

ORNL-58  
*cp. 5a*



3 4456 0360248 4

OAK RIDGE NATIONAL LABORATORY  
OAK RIDGE, TENNESSEE

TECHNICAL DIVISION  
M. D. Peterson, Director

REPORT FOR QUARTER ENDING JUNE 1, 1948

by

Stuart McLain

June 1, 1948

OAK RIDGE NATIONAL LABORATORY  
CENTRAL RESEARCH LIBRARY  
CIRCULATION SECTION  
4600N ROOM 175  
**LIBRARY LOAN COPY**  
DO NOT TRANSFER TO ANOTHER PERSON  
If you wish someone else to see this  
report, send in name with report and  
the library will arrange a loan.

NON-TRANSFERABLE



**DECLASSIFIED**

CLASSIFICATION CHANGED TO:  
BY AUTHORITY OF: T 107146  
BY: R. Morrison 7/8/57





DISTRIBUTION:

- 1. G. T. Felbeck
- 2. 706-A
- 3. 706-A
- 4. 706-A
- 5. 706-B
- 6. Biology Library
- 7. Training School
- 8. Central Files
- 9. Central Files
- 10. Central Files
- 11. Central Files
- 12. Central Files
- 13. E. Murphy
- 14. E. Taylor
- 15. A. E. Blaender
- 16. J. S. Felton
- 17. M. D. Peterson
- 18. H. E. Brington
- 19. W. H. Bridges
- 20. J. R. Hoffman
- 21. W. A. Johnson
- 22. J. A. Kiser
- 23. J. A. Lamm
- 24. R. N. Lytle
- 25. S. McLain
- 26. K. Z. Morgan
- 27. D. G. Reid
- 28. C. N. Ruckelshaus
- 29. F. L. Stearns
- 30. A. H. Weinberg
- 31. C. E. Winter
- 32. C. J. Collins
- 33. F. L. Culler
- 34. A. S. Householder
- 35. S. R. Sapirie
- 36. H. M. Roth
- 37-38. AEC, Oak Ridge National Laboratories
- 39-46. Argonne National Laboratories
- 47-50. Brookhaven National Laboratories
- 51-54. University of California, Radiation Laboratories
- 55-58. Los Alamos
- 59-61. AEC, Washington
- 62-63. New York Directed Operations
- 64-65. Hanford Engineer Works
- 66-67. General Electric, Schenectady
- 68. Patent Advisor
- 69-83. Technical Information Division, ORDO



3 4456 0360248 4



Technical Division Report for Quarter Ending June 1, 1948

0. Abstract

The Technical Division continued to exert its main effort on the High Flux Pile. The design of the pile mock-up or prototype, the chemical, and the metallurgical problems related to the pile were given the most attention. The developments of greatest interest in respect to the pile and associated plants were:

- 1) The design of the tanks for the pile mock-up or prototype were essentially completed; the design of the grids and supports nearly so; and the design of the mock reflector about 75 per cent completed. Bids were received for the steel tanks and purchase requisitions issued. Procurement of all items for the mock-up will be expedited.
- 2) Corrosion tests on beryllium have indicated that the best metal has satisfactory corrosion resistance. However, metal is continually being received that contains inclusions and exhibits very poor corrosion resistance. In addition this poor beryllium is very difficult to machine due to frequent fractures. An enlarged development program on the production of beryllium is indicated. A conference of the interested personnel will be called to determine the action to be recommended.
- 3) Studies of the solution of the uranium-aluminum assemblies have indicated that their solution may be carried out without the use of a heel of excess metal, eliminating danger of uranium build up in the solution tanks.
- 4) The Pilot Plant operation has resulted in completion of the runs using Oak Ridge Pile slugs (X slugs) with single cycle gross gamma decontamination factors of about  $10^3$  and average uranium losses of less than 0.01 per cent over the single cycle. It was not possible to determine the decontamination over the second cycle because of the extremely low value of the activity. Runs using Hanford slugs will be made during the next quarter.
- 5) The Pilot Plant also demonstrated that satisfactory crud removal can be obtained without the use of  $MnO_2$  as a filter aid. This results in increased filter rates and lowered losses of uranium.

The developments of most interest on programs not directly related to the High Flux Pile were:

- 1) The personnel and space allotted to the shielding program were considerably increased and an extensive program of the engineering tests developed.
- 2) Considerable thought was given to the programs to be taken up on completion of the work on the High Flux Pile. These programs will include uranium recovery from waste solutions, decontamination of wastes, expansion of the work on heat transfer, study of the theory of extraction, radiation tests, and other problems.

Technical Division Report for Quarter Ending June 1, 1948

1. General

The work conducted during the past quarter has been the continuation of the design of the Mock-up of the high flux pile, the engineering and metallurgical development tests related to the pile, and the chemical separation's processes. A small amount of work has been done on shielding, extraction theory, heat transfer, testing of pumps, etc.

Considerable planning has been done on programs to be undertaken on completion of the work on the pile for the Argonne National Laboratory. The most important work now planned will deal with recovery of uranium from the wastes and deactivation of all waste streams. The work on shielding and heat transfer will be considerably expanded.

2. Pile Design and Development

2.1 Design Progress (J. A. Lane)

In spite of heavy loss of personnel from the Design Section due to uncertainties concerning the future work at Oak Ridge National Laboratory, satisfactory progress has been made toward completion of the design details of the High Flux Pile mock-up or prototype. The drawings of the steel tanks which make up the top inlet sections and the bottom water outlet section were completed and bids obtained from sub-contractors. The drawings of the aluminum tank, or the "active" tank section, were completed and the drawings of the mock beryllium reflector approximately 75 per cent completed. It is estimated that these detailed drawings will be ready for bid requests by July 1, 1948. In order to expedite this work design changes have been held to a minimum. In some cases, however, design simplifications have been introduced in order to facilitate the fabrication of the parts. These changes will be discussed in detail in the following paragraphs.

The design of the aluminum grid support castings and the grids for the active assemblies is nearing completion. These drawings will be released for procurement within a week. Other outstanding problems such as the regulating rod details and changes in the shim rod design are scheduled for completion by August 1, 1948. It is expected that the top plug design with the supports, motors and gears for the control and shim rods, will be completed by September 1, 1948. The design of the unloading device is not yet worked out in detail. Very little direct work has been put on the design of the High Flux Pile itself. However, most of the mock-up design is directly applicable to the reactor.

## 2.11 Beryllium Reflector

The general scheme of the beryllium reflector design was described in detail in the last quarterly report, ORNL-8. In general, the reflector consists of vertical beryllium assemblies held in aluminum end boxes and supported by aluminum grids. Each assembly is made up of three sub-assemblies, each approximately one inch by three inches in cross-section. The numerous experimental holes which penetrate the active tank section introduce very complex designs. The design of all of these complicated assemblies is substantially complete. The detailed drawings for over thirty per cent of the assemblies have been completed and the rest will be completed by July 1, 1948.

The mock reflector will be fabricated from pure aluminum, rather than beryllium. In order to facilitate construction, the aluminum assemblies will be riveted rather than furnace brazed as originally contemplated for the beryllium assemblies. Present experimental work indicates that brazing the beryllium sections for the reactor is not very satisfactory. The method of joining these pieces must be decided upon later on the basis of additional tests. It appears, however, that bolting will be quite satisfactory.

Originally it was believed that the varying heat development and thermal stress in these assemblies would require different cooling surface areas. Results of recent tests and calculations have indicated that small holes will give more than sufficient cooling. This reduces the water content of the reflector to about two per cent. The use of these smaller channels has also permitted the design of more easily machined water channels around the beam holes.

Several actual beryllium assemblies will be made up and tested in the mock-up for corrosion resistance under actual operating conditions.

The aluminum tank drawings have been changed to eliminate the use of dowels for orienting the mock assemblies above and below the beam holes. Each two opposite aluminum assemblies above the beam holes will be bolted together and the resulting pieces will be dove-tailed to the adjacent sections to give positive locking. Below the beam holes the reflector pieces will be held together by adjacent sections and by bolting and dove-tailing the two front pieces only.

The Beryllium Corporation has begun the melting and casting of the production beryllium for the pile. Arrangements are underway to obtain a casting for tests and quality control work.

2.12 Mock-up Design

As noted above, the design of the aluminum tank with its associated parts is nearly complete. The Aluminum Company of America has suggested changes in the drawings to facilitate fabrication of the tank proper. Drawings of the group of supports and grids have been sent to the Aluminum Company for use in preparation of shop drawings of these castings. The sight glasses have been removed from the four offset beam hole thimbles of the aluminum tank to facilitate manufacture.

Bids have been obtained for the steel tanks and delivery is promised for October. The top plug design will be reviewed and it is expected that the drawings will be released by September 1st. A request for assignment of the necessary steel for this plug has been made.

The control rod design has been completed and the detailed drawings are being prepared. Drawings of the unloading machine, as prepared by the Kellex Corporation, will be reviewed and completed by September 1st.

2.13 Graphite and BeO Reflector Design

Due to the serious deterioration of the graphite under fast neutron bombardment, a protective annulus of beryllium oxide balls has been substituted for the graphite adjacent to the aluminum tank. This annulus is 5-1/2 feet inside diameter by 7-1/2 feet outside diameter and contains a seven-foot depth of 1-1/2 inch diameter BeO balls. Design data concerning this zone and the graphite reflector are summarized as follows:

BeO Annulus

Maximum ball temperature	540°F
Average ball temperature	380°F
Ball temperature at tank wall	430°F
Ball temperature at graphite	310°F
Tank wall temperature	110°F
Maximum ball temperature adjacent to Al sleeves	320°F
Air temperature corresponding to above ball temperature	400°F
Pressure drop thru balls	10" H <sub>2</sub> O
Average exit air temperature	180°F
Total air flow thru balls (at 130°F)	6,000 cfm

Aluminum Sleeves (for beam holes)

Dimensions: Two concentric 1/8" wall tubes with 1/2" cooling annulus between. Inner tube is 6" I.D.

Maximum wall temperature	220°F
Pressure drop	5" H <sub>2</sub> O
Axial flow rate	70 ft/sec
Total volumetric flow (6 sleeves)	2,000 cfm

Graphite Reflector

Dimensions: 12' x 14' x 10' high with 7-1/2' diameter hole at center. Fifty 2-1/2" holes with 1-1/2" plugs and eight 4" holes with 3" plugs provide cooling channels.

Maximum temperature	300°F - 350°F
Air flow rate	90 ft/sec
Volumetric flow at 130°F	11,000 cfm
Flow for lead around instrument holes	1,000 cfm
Total air requirement	20,000 cfm

The total flow requirement of 20,000 cfm represents a saving of about 25,000 cfm over the original design incorporating a replaceable graphite ring. However, the BeO is more expensive so that costwise the two schemes are comparable as indicated by the approximate costs given in the following table:

Graphite Ring

Power	\$180,000
Graphite	10,000
BeO Sleeves	<u>10,000</u>
	\$200,000

BeO Annulus

Power	\$ 80,000
BeO Balls	<u>120,000</u>
	\$200,000

The above power cost was estimated on the basis of 6 mills per kw hour, 20 years operation, 0.8 pile use factor and 40-inch water drop across the fans. The graphite was estimated as 50 cents per pound in place and the BeO estimated as \$1.50 per ball.

The real advantage of using the BeO balls is that all of the graphite will be protected from radiation damage. This assures continuous operation for long periods of time and eliminates the replaceable graphite ring - an awkward design at best.

Practically no detailing of the graphite reflector has been done. It is expected that Argonne National Laboratory will carry out this phase of the work along with the site drawings and shielding drawings.

### 2.14 Regulating Rod Design

Several designs for the regulating rod have been investigated in an attempt to obtain a rod of the lightest possible weight, consistent with stability toward shock and vibration. Although the best considered design meets the required specifications, factors of safety are somewhat lower than generally used. The behavior of such a rod under actual flow conditions, moreover, is difficult to predict. For this reason, it is expected that the final design of the regulating rod will be dictated by experiments in the pile mock-up or in a separate regulating rod set up.

The present design consists of a tubular aluminum rod, 1-1/2 inch diameter with a 3/32 inch wall thickness. This rod is supported by four bearings and is capable of remote coupling and uncoupling. Various types of remote couplings have been considered. The drive mechanism will consist of an electrically driven rack and pinion gear actuated by a Servo mechanism. Suitable shock absorbers will be provided for the rod. Operating characteristics and rod weights are summarized as follows.

Rod weight below coupling	10 lbs
Total rod weight	43 lbs
Load applied to lower rod	167 lbs
Load applied to raise rod	197 lbs
Velocity of rod achieved in 1/30 sec	4 ft/sec
Horsepower required to raise or lower the rod (including inertia of the gears)	2.0 - 2.2

2.15 Water Cooling System Design

Due to the cancellation of Kellex Corporation contracts, work on the design of the air and water cooling systems and the active waste disposal system was interrupted. These designs are being completed. The design of the cooling water system is essentially complete and will require very little work before starting construction drawings. The design of the active waste disposal system is approximately 70 per cent complete while the air cooling system is about 50 per cent complete.

A design and cost estimate has been developed for the cooling system of the so-called "Poor Man's Pile" reported in ORNL-26 and also for the operation of the High Flux Pile at approximately one-tenth power level. The equipment cost for the first of these two systems is roughly one-third that of the High Flux Pile as the result of a reduction in capacity and integration of the cooling system within and on the pile building. In contrast to this, the second system, which utilizes the same arrangement of cooling equipment as the high flux pile, costs approximately one-half as much due to the decreased capacity only.

Cost data and preliminary designs are in progress on several types of cooling systems for a 30,000 kw pile with a normal coolant flow of 15,000 gpm and a maximum shutdown flow of 1,500 gpm. It is assumed that water of reasonably good quality and quantity are available.

The systems being considered are listed as follows:

- 1) Once through strainer system - steel piping
- 2) Once through filtered water system - steel piping
- 3) Once through flocculated and filtered water system - steel piping
- 4) Recycle system with demineralized make-up water and 1% purge
  - a) stainless piping (or stainless clad)
  - b) aluminum piping
  - c) coated piping
- 5) Stainless steel recycle system with Kleinschmidt boiler (or equivalent) for make-up using hot demineralization and no purge
- 6) Stainless steel recycle system with 1% purge to a retention basin for decay to levels permitting purification for reuse by distillation or demineralization.

Preliminary indications show that the once through strainer system, 1), is the most economical although the induced activity in the water is on the border line of tolerance for  $\text{Na}^{24}$  (0.5 micro curies per liter). Preliminary results further show that the best system, considering economy of operation, minimum release of active waste, and safety is between 5) and 6). Data are required on hot demineralization systems before a firm evaluation can be made.

## 2.2 Engineering Development (C. E. Winters)

The engineering development tests related to the high flux pile are essentially complete except for work on thermal syphoning of water through the uranium alloy assemblies and beryllium reflector after shutdown. This work will be continued on a high priority basis.

### 2.21 Scale Formation and Water Treatment

Studies of the effect of scale formation on the coefficient of heat transfer between aluminum surfaces and flowing water have continued. These studies have shown that "protective" scales form naturally on hot aluminum surfaces cooled by water. After a certain maximum thickness is reached, the scale coefficient of heat transfer remains constant for a given metal temperature and given water conditions; i.e., no more scale is formed after the protective layer has been completely formed. The thickness of this protective scale is dependent upon aluminum surface temperature and water pH. The results reported below are all for demineralized water, with a 1 per cent purge and about .03 ppm Fe and .05 ppm Al ions in the make-up water. The average water temperature was about 50° C. The tests were conducted in a double pipe heat exchanger. The exchanger had 2S aluminum tubes about three feet long. All other piping, tanks, and pump were made of stainless steel.

It was found that the protective scale had a heat transfer coefficient of about 12,000 BTU/(hr)(ft<sup>2</sup>)(°F) at pH of 7.1 (with 5 ppm sodium dichromate and .005 Molar H<sub>2</sub>O<sub>2</sub>) and that it took about ten days to form this protective scale at a heat flux of 500,000 BTU/(hr)(ft<sup>2</sup>) and water velocity of 30 ft/sec. The protective scale at pH 6-6.5 (with .005 Molar H<sub>2</sub>O<sub>2</sub>) had a heat transfer coefficient from 30,000 to 50,000 BTU/(hr)(ft<sup>2</sup>). It took from two to three weeks to form this scale.

It seems apparent that if the scale reaches only a certain maximum thickness with no further decrease in scale coefficient of heat transfer, no defilming facilities need be supplied for the operation of the pile. The question remains whether or not corrosion of the aluminum continues after the protective scale has been formed; i.e., whether the scale is removed at the same rate that it is formed, or whether corrosion stops entirely.

It has been postulated that scale formation rates are proportional to corrosion rates. Accordingly, some experiments are being run to determine heat transfer coefficients of scales formed under similar conditions to those of the regular corrosion studies. Water is circulated through the tube at 87° C., no heat is transferred through the tube, and no purge water is added. Thus the temperature of the metal surface is the same as the water temperature. In the usual scale formation studies, heat is transferred through the tube to cooling water at a heat flux of 500,000 BTU/(hr)(ft<sup>2</sup>) and the tube surface temperature is maintained at a calculated average temperature of 87° C.

One combination scale formation and corrosion test has been completed using hot water. The results are summarized as follows:

Water Conditions:

pH 7.5, .005 Molar H<sub>2</sub>O<sub>2</sub>, 5 ppm sodium dichromate

Temperature - 87° C

No purge water added

Velocity - 30 ft/sec

Analysis:

	<u>Fe</u>	<u>Al</u>
Initial	.02 ppm	.05 ppm
After 6 days	.03 ppm	12.33 ppm

Scale Formation:

After 6 days, scale resistance = 36 - 41 units  
(corresponds to a coefficient of 24,000 - 28,000 BTU/(hr)(ft<sup>2</sup>)( F)

After 9 days, scale resistance = 60 - 71 units  
(corresponds to a coefficient of 14,000 - 16,700 BTU/(hr)(ft<sup>2</sup>)(°F)

The tube lost 10.6 grams in 9 days from 1.5 ft<sup>2</sup> exposed to the hot water. A corrosion rate of 3.6 mils/month is indicated by this weight loss.

The test was stopped after 9 days by pump failure.

The results of this test indicate a very high corrosion rate and a moderately high rate of scale formation. By the nature of the test it was impossible to follow the rate of scale formation from day to day. There were no indications that the scale formation rate had decreased with time under the conditions of this experiment.

A beryllium tube has been installed and has been operated for over two weeks at an estimated average wall temperature of about 87° C, and a water velocity of about 30 ft/sec under a variety of water conditions. Thus far no scale has formed on the tube, as evidenced by a constant over-all coefficient of heat transfer and visual inspection of the water-cooled surface of the tube. No pits can be seen. Beryllium appears to be as impervious to scale formation caused by corrosion as stainless steel.

## 2.22 Heat Dissipation after Shutdown

Recently an experimental program has been started for the measurement of heat dissipation from fuel assemblies under emergency shutdown conditions in which there is no forced circulation of cooling water. It is estimated that after shutdown of the High Flux Pile heat production of the order of less than 8 per cent of the normal operating power will continue for sufficient time to melt the fuel assemblies provided no heat is removed. Further, the heat transfer characteristics may be such that excessive temperature differences may occur between the fuel assemblies and the residual cooling water. The purpose of the experimental work is to measure the heat transfer characteristics of the fuel assemblies with natural convection of the residual cooling water; and to provide information for the design of the reactor; so that adequate cooling is maintained in case of failure of the cooling water circulation system.

