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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MAY 1963

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OAK RIDGE NATIONAL LABORATORY

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

MAY 1963

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

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROGRAM

GCR-ORR Loop No. 1. — The roughened-surface element that failed during operation in loop No. 1, as reported last month (ORNL-3451, p 4), was removed from the loop without difficulty. Observation of the element in the ORR hot cell revealed a narrow crack about 2 in. long near the vertical center line on the side of the element that was facing the reactor core center. Local melting had occurred in the copper braze that attached the 0.0063-in. stainless steel wire to the cladding surface, indicating that the surface temperature had been in excess of 2100°F. It appeared that the element bowed toward the reactor from asymmetrical cooling during the experiment, which was being conducted at low helium flow.

GCR-ORR Loop No. 2. — Operation of loop No. 2 continued without incident to the end of the reactor cycle. The low-power flux-calibration test assembly was removed to the segmenting cell and cut up. The cold finger was installed, and operation with it was satisfactory. The operation of the ORNL-designed regenerative compressors in loops Nos. 1 and 2 continues to be satisfactory.

ORR Poolside Capsule Irradiations. — Irradiation of helium-swept capsules containing 1-1/2-in.-diam spheres consisting of duplex pyrolytic-carbon-coated $(U,Th)C_2$ particles uniformly distributed in a graphite matrix (manufactured by National Carbon Company) was completed. An estimated accumulated burnup of 3.75 at. % of the heavy metal was attained. The particles had a thorium-to-uranium ratio of 0.6. Only barely detectable amounts of long-half-life fission gases were found near the end of the experiment. This indicates an exceptionally retentive fuel body having a ratio of fission-gas release rate to fission-gas birth rate (R/B) of less than 10^{-10}, which could be achieved only if no coated-particle failures had occurred. The capsule was irradiated at a fuel surface temperature of about 1500°F.

A helium-swept capsule containing pyrolytic-carbon-coated fuel particles in graphite inserts in machined graphite spheres was removed from the reactor after a burnup of 11 at. % of the heavy metal. This capsule,

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which was irradiated at a surface temperature of about 1100°F, had an R/B of about 5×10^{-5} for Kr^{88} at the time of removal from the reactor.

A capsule containing a $BeO-UO_2$ fuel mixture was irradiated at an estimated central temperature of 2100°F and was removed after about 2.4% burnup of the heavy metal. A high fission-gas release was observed for this capsule; the R/B for Xe^{133} was about 1×10^{-2}.

Three new capsules containing fueled-graphite spheres manufactured by various commercial vendors were installed in the poolside facility and are being irradiated. Each of these capsules is operating at an average power density of about 25 w/g for the fueled spheres, with surface temperatures of about 1500°F. One capsule containing fuel manufactured by the Westinghouse Corporation for the EGCR was removed, and two similar capsules were installed in the facility for irradiation.

Eight-Ball Experiment. — The first experimental assembly containing eight 1-1/2-in.-diam spheres consisting of coated-particle fuel in a graphite matrix was installed in the F-1 lattice position of the ORR on May 11. The design operating temperature of 1100°F in the graphite adjacent to the central fuel balls was achieved with the predicted $He-N_2$ gas mixture, which serves as the thermal barrier in the outer annulus of the assembly.

Compatibility of Fuel Cladding with Coolants. — Tests were initiated for determining the location of carbon absorbed by beryllium during exposure to carbon dioxide at elevated temperatures. Barium carbonate in which 5% of the carbon atoms were C^{14} was decomposed by heating to obtain the desired pressure of carbon dioxide, and a limited number of tests were completed at $C^{14}O_2$ pressures of 200 mm Hg at temperatures between 593 and 815°C for times up to 500 hr. The specimens were examined by metallographic, autoradiographic, and surface-counting techniques. The reaction products of beryllium with carbon dioxide were BeO and Be_2C. The reaction proceeded along preferred longitudinal directions and seemed to be influenced by the voids present in the as-received material. Three possible mechanisms were proposed to account for the greater rate in the longitudinal direction; the mechanisms were based on (1) an excess of vacancies in the oxide film from the voids, (2) segregation of impurity atoms around the voids, and (3) inability of the material to deform plastically in certain directions, because of preferred orientations of the grains. Although the beryllium specimens increased in carbon content during exposure to carbon dioxide, the carbon was found to remain associated with the reaction product; it did not diffuse into the beryllium.

Radiographic Examination of Fueled Graphite. — Several experiments were carried out for evaluation of radiography as a means of detecting internal discontinuities in fueled-graphite spheres. The sources used were Co^{60}, Ir^{192}, and x rays from 30 to 300 kv. One of the three 1.5-in.-diam fueled spheres radiographed had been broken into two pieces, which were fitted back together as closely as possible. Holes were drilled into the other two spheres. The spheres were then radiographed from three directions: through the poles, through the equator, and at 45° to the poles. The results obtained with 7.5 curies of Co^{60} at a 26-in. source-film distance and Eastman AA film indicate that elongated voids as small as 0.042 in. in diameter can be detected in the 1.5-in.-diam spheres with the technique described.

Long-Rod-Cluster Pressure-Drop Experiment. — A series of experiments was run for determining the Blasius friction factor of long, seven-rod clusters in circular channels, for rods with various surface roughnesses. The clusters had the same cross-sectional characteristics as the EGCR cluster but were much longer (52-in. unobstructed length). The system used two clusters in series; the three surface conditions studied were smooth rods, rods with No. 24 Whitworth thread having a root diameter of 0.850 in., and rods wrapped with 0.006-in. wire at a spacing-to-wire diameter ratio of 16:1. The friction factors were determined over the range of Reynolds moduli from 1.5×10^4 to 1×10^5.

The results were in good agreement with those of previous studies and were internally consistent. There was a consistent difference between the two clusters in the values of the friction factor. This was expected and was due to differences in inlet conditions at the entrances to the two clusters (the entrance to the first cluster was a section of long straight pipe, and the first cluster formed the entrance to the second cluster). In order to correct for any effects resulting from differences in the geometry of the two clusters, they have been reversed, and a short series of tests will be made with them in the reversed positions.

REACTOR FUELS AND MATERIALS DEVELOPMENT

Nondestructive Test Development. — Mathematical studies of the solution of eddy-current coil impedance are being made by relaxation methods. The relaxation method consists in satisfying the differential equations and boundary conditions for a problem, as outlined in the following steps. First, a lattice or "mesh of points" is imposed on the problem. The values of potential at each point in the lattice are averaged with a formula derived from a differential equation. The potential at each point throughout the mesh is averaged with the potential at its neighboring points a number of times until the value at each point becomes constant. The potentials associated with the eddy-current coil are complex (real and imaginary parts) and require a complex relaxation. In order to speed the process, an over-relaxation technique is used. The amount of correction of the potential at a point is determined by the averaging formula and then multiplied by a relaxation constant before the correction is added. The relaxation constant is empirically determined for a particular problem.

Too small a constant will cause the solution to converge slowly, requiring a large number of iterations (one complete averaging of the entire mesh). Too large a constant will cause the solution to oscillate and not converge at all. The system converged quickly when a constant of 1.6 was used for the condition in which the coil was in air and not near a conductor. The relaxation constant decreases from this value with the coil on a metal and with increasing conductivity.

Special cases have been run on the IBM 7090 computer to test the accuracy of the solution, to test the rapidity of convergence of the solution, and to determine the best relaxation constants. When satisfactory results are obtained, the impedances of coils of various geometries will be calculated for various operating frequencies. The results of this

study should allow custom design of optimum test coils for particular tests, and should be a major step toward reducing coil design to a science rather than an art.

Mechanical Properties. — A study of the behavior of Inconel at 1500°F under several realistic dynamic-stress states was completed. Its purpose was to evaluate critically the criteria used to solve low-cycle fatigue problems and to demonstrate their applicability to the more complex situations.

The results of these series of tests have been correlated with the results of previous static- and dynamic-stress tests. This investigation has furnished a general approximation for solving complex creep problems involving both static- and dynamic-stress states. The general solution is based on the developed relationship

$$t_r = (B/\bar{\sigma})^{\beta}(\bar{\sigma}/\sigma_1) ,$$

where

t_r = time to rupture,

$\bar{\sigma}$ = effective stress,

σ_1 = maximum principal stress,

B, β = constants,

and on Minor's damage-accumulation hypothesis. The damage accumulation, however, was shown to be independently additive in each principal stress axis.

