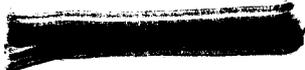




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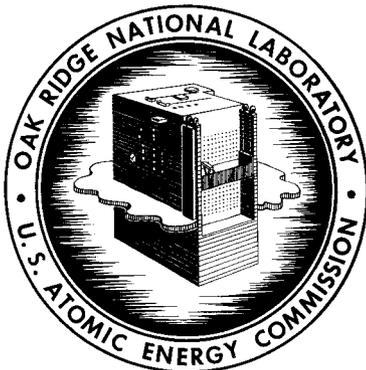
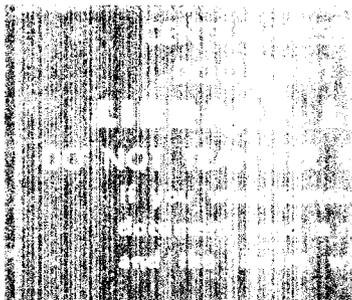


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OAK RIDGE NATIONAL LABORATORY
STATUS AND PROGRESS REPORT
DECEMBER 1960



OAK RIDGE NATIONAL LABORATORY
operated by
UNION CARBIDE CORPORATION
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U.S. ATOMIC ENERGY COMMISSION



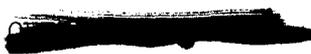
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

DECEMBER 1960

DATE ISSUED

JAN 9 1961

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
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U.S. ATOMIC ENERGY COMMISSION



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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

December 1960

This Status and Progress Report summarizes that portion of the Laboratory's work which is unclassified. Some of the topics are included every month, but the majority are reported on a bimonthly schedule.

SPECIAL NUCLEAR MATERIALS PROGRAM

Chemical Plant Criticality Studies. - A series of experiments related to nuclear safety in handling and storage of solutions of fissionable materials is in progress. A number of 5-3/8-in.-OD seamless polyethylene bottles, having a capacity of 12 liters and containing an aqueous solution of $UO_2(NO_3)_2$ with 92% U^{235} at a concentration of 410 g of uranium per liter, are being utilized. The arrays are nominally unreflected with no moderator present except that inherent in the individual units. The surface-to-surface separation distances required for criticality when the bottles are placed in various square-planar arrays with axes vertical have been determined for various solution heights in the bottles. For example, 81 half-filled bottles were critical at a separation distance of 4.78 in., while 81 full bottles must be separated 7.79 in. A comparison of square and triangular patterns in the arrays indicates that, for the same separation distance, the triangular pattern is more reactive. For example, with a separation distance of 3.90 in., 20 and 16 bottles, respectively, are required for criticality in the two patterns.

Dissolver Solution Analyses: Uranium-Molybdenum Fuels. - A coulometric titration for uranium-molybdenum samples in sodium tripolyphosphate was reported previously (ORNL-3038, p 1). In volatility process feed samples, uranium constitutes less than 1% of the total sample in fuels of this kind. Therefore, in order to concentrate the uranium and to remove some of the interferences, the titration has to be run after a preliminary resin column separation. The precision and accuracy of this method appear to be approximately within $\pm 2\%$.

Work is also in progress on a polarographic method for U(IV) in sodium tripolyphosphate medium.

Dissolver Solution Analyses: Uranium by Indirect Controlled-Potential Coulometry. - Exploratory work has been done on the application of the indirect controlled-potential coulometry, previously developed for plutonium, to the determination of uranium. It involves generation of an excess of intermediate reagent (in this case, ferric iron) at controlled potential within the titration vessel. The unreacted reagent is then electrolytically back-titrated after reaction is complete, and the amount of reagent consumed is measured by difference.

[REDACTED]

A small amount of data indicates that: (1) Cr(II) is a satisfactory reducing agent for valence adjustment prior to titration, (2) approximately 5 mg of uranium can be determined by this technique with a relative standard deviation of 0.2% or better, and (3) the method is not subject to interference from large quantities of iron, nickel, chromium, and chloride or from small amounts of nitrate. Accordingly, the method offers particular promise for the determination of uranium in stainless-steel-type dissolver solutions.

Dissolver Solution Analyses: Plutonium. - A brief investigation was made of a BiPO_4 precipitation technique for separating plutonium prior to its determination by controlled-potential coulometric titration, but it shows no advantage over the previously reported LaF_3 precipitation-coulometric titration method.

Molybdenum and zirconium were studied as potentially interfering elements in the controlled-potential coulometric titration procedure for plutonium. Molybdenum(VI) does not interfere when the Mo/Pu weight ratio in the supporting electrolyte solution is less than 10:1. An error of -0.5% was observed when the ratio was increased to 20:1 (approx 250 μg of Pu was titrated). Zirconium does not react electrolytically within the potential span of the plutonium titration but does interfere seriously in the titration, apparently by passivation of the working electrode.

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROJECT

Pebble-Bed Reactor Study. - A design study of a 330-Mw (electrical) pebble-bed reactor power plant with a net thermal efficiency of 40.5% has been completed. The reactor thermal output (800 Mw) is removed by helium entering at 550°F and leaving at 1350°F under a pressure of 700 psia. Three concepts of gas flow through the core were analyzed: axial down-flow, radial outflow, and axial upflow; the last was chosen for the design study.

The cylindrical core is 20.7 ft in diameter and 12.4 ft high. It is surrounded by a 3-ft-thick graphite reflector. The core and the reflector are contained in a 31.4-ft-ID spherical pressure vessel of 4-in.-thick carbon steel.

The fuel consists of 2.5-in.-diam graphite balls containing 0.44 wt % U^{235} and 8.45 wt % Th. The core power density is 6.6 w/cm^3 and is limited by the levitation of the fuel. Larger fuel balls, which could withstand higher gas flows without levitation, appeared to be limited to lower power densities because of thermal stresses. The conversion ratio, which is initially 0.75, will eventually rise to about 0.8. The fuel residence time in the core is 700 days.

The two steam generators are once-through boilers containing economizer, boiler, reheat, and superheat sections. Feed water enters at 400°F and leaves as steam at 2480 psia and 1055°F. Reheat steam enters at 470 psia and 623°F and leaves at 460 psia and 1005°F. The turbine-generator, a tandem-compound type, is rated at 345 Mw and is designed for indoor installation.

EGCR Physics. - Measurements of k_{∞} for the EGCR core lattice were made in the Physical Constants Test Reactor at Hanford. In order to establish these measurements as a base point for criticality calculations for the EGCR, k_{∞} was calculated for the test lattice, which is different in some details from the actual EGCR lattice. The calculated k 's are completely independent, in that they do not utilize any of the PCTR data. The results, for 1.8 and 2.6% enriched uranium, are as follows:

<u>Uranium Enrichment (%)</u>	<u>k (calculated)</u>	<u>k (measured)</u>
1.8	1.142	1.146 ± 0.004
2.6	1.266	1.264 ± 0.008

EGCR Experimental Loops. - The AEC has notified ORNL that completion of loop installation is to be deferred until at least July 1, 1965. Minimum provisions are to be made now in the EGCR construction for permanent portions of the eight loop assemblies to permit future installation of the loops. Present plans are to proceed with title II design of the upper and lower nozzle tee sections, the upper and lower horizontal piping chases, and the necessary chases for cooling duct work. All other loop design work will be carried through title I design only. A preliminary hazards report will be prepared.

GCR-ORR Loop No. 2. - Work has continued on the stainless steel cell liner, which is now about 60% complete. The framework for the decontamination room was erected. The 5-ton remotely operated crane, the omniscopes for cell viewing, and a regenerative compressor were received and will be tested before installation.

GCR-ORR Loop No. 1. - The two regenerative compressors installed in the twin-turbine vessel at GCR-ORR loop No. 1 have continued to operate satisfactorily during numerous tests. The compressors have operated a total of about 1350 hr with helium at 600°F. Operating speeds have ranged from 6000 to 12,000 rpm at a loop pressure of 45 psig.

Back-Diffusion Studies. - The studies of back-diffusion through labyrinth seals have been completed. Tests with both air and helium as the coolant gases gave no indication of back-diffusion of buffer water vapor against the flow of helium through the labyrinth.

Irradiation Experiment Evaluation. - A UO₂ pellet clad in graphite coated with a 1-mil-thick pyrolytic carbon coating was examined following irradiation in the C-1 facility of the LITR. It was found that the pyrolytic coating had flaked off in several places. When the graphite capsule was immersed in hot oil, gas leaked freely from two pinholes at the edge of the capsule. The gas leakage confirmed suspicions that the UO₂ had been exposed to the sweep gas during irradiation; surprisingly, there were no gross cracks in the graphite capsule.

Data are being obtained on the swelling of beryllium during postirradiation annealing of specimens irradiated at 50°C with 6×10^{20} neutrons/cm² (> 1 Mev). No swelling occurred upon annealing for 1 hr at

temperatures as high as 800°C. Longer-period annealing experiments are in progress.

Three samples of 93.5%-dense UO_2 manufactured by Mallinckrodt were irradiated in the ORNL Graphite Reactor and were annealed out-of-pile for a period of one week at 600, 800, and 1000°C respectively. A fourth sample was annealed for 2 days at 1100°C. The total activity of the gas released at 600°C was below the limits of detection. At higher temperatures an initial rapid release of approximately 0.001% of the Xe^{133} occurred in the first hour. Subsequent release was below the limits of detection. Calculations showed that the diffusion rate parameter (D') at 800 and 600°C was at least an order of magnitude below that predicted by recently reported United Kingdom data.

UC₂-Graphite Fuel-Element Fabrication. - Samples of graphite containing both uncoated and pyrolytic-carbon-coated UC_2 particles were prepared by dry-mixing the particles, binder, and graphite; cold-pressing at 45 tons/in.²; and firing at a rate of 20°C/hr to 1000°C in argon. Metallographic examination of the sample containing uncoated particles showed a reaction layer around each particle. Particles in the center of the pellet exhibited a reaction layer of approximately 0.5 mil. A traverse examination from the center of the pellet to the outer edge revealed an increasing amount of the reaction product. Particles along the outer edge of the pellet were completely converted to the reaction product. Final identification of this product has not been completed, but it is believed to be UO_2 . The pyrolytic-carbon-coated UC_2 particles appeared to withstand the fabrication process without microstructural change.

