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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JULY 1962

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OAK RIDGE NATIONAL LABORATORY
STATUS AND PROGRESS REPORT
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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JULY 1962

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

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROGRAM

EGCR. – The facility for testing the EGCR control-rod drive is essentially complete, and functional tests were started. Performance characteristics for gas flow inside and outside the top and bottom fuel element dummies were determined, and a variable orifice was designed for the bottom dummy to provide a linear relationship between orifice position and flow rate.

Work is progressing satisfactorily on the application of Stellite overlays to nozzle tee components for the EGCR experimental loops. All prefabricated loop piping was delivered to the EGCR for installation in the loop pipe chases in the EGCR concrete.

GCR-ORR Loop No. 1. – A test fuel element (7C) instrumented to simulate one element of an EGCR instrumented fuel assembly operated satisfactorily in GCR-ORR loop No. 1 for the scheduled 14-week irradiation. The fuel central temperature was measured by a tungsten-rhenium thermocouple installed in a molybdenum well. Four stainless-steel-sheathed Chromel-Alumel thermocouples were brazed with copper to the inner surface of the cladding and were fitted into notches cut in the UO_2 fuel pellets. Two of these thermocouples were 0.065 in. in diameter, and two were 0.040 in. in diameter. Corrections were made for the effect of displaced fuel. These thermocouple installations facilitated measurement of the cladding temperature without disturbance of the gas-coolant flow stream.

During the irradiation the desired fuel cladding temperature was maintained by adjusting the loop gas temperature. The test element operated at a constant maximum cladding temperature of 1500°F and a fuel central temperature that varied between 2100 and 2200°F. This irradiation, the fifth in a series, completed the testing of elements simulating the features of EGCR instrumented fuel assemblies.

GCR-ORR Loop No. 2. – Much of the installation of GCR-ORR loop No. 2 is now complete. The main piping was installed and leak-checked satisfactorily. Final leak checks of the Marman Conoseal joints are in

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progress. With the installation of four gas-throttling valves and a gas chromatograph, the gas purification and sampling systems will be complete. The chromatograph is being tested in the laboratory. Final modifications to the control systems and calibration of instruments associated with the main piping are in progress. The main (18-in.) plug for the beam hole is undergoing hot performance tests in a laboratory component test stand. Cold shakedown of the loop and facility components is in progress.

Pebble-Bed Reactor Experiment. – Twelve 1-in. Marman Conoseal joints were received for evaluation. A rig for testing six Marman and six Graylock joints is being designed, and a rig for testing a 36-in.-diam Graylock joint is being fabricated.

Performance and design criteria for PBRE gas-bearing blowers are being developed, and a cold unit for studying maintenance and operating problems of these machines is being assembled.

Calculations were carried out for estimating the amount of cerium that would be released from PBRE fuel, assuming that (1) the more volatile precursors (Xe, Cs, Ba) are held in the fuel compacts; (2) the coolant is saturated with gaseous cerium from the reaction $CeC_2(s) = Ce(g) + 2C(s)$ at 1700°F; and (3) Dalton's law approximates the amount of gaseous cerium saturating the coolant. This amount of cerium is calculated to be 0.0008 g/yr. Since assumption (2) is not likely under reactor operating conditions, the estimate of cerium release is conservative by several orders of magnitude. This negligible release of cerium will apply quite well to those isotopes that have no volatile precursors (Ce^{145} through Ce^{148}); Ce^{143} and Ce^{144} have very short-lived volatile precursors and should also be effectively held up.

Criticality calculations for the PBRE were previously made on the basis of a simple volume homogenization of the core material, but, since there is an average void fraction of 39%, there may be an appreciable error introduced by neglecting the effects of streaming within the core. Calculation of these effects is complicated by the partially random nature of the size, shape, and distribution of the voids. An estimate of the streaming was obtained by the method of Behrens. It was assumed, for calculational purposes, that the voids were regular tetrahedrons, regularly distributed, with one void for each fuel sphere. The corrections to the diffusion coefficient, when calculated in this manner, amounted to 2% in the energy range 10^7 to 1.35×10^6 ev and increased to 13% in the energy range 0.0131 to 10^{-4} ev. A calculation will be made to see how much k is changed.

GCR-3 Physics. – The study of GCR-3 fuel cycles was extended to determine the effects of core zoning on fuel lifetime and fuel-cycle costs. By use of the FUELMOVE program for the IBM 7090, calculations were made for the cases in which the core is divided into 2, 3, 4, and 5 equal-volume radial zones. The reactor is operated batchwise with fresh fuel until it ceases to be critical with all control poison removed, and the fuel in the central zone is then discharged. Fuel in the outer zones is moved one zone inward, with an axial inversion if the move occurs at approximately midlife. The results obtained for the GCR-3 core, with a 9.4-in. triangular lattice pitch and with 3% enriched fuel, are summarized below:

	Number of Zones			
	2	3	4	5
Average exposure of first core, Mwd per metric ton of U	15,479	17,023	17,498	18,197
Average discharge exposure at cyclic equilibrium, Mwd per metric ton of U	16,729	18,606	19,637	20,279
Maximum discharge exposure at cyclic equilibrium, Mwd per metric ton of U	18,758	20,739	21,349	21,959
Feed time interval at cyclic equilibrium, days	365.3	270.9	214.4	177.1

The effect of zoning on fuel-cycle costs was examined. Results are given below for the cases in which a fuel lifetime of 20,000 Mwd per metric ton of U is desired:

Number of Zones	Enrichment Required (%)	Fuel-Cycle Costs per Electrical Output (mills/kwhr)
1	4.08	2.095
2	3.42	1.800
3	3.20	1.725
4	3.09	1.680
5	3.02	1.640
∞	2.77	1.525

The results indicate that on-line fuel-handling equipment could save about 0.1 to 0.2 mill/kwhr in fuel-cycle cost.

Gas-Structural Materials Compatibility Tests. – A facility is being operated to test the compatibility of stainless steels and Inconel with hydrogen at 1000 psig and 1500°F, and another facility was completed for testing the compatibility of graphite with oxidizing impurities in helium at conditions of interest to the PBRE. In conjunction with the latter tests, appropriate analytical techniques are being developed for the measurement of impurities at very low concentrations by gas chromatography. The chromatograph was improved by adding shielded thermistors to minimize noise from flow fluctuations, by adding relief regulators to reduce variations with atmospheric pressure, and by improving temperature control.

Compressor Development. – The Bristol Siddeley experimental gas-bearing compressor, which performed satisfactorily in exhaustive tests,

was disassembled and studied and was reassembled successfully for additional operation and study. Bristol Siddeley compressors were received for GCR-ORR loop No. 2 and are now being tested prior to operation.

LITR Irradiation Experiments. – A new series of tests, designated L-HT, was designed for determining the fission-gas release of UO_2 at temperatures above 3650°F, where void migration and the resulting columnar grain growth are expected to occur. The enriched fuel pellets are insulated by bushings of depleted UO_2 and retained by a tantalum sleeve in the LITR Inconel capsules. Void spaces within the capsule are filled with an argon atmosphere. The first test is now operating at fuel temperatures of ~3900°F, as indicated by tungsten vs rhenium thermocouples.

Fission-Gas Release from Pyrolytic-Carbon-Coated Uranium Carbide Particles During Irradiation. – The fission-gas retention properties of pyrolytic-carbon coatings on uranium carbide particles are being studied by comparing various kinds of coated particles with bare uranium carbide particles in the B-9 and C-1 fission-gas facilities in the ORR. Three pyrolytic-carbon coatings have been studied: laminar, columnar, and duplex (combination columnar and laminar).

All the coatings leaked gas as soon as irradiation began. The release rates of fission gas did not increase with burnup. Two of the specimens, the columnar and the duplex, showed temperature-dependent gas release at constant fission power in the 1300 to 1700°F range; the others did not. The distribution of isotopes in the fission gas indicated that the primary release from the laminar-coated particles was by recoil. About 5% of the laminar-coated particles ruptured at initial startup, and few, if any, failures occurred with continued irradiation and temperature cycling.

The release from the columnar- and duplex-coated particles was primarily by diffusion through the coatings. It is difficult to estimate the proportion of particles with microcracks in the coatings. The release rates of two isotopes of xenon and three isotopes of krypton are averaged, and the ratios of release rates are given below to indicate the relative efficiency of the coatings in retaining fission gases. The fission-gas release rates of uncoated particles ranged from 7% Xe^{133} to 0.4% Kr^{87} at 1500°F. In the data given below, the gas retention is the ratio of release from bare particles to release from coated particles:

Coating Structure	Uranium Carbide Particle Size (μ)	Coating Thickness (μ)	Gas Retention at 1500°F	Uranium Burnup (%)
Bare particle	210		1	4.3
Laminar	235	100	55	14.9
Columnar	200	82	300	12.1
Duplex	275	42	1300	14.7

Instantaneous Fission-Gas Release from UO_2. – Specimens of thin-plate UO_2 are being irradiated in the ORR C-1 fission-gas facility to study the fundamental mechanisms of fission-gas release. Specimens of sintered stoichiometric and nonstoichiometric UO_2, as well as fused-crystal specimens, have been irradiated. A single-crystal specimen is now being irradiated. The following observations have been made:

1. Sintered and fused-crystal specimens have temperature-dependent gas release above about 500°C. The gas release from the single crystal is temperature-dependent above about 700°C.
2. Excess oxygen can increase fission-gas release from UO_2 by a factor of 100.
3. The activation energy for sintered and fused-crystal specimens, both stoichiometric and with excess oxygen, is from 30 to 40 kcal/mole for xenon, krypton, and iodine during irradiation.
4. About 45% of the 133 mass and 77% of the 135 mass released from the UO_2 escape as iodine. This proportion does not change with stoichiometry or flux.
5. The gas release from the single crystal is about a factor of 10 lower than that from the sintered and fused stoichiometric specimens for equivalent temperatures.
6. Both heating and cooling bursts were observed from the sintered nonstoichiometric specimens, while the fused-crystal stoichiometric specimens emitted only heating bursts.

Simulated Fuel-Element Studies with Chipped UO_2 Pellets. – A second experiment with chipped UO_2 pellets supplied by Westinghouse was discontinued after 50 thermal cycles. The results confirmed the ratchetting of the cladding and the cladding protrusions observed in the initial experiment.

Metallographic examination has shown that the cladding protrusions noted on both specimens were at or near the major chips on each pellet. More UO_2 particulate matter was observed in the pellet-to-cladding interface than previously. One UO_2 pellet had fractured in such a manner that a large segment, approximately 5% of the pellet, was able to move a distance of 0.002 in. In all previous metallographic samples the fracture had occurred so that no movement of large particles into the inner annulus was noted. These results, when compared with those of previous experiments, indicate that the resistance to fracture of these chipped pellets of UO_2 is less than that of high-quality pellets.

