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AEC RESEARCH AND DEVELOPMENT REPORT

LABORATORY RECORDS
1954

OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1954

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OAK RIDGE NATIONAL LABORATORY
STATUS AND PROGRESS REPORT

March, 1954

F. T. Howard

and

W. H. Sullivan

Date Issued: APR 7 1954

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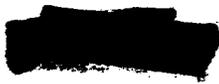
TABLE OF CONTENTS

	<u>Page</u>
PROGRAM 2000 - SOURCE AND FISSIONABLE MATERIALS	6
Waste Metal Recovery	6
Excer Process Development	6
Feed Materials	6
PROGRAM 3000 - WEAPONS	6
RaLa Production	6
PROGRAM 4000 - REACTOR DEVELOPMENT	7
HOMOGENEOUS REACTOR PROJECT	7
Homogeneous Reactor Experiment	7
Homogeneous Reactor Test	7
Reactor Physics	7
In-Pile Loops	7
Dynamic Corrosion Loop Tests	8
Engineering Development	8
Instrumentation and Controls	9
AIRCRAFT NUCLEAR PROPULSION PROJECT	9
Aircraft Reactor Experiment	9
General Aircraft Reactor Design	9
Experimental Engineering	10
Mathematics and Computation	10
Reactor Chemistry	10
Corrosion Research	11
Metallurgy - Mechanical Properties	12
Metallurgy - Welding	12
Reactor Control	12
Shielding Research	13
Fuel Recovery	13
GENERAL REACTOR	14
School of Reactor Technology	14
PROGRAM 5000 - PHYSICAL RESEARCH	14
REACTOR OPERATIONS	14



TABLE OF CONTENTS (Continued)

Program 5000 (Continued):	<u>Page</u>
ISOTOPE PRODUCTION	15
Radioisotope Production	15
Radioisotope Research	15
PHYSICS	15
Neutron Diffraction	15
Low Temperature Studies	16
High Voltage Program	16
Theoretical Physics	16
Interaction of Radiation and Matter	17
CHEMISTRY	17
Chemical Separation of Isotopes	17
Instrumentation for Nuclear Chemistry	18
Hot Laboratory Research	18
Chemistry of Corrosion	18
Ion Exchange Technology	18
THE ORACLE	19
PROGRAM 6000 - BIOLOGY AND MEDICINE	20
BIOLOGY	20
Cytogenetics	20
Microbiology	21
Radiation Protection	21
Biochemistry	22
Plant Biochemistry	22
Biophysics	22
Pathology and Physiology	23
Enzyme Chemistry	23
RADIOISOTOPE SALES AND COSTS	25
GROSS OPERATING COSTS	25
PERSONNEL SUMMARY	26



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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

March, 1954

The subject material of this report represents approximately half of the Laboratory's program, which is covered with some exceptions on a bimonthly schedule.

PROGRAM 2000 - SOURCE AND FISSIONABLE MATERIALS

Waste Metal Recovery - The metal recovery plant for the recovery of plutonium and americium from Hanford slag and crucible wastes began operation on March 10. Approximately 600 g of plutonium has been isolated with a solvent extraction loss of only 0.15%. (AEC Activity 2344)

Excer Process Development - In an exploratory run on the Excer process flow-sheet (Fig. 3.1 ORNL-1561) for the conversion of uranyl nitrate to dry uranium tetrafluoride, decontamination factors of 1500 for zirconium, 150 for niobium, and 17 for ruthenium were obtained. Dehydration of $UF_4 \cdot 3/4 H_2O$ in an atmosphere of HF or N_2 at $400^\circ C$ for 30 min gave a UF_4 product containing only 0.1% water. (AEC Activity 2604)

Feed Materials - Studies on reduction of thorium chloride in a proposed continuous process showed that $ThCl_4$ in propylenediamine solution could be reduced to thorium metal by lithium amalgam at $100^\circ C$. The thorium remained in the amalgam phase as a suspension. Lithium amalgam reduction of uranium tetrachloride in anhydrous isopropyl alcohol gave a poor yield and was accompanied by side reactions.

Ammonium diuranate precipitated from aqueous uranium solutions by treatment with urea (which hydrolyzes) were more crystalline and could be filtered 20 times faster than those precipitated with ammonium hydroxide. This precipitation technique may find application in the processing of uranium ores and in the recovery and separation of uranium from waste. (AEC Activity 2XXX)

PROGRAM 3000 - WEAPONS

RaLa Production - The standby purification equipment, which has been out of service for eight months because of a "thermowell" weak in the tantalum product evaporator was repaired. The product evaporator was replaced with a unit of a new design which is expected to allow closer operating control. The Teflon liners in the sampling valves were replaced because the high-intensity radiation had caused extensive cracking of the plastic. (AEC Activity 3702)

PROGRAM 4000 - REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Experiment - The final run for xenon production was completed and, after a 24-hour decay period, approximately one thousand curies Xe^{135} was separated for neutron cross section measurements.

Following the xenon collection, decontamination of the fuel circulating system was begun. The initial radiation level in the reactor cell, after removal of the fuel, was ~ 2000 R/hr. The activity was reduced to ~ 100 R/hr by rinsing with water, then circulating 8 M HNO_3 and 10% NaOH-tartrate-peroxide solution.

The heavy water was removed from the reflector system and dismantling of the reflector cell was almost completed. A 15-ft deep concrete pool was constructed for the storage of contaminated reactor components. (AEC Activity 4103.1)

Homogeneous Reactor Test - The major effort on the HRT is being applied to the design and fabrication of the larger components of both the fluid fuel and blanket systems. The heat exchanger designs were completed and sent to prospective fabricators for bids. The core and pressure vessel contract was revised to include mechanical and hydraulic experiments on a full-scale mock-up. The Westinghouse Electric Company agreed to provide titanium parts for the main circulating pumps of both the blanket and core systems. The method of pressurizing the reactor has not been decided; both steam and gas pressurization of both systems are being studied. (AEC Activity 4103.1)

Reactor Physics - Calculations of HRT statics are about 80% complete. Preliminary investigation of HRT dynamics shows the maximum pressure rise to be less than 200 psi for a 1.5% change in reactivity. The dynamics of the complete HRT system are now being studied. The equations of motion are being coded for solution with the ORACLE; results are expected by May 1, 1954. (AEC Activity 4109.2)

In-Pile Loops - A mock-up of the in-pile loop system was completed and put in operation. The mock-up consists of all components including the loop, loop container, shield plug, equipment chambers containing the auxiliary equipment, and the control panel. Preliminary tests indicated satisfactory performance of all components. The loop has run continuously for ~ 100 hours (total accumulated operating time is ~ 200 hours) with uranyl sulfate solution containing 40 g U/l and with 0.013 M copper sulfate as an internal catalyst for the recombination of hydrogen and oxygen. Daily additions of hydrogen and oxygen in stoichiometric ratios have indicated, in the preliminary tests, that the copper sulfate catalyst is performing as expected.

