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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

AUGUST 1960

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

AUGUST 1960

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

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

August 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

Dissolver Solution Analyses: Uranium. – Radioactive and nonradioactive solutions, similar to those to be found in power reactor fuel reprocessing, were analyzed for uranium. The procedure consisted in separating the uranium from the dissolver solution by means of triisooctylamine in xylene, stripping the uranium from the organic layer with acid, and reducing the uranium(VI) quantitatively by use of a controlled-potential coulometer. The results obtained indicate that power reactor fuel dissolver solutions can be analyzed for uranium to within ±1%. Complete details and data will be contained in ORNL-2987. Work is in progress to increase the sensitivity of the uranium determination so that accurate results may be obtained in the microgram range.

A method has been developed by which uranium may be determined coulometrically in solutions containing as much as 10 parts of molybdenum per hundred parts of uranium. Molybdenum is not coulometrically reduced in a basic medium; therefore a solution 0.06 M in sodium sulfate and 0.06 M in sodium tripolyphosphate at pH 8.5 is used as the electrolyte. Studies are now in progress to determine the precision and accuracy of this method.

Dissolver Solution Analyses: Plutonium. – The controlled-potential coulometric titration of plutonium has been described in earlier reports. The performance of that method has been tested in the High Radiation Level Analytical Facility by remotely analyzing six highly radioactive uranyl nitrate solutions (derived from declad uranium metal rod) for plutonium content. The results exhibited a relative standard deviation of 0.5% under those conditions and recovery was 99.3%. The results compared favorably with those obtained by TTA extraction and counting techniques.

A study was made of the determination of plutonium by carrying Pu(IV) on a precipitate of LaF_3, dissolving the precipitate in $Al(NO_3)_3$, and titrating the plutonium coulometrically in the resulting solution. This technique can be useful when the concentration of plutonium is very low or when a rapid separation is needed. The relative standard deviation was 0.3% and the average recovery was 99.8% when 256 μg of plutonium was determined by this procedure.

[redacted]

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROJECT

EGCR Control Studies. – The nominal fuel element surface temperatures and gas temperatures have been determined for another method of control-rod programming for the EGCR. In this case it was assumed that the central control rod was fully inserted and that the remaining rods operated as a bank. The depth of insertion of the bank was 62 in., giving a Δk value of 2.5% for the bank. It was found that the maximum nominal surface temperature of the fuel elements for this case was about 1400°F. This is 100°F above the value for the case of the central rod fully inserted and the inner ring of four rods one-half inserted. It is estimated that the maximum local temperature would be between 1600 and 1700°F. Based on the latest concept for controlling the reactor for a wide range of experimental conditions, the case discussed above probably gives the maximum fuel element temperatures.

Tests of a Simulated EGCR Fuel Element at Elevated Temperatures. – A simulated fuel element, 27-1/2 in. long, was subjected to an external pressure of 320 psi while being held for 32 hr at an average central UO_2 temperature of 2350°F and an average cladding temperature of 1420°F. The pressure within the capsule varied from 3 to 5 psi during the test. After approximately 30 hr the cladding had collapsed sufficiently to make contact with the UO_2. The capsule was then thermally cycled between the temperature limits of 400 and 2350°F for a total of 109 cycles. The expansion characteristics of the capsule did not change during the 109 cycles. The total permanent strain introduced in the cladding measured 0.45%. Gaps formed between the pellets during the thermal cycling. However, the gaps were very small (on the order of 0.020 in.) and were not present at each pellet interface throughout the capsule.

After examination the capsule was reheated and held at an average cladding temperature of 1475°F for 114 hr. The central UO_2 temperature during this period was 2750°F. After 65 hr at these temperature conditions, the cladding had collapsed completely, and the profile of the cladding followed very closely that of the pellets. The capsule was then thermally cycled an additional 48 cycles. The dimensional characteristics of the capsule changed markedly during this thermal cycling program. The largest single gap measured was 0.040 in. The total permanent strain introduced in the cladding, measured axially, was 2.5%.

The capsule was again placed in the experimental apparatus and held for 330 hr at an average cladding temperature of 1550°F and a central UO_2 temperature of 2870°F. No general collapse of the cladding into the voids between the pellets was noted.

Expansion of Dished UO_2 Pellets. – The expansion behavior of pellets dished on only one end and that of pellets dished on both ends have been compared. The average dishing was approximately 0.003 in. Within experimental error, it was found that the dimensional behavior of the double-dished pellets was the same as that of the pellets dished on only one end.

Experimental Tube-Buckling Studies. — Four 12-in.-long tubular specimens of type 304 stainless steel (0.750 in. OD, 0.025 in. wall) are being tested at 1200°F in argon with an external pressure of 400 psi. The test was interrupted at 508 and 1252 hr for specimen examination. No evidence of collapse was found, and the test is being continued.

A second, identical set of specimens is being tested at 1200°F in argon with a 600-psi external pressure. The test was interrupted at 475 hr, and no evidence of collapse was found; therefore, the test has been resumed.

A single specimen (12 in. long, 0.750 in. OD, 0.025 in. wall) was heated to 1200°F in argon, and the pressure was increased until collapse occurred. The results indicate that instantaneous collapse occurs at 800 psi at this temperature. This test will be repeated for verification of the result.

Reactions of Type 304 Stainless Steel with Atmospheres Containing CO and CO_2. — Sixteen tubular specimens of type 304 stainless steel were exposed to a CO_2-CO atmosphere in the temperature range from 941 to 1800°F for a period of 703 hr. The gas composition during the test was maintained at 0.1339% CO_2 and 0.301% CO, with the balance helium. Oxidation of the steel specimens ranged from 1.5 mg/cm^2 at 1800°F to 0.03 mg/cm^2 at 941°F. Oxidation appears to be of a superficial nature in this atmosphere at temperatures below 1300°F.

Removal of Fission Gases from Coolant. — A study was made of the effect of moisture in the carrier gas, in this case air, on the dynamic adsorption of krypton by Columbia HCC activated carbon, 12/28X. The air velocity employed was 0.07 fpm, the pressure was 1 atm, and the charcoal was at 25°C. After prolonged exposure of the charcoal to air having relative humidities estimated to be 51 and 59%, k values of 29 and 16 cm^3/g and N values of 20 and 18 ft^{-1}, respectively, were obtained. The k value is a measure of retention time, and N is the number of theoretical plates in an adsorber. These data illustrate the deleterious effect of moisture on adsorption efficiency and indicate that moist air carrying the fission gases krypton and xenon should be dried, if possible, before passing through an adsorber designed for removal of these fission gases.

Determinations of k and N with helium as the carrier gas were also made for PCB activated carbon, 12/30 mesh, with the helium at 1 atm and a velocity of 1.6 fpm and with the charcoal at both 25 and 0°C. The k values for the two temperatures were 52 and 122 cm^3/g and the N values were 58 and 65 ft^{-1}, respectively. These values are not much different from those obtained previously for HCC activated carbon under the same conditions; so it is expected that the two materials will behave similarly at the temperatures and helium pressures of actual interest.

Decontamination Studies: Noncorrosive Solvent for Massive UO_2. — In the search for a noncorrosive solvent suitable for dissolving broken UO_2 pellets in the charge and service machines of the EGCR, solutions of oxalate salts mixed with hydrogen peroxide were tested. For maximum UO_2 solubility the pH of the solution is controlled at a very slight acidity and the corrosion rates on mild steel are acceptably low. Two 39-g EGCR-type UO_2 slugs have been dissolved, each in 500 ml of 4% sodium oxalate—3% H_2O_2 at 95°C. At a pH of 4.0 to 4.5, one slug was 73% dissolved in 3 hr and totally dissolved in 7-1/2 hr. The corrosion rate on mild steel was 0.0069 mil/hr and

on type 347 stainless steel 0.0004 mil/hr. The second slug was 70% dissolved in 3 hr at a pH of 5.0 and, after solution renewal, was totally dissolved in 5 hr. Mild steel corrosion in this run was 0.0025 mil/hr, as compared with 0.0005 mil/hr on type 347 stainless steel. The reagents were fortified periodically with small amounts of peroxide and oxalate. The pH spontaneously rises to above 7, if not controlled by occasional addition of oxalic acid, and rapid H_2O_2 decomposition sets in. The lifetime of the oxalate-peroxide reagent is expected to be at least 20 min at 90 to 95°C, which is far superior to the 0.75-min lifetime of the carbonate-peroxide reagent at 90°C.

THERMAL-BREEDER REACTOR PROGRAM

Homogeneous Reactor Program

Homogeneous Reactor Test. — All planned HRT core-vessel modifications and repairs were completed in August. Both holes in the Zircaloy vessel were patched, and tests were conducted to measure the tightness of the patches. Leakage through the upper (second) hole, if any, was not measurable. Leakage through the lower (first) hole was such that minor upsets in the fuel-blanket pressure difference during operation will allow uranium to mix into the blanket region. Tests are planned to study problems connected with control of the pressure difference and the pressurizer level.