Materials Compatibility Studies. — The calculated free energies of formation for dilute oxygen-potassium solutions and oxygen-niobium solutions indicate that oxygen should be gettered from potassium by niobium. However, experiments in which niobium samples were in contact with liquid potassium at 815°C showed that, when the initial oxygen concentration in the niobium varied from 360 to 1500 ppm and the oxygen in the potassium was 125 to 1225 ppm, oxygen was redistributed from the niobium to the potassium. The reaction for this redistribution can be described by

$$Nb(\underline{O})_s + K(\underline{O}^*)_\ell \xrightleftharpoons{815\,^\circ C} Nb(\underline{O}^*)_s + K(\underline{O})_\ell ,$$

where

$(\underline{O})$ = a relatively large oxygen concentration in solution,

$(\underline{O}^*)$ = a relatively small concentration in solution.

These experiments indicated that, for the Nb-O-K system,

$$\kappa_{815°C} = C_{O \text{ in Nb}}/C_{O \text{ in K}} \cong 0.2 ,$$

where

κ = coefficient for distribution of oxygen between the refractory metal and the alkali metal,

C = oxygen concentration.

Two recent experiments were conducted to study further the equilibrium suggested by the reaction above. In one test, finely divided solid K_2O was placed in contact with a large quantity of niobium foil in a niobium tube and thermally equilibrated at 800°C for 50 hr. After equilibration, it was found that the K_2O had been reduced to potassium metal, probably according to

$$K_2O_\ell + Nb(\underline{O}^*)_s \xrightleftharpoons{800°C} 2K(\underline{O})_\ell + Nb(\underline{O})_s .$$

The reaction must proceed strongly to the right at 800°C; this is in agreement with predictions from thermodynamic data.

The second test consisted in heating a niobium specimen, previously oxidized until there was a layer of niobium oxide on the surface, with an excess of potassium at 800°C for 100 hr. Comparison of x-ray data taken before and after test showed the complete absence of any niobium oxide after test. The following reaction at the oxide-potassium interface may be postulated to describe the results of this test:

$$NbO_{2_s} + K(\underline{O}^*)_\ell \xrightleftharpoons{800°C} Nb(\underline{O}^*)_s + K(\underline{O})_\ell .$$

Fuel Element Development. — Interim development of the fabrication of uranium monocarbide with the volatile sintering-temperature depressant UAl_2 has netted great improvement in results. The use of the general technique described previously (ORNL-3368, p 8), but with close control of charge-material chemistry and powder-handling operations to avoid contaminant pickup, has resulted in high-quality pellets of 97% of theoretical density and higher at the low sintering range 1350 to 1400°C. The product was virtually free of secondary constituent, and 97% or more of the original aluminum (from UAl_2) was removed during the vacuum sintering. Such results were repeatedly obtained in consecutive experiments involving 1/2-in.-diam pellets weighing up to 13 g and several different batches of starting material, indicating the consistency of the process.

Preliminary evaluation of the method for production resulted in the fabrication of twenty-four 5/16-in.-diam pellets (~110 g total) in a single run. Density was uniformly in excess of 95% of theoretical, and quality was high. The pellets were unusually free of "hour-glassing," and edges were sharp. No pellets showed any physical defects. It is believed that the process is capable of even substantially higher scaleup with densities in excess of 97% of theoretical.

REACTOR COMPONENTS DEVELOPMENT

Reactor Controls Development. — The new PCP-II ionization chamber series [ORNL-3191, p 70 (1962)] was extended by constructing gamma-compensated neutron-sensitive sections. The parallel circular plates, made of nickel 0.012 cm thick and spaced 0.1 cm apart in N_2 gas at 800 torr, are arranged to form alternate sections, uncoated and coated with B^{10} (0.35 mg/cm^2). In the experiments described here, the test section had 16 signal plates, 9 positive high-voltage plates, and 8 negative high-voltage plates. The uncompensated sensitivities were 4×10^{-15} amp/nv and 1.3×10^{-12} amp-hr/r.

The compensation can be changed by varying the ratio of the positive high-voltage polarizing potential to the negative high-voltage polarizing potential. The signal plates are slightly smaller than the high-voltage plates, so the change in polarizing-potential ratio creates a slight radial distortion of the electric field, and therefore the effective volume, near the edges of the plates. This technique has the advantage that both the neutron and gamma sections are well saturated at all polarizing voltages ordinarily used, so that the electrical compensation is not obtained at the expense of saturation. This results in a measured compensation factor independent of gamma intensity over at least four decades. In the test chamber installed in the BSR, with +300 v applied, changing the negative high voltage from −300 to −200 v changed the compensation from 2.4% overcompensated to 0.6% undercompensated.

The accuracy of manufacture permits attainment of compensation of better than 3%.

Tests in the BSR with both "cool" and "hot" cores (as regards fission product gamma-ray intensity, and therefore energy) showed that the compensation of the test chamber, located in water at any point 15 to 40 cm from the core surface, varied less than 0.5% over a range of more than three decades. The signal from the compensated chamber followed the power of the BSR from source to full power — a range of 6-1/2 decades.

POWER REACTOR FUEL PROCESSING

Dissolution of PuO_2 Pellets. — Only 83% of a sintered PuO_2 pellet having a density 90% of theoretical was dissolved by refluxing for 86 hr in 14 M HNO_3–0.05 M HF. There was no appreciable variation of the apparent surface area during the dissolution. The plutonium dissolution rate was about 1% per hour and was constant throughout the dissolution.

Chloride Volatility–Solvent Extraction of HTGR Fuel. — Samples of High-Temperature Graphite Reactor fuel (7% Th–1.5% U–91.5% C) weighing about 30 g each were burned in oxygen at 800°C in 4 hr; the ash was chlorinated in 15% CCl_4–N_2 vapor at 500°C in 1 to 2 hr, and the nonvolatile chlorides were completely dissolved in cold water. The product solutions were evaporated to more than 500 g of thorium per liter and more than 100 g of uranium per liter and were made 1 M in H_2O_2 at 80°C to oxidize slightly soluble UCl_4 to the more soluble UO_2Cl_2, to remove free chloride, and to lower the freezing point to below room temperature.

Various methods of recovering uranium from the product solution by solvent extraction are being investigated. Over 99.9% of the uranium was extracted, using a volume of 30% TBP–Amsco equal to the volume of aqueous solution, in five batch extractions when the aqueous solution was made 1 M in HNO_3; seven batch extractions were required to achieve 99.9% uranium recovery when no nitrate was added. In both cases 92 to 96% of the thorium remained in the aqueous phase.

Physical Properties of $ThOCl_2$–Azeotropic HCl Solutions. – Thorium oxychloride was found to be miscible in all proportions with azeotropic HCl. The melting points of the solutions increased with increasing $ThOCl_2$ content and were found to be 35, 60, and about 140°C at 4, 5, and 8 M $ThOCl_2$ respectively. The melting- and boiling-point curves meet at approximately 8 M $ThOCl_2$. The melting point of azeotropic 5 M $ThOCl_2$–HCl solution was lowered from 60°C to –20°C by adding 1.5 moles of H_2O_2 per mole of thorium at 80°C. The H_2O_2 reacted with chloride to form chlorine, which volatilized out of the solution. After the addition of the H_2O_2, the solution was still stable at temperatures as high as the boiling point (over 100°C) and could be boiled down to any desired concentration. The solution began to appear viscous at 25°C if boiled down to more than 5 M in thorium.

Flowsheet Development for UC Fuel. – Laboratory development of an aqueous-process flowsheet for sheared UC reactor fuel was continued. Unirradiated UC (200 g, 5.2% C) containing UC_2 as an impurity was hydrolyzed in 300 ml of water at 90°C. It took 8 hr for complete hydrolysis, as evidenced by cessation of gas evolution. After cooling, 300 ml of 15.8 M HNO_3 containing Pu(IV) tracer was added to dissolve the UO_2 slurry in 2 hr. A wine-red solution was produced, and it contained traces of yellow organic solids.

The raw feed solution was treated with 0.04 M $KMnO_4$ for 4 hr at reflux. The MnO_2 precipitate was removed by filtration; the filtrate had the characteristic yellow uranyl nitrate color, a qualitative indication that the organic acids had been destroyed. The precipitate was washed three times with 50-ml portions of 4 M HNO_3. Analysis showed that, before washing, the precipitate retained 0.03 and 0.64% of the total uranium and plutonium respectively. Repeated washing reduced these losses to 0.028 and 0.13% respectively; however, analysis indicated that additional or more efficient washing would have recovered more of the plutonium. The filtered feed solution was treated with 0.1 M $NaNO_2$ for 1 hr at 40°C to reduce Pu(VI) to Pu(IV).

The feed was then processed batch-countercurrently through four volumetric throughputs of the Purex first cycle of solvent extraction.

Solvent extraction performance was satisfactory, and no emulsions or interfacial cruds were observed. Uranium recovery was quantitative within the limits of analytical detection (fluorometric). The maximum uranium loss occurred during stripping (0.3 μg/ml after seven stages). Plutonium extraction loss was 0.05% at the seventh extraction stage; however, the stage data show that plutonium was still being extracted at this point. Extraction coefficients for the last four stages suggest the presence of Pu(VI). Plutonium partition loss was nil after seven partition stages; no plutonium could be detected by pulse-counting the uranium product stream.