The experimental equipment is designed to duplicate as nearly as possible conditions of heat transfer and fluid flow in one channel of the fuel assembly. The heat transfer apparatus contains two electrically heated stainless steel plates with dimensions corresponding to those of the fuel plates. Demineralized water is held in the space between the plates. Power is supplied to the plates from a 50 KVA, 6000 ampere variable transformer. A temperature control is provided to shut off the power in case of overheating of the plates in order to prevent the "burn-outs" often associated with this type of apparatus.

Experimental data will show conditions of water flow necessary to provide the heat fluxes expected in the fuel assemblies for various conditions of reactor operation. Measurements will include: water conditions entering and leaving the apparatus; pressure drop through the channel; plate temperatures; and power input to the plates. Tests will be conducted both with and without outside free circulation. Theoretical calculations are also being made.

The first heat transfer apparatus designed and constructed according to the principles described above has been subjected to "shakedown" tests. These tests have shown that the method of mounting the plates allows buckling and consequent short-circuiting with severe operating conditions, and that the scheme for measurement of plate temperatures by use of thermocouples must be improved. The apparatus has been redesigned to eliminate these difficulties and is now being rebuilt.

### 2.23 Thermal Stresses in Beryllium Reflector

An experimental investigation has been conducted to determine the allowable heat fluxes and thermal stresses in the beryllium reflector of the high flux reactor. It has been considered in establishing the temperature and stress distributions in the beryllium reflector that the assemblies were composed of a number of cylindrical heat drainage areas, each area surrounding a cooling hole. The experimental models used represented the case of a long cylinder heated uniformly along the outer surface with the heat removed from the surface of an axial hole. Two beryllium cylinders, one 8 inches long with a 1.1 inch outside diameter and 0.125 inch inside diameter and one 5 inches long with a 1.6 inch outside diameter and 0.125 inch inside diameter, were tested. Heat was supplied to the outside surface of the cylinders by induction heating and the surface of the axial hole was water cooled.

A test model was subjected to thermal conditions 4.5 times more severe than expected in an operating pile and no detectable failure occurred. The 5 inch long test piece was subjected, further, to 50 cycles of alternate heating to 4.5 times the pile heat flux level and then cooling and no failure occurred. The 8 inch long test cylinder was not cracked or damaged after 100 cycles where the heat flux level was 3.2 times the expected pile level. Thermal stresses set up in the beryllium pieces were also greater than is expected in an operating pile by factors of 4.5 and 3.2 respectively.

### 2.24 Bending of Fuel Assembly Plates

An investigation was conducted to determine the dimensional stability of fuel assembly active plates when pressure was applied uniformly over the curved surfaces. A uniform pressure was produced in the interior of the test assemblies by means of nearly static water pressure. Measurement of dimensional changes of the plates was done using a reference plane technique. Critical bending pressure was considered the pressure at which the curvature of an active plate was reversed.

A uniformly distributed pressure of 23 - 25 psi is sufficient to reverse the curvature of a fuel assembly active plate when the pressure is applied to the convex side. A similar pressure on the concave side produces a deflection of 0.007 inch. Pressures of 10, 15, and 20 psi applied to the convex side of a plate in an assembly produce maximum deflections of 0.006, 0.017, and 0.035 inches respectively. The maximum permanent deformations at corresponding pressures are 0, 0.011, and 0.024 inches respectively.

These data when combined with the data concerning the hydraulic forces that can occur, indicate that the fuel assemblies as now designed are of adequate strength

2.25 Assembly Bending Tests

A series of tests were conducted on a fuel assembly to determine its rigidity when considered as a beam supported at the two ends and subjected to a uniform load along the length. Rigidity of the assembly was investigated when uniform pressure loads were applied, first, in a direction parallel to the surface of the active plates and, second, in a direction normal to the surface of the active plates.

The apparatus consisted of a typical aluminum assembly mounted on supports set 39 inches apart. In the experimental work a concentrated center load, applied by means of a Baldwin-Southwark testing machine, rather than a uniform load was utilized for convenience. The concentrated center load was then converted into an equivalent uniformly distributed load necessary to produce the same maximum stress and deflection. Deflection measurements were indicated by dial gages which were mounted in a reference stand. Load and strain data were obtained from the testing machine and SR-4 resistance wire strain gages.

Results of the tests showed that a fuel assembly acts as a beam when bending under a load applied either parallel or normal to the surface of the active plates. In both cases maximum deflection and stresses occurred at the longitudinal center of the assembly.

For case (1) in which loads were applied parallel to the surface of the active plates, a uniform pressure of 12 psi caused permanent deflection. Total deflection at this pressure was 0.043 inch. Uniform pressures of 8, 11, 16 and 18 psi produced deflections of approximately 0.028, 0.040, 0.080, and 0.100 inches respectively.

For case (2) in which loads were applied normal to the surface of the active plates, a uniform pressure of 13 psi caused permanent deformation. Total deflection at this pressure was 0.034 inches. Uniform pressures of 9, 16, 19 and 20.5 psi produced deflections of approximately 0.026, 0.054, 0.073 and 0.107 inches respectively.

2.26 Electromagnet for Shim-Safety Rod

The final results of tests on the #2 electromagnet (with SAE 1010 steel) are summarized in the data presented in Figures 1 through 5. The design requirements for a magnet which will release in less than 0.030 seconds at 200 milliamperes current with a 0.003 inch non magnetic shim while supporting a 600 lb. load are not satisfied by magnet #2. As explained in the last quarterly report magnet #2 was not constructed to design specifications in some important respects. These faults may be corrected in magnet #3 (with Permadrur laminations), still under construction. Difficulties have been experienced in insulating core laminations, in winding satisfactory pole coils, and in sealing the magnet within its stainless steel can.

In an operating pile magnets will release rods when power level exceeds 1.5 normal operating power or when a scram signal is received from safety circuits not related to power level (e. g., period meters, water flow). The operation of these circuits has been described by various Physics Division reports and will not be discussed here. The fastest release might be required at very low power levels when magnets are energized with maximum current and therefore are exerting maximum holding force. It is these conditions for which magnet #2 is inadequate because its release time is 40 to 50 milliseconds.

Several means of reducing this time are available as may be seen on Figures 1, 3, 4, and 5. Figure 1 shows that the use of a laminated armature on the control rod effects a 10 millisecond reduction. The design of the control rod is being altered to accomplish this change. Figures 1 and 4 show that a maximum current of 150 milliamperes instead of the 200 now specified would permit a 15 millisecond reduction in release time with substantially no reduction in holding force. The realization of this saving involves a change in the design of electronic supply circuits which may not be feasible.

Figure 3 and 5 indicate that release time may be reduced at the expense of holding force by increasing the thickness of the air gap between the magnet and the armature face. The allowable reduction in holding force will be determined in a hydraulic mock-up now under construction. If it is found that a 800# holding force is sufficient to support the safety rod under any operating condition, a shim thickness of .010 inch could be used and a reduction of about 20 milliseconds should result.

Figure 2 shows how a gradual reduction of holding force causes a reduction in release time. This would be the situation if the pile power level increased from N.F. to 1.5 N.F. over several milliseconds.

Further work with the shops will be done with the object in mind of improving the insulation between laminations and between turns on the coil. Magnets manufactured to date do not meet design requirements in either respect.

#### 2.27 Testing of Shim-Safety Rod

Installation of a full size thorium-uranium shim rod is now complete except for the electromagnet on the drive mechanism. Operation of the rod will begin as soon as the magnet is ready.

The actual weight of the shim rod is 125 lbs. However, the effective weight of the rod installed with bearings and submerged in water is only 50 lbs. The supporting force of the bearings was measured as ~50 lbs. (a friction coefficient ~.2 between the graphite and stainless steel) and the water buoyancy calculated as ~25 lbs. When a rod is being withdrawn, the bearing friction, of course, increases the force in

FIGURE 1

DATA # 1029

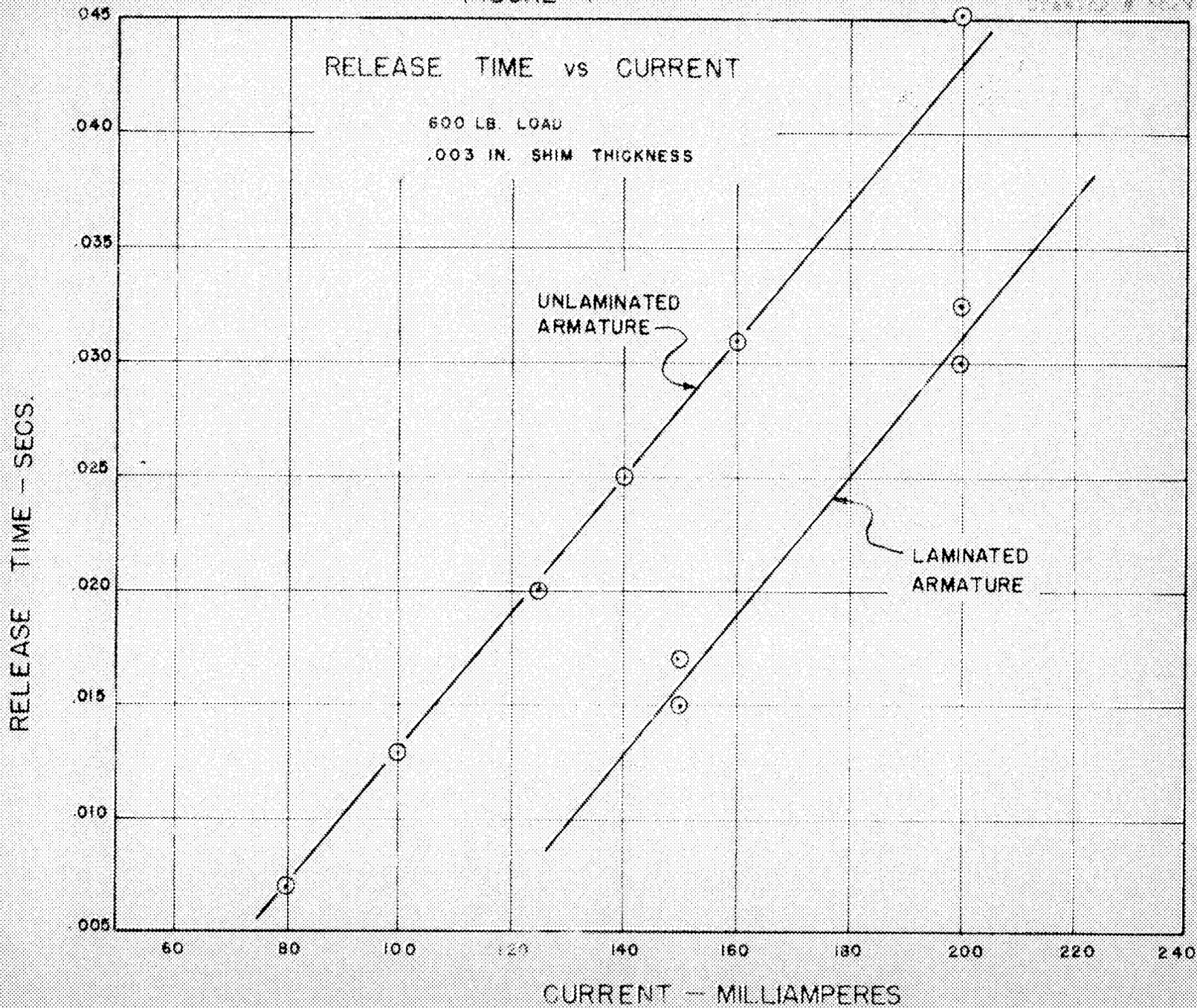


FIGURE 2

RELEASE TIME vs SCRAM SIGNAL TIME

INITIAL CURRENT 200 MA & 150 MA

UNLAMINATED ARMATURE, 800 LB. LOAD .003 SHIM THICKNESS

Drawing # 5630

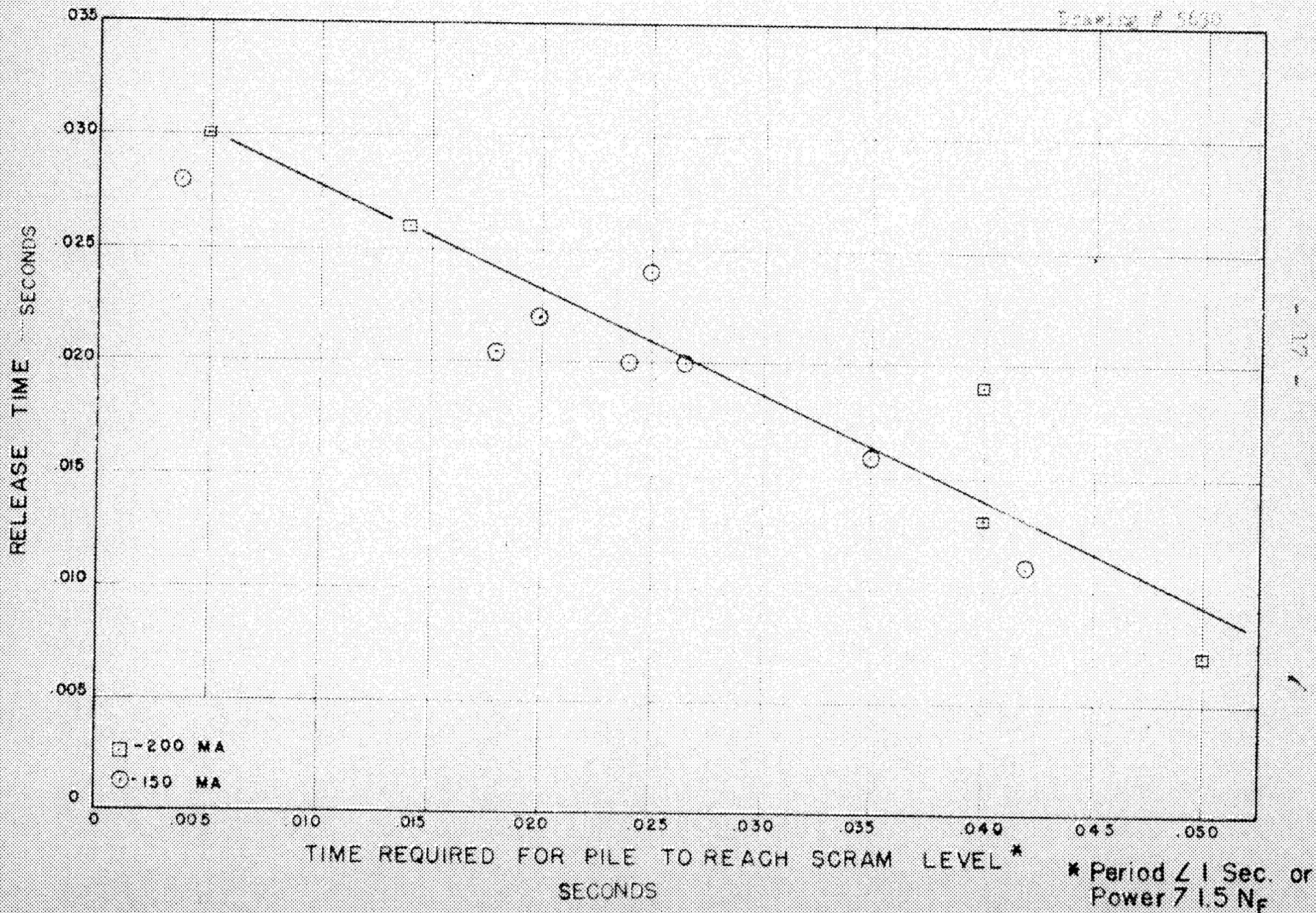
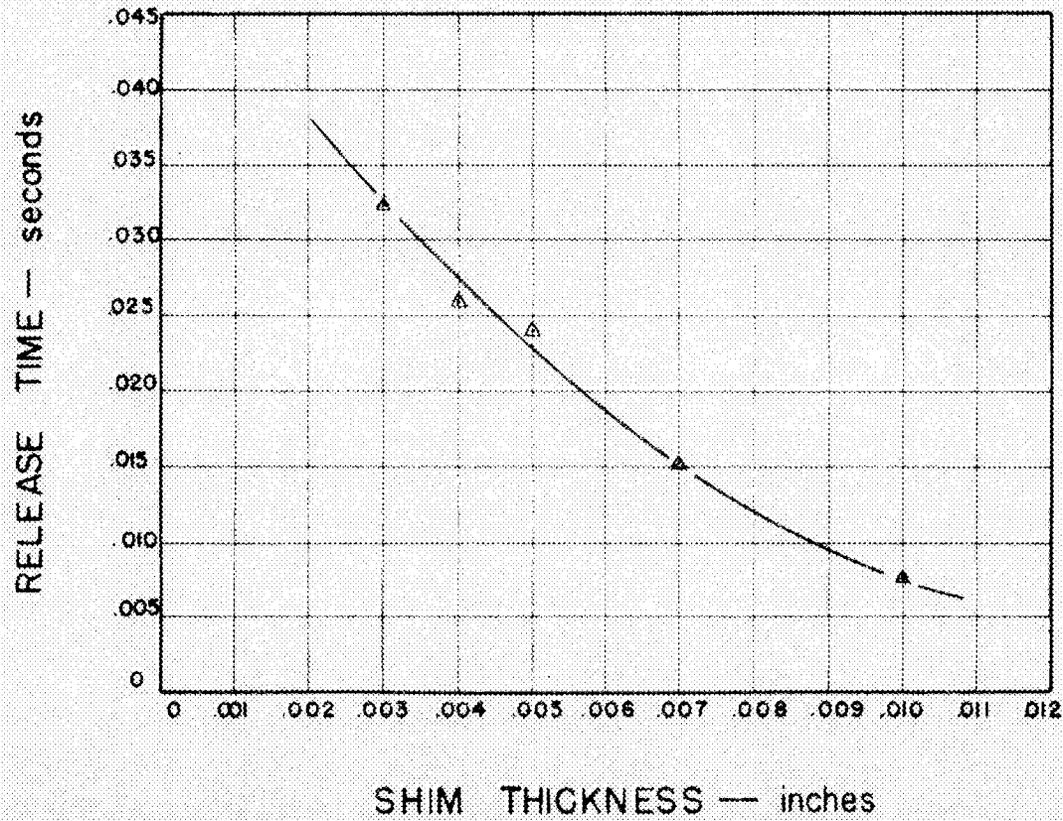