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. - The HRT "initial approach to full power" experiment with modified flow (through the core vessel from top to bottom) continued. Data were collected on inventory, power, and temperature behavior at a number of intermediate power levels. The reactor was operated primarily at a pressure of 1400 psig, at temperatures of 260°C in the core and 230°C in the blanket, and at power levels up to 4.2 Mw. There have been no indications of fuel instability to date with the reversed flow pattern. Normal power oscillations, however, have been "rougher" than they were before the flow was reversed.

Operation was interrupted for about ten days during the month because of a leak in the system. A fuel feed pump had leaked approximately 1 liter of fuel solution from a check valve chamber connection. The pump had a total operating history of approximately 23,000 hr.

After the leak was repaired, the experimental program (run 23) was resumed. Stepwise increase in power will continue above the current level of 4.2 Mw as satisfactory analyses of data at the intermediate levels are made.

HRT Chemical Pilot Plant. - During the first 550 hr of power operation in run 22, only 160 g of corrosion products was collected in the processing plant. Collection rates in excess of 1 g/hr were achieved with the

original multiple hydroclone in the comparable period of run 20; whether the lower removal rate of the revised multiclone is the result of lower efficiency or a reduced solids concentration in the fuel solution cannot at present be determined. The zirconium-to-iron ratio (1:1) found in the solids collected was lower by a factor of 2 to 3 than in any other operation since run 17.

Full-scale nonradioactive testing of the UO_4 fuel processing system was resumed. When a diatomaceous earth filter aid was used, the peroxide precipitate was retained quantitatively and a 4-kg batch could be filtered in about 6 hr.

Analytical Chemistry. - Improvements were made in the flame photometric method for determining calcium. When aqueous solutions are used, the emissivity of calcium, measured at 422.7 m μ , is seriously decreased by sulfuric or phosphoric acid (> 0.0001 M) and by nitric or hydrochloric acid (> 0.01 M); however, it has been found that in test solutions which contain 20% ethylene glycol, each of these acids may be present in a concentration as high as 1 M without significant interference. It was also observed that the tolerance limits for these anionic species are somewhat more favorable if an oxyacetylene flame is used instead of the oxyhydrogen flame, which has a lower temperature. By utilizing this improved method, 10 μ g/ml of calcium can be determined in molar solutions of phosphoric, sulfuric, nitric, or hydrochloric acid with a coefficient of variation of 5%.

In the case of basic phosphate solutions, produced by the addition of $(NH_4)_2HPO_4$, the tolerance limit, even with the addition of ethylene glycol, is 0.01 M. However, if the solution is acidified with nitric acid (to a pH of 0.5 to 1), this limit is increased to 0.1 M.

Slurry Circulation Experiments. - Run 200B4, in which the heat transfer coefficient of a 1600°C-fired 1.8- μ thoria slurry was measured as a function of velocity, temperature, and concentration, was completed. At velocities over 10 fps in the temperature range from 200 to 280°C, the observed heat transfer coefficients were not greatly different from those obtained with water.

A 200B loop test section for measuring friction factors in turbulent flow was designed and is being installed. The section will operate in the Reynolds number range from 10^4 to 4×10^5 .

Capillary-tube viscometer measurements were made with 200B4 and L041 thoria slurries. Each slurry was operated at several concentrations in both flocculated and dispersed conditions in laminar flow at high shear rates. It was shown that at high shear rates the coefficient of rigidity of the flocculated material tends to approach the viscosity of the same slurry when it is dispersed.

Engineering Development. - The HRT mockup loop was operated for about 700 hr at 260°C with an HRT-type fuel solution containing an increased acid content, 0.035 m H_2SO_4 . The corrosion rate averaged over the first 636 hr was 1 mil/yr, only slightly greater than normal.

The oxygen compressor for use in circulating contaminated gas from atmospheric pressure to 2500 psi was restored to test service, following the modification of the drive system to include an intermediate water system. It is expected that the intermediate system will eliminate the transport of dirt from the drive into the diaphragm heads.

The full-scale model of the HRT core was connected to a constant-head tank in order to study variations in mixing rate with time. The variations observed in preliminary runs are very similar to power variations observed in the reactor.

Tools for removing the lower patch from the HRT, for reaming the lower hole, and for installing a new tapered plug were fabricated. These tools were used to establish a more leak-free seal at the lower hole.

Design. - Designs were completed for two mechanisms that will furnish information on the behavior of the HRT core. The first mechanism is a corrosion specimen holder which will extend along the vertical center line of the core. Provision is made for exposing a number of zirconium alloy samples for determination of the effect of environment on corrosion and mechanical properties. Several Zircaloy-clad thermocouples will be mounted to indicate temperature in the vicinity of the specimens. The entire assembly is hydrodynamically streamlined to promote cooling.

The second mechanism is one for positioning a number of thermocouples in those regions of the core which are believed likely to experience variations in mixing rate, and therefore in temperature. Data obtained with this device may provide the basis for a relationship between random flow variations and variations in reactor power.

MOLTEN-SALT REACTOR PROJECT

MSRE Design. - The poison scheme described previously (ORNL-3048, p 6) has been expanded to provide four poison tubes instead of one. This provides enough poison to permit override of excess reactivity introduced by complete penetration of fuel into the graphite up to the theoretical limit. Core design is being modified to include these four tubes.

The design of the neutron thermal shield around the reactor has been completed. A nuclear instrument tube has been designed, giving access to the reactor from the outside for all nuclear instruments. Heater and thermal insulation design for the reactor vessel has been finished.

Piping drawings for the secondary salt circuit have been issued for comment. Radiator drawings with fabrication details have been issued for comment.

All major building modifications (structural) have been frozen, and detailing of footings, shielding, excavating, and partition alterations is now in progress.

The electrical service design is in preparation. Layout of the heater circuits has been started, and disconnect plugs for these circuits, as well as for associated thermocouple lines, have been designed.

Design memorandums have been issued covering the off-gas disposal and cover-gas supply portions of the system.

The hazards report is being written, using the "Preliminary Hazards Report on the Molten Salt Reactor Experiment," which was prepared by a group in the Hazards Evaluation Course of ORSORT. Additions, rearrangement, and updating are being done for completion by January 1, 1961.

Component Development. - Seven corrosion pump loops of INOR-8 have operated with various mixtures of fluoride salts at 1300 to 1500°F for

periods of from 6000 to 19,000 hr. Two loops were dismantled for metallurgical examination after 20,000 hr each at operating conditions.

Two INOR-8 thermal convection loops with graphite-molybdenum test sections have operated at 1300°F with MSRE salt.

The design of the engineering test loop was finished and the fabrication is 20% complete.

The 72% Ag-28% Cu solder alloy freeze valve for use with the sampler-enricher failed after 41 cycles with a total of 73 hr at 1500°F, apparently because of excessive oxidation of the solder.

The resistance-heated salt freeze valve has been cycled 74 times with an average thaw time of 3.5 min. Cycling of the induction-heated valve was discontinued after 60 successful cycles, and a replaceable resistance heater was substituted for the induction coil. For 17 cycles with a maximum heater sheath temperature of 1400°F, this heater has given thaw times of 3.5 min.

Thermal cycling of the 3-1/2- and 4-in. freeze flanges was terminated after 104 cycles between room temperature and 1300°F. No cracking was detectable; however, severe warpage occurred in the 4-in. flange, indicating excessive thermal stress.

The final suggested design report for the improved cross-sectional geometry flange for the MSRE was received from the project stress consultants.

Remote Maintenance. - Mockups of the pump flanges and a flanged section of the secondary piping are being constructed in the remote maintenance demonstration area to evaluate the remote maintenance problems for these components. A personnel bridge over the mockups will be used to simulate the work area used in a semidirect maintenance.

The 1/12-scale model of the primary reactor system is about 20% complete.

Development and test work on manipulator-operated disconnects for the auxiliary piping and off-gas lines is continuing. The test on a modified integral ring-joint 3/4-in. flange has indicated promise for use where inert-gas buffering is required. A 3/4-in. metallic seal Pushomatic-type disconnect with leak detection provisions is being procured for test.

Pump Development. - The test of the pump having a molten-salt-lubricated journal bearing with fuel No. 130 at 1225°F continued; 5940 hr of operation has been accumulated. The test of the PKP pump with beryllium salt (BULT-4-1U) at 1225°F continued; 2200 hr of operation has been accumulated.

Head, flow, speed, and power data have been obtained in the water test of the MSRE primary pump. The time required to reduce the concentration of dissolved CO₂ in water by a factor of 2 was measured for several different gas stripping configurations. Fabrication of the hot test MSRE primary pump was started, and the mechanical, electrical, and instrumentation design of the hot test loop is near completion. The contracts for casting the impellers and volutes for the primary and secondary pumps in INOR-8 were approved; delivery is anticipated by early April 1961.

Temperature distributions in the primary pump tank have been calculated for the following cases: uninsulated wall with and without internal heat generation, insulated wall with and without heat generation, and forced-convection air-cooling with internal heat generation.

Calculations for the first case indicated a value of 4900 psi for thermal stress at the junction of the pump tank sphere and cylinder in the gas region. Subsequent study revealed that the moments between the sphere and the cylinder, which had been neglected in the thermal stress calculation, must be considered. Resolution of this problem is being pursued in cooperation with the stress analysts.

As a result of the Division Radiation Officer's review of the back-diffusion experiment utilizing Kr⁸⁵, the equipment is being modified to provide additional safety.

Reactor Analysis. - The estimated shielding required to reduce the dose outside the radiator pit to tolerance was 2 ft of ordinary concrete; the radiation sources for this dose were the decay products from the $F^{19}(n,\gamma)F^{20}$ and $F^{19}(n,\alpha)N^{16}$ reactions. Activations of the INOR-8 in the radiator pit, resulting from (γ,n) reactions in beryllium, produced a dose of 1 mr/hr at a distance of 1 ft from the center of the secondary piping.

