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ORNL-1402
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 1952

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

September, 1952

J. P. Lane

Date Issued: OCT 7 1952

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OAK RIDGE NATIONAL LABORATORY

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TABLE OF CONTENTS

	Page
PROGRAM 4000 - REACTOR DEVELOPMENT	6
HOMOGENEOUS REACTOR PROJECT	6
Homogeneous Reactor Experiment	6
ISHR Design	6
Solution Chemistry	6
Radiation Chemistry	7
Dynamic Corrosion Studies	7
Boiling Reactor Experiment	7
AIRCRAFT NUCLEAR PROPULSION PROJECT	7
General Design	7
Aircraft Reactor Experiment	7
ANP Metallurgy	8
ANP Reactor Chemistry	9
ANP Corrosion	9
Heat Transfer and Physical Properties Research	10
Reactor Physics	10
ANP Experimental Engineering	11
PROGRAM 5000 - PHYSICAL RESEARCH	12
REACTOR OPERATIONS	12
PRODUCTION OF ISOTOPES	12
Radioisotope Production	12
Stable Isotope Production	12
PHYSICS	12
High Voltage Program	12
Neutron Diffraction	13
Neutron Velocity Selector	13
The 86-Inch Cyclotron	13
86-Inch Cyclotron Nuclear Physics	13
Electronuclear Machines	13
The 63-Inch Cyclotron	14
Isotope Analysis Methods Laboratory	14
Mass Spectrometer Laboratory	14
CHEMISTRY	15
Radiation Chemistry	15
General Reactor Chemistry	15
Chemistry of Uranium Raw Materials	15
Electrochemical Studies	16
Thorex	16

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TABLE OF CONTENTS Continued

	Page
METALLURGY	16
Ceramics Research	16
PROGRAM 6000 - BIOLOGY AND MEDICINE	18
BIOLOGY	18
Pathology and Physiology	18
BIOPHYSICS	18
Radioactive Liquid Waste Disposal Research	18
PROGRAM 9000 - RESEARCH FACILITIES	19
Conversion of Jones Construction Camp (Central Shops Building #7012)	19
Design, Various Projects	19
RADIOISOTOPE SALES	20
GROSS OPERATING COSTS	20
PERSONNEL SUMMARY	21

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

September, 1952

PROGRAM 4000 - REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Experiment (AEC Activity 4103.10)

Hydrostatic tests of the high pressure system of the HRE have been completed without disclosing any leaks. The system has now been filled with water and will be run in simulated operation at 250°C and 1000 psi pressure for a few days to see if any water is lost from the system during this period and as a final test of the system. If no losses occur and if all operations are satisfactory, fuel solution will be recharged and operation resumed.

ISHR Design (AEC Activity 4109.1)

Cell and equipment layouts have been made for a single region, intermediate scale homogeneous reactor that would be a prototype plutonium producer. Calculations indicate the feasibility of the proposed system and indicate that the reactor could be operated without control rods. The design was predicated on the use of UO_2SO_4 fuel solution at 250°C with the possibility of using UO_3 slurry fuel. Discouraging corrosion results for high concentration solutions at 250°C in stainless steel and the lack of information on slurry handling problems caused a re-examination of the ISHR program with the result that emphasis is now being placed on two-region (core and blanket) reactors that operate under conditions shown in the dynamic loop tests to be acceptable from the corrosion standpoint.

The two-region reactors being considered have a 4-foot core tank using 5 g U/l UO_2SO_4 fuel at 250°C with a 2-foot blanket. Calculations indicate that with a 1/8-inch thick stainless steel core tank and a 4000 g Th/l or thorium oxide blanket, one gram of U^{233} can be obtained in an unpoisoned reactor for each gram of U^{235} burned, assuming D_2O is used both in the core and in the blanket. Better conversion could be obtained with a 1-cm zirconium tank. Drawings of the core tank and pressure vessel assemblies and cell layouts are now being made.

