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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1964

OAK RIDGE NATIONAL LABORATORY
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1964

DATE ISSUED

April 6, 1964

OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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UNION CARBIDE CORPORATION
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MARCH 1964

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

Graphite Oxidation Experiment. — The third experiment in which we determined the rates of reaction between reactor-grade (TSF) graphite specimens and trace impurities in a helium coolant was terminated after 985 hr of operation. In this test the CO and CO_2 impurities in the circulating helium were controlled at 125 and 1000 ppm by volume respectively. Based on the graphite geometric surface area, the reaction rate ranged from 2.8×10^{-3} mg cm^{-2} hr^{-1}, for the graphite sample maintained at an average surface temperature of 1590°F, to 4×10^{-2} mg cm^{-2} hr^{-1}, for samples maintained at a surface temperature of 1800°F. The test data indicate an activation energy of 70 kcal/mole.

Development of Graphite Rupture Criteria. — Four types of EGCR graphite specimens, designated small tensile, small compressive, large bend, and small bend, were tested, and the data were analyzed. Stress-strain diagrams were prepared for each of the individual specimens, and composite curves were prepared for each type of specimen. We evaluated the data statistically to obtain the means and standard deviations of the modulus of elasticity both in tension and in compression, the fracture stress, the fracture strain, and the total strain energy at fracture. Confidence intervals at 90, 95, and 99% were computed.

The strength data show that on a statistical basis the modulus of elasticity in tension is less than that in compression. The mean tensile fracture stress and the mean fracture strain are about the same for the two sizes of bend specimens tested, but the mean values for the tensile specimens are much lower. Standard deviations range from 13 to 20% of the mean values. The largest spreads in the data were for the small tensile specimens. Additional studies must be conducted to identify the factors that caused the observed differences.

Viscoelastic Analysis of EGCR Graphite Fuel Sleeve. — Irradiation-induced creep data generated in the ORR were used to evaluate the behavior to be expected of the EGCR graphite fuel sleeves. We based the

analysis on an assumption of generalized plane strain for an average fast-neutron exposure equivalent to a fuel burnup of 10,000 Mwd/metric ton.

The maximum tensile stress obtained from the viscoelastic analysis was 213 psi for the assumed exposure, and it approached asymptotically a limit of 256 psi. The corresponding elastic calculation yielded a tensile stress of 771 psi that increased linearly with exposure.

Despite the large reduction in stresses, the strain distributions were unchanged by creep. The maximum strain, which was the same for both analyses, was of the order of 0.04%.

Design Criteria for Graphite Components. — We conducted further studies on the relative importance of the stress-strain relationship in predicting the behavior of graphite bodies. The current studies were made with the stress-strain diagram for type ATJ graphite at 5000°F, which exhibits a large strain to failure (12.7%), most of which is in the plastic range. Two linear elastoplastic approximations were made to the stress-strain diagram: an elastic perfectly plastic case and an elastic strain-hardening case.

The stress and strain distributions were calculated for thick-walled cylinders loaded with an internal pressure. The results of the study showed that the ultimate loads predicted by the two approximations were in good agreement; however, the distributions of the strains and stresses within the body may be quite different at pressures less than the maximum. We concluded that the degree of correlation was a strong function of the accuracy with which the stress-strain diagram was represented.

GCR-ORR Loop No. 1 Operation. — We continued the experiment for determining the behavior of radioactive fission products in a circulating helium stream following release of the fission products, from a small hole in a stainless-steel-clad UO_2 fuel element, into the helium stream. The loop operating temperature was increased gradually until the fuel element was operating at cladding and central temperatures, respectively, of 1400 and 2400°F. During initial operation, no release of fission gases into the loop was detected. After approximately three weeks of operation, the Xe^{135} concentration increased to approximately 10 dis min^{-1} cm^{-3} (STP). The Xe^{133} isotope was just detectable. The unexpectedly low release rate compared with that in the previous experiment may indicate either that the hole is partially plugged or that its smaller size and location have effectively restricted the release of noble gases. The arrangement of the vent hole and the MgO insulator spacers used at the top of this element should more effectively restrict the convection of fission gases within the fuel can. We will continue the experiment for at least one more ORR cycle.

LITR Capsule Irradiations. — In a new capsule for the irradiation of pyrolytic-carbon-coated particles, the particles will be in graphite containers instead of the tantalum cans used previously. The new design more closely approximates actual service conditions for the particles.

ORR Poolside Capsule Irradiations. — We continued the irradiation of one EGCR-type stainless-steel-clad capsule and two capsules containing fueled graphite spheres 6 cm in diameter. Special tests for determining

the fission-gas release as a function of power density and temperature were conducted on one capsule (05B-7) containing pyrolytic-carbon-coated particle fuel in spherical graphite matrix elements following ~1.2 at. % heavy-metal burnup. The ratio of release rates was slightly greater than the corresponding change in power density. Increasing the fuel surface temperature from 1135 to 1600°F resulted in an increase by a factor of 4 in the fission-gas release rate. Near the conclusion of these experiments, argon from the blanket gas region was observed in the sweep gas samples, suggesting that a leak had developed in the primary containment vessel. We therefore removed the capsule from the high-flux region and will withdraw it during the April ORR shutdown.

Eight-Ball Experiment. — The fourth eight-ball experimental assembly containing 1/2-in.-diam spheres fueled with pyrolytic-carbon-coated UC_2 fuel particles continued to operate. The sphere surface temperature has increased slightly, to ~1750°F.

Thermal Shock Effects on Fueled Graphite Spheres. — Fueled graphite spheres (1-1/2 in. in diameter) were dropped into a graphite resistance furnace operating at 2000°C, held at temperature for 15 min, and then dropped into a water-cooled graphite crucible to determine whether thermal effects were responsible for dimensional decreases noted after irradiation. After treatment, most spheres showed increases in diameter, indicating that the dimensional decreases of irradiated spheres were not associated with thermal shock.

Total Replication of Fueled Spheres. — A replication technique (Proc. Conf. Hot Lab. Equipment, 9th, Chicago, 1961, pp. 166–72) was used successfully on irradiated fueled spheres, both 1-1/2 in. and 6 cm in diameter. The process consisted in making a rubber mold of the sphere and filling the mold with epoxy resin. With the use of this technique, we can compare the surface of a sphere before and after a prescribed treatment without loss in detail. Because of the reduced radioactivity of the replica, the replicated surface of the irradiated sphere can be examined outside the hot cell.

APPLIED AND BASIC REACTOR PHYSICS

Basic Reactor Shielding: A Method for Analyzing Neutron and Gamma-Ray Spectra. — Many instruments, such as scintillation counters, commonly used to detect nuclear radiations produce such a large amount of data that it is not feasible to examine the results of a given experiment directly. Instead, the data are customarily plotted automatically or displayed on a television-type screen, with each different energy of the incident radiation producing a characteristic response curve. If there are several different energies present, however, the curves become scrambled or folded together, and only the mixture is seen. In order to determine the intensities of the different components, the scrambled curve must be unscrambled or unfolded. This is a common problem, and many methods and computer programs exist for its solution. All these methods, however, have the serious limitation that if there are too many components scrambled together, very large errors occur in the unfolding.

A code has recently been developed which largely overcomes the error problems associated with the other methods and makes possible the unfolding of the energy spectra of neutrons and gamma rays from reactors and accelerators routinely, even though the spectra contain an unlimited number of components. The new code is unique in that information usually ignored in other codes is used advantageously to obtain much smaller errors. It utilizes the relatively new technique of quadratic programming (a mathematical technique developed to aid in optimum economic scheduling) to make the best use of certain known properties of the radiation, such as continuity, decrease in pulse size with radiation energy, nonnegativity of response, etc.

The new code consists of a main program, known as SLOP, and four special programs which must be written for each different application. The first of these describes the shapes of the curves resulting from single-energy radiations, the second defines the shapes of the unfolded curves, the third reads the experimental data into the computer, and the fourth specifies restrictions to be placed upon the radiation.

Basic Reactor Shielding: Low-Energy Neutron Spectra in LiH. — Previous investigations have shown that in certain hydrogenous materials the low-energy neutron spectrum attains an asymptotic shape beyond some minimum distance from a fission source, which would indicate that the spectrum beyond that distance depends only on the scattering properties of the medium and not on the source term or the geometry. If this is found to be true for materials of interest as radiation shields, a measurement of the asymptotic spectrum for a particular medium should provide a sensitive check on shielding calculations for that medium, even though the calculations do not accurately reproduce the experimental conditions. Since lithium hydride is a frequently proposed neutron shield, the neutron spectrum through various thicknesses of the material was measured, and the results were compared with Monte Carlo, moments method, and NIOBE code calculations, as well as with some infinite-medium calculations performed at General Atomic.

The measurements were performed at the General Atomic linear accelerator, where a near-fission neutron source was obtained by placing a lead target in the electron beam. Three types of spectra were measured: spectra of the leakage flux of low-energy neutrons, spectra of the forward-directed angular flux at an internal point, and spectra of the scalar flux at an internal point. Time-of-flight spectrometry techniques were used to determine the energies of the moderated neutrons. The energy range covered was from about 0.01 to 1000 ev.

It is apparent from the measurements and the calculations, which were in agreement for energies above 0.4 ev, that the spectral details of neutrons moderated in LiH are practically invariant with position after a minimum penetration depth that lies between 2.5 and 5 cm. It is also shown that at any point in a shield where the gradient term vanishes, the spectrum of angular flux and the spectrum of scalar flux become identical. This spectrum should then be identical to the infinite-medium constant-source density spectrum. The spectrum measured in an almost-gradient-free region of LiH compared favorably at very low energies with an infinite-medium calculation that employed first a scattering kernel for hydrogen as

bound in water (σ_a = 72 barns) and then a scattering kernel for LiH. This agreement suggests that either hydrogen scattering kernel is adequate and that both the scattering by hydrogen and the ratio Σ_s/Σ_a, which depends on chemical binding, are most important in determining the spectral shape.

Radiation Shielding Information Center. — The Radiation Shielding Information Center (RSIC) has developed an information retrieval system for storing bibliographic and indexing information on magnetic tapes and for producing literature searches and shielding bibliographies. In preparing information for the system, each literature specimen is identified by an accession number and indexed under as many subject categories as are applicable. A total of 118 subject categories are currently in use, each a subcategory of nine major topics (for example, "Shield Design Techniques" and "Shielding Materials").

For each subject category number assigned to a literature specimen, an emphasis role indicator number is also assigned which gives a semi-quantitative evaluation of the emphasis or relevance placed upon the category by the specimen. Where applicable, another role indicator is assigned which indicates whether the specimen describes or merely uses a given technique, theory, or device.

The information retrieval system has been used to produce a shielding bibliography differing from the usual bibliography in that it is in a readily searchable form. It contains two parts: a list of documents under each of the 118 subject categories and an author index. Each subject category lists all the documents dealing with that particular subject, the information being repeated under other categories when appropriate. The author index gives all relevant document accession numbers and subject category numbers for each author.

The Center has also organized a computer code section to accumulate experience with various computer codes; to examine the codes as to operability, ease of dissemination, and production of the desired results; and to intercompare codes. Code packages are prepared for distribution.

REACTOR FUELS AND MATERIALS

Nondestructive Test Development. — Radiography has proved to be a valuable inspection method for nuclear materials. However, it is normally considered to be impractical or impossible in the presence of a high radiation background. Nevertheless, we are achieving some promising results in initial studies of the feasibility of performing useful radiography under those conditions. An aluminum specimen containing several penetrameters was radiographed numerous times with a technique designed to produce an average acceptable quality image. The exposed film was then subjected to varying amounts of background fogging radiation at energy levels of 50, 100, and 300 kv (constant potential) and 1.1 Mev. The films were subsequently chemically processed according to standard procedures to allow viewing and evaluation. The penetrameters were evident on film that had received fogging irradiation up to values of 1 r at 100 kv (constant potential), 2 r at 50 or 300 kv (constant potential), and 32 r at 1.1 Mev. It must be remembered that these are accumulated doses. Since the primary

exposures can be made in less than a minute, substantial dose rates can be tolerated. Further studies are being made to increase the tolerance to background radiation.

Mechanical Properties. – The strength and ductility of metals are often adversely affected by reactor radiations. Thus, reactor components that are adequate on the basis of conventionally measured properties may fail in service under irradiation. Since the defects introduced into materials by irradiation are affected by the temperature of irradiation, we have investigated the influence of irradiation temperature on the post-irradiation tensile properties of type 347 stainless steel. The alloy, ASTM grain size No. 8, received 5% cold work prior to being irradiated to an integrated dose of approximately 7×10^{20} neutrons/cm^2 ($E > 1$ Mev). To ascertain the effect of irradiation temperature, 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 are being determined for each test. 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 the alloy 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 least value found is 3.0% for 5%-cold-worked type 347 stainless steel irradiated at 700°F and tested at 1400°F.

Solid Reaction Studies. – The processes that limit the useful life of structural materials, such as creep and corrosion, often depend on rates of diffusion. We are continuing to study several factors governing diffusion in solid materials.

We have continued experiments on the diffusion of impurities in a metal under a temperature gradient, completing our investigation of the redistribution of Sb^{124} in silver. In four silver specimens containing Sb^{124} and annealed in a gradient, the antimony concentration increased at the hot side and decreased at the cold side. The steady-state concentration was described by a straight line in a log c vs 1/T plot, and the heat of transport was -29 ± 3 kcal/mole for the thermal diffusion of antimony in silver. The magnitude is remarkably high. In a similar experiment with a silver specimen initially containing uniformly distributed Ru^{103}, the concentration profile was quite different from what one would expect by thermal diffusion. Ruthenium had apparently diffused from both ends of the specimen into the tungsten barriers.