The dose received by the lubricating oil in the secondary pump was estimated to be 2×10^7 rads in one year if the reactor is operated at 10 Mw. This estimate is a factor of 5 below the lower limit of the dose range in which oil damage has been observed.

A number of one-dimensional multigroup calculations were done to determine the effect on critical mass of dividing the core into three zones having equal areas but different fuel volume fractions. For a uniform core 28 in. in radius and 63 in. high, with 24% fuel volume, the estimated core critical mass was 12.9 kg of U²³⁵ and the system inventory (assuming 40 ft³ external volume) was 46.6 kg. When the three core regions had fuel volume fractions of 0.25, 0.13, and 0.04 (from inside to outside), the estimated core critical mass was 10.6 kg and the system inventory was 44.9 kg. The smallest core critical mass for the volume fractions investigated was 8.6 kg; however, since the associated volume fractions corresponded to a large ratio of external to internal fuel volume, the system inventory was 59.5 kg for this case.

Heat Transfer and Physical Properties. - Heat transfer studies with the BULT-14 mixture (LiF-BeF₂-UF₄-ThF₄, 67-18.5-0.5-14 mole %) have been discontinued after a total exposure of approximately 7100 hr. During this time no significant change in the heat transfer coefficient was observed; re-evaluation of the data in view of compositional changes is continuing.

Continued analysis of the preliminary measurements on the viscosity of the fuel mixture LiF-BeF₂-ZrF₄-ThF₄-UF₄ (70-23-5-1-1 mole %) indicates that, while the over-all results scatter widely, kinematic viscosities based on the lower limits of the three individual sets of data show reasonable agreement. Thus, at 650°C the data range from 2.05 to 3.15 centistokes, and at 800°C from 1.2 to 1.8 centistokes.

Measurements have been completed on the enthalpy and heat of fusion of the MSRE fuel mixture (composition as listed above) over the temperature range from 100 to 800°C. For the solid (100 to 430°C), the enthalpy (in calories per gram) can be expressed as

$$H_t - H_{30} = -0.155 + 0.145t + (3.63 \times 10^{-4})t^2 ,$$

and for the liquid (475 to 800°C), as

$$H_t - H_{30} = -23.4 + 0.572t - (8.99 \times 10^{-5})t^2 .$$

The heat of fusion at 450°C was 77.0 cal/g.

Assembly of the apparatus for determining the density and surface tension of the MSRE fuel mixture is proceeding.

MARITIME REACTORS PROGRAM

Experiment No. 1 fuel clusters were visually examined in the ORR hot-cell facility. The specimens were observed to have a reasonably adherent, uniform, black film. No evidence of damage was noted. The flux monitor wires were removed for radioanalysis. The fuel clusters were cut from the experiment support structure and, after being photographed, were canned in preparation for shipment to the G-E Vallecitos facility, where they will be destructively examined.

Experiment No. 3 fuel clusters were inserted into the pressurized-water loop for further irradiation.

ANP PROGRAM

Shielding Research. - Assembly of the new Tower Shielding Reactor (TSR-II) at the Tower Shielding Facility was nearly completed. The control turret was connected with the system, and operation at a few watts demonstrated that the controls worked in a satisfactory manner. The main water system was operated with gradual increases in flow rate, and various leaks were rectified until satisfactory operation was achieved at 400 gpm. The full flow condition of 700 gpm at 30 psig has not yet been reached.

The silicon-diode neutron spectrometer was calibrated with neutrons from the reaction of deuterium with beryllium, and the spectrometer provided a reasonable reproduction of the known beryllium spectrum. In the light of this test and previously reported results, it has been demonstrated that the silicon-diode neutron spectrometer is suitable for taking spectral measurements around a bare reactor where there are not many neutrons with energies higher than 8 Mev.

Experiments were performed at the Lid Tank Shielding Facility to obtain data on the penetration of radiation through various arrangements of lead, steel, lithium hydride, and boral. Approximately 50 configurations were measured, with particular attention being paid to the intensity and the cause of the gamma transmission. Provisions were made to follow the production of secondary gamma rays by distributing gold, indium, and molybdenum foils throughout many of the configurations in order to determine the low-energy neutron flux distribution. These penetration data will be used by NDA for comparison with results obtained by moments-method calculations.

The development was completed of gamma-ray and neutron calculation codes based on kernel methods for calculating the leakage from the TSR-II reactor and the Pratt and Whitney shield mockup. These codes will be used in conjunction with the Convair code, which deals with air scattering, and the TRG code, which deals with the penetration of radiation through slabs

of crew-compartment material. An additional kernel type of code was developed for calculating the neutron and gamma-ray doses received in a crew compartment from sources in a reactor, including the effect of air scattering. This last code is not particularly accurate, but it is useful for checking the magnitude of results from other calculations.

Columbium Alloy Research. - A study was made of the aging of electron-beam welds in heat XM-339 of a Cb-1% Zr alloy. The aging effects at 1500, 1600, and 1700°F were shown to be less severe in these low-contamination welds than in tungsten-arc welds made in the XM-339 material.

The first of a series of in-pile experimental assemblies containing tube-burst-type specimens of a Cb-1% Zr alloy was inserted in the ORR poolside facility. The equipment was designed for tests at 1800 to 2000°F with helium pressures up to 5000 psi. It was determined that the over-all assembly was leak-tight, that several columbium-zirconium tubes contained weak welds, and that the heaters would burn out when providing the heat flux necessary to maintain the desired high temperature. The required heat flux was higher than anticipated, either because of overestimation of the gamma heating or underestimation of the radiative cooling. Improved tube welds and heavier-duty heaters were therefore incorporated in the experiment assembly under construction.

Liquid-Metal Technology. - Since oxidation during ground operation will be a problem to be circumvented in space power plants, a series of alloys being considered for the containment of boiling potassium were subjected to oxidation tests at 1800°F for 100 hr in air. The weight changes due to oxidation and spalling are listed below:

<u>Alloy</u>	<u>Weight Change (mg/in.²)</u>	<u>Thickness of Oxide Layer (mils)</u>
Type 310 stainless steel	+2.1	0.5
Type 316 stainless steel	-197	5
Type 318 stainless steel	-227	4
Type 446 stainless steel	+5.8	0.5
Haynes alloy No. 25	+3.7	0.5
Haynes alloy No. 26	+6.4	0.5

Oxide stringers penetrated to a depth of 2 mils in both the Haynes alloys.

Beryllium Oxide Research. - The porous collector method for determining eutectic temperatures and compositions was used to continue the phase studies of the binary systems MgO-BeO, CeO₂-BeO, and ThO₂-Al₂O₃. The eutectic temperature for CeO₂-BeO was shown to be 1900 ± 20°C.

Studies were made of sections from two BeO-CaO samples that had been polished and etched. The samples were prepared by heating mixtures of BeO-CaO (60-40 mole %) and BeO-CaO (80-20 mole %) to approximately 1550°C in air and furnace-cooling them. The sample containing 80 mole % BeO consisted of well-developed BeO grains in a matrix of two different phases. One of the matrix phases was water soluble and appeared to be free calcia;

the other phase was insoluble in water and was not positively identified. The sample containing 60 mole % BeO contained no primary BeO, but it did contain the "matrix phases" observed in the sample with 80 mole % BeO.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Release of Fission Products on In-Pile Melting of Reactor Fuels. - In previous reports (ORNL-2945, p 9; ORNL-2980, p 10; ORNL-3038, p 14) a brief description has been given for the design of an experiment for studying the release of fission products resulting from the in-pile melting of fuel-element specimens. This is a part of the AEC experimental program for reactor fuel-element catastrophe studies.

The reactor facility, at the ORR, for the first of these experiments, which is to melt UO_2 in helium atmosphere, is 95% complete. The facility consists of an entry tube leading into the lattice, a hydraulic lift for positioning the experiment in the lattice, a liquid-nitrogen-cooled charcoal trap for collecting the rare gases released during meltdown, and the control units such as position controls, a position indicator, cooling-water flowmeters, and temperature recorders.

Initial use of the facility will be to measure the vertical gamma heating profile for the F-9 lattice position. The heat calculations and the design for this apparatus are completed and have been approved by the Operations Division. The equipment is almost finished and is expected to go into the ORR during the present cycle. In addition to the heating power information, this experiment will provide a thorough checkout for the irradiation facility, including the controls and the reaction of the experimental unit to the positioning mechanism, since the construction of the gamma device is externally identical to that of the meltdown equipment. The test is to be carried out several times over the period of the reactor cycle.

Radiation Damage: Advanced Engineering and Development. - The post-annealing status of the stored energy of the ORNL Graphite Reactor has been evaluated by testing more than 100 core samples taken from 38 channels. The results of the calorimetric measurements clearly show that the success of the annealing operation is closely related to the maximum temperature profiles reached during the anneal.

The stored energy in a 15-ft-diam core region was reduced to a level where a spontaneous release is now not possible. The peripheral regions of the fuel zone, including the outer three rows, did not reach sufficiently high temperatures for annealing, primarily because of sharply dropping thermal gradients at the edges of the moderator.

Between 3.5 and 4.5 Mwhr of stored energy was released during the annealing operation. This was achieved with a maximum fuel-element temperature of $275^{\circ}C$ and a maximum graphite temperature of $236^{\circ}C$, which emphasizes the importance of the low-temperature method.

Power Reactor Fuel Processing: Processing of Beryllium-Containing Fuel. - In dissolution tests on Hastelloy X GCRE fuel cladding in boiling $2\ M\ HNO_3$ - $4\ M\ HCl$, about 5 moles of hydrogen ion, 1 mole of nitrate, and 1 mole of chloride are consumed for each mole (59 g) of alloy dissolved.

The uranium can be completely leached from the fuel pellets (70% UO₂-30% BeO) in 5 hr with boiling 6 to 13 M nitric acid.

In 24 hr, 97.8% of the uranium was leached from an MGCR fuel pellet (61% UO₂-39% BeO) with boiling 8 M HNO₃.

