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Progress Report for the Month of April, 1947

Contract No. W-38-094-eng-27

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April 30, 1947

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SUMMARY

In ore dressing, ³the last three samples of Idaho phosphate rock were treated so as to obtain from each of them ²two separate products, one of which was enriched in quartz and the other enriched in phosphate. Based on β -counts, ^uthe uranium contents of the quartz fractions were consistently low, the assays varying from one-fifth to one-half as high as the corresponding head assay. ^uThe uranium contents of the phosphate fractions were slightly but not significantly higher than the head samples. ²These data substantiate previous evidence that the uranium is disseminated throughout the phosphate and cannot be concentrated to any extent by means of a practicable physical separation.

²In the pyrometallurgical work a number of ^{tests} ~~ideas~~ were tried, the results of which are briefly summarized.

The removal of volatile ^uuranium compounds from the heated shale-ore charge (Sample 1090-4) is apparently not aided by sweeping with large volumes of ^Ssulphur chloride gas.

Results of distillation tests on the oil-shale sample indicate that it is possible to recover 5 gallons of oil ~~per~~ ton of shale as a distillate in a preheating period.

The thermic-reduction tests, using Mg, Al, Fe, or Zn gave no indication of ^uuranium volatilization. ¹³Microscopic examinations are being made on the products to try to find metallic uranium, if any, in the product.

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The vacuum furnace testing was carried out on a phosphorus furnace slag and on various charges of Ores 4, 8, and 11. There are few data to give basis for conclusive interpretations. Estimations of uranium concentrations in the residues by B-count assays gave indecisive indications that there was no appreciable removal of uranium from any of the experimental samples (4, 8, and 11) by heating, smelting, or chloridizing under vacuums of less than 1 micron.

^{3/}Excellent recoveries of uranium from synthetic phosphoric acid solutions containing ^uuranium were obtained by precipitating the ^uuranium with organic amines. The ashed products contained as much as 80% per-cent of ^uuranium. The recoveries ~~were~~ consistently over 70% per-cent. It was more difficult to get satisfactory precipitates from synthetic calcium phosphate solutions and still more difficult from leach solutions, but on the whole the results were highly encouraging.

^{4/}Ninety-four to 98% per-cent of the ^uuranium in superphosphate leach solutions was recovered by ether extraction in the presence of 30% per-cent ^{Fe(NO₃)₃} ferric nitrate. The recovery was lowered by reducing the amount of ^{Fe(NO₃)₃} ferric nitrate. / and

Attempts to prepare "saturated" superphosphate solutions and to use them for leaching the uranium from superphosphate were not very encouraging because of low net extraction. Too much uranium was lost to the residue in retained leach solution.

Attempts to reconstruct superphosphate from mixtures of leach residues and solutions were also highly encouraging. Such mixtures were evaporated to

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about the same weight as the original superphosphate and were then found to have about the same soluble matter. This indicates that little, if any, of the soluble phosphate was lost.

Summary reports on both chemical and physical methods of analysis for uranium in trace amounts are in preparation.

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CHEMICAL AND SPECTROGRAPHIC ANALYSIS

(E. J. Center, Assistant Supervisor)

Spectrographic

The possibility of using electron multiplier phototubes in the analysis of ores and rocks for small amounts of uranium is being investigated. Mr. W. M. Henry will talk to Dr. G. H. Dieke at Johns Hopkins University on April 25, 1947, about the application of these tubes to spectrographic trace analysis in general.

Wet Chemistry

Work is proceeding on a method for the direct extraction of uranium from solutions containing large amounts of phosphate. This problem is being considered from both an analytical and large-scale recovery point of view.

$\text{Fe}(\text{NO}_3)_3$ has proven to be the most effective salting-out agent of those investigated to date. By adding 30 per cent of $\text{Fe}(\text{NO}_3)_3$ to an aqueous superphosphate leach⁽¹⁾, and extracting with ethyl ether for one hour at room temperature in a continuous extractor, 100 per cent uranium recoveries have been obtained. Adding 15 per cent of $\text{Fe}(\text{NO}_3)_3$ and extracting the above solution in the same manner resulted in a 25 per cent uranium recovery, but if the extraction was carried out at 0°C., the recovery of uranium rose to 45 per cent⁽²⁾.