We are studying the effect of alpha irradiation on the anomalous near-surface diffusion of Pb^{212} in silver. In the temperature range 380 to 470°C without the radiation source, we find that this diffusion can

be described by an Arrhenius-type equation with an activation energy of 61,700 cal/mole. Bombardment by 10^{11} alpha particles cm^{-2} sec^{-1} (from a 10-curie Po^{210} source) at any temperature in the range 360 to 440°C increases the diffusion coefficient by 7×10^{-18} cm^2/sec within experimental error. This result is consistent with the existing theory of radiation-enhanced diffusion.

Materials Compatibility Studies. — The resistance of refractory metals to corrosion by alkali metals can be seriously impaired by the presence of oxygen in the liquid metal. Potassium is being considered as the working fluid in several proposed space reactors. In order to improve the lifetime and reliability of such systems, we are studying methods to remove and to analyze oxygen in potassium. Previously, we had demonstrated that oxygen in potassium-refractory metal systems can seriously affect the corrosion resistance of the solid metal. The oxygen content in refractory metals can be analyzed accurately by a vacuum fusion technique, but no proven method exists for measuring the oxygen concentration in potassium.

Recent experiments indicate that oxygen in potassium can be determined by a fast-neutron activation technique. Unlike most other methods, it is specific for oxygen. Bombardment with 14-Mev neutrons from the ORNL 150-kv Cockcroft-Walton neutron generator converts the O^{16} in potassium to N^{16} by an (n,p) reaction. The 7.4-sec half-life of N^{16} permits only a few seconds for transfer of the irradiated sample to the counter. The sample is transported rapidly in its sealed container through polyethylene tubing under the pull of vacuum. Molybdenum was selected for the sample capsule because it inherently has a low oxygen concentration, is compatible with potassium, and has a negligible induced activity after irradiation. An initial capsule, with a 0.030-in. wall and an O-ring seal, was found by analysis to contain 98 ppm O when empty and 328 ppm O when filled with purified potassium. In duplicate tests, known weights of K_2O, equivalent to 520 and 330 ppm O, were added to the potassium, and the system was thermally equilibrated at 200°C for 1 hr. Subsequent activation analysis of the oxygen in the potassium yielded 95 and 98% recovery respectively. Only one determination could be made on each encapsulated sample, since the sudden discharge of the container into the counting cubicle loosened the cap and permitted air contamination of the potassium. In similar experiments with a second type of molybdenum capsule, having a 0.020-in.-thick wall and sealed by a press-fit polyethylene cap, a recovery of 102% was found. However, this capsule also leaked after a single determination.

Fuel Element Development. — To improve the economy of the nuclear fuel cycle, simpler and more direct methods of fuel element fabrication are needed. We are investigating the pyrolytic decomposition of gases as a method of directly depositing fuel element materials. In previous work (ORNL-3517, p. 7) we have prepared tungsten tubing of unusual quality and refractory carbides with unique properties. We have now prepared uranium dioxide by the reaction of UF_6 with steam and hydrogen.

Uranium dioxide can be deposited as either powder or massive material. At 1300°C, lower steam concentrations favor the massive form. Both products have been identified by x-ray and chemical analyses as UO_2, and examination of the powder by electron microscopy gave a mixture of particle sizes ranging from 60 to 200 A. The massive UO_2 is highly crystalline, and the crystallite size depends on the deposition temperature. Too low a hydrogen-steam ratio allows the formation of UO_2F_2 and UF_4. In continuing work we are emphasizing the deposition of massive material, with the prime objectives being to determine the effects of steam and hydrogen proportions on the characteristics of the deposit and to achieve a dimensionally uniform deposit.

REACTOR COMPONENTS

Reactor Controls Development. — A wide-range counting channel [Trans. Am. Nucl. Soc. 3, 454 (1960)] has been assembled and tested by use of the production prototypes of the instrument modules recently developed (ORNL-3436, p. 7). A fission-chamber drive unit intended for the HFIR was used to control the position of a 3/4-in. fission chamber and preamplifier assembly [Trans. Am. Nucl. Soc. 5, 185 (1962)] at the BSR. After initial adjustment, the instrument indicated reactor power and period over the eight-decade range of the BSR. The transient response to a simulated 5-sec period was slightly underdamped, with 10% overshoot in indicated period. The effective time constant at 10 counts/sec is 6 sec. The positioning servo has an effective time constant of 300 msec when operating at its normal setting of 10,000 counts/sec. The pulse pickup noise is negligible even when the reactor control or other equipment is operated. Further testing will be directed toward optimizing fluctuations and time constants, evaluating other drive mechanisms, and gaining experience with different reactor configurations.

The two 3/4-in. fission chamber and preamplifier assemblies now in service at the ORR have operated continuously since their installation, one on August 28, 1962, and the other on August 29, 1963. The ultimate limitation on the service life of this instrument has not yet been established.

Three PCP-II ionization chambers (ORNL-3191, p. 70) are now installed in reactors. One, in the LITR since August 1963, has a gamma-compensated neutron-sensitive section operating the period safety channel, and an uncompensated section for future use in a power safety channel. A similar chamber is under test in the ORR. Also under test at the ORR is a gamma-sensitive ionization chamber for safety use [Trans. Am. Nucl. Soc. 6, 74 (1963)].

POWER REACTOR FUEL PROCESSING

Dissolution Studies with UO_2 Fuel Pellets. — In order to calculate, according to the dissolution rate, the time required to dissolve UO_2 fuel pellets, the variation in the surface area during dissolution must be

known. Therefore, a series of experiments was conducted in which batches of 17 unirradiated UO_2 pellets were dissolved in 4.5 to 10 N HNO_3. These tests showed a marked variation in the time for the dissolution with the different acid strengths. Regardless of acidity, however, the dissolution curves had similar shapes. An interesting observation was that pellets dissolved into thin wires when concentrated acid was used, but dissolved into dumbbells and left flat end pieces undissolved in low acidities.

Dissolution Studies with ThO_2-UO_2 Fuel Samples. — In order to compare the dissolution rates of both unirradiated and irradiated ThO_2-UO_2 (containing 4% UO_2) prepared by the sol-gel and arc-fusion processes, samples were reduced to powder and dissolved under identical conditions. The size distribution of the particles was: > 20 mesh, about 60%; 20 to 60 mesh, 20%; and < 60 mesh, 20%. All samples were digested for 7 hr in boiling 13 M HNO_3—0.04 M HF—0.04 M $Al(NO_3)_3$. The volume of dissolvent was sufficient in each test to produce a 1 M solution of thorium if the sample had totally dissolved. With unirradiated samples, 67% of the sol-gel mixed oxide dissolved in 7 hr, and 85% of the arc-fused material dissolved in the same time. Ninety-one percent of a sol-gel-derived sample irradiated to 35,000 Mwd/ton dissolved under similar conditions in a test conducted at the General Electric Company Vallecitos hot-cell facilities. These tests indicate that sol-gel-derived ThO_2-UO_2 dissolves slower than arc-fused fuel. However, for the oxide produced by the sol-gel process, dissolution is faster after irradiation. These tests are being continued.

Leaching Studies with UO_2-ZrO_2 Pressurized Water Reactor Seed. — Unirradiated samples of PWR fuel wafers (35% UO_2—65% ZrO_2) were ground to 200 mesh. A sample of this powder was then leached for 7 hr with boiling 13 M HNO_3. Only 11.2% of the uranium in the sample dissolved. This confirms prior results that nitric acid leaching, under virtually all conditions, will not yield acceptable uranium recoveries.

Stoichiometry of the Reaction of Uranium Monocarbide with Steam. — Previous studies showed that when uranium monocarbide is contacted with steam at high temperature (650 to 800°C) the reaction proceeds stepwise. The relatively fast reaction

$$UC + 2H_2O \longrightarrow UO_2 + C + 2H_2$$

occurs first, and then the free carbon is converted to oxides at slower rates according to the reactions

$$C + H_2O \longrightarrow CO + H_2 \ ,$$

$$C + 2H_2O \longrightarrow CO_2 + 2H_2 \ .$$

Several runs have been made to establish the stoichiometry of the overall reaction. The best results were obtained with a specimen of uranium monocarbide obtained from Atomics International which contained 4.51 wt % carbon. From analysis of the products of the reaction of this material with steam at 750°C, the overall reaction

$$UC + 0.06U + 3.71H_2O \longrightarrow 1.06UO_2 + 3.68H_2 + 0.66CO_2 + 0.27CO$$

was obtained. The material balance is not complete, since 0.07C and $0.03H_2$ remain unaccounted for. However, these slight disparities may fall within the limits of experimental and analytical error.

Pyrohydrolysis of Irradiated UC-PuC. – Two pyrohydrolysis experiments were made with irradiated samples of 80% UC–20% PuC to determine the radioactive components in the off-gas by filtration of the gas immediately after condensing the excess steam. One-gram samples irradiated to 1000 and 20,000 Mwd/metric ton were contacted with steam at 750°C for 2 hr in order to produce a stream of particles, some of which were trapped during condensation of the steam; the remainder were caught on a paper filter. The radioactivity trapped on the filter was due to Cs^{137}, leached from the specimen during the reaction with steam. Dry gas samples taken downstream from the filter were free of fission product contamination. These and previous results show that during the pyrohydrolysis of irradiated carbide fuels, about 0.5% of the cesium is carried by the steam off-gas stream. However, the cesium can be effectively trapped by steam condensation plus filtration to yield a nonradioactive off-gas.

Protactinium Adsorption. – Inorganic ion exchangers are being investigated as a possible means of separating protactinium from aqueous Th-U^{233}-Pa fuel solutions. In addition to the work with unfired Vycor, preliminary experiments show that 70- to 80-mesh Bio-Rad ZP-1, a zirconium phosphate exchanger, will also adsorb Pa^{233} from nitric acid solutions. The protactinium distribution coefficient from solutions containing thorium (4 g/liter) and Pa^{233} (1.5 to 3×10^5 counts min^{-1} ml^{-1}) ranged from 100 (when the concentration of nitric acid was 0.5 M) to 29,000 (for 12 M HNO_3). About 45% of the protactinium was in an unadsorbable form in 0.5 M HNO_3, but less than 0.4% was unadsorbable from solutions containing more than 3 M HNO_3.

Corrosion. – Type 304L stainless steel and "A" nickel were evaluated as construction materials for equipment in which carbide fuels will be pyrohydrolyzed at high temperatures. Welded specimens of both materials exposed to steam at 825°C and atmospheric pressure corroded at rates which averaged 0.2 and 0.7 mil/month, respectively, for 1008-hr exposures. "A" nickel tested in steam containing 1 vol % O_2 under these same conditions showed a weight gain equivalent to a corrosion rate of 1.8 mils/month for a 168-hr exposure.

One reagent being considered for chemically "burning" graphite and carbide fuels is 10% HNO_3–90% H_2SO_4. At 150°C, type 304L stainless steel showed a corrosion rate of 5.6 mils/month for a 168-hr exposure in the oxidizing mixture. Rates for type 309SCb and Carpenter 20 stainless steel (48-hr exposures) were 8.8 and 17.5 mils/month respectively. Since none of these rates are acceptably low, other alloys will be tested.

Welded specimens of type 309SCb stainless steel, Haynes alloy No. 25, Hastelloy F, and Corronel 230 tested in boiling 7 M HNO_3–0.05 M HF, a proposed dissolvent for high-fired UO_2-PuO_2 fuels, corroded at maximum rates of 14.8 (72 hr), 6.7 (192 hr), 31.7 (72 hr), and 1.6 (192 hr) mils/month respectively. In a solution containing 0.24 M UO_2 and 0.06 M rare-earth oxides (to simulate PuO_2), rates for 144-hr exposures were 0.4, 0.8, 0.6, and 0.2 mil/month respectively. These tests show the superior resistance of Corronel 230.

Fluoride Volatility Processing: Aluminum-Alloy Flowsheet Development. — A 67-21-12 to 52-16-32 mole % $KF-ZrF_4-AlF_3$ system is being studied for use in dissolving Al-U alloy fuels with HF. Supplemental to the studies of the corrosivity of the $KF-ZrF_4-AlF_3$ melt made at Battelle Memorial Institute (ORNL-3543, p. 7), the corrosion of Hastelloy N (INOR-8) was investigated during hydrofluorination in molten $KF-ZrF_4-AlF_3$ and in $LiF-NaF-ZrF_4$. These studies made possible the direct comparison of corrosion in processes for dissolving Al-U and Zr-U alloys. Gravimetry indicated that $KF-ZrF_4-AlF_3$ is less corrosive to Hastelloy N than $LiF-NaF-ZrF_4$, but the results remain to be confirmed by metallographic examination of the specimens.

The tests were made at 600°C, with each specimen exposed in both the salt and gas phases without fuel dissolution. If the computations are arbitrarily based only on the area of each specimen actually submerged in salt, corrosion rates of 0.006 and 0.013 mil/hr are found at 67-21-12 and 51.8-16.2-32 mole % $KF-ZrF_4-AlF_3$, respectively, for the Al-U alloy process case, and 0.022 and 0.018 mil/hr at 37.5-37.5-25 and 27.5-27.5-45 mole % $LiF-NaF-ZrF_4$, respectively, for the Zr-U alloy process. The compositions used represent the initial and final conditions of the two processes. The calculated corrosion rates should be considered only as relative and not as absolute values, inasmuch as the specimens were exposed to both the gas and salt phases.