The use of impure UC in this experiment represents a "worst-case" condition for the processing of UC fuel, since the actual reactor fuel is expected to be of much higher purity. The results indicate, however, that even such impure fuel as that in these tests can be processed satisfactorily.

Solvent Extraction. — An unidentified substance in slightly degraded di-sec-butylphenyl phosphonate (DSBPP) in diethylbenzene (DEB) caused inaccurate analyses for uranium. Uranium analytical data obtained in the countercurrent stripping of uranium from 2.5% DSBPP in DEB containing 2.5 g of uranium per liter, after the uranium-loaded solvent was stored for four days, were in error by a factor of at least 2 for all except the first stage.

The coefficient for the distribution of protactinium tracer in 30% tributyl phosphate in n-dodecane decreased from 4.72 ± 0.46, with (0.49 ± 0.06)% unextractable, in 5 N HNO_3 to 0.78 ± 0.05, with (66 ± 15)% unextractable, in 5 N HNO_3 containing 0.01 N HF.

Laboratory countercurrent batch-extraction experiments indicated that rongalite ($CH_2HOSONa \cdot 2H_2O$) is more efficient than ferrous sulfamate or uranium(IV) for selective stripping of plutonium from uranium in 30% TBP in n-dodecane. Experimental conditions were as follows:

Feed (1 vol):	100 g of U per liter, 6×10^6 counts min^{-1} ml^{-1} of Pu, 30% TBP in n-dodecane
Strip (0.083 vol):	0.06 M ferrous sulfamate, 0.05 M HNO_3
or	0.03 M U(IV), 0.1 M HNO_3
or	0.066 M rongalite, 1.0 M HNO_3
Scrub (0.1 vol):	30% TBP in n-dodecane
Stages:	Six strip, six scrub

The rongalite reduced the amount of plutonium in the uranium by a factor of 8000, whereas the ferrous sulfamate and uranium(IV) reduced the amount of plutonium by factors of 200 and 15 respectively. Reduction of plutonium with rongalite results in the liberation of SO_2, which decreases the amounts of solids in the aqueous waste.

Protactinium Adsorption. — The amount of protactinium adsorbed in 24-hr contacts with 10 g of unfired Vycor glass per liter of solution containing 2×10^6 counts min^{-1} ml^{-1} of Pa^{233} and 10 M HNO_3 ranged from 97.8 to 98.3% for three different batches of glass.

Shear-Leach Process. — Bench-scale leaching tests with 1/2-in. sheared lengths of stainless-steel-clad ThO_2-UO_2 unirradiated fuel in a 4-in.-diam cylindrical dissolver showed that, for a composite product containing about 125 g of thorium per liter and 9 M HNO_3, virtually the same time (about 12 hr) was required to achieve 99.9% dissolution whether the dissolvent was used in a single batch or in two successive half-batches.

Heat transfer tests made on the steam-jacketed leacher, using 40 psi steam pressure, showed that the boilup rate for the product solution rose from 0.09 to 0.14 lb/min as the air sparge, used to suspend ThO_2-UO_2 fines,

was increased from 0 to 0.06 scfm. Increasing the sparge rate up to 0.19 scfm produced no further increase in boilup rate but did increase the foaming.

Fluoride Volatility Processing: Aluminum Alloy Flowsheet Development. — According to visually determined liquidus data, molten $KF-ZrF_4$ mixtures can dissolve sufficient AlF_3 to be useful as the solvent in volatility processing of aluminum-alloy or aluminum-clad fuel elements. At 600°C, the maximum acceptable temperature for the process, a solvent containing 63 mole % KF dissolved up to 15 mole % AlF_3. Also, the addition of KF to this mixture permitted the dissolution of a substantial increment of AlF_3 without exceeding the 600°C temperature limit. The limit would not permit the use of more KF in the initial solvent. Recent results, obtained in a continuing investigation of $KF-ZrF_4-AlF_3$ phase equilibria, indicate that a single addition of 15 mole % KF during dissolution increased the solubility of AlF_3 from 15 mole % to 26 mole %.

Two immiscible liquids were found in the $KF-AlF_3$ binary system above 53 mole % AlF_3 at 980°C. The two-liquid region apparently extends into the $KF-ZrF_4-AlF_3$ ternary system but not to compositions of present interest as solvents in volatility processing.

Laboratory dissolution studies further indicate the apparent feasibility of the $KF-ZrF_4$ system for aluminum-based fuels. The dissolution of aluminum in $KF-ZrF_4$ salt at 600°C by hydrofluorination proceeded much more rapidly at 37 mole % ZrF_4 than it had in previous work at 30 mole % ZrF_4. Use of 70-30 mole % $KF-ZrF_4$ salt with 5 mole % AlF_3 was reported to give dissolution rates of 10 mils/hr, whereas in recent work with 63-37 mole % $KF-ZrF_4$ and no AlF_3 present, dissolution rates of 40 to 50 mils/hr were achieved. Although it is possible that the absence of AlF_3 promotes fast hydrofluorination, the more probable explanation is that the ZrF_4 reduction accounts for part of the aluminum dissolution. A black material or sludge is always formed, probably colloidal zirconium metal. This sludge sometimes adheres to the dissolving aluminum; at other times it disperses in the salt and in some cases leads to difficulty in passage of the HF through the salt. This problem does not appear serious, however, since several complete process dissolution tests have been successfully made, ending at about 15 mole % AlF_3, the approximate concentration limit imposed by the $KF-ZrF_4-AlF_3$ phase diagram in a single step. Also, tests have demonstrated that fluorination of the salt at 550°C results in volatilization of more than 99.9% of the uranium (initial fuel, 3.6% uranium in aluminum).

Fluoride Volatility Processing: Retention of TeF_6. — More laboratory tests were made to determine the absorption of TeF_6-F_2 gas mixtures in aqueous 10% KOH solution. By use of two consecutive packed columns 1 in. in diameter and 20 in. long, with countercurrent flow, TeF_6 absorption efficiencies of 46, 68, and 67% were obtained at 25, 50, and 70°C, respectively, for both columns together. In contrast, more than 99% of the fluorine was trapped in the first column alone at all three temperatures. Tellurium hexafluoride thus appears to be somewhat more stable than fluorine to hydrolysis with aqueous caustic solution.

Fluoride Volatility Pilot Plant. — Five complete runs were made on Zr-U alloy after a 30-week shutdown. Cold runs TU-13, -14, and -15 were

made to study equipment performance and to study selective desorption for improving product quality. Hot runs R-7 and -8 were made to study behavior of, and uranium decontamination from, fission products in 13- and 6-month-decayed alloy.

Cold-run dissolution rates ranged from 2.0 to 2.9 kg/hr (based on 90% completion to minimize tail-out), with HF utilization efficiencies of 24 to 34% per pass. Hot-run dissolution rates (3.4 and 3.7 kg/hr) and HF utilizations (41 and 49%) were higher, but perhaps not as a result of fuel burnup.

Uranium was separated as UF_6 in all runs by reaction with F_2 at 500°C. Average F_2 utilization efficiency was about 5% during cold runs, and 14 and 7% during runs R-7 and -8. Nonrecoverable uranium losses, mainly in the waste salt, were < 0.46%.

In all runs, MoF_6 was selectively desorbed from the UF_6 at 150°C and collected (with milligram amounts of uranium) on a chemical trap. The product was not improved in cold runs; up to 2000 ppm molybdenum collected in the product. This was attributed to HF dilution of the product (up to 30 wt %) and not to ineffective desorption. With the R-7 product, which was not contaminated with HF, only 9 ppm molybdenum collected — the lowest in the Zr-U alloy program (~1/20 of the AEC-specified maximum).

Total cation impurities in the cold-run products were ~3200 ppm, or ten times the AEC specifications (because of HF co-contamination). Cation specifications were met in run R-7 on 13-month-decayed alloy (< 171 ppm). In this run, Tc^{99} and Np^{237} were removed from the UF_6 to < 1 and 4 ppm, respectively, in the product by complexing with MgF_2 at 100°C. At least 200 g of uranium was retained on the bed by adsorption and deposition.

In run R-7, on 13-month-decayed alloy, overall (including dissolution and product recovery) decontamination factors (DF's) ranged from $> 10^5$ to $> 10^9$ for volatile Sb^{125}, I^{131}, Te, Ru^{106}, and Nb^{95} and from $> 10^8$ to $> 10^9$ for nonvolatile Zr^{95}, Sr^{90}, and Cs^{137}. The DF is the ratio of fission product activity in the fuel to that in the product. Specific fission product activities in the fuel were evaluated from reactor data, with the fuel burnup calculated by the CRUNCH code on the IBM 7090. Activities in the product were measured; when the activity was below the limit of detection, the limit was used for the DF calculation. All the dissolution DF's were small compared with the recovery DF's. Dissolution DF's for volatile fission products ranged from 10 for I^{131} to 750 for Ru^{106}; for Sr^{90} and Cs^{137} the dissolution DF was negligible (DF of 2 or 3).