Power Reactor Fuel Processing: Dissolution of ThO₂-UO₂ Fuel. - In dissolution tests on high-density Universal Match Company ThO₂-UO₂ pellets in boiling 15.8 M HNO₃-0.1 M Al(NO₃)₃-0.04 M NaF, the study of operation with a mixed oxide heel was continued. A 660-g batch of ThO₂-UO₂ was 95.5% dissolved in 31.5 hr; then a second 660 g was added and allowed to react for 32 hr; a cumulative heel of 6.15% remained.

Titanium-45A showed a maximum corrosion rate of 0.05 mil/month over 100 Thorex batch buildowns.

Power Reactor Fuel Processing: Mechanical Processing. - Additional shear encasement washing tests were made with the 1/4-scale wooden shear model. Talc was used to simulate UO₂ fines. The results indicate that 23 spray nozzles with a capacity of about 9.5 gpm at 40 psi, as compared with the previously reported 15 gpm, should be sufficient to wash and decontaminate the interior of the shear.

In tests of the mechanical operability of the inclined rotary drum leacher, discharge of short stainless steel rods (1/2 in. OD and 1 in. long) was satisfactory but occurred in spasmodic batches of one to ten pieces when the leacher was batch-charged (approx 14 lb of rods per batch) and operated in an oscillating motion [for 10 sec (50°) in the clockwise direction and 9 sec (45°) in the counterclockwise direction]. One batch charge of rods was discharged from the leacher for each revolution of the drum.

Preliminary compaction studies indicated that compaction or flattening of leached tubular fuel sections may not be economically attractive.

A review of the possible hazards of a runaway reaction during the dissolution of aluminum in nitric acid indicated there would be no dangers in using pieces of chopped aluminum-clad UO₂ for test work at acid strengths less than 10 M (50 wt %) HNO₃. Dissolutions of aluminum tubing in boiling 4, 6, 8, and 10 M nitric acid contained in glassware showed a uniform penetration rate of 2.6 mils/hr. Similar tests with the same acid strengths but with uranyl nitrate solution contained in stainless steel beakers showed a nearly uniform penetration rate of 1.6 mils/hr. The result indicates that a large amount of UO₂ pellets in 35-mil-wall aluminum tubing, now available at ORNL, could be chopped into 1-in. lengths and used for leaching tests in the inclined drum rotary leacher.

Power Reactor Fuel Processing: Solvent Extraction Studies. - Acid Thorex solvent extraction experiments in mini-mixer equipment were made with feed solutions containing as much as 5×10^8 counts min⁻¹ ml⁻¹ of gross gamma activity; decontamination factors were 10⁴, 5 × 10³, and 2 × 10⁵ from ruthenium gamma, zirconium-niobium gamma, and rare-earth beta, respectively. Owing to the limited number of extraction stages available in this equipment, the thorium loss was about 7%. No conditions have been found which will result in as high decontamination factors or avoid the formation of a second organic phase if the Acid Thorex flowsheet is modified so that the salting acid is added at the feed plate instead of lower in the extraction section.

Ninety-five per cent of the niobium may be removed from 10 M HNO_3 containing zirconium and niobium by passage through a column of unfired Vycor.

Power Reactor Fuel Processing: Corrosion Studies. - In Purex evaporator tests the maximum corrosion rate for titanium was 0.02 mil/month (504 hr), as compared with 3.55 for type 347 stainless steel (294 hr). The stainless steel suffered severe intergranular attack. Titanium-45A corroded at maximum rates of 1.4 and 3.3 mils/month in boiling 6 M HNO_3 -0.05 M H_2SO_4 and 15 M HNO_3 -1 M H_2SO_4 , respectively, over periods of 1000 and 360 hr. Some localized attack was observed in the heat-affected zone near the weldments.

Hastelloy F and Hastelloy C each showed corrosion rates of 0.4 mil/month or less in scouting tests for a multipurpose component material for Darex, Zirflex, and Sulfex decladding solutions at 35°C. Titanium-45A showed rates as high as 6 mils/month in Zirflex tests, and Carpenter 20 and LCNA (essentially the same as low-carbon Ni-o-nel) showed accelerated attack in the Darex solution with rates as high as 6.7 and 57 mils/month, respectively, for 506 hr exposure.

Power Reactor Fuel Processing: Processing of Graphitized Fuel. - More than 99% of the thorium was recovered from ungraphitized Th-U fuels by grinding to -10 mesh and leaching with 13 M HNO_3 -0.04 M NaF -0.1 M $\text{Al}(\text{NO}_3)_3$. Less than 2% of the thorium was recovered when 15.8 M HNO_3 was used as the leachant.

For graphitized Th-U fuels, maximum thorium recoveries of 90 to 93% were obtained by simultaneous disintegration and leaching with 90% (21 M) HNO_3 in two successive 4-hr exposures.

Treatment and Disposal of Radioactive Wastes: Low-Activity Wastes. -

ORNL process water was "spiked" with 5 mc each of Cs^{137} , Cd^{141} , and Sr^{85} , made alkaline with 0.01 M NaOH , allowed to flocculate and settle, and then filtered. The supernatant solution was passed through a 1- by 2-in. column of Duolite C-3 sulfonic-phenolic resin. Cesium appeared in the effluent after passage of 900 bed volumes, and the activity reached 1% of that in the feed after about 1600 bed volumes had been passed through. With mixed fission products the activity of the effluent remained relatively constant at about 2% (principally ruthenium) until breakthrough was reached at about 1890 bed volumes. Application of the process to HRT low-activity waste water gave effluent activities varying from 0.1 to 0.5% as the number of bed volumes of effluent increased from 0 to 1958. Drying of the solids flocculated and filtered from ORNL process water gave a product with a density of 1.18 g/ml.

An integrated laboratory-scale system was built and operated to demonstrate the feasibility of continuously decontaminating ORNL process water wastes by neutralizing to pH 11.7, flocculating the solids, filtering, and passing the filtrate through a cation exchange resin. A unit is being built to demonstrate this process on the ORNL waste stream itself.

Treatment and Disposal of Radioactive Wastes: High-Activity Wastes. -

Heating synthetic Purex waste to 900°C without additives to suppress sulfate volatility gave 34.2, 13.2, and 53.8% of the sulfate in the residue, condensate, and scrubbers respectively. These results indicate decomposition of bisulfates to H_2SO_4 and metal sulfates, with subsequent decomposition of all sulfates except Na_2SO_4 to oxides and SO_3 . Heating to 500°C

with 1.2 moles of NaOH and 0.2 mole of MgO per liter gave 37.0, 23.5, 40.9, and 1.4% of the ruthenium in the residue, equipment washings, condensate, and scrubbers respectively. The fraction of total ruthenium in the condensate was approximately 1/50 the fraction of total nitrate in the condensate at any given time until the final high-temperature stage of calcination, when the proportion of volatile ruthenium increased sharply. The ruthenium balance was 99.4% in each of two batch evaporation-calcinations to 900°C with NaOH and MgO added to prevent sulfate volatility, indicating that most of the ruthenium plating in the overhead system occurred in the de-entrainment section rather than in the condenser and evaporator flask. When NO was added to the calciner feed stream during semicontinuous evaporation and calcination, 17.6% of the ruthenium was found in the condensate, as compared with 31.8 and 45.6% when NO was introduced into the vapor space of the calciner pot. No net inert off-gas was produced, and about 0.43 mole of NO was consumed per liter of waste solution.

The thermal conductivity of the solids obtained by heating Purex waste to 900°C with NaOH and MgO added, as determined by the steady-state technique, varied almost linearly with temperature from 0.125 Btu hr⁻¹ ft⁻¹ (°F)⁻¹ at 300°F to 0.360 at 1350°F.

The operability of a continuous evaporator in-line with a pot calciner is under study by analog computer simulation and with a 50-liter-capacity (2 liters/min boilup rate) experimental stainless steel evaporator. It was computed that, after 3 hr run time, the flow rates in a production unit would reach maximum values of 0.44, 2.62, and 3.06 gal/min for the waste stream to the evaporator, the water strip stream to the evaporator, and the evaporator overhead stream respectively. The length of time for the experimental evaporator to reach steady-state conditions with various raw feeds varied from 13 to 24 min.

Waste Disposal Research and Engineering: Geochemical Studies. - Grundite, a commercially available clay mineral, is used to treat process waste water at ORNL. This material is suspected of being an interstratified montmorillonite mica, and previous studies have shown that montmorillonites exhibit an increase in cesium selectivity when heated to temperatures inducing irreversible collapse of the c axis. Thus it was felt that heating would be beneficial to grundite if it contained montmorillonite. Heating grundite to 500, 600, or 700°C did result in improved cesium selectivity. After heating to 600°C the cesium capacity was 1.7 times that of the unheated material. At 500 and 600°C, heating for 3 hr did not affect the initial rate or the final capacity of the material for cesium; at 700°C, heating for longer than 1 hr reduced the initial rate of sorption, but the total capacity remained constant. Studies also show that more efficient use of grundite can be made by allowing longer contact time or by re-using material which has been used previously for one application. Comparative studies with different materials heated to 600°C show that Wyoming bentonite (montmorillonite) is slightly better than grundite. These materials are especially valuable for waste disposal applications, since the cesium is "fixed" in the hexagonal network of oxygen atoms in the crystal and is thereby resistant to leaching.

Waste Disposal Research and Engineering: Disposal in Natural Salt Formations. - Temperatures in the neutralized waste cavity of the field

experiments at Hutchinson, Kansas, are falling very slowly, and the average waste temperature is now approximately 70°C.

Two of the four small field experiments started in September are still in operation. Difficulties with heaters forced the temporary termination of the slender-cylinder experiment. The experiment designed to measure the effect of several heat sources on the temperature of a single adjacent point has been completed. The temperature rise was considerably greater than would be obtained by summing the contributions from each of the sources considered separately.

Waste Disposal Research and Engineering: Disposal of ORNL Radioactive Wastes. - In selecting a new solid-waste disposal site (Burial Ground No. 5), items considered included topography, soil character, surface and ground water conditions, accessibility, and location with respect to the Laboratory and the White Oak Creek drainage basin; also, the area must have sufficient extent to meet the needs of disposal for about 5 yr.