-
- (1) No. 12803. See section on chemical work. The phosphate leach solution contained 4.4 mg. U and 18.7 g. P_2O_5 per 100 ml.:

$$\frac{\text{P}_2\text{O}_5}{\text{U}} = 4250$$

- (2) Three per cent nitric acid was added to the aqueous phase in all the $\text{Fe}(\text{NO}_3)_3$ extractions.

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Plans are being made to expand the extraction phase of the program.

A topical report on Battelle analytical methods for the determination of small amounts of uranium in phosphates, shales and their products is being prepared.

CHEMICAL WORK

(H. A. Pray, Supervisor)

Attention was paid almost entirely to preparation of and leaching with "saturated" superphosphate solutions and to recovery of uranium from the leach solutions. Some additional work was done on leaching phosphate rock with carbonates and shale with acid, but this is not reported because chemical analyses are not yet available.

SUPERPHOSPHATE LEACHING

As explained in last month's report, it was thought that a solution saturated with respect to superphosphate might be an efficient leach solution which would not dissolve much of the superphosphates, but would remove the uranium. Preparation of such a solution was attempted but was found to be difficult, if not impossible.

When an excess of superphosphate was stirred with water there was a steady increase in density until, at a density of 1.5, the solution became too syrupy to work. Furthermore, the solution became more acid. This indicated a shifting ratio between CaO and P_2O_5 in the direction of increased free P_2O_5 in solution. Apparently, in dissolving, the monocalcium phosphate was partly composed to dicalcium phosphate and phosphoric acid.

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Attempts were made to leach batches of superphosphate with solutions prepared in the manner described, but which had not yet reached the syrupy stage. For example, a superphosphate solution containing 63 mg. uranium per liter and which had a density of 1.29 was used to leach one batch of superphosphate. The results are given in Table 50.

The result was that uranium concentration of the solution was increased from 63 mg./l. to 80 mg./l. but the net amount of uranium extracted was only 2.5 mg., or 20 per cent of that present in the superphosphate. About 60 milliliters of the leach solution was left on the leached residue and this contained 4.8 mg. of uranium. If none of the leach solution had been left on the residue the extraction would have been about 7 mg., or over 50 per cent of that contained in the superphosphate. Although the amount of solution left on the residue may possibly be reduced by better technique, it appears that a high net extraction of uranium is unlikely by this method.

The solution with a specific gravity of 1.29 dissolved 20 per cent of the superphosphate, or nearly half as much as is usually dissolved by water. Its density increased to 1.33. Solution with densities higher than this were so syrupy that they were difficult to filter.

The only materials at present available for the leaching tests are superphosphates prepared in the laboratory, and a Sacco brand 20 per cent superphosphates purchased on the open market. The former necessarily varies somewhat in composition from batch to batch. The origin of the second is not known, or is the conditioning treatment to which it has been subjected known. Both contain about the same amount of uranium, 0.006 - 0.007 per cent by beta count. The leach solutions from the laboratory—prepared superphosphates were more acid than those from the Sacco product. The pHs of the solutions

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TABLE 50. LEACHING SUPERPHOSPHATE WITH A "SATURATED"
SUPERPHOSPHATE SOLUTION

	Volume, cc.		Weight, g.		Loss, %	Uranium				Extracted, %
	Before	After	Before	After		Before mg./l.	mg.	After mg./l.	mg.	
Solution	400	340	516	452	12	63	25	80	27.5	20
Superphos- phate	-	-	190	152	20	-	-	-	-	-

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were 0.5 to 1.0 and 2.5 to 3.0, respectively. About twice as much of the uranium in the laboratory product was water-soluble as of that in the other. Receipt of the superphosphate from the Florida rock would help this situation.

URANIUM RECOVERY

As indicated in last month's report, two methods for recovering uranium from leach solutions are being investigated. They are (1) precipitation with organic reagents, and (2) extraction with an organic solvent.

Precipitation of Uranium With Organic Reagents

Uranium was precipitated nearly quantitatively with amines from phosphoric acid solutions containing 500 and 50 mg. of U per liter. Table 51 gives the results by beta-count and chemical analysis. The beta-count analyses on the 500 mg./l. solutions are less reliable than the chemical analyses because they were based on a single reading taken a short time after the precipitation. The others were based on several readings made at intervals of several days and are believed to be more reliable.