Considerable I^{131} (~80%) and Nb^{95} (~97%) remained in the salt during fluorination. About 85% of the Ru^{106} and 95% of the tellurium in the feed salt were separated with the UF_6 during fluorination.

The UF_6 product from run R-7 met the AEC gamma specification and (within the limit of error) the beta specification. In all cases, specific fission product activities in the product were at or below the lower limit of detection.

THORIUM UTILIZATION PROGRAM

Planning and Evaluation Studies. — The nuclear performance of the High-Temperature Graphite Reactor, which utilizes BeO instead of graphite

in the spines of the fuel elements, was investigated by using calculation methods described in previous reports. The results indicate a maximum conversion ratio of 0.97 at a C/Th ratio of 130 and a Be/Th ratio of 43. This is 0.03 unit higher than that achieved in the best graphite-spine case, which occurred at a C/Th ratio of 200. Although 0.09 neutron per fissile absorption was produced by the (n,2n) reaction in beryllium, increased fission product losses and (n,α) absorptions reduced the gain to the given figure. However, the fissile inventory was reduced from 1144 kg with graphite spines to 1012 kg with BeO spines. This decreased inventory plus decreased net burnup costs resulted in a fuel-cost saving of 0.05 mill/kwhr (electrical). Incremental inventory charges and processing costs for beryllium were not estimated.

Testing of Thoria-Pellet and -Suspension Fuels. — Yield-stress data obtained on aqueous suspensions of thoria-urania recovered from the drain tank of the first in-pile slurry loop (L-2-27S) have been extrapolated as a function of crystallite size (or surface area) to permit estimation of values which might be anticipated at realistic reactor-blanket doses. At a dose of 2.5×10^{18} fissions per gram of solids, a flocculation index, τ_y/ϕ^3, of approximately 2000 is obtained, where τ_y is yield stress, in lb_f/ft^2, and ϕ is the volume fraction of solids.

Correlations imply that such a material at a concentration of 500 g of Th per liter would be in turbulent flow at velocities above 7 to 10 fps.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — The first MSR components were delivered to Building 7503. The reactor assembly jigs were set up, and the installation of components on the jigs was started.

Installation of auxiliary piping in the reactor cell, drain-tank cell, and coolant cell continued.

Procurement of all material and components for the MSRE continues as scheduled. Most procurement will be completed by July. The delivery of the MSRE graphite is expected by October.

The overall project completion as of May 1, 1963, was 71.8%.

Metallurgy. — Fabrication variables were investigated for the making of thin-walled cylinders of Gd_2O_3-Al_2O_3 mixtures of interest as control-rod elements for the MSRE. Shrinkage characteristics and bulk-density changes were determined as functions of green density, and tests were conducted to better understand the sintering characteristics of this system. The effects of varying time and temperature during the sintering of cylinders made with "prereacted" powders were studied in an attempt to resolve the distortion problem encountered in normal fabricating procedures.

Mixtures of Gd_2O_3-Al_2O_3 containing 30 wt % Al_2O_3 were successfully cold-pressed and sintered into cylinders with 95% of calculated density. Shrinkage of the mixed powders caused severe distortion, apparently due to the formation of a perovskite-type phase, $GdAlO_3$. Prereacting the powders to form this compound before sintering and the use of a prereacted Gd_2O_3-Al_2O_3 holding fixture were found to resolve the distortion problem. Mixtures with 20% Al_2O_3 resulted in less distortion when sintered at higher temperature.

Sample control-rod elements were tested in the control-rod test apparatus by thermal and mechanical stressing for 600 hr and approximately 11,000 cycles from room temperature to 1400°F. Hot-pressed Gd_2O_3-Al_2O_3 cylinders cracked under this treatment, but there was no resultant crumbling of the ceramic or distortion of the metal container.

Chemical Development. — The LiF-BeF_2-ZrF_4 ternary phase diagram contains a thoroughly investigated composition region around 2/3 mole fraction LiF (with low ZrF_4 content) that can be considered as the low-melting solvent for UF_4 in MSRE-type fuel mixtures. In further explorations of some of the high-melting regions that are of little reactor interest and that are at low LiF concentrations (10% or less) in this diagram, a region of liquid-liquid immiscibility was encountered. Although enough is known to provide assurance that the phenomenon is of no practical consequence for MSRE-type melts, the extent of the two-liquid region is being delineated as a matter of background information. Repeated fractionation of fuels by distillation could conceivably produce a condensate composition in such a two-liquid region, but this is most unlikely because of the low volatility of the MSRE fuel, for which the vapor pressure is 0.02 mm Hg at operating conditions (bp = ~1400°C).

Preparation of MSRE Materials. — Approximately 16,000 lb of Li^7F was received from the Y-12 Plant for use in the preparation of fluoride mixtures for the MSRE. For quality-control purposes, this material was classified into 66 lots; analyses for impurities and Li^7 assay were obtained on each lot. An additional 4000 lb of Li^7F will be prepared from Li^7OH to meet anticipated requirements for scheduled reactor operations.

Radiation Effects. — Continued refinements of data on the production of CF_4 by in-pile capsules containing MSRE fuel and graphite at operating conditions have confirmed that the yields, when detectable (by mass spectrometry of samples allowed to accumulate in-pile for several days), were generally near limits of detection, precluding a quantitative average of the yields. However, a rate of production of CF_4 corresponding to about 6% of the rate of production of stable xenon appeared to be qualitatively not too inconsistent with much of the data. Assuming that a rough extrapolation from capsules to MSRE performance could be made on the basis of ratios of graphite surface to fuel volume, the implied yield of CF_4 for the MSRE was 30% of the stable-xenon production, or about 6 ml/Mwd (≅10 ppm in the off-gas at MSRE design conditions). Such a rate is favorably low and might again be difficult to measure.

MSR Processing. — The design of the MSRE fuel processing system was reviewed by the operations, development, and metallurgy groups, and comments are being evaluated. The cost of the system was estimated to be $323,000, most of which will be spent in FY 1964. A proposal to the AEC is being prepared.

The Safety Analysis Report was issued. The most serious problem is the release of tellurium during processing. Since TeF_6 hydrolyzes very slowly, the caustic scrubber is not expected to have the required efficiency. Other means, such as reduction to metallic tellurium on nickel mesh, are being considered. Demonstration of at least 90% removal from the off-gas stream will be required before processing a fully irradiated fuel batch.

Analysis of Radioactive MSRE Fuels. — Manipulative testing of the mixer mill and powder-transfer device was completed in the mockup. Also tested was the prototype transport-container decoupling device. The testing showed a need for an additional device. A pulverizer-mixer aligner was developed. Modifications to all the above equipment are approximately 70% complete.

Instrumental checkout of the high-sensitivity coulometer is approximately 50% complete. The statistical evaluation of the spectrophotometric method for the determination of nickel in sulfuric acid solutions of MSRE salt is in progress in the mockup. Other phases of the work necessary for handling and analyzing the MSRE fuel sample in the hot cell are in progress.

An amperometric method for the titration of Cr(VI) with ferrous sulfate was developed for the determination of chromium in MSRE fuels; a titration-cell assembly containing a pyrolytic graphite electrode and a standard calomel electrode was specially designed. Chromium is oxidized to Cr(VI) with silver oxide in 0.5 M H_2SO_4. The Cr(VI) is subsequently reduced to Cr(III) by ferrous sulfate; the resulting decrease in diffusion current is measured electrically. The progress of the titration is followed by means of an ORNL polarograph, model Q-1160, with the pyrolytic graphite electrode at +1.0 v vs the S.C.E. The end point of the titration is a sharp deflection in the curve. When the MSRE fuel is dissolved in sulfuric acid, the titration of chromium can be performed without any prior separations. The precision on nonradioactive samples is 1%, the ultimate precision that can be expected.

ADVANCED REACTOR DEVELOPMENT

Advanced Test Reactor Corrosion Program

A series of tests was conducted with aluminum specimens to determine the rate of corrosion product buildup on the water-cooled surfaces as a function of temperature when the pH of the coolant was 5.7. In all tests the flow rate and heat flux were maintained at 45 fps and 1.5×10^6 Btu hr^{-1} ft^{-2}, respectively, but the coolant temperature was varied from run to run. In all cases the corrosion product formed on the water-cooled surfaces in proportion to the 0.778 power of the exposure time, as has been true at all heat fluxes and pH's. A plot of the logarithm of the proportionality constant vs the reciprocal of the absolute temperature at the specimen-water interface yielded a straight line parallel to, but 4.5 times as great as, that obtained by treatment of the pH 5.0 data in the same manner. In addition, all data obtained from previous runs conducted at pH 6.0 or in deionized water fell, when treated in the same manner, on the pH 5.7 curve, indicating that, when other conditions are constant, there is no difference in the rate of oxide growth in the pH range of 5.7 to deionized water.