Two in-pile loops for the test work were built, and a third loop, which is to be installed in the LTR, is under construction. All-metal bearings (Stellite 98M2-to-Stellite 98M2) were fabricated and are being tested as possible replacements for the Stellite 98M2-to-graphitar No. 14 bearings now in use in the small 5-gpm circulating pump.

PROGRAM 4000 - REACTOR DEVELOPMENT (Continued)

Modifications at the LITR required for installation of the loop in hole HB-4 are virtually complete. All service facilities and building alterations, including a hot drain, were completed. Shielding for the equipment chambers at the LITR face is ready for placement. All piping and valves were installed in the equipment chambers and all auxiliary equipment, with the exception of the Baldwin pressure transducers, and are now being put in place. Delivery of the pressure transducers is expected by April 1. Fabrication of the in-pile loop control panel was completed and all instruments are in place. Wiring of the instruments is underway and the panel should be ready for installation at the LITR by April 20. (AEC Activity 4109.4)

Dynamic Corrosion Loop Tests - An experiment recently completed in the all-titanium loop clearly demonstrates the resistance of titanium (and zirconium) to corrosion by uranyl sulfate solutions. The loop was operated at 275°C for 400 hours with a solution containing 1.34 M uranyl sulfate. At the end of this run the specimens showed no detectable change in weight but had developed a thin, blue film, probably less than 1000 Å thick. There was no change in the appearance of the interior loop surfaces, including the titanium impeller and welds. Zirconium specimens included in the loop developed thin, black films and showed very slight weight gains. Under the same test conditions stainless steel specimens would have corroded at a rate in excess of 1000 mils per year.

The corrosion resistance of stainless steel to 0.04 M UO_2SO_4 containing 0.006 M H_2SO_4 at 250°C was demonstrated in a series of tests extended to 3500-hours duration. The small amount of acid was sufficient to stabilize the solution but not to change, materially, the corrosion damage. Corrosion test pins exposed for 100 hours at flow rates of 19 and 28 fps (feet per second) showed average weight losses of 2 and 3 mg/cm², respectively; increasing the exposure time did not increase the weight loss. Pins exposed at 42 fps lost about 6 mg/cm² and showed no consistent increase in weight loss with increased exposure time. The data were not as consistent, however, at the highest flow rates as at the lower rates. The indications were that at slightly higher flow rates the oxide film would not have been stable and corrosion would have been severe. In other words, flow rates at least as high as 35 fps are tolerable under the above conditions. (AEC Activity 4109.4)

Engineering Development - The 4000-gpm loop was assembled and preparations are underway for thorough leak testing. Fabrication techniques specified for reactor work were used for the large pipe, and, thus, the leak testing operations should provide some indication of how tight one may expect these systems to be.

A modified HRE-type pulsafeeder was operated at 2000 psi and at 72 strokes per minute. This higher speed, almost double the HRE speed, was attained by improving the check valve design.

Preliminary designs of medium-size pumps and blowers for the HRT and for prototypes of future large reactor components are nearing completion at

PROGRAM 4000 - REACTOR DEVELOPMENT (Continued)

Westinghouse and Allis-Chalmers. These companies will make fixed-price proposals for the final design and fabrication of this equipment.

Abrasion tests of fluidized thoria pellets in water at room temperature on samples of stainless steel, zircoloy, and brass were summarized in memorandum CF-54-3-44. (AEC Activity 4109.4)

Instrumentation and Controls - The practicability of constructing the HRT so that it can be flooded for under-water maintenance is being investigated. A steel tank is being installed for use in testing instruments, components, and connecting cables under thirty feet of water. Water-proof and radiation-proof power wiring was requisitioned and similar thermocouple wire is being ordered for test purposes. A survey of waterproofing information in technical and manufacturer's literature was completed.

Several manufacturers were visited to discuss valve problems and to determine their interest in manufacturing shut-off valves with extremely small leakage across the seat when shut-off at the operating conditions of 2000 psi and 300°C. Bellows were ordered from two manufacturers for use in ORNL-designed valves for the above conditions. (AEC Activity 4109.6)

AIRCRAFT NUCLEAR PROPULSION PROJECT

Aircraft Reactor Experiment - A spare heat exchanger was opened for examination; it was found that coated welding rod had been used for some of the original welds. Since it was felt that because of the corrosion problem welds of this type would not be dependable in the ARE system, the fuel and the sodium heat exchangers were removed and are being refabricated. It is estimated that this delay will postpone initial testing of the ARE system until sometime in June. (AEC Activity 4401.1)

General Aircraft Reactor Design - A study was made to determine the effects of the reactor temperature, power density, and radiation doses inside and outside the crew compartment on aircraft gross weight. The results of the calculations for three design conditions show that the gross weight of the airplane is relatively insensitive to reactor power density and temperature for Mach 0.9, sea-level operation but that it is quite sensitive for supersonic high altitude conditions. Also, an increase in reactor temperature level of 100°F is more effective in reducing gross weight than is a factor-of-2 increase in power density.

An evaluation of the results of preliminary tests of a full-scale Lucite core model of the reflector-moderated reactor indicated the need for cut-offs in the pump volutes and turning vanes around one quadrant of the impeller periphery to obtain uniform flow distribution at the core inlet. Studies of flow in the core showed that a set of turbulator vanes was required in the core inlet passage to assure axially symmetric flow with no flow separation at the core shell wall. (AEC Activity 4401.1)

PROGRAM 4000 - REACTOR DEVELOPMENT (Continued)

Experimental Engineering - Two ARE pumps were tested at operating temperatures. Difficulties with bearings that appeared during cold tests were corrected. A plastic model of a horizontal-shaft, gas-sealed, sump-type, centrifugal pump for in-pile use was operated successfully with water. The three, gas-sealed, sump-type, vertical-shaft, centrifugal pumps for pumping fluorides in in-pile loops demonstrated satisfactory characteristics in pumping water. In addition, one pump operated satisfactorily for 523 hr at 1300 to 1400°F with NaZrF_5 salt, one operated for 65 hr at 1400°F with sodium, and the third is being tested with sodium at 1400°F. A plastic model of a centrifugally sealed pump was tested with water, and head-vs-flow curves and deaeration and sealing characteristics were determined.

