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OAK RIDGE NATIONAL LABORATORY

PROGRESS AND STATUS REPORT

MARCH 1957

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STATUS AND PROGRESS REPORT

March, 1957

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

March, 1957

This status and progress report summarizes somewhat less than one-half of the Laboratory's activities. A few of the activities are reported every month but most of them are reported on a bimonthly schedule. Program 4400 is reported separately.

PROGRAM 2000 - SPECIAL NUCLEAR MATERIALS

Waste Metal Recovery - A total of 28.5 tons of uranium has been recovered, to date, from ORNL tank waste with the use of the Metal Recovery Plant. The plant is now shut down for cleaning and modification prior to beginning recovery of uranium from Chalk River irradiated material. (AEC Activity 2350)

Excer Process - In experiments on the reduction of UO_2Cl_2 by metallic iron, the purity of the $UF_4 \cdot 3/4H_2O$, precipitated by HF, acting on the UCl_4 reduction product, was equal to that of the electrolytic reduction product when the product was thoroughly washed. The water used to wash the UF_4 precipitate, however, contained 0.1-1.0 g of uranium per liter in a soluble or colloidal form. The tap density of the dried product was increased, in a typical case, from 1.5 to 3.8 g/cc by tabletting under a pressure of 40 tsi.

In further electrolytic-reduction tests, an Ionics membrane cell was operated continuously for 173 hr and again for 336 hr. In the first run the cathode was lead-silver, and the anode was platinum-coated tantalum. The catholyte was 3 N HCl containing 200 g of uranium per liter, and the anolyte was 1 N H_2SO_4. The steady-state chloride concentration of the anolyte was 0.01 N, and the final uranium concentration was 0.04 g/liter. The average reduction efficiency was 88% for 100% reduction. The cathode was severely pitted, and a deposit (86% lead, 8.8% silver) plugged the cathode compartment. In the second run the cathode was of Hastelloy C, and there was no visible damage to the cell components.

Tests are being made in the 2-in.-dia contactor on the recovery of uranium from low-grade ores by ion exchange in a form suitable for feeding to the Excer reduction step. The contactor operated at a rate of 125 g of uranium per hour on feed prepared by dissolving the ore concentrate in HCl and saturating the solution with NaCl to get a favorable distribution of uranium to the resin. (AEC Activity 2702)

Fluorox Process - Approximate rate constants obtained for the reaction $2UF_4 + O_2 \longrightarrow UF_6 + UO_2F_2$ in the temperature range 600-800°C with the use of high-specific-surface-area UF_4 were of the order expected from experiments with

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the use of low-surface-area UF_4. A postulated side reaction, $UF_4 + UF_6 \longrightarrow 2UF_5$, was found to consume ~ 30% of the UF_4 at 800°C and 10% at 700°C. (AEC Activity 2702)

Metallex Process - Phase studies of the binary system thorium-mercury show two intermediate intermetallic phases, $ThHg_3$ and ThHg. The solubility of thorium in mercury ranges from about 100 ppm at room temperature to about 10 wt % at 300°C. Free sodium in mercury prevented the separation of thorium mercuride from the reaction by-products in dry reduction of $ThCl_4$ with sodium amalgam.

In shakedown runs of the large-scale continuous contactor (expected design capacity 1-2 lb/hr), temperature control and operation of the salt and amalgam feed systems were satisfactory. There was some difficulty in discharging the product. (AEC Activity 2704)

Power Reactor Fuel Reprocessing: Darex Process - The continuous dissolution of stainless steel-containing fuel elements in aqua regia and the removal of the chloride from the solution were successfully demonstrated in all-glass equipment. In five chloride removal runs in a 4-in.-dia 12-plate pyrex bubble-cap column, the average plate efficiency was 35%. At a constant vapor to liquid flow ratio of 2:1, an increase in the acid concentration of the stripping vapors from 32 to 38 mole % HNO_3 increased the chloride removal but decreased the recoverable chloride.

The continuous dissolution rate of stainless steel increased from 15 mg per min·cm^2 in 6M HNO_3-2M HCl to 130 mg/min·cm^2 in 6M HNO_3-4M HCl. The product streams were essentially constant in chloride and nitrate content. Product acidity increased somewhat with increasing feed rate.

First- and second-cycle solvent-extraction flowsheets for the dissolver solution, following chloride removal, were designed and tested. Uranium recovery in the first cycle was > 99.99% and gross β and γ decontamination factors were 4.8×10^5 and 6.1×10^4, respectively. The feed to the second cycle was not very radioactive, and the product activity was essentially background.

Tantalum showed negligible corrosion rates in all Darex dissolver and stripping solutions and atmospheres. Potentiometric studies indicated that hydrogen gas was evolved when tantalum was in contact with uranium dissolving in boiling 5M HNO_3-2M HCl. (AEC Activity 2924)

Power Reactor Fuel Reprocessing: Zircex Process - In hydrochlorination of a 12% U-88% Zr alloy to form volatile $ZrCl_4$, the preliminary results indicated that the uranium loss to the $ZrCl_4$ sublimate depended on the temperature. Losses were 2.2% at 694°C and 1.4% at 530°C.

In a single test of the hydrochlorination of Zircaloy-2-clad UO_2 pellets (PWR blanket rod), 0.013% of the uranium was lost in the acid-insoluble residue and 0.002% by entrainment or volatilization. The presence of water during

hydrochlorination at 340°C produced a surface that inhibited reaction. The presence of aluminum metal at the start of hydrochlorination of oxide-coated zirconium promoted the reaction.

Tantalum corrosion was negligible in all dissolver solutions and atmospheres. (AEC Activity 2924)

Power Reactor Fuel Reprocessing: Aqueous Dissolution - The Zircaloy-2 cladding was completely stripped from the UO_2 pellet core of an obsolete PWR blanket element by HF at 5 M or higher concentrations at a rate of 168 mg per min·cm^2 or higher. The uranium losses were $< 0.05\%$.

Dissolution rates for a 90% U-10% Mo alloy in nitric acid increased from 1.4 to 106.8 mg/min·cm^2 as the acid concentration was increased from 0.5 to 15.8 M. For a 90% U-10% Nb alloy, the rate increased from a negligible rate to 0.91 mg/min·cm^2 as the acid concentration was increased from 5 to 15.8 M. The dissolution rate of type 304L stainless steel, in 5 M HNO_3 containing HF, increased linearly as the HF concentration was increased from 1 to 5 M.