Solution Chemistry (AEC Activity 4109.31)

Studies of the corrosion inhibiting effect of alkali metal hydroxides and fluorides added to uranyl fluoride solutions, although carried out in a very preliminary way, give some encouragement. It seems clear that there is formation of a complex in these cases and that it is possible to bring about appreciable decreases in the acidity of uranyl fluoride solutions without adversely affecting solubility or thermal stability.

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Radiation Chemistry (AEC Activity 4109.31)

In-pile experiments in which uranyl sulfate solutions are exposed at 250°C in stainless steel bombs indicate that at least at the higher power densities the corrosion rate in the pile is greater than that under the same conditions outside the pile. Similar tests carried out in titanium bombs indicate good solution stability and very low corrosion. In a recently completed test, 6.5 days in the pile at 250°C with a power density of 1 KW/liter, the solution after exposure was clear with no precipitate. The amount of titanium in solution was undetectable analytically.

Dynamic Corrosion Studies (AEC Activity 4109.4)

A welded cross removed from the 347 stainless steel loop in which 100 g U/l UO_2SO_4 has been circulated for $\sim$ 3000 hours at 100°C shows none of the excessive pitting usually seen at such points of high turbulence in loops operating at higher temperatures with similar concentrations of uranium. The inner surfaces of the cross are free from pits and their appearance indicates no worse attack than the rest of the loop in which the general corrosion rate has been below 0.5 mils per year.

Boiling Reactor Experiment (AEC Activity 4112.1)

Design of a small scale boiling reactor ("teapot") for use in studying stability and power removal characteristics is now in progress. The layout of the HRE #2 building in which boiling reactor experiments will be carried out has been prepared and detailed design is being started. The building is nearly identical with the HRE building except that shielded compartments are located below floor level and a canal for storage has been included.

A better understanding of the mechanisms of power removal from boiling reactors has been obtained from a recently completed study.

AIRCRAFT NUCLEAR PROPULSION PROJECT

General Design (AEC Activity 4401.1)

Detail arrangements have been made with the critical experiment group for criticality experiments on the reflector-moderated (fireball) type of reactor. The first assembly will utilize uranium discs sandwiched between 1" thick cans of stainless steel containing Na. These will be loaded into the 3" square pigeonholes of the tables used for the GE air cycle reactor. If the critical mass of this assembly is reasonably close to IBM predictions, the canned Na-uranium disc assemblies will be removed and replaced with cans made of square aluminum tubing. These cans will be filled with the salt removed from the ARE critical assembly.

Aircraft Reactor Experiment (AEC Activity 4401.1)

Since dynamic studies of reactor behavior made on the reactor simulator indicated that the worst possible conditions occasioned by a leak of NaK into

the fuel was tolerable, NaK instead of BeF_2 - NaF was chosen as the reflector coolant. The reflector coolant circuit design was completed and specifications for the heat exchangers were released to the manufacturer of these units.

Pump designs have been frozen and some design drawings are already released to the shop. The pump incorporates a combination packed seal and frozen seal. The establishment of the pump design has permitted work to proceed on the surge tank design. The fuel injection equipment is being fabricated in the Y-12 shops.

Plans for the off-gas disposal system have been revised. Fission gases will now be held in storage tanks until the activity has decayed to a safe value for release up the stack. The change from the carbon cartridge absorber was made because of the excessive amount of liquid nitrogen required to cool the carbon.

The control equipment has not yet arrived but the vendors advise that shipment will be made early in October. An on-off type of servo has been checked on the analogue computer and its performance has been found to be satisfactory. Drawings of this servo are now being made to fit into the control assembly.

Installation in the 7503 Building is well under way. A considerable amount of water piping, electrical power wiring, and setting of auxiliary equipment has been done. The work in the building will accelerate materially when the pressure shell and heat exchangers arrive, delivery of which is expected in October.