Fluoride Volatility Processing: Flowsheet Development for Fuel Containing ThO_2. — Adaptation of the fused-salt volatility process to reactor fuels containing ThO_2 would require use of a salt system in which ThO_2 will dissolve rapidly by hydrofluorination and in which the solubility of ThF_4 is high. Dissolution rates in the $LiF-NaF-ThF_4$ mixture, recommended as a system with a high ThF_4 capacity, were found to be unsatisfactory. Hydrofluorination attack rates on ThO_2 pellets at 600°C were 0.7 and 0.9 mil/hr in 44.5-38.5-17 and 40-35-25 mole % $LiF-NaF-ThF_4$ respectively. In a single test with 37.5-37.5-25 mole % $LiF-NaF-ZrF_4$, a rate of 4.6 mils/hr was measured at 600°C, confirming previous work which indicated that ZrF_4 is helpful (ORNL-3153, p. 35). Consideration is therefore being given to use of the quaternary system $LiF-NaF-ZrF_4-ThF_4$ for a ThO_2 flowsheet.

Fluoride Volatility Processing: Stainless Steel Flowsheet Development. — The results of preliminary studies indicate that molten mixtures of KF and ZrF_4 may have sufficient capacity for dissolving FeF_2 to warrant consideration of their use in the application of the volatility process to fuels dispersed in a stainless steel matrix. As an example, a melt containing 40 mole % ZrF_4 and 60 mole % KF dissolved sufficient FeF_2 at 575°C to give a final concentration of 21.9 mole % FeF_2.

Fluoride Volatility Processing: Drop Fluorination Test. — A spray or gas-phase-continuous fluorinator is being considered as a means of eliminating fluorinator corrosion, for achieving rapid volatilization of plutonium hexafluoride, or possibly for use in developing a continuous fluorination step (ORNL-3543, p. 9). A new falling-drop fluorination tower 3 in. in diameter has been constructed and has operated with 47.5-47.5-5 mole % $NaF-ZrF_4-UF_4$, with results significantly better than those obtained with the first tower, which was 1 in. in diameter. In designing

the new equipment, some cold spots at flanges were eliminated to obtain a more uniform temperature. The larger diameter resulted in greater salt recoveries.

An experiment at 580°C produced droplets with a residual uranium content of less than 1 ppm (better than a 99.9% uranium recovery) at droplet diameter ranges of 63 to 88, 88 to 105, and 105 to 125 μ. Droplets in the diameter range 125 to 149 μ had a residual uranium concentration of only 3 ppm. The improved results in the new equipment are attributed mainly to the absence of cold spots along the column.

Fluoride Volatility Processing: Tellurium Removal from Process-Gas Effluent Stream. — The use of hot nickel beds is being considered as a means of absorbing Te^{127} and Te^{129} (as tellurium hexafluoride) from the gas effluent of the Volatility Pilot Plant, thus preventing its release to the stack system. Supplementing previous work (ORNL-3568, p. 8), further laboratory tests emphasizing material balances were made in order to study the effectiveness of hot nickel beds in removing tellurium hexafluoride from helium. The results again confirmed that a nickel bed at 400 to 500°C is highly effective in sorbing tellurium hexafluoride, thereby preventing tellurium from escaping with the helium carrier gas. The tellurium escaping through the hot nickel test beds was trapped for analysis in three consecutive bubbler traps containing aqueous potassium hydroxide and held at 80°C. Although the overall material balances for runs at 400, 450, and 480°C were only 65, 67, and 58%, a negligible amount of tellurium was found in the potassium hydroxide traps. Analyses made of different sections of the nickel beds indicated that 450° was better than 400°, since more sorption occurred at the inlet end.

Fluoride Volatility Processing: Fluoride-Catalyzed Destructive Oxidation of Stainless Steel. — Previous tests with APPR fuel elements established that the presence of fluorides permitted the destructive oxidation of stainless steel at temperatures of 600 to 700°C (ORNL-2271, p. 71). To explore the feasibility of such a process, a quantitative study was conducted in which HF-O_2 and HF-air mixtures were contacted with stainless steel (principally type 304L). The results confirmed the previous work by showing that high oxidation rates for stainless steel can be obtained at temperatures as low as 600°C. Conclusions concerning oxidation rates are still uncertain because of discrepancies between results from two lots of type 304L stainless steel. With one lot of type 304L specimens (cylinders 1/4 in. in diameter and 1 in. long), oxidation rates of 0.9, 15.2, 54.8, 67.5, 53.7, and 35.9 mils/hr were obtained at 10, 20, 30, 40, 50, and 60 vol % HF in oxygen at 650°C at a total flow rate of 200 ml/min (STP). At a flow rate of 100 ml/min, the oxidation rates were lower, but the utilization of oxygen was higher, as was expected. The first lot of specimens also yielded a high temperature coefficient. With a second lot of specimens, the maximum oxidative attack also occurred at about 40% HF in oxygen but at only 9.2 ml/hr at 575°C. The second lot had a much lower temperature coefficient, and the metal pitted severely, in contrast with the smooth surface noted for the first lot.

The reaction products from the above tests were predominantly Fe_2O_3, with no more than approximately a 1% fluoride content, consistent with a thermodynamic evaluation. Since little of the HF is used, a process based on this reaction would probably require the recycling of the HF.

Since nickel 200 might be considered as a constructional material for use with the stainless steel oxidation process, its resistance to $HF-O_2$ was studied. In 1 vol % $HF-O_2$ at 600°C, weight gains were somewhat erratic but increased with exposure time, at least up to 500 hr. Corrosion rates corresponding to average weight gains increased from 0.1 mil/month at 24 hr to 0.4 mil/month at 504 to 1008 hr.

Fluoride Volatility Processing: Vapor Equilibrium Studies. – One method of separating volatile fluorides from uranium hexafluoride in a fluoride volatility process is by distillation. In order to develop and design distillation equipment for separation, detailed data on the vapor-liquid equilibrium are necessary. Accordingly, an apparatus for determining the vapor-liquid equilibria of volatile fluorides has been built. Its ranges of operating conditions are 50 to 250°C and 0 to 8 atm of pressure. Provisions are made for obtaining both vapor and liquid samples and for determining the total pressure of the system. The apparatus is being used to establish the 150°C isotherm for the NbF_5-UF_6 system. The system has a marked positive deviation from Raoult's law in the lower range of UF_6 concentration, as shown below:

Mole Fraction of UF_6 in Liquid	Total Pressure (atm)	
	Experimental	Predicted
0.027	1.70	0.40
0.040	2.18	0.55
0.053	2.73	0.61
0.075	3.43	0.97
0.105	3.95	1.34

Fluoride Volatility Pilot Plant. – After completion of the series of runs in which short-cooled Zr-U alloy fuel elements were processed, the Volatility Pilot Plant was shut down for a cleanout and inspection prior to the start of Al-U alloy fuel processing. Decontamination to the extent desired was completed. The background at the bottom of cell 1 was reduced from 2500 mr/hr to 150 mr/hr.

Dissolver-wall thicknesses were measured from the penthouse by remote pulse-echo techniques. Since the last set of measurements, there had been nine complete dissolutions and two HF sparge tests. Comparison with the previous measurements gives a more encouraging picture of corrosion than the one appearing from earlier measurements. Data are summarized below for the first 29 runs and for the 40 runs which include last year's operations. The data are in terms of thickness loss in mils for each of four sections of the dissolver, and they show the maximum measured loss and the average of all the thickness-loss measurements in that section: In the enlarged vapor section, the maximums were 18 and

25 mils, and the averages were 10.6 and 10.1 mils for 29 and 40 runs respectively. In the conical section (normal interface), the maximums were 16 and 25 mils, and the averages were 12.4 and 15.3 mils for 29 and 40 runs. In the lower section, maximums were 16 and 21 mils, and averages were 9.84 and 10.25 mils for 29 and 40 runs. In the bottom 18 in., which was a new section and thus exposed to only the last 11 runs, the maximum loss was 27 mils, and the average loss was 8.7 mils.

THORIUM UTILIZATION PROGRAM

Uranium-233 Purification and Sol-Gel Oxide Preparation. — During February, 6.5 kg of U^{233} was purified by a solvent extraction process with 2.5% di(sec-butyl)phenyl phosphonate in diethylbenzene. The U^{233} feed solution consisted of 4200 g obtained by dissolution of Jezebel fuel section P-2 and 2300 g of material recycled from previous runs. The purified U^{233} met all sol-gel requirements. This run completes the U^{233} requirements for the Kilorod Program.

The replacement steam superheater for the thorium nitrate denitrator was installed, and operations were resumed to meet thoria requirements for process development and other requests. Sufficient thoria is available to complete the Kilorod Program.

During February, about 60 kg of mixed oxide was prepared for rod fabrication. A total of 960 kg has been prepared in the program. Oxide preparation is thus about 96% complete.

The radioactivity released by the blending, drying, and calcining steps was measured throughout each operation. Preliminary results indicate that significant quantities of long-lived alpha activity as well as short-lived radon activity are released in each operation. The long-lived activity from the dryer was identified as Th^{228} (5%) and U^{233} (90%), with the remainder being short-lived U^{232} daughter activities still present at the time the analysis was made.

The previously reported calciner control troubles were corrected. The air-driven variable transformer was found to be defective and was replaced. The control circuit was thoroughly serviced.

Thorium-Uranium Fuel Cycle Development Facility. — The TUFCDF is to be used in the development of processes for remotely recovering fissile and fertile materials and for fabricating Th-U^{233} fuel assemblies. Title I design of the facility was completed in January 1964, and project completion by early 1967 is scheduled.

Approval to start title II design was received on March 5, 1964. Work related to building design has been aimed at reductions in cost of building components or improvement of operational capability. The vertical-tube furnace and related components for calcination of gel to oxide are almost completely designed. The dryer design study has established the need for additional investigations of this operation, and these are now under way. Crushing and grinding experiments indicate that a disk pulverizer may be acceptable as a replacement for the roll and jaw crushers and that a small, continuously operated ball mill can produce large quantities of oxide

fines. Conceptual assembly drawings were made of a welding machine and a tube-transfer device. Studies and design effort continued on the various other items of fabrication equipment.

Rod Fabrication. — Operation of the rod-fabrication facility to meet the commitment to Brookhaven National Laboratory is continuing. The required 900 rods 42 in. long are now fabricated, and 133 of the required 200 rods 19 in. long were made during February. Part of the month was spent on facility cleanup for criticality control and on modifications to the system to permit production of the 19-in.-long rods. On the basis of the days spent in actual rod production, the average production rate was 18.5 rods/day.

The equipment for preparing the ThO_2-U_3O_8 pellets to be used in fabricating the pellet rods for use by BNL in calibration of their zero-power criticality tests was installed in the interim alpha laboratory in Building 3019. The proposed operating procedure was approved, and $U^{233}O_8$ was prepared for use in the pellet fabrication.

Sol-Gel Microsphere Preparation. — A tapered 12-liter glass column was used to prepare 850 g of thoria—8 wt % urania (highly enriched) microspheres having diameters in the range 150 to 300 μ (after calcination at 1150°C) for irradiation testing. Because of particle-agglomeration problems, it was necessary to change the surfactant (used to adjust the interfacial tension) from Ethomeen S-15 (tertiary amine) to Span 80 (sorbitan monooleate). The effectiveness of Ethomeen, which is cationic, appears to depend on the free nitrate or urania content of the sol. The system Span 80 and 2-ethylhexanol produced spherical particles of thoria-urania up to 1000 μ in diameter. In earlier tests with thoria sols, pear-shaped particles were produced with Span 80 for sizes greater than 300 to 500 μ. With a glass two-fluid nozzle for forming the droplets, the yield of thoria-urania product in the size range 150 to 300 μ varied from 50 to 80%.

The studies to optimize the procedure for forming microspheres are continuing. A laboratory-scale column for preparing spheres was used to prepare microspheres in experiments in which the effects of adding 5 to 10 vol % of various materials were determined. The most satisfactory additives found to date for forming urania-thoria-carbon spheres in 2-ethylhexanol are the secondary and tertiary alcohols.

Studies are in progress under subcontract at Southwest Research Institute to apply techniques that the Institute has developed to the encapsulation of thoria—3% urania sol droplets. The encapsulated droplets can be gelled and dried to microspheres. Most of the tests were made with methacrylate or cellulose acetate butyrate polymer solutions for forming the encapsulating shell. Good capsules of sol have been formed, but they usually collapse or break when the sol shrinks on drying to form gel. Procedures intended to make the shell shrink as the sol gels have not yet been successful.

In one test, unencapsulated sol was formed to spheres by passing it as droplets through hexane into diethylenetriamine.

Irradiation of Thorium Fuels. — Replicas of irradiated arc-fused and sol-gel oxides are being examined by electron microscopy to establish the nature of the dark spots observed. Additional dissolution studies are being made to further verify the higher rates observed on irradiated fuel, compared with unirradiated fuel.

X-ray diffraction data from Bettis Atomic Power Laboratory examination of pellets irradiated to 50,000 and 95,000 Mwd/metric ton showed a broadening of the high ThO_2—low UO_2 solid-solution peaks. An appreciable decrease in unit cell dimensions as a function of increased burnup was noted.

Vibratory Compaction. — Laboratory and equipment modifications have been completed to permit the filling and vibratory packing of 10-ft-long tubes with powdered fuel. Efforts to fill these thin-walled (0.012-in.) tubes with the aid of the new high-energy vibrator resulted in failure of the tube wall. A relatively high transverse component may be responsible, and some redesign is under way to correct this problem.