Stainless steel dissolved anodically in 2-3 M HNO_3 at a rate of 3 mg per min·cm^2, with current efficiencies of 90-95%. Zirconium dissolved anodically in 2-3 M HCl-0.15 M HF at rates as high as 50 mg/min·cm^2, and no metallic residue remained. With alternating current between two stainless steel electrodes in 3 M HNO_3, one electrode dissolved at the expected rate, but the other became passivated. (AEC Activity 2924)

Power Reactor Fuel Reprocessing: Hermex Process - In the Hermex process uranium is dissolved in boiling mercury and recovered from the solution by pressing and sintering. Uranium was not readily dissolved from uranium-zirconium fuel elements following degradation of the zirconium by hydriding or by molten magnesium. (AEC Activity 2924)

PROGRAM 3000 - WEAPONS

RaLa - The Laboratory was released officially from its standby responsibility for RaLa production. (AEC Activity 3641)

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test - Following the termination of water run No. 6 on February 25, after 164 hr at 275°C and 1700 psi, the HRT system was readied for test operation with depleted uranium solution. Both fuel feed pump heads and the fuel letdown valve were replaced because of corroded Stellite sealing surfaces.

Run No. 7 was started with water under the same conditions as run No. 6, but the run was briefly interrupted after three days for replacement of the fuel pressurizer purge pump, which had ruptured a diaphragm. The reactor was then operated continuously from March 3 until March 22 at 275°C and 1700 psi. Depleted uranyl sulfate, sulfuric acid, and copper sulfate were added on March 4 to yield a fuel concentration of approximately 5 g of uranium per kilogram of water with 50% excess acid. On March 11 the fuel concentration was increased to a nominal 10 g of uranium per kilogram of water and remained at that level for the rest of the run. The run was satisfactory, in general, but two difficulties were experienced. A bellows in the core-blanket equalizing valve developed a very small leak, which resulted in the loss of water vapor from the system. A diaphragm ruptured in the purge pump for the blanket-circulating pump and must be replaced at some future time.

The chemical processing plant was operated successfully with the reactor on several occasions during the 3rd week of March. No notable difficulties were encountered during joint operation. (AEC Activity 4103.3)

HRT Chemical Pilot Plant - Construction of the low-pressure system for the chemical processing plant was essentially completed. In tests of remote maintenance procedures, the circulating pump was lowered into place with the overhead crane. The bolt-hole aligning tool satisfactorily aligned the four vertically mounted flanges, and bolts were inserted by a magnet attached to an adaptor on the vertical-torque wrench. (AEC Activity 4103.1)

Mathematics and Computation--Homogeneous Reactor Development - An Oracle code was written for calculating the fuel costs in a one-region, thorium breeder reactor operating under steady-state conditions. Atomic species considered were U^{233}, U^{234}, U^{235}, U^{236}, Th^{232}, Pa^{231}, and fission-product poisons. An Oracle code was written for obtaining the critical mass and flux distributions in an n-region reactor, with the use of a two-group, one-dimensional model. (AEC Activity 4103.1)

Reactor Analysis - Results were obtained for steady-state fuel costs in one-region and two-region thorium breeder reactors (thorium in both core and blanket). For two-region reactors, the lowest fuel costs were associated with a UO_2SO_4 solution-type core region, a small (4-ft) core diameter, and a core-wall power density of 170 kw/liter. For relatively low wall power densities (20-40 kw/liter), the lowest fuel costs were associated with 6-7-ft-dia cores fueled with a mixed uranium oxide-thorium oxide slurry containing 100-200 g of thorium per liter. The difference in fuel cost between the 4-ft-dia solution core and a 6-ft-dia slurry core containing 150 g of thorium per

liter was 0.06 mill/kw-hr. For one-region reactors, the fuel costs based on steady-state results were about 0.6 mill/kw-hr higher than for two-region systems. Results based on unsteady-state calculations indicate that the difference is appreciably less than 0.6 mill/kw-hr. (AEC Activity 4103.1)

Homogeneous Reactor Instrumentation - A design report, covering wiring revisions and additions to the HRT control circuits, was issued that describes the addition of a time delay to allow for gas recombination before dumping, the rearrangement of control wiring to provide parallel connections in critical circuitry, the revision of the circuit to provide for the addition of oxygen to the high-pressure system, the addition of circuits to provide controls for the new demineralized cooling water loop, and the addition of waste disposal system alarms to the main control room annunciator system.

The HRT oxygen-metering valves were redesigned to utilize replaceable trim (needle and seat) which is available commercially in a wide variety of sizes and materials. A tungsten carbide set ground to a clearance of 0.0005 in. will be tested initially. The flow coefficient is adjusted by varying the volume of a small spline ground in the needle.

A valve was designed similar to the HRT letdown type which incorporates a plug that is removable from the bellows-stem assembly to permit the economical evaluation of a number of alternate materials.

Two stainless steel valve plugs capped with an 80% Pt-20% Ir alloy hardened to an R_c of 25, supplied by Baker and Company, were tested against type 347 stainless steel seats in the valve test loop. After 400 "dumps", the first set was found to be bubble tight with 2000 psi of nitrogen gas applied under the seat. After 400-dump testing cycles, the second trim set leaked 15 cc of water per minute with 2000 psi applied. However, the type 347 stainless steel seat had been nicked at the seating edge by a foreign particle, and therefore this leakage test was not indicative of the wear on the platinum-irridium alloy plug. Other platinum alloys will be simiarly tested.

Preliminary drawings of the front panel layout for the 22-ft control board, the control circuit schematic, the instrument flow sheet, the relay boxes, and a control area layout were completed for the ORR in-pile loop. (AEC Activity 4103.1)

HRP Design - The HRT refrigeration compressor was found to have been overrated by the vendor by nearly 50%. The vendor will make the necessary modifications to the unit to bring it to its rated capacity of 43,000 Btu/hr at the operating temperature of -40°F. Further modification of the refrigeration system, including the replacement of the chiller tank with a heat exchanger of greater capacity, will be required to compensate for the change from Freon F-11 to Amsco in the secondary system.

Design of the chemical feed station for the HRT steam system feedwater treatment system was completed, and all major purchased equipment was received.

Major emphasis was placed on the design of tools and facilities for the remote maintenance of the HRT. The design of manual handling facilities for the wall periscope was re-activated following a review of the problem and a decision against the use of remotely operated facilities.