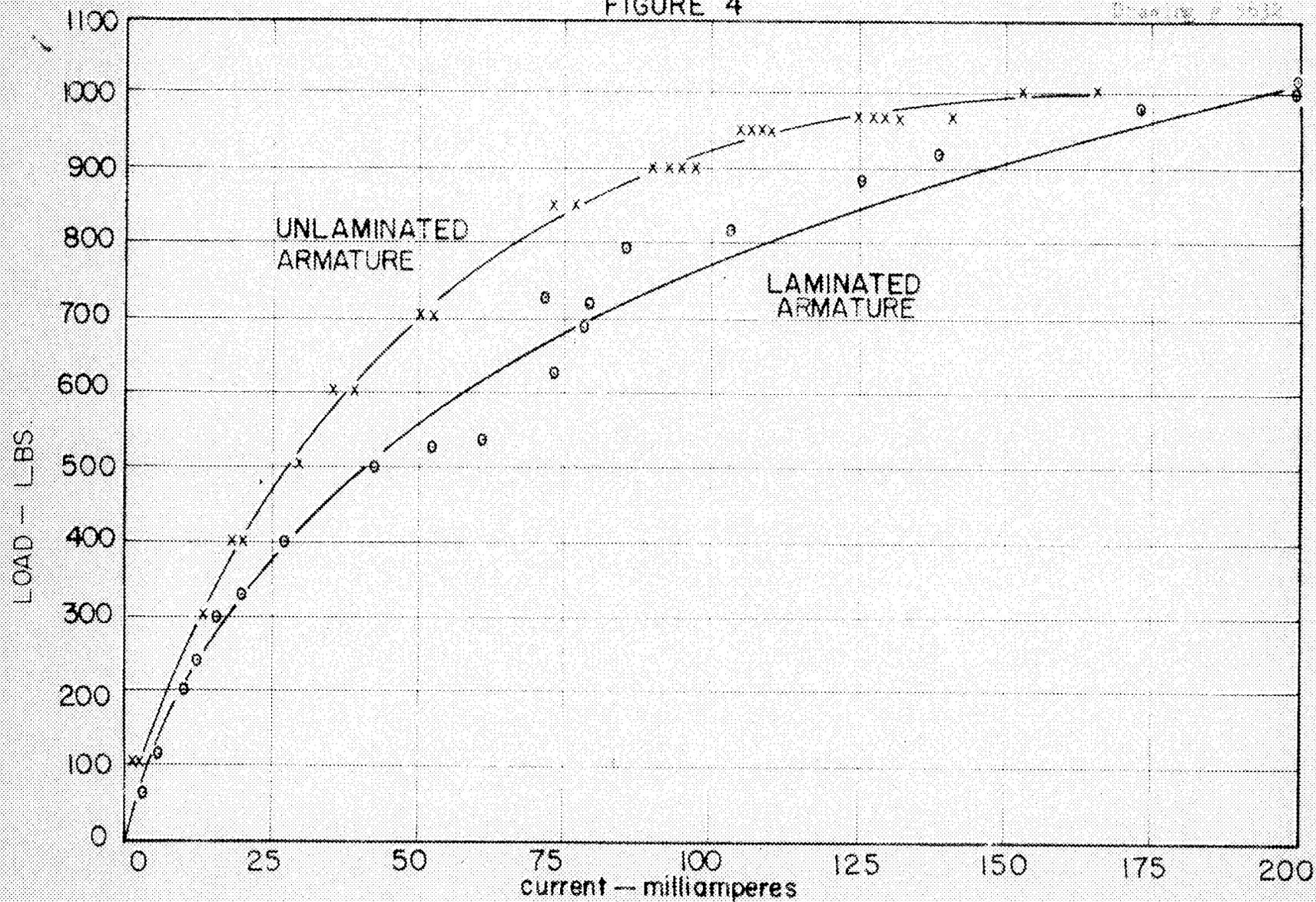
FIGURE 3  
RELEASE TIME vs SHIM THICKNESS  
150 M.A. — INSTANTANEOUS CURRENT DECAY  
600 Lb. Load



Scanned

FIGURE 4

Draw. No. 7 5632

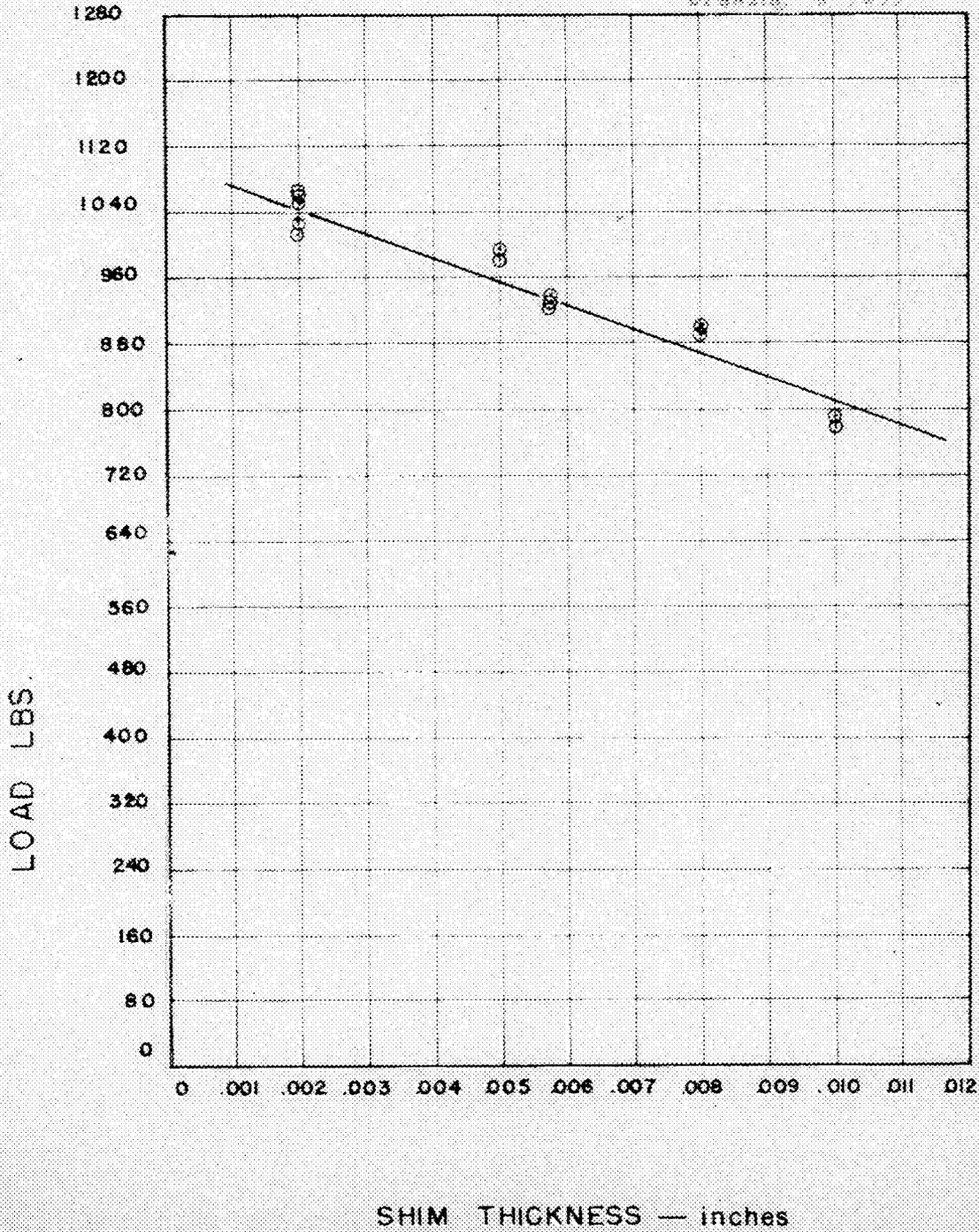


CURRENT vs LOAD  
003 MICA SHIM



FIGURE 5  
SHIM THICKNESS (AIR GAP) vs LOAD  
200 M.A. CURRENT

Drawing # 1633



opposition to the holding force of the magnetic clutch. Thus the total force opposing the magnetic force during rod withdrawal is:

Weight of Rod	125 lbs
Less water displaced	<u>25</u>
	100 lbs
Force of water at 50 psi pressure drop	410 lbs
Force due to bearing friction	<u>50</u>
	560 lbs

2.28 Mock-Up Construction

The J. A. Jones Construction Company has completed all foundations and footings for the mock-up. The brick work on both the switch-gear house and the control house is 50 per cent complete. The steel for the supports of the mock-up has been located but not obtained. The instrument panels for the control house are being designed. All instruments which were formerly located at various points around the mock-up area are being centralized in this control house. Both the circulating water pumps have been installed.

All large-dimension piping has been received but a review of coating resistance tests indicated the Amercoat #55 coating (supplied by prior specification with the piping) to be an inferior coating to other available Amercoats. It was decided that all pipe sections lying between the strainers and the mock-up should be re-coated with Amercoat #33. Inquiries to the manufacturer resulted in assurances that such over-coating was quite feasible. The #33 seal coats applied to date have peeled in practice. It may be necessary to revert to the existing #55 coating.

It was planned to coat Tank "A" and various auxiliary pipe sections with the Amercoat #31-33 combination and all tanks and tank parts below the Tank "A" - Tank "B" flange (except for Al and Stainless parts ) with Amercoat #77, a thermal setting coating. In view of coating results to date, the entire question is being reviewed with adherence tests on field prepared specimens underway.

Procurement

All major tank sections have been requisitioned as well as Class II replaceable reactor aluminum assemblies, "A" pieces and various aluminum pieces. Purchase requisitions for all other items needed will be written as design is completed.

Operating and Maintenance Procedures

Experiments with a hand manipulated tool to remove fuel and replaceable aluminum assemblies are planned. Replaceable reflector pieces will be provided with tapped holes for the insertion of eye-bolts to facilitate replacement.

No firm decisions on tools and techniques necessary for pile operation and maintenance will be made, pending the outcome of these experiments.

Mock-up Dimensional and Stress Studies

Conception studies are underway on techniques required to obtain adequate dimensional and stress information on the mock-up reactor and structural members.

An acceptance inspection of the "D" tank will include measurements of diameter, and roundness. An inside caliper arrangement has been conceived which will rotate about a central shaft. The shaft will be located by 3 spokes which project to the tank periphery. The caliper arm will be equipped with dial gages to indicate measurements.

Grid plate dimensions and hole spacing will be checked with standard micron calipers. Flatness of the grid plate will be determined using available surface plates. Assembly spacing will be checked with Go-No-Go gages which will be designed following standard practices. During mock-up operation, dimensional measurement will be restricted to those outside the tank using reference plane techniques to measure changes in tank dimensions. Strain and stress measurements will be obtained from standard gages which can be located as desired.

Fluid Flow in the Pile Mock-Up

Of principle concern is the flow pattern in the reactor. It is desired that measurements be made that show the direction of flow and water velocity in all parts of the reactor, and that these measurements be made for all significant loadings of the pile.

In the active portion of the reactor it will be necessary to determine the total flow through each fuel assembly and each beryllium assembly, and to determine the flow in the gap between the assemblies. In the reflector it will be necessary to determine the flow through all of the holes and cracks, and especially through the larger spaces such as the holes which provide cooling water to the beam holes, and the annulus around both sides of the interlocking ring of beryllium. Measurement of the flow through the assemblies in the active section will be made with pitot tubes. The spaces between fuel assemblies and the cooling channels in the beryllium reflector are, however, too small to permit mounting pitot

██████████ T

tubes without restricting the flow. Flow in these spaces will be determined by pressure drop measurements, made with very small diameter surgical tubing (.03-.04 inches) which will be buried in the assemblies and emerge flush with the side. The tubes will be led out either through the top or bottom of the assembly. In some cases it may be necessary to bury as many as 30 tubes in one assembly.

Direction of flow will be determined by two means. First, enough static pressure points will be measured so that lines of equal pressure may be drawn for any cross section through the reactor. The direction of flow will then be perpendicular to these isobars and in the direction of decreasing pressures. Second, velocity measurements will be made at several points along a vertical line, so that by observing the increase or decrease in vertical flow the horizontal flow may be determined.

It is not possible at this time to give more than a general picture of the procedure for determining the flow pattern in the reactor because much of the design data are still lacking. It appears now, however, that it will be sufficient to make nearly all of the measurements in one quarter of the reflector section and it will not be difficult to make measurements over the entire active section. By rotating the loading in the active section, conditions in one quarter of the reflector can be made to simulate conditions in any other quarter of the reactor. Flow and static pressure measurements will be made at the following locations.

Active and beryllium assemblies: The total flow through each assembly can be measured by installing pitot tubes up through the monitoring tubes into the lower end boxes. Static pressure tubes for measuring flow between the assemblies will need to be mounted on all four sides of about three of each type of reactor assembly. These assemblies can then be moved to all locations in the active section. It will be desirable also to measure static pressure at the entrance and exit of the fuel and beryllium assemblies, and to determine end effects on the assemblies.

Annulus on both sides of the Interlocking ring of beryllium: Static pressure taps mounted at about four elevations on the beryllium pieces can be used to measure the flow and change in flow. It will be desirable to make these measurements at frequent intervals to get a good picture of the direction of flow.

Cracks between the Reflector Beryllium: Static pressure taps mounted at four elevations on all four sides of about 30 assemblies should give a good cross section of the flow in one quarter of the reflector.

Holes in the Beryllium: Two pressure taps on one or two holes in each of the 30 pieces mentioned above should be sufficient.

Cooling Channels to the beam holes and annuli along the beam holes: All of these measurements will be made by means of pressure taps mounted in the adjacent beryllium.

██████████

Space between the Reflector and the Tank wall: This will be measured by pressure taps mounted in the beryllium. A measurement at every other assembly along the tank should be adequate. Pressure measurements are to be made at several elevations as previously mentioned.

Pneumatic tubes and Regulating rods: These flows can be measured in the same way as those in the small holes in the beryllium.

Plenum chamber below the beryllium: It is questionable that any flow measurements are necessary, but pressure drop across the chamber will be determined.

Traverse of flow just above the Reactor: This is thought to be unimportant, but can be measured with pitot tubes if desired.

The location of most of the above mentioned pressure taps and pitot tubes has been tentatively selected. Letters of inquiry have been sent out on the surgical tubing, manometers, connectors, and other equipment which will be needed. An experimental setup will be designed for calibrating the pitot tubes and the individual assemblies for velocity by pressure drop measurement.

## 2.3 Corrosion of Reactor Materials

### 2.31 Laboratory Tests (J. A. Kyger)

Extruded Beryllium: Investigations using sodium nitrate as a corrosion inhibitor for beryllium when exposed to static simulated pile operating conditions were completed. The results of these tests were as follows:

- 1) Low nitrate concentrations (2-5 ppm) were beneficial in that they tended to suppress general corrosion attack but were not completely effective in reducing localized attack.
- 2) High nitrate concentrations (10-40 ppm) reduced localized attack to negligible values but at the same time stimulated the general corrosion rates of the material.

A study using sodium nitrate under dynamic conditions of test is being conducted.

2S Aluminum: Static tests were conducted using sodium dichromate in concentrations of 10-40 ppm at 0.0005M hydrogen peroxide and pH 7.5-8.0. The corrosion rates for the aluminum increased with increasing dichromate concentrations. Pitting attack, however, was negligible in all cases.

Static tests were also conducted using concentrations of 10-40 ppm. of sodium nitrate and sodium pyrophosphate at pH 6 as corrosion inhibitors. In both cases, localized attack was more effectively reduced at the higher concentrations. The results from the sodium nitrate tests were more favorable than those from the sodium pyrophosphate or the sodium dichromate at these concentrations and testing conditions. At 30-40 ppm. of sodium nitrate, the results compared very favorably with those obtained using the generally accepted conditions of 5 ppm. sodium dichromate at pH 7.5-8.0

Panels of 2S aluminum were subjected to an Alodizing treatment which produced a chromic-phosphoric type of protective film. These panels were exposed to static simulated pile operating conditions with excellent results. Corrosion rates were much lower than rates obtained with untreated aluminum specimens exposed under similar conditions. This type of film appears to offer good possibilities for corrosion protection of aluminum, dependent upon the effects of irradiation on the film and its effect upon heat transfer. More extensive testing is being conducted with the Alodizing process.

No. 43 Aluminum Casting Alloy: Corrosion rates for this material were found to be much greater under dynamic conditions than when exposed to static conditions. Localized attack was quite severe in the former case. The use of 5 ppm. of sodium dichromate at pH 7.5-8.0 under dynamic simulated pile operating conditions was effective in reducing corrosion rates from 0.5 mils/month to 0.2 mils/month.

Sodium nitrate in concentrations of 3-5 ppm. at 0.0005M hydrogen peroxide under static conditions appeared effective. Corrosion rates of 0.005-0.009 mils/month were obtained as compared to 0.14 mils/month in uninhibited testing solutions.

347 Stainless-2S Aluminum Contact Couples: Sufficient test data have been collected on exposures of this couple to static pile conditions to state that the presence of a thin film of metallic zinc on the 2S aluminum acts as a retarder of galvanic corrosion. In all cases, corrosion attack of the aluminum was favorably reduced. Future tests are planned under dynamic conditions.

### 2.32 Engineering Tests (C. E. Winters)

Pierced Assemblies: Tests on a specimen of clad alloy consisting of a sandwich of approximately 0.020 in. 2S aluminum, 0.020 in. U-Al alloy (20% U), and 0.060 in. 2S aluminum indicate that pitting failures in the protective cladding will not cause serious corrosion effects in the alloy. The surface clad with 0.020 in. aluminum was exposed to demineralized water (0.005 molar in H<sub>2</sub>O<sub>2</sub>, pH 6, 39 to 51° C) circulating with a velocity of 20 ft/sec while the other side was heated with steam to produce a heat flux of 0.4 to 0.5 kw. per sq. in. across the plate. The depth of several small holes drilled through the cladding on the water side did not appreciably change after a 29 day exposure. No blisters were observed between the layers of metal, and no appreciable mushroom effect in the alloy layer was seen when the holes were examined.

2S and 43S Aluminum Interfaces: Tests conducted on various aluminum interfaces indicate that little or no seizure due to the formation of corrosion products will occur between the lower grid of 2S aluminum and the 43S aluminum bosses on the adapter of the fuel assembly in the proposed high flux reactor. Assemblies of 2S and 43S aluminum blocks, some of which were chemically treated to promote the formation of oxide films, were exposed for 40 days to stirred demineralized water (55° C, 0.005 molar in H<sub>2</sub>O<sub>2</sub>, pH 6). The various chemical treatments used are described in the handbook, Finishes for Aluminum, from the Reynolds Metal Company. At the conclusion of the test, no appreciable amount of seizure was encountered between any of the interfaces. However, the surfaces treated by Alrok and Bauer - Vogel processes were freer from corrosion products than the others. Additional information will be obtained from the operation of the mock-up.

Impure Beryllium and Q Process Beryllium: A Q Process (Powder Metallurgy Process used by Brush Beryllium Corporation) beryllium mock-up section of the proposed pile reflector has been tested for a period of 122 days under simulated pile conditions. The average expansion between interfaces parallel and perpendicular to the direction of flow was 0.0052 and 0.0113 mils/interface/day respectively. The differences in the expansion rate of vertical and horizontal interfaces may be attributed to differences in the tension in the bolts holding the pieces in contact and to effects attributable to the flow. The cooling channels remained free from corrosion products.

Tests on beryllium with inclusions indicate that this material should not be used under water in reactors where close tolerances are required or where it cannot be readily replaced. Specimens of poor quality extruded beryllium were exposed for 30 days to demineralized water (92° C, 0.005 molar in H<sub>2</sub>O<sub>2</sub>, pH 6.2) circulating at velocities of 10 and 20 ft/sec. At the conclusion of the test, all the specimens were covered with numerous pits accompanied by the formation of barnacles. Maximum depth of the pits was 18 mils. In many instances, thin slivers of metal adjacent to pits were raised to a maximum of 0.010 in. above the surface of the specimens. It is believed that attack was initiated by water entering small holes in the metal surface causing electrochemical corrosion to proceed around the holes. The accumulation of voluminous corrosion products in these pits pushed the metal above the surrounding surface. The average overall corrosion rate was 0.030 and 0.035 mils/mo. at water velocities of 10 and 20 ft/sec.

Effects of Irradiation on Corrosion of Beryllium: Specimens of extruded beryllium made prior to December 1, 1947 and extruded before the latest techniques were developed, were placed in the aluminum water tube in pile hole No. 19 in order to determine the effect of irradiation on corrosion of this material. A total of 12 specimens, 1.35 in. diameter and 4.00 in. long, were positioned in the center of the tube with aluminum spacers and located at various distances from the entrance of the

tube into the pile. Three of the specimens were located in the concrete shielded section of the tube at the entrance, six were located near the center of the pile, and the remainder were situated in or near the shielded section at the exit end of the tube.

Exposure conditions were checked continuously for 63 days and are listed below:

Demineralized water analysis (average)

Al content, entrance	0.05 ppm
Fe content, entrance	0.04 ppm
pH, entrance	5.1
pH, exit	5.2
H <sub>2</sub> O <sub>2</sub> exit	0.12 ppm (0.2 ppm, max.)
Water Velocity	
Past specimens	4.7 ft/sec
Through tube	0.9 ft/sec
Water temperature, entrance	90 to 95° C

The water velocity in the tube was regulated so that the water would receive about the same exposure as water moving at 30 ft/sec in a flux of  $10^{14}$  neutrons/cm<sup>2</sup>/sec.