Preparations are now being made for a back flush of the core system to remove scraps that have resulted from the core modification and repairs.

HRT Chemical Pilot Plant. — The laboratory study of the proposed HRT fuel purification process by uranium peroxide precipitation was completed. The effects of temperature and iron concentration on peroxide decomposition rates were studied briefly; the decomposition rates vary directly with iron concentration in the range studied up to 1 g/liter, and the temperature dependence is typical of many previous studies at higher temperatures. Full-scale equipment is being assembled for testing at the reactor site.

Thorium Blanket Studies. — In the preparation of rounded thoria pellets by pressing ceramic-grade thoria powders and calcining, two types of troublesome cracking were defined and resolved. One type, in which the spherical section of the pellet separates from the cylindrical, was greatly decreased by increasing the radius of curvature of the punch face from 0.144 to 0.25 in., by increasing the powder prefiring temperature from 800 to 1200°C, and by increasing the Carbowax lubricant-binder to about 8%. Another type, top and side cracking, which radiates from the poles parallel to the axis of the pellet, is caused by strains induced in the pellet by shrinkage during sintering. These strains, and cracks due to them, were eliminated or greatly lessened both by a calcination schedule which included soaking for 1.5 hr at 1200°C before the temperature was raised to 1750°C and by regulation of the powder granulation pressure to give a pour density of 2.5 to 2.75 g/cc.

Rounded ThO_2 particles, 3 to 5 μ in diameter, were prepared by flame calcination of a thoria sol at 1500°C. The sol was prepared by treating 650°C-fired ThO_2 with nitric acid and dispersing in water at a concentration of 2 M.

Chemical Research for Aqueous Homogeneous Reactors. — The solubility of ThO_2 at 200°C in HNO_3-H_2O was determined for solutions having nitrate concentrations between 2.4 and 9.0 M. The mole ratios Th:NO_3^- at saturation were found to be 0.19 at 2.4 M NO_3^-, 0.22 at 4.7 M NO_3^-, and 0.21 at 9.0 M NO_3^-. These results indicate that in 2.4 M NO_3^- the thorium solubility at 200°C is four times as great as the previously reported (ORNL-2980) solubility at 300°C; in 9.0 M NO_3^- the solubilities at 200 and 300°C were about the same.

A study was made of the removal of tracer quantities of Pa^{233} by coprecipitation with thorium peroxide at 55 to 65°C from thorium nitrate solutions over a range of thorium concentrations from 1 to 3 M and a range of

nitrate-to-thorium ratios from 4:1 to 5:1. Over 95% of the protactinium was removed from solution when as much as 1% of the thorium was precipitated. The concentration of peroxide required for this precipitation increased with increasing acidity.

Slurry Circulation Experiments. — Run 22A, in the 200A loop, is being made for observation of the high-temperature dropout behavior of a slurry of 3.65-μ-dia thoria fired at 1600°C. The run was interrupted so that a valve could be replaced in the slurry charging system. The valve was repaired, and the run is being continued.

Inspection of the 300-SM slurry system following completion of a series of slurry circulation tests at temperatures up to 300°C revealed that the system was in excellent condition. The slurry heat exchanger showed no observable attack on the tubes or tube sheets. The ball valves, which had been closed against flowing slurry approximately 40 times during the final test period, showed slight wear; the valve seats were in good condition, but there was some detectable out-of-roundness of the balls. The trim material in these valves was aluminum oxide. The 300-SM system was placed in standby condition after the inspection, as no tests are planned for fiscal year 1961.

Engineering Development. — Detailed measurements of fluid residence time at various locations in the HRT core model with reversed flow were completed. The data indicate that when the HRT is operated at 5 Mw core power, the maximum wall temperature will be 301°C if the core and blanket inlet temperatures are 250°C. The maximum temperature of the fuel near the wall will be about 308°C under the same conditions.

A 5-in.-dia cylindrical core model with two longitudinal inlets extending the length of the model and with an axial outlet at the top of the vessel was tested for possible application as a replacement vessel for the HRT. Measurements of local fluid residence time showed that such a core would operate with the cylindrical walls at a temperature about midway between the inlet and outlet temperatures and with the top and bottom of the core at temperatures slightly above the outlet temperature. The design of the inlet nozzle is being modified to provide more flow of inlet fluid along the top and bottom of the vessel.

The HRT mockup was operated with water to investigate control of the system under conditions which simulate HRT operation with a high rate of solution transfer between the core and blanket regions.

Design. — A preliminary design was prepared for a Zircaloy-2 corrosion-specimen holder for insertion in the HRT. The design involves a Zircaloy-2 mast of Y section; the corrosion specimens, oriented in the direction of flow, would be welded to the mast. The assembly would be inserted in the central jet of the reversed-flow core, supported from the 2-in. access flange at the top and attached to the No. 6 screen at the bottom.

Design of a replacement pressure vessel for the HRT was initiated. Both re-entrant and straight-through core designs are being prepared. Layouts of the HRT high-pressure system modified to receive a replacement vessel were started.

MOLTEN-SALT REACTOR PROJECT

MSRE Design. – The core design for the MSRE is complete, and detail drawings are in preparation. This design calls for an unclad-graphite-moderated cylindrical core 54 in. in diameter and 66 in. high. The graphite lattice is constructed of 2 × 2 × 66 in. stringers banded with molybdenum to restrain any tendency to bow because of the neutron flux gradient in the core. Fuel passages of 0.200 × 1.20 in. cross section are machined between the graphite stringers, resulting in ~ 88% graphite and 12% fuel salt by volume.

The fuel, a mixture of LiF, BeF_2, ZrF_4, ThF_4, and UF_4 (70-23-5-1-1 mole %), makes two passes through the reactor. It enters an annular ring at the top, flows down an annular peripheral passage to cool the INOR-8 vessel wall, into a flow-reversing plenum, and then upward through the graphite lattice to an exit plenum. Fuel residence time in the active lattice (at the design flow, 1450 gpm) is ~ 3 sec.

Design of the MSRE primary heat exchanger is complete. This is a baffled-shell U-tube exchanger designed to transfer 10 Mw of heat from fuel salt to coolant salt (LiF-BeF_2). The unit contains 162 tubes, 0.500 in. in outside diameter and 11 ft 8 in. long, assembled with a triangular pitch on 0.812-in. centers. The fuel is in the shell side and the coolant flows through the tubes.

The primary-system layout, now essentially complete, is designed to reduce stresses from thermal expansion and to facilitate the removal and replacement of all components by use of remote handling and viewing equipment. The pump is mounted ~ 3 ft above the core on a common center line. Both are mounted rigidly in the horizontal plane but are free to move in the vertical plane to the extent of the thermal expansion in the 3 ft of connecting 8-in.-dia pipe. The heat exchanger, mounted horizontally at a level intermediate to the pump and reactor, and the 6-in.-dia connecting piping are pitched at 3° to permit drainage. The heat exchanger and piping are mounted to float in the horizontal plane to eliminate stresses due to thermal expansion. Coolant piping located in the reactor cell is sufficiently flexible to avoid overstressing the system.

The fuel drain tank is designed to hold the entire 55 ft^3 of the reactor fuel charge with an additional gas volume of ~ 5 ft^3. It is a cylindrical tank 48 in. in diameter and 83 in. high. The cylindrical wall is 0.500 in. thick and the heads are 0.750 in. thick. The tank incorporates a boiling-water afterheat removal system contained in 40 thimbles 2 in. in diameter extending downward into the tank. Inside the thimbles are bayonet tubes in which demineralized water is boiled at atmospheric pressure. The water entry tube is 0.375 in. OD × 0.045 in. wall × 74 in. long. The steam return line is made of 1-1/4-in. sched-40 pipe.

The tank will receive the fuel at 1200°F mean temperature approximately 15 min after reactor shutdown, at which time the afterheat generation is calculated to be less than 100 kw. The water feed rate for removal of 100 kw of heat is 300 lb/hr. The fuel temperature levels off at 1300°F approximately 3 hr after draining.

Progress was made in the design of the MSRE fuel pump. This is a vertically mounted sump-type pump capable of delivering 1450 gpm at a head of

75 ft of fluid. The performance characteristic curve has been established, and casting molds are available for the impeller and volute.

The pump has approximately 30 in. of overhang between the center of the impeller and the lower shaft bearing. Oil-lubricated ball bearings and rotating face seals are used. A constant flow of inert gas down the shaft to the pump expansion tank keeps the bearing and seal regions relatively free of radioactive fission gases.

The pump is driven by a 75-hp squirrel-cage motor with power supplied through a variable-frequency alternator which gives a wide range of pump speeds. The motor is completely enclosed and operates in an inert atmosphere. It is close-coupled to the pump, and both are hermetically sealed as a single unit; however, the seal can be remotely broken and re-established to permit replacement of the motor without disturbing the pump. The rotary element of the pump can be replaced remotely after the motor is removed.