High Flux Isotope Reactor Design and Development

Fuel Element Development. — By controlling the fabrication sequence to yield plates of very uniform quality, it has been possible to greatly improve the reproducibility obtainable by low-pressure marforming. It was found that formability could be improved by either decreasing the hot-rolling temperature or increasing the percentage of cold work in the reduction sequence. It was not possible, however, to lower the rolling temperature, since the blister rejection rate then increased. The percentage of cold work was increased from 7.5% to 20%. It should be pointed out that these plates are not curved in the cold-worked condition, but in all cases are completely annealed. The thickness of the final plates was controlled within ±0.0002 in. by grading the hot-rolling schedule to obtain a tolerance at this stage of ±0.005 in. and then reducing the percent cold reduction per mill pass. In a small test run incorporating these changes, 95% of the curved plates were acceptable.

A complete set of fueled plates (depleted) for an inner annulus was fabricated by using the single cold-pressing operation. No cores were rejected for failure to meet the dimensional tolerances, and after roll-cladding to final size, only 3.3% of the plates were rejected for blisters, pits, or dimensions. This rejection rate is much lower than that obtained with the forged cores.

Physics. — Thirty-three-group, one-dimensional fuel-cycle calculations, using a basically improved set of cross sections, were made for an HFIR core containing 9.4 kg of U^{235}. The results indicated that in the radial direction the ratio of maximum to average power density (exclusive of hot-spot considerations) would not exceed 1.12 during a typical core lifetime of about 14 days. As the fuel cycle proceeded, the volume-averaged thermal flux in the plutonium target decreased by about 2% and the peak thermal flux in the beryllium reflector increased by about 50%.

In the above calculations, boron was used as a burnable poison and was located only in the filler piece of the inner-annulus fuel-plate core. With this initial combination of fuel and burnable poison, there were no realistic combinations of depleted burnable poison, fuel, and fission product concentrations for which the reactivity of the core was greater than that at the beginning of the fuel cycle.

These calculations concluded the studies required for specifying the HFIRCE-3 (third HFIR critical experiment) and initial HFIR fuel and burnable-poison loadings and distributions. The critical experiment is tentatively planned to commence on September 1, 1963.

Engineering Development. — Various bearings proposed for use in the HFIR are being tested. Bearing loads, surfaces, and part movements simulate those in the reactor. The tests are conducted in water which closely approximates (except for pressure) the water which will surround the bearings of the HFIR. Measurements and inspections include coefficients of friction, weight changes on bearings and shafts, radial and lateral play, and visual observations. Tests so far on the 1.125-in.-OD Haynes Stellite bearings (New Departure No. 77F8E) indicate good performance. One set of bearings, rolling with a 100-lb load per bearing against a simulated control plate, exhibited friction coefficients of ~0.009. The increase in

radial clearance after 75,000 revolutions was 0.001 in. The same bearings were then deliberately misaligned so that the 100-lb load was applied to the edge of the outer rod, and an additional 75,000 revolutions were accumulated. The coefficients of friction were approximately 0.02, and the radial clearance increased to 0.004 in.

Thirty-five materials of construction are required for the primary pressure vessel, the fuel and reflector, the control-rod drive assemblies, structural members, and experimental facilities in the HFIR. Numerous galvanic couples of metals and nonmetals are involved in both static and dynamic loading. The corrosion program is being continued to test possible materials as the design of components progresses.

The corrosion program has been established to permit the evaluation of most of the materials of construction. No severe corrosion has been found in galvanic couples between stainless steel and 6061-T6 aluminum, provided a stainless steel gasket is used; aluminum gaskets appear adequate for flange connections between beryllium and 6061-T6 aluminum; the special aluminum alloy No. 1 originally proposed for skid pads failed by intergranular attack; no evidence of stress-corrosion cracking has been observed in any of the austenitic, ferritic, or aluminum alloys planned for installation in the reactor.

Electrolyzed aluminum which had been considered for improved wear characteristics failed by spallation of the coating, whereas electrolyzed stainless steel appears satisfactory for threaded stainless-to-stainless connections; the Haynes Stellite 25 ball bearings proposed to replace the aluminum skids show no evidence of attack to date after 2200 hr of exposure.

Particular emphasis is being placed on materials for bearing and track combinations and on seals between the vessel extension and the control-rod drive rods at the bottom of the pressure vessel.

Hazards Report. — The descriptive portion of the hazards report is approximately 50% complete. This includes information and drawings (44) for all systems except the core and the instrument and controls systems; these are to be available by June 7. The site and meteorology portions will be compiled by June 5. The first draft should be complete by the latter part of June.

PHYSICAL RESEARCH PROGRAM

Reactor Operations. – During the first 20 weeks of 1963, the ORR operated 72.5% of the time. This decrease in operating time was caused by an extended shutdown for installation of loop No. 2 during the first quarter. A second miniature fission chamber was placed in service and was connected to the No. 2 count-rate channel. The core loading was altered by replacement of fuel on the north lattice with beryllium so that beam-type experiments could be installed in beam holes HN-1 and HN-4.

The LITR operated 90.9% of the time during the first 20 weeks of 1963. Approximately half the instrument renovation has been completed, along with about one-fourth the cable installation required for moving the remote controls to the ORR.

The ORNL Graphite Reactor operated 93.0% of the time during the first 20 weeks of 1963. Approximately 19,000 irradiated fuel slugs from the ORNL Graphite Reactor were shipped to Savannah River for reprocessing.

A report describing operating limits for the ORR was submitted to the AEC, and a draft of a similar report for the LITR was prepared.

Waste Disposal. – The release of Ru^{106} from the waste pit area, which had reached a peak of more than 600 curies/month during 1961, appears to have leveled off at a rate between 110 and 125 curies/month. A portion of the decrease may be attributed to the fact that the use of the pits for waste disposal was discontinued late in 1962, although a substantial decrease was noted before that time. The covered trenches currently used have shown no significant release of activity.

Hot-Cell Operations. – Operations in the hot cells were normal. Eight GCR capsules containing various UO_2 and UC_2 fuels irradiated in the ORR, the LITR, or the MTR were disassembled and examined. One SM-1 fuel element was examined visually, and crud and film samples were taken for chemical analysis. Disassembly and examination of experimental array ETR-ORNL 41-8, containing about 150 encapsulated BeO pellets, were essentially completed. Specimens were recovered from 14 ORR experiments in the ORR cells. Examination of molten-salt experimental assembly MTR-47-4 was completed, and the study to determine the rate of gas formation in experiment MTR-47-5 at various temperatures continued. Installation of in-cell equipment was started in the High-Radiation-Level Experiment Laboratory, and installation of the in-cell milling machine was completed.

PHYSICS AND MATHEMATICS RESEARCH

Charge Spectrometry. – To check whether shake-off of an outer electron might be greater for positron than for negatron decay, calculations of the probability for exciting the outermost electron were made for several atomic systems. These calculations are based on obtaining overlap integrals of the initial and final states of the outermost electron as given by Hartree-Fock solutions. The probabilities for any p electron

to vacate the outermost shell following beta decay for various atomic systems are:

Decay	% Probability
$Ne \xrightarrow{\beta^-} Na^+$	17.0
$Ne \xrightarrow{\beta^+} F^-$	24.9
$F \xrightarrow{\beta^+} O^-$	27.9
$Kr \xrightarrow{\beta^-} Rb$	14.6
$Kr \xrightarrow{\beta^+} Br^-$	19.8

The results indicate that the amount of electron shake-off from the outermost shell may be greater for positron decay, but the difference is not large.

Neutron Diffraction. – Neutron diffraction measurements were made on alloys of the heavy rare earths with yttrium and with each other in order to study the influence of alloying on the magnetic structure properties of the pure metals. It was found that the Néel temperatures of the pure metals (Curie temperature for gadolinium) and of the rare-earth alloys are proportional to the two-thirds power of $X = \xi(g - 1)^2 J(J + 1)$, where ξ is the concentration of the rare-earth atom for which $(g - 1)^2 J(J + 1)$ is the square of the projection of the spin S on the total angular momentum J. With the exception of concentrated Gd-Y alloys, the magnetic structures of the alloys near the Néel points are characterized by a sinusoidal modulation, the wave number for which can be measured from the diffraction data. To a fair approximation the initial values of the interlayer angles in the pure metals and in the alloys fall on a universal curve in the parameter X cited above. For a small X, $\omega_i = 51°$ per layer, as, for example, in thulium and erbium. As X increases, ω_i decreases smoothly, so that for terbium, $\omega_i = 20.5°$ per layer. To a first approximation both the Néel temperatures of the alloys and the initial values of the turn angles are determined only by the average value per site of the spin squared. As the temperature is reduced below the Néel point, the anisotropy energy becomes important, and the magnetic structures and their transformations depend strongly on the particular constituents of the alloy.