At the conclusion of the run, the general surface appearance of the beryllium was unchanged except for numerous small pits (approximately 20 per sq. in.) whose maximum depth was 8 mils. However, one of the specimens located in the exit shielded section of the tube was ruptured longitudinally for a distance of approximately 5/8 in. and to a maximum depth of 1/8 in. Metal adjacent to the rupture protruded to a maximum of 1/8 in. above the surface of the specimen. Apparently, the rupture was caused by corrosion which proceeded around impurities initially present in the metal. The average defilm corrosion rate for all the specimens was 0.06 mils/mo. Although the specimens located at the center of the pile were subjected to a neutron bombardment more than twenty times greater than those in the shielded section of the tube, no significant variation in the corrosion rate or severity of the pitting attack was observed.

TABLE I

<u>Location in Pile Tube</u>	<u>Flux, neutrons/cm<sup>2</sup>/sec</u>	<u>Defilm Corrosion Rate, Mils per Mo.</u>
Entrance end	3 x 10 <sup>10</sup>	0.0608
Entrance end	3 x 10 <sup>10</sup>	0.0584
Entrance end	3 x 10 <sup>10</sup>	0.0612
Center	7 x 10 <sup>11</sup>	0.0676
Center	7 x 10 <sup>11</sup>	0.0636
Center	7 x 10 <sup>11</sup>	0.0597
Center	7 x 10 <sup>11</sup>	0.0597
Center	7 x 10 <sup>11</sup>	0.0604
Center	7 x 10 <sup>11</sup>	0.0683
Exit end	1 x 10 <sup>11</sup>	0.0711
Exit end	3 x 10 <sup>10</sup>	0.0637
Exit end	3 x 10 <sup>10</sup>	0.0476*

\*Specimen ruptured

2.4 Metallurgical Development (J. A. Kyger)

2.41 Fuel Assemblies

Melting and Casting U-Al Alloys: Satisfactory procedures for melting and casting these alloys have been described in the "Preliminary Process Manual for Fabrication of U-Al Fuel Rods" by W. L. Cockrell. Efforts are currently devoted to refining this process and determining the effect of previously unstudied variables.

The following conclusions have been reached:

- 1) Recovery of uranium in the ingot increases with increasing uranium concentration of the ingot.

<u>% U</u>	<u>% Recovery in Alloy</u>
5	95.7
10	95.8
15	96.5
20	96.4
25	97.5

- 2) Recovery also increases with the size of the melt. (The following figures are for 18% U.)

<u>Melt Size, Gms</u>	<u>% Recovery</u>
250	95.6
500	96.6
1000	98.3
1500	98.5
2000	98.4

- 3) The mold shape should be such that the ingot width is not less than  $1/2$  the length, otherwise excessive piping occurs.
- 4) Ingot quality improves as the mold thickness increases from  $1/2$ " to 1".
- 5) A holding time of ten minutes with frequent stirring appears satisfactory, longer times give no improvement.
- 6) Piping is not reduced by the use of a hot top, but is reduced by using a flared head on the mold.
- 7) Operating technique is the most important variable.

Radiographs of 20, 30, and 40% U-Al ingots showed little segregation as cast. Rolling produced considerable random segregation in the 20% material, but not in the higher alloys.

#### Cladding U-Al Alloys

Work has continued on improving the quality of the aluminum clad uranium aluminum alloy sheets for the fuel assemblies. It is possible to make satisfactory sheets by hot rolling the alloy between two sheets of aluminum without the use of a frame around the alloy core, but operational difficulties make this method undesirable.

In the past, when an aluminum frame has been used around the core, blisters frequently occurred, after cladding, at the ends of the core. This situation has been considerably improved by the use of a punch and die for blanking frames and cores. The fit of the two mating pieces is this much better than can be practically obtained by machining the parts.

In addition, a tighter ultimate fit can be obtained by starting the rolling operation with the core material slightly thicker than the frame. The first pass then forces the core out against the frame. A series of plates fabricated with various thicknesses of core and frame indicate that optimum conditions obtain when the core is about 10% thicker than the frame. A greater differential gives poor edge bonding with the cladding sheets. It was also indicated that smaller percentage reduction per pass gave fewer blisters. With a core of 0.090" and frame and cladding of 0.082" and 10% reduction per pass, blisters were practically eliminated. However, multicore sheets rolled using these techniques still yielded 50% blistered plates.

A study of bond strengths on many plates clad by various techniques has led to the following conclusions:

- 1) In general, the edge bonding is least strong due to ready relief of roll pressure through metal flow. This implies the use of reasonably wide sheets; subsequently, to be sheared to size.
- 2) Bond strength can be tested in the sheared borders by severe flexing or prying apart. Regardless of bond strength the interface of the cladding sheets is revealed by caustic etching.
- 3) When no frames are used, the core edges are rounded. With frames and cores of the same dimension the edges are square. When the cores are thicker than the frames the core edge is somewhat concave.

A radiographic study of 27 clad plates was made to determine the dimensional variations. Three characteristics were measured; length, width, deviation from straightness per foot of length. The median values (such that half the plates are better, half poorer) are as follows:

Length	0.16"
Width	0.006"
Deviation/ft.	0.016"

From these results, it is felt that little difficulty will be encountered in meeting design specifications in these respects.

It is planned to start a production run on June 1, making four six-core sheets per day. By August 1, enough plates will have been turned out for 30 assemblies assuming a yield of 50% as satisfactory finished assemblies. Unsatisfactory assemblies will be sent to Semi-Works for dissolver studies.

To date, it has been planned to vary the uranium content of the core as enrichment decreases with irradiation time. Recent results, however, indicate that it may be advisable to vary the core thickness instead, keeping the uranium content at its present practical maximum of 25%. Benefits resulting from this procedure are described under "Melting and Casting", above. Operation-wise, this proposed method would be no more involved than the one considered previously.

#### Scrap Recovery

Little progress has been made on this phase of the work during the last period due to the fact that no suitable material has been found to resist the boiling  $\text{HNO}_3$  - HF mixture used in the leaching step.

The first step in the proposed process calls for dumping the molten slag into water, a step which leaves the slag in a very porous and easily leachable form. However, several mild explosions have resulted and a new type of spray tank must be designed to carry out this operation safely.

#### Assembly Fabrication

##### 1) Cleaning

The hot caustic dip previously used to clean assembly parts has a severe corrosive action and several mils of metal are usually removed from the surfaces during cleaning. Cleaning compounds (containing inhibitors), sold by the Oakite Company, show considerable promise and have yielded satisfactory brazed joints.

##### 2) Flux

Experiments on Eutectic No. 190 flux indicate that it may be possible to halve the lithium content if necessary. In a series of tests on the action of LiCl fluxes it was found that high lithium fluxes containing small amounts of etchants such as cryolite, zinc chloride, etc, produce sound joints. There is a possibility that fluxes of this type may reduce flux entrapment. Additional work along these lines depends upon the results obtained from current chemical and neutron absorption tests on joints made with No. 190 flux. Indications are that the present flux may be satisfactory. Chemical analysis indicates 0.002% Li by weight in the brazed assembly and this is believed to be acceptably low. Further tests are in progress.

3) Jigging

Conclusive flux exposure tests on various suggested ceramic materials during this period have resulted in the selection of a magnesium aluminum silicate, Alsimag No. 202.

The following properties have been investigated and have been found satisfactory for our purposes:

a) Resistance to Thermal Shock

This material differs considerably from Lava Grade A in this respect. Alsimag No. 202 resists the shock resulting from water quenching from a temperature of 600°C. Lava Grade A will develop cracks when subjected to such thermal conditions and may even crack during heating in contact with metal surfaces.

b) Resistance to the Attack of Aluminum Brazing Fluxes

Many ceramic materials show a tendency to structural breakdown under the attack of molten flux. Alsimag No. 202, after twenty hours' continuous exposure to molten flux, was not affected. In addition, ten complete brazing operation cycles failed to alter the properties of the ceramic. The cycle included washing, drying, exposure to alcohol-flux slurry, drying, and furnacing at 600°C for forty minutes. Lava Grade A shows tendencies of structural breakdown under these conditions.

c) The Alsimag No. 202 shows dimensional stability.

d) Effect on Brazing Aluminum

A joint brazed adjacent to Alsimag No. 202 was entirely satisfactory.

The complete jigs are being fabricated by American Lava Corporation. Contact with the firm has established May 29, 1948 as delivery date for these units. Jigs are being procured for two sizes of units, the fuel rod and the control rod sections.

4) End Boxes

Purchase Requisition No. 90177, for 56 end boxes (adapter castings) was issued on March 23. This order was urgent at the time, since the pile mock-up requires assemblies equipped with such castings. Considerable machining must be done on these castings before they can be fitted to our assemblies. Consequently, we must have trial castings before the vendor's foundry procedure can be approved. After conference with the Purchasing Department, a suitable vendor was selected for these items. The first two castings should be received about June 30, 1948.

Alodizing

From time to time consideration has been given to protection of the assembly against corrosion by some type of anodizing. Electrical methods are considered impractical due to the geometry of the assembly. Recently, tests on samples "Alodized" by the American Chemical Paint Company have yielded very good results. Ingredients for this treatment have been obtained and an assembly section has been "Alodized". It will be tested against a similar but untreated section.

2.42 Control Assemblies

Thorium Cladding

1) Purity

Previous reports have mentioned difficulties experienced in the rolling and curving of al clad thorium sheets. According to recent literature,<sup>(1)</sup> thorium is about as soft as annealed copper, having a Rockwell hardness of B-10. Our samples of rolled thorium show a Rockwell hardness around B-65. Annealing does not alter this value significantly and the increased hardness must be due to the presence of impurities. This is revealed by laminated structures in the rolled sheets

(1) G. Meister "Production of Thorium, Zirconium, and Uranium", Metal Progress, Vol. 43, No. 4, April 1948, p. 515-520.

and by darkened surfaces produced by acid pickling. Analyses in project reports have shown presence of small amounts of carbon, iron, aluminum, and considerable oxide. A sample checked recently by the spectrographic laboratory showed small amounts of copper, beryllium, and calcium; but no aluminum or iron. Carbon and oxygen were not determined. Carbon has been detected in other samples by the acetylene odor produced by water after treatment with molten AgCl.

2) Cladding

A total of nine sheets of thorium (0.200" x 2-1/2" x 10-1/2") were drilled with thirteen uniformly spaced 1/16" holes, arranged in three rows along the length. These were pickled in a boiling solution of 50% HNO<sub>3</sub> and 1% HF to remove the oxide coat and then dipped in an aluminum bath at various temperatures from 700°C to 900°C. It was found that immersion for 1/2 minute at 700°C gave a uniformly thick coat and also filled up the holes. Higher temperatures resulted in incompletely filled holes.

These dipped sheets of thorium were then fitted with 0.180" frames and 0.175" cores of 2S aluminum. Frames, covers, cores were all scratch brushed and the sheets rolled at 1000°F to 0.200". In several cases, the ends of the thorium cores broke through the cladding metal. Also, the cover sheets were poorly bonded to the frames. It is believed that a greater total reduction in thickness will solve the problem of poor cover to frame bond. Since the only available thorium is a supply of 0.200" thick plates, an order has been placed with the Atomic Energy Commission for thicker plates. When these arrive, additional rolling tests will be made.

Since the rolling of the sandwich fails to reduce the thorium at the ends, the cover sheet eventually breaks at this point. One alternative which seems practical is to have the thorium longer than required, cutting off the ends after cladding. The sandwich can then be sealed by Argon Arc welding; or an aluminum extension can be added to the plate by the same technique.

SECRET

3) Intermediate Silver Layer

In checking the possibility of silver as an intermediate layer between thorium and aluminum, attempts were made to deposit silver from a molten sodium chloride bath containing silver chloride. Results were inconclusive. Previously, attempts were made to electroplate silver on thorium but the results were not satisfactory.

To check the bonding of silver to aluminum a sample of alclad silver was prepared. The layers could be pried apart with pliers. Modified techniques might improve the bonding.

4) Powder Metallurgy

The entire problem of cladding the thorium sheets might be eliminated if a powder alloy could be made having an aluminum matrix and containing a large percentage of thorium as discrete particles imbedded therein. The Research and Standards Branch of the Bureau of Ships has had made some iron-aluminum powder alloys as a stand-in for Th-Al alloys, but results are as yet inconclusive.

Thorium Production

A large scale batch of thorium was rolled to rods by Westinghouse and has now been received. This batch contains material made by various processes similar to some small scale samples that were received some time ago. The small samples show no appreciable difference in neutron absorption, regardless of the purification process. If the same results are obtained on this production size run, a considerable saving can be realized in subsequent runs by eliminating part of the purification steps.

Thorium Assembly

Study is proceeding on the proposed design in which the clad thorium sheets are flat and the side plates are curved to fit the lattice. Although some extra machining is required in this design, no serious disadvantages have been found; and it may prove quite helpful not to have to bend the clad sheets.

Cadmium Assembly

The present design calls for a 0.020" cadmium foil to be wrapped around a square tube, inserted in a channel section, covered with a flat strip, and welded. Difficulty may be experienced with warping and cadmium melting or oxidation. A modified external closure consisting of two "L" sections may decrease warping. The possibility of casting the cadmium is being considered. A preliminary test has been made on thin annular sections 0.025" in thickness and 24" long. The lower portion showed fair penetration by the cadmium. Additional tests will be made. The possible use of cadmium alloys and proportionately wider insert spaces is being considered.

2.43 Beryllium Assemblies

Extrusions

Recent shipments of rectangular beryllium extrusions for the reflector are somewhat smaller in lateral dimensions than required. Specifications called for a 1-3/8" by 3-5/8" section, but after removal of the jacket the dimensions approach 1-1/4" by 3-1/2". This size is too small, particularly in the long cross sectional dimension. A slightly larger size is essential in order to insure removal of surface metal of questionable soundness. This will be discussed with Dr. Kaufmann of M.L.T.

Machining

During the past quarter, an experimental design for machine-fabricating the beryllium assemblies was proposed and tried. Initially, a full-sized Type TD-571, Class #2, with 1/8" diameter cooling water holes assembly was fabricated. Machining results indicate joining the beryllium slabs by bolting to be definitely feasible and practical. Machining tests have indicated that Carboloy tools tend to chip but that less hard and tougher tools are satisfactory. The use of light machines results in considerable chattering. Bolt threads are best out on a lathe and chased by male or female taps. Machine work on a second full-sized assembly, Class #1, with 0.080" by 1" slots, is underway.

Some cursory grinding tests were made on beryllium to determine if grinding is a suitable method of cutting the longitudinal cooling slots. Etching of the surface after grinding revealed the presence of minute thermal checks.

Some experimental work on the machining of beryllium has been started by the Sam Tour Company through the Bureau of Ships. Further work with this company will be discussed soon with the New York Office of the Atomic Energy Commission.

Brazing

The possibility of using phosphocopper as a braze for beryllium was checked some time ago. The beryllium can be "tinned" without the use of a flux by rubbing a heated sample with phosphocopper. The parts can then be joined by heating and pressing.

3.0 25 Separations and Development

3.1 Design Progress (J. A. Lane)

The recovery of enriched uranium and its separation from fission products is to be conducted in the 1200 Area. After decay the fuel assemblies will be dissolved, separated by three column solvent extraction cycles, the uranium precipitated, and refabricated in the 1400 Area.

During the past period the design of the 1200 Area has proceeded with emphasis on the design of the canal, recovery of rejects, solvent recovery, and waste disposal. The process drawings are being revised to include the latest pilot plant results.

3.11 Decay Storage of Irradiated Assemblies

Irradiated fuel assemblies will be stored in a canal for a period of approximately 145 days to allow for the decay of the 6.8 day beta emitting U<sup>237</sup> to a level safe enough for handling in metallurgical processing operations. The canal design is now being considered; its capacity will be slightly greater than the requirements imposed by the operation of the experimental pile with provision for enlargement to accommodate output of another pile. Considerable thought has been given to safety precautions in respect to this canal. Space arrangements will be designed to prohibit the location of an excess number of assemblies in one area. In addition, cadmium or another neutron absorber will be used to shield the assemblies. This design has not been fixed to date.

3.12 Recovery and Decontamination

The capacity of the irradiated fuel assembly processing area is outlined below. The figures shown are based on processing two fuel assemblies containing uranium of 60.3% isotopic enrichment per twelve hours.

Grams of 25	241
Grams of uranium, total	400
Grams of aluminum	9720
Grams of fission products	37.8
Column flooding rate	60%

Irradiated shim safety rods and control rods will be processed in the same equipment under identical chemical conditions. This basis for design will permit doubling the processing rate through the same equipment to accommodate an additional pile.

Chemical flowsheets using these capacities have been developed and issued for comment and correction. All chemical flowsheets have been so set up as to indicate a running material balance. In their present state the chemical flowsheets do not show the activity of the various process streams nor the overall uranium loss figures. These quantities will be added before the preliminary report is issued, after data on Hanford irradiated slugs have been obtained from current hot pilot plant operations. The chemistry has been changed to agree with current pilot plant chemical flowsheet conditions.

Equipment flowsheets for the continuous processing of irradiated assemblies showing tank capacity, instrumentation, service liner, and all process lines have been developed and are being checked.

### 3.13 Recovery of U from Rejects

The capacity of the reject processing cycle for 1400 Area reject material has been changed from two to one assemblies per day. For the purpose of flowsheet calculations, it has been assumed that this assembly will be fabricated of the most degraded uranium processed in the 1400 Area, i.e., an assembly which contains 219 gms of uranium of 65.2% enrichment and approximately 4600 gms of aluminum. This capacity is approximately 60% over that required by estimated wastes and rejects from the 1400 Area.

Because the size of the dissolver batch is only twenty gallons and since no remote operation will be required, it was decided to make the 1400 reject cycle a batch operation. In the selection of conditions for the batch extraction, advantage was taken of the high distribution coefficients ( $d_c = 100 \text{ org}$ ) for uranium from a 2.5 M.  $\text{Al}(\text{NO}_3)_3$  solution 0.2 M. in  $\text{HNO}_3$ . The high concentration of  $\text{Al}(\text{NO}_3)_3$  will be maintained by the addition of fluoride ion. Chemical flowsheets for this operation, TD-477 and 478, have been issued for checking.

### 3.14 Solvent Recovery

Facilities for virgin solvent pretreatment and used solvent recovery will be provided in the 1200 Area. Procedures now used in the hot pilot plant for pretreatment and recovery have been adopted. Virgin hexone will be washed with two 1/10 volume washes of 1 M.  $\text{Na}_2\text{Cr}_2\text{O}_7$ , 0.2M  $\text{HNO}_3$ , two 1/5 volume washes of water, and then sent to purified solvent storage. Used hexone will be washed with two 1/10 volume washes of 20%  $\text{NaOH}$  followed by a steam distillation over a caustic heel. Hexone will be stripped from all aqueous phases of column products and raffinates, and from the solvent recovery washes and azeotropes. The solvent recovery plant will process 370 gallons of hexone per day. Chemical flowsheet TD-479 has been issued for checking.

3.15 Uranium Salvage

Provision for the salvage of uranium from both radioactive and non-radioactive solutions will be provided on the same basis outlined in the quarterly report of March 1, 1948, ORNL-8.

