single-Stage Acid Leaching Tests of Lignite Ash - Muffle Roast at 450°C

Sample Number	Roast Time, Hours	% L.O.I.	Ash % U_3O_8	Grind pH	Leaching Conditions Time, Hours	Leaching Conditions % Solids	Leaching Conditions Temp °C	H_2SO_4, Lb/Ton Ash	H_2SO_4, Lb/Ton Lignite	Terminal Conditions pH	Terminal Conditions Emf (mv)	Residue Assay % U_3O_8	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay, g/l[1] U_3O_8	Mo	Fe	$SO_4^=$
185-1	2	42	0.47	--	6	33	75	875	510	--	-450	0.03	95	1.04	0.12	8.0	97
185-2	2	72	0.21	--	6	33	75	625	175	--	--	0.07	70				
185-3	2	68	0.52	--	6	33	75	425	135	--	--	0.16	70				
185-4	2	38	0.60	--	6	40	80	200	125	3.2	-280	0.12	77.5				
					6			400	250	2.9	-300	0.082	85.7				
					6			800	500	0.8	-465	0.044	94.0				
					6			1600	1000	0.0	-505	0.032	94.4				
					1/2			800	500	0.8	-470	0.052	92.8				
					2			800	500	1.0	-470	0.040	94.0				
					4			800	500	1.0	-460	0.042	94.3				
					16			800	500	1.2	-455	0.052	92.4				
185-5	2	42	0.41		6	50	35	480	280	--	--	0.069	85.2				
					6		80	600	350	--	--	0.043	91.8				
					6		80	900	520	--	--	0.034	93.6				
					18		80	480	280	--	--	0.062	87.3				
185-6	2	40	0.48		6	40	75	700	420	0.5	-450	0.02	97	1.24	0.69	4.2	92
185-7	3	82	0.90 [2]		6	40	75	300	55	3.3	--	--	65.5	1.47	0.76		
								500	90	1.7	--	--	80.6	1.81	1.05		
								700	125	0.9	-410	--	87.3	1.97	1.10		
								900	160	0.7	-440	--	89.1	2.00	1.51		
								1000	180	0.2	-450	--	89.5	2.00	1.54		
								1200	215	0.1	-460	--	90.2	2.02	1.57		
185-8[3]	2	46	0.52	11	6	40	75	1500	750	0.5	-475	0.02	97	1.1	0.35	14.2	125

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Table B-1 (Cont'd.)

Single-Stage Acid Leaching Tests of Lignite Ash - Muffle Roast at 450°C

	Roast		Ash			Leaching Conditions			H_2SO_4, Lb/Ton		Terminal Conditions		Residue Assay		Diluted Pregnant Liquor Assay, g/l			
Sample Number	Time, Hours	% L.O.I.	% U_3O_8	Grind pH	Feed Mesh	Time, Hours	% Solids	Temp °C	Ash	Lignite	pH	Emf (mv)	% U_3O_8	% U_3O_8 Ext'n	U_3O_8	Mo	Fe	$SO_4^=$
185-16	2	40	0.55	5	-10	6	40	75	615	370	0.5	-435	0.06	90	1.2	0.7	3.2	85
	↓	↓	0.57	[4]	↓	↓	↓	85-89	200	120	3.0	--	--	76.0	1.08	0.48		
	↓	↓	↓	↓	↓	↓	↓	↓	400	240	2.8	--	--	86.9	1.23	0.59		
	↓	↓	↓	↓	↓	↓	↓	↓	500	300	2.2	--	--	90.0	1.28	0.72		
	↓	↓	↓	↓	↓	↓	↓	↓	600	360	1.2	--	--	92.6	1.32	0.90		
	↓	↓	↓	↓	↓	↓	↓	↓	700	420	0.7	--	--	93.4	1.33	0.95		
	↓	↓	↓	↓	↓	↓	↓	↓	800	480	0.4	--	--	94.6	1.35	1.03		
185-18	2	52	0.47	11.5[4]	-10	6	30	90	400	190	4.0	--	0.28	31.7	0.37			
	↓	↓	↓	↓	↓	↓	↓	↓	500	240	3.4	--	0.14	63.7	0.75			
	↓	↓	↓	↓	↓	↓	↓	↓	600	290	3.2	--	0.08	77.3	0.91			
	↓	↓	↓	↓	↓	↓	↓	↓	700	335	3.4	--	0.09	79.0	0.93			
	↓	↓	↓	↓	↓	↓	↓	↓	800	385	2.9	--	0.05	89.0	1.05			
	↓	↓	↓	↓	↓	↓	↓	↓	1000	480	1.9	--	0.04	93.7	1.10			
	↓	↓		↓	↓	↓	↓	↓	1100	530	1.6	--	0.02	96.5	1.13			
	↓	↓	0.49	↓	↓	↓	↓	40-50	1100	530	2.3	-430	--	94.7	1.16	0.14		
	↓	↓	↓	↓	↓	↓	↓	70-75	1100	530	2.1	-430	--	94.9	1.16	0.09		
	↓	↓	↓	↓	↓	↓	↓	91-98	1100	530	1.6	-415	--	94.5	1.16	0.06		
	2	↓	0.46	↓	↓	↓	40	75	1460	700	-	--	0.015	97.1	--	--		
	4	↓	↓	↓	↓		↓	↓	1460	700	-	--	0.012	97.8	--	--		
	2	↓	↓	↓	↓	18	↓	↓	1460	700	-	--	0.012	97.5	--	--		
	4	↓	↓	↓	↓	18	↓	↓	1460	700	-	--	0.007	98.9	--	--		
185-19	2	43	0.50	-	-10	6	40	75	620	350	1.0	-470	0.085	84.1	1.05	0.8	-	77
	↓	↓	↓	6	-35	6	↓	↓	700	400	1.1	-460	0.056	90.9	1.13	0.9	9.7	--
	↓	↓	↓	↓	↓	6	↓	↓	800	455	0.4	-480	0.046	92.2	1.15	1.0	12.2	--
	↓	↓	↓	↓	↓	6	↓	↓	900	510	0.2	-495	0.039	93.6	1.17	1.3	15.5	--
	↓	↓	↓	↓	↓	6	↓	↓	1000	565	0.1	-495	0.033	94.7	1.18	1.5	20.7	--
	↓	↓	↓	↓	↓	18	↓	↓	1000	565	0.5	-470	0.037	94.0	1.18	1.0	17.0	123

Table B-1 (Cont'd.)

Single-Stage Acid Leaching Tests of Lignite Ash - Muffle Roast at 450°C

Sample Number	Roast Time, Hours	% L.O.I.	Ash % U_3O_8	Grind pH	Feed Mesh	Leaching Conditions Time, Hours	Leaching Conditions % Solids	Leaching Conditions Temp °C	H_2SO_4, Lb/Ton Ash	H_2SO_4, Lb/Ton Lignite	Terminal Conditions pH	Terminal Conditions Emf (mv)	Residue Assay % U_3O_8	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay, g/l U_3O_8	Diluted Pregnant Liquor Assay, g/l Mo	Diluted Pregnant Liquor Assay, g/l Fe	Diluted Pregnant Liquor Assay, g/l $SO_4^=$
185-19	2	43	0.58	--	-10	6	40	70	550	315	1.45	-450	0.088	84.0	1.09	0.30	3.3	57
								80	550	315	1.40	-430	0.080	84.3	1.09	0.036	1.2	49
								90	550	315	1.60	-370	0.080	84.2	1.09	0.015	0.7	48
								100	550	315	1.65	-370	0.080	84.0	1.09	0.013	0.5	43
								98	600	340	2.55	-365	0.071	86.9	1.24	0.008	0.4	--
									700	400	2.00	-400	0.064	88.7	1.27	0.020	0.6	--
									800	455	1.40	-430	0.046	92.1	1.33	0.036	2.3	--
									900	510	1.10	-445	0.041	93.1	1.38	0.069	4.9	--
									1000	565	0.70	-470	0.041	93.1	1.38	0.11	6.2	
185-20	3	68	0.50	--	-10	6	40	75	350	110	1.0	-430	0.11	78.2	0.98	0.55	-	56
				4.5	-35				500	160	0.4	-455	0.082	83.8	1.05	0.69	3.7	73
									600	190	0.1	-460	0.069	86.6	1.08	0.76	5.4	83
									700	225	0.0	-470	0.052	90.0	1.13	0.81	7.0	95
						18			700	225	0.2	-465	0.065	87.6	1.09	0.82	5.6	93
						2			700	225	0.0	-470	0.066	87.0	1.09	0.74	5.5	90
						4			700	225	0.0	-470	0.064	87.4	1.10	0.80	5.9	91
						6			700	225	0.0	-470	0.052	90.0	1.13	0.81	7.0	95
						18			700	225	0.2	-465	0.065	87.6	1.09	0.82	5.6	93
						6		25	700	225	0.0	-480	0.11	77.2	0.97	0.75	1.8	88
								50	700	225	0.0	-480	0.077	84.5	1.06	0.77	4.2	87
								75	700	225	0.0	-470	0.052	90.0	1.12	0.81	7.0	95
								100	700	225	0.2	-465	0.042	92.0	1.15	0.70	6.4	86
				--	-10			98	150	45	2.45	-285	0.19	63.0	0.80	0.30	0.3	--
									220	70	2.00	-315	0.15	71.0	0.87	0.30	0.5	--
									310	100	1.25	-380	0.13	75.1	0.93	0.25	0.9	--
									600	190	0.50	-470	0.080	85.8	1.10	0.65	4.8	--
									700	225	0.20	-480	0.065	88.5	1.15	0.72	5.6	--
									800	255	0.00	-490	0.059	90.1	1.15	0.84	7.0	--

Table B-1 (Cont'd.)

Single-Stage Acid Leaching Tests of Lignite Ash - Muffle Roast at 450°C

Sample Number	Roast Time, Hours	% L.O.I.	Ash % U_3O_8	Grind pH	Feed Mesh	Leaching Conditions			H_2SO_4, Lb/Ton		Terminal Conditions		Residue Assay	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay, g/l			
						Time, Hours	% Solids	Temp °C	Ash	Lignite	pH	Emf (mv)	% U_3O_8		U_3O_8	Mo	Fe	$SO_4^=$
185-21	2	47	0.18	3.9	-35 5/	6	40	75	500	265	0.75	-490	0.031	84.3	0.38	0.21	7.0	68
	↓	↓	↓		↓	↓	↓	↓	700	370	0.15	-515	0.018	91.5	0.41	0.36	13.4	92
	↓	↓	↓		↓	↓	↓	↓	800	425	0.0	-515	0.014	93.2	0.42	0.33	12.9	102
	↓	↓	0.19	-	-10	6	40	98	170	90	2.00	-375	0.057	66.0	0.34	0.001	0.3	--
	↓	↓	↓	-	↓	↓	↓	↓	275	145	1.55	-400	0.040	79.2	0.36	0.003	0.6	-
	↓	↓	↓	-	↓	↓	↓	↓	500	265	1.05	-450	0.020	90.0	0.43	0.032	2.8	--
	↓	↓	↓	-	↓	↓	↓	↓	600	315	0.70	-465	0.017	91.7	0.43	0.065	5.2	--
	↓	↓	↓	-	↓	↓	↓	↓	700	370	0.40	-485	0.014	93.3	0.44	0.094	6.6	--
	↓	↓	↓	-	↓	↓	↓	↓	800	425	0.20	-510	0.013	93.9	0.44	0.17	11.1	--
185-22	3	69	0.50	4.0	-35	6	40	75	300	90	1.9	-380	0.13	75.5	0.95	0.48	1.0	37
	↓	↓	↓		↓	↓	↓	↓	500	150	0.5	-450	0.080	85.3	1.07	0.61	3.2	68
	↓	↓	↓		↓	↓	↓	↓	700	215	0.0	-480	0.062	88.8	1.11	0.75	6.3	92
185-25	2	66	0.66	-	-35	6	40	75	600	205	2.1	-445	0.058	91.6	1.51	0.05	1.8	39
	↓	↓	↓	-	↓	↓	↓	↓	800	270	1.4	-485	0.035	95.2	1.59	0.46	8.6	68
	↓	↓	↓	-	↓	↓	↓	↓	1000	340	0.8	-515	0.022	97.1	1.60	0.87	14.4	90
	↓	↓	↓	-	↓	↓	↓	↓	1200	405	0.4	-520	0.016	98.0	1.62	1.25	19.1	111
	↓	↓	0.69	-	-10	6	40	98	600	205	2.65	-375	0.064	89.8	1.56	0.006	0.4	--
	↓	↓	↓	-	↓	↓	↓	↓	670	230	2.30	-440	0.050	93.0	1.60	0.031	1.2	--
	↓	↓	↓	-	↓	↓	↓	↓	800	270	1.55	-470	0.040	94.0	1.62	0.14	4.1	--
	↓	↓	↓	-	↓	↓	↓	↓	1000	340	0.90	-515	0.032	95.3	1.65	0.63	13.8	--
	↓	↓	↓	-	↓	↓	↓	↓	1200	405	0.40	-530	0.032	95.7	1.65	1.17	18.5	--
185-26	2	46	0.35	7.5	-35	6	40	75	900	485	1.1	-540	0.019	94.6	0.83	0.044	11.2	81
	↓	↓	↓	-	↓	↓	↓	↓	1200	650	0.5	-540	0.012	96.9	0.85	0.20	25.8	100
	↓	↓	↓	-	↓	↓	↓	↓	1500	810	0.0	-560	0.0062	98.6	0.86	0.32	39.8	137
	↓	↓	↓	-	↓	↓	↓	↓	1800	975	0.0	-560	0.0068	98.5	0.86	0.33	42.4	153

Table B-1 (Cont'd.)

