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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 1963

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 1963

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 1963

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

GAS-COOLED REACTOR PROGRAM

Fuel Element Irradiations in GCR-ORR Loop No. 1. – A roughened-surface fuel element consisting of UO_2 pellets clad in stainless steel and having a heat rating of approximately 75,000 Btu hr^{-1} ft^{-1} has operated satisfactorily for approximately three weeks. The surface roughness was formed by machining, leaving fins 0.0055 to 0.0075 in. high spaced at 0.040-in. intervals. The cladding wall between fins is 0.015 in. thick. This fuel element has a higher power density than any tested previously. The square-cut machined fins have improved the heat transfer, as compared with that from a smooth surface, but may be less effective than the brazed-wire roughness tested previously.

GCR-ORR Loop No. 2 Irradiations. – The second experiment conducted with three 6-cm-diam graphite-matrix spheres fueled with coated particles of uranium carbide is under way. Alteration of the in-pile assembly to bypass helium flow has made it possible to increase the fuel inlet gas temperature to 1150°F. The three spheres were manufactured by General Atomic. One is operating at a measured central temperature of 1650°F, one at 1460°F, and one at 1370°F. Fission-gas release to the loop, although higher than in the previous run, is not excessive and has caused no inconvenience in loop operation.

LITR Capsule Irradiations. – A new capsule containing pyrolytic-carbon-coated uranium carbide fuel particles in each of four compartments is operating satisfactorily. Measured fuel temperatures in two of the compartments are between 2300 and 2400°F. Both this and a similar capsule installed previously are scheduled for 6 at. % burnup of the uranium.

ORR Poolside Capsule Irradiations. – The three sweep capsules containing 6-cm-diam graphite spheres fueled with pyrolytic-carbon-coated uranium carbide particles which were installed in the poolside facility last month are operating approximately at design conditions. The central temperature of the separately compartmented sphere in each capsule, which

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is swept with helium, is approximately 2500°F. The ratio of fission-gas release to birth rate is approximately 1×10^{-4}. The fuel for each of these spheres was manufactured by General Atomic; two spheres are enclosed in a molded unfueled graphite shell, and the third is in a machined shell of ATJ graphite. Other spheres being irradiated were manufactured by National Carbon, General Atomic, 3-M, and ORNL.

Eight-Ball Experiment. – Irradiation of the third experimental assembly containing eight 1-1/2-in.-diam spheres fueled with coated particles of uranium carbide in a graphite matrix was begun. The power densities of the spheres in this capsule vary between 35 and 40 w/cm^3. Surface temperatures of the graphite adjacent to the fueled surfaces are approximately 1100°F. The measured central temperature of the top ball is 1400°F.

Postirradiation Examination of Fueled-Graphite Spheres. – Three types of fueled-graphite spheres that were irradiated in the ORR were examined: (1) molded spheres in which the uranium carbide–thorium carbide fuel was uniformly dispersed, (2) molded spheres which had unfueled graphite shells around the fueled-graphite core, and (3) machined spheres of unfueled graphite containing a fueled-graphite cylinder secured by a threaded graphite plug. All the spheres were irradiated at 1000–1500°F to 1–11% heavy-metal (U + Th) burnup.

There was no obvious damage to any of the spheres. Analyses of gas samples from static-atmosphere capsules indicated that 0.01 to 0.1% of the Kr^{85} was released. Dimensional measurements showed very few consistent changes; some of the molded spheres had shrunk as much as about 1% on the equatorial diameter. There were no significant changes in the polar or temperate diameters or in the weights of the spheres. A less than 10% reduction in strength was indicated by compression tests, which were performed on eight irradiated spheres. The analyses of fluidized coke surrounding some of the spheres revealed small quantities of solid fission products, amounting to less than 1% of the Sr^{89}, Zr^{95}, Ru^{106}, Cs^{137}, Ba^{140}, and Ce^{144} produced.

Preirradiation Evaluation of Fueled-Graphite Spheres. – A method was developed for rapidly measuring sphere diameters by utilizing a rotating fixture and an eddy-current coil to detect the relative position of a Teflon-tipped probe. The probe is held in contact with the sphere surface 180° from a fixed reference surface, and its position varies as the diameter varies. A strip-chart recorder continuously records the signal as the diameter of the sphere. The sphere is indexed to a new scanning circumference with each rotation. The record of one sphere on which a 0.020-in. flat was filed showed that the flat area passed under the probe or the zero reference surface an average of once in four rotations. The longest interval without an indication of the flat area on the chart was 12 rotations. A speed of 1 rps can be used without difficulty, and it is anticipated that the inspection can be done on a large number of spheres with an average time of less than 2 min/sphere. The instrument will detect diametral changes of ±0.001 in.

Preparation of Coated-Particle Radiographic Standards. — The furnace for applying pyrolytic-carbon coatings was used to produce radiographic standards that could be readily distinguished from ordinary particles. Tungsten wire 0.002 in. in diameter was chopped into 1/8-in. lengths to make a 0.05-g sample. The wires were charged into the coating furnace on top of a fluidized bed of angular alumina grit and coated along with the grit. The wires were coated uniformly to a thickness of ~100 μ, while the alumina grit received a 115-μ coating. There was a 1- to 2-μ reaction layer at the tungsten—pyrolytic-carbon interface that could be observed metallographically, but which did not affect the usefulness of the specimens as radiographic standards.

Optical measurements on one of the standards indicated a composite diameter of 230 μ. This standard was radiographed with several batches of coated particles as a reference for dimensional measurements.

Fission-Gas Release from UO_2 During Postirradiation Annealing. — In the investigation of the release of fission gas during postirradiation annealing of neutron-activated samples of UO_2, a definite effect of stress on the rate of release from a sample was observed. This effect appears to increase with increasing stress or temperature or both and to diminish with increasing time at a given temperature. Since these variations are also characteristic of the motion of lattice dislocations under stress at elevated temperatures, it is felt that there may be a correlation between the motion of dislocations and the release of fission gas.

Further evidence of the correlation between stress state or dislocation motion and the release of fission gas was found in an experiment in which the fractional release of Xe^{133} from bushings of ThO_2 + 2% UO_2 was recorded while the bushings were repeatedly heated to a temperature of 1450°C. The heating-burst releases decreased on repeated heating when the sample was cooled relatively slowly from temperature. The bursts were found to rise to or beyond the original magnitude when the sample was quenched drastically from a high temperature and then reheated. Since this release behavior parallels the expected behavior of dislocations in the sample, it appears to reinforce the proposition that the burst of fission gas observed on heating or stressing a sample is connected with the motion of dislocations within the sample.

Instantaneous Fission-Gas Release from Coated Uranium Carbide Particles. — Postirradiation examination of capsule B9-13 showed no broken coatings. Several uranium carbide cores were cracked, but the cracks did not continue into the coatings. No reaction zones were present at the core-coating interfaces. Capsule B9-13 contained uranium carbide particles with triplex pyrolytic-carbon coatings and was irradiated to 12 at. % burnup of the uranium at 2400°F.

Capsule C1-11, which contains triplex pyrolytic-carbon-coated thorium-uranium carbide particles, has been irradiated at 2050°F to 2.9 at. % heavy-metal burnup; and capsule B9-14, which contains triplex pyrolytic-carbon-coated uranium carbide particles, has been irradiated at 2500°F to 6.5 at. % uranium burnup. These irradiations are continuing. The coatings of the particles in both capsules are releasing about four times as much krypton as xenon.

Development of Graphite Rupture Criteria. — An analog computer was programmed to calculate the extreme fiber stresses in graphite bend specimens from strain measurements on the tension side and on the compression side while tests are in progress. Stress-strain diagrams are plotted on x-y recorders at the same time. Therefore the results of a test can be visually monitored during the test.

Because of the variations in mechanical properties of graphite, the stress-strain data will be evaluated statistically. Therefore the raw data and computer stresses are relayed via telephone line to a digitizer where the data are scanned at frequent intervals during the test, recorded on tape, and subsequently punched on cards. The experimental data are also recorded on an oscillograph for use in checking and in case of malfunction of either the analog computer or digitizer.

Design Criteria for Graphite Components. — One phase of the graphite mechanics program is a theoretical study of the relative importance of the stress-strain relationships in predicting the behavior of graphite bodies under thermal and externally applied loadings. The purposes of this study are (1) to determine analytically the stress and strain distributions in simple bodies according to various theories of solid mechanics; (2) to determine whether detectable differences in these distributions, as predicted by the different theories, will occur; (3) to define a test that will distinguish between the theories if such differences occur; and (4) to assess the consequences of using simplified methods in design.

Stresses and strains were calculated for thick-walled graphite cylinders loaded with an internal pressure. Four different stress-strain relationships were used. These relationships were approximations to the true stress-strain curve obtained from a specimen of AGOT graphite loaded in uniaxial tension. Two of the relationships were straight line: one tangent to the true stress-strain curve at zero load, and the second connecting the point of zero load with the failure point. The third relationship had an elastic portion with a perfectly plastic portion, and the fourth had an elastic portion with a linear strain-hardening plastic portion.

Cylinders with different wall thicknesses ranging from an inside radius to outside radius ratio a/b of 0.25 to 0.75 were studied by using each of the four stress-strain relationships. Since the circumferential strains on the inner and the outer surfaces are the most significant items which can be measured experimentally, these values were computed as functions of internal pressure.

The results indicate that cylinders with $a/b = 0.50$ are potentially suitable for use in identifying experimentally the stress-strain relationships and theories applicable to graphite bodies. When the pressure ranges from 1200 to 1350 psi, the differences in predicted strains on both surfaces are large enough to be detected by strain gages. Actually, differences may be seen between all curves at pressures above 690 psi when $a/b = 0.50$.

Graphite Oxidation and Mass Transport. — Operation of a facility for low-pressure tests of graphite oxidation was started. Tests are being

run to study the effect on moderator-grade graphite samples of low levels of impurities in a simulated reactor coolant stream. The loop has been operated for 425 hr with impurity levels of CO and CO_2 maintained at 150 and 220 ppm, respectively, by means of a copper oxide bed and a molecular sieve operating in parallel in the loop bypass system. The test piece is type TSF graphite, and the average inlet gas temperature is 1800°F. Gas samples are being taken periodically for analysis.