Intergranular cracks on the inner surface of the cladding were observed in the second experiment. There are indications that these cracks do not occur at positions corresponding to the pellet-to-pellet interfaces. Additional metallographic examinations will be needed to study the nature of this cracking.

Preparation of Fueled BeO Specimens for Irradiation. – Thirty BeO-UO_2 specimens were fabricated to dimensional specifications by cold-pressing and sintering in hydrogen at 1750°C. These specimens were carefully characterized with respect to dimensions, density, surface cracking, and uniformity of fuel distribution. On the basis of the results, 16 specimens were selected for use in the ORR poolside group VI experiment. The composition of these specimens is 60.1 ± 1.0 wt % UO_2 or 27

vol % UO_2, 65.3 vol % BeO, and 7.7% void volume. Of the total void volume, 5.1% is present in the fuel and matrix, and 2.6% is present as a 2-μ gap between the fuel particles and the matrix. The oxygen-to-uranium ratio on three analyses varied from 2.002 to 2.004. The average density of the specimens is 92.3% of the theoretical density of 5.35 g/cm^3. The average diameter of the fuel particles in the sintered specimens is 166 μ.

STUDIES AND EVALUATIONS

Reactor Evaluation Studies. – The reactor physics characteristics of the G-E 630A maritime reactor were evaluated. The following items were considered: maximum-to-average power density as a function of position and time; maximum fuel surface temperature; fuel reactivity lifetime; reactivity control associated with the shim and safety rods; and reactivity associated with water-flooding of the fuel region. Based on the design specified in GEMP-108, the maximum fuel burnup corresponded to 3.85 at. % fission of the fuel meat; the associated fuel temperature was about 1050°F; the average burnup was 2.1 at. % fission with a temperature of 1500°F; the maximum fuel temperature was about 1650°F at a fission burnup of 1.85 at. % (inclusion of a hot-spot factor would increase this temperature to about 1800°F). The specified design had a reactivity lifetime of about 14,000 hr at rated power. It was found that with the reactor flooded and all rods in, the effective multiplication constant was greater than unity.

The gross radial power distribution can be flattened by appropriate changes in the distribution of moderator and fuel. Based on an initial uniform heat flux, a gross radial power factor of 1.05 (maximum-to-average power), and a fuel lifetime of 15,000 hr, the maximum fission burnup of the fuel meat would be about 3.45 at. %; the associated temperature would be about 1125°F. The maximum fuel temperature would be about 1550°F if no hot-spot factor is applied; the corresponding fuel burnup would be about 1.80%.

REACTOR FUELS AND MATERIALS DEVELOPMENT

Nondestructive Test Development. – A study is being made of coil-impedance variations for different coil-to-specimen spacings and oscillator frequencies. These variations are being studied as a function of the physical dimensions of the coil: length, thickness, diameter, and number of turns. The most accurate measuring system has proved to be a balanced-bridge impedance network, with the test coil comprising one leg of the bridge. The specimen materials have been carbon steel (relatively high permeability) and aluminum (relatively high conductivity and low permeability). With these materials a complete series of impedance measurements are being taken for a number of coils, with the coil being adjacent to a single metal specimen and with the coil sandwiched between metal specimens. In this manner, spacing measurements can be simulated for all values from minimum to infinite dimensions.

Normalized impedance curves for each of these conditions are being plotted. It is recognized that there are some inaccuracies in the curves, since a large number of variables are being studied with a

limited number of coils and since second-order effects are being neglected. It is believed, however, that reasonably accurate relationships have been noted between frequency and coil diameter and that approximate relationships have been noted between coil length and the impedance per unit spacing change.

These data will provide quantitative design criteria for new space-measuring systems and perhaps allow improvements in those which have been developed for such problems as coolant-channel spacing measurements for the HFIR, ATR, and Fermi fuel elements.

Mechanical Properties. — Creep-rupture testing of Zircaloy-2 tubes is in progress to determine the deformation and fracture characteristics of metals with strongly preferred mechanical anisotropy. The tubing was found to exhibit a strong intensity of $\{10\bar{1}0\}$ poles in the axial direction, strong intensity of $\{0001\}$ poles with an absence of $\{10\bar{1}0\}$ or $\{11\bar{2}0\}$ poles in the tangential direction, and a moderate intensity of $\{11\bar{2}0\}$ poles in the radial direction. In accordance with the basal pole concentration and the lack of $\{10\bar{1}0\}$ or $\{11\bar{2}0\}$ poles in the tangential direction, room-temperature properties and creep-rupture strength demonstrate a greater resistance to deformation in this direction. The general behavior under complex stresses at room temperature is similar to the high-temperature behavior except for short-time rupture life. The effect of the mechanical anisotropy can still be described by the Hill constants in the general equation

$$1 = H(\sigma_z - \sigma_\theta)^2 + F(\sigma_\theta - \sigma_R)^2 + G(\sigma_z - \sigma_R)^2 ,$$

where F, G, H = anisotropy constants and σ_z, σ_θ, σ_R = axial, tangential, and radial stresses. The constants vary with time at high temperatures as indicated above; however, they approach values equal to those determined from creep-rate measurements for rupture times greater than 1000 hr. The high-temperature values are H = F and G = 2.1H; the room-temperature values are H = F and G = 3.7H. The observation that the G/H ratio is lower at the high temperature indicates a less-pronounced mechanical anisotropy at the elevated temperatures.

Solid Reaction Studies. — The previously reported unusual temperature dependence of the diffusion coefficient of Zr^{95} in body-centered cubic (beta) zirconium has been shown to be described in terms of the absolute temperature of the alpha-to-beta transformation of 1136°K by the following equation:

$$D = 3 \times 10^{-6} \left(\frac{T}{1136} \right)^{15.6} \exp \left[- \frac{19{,}600 + 31(T - 1136)}{RT} \right] \text{cm}^2/\text{sec} .$$

Subsequent studies on the diffusion of Nb^{95} in β-zirconium and of Ti^{44} in body-centered cubic β-titanium have also revealed marked deviations from linearity in the usual Arrhenius-type plots. For the former, the apparent activation energy varies from 23,000 to 60,000 cal/mole between temperatures of 900 and 1750°C; for the latter the variations are from 30,000 to 44,000 cal/mole between temperatures of 900 and 1550°C.

Materials Compatibility Studies. – Existing data on the free energies of formation of niobium and potassium oxides suggest that niobium should getter oxygen from potassium. However, recent studies in which niobium containing 70 ppm oxygen was equilibrated at an elevated temperature with potassium containing 400, 1400, and 2400 ppm oxygen showed that no transfer occurred. In fact, the data indicated that oxygen was transferred from the niobium. Therefore, at 815°C, κ, the distribution coefficient for the Nb-O-K system appeared to be < 1, where

$$\kappa_{815°C} = \frac{C_{\text{oxygen in Nb}}}{C_{\text{oxygen in K}}} .$$

Additional experiments were then conducted to determine more quantitatively $\kappa_{815°C}$ for the Nb-O-K system. In the first test a niobium specimen containing 360 ppm oxygen was equilibrated for 100 hr at 815°C with potassium containing 1225 ppm oxygen. Analysis of the niobium after test showed it to contain 310 ppm oxygen. In the second test a niobium specimen containing 1300 ppm oxygen was exposed for 100 hr at 815°C to purified potassium containing 125 ppm oxygen. Final analysis of the niobium indicated an oxygen concentration of 110 ppm.

From the above data, calculations show that $\kappa_{815°C} \cong 1/4$ for the Nb-O-K system.

Fuel Element Development. – Postirradiation examinations of aluminum-base fuels containing 64 wt % UAl_3, 63 wt % U_3O_8, and 60 wt % UC_2 were culminated on completion of the x-ray diffraction studies conducted at BMI to identify the reaction products observed in the fuel samples after irradiation. In the x-ray studies, a disordered form of UAl_3 was identified as one of the reaction products in the irradiated U_3O_8-bearing dispersion fuel. Although previous studies had revealed the formation of UAl_3 in this system after long exposures at 1112°F, its formation had always been accompanied by a volume increase. No such volume increase was observed in this system. The absence of U_3O_8 and UO_2 in the x-ray pattern for the irradiated sample indicates that these oxides had become amorphous. The analyses of the irradiated UAl_3-Al dispersions showed no indication of the formation of UAl_4; its formation had been considered a possibility from the metallographic examination. However, the UAl_3 appears to have undergone an ordered-disordered transformation as a result of irradiation. No apparent changes were noted in the analyses of the irradiated UC_2-Al dispersions. The UC_2 and UC particles originally present in the fuel samples appeared to remain crystalline after a U^{235} fission burnup of 35 at. %.

REACTOR COMPONENTS DEVELOPMENT

Reactor Controls Development. – Development and testing are continuing on the new series of multisection ionization chambers. A developmental two-section chamber was fabricated for testing the concept

of a dual safety chamber having neutron- and gamma-sensitive sections. The performance of the gamma-sensitive section when installed in the ORR has been excellent, with good sensitivity and saturation characteristics. When the reactor is started up or shut down, the current from the gamma-sensitive chamber follows closely the behavior of all the neutron-sensitive chambers. The neutron-sensitive section of the developmental chamber will be built with lower sensitivity to suit the proposed permanent location of the chamber.

Further studies of pickup noise in pulse-counting systems for reactor startup and operation have led to minor modifications of the circuit of the flexible preamplifier assembly previously described (ORNL-3211, pp 13–14) and its grounding and shielding layout.

A major difficulty in pulse-counting systems is excessive pickup noise arising from the flow of noise currents in the electrical shielding of signal cables; capacitively coupled ground loops will inevitably exist even when the shields are disconnected from obvious grounds. In the laboratory, such instrument systems can be truly isolated from the building ground, but in a reactor installation this is not possible. The results of an extensive series of laboratory and field tests indicated that the least noisy layout used multiple electrical shields, connected to each other at several points along their length. This prescription is contrary to previously held ideas and will be further tested in the field.

POWER REACTOR FUEL PROCESSING

Shear and Leach Process. – Measurements of particle size distribution for a carburized (0.8% max carbon) ORNL Mark I fuel prototype assembly sheared into separate batches of 1/2-, 1-, and 1-1/2-in. lengths showed 26, 44, and 20% more particles, respectively, in the fraction < 9520 μ than for a noncarburized assembly. Particles in the smallest fraction measured, < 44 μ, were 16, 20, and 22% less, respectively, than for a noncarburized assembly. The total stainless steel in the fractions < 9520 μ was 18.5, 5.3, and 3.3% of the original weight, respectively, and compares with measurements of 2.7, 2.0, and 1.1% for noncarburized elements.