Single-Stage Acid Leaching Tests of Lignite Ash - Muffle Roast at 450°C [6]

Sample Number	Roast Time, Hours	% L.O.I.	Ash % U_3O_8	Grind pH	% Mo Ext'n	H_2SO_4, Lb/Ton Ash	H_2SO_4, Lb/Ton Lignite	Terminal Conditions pH	Terminal Conditions Emf (mv)	Residue Assay % U_3O_8	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay, g/l U_3O_8	Mo	Fe	$SO_4^=$
185-27	2.5	32	0.45	4.5	13.7[7]	300	95	1.7	-410	0.12	75.7	0.85	0.41	1.2	38
						500	160	0.5	-460	0.068	86.3	0.97	0.57	3.3	69
						700	225	0.1	-490	0.051	89.9	1.01	0.68	6.2	92
185-28	2	46	0.16	4.2	Nil[5]	300	160	1.4	-435	0.042	75.3	0.30	0.008	6.9	38
						500	270	0.8	-490	0.028	84.4	0.34	0.16	8.5	67
						700	380	0.6	-510	0.013	93.1	0.37	0.27	9.1	90
185-29	2 1/4	51	1.18	8.2	36.0[7]	600	295	1.3	-435	0.18	86.4	2.5	0.053	2.7	70
						900	440	0.5	-475	0.13	90.7	2.7	0.20	6.4	109
						1200	590	0.05	-500	0.095	93.8	2.8	0.31	14.3	147
185-30	2	62	0.66	4.8	1.4[7]	325	125	1.4	-470	0.25	66.2	1.10	0.29	3.8	49
						650	245	0.7	-510	0.15	81.9	1.36	0.77	13.4	85
						975	370	0.3	-530	0.093	89.5	1.48	1.09	23.6	122
						1300	495	0.0	-545	0.063	93.5	1.53	1.29	31.5	154
185-31	2.5	57	0.82	10.6	14.3	550	235	1.8	-465	0.078	90.3	1.83	0.003	2.0	39
						875	375	1.2	-510	0.051	94.2	1.95	0.031	12.3	77
						1200	515	1.0	-530	0.028	97.1	1.97	0.11	25.5	120
						1530	660	0.6	-530	0.020	98.3	2.07	0.22	39.2	157
185-33	2.5	67	0.29	10.8	15.0	615	205	1.7	-460	0.061	80.1	0.58	0.012	2.5	61
						820	270	1.2	-490	0.028	91.7	0.67	0.11	11.1	93
						1020	335	1.2	-500	0.025	93.0	0.68	0.16	13.7	101
						1330	440	0.8	-520	0.022	94.2	0.68	0.38	22.8	131
185-34	2.5	60	0.42	4.4	1.0[7]	300	120	1.0	-490	0.12	73.8	0.77	0.16	4.6	42
						500	200	0.8	-515	0.087	81.7	0.86	0.30	10.9	63
						700	280	0.4	-530	0.063	87.5	0.92	0.46	18.5	89
						900	360	0.3	-540	0.050	90.6	0.95	0.54	25.0	110

Table B-1 (Cont'd.)

Single-Stage Acid Leaching Tests of Lignite Ash - Muffle Roast at 450°C

Sample Number	Roast Time, Hours	% L.O.I.	Ash % U_3O_8	Grind pH	% Mo Ext'n	H_2SO_4, Lb/Ton		Terminal Conditions		Residue Assay	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay, g/l			
						Ash	Lignite	pH	Emf (mv)	% U_3O_8		U_3O_8	Mo	Fe	$SO_4^=$
185-35	2	49	1.33	11.0	17.0	800	410	1.7	-460	0.097	93.1	3.10	0.022	1.7	69
	↓	↓	↓	↓	↓	1100	560	0.9	-515	0.047	96.9	3.22	0.098	6.5	101
						1400	715	0.4	-545	0.030	98.3	3.27	0.12	12.1	138
						1700	870	0.3	-545	0.021	98.8	3.28	0.38	13.7	163
185-37	2.5	67	1.04	7.8	21.3	500	165	2.4	-385	0.24	79.3	2.06	0.043	1.5	62
	↓	↓	↓	↓	↓	700	230	1.8	-420	0.19	84.3	2.20	0.064	2.6	82
						900	300	1.2	-450	0.15	88.0	2.29	0.12	3.9	107
						1100	365	0.8	-460	0.086	93.3	2.43	0.23	5.4	135
185-38	2	55	0.39	10.3	19.5	500	225	2.8	-370	0.083	78.4	0.77	0.002	0.7	42
	↓	↓	↓	↓	↓	750	340	1.4	-460	0.035	91.5	0.89	0.013	5.4	72
						1000	450	1.0	-480	0.019	95.8	0.94	0.065	11.4	101
						1250	560	0.5	-500	0.013	97.3	0.95	0.14	19.8	136
185-39	2.5 8/	60	0.88	10.5	N.D.	650	255	1.4	-470	0.072	92.4	2.03	0.06	3.9	65
	↓	↓	↓	↓	↓	1030	410	1.0	-505	0.025	97.6	2.15	0.35	14.9	102
185-40	2 8/	61	0.40	8.6	N.D.	350	135	1.4	-450	0.14	65.0	0.65	0.33	2.2	36
	↓	↓	↓	↓	↓	525	205	1.0	-485	0.10	78.5	0.78	0.67	7.9	64
						790	310	0.8	-495	0.066	85.7	0.86	1.0	15.0	99
185-41	2.5 8/	64	0.57	10.2	N.D.	560	200	1.5	-455	0.079	85.1	1.22	0.04	3.1	43
	↓	↓	↓	↓	↓	790	280	1.0	-500	0.080	85.7	1.23	0.29	11.6	71
						1070	380	0.5	-525	0.047	92.6	1.32	0.72	23.8	112
185-42	2	64	0.16	9.1	11.1	610	220	1.4	-455	0.040	75.9	0.30	0.01	4.3	59
	↓	↓	↓	↓	↓	840	305	1.0	-490	0.027	85.0	0.34	0.16	19.0	111
						1220	440	0.5	-515	0.017	91.5	0.37	0.28	27.3	135
185-43	3/4	14	0.105	6.4	11.0	300	260	1.0	-450	0.0068	94.0	0.25	0.03	5.7	37

Table B-1 (Cont'd.

Single-Stage Acid Leaching Tests of Lignite Ash - Muffle Roast at 450°C

Sample Number	Roast Time, Hours	% L.O.I.	Ash % U_3O_8	Grind pH	% Mo Ext'n	H_2SO_4, Lb/Ton		Terminal Conditions		Residue Assay	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay, g/1			
						Ash	Lignite	pH	Emf (mv)	% U_3O_8		U_3O_8	Mo	Fe	$SO_4^=$
185-44	2 1/4	59	1.21	6.9	4.5	575	235	0.5	-470	0.128	92.0	2.78	4.5	6.0	60
185-45	2	52	0.55	4.0	5.9 5/	440	210	0.5	-460	0.069	88.9	1.22	0.78	6.4	68
185-46	2 1/4	63	0.36	10.8	17.1	1630	600	0.5	-520	0.010	97.6	0.88	0.35	19.5	141
185-47	2	37	0.23	4.0	4.4	505	315	0.5	-480	0.021	92.0	0.53	0.59	5.7	75
185-48	2	66	0.30	10.5	10.4	970	330	1.6	-470	0.024	92.2	0.69	0.03	2.9	98
	↓	↓	↓	↓	↓	1210	410	1.0	-510	0.010	97.2	0.73	0.09	9.7	110
	↓	↓	↓	↓	↓	1420	480	0.6	-535	0.005	98.7	0.74	0.33	16.5	143

1/ All solution assays at a four to one solution to feed ratio.

2/ Sample 185-7 leached in water for four hours at 40 per cent solids prior to acid leach with 25 per cent Mo extraction included in pregnant solution assay.

3/ Minus 10-mesh leach feed for Samples 185-1 through 8.

4/ Samples 185-16 and 185-18 leached in water for four hours at 40 per cent solids prior to acid leach. Pregnant liquor assay includes water soluble molybdenum.

5/ About 25 per cent of the uranium was extracted from Sample 185-21 in the grinding water. Extraction and solution assay figures include this uranium.

6/ Samples 185-27 through 48: Minus 35-mesh ash leached six hours at 40 per cent solids and 75°C.

7/ Small amounts of U_3O_8 extracted in the grinding water.

8/ Feed to roaster contained 16-21 per cent moisture.

Table B-2

Acid Leaching of Samples 185-29 (A-I) Muffle Ash

Sample 185-29	Ash %	Calculated[1] Ash Analysis, %		Leaching Conditions[2] H_2SO_4, Lb/Ton		Terminal Conditions		Residue Assay	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay[3] Grams/Liter			
		U_3O_8	Mo	Ash	Raw Ore	pH	Emf (mv)	% U_3O_8		U_3O_8	Mo	Fe	$SO_4^=$
A	29	4.70	1.05	432	125	0.7	-410	0.79	85.5	9.13	1.90	1.5	61
B	48	0.66	0.33	835	410	0.65	-470	0.13	83.0	1.87	0.11	4.8	97
C	41	3.63	0.29	847	347	0.4	-480	0.44	90.1	8.27	0.19	5.3	101
D	38	0.86	0.25	1080	410	0.3	-500	0.051	95.0	1.85	0.17	8.1	113
E	80	0.016	0.087	1022	820	0.3	-585	0.0045	88.8	0.07	0.018	9.6	113
F	58	1.21	0.15	1504	875	0.3	-480	0.028	95.2	3.00	0.20	23.6	137
G	55	0.213	0.035	1197	660	0.5	-470	0.013	94.0	0.51	0.011	12.5	102
H	36	2.30	0.115	1270	458	0.3	-510	0.010	96.4	5.23	0.12	14.2	135
I	69	0.28	0.22	1051	725	0.45	-510	0.047	86.1	0.60	0.095	5.1	125

1/ Calculated from raw ore analysis and L.O.I.

2/ Minus 35-mesh feed leached for six hours at 75°C and 40 per cent solids

3/ 4:1 solution to feed ratio

Table B-3

Two-Stage Countercurrent Acid Leaching Tests of Lignite Muffle Ash

Sample 185-18 - 0.46% U_3O_8

Cycle Number	1	2	3	4	5	6	7	8	9	10	11
Neutralizing Leach											
Terminal pH		2.4	2.45	2.3	2.4	2.9	2.5	2.8	2.6	2.9	2.0
H_2SO_4, Lb/Ton		490	560	595	630	630	560	630	595	595	595
Pregnant Liquor, g/l											
U_3O_8		1.34	1.03	1.08	1.07	1.08	1.02	0.99	1.02	0.90	1.10
Mo		0.035	0.025	0.025	0.016	0.012	0.018	0.019	0.026	0.014	0.028
Fe^{+++}		1.45	1.14	1.05	0.64	0.52	0.72	0.81	0.90	0.47	0.76
Total Fe		1.98	1.59	1.42	0.97	0.88	1.16	1.22	1.29	0.81	1.11
$SO_4^=$		89.9	76.1	69.3			80.2	83.4	91.6	70.4	80.5
Strong Acid Leach											
Terminal pH	0.7	0.75	1.5	1.0	1.1	0.75	1.2	0.9	1.0	1.2	1.4
H_2SO_4, Lb/Ton	1400	420	420	350	420	490	490	490	490	490	490
Filtrate, g/l											
H_2SO_4		6.9	5.4	3.4	4.4	4.9	7.3	9.8	6.4	3.9	5.9
U_3O_8	0.94	0.36	0.22	0.11	0.19	0.16	0.12	0.18	0.16	0.28	0.16
Mo	0.15	0.089	0.074	0.026	0.039	0.10	0.039	0.091	0.059	0.062	0.074
Washed Residue, % U_3O_8	0.018	0.031	0.039	0.033	0.023	0.016	0.020	0.021	0.023	0.024	0.025
Total H_2SO_4, Lb/Ton	1400	910	980	945	1050	1120	1050	1120	1080	1085	1085
Lb H_2SO_4/Lb U_3O_8 Ext'd	158	106	115	110	120	127	119	127	124	124	124
% U_3O_8 Extraction	96.5	93.1	92.2	93.0	94.9	96.0	95.5	95.7	95.1	95.0	94.8

Notes: Grind: 3 minutes at 33 per cent solids, pH 10.7, 19 per cent Mo extraction.
Neutralizing Leach: 2.5 hours at 25 per cent solids and 90°C. Filter from 20 per cent solids with 0.2 pound Separan per ton. Pregnant liquor at 4.4:1 solution to feed ratio and pH about 2.5.
Strong Acid Leach: 5 hours at 25-28 per cent solids and 90°C. Filter using 0.1 pound Separan per ton to a 3:1 solution to feed ratio.
Average Soluble Loss: 1.3 per cent of dissolved U_3O_8.