Specifications were prepared and a purchase request was issued for replacement of the HRT bolts with newly designed bolts more satisfactory for use at high temperatures. Several preliminary conclusions were drawn as the result of the first series of runs of the flange test facility, which were that resiliency and uniformity of flange bolting are definite factors in flange leak tightness; that flange bolts should be retightened to assure minimum leakage of the flange following thermal cycling of a system; and that nearly imperceptible damage to the seating surfaces of either the flange or the ring joint gasket is sufficient to destroy the integrity of a flanged joint.

Preliminary design of HRE-3 was started. The proposed reactor is to operate on a thorium-uranium cycle at a thermal energy level of approximately 60 Mw. Design objectives include combination of breeding with a simplification of the system as compared with HRE-1 or the HRT. (AEC Activity 4103.1)

HRP Metallurgy - Mechanical property testing has shown that plates of both high purity titanium and zirconium are resistant to crack propagation even at low temperatures. Drop-weight testing (a mechanical test which measures the minimum temperature at which a moving crack will be arrested by the material) has shown the transition temperature for the Zircaloy-2 core tank material to be -250°F and for A-40 titanium to be -320°F. With both materials, cracks formed in the area around a weld even at room temperature but did not progress through the base metal. It has not been determined whether this cracking was caused by a brittle heat-affected structure or by contamination during the welding.

In a Charpy V-notch type impact test, the specimen and notch orientation are much more critical with the hexagonal zirconium alloys than they are with the more common cubic metals. With the notch oriented through the plate a room-temperature impact value of 34 ft-lb is obtained but, with the notch in the plane of the plate and parallel to the rolling direction, the value drops to 12 ft-lb. At 300°F the two values are 103 and 20 ft-lb, respectively.

Welding procedures were developed and qualified that will permit replacement of the leak detector tubing and flanges in the reactor pit. The small tubing will be butt-welded by using preplaced flat inserts and will be joined to the flanges through a transition piece. (AEC Activity 4103.1)

HRP Spectroscopy Development - An x-ray diffraction method of determining the relative abundance of UO_2 in solid solution with ThO_2 and a variable concentration of crystalline U_3O_8 was developed in conjunction with HRP analytical chemistry requirements. Under optimum conditions a shift in the lattice constant of 0.0002 Å can be detected; this corresponds to slightly better than 0.2% analytical accuracy. (AEC Activity 4103.1)

Laboratory Corrosion Studies - Stress-corrosion cracking in U-bends of type 347 stainless steel was produced consistently at 300°C in oxygenated distilled water containing 100 ppm chloride ion, added as HCl and NaCl, at a pH of 2.8. Cracks were observed on 26 out of 26 U-bends exposed for 100 to 350 hr in the solution. There was no apparent effect of dissolved oxygen content in the range of 25 to 1250 ppm on the incidence of cracks. Preliminary test results with pertechnetate ion in the chloride-containing distilled water were encouraging with respect to preventing stress-corrosion cracking. However, preliminary results indicate that the pertechnetate ion is not completely stable under the conditions of the test.

Elgiloy (a spring material considered for use in the liquid-level controller for the HRT pressurizer) has shown a susceptibility to stress-corrosion cracking in the cold-rolled condition in both the liquid and vapor phases of oxygenated 0.04 m UO_2SO_4 - 0.02 m H_2SO_4 - 0.005 m $CuSO_4$ solutions. Attempts to eliminate the crack susceptibility by various surface treatments, such as pickling and electropolishing, or by stress-relieving at 482°C (900°F) have been unsuccessful. Annealing in air at 1149°C (2100°F), followed by pickling, was effective, however, in preventing cracking in uranyl sulfate solutions at high temperature. (AEC Activity 4103.1)

Dynamic Solution Loop Corrosion Tests - It was previously reported that the addition of 0.04 m $BeSO_4$ to 0.04 m UO_2SO_4, containing 0.005 m $CuSO_4$, produced a solution that was phase-stable at 300°C for a 200-hr period; furthermore, the solution appeared to be less corrosive to stainless steel at temperatures between 200° and 250°C than did the solution proposed for use in the HRT (0.04 m UO_2SO_4, 0.02 m H_2SO_4, and 0.005 m $CuSO_4$). Recent experiments in which the beryllium sulfate concentration has been either 0.03 m or 0.08 m and the uranyl sulfate concentration 0.04 m have shown that such solutions are not stable at 300°C. In both cases, 15 to 20% of the beryllium precipitated from solution, probably as beryllium oxide, in less than 200 hr. With 0.03 m $BeSO_4$, the corrosion of stainless steel was about the same as at the 0.04 m level; but with 0.08 m $BeSO_4$, corrosion was much more severe. (AEC Activity 4103.1)

Small-Scale Dynamic Slurry Corrosion Tests - Slurries prepared with thorium oxide calcined at various temperatures were circulated in toroids at 26 fps and 250°C, with 50 psi oxygen. The slurries contained 500 g of thorium per kilogram of water, and the thoria used, which was prepared from thorium oxalate precipitated at 10°C, was calcined at 1000°, 1200°, 1400°, or 1600°C. In 314-hr runs, no significant effect of the calcining temperature on corrosion rates of type 347 stainless steel, titanium, Zircaloy-2, or carbon steel SA212 grade B was found. The average size of the particles in the slurry, ~ 1-1.5μ, changed little during these 314-hr runs.

Slurries containing 500 and 1000 g of thorium per kilogram of water, prepared from thorium oxides calcined at 550° and 650°C, were also circulated in toroids at 26 fps and 250°C, with 50 psi oxygen. In 309-hr runs, the corrosion rates were normal or slightly lower for type 347 stainless steel, titanium 75A, Zircaloy-2, or carbon steel SA 212 grade B. In general, the corrosion was

slightly less in the slurry containing the lower concentration of thoria that was calcined at the lower temperature. After these tests, the particle sizes were ~ 0.7μ. (AEC Activity 4103.1)

Dynamic Slurry Loop Corrosion Tests - For a series of three tests, approximately 3000 ppm of sulfate ion (based on thoria) was added as sodium sulfate, sulfuric acid, and thorium sulfate, respectively, to slurries containing average concentrations of 484 to 988 g of thorium per kilogram of water, prepared from ORNL-produced thorium oxide calcined at 800°C. In the three oxygenated-loop runs at 300°C the corrosion rates of austenitic stainless steel, Zircaloy-2, and titanium pin specimens were of the same order of magnitude as those observed during previous tests without sulfate ion. Slurry spheroids, 6 to >120μ, which formed during circulation in the three tests, had no apparent effect on corrosion rates.