A Yankee-type fuel assembly of 25 porcelain-filled tubes (5/16-in. OD with 1/4-in.-OD spacer ferrules) sheared into lengths of 1/4, 1/2, 5/8, 3/4, and 1 in. had 81, 30, 18, 15, and 11 wt % of the particles < 9520 μ. The tubes of this assembly, which is smaller than the ORNL Mark I assembly, were compacted in cuts more than 1/4 in. long, and there was ~38 wt % of particles in the fractions < 4760 μ.

The center step of the Squarkeen No. 3 (American Shear Knife Company) moving blade was chipped on the cutting edge during the 5894th cut of a porcelain-filled ORNL Mark I fuel assembly. Failure occurred while the solid steel end plugs were being sheared. A replacement blade of Kleenkut, obtained from the Heppenstall Company as an equal to Squarkeen, failed at 320 cuts.

Processing Studies on Uranium and Thorium Carbides. – Hydrolysis of uranium sesquicarbide in water or 6 M H_2SO_4 at 80°C yielded 60 ml (STP)

of gas per gram of carbide. The gas consisted of 58% hydrogen, 25% ethane, 6% C_3 to C_8 saturates, and 6% unsaturates. Of the total carbon in the specimen, half was in the gas phase and 11% in the wax, leaving nearly 40% unaccounted for and presumably in the solution. Reaction of the uranium sesquicarbide with 6 M HCl resulted in a slight increase in the gas volume, quantity of free hydrogen, and amount of wax produced; the amount of ethane and butane decreased slightly. These small changes in reaction products when 6 M HCl is substituted for water or 6 M H_2SO_4 are probably caused by the dicarbide impurity in the sample.

Hydrolysis of thorium monocarbide, like that of uranium monocarbide, yielded principally methane (82%) with some hydrogen and small quantities of the higher hydrocarbons. The gaseous products were independent of reagent – H_2O, 6 M H_2SO_4, or 6 M HCl; virtually all the carbon was in the gas phase.

Solvent Extraction Studies. – In laboratory countercurrent batch extraction experiments with stored full-activity-level U^{233} solutions, gross gamma decontamination factors of the U^{233} product were at least 200; the product contained no Th^{228}. Solutions containing up to 30 ppm of Pa^{231} in 0.5 M Th–11 M HNO_3 showed no indication of instability two weeks after preparation. Adsorption of protactinium from the $Th(NO_3)_4$-HNO_3 system on unfired Vycor glass and elution with oxalic acid separated it from thorium, ruthenium, zirconium-niobium, and rare earths by factors of > 50, 4000, 3, and 4×10^5 respectively.

The relation for the instantaneous dissolution rate of 96%-theoretical-density UO_2 pellets in boiling 2 to 15.6 M HNO_3 is:

$$\log \text{rate} = 2.116 \log (\underline{M}\ H^+) - 0.684 .$$

The addition of 0.1 M HF–0.3 M $Al(NO_3)_3$ increased the rate in 2 M HNO_3 by 80% but in 14 M HNO_3 by only 15%.

Fluoride Volatility Pilot Plant. – Four more successful runs (R-3 through R-6) were made with 5- to 6.5-yr-decayed high-burnup (> 15%) zirconium-uranium alloy fuel (~44 to 46 kg per run). Analytical data from runs R-3 and -4 indicated that the bulk of the uranium decontamination from fission products continued to occur during fluorination and sorption-desportion on NaF. Maximum decontamination factors from volatile fission products during dissolution ranged from ~5 to 8 for Sb^{125}, and specific fission product activities in the UF_6 product were below analytical limits of detection. In run R-3 on 5-yr-decayed fuel, the Ru^{106} decontamination factor increased to ~10^8; all other specific decontamination factors remained essentially the same (~10^8 to 10^{10} for Cs^{137}, Sr^{90}, and rare earths; ~10^4 to 10^7 for Sb^{125} and Zr^{95}-Nb^{95}).

For the four runs, dissolution rates ranged from 2.4 to 5.4 kg/hr and HF utilization efficiencies from 27 to 55%. Fluorine utilization in conversion of UF_4 to UF_6 was only 2 to 5%. After the first run, uranium recovery was >99.5%. The UF_6 product contained < 900 ppm of impurities in runs R-1 to -4, but in R-5 an unexplained increase in the molybdenum level occurred (560 ppm in R-4 and 4300 ppm in R-5). The plutonium level was < 1 ppb for runs R-1 through R-3.

Equipment performance was generally satisfactory. In run R-3 the hydrofluorinator off-gas line became plugged during dissolution, and

indirect cleaning through the fuel-slug chute was required. The NaF pellet bed was re-formed with difficulty in this run, but agglomerated pellets were broken up by inserting a metal tape in a rod-out line. Excessive leakage of a fluorinator flange in run R-6 required above-normal inert-gas buffering for UF_6 containment.

In run T-11, which followed run R-6, the molten-salt portion of the dissolver was partially decontaminated by dissolution of a Zircaloy-2 simulated fuel element. Inert-gas buffering of the leaking vessel flange without excessive inleakage was demonstrated. The dissolver was partially decontaminated, with decontamination factors of ~3 to 40 in the vapor section and of ~1 to 3 in the salt section.

Metallographic examination of two 3/8-in. sched-40 INOR-8 pipes removed from inside the hydrofluorinator after run TU-11 showed a maximum corrosion rate of 0.12 mil per hour of HF exposure at a point 1.5 in. above the HF distribution plate near the bottom of the vessel. The pipes had been exposed to 20/43–17/41–22/58 mole % $NaF-LiF-ZrF_4$ for 1359 hr, including 503.4 hr of HF exposure time. Temperatures were 380 to 620°C and 490 to 675°C in the vapor and salt regions respectively. The accentuated interface attack noted during corrosion tests in the laboratory hydrofluorinator was not observed. Only slight intergranular attack was noted – on one pipe in the upper half of the vapor region.

THORIUM UTILIZATION PROGRAM

Planning and Evaluation Studies. – Comparative capital costs of three possible energy-conversion systems for the Molten-Salt Converter Reactor (MSCR) were obtained by Sargent and Lundy Engineers. Only costs associated with facilities outside the reactor proper were considered. The associated capital costs (in dollars per kilowatt of electricity) were about 66 for the direct power cycle (in which steam was generated in a boiler heated with fuel salt), about 73 for an indirect power cycle (utilizing an intermediate salt and a Loeffler-type boiler), and about 71 for a mercury binary cycle (in which mercury vapor was generated in a boiler heated with fuel salt).

Fuel-cycle costs for MSCR reactor types were calculated as a function of processing method and of processing-plant capacity. Use of fluoride volatility processing in an on-site facility (2 ft^3/day processing capacity) gave fuel-cycle costs of 1.33 mills/kwhr; use of a central facility (12 ft^3/day processing capacity) gave fuel costs of 0.99 mill/kwhr. Use of Thorex processing in the AEC multipurpose processing plant gave fuel costs of 1.00 mill/kwhr, while use of a single-purpose central Thorex facility (capacity, 1 ton of thorium per day) gave fuel costs of 0.88 mill/kwhr.

The equilibrium reactor program (ERC-11 for the IBM 7090), which calculates fuel costs for solid-fueled reactors operating on an equilibrium fueling cycle, was completed, and final checkout is in progress.

Testing of Thoria-Pellet and -Suspension Fuels. – It is planned to operate an in-pile pellet loop in the HN-1 facility of the ORR, possibly next spring. The purpose of the experiment will be to observe the effects

of irradiation on thoria pellets and their attrition products. The pellet bed is to be mixed by fluidization from time to time, simulating the use of such pellets in a breeder reactor blanket. A pellet-bed system suitable for the in-pile loop is being designed and developed.

A series of laboratory attrition tests was conducted to evaluate the attrition characteristics of an experimental batch (code P-140) of nominal 0.2-in.-diam spheroidal ThO_2 pellets which were of interest for use in the development of the in-pile pellet-bed experiment. Batch P-140 was a composite of three lots of pellets prepared in the Metals and Ceramics Division by milling cubes which had been pressed from batch DT-100 thoria.

Preautoclaved (72 hr in 260°C H_2O) pellets displayed overall weight loss rates of 0.88%/hr after 50 hr of milling and 0.26%/hr after 10 hr of spouting in standardized ball-mill and spouted-bed tests.

In fluidized-bed tests (1 × 1 in. static beds with tap water as the fluidizing medium), overall weight loss rates after 200 hr of fluidization were 0.013%/hr at 30% bed expansion (57% void fraction, 0.6 fps) and 0.036%/hr at 100% bed expansion (72% void fraction, 1.0 fps).

Engineering of Thorium Reactor Systems. – Forced-convection nucleate-boiling heat transfer measurements were made for aqueous thoria slurries flowing past a heated stainless steel test strip at velocities between 2 and 14 fps, pressures between 17 and 31 psia, and with slurry concentrations of 0 to 1900 g of ThO_2 per liter. The heat fluxes observed were the same or up to 30% greater than those observed with water at the same temperature, driving force, and velocity. This provides evidence that no slurry film is forming on the heat transfer surface during nucleate boiling.

These runs were extended to measure subcooled burnout heat fluxes over the above range of velocity, pressure, and concentration. Whereas the water burnout data agreed quite well with the literature correlations, the slurry data were up to 50% below the expected heat flux. Additional runs are under way in order to determine which slurry properties are responsible for the deviation.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. – Design of the MSRE was continued, with major effort on support structures for equipment and piping and on the electrical systems and instrumentation.

Drawings of reactor cell structures are now about 90% complete; detail drawings of the drain-tank furnaces are 50% complete; drawings for the 480-v switch gear are 40% complete. Electrical-heater schematics and conductor routing designs were completed. Instrument air application diagrams were issued. The auxiliary and transmitter control panels and panel cutout designs were completed. Elementary electrical schematics of the control circuits are 60% complete. A critical-path schedule was made for design, procurement, and installation of the electrical and instrumentation systems. Design of the containment vessel and the proposed tests were reviewed, and minor changes were made in the design of the vessel to satisfy code requirements for testing at 48 psi.

The tubing for the radiator and heat exchanger is being inspected at the fabricator's plant. Partial delivery was made on both the 3/4-in.-OD tubing for the radiator and the 1/2-in.-OD tubing for the heat exchanger.

Fabrication work on the components is progressing on schedule in the Y-12 Machine Shops. This work is approximately 40% complete.

The 7-ft extension for the reactor containment cell was delivered to the site, and its installation is 75% complete. Approximately 95% of the concrete in the drain-tank cell has been poured.

The concrete work for the filter pit, the stack, and the cooling tower was completed; the filter racks and the duct work in the filter pit are being installed. The cooling tower was delivered and is being installed.