A 25-acre site was selected in Melton Valley after a preliminary field reconnaissance. The site is located within 1/2 mile of the Laboratory in the White Oak Creek drainage basin. From geologic studies it was found that the principal types of rock underlying the area are shale, siltstone, and limestone. In areas of higher elevation, weathered shale is found at depths up to 35 to 40 ft, whereas in low topography fresh rock is found within a few feet of the surface. Water-level measurements taken in auger wells were used to prepare depth-to-water and water-table contour maps. Five deep wells were drilled to determine the occurrence and circulation of ground water at greater depth. Pressure tests of these wells showed that the most permeable zones or fractures occur within the first 100 ft. Hydrographs for the wells showed a maximum water-level fluctuation of 14 ft and a minimum fluctuation of 1.5 ft over a period of approximately eight months. A procedure for solid-waste burial was recommended based upon these findings. A new trench, designed to improve monitoring and limit the downward percolation of rainfall, was evaluated.

Waste Disposal Research and Engineering: Process Waste Water and Treatment Plant Studies. - In addition to the normal dosage of lime, soda ash, and clay, various amounts of trisodium phosphate were added to process waste. Laboratory studies showed an optimum removal of 98% of the strontium at 125 ppm PO_4^{---} . While a similar efficiency was demonstrated when only phosphate was added (100 ppm excess PO_4^{---}), pH control of the solution with NaOH was necessary. Although the settling characteristics of the flocculent material formed in both cases require improvement, the lime-soda ash-clay-phosphate system has demonstrated superior qualities. During a one-week plant trial of phosphate feed, suspended solids in the effluent increased and thus confirmed the need for improved solids control. A combination of coagulant aids, Hagan No. 2 and Hagan No. 323, improves considerably the rate of settling of the suspended solids. Appropriate plant trials to test the effect of coagulant aids have been scheduled.

Waste Disposal Research and Engineering: Clinch River Studies. - A program of water sampling and water analysis for determining radionuclide and mineral concentrations in the Clinch and Tennessee Rivers downstream from Oak Ridge is now in full operation. Water samples are collected from (1) the Clinch River at the Oak Ridge water plant, (2) the Clinch River

near Center's Ferry, (3) the Tennessee River at Loudon, (4) the Tennessee River at Watts Bar Dam, and (5) the Tennessee River at Chickamauga Dam. Subsamples collected each day, the volumes of which are proportional according to predicted mean flows for the week and the daily actual flows, are composited weekly (Sunday through Saturday). The weekly composites are then thoroughly mixed to resuspend the settled sediment and split into two portions. A 5-gal portion is sent to the U.S. Public Health Service (RATSEC) at Cincinnati for a scan of the gamma spectra and a determination of the Sr^{90} concentration in the water and in the sediment. A 1-gal portion is expressed to the Tennessee Stream Pollution Control laboratory in Nashville for mineral analysis.

Quarterly reports will be prepared to show a continuous record (by weeks) of the concentration, and the total quantity, of radioactivity and minerals in the water and in the sediments at each of the sampling stations.

Waste Disposal Research and Engineering: Long-Range Evaluation of Over-All Waste Complex. - A review of factors affecting hazards of tank storage of radioactive wastes is now being completed.

An attempt is being made to define a simple "hazards index" for systems containing radioactive materials. A compartmental model expressing contact of escaped radionuclides with man gives an equation so unwieldy as to be useless for this purpose. Possible simplifications, and other models, are being investigated.

PHYSICAL RESEARCH PROGRAM

PHYSICS AND MATHEMATICS RESEARCH

High-Energy Physics. - Interactions of K^- mesons at rest in helium were searched for events that could be interpreted as the production of a Λ hyperon, a π^- , and He^3 . All events in which the decay of a Λ was visible and which had either a single π^- or a π^- and a short heavily ionizing track were analyzed kinematically and checked for coplanarity. Twenty-one candidates were found, of which only six could be interpreted as $\Lambda\pi^-He^3$ events.

Scintillation Spectrometry and Instrument Development. - The decay of I^{132} has been investigated by means of scintillation spectrometers. Energies (and relative intensities) of the gamma rays that were observed are 0.240 (1.3), 0.518 (15), 0.667 (100), 0.72 (5), 0.775 (63), 0.953 (15), 1.14 (1.2), 1.142 (2.7), 1.30 (2.4), 1.392 (6.4), 1.45 (1.1), 1.75 (0.3), 1.91 (0.7), 1.99 (0.3), 2.08 (0.18), 2.18 (0.13), and 2.39 (0.11) Mev. The data indicate that the gamma-ray peak at 0.667 Mev actually represents four gamma rays with energies between 0.62 and 0.68 Mev. An energy-level diagram of Xe^{132} based on the present spectral studies and gamma-gamma angular correlation measurements has been proposed. Energies (and spins) of the levels are 0.673 (2^+), 1.32, 1.448 (4^+), 1.81, 1.966 (3), 2.10 (3, 4, or 5), 2.401 (4 or 5), 2.59 (3 or 5), and 2.84 (3, 4, or 5) Mev.

High-Voltage Experimental Program: Total Cross Section of Pb^{208} . - The total neutron cross section of Pb^{208} has been determined with 3 kev resolution in the 1.4 to 1.9 Mev energy range. The measurements were made by the standard transmission technique; the neutron source was the $Li(p,n)$ reaction produced by protons from the 5.5-Mev Van de Graaff accelerator. The sample was in the form of a 116-g cylinder, 1-1/8 in. in diameter, of 99.75% pure Pb^{208} . Neutrons were detected with a stilbene crystal from which pulses due to gamma rays were depressed by means of the Brooks-Owen technique. With the bias settings used, 1.9-Mev neutrons were counted with about 10% efficiency, which was 3000 times the efficiency for counting 1.3-Mev gamma rays. Corrections to the data for in-scattering were calculated by use of published differential cross sections of normal lead, and preliminary corrections for the second group of $Li(p,n)$ neutrons were made by use of previous total cross section measurements of Pb^{208} obtained with 10 kev resolution. In the energy interval between 1.4 and 1.9 Mev, at least 24 levels were observed. These had peak cross sections corresponding to J values in the range 1/2 to 7/2. The widths of the levels varied from less than 3 kev to about 100 kev.

High-Voltage Experimental Program: Neutrons from the He^3 Bombardment of Some Light Nuclei. - The excitation functions for the reactions $Be^9(He^3,n)C^{11}$ and $Li^7(He^3,n)B^9$ have been studied at 0 and 90° for He^3 bombarding energies from 1.0 to 2.5 Mev. The reaction $C^{13}(He^3,n)O^{15}$ has

been studied at 0° for the same bombarding energy range. Angular distributions for the first two reactions have been measured at a bombarding energy of 2.1 Mev. The ground state and the first five excited states of C^{11} were observed. All the neutron groups were clearly resolved except those associated with excitation of the fourth and fifth excited states of C^{11} at 6.50 and 6.77 Mev. These two groups were resolved graphically. The ground state of B^9 was clearly resolved from the first excited state at 2.37 Mev, but the first and second excited states were unresolved. The three-body continuum was observed for all neutrons with energies not exceeding that for the ground state. At higher bombarding energies the three-body continuum becomes more prominent relative to the B^9 ground state. The spectra from the reaction $C^{13}(He^3, n)O^{15}$ show the ground state and the third excited state (6.15 Mev) clearly resolved, but the first and second excited states at 5.20 and 5.25 Mev were unresolved.

Heavy-Particle Physics. - The differential cross section for the reaction $B^{10}(N^{14}, O^{15})Be^9$ was measured; a peak in the angular distribution was observed at about 30° c.m. corresponding to a value of $r_0 = 2$ fermis in the classical tunneling theory. Measurements on the angular distribution of N^{13} from $N^{14} + B^{10}$ were initiated. Again a peak in the forward direction is observed. Inelastic scattering of N^{14} from the first state in Mg^{24} was measured from 70 to 100° in the center-of-mass system. The results are being analyzed.

The 86-Inch Cyclotron: Nuclear Physics. - Two surface-barrier counters made on thin silicon wafers were obtained from the Instrumentation and Controls Division for testing. One counter was made on an 0.008-in. wafer and the other on a 0.003-in. wafer. Each in turn was used in place of the gas proportional counter of the dE/dx -E counter telescope. Both counters performed very well and were found to have several advantages over the proportional counter. Their small thickness permits construction of a telescope in which multiple scattering in the dE/dx counter cannot cause particles to scatter out of the telescope. The observed long-term gain stability has been excellent over periods of a week. The silicon counters can tolerate at least ten times the counting rate of the proportional counter without noticeable drift in gain. The 0.003-in. counter has been in routine service for measuring (p,d) and (p,t) energy spectra for about two weeks. Data taking was much more efficient due to the shorter setup time resulting from the gain stability and to increased counting rates permitted by the short recovery time of the counter. The counter apparently will operate satisfactorily at rates of 10^4 counts/sec, perhaps even higher.

An attempt was made to evaluate a technique for measuring total reaction cross section for protons by placing the target material between a silicon dE/dx counter and a sodium iodide E counter. It was found that a two-coordinate record of the coincident pulses is required to obtain unambiguous results. In the absence of a two-coordinate pulse-height analyzer, transistor circuits were built to be used with other available equipment to permit recording in four dE/dx "slices" simultaneously.

Examination of the (p,d) spectra from iron isotopes and cobalt shows a systematic shifting of the p and f levels from isotope to isotope.

Comparison with Nilsson calculations for single-particle levels in deformed nuclei suggests that Fe^{54} and Co^{59} are spherical, while Fe^{56} , Fe^{57} , and Fe^{58} have prolate distortions. This conclusion is based on taking the Nilsson diagrams at face value.