3.16 Waste Storage and Disposal

The waste disposal for the combined 1100, 1200, 1300 and 1400 Areas system is now under consideration. The facilities to be provided can be divided into three classes:

- 1) Canyon waste hold tanks for collecting wastes such as process condensates, cooling water, steam condensate, etc, for analysis before disposal to the storage system or retention pond.
- 2) Permanent storage facilities for highly radioactive solutions.
- 3) Waste disposal system of neutralizers and retention pond.

Facilities will be sized so that the capacity required by the operation of the research pile alone is provided initially. If another pile is constructed in the future from which fuel material will be processed in the 1000 Project chemical facilities, the waste system will be expanded to accommodate the additional load.

3.17 Analytical Control

A central laboratory for performing all analyses for the 1200, 1300, and 1400 Areas will be provided. A new estimate of the number of control samples to be taken, patterned after sampling procedures used in the hot pilot plant, has been made. The analytical group is now making a new estimate of manpower and space requirements for the analytical laboratories. On the basis of this new estimate the size of the analytical facilities may be altered.

3.18 Building Layout

The study of the economy of stainless steel liners for canyon cells was completed and issued as a memorandum to file. The cost of cell liners per square foot was estimated to be approximately \$5.00. The lining of a typical cell, 20' x 20' x 15', would cost approximately \$8,000.

Based on information from the operations group, the savings made possible by a stainless lined cell were approximately \$1,900. Thus, a cell liner would pay for itself in four decontaminations. Past experience has shown that one such decontamination per year is probable. There would, therefore, be a definite economy provided by a stainless lined cell if the plant operates for ten years. However, pin hole leaks or bad welds in the liner might make it possible for active waste solutions to seep between the cell wall and liner, thus making decontamination almost impossible. Other methods of coating concrete will be investigated before stainless lined cells are adopted. In addition, remote operations will be studied further.

### 3.19 Decontamination

Facilities for the decontamination of cells and equipment for the 1200 and 1300 Areas are now being outlined. It has been assumed that decontamination of the entire processing canyons will be required once per year for repairs and/or alterations. Before an actual decontamination begins the process equipment will be washed out with  $Al(NO_3)_3$  solution made up as a dummy feed and run through the process as a dummy run to remove the uranium from the process equipment. This solution, with its uranium, will be recovered in the salvage area or fed back into the main process stream. The first cycle equipment will receive two washes with 60%  $HNO_3$ , followed by two washes of 20%  $NaOH$ , one day of steaming with low pressure steam spiked at the tank inlets with 60%  $HNO_3$ , and finally three water washes. All decontamination wastes will be collected in the canyon catch tanks where neutralization, concentration, and analysis for uranium will be performed before ultimate disposal. The quantities of waste solution from one such a decontamination have been estimated as follows:

60% $HNO_3$	1,850 gal
20% $NaOH$	1,850 gal
Steam condensate	1,400 gal
Water washes	19,000 gal

Estimates for decontaminating cell walls and process vessel and equipment exteriors are now being made.

Other phases of the 1200 Area design such as the process tank overflow system, canal layout, shielding, service requirements, change house facilities, building layout, duplication of equipment, activity study, ventilation, and security will be reviewed before the final preparation of the design report.

3.2 Laboratory and Semi-Works (F. L. Steahly)

3.21 Metal Solution

The dissolving of the pile assembly was investigated on full scale. Originally the object of the study was to dissolve each assembly at a uniform rate over 24 hours and to obtain a basic solution. This was accomplished by adding the nitric acid to an excess of metal at a uniform rate in 12 hours at 65° C, and then digesting for 9 to 10 hours at 90° C. to reduce the acid concentration of the final solution. This procedure is not necessary because the irradiated assemblies are to be cooled for 145 days after which time very little radioactive gases will remain and a slow solution rate is not required.

A study is now in progress to determine the feasibility of completely dissolving each charge of pile assemblies in less than 12 hours, so that a critical mass build up will be impossible. The variables to be investigated are as follows: rate of acid addition, moles of acid per mole of aluminum present, reaction temperature, and crud characteristics. In the first runs the acid was added in 1.5 hours at 90° C. The reaction was quite vigorous and somewhat unsteady. By increasing the time of acid addition to 3 hours and maintaining the temperature at 90° C, a more controllable reaction was obtained. In two runs, 3.75 moles of nitric acid per mole of aluminum was added leaving 1.7 and 2.7% of the metal undissolved at the end of 24 hours. In the third run the acid to aluminum ratio was changed to 4.02 and under similar conditions of acid addition rate and temperature, the metal was completely dissolved at the end of seven hours giving a final solution of 1.7M aluminum nitrate and 0.75M nitric acid. The acidity of the other two runs at the end of seven hours had been 0.9 and 1.7M. In all runs, the solution temperature was raised to boiling (106°-107° C) after the acid was added.

Further work is in progress to determine optimum conditions for dissolving the uranium-aluminum alloy and to evaluate the crud situation.

The preliminary investigation of the pH and specific gravity instruments for remote analysis of the metal solution was not successful due to failure of the recirculating system that was proposed, the relatively short life (9 hours) of the pH electrodes, and the failure of the specific gravity instrument. Since then more rugged pH electrodes have been obtained from Leeds and Northrup. A pneumatic specific gravity meter is to be tested to determine if sufficient accuracy can be obtained.

### 3.22 Complexing Agents in Extraction

The organic soluble complexing agents, tri-n-butylamine and 2-hexyl pyridine, were not effective in increasing uranium decontamination under bare flowsheet conditions (1.3M  $\text{Al}(\text{NO}_3)_3$ , pH 1.8). However, it was found that at 0.8M  $\text{Al}(\text{NO}_3)_3$ , 0.02M  $\text{HNO}_3$ , the addition of tri-n-butylamine increased uranium decontamination by a factor of 40, but the decontamination was less by a factor of two than that obtained under bare flowsheet conditions. Therefore, these complexing agents would be effective, only if low aluminum nitrate and acid conditions were required. The 2-hexyl pyridine flowsheet was demonstrated to give no significant increase in decontamination factor in both semi-works and pilot plant runs.

Hydrazine as a complexing agent has been more difficult to evaluate. By increasing the equilibration time from five minutes to one hour with 0.4M hydrazine the uranium distribution coefficient increased from 0.9 to 138. The effect of digestion time and temperature was then studied. The feed solution (1.3M  $\text{Al}(\text{NO}_3)_3$  pH 1.8, 0.5M  $\text{N}_2\text{H}_4$  and 0.05  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$  was digested at 25° C and it was found that the fission product distribution coefficient (aqueous:organic) decreased from 0.05 at the end of one hour to 0.008 at the end of 96 hours. The digestion was repeated with 0.03M  $\text{N}_2\text{H}_4$  at 85° C and in one hour the fission product distribution coefficient was 0.002. This indicated that by feed digestion at 85° C the fission product decontamination in extraction may be increased from  $10^3$  to  $10^4$ . This is to be further investigated.

The use of diphenyl thiourea, another organic soluble complexing agent, was demonstrated in a series of semi-works runs to give a ruthenium decontamination factor of  $2 \times 10^4$ , as compared to 210 for a bare flowsheet run. The gross beta decontamination was increased by a factor of ten, but the gamma decontamination was not affected. The DPT was added to the organic phase after scrubbing, heated to 50° C for 20 minutes, cooled, and then passed on to the strip column. The pilot plant demonstration of this process failed due to precipitation of uranyl sulfate in the digestion chamber. No precipitate was found in the semi-works digestion chamber after 700 hours of operation. The pilot plant conditions differed somewhat from the semi-works conditions in respect to feed uranium concentration, time and temperature of digestion, and DPT concentration.

The addition of 0.001M thenoyl tri-fluoroacetone (TTA) to the hexone solution before stripping complexed the zirconium holding it in the hexone phase during stripping. Batch laboratory data indicated that an additional zirconium decontamination factor of 7-10 was obtained.

### 3.23 Reducing Agents

Before starting the pilot plant demonstration of the 25 process with W metal, it was necessary to choose conditions so that the major portion of the plutonium present would concentrate in either the

waste or product stream. Previous work done at Knolls Laboratory indicated that the use of sulfamic acid-ferrous ammonium sulfate reduced the plutonium making it less extractable. In laboratory tests ammonium sulfamate was substituted for sulfamic acid to minimize the effect on pH of the aqueous feed. It was shown that the addition of 0.05M ammonium sulfamate - 0.05M ferrous ammonium sulfate to the 1.4M aluminum nitrate, pH 1.85 feed solution decreased the plutonium distribution coefficient (organic to aqueous) from  $5 \times 10^{-2}$  for the bare flowsheet to  $6.5 \times 10^{-4}$  under reducing conditions. The uranium distribution coefficient was also reduced from 3.8 to 2.1, but the overall effect was to increase the uranium-plutonium separation factor from 76 to 3230.

The substitution of ferrous sulfamate for the combination reducing agent ammonium sulfamate - ferrous ammonium sulfate was suggested, and demonstrated to be satisfactory. The ferrous sulfamate was produced by agitating an excess of powdered iron in a 30% sulfamic acid solution for four hours at room temperature. The excess iron was then filtered off. The resulting ferrous sulfamate was stable when stored in a refrigerator but at room temperature, a small amount of yellow solid precipitated but this had little effect on the ferrous sulfamate concentration.

The addition of ferrous sulfamate to the feed and scrub caused an increase in fission product decontamination in pilot plant runs. The study of ferrous sulfamate concentration, and pre-digestion time has not shown a significant increase in uranium decontamination. Temperature and extraction time effects are being investigated.

The use of hexone-soluble sulfanilamide was proposed as a reducing agent in the hexone phase to improve ruthenium decontamination. This was postulated on the basis of the favorable results of the aqueous phase increased decontamination in the pilot plant runs. However, laboratory batch tests have not indicated any effect when the sulfanilamide was added to the organic phase.

It was thought that the presence of nitrite would complex ruthenium making it organic soluble and thereby lowering its decontamination. However, the addition of 0.003M nitrite had no appreciable effect on the ruthenium distribution coefficients.

### 3.24 Neptunium Separation

Neptunium separation was demonstrated in one semi-works run giving an 88% yield with a fission product decontamination factor of approximately  $10^3$ . Laboratory determined conditions of 1.0M  $\text{Al}(\text{NO}_3)_3$ , pH 1.5, 0.05M  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ , 0.1M  $\text{N}_2\text{H}_4$  and 0.4  $\text{N}_2\text{H}_4$  in the organic phase were used in the extraction section along with the addition of DPT before stripping to increase ruthenium decontamination. Under these conditions, the neptunium was carried along with the uranium to be separated in a second cycle. It was concluded that the neptunium could be recovered by this procedure with satisfactory fission product decontamination.

### 3.25 Scrub Study

The effectiveness of the scrub section in the first column has been less than anticipated. Laboratory column runs are now in progress to determine the effect of varying the aqueous to organic flow ratio, the scrub column height, and the salt concentration. The flow ratio (aqueous to organic) was varied from 0.1 to 0.33 with no significant effect on decontamination. The scrub column height with 3/16" helices in a 1" glass column was increased from one to five feet with the scrub decontamination factor increasing from 3.4 to 14.8. Under these conditions the HETS for scrub was approximately 1.5 feet, and four feet (2.7 stages) seemed to be the optimum scrub height. The study of variation of the salt concentration in the scrub section has not been completed.

### 3.3 Pilot Plant Development

The 1200 Area Pilot Plant has completed process investigations using uranium bombarded in the Clinton pile. Eight runs have been made with this material in the past period through the first cycle equipment from which data were obtained assuring adequate fission product and transuranic element decontamination for the first two cycles of extraction and stripping. Operation of the second cycle was curtailed during the past period because of the low level of activity present. At the conclusion of these Clinton runs the first cycle equipment was easily decontaminated for inspection and minor modifications during which no major equipment fault, leaks, or corrosion was found.

In the past period investigations have been made in which the reducing agent combination of ferrous ammonium sulphate and sulphamic acid was tested and replaced with ferrous sulphamate and a tentative decision made against including 2 hexyl-pyridine as a uranium organic complexant in extraction and diphenylthiourea as a scrub during stripping. Other minor changes made to improve decontamination and operation include the use of 0.04N HNO<sub>3</sub> for stripping and doubling of the previously reported scrub rates. Filtration study on the column feed has eliminated the use of MnO<sub>2</sub> as a filter aid because of the high uranium losses and poor operability.

### 3.31 Construction Costs

The cost summary of construction of the 1200 Area Pilot shows that approximately \$235,000 have been expended in the construction of this plant and major related facilities. This expenditure does not include the building or the tanks salvaged from the bismuth phosphate Pu separation pilot plant and is exclusive of any overhead incurred by the Oak Ridge National Laboratory for material procurement, shipping charges, accounting or operating overhead during construction, or expenditures incurred during plant design and any minor changes made through the medium of repair orders. Considering the lack of overhead and miscellaneous charges, the figure quoted is considerably low for the true overall cost of the installation.

### 3.32 Dissolution

Dissolution of uranium metal in nitric acid in the presence of excess metal continues to give satisfactory operation and resultant products. Inspection of the type 347 stainless steel dip pipes that have been in service in the type 309S Cb stainless dissolver during the dissolving of ten batches of uranium showed that the etching was no more pronounced than in the previous inspection which was made after dissolving two batches of metal. The service between inspections represents 250 hours of dissolver operation at temperatures of 100-105° C and approximately 2000 hours of storage of 15% UNH solution at 30° C.

### 3.33 Feed Makeup - First Cycle

Feed makeup to the desired pH, described in the last quarterly report as being done by the acid analysis of all component streams, has become routinized to the extent that stock quantities of the various solutions are combined, filtered, and then sampled for analysis and pH adjustment just prior to feeding into the columns, reducing the number of necessary samples in the feed makeup. The concentration of the aluminum is controlled by specific gravity measurements and the pH measurements are made using a one-drop pH meter. Investigations during the past period into the use of indicators in the recirculating samplers as a process control for pH show promise.

### 3.34 Crud Removal Filtration

The feed solution, which is filtered through a sintered stainless steel disk of porosity G having an area of 240 sq. in., continues to give satisfactory operation without filter aid. The second expendable filter disk had to be replaced when it became plugged with MnO<sub>2</sub> fines during one of the runs (9X) which gave a filter life of only 700 gallons of solution. The subsequent disk has been in operation for a total of eight runs and has processed approximately 2000 gallons of solution with evidence of only slight plugging. This increase in length of service has been a combination of improved operation and method of fabricating of the disk. The use of NaOH washes to remove plugging material, presumably aluminum oxide, is dependent on filtration rates. In some cases it is not necessary to clean the filter for an entire batch of 250 gallons while in other cases cleaning is required twice during the run. The improvement in fabrication procedures is that the sintered stainless steel is heliarc welded to a 1/4" stainless steel backup plate, making a mechanically strong welded unit as compared to those formerly made by resistance welding.

One run (9X) was filtered using 2 grams/liter of co-formed MnO<sub>2</sub> as a filter aid. Using the best precipitating and filtering conditions, as determined by the laboratory, the filtration took 120 hours as compared to 20 - 30 hours when using no filter aid. Filtering with and without MnO<sub>2</sub>

showed that the uranium losses were 1.3% as compared to 0.06% despite repeated washings of the cake. Decontamination factors across the filtration step without filter aid are negligible as compared to 1.3 for gross betas, 6.0 for gross gammas, 19 for Ru betas, and 25 for Zr betas when  $MnO_2$  was used. The high losses, excessive filtration time, and traces of manganese in the extraction column raffinates interfering with the fluorophotometric method of uranium analysis making extraction losses unreliable, resulted in deciding against including  $MnO_2$  in the 1200 Area flowsheet.

### 3.35 First Cycle

A total of eight first cycle extraction and stripping runs using Oak Ridge Pile irradiated uranium which gave material having  $1.5 \times 10^9$  beta cts/min/gm U and  $4 \times 10^6$  gamma cts/min/gm U have been made in the past period. The flowsheet conditions given in Figure III of the last quarterly report (ORNL-8) were modified one at a time during these runs to study process variables. The study of the data, summarized in Table II resulted in revising the flowsheet to that given in Figure 6. Decontamination in the first cycle continues to be adequate with factors of  $3 \times 10^3$  for betas and  $2 \times 10^3$  for gammas even at the low level of activity being investigated. Ru activity continues to be the limiting factor in decontamination. The total losses of uranium in both the extraction and stripping column of the first cycle gives adequate recovery, being less than 0.005%.

The use of the reducing agent combination of ferrous ion plus sulphamic acid did not affect the losses of uranium. The use of this combination increased fission product decontamination, with the increase being more noticeable in the second cycle data, given in Table III. The ferrous ion plus sulphamic acid was added in the scrub stream in Runs 9 and 10 and gave adequate decontamination showing that the reduction reaction takes place rapidly. The use of the reducing ion combination of ferrous ion plus sulphamic acid gave Pu decontamination factors of  $2 \times 10^3$  as compared to 3 to 5 when neither was used and 1 to 3 when sulphamic acid alone was used. This indicates that sulphamic acid by itself is not a strong enough reducing agent to give adequate Pu decontamination.

Instability of the ferrous ammonium sulphate in the strong aluminum nitrate solution gave rise to the use of ferrous sulphamate, which is stable. This material was added in excess of the amount for maximum effectiveness in Run 14X, causing a lower decontamination than was predicted. Evidence of a maximum reducing agent concentration for best decontamination of fission products is confirmed by experimental laboratory evidence.