Chemical Development. — MSRE fuels which contain UF_4 but no ThF_4 are being studied. Careful exploration of the liquidus contours in the system $LiF-BeF_2-ZrF_4$, the solvent for the MSRE fuel, shows that if the ZrF_4 concentration is held at 4 mole % the maximum LiF concentration is 67 mole % when the liquidus is to be at 450°C or below. Both the vapor pressure and the viscosity should decrease as the LiF concentration of the melt is increased. Accordingly, if the ZrF_4 concentration is maintained at 4 mole %, the optimum fuel solvent almost certainly contains 67 mole % LiF and 29 mole % BeF_2; fuels of uranium content below 0.25 mole % would be formed by decreasing the LiF by an amount equal to the UF_4 added. In addition, it is likely that the vapor pressure and viscosity would be improved slightly if the ZrF_4 concentration was reduced while the LiF concentration was held constant. If adequate protection against oxygen contamination is obtained at lower ZrF_4 concentrations, an appreciable improvement in these two properties and in the liquidus temperature should be provided by a fuel such as 67-30-2.85-0.15 mole % of $LiF-BeF_2-ZrF_4-UF_4$.

Recently available graphite of the type that will be used in the MSRE, and which is being used in the Engineering Test Loop, was permeated to the extent of 0.5 wt % by MSRE fuel under 75 psi. This meets MSRE specifications.

The desorption characteristics of unclad graphite moderator were studied, under conditions similar to those expected to exist during the initial heatup of the reactor, with a 6-in. length of AGOT graphite having the same cross-sectional configuration as MSRE moderator elements. The temperature of the graphite was raised to 700°C at a programmed rate of 30°C per hour. Two experiments on the desportion of water were performed: the first with the graphite as-machined and the second with the same graphite specimen after it had been exposed to helium vapor saturated with water at room temperature. In both experiments, two distinct desorption peaks were found: an evolution of what was assumed to have been physically adsorbed water at an initially high rate which decreased to a minimum value by the time a temperature of 200°C was reached, and an evolution of what was assumed to have been chemically adsorbed water in the temperature region of 250 to 450°C. No significant quantities of water vapor appeared in the circulating gas stream at higher temperatures. Further experiments are in progress with MSRE-grade graphite.

Preparation of MSRE Materials. – The production of purified fluoride mixtures for MSRE operations is scheduled to begin in September 1962. Modifications to the production plant are near completion; they include the addition of a premelting furnace assembly and the relocation of the raw-material handling facility.

The status of the procurement of raw materials for production operations is as follows: About 5000 kg of isotopically pure Li^7, as lithium hydroxide, has been selected and set aside for MSRE use. This material is being converted to lithium fluoride at Y-12. Tentative chemical and physical specifications have been determined for the purchase of some 10,000 lb of beryllium fluoride. Sufficient hafnium-free zirconium tetrafluoride (2300 lb) was received to prepare one fuel loading for the MSRE.

Radiation Effects. – Analyses for corrosion products in additional samples from fuel irradiated in graphite boats (test 47-3) gave results different from those previously reported. The corrosion products, sought as evidence of attack on coupons of INOR-8 and of molybdenum, were later found by improved analytical procedures to be present at a concentration of about 100 ppm, rather than several hundred or 1000 ppm. Such results for Cr, Fe, and Ni are representative of reducing conditions and still have some puzzling aspects in that no differences are discernible with capsules that produced lesser amounts of CF_4 and thus should have been much less strongly reducing. Additional evidence of the reduced nature of the fuel was found in the absence of molybdenum (below the 50-ppm limit of detection) in spite of an obvious attack of the molybdenum coupons and of the molybdenum produced as a fission product; the implication is that molybdenum deposits as a solid and does not remain in the fuel.

Further attempts to manipulate the pyrolytic graphite coupons from irradiation test 47-3 have confirmed the suspicion that the exposure caused some of the specimens to crumble rather easily; the reason for this deterioration will be sought in further examinations of the pieces.

Irradiation Tests. – Experiment ORNL-MTR-47-4 (see ORNL-3282), designed to determine if CF_4 exists in the cover gas over MSRE fuel in contact with submerged graphite, was irradiated for 12 weeks, removed from the MTR on June 4, 1962, and returned to ORNL for examination. Total irradiation time was 1550 hr, and about 95% of this time was at full power. The maximum measured temperature among the four instrumented capsules was maintained at 1400 ± 50°F. The mean measured temperatures of the other three instrumented capsules were ~1380, ~1370, and ~1310°F respectively. These temperatures, according to the design temperature distribution obtained by use of TØSS (see report K-1494) on the IBM 7090 computer, are essentially the graphite-to-salt interface temperatures. The corresponding calculated INOR-8 capsule wall-to-salt interface temperatures are 1130, 1125, 1115, and 1100°F. The temperature of about 12% of the INOR-8 surface – one end cap in contact with the fuel salt – was calculated to be the same as the temperature at the graphite-to-salt interface.

The temperature history of the in-pile experiment is being duplicated with out-of-pile control capsules. The temperature history of the uninstrumented crucible capsules was calculated on the basis of temperature measurements in the instrumented capsules and the surrounding liquid sodium.

In the next experiment, ORNL-MTR-47-5, a helium purge is to be used to sweep CF_4 and/or other reaction products to a sampling station for collection and analysis. The experimental equipment has been designed and is about 50% constructed. The capsules to be irradiated have been fabricated and loaded with fuel salt. The off-gas collection system design has been verified by bench test.

Analysis of Radioactive MSRE Fuels. – The Analytical Mockup Hot Cell for the evaluation of methods of analysis of the MSRE fuel has been erected, and most of the service piping has been installed. Cell equipment is being designed to remove the sample-filled ladle from the transport container and to reload the transport container with an empty ladle.

A copper pulverizer-mixer was designed for homogenizing the fuel sample. The copper ladle that contains the solidified fuel is placed in a pulverizer-mixer, which is agitated on a mixer mill. The fuel is removed from the ladle by fracturing, pulverized into a homogeneous powder, and transferred to a storage bottle. The homogenized fuel sample is then available for analysis. A detailed report about this pulverizer-mixer has been written (ORNL TM-291).

Procurement, fabrication, and design of the analytical equipment necessary for analyzing the sample in the cell are also in progress.

ADVANCED REACTOR DEVELOPMENT

Advanced Test Reactor Corrosion Program

The effect of pretreatment on corrosion and on corrosion-product accumulation on aluminum through which heat is being transferred has been investigated. The three pretreatments examined were: exposure to a dilute sodium dichromate solution at 150°C for 30 hr, "alodizing" (a chemical chromate-phosphate conversion coating), and anodizing. All treated specimens were subjected to a heat flux of 1.5×10^6 Btu hr^{-1} ft^{-2} and were cooled with water adjusted to a pH of 5 with nitric acid. No beneficial effect of any of the pretreatments was observed; the rates of corrosion-product buildup and of corrosion of the aluminum were essentially the same as those observed with as-machined specimens exposed under the same conditions.

QMV beryllium specimens have been exposed to water (pH 5.0, 4 ppm oxygen) at 100°C for 1300 to 1560 hr. Corrosion rates have been relatively constant at 1.7 to 2.5 mils/yr and independent of velocity in the range 22 to 81 fps. The results indicate that corrosion of the beryllium reflectors in the HFIR and the ATR will not present a serious problem, but the tests are being continued in order to determine whether the corrosion rate changes with prolonged exposure.

High Flux Isotope Reactor Design and Development

Metallurgy. – Proposals to manufacture HFIR fuel elements under a cost-plus-fixed-fee arrangement rather than on a lump-sum basis have

been requested of commercial vendors. This change in the procurement plan was brought about mainly by the lack of industrial experience in the making of fuel plates with a graded fuel distribution to the close specifications required. In addition, commercial vendors lack experience and equipment for the nondestructive inspection for homogeneity of U^{235} in the fabricated fuel plates. As a consequence of the latter point, efforts to scan for homogeneity by x-ray attenuation have been significantly increased; the objective is the development of a pilot model and later a production model for rapid nondestructive testing of fuel plates in the very small local areas required by the specifications.

Destructive chemical analyses of the uranium in the fuel-bearing portion of the inner-annulus composite plates have indicated regions 55% higher than the nominal concentration. These segregated regions in the thin portion of the fuel core are thought to be caused principally by the large particles in the U_3O_8 powder gradation used. It is planned to investigate the use of a more restricted particle-size distribution.

Re-forming of composite fuel plates with improved Marforming equipment in which the rubber is completely contained has been encouraging. Fuel plates have been re-formed to tolerances only obtained previously when unfueled aluminum stock was used.

An advanced concept in making HFIR fuel elements is to manufacture segments containing 14 to 16 plates and to join the segments together into an annulus element. A 16-plate segment containing unfueled plates has been assembled and mechanically joined to well within the specifications.

Composite plates containing 37% Eu_2O_3 or 37% tantalum dispersed in aluminum showed no dimensional changes or chemical reactions in preliminary compatibility studies. Plans have therefore been laid to develop the neutron-absorber components for the control rod with these materials being used for the "black" and "gray" regions.

Design and Analysis. – The analog transient hot-spot analysis of the HFIR fuel element has been primarily concerned with evaluation of the ability of various control systems to protect the reactor during ramp reactivity insertions. The method of approach to this problem has been to examine the capabilities of the proposed HFIR control system and then investigate seemingly feasible modifications to this system. Considerable effort was devoted to checking out the transient-analysis model to assure that it is applicable to the cases under consideration.

Some thermal deflection experiments have been run on a 50-in.-long mockup of the HFIR outer nickel control plate. The experiments thus far indicate that the maximum axial deflection of this plate is about 0.8 mil/°F and the maximum radial deflection is 0.6 mil/°F.

A new code for the IBM 7090 computer has been started which will predict the flow distribution, pressure distribution, and the coolant-water temperature in the HFIR control region. This code will consider all three cooling channels. Use will be made of the experimental and calculated results obtained previously.

Equipment is being designed to position the three ion chambers in the thimbles provided in the pressure vessel. The equipment will support the ion chambers, protect the cables in the pool, and allow for adjustment of chamber position.

Assembly drawings were prepared which show the current concept of the reactor components and experimental facilities. The major changes from the previous concept are in the target assembly, outer shroud, control-rod access plugs, and the vertical experimental facilities.

Instrumentation. – The package II instrument drawings were completed, and the bid invitations were submitted to vendors with an August 10 closing date. This group of equipment includes the process panel boards, the control console, and the relay cabinets, all with the necessary equipment, and the nuclear instrument cabinets without equipment.

Engineering Development. – The assembly of the HFIR mockup is 90% complete. All major components due from vendors are on hand. The installation of instrumentation such as control-rod drives, position indicators, strain gages, and flow-measurement piping is 90% complete. The mockup is now scheduled to begin operation during the week of August 6, 1962.