ANP Metallurgy (AEC Activity 4401.5)

The calibration of the electrode force gage on the electric resistance spot welder was completed. Since electrode force is an important variable in spot welding, this calibration should permit the successful welding of materials, such as stainless steel and Inconel, whose welding conditions have previously been determined. This will also allow the determination of the electrode force needed to weld such materials as columbium, molybdenum, and thorium, whose welding conditions are not known.

The liquidus temperatures of the Pd-Ni-Be brazing alloys with 2, 3, and 4% beryllium additions were found to be above 2250°F. Since the 1% beryllium alloy melted well at 2150°F, it appears that these later alloys have an excess beryllium content. Therefore, beryllium additions of 0.25%, 0.50% and 0.75% will be tried for the purpose of lowering the melting point.

A BeO block has been immersed in molten sodium at 1500°F for 215 hours to simulate certain ARE operating conditions. The block was observed to be free of cracks and surface spalls at the end of the test, but to have a weight gain of 1% and a growth of 0.05 inches both in length and width. Some surface spalling was observed after the BeO was placed in water to remove the sodium.

Results of 100 hour corrosion tests of NaOH and stainless steel are as follows:

Material	Temp., °C	Metallographic Notes
304	350	No attack.
304	450	No attack.
304	550	One-half to one mil of light intergranular penetration.
304	650	Unattacked material decreased from 34 to 24 mils.
316	350	No attack.
316	450	No attack.
316	550	One to two mils intergranular attack.

In some tests run with (NA-K-Li-U)F_x, sections of the testing tube were taken both above and below the bath level. Corrosive attack was noted on all sections examined. This indicates that attack can be expected on metals exposed to the vapor phase of the fluoride mixtures.

Details of these tests are presented below:

Material	Liquid Phase Notes	Vapor Phase Notes
Inconel	Erratic attack; subsurface voids to 1-1/2 mils.	Erratic attach, subsurface void to 1 mil.
304	Erratic attack; intergranular with a few subsurface voids to 1 mil.	Intergranular attack to 5 mils.
309	Subsurface voids to 3 mils.	Subsurface voids to 3 mils.

ANP Reactor Chemistry (AEC Activity 4401.5)

The cooling curves of the fuel containing 55% NaF; 40% BeF_2: 5% UF_4 (mol basis) were found to have breaks at 465°C and 340°C. The solidified fuel was composed of two layers; the lower layer was somewhat more than half the total and contained most of the $NaUF_5$.

Systems containing sodium and potassium fluoborates and systems containing aluminum fluorides have been studied, but no promising fuel or coolant have been found so far.

ANP Corrosion (AEC Activity 4401.5)

Experimental studies of corrosion-inhibiting additives to fuel mixtures were continued with some evidence that a 10% addition of $NaAlO_2$ to sodium hydroxide in a tilting furnace type experiment inhibits corrosion of nickel, and

additions of zirconium metal or zirconium hydride to the fuel 50% NaF; 46% ZrF_4: 4% UF_4 (mol basis) reduces corrosion of Inconel and #316 stainless steel.

Heat Transfer and Physical Properties Research (AEC Activity 4401.5)

The thermal conductivity of the fuel, NaF-ZrF_4-UF_4 whose composition is 50-46-4 mole % respectively, has been measured between 600°C and 800°C. Over this temperature range the average conductivity is 1.4 Btu per hour/ft/°F. It is interesting to note that for those fluoride salts whose thermal conductivity has been measured there is a definite correlation between the conductivity and the uranium concentration of the mixture, i.e., the salts with the lower thermal conductivities have the higher uranium concentration.

The viscosity of the proposed fuel carrier, NaF-BeF_2, whose composition is 57-43 mole % respectively, has been measured at several temperatures. These data are tabularized below.