Chemistry. – In a search for ways of alleviating the potential hazard arising from the precipitation of UO_2 in the MSRE fuel (nominal UF_4 content is 1 mole %) by accidental contamination with oxide, the effect of adding ZrF_4 as an oxide scavenger was explored. The initial results indicate that if there is about five times as much ZrF_4 as UF_4, ZrO_2 will be produced in preference to UO_2 when additions of oxide are made to the melt. Experiments to confirm in a quantitative manner the absence of UO_2 in association with the precipitated ZrO_2 have not been completed. These initial results on the scavenging action of ZrF_4 are presently being confirmed by a variety of approaches.

Metallurgy. – Radial-heat-flow apparatus was prepared for measuring the thermal conductivity of INOR-8 alloy between the temperatures of 100 and 1400°F. Initial results of measurements made at 680 and 736°F were 0.168 and 0.174 w cm^{-1} $(°C)^{-1}$ respectively; these results verify previous work to within 3%.

Tensile tests were performed to determine the effect of low creep strain on the strength and ductility of INOR-8. Rod specimens which had previously been subjected to creep strains at 1250°F showed negligible change in tensile and yield strength when tested at 1300°F. There was a loss in ductility in one specimen exposed for 7939 hr with 0.48% creep strain; the elongation was 36.5%, as compared with an initial value of 61%.

Two types of solidified metal seals were developed for use with molten fluorides at elevated temperatures; one contains an alloy sump with a tongue-and-groove joint design, and the other has an alloy-impregnated metal fiber compact. Components of both types were fabricated and were tested by breaking and forming the seals a number of times in an argon atmosphere and subsequently leak-checking with helium. Joints of these types containing gold-copper braze alloys were leak-tight.

Permeation tests of grades S-4 and AGOT graphite with $LiF-BeF_2-ThF_4-UF_4$ (67-18.5-14-0.5 mole %) at 1300°F at pressures of 25, 26, and 150 psig in 100-hr exposure periods indicated that (1) there were small differences in the permeation of these grades by salt for the different pressures used and (2) actual and theoretical salt permeations were essentially the same except for the permeation of grade S-4 at 25 psig. Grades S-4 and AGOT, respectively, are moderately low- and high-permeability grades of graphite.

Corrosion Tests. – The compositions of thin corrosion films found on several of the long-term INOR-8 corrosion loops were investigated by means of an electron-beam microprobe analyzer. The results of the analyses indicated an increase in molybdenum content and virtually complete depletion of chromium and iron as compared with the composition of the base metal. An analysis made by scanning along a cross section of a specimen containing a film approximately 1/4 mil thick indicated that the concentration gradient was limited to about 1/2 mil.

In-Pile Tests. – The hot-cell disassembly of experiments ORNL-MTR-47-1 and -47-2 was started at Battelle Memorial Institute during this period. Samples of each graphite specimen from these two experiments will be removed by core drilling and subjected to radiochemical analysis. The analytical data thus obtained will be utilized in predicting the behavior of rare-earth and other fission products in a graphite–fused-salt system under conditions simulating those that will occur in the MSRE.

Reactor Analysis. – Two-group, two-dimensional calculations were performed to obtain an estimate of the reactivity change introduced by going from isothermal startup to MSRE operating conditions. In going from a uniform temperature of 1200°F to mean inlet and outlet temperatures of 1175 and 1225°F respectively, the associated decrease in reactivity was found to be 5.0×10^{-4} $\delta k/k$ on the basis of criticality calculations. By first-order perturbation theory, the above decrease was calculated to be 3.9×10^{-4} $\delta k/k$.

Estimates of critical mass and total fuel-system inventory have been computed for graphite-moderated bare cylindrical reactors 4.5 ft in diameter and 5.5 ft high. For a fuel salt containing Be, Li, Zr, and U, the critical mass was about 9 kg of U^{235} for salt volume fractions between 0.08 and 0.16; the U^{235} inventory of the system decreased with increasing salt fraction. With 4 mole % thorium present in the fuel salt, the critical mass increased from about 16 kg of U^{235} at a salt volume fraction of 8% to about 35 kg at 16%.

Heat Transfer and Physical Properties. – Analyses of experimental data on the surface tensions of three $NaF-BeF_2$ mixtures were completed. The results over the temperature range 500 to 800°C can be represented by the following equations:

$$\text{for } NaF-BeF_2 \ (57.1\text{-}42.9 \text{ mole } \%),\ \sigma = 264.4 - 0.116t; \qquad (1)$$

$$\text{for } NaF-BeF_2 \ (57.5\text{-}42.5 \text{ mole } \%),\ \sigma = 269.4 - 0.136t; \qquad (2)$$

$$\text{for } NaF-BeF_2 \ (63.0\text{-}37.0 \text{ mole } \%),\ \sigma = 293.2 - 0.165t. \qquad (3)$$

In these equations, σ is in dynes/cm and t in degrees C. The 6% discrepancy between samples 1 and 2 may relate to differences in impurity content arising from differences in exposure time in the circulating loop from which the samples were drawn.

Corrections to the original data and the inclusion of more recent results have yielded a revised correlation of the mean heat capacity of BeF_2-containing salt mixtures. The new equation reads:

$$\bar{c}_p = 4.21(\overline{MN})^{-0.770} \quad ,$$

where $\bar{M}$ is the average molecular weight and $\bar{N}$ is the average number of atom species of the mixture.

Heat transfer studies with $LiF-BeF_2-UF_4-ThF_4$ (67-18.5-0.5-14 mole %) were interrupted after 5560 hr of operation so that the circulating pump could be replaced; damage appears to be restricted to the upper shaft bearing. Recent chemical analyses show that the composition of the salt differs somewhat from the nominal; a re-evaluation of the data using corrected thermal properties will be necessary.

Fuel Processing. — Thorium fluoride present in MSR fuels follows the rare earths in the HF dissolution process, ending up in the waste stream. Leaching this waste with SbF_5-HF solutions offers promise as a method for extracting the rare earths (the major poisons) from the ThF_4, thus permitting the ThF_4 to be recycled to the reactor. The LiF present reacts with SbF_5 in HF to form an insoluble compound, rare-earth fluorides form very soluble compounds, and ThF_4 neither reacts nor dissolves appreciably. Quantitative solubilities have not yet been determined.

MARITIME REACTORS PROGRAM

Four-group, two-dimensional calculations of the characteristics of the NMSR core I operating at 68°F have been completed. Previous calculations, although of the multigroup type, allowed for heterogenous flux variations only in the thermal group. The present calculations accounted for heterogenous flux variations in four energy groups, and the results are in much better agreement with the results of experiments at zero power.

Similar calculations of the characteristics of an NMSR core containing Zircaloy spacers instead of the present stainless steel spacers indicate that the Zircaloy spacers would increase the control-rod worth by some 5% in δk. The four-group method has been successfully applied to analyses of Marty critical experiments containing either stainless steel or aluminum spacers.

NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Release of Fission Products on In-Pile Melting of Reactor Fuels. — Design of the equipment necessary for in-pile melting of UO_2 specimens clad in stainless steel has been completed. Calculations of the temperature profile for the apparatus on insertion in the reactor have been completed. Adequate temperature control can apparently be obtained by control of the position of the experiment in the lattice. Melting temperatures of the UO_2 specimens can be reached when the assembly is inserted 2 in. into the lattice of the ORR. Information and drawings necessary for final approval of the experiments are about 75% completed. Fabrication of the reactor entry tube and of some of the ceramic pieces required has been completed.

Fuel Cycle Development. — It has been observed that UO_2 dissolves in molten ZrF_4 with the formation of ZrF_4-UF_4 solid solutions. The phases identified in cooled melts originally containing LiF-BeF_2-ZrF_4-UF_4-BeO indicate that the presence of ZrF_4 in such mixtures results in formation of ZrO_2 instead of UO_2 if the ZrF_4/UF_4 concentration ratio is 4 or greater. These data suggest that fuel elements containing uranium oxide may be effectively dissolved with a molten salt mixture containing ZrF_4.

Power Reactor Fuel Processing: ThO_2 Fuels. — The densities (g/ml) of sulfuric acid solutions in which the type 304 stainless steel cladding had been dissolved from ThO_2-UO_2 fuel (Sulfex process) were shown to fit the empirical equation

$$d = 1 - 4.31 \times 10^{-6}t^2 + 0.0275[H^+] + 0.00262[SS]$$

in the temperature (t) range 20 to 80°C for stainless steel concentrations [SS] of 0 to 40 g/liter and sulfuric acid concentrations $[H^+]$ of 0 to 10 N.