Oxygenated slurries containing average concentrations of 478 g of thorium per kilogram of water, prepared from ORNL-produced thorium oxide calcined at 650°C, were circulated at 300°C for 304 hr at several different velocities. Average corrosion rates of pin specimens exposed in slurry circulated at relative velocities of 10, 20, and 30 fps were, for austenitic stainless steels, 4, 6, and 6 mpy for the respective velocities; for Zircaloy-2, 0.8, 2, and 4 mpy; and, for titanium and its alloys, 2, 2.5, and 3 mpy. (AEC Activity 4103.1)

In-Pile Solution Corrosion Loops--LITR - Dismantling of loop L-4-13 was completed. This was the first loop experiment in which heavy water was used as the solvent. The loop was fabricated from type 347 stainless steel and contained 0.17 m UO_2SO_4. The operating temperature was 250°C in the main stream. The corrosion rates of zirconium specimens in the core were in general agreement with rates observed at the same power density in previous experiments.

Irradiation of in-pile loop L-4-16 was completed. The total circulation time after insertion was 898 hr. The energy output of the LITR during this time was 2325 Mwhr, and essentially all the energy was liberated at the 3 Mw level. The loop was fabricated from type 347 stainless steel and contained 0.17 m UO_2SO_4 solution at 280°C. The overall stainless steel corrosion rate was 0.5 mpy, based on oxygen data, and 0.5 mpy, based on nickel-in-solution data. (AEC Activity 4103.1)

In-Pile Solution Corrosion Loops--ORR - The ORR loop facility design was modified to include a retraction mechanism with an 18-in. stroke. This permits a reduction in neutron and gamma flux in the experiment by a factor greater than 10 without reduction in reactor power. The primary need for the retraction is to avoid the possibility of overheating, which might otherwise result under some circumstances because of the high rate of gamma heating which will prevail near the lattice after reactor shutdown. (AEC Activity 4103.1)

Autoclave Radiation Corrosion Studies--LITR - A Zircaloy-2 autoclave was recently exposed in HB-5 of the LITR at a sequence of temperatures: 250°, 280°, 330°, and 300°C. This autoclave contained a solution of 0.04 m UO_2SO_4,

0.03 m H_2SO_4, and 0.005 m $CuSO_4$. The calculated power density varied from 4.5 w/ml at 250°C to 3.7 w/ml at 330°C because of the effect of temperature on solution density. The corrosion rates, in their chronological order of observation, were 7.7 mpy at 250°C, 14.5 mpy at 280°C, 22.6 mpy at 330°C and 12.5 mpy at 300°C.

It is interesting to compare these results with those of an earlier Zircaloy-2 experiment for which the solution used was 0.04 m UO_2SO_4, 0.05 m $CuSO_4$, and 0.02 m H_2SO_4. In the earlier experiment the exposure was to a fission power density of about 4 w/ml, and the Zircaloy-2 was corroded at a rate of 10.3 mpy at both 280 and 300°C. The results from these experiments are similar in that they both show a discontinuity in the temperature effect on corrosion in the range 280 to 300°C. (AEC Activity 4103.1)

GENERAL REACTOR RESEARCH

Long Range Planning Studies - Calculations of reactivity lifetimes in heterogeneous, graphite-uranium reactors were extended to include slightly-enriched systems. Neutron-absorption in U^{235}, U^{238}, Pu^{239}, Pu^{240}, Pu^{241} and fission-product poisons were considered, and k_e was obtained as a function of fuel exposure. Reactivity lifetimes of 7,800, 11,000, 19,000, and 42,000 Mwd/ton were calculated for reactors containing 0.71, 1.0, 2 and 5% enriched-uranium, respectively, based on reactors having equal initial investments in fuel and moderator.

Initial results were obtained for the steady-state temperature distribution within the ORNL Graphite Reactor based on the assumption that the reactor contains highly-enriched, annular-type, cylindrical fuel elements. The air passing through the central region of the fuel has a spatial temperature distribution nearly the same as that of the air passing outside the fuel elements. There was little temperature variation in the radial direction within the graphite for a given cell. (AEC Activity 4510)

Basic Reactor Research - The ratio of the number of neutrons released per fission of U^{233} to the number released per fission of U^{235} was determined by a fast-coincidence method to be 1.024 ± 1%. The results of two different runs agreed to within 0.5%. The values for this ratio published previously in BNL-325 and KAPL-1464 were 1.033 ± 2% and 1.017 ± 2.2%, respectively. (AEC Activity 4520)

Basic Shielding Research - Measurements of the radiation attenuation of ordinary concrete slabs, separated by varying thicknesses of water, were analyzed with respect to the effect of the water content of the shield on its neutron attenuation. It was found that 7 wt % water content is adequate to ensure that penetration of neutrons in the intermediate-energy region is not excessive. Greater water content than this improves the overall neutron attenuation according to the removal cross section theory. A calculation of gamma-ray attenuation, with the use of buildup factors obtained by the moments method (Goldstein and Wilkins, NYO-3075), gave a value in good agreement with the measured quantity. (AEC Activity 4570)

Metallurgical Materials and Processing - Carburization and intergranular corrosion are being investigated as a method for assisting in the recovery of unburned uranium from spent stainless steel-clad fuel elements. A sequence of metallurgical treatments, including carburization, homogenization, and sensitization, develops extreme sensitivity to intergranular corrosion in both types 304 and 347 stainless steel materials used in APPR fuel elements. A total time of about 5 hr is required for the metallurgical treatments. The material is then reacted for 2-6 hr with a boiling solution of copper sulfate and sulfuric acid to corrode the material sufficiently to allow it to be crumbled into individual grains of metal. Uranium is not dissolved but is recovered by subsequent leaching with boiling 10 M nitric acid for 2 hr. A few laboratory tests have indicated that 99.6% of the contained uranium is recovered in the leaching solution and that 80% of the stainless steel is left as a granular powder.