Temperature °F	Viscosity cp
620	12
730	8.5
870	5

With the exception of BeF_2-containing fluorides (whose viscosities are also dependent upon the BeF_2 content), the more viscous fluoride mixtures are those with the higher uranium concentration.

In order to interpret properly corrosion results in thermal convection loops, an analysis has been undertaken to determine the mean circulation velocity of the fluid. One analysis of a particular loop of 1/2" IPS schedule-40 Inconel pipe circulating the fuel NaF-KF-LiF-UF_4, composition 10.9-43.5-44.5-1.1 mole % respectively, was calculated to have a circulating velocity of only 0.2 ft/sec - corresponding to a Reynolds number of about 800. As this Reynolds number is well within the laminar flow region, corrosion data from these loops should not necessarily be considered to be a good test of the ARE system (wherein turbulent flow will exist) should the dynamic effect play a major role in corrosion. Higher velocities may be obtained in thermal convection loops by heating and cooling the hot and cold legs in such a manner as to get uniform hot and cold leg temperature distributions.

Reactor Physics (AEC Activity 4401.7)

The kinetic equations of the circulating fuel reactor have been slightly generalized to include the case in which there are several alternative paths for the fuel elements, each path having a different transit time and different power density. (The power density is still assumed to be independent of the position along a path.) The result of this investigation confirmed what was anticipated; namely, that oscillations are always damped. The only exception is that stationary (but not antidamped) solutions can exist, if all transit times are multiples of the period of the oscillation - a condition which hardly will be fulfilled.

ANP Experimental Engineering (AEC Activity 4401.1)

Two Inconel loops were filled with purified chemical lead. The first which had been cleaned by circulating NaK and then evacuating while hot did not circulate at all. The second loop was similarly cleaned with NaK followed by a water wash and hot evacuation. This loop circulated only 2 1/2 hours. A 430 series stainless steel loop which had been cleaned by a nitric acid rinse, water rinse, and a hot cleaning cycle circulated lead for 16 1/2 hours. A 410 series stainless steel loop which had been NaK cleaned followed by a water wash, nitric acid rinse, a second water wash and hot evacuation circulated 351 hours before plugging. The plugs will not melt at 1575°F, and analysis shows them to have high nickel content. However, the diffraction patterns do not check with any known lead or nickel system.

PROGRAM 5000 - PHYSICAL RESEARCH

REACTOR OPERATIONS

The total accumulated KWH for X-10 pile operations during September were 2,179,073 averaging 3353 KW per operating hour. Pile down time was 9.7% as compared with 10.1% in August. The pile has been completely charged with silicon bonded slugs.

The total accumulated KWH for LITR operations during September were 964,316 averaging 1493 KW per operating hour. Down time was 10.3% as compared with 22.0% in August.

PRODUCTION OF ISOTOPES

Radioisotope Production (AEC Activity 5111)

There were 870 radioisotope shipments during September.

Stable Isotope Production (AEC Activity 5121)

Titanium and lithium were processed during the month of September yielding seven isotopes. Overall production innage for the four calutrons was 1634 hours. Outage time was 27.6% as compared with 37.3% during August. The integrated output was 595,050 ma hours for the Alpha channels and 38,921 ma hours for the Beta channels.

Basic experimental work preliminary to the recovery of enriched isotopes of the inert gases was carried out.

Chemical processing refinement was conducted on isotopes of chlorine, tin, boron, barium, lithium and calcium. Chemical services were supplied to the calutrons for the separation of isotopes of lithium and titanium.

There were 23 stable isotope shipments during September.

PHYSICS

High Voltage Program (AEC Activity 5211)

The fission cross section ratio of U^{238} to U^{235} has been determined from threshold to 4 Mev. The curve is flatter than that previously assumed.

The elastic scattering of neutrons from nitrogen has been determined over a neutron energy range from 0.4 Mev to 2.3 Mev. The distribution characteristic is a high probability of forward scattering. At the 1.6 Mev resonance the angular distribution changes markedly.