Critical Experiments. – Additional control-rod calibrations by the pulsed-neutron method were made in the critical experiment for three delayed critical positions obtained with different boron concentrations in the moderator water. These calibrations extended over displacements where the control-plate sensitivities were a minimum. The safety cylinder was also calibrated for these conditions. The reactivity effect of a cadmium cylinder between the fuel-element annuli was measured as a function of insertion distance. These data were necessary to evaluate the adequacy of the present control-plate design and to demonstrate the feasibility of alternative concepts. The control plates have been modified to eliminate the "gray" region, and more control-plate studies are in progress to evaluate the benefit in terms of gain in control-rod sensitivity and the loss in terms of peak-to-average heat flux.

WASTE DISPOSAL DEVELOPMENT

Foam Separation Studies. – Calcium precipitation is the first step in new ion exchange and foam separation processes for decontaminating waste water. Beaker tests showed that when precipitation is made from 0.01 M NaOH, then sodium hexametaphosphate, sodium tripolyphosphate, tetrasodium pyrophosphate, sodium orthophosphate, Fab, and Turco 4234 in concentrations of 1 to 3 ppm strongly interfered; fluoride and oxalate ions do not interfere. Phosphate interference was nearly eliminated by making the water 0.005 M each in NaOH and Na_2CO_3 and 1 to 5 ppm in Fe^{3+}, pH 11.3. With 0.01 M NaOH instead of 0.005 M, the treatment was less effective. In a 100-hr continuous precipitation run with ORNL low-activity waste, containing ~3 ppm of phosphorus as PO_4^{3-}, the total average dissolved hardness was decreased from ~70 ppm to 4.7 ppm, as $CaCO_3$.

Beaker tests also showed that phosphate interference in calcium precipitation can be eliminated by using orthophosphate, 40 to 50 ppm, in 0.004 M NaOH as the precipitant. In a continuous run with precipitation from 0.004 M NaOH–50 ppm PO_4^{3-}, the hardness was decreased from ~70 ppm to 5.8 ppm, as $CaCO_3$.

Values of heights of transfer units for foam-separation stripping of Sr^{89} from a dodecylbenzenesulfonate solution varied from 2.2–2.6 cm at 130–160 gal ft^{-2} hr^{-1} to 4–10 cm at 80–110 gal ft^{-2} hr^{-1} with a weir-type liquid-feed distributor. Values were lower with a spider-type distributor of seven capillary tubes, but the difference was small at the higher flow rates.

Application of the χ^2 statistical test confirmed the fit of normal distributions for foam bubble diameters from spinnerettes with 50- or 80-μ-diam holes; however, normal distributions are rejected for the bubble diameters from porous fritted glass or sintered-metal gas spargers. Correct average diameters for calculating the foam surface area were derived from these normal diameter distributions.

PHYSICAL RESEARCH PROGRAM

Reactor Operations. – During the first 27 weeks of 1962, the ORR was in operation 82.3% of the time. Installation of a second off-gas system for ORR experimenters was completed. The new system can be pressurized to 90 psi.

Reliability of the ORR building emergency ventilation scrubber was increased markedly by making a number of modifications, including the circulating of water through the scrubber continuously.

Waste Disposal. – The filling of pit No. 3 was completed. The design of trench No. 7, which will replace one or both of the other two open pits, was also completed. The new trench is scheduled to be put into operation in October of this year.

The main components of the liquid-waste-monitoring system have been installed and are operating satisfactorily except for minor details. The installation of the 3039 stack monitoring system is scheduled for completion by August 1, 1962.

Hot-Cell Operations. – Operation in the hot cells was routine, and the backlog has been brought to a current basis. Examination of fuel element S-81 from the SM-1 reactor at Fort Belvoir was begun. One UO_2 meltdown experiment was disassembled and examined. Examination of ORR and LITR capsules containing UC_2 and UO_2 fuels continued. Experiment ORNL 41-7, containing BeO pellets irradiated in the MTR, was disassembled and examination was essentially completed.

PHYSICS AND MATHEMATICS RESEARCH

Neutron Diffraction. – Neutron diffraction studies on rare-earth yttrium alloys have been undertaken in order to investigate further the nature of the exchange interactions responsible for the magnetic ordering of the pure rare-earth metals. A preliminary survey of Tb-Y alloys shows that the Néel temperature decreases linearly, or nearly so, with Y concentration; the Curie point falls rapidly with concentration. An alloy 70% Tb–30% Y retains the antiferromagnet helical structure to 4.2°K. The minimum values of the interlayer turn angle in the helical structures show a more or less regular increase with increasing Y concentration. On simple theoretical models the value of the interlayer turn angle is related to the ratio of 2d to 3d neighbor interactions. Introduction of just 10 at. % Y into Dy eliminates entirely the Curie point for the spontaneous transformation of the helical structure to a ferromagnet observed in pure Dy.

Theoretical Physics. – The computer programs, previously described (ORNL-3240), for calculations employing the distorted-waves theory of direct nuclear reactions, have been used to study a variety of reactions:

1. Inelastic scattering. It has been found that the differential cross sections for strong transitions in the inelastic scattering of neutrons, protons, and alpha particles are well described if the nonspherical potential well interaction suggested by the nuclear collective and "deformations" obtained from Coulomb excitation measurements is used.

2. Quasi-elastic scattering. The strong (p,n) transitions to states which are the analog of the target ground state have been studied by using the isotopic-spin dependence of the optical potential suggested by Lane [Phys. Rev. Letters 8, 171 (1962)]. The model was also extended to (He^3,t) reactions. Reasonable agreement with experiment is obtained in both cases.

3. Stripping and pickup reactions. The optical potentials obtained from analysis of elastic deuteron scattering are being applied to the calculation of deuteron reactions, especially (d,p) and (p,d). Typical of the questions being studied is whether contributions from the nuclear interior should be included.

In addition, the effects on various reactions of including spin-orbit coupling in the optical potentials are being investigated.

Fast-Chopper Time-of-Flight Spectrometer. — The ORNL fast-chopper time-of-flight neutron spectrometer was utilized to make transmission measurements on isotopically enriched samples of Zr^{90} (97.8% enrichment), Zr^{91} (90.9%), Zr^{92} (95.7%), Zr^{94} (96.5%), and Zr^{96} (57.3%). In the energy range from ~0.5 ev to 40 kev, three resonances were observed in Zr^{90}, five in Zr^{92}, five in Zr^{94}, and about seven in Zr^{96}. Eleven resonances were resolved in Zr^{91} up to 3000 ev, and an s-wave strength function of $(1.0 \pm 0.3) \times 10^{-4}$ is assigned to Zr^{91}. A new weak resonance was observed in Zr^{91} at ~450 ev in these transmission measurements; this level was also observed in capture measurements with the Rensselaer Polytechnic Institute linear accelerator. It is interesting to note that of the seven resonances observed in Zr^{91} below 1600 ev, four had a Γ_n^0 of less than 3% of the average Γ_n^0 for all seven levels. This indicates that some of the small resonances in Zr^{91} may be due to p-wave neutrons.

Low-Temperature, Nuclear and Solid State Physics: Angular Distribution. — Previous studies (ORNL-2910, p 60) of the angular distribution of fission fragments from thermal-neutron-induced fission of aligned U^{235} and U^{233} in the temperature region 77 to 1.3°K have been extended to 0.45°K; the study of resonance-neutron-induced fission has also been made. Such angular distributions are primarily determined by the K quantum number values involved in passing over the barrier.

The attainment of nuclear alignment at 0.45°K was accomplished with a continuously circulating He^3 refrigerator and the use of He^3 heat exchange gas to cool the single-crystal sample. Low counting rates required development of an automated counting and sample-rotating apparatus. Monochromatic neutrons were obtained from a beryllium crystal monochromator at the ORR.

The results obtained thus far are as follows:

1. The anisotropy for 0.14-ev neutron-induced fission of U^{235} is found to be consistent with an angular distribution given by

$$W(\theta) = 1 + \frac{0.0495 \pm 0.0025}{T} P_2(\cos\theta) .$$

Within the accuracy of the old results, this agrees with the results previously obtained (ORNL-2910, p 60) for U^{235} with reactor spectrum neutrons from the ORNL Graphite Reactor.

2. The 1.14-ev fission resonance in U^{235} gives an equivalent angular distribution $W(\theta) = 1 + \frac{0.044 \pm 0.006}{T} P_2(\cos\theta)$, which also agrees with the thermal-neutron effect within the accuracy of the results.
3. First results for the 8.8-ev resonance in U^{235} suggest that there may be a rather different angular distribution. If this is correct, it would suggest a different J value for this resonance, together with different K values.

In addition to the preceding results, previous measurements (ORNL-2430, p 50) of the anisotropy of alpha-particle emission from U^{235} have been extended from 1.2 to 0.45°K. The alpha-particle angular distribution obtained from these results is $1 - \frac{0.074 \pm 0.002}{T} P_2(\cos\theta)$, which agrees with the earlier data within the accuracy of the earlier data.

Rather surprisingly, no nuclear spin lattice relaxation effects were observed, even at the lowest temperatures.

Low-Temperature, Nuclear and Solid State Physics: Mössbauer Effect. — Further measurements, and analyses of previous results, are reported on the Mössbauer effect for the 77-kev excited state in Au^{197}. From the results of transmission experiments on four metallic gold absorbers ranging in thickness from 44.5 to 1410 mg/cm^2, the natural line width of the 77-kev state in Au^{197} was determined. This corresponds to a half-life for this state of $(1.93 \pm 0.10) \times 10^{-9}$ sec, in agreement with a direct measurement by Sunyar [Phys. Rev. 98, 653 (1955)]. Also determined was the recoilless fraction in the source (consisting of Au in Pt), $f = 0.27 \pm 0.03$, and in the gold absorber, $f' = 0.14 \pm 0.015$. The value for f' is dependent upon a theoretical estimate for the conversion coefficient of the 77-kev state, $\alpha = 3.96 \pm 0.14$, by Shirley et al. [Phys. Rev. 123, 816 (1961)]. These values for f and f' at 4.20°K may be compared with values calculated from the Debye model, $f = 0.30$ and $f' = 0.18$, by using $\theta_{Au} = 164$°K and $\theta_{Au\ in\ Pt} = \theta_{Pt} = 233$°K.

A partial resolution of the six-line hyperfine spectrum of the 77-kev gamma ray has been seen by using absorbers consisting of ½% Au in Fe and 1% Au in Co and Ni. Analyses of these spectra have made possible a determination of the 77-kev state magnetic moment as $\mu = +0.38 \pm 0.08$ nuclear magneton. For all the magnetic moments yet seen, this moment appears to be farthest from the Schmidt limit. The hyperfine structure coupling is found to be proportional to the magnetic moment/atom of the host ferromagnetic metal, with a constant of proportionality of $(13.1 \pm 0.3) \times 10^{-3}$ cm^{-1}/Bohr magneton. This hyperfine structure coupling is equivalent to an effective magnetic field H' at the gold nucleus. For Au in Fe, Co, and Ni, the field H' is 1.46 ± 0.16, 1.18 ± 0.12, and 0.53 ± 0.16 megagauss respectively.