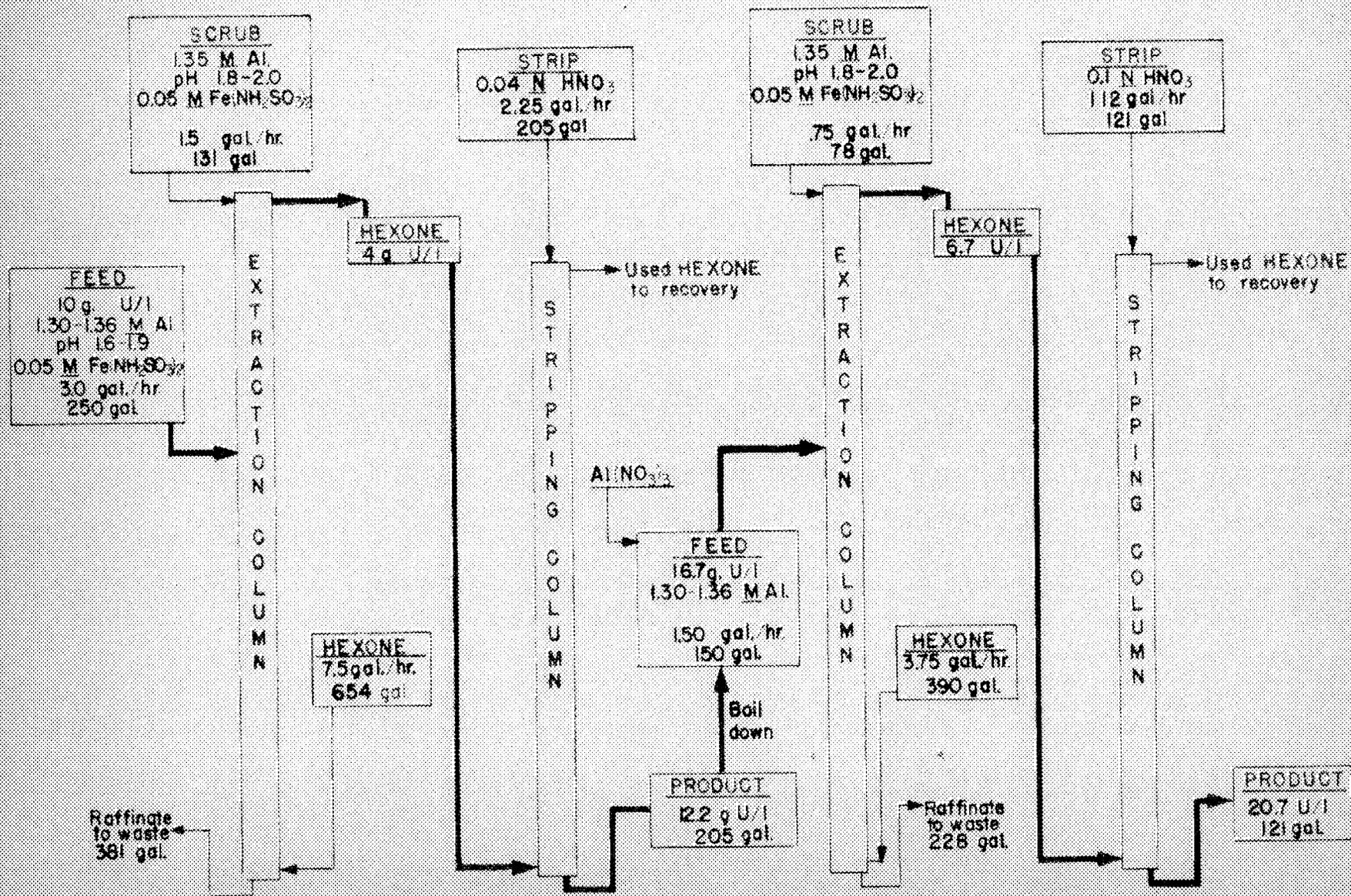
TABLE II

First Cycle Oak Ridge Laboratory Pile U  
Flowsheet Conditions as in Figure 6 Except as Noted

Run	<u>7X</u>	<u>8X</u>	<u>9X</u>	<u>10X</u>	<u>11X</u> (a)	<u>12X</u> (b)	<u>13X</u> (c)	<u>13 X B</u> (c)	<u>14X</u>
<u>Feed</u>									
pH	1.78	1.56	1.81	1.02	1.52	1.60	1.64	1.64	1.70
NH <sub>2</sub> SO <sub>3</sub> <sup>-M</sup>	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
Fe <i>H</i>	0.05 (d)	0.02 (d)	0.02 (d)	0.02 (d)	None	None	None	None	0.10
<u>U Losses %</u>									
Extraction	0.0011	0.0004	-	0.0008	0.0007	0.0008	0.0011	0.0005	0.0002
Stripping	0.010 (e)	0.012 (e)	0.0025	0.0016	0.0005	0.027	0.0009	0.0002	0.0014
<u>Decontamination Factor</u>									
Gross beta	4x10 <sup>3</sup>	3.4x10 <sup>3</sup>	8.9x10 <sup>3</sup>	0.9x10 <sup>3</sup>	0.2x10 <sup>3</sup>	7.3x10 <sup>3</sup>	2.9x10 <sup>3</sup>	0.9x10 <sup>3</sup>	1.5x10 <sup>3</sup>
Gross gamma	2.3x10 <sup>3</sup>	1.7x10 <sup>3</sup>	7x10 <sup>3</sup> (f)	0.4x10 <sup>3</sup>	0.1x10 <sup>3</sup>	4.1x10 <sup>3</sup>	1.9x10 <sup>3</sup>	0.7x10 <sup>3</sup>	1.1x10 <sup>3</sup>
Ru	200	210	1.8x10 <sup>3</sup>	40	10.9	330	152	82	99
Zr	7.5x10 <sup>3</sup>	1.1x10 <sup>4</sup>	2.1x10 <sup>4</sup>	3x10 <sup>3</sup>	0.7x10 <sup>3</sup>	4x10 <sup>3</sup>	1.4x10 <sup>4</sup>	7.8x10 <sup>3</sup>	2.5x10 <sup>4</sup>
Pu	72 (g)	1.3x10 <sup>3</sup>	1.8x10 <sup>3</sup>	3x10 <sup>3</sup>	1.4	2.8	1.3	1.02	33
Np	-	-	-	-	-	-	250	64	3.5x10 <sup>3</sup>

Table II. Addenda

- (a) 2-hexyl pyridine added to hexone.
- (b) DPT added to hexone stream enroute to stripping column.
- (c) 13A and 13B equal portions of the same run with A receiving twice normal scrub rate; B no scrub.
- (d) Ferrous ion introduced as  $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$ .
- (e) Stripped with 0.1N  $\text{HNO}_3$ , others with 0.04N  $\text{HNO}_3$ .
- (f) Includes decontamination over filtration step - extraction and stripping decontamination factors normal.
- (g) Contaminated from previous heel.



1200 AREA PILOT PLANT FLOWSHEET

5-5-48

56 27

FIGURE 1

Hexone with 0.1M 2-hexyl pyridine for complexing the uranium into the organic phase was used in Run 11X. This material caused a large decrease in decontamination factors due mainly to chemical reasons, as evidenced by the fact that although the decontamination factors decreased, the Ru and Zr still constituted the usual 95% of the fission product activities in the product stream.

A study of the use of diphenylthiourea (DPT) to reduce the fission product distribution ratio into the aqueous stream during stripping was made in Run 12X. The DPT was added to the uranium-hexone stream from the extraction column just ahead of a mixing chamber, maintained at a temperature of 60° C. with sufficient quantities being added to give a concentration of 20 grams DPT/liter in the stripping column. The use of DPT increased decontamination over the stripping column to 4.0 instead of the usual 1.2 to 1.7, but was less than that predicted by laboratory and small scale column runs. The inclusion of DPT into the flowsheet at this time was not warranted because  $UO_2SO_4$ , formed by decomposition of the DPT, precipitated in the mixing chamber plugging it after 85 hours of operation, and the problems of removing the Ru activity and DPT from the used hexone are unsolved.

The decontamination of undesired transuranic elements (Np and Pu), as shown in Run 14X indicates the latest flowsheet will be adequate for the 1200 Area. The elements will be mainly eliminated in the raffinates of the first cycle and should their recovery be desired, they can be obtained by processing these raffinates.

Stripping losses averaged 0.01% when 0.01N  $HNO_3$  was used, 0.003% using 0.04N  $HNO_3$ , and 0.001% using 0.1N  $HNO_3$  (last report). Inasmuch as the use of 0.04N  $HNO_3$  gives adequate recovery and that amount of acid can be tolerated in the second cycle feed makeup, the use of this strength acid was incorporated into the flowsheet.

The effect of pH on decontamination cannot be definitely established in the range of the given data due to the changing of the other variables. The one exception to this is Run 10X which was made at a pH of 1 to determine the lower limits of this variable and gave decontamination factors of 900 for beta and 400 for gamma, considerably lower than decontamination factors obtained under identical conditions at a pH of 1.7. Additional pH investigations will be made in the coming period.

S. E. Galt

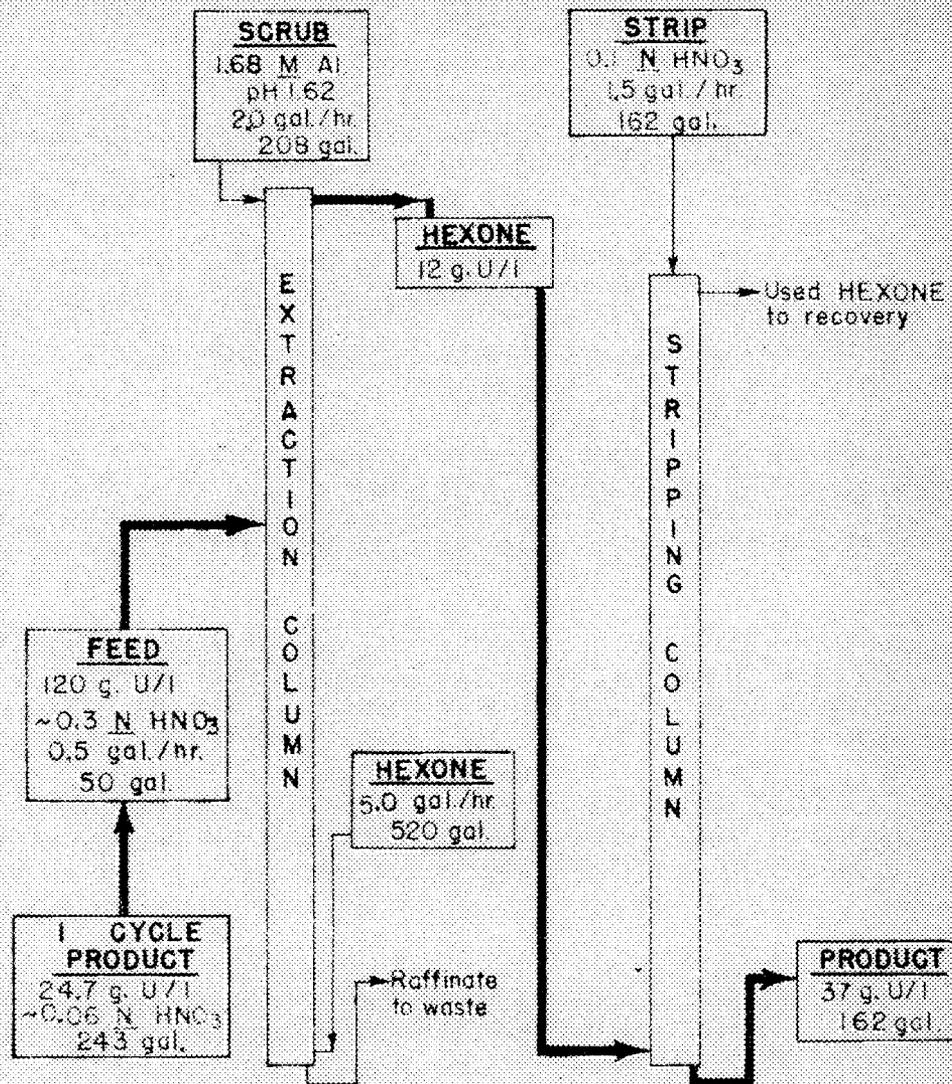
Scrub flow studies made in Run 13X showed that decontamination improved with increased scrub rates. This run was a continuous run; in the first part (A) scrub was introduced at twice the flowsheet conditions (1.5 gal/hr) and in the second part (B) the scrub was shut off. The decontamination factor over the strip column in part A was 2.0 as compared to the usual 1.2 to 1.7. This fact indicates that the scrubbing was very effective in taking out the water soluble fission products, leaving the ratio of the hexone soluble fission products larger than usual in the product stream. In Run 13X B the Ru and Zr constituted less than 36% of the activity in the product stream as compared to the usual 95%, indicating non-removal of some physically carried aqueous droplets when the scrub section is stagnant.

The first cycle extraction column has operated a total of 1400 hours without any evidence of fouling or plugging due to crud formation. Inspection of this column after completion and decontamination from the Oak Ridge Pile activity runs showed no appreciable build up of solids in the column. This lack of crud formation, it is felt, is due to the care taken in feeding only clean solutions into the column and the relatively large size 1/4" x 3/8" Raschig rings which would permit any small particles to leave via the raffinate stream.

### 3.36 Second Cycle

The feed to the second cycle continues to be made up by boiling down the first cycle product and adding the various reagents before introduction into the extraction column. An alternate flowsheet shown in Figure 7 was used in Run 9X with satisfactory results. This flowsheet provides for the mixing of the various reagents and salting agents in the column to obtain the desired conditions rather than in the mix tank, eliminating the tedious feed makeup procedure and taking advantage of the increased scrub rate.

The activity in the second cycle product continues to be below uranium background by a factor of 10, making determination of gross beta and gamma decontamination factors extremely unreliable. The decontamination of specific fission products are given in Table III and are the only reliable decontamination factor data obtainable at the very low levels of activity in the second cycle, and they are at the lower limits of accuracy. Run 9X, made with the alternate flowsheet shown in Figure 7, gave satisfactory operation and uranium recovery. During the coming period the evaluation of the alternate second cycle flowsheet will be made, and it is hoped that the processing of the Hanford irradiated uranium will give sufficient activity in the second cycle to make it possible to obtain more accurate decontamination factors.



ALTERNATIVE SECOND CYCLE PILOT PLANT FLOWSHEET

TABLE III

Second Cycle Oak Ridge Laboratory Pile U  
Flowsheet Conditions in Figure I Except as Noted

Run	5X	6X	7X	8X	9X
<u>Feed</u>					
pH	1.68	1.67	1.58	1.0	See
NH <sub>2</sub> SO <sub>3</sub> <sup>-</sup> M	-	0.05	-	0.06	Figure
Fe <sup>++</sup>	-	0.02	-	0.02	7
<u>U Losses %</u>					
Extraction	0.0009	0.0018	0.0004	0.0011	0.0021
Stripping	0.0064	0.0056	0.0010	0.0031	0.0055
<u>Decontamination Factor</u>					
Ru	82	173	44	28	10
Zr	81	33	74	14	20

- 54 -

### 3.37 Solvent Treatment and Recovery

The pretreatment and recovery of hexone has settled down into a smooth routine operation which is essentially the same as described in the last quarterly report. The pretreatment of the commercial hexone consists of a one molar sodium dichromate wash and steam distilling from a small caustic heel. The recovery of used process hexone consists of a continuous steam distillation from a caustic heel by adding used material in a steady stream to the still pot. The process has shown to give a recovery of 94% with the purity of the product being dependent to some degree on entrainment so that the distillation rate must be kept reasonably low. The purified hexone in each case exceeds the specification of less than 0.002N total reducing power and less than 0.01% acidity calculated as acetic acid. Recent changes to the distillation equipment have increased the distilling capacity and simplified operations. The residual activity in the used hexone has continued to be mainly betas and there has been no measurable radiation in the used solvent storage area and no difficulties have been experienced during the solvent recovery operation.

### 3.38 Analytical Developments

A study of the accuracy of the pH determination of process solutions was made which showed that the determination remained within the useful control with a standard deviation of 0.03 pH units. The study made with the "one-drop" glass electrode showed similar results. A similar study using the rapid titrimetric analysis of aluminum showed that the method was undesirable, resulting in a change to the use of specific gravity as a process control. A study of this method showed that the accuracy was within the allowable limits having a deviation of 2.5% with a slight, but not warranted, greater precision when corrected for temperature. The development of a falling drop specific gravity determination by the Analytical Section shows promise. Similar analytical accuracy determinations on other analysis methods will be made in the coming period.

### 3.39 Decontamination of Equipment

At the conclusion of a series of runs investigating process variables using Oak Ridge pile irradiated uranium, the Pilot Plant was shut down to permit inspection of the equipment. Only the equipment in the first cycle required decontamination using alternate washes of hot soda ash, hot nitric acid, hot citric acid, and hot caustic. The study of the effect of the various decontaminating agents showed no decided advantage of any one because of the relatively low level of activity in the equipment and close proximity of the various tanks in the cell. After the completion of the washings, the entire cell and insides of all equipment pieces were sprayed with steam, which reduced all radiation readings from 20 to 50%. Another decontamination study will be made at the end of the Hanford runs in an effort to determine more specific information on decontamination with liquid reagents.

3.310 Program

The program for the next period includes completion of one half of the process investigation phase using Hanford irradiated uranium. Variables to be investigated include feasibility of the alternate second cycle flowsheet, the optimum concentration of the ferrous and sulphamic ions, effect of pH, and possibly some investigation on the effect of flow rates.

4.0 23 SEPARATIONS PROCESS DESIGN AND DEVELOPMENT

4.1 Design Progress 1300 Area (J. A. Lane)

A brief review of the work accomplished so far in 1300 Area design is given below. A comprehensive review of design work previous to March 1, 1948, was presented in the previous quarterly report.

The design capacity of the 1300 Area has been selected as fifteen kilograms of irradiated thorium per day. This will provide capacity for the output of the research pile plus one other pile of equal size. A typical delivery of irradiated thorium from the research pile will contain about twenty grams of 23 and 13 and two-thirds of a gram of fission products per fifteen kilograms of thorium. The shortest cooling period considered at the present time is eight months. With this cooling period the total 13 activity in a fifteen kilogram batch is about two watts (thirty milligrams of 13), and the total fission product activity is about one watt. With a cooling period of eight months and for one hundred gram batches the requirements of the 23 recovery process are:

Maximum total 23 loss, per cent	0.1
Thorium reduction factor	$10^6$
Decontamination from 13	$2 \times 10^5$
Fission product decontamination	$10^6$

This purity appears to be satisfactory for production quantities of 23. Whenever higher purities are needed, the additional purification operations may be designed by the investigators concerned.

A comparison between batch versus continuous processing for 23 recovery has been made and published as report ORNL-20. For a plant with the capacity of fifteen kilograms of thorium per day, a continuous column process is more favorable than one using intermittent batch extractions. The estimated investment and operating costs for each process are essentially the same.

The cooling period for irradiated thorium has been selected for the four classes of assemblies to be delivered to the 1300 Area. Table IV summarizes the exposure condition for each class of thorium with the consequent production of protoactinium and fission products.



TABLE IV

Estimated Activities of Irradiated Thorium

	<u>Thermal Flux</u>	<u>Period of Irradiation</u>	<u>Mass of Thorium</u>	<u><sup>13</sup></u>	<u>Fission Products</u>	<u>Cooling Period</u>
Production Rod	$10^{13}$	365 days	12.5 kg	2.9 g	1.65 g	160 days
Production Rod	$5.5 \times 10^{13}$	75	12.5	12.2	1.12	250
Production Rod	$5.5 \times 10^{13}$	15	12.5	4.6	0.11	310
Shim-Safety Rod	$2.3 \times 10^{14}$	15	8.1	12.2	0.32	300

The half-life of <sup>13</sup> is 27.4 days, the total energy released per disintegration was considered to be 0.6 Mev. The first-heeler curves were used to estimate the decay curves for fission products.

A series of curves for the decay of fission products from <sup>23</sup> has been prepared. The curves show the total <sup>23</sup> fission product activity associated with one kilogram of thorium irradiated for various periods of time at various thermal neutron fluxes. A report discussing these decay curves has been issued as report ORNL-35.

Other phases of the 1300 Area Design that have received consideration are: Chemical Flowsheets for both batch and continuous processes, space requirements for the storage of irradiated thorium, wall thickness for the 1305 building, salvage facilities, activity level of analytical samples, waste disposal, and side reactions which occur during the irradiation of Th<sup>232</sup>. A summary of design progress to date with bibliography and drawing lists has been issued as report ORNL-35.

#### 4.2 1300 Product Semi-works Design

During the past quarter design work has been undertaken for the installation of <sup>23</sup> semi-works equipment in the solvent building now under construction.

The design of the <sup>23</sup> semi-works has proceeded for chemistry conditions in which either diisopropyl ether or dibutyl cellosolve may be used. Since the volumes of process solutions using dibutyl cellosolve are greater than those using diisopropyl ether, the process equipment has been sized for dibutyl cellosolve conditions. The plant capacity is as follows:

Maximum No. of Hanford 4" thorium slugs	- 23
Minimum No. of Hanford 4" thorium slugs	- 10
Processing time per batch	- 110 hrs

Preliminary design for the 1300 product semi-works was completed on March 15, 1948, and the chemical flowsheets, equipment locations, equipment flowsheets, cost estimate, and job description were transmitted to the Plant Engineering Group for detailed design.

At present the status of the work is as follows:

1. The formal project request has been completed.
2. The preliminary cost estimate for the project is approximately \$140,000.
3. Detailing of tanks and equipment is proceeding.
4. Purchase requests for many items have been issued.
5. Project approval has not yet been obtained from the AEC.

#### 4.3 Semi-Works Results (F. L. Steahly)

##### 4.31 Comparison of Solvents

Four possible solvent extraction processes have been developed based on the use of hexone, dibutyl cellosolve, or diisopropyl ether. Two of these processes make use of the last solvent. The tests conducted to date have not differentiated between these solvents because insufficient activity can be added to follow the decontamination accurately. A decision will be made on the basis of flooding rates, required column height and decontamination factors obtained in the semi-works now under construction. Table V presents a summary of the results obtained to date.