A mockup of the ARE fuel flow system, which is being constructed for redetermining the filling characteristics of the system at atmospheric pressure rather than under vacuum, as tested previously, is about 75% complete. Apparatus was assembled for testing hydraulic motors to determine their suitability for operating in-pile pumps.

The addition of lead nitrate to a strong nitric-acid--boric acid solution was found to retard corrosion of Inconel markedly during the removal of UF_4 salts from Inconel parts.

Compatibility tests of bearing materials were run with chromium carbide and titanium carbide specimens sliding against each other in molten fluorides contained in nickel and Inconel systems. An appreciable metallic deposit was noted on the specimens from the nickel system, but little deposit formation was found on the specimens from the Inconel system. (AEC Activity 4401.1)

Mathematics and Computation - Calculations were made for 30 of a set of 48 reflector-moderated circulating-fuel reactor designs to determine the critical concentration of U^{235} in the fuel as a function of fuel annulus thickness, reflector thickness, and core radius. The following parameter values were used:

Fuel annulus thickness	5, 10, 15, and 20 cm
Reflector thickness	20, 30, and 40 cm
Core radius	20, 30, 40, and 60 cm

Analyses of the calculations indicated that to keep the U^{235} concentration below 4 mole % a combination of fuel annulus thickness of at least 10 cm, a reflector thickness of at least 30 cm, and a core radius of at least 30 cm will be necessary if the beryllium reflector is completely canned in Inconel. (AEC Activity 4401.2)

Reactor Chemistry - The solubility of UF_3 in a NaF-ZrF_4 mixture was measured experimentally at several temperatures. The UF_3 was produced in situ by the

PROGRAM 4000 - REACTOR DEVELOPMENT (Continued)

addition of metallic uranium to purified NaZrF_5 so that the ultimate composition of the mixture was 3.2 mole % UF_3 , 46.8 mole % ZrF_4 , and 50 mole % NaF . Chemical analyses of filtrates showed the solubility of UF_3 to be 4.1 wt % at 600°C, 5.8 wt % at 700°C, and 8.4 wt % at 800°C. All the UF_3 present was dissolved at 850°C and above. While analytical data are not yet available, there is some evidence that the solubility of UF_3 is increased by increasing the concentration of ZrF_4 . In a mixture containing 3 mole % UF_3 , 57 mole % ZrF_4 , and 40 mole % NaF , it appears that all the uranium may be soluble at temperatures below 550°C.

A plot of the logarithm of the concentration of HF vs. the logarithm of the concentration of FeF_2 for the reaction $\text{FeF}_2 + \text{H}_2 \longrightarrow \text{Fe}^0 + 2\text{HF}$ as carried out with 0.3 wt % FeF_2 in NaZrF_5 at 800°C, showed that the reduction was a "one-half order" process. This is presumed to be due to the removal of the HF under a near-equilibrium condition so that its concentration is controlled by the equilibrium constant

$$K = \frac{[\text{HF}]^2 [\text{Fe}^0]}{[\text{H}_2] [\text{FeF}_2]}$$

Since the hydrogen pressure was held constant at about one atmosphere, the concentration of HF was proportional to the square root of the concentration of FeF_2 .

A mixture of $\text{NaF-ZrF}_4\text{-UF}_4$, to which fission-product elements were added in concentrations roughly 1000-fold higher than those expected from the ARE, was shown to be considerably more corrosive to Inconel than is a pure fuel mixture. It has not yet been established which of the added materials was responsible for the roughly tenfold increase in corrosion.

A total of 1750 lb of purified fluoride mixtures (NaF-ZrF_4 and $\text{NaF-ZrF}_4\text{-UF}_4$) was produced in March; 900 lb of these materials will be shipped to the Pratt and Whitney Company. (AEC Activity 4401.5)

Corrosion Research - Oxidation tests at 800°C were made on Inconel joints brazed with various alloys, since the final application of brazing alloys will be in the construction of air radiators. The commercial alloys, such as Microbraz, low-melting-point Microbraz, and Coast Metals alloy No. 52, showed pitting attack to a depth of only 1 mil. Experimental alloys, such as the General Electric Company nickel-chromium-silicon alloy No. 81 and the "electroless" nickel brazing alloys (nickel-phosphorus and nickel-phosphorus-chromium), showed pitting attack to a depth of less than 1 mil. A gold-nickel alloy showed no attack.

In additional work on the evaluation of container materials for high-purity liquid lead, a quartz thermal-convection loop with inserts of type 347 stainless steel was used to circulate liquid lead at hot- and cold-leg temperatures of 810 and 525°C. The loop plugged after 253 hr of operation, which was 100 hr longer than was expected. The insulation of the cold leg was accidentally disturbed during operation, and the cold-leg temperature increased

PROGRAM 4000 - REACTOR DEVELOPMENT (Continued)

to 595°C for 15 minutes. It is thought that this temperature excursion caused a partial plug in the cold leg to break loose.

Cermets from the Kennametal and Sintercast Companies were tested in the fluoride mixture, $\text{NaF-ZrF}_4\text{-UF}_4$, in "seesaw" furnaces for 100 hours in the search for materials suitable for valve seats and pump seals. The average hot- and cold-leg temperatures were 800 and 675°C, and the samples and the fluoride mixture were contained in Inconel tubes. The tests indicated that Kennametal K-161-B, which is titanium carbide bonded with nickel, and Kennametal D4675, which is 94% tungsten carbide, with 6% cobalt binder, have good resistance to attack. One Sintercast sample of titanium carbide bonded with nickel also showed good resistance to corrosion.

Austenitic stainless steel thermal-convection loops, which had operated previously with lithium, failed after approximately 250 hr of operation because of plug formation. Improved techniques for handling lithium were developed, and two type-316 stainless steel loops are being operated with more nearly pure lithium at hot- and cold-leg temperatures of 816 and 600°C. One loop has operated for over 500 hr and the other for about 260 hr without plugging. (AEC Activity 4401.5)

Metallurgy - Mechanical Properties - Creep tests in vacuum, argon, and hydrogen of specimens from a heat of commercial or "A" nickel showed that at a stress of 1500 psi and a temperature of 1500°F there was little environmental effect and that rupture occurred in 950 to 1100 hours. However, when the specimens were tested under the same conditions in air, an asymptotically decreasing creep rate was found. In a test which was discontinued after 2000 hr, the total elongation was only 0.9%. A specimen that has been in test for 4000 hr at 2500 psi has not yet ruptured. The creep curve shows the usual asymptotic pattern, although at a slightly higher stress level. Thus, the creep strength of nickel specimens in air indicates a strengthening effect of the oxidizing atmosphere. (AEC Activity 4401.5)

Metallurgy - Welding - A commercially available drive mechanism designed to guide an oxygen cutting torch around intricate contours was converted to hold a Heliarc welding torch for tube-to-header welding studies. This machine, which incorporates a magnetic template, was found to be satisfactory for producing peripheral tube-to-header welds in tube sizes as small as 3/16 in. in diameter. The optimum welding conditions for joining 3/16-in.-dia. Inconel tubing to a 1/16-in. Inconel header plate were determined, and a heat exchanger header with 90 welds was fabricated. All 90 welds were consistent in quality and soundness. (AEC Activity 4401.5)

Reactor Control - The control system for the Tower Shielding Facility was completed, checked, and placed in good operating condition. The lacing and securing of the associated cables was nearly completed. The aerial cable terminals, gas flow system, reactor positioning drives, and position indicators (inside reactor tank) were also completed.