Pellet Fabrication. — The time remaining before fabrication of the ThO_2-U_3O_8 pellet rods for use at Brookhaven National Laboratory is being used to continue to look for ways to improve the fabrication techniques. Isostatic pressing is being used to produce pellets of very uniform axial density. Whether or not the BNL pellets are fabricated in this way, the method may be useful to produce very uniformly dense ThO_2-UO_2 pellets for use as reference standards in the gamma scanning of rods filled by vibratory compaction.

Dispersion Hardening of Thorium Alloys. — Hot tensile tests to evaluate hardening by dispersion and alloying were completed. The tests were conducted at 800°C, using thorium strengthened by alloying with zirconium or indium, or using thoria in the form of a dispersoid. Alloys containing 5 wt % indium and dispersions containing 4 vol % thoria were much stronger than the other compositions studied. A spread of 1500 to 2000 psi in tensile strength was found for samples prepared by presumably identical methods. The fact that 4 vol % ThO_2 produced yield strengths about 60% higher than the yield strengths produced by 7 to 13 vol % ThO_2 was unexpected, and the reason is being sought.

Coated-Particle Development. — A coated-particle-development laboratory is being set up to evaluate methods proposed for preparing coated-fuel microspheres by sol-gel techniques.

The design of structural modifications of the building in which the laboratory is to be located has been completed. Layouts for air handling, piping, and electrical installation are being prepared. Procurement specifications for the hood, glove box, and coating furnace will be issued shortly, and the design of the furnace enclosure is about 30% complete. The concept of the coater and, more specifically, the concept of its gas distributor have been reviewed in the light of theoretical calculations, observations of fluidization models, and consultations. Detailed design of this equipment was started.

Microsphere Coater Development. — The analysis of the effect of the many variables on fluidization and on the rates of pyrolytic carbon coating of microspheres has been completed. A nomograph was developed for determining the interrelations of the flow rate of the coating gas, its viscosity, its density, and particle diameter. Scale models of various designs of coating-gas distributors were studied, and a detailed design for a coater is under way.

Equipment Development. — Experimental studies were started to obtain data for drying sols to gels by using radiant heating panels, as proposed for the dryer at the Thorium-Uranium Fuel Cycle Development Facility. The only data on drying previously available were those obtained incidental to the process development for the Kilorod Facility and in its operation. Rate data are being collected for a special radiant element and a 0.42-ft^2 tray in a small oven that employs overhead heating. Preliminary results indicate that drying rates 5 to 10 times those used in the Kilorod Program may be practical.

Preparation of the area in which the 3-in.-diam, vertical-tube calcining furnace will be tested has begun. Unused equipment was removed, the floor was painted, and the drain system was upgraded. Relocation of a vacuum dryer and installation of a slab-tank sol-preparation system should be completed during April. Since the rotary denitrator for the thorium nitrate, which is also located in this area, will not be moved, the area will house a complete sol-gel system for the conversion of thorium nitrate to the dense oxide product.

Extension of Sol-Gel Process to Newer Fuel Types. — The preparation of 850 g of thoria—8% urania (highly enriched) microspheres (150 to 300 μ in diameter) for irradiation studies to be conducted at General Atomic, Battelle Memorial Institute, and the ORNL Metals and Ceramics Division has been completed up to the 4% hydrogen—argon firing step. Measurements of physical properties were made on the gel spheres dried at 100°C and on the air-fired spheres obtained by calcining the gel spheres in air for 4 hr at 1150°C. The gel spheres had a density of 4.53 g/cm^3 as determined by mercury displacement and a density of 7.1 g/cm^3 as determined by helium displacement. Their BET (nitrogen-adsorption) surface area was 56 m^2/g. The air-fired oxide spheres had a mercury displacement density of 9.99 and a helium displacement density of 10.03. Their BET surface area was 0.004 m^2/g. Extremely small spheres (1 to 30 μ) have also been prepared from this material. Those larger than 10 μ looked hollow, but this has not been established.

Work on the preparation of a variety of types of dense mixed oxides by the sol-gel procedure was started. A 20% urania—thoria, a thoria—molybdenum oxide gel, and a dense zirconium oxide have been prepared so far.

Physical and Chemical Properties of Molten Chlorides. — Studies are continuing in the irradiation of mixtures of $AlCl_3$ and $ThCl_4$ in order to produce Pa^{233} and then to try to sublime it and the $AlCl_3$ away from the $ThCl_4$. Results show that protactinium chloride is not volatile from mixtures of 50 mole % $AlCl_3$—50 mole % $ThCl_4$ when heated at 250°C for a number of hours, even though the $AlCl_3$ sublimes readily.

The solubility of $ThCl_4$ in $AlCl_3$ at 250°C was indicated to be slightly above 1.0 mole %.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — Progress was made in all activities concerned with the fabrication and installation of the MSRE.

Machining, cleaning, and final inspection of the reactor vessel were completed, and work was started on installing the graphite moderator assembly in the vessel.

A water flow test was completed on the heat exchanger tube bundle. Binding of the tubes with INOR-8 spacing bars was nearly completed.

All fuel piping, including freeze valves, was completed in the drain tank cell; leak checking of this system was started. The entire coolant system was checked for leaks with helium. Heaters and insulation were installed on the coolant drain tank, and work continued on the installation of other heaters and insulation in this area. The cooling-air duct was completed around the radiator. The radiator door drive mechanism was installed. The radiator system is almost ready for air flow test.

The critical-path schedule showed the MSRE project to be 91% complete on March 1.

Metallurgy. — Studies were continued to evaluate the effect of heat treatment on the properties of Hastelloy N. In previous work we showed that the ductility of this alloy went through a minimum between 1400 and 1800°F and that the low fracture strains were related to the heat treatment.

Annealing studies showed that the precipitate present in a vacuum-melted heat of Hastelloy N (heat 2477) could be dissolved by annealing the alloy at 2400°F for 1 hr. These studies also showed that a fine precipitate formed if the solid-solution alloy was held at temperatures near 1500°F. This reprecipitation influenced the tensile properties only slightly. Cold working the alloy after the solution anneal and before heating to the reprecipitation temperature resulted in a reduction in ductility and an increase in strength. This is probably due to the influence of the additional nucleation sites brought about by cold working.

Chemical Development. — The main oxidizing agent in the MSRE fuel ($LiF-BeF_2-ZrF_4-UF_4$; 65-29.1-5-0.9 mole %) is UF_4, a few percent of which is converted to UF_3 as equilibrium is approached. Measurements of the solubility of UF_3 in the fuel, along with results on the UF_3-UF_4 equilibrium in the presence of H_2 and HF, suggest that UF_3 may be rather more stable in the MSRE fuel than in some of the fuel solvents previously studied. Also, the solubility of UF_3 is apparently considerably lower than that predicted by analogy with rare earths or plutonium in $LiF-BeF_2$ melts. Thus, while the measured solubility provides a margin of tenfold or so at operating temperatures, the possibility that the used fuel freezes as a five-component system, rather than a four-component system, is being studied. So far, no evidence of a different freezing pattern due to the presence of UF_3 has been encountered in the examination of frozen deposits from the Engineering Test Loop.

Estimates of the surface tension of MSRE fuel, based on the shape of a sessile drop (1 cm in diameter) on graphite, give, at MSRE operating temperatures, about 230 dynes/cm, compared with previously accepted estimates of about 200 dynes/cm or slightly lower. The temperature coefficient of surface tension was about 0.1 dyne cm^{-1} $(°C)^{-1}$, as is also characteristic of alkali fluorides. Correlation of the behavior of MSRE melts with that of other fluorides indicated that fluorides change (contact angle of 90°) from wetting to nonwetting on graphite as the surface tension at the

melting point rises through 180 dynes/cm. On platinum or nickel the change occurs at 210 dynes/cm; presumably the change occurs similarly on INOR-8 when the latter is sufficiently clean. Based on comparison with nickel, a contact angle of 130° is predicted for fuel or coolant on INOR-8 at 650°C.

Preparation of MSRE Materials. — The horizontal rotary kiln, installed for use in the densification of the Li^7F feed material, was put into service. A total of 911 kg of material was treated (11 batches); excess water was removed, and the bulk density of the product was increased from an average of 0.5 to about 1.0 g/cm^3. It proved possible to raise the temperature of the treated material only to 500°C, which was lower than that desired on the basis of development work. Since enough material had been treated to provide for initial fluoride production operations, the kiln was shut down for installation of additional heaters.

The fluoride production facility was put into operation with Li^7F and BeF_2 raw materials for the purpose of making the coolant salt. The startup operation proceeded more smoothly than had been anticipated; nine batches of coolant (120 kg each) were prepared in a 20-day period. Following this, the facility was shut down for inspection of the meltdown and treatment tanks, routine maintenance, review of the analytical results, and the production of fuel samples containing enriched uranium for use in the radiation experiment now in preparation (ORNL-MTR-47-6). It seems probable that the rate of coolant and flush salt production can be increased substantially over the initial rate.

Radiation Effects. — Parts from previous in-pile exposures of INOR-8 capsules containing MSRE fuel were examined further. For example, the metallography of coupons from exposure 47-3 was finished. These coupons, which were exposed in contact with graphite at higher temperatures (~800°C) than the previously examined walls, suffered changes that were more readily detectable, such as carburization and corrosion. The extent to which these effects are inherent with molten fuel remains in doubt, because the corrosion was generally worse in the capsules that presumably evolved the most F_2 from frozen salt during shutdowns.

Half of the capsule that received the least exposure (1.3% burnup) in assembly 47-4 was reexamined after more than a year in storage; whereas the fuel crystals had formerly been uniformly darkened by radiation damage, the outer two-thirds was now found to have returned to a normal green color. The mechanism of this apparant bleaching has not been established. The fuel in a companion capsule that underwent 11.3% burnup, and was thus even darker, was unbleached.

The G_{F_2} (molecules of F_2 per 100 ev of absorbed energy, not counting the induction period) in exposure 47-5 was reassessed on the basis of neutron flux determined from the Co^{60} contents of pieces of the capsules. Power densities determined in this manner were approximately twice the design values. Thus, the G_{F_2} values ranged from 0.005 to 0.031 during shutdowns, and further revisions of previously reported values may be necessary when better estimates of the power densities are obtained from isotopic analyses of the uranium in the fuel.

Irradiation Tests. — The sixth capsule irradiation experiment of the ORNL-MTR-47 series in support of the MSRE is in the assembly stage, and the instrument panels and gas-flow systems are being modified at the MTR. This experiment is designed primarily to further the study of CF_4 generation and general compatibility of reactor fuel and materials. The experiment will also provide an additional opportunity to examine the purge gas for the presence of fission products, such as iodine, which would affect the design of the reactor off-gas system. Irradiation at the MTR is scheduled to begin during the last quarter of FY 1964.

The gas-sample station associated with the two capsules equipped for purging has been built, and external instrumentation is being connected for final tests of the system and the sampling technique. The sample station is designed for measuring the generation and radiolytic decomposition rates of CF_4 from fissioning MSRE fuel. The CF_4 is adsorbed in molecular-sieve traps cooled by liquid nitrogen. Samples of the trapped gas are analyzed for CF_4 to obtain generation and decomposition data. A thermal conductivity cell calibrated for CF_4-He mixtures will be used as an alternate means of obtaining data on the rate of radiolytic decomposition of CF_4 within the capsules.

The first attempt to load the capsules with fuel was unsuccessful due to severe crumbling of the precast fuel pieces — a condition not experienced with trial castings. Another set of fuel castings is being made, and equipment is being prepared for an alternate technique of filling the capsules with powder.

Analysis of Radioactive MSRE Fuels. — The equipment for use in preparing the fuel-salt samples for analysis was tested and was satisfactory after a few minor improvements were made. The tests were on samples obtained from the Engineering Test Loop by means of the prototype of the sampler-enricher. Maximum recovery of the sample after preparation for analysis was ~95%; average recovery was ~90%.

Fractions of each of the prepared samples were dissolved. These solutions are being used to evaluate the analyses of U, Zr, Cr, Be, and F.

Investigations were continued on the determination of oxide in MSRE fuel salt by means of the inert-gas fusion technique. The salt, packed in a closed graphite container, is ignited by induction heating for 1 min in an argon atmosphere at temperatures of the order of 2500°C. Oxide ion is released as CO, which is then oxidized to CO_2 and measured by any of several methods. Gas chromatography appears to be the most feasible method for hot-cell application. The graphite container is shielded to prevent arcing from the graphite to the quartz assembly. Preliminary results indicate higher oxide values than those previously reported by the $KBrF_4$ technique.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. — Proposals to fabricate 33 HFIR fuel elements were received from six qualified vendors. Representatives from all vendors had visited ORNL and observed our techniques before submitting their proposals. Visits have been made to evaluate the plants and capabilities of all the vendors. The final evaluations of the proposals are now being made.

A segment of an HFIR cylindrical control plate was formed without occurrence of the core cracking, which has been a problem. Two changes had been made in the plate: (1) A second aluminum plate was glued to the plate to be formed so as to move the neutral axis of the composite into the all-aluminum plate; this made all stresses in the cored plate compressive stresses, which are less likely to cause cracking. (2) The europium oxide dispersion core was fabricated as a single piece rather than made up from small segments. Additional work is required to show whether both changes are necessary to eliminate the cracking.

The homogeneity inspection of all plates for the critical inner annulus has been completed, and for the outer annulus over two-thirds have been inspected. The scanner has worked surprisingly well (even at high speeds) for a first test. The tremendous volume of data being obtained has not been examined in detail, but seems to confirm the spot-testing results and shows the plates to be acceptable.