Fast-Chopper Time-of-Flight Spectrometer. – Transmission measurements were made with the 45-m spectrometer for samples enriched in the six hafnium isotopes $Hf^{174,176-180}$. Measurements below 100 ev were made with an energy resolution of 1-1/2% and above 100 ev with a resolution of 60 nanoseconds/m. The data show many new resonances which had not been observed in previous transmission measurements. The data are being analyzed

to obtain the parameters of the resonances. These transmission data will be compared with the capture-cross-section data measured with the RPI Linac to obtain radiation widths for the resonances.

High-Voltage Experimental Program. – Since recent theoretical analysis offers an explanation for the origin of certain types of compound nuclear states, it is important to identify the quantum parameters of resonance states in relatively simple nuclei such as Pb^{209}. Measurements [Bull. Am. Phys. Soc. 8, 82 (1963)] of the differential cross sections for neutron scattering from Pb^{208} together with published total-cross-section data make it possible in principle to assign the quantum parameters for the prominent resonances between 0.715 and 1.761 Mev neutron energy. Since in this energy range there is evidence that phase shifts for partial waves up to $g_{7/2}$ may be significant, a FORTRAN code has been written in order to compute differential cross sections as a function of the eight possible partial-wave phase shifts and to average the theoretical curves over the energy spread of the experimental measurements. For the first several resonances an approximate fit to the experimental data verifies the previous parameter assignments which were made on the basis of qualitative arguments [Bull. Am. Phys. Soc. 8, 82 (1963)]. This code is being incorporated into a search routine to obtain a least-squares fit to the experimental data.

Spectroscopy Research Program. – A brief study has shown that gas laser oscillations may be used to determine the instrumental resolution of infrared spectrophotometers. Monochromatic radiation may be produced at a number of different wavelengths in the infrared, with as many as eight wavelengths simultaneously in oscillation in an rf-excited mixture of He-Ne-Xe. A frequency scan by the spectrophotometer over a monochromatic line provides a precise description of the spectral slit function at that particular wavelength; thus the resolving power may be accurately determined.

Mathematics and Programming Research: ALGOL Compiler. – The Oak Ridge ALGOL compiler written for the CDC 1604 computer has been extensively tested by actual use on routine scientific calculation. Its high speed of compilation – faster than that of any other algebraic language compiler in use in Oak Ridge – makes it ideal for short problems. A programming manual, ORNL-3460, has been written.

The input-output and intermediate tape facilities utilize most of the existing FORTRAN routines. This reduced the total programming effort. The nature of these facilities is such as to require additions to the language, including the declarators format and list. A non-FORTRAN input routine has also been added. It accepts numbers in any ALGOL form, making the necessary type conversions automatically (i.e., without a format declarator indicating field widths and whether type real, integer, or Boolean).

Modifications have been made in the ALGOL Control System to allow unrestricted mixing of ALGOL and CODAP-1 (assembly language) subprograms. Each subprogram to be compiled separately is connected to the calling program through the declarator external, which is an addition to the ALGOL language. These changes give the ALGOL compiler the same precompilation features that FORTRAN has.

Mathematics and Programming Research: Graphical Output. — A new subroutine package to facilitate plotting data from the CDC 1604-A computer has been added to the basic monitor library at the Laboratory's computer. This subroutine package converts machine data into a special format and writes magnetic tapes used to drive a Calcomp digital plotter. The package is one of the fastest of its kind in existence, thus making computer time a negligible factor in its use, and is comprehensive enough to be adaptable to almost all types of graphical applications. Its use in processing experimental data has become one of the standard computer applications.

The advantages of a graphical output are readily seen. The scientist may choose from a graph those values which are important to him, rather than scanning thousands of printed numbers. He may thus verify expected trends or make new discoveries in a much shorter time and, just as important, reduce the time wasted in false channels.

CHEMISTRY RESEARCH

Chemical Engineering Research. — The flow capacity of pulsed columns at a high flow ratio (100:1) of aqueous to organic phase is virtually the same as that at a more conventional ratio of 5:1. For the system 30% tributylphosphate—Amsco vs 4 M $NaNO_3$, the total flow rate in a standard sieve-plate (1/8-in. holes, 23% free area) column, operated with the aqueous phase continuous, increased from 520 to 3100 gal hr^{-1} ft^{-2} as the pulse frequency was decreased from 90 to 35 cpm. The flow capacity of a nozzle-plate (1/8-in. holes, 10% free area) column operated with the organic phase continuous was significantly less, ranging from 190 to 820 gal hr^{-1} ft^{-2} as the pulse frequency was decreased from 70 to 35 cpm.

Solvent Extraction Research. — Large separations between the lanthanide and transplutonium groups were obtained by extraction from chloride-free solutions. The extractant was di(2-ethylhexyl)phosphoric acid in a hydrocarbon diluent with or without a modifier. The aqueous phase was a mixed solution of a sodium salt of ethylenediaminetetraacetic acid, N-hydroxyethylethylenediaminetriacetic acid, or diethylenetriaminepentaacetic acid with any of several common carboxylic acids, particularly glycolic acid. Cerium extracted better than americium by factors up to more than 50, while europium was usually still more extractable. Differences within each group were small. The presence of nitrate did not decrease separations. Distribution coefficients, separation factors, and the order of preference were dependent on the identity and both relative and absolute concentrations of the pair of mixed acids and on the composition of the diluent.

Hydroxamic acids, $R-\overset{\overset{O}{\|}}{C}-\overset{\overset{H}{|}}{N}-OH$, showed potential for extraction of zirconium, niobium, hafnium, and strontium. Strontium was extracted by 0.1 M octanohydroxamic acid in hexone from 0.5 M $NaNO_3$, with a maximum extraction coefficient of $E_a^o = 8$ and a separation factor from sodium of

$SF_{Na}^{Sr} \approx 50$ at pH 10.5. Cesium was not extracted. The available hydroxamic acids decomposed when exposed to nitric acid or alkaline solutions but appeared to be stable in extraction from hydrochloric acid.

It was shown previously that tributyl phosphate (TBP) when used in aromatic rather than aliphatic diluents gave improved performance with regard to radiation stability, uranium extraction power, and separation of uranium from fission products. Similar results have now been obtained with a wider range of aromatic diluents. Solutions of TBP in diethylbenzenes, butylbenzene, or trimethylbenzenes extracted uranium from 2 M HNO_3 with coefficients about 50% higher than those obtained with TBP in Amsco 125-82 solutions. Factors for separation of uranium from hafnium were about 2 times higher.

Process Chemistry of Uranium and Thorium. – In mineral beneficiation studies on Conway granite, about 70% of the thorium was recovered in a heavy mineral separate which comprised 8% of the ore weight. Magnetic fractionation of the ore gave a magnetic product representing about 20% of the ore weight and containing about 60% of the thorium.

Volatility Studies. – The results of several stirred reactor tests have demonstrated that mechanical agitation does not significantly improve the volatilization of PuF_6 from 31-24-45 mole % $LiF-NaF-ZrF_4$ upon fluorination at 600°C. In all cases the half-reaction time value was 3 to 5 hr, or about the same as in unstirred melts. In one 4-hr test where optimum conditions prevailed, the stirrer was the centrifugal-tube type operated at 2200 rpm.

With an alundum reactor and an alundum-fluorine inlet dip tube, a half-reaction time of 10 hr was obtained in another unstirred PuF_6 fused-salt volatilization test. This run also indicated no significant difference from the normal case, since use of about twice the usual amount of salt probably led to the long half-time.

From the results of the stirring tests and the alundum-vessel run, it appears that the slow rate of PuF_6 volatilization is due neither to diffusion-rate-controlling mechanisms nor to back reaction of the oxidized plutonium with the nickel container wall. Tests are now being planned to determine whether a higher fluorine pressure (at the same flow rate) or a change in the salt composition will result in some improvement in the volatilization rate.

Ion Exchange Studies. – An examination was made of effluent-water solutions collected from previously reported flowing-water irradiation experiments of Dowex 50W cation exchange resin. Each liquid volume was reduced by a factor of about 50 in a vacuum distillation unit that maintained the solution at or below room temperature to minimize the possibility of volatilizing or decomposing any organic constituents. Chemical analyses showed that most of the concentrates of each run contained soluble sulfates and sulfonates. Visually, the first concentrates of each run were colorless; remaining concentrates became progressively darker, varying from light straw to dark brown and containing increasing quantities of suspended solids. Ultracentrifuge and light-scattering studies indicated that these suspended solids had a density of about unity and a molecular weight of several million.