The aluminium (p,n) threshold was found to be 5.819 $\pm$ .01 Mev proton energy. The B^{11} (p, γ) yield has been run and several resonances were found. In the tritium (p, γ) reaction the 90° yield has been determined up to 5.1 Mev. The angular distribution of neutrons from the C^{13} (p,n) reaction has been measured as a function of energy up to 4 Mev.

The total reaction cross section for the (He^3, H^3) reaction has been run from 100 Kv to 800 Kv when a thin ZrT_2 target was used. When angular isotropy was assumed for the decay products, the total cross section rose continuously over this range.

Neutron Diffraction (AEC Activity 5211)

Work is underway on ferro-magnetic alloys containing two transition elements. The magnetic moments of the elements such as Fe, Co, and Ni will be determined by neutron diffraction techniques when the elements are mixed in various proportions.

Neutron Velocity Selector (AEC Activity 5261)

The measurement of the transmission of a thin sample of U^{236} reveals a resonance of 4.4 electron volts. Measurements of Th^{230} are contemplated in the near future.

The 86-Inch Cyclotron (AEC Activity 5261)

A beryllium target has been bombarded at one milliampere for the production of fast neutrons. The Biology Division has found that under these conditions there are regions near the cyclotron suitable for fast neutron irradiation of biological materials; the gamma ray and slow neutron backgrounds can be controlled and a suitable range in neutron flux is available.

86-Inch Cyclotron Nuclear Physics (AEC Activity 5261)

The study of the products of proton induced fission of natural uranium has been extended to include the yield ratios for eleven elements; bombardments and chemical separations have been completed and the final samples are being counted.

Electronuclear Machines (AEC Activity 5261)

An apparatus has been built for finding the median plane in a cyclotron magnetic field and for locating the center of the shimmed field. Use of the apparatus indicates that for the 22-inch cyclotron the median plane of the magnetic field corresponds within $\pm$ 0.125" to the geometric median plane of the tank with the electromagnetic shims in operation. Magnetic and geometric centers were determined to agree within $\pm$ 0.5".

The 63-Inch Cyclotron (AEC Activity 5261)

The range distribution of nitrogen ions was obtained by exposing Ilford C-2 photographic plates to the internal cyclotron beam. The maximum range in the emulsion is 15.5 microns, corresponding to an energy of 26 Mev; the center of the range distribution curve is 10.5 microns, corresponding to an energy of 21 Mev. Measurements of the range distribution as a function of absorber thickness were made with aluminum absorbers of 0.18, 1.46, and 1.94 mg/cm^2 placed in front of the emulsion.

Isotope Analysis Methods Laboratory (AEC Activity 5261)

All five of the predicted J = 2 to J = 3 rotational transitions in vinylacetylene have been measured in the region 26,600 mc to 27,846 mc to $\pm$ 0.05 mc. Molecular parameters have been calculated as follows:

$$b = 4744.8717 \text{ mc}$$
$$c = 4329.7550 \text{ mc}$$
$$\delta = 8.90547 \text{ kc}$$
$$D_{jK} = -87.6 \text{ kc}$$

A new mathematical treatment of the rotational data has been devised to permit rapid evaluation of the centrifugal distortion constants.

The X-ray fluorescence method of analysis for zirconium in uranium has been investigated for the cases of 40% lead and 20% tin impurity with no deleterious effect on the analysis. Attempts to observe the arsenic spectrum from K capture decay of Se^{75} gave negative results, possibly due to the very high γ ray background from the Se^{75} decay.

Mass Spectrometer Laboratory (AEC Activity 5261)

A sequence of tests have been completed on UO_2, U_3O_8 and UO_2Fe_2. It was found that the U_3O_8 disproportionated into UO_3 and UO_2. The vapor pressures of the product oxides are enough different to allow them to be studied individually at different temperatures. No thermal positive ions are found from UO_3 but UO and UO_2 thermal positive ions are found from UO_2 as was previously reported. The UO_2Fe_2 gave rise to UF_5 ions at low temperatures and UO_2 ions at high temperatures. UO_2SO_4 is also under investigation.