Table B-4

Two-Stage Countercurrent Acid Leaching Tests of Lignite Muffle Ash

Sample 185-27 - 0.27% U_3O_8

Cycle Number	1	2	3	4	5	6	7	8	9	10	11
Neutralizing Leach											
Terminal pH	2.5	2.3	2.5	2.7	2.8		2.6	2.4	2.4	2.4	2.5
H_2SO_4	112	35	35	0	0	0	0	0	0	0	0
Pregnant Liquor, g/l											
U_3O_8	0.76	1.33	1.0	1.67	1.76	1.67	1.83	2.21	1.82	1.88	1.69
Mo	0.13	0.053	0.067	0.1	0.11	0.084	0.070	0.067	0.043	0.21	0.046
Fe^{+++}	0.13	0.35	0.06			0.53	0.30	0.44	0.20		
Total Fe	0.71	2.00	0.75			2.77	2.64	3.14	2.72	3.06	2.52
$SO_4^=$		60.1	38.5	71.0	74.5	77.4	74.2	94.1	81.6	103.9	69.7
Strong Acid Leach											
Terminal pH	0.5	0.5	0.0	below scale------							
H_2SO_4, Lb/Ton	280	280	315	350	365	365	365	365	365	365	
Filtrate, g/l											
H_2SO_4			24.5	28.4	31.3	42.2	40.1		49.0	39.5	
U_3O_8	0.86	0.87	1.78	1.31	0.99	1.10	0.83	0.73	1.13	1.13	
Mo	0.69	0.65			1.20	1.36	1.19	1.28	1.41	1.70	
Washed Residue, % U_3O_8	0.035	0.028	0.026	0.024	0.024	0.022	0.022	0.019	0.019	0.020	
Total H_2SO_4, Lb/Ton	392	315	350	350	365	365	365	365	365	365	
Lb H_2SO_4/Lb U_3O_8 Ext'd	82	64	71	71	74	73	73	72	73	73	
% U_3O_8 Extraction	88.4	90.4	91.1	91.4	91.7	92.3	92.9	93.3	92.7	93.1	

Notes: Grind: 3 minutes at 33 per cent solids at pH 6; 8 per cent Mo extraction.
Neutralizing Leach: 2.5 hours at 50 per cent solids and 90°C. Pregnant liquor at 1.5:1 solution to feed ratio and pH about 3.2.
Strong Acid Leach: 5 hours at 60 per cent solids and 90°C.
Average Soluble Loss: 1.2 per cent of dissolved U_3O_8.

Table B-5

Two-Stage Countercurrent Acid Leaching Tests of Lignite Muffle Ash

Sample 185-28 - 0.16% U_3O_8

Cycle Number	1	2	3	4	5	6	7	8	9	10	11	12
Neutralizing Leach												
Terminal pH	2.0	2.1	2.2	2.6	2.3	2.4	2.6	2.6	2.6	2.6	2.4	2.5
H_2SO_4, Lb/Ton	189											
Pregnant Liquor, g/l												
U_3O_8	0.34	0.83	0.93	0.77	0.78	0.78	0.76	0.79	0.81	0.61	0.44	0.79
Mo	0.010	0.011	0.014	0.008	0.011	0.006	0.083	0.009	0.009	0.005	0.042	0.011
Total Fe	2.02	2.0	2.60	1.75	2.47	1.38	1.71	1.85	1.69	1.80	6.05	2.30
$SO_4^=$	42.5	70.2	86.4		84.2	46.2	108.0	65.8	65.5	58.2	87.7	77.9
Strong Acid Leach												
Terminal pH	0.0	0.0	0.0	0.0	0.0	0.2	0.0	0.1	0.0	0.0	0.0	0.1
H_2SO_4, Lb/Ton	350	400	393	393	393	393	393	393	393	393	393	393
Filtrate, g/l												
H_2SO_4	13.7	14.7	7.3	12.7	15.7	19.6	16.7	26.0	25.5	67.0	47.5	25.0
U_3O_8	0.21	0.33	0.26	0.33	0.25	0.34	0.33	0.38	0.46	0.16	0.43	0.39
Mo	0.20	0.23	0.18	0.20	0.23	0.19	0.15	0.23	0.24	0.33	0.46	0.23
Washed Residue, % U_3O_8	0.0091	0.012	0.010	0.013	0.011	0.015	0.014	0.015		0.005	0.010	0.011
Total H_2SO_4, Lb/Ton	539	400	393	393	393	393	393	393	393	393	393	393
Lb H_2SO_4/Lb U_3O_8 Ext'd	177	135	130	134		135	134	135		128	130	131
% U_3O_8 Extraction	95.2	92.8	94.6	92.0		90.8	91.8	90.8		96.4	94.2	93.4

Notes: Grind: 3 minutes at 50 per cent solids with 13.2 pound $Ca(OH)_2$ per ton added to pH 11; 23 per cent Mo ext'n.
Neutralizing Leach: 2.5 hours at 40 per cent solids and 95°C. Pregnant liquor at 1.5:1 solution to feed ratio and pH about 2.7.
Strong Acid Leach: 5 hours at 50 per cent solids and 95°C.
Average Soluble Loss: 2.0 per cent of dissolved U_3O_8.

Table B-6

Two-Stage Countercurrent Acid Leaching Tests of Lignite Muffle Ash

Samples 185-30, 31, 33 Composite Ash - 0.60% U_3O_8

Cycle Number	1	2	3	4	5	6	7	8	9	10	11	12	13
Neutralizing Leach													
Terminal pH	2.3	2.8	3.0	2.5	2.6	2.5	2.6	2.2	2.2	2.3	2.3	2.4	2.0
H_2SO_4	252	35	70	140	0	0	0	0	0	0	0	0	0
Pregnant Liquor, g/l													
U_3O_8	1.7	1.8	2.58	2.96	2.50	2.74	2.24	2.74	2.23	3.41	2.37	1.81	2.96
Mo	0.007	0.007	0.020	0.026	0.015	0.025	0.011			0.058	0.047	0.011	0.077
Total Fe	0.65	0.58	1.40	2.47	2.00		1.70	4.13	7.81	4.46	3.46	1.50	4.64
$SO_4^=$	36.2	30.9	49.9	93.2	82.7		68.6	90.0			80.7	52.9	98.8
Strong Acid Leach													
Terminal pH	0.4	0.2	0.5	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
H_2SO_4, Lb/Ton	329	371	420	532	532	532	700	700	700	700	700	750	750
Filtrate, g/l													
H_2SO_4		8.3	8.8	17.1		18.6	68.8	34.8	32.4	38.2	35.3	53.0	60.8
U_3O_8	1.6	1.73	2.19	2.13		1.87	2.17	1.73	2.71	1.96	1.77	2.39	2.43
Mo	0.26	0.085	0.23	0.54		1.00		1.46	1.68	1.63	1.62	2.02	
Washed Residue, % U_3O_8	0.10	0.12	0.11	0.076	0.13	0.092	0.069	0.072	0.064	0.10	0.10	0.098	0.083
Total H_2SO_4, Lb/Ton	581	406	490	672	532	532	700	700	700	700	700	750	750
Lb H_2SO_4/Lb U_3O_8 Ext'd	58	43	50	63	56	52	65	66	66	70	70	75	72
% U_3O_8 Extraction	83.3	79.0	81.8	88.5	79.2	85.5	89.3	88.8	88.7	83.3	83.3	83.7	86.1

Notes: Grind: 3 minutes at 50 per cent solids at pH 9; 10 per cent Mo extraction.

Neutralizing Leach: 2.5 hours at 40 per cent solids and 95°C. Pregnant liquor at 1.5:1 solution to feed ratio and pH about 2.3.

Strong Acid Leach: 5 hours at 50 per cent solids and 95°C.

Average Soluble Loss: 2.8 per cent of dissolved U_3O_8.

Table B-7

Two-Stage Countercurrent Acid Leaching Tests of Lignite Muffle Ash

Sample 185-28 - 0.14% U_3O_8

Cycle Number	1	2	3	4	5	6	7	8	9	10	11	12	13
Neutralizing Leach													
Terminal pH	1.6	1.3	0.6	0.8	1.0	1.3	1.3	1.2	1.5	1.5	1.6	1.5	1.5
H_2SO_4, Lb/Ton	None----												
Pregnant Liquor, g/l													
U_3O_8	0.34	0.70	0.77	0.71	0.63	0.63	0.65	0.69	0.71	0.67		0.61	0.64
Mo	0.014	0.019	0.14	0.17	0.13	0.055			0.010	0.016	0.012	0.007	0.016
Total Fe	0.72	3.86	13.8	13.9	10.8	6.70	4.10	5.34	3.27	3.71	2.82	1.60	3.74
Al_2O_3	2.6		22.0	18.0			14.1		15.0	13.4	12.9	12.0	13.8
SiO_2					1.28	1.53		1.74	1.52	1.79	1.61		
$SO_4^{=}$						71.2	72.0		66.7	64.7	59.9	45.4	59.0
Strong Acid Leach													
Terminal pH	0.0	0.0	0.0	0.0	0.3	0.4		0.4	0.4	0.4	0.4		
H_2SO_4, Lb/Ton	440	440	393	360	300	290		270	270	270	270	270	270
Filtrate, g/l													
U_3O_8	0.43	0.24	0.26	0.21	0.23	0.19	0.22	0.22	0.22	0.21		0.23	0.22
Mo	0.35	0.59	0.66	0.62	0.59	0.51	0.36	0.40	0.36		0.28	0.37	0.38
Washed Residue, % U_3O_8	0.038	0.037	0.024	0.022	0.024	0.044	0.025	0.031	0.027	0.029	0.031	0.033	0.034
Total H_2SO_4, Lb/Ton	440	440	393	360	300	290		270	270	270	270	270	270
Lb H_2SO_4/Lb U_3O_8 Ext'd	206	203	163	147	125	143		120	115		120	120	121
% U_3O_8 Extraction	75.1	76.3	85.1	86.7	85.0	71.4	83.1	79.5	82.3		79.5	78.9	78.4

Notes: Grind: 3 minutes at 50 per cent solids with 13.2 pounds $Ca(OH)_2$ per ton added to pH 11.3; 23 per cent Mo ext'n.
Neutralizing Leach: 2.5 hours at 40 per cent solids and 70°C. Pregnant liquor at 1.7:1 solution to feed ratio and pH about 1.6.
Strong Acid Leach: 5 hours at 50 per cent solids and 70°C.
Average Soluble Loss: 0.8 per cent of dissolved U_3O_8.

Table B-8

Two-Stage Countercurrent Acid Leaching Tests of Lignite Muffle Ash

Sample 185-28 - 0.14% U_3O_8

Cycle Number	1	2	3	4	5	6	7	8	9	10	11	12	13
Neutralizing Leach													
Terminal pH	2.4	2.0	1.0	1.1	1.4	1.5	1.9	2.1	2.1	2.5	2.6	2.6	2.5
H_2SO_4, Lb/Ton	None----												
Pregnant Liquor, g/l													
U_3O_8	0.38	0.97	0.70	0.65	0.69	0.63	0.71	0.65	0.76	0.60		0.61	0.58
Mo	0.007	0.009	0.034	0.047	0.058	0.022		0.009	0.009	0.008	0.008	0.026	0.010
Total Fe	0.53	2.11	6.40	7.51	8.69	4.71		2.21	3.43	1.87	1.99	3.92	1.89
Al_2O_3	1.4		20.0	17.0	15.4		16.8	12.5	16.0	11.4	12.9	13.3	
SiO_2						1.45				1.49	1.22		
$SO_4^=$						65.2	63.4		60.5	48.3	52.4	58.4	45.0
Strong Acid Leach													
Terminal pH	0.0	0.0		0.0	0.4	0.5		0.6	0.6	0.6	0.6		
H_2SO_4, Lb/Ton	393	393	373	350	300	250		240	230	230	230	230	230
Filtrate, g/l													
U_3O_8	0.38	0.26	0.26	0.25	0.23		0.25	0.28	0.22	0.23			0.26
Mo	0.42	0.38	0.45	0.44	0.37		0.16	0.22	0.15		0.12	0.13	0.18
Washed Residue, % U_3O_8	0.039	0.025	0.023	0.024	0.022	0.024	0.045	0.028	0.030	0.045	0.033	0.034	0.035
Total H_2SO_4, Lb/Ton	393	393	373	350	300	250		240	230	230	230	230	230
Lb H_2SO_4/Lb U_3O_8 Ext'd	185	166	155	144	123	105		104	101		105	103	104
% U_3O_8 Extraction	74.7	83.5	85.1	85.1	85.8	84.0	69.0	81.3	80.0		77.7	78.3	77.7

Notes: Grind: 3 minutes at 50 per cent solids with 13.2 pounds $Ca(OH)_2$ per ton added to pH 11; 23 per cent Mo ext'n.
Neutralizing Leach: 2.5 hours at 40 per cent solids and 70°C. Pregnant liquor at 1.7:1 solution to feed ratio and pH about 2.6.
Strong Acid Leach: 5 hours at 50 per cent solids and 70°C.
Average Soluble Loss: 1.0 per cent of dissolved U_3O_8.

Table B-9

Thickening Test Data - Unleached Lignite Ash

Ground to Minus 35-Mesh

Sample Number	pH	Separan Lb/Ton Ash	F	D	R	A
185-29 Composite	8.1	None	6.3	1.0	2.6	2.7
		None	4.9	1.0	2.1	2.5
		None	3.6	1.0	1.13	3.3
		0.05	6.3	1.0	15.3	0.46
185-30,31,33 Composite	8.3	None	6.3	1.1	2.4	2.9
		None	4.9	1.1	1.9	2.7
		None	3.6	1.1	0.74	4.5
		0.05	6.3	1.2	17.3	0.39
		0.05	4.9	1.2	6.8	0.72
		0.05	3.6	1.2	2.3	1.4
185-34	4.1	None	6.3	1.4	1.13	5.8
		None	4.9	1.4	0.68	6.9
		None	3.6	1.4	0.31	9.5
		0.05	6.3	1.4	9.5	0.69
185-35	10.4	None	6.3	2.3	1.87	2.8
		None	4.9	2.3	1.17	3.0
185-37	7.5	None	6.3	1.3	1.56	4.3
		None	4.9	1.3	1.13	4.2
		None	3.6	1.3	0.59	5.2
		0.05	6.3	1.3	12.2	0.55
185-38	9.0	None	6.3	1.1	2.8	2.5
		None	4.9	1.1	1.8	2.8
		None	3.6	1.1	0.86	3.9

F = Feed dilution, pounds of solution per pound of dry solids.
D = Terminal dilution, pounds of solution per pound of dry solids.
R = Settling rate, feet per hour.
A = Thickener area, square feet per ton of solids per 24 hours.