One interpretation of the available data on the radiation degradation of this cation exchange resin is as follows. Initially, the only degradation process involves cleavage of active sulfonate groups from the resin. These dissolve, while being partly converted to sulfate, in the flowing-water stream as indicated by the occurrence of maximum acidity in the effluent stream at the beginning of each exposure period. The resin matrix does not start to break up until a substantial fraction of active sulfonate sites is lost. Then, along with the formation of water-soluble radiolysis products, small particles of the matrix, of several millions in molecular weight, begin to break off the beads and enter the effluent stream in some manner that does not lead to fragmentation of the resin beads.

Mechanisms of Separations Processes. – The transpirational vapor-pressure method is being used to obtain the activities of tributyl phosphate (TBP) in two-phase systems of uranyl nitrate, TBP, and water at 25°C. The TBP transpired is being determined by P^{32} beta counting of the labeled phosphate. As the uranium concentration in the organic phase decreases from 1.59 to 1.43 M, the vapor pressure of TBP increases from 0.0044 to 0.055 μ Hg; the corresponding rational activities, based on a vapor pressure of anhydrous TBP of 0.527 μ, increase from 0.0083 to 0.104. The standard deviations of these values have not yet been established.

Analysis of previously reported data shows that the excess partial molar free energy of TBP in water-saturated TBP is +256 cal per mole of TBP. As nitric acid is added to a two-phase system, the excess partial molar free energy of TBP decreases but becomes negative only when the concentration of acid in the organic phase is about 1 M, corresponding to a ratio $(HNO_3)_{org}/(TBP)_{aq}$ of about 0.3. The sign of this excess free energy and its magnitude suggest that there is a weak repulsion between TBP and water molecules; they do not support the concept of formation of a complex such as $TBP \cdot H_2O$.

Thermochemistry and Calorimetry. – The low-temperature heat capacity of $ReCl_3$ was measured from 7 to 310°K. No lambda-type transition was observed over this temperature range. There does appear, however, to be a Schottky-type transition with a heat-capacity maximum at approximately 15°K.

Chemistry of Technetium. – The Raman spectra of aqueous TcO_4^{2-} and of crystalline $KTcO_4$, $KReO_4$, Na_2MoO_4, Na_2WO_4, $Na_2MoO_4 \cdot 2H_2O$, and $Na_2WO_4 \cdot 2H_2O$ were measured. All the salts except $Na_2MoO_4 \cdot 2H_2O$ reveal lattice vibration bands below 200 cm^{-1}. The infrared spectrum of $KReO_4$(cryst) was measured from 600 to 2300 cm^{-1} using the Nujol-mull technique. A complex group of bands centered at 915 cm^{-1} was observed. In addition, a series of bands was observed in the overtone region 1800 to 1900 cm^{-1}, which is readily interpreted as a single overtone in combinations with three lattice vibrations observed in the Raman spectrum. The Raman spectrum of Na_2WO_4(cryst) confirms a deduction given in the literature that the previously unobserved fundamental $\nu_2(E)$ lies quite close to the $\nu_4(F_2)$ fundamental for WO_4^{2-} in T_d symmetry. Examination of all the spectra leads to the conclusion that the structure of TcO_4^-(aq) is tetrahedral in spite

of the fact that only two Raman bands were observed instead of the four expected from T_d symmetry. The two observed bands are accidentally two-fold degenerate. Comparison of the Raman spectra of Na_2WO_4(cryst), $Na_2WO_4 \cdot 2H_2O_4$(cryst), and WO_4^{2-}(aq) shows that bands observed on WO_4^{2-}(aq) are more nearly coincident with those of the dihydrate than with those of the anhydrous salt. This suggests that the WO_4^{2-}(aq) is a distorted tetrahedron, since the ion in $Na_2WO_4 \cdot 2H_2O$ is of lower symmetry than it is in the Na_2WO_4, where the site symmetry permits T_d symmetry for the WO_4^{2-}. The same observation and conclusion are made for the molybdates.

Mechanisms of Radiation Corrosion of Zirconium and Zircaloy-2 and Electrochemistry of High-Temperature Aqueous Corrosion of Metals. — In continued investigations of the electrochemistry of Zircaloy-2 corrosion in oxygenated 0.05 m H_2SO_4, the rates and activation energies of the partial processes (oxidation and reduction) at 293 and 206°C were determined as functions of film thickness starting at 60 min exposure.

At 293°C, complete current-voltage (Tafel) measurements demonstrated that the rate of the anodic partial process decreased continuously with film thickness. The rate of the cathodic partial process also decreased with film thickness for the first 6000 min and then slowly increased upon continued exposure. The rate of the overall corrosion reaction at the rest potential decreased with film thickness as previously reported. A logarithmic rate law was demonstrated for the partial-process rates and for the overall corrosion rate during the initial exposure period. The activation energy measurements were in the range 250 to 293°C. The anodic activation energy was 32.5 ± 2 kcal/mole and was independent of film thickness. That for the cathodic process was 7 ± 2 kcal/mole starting at 300 min. Measurements of the cathodic process at earlier times were unreliable.

At 206°C, the duration of the measurements was 3000 min. The activation energy measurements were in the range 160 to 206°C. The results of the current-voltage and activation energy measurements were the same as those for comparable times at the higher temperature.

Recent determinations of corrosion at very long exposure times have shown that the rate continues to decrease with time. The previously reported conclusion that an apparent steady state existed was based on observations made during shorter periods. The conclusion drawn from these short-period observations is now considered to be invalid.

Chemical Separation of Isotopes. — The Raman spectrum of liquid $XeOF_4$ has been recorded. A square pyramid structure is indicated with the O at the apex of the pyramid. The observed frequencies and their assignments are presented in the following table:

Assignment	Frequency (cm^{-1})	Intensity	Description (ν, stretch; δ, bend)
ν_1	920	20	ν (Xe-O)
ν_2	567	100	ν (Xe-F) symmetrical
ν_3	285	2	δ (F-Xe-F) symmetrical, out of plane
ν_4	527	40	ν (Xe-F) antisymmetrical
ν_5	233	6	δ (F-Xe-F) antisymmetrical, out of plane
ν_8	365	15	δ (F-Xe-O)
ν_9	161	3	δ (F-Xe-F) antisymmetrical, in plane

Chemical Applications of Nuclear Explosions. – Acid leaching of water-insoluble residue from a composite Gnome sample showed that 80% of the plutonium and the total rare earths are leached by 10 ml of 5 N HNO_3 per gram of residue at room temperature (27°C) with 73% dissolution of the residue. Increasing the acid excess to 100 ml of 5 N acid per gram only increased the plutonium and gross beta extraction by about 10% and the dissolution of residue by about 10%. The data indicate that dissolution of the residue is necessary to leach appreciable quantities of activity from the residues. The amount of activity and dissolved residue leached increased nearly linearly when the acid volume was increased from 3 ml to 10 ml per gram, but it increased only 10% for a tenfold increase in acid volume thereafter. This indicated that all readily soluble components are dissolved by 10 ml of acid per gram of residue. Increasing the leaching temperature from room to reflux temperature increased the amount of plutonium and total rare earths leached by 10%. Below about 30 volumes of acid per gram, the samples were difficult to filter. Varying the nitric acid concentration at a constant 30 volumes of acid per gram increased the amount of plutonium and total rare earths leached from 77 to 92% and from 84 to 91%, respectively, when the acid concentration was increased from 1 N to 10 N. Acid consumption was 9 to 13 meq/g.

METALLURGY AND MATERIALS RESEARCH

Ceramics Research: Sintering Studies. – Previous investigations have shown that sintering ThO_2 powder compacts have a temperature-dependent densification end point, in that the density is essentially

constant on reaching a given temperature regardless of heating rate. The temperature-dependent densification end point was observed for heating rates of 1 to 8°C/sec. Additional tests were made by placing the sintering compacts on a similar compact containing a thermocouple and moving the system into a high-temperature furnace at a rate adjusted to give the desired heating rate. Data obtained over an extended range of heating rates indicate that the temperature-dependent densification end point holds to at least heating rates of 100°C/sec. At heating rates less than 1°C/sec the density increases with decreasing heating rate. At low heating rates the contribution of diffusion-controlled processes to the densification becomes appreciable, probably after plastic flow ceases, because of the changes in geometry due to the initial densification.