High-Voltage Experimental Program: Excitation of Collective States by Inelastic Scattering of 14-Mev Neutrons. — Neutrons with an energy of 14 Mev from the (d,t) reaction have been used to excite collective states in Fe, Sn, Pb, and S. Pulses of deuterons with widths less than 2 nanoseconds and with a current intensity greater than 3 ma were obtained by a combination of beam chopping and klystron bunching. Besides the elastic peaks, the neutron spectra contain peaks which correspond to excitation

of the 0.84-Mev quadrupole state in Fe, the 1.15-Mev quadrupole and 2.3-Mev octupole states in Sn, the 2.5-Mev octupole and 4.1-Mev hexadecapole states in Pb, and the 2.2-Mev quadrupole state in S. The absolute differential cross sections for excitation of these states have been determined and compared with the direct interaction theory of Bassel, Drisko, and Satchler. A value of 0.12, which is extracted from this comparison for the quadrupole deformation parameter of Sn, is in good agreement with the value of 0.11 found from Coulomb excitation studies.

High-Voltage Experimental Program: Scattering and Reactions by Charged Particles. – Compound states of high excitation in Ne^{22} have been observed in the total neutron yield by bombardment of O^{18} with alphas and detection of the neutrons in 4π geometry with the graphite sphere. Twenty-five resonances were observed, varying in width from 5 to 140 kev, for alpha bombarding energies from 2.4 to 5 Mev (excitation energy in Ne^{22} from 11.7 to 13.8 Mev). Absolute cross sections were measured. To aid in the analysis of the level parameters, differential elastic scattering cross sections for alphas on O^{18} were obtained at center-of-mass angles of 90, 125, 140, and 152° for alpha energies from 2.4 to 3.4 Mev.

CHEMISTRY RESEARCH

Ion Exchange Studies. – Specific conductivity and pH data obtained as a function of irradiation time for solution effluent from columns of Dowex 50W-X8, -X12, -X16, and -X20 resin exposed in a Co^{60} gamma-radiation source in a system of flowing demineralized water should be calculable from each other, if other, as yet unidentified, radiation products do not affect them. Identified radiation products in the effluent included sulfate, sulfonate, and oxalate ions in the acid form. Published specific conductivity data suggest that, in the concentration range of interest, 10^{-3} to 10^{-5} N, ionization of these acids is essentially complete. Experimental data indicate that the three acids found in the effluent constitute the majority of the ionizable radiolysis products. Maximum specific conductivity in each run was 400 to 500 micromhos/cm, which corresponds to a calculated pH of ~3. The minimum pH was 2.7 to 2.8. Sulfur determinations on the resin samples and the effluent gave a G(–S) of 0.5 to 1.0 atom of S per 100 ev of energy absorbed. This corresponds to 50–100 micromhos/cm, or 10 to 25% of the maximum specific conductance to be accounted for. Thus organic radiation degradation products of the resin polymer appeared to contribute the major amount of acidity.

Chemical Engineering Research. – The flow capacity of sieve-plate extraction-scrub pulsed columns for a flowsheet in which di-sec-butylphenylphosphonate in diethylbenzene is used to separate uranium from thorium was 490 to 1340 gal ft^{-2} hr^{-1}, increasing with decreasing pulse frequency from 90 to 35 cpm. The flow capacity for the stripping column increased from 380 to 730 gal ft^{-2} hr^{-1} as the pulse frequency was decreased from 70 to 35 cpm.

Solvent Extraction Research. – Preliminary tests indicated the possibility of separating transplutoniums from lanthanides by extraction with

dialkylphosphoric acids from organic acid solutions. In extraction by di(2-ethylhexyl)phosphoric acid from tartaric acid, americium and curium were shifted from their usual similarity to the medium-weight lanthanides to positions below cerium. Neither transplutoniums nor lanthanides were extracted from K_2CO_3 and K_2CO_3-$KHCO_3$ solutions by amines or organophosphorus extractants. Hydrazine hydrochloride prevented oxidation of Ru(III) to Ru(IV) by air or H_2O_2, so that ruthenium was not extracted by tertiary amines from concentrated lithium chloride solutions.

In further studies on extraction of metal ions from 0.5 to 10 M HCl and LiCl–0.2 M HCl (0.5 to 10 M total Cl) solutions with typical primary, secondary, tertiary, and quaternary amines in diethylbenzene diluent, the extraction power was in the order quaternary > tertiary > secondary > primary. Maximum coefficients (0.1 M amine) were 1.5 for Ti(III), 3 for Ti(IV), 60 for Mn(II), 600 for Ca(II), 0.03 for Ni(II), 100 for Cu(II), 2500 for Zn(II), and 2 for As(III). Extraction coefficients generally increased with increased chloride concentration and were higher from the salt than from the pure acid system.

Continued studies of strontium extraction from sodium nitrate solutions by di(2-ethylhexyl)phosphoric acid (D2EHPA) and its sodium salt (NaD2EHP) in benzene confirmed that $E_{Sr} = [Sr]_{org}/[Sr]_{aq}$ is proportional to $[H^+]^{-2}$ with up to 15% of the reagent as NaD2EHP. At higher NaD2EHP fractions, E_{Sr} was proportional to $a^{\pm -2}_{NaNO_3}$ ($a^{\pm}$ = mean ionic activity) up to $a^{\pm}_{NaNO_3} = 1.5$ (~3.5 M $NaNO_3$), changing gradually to $a^{\pm -1}_{NaNO_3}$ at higher $NaNO_3$ concentrations. These results suggest the general equation

$$Sr^{2+} + (n/y)(ZR)_y \rightarrow SrR_2 \cdot (ZR)_{n-2} + 2Z^+ ,$$

where Z = H or Na and R = di(2-ethylhexyl)phosphate. On the basis of this equation, reagent dependence for this system has been reexamined at several constant NaD2EHP fractions by plotting log $[H^+]^2_{aq}\ E_{Sr}$ vs log total reagent concentration. The slopes of the lines obtained (n/y in the above equation) varied from ~3 at 100% HD2EHP to 0.95 at 100% NaD2EHP. This suggests an initial reaction $Sr^{2+} + 3(HR)_2 \rightarrow SrR_2 \cdot 4HR + 2H^+$, since HD2EHP is a dimer. Diminishing values of n/y may reflect changes in n or y or both as more of the reagent is converted to the sodium salt.

Nuclear Chemistry: Neutron Cross Sections. – The neutron cross section of the 2.6×10^6-yr Bi^{210} to form the 2.16-min alpha-emitting Bi^{211} (AcC) was measured in the pneumatic tube facility of the ORR by use of a cadmium filter technique. Quantities of Bi^{210} ranging from about 3 to 120 mμg were irradiated for times up to 4 min at thermal and resonance fluxes per unit ln E of 6×10^{13} and 0.26×10^{13} neutrons cm^{-2} sec^{-1} respectively. A dilute alloy of manganese in aluminum was used to monitor the irradiation. The thermal cross section and the resonance integral were found to be 0.054 ± 0.05 and 0.20 ± 0.02 barn respectively.

Electrochemical Kinetics and Its Application to Corrosion. – The dissolution of zirconium in aqueous solutions containing HF was investigated by use of electrochemical techniques. Rates of dissolution were estimated by application of an oscilloscopic pulse technique, and pertinent electrochemical constants have been determined. The overall rate of reaction is

controlled almost completely by the diffusion of undissociated HF molecules to the surface. The rate of the reaction in unstirred solution at 25°C is given by the expression, rate (amperes/cm^2) = 6.5×10^{-3} (HF), where (HF) is the concentration of undissociated HF in moles/liter. The cathodic reaction is first order with respect to hydrogen ion concentration, and Tafel slopes vary from 85 to 110 mv/decade. Anodic Tafel slopes and capacity measurements indicate the presence of a film and suggest that the dissolution reaction proceeds through continuous formation and dissolution of the film.

Physical Chemistry of Molten Salts. — In a determination of the phase relationships in the system CsF-ZrF_4, a compound of composition $3CsF \cdot ZrF_4$ was obtained, and single crystals of suitable size for x-ray diffraction studies were isolated. Single-crystal precession photographs were used to ascertain the unit cell and possible space groups, and from this information several features of the structure were inferred.

Cesium heptafluorozirconate is face-centered cubic, a = 9.70 ± 0.02 A, with four formula weights of Cs_3ZrF_7 per cell. The x-ray density is 4.53 g/cm^3, quite close to that estimated by assuming that 3CsF and $1ZrF_4$ mix without volume change, namely 4.62. The diffraction symmetry is m3mF ..., for which the possible space groups are $F\bar{4}3m$, F43, and Fm3m. The Zr^{4+} and Cs^+ ions are readily placed in fourfold and fourfold plus eightfold positions of either of these space groups. However, there are no positions of appropriate multiplicity to accommodate the F^- ions; so it was concluded that there is some disorder in their arrangement that causes them to be statistically distributed over certain, as yet undetermined, space-group positions of high multiplicity.

Oxygen-17 Pilot Plant. — Preoperational testing of the water distillation cascade was completed. The unit is now "on stream." Barring mechanical failures, the cascade is expected to reach steady-state operation during the summer or fall of 1963. Projections of the cascade's separative capacity, based upon transient isotopic gradients, are favorable; it appears that design performance will be attained.

High-Temperature Aqueous Solution Chemistry: Liquid-Liquid Immiscibility and Critical Phenomena in the Condensed Systems UO_3-CuO-SO_3-D_2O and UO_3-NiO-SO_3-D_2O. — Investigations were essentially completed of liquid-liquid immiscibility and critical phenomena in the condensed systems UO_3-CuO-SO_3-D_2O and UO_3-NiO-SO_3-D_2O. These studies were carried out by the synthetic method at temperatures between 260 and 450°C and at concentrations of SO_3 varying from 0.02 to 1.0 m. The results were combined with data obtained previously by the direct sampling procedure, and three-dimensional models were constructed to show the change of phase boundaries with variations in the temperature or the concentration of any component.

High-Temperature Aqueous Solution Chemistry: Effect of Hydrostatic Pressure on Liquid-Liquid Immiscibility; the System UO_3-SO_3-D_2O. — In a further investigation, the effect of hydrostatic pressure on the temperature of liquid-liquid immiscibility was determined for 11 aqueous, stoichiometric UO_2SO_4 solutions varying from 0.14 to 4.5 m in concentration and at pressures up to 300 atm. This same effect of pressure on liquid-liquid immiscibility was determined for UO_3-SO_3-D_2O solutions, 1.0 m in SO_3, at a molal ratio, R_U (UO_3:SO_3), of 0.2. A value for $\Delta t/\Delta p$ of ∓ 0.08°C

per atmosphere for stoichiometric solutions, as observed preliminarily (ORNL-3256, p 23), was found near 300°C, and +0.4°C per atmosphere was found near 400°C for the solutions of $R_U = 0.2$. These values correspond approximately to the values of $(\partial t/\partial p)_d$ for H_2O and thus may indicate a first-order effect of solvent density on liquid-liquid immiscibility.