Component Development. — Dynamic corrosion tests are being conducted on 0.250-in.-thick platelets consisting of europium oxide clad with 6061-TO aluminum. The platelets are in contact with 2-in.-diam rollers of Stellite 3, type 304 stainless steel, and 6061-T6 aluminum. These specimens are immersed in pH 5.0 demineralized water at 195°F throughout the tests. The europium oxide specimens were prepared by the Metallurgy Division and are considered representative of "as fabricated" HFIR control plates. The specimens are mounted vertically in the bearing tester, and a force of 100 lb is applied by the roller. Intermittent reciprocating motion is used to simulate motion that is typical of reactor operation. After one week of operation, pits 0.002 in. deep were noted on all the plates, regardless of roller material. These pits were concentrated principally at the ends of the surface over which the roller tracked. After 18 days of exposure, the initial pits were 0.005 in. deep and new pits were noted. This test is being continued.

The prototype HFIR outer control-rod drive has been tested under full pressure and temperature in the high-pressure test stand. Dummy weights were used to load the drive rather than a control plate. The drive performed satisfactorily during the tests. The prototype coincidence circuit magnet has been received and is presently being tested.

Fabrication of the HFIR structural components is progressing, and all major welding operations on structural parts are now complete. All the beryllium components are under fabrication. The permanent reflector, semipermanent reflector, vertical experiment facility plugs, and control-rod access plugs will be completed in April 1964. Welding procedure development is continuing for control-rod tracks.

Design and Analysis. — The fuel-plate slot depth in all of the fuel-element side plates except the outer side plate of the outer annulus is being increased 25 mils to improve the weld integrity.

A brief study of the effects of partial blockage of the inlet to the fuel-element coolant channels was undertaken. Assuming this restriction

to result in only a contraction and expansion loss at the channel inlet, with a uniform distribution of coolant across the channel width downstream of the restriction, the results of this study indicate that over 50% of the channel inlet could be blocked before hot-spot incipient boiling would occur at the design power of 100 Mw.

An effort was made to correct the digital computer code being developed for predicting the velocity and temperature distributions of the control-region coolant. It was found that use of correction factors on the momentum equations (called momentum coefficients) of values other than 1 resulted in predicted pressure drops through the system close to those measured experimentally.

The outer control-rod thermal deflection experiment is being modified to investigate the effect of a step change of the temperature in the axial direction on the deflection.

Instrumentation and Controls. – The coincidence element for the HFIR safety system is the electromagnet. (The theory of operation of a magnet of this type is described in ORNL-3191, pp. 66–72.) The magnet must produce a sufficient excess holding force to ensure that the rod is not inadvertently dropped. The holding force is based on the design load of the HFIR shim system, which is 880 lb.

A prototype FeNi magnet having the following properties has been designed and tested. With one coil energized, a holding force of 100 lb is produced; two coils produce 1380 lb, and three coils energized produce 1620 lb. Thus with only two coils energized a holding-force margin of 500 lb is produced. The release time of this magnet was measured by using the pulse current technique described in ORNL-2318. Also, a linear differential transformer was used to detect the first armature motion. With a load of 880 lb the release time is 4.3 msec when three coils are de-energized, and 7.5 msec when only two of the coils are de-energized.

Further investigation is now under way to determine the performance of a system composed of the above magnet and the mechanical components of the scram latch.

PHYSICAL RESEARCH

Reactor Operations. — The ORR operated 70.0% and the LITR 95.0% of the time during January and February 1964.

A prototype of the newly designed ORR control-rod drive mechanism was installed in the ORR. If this proves to be successful, five more drive mechanisms will be installed in June. The new drives are expected to be more reliable and to cause less trouble.

Four new beryllium reflector core pieces were installed in the ORR. All reflector pieces are being replaced, as the radiation damage causes dimensional tolerances to be exceeded.

The north large engineering facility has been converted to two beam holes, and one beam experiment is in operation. Equipment for the second hole is being installed.

Waste Disposal. — A review of the liquid waste discharges during 1963 has shown that less than one-half of the radioactive strontium released to the Clinch River went through normal radioactive waste system channels. The remainder was released as a result of leaks in the underground waste pipes through other routes, such as the sanitary sewage disposal system and storm sewers. In spite of these failures, the monitoring system built up during the last several years enabled the operators to detect the line leaks soon after they occurred, and the total release of strontium (8.8 curies) compares favorably with those of previous years.

Hot-Cell Operations. — Operations in the hot cells were normal. Four fueled capsules from the ORR were dismantled and examined; three of the capsules contained fueled graphite balls containing UC_2, and the other contained pyrolytic-carbon-coated UC_2 particles. The NaK was removed from four UO_2-fueled capsules irradiated at the ETR; these were stored for future examination. Two UO_2 meltdown experiments designed to study fission-gas release were disassembled and examined. Two experimental Army PM fuel bundles were disassembled and examined visually, and their dimensions were checked; they were reassembled for reinsertion in the ORR. Bend test measurements were made on about 128 irradiated specimens. Physical measurements were made on nine ORR beryllium reflector pieces, and several ORR fuel elements were visually examined for scratches.

Installation and final check of equipment continued at the High-Radiation-Level Examination Laboratory.

PHYSICS AND MATHEMATICS

Neutron Diffraction. — Neutron diffraction measurements were made on a series of rare-earth intermetallic compounds to determine the type of magnetic order and its relationship to the number of valence electrons in the system. The compounds studied included TbCu, TbZn, TbGa, TbAg, TbHg, and TbIn. The compounds with Cu, Zn, Ag, and Hg were found to have the CsCl type of structure, TbGa the CrB type, and TbIn a complex tetragonal unit cell. The compound TbGa is ferromagnetic with a Curie temperature of 155°K, and TbIn is antiferromagnetic below 190°K. For the compounds with the CsCl structures, TbCu and TbAg are antiferromagnetic with

Néel temperatures of 115 and 100°K, whereas TbZn and TbHg are ferromagnetic below 160 and 80°K respectively. This difference in magnetic behavior between the compounds with group Ib and IIb metals must be associated with the number of valence electrons rather than with interatomic separations, because the lattice constants of the two groups overlap.

Low-Energy Neutron Time-of-Flight Spectroscopy. — Neutron transmission and radiative capture measurements have been carried out from 1 ev to 30 kev at the Rensselaer Polytechnic Institute electron linac laboratory. The measurements were made with enriched samples of V^{50}, La^{138}, Sn^{120}, the W isotopes, and the Hf isotopes and with naturally abundant samples of V, La, Sn, W, Pr, Cu, Y, F, Sc, Zr, Zn, and Ga. The fast-chopper group's 4096-channel analyzer was moved from ORNL to RPI to facilitate this experiment; and it was possible, by using all the RPI analyzers as well, to simultaneously accumulate data on neutron radiative capture as a function of time of flight, neutron transmission as a function of time of flight, and the pulse-height distribution from the large liquid scintillator capture detector as a function of time of flight. All these data are being processed, but some preliminary results can be stated. A resonance has been observed in capture measurements upon tungsten at 15.5 ev which is assigned to W^{180}; this confirms partial capture measurements at Argonne, in which the resonance was observed but an isotopic assignment was not possible. Measurements have been completed on the detection efficiency of the liquid scintillator in the case of the tungsten isotopes (approximately 80% for all the isotopes), and the tungsten resonance parameters have been sent to the Sigma Center at Brookhaven. Capture measurements upon fluorine indicate resonances at 11.8, 13.5, 15.0, and 27.8 kev, whereas thick-sample transmission measurements exhibit only the strong resonance at 27.8 kev and a very weak (if real) resonance at ~22 kev. Resonances were observed in La^{138} at 21.0, 67.1, 90.5, 132, 220, 240, 261, and 308 ev; no new resonances were observed in V^{50} below 4 kev. Capture measurements upon Sn^{120} indicated resonances at 366, 427, 926, 955, 1282, 1441, 1730, 2900, 3170, 3930, 4330, 4470, 5230, 5350, 6780, (7120), 7630, (8420), and 8880 ev, where the energies within parentheses indicate weak resonances. A measurement of the resonance at 62 ev in tin (due to Sn^{124}) indicates a possible weak satellite resonance at 62.2 ev.

Low-Temperature, Nuclear and Solid State Physics. — The electrical resistances of nickel and of an alloy of nickel with 2 at. % gold have been measured in the temperature range 4.2 to 1240°K. The difference in resistance $\Delta\rho$ between the two samples rises from a value of 0.76 microhm-cm at 4.2°K to a highest measured value of 3.3 microhm-cm near the Curie point. At temperatures above the Curie point, $\Delta\rho$ first falls sharply, and then decreases slowly to a value near 1.2 microhm-cm at 1240°K. The sharp rise of $\Delta\rho$ below the Curie point may be associated with magnetic scattering, whereas the relatively constant value for $\Delta\rho$ above the Curie point as well as the value at 4.2°K may be related to the gold impurity scattering.

High-Voltage Experimental Program: A Search for Interference Effects Between Nuclear Resonance and Coulomb Excitation in the $Na^{23}(p,p')$ Reaction. — The narrow, isolated resonance in the $Na^{23}(p,p')$ reaction at 873

kev, situated upon a continuum due to Coulomb excitation, would appear to be a favorable case for observing interference effects between compound-nucleus inelastic scattering and Coulomb excitation. The yield of 439-kev gamma radiation emitted from the first excited state of Na^{23} was measured from 828 to 1100 kev incident proton bombarding energy. To within the accuracy of the experimental data, about ±8%, the noninterfering compound-nucleus and Coulomb-excitation contributions adequately account for the shape of the resonance, although the presence of an interference effect cannot be definitely excluded.

High-Voltage Experimental Program: Search for a Spin-Spin Effect in the Interaction of Polarized Neutrons with a Polarized Ho^{165} Target. — Using the Bureau of Standards portable He^3 refrigerator [Advan. Cryog. Eng. 8, 443 (1963)], a single crystal of holmium metal was cooled to 0.34°K. At this temperature, and with a magnetic field of 3 kilogauss, a nuclear polarization of approximately 75% is obtained owing to the extremely large hyperfine interaction. The magnetic field was produced by a superconducting split solenoid. To investigate the spin-spin interaction at 350 kev, where the neutron polarization from the $Li^7(p,n)$ reaction is about 55%, measurements were made of the transmitted intensities with the target nuclei polarized parallel and antiparallel to the neutron polarization. The observed difference in intensity, (−0.11 ± 0.32)%, corresponds to a difference in cross section of +30 ± 85 mb, where the plus sign corresponds to a larger cross section for the parallel orientation. Since the Ho^{165} nucleus is highly deformed, a transmission measurement was made to determine the difference in cross section between a polarized and an unpolarized target. The transmission for the polarized target was smaller by (1.3 ± 0.4)%, which corresponds to a cross-section increase of 350 ± 100 mb upon polarization. This deformation effect on the cross section is in agreement with the effect from the coupled channel calculation.

CHEMISTRY

Effects of Radiation on Analytical Methods. — The investigation of the effect of gamma radiation on the polarographic determination of uranium(VI) in uranium(IV) solutions has been continued. It has been found that radiolytic oxidation of uranium(IV) in 85% phosphoric acid is much slower when the solution is saturated with argon rather than air. The effect of the uranium(IV) concentration on this radiolytic oxidation is under examination.

The study of the effect of gamma radiation on the nickel-dimethylglyoxime complex has been continued. It has been found that the nickel-dimethylglyoxime color, due to the complex, is quite sensitive to gamma radiation. It has been observed, however, that on removal of solutions containing the complex from the radiation field, the color grows back to its original intensity. Tests are being conducted to ascertain the probable mechanism of this reaction. It is hypothesized that the nickel(III) in the color complex is radiolytically reduced and then is reoxidized after removal from the radiation field.

Separations Chemistry Research. — Commercial diethylbenzene (61% meta, 28% para, 5% ortho), a diluent used in radiochemical solvent extraction processing, was irradiated for approximately 100 whr/liter in a 10,000-curie Co^{60} source while being mixed with 2 M aqueous nitric acid. The irradiated material contained 0.7% nitrogen, and gas-liquid chromatographs showed that the products formed were the same as those previously observed (ORNL-3568) after nitration in the absence of radiation. Irradiation of a 1 M tributyl phosphate (TBP) solution (in diethylbenzene) incorporated more nitrogen (0.85%), with no change in the types or distribution of products.

In a previously reported study of interaction between the components of synergistic extraction combinations (ORNL-2471), molecular association between di(2-ethylhexyl)phosphoric acid (HA) and tri-n-octylphosphine oxide (TOPO) was demonstrated by means of infrared spectrophotometry. However, the possibility of a corresponding molecular association between HA and TBP could not be examined because of a fortuitous overlapping of the infrared absorption bands. Reexamination by means of dielectric measurements reconfirmed the association between HA and TOPO, and also showed association between HA and its complex salt NaA·3HA, but showed almost no association between HA and TBP.

Separations Process Development. — In testing the use of the phenol extraction (Phenex) process for recovering cesium from pollucite ore leach liquors, a carbonate stripping process was successfully demonstrated in continuous mixer-settler equipment. The feed liquor was prepared by roasting the ore with a mixture of sodium chloride and sodium carbonate at 700°C and leaching with water. About 99% of the cesium was recovered in four extraction and five water-scrub stages with 1 M 4-sec-butyl-2-(α-methylbenzyl)phenol (BAMBP) in kerosene. Contact of the extract with carbon dioxide and water gave a pure cesium carbonate product solution containing only 0.008% combined alkali-metal impurities. Overall cesium decontamination factors for the solvent extraction process were 360 from rubidium, >40,000 from potassium, 500,000 from sodium, >9000 from lithium, >37,000 from silica, 500 from iron, and >260 from aluminum.