Specimens of type 347 stainless steel-clad fuel plate material were given similar metallurgical treatments and then corroded with a solution of 4 M nitric acid and 2 M hydrofluoric acid for 3 hr. After these treatments, 40% of the stainless steel was left as a powder, and in excess of 99.9% of the uranium was recovered in the solution. (AEC Activity 4610)

Waste Studies - An ion-exchange resin with phosphoric exchange groups, Duolite C-65, was tested for waste treatment application. Its affinity for radioactive zirconium and niobium was superior to that of the corresponding sulfonate resin, but its affinity for rare earths, strontium, and cesium was inferior. (AEC Activity 4620)

PROGRAM 5000 - PHYSICAL RESEARCH

Reactor Operations - Reactor operations were normal. The occurrence of ruptured slugs in the graphite reactor decreased from three in January to one in February. Twelve discharged slugs, chosen at random, were measured and found to have grown from 0.090 to 0.637 inches beyond their original 4-in. length.

At the LITR the unexplained dropping of shim rods was found to be caused by slight drifts in the output of the magnet amplifiers. The magnet amplifiers were adjusted to a low scram level during routine magnet drop tests and therefore the slight drifts resulted in scrams. An improved method of setting magnet currents has eliminated scrams associated with magnet current drifts.

ORR Project - Construction work on the reactor building, the reactor shielding, and the cooling system is estimated to be 95% complete; the contractor is now estimating release of the building to the Laboratory as about June 1, 1957. It is still anticipated that six months will be required for installation of the reactor and its control system and for overall checking. Thus, a tentative date for initial full power operation is December 1, 1957.

The placement of the barytes concrete shield is essentially complete. The remaining work will be primarily mechanical and electrical, along with general clean-up of many small items.

The reactor tank was sent to the Portsmouth Naval Yard for checking and machining. Completion of this work is expected in early May.

The design of the combination collimator-plug arrangement for the horizontal beam holes was completed and reviewed by the interested experimenters. Final details of the fabrication drawings are being completed prior to initiating shop work.

ISOTOPE PRODUCTION

Radioisotope Production - Cesium chloride powder containing approximately 8,100 curies of Cs^{137}, with a specific activity of 21 curies per gram of dry powder, was obtained from purification of Cs^{137} feed from tank-farm waste solutions.

Testing and tracer runs were started on the short-lived fission products processing installation.

High-purity sulfur target material that is void of the usual organic contaminants was obtained by boiling commercial-grade sulfur with MgO at 800°F for one week and distilling off the sulfur produced. (AEC Activity 5111)

Radioisotopes, Process Development - Phase I construction of the Fission-Product Pilot Plant is essentially complete, and responsibility for the building has been accepted by ORNL. The Phase II equipment installation is under way.

The quartz containers for sulfur samples were found to be broken after irradiation in the LITR, apparently as a result of phase changes which brought about expansion of the sulfur. Platinum containers are therefore being fabricated for the irradiation of sulfur to produce P^{32}.

The target material for Cs^{134} production was changed from Cs_2CO_3 to CsCl because of gas pressure difficulties in opening ampoules containing irradiated Cs_2CO_3.

A relatively pure Ag^{111} product was obtained by dissolving the Pd target in aqua regia, evaporating to a small volume, and converting to the chloride

with 12 N HCl. The negatively charged complexes of Pd, Ir, and Au were adsorbed on an anion exchange resin, Dowex-2. The Ag activity was not adsorbed. A 3-g Pd-metal target was processed to recover 154 mc of $Ag^{111}NO_3$ solution with no detectable nonvolatile matter or radiocontaminants and ~1 mg/ml total solids. (AEC Activity 5112)

PHYSICS

Recoil Spectrometry - Molecular break-up following radioactive decay of C^{14} in CO_2 has been studied in the charge spectrometer. The CO_2, isotopically enriched to 38.6% C^{14}, was generated from $BaCO_3$ and introduced into the apparatus at an indicated pressure of about 1.5×10^{-5} mm of Hg. (The spectrometer was described in detail in ORNL-2076, p. 39.) The CO_2 was then subjected to magnetic analysis to determine the relative abundances of positively charged ions following the radioactive decay of the constituent C^{14} atom.

Preliminary results yield the following distribution:

e/m	Ion	Relative Intensity
1/46	$(NO_2)^+$	81.4 ± 1.6
1/30	$(NO)^+$	8.4 ± 0.4
1/16	O^+	5.9 ± 0.6
1/14	N^+	3.6 ± 0.4
2/46	$(NO_2)^{++}$	0.4 ± 0.06
1/32	O_2^+	0.2 ± 0.4
2/30	$(NO)^{++}$	0.06 ± 0.04
2/14	N^{++}	0.02 ± 0.01
2/16	O^{++}	0.01 (upper limit)

(AEC Activity 5220)

Neutron Diffraction - Neutron diffraction patterns were obtained for polycrystalline manganese sesquioxide at a series of temperatures in the range 4.2-298°K. Coherent magnetic reflections are observed in the low-temperature patterns at superlattice positions of the chemical unit cell. A study of the temperature dependence of these reflections yields a Neel temperature of 88°K, in reasonable agreement with that indicated by the λ-type specific heat anomaly at 79°K that was observed for the system. The paramagnetic diffuse scattering at 298°K, as obtained from a 4.2-298°K difference pattern, corresponds to an effective spin value of 1.8. This value is low because complete magnetic order was not attained at 4.2°K, but it shows fair agreement with the value S = 2 for the Mn^{3+} ion. A unique structure determination has not been made, but a qualitative correlation with the data was obtained on the basis of a four sublattice model involving previously discussed structure types A, C, G, and E (E. O. Wollan and W. C. Koehler, Phys. Rev. 100, 545, 1955). (AEC Activity 5220)

High Voltage Program - A preliminary measurement of the polarization of fast neutrons was undertaken with the use of elastic neutron scattering from helium as a polarization analyzer. Neutrons with an unknown degree of polarization were produced through the $T(p,n)He^3$ reaction by 5.2 Mev protons accelerated with the 5.5-Mv Van de Graaff. Neutrons with energies of 2.66 Mev were observed at an angle of 70° to the proton beam with a helium-filled cloud chamber. The volume-controlled expansion-type cloud chamber was 10 in. in diameter and 4 in. deep, and it was filled with helium gas at a pressure of 18 psi. The number of helium recoil tracks to the right (R) and to the left (L) give the product of the reaction polarization (P_R) and the analyzer polarization (P_A) by the expression

$$P_R P_A = \frac{L - R}{L + R} .$$

All tracks that make an angle between 10° and 80° with and to the right and left of the line joining the target and the chamber were counted, subject to a restriction on the length of the track, to give a value of $P_R P_A = -0.052 \pm 0.048$. The narrower angular spread of 30° to 60° gave $P_R P_A = -0.005 \pm 0.073$. These values are in agreement with a small value for P_R and/or P_A. The energy and angles chosen correspond to $P_A = -0.09$ (calculated). This small value was purposely chosen to check for possible instrumental asymmetries in this preliminary measurement. (AEC Activity 5220)