Table B-10

Thickening Test Data - Acid Leached Lignite Ash[1]

Sample Number	H_2SO_4 Lb/Ton Ash	Pulp pH	Separan Lb/Ton Ash	F	D	R	A	O'flow Quality
185-29 Composite	1200	1.2	None	8.2	1.4	1.07	8.5	Clear
			0.05	8.2	1.4	1.4	6.5	Clear
			0.1	8.2	1.4	1.5	6.0	Clear
185-30,31,33 Composite	900	1.7	None	8.1	1.5	0.18	49	Clear
			0.05	8.1	1.5	0.31	35	Clear
			0.1	8.1	1.5	0.94	9.3	Clear
			0.2	8.1	1.5	3.0	2.9	Clear
			0.2	6.4	1.5	1.09	6.0	Clear
			0.2	4.7	1.5	0.39	10.9	Clear
185-34	700	1.6	None	7.6	1.4	0.35	23.6	Cloudy
			0.1	7.6	1.4	0.66	12.5	Cloudy
			0.2	7.6	1.4	5.4	1.5	Cloudy
			0.2	6.0	1.4	0.63	9.7	Cloudy
185-35	1100	1.8	None	No settling				
			0.8	7.9	3.5	0.41	14.3	Cloudy
185-37	1100	1.9	None	7.9	1.4	0.11	79	Cloudy
			0.7	8.8	1.4	0.34	29	Cloudy
185-38	1000	1.7	None	No settling				
			0.1	7.3	1.9	1.25	5.7	Cloudy
			0.2	7.3	1.9	3.9	1.8	Cloudy
			0.2	5.8	1.9	1.87	2.8	Cloudy
			0.2	4.2	1.9	0.70	4.4	Cloudy

[1] Minus 35-mesh ash leached at 40 per cent solids for six hours at 75°C.

F = Feed dilution, pounds of solution per pound of dry solids.
D = Terminal dilution, pounds of solution per pound of dry solids.
R = Settling rate, feet per hour.
A = Thickener area, square feet per ton of solids per 24 hours.

Table B-11

Filter Tests of Acid Leached Lignite Ash[1]

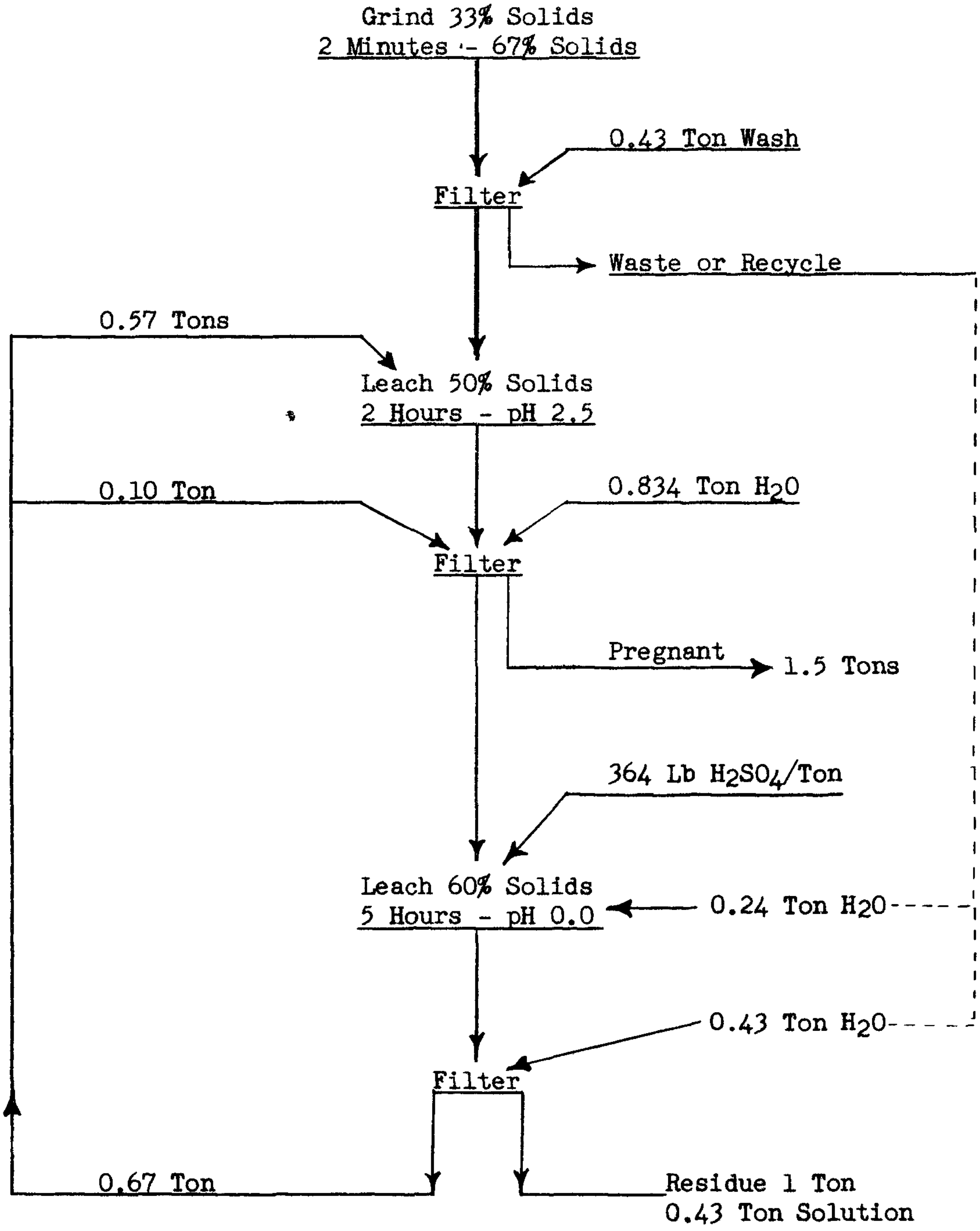
Sample Number	Ash % U_3O_8	Leach H_2SO_4 Lb/Ton Ash	Thicken from % Solids	Thicken to % Solids	Separan Lb/Ton Ash	Cycle Time, Minutes	Pickup Vacuum in Hg	Wash & Drain Vacuum in Hg	Cake Thick. Inches	% Moisture	Capacity Lb/Sq Ft/ Day	Soluble Loss %
185-16	0.50	650	25	50	0.25	1	8	8	1/2	46	2518	2.0[2]
						2	8	8	5/8	43	1960	6.0[2]
						4	8	8	1	43	1668	6.0[2]
185-19	0.50	1000	32	35	0.10	2	20	20	1/2	57	1240	18.7
						2	20	20	5/8	58	1410	26.0
185-22	0.50	700	32	45	0.15	2	10	10	3/16	40	715	--
						2	20	20	3/16	40	795	9.6
						4	20	20	5/16	36	580	--
185-27	0.45	700	22	46	0.10	2	21	21	1/2	44	1770	8.2
						2	10	10	7/16	46	1380	9.5
						2	10	21	9/16	43	1790	7.2
185-28	0.18	600	32	44	0.30	2	10	10	5/8	46	1800	14.7
						2	10	10	5.8	46	1740	13.2
185-29	1.15	1200	22	43	0.10	2	10	25	5/16	46	740	0.6
						4	10	25	7/16	34	535	0.4
185-30, 31,33	0.60	890	27	43	0.15	2	21	21	5/8	49	2160	20
						2	21	21	11/16	47	2600	16
185-34	0.42	700	20	46	0.20	2	10	25	5/16	46	835	0.6
						2	10	25	1/4	44	850	2.0
						4	10	25	3/8	43	530	0.6
185-35	1.33	1100	26	26	0.30	2	25	25	1/8	67	209	19.5
						4	25	25	5/32	64	135	9.6
						8	25	25	7/32	63	94	6.6
						4	10	25	1/8	63	113	2.6

Table B-11 (Continued)

Sample Number	Ash % U_3O_8	Leach H_2SO_4 Lb/Ton Ash	Thicken from % Solids	Thicken to % Solids	Separan Lb/Ton Ash	Cycle Time, Minutes	Pickup Vacuum in Hg	Wash & Drain Vacuum in Hg	Cake Thick. Inches	% Moisture	Capacity Lb/Sq Ft/ Day	Soluble Loss %
185-37	1.04	1100	21	39	0.30	4	10	25	1/8	48	147	0.7
						8	10	25	5/32	50	78	0.9
185-38	0.39	750	21	35	0.20	2	10	25	1/4	52	550	0.9
						2	10	25	1/4	51	600	1.9
						4	10	25	5/16	50	375	0.9

1/ Minus 35-mesh ash was leached for six hours at 750°C and 40 per cent solids. Filter tests were made at 25 per cent submergence. Soluble losses after one stage of thickening and one stage of filtration are expressed as per cent of total U_3O_8 extracted.

2/ Soluble loss as per cent of dissolved U_3O_8 in filter feed after two prior stages of thickening.



Two-Stage Leaching of Roasted Lignite
Sample 185-27

Figure B-1

APPENDIX C

Table C-1

Solvent Extraction of Northgate Leach Liquor

Sample 185-28

Feed Liquor	U_3O_8	Fe^{+++}	Fe^{++}	Mo	pH
Grams/Liter	0.43	9.16	1.42	0.3	0.80

Solvent: 0.092M Amine S-24, 2.5 per cent PDA in kerosene.

Organic to Aqueous Ratio Extraction	1:7
Number of Extraction Stages	4
Uranium Extraction	99%
Organic to Chloride Strip Ratio	5:1
Stages of Chloride Stripping	3
Organic to Carbonate Wash Ratio	15:1
Stages of Carbonate Wash	1
Product Grade	81.0% U_3O_8 0.3% Mo

Operation Characteristics - No emulsion in internal mixer-settlers. Emulsions in conventional mixer-settlers.

APPENDIX D

Table D-1

Sample 185-36

Flotation Test Data

CONDITIONS AND REAGENTS

POINT OF ADDITION	CONDITIONS TIME MINS.	% SOLIDS	PH	REAGENTS POUNDS PER TON No.2 Fuel Oil	Pine Oil								
		Calcine was leached with 520 pounds of H_2SO_4 per ton of feed for six hours at 50 per cent solids and 75°C. Terminal pH was 0.95. The residue was filtered, washed, and treated by flotation.											
Condition	2-3	67	3	1.5									
Ro Flot	5	20			As Rec'd								
Cl Flot	3	5											

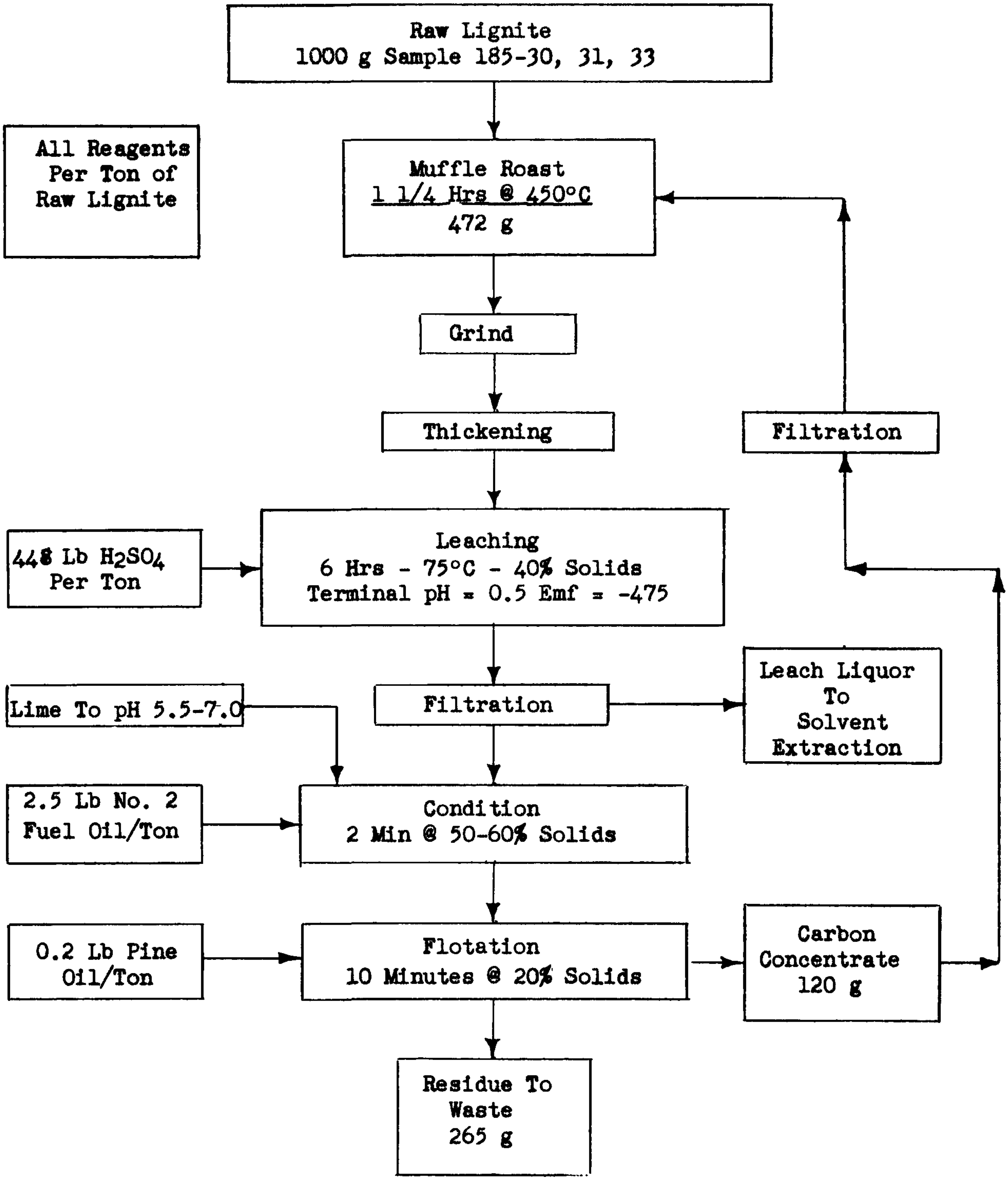
REMARKS:

METALLURGICAL RESULTS

PRODUCT	Weight or Volume	ASSAYS U_3O_8					% Dist'n U_3O_8	U_3O_8			
Calc. Feed	500.0	0.27					100.0				
Liquor	2100 ml	0.49 g/l					83.0				
Residue	461.2	0.050					17.0				
	% Weight						% Res.	% of Feed			
Residue	100.0	0.05					100.0	17.0			
Cl Conc	3.0	0.94				Flotation Leach	56.8	9.7			
Cl Tail	5.1	0.10					10.0	1.7			
Ro Tail	91.9	0.018					33.2	5.6			

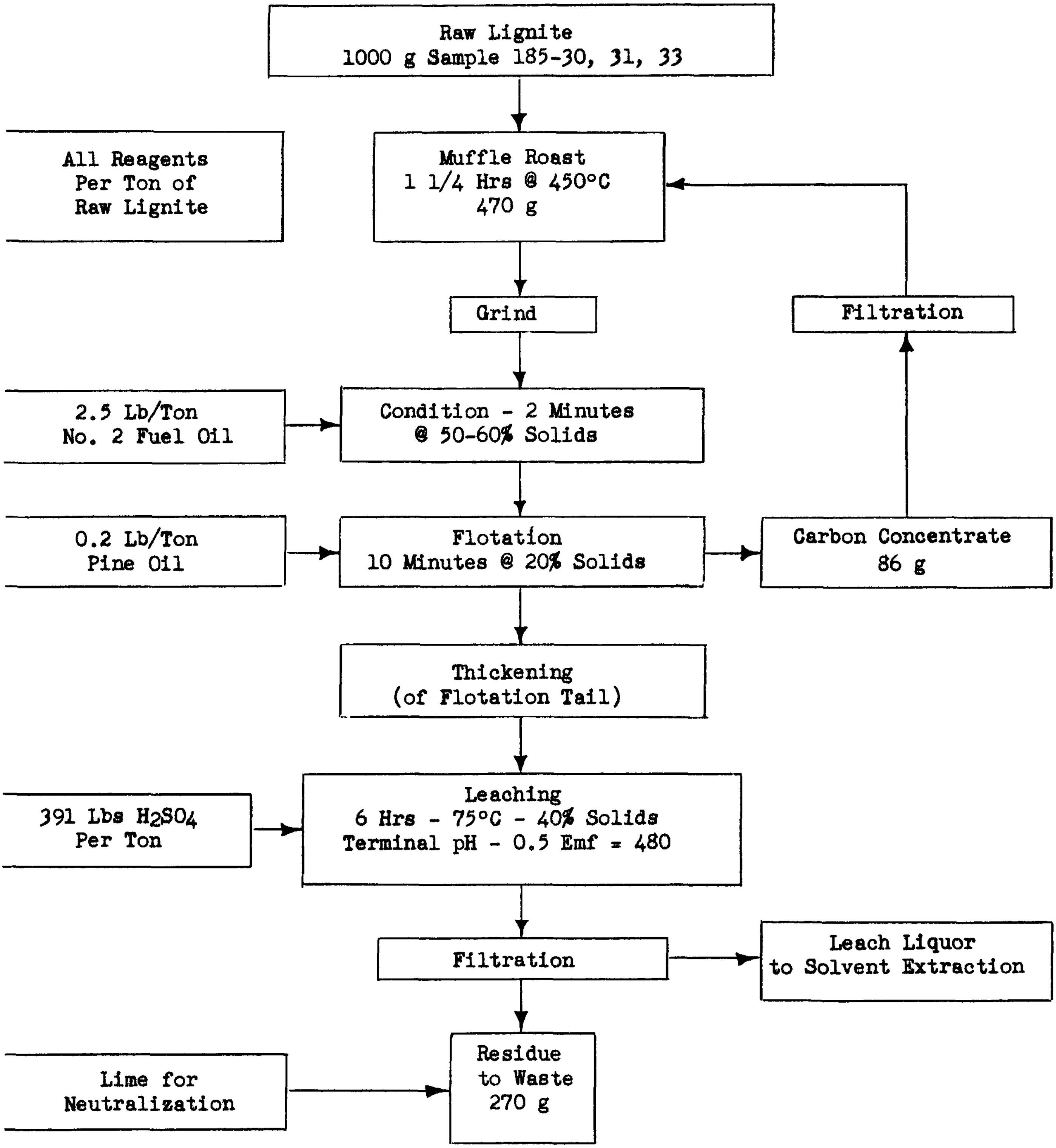
RATIO OF CONCENTRATION

REMARKS:



Leach - Flotation

Figure D-1



Flotation - Leach Flowsheet

Figure D-2

Table D-2

Summary of Batch Roasting, Leaching, and Flotation Test Results Where Roasting Time is the Principal Variable

Test Number	1	2	3	4	5	6	7
Feed:							
Weight, Grams	912	912	912	912	912	912	912
Calc. Assay, % U_3O_8	0.23	0.24	0.23	0.23	0.22	0.21	0.24
Roasting:							
Time, Hours	1/2	3/4	1	1 1/4	1 1/2	2	2 1/2
Time, % Dead Roast Time	20	30	40	50	60	80	100
Calcine Assay, % U_3O_8	0.42	0.456	0.491	0.522	0.537	0.526	0.60
Leaching:							
Terminal pH	0.55	0.50	0.55	0.60	0.50	0.60	0.50
Terminal emf (mv)	-425	-440	-460	-460	-485	-510	-535
H_2SO_4 Cons. Lb/ Ton Raw Lignite	407	421	418	431	453	461	480
Filtrate:							
Volume, ml	1180	1160	1150	1140	1470	1420	1370
Assay, g U_3O_8/l	1.18	1.26	1.40	1.51	1.23	1.24	1.48
% U_3O_8 Ext'n	65.0	67.5	77.0	81.7	90.5	91.9	93.5
Residue:							
Weight, Grams	438	404	336	318	273.4	279.2	274.5
Weight, % Raw Lignite Feed	48.0	44.3	36.8	34.9	30.0	30.6	30.1
Assay, % U_3O_8	0.274	0.174	0.143	0.121	0.069	0.056	0.051
U_3O_8 Loss, % U_3O_8 in Feed	35.0	32.5	23.0	18.3	9.5	8.1	6.5
Flotation:							
Reagent Consumption (Lb/Ton Raw Lignite):							
Lime	11.6	9.9	6.6	7.3	6.3	5.9	
No.2 Fuel Oil	25.0	10.0	5.0	2.5	1.5	1.0	
Pine Oil	0.58	0.33	0.25	0.2	0.15	0.15	
Flotation pH	5.5	6.5	5.8	5.7	5.3	7.0	
Flotation Concentrate:							
Weight, Grams	300	220	138	100	33.4	11.2	
Weight, % Raw Lignite Feed	32.9	24.1	15.1	11.0	3.7	1.2	
Assay, % U_3O_8	0.246	0.293	0.307	0.300	0.236	0.226	
U_3O_8 Recovery, % Lignite Feed	34.5	29.8	20.3	14.2	4.0	1.3	
U_3O_8 Recovery, % Residue	98.7	91.6	88.0	77.9	41.8	16.0	
Flotation Tail:							
Weight, Grams	138	184	198	218	240	268	
Weight, % Raw Lignite Feed	15.1	20.2	21.7	23.9	26.3	29.4	
Assay, % U_3O_8	0.007	0.032	0.030	0.039	0.046	0.049	
U_3O_8 Loss, % Lignite Feed	0.5	2.7	2.7	4.1	5.5	6.8	
U_3O_8 Loss, % Residue	1.3	8.4	12.0	22.1	58.2	84.0	

Table D-3
Summary of Batch Roasting, Flotation, and Leaching Test Results Where Roasting Time is the Principal Variable

Test Number	1	2	3	4	5
Feed:					
Weight, Grams	1000	1000	1000	1000	1000
Calc.Assay,% U_3O_8	0.205	0.216	0.222	0.219	0.226
Roasting:					
Time, Hours	1/2	1	1 1/4	1 1/2	2
Time, % Dead Roast Time	20	40	50	60	80
Calcine Weight, % Raw Lignite Feed	52.6	45.0	42.6	40.5	38.9
Calcine Assay,% U_3O_8	0.39	0.48	0.52	0.54	0.58
Flotation:					
Reagent Consumption (Lb/Ton Raw Lignite)					
No. 2 Fuel Oil	50	10	5	3	2
Pine Oil	--	--	0.5	0.3	0.25
Flotation pH	8.5	8.8	8.7	9.2	9.1
Flotation Concentrate:					
Weight, % Raw Lignite Feed	33.3	16.1	9.4	5.6	1.6
Assay, % U_3O_8	0.41	0.47	0.47	0.49	0.48
U_3O_8 Recovery, % in Lignite Feed	65.7	35.5	19.9	12.2	3.4
Flotation Tail:					
Weight, % Raw Lignite Feed	19.3	28.9	33.2	34.9	37.3
Assay, % U_3O_8	0.39	0.48	0.53	0.56	0.60
U_3O_8 Recovery, % in Lignite Feed	34.3	64.5	80.1	87.8	96.6
Leaching:					
Terminal pH	0.6	0.5	0.5	0.5	0.5
H_2SO_4 Cons.Lb/Ton of Raw Lignite	186	262	350	291	307
Filtrate:					
Volume, ml	1065	1170	1660	1400	1590
Assay, g U_3O_8/l	0.65	1.0	0.95	1.20	1.20
U_3O_8 Ext'n, % in Raw Lignite Feed	31.2	57.9	73.5	78.2	85.9
Residue:					
Weight, % of Raw Lignite Feed	17.0	23.2	25.9	28.6	29.9
Assay, % U_3O_8	0.040	0.058	0.055	0.072	0.079
U_3O_8 Loss, % U_3O_8 in Rougher Tail	9.0	10.3	8.3	10.9	11.0
U_3O_8 Loss, % U_3O_8 in Raw Lignite Feed	3.1	6.6	6.6	9.6	10.6

Table D-4
Results of Locked Test Using Leach - Flotation Flowsheet

		Filtrate From Flotation Product			Flotation Concentrate			Leach Liquor			Flotation Tail		
Number	Calcine Weight, Grams	Volume ml	Assay U_3O_8, g/l	Content g U_3O_8	Wt. Grams	%U_3O_8	Content Grams	Volume ml	Assay U_3O_8, g/l	Content Grams	Wt. Grams	Assay %U_3O_8	Content Grams
1	406.4	2600	0.0017	0.0044				1130	1.5	1.69	258	0.045	0.11
2	449.5	2600	0.0015	0.0039				1180	1.69	1.99	252	0.042	0.10
3	472.1	2660	0.0002	0.0005				1200	1.68	2.01	266	0.048	0.12
4	472.5	3140	0.0001	0.0003	120	0.261	0.3132	1110	1.75	1.94	248	0.039	0.09
Total				0.0091			0.3132			7.63			0.42

Product	Content g U_3O_8	Distribution % U_3O_8	Weight, Grams	Assay % U_3O_8
Leach Liquor	7.63	91.1[1]		
Residue (Flot. Tail)	0.42	5.0	1,024	0.041
Flotation - Soluble Loss	0.0091	0.1		
Carbon Concentrate - Circulating Load	0.3132	3.8		
Calculated Raw Lignite Feed	8.3723	100.0	4,000	0.029

[1] Assuming a 91.1 per cent U_3O_8 extraction from the circulating load of carbon concentrate upon ultimate roasting the U_3O_8 extraction is 91.1 per cent plus 3.5 per cent or 94.6 per cent.

Table D-5
Results of Locked Test Using Flotation - Leach Flowsheet

		Filtrate From Flotation Product			Flotation Concentrate			Leach Liquor			Residue		
Charge Number	Calcine Weight, Grams	Volume ml	Assay U_3O_8, g/l	Content g U_3O_8	Wt. Grams	Assay %U_3O_8	Content Grams	Volume ml	Assay U_3O_8 g/l	Content Grams	Wt. Grams	Assay %U_3O_8	Content Grams
1	391.1	--	--	--	--	--	--	1140	1.5	1.71	242.0	0.046	0.11
2	429.6	540	0.0011	0.0005	--	--	--	1260	1.54	1.94	276.0	0.048	0.13
3	436.3	2690	0.0022	0.0059	--	--	--	980	1.83	1.79	262.0	0.062	0.16
4	470.0	2615	0.0009	0.0023	85.6	0.507	0.434	1000	2.00	2.00	300.0	0.080	0.24
Total				0.0087			0.434			7.44			0.64

Product	Content g U_3O_8	Distribution % U_3O_8	Weight Grams	Assays % U_3O_8
Leach Liquor	7.44	87.3[1]		
Residue	0.64	7.5	1,079	0.0695
Flotation - Soluble Loss	0.0087	0.1		
Carbon Concentrate - Circulating Load	0.434	5.1		
Calculated Raw Lignite Feed	8.5227	100.0	4,000	0.213

1/ Assuming a 87.3 per cent U_3O_8 extraction from the calculated load of carbon concentrate; Upon ultimate roasting the U_3O_8 extraction is 87.3 per cent plus 4.5 per cent or 91.8 per cent.