Advanced Gas-Cooled Reactor Studies. — The Westinghouse Electric Company completed a study requested by ORNL of combined gas turbine—steam turbine power cycles for use with gas-cooled reactors with gas temperatures up to 1500°F; design and cost estimates of equipment for two versions of 1000-Mw (electrical) power plants were included. One system, using 1475°F gas in gas turbines to drive the circulators and generating all electric power with steam turbines, yields a net plant efficiency of 43% and a total cost of $49.16 per kilowatt of net plant power for all power conversion equipment, including steam generators and gas circulators. In another system, with the same gas temperature but using the gas turbine to generate approximately 10% of the total electric power in addition to driving the circulators, the net plant efficiency is 44% and the corresponding equipment cost is $59.13 per kilowatt of net plant power output. The corresponding cost for the GCR-3 equipment was $56.34 per kilowatt of electricity, and the gas temperature was only 1150°F. For the conditions assumed for the study, the gas-turbine topping cycle for generating power has no advantage over the steam cycle with steam-driven circulators, and the cycle using the gas-turbine-driven circulator has only slight advantage.

REACTOR FUELS AND MATERIALS

Nondestructive Test Development. — We are establishing criteria needed for the design and construction of eddy-current probes to measure the critical coolant-channel dimensions in reactor fuel elements. From measured and calculated values of coil impedance, several sets of graphs have been prepared, from which the proper coil dimensions and number of turns can be chosen. The impedance change for the desired spacing change can be approximated from the curves, and the range, sensitivity, and linearity of a particular coil can be predicted. This study will enable relatively inexperienced personnel to design and construct coolant-channel spacing probes.

Mechanical Properties. — Tensile properties are being measured on austenitic stainless steels that have been exposed to fast-neutron doses ($E > 1$ Mev) of 7 to 11 $\times 10^{20}$ nvt. The effects of metallurgical condition, irradiation temperature, postirradiation test temperature, and strain rate are being evaluated for more than 200 subsize tensile specimens. Results measured and derived include the 0.2% offset yield stress, ultimate engineering stress, true-stress—true-strain relationship, fracture stress, uniform and total elongation, reduction in area, and the energy necessary to cause plastic instability.

Several conclusions can be drawn from the first measurements. The strength of stainless steel, determined at any temperature in the range 70 to 1400°F, is not significantly affected by irradiation at temperatures above 1100°F. For alloys irradiated at lower temperatures, the yield strength measured above 1100°F is unaffected, but the yield strength at lower temperatures increases as the irradiation temperature decreases. The 1100°F temperature is probably important because it is half the absolute melting point, the temperature at which diffusion generally becomes important. For a given alloy, test temperature, and strain rate, the true stress at which plastic instability starts appears to be unaffected by irradiation at any temperature. The uniform elongation, which is a measure of ductility, depends on both irradiation temperature and test temperature; the lowest value found to date is 3.5% for 5%-cold-worked type 347 stainless steel irradiated at 400°F and tested at 750°F.

Solid Reaction Studies. — A new and original apparatus containing unique features was developed for measuring the diffusion of impurities in a temperature gradient. It was tested by measuring the redistribution of Sb^{124} initially uniformly distributed in a 0.4-cm-long silver specimen. After 240 hr the antimony concentration was 0.75 times as great at the end maintained at 890°C as at the end at 820°C. Steady state had not been reached.

A FORTRAN program was written for the calculation of diffusion coefficients on the CDC 1604 computer. Solving Fick's law for the case of an initially thin layer of tracer on the end surface of a long cylinder, the program calculates both the diffusion coefficient and the 90% confidence limit on it. The program also prepares a magnetic tape from which the Calcomp plotter can graph the logarithm of the specific activity against the square of the depth of penetration.

The diffusion of Pb^{212} in pure silver under alpha irradiation is being measured by the alpha recoil method, which is based on changes in surface activity. The first results indicate that in the temperature range 325 to 425°C, lead diffuses in silver one-thousandth as rapidly as expected by extrapolation from published measurements in the range 700 to 800°C. The discrepancy probably results because diffusion near the surface, which is emphasized in the present method, is not governed by the laws of bulk diffusion.

Materials Compatibility Studies. — A satisfactory method of analyzing for oxygen in potassium is still being sought. The amalgamation technique, in which potassium is extracted by mercury, leaving an oxide residue, has been tested on 20 potassium samples with controlled oxygen additions. When the residue was analyzed for potassium and assumed to contain the oxygen as K_2O, the oxygen content determined ranged from 8 to 81% of that added. The recovery percentage generally increased with the temperature at which the sample had been equilibrated after the solid oxygen source was dissolved. Oxygen losses to the wall or through reaction with impurities have been found not responsible for the low results. Either the oxygen is precipitated not as pure K_2O or some oxygen is retained by the amalgam. This latter possibility is now being examined by hot gettering the amalgam with zirconium metal.

We are studying the effect of zirconium concentration on the resistance of niobium-zirconium alloys to lithium. Oxygen contamination sometimes impairs the corrosion resistance. In specimens of Nb–1% Zr in the corrodable state, a zirconium-oxygen interaction peak has been observed in internal-friction experiments. This has been interpreted to mean that the oxygen, although in solution in the alloy, is in some way associated with the zirconium. Annealing at 1000°C removed this peak by precipitating the oxygen, presumably as zirconium oxide, and restored the resistance to lithium. We found that alloys of niobium containing zirconium and oxygen, which were added systematically, could be made resistant to lithium attack by annealing, only if the oxygen-zirconium atom ratio does not exceed 2. Since this ratio corresponds to the stoichiometry of ZrO_2, we propose that corrosion protection in niobium-zirconium alloys results from immobilization of oxygen in this stable phase.

Fuel Element Development. – The thermochemical deposition of refractory metals and carbides is continuing, with prime emphasis on deposition of fuel compounds. We have completed the investigation of the effect of temperature on uniformity and deposition rate in the hydrogen reduction of tungsten hexafluoride to form dense tungsten cladding material. Various lengths of tubes have been produced, with wall thicknesses ranging from 0.001 to 0.060 in. and diameters from 0.040 to 0.750 in. We are still investigating the purity and the structure of the tungsten deposits.

Stoichiometric B_4C and SiC have been deposited by hydrogen reduction of the appropriate chlorides in the presence of a hydrocarbon at elevated temperatures. We have varied the composition of deposited boron carbide from 5 to 81% boron by varying the deposition temperature and concentrations of the reactants.

Thermochemical reduction of UF_6 in the presence of methane has produced some small unidentified uraniferous carbide deposits. However, in the cold-wall hot-filament apparatus currently used, the major products are undesired intermediates such as UF_4. The equipment is being modified.

REACTOR COMPONENTS

Reactor Controls Development: Electronic Instruments. – The transistor electronic modules discussed previously (ORNL-3436, p 7) have been brought to the production prototype stage. A total of 352 modules of 24 types are being produced for initial testing and operation of the HFIR and the MSRE. The design of these instruments will be refined as experience is gained in the production, testing, and use of these modules in actual reactor instrumentation systems.

Reactor Controls Development: Fluctuation Analysis. – An on-line multichannel frequency analyzer comprising analog computer elements has been assembled and tested. This analyzer will be used for experiments on measuring shutdown reactivity (ORNL-3388, p 8) by analyzing neutron density fluctuations. A special low-noise preamplifier has been developed for use in these measurements. It has variable gain, up to 10^4 maximum, and bandwidth, with gain stability of better than 0.1% for 60 hr.

The noise referred to the input is 3.8 μv rms with a passband from 0.013 to 3500 cps. The input is differential, with 50 db of common-mode rejection for freedom from electrical pickup noise.

THORIUM UTILIZATION PROGRAM

Uranium-233 Purification by Solvent Extraction. — Fifteen kilograms of U^{233} for use in fabricating fuel rods for experiments at Brookhaven National Laboratory was purified by solvent extraction (4 kg of U^{233} per day) from nitrate solution with di-sec-butyl phenylphosphonate—diethylbenzene. Decontamination factors were 4600 and 400 for thorium and gross gamma activity respectively; recovery was 99.7%; the nitrate/uranium mole ratio was 2.2. The uranyl nitrate product was satisfactory for use in the sol-gel process installed in the fuel-rod facility.

Mixed Thoria-Urania Preparation in the Fuel-Rod Facility. — Two hundred twenty kilograms of mixed ThO_2-$U^{233}O_2$ satisfactory for use in fabricating fuel rods for experiments at Brookhaven National Laboratory was prepared by the sol-gel process in the fuel-rod facility, with no mechanical difficulty. Removal of Rn^{220} and Ra^{224} by volatilization during calcination resulted in decreased radiation levels.

Rod Fabrication. — One hundred fifty rods were completed for use in the Brookhaven National Laboratory criticality experiments. The first shipment (120 rods) has been sent to BNL. Unacceptable density variations along the length of the rod are still found, and failures of the grinding and vibratory compaction units limit the rod fabrication rate.

Thorium-Uranium Fuel Cycle Development Facility. — The Thorium-Uranium Fuel Cycle Development Facility (TUFCDF) is to afford adequate shielded space for development of processes suited to the remote processing and fabrication of Th-U^{233} fuel assemblies of different types. Sufficient shielding (5-1/2 ft of normal concrete) is being provided so that highly irradiated fuel assemblies can be accommodated after a three-month decay period. Because of the heavy shielding, no limitations will need to be imposed on U^{232} content or decay time after removal of U^{232} decay daughters. The four operating cells that comprise the facility are each 20 ft wide and have a combined length of 106 ft. The total estimated cost of the facility with minimal sol-gel vibratory-compaction equipment is $7,275,000. Facility planning is in the preliminary design stage (Title I), with Giffels and Rossetti, Inc., serving as architect-engineer. Design has been in progress since January 3, 1963. Title I design for the process equipment is being prepared by ORNL, and it has been completed except for final minor revision of otherwise completed drawings and for reworking of the initial draft of the Title I report. The major items remaining to be completed are: (1) specifications for cell cranes and manipulators, (2) specifications for shielded viewing windows, and (3) cell fire protection design.

Particle Sizing of Sol-Gel Thoria and Thoria-Urania. – Thoria and thoria-urania may be sized for vibratory compaction into fuel rods by simple fracturing and ball milling at the gel stage of the process. The density of the vibratorily packed powder approaches 90% of the theoretical density. One-kilogram batches of the mixed oxide have repeatedly been sized successfully in the laboratory. Recycle of the oxide is accomplished by simple redispersion in water, and overall process simplifications appear likely.

Sol-Gel Microsphere Preparation. – In preparing thoria and thoria-urania gel microspheres from the sol, a solution of 0.1 vol % sorbitan mono-oleate (Span 80) in 15 to 30 vol % isopropyl alcohol–CCl_4 in a mechanically agitated vessel gave the best results. Distillation of a 30 vol % isopropyl alcohol–CCl_4–0.1 vol % Span 80 solution after it had become nearly water saturated was shown to be a means of regenerating it for use in gel microsphere formation.