The ignited precipitates were high in uranium. They contained 50 to 80 per cent of the metal. Tetraethylenepentamine was the best precipitant of those tried. The major part of the tests on the more dilute uranium solutions were made with it.

Tests made on synthetic leach solutions which had been prepared from monocalcium phosphate phosphoric acid and uranyl nitrate, water leach solutions from laboratory prepared superphosphate, and water leach solutions from Sacco brand superphosphate purchased on the open market have not been so definite.

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TABLE 51. RESULTS OF URANIUM PRECIPITATION BY AMINES FROM 50 mg. OF U/100 cc. OF SOLUTIONS, pH SLIGHTLY BELOW 2

Test No.	Amine	Beta-Count Analyses*		Chemical Analyses	
		mg. of U	Recovery, %	mg. of U	Recovery, %
<u>Solution: 500 mg. U per liter. pH 1.5-2</u>					
2939-10-1	2-aminopyridine	36	72	31	62
2939-10-2	"	38	76	30.4	60.8
2939-17-3	Tetraethylene pentamine	50	100	49.2	98.4
2939-14-1A	Ethylenediamine	46	92	43.7	87.4
2939-15-1	Hexamethylenetetramine	50	100	32.8	65.6
2939-15-2	Melamine	46	92	32.8	65.6
2939-16-1	Mono-n-amyamine	48	96	42.2	84.4
2939-16-3	O-phenylenediamine	43	86	37.3	74.6
<u>Solution: 50 mg. U per liter. pH 1.5-2</u>					
2939-21-1	2-aminopyridine	50	100	-	-
2939-21-2	Ethylenediamine	44	88	-	-
2939-21-3	O-phenylenediamine	40	80	-	-
2939-22-2	Tetraethylene pentamine	44	88	-	-
2939-22-3	Melamine	38	76	-	-
2939-23-1	Hexamethylenetetramine	40	80	-	-

* B-count analyses on the 500 mg./l. solutions based on a single count; the others on three, or more.

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Synthetic calcium phosphate solutions and leach solutions from superphosphate did not react similarly to precipitation, at least in so far as could be determined from beta-count analyses. Chemical analyses are not yet complete. The beta-count indicated higher uranium precipitation from the synthetic calcium phosphate solutions. In general, however, the results are encouraging.

On the basis of preliminary experiments it was thought that carbon dioxide in the superphosphate leach solution might prevent precipitation of the uranium. Further tests with CO₂ saturated and unsaturated solutions indicate that carbon dioxide may be of minor importance.

Another possible interfering ion is fluoride. The effect of this on uranium precipitation will be investigated.

So far only amines have been studied as precipitants. Other classes of reagents, notably nitrogen-sulphur compounds and phenolic acids, will be tried.

Uranium Extraction with Ether

The extraction of uranium from superphosphate leach solutions is described in the analytical section of this report. In the presence of 30 per cent of ferric nitrate in the leach solution, 94 to 98 per cent of the uranium was extracted in one hour. When the amount of ferric nitrate salting-out agent was reduced to 15 per cent, the extraction dropped to 45 per cent. Ethyl ether is the only solvent tried so far.

Solutions containing as much as 30 per cent of ferric nitrate could not be used for leaching superphosphates. When such a solution was mixed with the latter, the mixture set to a gel. What effect ferric nitrate and other similar salts would have on the availability of the phosphate remains to be determined.

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SUPERPHOSPHATE RECOVERY

Recently consideration has been given to recovery, or reconstruction, of superphosphate from leach residues and leach solutions which have been stripped of uranium. Slurries were prepared from proper proportions of these products and evaporated to the same weight as the original superphosphate.

The products were examined for water-soluble material, physical properties as compared to superphosphate, and available phosphate. No loss in water solubility was observed on the reconstructed superphosphate. Its physical properties, i.e., granulation, crushability, dryness, color, and similar properties were hardly distinguishable from those of the original superphosphate. Chemical analysis has not yet been completed but the observation that no loss in water solubility occurred strongly indicates that none of the soluble phosphates were lost by leaching and evaporation.