After exposure to 54 successive Thorex feed adjustment boildowns, titanium 45A showed negligible corrosion. In boiling Sulfex solutions, low-carbon Ni-o-nel showed maximum rates of 3.5 and 1.8 mils/month for the solution and vapor phases, respectively, in 6 M H_2SO_4, and 0.7 and 2.5 mils/month in 5.8 M H_2SO_4 containing 10 g of stainless steel per liter. Repeat exposures of type 304L stainless steel to Darex-Thorex waste solutions at 80°C for 600 hr resulted in intergranular attack. In 48-hr exposures in waste evaporator environments, low-carbon Ni-o-nel and Hastelloy F showed maximum rates of 0.8 and 1.2 mils/month respectively.

Power Reactor Fuel Processing: Uranium Carbide Fuels. — The composition of the explosive gases produced when UC reacts with water or acids is being determined as a prerequisite for devising safe operating methods. Uranium monocarbide reacted with distilled water at 90°C in a helium atmosphere to yield a gaseous mixture consisting of 12.5 vol % hydrogen, 84.0 vol % methane, 2.7 vol % ethane, and 0.7 vol % propane. No olefins or carbon oxides were found by gas chromatography. The solid product was either UO_2 or $U(OH)_4$; 97% of the uranium was tetravalent. The reaction was slow at first, often showing an induction period, but once initiated proceeded rapidly. A 10-g specimen was virtually completely hydrolyzed in 1 hr.

Power Reactor Fuel Processing: U-Mo Fuels. — A stable solvent extraction feed solution of 1.0 M U, 0.017 M Mo, 0.003 M Al, 175 ppm Cl, and 3 M HNO_3 was prepared from prototype CPPD fuel, an alloy of uranium with 10% molybdenum. The aluminum can was removed as volatile aluminum chloride by treatment with a mixture of hydrogen chloride and nitrogen at 300°C, and the core was oxidized with a mixture of hydrogen chloride and air at 400°C. The molybdenum oxychloride that formed was volatilized, and the uranium oxide residue was dissolved in nitric acid. This procedure is based on the assumption that the original stainless steel cladding and sodium bond have been replaced in a mechanical processing facility by an aluminum shipping can.

Power Reactor Fuel Processing: Zirconium-Containing Fuels. — In engineering-scale development work on the Zirflex process for decladding Zircaloy-2-clad fuels, solutions ~ 0.8 M in zirconium that were stable at room

temperature were produced by dissolution of Zircaloy-2 in 4.5 M NH_4F–0.5 M NH_4NO_3. The average dissolution rate was acceptable, ~ 2 mg min^{-1} cm^{-2}. Neither increasing the nitrate concentration nor sparging with air produced higher dissolution rates or decreased the amount of solids formed. This direct method may be preferable to the conventional dissolution in 6 M NH_4F–0.5 M NH_4NO_3 to produce a solution 0.9 M in zirconium which must be diluted to ~ 0.8 M to remain stable. Solids centrifuged from the solutions were soluble in 8 M HNO_3 at 75°C.

Power Reactor Fuel Processing: Solvent Extraction. – Synthetic aqueous waste from the processing of Consolidated Edison fuel by the Acid Thorex process was concentrated by a factor of 18 without crystallization at room temperature. The solution – 0.043 M $Al(NO_3)_3$, 0.043 M H_3BO_3, 0.043 M $NaHSO_3$, 0.017 M NaF, 0.0043 M H_3PO_4, 0.0043 M $Fe(NH_2SO_2)_2$, and 2.6 M HNO_3 – was first evaporated to 1/75 of the initial volume to remove excess acid and then diluted back to 1/18 of the original volume.

A source of pure niobium tracer is required for the study of niobium in solvent extraction. In initial tests on separating trace amounts of Zr-Nb in 8 to 16 M HNO_3, more than 85% of the niobium and less than 10% of the zirconium were sorbed on passage through a 10-mm-dia column containing 100 to 200 mesh Vycor glass.

In further tests to establish the relation between tributyl phosphate concentration, saturation with uranium, and fission product decontamination, twice as much activity was extracted in a countercurrent batch extraction in the absence of uranium by 15% TBP as by 2.8% TBP; 20% as much activity was extracted with 15% TBP that was 60% saturated with uranium as by 2.8% TBP that was 40% saturated.

Power Reactor Fuel Processing: Feed Clarification. – The decladding of ceramic nuclear fuels by dissolution in acids produces solutions containing both siliceous and UO_2 or ThO_2-UO_2 solids. More than 90% of the UO_2 was recovered from an aqueous slurry containing 30 g of UO_2 (particle size, d_{50} = 20 μ) and 5.6 g of silica (particle size, d_{50} = 4.5 μ) per kilogram of water by passage of the slurry through a steam jet–hydroclone. Less than 1% of the silica was retained in the induced underflow pot of the clone during continuous operation when the liquid was recycled through the clone six times. The diameter of the clone was 0.4 in. and that of the underflow port was 0.143 in. Because of the tendency of solids to collect in stagnant pockets, UO_2 recoveries were not reproducible.

Treatment and Disposal of Radioactive Wastes: High-Activity Waste. – Synthetic Purex waste, with NaOH and MgO added to decrease sulfate volatility, was calcined to 900°C continuously in a 4 × 18 in. stainless steel pot. The solid residue, about 6 gal per ton of uranium processed, had a density of about 1.32 g/ml (porosity about 58%); the thermal conductivity was 0.155 Btu hr^{-1} ft^{-1} $(°F)^{-1}$ at 475°F and 0.207 at 676°F. The condensate and off-gas scrub liquid contained 0.28 and 0.02%, respectively, of the feed sulfate. A closed system was used, with gas recirculation, and nitric oxide was added as required to keep the system pressure near atmospheric. There was practically no noncondensable off-gas.

Calcination of Purex waste containing Na_2SO_4 and NaOH to produce a lower-melting solid residue gave a solid melting at 750°C and occupying 40%

less volume than that from waste calcined without additives. This calcination was carried out in an 8 × 84 in. stainless steel pot.

In a wetted-wall tower constructed to study the growth of soluble aerosols at constant temperature, the average diameter of a homogeneous NaCl aerosol increased from 2.89 to 5.05 μ in passing through the tower with the water phase at 27°C.

Ruthenium volatility during batch evaporation and calcination of synthetic Purex waste to 400 to 500°C in 125-ml glass equipment was about 70% with no additives. Most of the ruthenium came off during the last half of the boildown, but a significant fraction came off during the calcination after the residue went dry. The effect of other variables was as follows:

Conditions	Ruthenium Volatility	
	Boildown	Calcination
Nitric oxide atmosphere*	~ 0	0.9–1.5%
0.01 M tributyl phosphate	~ 0	3.1–4.5%
0.01 M dibutylphosphate*	Less effective than TBP	More effective than TBP (total 13%)
0.01 M TBP + 0.01 M DBP	~ 0	0.9%
0.01 M monobutylphosphate	Less effective than TBP	More effective than TBP (total 7%)
0.01 M inorganic phosphate	No effect	No effect

*Effectiveness decreased by NaOH, $Ca(OH)_2$, MgO, or Na_2SO_4.

Treatment and Disposal of Radioactive Wastes: Low-Activity Waste. — In studies of removal of cesium and strontium from ORNL process-water waste by phenolic ion exchange resin at pH 11.7, the presence of 10 ppm ethylenediaminetetraacetate, which might be present as a result of decontamination procedures, had negligible effect on the performance of Duolite C-3 phenolic-sulfonic resin. This amount of EDTA does decrease the efficiency of the lime-soda treatment currently in use.

Duolite S-30, a phenolic resin, had about half the breakthrough capacity of phenolic-sulfonic (Duolite C-3) and phenolic-carboxylic (Duolite CS-100) resins. The S-30 resin, however, required only about half as much regenerant acid as CS-100 and only about one-tenth as much as C-3.

Waste Disposal Research and Engineering: Geochemical Studies. — With a column containing an alumina bed, relatively efficient removal of strontium from simulated waste 0.1 M in $NaNO_3$ was obtained. The waste solution was 1.1×10^{-5} M in strontium ions (1 mg/liter) and 10^{-3} M in Na_2HPO_4. Further tests using a slurry technique have shown that alumina without added phosphate is also effective for strontium removal. Comparative slurry tests at pH 7.5 (1 g of Al_2O_3 to 50 ml of waste) showed that 63% of the strontium was removed when the waste was 10^{-3} M in Na_2HPO_4 and 50% when no phosphate was added. Further experiments showed that pH markedly influenced the sorption of strontium from this system; the higher pH favored its removal. Thus

at pH 10.0 and with no added phosphate, strontium removal was found to be 85%.

Increasing the $NaNO_3$ concentration decreased the amount of removal; increasing the concentrations of alumina and phosphate ion improved the removal of strontium as expected. Sodium fluoride (10^{-3} M) was also found to improve the removal of strontium from this system, though the same concentration of NaCl had no measurable effect on the removal of strontium from simulated waste 0.1 M in $NaNO_3$.