Dispersion Hardening of Thorium-Zirconium Alloys. – Compacts of thorium metal containing dispersions of thoria ranging from 4 to 10% were successfully prepared. The effect of combining alloying with dispersion strengthening is under investigation. An alloy containing 2% zirconium and several others containing from 3 to 5% indium have been prepared for testing.

Irradiation of Thorium Oxide Fuels. – Capsules tested in the ORR (poolside position) at an $\int k d\theta$ (a measure of the severity of thermal treatment) of 57 w/cm were examined. The loose particles were sintered, and considerable grain growth had occurred at the center of the capsule. A reaction between the fuel and the stainless steel clad was detected. This result appears similar to that reported previously on EGCR tests and by the British for UO_2. Capsules that had achieved a burnup of 68,000 Mwd per metric ton of thorium were removed from the MTR and are being examined.

Irradiation of Thorium-Base Alloy Fuels. – Two of the capsules made by Advanced Technology Laboratories for use in radiation-swelling tests failed and were removed from the General Electric Testing Reactor. Efforts are being made to repair one capsule, and a lower-temperature capsule has been substituted for the other.

Oxide Sizing and Vibratory Compaction. – Equipment is being developed for testing a method for sizing thoria and thoria-urania which employs the ball-milling of fired sol-gel material and uses what passes a 6-mesh screen as feed for vibratory compaction. Comparative evaluation of vibrators for use in the vibratory compaction step of fuel rod fabrication is continuing.

Fission-Gas Release from Ceramic Fuels. – The application of stress to a ceramic fuel has been observed to enhance the rate of fission-gas release. The manner in which this occurs is a function of stress, temperature, and time. This correlates with the expected behavior of dislocations under similar conditions. Thus it appears that dislocation movement may be a prime factor affecting the release rate of fission gases from fuels.

Sol-Gel Preparation and Properties of ThC_2 and UC_2-ThC_2. – Gels produced from four separate thoria-carbon sol preparations had the same ThO_2/C ratio (to within ±1%) in a given particle size range, and the same ThO_2/C ratio to within ±3% between coarse and fine fractions. Such homogeneity is essential in a material to be used in fuel elements. The sols used in these studies developed a settled solid phase upon standing for 48 hr; however, thoria-carbon sols stable for a week have now been produced.

The carbon content of the gels has been found to be the controlling factor in determining the carbide composition of the fired gel.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. – The design, procurement, and construction of the MSRE was 82% complete on September 1, 1963.

Heater control panels and main reactor control panels were set into position. Progress continued to be made in the installation of auxiliary piping, instrumentation, and electrical services. One of the fuel drain tanks and its cooler were installed. The second fuel drain tank was placed in the assembly jig. Fuel piping was welded to the heat exchanger and the freeze flanges. One of the coolant lines was installed on the jig and welded into the system. Installation of the coolant piping in the coolant cell was started. Both the fuel and coolant pump bowls were delivered to the job site and prepared for installation.

The H. K. Ferguson Company continued to install electrical and auxiliary systems outside the cells. The portion of the work being done by Ferguson is ~25% complete.

Metallurgy. – A spectrum of open-pore entrance diameters was determined for grade CGB graphite. The material was selected from bars fabricated for the MSRE core moderator. Results indicated that the pore size range is lower than that reported for experimental grade CGB-X. Grade CGB graphite had approximately 10 vol % of open pores, but the major portion of these have entrance diameters of less than 0.2 μ. At a reactor operating temperature of 1300°F, a molten-salt pressure in excess of 500 psia would be necessary to force salt into the bulk of these voids. This information explains the observation on CGB graphite in the salt-permeation screening tests that at 165 psia only cracks were permeated by salt.

Chemical Development. – The wetting behavior of MSRE fuel and coolant, with particular attention to the effect of the purity of the cover gas, is being surveyed by observations of sessile drops. No significant differences were noted between the fuel and the coolant; under helium at 600°C the contact angle of sessile drops with graphite was about 160°, and with nickel or copper, 90° or higher. In the presence of 10 ppm of H_2O in flowing helium, the contact angle on graphite decreased at rates as high as 2°/min, presumably in response to the formation by hydrolysis of oxides such as BeO or ZrO_2 at the melt-graphite interface. Elemental O_2 had much less effect than H_2O on the wetting of graphite under similar

conditions, but, in marked contrast, the role of O_2 in causing wetting of metal was easily observable. The wetting of metal by MSRE molten salts in the presence of trace amounts of O_2 or H_2O in the cover gas appeared to result from the formation of easily wetted metal oxide on the surface of the metal.

The sessile-drop behavior confirmed previous conclusions that wetting of graphite by circulating fluoride melts is not expected in the MSRE operations, but reopened questions concerning whether thoroughly clean nickel or nickel alloys are wetted by the molten fluorides.

Preparation of MSRE Materials. – An order for 12,000 lb of beryllium fluoride was placed with the Brush Beryllium Company for delivery beginning the first week of October 1963. As anticipated at the time bids were first received (ORNL-3388), it was possible, by deferring the purchase until the present time, to obtain a more competitive price for material of the best available quality. The final purchase price was approximately 85% of the lowest earlier bid price for the same quantity of material.

Radiation Effects. – Along with the two capsules from which gas samples were removed during the in-pile exposure, there were also four sealed capsules in the most recent irradiated assembly involving MSRE materials (experiment ORNL-MTR-47-5). The gas from the sealed capsules was collected with a puncturing apparatus and analyzed by mass spectrometry. Two of the capsules were small and contained cylinders of AGOT graphite (3 g) impregnated with fuel (0.3 g); these yielded only the original cover gas and the expected amounts of xenon and krypton. A larger capsule containing 25 g of fuel (under ~4 cc of helium cover gas) and distinguished by a relatively small graphite-fuel interface gave 115 cc of gas that contained 63% CF_4 and 19% F_2; whereas a similar capsule with 19 g of fuel, distinguished by a relatively large graphite-fuel interface, gave only 5.7 cc of gas with an analysis of 10% CF_4 and 35% F_2. The concentrations of uranium (0.66 mole %) in the fuel as well as the neutron fluxes in the two large capsules were essentially identical.

Based on evidence from other capsules, the F_2 must have evolved from the frozen fluoride fuel salts at room temperature during the decay period, and the CF_4 presumably resulted from reaction of the F_2 with graphite; the discordant results from the various configurations suggested that the amount of F_2 evolved in a given capsule is strongly influenced by some obscure and as yet unidentified factor in addition to the more obvious ones such as energy liberated during the decay period.

MSR Processing. – A directive was received authorizing construction of the MSRE fuel processing system. Drawings are being revised, and fabrication of the fuel storage tank was started. An initial meeting with the Criticality Committee was held in which most concern was felt over the possibility of a uranium breakthrough to the caustic scrubber. This possibility will be investigated further.

Analysis of Radioactive MSRE Fuels. – The devices known as the pulverizer-mixer aligner-clamp and the powder-transfer apparatus for use in processing samples of solidified salt were improved and refabricated and were tested satisfactorily in the hot-cell mockup.

The copper ladle for sampling the fuel was redesigned, and this necessitated the rejection of a shearing device that was to be used for cutting the attached stainless steel wire and the promotion of a modified tube cutter for cutting the capsule. A prototype tube cutter was fabricated and preliminarily tested.

One of the redesigned copper ladles, filled with sample, was tested in the mockup. The operation of decoupling the transfer container was omitted, but the ladle was cut open with the modified tube cutter and carried through the powder transfer stage. Additional modification and testing will be required to obtain a satisfactory operation.

Methods are being investigated for determining the reducing power in the MSRE fuel. Previous tests have shown that in an inert atmosphere the MSRE salt is soluble in molten potassium bisulfate—boric acid ($KHSO_4 \cdot H_3BO_3$) mixture at temperatures which exceed 225°C, while the UF_3 is essentially insoluble under these conditions.

Tests were conducted in which a 0.5-g sample of MSRE salt was dissolved in a mixture of 3 g of $KHSO_4$ and 0.5 g of H_3BO_3 at about 300°C in a filter stick. After the fusion was completed, usually in about 10 min, the melt was passed through the frit, 30-mm fine porosity, by the aid of a slight vacuum. No insoluble material remained on the frit. When UF_3 was added to a sample of MSRE salt and the experiment was repeated, more than 95% of the UF_3 was found on the frit. Cursory tests with CrF_2 added to the salt showed that the CrF_2 was completely soluble on MSRE salt. It thus appears possible to achieve a specific separation of UF_3 from the fuel salt. The UF_3 can then be determined in the usual manner by the hydrogen evolution method.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. — Full-size inner and outer aluminum-base composite plates for the HFIR control rods have been successfully hot rolled to within the desired dimensional tolerances. The finished plate, which is 13 in. wide, 64 in. long, and 1/4 in. thick, contains a 22-in.-long section consisting of 33 vol % rare-earth oxide mixed with aluminum and a 5-in.-long section composed of 40 vol % tantalum dispersed in aluminum. The rare-earth oxide (Lindsay mix) is a substitute for the more expensive europium oxide that will subsequently be placed in the plates for reactor operations. Room-temperature mechanical property data on this composite indicate that both the rare-earth and tantalum mixtures have rather low ductility — no greater than 6% elongation. Anisotropy in ductility has been observed in the rare-earth—aluminum material, where elongation values as low as 2% in the transverse direction have been recorded.

Thermal conductivities of these dispersions were measured with a thermal comparator. The values reported were 65 and 55 w m^{-1} $(°C)^{-1}$ for 40 vol % Ta—Al and 40 vol % rare-earth—Al mixtures respectively.

To achieve the formed dimensions that are desired within the strict tolerances imposed, the plates are first preformed to approximately a

9.2-in. radius and subsequently sized to final curvature by explosive sizing. Dimensional results of one outer control plate sized in this manner have been very encouraging. In the preforming operation, however, logitudinal cracks have generated in the rare-earth oxide—aluminum section. These cracks appear to be related more to the billet design than to the low ductility of the material. A change in design is expected to alleviate this problem.

Procedures were developed for welding formed plates into a cylinder representing the inner control rod. One mockup all-aluminum tube was welded together and expanded close to the finished diameter with no failures at the welds. The tube will be explosively sized to finished dimensions.