ANALYSIS OF SAMPLES FROM ANACONDA PHOSPHORIC ACID PLANT

Samples of raw rock, filter cake, and vanadium red cake were received from the U. S. Geological Survey. Analyses, given in Table 52, show that the uranium must be extracted with the crude phosphoric acid as neither the red cake nor the filter cake contain it. A sample of the acid was not included with the others.

TABLE 52. ANALYSES OF ANACONDA PRODUCTS

Product	U, %
Raw rock	0.010
Red cake	0.002
Filter cake	0.002

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BETA-COUNTING METHODS OF ANALYSIS

(H. R. Nelson, Supervisor)

Spot tests to determine whether the beta-count analyses of the ores and diluted synthetic ores are in error because of high-emanating power (continual loss of radon) were made on Ore 1 (Tennessee shale), Ore 8 (Idaho phosphate rock), Ore 11 (Florida pebble phosphate rock), and NBS-2230-1-KS-UL (Canadian uraninite). These tests were made by sealing the ores in polystyrene briquettes to trap possible emanation, and measuring the change of activity with time. An increase of activity proceeding according to the half-life of radon would indicate an increase in the amount of radon in the sample caused by entrapment in the plastic. No sensible increase was observed in any of the samples; it is concluded that there is no error in beta-count analyses of these ores owing to radon emanation.

The topical report on beta-counting methods is being revised and edited.

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ORE-DRESSING AND PYROMETALLURGICAL STUDIES

(A. C. Richardson, Supervisor)

ORE-DRESSING STUDIES

The progress reports for February, 1947, and March, 1947, stated the conclusion that no significant concentration of uranium was obtained by the concentration of the major phases of Samples 1090-1, -4, -7, and -9, the Tennessee shales; Sample 190-11, a Florida pebble phosphate; and Sample 1090-8, an Idaho phosphate rock.

This month similar data have been obtained on the three remaining samples, 1090-3, -5, and -6, all Idaho phosphates.

The procedure used in separating the phases of these three samples was that developed as a result of the experience with Sample 1090-8. In each case the organic matter was removed by roasting (at 1400°F. for Samples 1090-3 and 1090-6; at 1900°F. for Sample 1090-5), and the roasted product was ground and separated into three fractions by water elutriation. These were as follows:

<u>Product</u>	<u>Settling Rate</u>	<u>Nominal Size, Microns (Based on Quartz Spheres)</u>
Coarse sand	Over 5 cm./min.	Over 30
Intermediate	Between 1.4 and 5 cm./min.	15 to 30
Fine	Under 1.4 cm./min.	Under 15

Only the intermediate fraction was separated with heavy liquids; the coarse was not used because of insufficient liberation, the slime product was discarded because its persistent flocculation gave a poor separation. Two specific gravities were used to closely bracket quartz, and to give a heavier

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and lighter product as well. The products were all analysed for uranium content by the beta-count method; the product high in quartz was also analysed for silica content.

The results are shown in Table 53. The conclusions for all three samples are similar and can be discussed as one. No change is shown in the uranium content with change in particle size. The 2.60 to 2.68 specific gravity portions, which are the fractions enriched in quartz, contain from 76 to 87 per cent of SiO_2 and only 0.001 to 0.004 per cent of uranium. This is only 1/5 to 1/2 of the uranium assay of the head samples and indicates that there is no concentration of the uranium with the quartz. The fractions over 2.68 specific gravity, which consist of relatively clean phosphate, are slightly but not significantly higher in uranium contents than the head samples. It is concluded that no practicable concentration of uranium by physical methods is possible with these samples.

When the chemical analyses are completed on these samples it is planned to prepare a table that will show the approximate mineral composition and the uranium distribution in the major phases for all of the samples examined. Other than this, no further ore-dressing studies are contemplated on these particular samples.

PYROMETALLURGICAL WORK

This report covers the work during the period April 1 to April 30, 1947, on the elimination of uranium from low-grade ore samples by pyrometallurgical means. The work is divided into the following phases: use of sulphur chloride as a volatilizing agent, thermic reductions by metals, oil-shale distillations, and vacuum testing.