Table D-6

A Comparison of Leaching on Various Dead Roasted Lignite Ore with Flotation-Leach and Leach-Flotation on Ores Roasted for 50 Per Cent of Dead Burned Time

	Sample 185-27			Sample 185-39			Sample 185-40			Sample 185-43			Sample 185-44		
	L	L-F	F-L	L	L-F	F-L	L	L-F	F-L	L	L-F	F-L	L	L-F	F-L
Feed:															
Weight, Grams	625.0	983.0	994.2	873.0	889.6	901.9	882.0	925.2	910.9	500.0	953.0	476.5	1000.0	895.6	898.5
Calculated Assay, % U_3O_8	0.148	0.135	0.144	0.34	0.37	0.33	0.171	0.174	0.164	0.093	0.102	0.090	0.46	0.521	0.518
Roasting:															
Time, Hours	2 1/2	1 1/4	1 1/4	2 1/2	1 1/8	1 1/8	2.0	7/8	7/8	3/4	No Roasting	No Roasting	2 1/4	1 1/8	1 1/8
Time, % Dead Roast Time	100	50	50	100	50	50	100	50	50	100			100	50	50
Calcine Weight, % Raw Lignite Feed	32.0	48.2	43.0	39.5	44.3	42.3	39.0	47.7	48.9	86.0			41.1	51.8	51.0
Calcine Assay, % U_3O_8	0.464	0.279	0.336	0.861	0.845	0.855	0.439	0.365	0.336	0.109			1.11	1.004	0.995
Leaching:															
Terminal pH	0.5	0.5	0.6	1.0	1.15	1.1	0.8	1.0	0.8	1.0	0.85	0.90	0.5	0.55	0.5
Terminal Emf (mv)	-460	-340	-355	-505	-440	-460	-495	-415	-440	-450	-460	-470	-470	-420	-430
H_2SO_4 Consumption, Lb/Ton Raw Lignite	160	160	123	407	413	388	310	234.5	235.5	260	220	183.4	235	263	233
Filtrate:															
Volume, ml	1116	1100	1115	1400	1170	1380	887	1270	1180	1780	1300	1670	2300	925	1330
Assay, g U_3O_8/l	0.72	0.719	0.650	1.20	2.80	1.97	0.86	0.833	0.717	0.25	0.306	0.209	1.81	3.64	2.36
% U_3O_8 Extraction	86.7	63.8	50.6	97.6	83.9	83.2	86.9	65.6	56.5	94.2	82.1	81.2	91.3	72.2	67.4
Residue:															
Weight, Grams	181.5	426.5	385.5	166.0	330.5	307.5	174.0	389.5	402.5	400.0	451.5	437.5	310.0	430.0	432.5
Weight, % Raw Lignite Feed	29.0	43.4	38.7	32.8	37.2	34.1	33.9	42.1	44.2	80.0	94.7	91.8	31.0	48.0	48.1
Assay, % U_3O_8	0.068	0.112	0.184	0.025	0.162	0.178	0.066	0.142	0.161	0.0068	0.019	0.018	0.128	0.302	0.350
U_3O_8 Loss, % U_3O_8 in Feed	13.3	36.2	49.4	2.4	16.1	16.8	13.1	34.4	43.5	5.8	17.9	18.8	8.7	27.8	32.5
Flotation:															
Reagent Consumption, Lb/Ton Raw Lignite															
Lime		1.5			3.4			3.2			22.2			4.2	
No. 2 Fuel Oil		2.5	2.5		2.5	2.5		2.5	2.5		1.25	2.5		2.5	2.5
Pine Oil		0.2	0.25		0.2	0.2		0.2	0.2		0.05	0.15		0.2	0.2
Flotation pH		7.4	6.6		7.2	10.7		7.2	8.5		4.6	6.5		4.1	7.8
Flotation Concentrate:															
Weight, Grams		140.5	143.5		92.0	62.0		141.5	98.0		43.5	58.0		112.0	98.5
Weight, % Raw Lignite Feed		14.3	14.2		10.3	6.8		15.3	10.7		9.1	12.2		12.5	10.9
Assay, % U_3O_8		0.171	0.329		0.519	0.754		0.285	0.334		0.023	0.086		0.675	1.16
U_3O_8 Recovery, % Lignite Feed		18.1	32.9		14.3	14.3		25.0	21.8		2.1	11.4		16.2	22.4
U_3O_8 Recovery, % Residue		50.0	66.6		88.8	85.1		72.7	50.1		11.7	60.6		58.3	68.9
Flotation Tail:															
Weight, Grams		286.0	242.0		238.5	245.5		248.0	304.5		408.0	379.5		318.0	334.0
Weight, % Raw Lignite Feed		29.1	24.3		26.8	27.2		26.8	33.4		85.6	79.6		35.5	37.2
Assay, % U_3O_8		0.084	0.098		0.025	0.033		0.061	0.106		0.019	0.0086		0.170	0.141
U_3O_8 Loss, % Lignite Feed		18.1	16.5		1.8	2.5		9.4	21.6		15.8	7.4		11.6	10.1
U_3O_8 Loss, % Residue		50.0	33.4		11.2	14.9		27.3	49.9		88.3	39.4		41.7	31.1
% U_3O_8 Recovery in Circuit Based on Tail Loss	86.7	81.9	83.5	97.6	98.2	97.5	86.9	90.6	78.4	94.2	84.2	92.6	91.3	88.4	89.9
% Ignition of Total Hydrocarbon	100	75	84	100	92	96	100	86	84	100	0	0	100	82	82

APPENDIX E

Table E-1

Conditions and Results of Acid Leaching Tests on Raw and Roasted Dakota Lignite Ore - Sample 185-4

Roasting Conditions		Leaching Conditions						Terminal Conditions		Residue Assay	% U_3O_8 Ext'n
					Lb/Ton of Raw Ore						
Time, Hours	Temp °C	Time, Hours	Temp °C	% Solids	H_2SO_4	MnO_2	Fe	pH	Emf(mv)	% U_3O_8	
Winchester Retort Roast											
Effect of Varying Roasting Temperature, Leaching Temperature, and Acid Additions											
2	200	6	80	40	200	--	--	1.15	-380	0.17	59.6
2	200	6	80	40	400	--	--	0.5	-415	0.082	81.5
2	200	6	80	40	600	--	--	0.1	-430	0.055	87.3
2	200	6	80	40	800	--	--	0.0	-435	0.047	89.7
1	300	6	80	40	361	--	--	0.9	-360	0.17	64.3
1	300	6	80	40	722	--	--	0.1	-380	0.12	76.4
1	300	6	40	40	361	--	--	0.7	-390	0.18	63.5
1	300	6	40	40	722	--	--	0.2	-410	0.13	73.2
2	330	6	40	40	370	--	--	0.4	-430	0.074	82.2
2	330	6	40	40	555	--	--	0.0	-440	0.050	89.0
2	330	6	40	40	740	--	--	0.0	-445	0.047	91.7
1	400	6	80	40	555	--	--	0.25	-355	0.064	87.8
1	400	6	80	40	740	--	--	0.05	-365	0.053	89.6
1	400	6	40	40	555	--	--	0.15	-380	0.062	86.0
1	450	6	40	40	555	--	--	0.25	-340	0.12	74.8
Effect of Adding MnO_2 and Metallic Iron											
1	400	6	40	40	555	--	--	0.15	-380	0.062	86.0
1	400	6	40	40	555	20	--	0.15	-415	0.062	86.0
1	400	6	40	40	555	--	--	0.15	-380	0.060	86.7
1	400	6	40	40	555	20	2	0.15	-415	0.060	87.3
1	400	6	40	40	555	20	8	0.25	-380	0.072	85.0
1	400	6	40	40	555	40	8	0.15	-425	0.071	84.5

Table E-1 (Continued)

Conditions and Results of Acid Leaching Tests on Raw and Roasted Dakota Lignite Ore - Sample 185-4

Roasting Conditions		Leaching Conditions						Terminal Conditions		Residue Assay	
					Lb/Ton of Raw Ore						
Time, Hours	Temp °C	Time, Hours	Temp °C	% Solids	H_2SO_4	MnO_2	Fe	pH	Emf(mv)	% U_3O_8	% U_3O_8 Ext'n
Effect of Varying Leaching Temperature											
1	400	6	Amb.	40	555	20	8	0.2	-390	0.075	83.0
1	400	6	40	40	555	20	8	0.25	-380	0.072	85.0
1	400	6	80	40	555	20	8	0.3	-350	0.040	91.2
U.S.B.M. Retort Roast Effect of Varying Roasting and Leaching Temperature											
1	200	6	40	25	600	--	--	0.5	-490	0.072	82.8
1	300	6	40	25	600	--	--	0.5	-420	0.065	85.0
1	350	6	40	25	600	--	--	0.45	-440	0.058	87.1
1	400	6	40	25	555	--	--	0.6	-400	0.056	88.0
1	450	6	40	25	555	--	--	0.4	-420	0.15	64.7
1	200	6	80	25	600	20.0	--	0.5	-430	0.054	88.0
1	300	6	80	25	600	20.0	--	0.5	-390	0.058	85.7
1	400	6	80	25	555	18.5	--	0.6	-360	0.066	86.2
1	500	6	80	25	555	18.5	--	0.5	-360	0.24	45.8
1	600	6	80	25	555	18.5	--	0.95	-355	0.34	11.1

APPENDIX F

Table F-1

Screen Analyses - Ground Raw Lignite

	Sample 185-19	Sample 185-20	Sample 185-21
Grind, Minutes[1]	2	15	2

	Weight, Per Cent		
Tyler Mesh	Sample 185-19	Sample 185-20	Sample 185-21
+35	0.2	0.5	0.8
-35 +65	6.8	35.7	14.2
-65 +100	11.3	12.8	13.5
-100 +200	19.0	12.0	16.3
-200	62.7	39.0	55.2

1/ Five hundred grams of minus 10-mesh lignite ground at 50 per cent solids in a rod mill.

Table F-2

Single-Stage Acid Leaching of Raw Lignite

				Leaching Conditions				Terminal Conditions		Residue		Diluted Pregnant Liquor Assay, g/1			
Sample Number	% U_3O_8	Grind pH	Feed Mesh	Time, Hours	% Solids	Temp °C	H_2SO_4 Lb/Ton	pH	Emf (mv)	Assay % U_3O_8	% U_3O_8 Ext'n	U_3O_8	Mo	Fe	$SO_4^=$
185-1	0.275	-	-10	16	50	Amb	340	-	--	--	60	--	--	--	--
185-4	0.37	-	-10	6	50	35	258	0.7	--	0.087	78				
Effect of Varying Acid Concentration and Leaching Temperature															
				6	40	40	200	1.0	-470	0.16	58.2				
							400	0.4	-505	0.12	69.5				
							600	-	-525	0.063	84.0				
							800	-	-530	0.055	86.6				
				6	40	80	200	1.2	-410	0.13	64.6				
							400	0.5	-445	0.063	83.6				
							800	0.1	-465	0.039	89.4				
Effect of Varying Leaching Time															
				1/2	40	80	800	0.3	--	0.054	85.9				
				2				0.3	-480	0.050	88.0				
				4				0.2	-475	0.050	88.5				
				6				0.1	-465	0.039	89.4				
				16				0.2	-460	0.034	92.1				
185-16	0.35	-1/	-10	6	40	75	356	0.4	--	0.099	71.5				
				16			370	0.2	--	0.078	80.5				
				24			370	0.4	--	0.080	79.5				
				58			370	0.5	--	0.069	82.9				
				96			396	0.6	--	0.052	86.5				
185-18	0.22	-2/	-10	6	33	75	500	1.9	-435	0.026	88.6				
							750	1.2	-445	0.008	96.8				
							1000	0.9	-440	0.012	95.9	0.48	0.12	7.9	101

Table F-2 (Continued)

Single-Stage Acid Leaching of Raw Lignite

				Leaching Conditions				Terminal Conditions		Residue Assay		Diluted Pregnant Liquor Assay, g/l			
Sample Number	% U_3O_8	Grind pH	Feed Mesh	Time, Hours	% Solids	Temp °C	H_2SO_4 Lb/Ton	pH	Emf (mv)	% U_3O_8	% U_3O_8 Ext'n	U_3O_8	Mo	Fe	$SO_4^=$
185-18	0.22	--[2]	-10	6	40	75	990	0.4	--	0.043	81.9				
				16			990	0.4		0.044	81.3				
				24			990	0.4		0.033	85.9				
				48			990	0.2		0.026	90.4				
				96			915	0.4		0.023	90.5				
				Effect of Varying Acid Concentration											
185-19	0.28	5.5	-35	6	40	75	200	2.0	-380	0.16	43.2	0.31	0.01	0.64	17
				6			400	0.8	-455	0.033	88.3	0.63	0.19	4.9	41
				6			600	0.2	-465	0.016	94.6	0.67	0.48	8.7	64
				6			800	0.1	-470	0.015	95.4	0.68	0.80	12.2	90
				Effect of Varying Leaching Temperature											
				6	40	25	500	0.4	-570	0.025	92.4	0.66	0.25	6.4	54
				6		50	500	0.5	-510	0.019	94.1	0.67	0.36	7.5	52
				6		75	500	0.5	-475	0.014	95.8	0.68	0.41	8.0	55
				6		100	500	0.7	-425	0.018	94.7	0.67	0.30	7.2	53
				Miscellaneous Leaching Tests											
				18	40	75	600	0.6	-465	0.017	94.9	0.68	0.53	9.2	67
				18[3]		Amb	328	1.0	-510	0.11	72.0	0.49	0.08	2.9	28
				24		Amb	500	0.4	-560	0.031	89.8	0.63	0.32	6.8	74
				Effect of Varying Acid Concentration											
185-20	0.16	4.8	-35	6	40	75	150	1.0	-310	0.051	69.0	0.27[7]	0.003	3.4	25
							300	0.2	-330	0.027	83.6	0.33	0.004	4.1	43
							450	-	-335	0.019	88.5	0.35	0.006	4.2	60
							600	-	-330	0.016	90.4	0.37	0.009	4.3	80

Table F-2 (Continued)