Component Development. — Journal bearings with Graphitar No. 14 inserts were tested in pH 5 demineralized water at 195°F. The bearings were tested through load ranges of 25 to 100 lb per bearing, and the coefficients of friction varied from 0.12 to 0.15. The bearings turned freely during all phases of testing, although some Graphitar smearing was noted on the bearing shafts, which were machined from types 304 and 17-4 PH stainless steel. Bearing shafts of either Stellite No. 1 or No. 3 will also be tested. A sleeve-type bearing of Stellite No. 6 was tested against a shaft overlaid with Stellite No. 1 at loads of 25 and 50 lb/bearing. The surfaces of both the bearing and the shaft were in good condition after the tests. Previous tests with type 304 stainless steel shafts had resulted in severe score marks on the shafts.

A preliminary series of tests was completed on the magnetic actuator and ball-latch mechanism of the prototype HFIR control-rod drive. A response time of 7.0 to 7.8 msec was observed under various conditions for a total of over 2000 scrams. This response time is the time from initiation of the scram signal until initial motion was observed by means of a linear potentiometer attached to the ball-latch plunger. Initial motion of the magnet armature was observed in 4.7 to 5.3 msec, also by means of a potentiometer.

Strain gages were attached to the push rod to observe the dynamic strain of the push rod during scram and during impact on its shock absorber. The results were well within the limits of allowable strain.

The unit was disassembled and inspected both during and after the tests. No significant wear was noted.

Mechanical testing of a prototype fission chamber drive was completed. This unit is now being prepared for installation at the Bulk Shielding Facility, where it will be tested by the Reactor Control Department as a part of a complete fission chamber system.

A prototype chamber drive and support assembly was fabricated and assembled. This unit is being tested for operability and maintainability prior to beginning fabrication of the reactor drives.

Fabrication of the reactor components has been under way since July 15, 1963. The N^{16} probes, the fission chamber thimbles and flanges, the

vessel lower head, and the lower shield plug are 90% complete. The pedestals are 15% complete, and the permanent reflector is 40% complete. The reactor control-rod drives and servo input drive units were tested and accepted at the seller's plant.

Design and Analysis. — Review of the reflector design in light of the beryllium radiation damage data from the MTR is continuing. The present removable-reflector design will require some changes, the extent of the changes being dependent primarily upon the type of control-rod guidance package to be used and the necessity for a water seal between the removable reflector and control region. The removable beryllium will no longer serve as a structural member in the control-rod guidance package.

An analysis of the most recent data obtained on the control-region hydraulic experiment indicates that the hydraulic load on the outer control rods may be as high as 140 lb for some rod positions.

The analysis of the horizontal beam tubes indicated the desirability of increasing the minimum thickness of the hemispherical heads from 0.170 in. to 0.250 in. A review of the increased temperature in the thicker head showed a metal temperature not exceeding 220°F.

Radiographic examination of repaired weld areas in the pressure vessel following the third stress relief revealed no indication of defective welds. Lines which appeared on radiographs of the RH extension to nozzle dissimilar-metal welds were attributed to the joint geometry and the differences in density of the three different metals involved. Radiography of vessel welds is now complete except for ion chamber welds and EF extension to nozzle welds, which must be machined prior to radiography.

Dimensional inspection of the vessel following radiography indicated that the HB nozzles, IC nozzles, RH nozzles, and coolant-water inlet nozzles would finish-machine to approximately 5/16 in. short of nominal. Sufficient machine stock was available to obtain other dimensions within tolerances. A study was made of the significance of deviation from the specified dimensions for the nozzles out of tolerance, and it was found that the deviations could be accommodated.

The vessel, now being machined, is scheduled for completion by November 15.

Design of the steel and lead shielding to be placed around the ion chamber thimble in the vessel was completed.

PHYSICAL RESEARCH

Reactor Operations. — Through September 15 of this year, the ORR, the LITR, and the ORNL Graphite Reactor have operated 76, 87.1, and 82.4% of the time respectively.

The Graphite Reactor was placed on a 40-hr-week operating schedule on August 5, preparatory to complete shutdown on November 4. Facilities are being prepared in the LITR, the ORR, and the BSF for some of the experiments now done in the Graphite Reactor. The remote-control desk of the LITR has been moved from the Graphite Reactor to the ORR, and personnel relieved by the curtailed operation have been reassigned.

Waste Disposal. — During August, fabrication and testing of the new process waste alpha monitor were completed, and the instrument was installed at the diversion box. A calibration was carried out with U^{238} solution, and it was established that a 0.5 MPC_W (168 hr) level could be detected. This value will vary for other alpha emitters because of differences in MPC_W and in specific activity. Uranium-238 was chosen as the testing material not only because it is one of the least toxic alpha emitters but also because it has one of the lowest particle energies. On the basis of particle energy alone, the calibration showed that the new monitor will be capable of detecting any alpha emitter. The actual sensitivity will depend largely on other factors, as noted above.

Hot-Cell Operations. — Operations in the hot cells were normal. Eight fueled capsules from the ORR and the MTR were examined; the fuels included fueled graphite balls, UO_2, and pyrolytic-carbon-coated UC_2 particles. APPR fuel elements S-80 and Eu-SS-4 were dimensionally checked and visually examined. Examination of experiment ORNL-10, containing about 180 BeO pellets encapsulated in stainless steel, was completed; and examination of ORNL 41-8, a similar BeO experiment, was begun. The first fueled experiment from ORR-GCR loop No. 2 was disassembled. Specimens were recovered from 12 ORR experiments in the ORR cells, and profile dimensions were made on three ORR shim rods. Disassembly of molten-salt experiment MTR 47-5 was begun.

Installation of in-cell and out-of-cell equipment continued at the High-Radiation-Level Examination Laboratory.

PHYSICS AND MATHEMATICS

Charge Spectrometry. — The magnetic mass spectrometer has been adapted to measurements of charged ions that are formed as a result of inner-shell vacancies produced by x rays.

Krypton was irradiated by x rays from a molybdenum target. An estimated 90% of the vacancies were produced in the K shell by the characteristic Mo K radiation and the bremsstrahlung extending to 50 kev. Ions with charges up to +12 were observed, the most abundant ion being Kr^{5+}. The charge distribution compares favorably with the charge spectrum — corrected for the β^+ decay — that Snell and Pleasonton obtained in 1959

for the electron-capturing nuclide Kr^{79}. An enhancement of the more highly charged ions in the decay of Kr^{79} over those in the irradiation of krypton by x rays may be attributed to a successive internal conversion in the radioactive Kr^{79}. The intensity of Kr^{2+} is higher in either case than the theoretical expectation according to Rubenstein. It is, however, in fair accordance with experimental data of Bower [Math. Phys. Sci. Proc. (1936)] on the L fluorescent yield, tabulated values for the K fluorescent yield, and the reasonable assumption of unity probability for the transition $M_{4,5}$—NN. The intensity of Kr^{+} could not yet be determined because of the interference from ionization by secondary electrons.

The charge spectrum of neon, ionized in the K shell, has been reexamined using the radiation from a molybdenum and a titanium anode. Charges greater than +2 were observed in about 27% of the cases; theory would yield only 16%, while an earlier experiment, using a time-of-flight spectrometer and Al K_α radiation, yielded 31%.

Theoretical Physics: Multipole Moments of a Rounded Charge Distribution. — The multipole moments of a diffuse-edge charge distribution have been calculated, and compared with those obtained for a uniform, or "square," distribution. It has been shown that serious errors may arise if a single "equivalent uniform" radius is used to compute these quantities.

Tables of numerical values have been prepared for some integrals over the Fermi function. They are available in a form suitable for use in evaluating the various multipole moments of the rounded charge distributions of nuclei, both explicitly and in terms of the radii of "equivalent" uniform distributions which have the same moments. Analytic expressions for the integrals were studied.

Theoretical Physics: Spin-Spin Interactions and Scattering from Polarized Nuclei. — The elastic scattering of polarized nucleons from polarized targets was studied, assuming an optical-model potential which includes an interaction of the form $f(r)\ I \cdot S$. Numerical results were obtained, using the distorted-wave Born approximation, for some typical cases, namely, for neutrons of 0.3, 2, and 14 Mev and for protons of 5 and 14 Mev incident on Mn^{55} and Ho^{165} target nuclei. Comparison with experiment can yield the sign and magnitude of the $I \cdot S$ interaction.

Low-Energy Neutron Time-of-Flight Spectroscopy. — A program has been written for the CDC 1604 computer to correct the observed level spacing and the observed $\overline{\Gamma}_n^0/D$ obtained from neutron resonance data for the effects of small "missed" resonances. The correction is based upon a Porter-Thomas distribution of the reduced neutron widths. The sensitivity for finding small resonances "or the cutoff of the Γ_n^0 distribution" is expressed in the form $\Gamma_n^0\ (\text{cutoff})/\overline{\Gamma}_n^0 = cE^b$, where b is ~2 for most of the chopper time-of-flight data, and c depends on the sample thickness and the number of counts over a resonance. The data on the tin isotopes have been corrected for the effects of "missed" resonances. The correction to the observed level spacing for the tin isotopes varied from ~15% to a factor of 3, whereas the correction to the observed $\overline{\Gamma}_n^0/D$ varied from 1% to 50%.

High-Voltage Experimental Program: Possible Evidence of Electric Polarizability of the Deuteron in a Coulomb Field. — Deuteron polarizability, $\alpha = -2\eta/E^2$, where η is the change in binding energy of the deuteron, can be determined by measuring the deviation of the deuteron elastic scattering cross section from the Rutherford expression if the nuclear effect is ignored [Phys. Rev. 128, 2728 (1962)]. The deviations were measured at a scattering angle of 178°, using an annular detector on thin targets of Ni^{58}, Ni^{62}, Ag^{107}, and Ag^{109}. The deuteron beam of negligible energy uncertainty (±0.2%) and energy spread ($\Delta E \approx 0.1\%$) was provided by the ORNL 5-Mv Van de Graaff. The ratios, σ/σ_c, of the deuteron elastic scattering cross section to the Rutherford expression were determined within ±2%. Although the absolute target thicknesses were known within ±8%, observed $1/E^2$ dependence of σ made the absolute normalization possible. The ratios, σ/σ_c, begin to deviate from unity at $E_d \approx 2.2$ Mev, and the deviation at 3.6 Mev is (15 ± 2)% for Ni^{58} and Ni^{62}. The deviation is (5.5 ± 1.5)% for Ag^{107} and Ag^{109} at 3.6 Mev. The polarizability of the deuteron determined from the measured deviations, by neglecting the possible nuclear effect, is $\sim 0.6 \times 10^{-39}$ cm^3, in agreement with the calculated value. The deviation expected due to an optical potential is being calculated by the distorted-wave Born approximation to estimate the contribution due to the specific nuclear effect.