Neutron Velocity Selector - The housing, stators, collimators, optical system, etc., for the new LITR time-of-flight spectrometer are completed and are being assembled. The final machining of the slits in the stainless steel rotor is in progress. Thick samples of U^{234} and Pu^{240} were prepared and measurements in the thermal energy range are in progress with the old spectrometer. (AEC Activity 5220)

CHEMISTRY

Volatility Studies - In tests on the simulated fluorination step of the fused-salt-dissolution fluoride-volatility process for recovering uranium, changing the diameter of the fluorine inlet tube did not affect the efficiency of the fluorine--fused salt contact at gas flow rates below 5.0 cfm. The tube diameters investigated were from 2-15/16 to 6 in. When the liquid level was allowed to fall below the top of the draft tube, the efficiency was greatly decreased except at the highest gas flow rate tried, that is, 5 cfm. A disk-and-doughnut baffled draft tube was much more efficient than an unbaffled tube at gas flow rates below 1 cfm, but was somewhat less efficient at higher rates. The tests were made with CO_2 gas and NaOH rather than with actual process chemicals.

In laboratory studies, a micrometallic nickel filter removed some entrained fission products from the UF_6-F_2 fluorinator exit gas stream. Uranium and plutonium were not removed.

Preliminary results of a single run with nonirradiated material in the UF_6 processing equipment of the fused salt--fluoride volatility process pilot plant were satisfactory. In further laboratory studies, kinetic tests indicated that absorption of UF_6 on NaF was complete when the NaF was 20% saturated. Higher saturations were not studied. Decontamination of the UF_6 product was increased when the first NaF absorption bed was flushed with excess fluorine during the absorption cycle.

The rapid oxidation of pilot plant stainless steel equipment in the presence of HF (ORNL-2261) at 650°C indicated the possibility of oxidizing stainless steel-containing fuel elements under these conditions. In preliminary tests on such a procedure various types of stainless steel were penetrated at rates ranging from 0.5 to 27 mils/hr. In one test, a 30-mil-thick type 304 stainless steel element with a UO_2-type 304 stainless steel core was completely converted in 1 hr to a substance from which 95% of the uranium was recovered by either aqueous acid leaching or high-temperature fluorination. (AEC Activity 5310)

Chemical Engineering Research Studies - An Elox electrical-discharge cutting machine is being investigated for disassembling fuel elements. Demineralized water was successfully substituted for the oil coolant usually used with this machine and which decomposes under irradiation. There were no indications of spark hazards, and stainless steel and zirconium disintegration rates were 0.6 and 0.8 cm^3/hr, respectively. It is estimated that 1.64 hr would be required to make one cut through an APPR stainless steel subassembly side plate 0.05 in. thick and 24 in. long. (AEC Activity 5310)

Neptunium Process Development - The Purex process first-cycle flowsheet (Report ORNL-846) was modified to permit recovery of Np^{237} in the second uranium cycle by selective stripping in an added partitioning column. In batch tests of the modified flowsheet, 0.01M $NaBrO_3$ added to the aqueous scrub oxidized the Np(IV) or (V) to the extractable Np(VI) form, which was carried to the partitioning step. Neptunium distribution coefficients under process conditions were 0.52 and 1.20 after 1 and 45 min, respectively, which indicates that 6-12 min contact will be satisfactory under extraction column conditions.

Batch equilibration and countercurrent experiments under partitioning-column conditions indicated that neptunium would reflux but would remain in the solvent and be carried into the stripping column.

Tests of the modified flowsheet in the 0.75-in.-dia pulsed columns indicated that, as long as the partitioning column acidity is at the equilibrium value, the neptunium will follow the uranium. If the acidity is lower, it will follow the plutonium. (AEC Activity 5310)

Extraction Reagent Performance - The amount of unsaturated compounds formed in the kerosene diluent Amsco 125-82 increased with increasing irradiation up to 68 watt-hr/liter ($\sim 1.76 \times 10^7$r). However, the rate of formation decreased above 50 watt-hr/liter. Massive dosages of radiation produced measurable amounts of light fractions and gases and a viscous residue of polymers of higher boiling point than that of unirradiated Amsco. (AEC Activity 5310)

High-Level Nuclear Chemistry - In the final purification of 30 g of recently isolated Np^{237} to be used for neutron threshold detection work, it was necessary to assure maximum purity, especially with respect to fissionable contaminants such as plutonium and americium. For this purpose several ion-exchange techniques were applied to a product of 95.95% purity that had resulted from a conventional chemical treatment.

For the specific decontamination from uranium and all metals except calcium and the transuranic elements, precipitation of NpF_4 was used most effectively. In a single step of anion adsorption of the Np(IV) chloride complex, Pu^{239} was removed as Pu(III) by reduction with SO_2 and its concentration determined by the method of isotopic dilution with Pu^{238}. This step also successfully removed calcium, americium, and curium. An indirect method, based on the stability of Np(V) in the 8 M HNO_3 system in the presence of nitrite, gave an equally good decontamination (D.F. ~ 10^6) by adsorption of Pu(IV). The removal of iron by anion exchange based on its higher adsorbability at 6 M HCl was found to be unreliable in the presence of large quantities of neptunium. This step was supplemented by the usual diisopropyl ether extraction method. (AEC Activity 5330)

High-Temperature Aqueous Solution Chemistry - With the recently-constructed high-temperature isopiestic unit, preliminary thermal-dependence data were collected in the range 50-100°C on the alkali chlorides and bromides and on the sulfate salts, Na_2SO_4, UO_2SO_4, and $MgSO_4$. Because of loss of water to the vapor phase all the salt solutions became more concentrated as the temperature was raised; however, there were some pronounced differences in behavior. For example, the molality of NaBr went from 0.636 at 46.4°C to 0.666 at 74.3°C to 0.683 at 86.9°C, as compared with Na_2SO_4, which remained nearly constant at 1.02 m over this range. The total over-all error in the system appears at present to be approximately 1%. (AEC Activity 5330)

Ion-Exchange Studies - The self diffusion of bromide ion at 25°C in Stamex-S-44, a phenol-formaldehyde sulfonium type of anion exchanger, was found to be 1.18×10^{-7} cm^2/sec. This value is considerably smaller than that reported for Dowex-2, 6% DVB (divinylbenzene), where 3.87×10^{-7} cm^2/sec was obtained under the same conditions. These findings and the selectivity scale and moisture isotherms previously reported indicate that the sulfonium type of anion exchanger differs from the more common polystyrene quaternary ammonium type of resin principally in its rate of exchange.