Waste Disposal Research and Engineering: Disposal in Natural Salt Formations. — In the field experiments at Hutchinson, Kansas, the application of power to the acid cavity was discontinued on August 2 with the liquid temperature at 63°C. The temperature had dropped to 35° by August 15. Operation of the neutralized cavity is continuing, with an average liquid temperature of 71°C.

Study of the steady-state radiolytic-gas pressures produced by Co^{60} gamma irradiation of synthetic Purex waste solutions has been continued. Results of the study show that these pressures are influenced by the waste composition and can be reduced to 2.7 atm by decreasing the NO_3^- concentration and neutralizing. Metallic salts were not effective as recombination catalysts for reducing the steady-state gas pressures.

Waste Disposal Research and Engineering: Disposal of ORNL Radioactive Wastes. — Four naturally occurring materials, namely, vermiculite, variscite, Tennessee rock phosphate, and Florida pebble phosphate, were studied to investigate the feasibility of using them in columns for sorption of strontium from the high-pH intermediate-radiation-level wastes produced at ORNL. All except vermiculite were found to be effective in strontium removal.

The addition of soluble phosphate to the solution markedly improved the strontium-sorption characteristics of vermiculite. With 4 g of vermiculite and 100 ml of waste solution, strontium removal increased from 40% to 92% when 100 ppm of PO_4^{---} ions were added to the waste. Decreasing the pH of the solution adversely affected the strontium removal properties of all the materials.

All the materials showed beneficial, but not marked, effects with increase of the Ca^{++} concentration in these highly basic solutions. Increasing the strontium or sodium concentration did not produce any apparent beneficial effects for strontium sorption.

The commercially available Florida pebble phosphate material has good structural and hydraulic properties, and studies with a 1-in.-dia column gave promising decontamination. This material will therefore be tried in a larger column.

PHYSICAL RESEARCH PROGRAM

PHYSICS AND MATHEMATICS RESEARCH

High-Energy Physics. — An analysis was made of Λ hyperons produced by K^- mesons interacting with helium. Of 159 identified Λ hyperons which decayed into a proton and a π^- meson, 101 were produced by stopped K^- mesons. The direction of emission of the pion in the center-of-mass system was found to be isotropic within statistical variation. This result is in agreement with theoretical expectation.

Nuclear Measurements for Reactor Design. — An experiment is being carried out to measure the energies of correlated fission fragment pairs from the spontaneous fission of Cf^{252}. Silicon surface-barrier detectors are used in conjunction with specially designed low-noise amplifiers and a two-dimensional 100 × 100 channel recorder.

Scintillation Spectrometry and Instrument Development. — The Mössbauer effect has been observed for Ni^{61} at liquid-nitrogen temperature. The width of the resonance absorption curve is difficult to interpret without further experimentation. Present estimates put the lifetime of the 72-kev first excited state at approximately 1.3×10^{-9} sec. The curve may be broadened significantly because of hyperfine interactions. Measurements of the temperature dependence and foil-thickness dependence are now under way.

High-Voltage Experimental Program. — Experimental information on level positions and transition rates has been summarized for nuclei which exhibit enhanced E2 transition rates and yet do not belong to the spheroidal groups. The E2 transition rates indicate that the dynamic quadrupole deformations of many nuclei in this group are comparable with the permanent quadrupole deformations of spheroidal nuclei. It is well known that the concept of phonon vibrations has considerable success in describing the properties of the low-lying states of a large fraction of the nuclei in this group. Several theoretical studies have been made in order to obtain better quantitative agreement with experimental results. The effects of the coupling of intrinsic states to the phonon states have been considered. Alternatively, the expected spectra and transition rates have been worked out for several quite different shapes of the nuclear potential energy surface. The different theoretical results are compared with the experimental results for some nuclei. Since the magnitudes of the changes in level spectra and transition rates caused by interference from intrinsic states and by different shapes for the energy surface are comparable, it is difficult, with the still limited experimental information, to extract preferred descriptions for these nuclei.

In conjunction with measurements of internal conversion electrons made at Rice Institute and Wisconsin, an interpretation of the gamma-ray yields from Th^{232} was carried out taking a γ-vibrational state at 790 kev and a β-vibrational state at 773 kev. The values of $B(E2)_d$ for the vibrational states in Th^{232} and U^{238} are two to three times $B(E2)_{sp}$. For the E0 transition $2^+ \rightarrow 2^+$ of 723 kev, ρ is $(8 \pm 2) \times 10^{-2}$.

Millimicrosecond time-of-flight techniques, used for studying the total neutron cross sections of separated isotopes from 3 to 50 kev for nuclei with mass numbers from 45 to 88, have resulted in the determination of s-wave level densities and strength functions. Average radiative neutron capture cross sections for nuclei with mass numbers between 79 and 197 for energies between 7 and 170 kev provided p-wave strength functions and estimates of s-wave level densities.

The 63-Inch Cyclotron Operations. — The 63-Inch Cyclotron was available for experimental work for approximately 77% of the scheduled operation time during July and August. About two weeks of nonbombardment time resulted from leaks developing in the cooling water jackets in the liner. These leaks have been repaired, the machine is being baked out, and minor adjustments are being made in order to improve the beam. The breakdown of the down time of the machine is as follows: beam improvement 4.0%, routine source changes 2.0%, minor electrical troubles 2.0%, vacuum troubles 8.0%, and baking out the machine 7.0%.

Heavy-Particle Physics. — A method has been developed for the manufacture of thin (20 to 75 $\mu g/cm^2$) self-supporting boron targets; these are now being used to measure the differential cross section for the elastic scattering of nitrogen from B^{10}. Data on the elastic scattering of heavy ions have been analyzed with the aid of a recent semiclassical model proposed by Ford and Wheeler; the calculations were made at the IBM-704, and best fits to the experimental data indicate that the nuclear surface thickness is $\sim$ 20% of the radius. The differential cross section for the reaction $N^{14} + N^{14} \rightarrow C^{12} + O^{16}$ at 25.8 Mev (in the laboratory system) was measured at eight angles in the region from 0 to 60° in the center-of-mass system. No reactions were seen in which both products were left in the ground state. The upper limit was found to be $\sim$ 2 microbarns per steradian.

The 86-Inch Cyclotron: Nuclear Physics. — The measurement of (p,d) spectra from the iron isotopes 54, 56, 57, and 58 has been completed. Rather extensive angular distributions were obtained from all the isotopes to aid in the identification of the character of the nuclear levels producing the observed deuteron groups. Interpretation of the observed spectra is somewhat complicated by the effects of the unclosed proton shell. Deuteron and triton spectra and angular distributions are now being obtained from separated isotopes of nickel. The nickel isotopes possess a closed proton shell (Z = 28), and the low-lying levels should be free of the effects of proton excitation. This simplification should yield spectra that are more easily interpretable and should serve as an aid in explaining the more complicated iron spectra.

A closed-circuit television system has been set up between the experimental area and the counting room. This equipment has been used to obtain more accurate aligning of the beam on the targets and help eliminate uncertainties of spectrum contamination due to misalignment of the smaller separated-isotope targets.

The 86-Inch Cyclotron: Applied Physics. — The National Aeronautics and Space Administration utilized the external beam of the 86-Inch Cyclotron in a preliminary study of the effects of the radiation belt which envelops the earth starting at a height of several hundred miles and extending to a few

thousand miles. The 22-Mev proton component represents a sizable fraction of the spectrum. Various components of satellites, such as solar cells, quartz-windowed solar cells, metals, alloys, transistors, and condensers, were subjected to the 22-Mev proton beam. A series of irradiations was made to simulate a 5-yr exposure in space. Various measurements of electrical characteristics were made during the bombardments.

The radioisotope Co^{61} was produced by a (p,α) reaction on enriched Ni^{64} for a study of the Mössbauer effect in Ni^{61} being carried out in the Physics Division. This work was performed in 10 μa of external beam. An associated determination of the lifetime of the 70-kev state in Ni^{61} by electronic timing techniques is also utilizing the same target.

The Chemistry Division is now routinely utilizing 20 μa of external beam in an experiment designed to collect and study the recoils in the bombardment of 1 μg of Pa^{231}.

The isotopes Be^7, V^{48}, Mn^{52}, Co^{57}, Co^{61}, As^{74}, Rh^{102}, I^{125}, and Pm^{148} were produced as service irradiations for customers in the United States (civilian and military) and India. These irradiations represented approximately 170 hr of bombardment. The production of As^{74} continued to rise: the average for the last four 10-hr bombardments was 232 mc, as compared with an average of 143 mc during 1959 – an increase of 62%. The last increase, from 41%, was accomplished by using 80-mesh germanium metal instead of 200 mesh. Sintering has now been reduced, so that dissolution of the target is no longer a problem. Auxilary beam sweeping greatly increased the yields of V^{48}, Mn^{52}, and Sr^{85} in capsule targets. The same technique applied to a 6 × 5-5/8 in. flat-plate nickel target increased the yield of Co^{57} substantially.