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TABLE 53. RESULTS OF CLASSIFICATION AND HEAVY LIQUID SEPARATIONS
ON ROASTED SAMPLES OF IDAHO PHOSPHATE ROCK

Classified Product	Weight, %	U, %	Heavy Liquid Product	Weight, %	U, %	SiO ₂ , %
<u>Sample 1090-3</u>						
Fine	54.1	.005	--	--	--	--
Coarse	22.1	.005	--	--	--	--
Intermediate	23.8	.005	Over 2.68 2.68 to 2.60 Under 2.60	13.6 9.0 1.2	.007 .001 .004	-- 87.1 --
Composite	100.0	.005		23.8	.005	--
<u>Sample 1090-5</u>						
Fine	29.2	.008	--	--	--	--
Coarse	57.6	.008	--	--	--	--
Intermediate	13.2	.008	Over 2.68 2.68 to 2.60 Under 2.60	10.9 1.9 0.4	.009 .004 .006	-- 76.3 --
Composite	100.0	.008	--	13.2	.008	--
<u>Sample 1090-6</u>						
Fine	37.1	.010	--	--	--	--
Coarse	45.5	.012				
Intermediate	17.4	.012	Over 2.68 2.68 to 2.60 Under 2.60	14.2 2.6 0.6	.012 .003 .005	-- 82.2 --
Composite	100.0	.011	--	17.4	.010	--

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Use of Sulphur Chloride

The chlorination tests on Sample 4, as described on pages 172-173 in the report dated March 21, 1947, were repeated using sulphur chloride in place of pure chlorine gas. The sulphur chloride was prepared by bubbling metered amounts of chlorine through a molten sulphur bath. This sulphur bath was held at 275°F. At this temperature the chlorine formed sulphur chloride readily, which was then passed over the ore charge contained in a Pyrex brand pipette in an electric tube furnace. Each sample was held at 662°F. for 3 hours. The amount of chlorine (and, therefore, a proportionate amount of sulphur chloride) was varied from 15 cc. to 1200 cc. per minute. It was hoped that an excess of the reacting gas would tend to sweep away any volatile uranium compounds that might be formed. The results of these tests, however, indicated that such an effect was not attained. The tests in which large amounts of sulphur chloride were used gave residues of approximately the same uranium content as those tests in which smaller amounts of sulphur chloride were used.

These results were checked by both beta-count and chemical assays and are in agreement with those results obtained from similar tests (pages 172-173) in which pure chlorine was used instead of sulphur chloride. They also substantiate the previous indication that sulphur chloride will, under similar conditions, remove about 5.0 per cent more uranium than chlorine.

Oil-Shale Distillation

In the report of March 31, 1947, it was stated (page 174) the sample of oil shale, Sample 1090-4, had undergone a series of destructive distillation tests. The distillations were carried out in a quartz retort in a vertical

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electric tube furnace with an attached water cooled condenser for the effluent gases. The samples were heated to about 800°F. for 20 hours and then the temperature was raised to 1100°F. for the subsequent 4 hours of treatment. The results of the tests were as follows:

The ore lost, on an average, was 14.5 per cent of the original weight. The amount of free carbon remaining in the residues was 10.2 per cent. The total loss on ignition of this ore was 24.7 per cent. The water cooled condenser recovered about 4 per cent of the weight of the raw ore, leaving about 10.5 per cent unrecovered. The condensate contained 44 per cent oil and 56 per cent water. The oil, which amounted to 5 gallons per ton of shale, was roughly 1/3 unsaturated aromatics and olefines, and 2/3 saturated paraffins. Since it is probable that this ore would be heated prior to any metallurgical treatment, the above tests indicate some valuable products might be recovered during this period of preheating. Assays by beta-count and chemical analyses showed there was no apparent uranium elimination from the ore by this treatment.

Thermic Reductions

Since metallic uranium is being prepared at the present time by the reduction of uranium tetrafluoride with magnesium, the effect of reducing agents such as Mg, Al, Fe, and Sn, on Samples 1090-4, -5, and -11, was investigated.

The finely ground ore was mixed with 10 to 20 per cent, by weight, of the metal and heated to 1472°F. When magnesium was used, a flash point was reached which indicated a violent exothermic reaction. Charges with the other metals gave no such indications.