Volatility Studies. — The compound $NaF \cdot UF_6$ was prepared by gas-solid reaction for the first time, and the decomposition-pressure curve was determined. The compound forms readily with pulverized Harshaw pellets of sodium fluoride or from HF-activated sodium fluoride by reaction with UF_6 at 55 to 85°C. The compound is white. The decomposition data conform to the equation $\log P = 10.965 - 3.45 \times 10^3/T$, where P is the decomposition pressure in millimeters and T is the absolute temperature.

The compound $2NaF \cdot UF_6$ was also prepared for the first time by gas-solid reaction from Harshaw pellets of sodium fluoride without HF activation; the pellets were pulverized for this preparation.

Repeated, alternating sorption and desorption of UF_6 from 1/8-in.-diam Harshaw sodium fluoride pellets, on the same pellets broken to 12 to 16 mesh, and on powder made by pulverizing the same pellets demonstrated some interesting differences. The initial loss of sorption capacity noted on the second and third cycles was diminished in the case of the broken pellets and absent with the powder. The gradual increase in capacity after the third cycle with the pellets was accentuated with the broken pellets.

The initial complete conversion to $2NaF \cdot UF_6$ obtained with powdered sodium fluoride on the first cycle was retained on subsequent cycles for the powder.

Repeated sorption-desorption cycling showed another interesting difference between the pulverized pellets and the HF-activated sodium fluoride: $2NaF \cdot UF_6$ could be formed repeatedly on cycling for pulverized pellets, whereas the HF-activated material gradually lost that ability with repeated cycling.

Radiolysis of Metal Fluorides. — Continuation of irradiation of MSRE fuel salt of <0.002 in. in diameter (0.52 m^2/g) with 250-kv x rays gave $G(F_2)$ values as high as 0.07 (calculated from F_2, CF_4, and COF_2). In all cases, analysis showed oxygen and carbon dioxide in excess of that expected from the air content (based on argon and nitrogen). If it is assumed that the O_2 and CO_2 were generated by fluorine reactions with impurities in the salt, even higher G values are obtained.

Since the effects of impurities might be overridden by using a much higher rate of energy input, a beryllium-window type of x-ray tube was obtained in order to test the effect of the very high output available. Cells for containing the test salts were designed with very thin beryllium windows in place of the thin nickel diaphragms previously used. A ferrous sulfate dosimeter indicated an average dose rate of 10^{20} ev g^{-1} hr^{-1} into a 10-g sample of MSRE fuel-type salt. This input rate is about 15 times that obtained with the tube used in previous experiments. Subsequent irradiation experiments with the 150-kv beryllium-window tube and a portion of the long-irradiated MSRE fuel salt gave results consistent with those obtained with the lower-dose-rate apparatus. An elemental fluorine concentration of 3.6% was obtained during a 69-hr irradiation of fuel salt, which was at least an order of magnitude higher than might have been expected under previously used conditions.

Nuclear Methods of Chemical Analysis. — During March we completed and put into final form the first portion of the report 14-Mev Neutron Reactions. This report contains a description of the reactions observed when pure, naturally occurring elements (Z = 1 to 30) are irradiated with unmoderated 14-Mev neutrons produced by the $H^3(d,n)He^4$ reaction. Cross sections are calculated where possible, and normalized standard gamma-ray spectra are collected. These standard spectra are equivalent to the gamma radioactivity that will be produced when 1 g of the pure element is irradiated for a given length of time at a 14-Mev neutron flux of 5×10^8 neutrons cm^{-2} sec^{-1}. If more than one radionuclide is produced, a spectral decay is presented. These spectra may be utilized in accurately predicting the sensitivity of nondestructive spectral analysis of almost any sample.

Nuclear Chemistry: Neutron Cross Sections. — A measurement was made of the thermal neutron capture cross section and the resonance capture integral of U^{232}, the 73.6-year alpha-emitting isotope of uranium. Microgram quantities of U^{232} initially containing less than 1% of U^{233} were irradiated in the core of the ORR for times up to 150 hr. Measurements of the U^{233} formed during the course of the irradiation were carried out on submicrogram quantities of uranium with a two-stage mass spectrometer. By comparing the results obtained in irradiations made with and without

cadmium filters, a thermal (subcadmium) cross section of 79 barns and a resonance integral of 277 barns were found for radiative capture in U^{232}. The uncertainty in the cross sections is thought to be less than 10%. These values are based upon the Co^{59} cross sections, since dilute alloys of cobalt in aluminum were used to monitor the irradiations, and to a minor extent upon the U^{233} absorption and U^{232} fission cross sections, which must be known to account for the burnup of these species during the irradiation.

Organic Chemistry. — In the course of our study of the mechanisms of rearrangement reactions by means of labeling techniques with the radioisotopes C^{14} and tritium, we have now completed experiments on the acid-catalyzed rearrangement of 2-phenylnorbornane-2,3-cis-exo-diol. The reaction proceeds with intramolecular rearrangement of hydrogen from one carbon to the next one, that is, to the carbon bearing the phenyl group. The phenyl group remains attached to the same carbon atom throughout the rearrangement. Stereochemical studies show that the configuration of the product is inverted with respect to the configuration of the reactant.

Electrochemical Kinetics and Its Application to Corrosion. — In many and perhaps all cases, the dissolution of zirconium in solutions containing HF involves the continuous formation and dissolution of a film. The true rates of the surface reactions between HF and the ions at the surface of the film have been investigated with a rotating-disk electrode. With this apparatus, the effects of mass transfer are eliminated by measuring the current as a function of rotational velocity. The following systems have been investigated: zirconium in $HF-HNO_3$, zirconium in $HF-H_2SO_4$, and zirconium—15% niobium in $HF-H_2SO_4$. The rate of the surface reaction of zirconium in $HF-HNO_3$ is first order with respect to HF, but there is a small effect tending to reduce the rate at low concentrations of HF. For HF concentrations varying from about 10^{-3} to 5×10^{-2} N, the rate varies from about 1.2×10^{-3} amp/cm^2 to 2×10^{-1} amp/cm^2. The dissolution of zirconium and zirconium—15% niobium in $HF-H_2SO_4$ is much more complicated, since the rates are lower than in $HF-HNO_3$ and sharply decrease at low concentrations of HF. A mechanism involving the adsorption of sulfate on the surface accounts for most of the observations.

Chemical Physics: Microwave and Radio-Frequency Spectroscopy. — Single crystals of dimethylglyoxime after irradiation at 77°K with gamma rays show two prominent paramagnetic resonance spectra. One of these spectra is stable at room temperature, whereas the other slowly disappears over a period of several days. We have studied both spectra at room temperature and have evaluated the appropriate tensors of the spin Hamiltonians. A more precise evaluation than usual was made possible by developing programs for a computer that allowed all parameters to be found with a least-squares procedure.

The species stable at room temperature has been previously described. The spectrum is typical of a paramagnetic species with a single unpaired electron (doublet state) with a hyperfine coupling to a single nitrogen nucleus.

The second species contains two unpaired electrons (triplet state), and the g tensor, the hyperfine tensor, and the electron spin-spin interaction tensor have all been evaluated. To our surprise, the g and hyperfine tensors (a total of 12 numerical parameters) were almost identical

with those found for the stable, doublet-state species. This strongly indicates that the triplet-state species is an interacting pair of the doublet-state species. The characteristic directions and intensity of the spin-spin interaction substantiate this interpretation and show that the paired entities lie along one of the crystal axis directions displaced just one unit cell distance apart. The doublet species is probably formed by the removal of a hydrogen atom from the parent molecule. The triplet-state species would then form by this atom selectively abstracting a hydrogen atom (to form H_2) from a neighboring parent molecule.

Physical Chemistry of Molten Salts. — In a study of the wetting behavior of ionic melts, linear relationships were noted between the surface tension of molten fluorides and the cosine of the contact angle θ between the melts and either graphite or platinum (transition from wetting to nonwetting at $\cos \theta = 0$). A similar relationship for families of organic liquids and solids had been reported in the literature, but application to the interfacial behavior of molten salts has only begun to be explored.

Interest in the atomistic structure of the surface of an ionic melt led to consideration of a family of linear relationships between the crystal energy U_0 and the surface tension of alkali halides at their melting point. For mixtures with a common anion, since the surface might be expected to be richer in the component of lower surface tension, surface tensions that were low relative to the linear relationship were not surprising; however, eutectic melts fell on the line. The linear relationship was interrupted by a break or a shift to a new line for CsI, RbI; CsBr, RbBr; and CsCl, RbCl; this may be related to a different surface structure for these melts, since the anomaly is present only for those salts known to have a CsI structure under certain temperatures and pressures and is absent with fluorides. The usefulness of such apparent correlations, which may involve the structure of the surface, has not yet been explored in any detail.

High-Temperature Aqueous Solution Chemistry. — In high-temperature pressurized water systems operated under reducing conditions (e.g., pressurized water reactor systems), the principal species of dissolved iron believed to be present are Fe^{2+} and any soluble hydrolysis products. In a continuing program of high-temperature acidity measurements, the hydrolytic behavior of Fe^{2+} was investigated at temperatures approaching those employed in such systems (e.g., 260°C). For these measurements a hydrogen electrode concentration cell of the type previously employed (ORNL-3307) is being used. As anticipated, the presence of a hydrogen atmosphere prevented oxidation of Fe^{2+}. However, rapid reduction of traces of Fe(III) initially present appeared to occur only when sufficient acid (>0.01 m) was present at the beginning of an experiment to prevent extensive hydrolysis of Fe^{3+}.

The initial results (in 0.1 m NaCl medium at 95, 150, and 200°C) were found to be consistent with the simple hydrolysis reaction $Fe^{2+} + H_2O \rightleftharpoons FeOH^+ + H^+$. A value of $\sim 10^{-9.5}$ for $Q_{1,1} = [FeOH^+][H^+]/[Fe^{2+}]$ at 25°C in 1 M $NaClO_4$ has been reported. Hydrolytic precipitation occurs, however, before the reaction has reached 10% completion. Values of $Q_{1,1}$ are presently estimated as $10^{-7.5}$, $10^{-6.3}$, and $10^{-5.9}$ at 95, 150, and 200°C respectively.

Chemical Separation of Isotopes. — The infrared spectrum of gaseous chloryl fluoride has been examined in detail from 300 to 3000 cm^{-1}. The chlorine and oxygen isotopic structures of many of the bands were resolved. The Raman spectrum of liquid chloryl fluoride was observed from 0 to 1500 cm^{-1}, and polarization measurements were recorded. All fundamental frequencies of the molecule were observed in both infrared and Raman spectra. Valence force constants were calculated which closely reproduce the observed fundamental frequencies, as well as the isotopic shifts observed when the molecule was labeled with Cl^{37} and O^{18}. The observed and calculated fundamental vibrational frequencies are as follows:

	$FCl^{35}O_2$		$FCl^{37}O_2$		$FCl^{35}O_2^{18}$		$FCl^{37}O_2^{18}$	
	Obs	Calcd	Obs	Calcd	Obs	Calcd	Obs	Calcd
ν_1	1105.8	1105.8	1098.4	1098.4	1060.4	1058.8	1052.6	1050.9
ν_2	630.2	630.2	621.6	621.6	624.7	626.3	616.0	617.7
ν_3	547.9	547.9	544.4	544.4	528.0	524.7		521.3
ν_4	401.6	401.6	400.0	400.0		393.5		391.9
ν_5	1271.4	1271.3	1258.6	1258.5	1229.6	1226.1	1215.0	1212.8
ν_6	367.0	367.0	(365.5)	365.5		357.0		355.6

TRANSURANIUM-ELEMENT PRODUCTION

Chemical Process Development: Tramex Modification to Produce Chloride-Free Curium. — Since a potential hazard would exist if solvent were present when curium product is converted from chloride to nitrate by distillation, a modified Tramex flowsheet, which will produce chloride-free curium in 1 M HNO_3, was developed.

In this process, americium and curium are extracted with 0.6 M Alamine 336·HCl in diethylbenzene from 11 M LiCl feed as before; however, the Alamine 336 containing the extracted americium and curium is then converted from the chloride to the nitrate by contacting it with 8 M $LiNO_3$ in a partition column. Solvent from the partition column is then stripped with 1 M HNO_3. In a laboratory-scale mixer-settler demonstration of this process, made with tracer activities, product from the strip contactor contained 98% of the curium, about 30% of the ruthenium, and no detectable zirconium.

Although chloride analyses have not yet been made, previous results indicate that a good curium product in nitric acid can be obtained. In order for this process to be acceptable, ruthenium decontamination must be improved. As yet, there are no plans for installing the necessary third contactor in cell 4 of the Curium Recovery Facility.

Chemical Process Development: New Actinide-Lanthanide Extraction Systems. — Various solids are being tested as supports for solvents in

extraction chromatography (known also as reversed-phase chromatography). Johns-Manville Chromasorb W (60 to 100 mesh), manufactured for use in gas chromatography, absorbed about 0.8 ml of 1 M di(2-ethylhexyl)phosphoric acid (HDEHP) in diisopropylbenzene per gram of solid. Americium and europium were separated by elution with a mixture 1 M in glycolic acid and 0.025 M in pentasodium diethylenetriaminepentaacetate from a column of this composition, but the separation was not so sharp as expected. Europium was extracted by HDEHP absorbed on a column of porous unsulfonated polystyrene-divinylbenzene copolymer beads (60 to 100 mesh), but elution by nitric acid indicated that diffusion within the beads was very slow.