The hexone process was superior to the diisopropyl ether and both dibutyl cellosolve processes in flooding rate (1090 versus 600-700 gal/hr x ft<sup>2</sup>) and required column height (17 ft versus 25-33 ft.). The thorium reduction factors for all processes were of the order of 10<sup>4</sup>. These data indicate that the fission product decontamination obtained for the hexone process is lower by a factor of 10 than that obtained for the other two solvents.

TABLE V

## Comparison of Solvents for the 23 Chemical Separation Process

Process Description	Aqueous Feed		Scrub Solution			Flow Ratios				Temp.		
	Th(NO <sub>3</sub> ) <sub>4</sub>	HNO <sub>3</sub>	Al(NO <sub>3</sub> ) <sub>3</sub>	Ca(NO <sub>3</sub> ) <sub>2</sub>	HNO <sub>3</sub>	pH	Org:	Feed:	Scrub:		Strip:	
Diisopropyl Ether	2.5M	0.1M	2.5M	None	0.4M	basic	-	5:	4:	1:	1.25	25°C
Hexone	1.3M	0.05M	1.3M	None	0.05M	"	-	5:	4:	1:	1.25	20°C
Dibutyl Cellosolve (Low Acid in Feed)	2.0M	0.2M	2.4M	None	-	1.8**	4:	3:	1:	1	1	25°C
Dibutyl Cellosolve (High Acid in Feed)	1.25M	1M	2M	1M	1M	1.5*	4:	2:	2:	1	1	20°C

	Flooding Rate gal/hr/ft <sup>2</sup>	HETS at 20% Flood- ing (ft.)	Height of Column for 0.01% Loss	Thorium Reduction Factor***	Beta Decontamination Factor	Gamma Decontamination Factor****
Diisopropyl Ether	675	5.5	33'	2x10 <sup>3</sup>	7x10 <sup>4</sup>	>10 <sup>4</sup>
Hexone	1080	2.5	17'	90	9x10 <sup>3</sup>	>10 <sup>3</sup>
Dibutyl Cellosolve (Low Acid in Feed)	900	4.1	25'	4x10 <sup>2</sup>	2x10 <sup>5</sup>	>10 <sup>4</sup>
Dibutyl Cellosolve (High Acid in Feed)	600	5.7	25'	1x10 <sup>2</sup>	8x10 <sup>4</sup>	>10 <sup>4</sup>

\*This pH adjusted with Ca(OH)<sub>2</sub>

\*\*This pH adjusted with concentrated NH<sub>4</sub>OH

\*\*\*No scrub section was used in these determinations. Two stages of scrubbing have been found to raise the thorium reduction factor to more than 10<sup>4</sup> for all four of these systems.

\*\*\*\*Both gross gamma counting and FP-54 readings were used to determine the gamma decontamination, in all cases the gamma activity in the stripped product was background.

#### 4.32 Concentration of 23

During this quarter 3281.1 milligrams of 23 were concentrated by the batch diisopropyl ether extraction. Of this total 1735 milligrams of 23 was concentrated from the Chemistry Division dibutyl cellosolve extracted product with a 0.0007% loss. The balance of 1546.1 milligrams obtained from the Technical Division columns was processed with a 0.022% loss.

The investigation of the alternate thenoyl tri-fluoroacetone (TTA) chelation process for the concentration of 23 in a continuous counter-current extraction column showed no advantage over the batch diisopropyl ether process. In order to make the process operable it was necessary to obtain a greater density differential by substituting carbon tetrachloride for benzene as the solvent. Batch data indicated that this substitution did not affect the extraction coefficients.

In connection with the TTA concentration process the reaction characteristics of various fission products have been studied. The effect of TTA and acid concentration on the distribution coefficient and equilibration time was studied for ruthenium, zirconium, cerium, and columbium. Scouting work also covered thorium, aluminum, iron, and plutonium. A final report on this work is being prepared.

The study of zirconium has led to development of a chelation process for its separation from columbium in oxalate solutions.

#### 4.4 Solvent Studies

Commercial hexone has been found to contain methyl isobutyl carbinol, mesityl oxide, mesitylene, isopropanol, formic acid, acetic acid, isobutyric acid, and isovaleric acid. Representative samples of untreated Shell hexone were analyzed and showed average concentrations by weight of 0.22% carbinol and 0.23% mesityl oxide. Tests on the extractibility of 23 from salted aqueous solution was not affected by the addition to pretreated hexone of up to 5 per cent of each of the above compounds. No tests were run with more than one impurity. These studies will be continued.

Procedures for the recovery of hexone from diphenyl thiourea and 2-hexyl pyridine were developed. 2-hexyl pyridine contaminated hexone was recovered by washing with two one-tenth volume passes of 8N nitric acid. DPT contaminated hexone was recovered by steam distillation to remove the bulk of the DPT, and this was followed by sodium dichromate, caustic and water washes, and a final steam distillation. By means of vacuum distillation the hexone might be recovered in a single operation. The recovered hexone was used in laboratory and semi-works column runs and showed normal operability.

Approximately 72% of the DPT contaminated hexone was recovered by the steam distillation procedure. The dibutyl cellosolve flowsheet runs have shown variations in uranium distribution coefficients which were suspected to be due to the solvent. An investigation of DEC from various sources and various treatments showed little effect on the uranium distribution coefficient. Fractionation of the DEC yielded a low boiling cut (2.5% by volume) which showed a 15% improvement in uranium distribution coefficient. This was thought to be ethylene glycol mono-n-butyl ether.

## 5.0 Development Tests

### 5.1 Heat Transfer Research (R. H. Lyon)

During the quarter, the NaK circulating equipment has been erected and is almost ready for use. The pump designed as a copy in steel of the cast iron Gusher Pump has been found to deliver only about a 6 to 7 ft. head. A new closed impeller of  $5\frac{1}{2}$  inch diameter instead of the former semi closed impeller of 4 inch diameter has been designed, and is now being constructed. Calculations indicate that it should deliver the desired twenty foot head.

Theoretical considerations have progressed. In particular, study of a paper by Reichardt reveals methods of improving the flow development presented in the last quarterly report. The proposed improvements will include a more confident extension into the realm of higher Prandtl number fluids, and the possibility of corrections for changes in properties due to the radial temperature gradient in the fluid.

### 5.2 Monitoring the ORNL Reactor (C. E. Winters)

Of the several methods which have been considered for monitoring the pile, the use of a pitot tube for air velocity measurement has been selected as the best. An alternate method which measured air velocity by the use of thermally sensitive resistors was discarded when it was found that the thermistors became extremely active when placed in the pile.

The pitot tube, which is inserted in a venturi to keep it from being directional, is mounted on the end of a shield plug with the static and total pressure tubes coming out through the plug. The pitot is located about one foot inside the graphite. Two such tubes have been in the pile for 30 days. The data, taken on a manometer, is shown on Figure 8. Fluctuations in velocity have never been over 10% at ordinary operating conditions, and have generally been less than 5%. When fluctuations do occur they show up proportionately in both tubes, which indicates that, by comparing the velocity in each channel to that of a reference channel, the method could be made quite sensitive.

Drawing # 5718

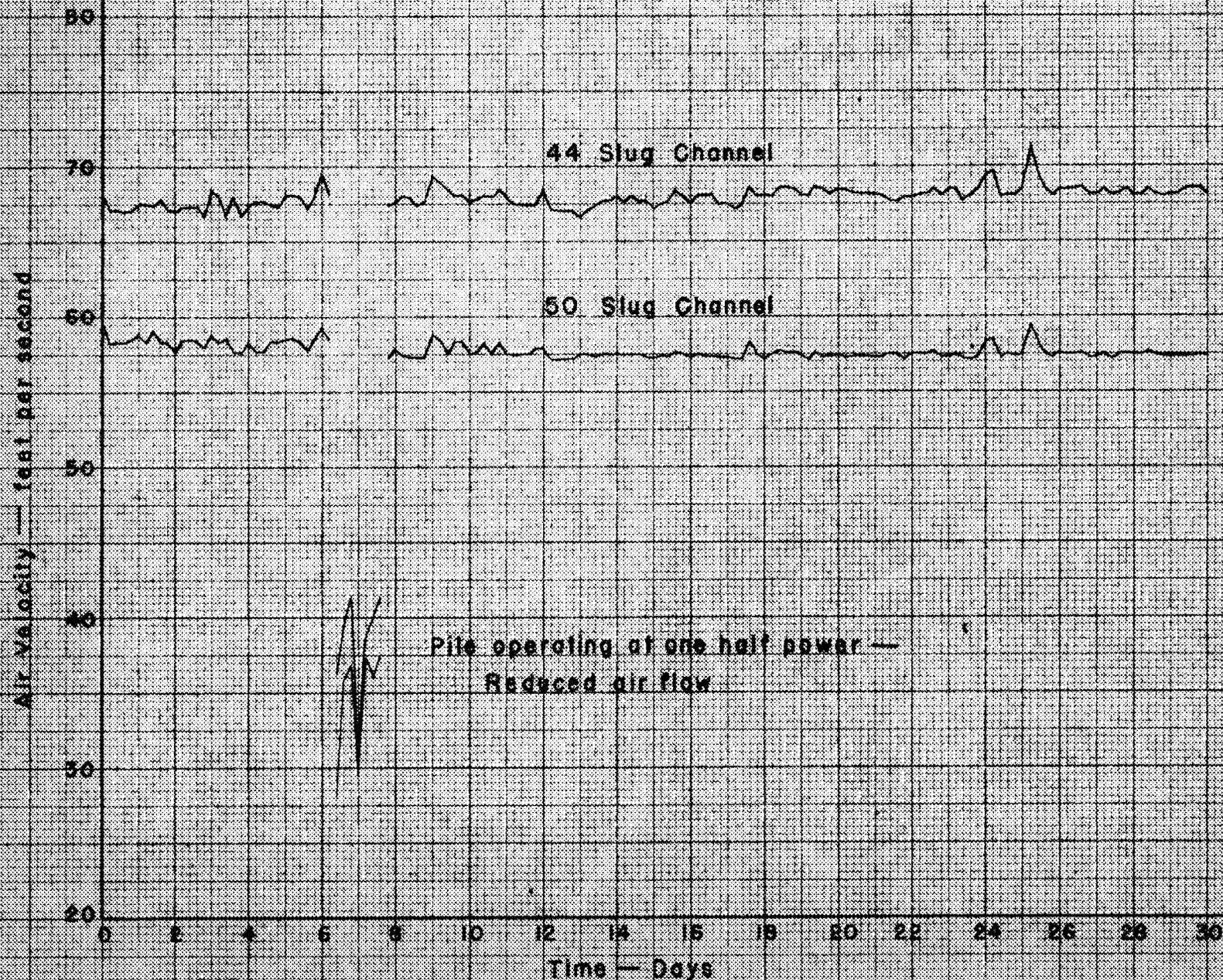


FIGURE 8 FLUCTUATIONS IN PILE AIR FLOW

- 53 -

It is proposed that the manometers be replaced with differential pressure gages of the strain gage type so that flow can be easily recorded on a multipoint continuous recorder. These gages are small enough to be mounted on the end of the shield plug, and can be made more sensitive than a manometer. Two gages have been received, and will be installed on the pitot tubes as soon as the auxiliary instrumentation is assembled.

### 5.3 Shielding (J. A. Kyger)

Several tests have been run as listed in the following paragraphs.

At the request of Hanford Engineering Works, further tests have been conducted on the strength of MO type concrete after subjection to elevated temperatures or pile radiation. Several small samples were constructed, containing shot only, canned, and inserted into a water cooled hole which extends to the center of the pile. The samples were exposed for one month, and compression tests were run on the irradiated samples as well as standards which were prepared at the same time. No deleterious effect on strength was observed. No change in physical appearance of the sample or distortion of the gas tight aluminum cans was detected.

Several MO samples were prepared and placed in a furnace at a temperature of 105°C for varying periods up to a month. Standard ASTM compression tests were run and no decrease in strength was indicated.

Plans are underway for a massive test to be conducted to investigate pouring and handling characteristics as well as other physical properties of MO type concrete.

Samples were prepared and shipped to Hanford for tests of radiation attenuation in a 3" hole in the Hanford shield. Also, small samples were prepared for further radiation stability tests in the high flux available at Hanford.

Arrangements are being made to obtain several 3" cores from the Oak Ridge Pile Shield in order to determine the change, if any, of the strength and composition of the concrete used in this shield. It is planned to use diamond core drills of a commercial type. Equipment and personnel for this drilling have been located and negotiations are underway to secure their services.

Considerable time has been spent on planning of future requirements of a broad shielding program. A study was prepared to indicate the facilities and manpower requirements of such a program, with respect to the technical aspects and the facilities required for radiation and attenuation measurements.

Discussion of the isotope container problem is now underway with regard to the development of throw-away containers to decrease expense in handling, decontamination, and clerical expense involved in the present type containers. The Operations Department has already begun a preliminary investigation of this problem.

#### 5.4 Electrical Flowmeter (C. E. Winters)

A flowmeter operating on the electrocaloric principle has been developed for remote measurement of radioactive flows in the range of 20 cc to 200 cc per minute.

Figure 9 presents a schematic arrangement of the meter with its control and indicating devices. A nichrome heater element (2) is wound about the insulated central section of 1/4 in. tubing (1) through which the liquid is flowing. Temperature sensitive resistors (3,4) ("Thermistors," Westinghouse Electric Company) located at the entrance and exit of the heated section form two elements of a Wheatstone bridge across which is connected a Brown "Elektronik" null detector (7). With the proper selection of resistors, (5, 6) a desired constant temperature difference may be maintained across the heater section by varying the energy input of the heater with changes in flow rate. This is accomplished automatically by the "Elektronik" circuit which amplifies unbalance signals resulting from flow changes and transmits the signals to a motor operated Variac transformer (8, 9) on the 110 volt heater power line until bridge balance is attained. A watt meter on this line indicates instantaneous flow and a watt hour meter serves as a flow integrator. The typical calibration shown in Figure 10 indicates the accuracy which may be expected of this flowmeter.

An attempt is being made to substitute thermocouples and a simple potentiometer circuit for the "Thermistors" and Wheatstone bridge shown in the sketch.

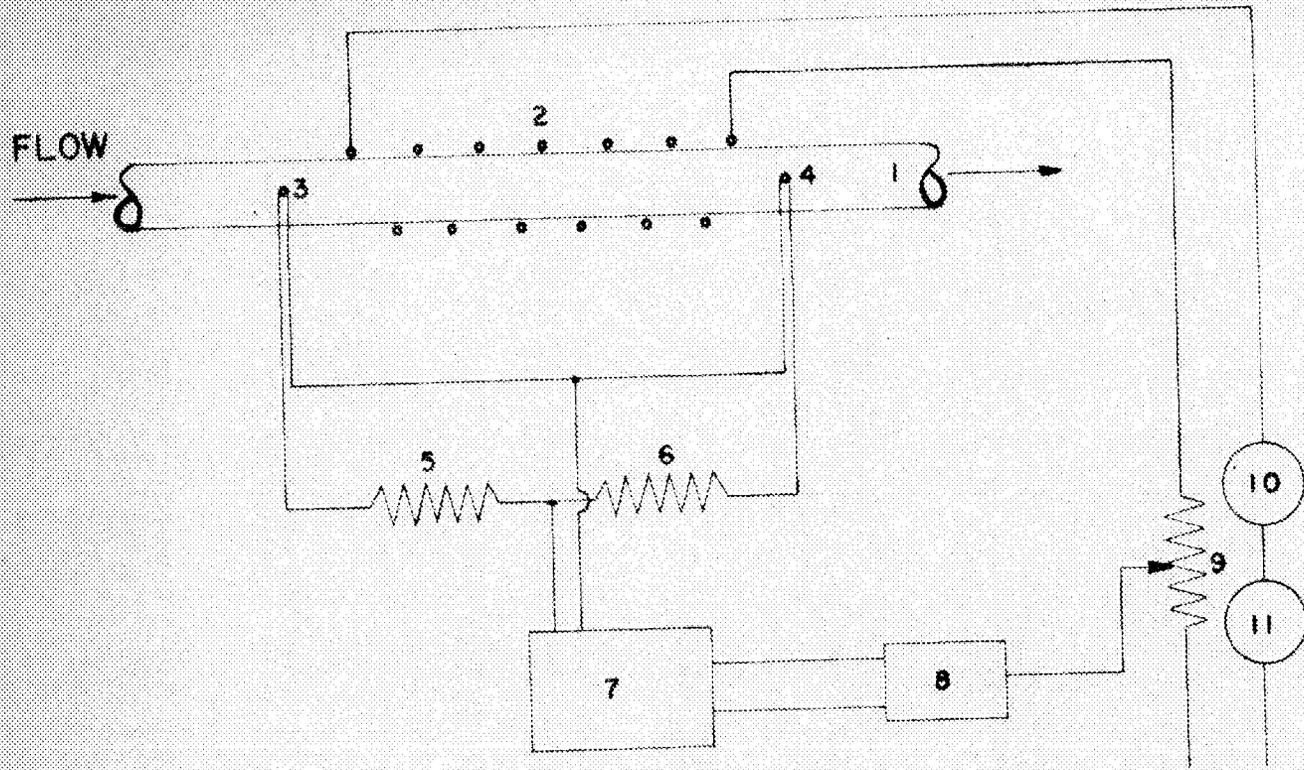
This device will be installed in the Hot Pile Plant as soon as parts now on order are received and assembled.

#### 5.5 Radiation Stability

This is to be a long range problem, the object of which is to study the physical changes that occur in materials that have been subjected to pile radiation.

Very little effort is being expended on this problem at present due to a lack of manpower. It is expected, when manpower becomes available, that the procedure for undertaking the problem will be as follows:

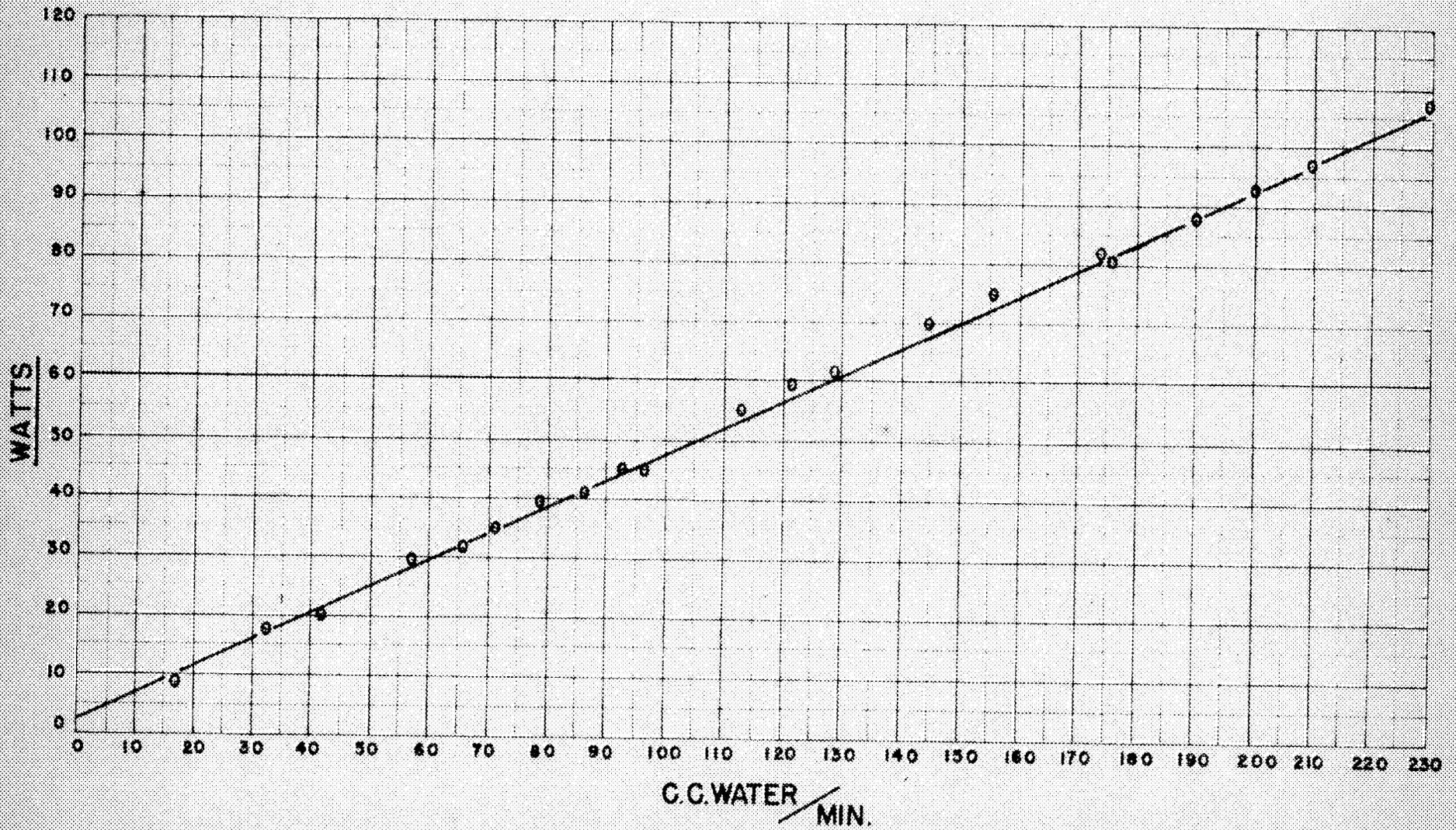
1. A survey will be made of existing data and this will be compiled for future reference.