The 86-Inch Cyclotron Operations. – The operation of the 86-Inch Cyclotron has been relatively routine. Available beam time for experiments has been approximately 74% of the operational time, an average of 81.2 hr per week. Operational down time during this period was distributed as follows: minor vacuum troubles, 1.0%; baking out machine, 7.0%; routine target setup time, 8.0%; electrical troubles, 2.0%; miscellaneous (shutting down machine, source changes, etc.), 8.0%.

An automatic beam cutoff, operating on the high voltage, has been designed, tested, and installed. It is being used during capsule bombardments for the applied physics program. Capsule targets are normally bombarded at ~ 200 μa beam intensity. Increases in beam of ~ 10%, which may result from changing arc conditions or sparking and are not uncommon, will normally rupture such targets. This instrument will respond in ~ 1/60 sec, far quicker than the operator can respond.

Changes have been made in the beam pipe that is located in the pit. These changes involved water-cooling and evacuation of the beam pipe, and result in quicker changes of experimental apparatus. The normal setup time has been reduced from 25 min to 5 min.

Electronuclear Machines. – The Cyclotron Analogue II, an eight-sector, spiral-pole electron cyclotron, is being built to model the beam dynamics of the proposed 850-Mev proton AVF cyclotron. All magnetic field calculations for the sector coils are now complete; the sector coils are on order. The sector coils produce only a fraction of the isochronous average field, which

rises from 42 gauss at the center of the machine to 75 gauss at the extraction radius (~ 14 in.). Five circular coils, located 2.5 in. from the median plane, will produce the isochronous average field to within 0.5% except at the center. Thirty-two circular coils with a radial spacing of 0.5 in., located 0.8 in. from the median plane, are used for final correction of the average field. With the trimming coil currents adjusted by least-squares fitting, the field error is reduced to ±0.02%. The final design of the coils and their support structure is 80% complete.

The Isochronous Cyclotron. — Model magnet measurements have been analyzed to find the optimum iron configuration. The latest measurements have shown that it is possible to accelerate protons to full energy with a maximum phase slip less than 5°. The same pole geometry allows heavy ions to be accelerated to full energy with negligible phase slip. The focusing properties of these magnetic fields appear to be adequate.

A series of measurements on the full-scale r-f model has been completed. Values of Q and excitation power have been obtained over the entire 7.5 to 22.5 Mc tuning range which are accurate to within 10%. The value of Q varies from approximately 3000 at 22.5 Mc to approximately 7500 at 7.5 Mc. The excitation power required for a 100-kv peak dee voltage varies from approximately 45 kw at 7.5 Mc to approximately 160 kw at 22.5 Mc. Tuning measurements were used to estimate spider and trimmer resolution requirements. The spider position will need to be known within a tolerance of 0.1 in. The tips of the trimmers, which are approximately 6 ft from their pivot points, will require adjustments as small as ±0.001 in. without backlash.

The building is now totally enclosed except for doors and windows. Work on the interior walls of the laboratory-office area is progressing well. Much of the major equipment is now in the third-floor equipment room. Three of the four shielding doors are installed and manually operable. As of August 21 the building is about 70% complete.

Stable Isotope Development. — A second-pass separation of Pb^{204} has provided 126 mg of 99.7% product. A similar separation is in progress for Pb^{208} and one is planned for Pb^{206}.

A study of the contaminants present in enriched Ca^{46} samples revealed the presence of normal calcium in widely varying amounts, too variable to be attributable to processes occurring within the calutron. The receiver pocket material has been changed from graphite to copper in order to determine and minimize the sources of contamination.

Collector pockets and material from routine separation runs are being processed to accumulate data pertinent to the sputtering of graphite by 38-kev iron ions.

An improved leak valve and a low-temperature ion source were developed. Both features reduced maintenance costs.

Special Separations. — Experiments to determine the applicability of ion exchange to the purification of plutonium for recycle are being run.

Decontamination of the old wash area is complete, and the area has been painted. Steam and air lines have been removed from the outside of the plutonium lot in preparation for the removal of the old hot process vacuum line. Plywood boxes have been obtained in preparation for the burial of the wooden plutonium recycle boxes, which have been disconnected from their service lines.

CHEMISTRY RESEARCH

Thorium Oxide Studies. — Sedimentation particle size analyses of thoria powders fired at 650, 800, 900, 1100, and 1500°C and exposed to 6×10^{20} neutrons/cm^2 in the LITR showed decreases in the average particle sizes of the oxides and substantial production of fines less than 0.6 μ in size. The fraction of fines below 0.6 μ in the irradiated material decreased from 34 wt % in the 650°C-fired oxides to 13 wt % in the 1500°C-fired oxides. This irradiation produced about 4000 g of mass 233 per ton of thorium; about 0.1% of the initial number of thorium atoms were fissioned.

A heavy-water slurry of Houdry ThO_2 spheres, 18 μ in average size, containing 0.12% adsorbed natural uranium and exposed to 8×10^{19} neutrons/cm^2 in the LITR in a settled condition showed a substantial increase in the particle size fraction between 1 and 9.6 μ. About 0.06% of the thorium was converted to mass 233 isotopes and 4×10^{-4}% was fissioned. A similar sphere preparation of 11 μ average size showed essentially no degradation upon pumping as a slurry for 550 hr at 175°C in a 100A loop.

Analytical Chemistry Research. — An assembly for making differential thermal analyses up to 1000°C was completed, tested, and applied to the observation of the effects of gamma radiation on the bromates of the alkali metals. The sensitivity of the device was evaluated by carrying out differential thermal analyses on standards of SiO_2 and $NaNO_3$, with Al_2O_3 as the reference material. By this method, together with thermogravimetric methods, it was established that, in general, alkali-metal bromates subjected to gamma radiation have lower melting points and decompose more rapidly when heated than similar unirradiated compounds.

Organic Chemistry. — The secondary isotope effects in the formation of the 2,4-dinitrophenylhydrazones of acetophenone-β-C^{14} and of acetophenone-1-phenyl-C^{14} were found to be 1.0085 ± 0.0004 and 1.0038 ± 0.0003, respectively. The data were obtained by determining the molar radioactivities of successive small aliquots of product for known fractions of reaction, and were analyzed by means of a nonlinear least-squares code with the IBM-704 computer. The uncertainties are expressed as standard deviations.

Radiation Chemistry: Radiolysis of Solid Potassium Nitrate. — In the gamma-ray-induced decomposition of potassium nitrate dispersed in a pressed potassium bromide matrix (< 0.1 mole % nitrate) the initial yield for nitrite formation is equal to the yield for nitrate decomposition, as indicated both by infrared measurements and by chemical analysis of the dissolved disk. No other oxides of nitrogen are formed. In KBr disks containing either KNO_3 or KNO_2, initial yields both for nitrate decomposition and for nitrite decomposition increase with concentration, finally reaching limiting values at ~ 0.1 mole %. This indicates competition of the nitrate or nitrite acceptor with other energy traps present in the disk. Over this concentration range, initial yields can be expressed by $G_0 = Kc_0/(\beta + c_0)$, where K and β are 1.88 and 0.027 for KNO_3, and 0.75 and 0.009 for KNO_2; c_0 is the initial concentration. The equation is similar to that relating light emission to concentration of thallium activator in alkali-halide scintillators.

Radiation Chemistry: Radiolysis of Sulfuric Acid Solutions. — The gamma radiolysis of sulfuric acid solutions gave evidence that the decomposition of H_2SO_4 is analogous to that of H_2O. The effect with added scavengers such as Fe(II) and Ce(IV) can be explained by assuming that the active chemical intermediates arise both from H_2O and from H_2SO_4. In addition to the intermediates H, OH, H_2, and H_2O_2 from H_2O, the following are present from the acid: H, HSO_4, H_2, $H_2S_2O_8$, and H_2SO_5. The molecular products presumably are produced by combination of radicals as they diffuse away from the sites of formation.

The total number of solvent molecules decomposed (H_2O + H_2SO_4) appears to be nearly constant over the entire acid range studied — 95 wt % H_2O to 95 wt % H_2SO_4. A detailed study of the air-saturated Fe(II) system showed that the Fe(III) yield decreases from 15.6 molecules oxidized per 100 ev absorbed in 0.4 M H_2SO_4 solution to about 11.0 in 17 M solutions. The change in yield is concomitant with a change in the oxidation mechanism. In solutions 12 M or less in H_2SO_4, Fe(II) is oxidized by H atoms in one of two ways, either through the HO_2 radical with consumption of O_2 or directly by H with an increased yield of H_2. In solutions more than 12 M in H_2SO_4, the mechanism is more complicated; for example, SO_2 is produced in appreciable yields in the most concentrated solutions. The SO_2 yield was found to follow that reported [J. Am. Chem. Soc. 77, 3215 (1955)] for air-free solutions. The presence of O_2 was reported to inhibit the formation of SO_2, presumably by oxidizing the SO_2 back to sulfate. The presence of Fe(II) apparently prevents the oxidization of SO_2 by reacting with the oxidizing intermediates such as HO_2, thereby allowing SO_2 to build up.