It has been suggested that an irreversible distention of the copolymer network occurs when large organic cations such as the tetra-alkyl-ammonium ions are absorbed by cation exchangers. If this is the case, the equation of state for the exchanger should differ from that obtained for salt-forms with smaller ions. Swelling pressures for several tetra-alkyl-ammonium salt forms of Dowex-50, 8% DVB, were computed from previously obtained moisture absorption isotherms; molal volumes were determined pycnometrically; and pressure-volume isotherm plots were constructed and compared with the plot for the hydrogen form of the same resin. The slopes of the isotherms for these large cation salt forms were found to differ considerably from that for the hydrogen,

and, in the case of the tetra-butyl-ammonium form, negative pressures occurred. These results may be interpreted as the consequence of a breaking of cross-links in the exchanger. (AEC Activity 5330)

Chemical Separation of Isotopes - The heat of reaction of BF_3 with anisole determines the cooling requirements for the recombiner in the Anco system for the separation of boron isotopes. The heat of reaction was calculated from the vapor pressures above various compositions of the complex, taken at several temperatures. In the temperature range of 15-40°C, $\Delta H = -12.3$ kcal/mole.

The fractionation of oxygen isotopes between gaseous CO_2 and CO_2 dissolved in acetone was measured. The separation factor at -78°C and 720 mm of Hg is 1.006 ± 0.005 (95% C.I.) with O^{18} enriching in the liquid phase. (AEC Activity 5340)

PROGRAM 6000 - BIOLOGY AND MEDICINE

BIOLOGY

Cytology and Genetics--Paramecium - Previous work showed that the number of lethal mutations produced by X-irradiation of Paramecium could be decreased by treatments that delayed cell division, such as exposure to streptomycin. It has now been shown that the lethal mutations produced by alpha particles can also be decreased in this way. This finding is especially significant because damage by irradiation with heavy particles has proved refractory to several other modification procedures. (AEC Activity 6130)

Cytology and Genetics--Plants and Fungi - In Neurospora evidence from reconstruction filtration experiments on macroconidial heterokaryons with disproportionate nuclear ratios indicates that the recovery of pre-existing mutants in the original population is not increased in proportion to the increase in the uninucleate population resulting from X-irradiation. These results suggest the existence of an interaction between viable mutant nuclei and X-ray inactivated wild-type nuclei in multinucleate conidia which precludes the recovery of such mutant conidia with the use of the "filtration-concentration" technique.

In Neurospora exceptional pseudo-wild types, believed to originate as nondisjunctional disomic nuclei, are unusual in that mitosis is abnormal and results in the formation of haploid nuclei. Studies now in progress show that among the ascospores the proportion of disomic pseudo-wild types in survivors of X-irradiation is drastically reduced in comparison to that in the unirradiated controls. This indicates that the reduction of the disomic into normal haploid nuclei occurs at least as early as the second postmeiotic division. (AEC Activity 6130)

Pathology and Physiology - RF mice were exposed to 250-kvp X-rays, to cobalt-60 gamma rays, and to neutrons of 14-Mev energy produced in a Cockcroft-Walton accelerator by the $T(d,n)He^4$ reaction. The $LD_{50}/30$ days was observed to be 917 rad of gamma rays, 660 rad of X-rays, and 627 rad of neutrons. If the RBE of gamma rays is assumed to be 1.0, then the RBE of X-rays is 1.39 and the RBE of 14-Mev neutrons is 1.47. The latter value is significantly lower than those reported previously for neutrons of lower energy. This is consistent with the evidence that the RBE of neutrons varies with their ion density.

Mice of the RF strain were exposed to Co^{60} gamma rays at varying intensities, beginning at 10 weeks of age. After exposure the animals were examined periodically throughout life, with the slit lamp, and the incidence and severity of lens opacities were recorded. The extent of lens injury varied not only with the total dose but with the intensity of radiation. The rapidity of onset, rate of progression, and final severity of lens opacities were several times greater if the radiation was administered at relatively high intensities than if administered at much lower intensities. For example, 7000 rad delivered at a rate of less than 0.06 rad/min was less damaging to the lens than 900 rad administered at a rate of 7 rad/min. (AEC Activity 6130)

Cell Physiology - An intermediate-scale gradient centrifuge was developed to the testing stage. Separation of rat liver mitochondria in one sedimentation was achieved. The fractionation of rat serum proteins progressed. Albumin and gamma-globulin were isolated that are free of contamination that is detectable electrophoretically or ultracentrifugally. The purification of other serum proteins is proceeding rapidly. Purified proteins are used to produce antisera which serve as specific reagents for the detection of the protein antigen in unresolved mixtures. (AEC Activity 6130)

Biophysics - It seems to be the invariable rule that, when two chromatid-type Breakage-Fusion-Bridge cycles are generated in the same nucleus, they continue in subsequent cell divisions as separate, independent chromatid-type cycles, and they never fuse to yield a chromosome-type cycle. This is probably an expression of the fact that free chromosome ends have to be in close physical proximity in a cell for an exchange to occur, as was indirectly deduced from various radiation experiments. The evidence in the present work is essentially of an all-or-none qualitative nature, and some effort was devoted to making a good photographic record. The examination of this material was completed.

Studies of the variations in radical production as a function of linear energy transfer in irradiated amino acids were extended to include 5.3-Mev polonium alpha particles. For equal energy absorbed these measurements show alpha particles to be approximately 0.1 as effective as gamma rays. In glycine the structure of the resonances from gamma rays, D-T neutrons, and alpha particles are identical, and thus it is shown very definitely that the type of radicals produced is independent of linear energy transfer in these simple amino acids. Studies of the effect of intensity and dose are in progress.