High-Voltage Experimental Program: Some Stopping Powers for Iodine Ions. — The multicomponent heavy-ion beam from the Oak Ridge Tandem Accelerator was used to measure the atomic stopping cross sections of Au, Ni, Al, and C for I^{127} ions in the energy range from approximately 25 to 115 Mev. The results have been published [see Phys. Rev. Letters 11, 284 (1963)].

Mathematics and Programming Research: Partial Differential Equations. — A one-group, two-dimensional criticality calculation is mathematically equivalent to a vibrating membrane. It is well known that when the region governed by the associated partial differential equation is not convex the convergence of the finite difference solution is slow, particularly in the vicinity of a nonconvex corner. For this reason the L-shaped membrane has been widely studied. The customary handling of this problem is to force points of a regularly spaced grid to fall exactly on the boundary. The fundamental frequencies for the finite difference problems thus defined for various grid spacings form a monotonically decreasing sequence (except for very coarse grids) as more and more grid points are introduced. Each value in this sequence is therefore an upper bound for the desired fundamental frequency of the physical membrane. Sharp lower bounds have not been reported. In recent work at ORNL an alternate approach to handling the boundary condition was tried; this approach forces boundaries to fall midway between two rows of grid points. It was found that this leads to a monotonically increasing sequence for the fundamental frequencies as more and more grid points are used. This has produced lower bounds which are just as sharp as the previously available upper bounds. With a grid spacing $h = 1/80$, the lower and upper bounds for the fundamental frequency of vibration of an L-shaped membrane formed by joining three squares, each one unit on a side, were found to be 5.3755 and 5.3792 respectively. The corresponding bounds for the smallest eigenvalue are 9.6323 and 9.6452.

Mathematics and Programming Research: Simultaneous Multiple Processing of Computer Input and Output. — Associated with any large-scale computer is the need to process a job and return the output to the originator as quickly as possible. This process may be broken into three relatively separate operations: (1) preparation of the job as input for the large computer; (2) performing the calculation on the large computer; (3) printing or punching its output. Since printers, card readers, and card punches are slow compared with the large computer, operations 1 and 3 are usually committed to a small computer. In operation 1, cards are copied onto magnetic tape (intermediate in speed between the two computers), which is then moved to the large computer for the computation. Output from the computation is placed on magnetic tapes, which are then moved to the smaller computer and are printed or punched by it.

A rough breakdown of the use, per hour, of the small computer at ORNL has been: printing, 45 min; punching cards, 5 min; reading cards, 10 min. If each of these operations must have sole use of the small computer, an obvious pileup occurs on the printed output if the large computer is to be used as continuously as possible (the large computer normally generates 45 min of output in about 30 min). Clearly, the printing time of 45 min cannot be reduced. However, if the card reading and punching could be made to overlap completely with the printing operation, then one-fourth of the small computer time could be made available for further printing. Pursuing this line of attack, a simultaneous processing code has been written for the CDC 160-A computer and is in use. Card-to-tape and tape-to-punch operations are performed simultaneously at near their maximum speed while printing proceeds at full speed.

When the simultaneous processing code was put into operation, the small computer was used 16 hr/day. After the change to the new method the use was cut to 12 hr/day.

CHEMISTRY

Effects of Radiation on Analytical Methods. — Studies on the effect of gamma radiation from a Co^{60} source on solutions of arsenazo III were continued. The examination of radiolytic decomposition as a function of arsenazo III and acid concentration reveals that, in the strong acid (HCl and H_2SO_4) systems that have been studied, maximum decomposition occurs at the highest acid concentration (9 N) and the highest arsenazo III concentration (10^{-4} M). The rate of radiolytic decomposition, in this case, averages approximately 4×10^{-6} mole of arsenazo per liter per kilorad of absorbed gamma radiation. The least amount of radiolytic decomposition was observed in 1 M acetic acid solutions, where, for a 10^{-4} M arsenazo III solution, the rate of decomposition averages approximately 9×10^{-8} mole per liter per kilorad of absorbed gamma radiation. This impressive radiation stability in acetic acid solutions is observed, however, only for the first 72 kilorads of absorbed radiation. On further exposure to gamma radiation, the rate of decomposition increases approximately 100-fold, after which a postirradiation decomposition also occurs.

In the study on the effect of gamma radiation on the colorimetric nickel dimethylglyoxime (DMG) method, it was found that the radiation effect is a function of the DMG concentration. The lower the DMG concentration, the less is the effect of a given quantity of radiation. The postirradiation effect noted in a previous report has been tentatively ascribed to those DMG solutions containing ethanol. Those solutions containing only the sodium salt of DMG apparently do not show this effect.

Some effort has also been directed toward an unambiguous identification of chlorine as one of the products of the radiolysis of acidic 10 M LiCl solutions. The o-tolidine test is not affected by hydrogen peroxide, but it is not completely specific for this purpose. The irradiated solution reduces bromide to free bromine, thus indicating an oxidizing potential at least greater than that of bromine. Work is in progress to extract the chlorine from solution, for positive identification of this component by gas chromatography.

Chemical Engineering Research: Stacked-Clone Contactor. — The development of the stacked-clone contactor for solvent extraction has reached the point of proven reliability as a low-holdup, high-efficiency device. A parametric study produced a prototype (Mark X) which has a total flooding capacity of 4 liters/min at an aqueous/organic phase ratio of 3 for the system 1 M $NaNO_3$–18% TBP-Amsco at 40°C. The flooding locus is expressed by $\Phi = \Phi_0 - 0.32A$, where Φ is organic flow, A is aqueous flow, and Φ_0 is the intercept, which is about 2 liters/min, varying with configurations. The stage efficiency based on both the extraction and the stripping of uranyl nitrate is greater than 70%, which, with a volume of 212 cc per mechanical stage, results in a residence time of less than 4.5 sec per theoretical stage.

Chemical Engineering Research: Solvent Extraction. — The instantaneous pressure drop across pulsed columns containing both sieve and nozzle plates was determined in order to calculate an effective orifice coefficient for the plates. The coefficients ranged from 0.6 to 1.1, increasing with increased pulse velocity. Comparison with coefficients measured with steady flow (no pulse) showed good agreement at the same Reynolds number.

Separations Chemistry Research. — Di(sec-butyl)phenylphosphonate, a reagent very effective in separating uranium and thorium by solvent extraction, must be used in a predominantly (more than 75%) aromatic diluent to ensure the organic-phase solubility of the extracted uranium. A similar compound, but with a methyl group replacing one of the hydrogens on the benzene ring, permitted the use of a diluent that was 25% aromatic and 75% aliphatic. The uranium extraction and separation from thorium was unaffected by the change in reagent structure.

Separations Process Development. — A process was outlined for recovering beryllium from sulfuric acid leach liquors of low-grade beryllium ores by extraction with 1-(3-ethylpentyl)-4-ethyloctylamine in hydrocarbon diluent at pH ~2, scrubbing with sulfuric acid (possibly containing a small concentration of a sequestering agent to improve the separation from aluminum) at pH 1.5, and stripping with 0.5 M H_2SO_4.

Thorium recoveries in leaching Conway granite with sulfuric acid were about the same (about 80%) for coarse-ground (–4 mesh) ore as for ore ground to –48 mesh. Acid consumption was significantly lower for the coarse material.

A new method for separating trivalent actinides from lanthanides consists in extraction by a monoacidic phosphate or phosphonate from a solution containing carboxylic and aminopolyacetic acids. Results of continued studies of the method support the following conclusions: Diethylenetriaminepentaacetic acid gives the largest group separations of several aminopolyacetic acid complexing agents tested; lactic acid gives the best performance, especially in phase separation, of numerous carboxylic acids that could be used as the major aqueous constituent; di(2-ethylhexyl)phosphoric acid gives better group separation factors, though lower distribution coefficients, than 2-ethylhexyl phenylphosphonic acid; and diisopropylbenzene is the preferred diluent for use in the first cycle of a process wherein the lanthanides are extracted from the actinides.

Preparation and Properties of U(IV) Sols and UO_2. — Attempts to produce urania in a manner analogous to that used in the sol-gel process for thoria preparation have been hampered, in part, by uranium oxidation by nitrate ion as nitrate-stabilized sols are evaporated to form gels. Efforts to remove nitrate by addition of formic acid to the catholyte during the electrolytic reduction of uranyl nitrate in a two-compartment cell using an anion exchange membrane were successful. However, flocculated suspensions rather than sols were formed both in this system and in one involving the electrolytic reduction of uranyl formate solutions. Dried (90 to 100°C in argon) products from these flocculated suspensions showed some promise of producing dense UO_2. Efforts to reduce residual ammonia content by washing the hydrous oxides precipitated with ammonia from uranium(IV) nitrate solutions resulted in improved dried and fired UO_2 products. Nitrate-stabilized sols prepared from the above hydrous oxides flocculated while being dried at 90 to 100°C in an argon atmosphere.

Radiolysis of Metal Fluorides. — The detection of elemental fluorine and carbon tetrafluoride in the cover gases following in-pile irradiation of MSRE fuel salts suggested that several aspects of the radiolysis of metal fluorides could conveniently be studied by using x rays. In experiments to date we have used 250-kv x-ray units. The test salts have been contained in prefluorinated thin-walled nickel containers in the presence of helium gas. The energy absorbed by the salt was determined by difference between the energy passing through an empty container and that passing through a container filled with salt.

The first test salt was an MSRE fuel type, containing lithium, beryllium, zirconium, thorium, and uranium fluorides in mole fractions of 0.7, 0.23, 0.05, 0.01, and 0.01 respectively. Irradiation resulted in the generation of CF_4 and COF_2 but no fluorine. A second sample of this salt was fluorinated at 150°C in an irradiation container until the appearance of traces of UF_6 in the cover gas. Irradiation of this sample resulted in liberation of F_2 and CF_4 with traces of COF_2 and SiF_4.

The first test salt was found to contain a wide range of particle sizes. Additional samples of this salt were screened, and two size ranges were selected for further irradiation tests. Seventy grams of fine particles (< 2 mils in diameter) was irradiated to an absorption of 9.5×10^{16} Mev, with no evidence of volatile fluorine compounds in the helium cover gas. It was believed that contamination of the particles with water vapor during the sieving operation had interfered with the liberation of fluorine compounds. To remove surface moisture and oxide, the salt was

fluorinated at about 50°C and reirradiated to a total dose of 7.9×10^{16} Mev. Mass spectrometric analyses of the cover gas during this period again disclosed CF_4, COF_2, and higher fluorocarbons, but no elemental fluorine. The yields of these compounds were equivalent to an average $G(F_2)$ value of 0.02, with no apparent dependence on dose. A coarse fraction of the same salt, containing particles 23 to 33 mils in diameter, was fluorinated at 50°C. Irradiation of this salt to a total dose of 6.3×10^{16} Mev generated no volatile fluorine compounds. Comparison of these results with those previously reported for particles 30 to 50 mils in diameter exposed to electron bombardment (ORNL-3491, p 19) suggests that particle size may be an important parameter affecting the release of fluorine from irradiated salt.