The 86-Inch Cyclotron: Applied Physics. - The isotopes Be^7 , Na^{22} , Mn^{52} , Co^{57} , Co^{61} , As^{74} , $\text{Y}^{87,88}$, Pd^{103} , Cd^{109} , $\text{I}^{124,125,126}$, Ce^{139} , Lu^{170} , and other isotopes were produced during 128 hr of service irradiations for customers, including three universities, two radioisotope processors, two hospitals, the National Aeronautics and Space Administration, the United Kingdom Atomic Energy Authority, and three ORNL divisions. A beam current of $0.01 \mu\text{a}$ was used to simulate the radiation damage of satellite components; $2600 \mu\text{a}$ was used in the production of Cd^{109} in a solid silver target. Window-type target irradiations using a degraded beam are utilized to produce Co^{57} in millicurie quantities. Instead of using only aluminum foils to degrade the beam energy, the standard practice now is to use extremely high-purity, low-cobalt nickel foils along with the aluminum foils; Co^{57} is thus produced in the foremost nickel foil at a rate of approximately 1 mc/hr. This by-product from the irradiation of various targets can be stockpiled.

Electronuclear Machines. - The Cyclotron Analogue II, an eight-sector spiral-pole electron cyclotron, will model the beam dynamics of the proposed 850-Mev fixed-frequency proton cyclotron. The designs for the sector coils to provide the azimuthally varying field for focusing and the trimming coils to provide for isochronous operation were completed and orders placed. The vacuum tank, pumping system, and radio-frequency system are complete. Installation of the machine has begun; the remaining components are expected in February, and initial operation is expected in March or early April.

The Isochronous Cyclotron. - The ORIC building (Building 6000) is 96% complete; only some painting, installation of laboratory furniture, and other detail work remain. A schedule has been prepared for the installation of cyclotron components to begin in January.

The 19 power supplies for the various magnet coils were received, or are in transit; the regulators are being designed and tested. A new regulator for the 1750-kva motor generator was delivered and will be used first in connection with the model magnet work.

The rf drive line which couples to the dee was fabricated and installed for testing in the full-scale rf model. The final amplifier tube, RCA 6949, was received. Laboratory-scale tests of the oscillator, the offset oscillator, and the frequency regulator systems are being made. The console and the control board are being fabricated by a contractor.

The design of the valley coils, harmonic coils, and trimming coils was completed, and the valley coils are being fabricated. Major components remaining to be designed are the dee, the dee-stem transition, the tank liner, and the deflector system. The delivery of the magnet base, the pole tips, and other components is expected well ahead of the assembly schedules.

Stable Isotope Development. - Chlorine separations have received considerable attention in an effort to provide isotopic purities that have heretofore been unobtainable. Two new receiver designs were employed which place maximum emphasis on high-purity collections. A separation was also made in which NaCl was used as the charge material. It was thought that the NaCl⁺ side band might be more easily collected than the elusive chlorine ions. However, operational difficulties were experienced, and this approach has not as yet been successful.

As part of the program to provide Ca⁴⁶ at higher isotopic purities, certain receiver entrance slots were decreased in length from 5-1/2 in. to 3-1/2 in. on an experimental basis. It had been noticed that the amount of contaminant in a pocket appeared to be dependent on the area of the opening. If this conclusion is correct, then the receiver modification may reduce contaminants by 40% while not substantially reducing the amount of collected Ca⁴⁶.

The feasibility of using the direct combination of iridium with chlorine in a quartz charge oven in a calutron ion source has been tested. The rate of reaction proved insufficient to provide satisfactory ion currents. Modifications to the source are planned which may remedy this situation. This technique may prove more successful when applied to separations of platinum and palladium, since these elements react more readily with chlorine.

A detailed theoretical investigation of the applicability of an inhomogeneous magnetic focusing system in the beta calutrons has been completed. The results indicate that better focus, greater mass dispersion, and higher transmission should be possible in an inhomogeneous system. A beta tank is being modified to achieve such a magnetic field.

Significant improvements have been made in the refinement of the isotopes of chlorine and cadmium. Pure chlorine can be recovered quantitatively from silver chloride by hydrogen reduction followed by trapping of the resulting HCl in NaOH solution. Preliminary investigation indicates that cadmium can be quantitatively and quickly separated from large amounts of copper by adsorbing the cadmium chloride complex on an anion exchange resin.

Special Separations. - Decontamination of the outmoded plutonium processing equipment has continued. Difficulties encountered with the radiation monitor system have been corrected.

Decontamination of the old uranium area to be stripped for the plutonium laboratory has continued. The uranium storage area in the basement has been cleared, old showers removed, and needed equipment decontaminated and rearranged.

CHEMISTRY RESEARCH

Thorium Oxide Studies. - The black vitreous material formed (see ORNL-3038, p 23) on irradiation of a 1750°C-fired thoria slurry was examined. It was 1 wt % of the total solids and was composed of > 76% volatile matter and contained only 0.23 wt % Th, 0.07 wt % Fe, and 0.007 wt % U. Its density was 1.18 g/cc. Qualitative indications are that the material is carbonaceous and that despite the 1750°C firing the pellets contained carbon.

Recalibration of the gas injection equipment used in the gas recombination catalyst development showed that there is a significant holdup of gas in the capillary tubing connecting the gas charging system to the reaction autoclave containing the slurry-catalyst system. This holdup under certain conditions may amount to as much as 80% of the gas charged and has led to several erroneous conclusions as to the activity and properties of the palladium catalyst being used in the gas recombination work. Re-evaluation of the gas recombination data indicates that, with slurries containing the sol-prepared palladium catalyst, there is probably a first-order dependence of rate on the hydrogen partial pressure and 0.5-order dependence on the oxygen partial pressure; there is little or no deactivation of the catalyst under excess oxygen; the apparent threshold partial pressure for hydrogen to react in the presence of excess oxygen is not real, and reaction is obtained even at low hydrogen partial pressures; recombination of the oxygen and hydrogen in the reaction vessel is complete, and the apparent stoppage of reaction when significant partial pressures of both hydrogen and oxygen were present in the reaction vessel is not real; the radical increase in specific catalyst activity when excess hydrogen is present, while real, may be consistent with the kinetics of the recombination process and does not necessarily indicate a change in the nature of the catalyst as previously surmised; and the solubilities of both oxygen and hydrogen in the slurry catalyst system are consistent with those predicted from the Henry's law constants obtained at both BMI and ORNL.

Radiation Chemistry of Organic Moderators. - A supply of purified biphenyl sufficient for the present study has been received. A chromatogram of the material showed no peaks other than air, solvent (tetrahydrofuran), and biphenyl.

A vacuum line has been set up for handling the organic materials after pyrolysis. The line was designed to allow separation of the organic products into several categories dependent on boiling points: (1) permanent gases and low-boiling hydrocarbons, (2) intermediate products with boiling points ranging from room temperature to 260°C, and (3) high-boiling polymeric products.

Gas and vapor chromatographic equipment is being obtained to facilitate the analysis of the organic decomposition products. The gases and low-boiling compounds will be analyzed on a dimethylsulfolane column in series with a Linde 5A molecular-sieve column at 0 and 25°C respectively. Intermediate-boiling compounds are to be analyzed on a didecyl phthalate or silicone-rubber column. The polymeric products are to be analyzed on a molten-salt column which is useful up to 500°C. A eutectic of lithium, potassium, and sodium nitrates supported on firebrick makes up the high-temperature column. All analyses above room temperature are to be performed on an F and M model 500 chromatograph, and the very-high-boiling materials will be analyzed by using temperature programming. Retention times for the expected products are available, and the use of internal standards will be held to a minimum.

Analytical Chemistry Research. - In connection with a spectral study of fluoride solutions of the 3d transition elements in molten alkali fluorides, spectra of cobaltous fluoride in molten rubidium fluoride and in

molten cesium fluoride were recorded over the temperature range 775 to 900°C. A specially designed compartmented lid was fabricated for the high-temperature cell assembly so that these very hygroscopic salts could be transferred from a dry box to the cell assembly without exposure to the atmosphere. Although the general shapes of the absorbance peaks for Co(II) in molten LiF, RbF, and CsF are similar, a significant shift of these absorbance maxima to longer wavelengths is observed as the size of the alkali-metal cations is increased. The data are being evaluated in terms of closest-neighbor interactions in order to gain a better understanding of the nature of the molten fluoride salt solutions.

An investigation of the analytical possibilities of absorbance measurements in molten borax is under consideration since this salt has excellent solvent properties for many metal salts and oxides. At the present time it is necessary to confine these salts as pendent drops for spectral study since no window material has been found which is inert to molten borax. Spectra for CoCl_2 , CoF_2 , NiCl_2 , and NiF_2 in borax have been obtained over the temperature range 25 to 900°C. In the case of the nickelous ion, a single absorbance peak was found which became somewhat less intense and shifted to longer wavelengths with increasing temperature. Approximately 60 ppm of nickel in a borax bead, 1-cm path length, would yield an absorbance of 0.1 at the wavelength of maximum absorbance. Significant differences in the wavelengths of absorbance maxima were noted in comparing the spectra of CoCl_2 and CoF_2 in borax. Approximately 30 ppm of cobalt in a borax bead, 1-cm path length, however, is required to yield an absorbance of 0.1 at the wavelength of maximum absorbance.

High-Temperature and Structural Chemistry. - The electrical conductivity was found to increase much more slowly with metal concentration in solutions of calcium in molten calcium chloride than in solutions of the alkali metals in their halides and of cerium in cerium trichloride. The conductivity of the saturated solution (2.8 ± 0.2 mole % Ca at 855°C) was found to be only 12 to 15% higher than that of the pure calcium chloride.

Transuranium Elements. - Ion exchange and solvent extraction methods of separating actinide and lanthanide elements were continued along with separation processes for transplutonium elements. In the ion exchange method the effects of LiNO_3 loading vs $\text{Al}(\text{NO}_3)_3$ loading, resin particle size, and column temperature were investigated. More column gassing and greater pressure drop occurred with $\text{Al}(\text{NO}_3)_3$ than with LiNO_3 . Iron, however, was more readily removed in the aluminum (99% vs 70%) than in the lithium system. Temperature appeared to be important. At 85°C, cerium removal in seven column volumes of 10 M LiCl was > 99.9%, compared with 93.5% at 65°C. The resin particle size appreciably affected the separation, since 100 to 200 mesh resin gave > 99.9% cerium removal with seven column volumes of 10 M LiCl at 85°C, as compared with 92.1% removal for 50 to 100 mesh resin. In seven column volumes of 10 M LiCl no americium should be removed.