PROGRAM 4000 - REACTOR DEVELOPMENT (Continued)

The instruments and associated sensing devices were tested and found to have a few mechanical and electrical defects, which were corrected or are being corrected.

The communication system, consisting of public address and sound-powered telephone systems, is being modified to increase its flexibility and efficiency. The television system for viewing the reactor curing operation is being installed.

In a preoperating test, the servo system of the ARE was found to function properly. (AEC Activity 4401.6)

Shielding Research - The Tower Shielding Facility reactor was loaded to criticality in the narrow section of the pool on March 12. It was later installed in the reactor tank and loaded to criticality in the pool with a 5 by 6 element array. The reactor was operated a third time at an altitude of 200 ft, and then the initial experiment for determining the neutron-flux distribution in the water shield surrounding the reactor was started. The 5 by 6 element array will also be used for the differential shielding experiments and for reactor power determinations.

The Lid Tank Facility measurements of the slant penetration of fast neutrons through water were extended to slant thicknesses of 40 centimeters. A complete analysis of the data is expected to provide sufficient information for predicting the radiation dose from air-scattered neutrons incident at large angles to the normal on a crew shield.

In a Lid Tank Facility experiment for investigating a new method of inserting a boron layer in a shield, a ceramic coating of B_2O_3 (0.011 g of boron per cm^2) was applied to the source side of a 1-5/8-in. iron shield. The coating was sufficiently durable for the experiment, and it might be useful in some applications.

A pulse-height integrator for extending the range of the anthracene scintillation counters used at the Lid Tank Facility was examined. The pulse-height integrator replaced a considerably less-sensitive electronic circuit which was used to measure the integrated photocurrent. The difficulties encountered previously appear to have been electronic rather than systemic. (AEC Activity 4401.7)

Fuel Recovery - Further study of tributyl phosphate solvent extraction processing of ARE fuel solution indicates that the best decontamination can be obtained with 5 to 7.5% tributyl phosphate in Amsco 125-90W when the feed nitric acid concentration is 0.5 N. With 3 N nitric acid feed, the maximum decontamination occurs at a lower tributyl phosphate concentration. The decontamination factors obtained were of the order of 10^4 in runs on synthetic feed solutions containing no fluoride. With actual feed solutions, which contain 1 M fluoride, decontamination factors of the order of 10^3 were obtained.

PROGRAM 4000 - REACTOR DEVELOPMENT (Continued)

The feasibility of a fluorination method for recovering uranium from the fuel for an actual aircraft reactor was demonstrated. Scouting runs showed that more than 99% of the fission products and less than 0.05% of the uranium remained in the residue when the UF_4 in molten $NaF-ZrF_4-UF_4$ salt was volatilized as UF_6 following conversion to the hexafluoride with elemental fluorine. The UF_6 thus recovered would be reduced in the gas phase to UF_4 (method developed at K-25), and the molten salt fuel would be refabricated by the method currently used for preparing ARE fuel. The process would be low in cost and would produce a low volume of radioactive waste. Gross beta decontamination factors of the order of 200 were obtained in the volatilization step; resublimation of the deposited UF_6 gave an overall decontamination factor of around 5000. (AEC Activity 4401.81)

GENERAL REACTOR

School of Reactor Technology - The deadline date for receipt of applications for the 1954-55 session was March 15. Applications are now being processed by the faculty for submission to the Committee on Admissions early in April.

Interview appointments are being scheduled for placement of unsponsored students in the 1953-54 class. (AEC Activity 4841)

PROGRAM 5000 - PHYSICAL RESEARCH

REACTOR OPERATIONS

Operation of the graphite reactor (3800 kw) and the LITR (3000 kw) was satisfactory. The wall temperature of LITR hole HB-2 was measured with a thermocouple of special design and found to be below $100^{\circ}C$, indicating that there is little danger of water boiling around the tube. Prior measurements had indicated temperatures up to $350^{\circ}C$, but this was shown to be a result of gamma-ray heating in the thermocouple assembly.

Work on the installation of circulating fuel loops in holes HB-2 and HB-4 of the LITR is progressing satisfactorily. A charcoal trap for collecting radioactive gases from the loop equipment was installed in a hole in the floor 12-ft deep.

A 200-gram MTR-type fuel element was placed in the LITR in place of a 168-gram element. Observations will be made to determine which element is preferable.

PROGRAM 5000 - PHYSICAL RESEARCH (Continued)

ISOTOPE PRODUCTION

Radioisotope Production - Radioisotope shipments remained normal at 1030 for January and 1048 for February. Sales of Sr^{90} (43 curies) have shown a notable increase during the last two months; this is about two-thirds of the amount shipped during 1953. (AEC Activity 5111)

Radioisotope Research - Two pellets of fission product Cs^{137}Cl , one containing 760 curies and the other 780 curies, were made in a hydraulic press at 20,000 psi pressure. Both pellets were placed in a stainless steel capsule which was then sealed by silver soldering. This capsule was inserted into another stainless steel capsule which was closed with a screw fitting and also sealed by silver-soldering. The dimensions of the active material in the combined 1540-curie source are 3.2 cm in diameter by 2.6 cm long. The specific activity, based on gross weight of the pellets, is 25.3 curies/g. A density of 2.91 g/cc was achieved, giving 73.6 curies/cc. The radioactive pellets were quite luminescent and photographs were made through a two-foot-thick high-density glass window. The finished source was stored for observation and testing before shipment to ORINS Medical Division for use in a teletherapy unit.

Two methods for final purification of carrier-free I^{131} were tested: (1) deposition on metallic silver surfaces followed by elution in 0.01 M H_2S -water solution and (2) distillation through an eleven-plate bubble-cap column. The silver-deposition method was found to be efficient, but elution of the iodine became progressively more difficult after each cycle. The distillation method was effective in separating iodine from nitric acid, hydrogen peroxide, and water. The results were quite reproducible and this method, an improvement of the simple distillation method used for the past six years, was selected for use in the new I^{131} plant.