The various components of the MSRE fuel salt are being tested to determine whether they are equally affected by x-ray irradiation. Lithium fluoride and zirconium fluoride have both been irradiated to higher total absorbed doses than those given to the MSRE fuel salt without any observed generation of volatile fluorine compounds. The zirconium fluoride was then fluorinated at 150°C and reirradiated; again no volatile fluorine compounds were generated. Seventy grams of the ternary salt $6LiF \cdot BeF_2 \cdot ZrF_4$ was irradiated to an estimated absorbed dose of 1.7×10^{17} Mev without generation of fluorine compounds. After fluorination at 160°C, irradiation to a dose of 1.6×10^{17} Mev resulted in an average $G(F_2)$ value of 0.011. Five such values were determined during the exposure with only minor variations and no evidence of dose dependence. From these results it appears that the composition of the test salt may also be an important parameter affecting its susceptibility to radiation decomposition.

Inorganic and Nuclear Chemistry of Technetium. — The infrared spectra of Na_2MoO_4(cryst) and Na_2WO_4(cryst) were observed from 2300 to 300 cm^{-1}. Consistent with the tetrahedral ions being in T_d site symmetry, each spectrum exhibited two fundamental bands essentially coincident with two of the four Raman lines previously observed. The significant information obtained is that the triply degenerate vibration, $\nu_4(f_2)$ (infrared active), lies below the doubly degenerate vibration $\nu_2(e)$ (infrared inactive). This represents an inversion of the normal behavior shown by ClO_4^-, SO_4^{2-}, PO_4^{3-}, and SiO_4^{2-}, where $\nu_4(f_2) > \nu_2(e)$. Since the Heath-Linnett orbital valence force field calculations always give $\nu_4 > \nu_2$, this type of force field calculation is not applicable to these ions. The Raman spectra of VO_4^{3-}(aq), ReO_4^-(aq), and $OsO_4(\ell)$ are similar to those of MoO_4^{2-}(aq) and WO_4^{2-}(aq); that is, all have $\nu_1(a_1) > \nu_3(f_2)$, which also represents an inversion of normal behavior ($\nu_3 > \nu_1$). The applicability of the Heath-Linnett type of calculation to these two ions and this molecule, as well as its applicability to TcO_4^-(aq) (where $\nu_1 \approx \nu_3$), is at least questionable. The infrared and Raman spectra of Na_2MoO_4(cryst) and Na_2WO_4(cryst) afford the first unequivocal assignment of $\nu_2(e)$ and $\nu_4(f_2)$ for any tetrahedral oxyanion in this region of the periodic table.

Nuclear Chemistry: Neutron Cross Sections. — A measurement was made of the thermal cross section and the resonance integral of the reaction $U^{232}(n,\gamma)U^{233}$. A sample of U^{232}, free of U^{233} to less than 1%, was obtained from neutron-irradiated Pa^{231}. Using a cadmium filter technique and dilute alloys of cobalt to monitor the flux, samples were irradiated up to about 6×10^{19} neutrons/cm^2. Following chemical purification of

the uranium, determinations of the U^{233}-U^{232} content were carried out with a 12-in. two-stage mass spectrometer using thermal ionization and electron multiplier detectors. Values of 79 ± 5 and 280 ± 20 barns were found for the thermal (subcadmium) cross section and the resonance integral, respectively, for neutron capture in U^{232}. These values may be compared with the estimate of Elson et al. [Phys. Rev. 89, 320 (1953)] of 200^{+300}_{-200} barns for a reactor spectrum cross section of U^{232}.

Chemical Physics: Microwave and Radio-Frequency Spectroscopy. — Five different paramagnetic resonance spectra were observed in single crystals of H_2O_2 after photolysis with ultraviolet light. One of these spectra arises from a triplet-state species, is indefinitely stable at 77°K, and is formed in very low yield. The predominant spectrum of the five is formed in high yield and disappears with first-order kinetics with a half-life of about 1 hr at 77°K, as described previously. Detailed measurements of this spectrum were completed for two crystallographic planes, and the analysis of the data progressed far enough to show clearly that this species is also in a triplet state (S = 1). The experimental observation of half-field transitions confirmed this analysis. The D term of the Hamiltonian (strength of electron spin-spin interaction) is about 0.01 cm^{-1}. Hyperfine effects from protons are present but are not yet analyzed. The OH and HO_2 radicals have always been likely candidates for any spectrum formed in H_2O_2, but neither can be responsible by itself for this predominant species.

Physical Chemistry of Molten Salts. — The crystal symmetry of $NaF \cdot 2UF_4$ was inferred from single-crystal x-ray data. A single crystal of the solid solution between $NaF \cdot 2UF_4$ and $KF \cdot 2UF_4$, with an Na:K mole ratio of about 2.5, was examined by x-ray diffraction. It was determined to be orthorhombic, with a = 8.60 A, b = 7.02 A, and c = 11.17 A. From systematic absences of possible diffraction maxima, the probable space group was deduced to be $Pna2_1$ or Pnam. The close similarity of these crystal parameters with those of $KF \cdot 2UF_4$ (a = 8.68 A, b = 7.02 A, c = 11.44 A; Pnam), reported by Zachariasen [J. Am. Chem. Soc. 70, 2147 (1948)] on the basis of x-ray powder data, and the results of phase studies which showed complete miscibility of $NaF \cdot 2UF_4$ and $KF \cdot 2UF_4$ indicate that these compounds are isomorphous.

High-Temperature Aqueous Solution Chemistry. — The electrical conductance of aqueous solutions of $KHSO_4$ (0.0000737 to 0.00505 m) has been measured at temperatures from 0 to 800°C and at pressures up to 4000 bars. From some of these results the second dissociation constant of sulfuric acid has been calculated to 300°C. The dissociation constant decreases with increasing temperature and increases with increasing pressure. Above 400°C, even at pressures as high as 4000 bars, the dissociation constant is too small ($\sim 10^{-7}$) to be evaluated accurately by the present method. At temperatures below 300°C, pressurizing the solution results in a large increase (~40% increase from 1 to 4000 bars at 100°C) in the specific conductance of the solutions. From the change in dissociation constant with pressure at constant temperature, the volume change for the reaction $HSO_4^- \rightarrow H^+ + SO_4^{2-}$ is estimated to be approximately −31 cc/mole, at temperatures to 300°C.

Chemical Separation of Isotopes. — The effect of eluent (HCl) concentration on the fractionation factor, α, for lithium isotopes on Zeo-Karb was measured at ~25°C. A small but uniform dependence of α on acidity was observed, as shown by the data in the following table:

Normality of HCl	Flow Rate (cm/sec)	Fractionation Factor, α
5	1.31×10^{-3}	1.00078
1	1.55×10^{-3}	1.0013
0.5	5.40×10^{-4}	1.0017
0.1	1.27×10^{-3}	1.0022

The decrease in the fractionation factor with increasing acidity appears to result chiefly from changes in the composition of the secondary solvation sphere of the aqueous-phase ions.

TRANSURANIUM-ELEMENT PRODUCTION

Chemical Process Development: Tramex Process. — The following studies were directed toward improving and testing the Tramex process, which will be used in both the Transuranium Element Program and the Curium Processing Program, for separating transplutonium elements from impurities.

Tramex feed solution at 10 w/liter loses nearly 0.1 mole of HCl per liter per day through radiolysis. This is a potentially serious problem, since acid concentration must be 0.1 M or less for satisfactory extraction. However, it has been discovered that certain reducing agents added to the feed can effectively eliminate acid loss by radiolysis. Over a 25-day period, the acid concentration in feed containing 1 vol % formaldehyde gradually increased to 0.22 M HCl and then decreased until curium hydroxide precipitated. Feed containing 10 vol % methanol was stable even after 25 days; during the entire 25 days, the acid concentration was 0.03 to 0.06 M HCl. Effects of methanol on the Tramex extraction must be determined.

A series of five tests made in small-scale (5 to 10 ml) batch countercurrent equipment demonstrated that no adverse effects on process chemistry resulted from Cm^{242} activity levels of 5 to 10 w/liter. The overall results indicate that the Tramex process is capable of producing completely decontaminated curium in high yield; however, good recovery and high decontamination were not both demonstrated in any one of the five tests. Behaviors of curium and fission products in these tests were all in agreement with previous results. The most significant items reemphasized by this series of high-activity-level tests are: (1) curium will reflux in the extraction section if the feed acidity is more than about 0.1 M; (2) rare-earth decontamination is poor because of slow rates in

the scrubbing section unless contact times are about 1 min or the operating temperature is about 60°C; (3) zirconium decontamination is good if the acidity in the stripping section is high enough (about 8 M HCl); and (4) small amounts of ruthenium are stripped with the curium unless nitrite is used in the stripping section.

The Cm^{242} product from the process will be in 8 M HCl. Conversion to the required nitric acid solution by repeated additions of concentrated nitric acid followed by evaporation offers a possible method for obtaining additional ruthenium decontamination if RuO_4 can be distilled from the final product. Scouting tests show that strong oxidants must be present to accomplish this. With 0.0004 M $NaBiO_3$ in boiling, concentrated nitric acid, decontamination factors were 70 to 100. With ozone bubbling through boiling, concentrated nitric acid 0.02 M in nitrite, the decontamination factor was about 5.

Small quantities of nitrate in the Tramex process greatly increase actinide distribution coefficients. However, lanthanide distribution coefficients are increased more, and the resulting actinide-lanthanide separation factors are reduced. Furthermore, distribution coefficients are more sensitive to acid changes when nitrate is present. These results indicate that control of process conditions will be much easier if nitrate is not present.

Chemical Process Development: Separation of Actinides and Lanthanides by Carboxylic Acid Complexing. — Cerium-americium and europium-americium separation factors of nearly 100 or more can be obtained by the selective extraction of lanthanides from mixed solutions of carboxylic acid and pentasodium diethylenetriaminepentaacetate (Na_5DTPA) into either 2-ethylhexyl phenylphosphonic acid (2EHΦPA) or di(2-ethylhexyl)phosphoric acid (HDEHP). At conditions for optimum separation of americium and curium from the lanthanides, europium was usually less extractable than cerium, but at lower Na_5DTPA concentration or lower pH, europium was more extractable than cerium. Lanthanide-actinide (americium-curium) separation factors of greater than 100 were obtained with the use of any of the following carboxylic acids: glycolic, lactic, malonic, and citric. Somewhat poorer separation was obtained when mandelic acid was used. Americium extraction into HDEHP in n-dodecane diluent was about 10 times as great as extraction into HDEHP in diisopropylbenzene diluent.