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An evaluation of the products of these tests is now under way. Preliminary assays by beta-count indicate there was no appreciable volatilization of uranium from the samples of 4, 8, or 11 in combination with any of the four metals mentioned above.

Microscopic examinations of super-panner concentrates of the products are being made to detect any metallic uranium that may have been formed by this reduction.

Pyrometallurgical Tests in a Vacuum Furnace

A series of tests was performed last month in which Ores 4, 8, and 11 were tested in a furnace under vacuum.

Figure 3 shows the details of the construction of the induction furnace used in the vacuum testing. The drawing is to scale and the quartz furnace tube is 24-inches long and 4-1/4-inches outer diameter. The furnace was designed to give a positive vacuum seal and to permit easy access to the charge. By removing the flange on the upper portion of the furnace it is possible to remove the crucible containing the charge and replace it with another crucible in a matter of 2 or 3 minutes.

Figures 4 and 5 show two views of the PSE pumping system with attached electric reduction furnace. The system of valves permits any combination of pumping from the furnace. The procedure employed was to preheat the charge slightly while open to the atmosphere. Then the furnace was sealed airtight and the entire system (furnace, diffusion pump, and lines) evacuated by the mechanical fore-pump to the nominal operating pressure of the fore-pump. The diffusion pump was blanked off from the furnace and was started in operation

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FIGURE 3 -ELECTRIC INDUCTION FURNACE FOR VACUUM TESTING

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Figure 4, Legend

- A. Cold trap
- B. Cold-trap housing
- C. Air-inlet valves (2)
- D. Control box, front view
- E. Pirani vacuum gauges (3)
- F. Switch box for mechanical pump
- G. Cooling water hoses
- H. Frame, PSE
- I. Caster wheel
- J. Valve between furnace and diffusion pump
- K. Cooling coils on furnace (2)
- L. Valve between furnace and forepump
- M. Quartz furnace tube
- N. Valve between forepump and diffusion pump
- O. Exhaust port
- P. Flywheel, forepump
- Q. Mechanical forepump housing
- R. Thermoswitch

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Figure 4. PSE Pumping System and Induction Furnace
Front View

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Figure 5. Legend

- A. Cold trap
- B. Vision port, eye piece
- C. Removable flange on top of furnace
- D. Valve between diffusion pump and furnace
- E. Cooling coils (2)
- F. Induction-furnace coil
- G. Quartz furnace tube
- H. Mechanical forepump housing
- I. Frame, PSE
- J. Caster wheel
- K. Stand, for taking readings
- L. Cold-trap housing
- M. Control box, rear view
- N. Cooling water tubes
- O. Diffusion pump
- P. Calrod heater unit in diffusion pump
- Q. Cooling water hoses
- R. Power lines
- S. Neoprene gasket
- T. Air-inlet valve

Figure 5. PSE Pumping System and Induction Furnace
Rear View .

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with the fore-pump continuing to hold the vacuum on the furnace, as well as act as fore-pump or rougher pump for the diffusion pump. When the diffusion pump is operating efficiently, valves are adjusted so as to place it in series with the mechanical fore-pump, and both pumps are then evacuating the furnace, the diffusion pump acting as the finishing pump and the fore-pump as the primary or rougher pump.

One of the samples tested in this vacuum furnace was a slag comparable to a standard phosphorus furnace product. This slag was prepared from Sample 1090-11 by smelting with silica and coke. The phosphorus content was less than 0.05 per cent, and there had been an elimination of 20 per cent of the uranium, as determined by beta-count assays.

The slag underwent further preparatory treatment by being oxidized by roasting at 1652°F. for 4 hours with sodium nitrate. The amount of nitrate added was 10 per cent of the weight of the slag. The roasted product was cooled, ground, and mixed with sodium fluoride added to the amount of 10 per cent of the weight of the sample. The mixture was melted in the vacuum furnace and held at the fusion temperature of 1112°F. for 4 hours, under a pressure of less than 1 micron. The final slag is undergoing chemical analysis to determine the extent of uranium elimination.

Other tests under vacuum included a FeS_2 -NaCl roast, smelting, and roasting of the three representative ores. Results have not yet been confirmed by chemical assays, but preliminary beta-counts on products do not indicate any appreciable elimination of uranium values by this method of treatment.

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