Additional tests were made on the alum crystallization method now used for separating cesium in order to determine the efficiency of the method for Cs^{137} decontamination of wastes having a wide range of chemical compositions. It was found that a decontamination factor of 1000 was consistently obtained. (AEC Activity 5131)

PHYSICS

Neutron Diffraction - A neutron diffraction study was made of the magnetic structures of a series of perovskite type compounds varying in composition from (100% La) MnO_3 to (100% Ca) MnO_3 . In the composition range from about (90% La, 10% Ca) MnO_3 to (50% La, 50% Ca) MnO_3 the compounds were found to be ferromagnetic with Curie temperatures ranging from near 20°C downward, in agreement with published values. Furthermore, the neutron studies indicate that, in this composition range, ferromagnetic and anti-ferromagnetic structures coexist, while, for compositions outside this range, there exists a

PROGRAM 5000 - PHYSICAL RESEARCH (Continued)

series of different anti-ferromagnetic structures. These various anti-ferromagnetic structures can be accounted for at least partially on the basis of magnetic cells which have twice the linear dimensions of the chemical cell (a) along one axis, (b) along two axes, and (c) along three axes. (AEC Activity 5211)

Low Temperature Studies - The specific heat of a polycrystalline sample of anhydrous MnCl_2 was measured in the temperature range from 1.3°K to 4.2°K by the techniques of adiabatic calorimetry. The sample was prepared by the dehydration of pure $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ in a dry HCl atmosphere. The results of six experiments indicate that in this temperature range the specific heat is anomalously large and passes through sharp maxima at 1.8°K and 2.0°K . This behavior is tentatively associated with a transition from the paramagnetic to the antiferromagnetic state. The existence of two maxima may be indicative of two separate phases in the crystal lattice.

An approximate partition function was obtained for certain paramagnetic salts of the iron group at low temperatures. For these salts the predominant interaction in the "spin" Hamiltonian of the magnetic ion is that due to the crystalline electric fields; this term is related to an anisotropy in the magnetic properties of the salts, as, for example, in the adiabatic dependence of temperature on magnetic field and entropy. With this approximate partition function, calculations were made of the entropy and $T = T(H)$ at constant entropy. (AEC Activity 5211)

High Voltage Program - The measurements of the differential cross section for elastic scattering of neutrons from nitrogen were continued. Neutrons scattered from LiN_3 at the nitrogen resonance of 1.40 Mev show increased scattering for forward angles when compared with non-resonance scattering. The 1.12-Mev resonance was investigated at a neutron energy resolution of 10 kw by the method of measuring the nitrogen recoil energies in a counter. This resonance also shows increased forward scattering of neutrons.

The cross section for the activation of the 0.8-sec metastable state in Pb^{207} by inelastic neutron scattering was remeasured in more detail. The absolute cross section was determined by calibration of the counter with a Bi^{207} source. The cross section rises approximately linearly from the threshold at $E_n = 1.6$ Mev to $E_n = 2.2$ Mev. It then levels off and rises abruptly at $E_n = 2.75$ Mev. The cross section is going up sharply at 3.3 Mev, the highest neutron energy for which it was measured. The experimental curve was compared with a theoretically derived curve in the region near threshold. The agreement is reasonably good. (AEC Activity 5211)

Theoretical Physics - Work on an ORACLE code for the computation of high energy scattering of electrons by nuclei was continued, and results from part of the code are now available. The UNIVAC tabulation of Racah coefficients was issued, as ORNL-1679, and the similar tabulation of Clebsch-Gordan coefficients is now in reproduction. The study of the relativistic corrections

PROGRAM 5000 - PHYSICAL RESEARCH (Continued)

to the Lamb shift is now complete, with the negative conclusion that no possible correction of α^6 order can compensate the omission of the positron effects, which were in question. (AEC Activity 5211)

Interaction of Radiation and Matter - In continuation of charge-exchange studies, measurements were made of the cross sections for electron loss by fast (20 to 250-kev) hydrogen atoms passing through various gases. These cross sections increase rapidly to a maximum at approximately 40 kev and then decrease slowly at higher energies. The maximum values are 7.2, 13.0, 23.5, 20.5, and $20.0 \times 10^{-17} \text{ cm}^2$ in hydrogen, helium, nitrogen, oxygen, and neon, respectively. (AEC Activity 5211)

CHEMISTRY

Chemical Separation of Isotopes - The system for the concentration of N^{15} by using ammonia (gas) versus ammonium carbonate (aqueous) was investigated further. This system, which has been demonstrated to give isotopic enrichment, appears quite attractive because of the closed reflux cycle possible and the attendant savings in the cost of reflux chemicals. The apparatus described previously (ORNL-1651) was used in two successful exchange runs. A saturated solution of commercial ammonium carbonate was fed from an infinite reservoir into the top of the exchange column. A sodium hydroxide solution was used as a refluxing agent at the bottom of the exchange column; when calcium hydroxide was used as the refluxing agent the calcium carbonate which formed tended to plug the packed stripping column after several hours of operation. Redesign of the stripping column should permit the use of calcium hydroxide for refluxing. The precipitate previously encountered in the exchange column was avoided by raising the temperature of the exchange column to 32.5°C . Analysis of the effluent from the stripper showed the ammonium ion to be as low as 0.002 M. The ratio of dissolved ammonia to dissolved ammonia plus ammonium ion in the exchange solution was 0.63. Isotopic assays of feed solution and samples taken from the bottom of the exchange column showed a total separation of 1.22 in a seven-hour run. Approximate extrapolation to equilibrium gave a total separation of 1.36.