Single-Stage Acid Leaching of Raw Lignite

				Leaching Conditions				Terminal Conditions		Residue Assay		Diluted Pregnant Liquor Assay, g/l			
Sample Number	% U_3O_8	Grind pH	Feed Mesh	Time, Hours	% Solids	Temp °C	H_2SO_4 Lb/Ton	pH	Emf (mv)	% U_3O_8	% U_3O_8 Ext'n	U_3O_8	Mo	Fe	$SO_4^=$
				Miscellaneous Leaching Tests											
185-20	0.16	4.8	-35	18	40	75	600	0.2	-310	0.018	89.3	0.38	0.006	4.5	74
				18[3]		Amb	145	0.8	-390	0.058	64.2	0.26	0.003	1.8	15
				24		Amb	300	0.1	-430	0.031	82.3	0.33	0.007	2.4	42
				6[4]		75	300	0.3	-365	0.033	80.2	0.32	0.006	3.9	44
				6[5]		75	300	0.4	-420	0.031	81.1	0.33	0.008	3.6	--
				6[6]		75	300	0.2	-415	0.030	81.9	0.33	0.011	4.1	--
185-21	0.10	3.8	-35	6	40	75	200	0.6	-430	0.022	79.4	0.20	0.07	4.2	31
							300	0.2	-450	0.015	86.2	0.22	0.12	5.9	44
							400	0.0	-465	0.011	90.6	0.23	0.16	8.0	60
185-22	0.15	4.8	-35	6	40	75	100	1.7	-270	0.077	49.4	0.16[7]	0.005	2.2	17
							200	0.8	-335	0.040	74.5	0.28	0.003	3.3	30
							300	0.4	-345	0.030	81.1	0.29	0.003	3.7	41
							400	0.1	-340	0.024	85.2	0.32	0.007	3.7	54
				24[3]		Amb	500	0.1	--	0.023	85.5	0.33	0.006	2.7	--
185-25	0.23	-	-35	6	40	75	200	1.7	-365	0.15	36.0	0.19[7]	0.03	1.0	16
							300	0.8	-435	0.053	78.5	0.47	0.11	2.8	30
							400	0.4	-445	0.039	85.3	0.50	0.18	4.9	42
							500	0.1	-450	0.035	86.9	0.52	0.22	6.0	55
185-26	0.18	7.5	-35	6	40	75	400	2.0	-400	0.089	50.0	0.22	0.001	1.7	21
							500	1.4	-445	0.036	75.7	0.33	0.011	7.2	34
							600	1.2	-450	0.037	81.5	0.36	0.026	10.6	43
							700	0.8	-455	0.017	91.1	0.40	0.072	16.9	64
							800	0.4	-475	0.015	91.3	0.40	0.16	19.5	69
							900	0.1	-480	0.009	94.6	0.42	0.25	23.0	91
							1000	-	-480	0.009	94.9	0.42	0.28	23.2	98

Table F-2 (Continued)

Single-Stage Acid Leaching of Raw Lignite

Sample Number	% U_3O_8	Grind pH	Feed Mesh	Leaching Conditions Time, Hours	% Solids	Temp °C	H_2SO_4 Lb/Ton	Terminal Conditions pH	Emf (mv)	Residue Assay % U_3O_8	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay, g/l U_3O_8	Mo	Fe	$SO_4^=$
185-27	0.145	4.5	-35	6	40	75	200	0.6	-290	0.050	67.6	0.25 7/	0.003 8/	3.7	28
							300	0.2	-310	0.032	79.4	0.29	0.004	3.6	41
							400	0.0	-350	0.019	87.9	0.32	0.006	4.2	55
185-28	0.09	4.3	-35	6	40	75	200	0.8	-450	0.019	80.0	0.17	0.038	3.2	28
							300	0.4	-465	0.013	86.9	0.18	--	5.0	40
							400	0.2	-475	0.010	89.9	0.19	0.093	6.4	52
185-29	0.61	-	-35	6	40	75	350	1.4	-380	0.18	70.8	1.08	0.008	2.8	38
							550	0.6	-410	0.065	90.5	1.38	0.027	5.2	63
							750	0.3	-420	0.045	93.5	1.43	0.050	7.8	88
185-30	0.27	4.2	-35	6	40	75	150	1.9	-380	0.17	40.7	0.27	0.022	1.5	18
							300	1.0	-440	0.047	85.0	0.57	0.13	5.5	41
							450	0.4	-455	0.032	90.1	0.60	0.23	8.8	58
							600	0.1	-460	0.019	94.7	0.63	0.30	11.4	79
185-31	0.36	5.6	-35	6	40	75	300	1.8	-405	0.19	48.8	0.45	0.001	2.1	23
							450	1.1	-440	0.087	76.6	0.69	0.011	6.5	41
							600	0.6	-460	0.039	89.1	0.80	0.045	12.6	64
							750	0.2	-465	0.034	91.2	0.83	0.068	16.3	82
185-33	0.10	5.0	-35	6	40	75	250	1.6	-390	0.046	57.3	0.14	0.010	1.7	25
							350	0.8	-425	0.027	76.7	0.20	0.032	3.5	41
							450	0.5	-445	0.018	85.0	0.22	0.044	5.2	52
							600	0.2	-450	0.016	87.4	0.22	0.062	6.7	67
185-34	0.18	4.2	-35	6	40	75	200	1.4	-440	0.075	63.2	0.28	0.017	2.7	25
							300	1.0	-465	0.039	81.9	0.37	0.044	5.8	39
							400	0.5	-480	0.027	88.0	0.40	0.12	9.3	55
							500	0.3	-485	0.023	90.0	0.40	0.15	10.7	65

Table F-2 (Continued)

Single-Stage Acid Leaching of Raw Lignite

Sample Number	% U_3O_8	Grind pH	Feed Mesh	Leaching Conditions Time, Hours	% Solids	Temp °C	H_2SO_4 Lb/Ton	Terminal Conditions pH	Emf (mv)	Residue Assay % U_3O_8	% U_3O_8 Ext'n	Diluted Pregnant Liquor Assay, g/l U_3O_8	Mo	Fe	$SO_4^=$
185-35	0.68	8.0	-35	6	40	75	500	1.5	-430	0.13	82.9	1.41	0.092	3.6	49
							600	1.0	-450	0.11	85.9	1.46	0.095	5.7	58
							700	0.7	-460	0.091	88.5	1.50	0.17	7.5	72
							800	0.4	-470	0.070	91.2	1.55	0.15	9.1	85
185-37	0.35	5.8	-35	6	40	75	300	1.3	-335	0.13	63.0	0.55[7]	0.001	2.6	34
							400	0.8	-355	0.092	74.7	0.65	0.002	3.2	47
							500	0.6	-360	0.067	82.7	0.72	0.002	3.4	60
							600	0.3	-365	0.060	84.7	0.74	0.002	3.5	72
185-38	0.18	5.3	-35	6	40	75	300	1.8	-405	0.089	51.1	0.22	0.003	1.5	25
							400	1.3	-430	0.043	75.0	0.33	0.010	3.4	39
							500	0.8	-455	0.018	90.3	0.40	0.024	6.2	58
							600	0.5	-465	0.023	88.1	0.38	0.032	7.2	63
185-39	0.35	6.1	-35	6	40	75	650	0.4	-460	0.032	92.1	0.81	0.18	7.9	75
185-40	0.16	5.0					515	0.4	-470	0.016	90.8	0.36	0.42	9.2	64
185-41	0.20	5.4					495	0.5	-480	0.029	86.3	0.43	0.19	10.3	57
185-42	0.06	5.2					525	0.5	-480	0.009	87.9	0.13	0.09	12.0	63
185-43	0.09	6.5					215	1.0	-465	0.004	95.8	0.22	0.07	4.5	24
185-44	0.50	4.5					380	0.4	-440	0.060	90.0	1.13	0.65	5.0	47
185-45	0.26	3.9					390	0.5	-460	0.022	93.3	0.61	0.30	5.6	58
185-46	0.13	8.1					700	0.5	-480	0.034	79.0	0.26	0.07	6.3	69
185-47	0.14	4.6					350	0.3	-440	0.018	88.8	0.31	0.24	6.8	50
185-48	0.12	6.7					595	0.4	-460	0.014	91.2	0.27	0.08	6.8	71

Table F-2 (Continued)

Single-Stage Acid Leaching of Raw Lignite

1/ Natural pH in water about 5.

2/ Two hour leach with cold water at 25 per cent solids dissolved about 25 per cent of the Mo and considerable organic material. The solution pH was about 8.

3/ Constant pH leaching.

4/ Added 20 pounds of MnO_2 per ton of ore.

5/ Added 80 pounds of MnO_2 per ton of ore.

6/ Added 30 pounds of $NaClO_3$ per ton of ore.

7/ Very little organic material in solution.

8/ Contains 0.26 to 0.30 grams of Fe^{+++} per liter.

Table F-3

Acid Leaching of Raw Sample 185-29 (A-I) Raw Lignite

Sample	Leaching Conditions[1]	Terminal Conditions		Residue Assay	% U_3O_8	Diluted Pregnant Liquor Assay[2] Grams/Liter			
185-29	H_2SO_4, Lb/Ton	pH	Emf(mv)	% U_3O_8	Ext'n	U_3O_8	Mo	Fe	$SO_4^=$
A	400	0.4	-285	0.32	77.8	2.45	0.003	2.3	49
B	500	0.65	-415	0.038	89.1	0.70	0.037	3.4	58
C	720	0.5	-440	0.121	92.9	3.61	0.10	6.5	80
D	648	0.35	-440	0.046	81.7	0.77	0.023	3.1	84
E	612	0.45	-470	0.0043	70.8	0.045	0.016	4.2	68
F	874	0.4	-460	0.042	94.8	0.046	0.046	11.6	84
G	756	0.5	-440	0.015	87.8	0.27	0.007	7.5	70
H	526	0.3	-450	0.092	90.0	1.84	0.024	4.2	60
I	863	0.5	-465	0.019	92.1	0.47	0.045	5.8	95

[1] Minus 35-mesh ore leached for six hours at 75°C and 40 per cent solids.

[2] Four to one solution to feed ratio.

Table F-4

The Effect of Varying Acid Concentration on Resin-in-Pulp of Lignite Slurries

Test	H_2SO_4 Added Lb/Ton	pH Initial	pH Final	Residue Assay % U_3O_8	% U_3O_8 Extraction
1	0	4.0	4.0	0.17	40.6
2	36	3.0	3.15	0.11	61.5
3	150	1.9	2.15	0.042	85.3
4	250	1.55	1.7	0.028	90.2
5	400	1.2	1.3	0.022	92.3
6	600	0.8	0.8	0.016	94.4

1/ U_3O_8 extraction based on a feed assaying 0.29 per cent U_3O_8.

Table F-5

Thickening Tests of Raw Lignites

Sample Number	Description	Separan Lb/Ton	F	D	R	A	Overflow Description
185-18	Unleached	0.50	4.6	2.3	4.4	0.7	Cloudy
	Leached	0.50	6.7	3.2	0.5	10.2	Cloudy
185-19	Unleached	None	4.0	1.34	0.31	11.4	Clear
	Unleached	0.05	4.0	1.14	1.54	2.5	Clear
	Unleached	0.10	4.0	1.14	3.15	1.2	Clear
	Leached	0.20	4.0	1.80	0.56	5.2	Cloudy
185-20	Unleached	None	4.0	1.66	0.14	22.3	Slightly Cloudy
	Unleached	0.05	4.0	1.40	0.42	9.2	Slightly Cloudy
	Unleached	0.10	4.0	1.40	1.12	3.1	Slightly Cloudy
	Unleached	0.15	4.0	1.40	3.50	1.0	Slightly Cloudy
	Leached	0.10	4.0	1.54	2.80	1.2	Clear
185-21	Unleached	0.10	4.0	1.38	0.53	6.6	Very Cloudy
	Unleached	0.15	4.0	1.38	1.22	2.9	Very Cloudy
	Unleached	0.20	4.0	1.38	4.20	0.8	Very Cloudy
	Leached	0.20	4.0	1.46	0.12	28.2	Very Cloudy

F = Initial dilution, tons of solution per ton of ore.
D = Terminal dilution, tons of solution per ton of ore.
R = Settling rate, feet per hour.
A = Required thickener area, square feet per ton per day.