High-Temperature Aqueous Solution Chemistry: Electrical Conductance at High Temperature and Pressure. — In a continuing study of the electrical conductivity of dilute aqueous solutions of K_2SO_4 at temperatures up to 800°C and at pressures up to 4000 bars (ORNL-3256, p 24), the equivalent conductances, at constant temperature and molality, were plotted against the density of the solutions. These isothermal plots were made by using experimental data obtained at temperatures varying from 300 to 800°C and corrected for the conductance of H_2O. It was found that the equivalent conductance for a solution of constant molality increases rapidly to a maximum at a density between 0.6 and 0.7, and then decreases slightly at higher densities. At densities between 0.5 and 0.9 the equivalent conductance was essentially independent of temperature. The equivalent conductances at the maximums were about 900, 800, and 750 cm^2/ohm for 0.000467, 0.00218, and 0.00467 m K_2SO_4 solutions, respectively, or about five times as large as the room-temperature values. In a comparison of the data with those for another electrolyte, it was found that at temperatures near 500°C, the equivalent conductance of an H_2SO_4 solution, experimentally determined in this program, is about one-half that of a K_2SO_4 solution at the same concentration. This indicates that the HSO_4^- ion is only slightly ionized under these conditions and that in an H_2SO_4 solution there are only H^+ and HSO_4^- ions, whereas in a K_2SO_4 solution K^+, OH^-, and HSO_4^- ions are present.

Chemical Applications of Nuclear Explosions. — In further studies on the mechanism of the reduction of sulfates by hydrogen, thermal decomposition of $CaSO_3$ and $MgSO_3$ showed that these compounds were likely intermediates. Eighty to 90% of the $MgSO_3$ formed MgO and SO_2; the rest formed $MgSO_4$ and free sulfur. The large amount of SO_2 formed by decomposition of an $MgSO_3$ intermediate would lead to large quantities of free sulfur and H_2S in hydrogen reduction of $MgSO_4$, and such was observed.

Ten percent of the $CaSO_3$ formed CaO and SO_2; the remaining 90% formed $CaSO_4$ and CaS in a 3/1 mole ratio. There was no free sulfur. When the $CaSO_3$ mole ratio of SO_2/CaO is 1, the compound undergoes autoxidation:

$$4CaSO_3 \rightarrow 3CaSO_4 + CaS \ .$$

The MgO was practically inert to SO_2, but CaO was very reactive. When the SO_2/CaO mole ratio was 12, 90% of the CaO reacted according to

$$CaO + 1.5SO_2 \rightarrow CaSO_4 + 0.25S_2 \ .$$

Free sulfur is formed from excess SO_2:

$$CaO + SO_2 \rightarrow 0.75CaSO_4 + 0.25CaS \ ,$$

$$CaS + 2SO_2 \rightarrow CaSO_4 + S_2 \ .$$

METALLURGY AND MATERIALS RESEARCH

Ceramics Research: Sintering Studies. – The diffusion of thorium in ThO_2 has been measured by an isotope-tracer technique. A thin layer of $Th(NO_3)_4$ enriched in Th^{230} was deposited on the polished surfaces of thoria pellets having 97.5% of theoretical density. After annealing, the pellets were sectioned by grinding thin layers, and Th^{230} was determined by mass spectrographic methods at the ORGDP. Preliminary results indicate an energy of activation for thorium self-diffusion in ThO_2 of 38 kcal/mole in the temperature range 1200 to 1700°C. This compares with an activation energy of 15 kcal/mole for diffusion of oxygen in ThO_2.

Physical Property Studies. – The thermal conductivity of cold-pressed pellets of aluminum powder and 0, 10, 15, 20, and 25 vol % Gd_2O_3 was measured at 75°C in the thermal comparator apparatus. The thermal conductivity values of the pellets decreased rapidly with increasing Gd_2O_3. This change was much more than would be expected if the aluminum were the continuous phase. The Rayleigh-Maxwell relation for calculating the thermal conductivity of two-phase structures was applied, assuming the dispersed phase to be aluminum and the continuous phase to be Gd_2O_3. The calculated values agreed with the measured values, and photomicrographs of the pellets substantiated the above assumption.

The thermal conductivity of 93.4% dense UO_2 was measured to 1200°C in the radial heat flow apparatus on the third heating cycle. The data to 1100°C can be fitted to ±1% by $K = \rho/[5.555 + 0.02183T(°K)]$, where K is the thermal conductivity in w cm^{-1} $(°C)^{-1}$, and ρ is the fraction of theoretical density. Below 600°C the K values are lower than those obtained during the second heating cycle, but higher than those of the first heating cycle. This effect is thought to be due to a slight increase in the O/U ratio of the specimen during storage. Above 600°C the K values of the three cycles differ by only ±1.7%. Although difficulties were encountered above 1100°C, the K values obtained possess a negative deviation from the above equation; thus the thermal resistance of UO_2 does not continue to increase linearly with temperature, indicating that lattice conduction is being supplemented by another heat transfer mechanism, possibly that of radiation transmission.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. – The installation of the high-vacuum liner was completed, with two changes as compared with previous runs: (1) the entrance and exit apertures for the molecular ion beam were enlarged to 2-1/4-in.-diam circles; (2) the Z extent, formerly ±6-1/2 in., was increased through the inboard coil throats to ±17 in., and the walls at these locations were electrically insulated from the rest of the liner and connected to high-voltage bushings that go through the vacuum-tank walls.

A number of gas dissociation runs were made in this revised geometry. The plasma volumes, as deduced from full width at half maximum of the Z distributions of the flux of charge-exchange neutrals, and the containment times were not affected by dc potentials applied to the insulated end walls (the range ±15 kv was investigated). However, with high molecular

ion currents and long containment times, the previously determined conditions for maximum volume, the neutral-particle-detector data indicated a reduction in plasma volume by factors of about 4 when compared with earlier runs.

In order to determine the cause of the reduction in plasma volume, other runs were made with the 2-1/4-in. beam apertures, but with an arrangement whereby the liner walls could be closed at Z = ±6-1/2 in. or opened through the coil throats to Z = ±17 in. As functions of the position of the end walls, measurements were made of the Z distributions of fast neutrals to a silicon barrier detector on the median plane, of the steady-state rf signals, and of the plasma containment times. Only the rf signals appeared to change; the bursts of rf came more often with the liner open to Z = ±17 in.

Experiments keeping the position of the liner end walls constant and varying the molecular-beam entrance aperture are planned.

High-Current Injection. — Modifications of the epoxy insulating skirts have made it possible to operate the accelerator tube at greater than 600 kv without a freon-filled bag. Skirted sections are being assembled for the DCX-2 accelerator.

The fact that Philips ion gage discharges occurred in the tube at high lens current and low accelerating voltage made it necessary to reduce the lens current when turning on the power supply. A plot of the lens magnetic field indicated a change in the shape of the ground electrode which has eliminated this necessity.

Beam-analyzing equipment has been installed but still lacks some instrumentation for power measurement. Ion beams have been used to check alignment.

Theory. — It has been shown that, taking collision damping into account, plasmas stable according to the linearized Vlasov-Boltzmann equation are in fact stable nonlinearly against small perturbations. The theorem is an adaptation to plasmas of a well-known orbit stability theorem by Lyapunov. A necessary and sufficient condition for linear stability with collisions is that there exists a macroscopic quantity which is increased by any perturbation and which damps monotonically in time. For a Maxwellian plasma, the quantity is free energy. Landau damping persists nonlinearly and can, like collision damping, promote nonlinear stability.

Radio-Frequency Heating. — The hot electron plasma in the Physics Test Facility (PTF) has been illuminated with up to 5 kw of microwave radiation, and stable operation has been achieved up to 4 kw. Preliminary measurements of diamagnetism indicate that energy density increases linearly with input power from 1 to 4 kw. Decay times of microwave noise and diamagnetism following power turnoff also appear to increase. Under conditions in which the plasma is stable, neutrons have been observed at a source strength of $\sim 2 \times 10^5$/sec for 3 kw of radio-frequency power input. Neutron observations were made by activation of Rh^{104} foils and by direct counting with a fission chamber. Both detection methods have shown that most of the neutrons originate near the center of the PTF and not at its ends, but these detectors were not capable of showing whether or not some of the neutrons originated at the walls of the microwave cavity. Calculations indicate that production of the observed neutron flux by deuterons

striking deuterons adsorbed at the walls would require more power than that applied to the entire plasma. A series of experiments carried out to investigate photoneutron production showed that this is less than 5% of the total flux.

Since the plasma instabilities are not observed when neutron output is greatest, it seems likely that the production is a steady-state process. An intensive study is continuing in order to ascertain whether or not the neutrons are of thermonuclear origin even if they originate in the plasma. No doubt some neutrons are produced from dissociation of deuterons by Coulomb interaction with electrons in the high-energy group ($T_e \sim 100$ kev). Gamma-ray flux measurements indicate, however, an insufficient number of electrons in the "tail" of this group with the requisite energy, 2.2 Mev, to produce the observed neutron flux. A measurement of neutron intensity with a mixed hydrogen-deuterium plasma was made. Assuming that the protons are heated by the electrons twice as rapidly as deuterons and that there is thermal equilibrium between protons and deuterons, the (D,D) reaction rate should increase with added hydrogen concentrations up to ~50% and then drop rapidly to zero with 100% hydrogen. On the other hand, if the reactions giving rise to the neutrons are electron dissociation of deuterons, the neutron output should decrease linearly with hydrogen concentration, other plasma conditions being constant. Results of the experiment show a neutron output which is above that expected from electron-deuteron interaction, but not so much as expected for the assumed model if the production is by (D,D) reactions. Statistical accuracy of the data is not great, however, due to the low counting rates. Very preliminary proton spectra taken on silicon barrier detectors indicate that most of the protons have a discrete energy of ~3 Mev. This would be true if the protons originate from (D,D) interactions and not true if they come from Coulomb dissociation of deuterons, since in this case the protons could have a continuous range of energies up to one-half the excess gained by the deuteron.

BIOLOGY AND MEDICINE PROGRAM

RADIATION EFFECTS

Pathology and Physiology. – After injection of homologous, immunologically competent cells into sublethally irradiated recipient mice, a severe disease may occur. Clinical and pathological findings suggest the presence of marked physiological and metabolic disturbances. Attempts to study liver derangement in this syndrome were undertaken by use of the bromsulfophthalein (BSP) liver function test correlated with the histological examination of liver tissue sections. Increasing dye retention by BSP test, fat deposition, and liver cell vacuolation was found to parallel the severity of the reaction up to the time of peak mortality at about 12 days. Animals receiving irradiation alone at these levels (300 r, 500 r) did not demonstrate this marked liver change and loss of function.