Work on catalysis of the exchange between gaseous hydrogen and liquid water is underway. A gas-liquid contactor running at 1800 rpm was employed to equilibrate normal hydrogen with deuterium oxide at room temperature and 400 mm Hg pressure. Catalysts tested so far, with little or no exchange, are nickel dimethylglyoxime, palladium dimethylglyoxime, cobalt dimethylglyoxime-dimethyl sulfide, hydrolyzed gadolinium chloride, gadolinium chloride, palladium colloid (Baker Colloid 46), lanthanum chloride, tetrahydrofuran, dimethylcellosolve, triethyl phosphate and propylenediamine. (AEC Activity 5361)

PROGRAM 5000 - PHYSICAL RESEARCH (Continued)

Instrumentation for Nuclear Chemistry - In order to measure the thermal neutron capture cross section of Np^{239} it is necessary to count spontaneous fissions of Pu^{240} at a rate of 1 to 10 counts per hour over a Pu^{239} alpha background of 10^8 to 10^9 counts per minute. A fast amplifier and chamber were designed for this purpose. (AEC Activity 5361)

Hot Laboratory Research - In the terminal operation of the Homogeneous Reactor Experiment, three runs were specially scheduled at 400 kw to provide sources approaching 1000 curies of 9.2-h Xe^{135} for neutron transmission studies with the wide range velocity selector. Two of the three samples collected were successfully mounted in a sufficiently short period to approach the kilocurie limit. The initial run was not processed within this time limit due to close scheduling of equipment installation. On the basis of transmission values, the second and third run samples were estimated to have contained about 500 and 1000 curies, respectively. Final counting of the Xe^{133} activity remaining and a mass spectrographic assay of the Cs^{135} produced will accurately determine the Xe^{135} content of each sample. (AEC Activity 5361)

Chemistry of Corrosion - Further study of osmium tetroxide as a corrosion inhibitor was made at 23°C . It was found that an OsO_4 concentration as low as 4×10^{-3} f in distilled water is protective to carbon steel; the tests have continued for five weeks. The effect of the osmium compound is associated with formation of a visible film of reaction products, and the electrode potential is ennobled. The passivated carbon steel will not displace copper from a cupric sulfate solution. This behavior may be contrasted with the action of the pertechnetate ion, which inhibits corrosion but does not ennoble the steel or prevent precipitation of copper.

It was found that a specimen of carbon steel, the corrosion of which was inhibited in a suitable concentration of potassium pertechnetate, was no longer inhibited when the solution was diluted below the "inhibiting" limit. This shows that the protective action is not due to a stable film formed at the higher concentration, but to a dynamic state that requires a certain minimum concentration.

Further experiments on the breakdown of oxide films on stainless steel, type 347, in dilute sulfuric acid confirmed the characteristic potentials described in the previous report (ORNL-1676). It was shown that the electropolished specimens, in particular, may pass from the noble state, having a golden film that is unstable under the experimental conditions used, to the less noble stable state with no visible film, without passing through an intermediate state characterized by an active electrode potential. (AEC Activity 5361)

Ion Exchange Technology - In studies on the cobalt cyanide poisoning of ion exchange resins (ORNL-1651) about 97% of the uranium capacity of the resin was restored by treatment with 2 M ammonium thiocyanate followed by sodium hypochlorite (5% free chlorine). The cobalt cyanide is present in South African ore leach liquor.

PROGRAM 5000 - PHYSICAL RESEARCH (Continued)

In preliminary studies more than 95% of the uranium was sorbed from a sulfuric acid leach solution of ground carnotite ore on IRA-400 resin in a Higgins continuous contactor. Only a small amount of the solid material suspended in the leach solution was filtered out by the resin, and this was easily removed from the resin by washing. (AEC Activity 5361)

THE ORACLE

During the last two months, the ORACLE was used primarily for "debugging" or "checking out" problems, with 152 hours being devoted to computations. Part of each morning was used for routine engineering and maintenance, while the remainder of the regular day shift was used for computations. In addition, three evenings per week were regularly used for computing.

A number of interesting and useful problems and subroutines have been run on the ORACLE and are ready for further production. Among these are the following codes:

1. A code for calculating any hypergeometric function; these functions include most of the customary functions of mathematical physics.
2. An inversion routine for matrices, which can be used for solving systems of linear algebraic equation.
3. A code for calculating the eigenvalues and eigenfunctions associated with the normal form of a linear second order ordinary differential equation.
4. Certain subroutines dealing with floating-point operations.
5. A routine which calculates determinates. (It gives insufficient accuracy at present.)
6. A least squares estimate of the parameters in the Fourier analysis of crystal diffractions.

The photoelectric tape reader was installed and found to operate satisfactorily. Engineering design was continued on the installation of the magnetic tape memory unit. Preliminary investigations are under way to determine the feasibility of using magnetic tape as an output device for the ORACLE. (AEC Activity 5511)

PROGRAM 6000 - BIOLOGY AND MEDICINE

BIOLOGY

Cytogenetics - In previous experiments it was only when tradescantia inflorescences were simultaneously centrifuged and X-irradiated that an increase in the number of chromosome aberrations, over the number induced by irradiation alone, was obtained. In more recent experiments, however, the aberration yield was enhanced if the centrifuge was started immediately after irradiation. It was also noticed that centrifuging for long periods of time, either previous to or during irradiation, decreased the aberration yield, apparently because the temperature rise due to friction of the centrifuge is enough to hasten the rejoining of broken chromosome ends.

The fact that post-irradiation centrifugation can increase the chromosome aberration yield indicates that radiation-induced breaks are not latent. This suggests a new tool for determining whether or not the same number of breaks are induced by irradiation in air as in nitrogen.

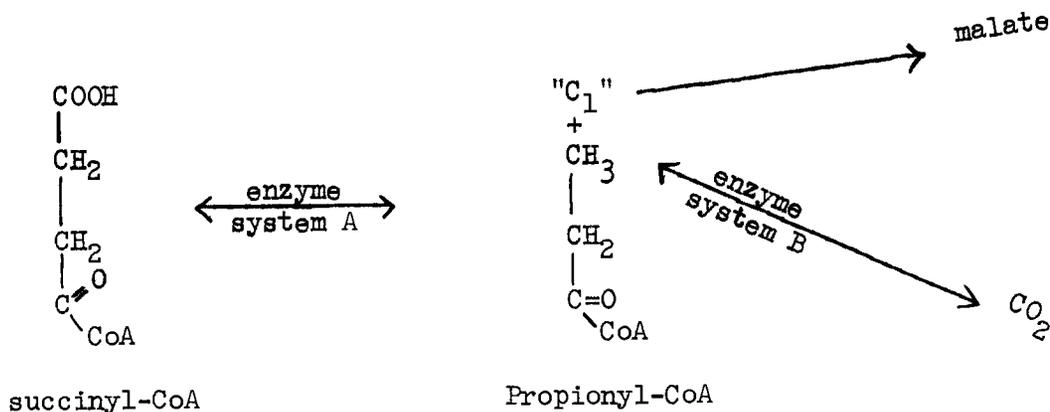
This work is being extended to Vicia faba wherein there is an extensive post-irradiation period during which centrifugation may increase the aberration yield.