Although lanthanide-americium and lanthanide-curium separation factors of nearly 100 can be obtained for this system, lanthanide-californium separation factors are considerably less. Separation factors are different when different aminopolycarboxylic acids are used to complex the actinides. The maximum europium-californium separation factor obtained to date is about 20.

A product containing 80% of the curium contaminated with only a trace of Ba^{140} was obtained from a mixer-settler demonstration of the new extraction process in which the feed was spiked with neutron-irradiated Am^{241}. For this test, lactic acid and Na_5DTPA were used, and the extractant was 2EHΦPA in diethylbenzene. Conditions were adjusted to extract lanthanides in the first contactor and to extract americium-curium into 1 M HNO_3 in the third. Lanthanides were quantitatively extracted in the first contactor and were not contaminated with any other fission products. About 20% of the curium remained in raffinate from the second contactor, apparently because of the slow rate at which the curium-Na_5DTPA

complex broke. Traces of Zr^{95} and Ba^{140} extracted in the second contactor, and in the stripping contactor this Zr^{95} remained in the solvent, but the Ba^{140} stripped with the curium. Ruthenium remained in the raffinate from the second contactor. Fission product behaviors in this demonstration run were consistent with distribution coefficient data obtained in batch tests with tracers.

Equipment Development. — Mockup tests are continuing in order to demonstrate and improve equipment design concepts. Representative jumper lines were fabricated to determine if they could be installed remotely in the cubicle with the master-slave manipulators. The problem of remote connection at the equipment-rack service lines was investigated; an alignment jig will be required to ensure proper fit. Preliminary testing of the transfer case for equipment removal was started.

Batch phase-separation tests in the system 5 M HCl—1 M HNO_3 vs 30% Alamine 336 in diethylbenzene gave break times considerably shorter than those for the Tramex flowsheet (10 M LiCl vs the same organic). The former system is used for nitrate removal to prepare feed for the Tramex cycle. Pulsed-column tests confirmed the greater ease of phase separation; in 3/4-in.-diam columns with 5% free area, total throughputs of 12 cc/min were achieved at 25°C, comparable to throughputs with the Tramex system at 50°C.

Transuranium Facility. — Installation of equipment for the TRU development facility in cell 4, Building 4507, is nearly completed, and cold testing is under way. Design for the supporting facility in cell 3, including the dissolver installation, was begun; procurement of the dissolver was initiated. Construction of the Transuranium Processing Plant, started August 1, is continuing; installation of underground sewer lines, the 30-in. cell-ventilation duct beneath the cell bank, and the floor pad for the waste-tank pit was completed. Procurement of critical items by ORNL for installation by the contractor is proceeding on schedule. A simulated removal and replacement of an alpha-seal window was satisfactorily demonstrated by the vendor this month prior to his proceeding with window fabrication. Design of the chemical process equipment is 33% complete. Drawings of the sampler and the first-cycle solvent extraction equipment rack were approved and issued. Fabrication of the latter was started in the ORNL shops. Procurement of 16,000 ft of Zircaloy-2 tubing for the process piping system was initiated.

Analytical Chemistry Development. — The very high levels of alpha activity associated with solutions of the transuranium elements create a serious hazard in the analytical laboratory. Methods for separating various fission product nuclides from the alpha-active materials prior to analysis are being devised. In the case of fission product zirconium, this may be accomplished by preferentially extracting the zirconium with 0.5 M trifluorotetraacetic acid in xylene from 6 M HNO_3—0.5 M $NH_2OH \cdot HCl$. For cerium and other rare earths, extraction was with 0.1 M 2-ethylhexyl phenylphosphonic acid in pentane from 1 M α-hydroxyisobutyric acid—0.1 M pentasodium diethylenetriaminepentaacetate (Na_5DTPA). The trivalent rare earths are then preferentially stripped from the solvent with 10 M HNO_3—2 M $NaBrO_3$, which oxidizes the Ce^{3+} to Ce^{4+}; the Ce^{4+} remains in the solvent.

A new, direct, nondestructive method for the determination of Cf^{252} was developed. The method is based on gamma scintillation spectrometry involving the measurement of the prompt gamma rays coincident with the spontaneous fission of Cf^{252}. Californium-252 has an alpha half-life of 2.55 yr and a spontaneous fission half-life of 66 yr. The gamma-ray spectrum in coincidence with the spontaneous fission extends to about 5 Mev. Each fission event yields an average of 10 to 12 gamma rays. The technique may be used either for the rapid qualitative or for the quantitative determination of Cf^{252} in the presence of associated fission products and actinides. As little as 10^{-2} nanogram of Cf^{252} can be quantitatively measured. The method has several useful analytical and process applications.

HFIR Target Fabrication Development. — The final phase in the fabrication of prototype targets for irradiation experiments is under way. Continued difficulty is found in producing porosity-free welds, although no difficulty is encountered in making them leaktight to helium. Mechanical tests indicate that the strength of the welds containing some porosity is sufficient to withstand pressures that burst the tube wall. Some additional conceptual design for the remote fabrication equipment was initiated. The completed models of the cap-powder dispenser, blender-dispenser, and transfer arm were received from the fabricator and are being bench tested. Some changes in the process line were made in order to provide an inspection step before the pellets are irretrievably locked into the target tube.

METALLURGY AND MATERIALS

Physical Properties Studies. — We have measured the thermal conductivity of Armco iron from 100 to 950°C in the radial heat flow apparatus. We also measured the electrical conductivity and total hemispherical emittance of the same material. The thermal conductivity to 780°C could be described within ±1.5% by two linear equations, with a slope change at 410°C. The thermal conductivity varied little between 780 and 910°C. The electronic contribution to the thermal conductivity was calculated from the measured electrical conductivity, and the lattice contribution was obtained by difference. The electronic contribution dominates the thermal conductivity and its temperature dependence. The lattice contribution, however, is strongly temperature dependent, showing a maximum at 300°C of 0.114 w cm^{-1} $(°C)^{-1}$, 21% of the total thermal conductivity at that temperature. The Curie transformation at 768°C significantly affects the temperature dependence of the electronic contribution but has little effect on the lattice contribution. The slope change at 410°C can be predicted from the electrical conductivity and coincides with a minimum in the thermoelectric power of iron. The alpha-gamma transformation at 910°C lowered the total thermal conductivity by about 4%, although the electronic portion was affected by less than 1%; thus the transformation affected primarily the lattice portion. The total hemispherical emittance varied with temperature less than predicted by classical models based on electrical resistivity.

We have determined the total hemispherical emittance to 1000°C on a series of type 316 stainless steel specimens possessing a variety of surface conditions. The emittance of bright-polished specimens depended on the system pressure in the temperature range 600 to 950°C. At 5×10^{-6} torr the emittance increased significantly with time below 800°C, because of oxidation, but decreased at higher temperatures, because of oxide decomposition. A specimen preoxidized to form a surface layer of Cr_2O_3, which had an emittance 3 times that of the bright-finished specimen, also decreased in emittance.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. — Some plasma studies were performed in order to determine the effects of adding electron cyclotron heating in the confining mirror field. These experiments were performed in a slightly asymmetric mirror that resulted from a 10% short in one coil. The maximum microwave power levels available were of the order of 20 w. The use of 35-kMc microwaves (appropriate for heating in resonance zones off the median plane) had no conclusive effect on the plasma parameters studied (the axial distributions of the flux of charge-exchange neutrals, the energy distributions of these neutrals in the median plane, and the proton containment time) at either high or low pressures. With 26.8-kMc microwaves (for heating in the median plane) effects were observed at the lower pressures. In operation at maximum power input with containment times longer than 0.6 sec, the axial distributions of the charge-exchange-neutral flux changed to indicate a smaller plasma volume. The central fast-proton density increased 20 to 25% at the longest containment times (about 7 sec). The energy distributions showed evidence of more proton energy degradation, but this increased degradation was so slight that it did not affect the measured containment times. Studies of these effects as a function of the 26.8-kMc power level at the longest containment times showed essentially no changes as the power was reduced two orders of magnitude. Further reductions resulted in a decrease in the magnitude of the plasma effects, but they were still observable at the lowest nonzero level available, which is estimated to be at least three orders of magnitude below maximum power.

The shorted coil was replaced, and a new liner that permits a plasma extent from Z = ±6-1/2 in. unobstructed out to a radius of 11 in. was installed.

A series of rf measurements of the signals from the plasma instability was made under a variety of operating conditions, using the most sophisticated of the probe and electronic systems that have been devised. Some time will be required for complete analyses of these data, but a preliminary study of the results has shown that they will add substantially to an understanding of the nature and role of the rf fields. In particular, it is now realized that there is not one, but rather several modes of collective behavior at low pressures. Some of the experiments were performed in conjunction with the 26.8-kMc microwave studies described above. In operation at low pressures without microwave power, the rf signals show polarizations that indicate the presence of both azimuthal

and axial currents, the axial currents being associated with electron currents to the end walls. The application of sufficient microwave power damps out both the axial rf and the axial current signals, and to some extent smooths and makes more regular the azimuthal current signals. The power threshold for these effects is only slightly above the minimum non-zero level available.

Beam-Plasma Interaction. — An effort was made to find the direction that future work with the hot-electron plasma by beam-plasma interaction should take. An extrapolation of the $1/\tau$ dependence on pressure passed through the origin. It seems then that stability is not a problem with a hot-electron plasma generated in this manner. This is in agreement with the calculated τ value based on only scattering and mirror losses. The electron temperature was found to vary inversely linearly with background pressure. An extrapolation to a background pressure of 10^{-6} torr indicated that we might expect an electron temperature of 2.5 Mev. It should be possible to maintain this degree of vacuum with but little additional pumping.

Measurement of the local energy density in the hot-electron plasma by means of the diamagnetic deflection of an ion-beam probe was studied theoretically. Calculations indicate that in the cigar-shaped plasma of the arc research facility, the method should work well. In one preliminary experiment, an ion-beam deflection of the correct order of magnitude was observed.