A new General Electric Mass Spectrometer arrived September 1952 and is in operation.

CHEMISTRY

Radiation Chemistry (AEC Activity 5311)

Preliminary experiments in a study of bubble nucleation by fission recoil tracks indicate that single fissions provide bubble nuclei which cause ebullition in uranyl sulfate solutions superheated above a reproducible minimum temperature.

A constant initial yield has been observed for hydrogen peroxide in water irradiated as ice at temperatures from $4^{o}K$ to $77^{o}K$.

General Reactor Chemistry (AEC Activity 5361)

The neutron capture cross section of Pa^{233} has been determined to be 130 ± 30 barns. This preliminary value is about three times as high as other reported values. The technique of this determination was to measure the U^{234} produced by irradiation of thorium samples in the LITR and the Hanford piles; adjustments were made for Th^{233} and U^{233} neutron captures which also produce U^{234}.

Chemistry of Uranium Raw Materials (AEC Activity 5361)

Water leaching of beneficiated Leached Zone ore after baking with ammonium bisulfate has been carried through four countercurrent stages. Ninety percent of the phosphate was extracted. The liquor contained 140 g P_2O_5 /liter and 0.14 g U/liter after most of the aluminum had been removed as alum.

Unbeneficiated Leached Zone ore was leached satisfactorily at 50% pulp density after a low-temperature nitration bake. The nitrate content of the leach liquor was within the limit required for an acceptable nitrogen-phosphorus fertilizer product.

Approximately 75% of the uranium and 50% of the molybdenum can be extracted from an $800^{o}C$ lignite ash with ammonium carbonate solution. Roasting temperatures above $800^{o}C$ render the uranium insoluble in a subsequent carbonate leach. About 90% of the uranium has been extracted from raw lignite by severe treatments with 40% sulfuric acid.

The study of the solubility behavior of uranium $^{+6}$ phosphate in 0.001 to 15M phosphoric acid has been completed. The following compounds have been identified to be the stable solid phases in the concentration ranges indicated.

0.001----0.014M	H_3PO_4:	$(UO_2)_3(PO_4)_2 \cdot 4H_2O$
0.014----6.1M	H_3PO_4:	$UO_2HPO_4 \cdot 4H_2O$
6.1 ----15M	H_2PO_4:	$UO_2(H_2PO_4)_2 \cdot 4H_2O$

Calculations based on pH titration data and solubility data in uranyl phosphate, perchloric acid solutions indicate the following complexes along with their formation constants.

$UO_2H_3PO_4^{++}$	k = ca. 17
$UO_2H_2PO_4^{+}$	k = ca. 20
$UO_2(H_2PO_4)_2$	k = ca. 18
$UO_2[(H_2PO_4)_2H_3PO_4)]$	k = ca. 200

These k values apply to 1M perchloric acid solution and are defined as

$$\frac{[UO_2H_x(PO_4)_n^{(2+x-3n)}][(H^+)^{3n-x}]}{[UO_2^{++}][H_3PO_4]^n}$$

wherein the bracketed quantities refer to concentrations in moles per liter.

Electrochemical Studies (AEC Activity 5361)

In the search for suitable electrodes for a water decomposition cell for deuterium separation, the use of a palladium cathode and a hydrogen anode gave a hydrogen-deuterium separation factor of 6 when operating at 0.25 volts. This voltage is a factor of 10 less than that of conventional cells.

Thorex AEC Activity 5361)

The chemical flow sheet for the thorex process has been established; the equipment flow sheet is being evaluated. The Purex pilot plant is being decontaminated prior to conversion to Thorex pilot plant operations.