The extraction of lanthanides and americium by a naphthenic acid, Sunaptic acid A, was highly dependent on acidity. Distribution coefficients between 1 M Sunaptic acid in Amsco 125-82 and 0.1 M citric acid at pH 6.8 were 0.4, 0.7, and 1.1 for europium, americium, and cesium. At high pH's, coefficients greater than 100 were attained, but more than 24 hr was required to reach equilibrium.

A survey is being made of various substituted acetic, propionic, and stearic acids as extractants.

Curium Recovery Facility. — Cells 3 and 4 in Building 4507 are being equipped to test transuranium process chemistry at full-scale activity levels and to recover gram quantities of Cm^{242} and Cm^{244}. Design and installation of equipment are nearly complete, and cold runs for testing chemical flowsheets are in progress.

Flowsheet and equipment tests have been completed on simulated target dissolutions, Tramex and Clanex (co-lanthanide-actinide nitrate extraction) feed adjustments, and conversion of product from hydrochloric acid to nitric acid solutions. Flowsheet testing with the mixer-settler was hampered by air locking of the gravity flow lines and by severe emulsion problems encountered with Tramex reagents. The emulsions are now believed to have been caused by molybdenum in the lithium chloride solution.

Process Equipment Development. — Four process pumps are being operated simultaneously, two with annealed Zircaloy-2 diaphragms and two with the material as received (10% cold work), as part of a program to determine and specify the optimum diaphragm material. Six pumps were fabricated for use on the first-cycle solvent extraction system according to the tentative final design and are being tested. These tests show that closer tolerances are required for certain parts to ensure that spare parts are completely interchangeable. Remote assembly and disassembly of a pump were demonstrated with the impact wrench, the master-slave manipulators, and a simple alignment jig.

In a cell mockup study to demonstrate a remote maintenance procedure, the hot disconnect well and tube bundle were removed and replaced completely by remote operations. Some minor modifications are needed to provide better support and to simplify the operation, but the design and procedure were shown to be adequate.

HFIR Target Development. — The six Pu^{242}-containing target rods to be irradiated in one of the Savannah River Laboratory production reactors were fabricated in the new alpha laboratory in Building 3019. After being delayed for several days because of difficulties with the off-gas system for the glove box, operation with Pu^{242} was begun on March 1. Two batches

of target pellets (38 pellets each) were successfully fabricated with no difficulties.

The finned tubes for containing the pellets were machined into the required configuration, and the closure welds were completed. The round spacing can that will be attached to the finned tubing was drawn to the desired dimensions, and the required machining operations were finished. Several tests were performed to determine the adequacy of a mechanical staking procedure for attaching the spacing can to the finned tubing. The tests showed that the pull strength of the joint was about 175 lb, which should be more than adequate. Fabrication of the targets was completed on March 26.

The design of a glove box to enclose the metallograph for the plutonium metallographic section has been completed. An order for this glove box, along with the remainder of the required metallographic equipment, will be placed in the near future.

Transuranium Facility. — Construction of the Transuranium Processing Plant continues on schedule. The first- and second-floor pads in the operating laboratory wing of the building have been poured, and the south, east, and west walls of the south half of the building are nearly complete. The 50-ton crane, the chiller units for the building air conditioning system, and the instrument air compressors were installed. All items made of Hastelloy C by the Nooter Corporation, including several pipe assemblies, the tank pit sumps, and the engineering-model cubicle floor pan, have been received. A contract was let with Nooter for providing the remaining eight floor pans, the design of which was greatly simplified in order to reduce costs. Also, the ten cell-shielding-window sleeves were received.

METALLURGY AND MATERIALS

Ceramics Research: Sintering Studies. — Observations of grain growth and pore closure in ThO_2 have continued with a more thorough study of etch pits on the surface of highly sintered ThO_2 compacts. Electron microscopy showed that the pits were formed by crystallographic planes tentatively identified as (100), (110), and (111). Observation of the pits in the optical microscope indicated that they may start to develop by thermal faceting and that a preferred orientation of grains may exist in a grain-growth specimen that had been heated for several hours at elevated temperature. In agreement with this, an x-ray analysis of a grain-growth specimen heated for 12 hr at 2220°C showed definite indication of a preferred orientation of grains. Further x-ray analysis will be used to determine the distribution of grain orientation in more detail.

Physical Properties Studies. — We are measuring the effect of controlled variables on heat transport in solids by several methods. Using our single-sphere thermal comparator, which rapidly measures specimens 1/4 in. thick and 1 in. or less in diameter in the range 50 to 400°C, we found that the thermal conductivity of ThS was 44.8 w m^{-1} deg^{-1} at 75°C and 37.3 w m^{-1} deg^{-1} at 300°C. Oxygen-deficient UO_2 was found to be a 15% better heat conductor at 300°C than the stoichiometric material. Since

the comparator will not work on coarsely dispersed mixtures or anisotropic materials such as graphite, we have built a longitudinal heat-flow apparatus that gives an absolute accuracy of ±3.5%. This apparatus will be used to complement the radial heat-flow measurements on graphite.

With the radial heat-flow apparatus, we measured the thermal conductivity of CGB graphite of 1.824 g/cm^3 density in the range 50 to 900°C in a direction perpendicular to the extrusion direction. It decreases with increasing temperature, obeying the equation $1/k = 0.5454 + 12.65 \times 10^{-4}T$ within ±5% at 100°C and ±1% at 900°C, k being the conductivity in watts per centimeter per degree, and T the temperature in degrees Kelvin.

Erratic thermocouple behavior at 1100°C and higher is a major limitation to high-temperature thermal conductivity measurements. To solve this we are drift-testing thermocouples at high temperatures under high vacuum. Thermocouples made from 90% platinum–10% rhodium vs platinum, 94% platinum–6% rhodium vs 70% platinum–30% rhodium, tungsten vs 74% tungsten–26% rhenium, and 95% tungsten–5% rhenium vs 74% tungsten–26% rhenium drifted less than ±5°C when exposed for 350 hr at 1300°C and 2×10^{-7} torr and less than ±10°C in 1000 hr at 1450°C and 4×10^{-8} torr. This was true for all thermocouples positioned in reentrant wells in an isothermal niobium block. However, thermocouples welded to the niobium block and exposed to a severe temperature gradient drifted seriously.

We have measured the total hemispherical emittance and electrical resistivity of Nb–10% W–1% Zr–0.1% C, a promising high-temperature alloy, at temperatures up to 1500°C. This alloy is inert in a vacuum of 10^{-7} torr and does not exhibit the oxidation and subsequent rise in emittance that have been observed for the Nb–1% Zr alloy above 400°C. The electrical resistivity was only 2 to 4% greater than that of pure niobium, which may be indicative of ordering occurring because of the tungsten addition.

We have investigated a method of determining specific heat with the emittance apparatus, in which we measure the time-temperature response of a rod specimen given an incremental increase in power. The results on iron at 520°C were within 2% of literature values, when the data were reduced by an unrefined computation. We expect agreement within 1% when a more refined computation is made.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. — Most of this report period has been devoted to detailed studies of plasmas established by Lorentz-force trapping. These experiments have usually involved injecting the maximum H_2^{+*} beam (260 to 320 μa) and making observations as the containment time τ was varied by controlling a gas leak into the plasma region. The τ range investigated was from the τ value at base pressures (up to 40 sec decay time on the central charge-exchange detectors) to tenths of a second. Lorentz-force trapping dominates gas-dissociation trapping throughout this τ range, so for a fixed H_2^+ current the proton current injected into the magnetic trap is a constant. Analyses of the charge-exchange signals from the usual array of neutral-particle detectors (foil-covered Faraday cups) indicate (1) that the total charge-exchange loss current is constant over the τ

range from 0.1 sec to about 2 to 4 sec, and by indirect argument, that this loss current is equal to the proton current input to the trap; (2) that the charge-exchange loss current then falls off markedly as τ is further increased; (3) that as τ is increased, axial scattering of the plasma rapidly becomes more pronounced, the central (maximum) plasma density approaches a limit of about 10^8 cm^{-3}, and the total number of stored fast protons approaches a limit of about 10^{12}. Studies of the charge-exchange signals as a function of the outer radial limit of the plasma have shown that the τ range of rapidly increasing axial scattering is also one of rapidly increasing radial scattering, and as a consequence it is believed that the apparent limitations are in large part the result of plasma losses to the radial walls. The experiments now in progress involve the use of energy analyzers and secondary-emission detectors for the charge-exchange loss currents. These studies should provide information on the extent to which the present data are influenced by the transmission characteristics of the foil detectors.

Beam-Plasma Interaction. — A factor of 5 scale-up in size (to 10 cm^3) has been made of the high-pressure region of the pressure-gradient (mode II) arc. This high-pressure region is thought to be a basic ingredient in our beam-plasma interaction studies. It is required for the hot-electron plasma described in these reports and is the region which a theoretical treatment describes as completely ionized or burned out of neutrals. Experiments with the scaled-up version of this burnout region give qualitative data which are in full agreement with the theory. Spectral light virtually disappears at the transition to the mode II regime. Strong radio-frequency radiation (200 v/m at 1/2 m from the plasma) at the ion cyclotron frequency is measured in the vacuum chamber housing the apparatus. Over 90% of the input power (3 kw in this instance) was accounted for as a radial loss to the plasma liner.

Theory. — A program is under way to analyze the dispersion relation for electrostatic instabilities in injection machines in much greater detail than was previously possible. The effects of electron temperature, anisotropy in the ion velocity distribution, and the finite size of the plasma are being surveyed. One effect of finite size is that a diamagnetic current must flow. In this respect, the plasma is similar to an ion beam and should exhibit the two-stream instability characteristic of beams. On the other hand, anisotropy also produces electrostatic instabilities, but generally over a more narrow range of parameters than the two-stream type. Preliminary results indicate that the anisotropy instability dominates over the beam instability when both are possible.

Radio-Frequency Heating. — The EPA Facility has been under extensive modification for the last two months for the purpose of preparing an energetic neutral-beam injection experiment. This experiment will involve the injection of a 15- to 22-kev neutral hydrogen beam equivalent to several milliamperes of ions into the hot-electron plasma. The energetic neutrals will be ionized by the hot electrons and trapped in the magnetic field. This hot-electron plasma is prepared by the application of several tens of kilowatts of 3-cm microwave power to deuterium gas in a 3:1 magnetic mirror.

This injection experiment will check the "blanket" effect of the plasma, that is, the reduction of neutral gas at the center of the plasma by ionization by the thick blanket of hot electrons. Measurements of the charge-exchange neutrals from the trapped hydrogen ions will measure the effectiveness of the blanket. In addition, the heating of the ions in the plasma by the trapped injected ions will be studied.

In the Physics Test Facility, a new 4-mm microwave interferometer has been constructed with a focusing arrangement improved over previous models. There are insufficient data at the present time to draw any conclusions about the density as measured by this interferometer. Further measurements with the interferometer are continuing in connection with gridded probe studies and energy density measurements. The gridded probes are also being used to study the peculiar instabilities that can be generated in the plasma. A new type of gridded probe and circuit has been designed to suppress possible discharges in the probe.

Construction of the redesigned Elmo Facility is under way and is expected to be completed in the late spring. The new facility will be involved in electron-cyclotron experiments with 3-cm microwave power in the double-folded cusp configuration and also in experiments with 1 or 2 kw of 8-mm power with the device operated as a mirror.

Cross Sections. — A proportional counter has been fabricated and evaluated for the detection of low-energy neutral hydrogen atoms. The entrance window consists of a thin Formvar film mounted on a 0.001 × 1 in. slit which is scanned across the spatial extent of the beam. The counter is filled with methane at a pressure of 1 to 2 mm. The energy resolution was 70% for 2.5-kev neutral hydrogen atoms and was 10.7% at 30 kev. Particles whose energy is 1 kev are detectable, but with less efficiency.

The total dissociation cross sections of H_2^+ and H_3^+ in hydrogen gas have been measured in the energy range 40 to 220 kev. The cross sections decrease monotonically with increasing particle energy. The cross section for H_3^+ decreases from a value of 8.3×10^{-16} cm^2/molecule at 40 kev to 2.8×10^{-16} cm^2/molecule at 200 kev.

Angular distribution measurements of the H_2^+ ions resulting from dissociation of H_3^+ have been completed. The measurements were performed over the energy range 60 to 200 kev and for target gases of hydrogen, water vapor, and xenon. Analysis of the data by conversion of the angular distribution in the laboratory system to the center-of-mass systems yields information regarding the reaction kinetics. For a given energy the angular distribution was constant as the target gas was changed, indicating that the kinetics are independent of the mass of the target atom.

BIOLOGY AND MEDICINE

RADIATION EFFECTS

Statistical Services and Research. — Chromosome-type aberrations induced by 2.5-Mev neutrons in humans have been observed to be functionally related to the dose of neutrons. Irradiated blood samples from five humans

were analyzed for deletions, rings, and dicentrics. These data were examined for homogeneity among the five humans; the deletion data and the data on rings and dicentrics were then fitted by least squares to the model $Y = \alpha + \beta D$, where Y is the number of aberrations per cell and D is the dose of neutrons.