A liquid-liquid solvent extraction system was found which appears similar to the solid-liquid anion exchange system. It involves selective extraction of the actinide elements into tertiary amines from concentrated LiCl solutions. Separation factors of approximately 100 between americium and europium were readily obtained when 30% triisooctylamine in

xylene was used to extract from 10 to 12 M LiCl containing 0.1 M AlCl₃. The order of extractability with tertiary amines was Cf > Am \approx Cm \gg Eu > Ce \approx Y. The distribution coefficients were directly proportional to the square of the organic concentration and inversely proportional to the square of the HCl concentration. The distribution coefficients were also proportional to approximately the 17 power of the LiCl concentration, with an americium coefficient of about 1 at 10 M.

METALLURGY AND MATERIALS RESEARCH

Preparation of Pure Single Crystals. - A crystal of LiF was grown in a nickel capsule (0.025 in. wall thickness) under a helium atmosphere. The LiF separated cleanly from the nickel without rupturing the capsule. The crystal is colorless, but strained, and has a refractive index of 1.392 ± 0.001 in sodium D light. There is no selective infrared absorption. The infrared and ultraviolet cutoff values are approximately 6.5 and 0.12 μ respectively, but the crystal does transmit a few per cent of the incident radiation at 8.0 and 0.11 μ .

Very pure Li⁷F has been prepared from Li⁷OH·H₂O for use as a starting material to grow an Li⁷F crystal.

Crystal Physics of Ceramics. - Crystals of magnetite, Fe₃O₄, have been synthesized hydrothermally through an oxidation-reduction reaction involving low-carbon steel and initially 0.5 N NaOH at temperatures near 420°C and estimated pressures of about 4500 psi. The largest crystal prepared thus far was almost 3 mm on an edge and is as large as any reported to have been grown hydrothermally. A better-quality crystal, having well-defined facets, with edge lengths of about 1.5 mm, grew at the rate of at least 0.09 mm/day in the direction of the cube axis. This growth rate is at least twice that reported in the literature.

High-Temperature Reactions in Metals and Ceramics. - Spectroscopic studies of dilute solutions of bismuth metal in molten BiCl₃ have revealed that the solute bismuth is partitioned into more than one species. The data are consistent with a two-species model. Studies of the Bi-BiBr₃ system have progressed far enough to show that partitioning occurs here also but that the equilibrium is shifted quite far in favor of the chromophore up to a concentration of 0.7 M. Various quantitative spectroscopic parameters have been evaluated for both systems.

Fundamental Investigation of Radiation Damage in Solids: Irradiation Effects on Alloys. - Electrical resistivity measurements on solid-solution alloys of aluminum in copper, near the composition 15 at. % aluminum, are being made as a function of neutron, electron, and gamma irradiation. It is found that a decrease in resistivity of about 2% takes place for each type of irradiation at 100°C. However, if the irradiation is carried out at temperatures near that of liquid nitrogen, no decrease in resistivity is observed, although the decrease will set in upon subsequent warming above about -50°C. The decrease in resistivity is believed to be due to an atomic rearrangement, probably corresponding to increased short-range order, that takes place in the course of the motion of radiation-produced defects. Two kinds of electron-irradiation experiments are particularly

useful in providing information concerning the details of the defect production and motion. In the first of these, electrons of various energies are used to bombard the alloy at constant flux and temperature, from which a measure of the cross section for the production of displacements is obtained as a function of electron energy. Secondly, the electron flux is varied while the energy and the temperature are kept constant, providing information concerning the processes by which the radiation-produced defects are diffused and annihilated in the alloy. The rate of the decrease in resistivity is observed to increase as the square root of the flux, which suggests that the annihilation of defects occurs, at least partly, as a result of the recombination of vacancy-interstitial pairs.

Fundamental Investigation of Radiation Damage in Solids: Minority Carrier Lifetime Studies of the Annealing of Radiation Damage in Germanium. - The nature of gamma-ray-induced damage is expected to be simpler than that for neutron bombardment, and since the recombination process is better understood in n- than in p-type material, efforts have been concentrated on n-type germanium irradiated with Co^{60} gamma rays. Isochronal anneals were performed on antimony-doped samples with six different impurity concentrations, arsenic-doped samples with three different impurity concentrations, and one p-type indium-doped sample. The anneals were for 1 hr each at up to 11 different temperatures, equally spaced on the $1/T$ scale. The important feature displayed by the annealing curves for antimony-doped material is that the annealing behavior depends markedly on impurity concentration (as determined from conductivity) throughout the resistivity range. The results for arsenic-doped material are entirely different. There was an early conductivity-dependent anneal ranging from about 15 to about 60% in the three samples used. At higher temperatures the annealing curves for the two lower resistivities (higher impurity concentration) very nearly superimpose when normalized. The annealing behavior of high-resistivity arsenic-doped material is rather similar to that for high-resistivity antimony-doped material. This might be expected in these higher-resistivity samples, since the impurity concentration is rather low and might, therefore, play a less important role in the annealing process.

CONTROLLED THERMONUCLEAR RESEARCH

Arc Research. - Initial assembly of the long solenoid was completed in late September. Since that time, operation has been for the purpose of studying lithium arcs.

Lithium arcs have been run with the cathode at three different positions in the tank: first, with the cathode at the end, giving an arc length of 120 in.; second, with the cathode mounted six windows from the anode end to obtain an arc approximately 40 in. long; and third, in the eighth window from the anode to obtain an arc approximately 52 in. long. No particular difficulty was encountered in operating with the cathodes inserted in the windows. By using this arrangement it was possible to observe the back-streaming of the arc. Preliminary data with the mass analyzer looking at the back-streaming showed Li^+ only. The radiometer gave the same reading with and without a quartz window when looking at

the back-streaming; this is consistent with the mass spectrometer data. The back-streaming was green, and only a small amount of red due to excited Li^0 was seen around the cathode.

Several anodes have been tried with the arc as a heat source. The most successful to date has been a water-cooled copper cup with two 1/4-in. tungsten disks in the bottom and a tungsten cylinder for a nose.

The magnet coils have been connected so that 11 small coils in the center are energized by generator A and the rest of the coils by generator B. This makes it possible to have a uniform field, a mirror field, or a reverse mirror field.

During operation several things concerning lithium arcs have been observed. The voltage between electrodes is determined by the anode configuration. The voltage is lower when there is good heat transfer to the lithium and high when there is poor transfer to the lithium. For an arc current of 100 amp, the voltage has run as low as 80 v and as high as 250 v for the same arc length. An anode where the lithium is heated by a separate source is under construction.

The cathodes have been 1/2-in. tantalum tubing. Lithium is condensed inside the cathode at the cool end next to the holder. If a floating water-cooled baffle is placed around the cathode, lithium is condensed on this baffle also. The lithium stream has approximately the same diameter as the hole in the anode end.

There is a red glow due to excited neutral lithium at both the anode and the cathode end. The window opposite the hot end of the cathode does not become covered nearly as rapidly as the window opposite the baffle, which is near the other end of the cathode. This indicates that Li^0 will not go through one wall of the arc without being ionized.

Mass analyzer data show that the arc contains Li^+ and Li^{++} . The ratio varies considerably during a run. On at least two occasions the Li^{++} peak was higher than the Li^+ . The variation in the ratio is due to changes in the anode or to heating of the nose of the probe, or both. A hydrogen peak is often observed near the end of a run; this is probably due to some compound of lithium being vaporized. Excited states of Li^{++} have not been observed spectroscopically.

Radiometer data show that the radiation from the arc decreases as the vacuum gets better. Also, placing a quartz window between the arc and the radiometer decreases the reading by more than 90%.

Future work will be directed toward obtaining an anode which can be run for longer periods of time and obtaining control of the lithium pressure so that more precise data can be obtained.

DCX-1 Facility. - The DCX vacuum system has been modified to permit three-stage differential pumping. Evaporative titanium pumping has been installed in the plasma region. Titanium pumps and well-trapped diffusion pumps are used for an intermediate region, and four 32-in. diffusion pumps are used for pumping in the outer region. Preliminary tests indicate that a static base pressure of 3×10^{-8} mm Hg is readily obtainable in the plasma region. Hydrogen pumping speeds of 60,000 liters/sec at 10^{-7} mm Hg have been measured.

The ion source on the DCX accelerator has been replaced by one designed and constructed by the High Voltage Engineering Corporation. The ion beam emerging from the ion source is passed through a magnetic

analyzer. At the entrance to the accelerator tube, H_2^+ currents of 28 ma have been obtained. The beam has a diameter of approximately 2 cm and a divergence of less than 1° . The source has been installed, and the parameters of the accelerated beam are being studied (600 kv acceleration).

Larger Magnetic Facility. - The design for the DCX-2 is about 5/8 complete.

Trajectories of molecular ion input beams have been traced stepwise through the region where reflection takes place. It has been found that for H_3^+ injection with a 15-in.-diam orbit, sufficient precession of the orbit may be obtained to cause the beam to miss the injector on its return path. Means of controlling the field variation at large radii, responsible for this precession, are being studied.

Vacuum. - Tests of metal film pumping systems have been run with titanium, molybdenum, and chromium. The greatest pumping speeds observed so far for the three metals are 7000, 2000, and 650 liters/sec respectively.

Five of the porcelain electrical insulators, 28 in. in diameter and 12 in. high, for an accelerator tube have been leak-tested. The greatest leak rate measured was 10^{-10} liter/sec (of helium at standard conditions).

BIOLOGY AND MEDICINE PROGRAM

BIOMEDICAL PROBLEMS IN ATOMIC ENERGY OPERATIONS

Physics of Tissue Damage. - Preliminary thermoluminescence spectra of three amino acids, phenylalanine, tryptophan, and tyrosine; the enzyme trypsin; and spores of Bacillus megaterium have been obtained with filters. The samples were irradiated in the dry state at liquid-nitrogen temperature ($77^\circ K$) with Co^{60} gamma rays. Each sample was exposed to approximately 6×10^5 r. The samples were warmed up at $12^\circ C/min$, and the emitted light was analyzed by interposing filters between the sample and a photomultiplier. The spectral resolution was about 250 A.