Radiation Chemistry: Decomposition of Ethylene by Alpha Particles. — The radiolytic decomposition yield of ethylene obtained with alpha particles of radon agreed well with the early value of Lind, Bardwell, and Perry (molecules per ion pair, 5.1; molecules per 100 ev absorbed, 18). The ratio of the increase of gas noncondensable in liquid nitrogen to the decrease of condensable gas increases more than 25 fold during the course of the reaction. This indicates that the noncondensable gas (probably hydrogen) comes largely if not entirely from the irradiation of the condensed products. These products first appear as a colorless liquid, which, however, on continued irradiation solidifies and turns dark brown.

Nuclear Chemistry: Spectroscopy. — Beta-ray polarization measurements were continued. The apparatus was redesigned in order to relax somewhat the rigid requirements which the old equipment imposed on the beta-ray source. It is hoped that these improvements will permit a search for a possible pseudoscalar interaction in beta decay. Preliminary measurements with P^{32} were made in the rebuilt apparatus.

High-Temperature and Structural Chemistry. — An electrochemical investigation of solutions of cerium in molten cerium trifluoride was started. Some data in the recent literature, obtained with the use of alumina crucibles, were shown to be erroneous, as dissolved cerium reacted readily with alumina to form CeOCl and aluminum metal.

The electrical conductivities of three molten salts, LiI, CsF, and $CeCl_3$, were found to be quite different from values reported by others, which are thought to be in error.

A neutron and x-ray diffraction study of molten cesium fluoride yielded a radial distribution function and an estimate of 3.7 ± 0.1 nearest neighbors of opposite charge at a most frequent distance of 2.85 A. The number of nearest neighbors is significantly smaller than the number 6, at 3.12 A, in the solid at the melting point.

On the basis of new x-ray diffraction studies on molten and solid bismuth(I) chloroaluminate, the assumption of a trimeric ion, $(Bi_3)^{+++}$, was arrived at, which may replace an earlier proposal of a dimeric ion, $(Bi_2)^{++}$, thought to occur in $(BiCl)_x$.

Chemical Separation of Isotopes. — Installation of the water distillation cascade of the O^{17} pilot plant is complete except for instrumentation and insulation. Completion of the remaining work is expected within three weeks. Leak-testing is now in progress. The first unit of the thermal diffusion cascade has been assembled and will be tested early in September.

METALLURGY AND MATERIALS RESEARCH

Preparation of Pure Single Crystals. — A furnace assembly for growing single crystals of high-melting fluorides has been built and tested. The techniques for treatment of molten fluorides with anhydrous HF and hydrogen have been successfully applied to pure molten LiF at 900°C. Techniques were also demonstrated for the transfer of this pretreated molten material, without exposure to the atmosphere, into crucibles suitable for growth of the single crystals; the crucibles can be sealed under an inert atmosphere so that the crystal-growing operation can be performed within the sealed crucible system. Some difficulties with rupture of the thin-walled crucibles have been encountered, but it appears that this problem has been alleviated by improved crucible design.

Deformation of Crystalline Solids. — Progress has been made recently in measuring calorimetrically the heat liberated during a tensile test of a metal sample. This heat is used to vaporize some suitable fluid, such as Freon, and is measured by the volume of the gas evolved. The increase in the internal energy (stored energy) of the sample is given by the difference between the mechanical input and the heat liberated. Some data have been obtained for copper, nickel, brass, and aluminum. However, certain experimental difficulties prevent their complete acceptance. It is expected that improvements in the calorimeter design will correct this situation.

Alloy Theory Research. — The electrical resistivity of α-zirconium at low temperature is increased strongly by small additions of Ag, Cd, In, Sn, or Sb. The large effect is evidently due to the nonperiodicities introduced into the unfilled d band of zirconium by solutes of the above type, which have complete d shells.

A proportionality of the effect per atomic per cent of solute to the square of the excess positive charge on the solute ion was not found, the effects being more nearly represented by a linear increase with increased solute valency. Matthiessen's rule, that impurity and thermal scattering

are additive, is obeyed approximately in the most dilute alloys. With higher amounts of solute, a systematic negative deviation occurs in which the high-temperature resistivity is smaller than the sum of the impurity resistivity and the resistivity of pure zirconium at the same temperature. This effect is believed to be due to an increased tendency of the electrons to overlap into more mobile electron states.

Metal Surface Studies. — Single crystals of niobium have been used in a study of the phenomena associated with the "break-away" behavior of oxide films on niobium. The (100) planes were established as the most slowly oxidizing planes of niobium, whereas the (111) and (110) planes oxidized most rapidly. Oxidation rate curves for various crystallographic planes of niobium were determined and were correlated with the surface topographies of the oxide films. Oxide films on all crystal planes exhibited a characteristic break-away behavior. The density of cracks in the oxide film was shown to be much higher on the high-rate planes, and differences in the nature of the cracks formed on high- and low-rate planes were noted. Evidence of severe stress generation in the oxide and in the metal substrate during oxidation was also observed.

Fundamental Investigation of Radiation Damage in Solids: Sensitization of Alkali Halides by Calcium. — A recent study of the x-ray-induced absorption spectrum of potassium chloride containing calcium as an impurity suggests an explanation for the greatly enhanced sensitivity of alkali halide crystals containing a divalent impurity. Exposure to x rays at 195°K produces a 325-mμ absorption band, which prior electron spin resonance studies at the Argonne National Laboratory have identified as being caused by a Cl_2^- molecular ion oriented along < 100 >. This band is thermally unstable above 230°K, and the present studies have shown that on disappearance it is replaced by a band at 218 mμ, quite near the V_3 absorption band. The 325-mμ band may be regenerated and the 218-mμ band bleached by F-band excitation at 195°K. These results suggest that the sensitization of the crystal by calcium ion occurs through hole capture at the cation vacancy, which is taken into the crystal with the impurity ion to preserve electrical neutrality, followed by relaxation of a chlorine atom into the vacancy, thereby forming an anion vacancy and the Cl_2^- ion. The anion vacancy produced in this way may be the source of the much enhanced F-center development in crystals containing divalent ions. On warming, the Cl_2^- ion appears to ionize, producing the much more stable linear Cl_3^- molecular ion.

Fundamental Investigation of Radiation Damage in Solids: Lattice Defects in Alkali Halides. — One mechanism proposed to explain the creation of lattice defects in alkali halide crystals by ionizing radiation is that recombination of excitons at dislocation jogs may evaporate pairs of cation and anion vacancies from these sites. Such a process leads to dislocation climb and should be detectable as a displacement of the point at which the dislocation intersects the surface. A concentration of F centers greater than 4×10^{17} cm^{-3} was introduced by Co^{60} gamma-ray exposure into a sodium chloride crystal which contained $\sim 5 \times 10^5$ dislocations/cm^2. The F-center content should be an index of the number of anion vacancies created, and it was found that for this mechanism 4×10^{17} F centers should cause a dislocation climb greater than 3 μ for each dislocation present. Microscopic

examination of the location of dislocation etch pits before and after exposure showed no detectable climb greater than 0.5 μ. It is therefore concluded that, under the conditions of the experiment, evaporation of vacancies from dislocation jogs is not the principal mode of defect production by ionizing radiation.

Fundamental Investigation of Radiation Damage in Solids: Magnetic Susceptibility of Rhenium Compounds. – The rhenium atom, having a partially filled d shell, may be expected to have a magnetic moment, which might cause its compounds to become ferro- ferri-, or antiferromagnetic at low enough temperatures, although no such structure has yet been found by neutron diffraction techniques. Measurements of the magnetic susceptibility of K_2ReCl_6 and K_2ReBr_6 were performed between 4 and 300°K. In both compounds clear evidence of a magnetic phase change appeared between 10 and 20°K. In the bromide the magnetic phase change is at 15.5 ± 0.5°K; a specific heat peak occurs at the same temperature. The behavior of the susceptibility below the phase changes indicates that both compounds become antiferromagnetic at these low temperatures.

CONTROLLED THERMONUCLEAR RESEARCH

Arc Research. – A temporary vertical solenoid consisting of five of the coils for the 120-in. solenoid was assembled and put into operation during the latter part of May. From late May until early August a study of lithium arcs was conducted.

Several anodes were constructed and tried in an effort to overcome motor action of the molten lithium in the anode cup. Stable operation was obtained for arcs from 50 to 150 amp. Higher-current arcs could be run only for a short time because of inadequate heat dissipation in the anode and cathode.