Small samples of pure U^{233} are being prepared for neutron capture cross section measurements. Pa^{233} will be adsorbed on a silica gel column and allowed to decay to U^{233}. The U^{233} can be stripped with nitric acid.

METALLURGY

Ceramics Research (AEC Activity 5411)

A glass with beryllium silicate as a constituent and containing particles of dissolved beryllium oxide has been developed as a reflector for reactors. Since the reflector temperatures would be above the annealing temperature of the glass, the problem of heat fracture is eliminated. In the case of the ANP "fireball" reactor the critical mass of U^{235} was calculated to be about 35 pounds when this type reflector was used.

Hafnium carbide has been successfully synthesized. This material, although brittle, is very hard; the hardness was measured at a Vickers number of 2910. This somewhat harder than boron carbide.

PROGRAM 6000 - BIOLOGY AND MEDICINE

BIOLOGY

Pathology and Physiology (AEC Activity 6400)

Experiments have shown that radiothyroidectomy has a marked enhancing effect on a 24-hour retention of radioiodine and that pituitary tumors grafted in the leg muscle of mice have an enhancing effect on the uptake of radioiodine.

BIOPHYSICS

Radioactive Liquid Waste Disposal Research (AEC Activity 6510)

In the program of developing less expensive means of waste disposal, a large concave-shaped pit, 100 by 200 feet by 15 feet deep was dug in the shale soil formation near White Oak Lake in the ORNL Area for the purpose of storing radioactive liquid waste. To date about 16,000 gallons of waste containing 300 curies of activity have been stored here. Wells 90 feet deep have been placed in the vicinity of the pit to test the dispersal of radioactive material by ground waters; the activity in the test wells has increased, but analytical results indicate that the activity is solely ruthenium. Several soils are being tested to compare their ability to adsorb radioactive material with the shale soil of the pit.

Conversion of Jones Construction Camp (Central Shops Building #7012)

The concrete floor slab has been poured. The septic system has been installed.

Design, Various Projects

In the design stage are these projects: the I^{131} Plant, the Boiling Homogeneous Reactor Experiment Building, the Tower Shielding Experiment, the Research Reactor, and the OCRA Water Supply.

RADIOISOTOPE SALES

Sales	July	August
Transfers within AEC	$ 30.00	$ 120.10
Cash Sales	5,876.72	4,767.85
Foreign	181.70*	1,105.90
Cancer Program (Free)	14,767.52	19,863.12
Technical Cooperation Plan	-0-	-0-
Total Sales and Transfers FY 1953 to Date	65,378.42	151,058.32
Total Cancer Program (Free) to Date	1,433,483.16	1,453,346.28
Technical Cooperation Plan (Shipment to Date)	15,152.55	15,152.55

GROSS OPERATING COSTS
(Including X-10 & Y-12)

(a)	Actual Cost for August 1952	2,400,026	
	Actual Plant and Equipment Cost	371,813	
	Construction Program "H"	19,397	
	Total Operating and Construction Cost for August 1952		2,791,236
(b)	Estimated Operating Cost for September 1952		2,800,000
(c)	Actual Accumulative FY 1953 Operating Cost through August 1952	4,750,987	
	Actual Accumulative FY 1953 Plant and Equipment Cost through August 1952	727,333	
	Actual Accumulative FY 1953 Construction Program "H" Cost through August 1952	44,223	
	Total Accumulative FY 1953 Operating, Construction, and Plant and Equipment Cost through August 1952		5,522,543
(d)	Estimated Operating Cost FY 1953 through August 1952		8,322,543

PERSONNEL SUMMARY

	Number of Employees September, 1952	New Hires September	Terminations September
Administration	78	1	2
Operations*	119	0	1
Engineering, Shops and Mechanical	915	12	7
Laboratory and Research	1483	43	75
Protection	181	0	2
Service	395	3	2
Total	3171	59	89

* Includes Electrical Distribution and Steam Plant as well as the Operations Division.