The newly laid Habrobracon egg has the nucleus located at the convex surface. Previous work has demonstrated that egg survival follows an exponential curve when the convex side is irradiated with ultraviolet light, and a sigmoid curve when the concave surface is treated. These data are consistent with the hypothesis that death from ultraviolet exposure of the convex side is from nuclear inactivation while death from treatment of the concave side is predominantly cytoplasmic in nature. If visible light of strong intensity is applied after ultraviolet irradiation of the convex side of the egg, egg survival is significantly higher than when ultraviolet light alone is used. On the other hand, visible light does not affect survival when the concave side is irradiated. This suggests that photoreactivation of damage induced by ultraviolet light is a phenomenon associated predominantly with the egg nucleus.

It was recently demonstrated that treatment of Paramecium aurelia with low concentrations of H_2O_2 after irradiation decreases the amount of detectable mutation. This finding is contrary to all previous findings which had indicated that the peroxide can act as a mutagen. It also shows that peroxide, acting after irradiation, does not enhance the effect of the radiation, as had been reported in England for bacteriophage. In recent experiments, a decrease in detectable mutation was found when the paramecia were treated either before or after irradiation. The effect decreased with time after irradiation but was still detectable a day later, following four cell divisions. A large effect was found even when none of the paramecia were killed and there was only a slight prolonging of the interdivision interval by the radiation. Thus it seems unlikely that a simple selection of non-mutant animals is involved. No satisfactory hypothesis can yet be offered. (AEC Activity 6400)

PROGRAM 6000 - BIOLOGY AND MEDICINE (Continued)

Microbiology - Previous reports described the following findings in connection with succinate decarboxylation by enzyme extracts of Propionibacteria and Veillonella: (1) coenzyme-A dependence; (2) presence of potent coenzyme-A transphorases; (3) succinyl- and propionyl-deacylases; (4) formation of an active carbon fragment ("C₁") produced during succinate decarboxylation, and which acts as a precursor to malate carboxyl carbon as well as to CO₂. The following reaction sequence depicts reaction steps, compounds, and enzyme systems involved in current studies.



Enzyme system A from Propionibacterium enzyme extracts was partially purified by protamine treatment followed by (NH₄)₂SO₄ fractionation; the main activity of enzyme A was found in the 50-70 fraction. Enzyme system B was obtained from Veillonella enzyme extracts by centrifugation at 70,000 g; 35% of the activity was found in the dark (particle) layer of the residue, and the remainder is the supernatant. Enzyme B is most stable on the particle. (AEC Activity 6400)

Radiation Protection - In studies carried out with Escherichia coli strain P 82/r (purine⁻) and strain T 83-8 (tyrosine⁻) it was found that cell suspensions X-irradiated under anoxic conditions (N₂ saturated) show the same response (induced mutation rate) to postirradiation temperature treatment as they do when exposed under an atmosphere of oxygen. In both cases an increasing mutation rate was found with increasing incubation temperatures during the first 48 hours. It was also observed, by exposure of E. coli strain T 83-8 in either O₂ or N₂ to several doses of X rays, that for equivalent survival values the mutation rate is the same, i.e., independent of the presence or absence of O₂ during exposure. This is in agreement with similar data obtained on strain P 82/r.

It was found that, in Homo sapiens, the bacterial recovery factor is present in the urine of both males and the females, including pregnant females. A 95% ethanol extraction of fresh urine yields a product purified approximately 10-fold. Paper partition chromatography is being used in attempts to purify this product. Experiments, designed to duplicate the recovery effect on a complex synthetic medium, are in progress. (AEC Activity 6400)

PROGRAM 6000 - BIOLOGY AND MEDICINE (Continued)

Biochemistry - In the course of studies concerning the action of bacterial deoxyribonuclease, isolated from bacteria lysed by bacteriophage, on bacteriophage deoxyribonucleic acid, there were indications that there exists in bacteriophage some protein other than the protein forming the membrane and that perhaps this non-membrane protein did not remain outside the bacterial cell during phage infection, as does the membrane protein, but entered along with the deoxyribonucleic acid. Since the existence and behavior of such a protein is at variance with present concepts of bacteriophage structure and function, more definite proof concerning it was sought. Osmotic lysis of purified bacteriophage T₄r⁺ and quantitative removal by centrifugation of residual phage and of the "ghosts" (membranes) leaves a protein as well as the nucleic acid in the supernatant solution. This protein amounts to 25% of the total phage protein, has a different amino acid composition than the membrane (ghost, insoluble) protein, and differs in its sensitivity to proteolytic enzymes. By the use of phage grown on glycine-C¹⁴, which labels both the nucleic acid and the protein of the new phage, it can be shown that the ratio of labelled nucleic acid to labelled protein in ghost-free phage lysates is also found in the infected bacteria, showing equal degrees of penetration by both components. Because of the conflict between these observations and current concepts, based largely on Hershey's work with P³² (which labels DNA only) and S³⁵ (which labels membrane protein only), these observations are being repeated under a wide variety of conditions to confirm their validity. (AEC Activity 6400)

Plant Biochemistry - It was shown previously that carbon monoxide treatment of barley leaves inhibits subsequent C¹⁴O₂ fixation. This inhibition was prevented by illuminating the leaves during CO exposure, which implicated cytochrome oxidase, the terminal enzyme of the respiratory chain that is complexed by CO. The photodissociation spectrum of this complex has a peak at 590 mμ. It has now been shown that the photosynthetic inhibition induced in barley leaves by CO is removed 50 times more effectively by yellow light (589 mμ) as by red light (640 mμ). This is strong evidence that cytochrome oxidase is involved, probably in formation of high energy phosphate bonds, in the chain of events leading to photosynthetic CO₂ fixation. (AEC Activity 6400)

Biophysics - Irradiation of Tradescantia pollen with 250-kv x rays at constant dose over an intensity range from 3 to 300 roentgens per minute showed no significant variation in observed aberration frequencies. These experiments were designed to determine the effective life time for chromosome breaks in dry pollen. The results indicate that breaks remain open in this material for at least several hours. Split-dose experiments have confirmed these results and indicate that there is no difference between a dose given in a few minutes and that given in two fractions with a 3-hour rest period between. Further work with longer rest periods is in progress.

Experiments using a continuously indicating ion-chamber dosimeter with polyethylene walls and filled with ethylene gas indicate that this instrument

PROGRAM 6000 - BIOLOGY AND MEDICINE (Continued)

can be used as a dosimeter for measuring dosage of fast neutrons. Studies of the effect of scattered neutrons on the Hansen-McKibber long counter indicate that acceptable scatter corrections can be made, yielding acceptable neutron flux calibrations. (AEC Activity 6400)

Pathology and Physiology - Mice were injected intravenously with 40-160 μc of colloidal gold-198; over 80% of which was taken up by the liver soon after injection. From 84 to 90% of the mice developed cirrhosis, accompanied by tumors of the liver (hepatomas) in 20 to 38% of these cases. The incidence of leukemia was not affected.