Application of the resonance techniques to the study of radiation protection in bacteria is continuing. Escherichia coli irradiated in the presence of AET (the S,2-aminoethylisothiuronium·Br·HBr compound) shows a free-radical resonance considerably less than that observed in the unprotected controls. Experiments now in progress also indicate a difference in shape, as well as in magnitude, in the signals obtained from the protected and unprotected bacteria. (AEC Activity 6130)

Microbial Protection and Recovery - The lethal and membrane effects of short and long ultraviolet X-rays were examined. X-ray and short ultraviolet (2537 Å) radiation have striking similarities in that the ability of the cells to retain potassium is much less sensitive than is the lethal effect. Exactly the opposite is true for long ultraviolet radiation (3000-4000 Å). No satisfactory explanation has been found to date for this phenomenon. (AEC Activity 6230)

Mammalian Recovery - The thymus of the irradiated mouse, treated with rat bone marrow, was studied to determine whether it stayed that of a mouse or changed to rat type. For the first three weeks after irradiation, the thymus cells reacted toward antisera like those of a mouse. After this, they reacted like those of a rat.

The pathologic changes in mice dying after rat bone marrow treatment were studied and shown to be essentially identical to those seen after homologous bone marrow therapy.

Cell lines, originally obtained in other laboratories from mouse bone marrow (CF1C9 and MC32), rat bone marrow (R), and a mouse lymphosarcoma (MBIII), are being tested as possible agents for modification of the radiation syndrome of lethally irradiated mice. The results to date indicate that cell lines MC32 and MBIII are negative in this respect, while CF1C9 cells have prolonged the survival time of a small percentage of lethally exposed C_3H X $101F_1$ mice in one of two experiments. The development of alkaline phosphatase positive material in C_3H X $101F_1$ mice exposed to 950 r and then given the R cells was observed at 3 and 5 days.

During the first 30 days after X-irradiation the hematocrits, leukocyte, and platelet counts, and the spleen, thymus, and body weights were essentially the same in leukemoid leukocyte-treated mice as they were in bone marrow-treated mice. (AEC Activity 6230)

Radiation Immunology - Study of the physical properties of rat-in-mouse red blood cells revealed that the surface molecular configuration and the hemoglobin of these cells were identical to normal rat red blood cells. However, these cells possessed dualistic osmotic properties; the relative amount of hemoglobin released when cells were lysed in water was more comparable to the rat red blood cells, but their temperature-dependent property was more comparable to the mouse red blood cells.

Studies of the time of X-irradiation in relation to the time of antigen injection showed that 710 r given 5 days before antigen injection caused an increased antibody response. Marked decreased antibody response was observed when X-radiation was given 3 days before to 5 days after antigen injection. (AEC Activity 6230)

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STATUS OF CONSTRUCTION

Additional Boiler for Steam Plant, Building 2519 - The ORNL steam plant, Building 2519, which was placed in operation in 1948, included sufficient space and foundations for the installation of an additional boiler at some future date to provide for the anticipated expansion of the Laboratory's programs and facilities. The three coal-fired boilers in the original installation had a total rated capacity of 150,000 lb of steam per hour; however, the conversion of these boilers, in 1950, from coal to gas firing reduced the output of the boilers to approximately 120,000 lb/hr under normal conditions and a maximum of 135,000 lb/hr under short-period emergency operating conditions. The actual peak steam-load demand increased from 61,000 lb/hr in 1948 to 116,000 lb/hr in 1954, which approached the normal capacity of the three converted boilers.

The increase in the steam usage plus the anticipated programmatic expansions thus dictated the installation of additional steam-producing facilities. Consequently, the Atomic Energy Commission authorized the installation of a 50,000 lb/hr gas-fired (oil stand-by) boiler with the required accessory draft fans, pumps, safety valves, flues, etc. The physical installation of the boiler and its appurtenances, which was started in December 1956 by A. Dierks and Company under an AEC prime construction contract, is to be completed by the end of March; however, final tests and the minor adjustments required before the facilities are placed into operation will not be concluded until some time in April.

The major portion of the engineering design on this installation was automatically supplied by the vendor for the boiler as part of the boiler purchase contract. The Laboratory provided the balance of the engineering services, which consisted of establishing the design criteria and specifications, preparing the drawings for the extension of utilities, and coordinating and inspecting the work. The Laboratory also assisted the contractor with miscellaneous field work.

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Ramsey, D. A., National Research Council of Canada

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Weiller, R., IBM Corp. (Citizen of Switzerland

RADIOISOTOPE SALES AND COSTS

Type of Transaction	February 1957	FY 1957 to Date
Domestic Sales	$103,354	$1,151,233
Foreign Sales	3,818	86,112
Project-Transfer	1,886	9,337
Project-Cash Sales	9,461	37,696
Technical Co-operation Program Credits	0	3,395
Plant Credits	-4,139	26,084
AEC Credits	20,941	111,159
Total Radioisotope Income	$135,321	$1,445,957
Total Radioisotope Costs	$199,943	$ 962,994
Total Radioisotope Shipments	1,068	8,805
Helium		
Income	$ 855	$19,077
Costs	$1,014	$ 5,955
Shipments	2	20

GROSS OPERATING COSTS

	Cost for February	FY 1957 Cost to Date
Programmic Operating Cost - Net	$4,525,213	$33,909,323
Equipment	230,153	1,717,685
Construction	178,031	1,254,657
Work for Others - Transfers	59,892	395,530
Inventory Changes	6,587	6,018
Reimbursable Work for Others	20,775	577,233
Deferred Charges	(3,557)	(264,406)
Total Laboratory Cost - Net	$5,017,094	$37,596,040
Estimated Cost for Next Month	$5,300,000	$42,896,040

()Credit

PERSONNEL SUMMARY

	Number of Employees March, 1957	New Hires March	Terminations March
Administration	73	0	0
Operations*	132	2	0
Engineering, Shops, and Mechanical	861	4	5
Laboratory and Research	2178	25	32
Protection	117	0	0
Service	374	0	9
	3735	31	46

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

939 Laboratory employees are working in the Y-12 Area.

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Reports in this series issued during the past year:

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November 1956	ORNL-2232
December 1956	ORNL-2248
January 1957	ORNL-2261
February 1957	ORNL-2281

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