Theory. — A threshold condition has been calculated for onset of the usual two-stream instability in an infinitely long, cylindrical beam—plasma system of radius A which is coaxial with and contained in a cylindrical conducting wall of radius B. The resulting dispersion relation is identical with the infinite beam—cold plasma dispersion relation if the electronic charge, e, is replaced by

$$kA\left(\frac{1}{2}\,\ell n\,\frac{B}{A}\right)^{\frac{1}{2}} e\ ,$$

where k is the propagation constant of the wave, and A and B are the geometrical constants defined above.

Radio-Frequency Heating. — The new scaleup of the electron cyclotron experiment in the EPA facility has been in successful operation since the end of July. The plasma is created in a 3:1 magnetic mirror, by a 50-kw continuous-wave 10.6-gigacycle power source. Preliminary results at 20-kw continuous-wave input power show that the plasma contains ~150 j of stored energy and occupies a volume of ~50 liters. The ratio of plasma pressure to magnetic pressure, β, is about 0.2. The plasma thus appears to have scaled in stored energy and volume directly with the microwave power.

The containment time shows an increase over that obtained at a few kilowatts in the PTF. The decay time of the energy density is ~0.4 to 0.5 sec, as opposed to ~0.1 to 0.2 sec in the PTF. In addition, the decay time of the microwave noise has increased by a factor of about 2. The neutron emission, mainly from Coulomb dissociation of the deuterium gas, has increased by an order of magnitude, approximately proportional to power. The neutron spectrum has not yet been determined.

BIOLOGY AND MEDICINE

CHEMICAL CO-CARCINOGENESIS

Effects of Chemical Carcinogens on Microorganisms. — Wild-type (prototrophic) *Aerobacter aerogenes* has been rendered deficient for leucine (by the chance isolation of a spontaneous auxotrophic mutant), for methionine (by selection of a spontaneous auxotrophic mutant with the penicillin cycling technique), for uracil (also by the penicillin cycling technique), and for thymine (by cultivation in the presence of aminopterin and 5-bromodeoxyuridine). Currently, this quadruple auxotroph of *A. aerogenes* is being manipulated so as to acquire slow-growing, starvation-resistant derivatives. Once acquired, the slow-growing quadruple auxotroph will be compared with the rapid(normal)-growing quadruple auxotroph. The purpose of introducing the multiple auxotrophy is to enable specific labeling of intracellular macromolecules. The general aim is to discover the controls affecting growth rate and survival, and the possible effect of chemical carcinogens on these controls.

RADIATION EFFECTS

Statistical Services and Research. — A nonlinear least-squares computer program was modified to provide automatic plots of the fitted equation. For those problems in which a random variable, Y, is a nonlinear function of a nonrandom variable, X, the program will furnish a linear or a semilogarithmic graph of the function f(X) on a plot of the observed points (X,Y). The vertical and horizontal scales are labeled in the units of the observational data at intervals which are multiples of 1, 2, 4, and 5 units per inch. Curves of the same family for as many as six sets of data may be plotted on the same graph.

RADIATION GENETICS

Mammalian Biochemical Genetics. — Hemoglobins possessing different physicochemical properties have been found among inbred strains of laboratory mice. The properties of the hemoglobin molecule are reflections of the primary structures of their α and β chains of amino acids. The chains can be fragmented through trypsin digestion. Separations of these tryptic peptides can be accomplished by column chromatography on Dowex 1-X2, Dowex 50-X2, and Sephadex G-25 resins. The α chains of C57BL and BALB/c hemoglobins contain 14 tryptic peptides. Analyses with the Spinco automatic amino acid analyzer reveal one amino acid difference; namely, a substitution of aspartic acid for threonine in peptide αT-9. This peptide contains 29 amino acids, and the exact position of the substitution is as yet unknown. The substitution of this amino acid markedly reduces

the solubility of BALB/c, as compared with C57BL, hemoglobin in KH_2PO_4-K_2HPO_4 buffer. However, BALB/c hemoglobin remains soluble in physiological fluids, in contrast to the insolubility of sickle-cell hemoglobin of humans under similar conditions.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — Lethally irradiated $1C3F_1$ mice treated with homologous (LAF_1 or $B6D2F_1$) bone marrow cells (BMC) irradiated with 400 r in vivo survived 90 days as well as did those treated with similar, but unirradiated, BMC. All surviving chimeras from both groups had exclusively donor-type erythrocytes at 90 days. All chimeras tested (19, representing both groups) had donor-type lymph node cells in the fourth month after transplantation. Ten additional survivors, tested ten months after irradiation and treatment, retained lymph node and red cell transplants. Data were not obtained that would suggest that sublethal irradiation of donor BMC suppresses secondary disease. The present findings are in agreement with evidence that the radiosensitivity of immunologically competent cells is similar to that of hemopoietic cells. Thirty-seven chimeras were tested for the presence of recognizable chromosomal aberrations. These were found to be more frequent in the recipients of irradiated marrow than in the recipients of unirradiated marrow. Cloning of cells containing aberrations, evident in both groups, was also more frequent in the animals treated with irradiated marrow.

Tissue Culture. — Blood samples were obtained 1 yr after irradiation from three men involved in the Puerto Rico Nuclear Center accident. The samples have been scored for chromosomal aberrations, and karyotype analyses are in progress. Scoring and karyotype analyses of the samples taken 5 yr after irradiation from the eight men involved in the Y-12 accident have been completed.

Fractionation experiments have been made on the Chinese hamster in order to test the effect of dose rate on rejoining and repair of chromatid aberrations produced by ionizing radiations. In addition, an attempt is being made to synchronize mitoses in tissue culture by the addition of 5-aminouracil to the culture media.

MOLECULAR- AND CELLULAR-LEVEL STUDIES

Cell Growth and Reproduction. — Earlier histological observations led to the notion that in the growing lens of vertebrates the lens epithelium is an actively dividing system and gives rise to cells which migrate into the interior of the lens and transform into lens fibers without further cell division. Some of the data obtained in our study of the regenerating lens furnished information concerning protein synthesis, deoxyribonucleic acid synthesis, and appearance of lens antigens during the cellular transformation. Autoradiographic data presented here concern the pattern of ribonucleic acid synthesis occurring in the cells involved in this transformation.

Fifty microcuries of H^3-uridine (1.0 curie/millimole) was injected into the body cavity of the adult newt (Triturus viridescens) in which the regenerated lens shows well differentiated primary lens fibers. Twelve hours later the animals were sacrificed, and serial sections through the regenerated lens were studied with autoradiography. According to a control with ribonuclease, about 90% of the silver grains observed in the autoradiographs should be due to activity in RNA.

Studies of grain counts per unit area led to the following results: (1) The lens epithelium shows a grain count (12.81 ± 3.41), which is significantly higher than that of the lens fibers of the same lens (1.69 ± 2.01). (2) Within the lens epithelium a gradient in the grain counts is present, which has its apex at the anterior pole and decreases toward the equatorial zone. (3) With the progress of growth, the ratio between the grain counts of lens epithelium and lens fibers shifts in favor of the former. The shift is mainly due to the decrease of grain counts in the fiber area. (4) The cytoplasmic grain count varies from 36 to 50% of the total grain count in the different areas of the growing lens. The highest value is found in the equatorial zone of the lens epithelium.

Autoradiography of the same system, using H^3-thymidine, revealed that DNA synthesis is restricted to the lens epithelium. This is in harmony with the strict localization of mitosis in the same area. By contrast, RNA synthesis is demonstrated in both epithelium and fibers. If we assume that the activity in cytoplasmic RNA suggested in the present experiment is due to RNA synthesized in the nucleus and transferred to the cytoplasm, then our data indicate a quick RNA transfer in the equatorial zone of lens epithelium, where cells are being transformed into lens fibers. Probably this is connected with the following earlier observations: (1) The lens fibers show a percentage of cytoplasmic protein synthesis which is significantly higher than that of the lens epithelium. (2) The main part of lens-specific antigens appears in the cytoplasm of the cells which are beginning to transform into lens fibers. Those data taken as a whole suggest a hypothesis that the lens epithelium cells are very active in RNA synthesis and that a part of the RNA thus produced is transferred into the cytoplasm and forms the protein-synthesizing apparatus that carries information for lens-specific macromolecules.

Biophysics: Cytochemical Studies of Radiation Damage and Repair. — Physarum polycephalum, a slime mold which undergoes synchronous mitotic divisions, is delayed mitotically for 6 to 8 hr after exposure to 25 kr of x rays. Apparently normal divisions occur thereafter. Preliminary experiments indicate that H^3-thymidine, a labeled precursor of deoxyribonucleic acid (DNA), is incorporated into DNA during the x-ray-induced mitotic delay, whereas microspectrophotometric measurements show no net increase in total DNA. These results, although qualitative and preliminary in nature, suggest that repair or recovery from x-ray-induced damage may involve replacement of parts of the DNA molecule.

Biophysics: Activation Energies of Free Radicals Produced by Irradiation of Amino Acids. — Preliminary analysis of our decay data for the concentrations of radiation-induced free radicals in polycrystalline amino acids has yielded the following set of activation energies (in ev/molecule): glycine, 0.53; alanine, 0.85; glutamic acid, 0.26; and glycyl glycine, 0.47. Measurements are being continued. Measurements on leucine

have been started. Plans for more rigorous statistical analysis are under way.

Biophysics: Molecular Mechanism of Radiation Repair. — Bacteria that are resistant to ultraviolet irradiation have mechanisms which remove the principal ultraviolet lesion (a thymine dimer) from their DNA. We are attempting to determine whether the resulting gaps in DNA strands are repaired and, if so, how. If only normal TT sequences replace the lesion, all the radioactivity incorporated by a bacterial culture that has been irradiated and then placed in H^3-thymidine will be associated with thymine pairs. We have devised a technique for measuring the number of thymine pairs in a DNA. Our data indicate that in the process of radiation repair, other nucleic acid bases than just thymine pairs are incorporated.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Basic Instrumentation. — A spatial radiation detector was recently developed. This semiconductor detector yields information concerning not only the energy of a nuclear event, but also its location along a line. In the last few months, detector geometries capable of determining the location of an event in two dimensions were studied. In addition, a vacuum chamber, within which it is possible to precisely move the detector relative to the radiation source, was designed and constructed in order to study these devices.

Low-noise semiconductor preamplifiers were studied to determine their optimum performance characteristics in nuclear applications. The following transistorized instrumentation was developed for use in measuring the noise of semiconductor devices and amplifiers: a low-noise voltage-sensitive preamplifier, a resistance-capacitance wide-band passive filter, and a twin-tee narrow-band active filter. Three charge-sensitive preamplifier configurations employing field-effect transistors in the input stages were developed and tested.

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