The spectra of all the materials are rather broad; this is only partly due to the low resolution from the filters and the system of data analysis employed. The relative spectral composition was found to remain approximately constant for the major peaks of the glow curves. After the samples were warmed beyond the major peak of the glow curve, all the spectra shifted toward longer wavelengths.

Since the analysis has not been completed at this time, the following peak wavelength values (in angstroms) are only approximations: phenylalanine, 4875; spores, 4625; trypsin, 4625; tryptophan, 5625; tyrosine, 5375.

Radiation Physics and Dosimetry. - The instability resulting from the interaction of a beam of charged particles with a plasma has been studied by Akhiezer and Fainberg under the assumption that $\theta = 0$, where

θ is the angle formed by the beam and the direction of the growing wave. Under these conditions the interaction is electrostatic. In this investigation the assumption is generalized so as to include $\theta \neq 0$ and the effect of electromagnetic interaction. For $\omega \approx \omega_1$, where ω_1 is the Langmuir frequency of the plasma, the interaction is electrostatic for all values of θ , and the resulting instability, which produced a longitudinal wave, increases exponentially in accordance with the term

$$\exp (3 \sqrt{3} \omega_0^2 k v_0 \cos \theta / 8)^{1/3} t ,$$

where ω_0 is the Langmuir frequency of the beam. For a frequency range below ω_1 the instability is less pronounced. However, this instability is significant, since the interaction is electromagnetic and the resulting "growing wave" is characterized by an electric vector having both transverse and longitudinal components. An assumption was made that the density of the incident beam is small, and the results cover all values of θ except those close to $\pi/2$. For $\theta \approx \pi/2$ the assumption is more general; thus the results apply to any density of the beam.

Internal Dose Estimation. - A maximum permissible concentration in air has been determined for insoluble Ra^{226} compounds on the basis of the lung as the organ of reference. In calculating this $(\text{MPC})_a$ it is necessary to assume a value for the retention of Ra^{226} daughters in the tissue, because the effective energy per disintegration depends on the retention.

In experimental studies with dogs and from the data obtained on human beings who have long-term skeletally stored radium, a value of 30% has been obtained for the retention. If this value is assumed to apply to the case of lung-stored Ra^{226} , the effective absorbed energy per disintegration is 110 Mev and the $(\text{MPC})_a$ is 5×10^{-11} $\mu\text{c}/\text{cc}$ (for a 40-hr week). This might be considered to be a conservative value, because in bone the remodeling process and the resulting formation of new bone crystals produce conditions favoring a diffusion lock of radon, thereby increasing the retention. Actually, at one day after injection the retention is about 4%, and it takes on the value of 30% only after a considerable period of time (about 20 yr). If the $(\text{MPC})_a$ based on lung is calculated on the assumption of zero retention, then the effective energy is 49 Mev and the corresponding $(\text{MPC})_a$ is 1×10^{-10} $\mu\text{c}/\text{cc}$.

Ecology and Marine Studies. - The rate at which crayfish, an important link in aquatic food chains, accumulate Co^{60} from solution follows the first-order rate equation. For an experimental system in which a solution containing Co^{60} at a concentration of 2.52×10^3 disintegrations $\text{min}^{-1} \text{ml}^{-1}$ was pumped over crayfish continuously, the rate constants were 0.238 and 0.252 per day for groups of crayfish with average weights of 0.9 and 6.4 g respectively.

Excretion rates were determined by placing radioactive crayfish in clean spring water and counting them at daily intervals. The first-order rate constants for excretion were 0.019 and 0.010 per day for groups of crayfish with average weights of 0.87 and 5.02 g respectively. Experiments including dead crayfish showed that about 50% of the uptake was due to adsorption on the exoskeleton. The rapidity of this adsorption

accounts for the higher rate of uptake by large crayfish. The higher rate of excretion by small crayfish is due to the greater metabolic rate of these individuals, which results in rapid elimination of metabolically available cobalt. Low and equal levels of Co^{60} were found in the blood, muscle, and gonads, whereas greater concentrations were found in the gut, hepatopancreas, and exoskeleton. The exoskeleton contained 95% of the body burden of Co^{60} .

NUCLEAR ENERGY CIVIL EFFECTS

Dosimetry Applications. - An extended source field has been designed and constructed at the Nevada test site for standardization of air-borne radiation detection equipment. The sources (Co^{60} or Cs^{137}) may be placed in holders set at 100-ft intervals in a square array measuring 2000 ft on an edge. The central 100-ft square is further segmented so that 100 sources having 1/100 of the activity of the usual source can be placed on 10-ft centers. The field may be readied for exposures to Co^{60} or Cs^{137} within a matter of hours; point sources are also available for comparison. Several laboratories collaborated in the initial experiments with the radiation field and air-borne scintillation spectrometers and dosimeters. Measurements were successfully carried out at heights of from 100 to 1000 ft.

A dual-detector neutron spectrometer has been constructed for use in coexistent neutron and gamma radiation fields. Two anthracene crystals (1 mm thick) are mounted in a vacuum facing each other and separated by about five times their diameter. The two preamplifier outputs are connected to their respective DD-2 amplifiers, the outputs of which are used in a coincidence and gating circuit for a multichannel analyzer. An A-8 amplifier is connected to a mixing circuit between the outputs of the preamplifiers, and the signals from the A-8 are fed through a delay line to the multichannel analyzer.

ISOTOPE DEVELOPMENT PROGRAM

Radioisotope Research and Development. - The results of the multi-kilocurie Sr^{90} purification run in the solvent extraction equipment at the Fission Product Pilot Plant indicated that several changes should be made to the flowsheet that was tested. The difficulties that were encountered were due to the presence of gross quantities of extraneous non-extractable ions and to iron and chromium. The nonextractable ions were either Na^+ or NH_4^+ which was not completely removed from the calcium and strontium carbonate centrifuged cake prior to the acetic acid dissolution. The presence of sodium or ammonium acetate in the aqueous stream buffered the pH to a relatively high value. Since the quantity of unextracted ion was not known, control of the cascade was lost. Even if the concentration of Na^+ or NH_4^+ were known, control would be difficult. The iron and chromium produced a precipitate which interfered with the operation of the equipment.

Suggested flowsheet changes for future runs are as follows: (1) The mixer-settler feed will be prepared by extracting the strontium and calcium into a solvent consisting of di-2-ethylhexylphosphoric acid (D2EHPA) in Amsco. This organic extract will then serve as a feed stream to the mixer-settlers. With this feed preparation step, essentially all the Na^+ and NH_4^+ can be removed along with some of the Ba^{++} . (2) A 0.05 M HNO_3 -D2EHPA system will be used in the mixer-settlers. The aqueous stream will be unbuffered. This is possible because the bulk of the feed (calcium) is extractable and is already in the organic phase when it enters the cascade; thus it produces only small pH changes near the feed stage. The strontium and barium which are removed in the aqueous phase are not present in sufficient quantities to upset the cascade operation. (3) The BaCrO_4 precipitation step, which contributed excess chromium to the feed, will be performed after the solvent extraction process rather than before the extraction step.

Tracer-scale tests of the organic feed-0.05 M HNO_3 system which are now in progress indicate that the continuous flow system is much more stable in regard to flow rate variations than the acetic acid system. A process efficiency of more than 95% is also indicated. The remaining experimental work is principally the investigation of the feed preparation process conditions.

Radioisotope and Stable Isotope Production. - Charge materials were prepared for the separation of the isotopes of Ga, K, Zn, Cl, Fe, Ca, and Ni. Dysprosium chloride and samarium chloride have been prepared for isotope separations, although collections have not yet been started. The chemical recovery of osmium that was used as charge material in the recent isotope separation has been continued in an attempt to obtain 300 to 400 g of very pure osmium metal for cyclotron use. The following have been provided for cyclotron use: Eu_2O_3 , 370 g; Re metal, 375 g; Ir metal, 375 g; and Ga_2O_3 , 432 g. Collections of the isotopes of Ca, Pb, Fe, Ga, Ni, Cd, Zn, and K were made with ion currents equivalent to 2704 g. Separations were performed in the four calutrons in Building 9731 and in 16 of the calutrons in Building 9204-3.

Fourteen new lots of the separated isotopes of Pb and Os and six returned lots of Cl, Lu, Rb, and Tl were recovered, refined, and prepared for inventory. The following table summarizes the quantity and quality of the osmium isotopes released to inventory:

<u>Osmium Isotope</u>	<u>Lot No.</u>	<u>Amount (g)</u>	<u>Mass Assay (at. %)</u>
186	JS 1176 (a)	0.758	61.27
187	JS 1177 (a)	1.051	45.76
188	JS 1178 (a)	5.451	87.7
189	JS 1179 (a)	6.665	87.3
190	JS 1180 (a)	9.501	95.46
192	JS 1181 (a)	15.790	98.68

Sixty-seven samples of Cd, Ca, Cl, Ga, Fe, Pb, and Ni were recovered from individual collector pockets of graphite or copper in order to obtain development data on retention, mass analysis, sputtering, etc.

Thirteen inventory items were converted to more suitable compounds or to the element.

One sample of Hg^{199} metal and one sample of $\text{Hg}^{202}(\text{NO}_3)_2$ were sealed in Pyrex ampoules for shipment. A small sample (0.250 g) of $\text{Ca}^{40}\text{CO}_3$ was returned and repurified to remove a small activity due to Zn^{65} and Hg^{203} formed during a neutron irradiation.

The conversion of 8 kg of $\text{Pb}^{206}\text{Cl}_2$ to Pb in the form of a cylinder to be loaned to Los Alamos was the most extensive special conversion service. As the lead was of radiogenic origin and contained an appreciable quantity of Pb^{210} , it was necessary to carry out the operation under controlled conditions.

Eighteen kilograms of a depleted uranium batch has been purified and converted to oxide. The remainder of the batch is in process. Four Pu^{239} batches have been combined and purified. The following shipments were made:

<u>Element</u>	<u>Number of Shipments</u>
Uranium	12
Plutonium	1
Neptunium	6
Thorium	2

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