Recovery from Somatic Effects of Radiation. – It has been reported that lymphoid leukemia cells and antibody-forming cells of C57BL mice do not grow as well in F_1 hybrid as in isogenic recipients or as cells of

other parental strains. The inability of the C57BL cells to proliferate in the F_1 hybrid mice has been ascribed either to "allergic death" of such cells, owing to the presence of excessive amounts of antigen, or to F_1-hybrid anti-parent immune reactions against such cells. The latter explanation implies, however, that transplantation antigens of the C57BL strain are not expressed in the F_1 hybrid, an implication which is at variance with the general experience in mouse immunogenetics. The present study was undertaken to explore the immunogenetic mechanisms for the failure of C57BL cells to grow in F_1 hybrid as well as in isogenic hosts. As a first step, the C57BL parent was mated with isogenic resistant partners differing for a single allele at the H-1, H-2, or H-3 histocompatibility locus. The F_1 hybrid offspring were x-irradiated (700 r), and 10^6 nucleated parental (C57BL/10 Sc Sn) marrow cells were injected intravenously immediately thereafter. Five days later, growth of the injected cells was evaluated by determining the uptake in the recipient's spleen of I^{131}-deoxyuridine, a specific deoxyribonucleic acid precursor. The results indicate that when the parents of the F_1 hybrid recipients differed at the H-1 (C57BL/10.BY strain) or H-3 locus [(C57BL/10. 129 (14M) strain], the C57BL/10 cells grew as well in the hybrid as in an isogenic host; however, when the parents differed at the H-2 locus alone (C57BL/10.D2 strain) or at the H-2 and other loci (C3H/Di Sn strain), growth of the C57BL cells was not detected in the F_1 hybrid recipients. Cell growth, however, could be detected in such recipients by increasing the number of marrow cells injected by a factor of ~10. These data suggest that the inability of C57BL cells to grow in F_1 hybrids is related to the influence of the H-2 genotype. Further studies with various combinations of parental H-2 genotypes will clarify the role of the $H\text{-}2^b$ allele in the donor and in the F_1 host in this phenomenon.

Physiology of Blood Platelets. — Previous studies in blood serum obtained from platelet-depleted donor rats have demonstrated the presence of an agent that can stimulate an increase in the production of blood platelets. To investigate the nature of the active agent further, active serum was acidified and boiled, and the filtrate was assayed. Such filtrates induced only a relatively small increase in the number of circulating blood platelets compared with that caused by injection of untreated active serum. It appears, therefore, that the active agent is at least partially heat-sensitive. This contrasts with findings on the nature of erythropoietin, a regulator of red blood cell production, which is not destroyed by acidification and boiling.

Medium(Low)-Level Long-Term Effects of Radiation. — Phagocytosis and killing of bacteria by mouse Kupffer cells were studied in irradiated and unirradiated mice by use of organ bacterial counts. Organs were removed aseptically, homogenized, and diluted. X radiation was administered in doses of 50 r every 48 hr. Forty-eight hours after having received 500, 1000, 1500, and 1650 r of accumulated dose, groups of mice were injected with a suspension of *Pseudomonas aeruginosa* containing approximately 10^6 cells. A steady decline was noted in the number of bacteria in the liver of unirradiated animals. Spleen and sternal marrow at all times after injection of bacteria contained less than 10% of that found in the liver. The blood levels decreased rapidly for the first half hour after injection, and no bacteria were detected between 4 and 12 hr. No more bacteria

were present in the livers of irradiated mice than in the livers of unirradiated mice 0.5, 4, 8, and 12 hr after the injection of bacteria. At 24 hr after injection, however, elevated liver bacterial counts (greater than the control mean plus three standard errors) were found in some individuals in all irradiated groups. The proportion of such individuals increased as the accumulated dose increased. All 24-hr values were elevated after 1500 and 1650 r. The elevated liver counts may be due to a radiation-induced defect of the Kupffer cells or to an interference with the subcutaneous inflammatory response at the site of the bacterial inoculation.

RADIATION GENETICS

Mutagen Mechanisms: Bacterial Metabolism. — A number of attempts have been made to explain the mechanism by which radiation resistance develops in unbuffered, glucose-containing media. Resistant cells are always larger than their sensitive counterparts and contain approximately twice the amount of macromolecular components per cell. It is not necessarily true that large cells with the higher concentration of deoxyribonucleic acid (DNA) and ribonucleic acid (RNA) are maximally resistant. Studies of the kinetics of development of resistance show that the large cells develop in cultures several hours before maximal resistance is measurable. These large cells yield survival curves that have a multiplicity and slope in accord with the increased concentration of DNA and RNA. Nevertheless, without a measurable change in size or content of macromolecular components, the resistance proceeds to increase for 6 to 8 hr.

Although earlier studies indicated rather elaborate nutritional requirements for development of resistance, recent investigations show that the processes involved in the increased cell size and the further development of radioresistance can be dissociated; the former require organic nutritional support, while the latter proceed at a maximal rate in an inorganic salt—glucose medium. This finding should make it much easier to understand what previously seemed to be a highly complex set of interactions.

Mutagen Mechanisms: Bacterial Genetics. — Recent experiments investigating the genetic control of radiation resistance in *Escherichia coli* were performed with two mating strains not previously employed. The results of crosses between these strains are not interpretable because of an unusual behavior of these organisms on various plating media. Logarithmic phase cells of these strains will not produce colonies on a synthetic medium known to be adequate for stationary phase cells. Experimental manipulation of diluting and plating media suggests that this phenomenon is due to a leakage from the cells of materials essential for the initiation of growth on synthetic media. The data suggest that it will be possible to prevent this leakage by alteration in the composition of the liquids used for dilution of cells prior to growth on plates. It is essential that this problem be solved if analyses of radiation resistance are to be made with these strains of *E. coli*.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. – It would be advantageous to have a rapid, reliable means of ascertaining the degree of cellular proliferation of transplanted hematopoietic tissue in irradiated animals. Preliminary investigation of Fe^{59} uptake by hematopoietic tissues suggested that a spleen assay could be used. In this study, graded doses of isologous bone marrow are injected into irradiated mice. The subsequent erythropoietic activity in spleen which results from proliferation of transplanted marrow can be assessed in terms of 24-hr Fe^{59} uptake by that organ. In an attempt to obtain the maximum reproducible cell dose response, major variables of the system were studied and the following results obtained: the x-ray dose should be an LD_{100} or slightly higher; the Fe^{59} can be injected as early as eight days after irradiation; the intraperitoneal injection of the isotope is comparable to the more time-consuming intravenous route; in terms of percentage uptake of Fe^{59} by the spleen, there is no isotope dose dependence; with an eight-day assay the uptake of Fe^{59} is linear (slope ~0.4) with bone marrow cell dose over the range 0.05 to 0.4×10^6 cells.

MOLECULAR- AND CELLULAR-LEVEL STUDIES

Biophysics: Ultraviolet Studies. – Photochemical products may be isolated from ultraviolet-irradiated deoxyribonucleic acid (DNA) by degradation of the DNA with venom diesterase followed by chromatographic separation. The major products are the four trinucleotides $pAp\widehat{TpT}$, $pGp\widehat{TpT}$, $pCp\widehat{TpT}$, and $pTp\widehat{TpT}$ ($\widehat{TT}$ indicates a dimer) (ORNL-3267, p 113). The dimers in the trinucleotides are split by 2390 A, and the cross section for splitting is the same for all four trinucleotides, even though they have different absorption spectra. The trinucleotides pXpTpT, which contain no dimers, have also been isolated, and the cross sections, at 2800 A, have been determined for the reaction $pXpTpT \xrightarrow{2800\ A} pXp\widehat{TpT}$. These cross sections depend on whether the third base, X, is A, G, C, or T. They decrease in the order T, C, A, G for reasons that are not known.

An attempt has been made to identify the other photochemical products in DNA by the following procedure: DNA's have been made, by use of the calf-thymus polymerase, that are labeled in one of the four bases; these synthetic DNA's are irradiated, degraded enzymatically, and chromatographed; the unidentified photochemical products contain both G and C but not T.

Biophysics: Electron Spin Resonance Studies. – Measurements have been made of electron spin resonances (ESR) in dry hematoporphyrin, irradiated with visible and ultraviolet light and without known previous irradiation. In all cases a broad (> 100 gauss wide) and a narrow ESR signal are found. Irradiation with visible or ultraviolet plus visible light clearly increases the narrow signal, and the kinetics of this phenomenon as well as variations in the broad signal are being studied.

Previous work (ORNL-3201, p 156) on electron spin resonances in organic dyes is being extended to include studies throughout the temperature range −180 to +300°C (solids and frozen solutions), power saturation, and effects of visible light.

The power saturation data for diphenylpicrylhydrazyl, pitch, and irradiated sucrose were analyzed in accord with the theory that the relation between areas of absorption curves (A) and microwave power (P) is

$$A = \frac{PN}{a + bP} ,$$

where N is the number of spins present, and a and b are constants characteristic of the sample and spectrometer. From this method smaller corrections for saturation are found than reported previously.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND INSTRUMENTATION

Basic Instrumentation. — A dc-to-dc converter was assembled and tested. A CK66 transistor oscillator operated at 1.4 v input excites a 1/8-in.-wide, 1-in.-long bar of piezoelectric ceramic, which is used as a voltage transformer, in its fundamental longitudinal mode. A rectified open-circuit output voltage of greater than 750 v was obtained, which indicates a piezoelectric transformation ratio of 550 to 1.

By refining previously described techniques for fabrication of lithium-drifted p^+-i-n^+ semiconductor detectors (ORNL-3279, p 29), improved detectors were prepared which have a dead layer less than 1.5 μ thick, whereas detectors prepared previously had a 7-μ-thick dead layer.

The resolution for 655-kev conversion electrons for 195°K was about the same, 6.5 kev (full width at half maximum), with detectors prepared by either fabrication procedure. However, for 5.8-Mev alpha particles, a resolution of 2% was achieved with the improved detectors; the resolution of the older detectors was too poor to measure.

A bias voltage of 150 v was required for measurement of alpha particles with detectors prepared by either procedure, but for beta particles a 50-v bias voltage was required with the new detectors as compared with up to 150 v with the older detectors. This indicates that higher electric fields are required for collecting the charge produced by high-specific-ionization particles.

With the new detectors it was not necessary to cool the detector to 78°K to obtain optimum resolution. In most cases, cooling to 273°K (water-ice temperature) was sufficient to obtain optimum resolution. In only a few cases was it necessary to cool to 195°K (dry ice—alcohol temperature).

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