Spectrographic data showed strong Li^0 and Li^+ lines near the anode. A red glow from Li^0 was visible at the anode end, but not from other portions of the arc. Mass-analyzer data with the probe approximately 1/8 in. from the arc and approximately 24 in. from the anode indicated primarily Li^+ with a small amount of Li^{++}. No indication of Li^{+++} was observed.

Early in August the temporary vertical solenoid was disassembled and assembly of the 120-in. horizontal solenoid was started. The estimated completion date for the 120-in. solenoid is October 1, 1960.

DCX-1 Facility. – The analyses of the mass spectrometer data on liner gas composition, which were taken simultaneously with gas-breakup neutral-particle detector data, have been completed. The pressure variations of the impurity components led to a qualitative understanding of the gas-breakup proton containment time vs pressure curves.

The energy-loss experiments, using the neutral-particle spectrometer, were continued but have been interrupted by ground faults in two magnetic field coils. Spare coils were available and are being installed.

Larger Magnetic Facility. – AEC approval for the construction of this research facility has been received. Design work on the equipment is continuing. Magnetic tests on a scale-model injection snout are still in progress.

Vacuum. – The feasibility of enhancing the performance of an oil diffusion pump by raising the top diffusion stage has been under investigation. Such a modification of a Westinghouse 20-in. pump with improved DPI jets has been made by removing a 20-in. section of the pump barrel. The original jet assembly, when installed in this shortened barrel, projected 9 in. into a 36-in.-dia test dome, thus wholly exposing the uppermost vapor stream. Changes of the top jet cap and of the geometry of the annular nozzle defining the vapor stream resulted in pumping speeds which varied from 2000 to 13,000 liters/sec; these may be compared with the probable unbaffled speed of about 8000 to 10,000 liters/sec. A study of oil collection from the dome, coupled with observations of localized heating of the dome, indicated a wide divergence of the primary vapor stream. Although an optimum nozzle design has not been established, base pressures in the dome have been surprisingly low – usually about 2×10^{-7} mm. The limiting fore pressure was 150 μ (characteristic of the 8-in. booster pump) and was independent of changes made in the 20-in. pump.

Filaments made of an alloy of titanium and tantalum offer promise of high evaporation rates for pumping by vapor-deposited metal films. Such filaments appear to be self-healing during use. A local melting of part of the filament depletes that part in titanium, which results in a melting-range increase. (Similar behavior might be expected for the molybdenum-tungsten system.) Utilization has been made of the niobium-titanium system, which is characterized by an extraordinarily wide two-phase region. The alloy flows with difficulty over a large temperature interval, on account of the presence of both solid and liquid. Such an alloy has been used to evaporate titanium successfully with arc heating.

BIOLOGY AND MEDICINE PROGRAM

BIOMEDICAL PROBLEMS IN ATOMIC ENERGY OPERATIONS

Waste Disposal Research and Engineering: Evaluation of Soil Disposal. – Primary emphasis has been shifted from an evaluation of specific disposal operations to a comprehensive study of the nature and extent of fission product accumulation and transport in the White Oak Creek drainage system, resulting from general and Laboratory fallout, as well as waste disposal practices.

The formation of White Oak Lake (a 44-acre impoundment now maintained almost empty for emergency containment) and an intermediate pond on White Oak Creek (an 8.3-acre impoundment no longer in use) resulted in the deposition and accumulation of contaminated sediments. The quantity of fission products in temporary storage in deposited sediments is of importance, since solution and sediment transport will carry these radionuclides to the Clinch River.

From cores of sediment recovered from the lake bed and the pond bed, it is estimated that 11.2 curies of Sr^{90}, 477 curies of Cs^{137}, and 58.2 curies of Co^{60} are present in the lake sediments, and that 3.7 curies of Sr^{90}, 17

curies of total rare earths, 114 curies of Cs^{137}, and 12.6 curies of Co^{60} are present in the pond sediments. The above total for Sr^{90}, 14.9 curies, represents a decrease of about 30 curies from an estimate for 1956 of 47.8 curies.

TRAINING, EDUCATION, AND INFORMATION PROGRAM

Biology and Medicine Program. — The summer training program in applied health physics, which began June 1, was completed August 24. The group consisted of 13 AEC Fellows in Health Physics, three Air Force officers, and four foreign students (two from Greece, one from the Philippines, and one from India).

ISOTOPE DEVELOPMENT PROGRAM

Radioisotope Research and Development. — The aluminosilicate cation exchange material manufactured by the Permutit Company under the trade name Decalso was found to be suitable for the adsorption of Sr^{90} fractions. It was shown that 40,000 curies could be shipped in one shielded transfer tank containing Decalso and subsequently eluted with NH_4NO_3 solution to provide a feed for processing in the Fission Product Pilot Plant.

A precipitation method was perfected whereby a filterable titanium strontium oxalate compound can be prepared by urea hydrolysis followed by treatment with gaseous ammonia to slowly raise the pH of the solution to 8.5.

A compact furnace was designed to utilize the decay heat of $Ce^{144}O_2$ and $Sr^{90}TiO_3$, augmented by resistance heating in platinum wires, to form ceramic pellets from these compounds at 1400 to 1500°C.

A 400-g quantity of NH_4TcO_4 was prepared for the Tc^{99} inventory. In addition, approximately 1 kg of Tc^{99} in a concentrate is available for final purification. The indicated rate of processing of Tc^{99} at ORNL is about 400 g/month.

Radioisotope and Stable Isotope Production. — Charge materials were prepared for the separation of the isotopes of tantalum, osmium, magnesium, and calcium in Building 9731, and of lead (radioactive and enriched in Pb^{208}), iron, and calcium in Building 9204-3 (expanded facilities). Fifty-two kilograms of anhydrous fused $FeCl_2$ and 1100 kg of AgCl were also prepared. The chemical recovery of osmium, which was used as charge material but was not successfully resolved into its isotopic components, was initiated. This chemical processing has been restricted to the graphite parts associated with the ion sources and accelerating electrodes. Approximately 6 kg of $PbCO_3$ and 1.7 kg of $PbCl_2$, both enriched in Pb^{206}, have been recovered from used charge material. Collections of isotopes of calcium, iron, lead, and tantalum were made, with ion currents equivalent to 1510 g. Separations were performed in the four calutrons in Building 9731 and the 14 calutrons in Building 9204-3 (expanded facilities).

Thirteen new lots of the separated isotopes of barium, calcium, chromium, and lead and seven lots of isotopes returned from loans were recovered,

refined, and prepared for inventory. Fifteen new lots and two returned lots were prepared for inventory following chemical refinements and spectrochemical and mass analyses. The isotopes Br^{79}, Br^{81}, and Pb^{207} were collected and refined under research and development conditions to obtain products of greater than 99% isotopic purity. The bromine isotopes were separated for the National Bureau of Standards and were ultimately recovered as sodium bromide.

Sixty-two samples were recovered on a preliminary basis for study of retention, mass analysis, sputtering, and other developmental purposes. Among these were Ca^{46} collected on single crystals of copper, Mg^{26} separated in a 270° mass separator, and Pb^{204} collected in the $(Pb^{204}Cl^{35})^+$ position.

Conversions of inventory forms to more suitable forms included Li^6 metal to Li_2CO_3, Li^6 metal to LiF, Cr_2O_3 to CrO_3, $Ge^{74}O_2$ to germanium metal, and Hg^{198} in the form of aqueous nitrate to mercury metal.

Samples of Hg^{199}, Hg^{200}, Hg^{202}, and N^{15} stocked as aqueous nitrate were encapsulated in Pyrex ampoules. One sample of Hg^{199} metal was encapsulated in a quartz ampoule.

Target preparations included 35 conversions and 17 targets.

Thirty grams of Np^{237} was purified from uranium by an oxalate precipitation process and converted to neptunium oxide. This material now contains only 300 ppm uranium. Preparations are being made to load about half this material into a container for cross-section measurements. The rest of the NpO_2 has been shipped.

Recovery of 738.5 g of uranium as U_3O_8 was made from the carbon ash from the last series of U^{238} runs.

Recycling of enriched Pb^{208} is approximately 60% complete. The Pb^{210} product recovery from this run has been started.

Four plutonium return samples have been purified and added to the inventory. Two other impure plutonium samples are in process.

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August 1960

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233. B. S. Weaver
234. A. M. Weinberg
235. M. E. Whatley
236. J. C. White
237. C. E. Winters
238. E. O. Wollan
239. R. E. Worsham
240. W. Zobel
241. A. Zucker
242-243. Central Research Library
244. Health Physics Library
245. ORNL — Y-12 Technical Library, Document Reference Section
246. Reactor Experimental Engineering Library
247. Biology Library
248-439. Laboratory Records Department
440. Laboratory Records, ORNL R.C.