The same dose of radiation of different types may affect living organisms with varying degrees of severity. It is important therefore to define a term such as "relative biological effectiveness" (RBE), which is merely the number of units of type I radiation which yields the same response as one unit of type II radiation. The RBE is usually estimated from the ratio of the slopes of two fitted linear regressions. If the doses of the two types of radiation are known precisely, and the dose-response curve is linear, there is no difficulty in estimating the RBE. Because of the physical limitation of certain radiation sources, however, the apparent total dose of type II radiation may be a mixture of both type I and type II radiation. For instance, a dose of 100 rads of neutrons from a neutron source might really be 90 rads of neutrons and 10 rads of gamma rays. If an estimate of the RBE of neutrons with respect to gamma rays is being sought, such a confounding of radiation types may cause some difficulties. Several techniques have been developed to overcome these difficulties.

Pathology and Physiology. — An experiment was done to study the effect of variables on the induction of glomerulosclerosis in irradiated mice. LAF_1 mice were exposed to 500–1200 r of whole-body or partial-body x rays at 3 or 12 months of age, at a dose rate of 90 r/min. In certain groups, injection of radioprotective chemical (AET) was given prior to irradiation. Necropsy showed that the incidence of spontaneous glomerulosclerosis was less than 3% in controls. An increased incidence was noted only in those groups receiving x-ray exposures of 650 r (approximately the LD_{50}) or over and only when the kidneys were directly exposed, ranging from 31% at 650 r to 93% at 1200 r. Gross observation of kidneys at intervals after irradiation indicated that there was a long latent period (about nine months) before such changes of glomerulosclerosis became evident. When radiation exposure sufficient to induce glomerulosclerosis was given at one year of age, the resulting final incidence was lower. It is felt that this was at least partly due to the length of the latent period, allowing death of many animals in such groups before the kidney lesions could become manifest. When one kidney was shielded and the other exposed to radiation, glomerulosclerosis subsequently appeared only in the irradiated kidney. The use of AET pretreatment did not significantly alter the final incidence of glomerulosclerosis at the three radiation dose levels tested.

Recovery from Somatic Effects of Radiation. — Studies on the morphology and function of bone marrow regenerating after sublethal irradiation, transplantation into irradiated recipient animals, and long-lasting plethorization strongly suggest that the proliferative capacity and hemopoietic competence of the marrow do not depend on its content of small, lymphocyte-like, mononucleated cells, which are capable of incorporating tritiated thymidine. The structure and function of these lymphocytes, as opposed to the lymph-node and lymph lymphocytes, have not been thoroughly

investigated, however, probably because of the complexity and heterogeneity of the marrow's cellular composition. Described here is an approach to reduce such heterogeneity by removing most, if not all, of the large blastic elements from mouse marrow cell suspensions.

Marrow cells from long bones of adult C3H and (C3H × C57BL)F_1 mice were suspended in Tyrode's solution at a concentration of 10^7 cells/ml. Aliquots of the cell suspensions were filtered twice through packed glass-wool columns of varying compactness to obtain varying degrees of lymphocyte enrichment. Marrow samples were smeared on glass slides for microscopic examination and stained with Ralph's hemoglobin and Wright-Giemsa stains. It was possible by filtration to remove all large cells, that is, all blast forms, reticulum cells, histiocytes, and monocytes, but to retain small and medium lymphocytes, normoblasts with pyknotic nuclei, metamyelocytes, and granulocytes. The lymphocyte enrichment was inversely related to the total cell yield and directly related to the compactness of the columns. Known amounts of the original and of the filtered marrow were transplanted into isogenic recipient mice exposed a few hours earlier to 900 r of x rays. The proliferative capacity of the transplanted marrow was estimated by measuring the marrow's ability to promote DNA synthesis in the spleens of the recipients, as judged by incorporation of labeled 5-iododeoxyuridine (I^{131}UdR), a specific DNA precursor, five days after transplantation. The hemopoietic function of the transplanted marrow was estimated by examining histologic sections of the recipients' hemopoietic sites 10 to 30 days later. Neither the filtration procedure nor the absence of blast cells was observed to reduce the proliferative and hemopoietic competence of the marrow, since a normal degree of I^{131}UdR incorporation, of hemopoietic colony formation, and of 30-day survival was accomplished by the filtered marrow. The uptake of I^{131}UdR in the recipient spleens appeared, furthermore, to be correlated with the content of small and medium lymphocytes in both the unfiltered and the filtered marrow, and not with any other cell types. The results indicate, therefore, that the marrow lymphocyte is indeed a multipotent hemopoietic stem cell in the mouse.

Physiology of Blood Platelets. — As part of an investigation of factors that regulate platelet production and maintain circulating numbers, the platelet levels of otherwise normal rats were raised by transfusion of freshly collected platelets to two or three times the normal level to study the effect on megakaryocytes and on platelet numbers at intervals after transfusion. The number of megakaryocytes in the bone marrow of rats killed at intervals between 2 and 8 days after the beginning of a course of two or three transfusions was not altered. In addition, platelet numbers were not appreciably depressed below normal levels after their decline from the hypertransfusion level. These results indicate that the presence of abnormally high numbers of platelets in the circulation does not reduce the numbers of platelet precursors, although it may arrest megakaryocyte maturation.

Medium(Low)-Level Long-Term Effects of Radiation. — Male and female mice of the RF/Up strain were exposed to whole-body irradiation, beginning at 8 to 10 weeks of age. The radiations and dose rates were as follows: 250-kvp x rays (70–80 rads/min), Co^{60} gamma rays (0.003–50 rads/min), cyclotron fast neutrons (50–120 rads/min, ~1.3 Mev mean energy), and Po-Be fast neutrons (0.0003–0.01 rad/min, ~5 Mev mean energy). Irradiation at

a given dose rate was continued until either a predetermined dose had been accumulated or 50% of the exposed population had died. The duration of survival decreased with increasing dose and dose rate, but the life-shortening effectiveness of the radiation also diminished at high dose levels, especially in those chronically exposed populations subject to onset of mortality before the end of exposure. Under the latter conditions, the extent of life-shortening by the gamma rays was only about one-third as high (~0.02–0.07 day/rad, depending on the dose rate) as it was when the daily gamma irradiation was discontinued before the onset of mortality, and about one-tenth as high as when the gamma irradiation consisted of a single brief exposure. In the case of fast neutrons, the life-shortening effectiveness under such conditions was about two-thirds as great (~0.3–1.0 day/rad, depending on the dose rate) as it was when the daily neutron irradiation was discontinued before the onset of mortality or when the neutron irradiation consisted of a single brief exposure.

RADIATION GENETICS

Mammalian Biochemical Genetics. — A single band of esterase activity in the serum of C57BL mice and two esterases in the sera of most other strains of mice are observed in the region of serum albumin following electrophoresis in starch gels. Studies on the substrate specificities and gene-controlled molecular alterations of the allelomorphic forms of these serum esterases require isolation of the enzyme. The esterase of C57BL serum was separated from most serum proteins on diethylaminoethyl (DEAE) cellulose by gradient elution with sodium phosphate and sodium chloride. Sephadex G-200 was used to remove the small quantities of macro- and gamma-globulins that remained with the esterase following passage through DEAE cellulose. Ultracentrifuge data indicated that the esterase has a molecular weight of approximately 70,000, which is similar to that of albumin. Guanidine hydrochloride did not dissociate the esterase into smaller components, indicating that it is not an isoenzyme.

Mutagen Mechanisms: Bacterial Genetics. — *Escherichia coli* K-12 AB1899 is an encapsulated mutant bacterium sensitive to x-ray and ultraviolet (2537-A) radiation. The mutation leading to enhanced sensitivity involves an alteration in the genome close to the region controlling sensitivity to the bacteriophage T6 [Genetics 49, 237 (1964)]. An independently isolated encapsulated mutant derived from *E. coli* K-12 HFrH has also been shown to involve an alteration in the region of the T6 locus [Bacteriol. Proc., p. 107 (1963)]. We have established that this mutant is radiation sensitive. It, like AB1899, forms long nonseptate filaments after exposure to ionizing radiation. These filaments can be induced to form cross walls and divide normally by the incorporation of pantoyl lactone in the postirradiation plating medium.

The apparent linkage between the formation of capsules and the failure of the mechanism responsible for cross-wall formation observed in these strains led us to search for uncapsulated mutants. One such mutant has been obtained that, although devoid of capsular material by chemical

and morphological criteria, retains the radiation sensitivity and other characteristics of the AB1899 culture from which it was derived.

The isolation of this new mutant establishes that the formation of capsules is not necessarily related to the radiation phenomena described for these E. coli strains. The genetic basis for the new alteration is under investigation.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — Although short-term culture techniques have been successfully applied to the leukocytes of a variety of animals including man, no reproducible method has yet been devised for the mouse. Since the metabolic rate of most dividing cells is higher than that of nondividing cells, attempts were made to vary the culture conditions in order to vary the physiological state of the cultured cells. To minimize the errors, a single variable was studied at a time. Cells were generally maintained in tissue culture 199, with added 25% calf serum, phytohemagglutinin, and antibiotics. The variables tested included age, sex, and strain of mice; method of narcotization; methods of leukocyte separation; various commercial culture media; and serum at varying concentrations from a variety of animals. Physical variables were also studied: variation of the gas phase above the cultures, regulation of pH over a wide physiological range, variation of culture temperature, and variation of culture vessel and volume.

Cells were examined at daily intervals up to seven days, when most cells showed signs of disintegration. Although mouse blood contains a high percentage of lymphocytes — cells that have been implicated in mitotic activity in leukocyte cultures of other species — there was no evidence of mitosis under any culture condition in our laboratory. However, successful cultures were obtained from human, rat, and rabbit blood. Three possibilities may be suggested as reasons for the apparent inability of mouse leukocytes to grow in culture: (1) the culture conditions may not be optimal; (2) mitotic activity in other animals may be an allergic response to phytohemagglutinin from a previous encounter in nature; (3) the type of mononuclear cell capable of division may not be present in the mouse. The last possibility seems to be the most likely one at present.

Tissue Culture. — Peripheral blood samples have been obtained from persons irradiated in the radiation incident at the Puerto Rico Nuclear Center approximately 18 months ago, and the material is being scored for chromosomal aberrations. Scoring of human leukocyte samples irradiated with 730-Mev protons has been completed, and the scoring of samples irradiated with fission-spectrum neutrons from the Health Physics Research Reactor operated in both steady-state and fast-burst modes is in progress.

An electron-microscopic study of the changes induced in human leukocyte populations by treatment with phytohemagglutinin has been completed, and further studies of this system by means of fluorescence microscopy have been begun.

An experimental "package" in which human blood samples will be irradiated with P^{32} beta rays during the weightless phase of the Gemini-III manned space flight has been designed, fabricated, tested, and delivered

to the NASA Manned Spaceflight Center. The experiment has received NASA approval for the flight. Further testing, both physical and biological, of individual components of the device has been completed.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Basic Instrumentation. — The application of field-effect transistors in charge- and voltage-sensitive preamplifiers was continued. Optimization of a charge-sensitive preamplifier with a field-effect transistor in the input stage produced a minimum noise line width of 1.5 kev full width at half maximum referred to silicon (zero detector capacitance) and a slope of 0.05 kev/picofarad. The RC filter time-constant was 4.8 μsec, and the field-effect temperature was approximately 123°K. A wide-band voltage-sensitive preamplifier with a field-effect transistor as the input stage was developed. The preamplifier voltage gain was 22, which was flat from 60 cps to 10 Mc; the input resistance was 20 megohms shunted by 9.5 picofarads capacitance; the output resistance was 100 ohms; and the equivalent noise voltage was 6.55 nanovolts/$cps^{1/2}$.

Lithium-drifted germanium gamma detectors were produced. At 77°K, typical resolutions for 1.33-Mev gamma energies from Co^{60} were 6.5 to 8 kev full width at half maximum for depleted volumes of about 160 mm^3 and with a leakage current of approximately 10^{-11} amp.

ISOTOPE DEVELOPMENT

Analysis of Gamma-Ray Nuclides by Electronic Spectra Resolution. — The number of spectra in the standard response-function library has been increased and now includes the spectra of 43 radionuclides. A computer program for the recall of standard spectra on paper tape or in graphical form has proved satisfactory. Reliability testing of the standard spectra is now under way.

The absolute standardization techniques of this program were utilized in assaying an intercomparison sample of Cs^{137} distributed by the International Atomic Energy Agency. In addition to the IAEA, nine laboratories in the United States participated in the evaluation of the intercomparison sample. Values for the specific activity of the solution ranged from 8.58 to 10.3 μc/g in a total of 16 determinations, of which seven "absolute" determinations gave a mean of 9.02 ± 0.07 μc/g; three independent methods used at this laboratory yielded a mean value of 8.98 ± 0.07 μc/g. The results of this intercomparison have helped clarify the decay-scheme parameters for this important radionuclide.

CIVILIAN APPLICATIONS OF NUCLEAR EXPLOSIVES

Chemical Applications of Nuclear Explosions. — In laboratory development work for Project Coach, leaching experiments with the most recent sample obtained from the Gnone cavity show that the acid-leaching properties of the water-insoluble residue differ markedly from those of previous samples. Treatment with 30 ml of 1 N HCl per gram of the water-insoluble

residue at room temperature for 4 hr leached only 20 to 40% of total-rare-earth beta activity, plutonium, or americium, whereas with previous samples, 75 to 90% of these activities were leached under the same conditions. Refluxing with 6 N HNO_3 or 6 N HCl (15 ml per gram of water-insoluble residues) for 4 hr leached about 90% of the above-mentioned radioisotopes. Complete leaching of these radioisotopes was achieved only when the iron in the water-insoluble residue was completely solubilized. Petrographic analysis showed the residue to contain Fe_3O_4, $CaSO_4$, quartz, and olivine ($MgFeSiO_4$) as principal constituents: 10% Fe, 20% $CaSO_4$, and the balance presumably quartz and silicates.

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