Administration of desiccated thyroid and of thyroxine, beginning on the day of irradiation and continuing daily thereafter for 38 days, failed to alter the blood counts or survival of adult rats exposed to an LD_{50} dose of x rays.

Adult mice and rats surviving a nearly lethal dose of fast neutrons, produced in the 86-inch cyclotron, developed complete cataracts within 6 and 9 months, respectively; however, guinea pigs given the same dose developed only relatively mild opacities of the lens within the first year. These observations indicate the relatively high radiosensitivity of the lens of the mouse and the rat and suggest that severe cataract in the guinea pig results only from a dose of neutrons which exceeds the lethal dose for whole-body radiation.

Histone was isolated from chicken erythrocyte nuclei by several different procedures. Electrophoretic studies, in acetate buffers (pH 5.2 - 5.8), showed that five peaks were present. Similar patterns were seen under a wide variety of conditions suggesting that at least five components are present in the undegraded substance.

Histological evidence was produced to show that immersion of embryos of Aneides anaeus in a solution containing 50 $\mu\text{c}/\text{ml}$ of I^{131} for 24 hours at the developmental stage marking the beginning of thyroid function completely destroyed the thyroid, whereas immersion in solutions of concentrations up to 20 $\mu\text{c}/\text{ml}$ had no observable effect on the gland. Such thyroid destruction did not inhibit development.

A series of studies was undertaken to measure the exchangeability of radioactive potassium in frog embryos. A preliminary experiment establishing control levels was performed. The present work is concerned with a comparison of the exchangeability of potassium at various stages of development. Present findings indicate that the movements of lithium into and out of frog embryos is closely related to reciprocal sodium movements but not to potassium movements. (AEC Activity 6400)

Enzyme Chemistry - During earlier studies of firefly-lantern extract luminescence (Strehler and Totter, Arch. Biochem. and Biophysics 32, 397, 1951), it was noted that chloride ion reduced the response to adenosine triphosphate (ATP) and therefore interfered with the application of the luminescence method

PROGRAM 6000 - BIOLOGY AND MEDICINE (Continued)

to the assay of ATP. The influence of other anions on the luminescence and fluorescence of firefly lantern extracts and purified luciferin was determined. The results show that the suppression of luminescence and of fluorescence is related to the ionic deformabilities of the anions tested. This result suggests that the effect is analogous to the well known quenching of fluorescent dyes by anions. A comparison of the concentration of iodide ion (0.011 M) required to reduce luminescence by 50% with the concentration (0.10 M) required to reduce luciferin fluorescence a similar amount excludes the possibility that the emitting molecules are identical in the two cases.

The action spectra for the delayed light emission of blue-green and red algae is being investigated. The blue-green algae used, Anacystis nidulans and a strain of Lyngbya which is not yet free of bacterial contamination, were grown in an enrichment medium, Chu No. 10. The red alga, Porphyridium cruentum, was grown in R. H. Swain's medium for Porphyridium. All forms were grown in the presence of light from sodium and neon lamps. Filters were used with the neon lamp to reduce the light intensity at the wavelength at which chlorophyll absorbs light. Blue-green algae contain the pigments phycocyanin and chlorophyll, and the red algae contain phycoerythrin in addition to phycocyanin and chlorophyll. Action spectra obtained for the delayed light emission in the blue-greens show a peak only in the region where phycocyanin is capable of absorbing light. The action spectra for the red algae show a large peak in the region where phycoerythrin absorbs light and small peaks in the phycocyanin and chlorophyll regions. However, lighting conditions have not yet been obtained where the wavelengths absorbed by chlorophyll are completely eliminated in the red algae. (AEC Activity 6400)

[REDACTED]

RADIOISOTOPE SALES AND COSTS

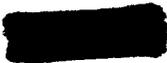
<u>Type of Transaction</u>	<u>February</u>	<u>FY to Date</u>
Domestic Sales	\$ 67,527	\$588,460
Foreign Sales	6,625	39,765
Project-Cash Sales	14,632	82,322
Project-Transfer Credits	---	3,995
Technical Cooperation Program Credits	946	6,778
Plant Credits	1,416	78,942
Civilian Defense Credits	---	390
Cancer Program Credits	19,916	147,482
*Miscellaneous Income	<u>82</u>	<u>334</u>
 Total Income	 \$111,144	 \$948,468
 **Income - No production costs incurred, subtract from Total Income	 <u>3,152</u>	 <u>21,677</u>
 Net Radioisotope Income	 <u>\$107,992</u>	 <u>\$926,791</u>
 Radioisotope Costs	 <u>\$ 93,458</u>	 <u>\$741,152</u>
 Radioisotope Shipments	 <u>1,045</u>	 <u>8,076</u>

*Miscellaneous income - D₂O, freight, missing parts, TCP, etc.
 **Income from H³, He³, B¹⁰, and miscellaneous items in excess of handling cost.

GROSS OPERATING COSTS

	<u>Cost for February</u>	<u>FY 1954 Cost to Date</u>
Programmatic Operating Cost -- Net	\$ 2,261,108	\$ 18,932,863
Plant and Equipment Cost	356,493	2,574,274
Construction Program "H"	747	24,955
Work for Other Parties - Transfers	44,369	347,011
Inventory Changes	19,503*	209,480*
Reimbursable Work for Other Parties	142,657	1,048,999
Deferred Charges	<u>5,105</u>	<u>5,714</u>
 Total Laboratory Cost - Net	 <u>\$ 2,790,976</u>	 <u>\$ 22,724,336</u>
 Estimated Cost for Next Month - Net	 <u>\$ 2,850,000</u>	 <u>\$ 25,574,336</u>

*Credit



PERSONNEL SUMMARY

	<u>Number of Employees</u> <u>March, 1954</u>	<u>New Hires</u> <u>March</u>	<u>Terminations</u> <u>March</u>
Administration	63	0	1
Operations*	115	0	0
Engineering, Shops and Mechanical	782	26	2
Laboratory and Research	1691	30	15
Protection	149	0	0
Service	<u>375</u>	<u>5</u>	<u>2</u>
Total	3175	61	20

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

A total of 630 Laboratory personnel are located in the Y-12 Area.

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Previous reports in this series:

March 1953	ORNL-1529
April 1953	ORNL-1545
May 1953	ORNL-1562
June 1953	ORNL-1576
July 1953	ORNL-1600
August 1953	ORNL-1612
September 1953	ORNL-1622
October 1953	ORNL-1643
November 1953	ORNL-1651
December 1953	ORNL-1665
January 1954	ORNL-1676
February 1954	ORNL-1695