CIRCUIT FOR ELECTRICAL FLOWMETER

- 1. HEATER TUBE SECTION
- 2. HEATER COIL
- 3, 4. "THERMISTORS"
- 5, 6. FIXED RESISTORS
- 7. BROWN "ELECTRONIC" NULL DETECTOR
- 8. VARIAC MOTOR
- 9. VARIAC
- 10. WATTMETER
- 11. WATT-HOUR METER

Figure 4.



CALIBRATION OF ELECTRICAL FLOW METER

Figure 10.

- 67 -

- 2. A study will be made to determine the types of materials which should be tested first, and to determine the specific tests that need to be made.
- 3. Materials will be placed in the pile for a specified length of time and compared to non-irradiated samples.

The most urgent need for this work at the present time is on plastics, insulating materials, oils, and greases. A few very limited tests have been done to gain information on materials for use in the pile magnets. A summary of these tests is given below. The materials listed were kept in the pile for 35 days at an average temperature of 100° F. A comparison of the flux to which the samples were subjected with that of the flux at the magnets of the high flux reactor is as follows:

	<u>At Magnet on High Flux Reactor</u>	<u>ORNL Reactor</u>
Thermal neutron flux - $n/(cm^2)(sec)$	$3 \times 10^7$	$9 \times 10^{11}$
Epithermal neutron flux - $n/(cm^2)(sec)$	$3 \times 10^7$	$9 \times 10^{10}$
Gamma flux - photons/ $(cm^2)(sec)$	$10^{10}$	$9 \times 10^{11}$

<u>Sample</u>	<u>Remarks</u>
Ceroc covered wire	The insulation is very easily damaged by handling, and seems to be quite porous. When removed from the pile it was covered with a green powder of copper oxide. During irradiation the insulation resistance dropped from 1,500 megohms per inch to 0, and the breakdown voltage dropped from 800 volts to 0.
Formex covered wire	The insulation appeared to be slightly more brittle after irradiation. Insulation resistance changed from >200,000 megohms/inch to 86,000 - breakdown voltage changed from 5000 volts to 2,000.
Formvar covered wire	No change in appearance. No change in insulation resistance. Breakdown voltage went from 3,000 volts to 950 volts.

[REDACTED]

<u>Sample</u>	<u>Remarks</u>
Wire covered with rubber and varnished rayon braid.	The varnish was badly charred, but the rayon was not appreciably affected. Insulation resistance dropped from 290,000 megohms/inch to 40,000. The breakdown voltage dropped from 16,000 volts to 15,000 volts.
Amphenol and rubber covered wire	The rubber turned black, and brittle, but the amphenol was not noticeably affected. Insulation resistance went from >700,000 megohms/inch to 0. Breakdown voltage dropped from >16,000 volts to 800 volts.
Lacquered glass braid insulating spaghetti	Turned black, hard and brittle.
Birnback insulating spaghetti	Turned black, hard and brittle.
Transite	No change.
Mica	No change.
Bakelite	A great number of small cracks had formed, all in parallel planes. Blisters formed on the face parallel to the planes of the cracks.
Silastic 160 (silicone rubber)	Became very hard and brittle.
Silastic 150 (silicone rubber)	Became very hard and brittle.
Silicone stop cock grease	A small amount of grease was sealed in a glass tube and inserted in the pile. When the tube was removed it was connected to a manometer to see if gassing had occurred. The tube exploded when an attempt was made to open it to the manometer. The grease had become very hard and brittle.

[REDACTED]

The following three specimens were samples of adhesives to be used in holding together the laminations in the magnet core. Two thin strips of aluminum were glued together with each adhesive and placed in the pile.

Bakelite cement	Became hard and brittle but still showed good adhesive properties.
Clear glyptal	Remained tough and plastic between the metal strips. Seemed to have better adhesion than the bakelite cement.
Silicone resin	Poorest of the three, but did show fair adhesion.

#### 5.6 Induced Activities

It is very often difficult to determine accurately the amount of gamma activity to be expected when materials are removed from the pile. This is especially true of materials of construction that have been in the pile for a long time. It is proposed that a large number of materials be subjected to pile radiation, and decay and absorption curves measured upon removal from the pile. These curves can then be analyzed for the proportion of each gamma contributor, its half life and energy. In most cases it should be possible, with these data, to determine the elements which are involved.

A small amount of preliminary work has been done on this problem, but due to a lack of manpower, it is progressing slowly at the present time. A few samples of metals have been removed from the pile, after two months irradiation, and data are now being collected. These preliminary data will serve mostly to determine the size samples that should be used and the method for taking the data. All of the necessary equipment has not yet been assembled.

#### 5.7 Hanford Slug Carrier

Apparatus is being assembled for an experimental investigation of the maximum slug temperature in a modified carrier to be used for shipment of hot Hanford slugs. J. A. Lane has calculated this maximum temperature to be 245° F based on a heat output of 660 watts for 12 slugs. If the carrier is loaded to its capacity of 19 slugs, the heat output and maximum slug temperature will be correspondingly greater.

Dummy aluminum slugs with a resistance wire imbedded in the center will be used in test work. Heat input will be supplied electrically. Maximum slug temperature, rate of temperature rise, maximum carrier temperature, and the time required for slugs and carrier to reach an equilibrium temperature can be determined with properly placed thermocouples.

5.8 Absorption of Radiation from an Oak Ridge Pile Slug

The final report on this problem is now being typed (ORNL-53). It shows the decay of a slug over a period of 150 days taken through several thicknesses of lead. A comparison is shown between experimental and theoretical values.

5.9 Packless Valve Development

Three companies are now collaborating with this laboratory in the development of a satisfactory bellows sealed valve, The Robert Shaw Control Company, Alloy Steel Products Company, and Hammal Dahl Company. The Robert Shaw valves, two manual and one remote controlled, are to be delivered in June for testing. The other companies are still in the design stage.

5.10 Steam Jet Tests

A Schutte and Koerting Steam Syphon Catalogue No. 217, size 1" suction by 1" discharge by 3/4" steam, made of 316 stainless steel was tested for steam pressure of 10 to 80 psi. The minimum and maximum flow rates were 21.4 and 64.2 liters per minutes (5.6 to 16.9 gallons per minute). Its starting suction was 19.5" of mercury. A 1/2-inch jet is now being tested.

5.11 Waste Treatment

A survey of the radioactive waste problem at this laboratory is being started to record the source, quantity and chemical composition of the various wastes. Two waste disposal processes are being investigated; the first for the 25 process aluminum nitrate wastes, and the second for decanted effluent from the chemical waste tank which is passed through the settling pond to the creek. In both problems it is expected that a combination of ion exchange, precipitation, and evaporation will be required to obtain the required concentration of the active wastes.

Adsorption of 25 aluminum nitrate waste on Dowex 30 (80 to 100 mesh) and Dowex 50 (300 to 500 mesh) cation exchange resins was studied in the laboratory to compare elution with nitric and sulfuric acids at 25° C and 100° C. The aluminum exhibited a tailing out effect under all conditions. The decontamination factor for beta was 6 and for gamma was 3 using Dowex 30 resin at 25° C when 94% of the aluminum was eluted to give the following decontamination factors: beta 2.7, gamma 1.3. In the semi-works columns, the Dowex 50 resin was investigated using 0.5M nitric acid and 5% oxalic acid at 25° C and 90° C. The highest beta decontamination, a factor of 25, was obtained with oxalic acid at 25° C with approximately 97% of the aluminum eluted. At 90° C the beta decontamination factor was 15 which was equal to that for nitric acid at 25° C and 90° C.

A sample of the waste effluent from the chemical waste tank (W-6) was adsorbed and eluted to give the following decontamination factors: beta 15, gamma 30.

The results of the ion exchange work indicated that the removal of the aluminum before adsorption would be desirable. Two precipitation procedures have been demonstrated to yield compact precipitates: By the crystallization of aluminum sulfate from an ethanol-water system, gamma decontamination factors of 10-20 were obtained. A granular aluminum hydroxide can be precipitated by the addition of formic acid and urea. Both of these procedures require large amounts of reagents and may not be practical. The use of ferric hydroxide as a scavenging precipitate is being considered.

## 6. Physical Facilities

### 6.1 Architectural Design

The design of a standard hood to handle  $\beta$  and  $\gamma$  activity up to 10 millicuries has been completed and accorded favorable comment from all interested parties. The novel feature of such a hood involves a floor level bypass which closes simultaneously when the hood doors are opened, providing a uniform flow of air through the hood. The behavior of the hood under usual operating conditions will be determined experimentally by means of a model, on the basis of which final drawings will be prepared. A sketch will be included in the next quarterly report.

### 6.2 Metallurgical Engineering Equipment

The new controls for the 20" x 30" Mesta Mill are being installed. This new equipment will provide dynamic braking and reverse as well as speed control for this mill. The 12" x 14" United Rolling Mill is being installed.

Several furnaces have been located at Y-12 which are available for transfer here. They are:

3 Type 56 - Hevi Duty, Used, Muffle Size, 5" x 7" x 15"

1 Type HD 10187, Hevi Duty, Used, Muffle Size, 7" x 10" x 18"

3 Type LA213N, Hayes, New, Muffle Size, 10" x 10" x 54"

The four Hevi Duty furnaces have been requisitioned. The Hayes furnaces are of a special design for use with 110 V, 3 phase current. Transformers are not available. If a source of power can be found for them, one will be set up to be used with the 12" x 14" mill. The type HD 10187 Hevi-Duty is not equipped with a transformer, but one can be acquired since this is a stock furnace.

Plans are being formulated for the future layout of Bldg. 101-B. In order to pursue the program which has been proposed for this section, some new equipment will be needed. A partial list follows:

1. Degreaser
2. Vacuum melting equipment
3. Cleaning tanks
4. 100 ton hydraulic press - 2 ram type
5. Forge hammer
6. Spot welder
7. Bar rolls for 12" x 14" Mill
8. Small roller leveler
9. Welding equipment
10. Milling machine
11. Lathe
12. High temperature furnace
13. Controlled atmosphere furnace

#### 7. Service Work

7.1 During the Quarter 60 lbs. of braze metal, 11.5% Si-Al alloy, was prepared for Brookhaven National Laboratory. This was cast into 4000 gm. ingots. It was rolled until a 0.060" sheet resulted with enough cross rolling to give a sheet 24" wide. It was then sheared into strips from 0.100" to 0.110" wide.

7.2 239 enriched uranium aluminum alloy slugs have been prepared for radiation at Hanford for subsequent use in the Pilot Plant. The enriched material was received as  $U_3O_8$ , and reduced in the usual manner to give an 18% alloy. The average uranium recovery for the 13 reduction heats was 98.27% and the uranium percentage varied from 18.06 to 18.51. There were 39 remelt heats and 19 scrap heats in which the alloy was diluted to 4.25%. The recovery in all these heats was within the analytical error; i.e., approximately 1%. The uranium percentage varied from 4.23% to 4.45% in the scrap heats.

These slugs are now ready for shipment, pending clearance at Hanford.

7.3 U-Al alloys of 30% to 40% U were cast and rolled to 0.035" for the Knolls Laboratory at Schenectady. No difficulties were encountered and yields were good.

7.4 Some progress has been made toward fabrication of a pile exposure tube for S. Siegel of the Physics Division. This tube is to be used as a source of fast neutrons for irradiating samples in the X-10 Pile. It is to be 12" long, have a mean diameter of 3.3", and be composed of an clad U<sup>235</sup> Al alloy. Using the punch and die which had been made for the fuel rod active plates, four 0.160" cores of 25% U-Al alloy (natural) were punched. These were fitted in a frame punched so that the cores were side by side, and separated by 1/4" dead aluminum with 2" aluminum around the outside. Frame thickness was 0.125". A cover of 0.080" was applied and the sheet rolled to 0.125". This gave a core with outside dimensions of 9.5" x 10-3/4". The sheet was trimmed to 13" x 10.4" and rolled in the sheet metal shop into a tube 13" long. The edges were then welded. Maximum variation from roundness was about 1/32". Plans are being made to fabricate the tube with enriched uranium.

7.5 The possibility of producing dispersions of bismuth in aluminum was tested briefly. According to the phase diagram the two metals are largely insoluble in the liquid region. However, the mutual solubility increases with increase in temperature. It should then be possible to produce dispersion by rapid cooling from a high temperature region of complete solubility. Two mixtures containing 10 and 20 weight per cent Bi were heated to 1400° C and cast in an iron mold. Re-heating of polished sections indicates Bi dispersion. The 20% Bi sample showed some separation of Bi at the bottom which might be decreased by more rapid cooling. The latter sample darkens rapidly on exposure to moisture.

7.6 Two small preferentially cooled ingots of gold and silver were prepared for use in thermal conductivity experiments by the Metallurgy Division.

7.7 Scrap Indium metal collected by the Chemistry Division was remelted, cast into billets, and cold rolled into various foil thicknesses.

## 8. Personnel

The Technical Division contained 91 technical and 89 non-technical employees as of May 28, 1948. This is a decrease of 10 technical and an increase of 2 non-technical employees over the figures shown for the last quarter ending March 1, 1948.

### Hires

- 1 Chemical Engineer (Kitzes)
- 2 non-technical employees (Burnett and Thomas)

Terminations

- 5 Chemical Engineers (Leverett, Briggs, Hanson, Weills and Levenson)
- 2 Civil Engineers (Hovorka and Kocur)
- 2 Metallurgists (Cockrell and Lawson)
- 2 Mechanical Engineers (Rochrenbeck and Hill)

Transferred in

- 1 Chemist from Chemistry Division (Gost)
- 1 Chemical Engineer from Chemistry Division (Ellison)
- 1 non-technical employee from Operations (Keck)

Transferred out

- 1 Stenographer to Power Pile Division (Tuttle)
- 1 Clerk to Chemistry Division (Snow)
- 1 Non-technical employee to Chemistry Division (Houser)

*Stuart Mc Lain*

STUART McLAIN

# PROGRESS STATISTICS FOR RESEARCH PILE PROGRAM

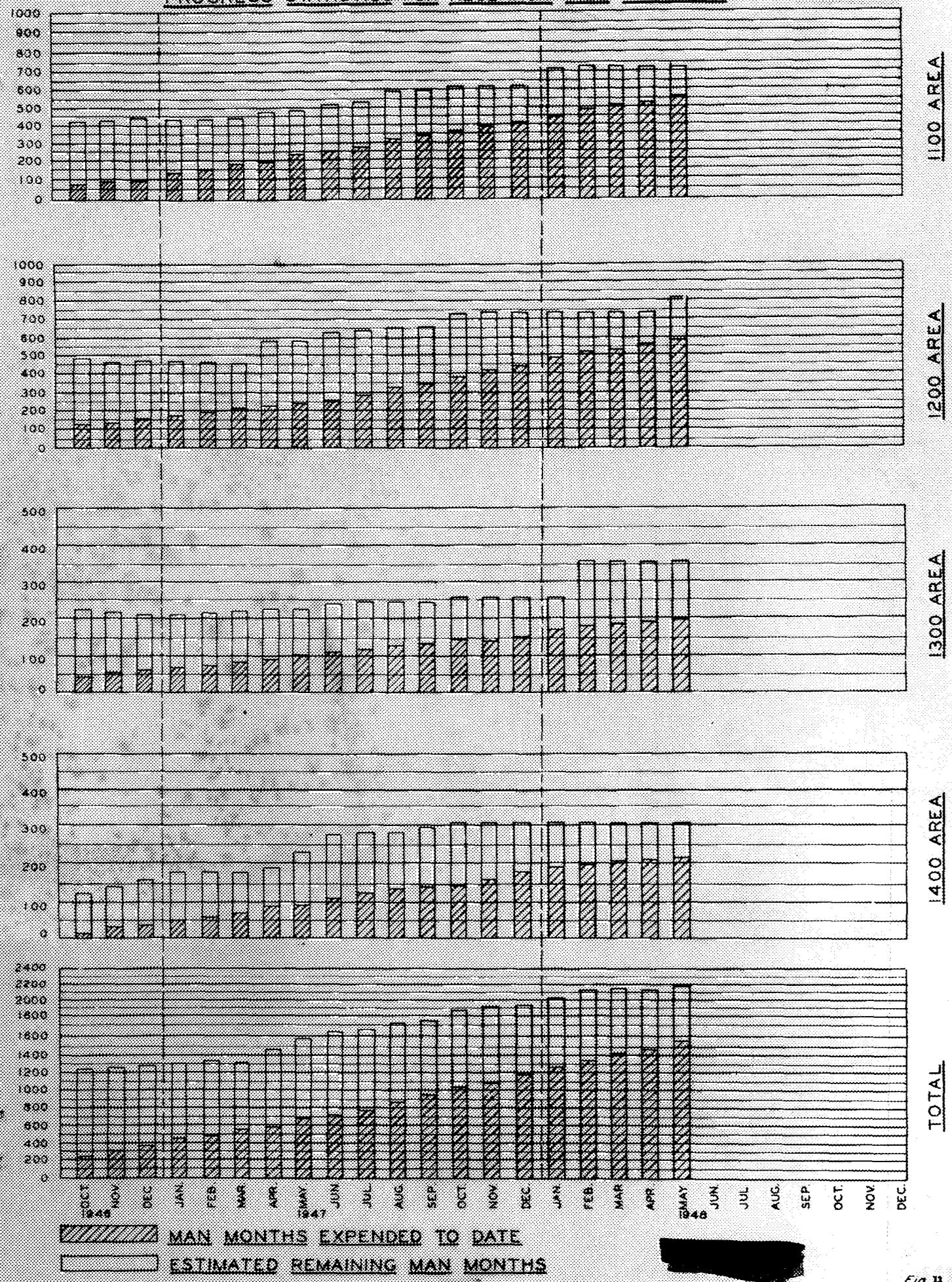


Fig. 11