Table F-6

Filter Tests of Acid Leached Raw Lignite

Sample Number	% U_3O_8	Leach, H_2SO_4/Lb Lignite	Thicken From % Solids	Thicken To % Solids	Separan Lb/Ton Ore	Cycle Time, Minutes	Pickup Vacuum in Hg	Wash & Drain Vacuum in Hg	Cake Thickness Inches	% Moisture	Capacity Lb/Sq Ft/ Day	Soluble Loss %
185-18	0.22	800	13	24	0.50	3.5	20	20	--	60	1000$^+$	--
185-19	0.28	500	32	42	0.25	2	20	20	5/16	51	830	18.2
						4	20	20	7/16	50	570	16.7
185-22	0.15	400	32	42	0.10	2	20	20	1$^+$	48	3600	11.7
						2	10	10	1/2	42	1840	—
185-27	0.14	400	25	38	0.15	2	10	22	1$^+$	47	3840	8.6
						2	10	22	1$^+$	48	3580	12.3
185-28	0.09	400	35	45	0.30	2	20	20	3/16	48	635	19.3
						4	20	20	1/4	46	405	18.7
185-30, 31,33	0.22	520	29	34	0.60	2	21	21	3/16	51	450	11.0
						6	21	21	3/8	48	340	12.0

Table F-7

Two-Stage Countercurrent Acid Leaching Tests of Raw Lignite

		Natural Leach		Strong Leach					Pregnant Liquor Grams/Liter			
Sample Number	Cycle	H_2SO_4 Lb/Ton	pH	H_2SO_4 Lb/Ton	pH	Residue Assay % U_3O_8	% U_3O_8 Ext'n	Total H_2SO_4 Lb/Ton	U_3O_8	Mo	Fe^{++}	Total Fe
185-18	1	130	2.4	630	0.4	0.011	94.7	790	0.71	0.074	--	--
	2	130	2.5	630	0.5	0.015	92.8	760	0.44	0.042	3.92	5.33
	3	147	2.5	560	0.5	0.014	93.3	707	0.47	0.042	4.16	6.69
	4	175	2.5	560	0.5	0.017	91.9	735	0.53	0.047	2.46	4.29
	5	140	2.5	580	0.5	0.013	93.8	720	0.53	0.053	3.54	5.95
185-19	1	0	2.8	404	0.5	0.026	90.7	404	0.80	0.063	1.51	3.59
	2	37	2.3	404	0.4	0.020	92.8	441	0.86	0.12	--	5.92
	3	0	2.5	404	0.4	0.025	91.1	404	0.71	--	2.10	5.46
	4	0	2.5	368	0.4	0.026	90.7	368	0.90	--	2.30	8.38
185-20	1	0	1.5	289	0.3	0.026	83.8	289	0.58	0.006	8.80	No Ferric Iron
	2	0	1.4	258	-	0.026	83.8	258	0.44	0.005	6.65	
	3	0	1.4	221	0.4	0.035	78.2	221	0.32	0.004	5.60	
	4	0	1.4	258	0.4	0.025	84.0	258	0.35	--	5.07	
185-21	1	0	-	295	0.4	0.019	81.0	295	0.37	0.076	4.12	8.80
	2	0	1.6	258	0.4	0.010	90.0	258	0.23	0.061	3.44	6.08
	3	0	1.5	258	0.3	0.011	90.0	258	0.28	0.070	--	6.63

APPENDIX G

Table G-1

Treatment of Lignite Liquor for Ion Exchange

Effect of Recycle of Repulp Solution

	Leaching 1190-Gram Charges				Neutralization with $CaCO_3$					
Test	H_2SO_4 Lb/Ton Ash	Residue Assay % U_3O_8	% U_3O_8 Ext'n		pH	U_3O_8	Mo	Fe^{+++}	SO_4	$CaCO_3$ Lb/Ton Ash
1	700	0.038	95	Before	1.5	1.57	0.82	3.42	102	380
				After	3.8	1.00	0.050	0.03	27.4	
2	700	0.027	96	Before	1.5	1.69	0.90	4.82	116	500
				After	3.8	1.17	0.024	0.01	20.3	
3	700	0.055	92	Before	1.9	1.84	0.65	2.16	136	500
				After	3.5	1.32	0.124	0.08	28.6	
4	700	0.046	93	Before	2.1	1.71	0.61	2.87	116	500
				After	3.3	1.16	0.049	0.01	20.0	

	Repulp						Ion Exchange			
	Filtrate[1/]				Residue		Bed Volumes To:			Loading g U_3O_8/l WSR
		Gram/Liter								
Test	Volume (ml)	U_3O_8	Mo	Fe	% U_3O_8	% Loss U_3O_8	Breakthru	Saturation	Elution	
1	3200	0.13	0.33	1.62	0.004	0.2	62	128	10	94.8
2	3200	0.20	0.02	1.10	0.024	1.7	60	128	10	97.2
3	3500	0.27	0.51	2.37	0.003	0.2	57	124	10	100.0
4	3450	0.26			0.012	1.5	58		10	103.0

1/ Filtrate was recycled to the next acid leach.

APPENDIX H

Part I

Pilot Plant Roasting of Dakota Lignites at U. S. Bureau of Mines Coal Branch, Denver

Pilot Plant Roast Test

The USBM Coal Carbonizer Pilot Plant tested Bryco Mining Company lignite ore on October 13, 1955 at Denver, Colorado. In this 18 hour test, a series of six operating conditions were investigated and char samples were taken for leaching studies. The roasting test data for this run are presented in Table H-1. The objective of this experiment was to bracket a roasting condition which would produce a product suitable for uranium alkaline leach extraction.

The char products were sent to Winchester for laboratory leach testing to evaluate the effect of the roasting conditions on the leaching and filtering characteristics of the char.

Although the products from these roasts filtered quite well, the uranium extractions obtained were not acceptable.

Leaching Conditions

All samples, which were approximately 10-mesh, were leached for 24 hours at 80°C and 20 per cent solids with a solution containing 100 grams of Na_2CO_3 and 50 grams of $NaHCO_3$ per liter. Air was added as an oxidant. The test results on the Denver samples received are shown in Table H-2.

Table H-1

USBM Carbonizer Pilot Plant Test Number 133 Operating Summary

Bryco Mining Company Dakota Lignite
Sample 185-4

Preparation: The as-received sample was minus one inch and contained 33 per cent moisture. This material was stage-crushed to minus 1/4 inch and dried in the pilot plant to six per cent moisture, 28 per cent volatile matter, and 51 per cent ash.

Reactor: Eight inch diameter by 15 feet long; all test conditions operated at 11 minute retention time.

Period	Temp °C	Air Added cfm/ Lb Coal	Sample Weight Char Lb	Weight Dust Lb	Make Gas Value Btu/cf
"A"	500	None	257	13.0	85
"B"	460	None	222	12.0	90
"C"	420	None	302	8.0	92
"D"	435	2.0	270	8.5	30
"E"	460	2.5	317	8.5	32
"F"	500	2.5	139	4.5	28
Non-Sample Char Produced			896	---	
Total Coal			2403	31.0	
			85.5%		

Table H-2

Carbonate Leaching of Denver Roasted Samples

Bryco Mining Company Dakota Lignite
Sample 185-4

Char Sample	Temp °C	Head Assay % U_3O_8	% U_3O_8 Ext'n	Na_2CO_3 Consumption Lb/Ton	Comparative Filter Rate Minutes 1/
"A"	500	0.491	44.7	184	17
"B"	460	0.431	82.0	168	17
"C"	420	0.415	81.2	164	69
"D"	425-435	0.402	81.4	127	40
"E"	460	0.455	70.4	176	16
"F"	500	0.503	50.3	178	11

1/ Two hundred and fifty grams of a 24 cm Buchner funnel followed by wash of 250 ml of hot water.

Carbonate Leaching of USBM Retort Roasts

A pilot plant scale carbonizing test was run at USBM Coal Division, Denver, Colorado on about 2000 pounds of Peter Kiewit lignite from Harding County, Souch Dakota (Sample 185-9). Details of the initial 400°C test are presented in Tables H-3 and H-4.

Table H-5 presents the results of standard carbonate leaching tests on the various products from the pilot plant carbonizing tests and on chars produced in the assay retort in the temperature range 400°C to 500°C.

The extraction from the primary separation in the pilot plant run was about 89 per cent but the residue did not filter at a practical rate. A comparable extraction was also obtained from the 400°C assay retort test, but the filter rate was extremely slow. Good filterability was obtained only from the assay retort test at 500°C but the extraction dropped to 2.4 per cent.

From analysis of the data it appears that, at the short retention time (approximately 12 minutes) of the Denver pilot unit, the temperature of 400°C was not high enough to reduce the volatile content of the Peter Kiewit sample to the point of good filterability. Consequently, a second pilot plant test is scheduled to investigate higher temperatures.

Table H-3

Pilot Plant Drying Test Number 171-12"

U.S.B.M. Coal Division, Region IV, Denver, Colorado

Sample:	Peter Kiewit, Winchester Sample 185-9
Reactor:	Twelve Inch Diameter by Eight Feet Long
Capacity:	Four Hundred Pounds of Moisture Removed Per Hour
Fuel:	Natural Gas, 814 Cubic Feet Per Hour. 421 Btu Per Pound of Raw Product Charged, 383 Btu Per Pound Required to Dry Product

Product	Moisture %	Weight Lb/Hour	Weight Distribution %	U_3O_8 Content %	U_3O_8 Distribution %	Temp °F
Feed	33.6	1354	100.0	0.409	100.0	60
Primary Separator	5.4	959	96.0	0.344	95.7	300
Cyclone	0.0	29.2	3.25	0.400	3.8	250
Scrubber	-	6.8	0.25	0.250	0.5	-

Gases Required

	Volume SCFM(60-30)	Temp °F
Natural Gas	945	1600
Air with Gas	7581	-
Recycle Gas	3144	250
Cooling Gas	2670	250

Table H-4

Pilot Plant Carbonizer Test Number 129

U.S.B.M., Coal Division, Region IV, Denver, Colorado

Sample: Peter Kiewit, Winchester Sample 185-9
Reactor: Eight Inch Diameter by 15 Feet Long

Product	Weight Lb/Hour	Weight Distribution %	U_3O_8 Content %	U_3O_8 Distribution %	Temp °F
Feed	358.9	100.0	0.344	100.0	175
Primary Separator	286.9	86.4	0.350	95.2	740
No. 1 Dry Cyclone	16.3	4.9	0.250	3.84	625
No. 2 Dry Cyclone	2.4	0.7	0.290	0.63	-
No. 3 Dry Cyclone	1.2	0.4	0.210	0.02	413
Hot Precipitator	1.0	0.3	0.130	0.01	235
Tar, Oils, Water	10.4	3.1	0.002	0.001+	165
Gases Produced	13.8	4.2	-	-	73
	332.0	100.0		99.65+	
Total Dry Solids	339.5				
Not Accountable	6.5				

Flow Rates

Feed Solids	358.9 Lb/Hour
Transport Gas	68.2 Lb/Hour
Heat Gas (Natural Gas)	286 SCFH
Solids Carrying Make Gas	608 SCFH
Gas Produced	125 SCFH

Table H-5

Sodium Carbonate Leaching Results on Peter Kiewit Group Lignite Products

Sample 185-9

(All Leaches were 24 Hour, 80°C, with 200 lb. Na_2CO_3/Ton, 100 Lb. $NaHCO_3$/Ton at 20% Solids)

		Heat Treatment			Residue	
Test	Calcine Origin	Temp °C	Time, Hours	Feed Assay % U_3O_8	Assay % U_3O_8	% U_3O_8 Extraction
1	Assay Retort	400	1.0	0.47	0.064	85.8
2	Assay Retort	450	1.0	0.47	0.240	47.0
3	Assay Retort[1]	500	1.0	0.49	0.460	2.4
4	Assay Retort on Sample 185-4	400	1.0	0.40	0.014	96.5
5	Pilot Plant Primary Separator	400	0.17	0.35	0.038	88.8
6	Pilot Plant Hot Precipitator Dust	400	0.17	0.13	0.009	86.0
7	Gas Scrubber Dust	400	0.17	0.011	0.002	83.5

[1] All leaches were filtered on a 24 cm Buchner funnel. All filtered slowly except Test 3 which filtered rapidly.

Part II

Pilot Plant Testing of Dakota Lignites at Bozeman Montana in the Porter-Dillon-Pringle (PDP) Retort

Pilot Plant Roast Test

About five tons of Bryco Mining Company uraniferous lignite was processed in the PDP Retort at Bozeman, Montana on October 24, 1955. This pilot plant test was a 16-hour continuous temperature series test similar to the test No. 133 run at Denver on October 13, 1955. This furnace operated at a 30 minute retention time (or with a char rate of 340 pounds per hour) at temperatures ranging from 413 to 538°C. The furnace operated with minor interruptions and char samples were collected for each temperature condition. The char samples were leached at Winchester to see if a satisfactory leachable product was produced in this furnace. The summary of leaching tests on the chars from Bozeman, given in Table H-6, would indicate that no satisfactory char product was made in the PDP Retort and that the filtering characteristics of these char leach residues were not as good as similar products made in the USBM Carbonizer.

Leaching Conditions

All samples which were approximately 10-mesh, were leached for 24 hours at 80°C and 20 per cent solids with a solution containing 100 grams of Na_2CO_3 and 50 grams of $NaHCO_3$ per liter. Air was added as an oxidant. The test results on the Bozeman samples received are shown in Table H-6.

Table H-6

Carbonate Leaching of Bozeman Roasted Samples
Bryco Mining Company Dakota Lignite

Test Number	Temp °C	Assay % U_3O_8	% U_3O_8 Ext'n	Na_2CO_3 Consumption Lb/Ton	Comparative[1] Filter Rate Minutes
16	413	0.44	37.2	233	3500
17	432	0.39	25.6	-	600
18	455	0.40	32.5	205	420
19	493	0.41	61.7	137	97
20	538	0.48	18.3	142	89
21	Tar	0.0085	17.4	84	-
22	Dust	0.44	84.1	-	11

[1] One hundred grams on a 12 cm Buchner funnel.

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Table J-1

Table Concentration of Peter Kiewit Lignite

Sample 185-18

				Acid Consumption (Lb/Ton)		U_3O_8	
Product	Weight	% Weight	% of Raw Feed Weight	Calcine	Original Feed	% or g/l	% Dist'n
Table Feed:	5777.0		100.0			0.211*	100.0
Roaster Feed	4975.0		86.1			0.24	98.1
Reject or Tails	802.0		13.9			0.032	1.9
Roaster Feed: Calcine 2 Hours @ 450°C							
Roaster Feed	1000.0	100.0	86.1			0.24	
Calcine	351.4	35.1	30.2			0.68*	
Leach Calcine @ 30% Solids, +90°C, 6 Hours - Terminal pH and Emf = 0.85 and -510 (94 ml H_2SO_4 Added = 164.5 g)							
Calcine	200.0			1645	497	0.65*	100.0
Residue	184.0					0.064	9.0
Pregnant	435 ml					1.60	53.4
Wash	500 ml					0.98	37.6
Reject - Calcine 2 Hours @ 450°C							
Reject	500.0	100.0	13.9			0.032	
Calcine	430.6	86.1	12.0			0.037*	
Leach Calcine @ 30% Solids, +90°C, 6 Hours - Terminal pH and Emf = 0.85 and -505 (35.2 ml H_2SO_4 Added = 61.6 g)							
Calcine	200.0			616	74	0.0364*	100.0
Residue	169.0					0.0023	5.3
Pregnant	400 ml					0.15	82.3
Wash	485 ml					0.020	12.4
Roaster Feed Filtrate	52.6 liters					0.001	0.43 (Dissolved)

*Calculated