Physical Properties Studies. – An extended thermal-conductivity determination from 100 to 1350°C on 93.4% dense UO_2 in the improved radial-heat-flow apparatus was completed. This was followed by a reinstrumentation of two of the three measuring planes with new thermocouples and a run to 600°C. These tests have yielded several significant results. First, the thermal-resistance data obtained to 1100°C are linear to ±1%, as expected for pure phonon conduction, and are reproducible to ±0.5%. Second, the data above 1100°C showed as much as a 6% deviation from this linearity at 1350°C, and a permanent decrease of 3 to 4% in the thermal resistance of UO_2 between 900 and 1050°C upon cooling from 1350°C. This behavior had been interpreted as the possible onset of a supplementary high-temperature heat transfer mechanism accompanied by a permanent change in the specimen stoichiometry. However, the run to 600°C showed the data above 1100°C to be suspect because of a permanent decrease in the thermal emf of the Pt vs 90% Pt–10% Rh measuring thermocouples above 1100°C in the system environment. The run to 600°C indicates that the 1350°C measuring treatments did result in a specimen change accompanied by an increase in thermal conductivity. Finally, these tests have demonstrated that the present radial-heat-flow apparatus is operable to 1100°C, and plans to retest specimens to at least this temperature are being pursued. Simultaneously, a short-time, high-temperature environmental thermocouple study is in progress with the goal of establishing a more stable thermocouple for use above 1100°C. The temperature dependence of the electrical resistivity of the 90% Pt–10% Rh core heat was measured, and it appears to be a satisfactory secondary temperature standard for this system.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. – The circuit to the DCX coils was modified to permit shifts of the magnetic median plane by ±1 in. with respect to the geometric median plane in order to determine the effects on the plasma spread at low operating pressures. Variations of this amount resulted in no change in the plasma width or spreading time but did shift the position of the maximum of the plasma profile in the same direction as that of the magnetic median plane shift.

Previous observations of decay times at positions off the median plane indicated progressively shorter decay times with larger Z. From these data it was concluded that this difference was a result of lower-energy particles (50 kev lower average energy) in the plasma off the median plane. Measurements with silicon-barrier energy analyzers at Z = 0, 2-1/2, and 5 in. have confirmed that the escaping neutrals have lower average energies at larger Z values (~50 kev at Z = 5). However, when these measurements are corrected to give the average energy of the trapped particles, the averages are the same (~300 kev) at Z = 0 and Z = 5 in. These results imply a larger energy spread at larger Z.

The cold-plasma loss rates and some features of the energy distributions were measured as a function of deuterium and hydrogen gas pressures from 1×10^{-7} to 1×10^{-4} torr. Almost linear increases of the cold-plasma production rates with pressure were noted, and there were no sharp increases of the rates at the suggested critical pressures. However, some features of the energy distributions changed abruptly at these critical pressures – a result unanticipated and as yet unexplained.

Emphasis during this report period has been placed upon optimizing the parameters that would permit full utilization of proton trapping by Lorentz dissociation. The trapping fraction for H_2^+ obtained from H_3^+ remained at 10^{-4}, independent of water-vapor cell pressure up to a neutral areal density of $4 \times 10^{14}/cm^2$. Because trapping of protons through Lorentz dissociation is independent of pressure and because the average proton lifetime is increased by lowering the base pressure, modifications are being incorporated which will permit lowering the base pressure from 1×10^{-9} torr to perhaps 1×10^{-10} torr. This has necessitated lengthy leak hunting, repairing, and the installation of yet another liner inside the present liner. This new liner will be maintained at slightly lower temperatures (77°K) than those for the usual liner. Studies of the ion-source focal properties and optimization of its parameters for H_3^+ production are also being made. A new water-vapor cell that should permit higher neutral density and hence a higher efficiency for the conversion of H_3^+ to H_2^+ (to the reported maximum of 13%) was installed. The combination of higher conversion efficiency and lower base pressure should permit a hundredfold increase over the maximum number of protons that has previously been trapped via gas dissociation.

Computations are being made for optimization of magnetic-field shape and injection parameters to permit the maximum Lorentz trapping.

High-Current Injection. – The H_2^+ ion current injected into the DCX-2 is still limited to 60 ma, even at a total current of 150 ma. This problem has been investigated with the High-Intensity-Beam Facility. The results, although not complete, are almost identical with those found in the DCX-2. For example, with 100 ma total current and optimized source parameters, current ratios of 60 ma H_2^+, 30 ma H^+, and 10 ma H_3^+ have been obtained. However, when the total current is increased from 100 ma to 150 ma, there is essentially no change in the H_2^+ ion current focused through the 1-5/8-in. aperture. It appears that the injected-current limitation is a decreased relative production of H_2^+. If this proves to be the case, a variety of ion source modifications will be evaluated at total currents above 100 ma.

Except for the H_2^+ current limitation the DCX-2 injector system performed very satisfactorily until May 17, when a small hole developed in one of the porcelain bushings used in the accelerator tube. This hole appears to be identical to two previous holes developed in similar bushings on the DCX-1. The reason for these failures is not understood.

An electron gun has been built for injecting along field lines into the DCX-2. Up to 5 amp of electrons at 3 kv have been produced, injected along field lines, and collected on the other end of the machine.

Theory. — The influence of the shape of the free-electron energy distribution on the steady-state de-excitation radiation resulting from electron collisions with not-completely-stripped impurity ions has been investigated for homogeneous, spherical plasmas. The equation of radiative transfer and that giving the rate of change of the impurity atomic populations are solved, taking into account all radiative and collisional processes involved. As examples, Druyvesteyn and Maxwellian electron distributions of the same mean energy, $\bar{E}$, are compared. When $E_{12}/\bar{E}$ is large (E_{12} being the transition energy between the one impurity-excited level considered and the ground state), details of the high-energy tail of the distributions are emphasized, and large differences in energy loss are found. For $E_{12}/\bar{E} = 5$, the radiation from the Maxwellian distribution is 100 times that from the Druyvesteyn distribution.

In an effort to explain a possible discrepancy between the calculated and observed refractive index in hot electron plasmas, the index of a homogeneous plasma in a magnetic field with $\vec{E}$ parallel to the field has been reexamined. With relativistic kinematics and monoenergetic electrons, the index is found to be that for cold plasmas for electrons up to ~100 kev and only begins to fall significantly at energies around the electron rest mass. Also, temperature anisotropy has only modest effects.

Radio-Frequency Heating. — Measurements have been made of several plasma parameters when metal shot is dropped through the electron cyclotron plasma. A mechanical device drops 1/8-in.-diam lead or stainless steel shot from a hopper over the microwave cavity. Coils wound around the outside of the cavity at the entrance to the mirrors monitor the flux change due to the plasma dumping a large fraction of its energy on the shot. An x-ray detector, a microwave noise radiometer, and other monitors indicate the progress of the shot through the plasma. An x-ray detector viewing the axis of the machine through a narrow collimator indicates the time when the shot crosses the axis by the burst of x rays observed at that time.

Measurements of the emf from the coils monitoring the flux change indicate that between 40 and 50% of the total stored energy of the plasma is removed when the shot passes through the plasma. The microwave power is kept on as the shot drops. It is apparent from the measurements that the plasma is being replaced at a rate somewhat slower than it is removed by the shot. Calculations of the energy removed as a function of time and hence as a function of plasma radius indicate that the plasma energy density is concentrated in the center of the machine in the form of a football or a cylinder with a fairly sharp outer radius of about 10 cm. The x-ray monitors indicate a belt of hot electrons outside this hot

central core which produces copious x rays upon striking the shot, but which contains little stored energy (as monitored by the flux coils). Pinhole x-ray photographs made from the side of the machine tend to confirm the picture of the hot central ball.

BIOLOGY AND MEDICINE PROGRAM

RADIATION EFFECTS

Long-Term Somatic Effects of Radiation: Mathematics and Computation. – Mortality intensity studies on data collected in experiments with RF mice continue. A computer program has been written to compute the mortality intensity by fixing the number of animals dying in an interval and allowing the time to vary accordingly. This method provides estimates of mortality intensity which have good statistical properties. More recently, consideration is being given to the adjustment of this technique so that mortality intensities for different causes of death may be studied in the same way.

Discussions continued on the standardization of record keeping for animals in the somatic-effect study. A coding procedure was agreed upon for a standard animal identification number which will include a complete genealogy as well as all other information specific to each animal. The standardization of coding procedures for this study is extremely important because of the many scores of thousands of animals which will be involved over a long period of time.

A new nonlinear mathematical model expressing survival as a function of radiation dose was developed and applied to grain counts made on cells following the incorporation of H^3-thymidine and H^3-uridine. The model purports to explain the probability of survival in terms of the relative number of colonies which develop after incorporation of known doses of radiation.

RADIATION GENETICS

Mutagen Mechanisms: Bacterial Genetics. – A method has been developed to selectively isolate stable partial diploid strains of *E. coli* K-12. Strains have been isolated which are diploid for various lengths of the genome in the region between the loci for thiamine and tryptophan biosynthesis. The process of ultraviolet-induced genetic recombination has been studied by using a partial diploid strain which is diploid from the locus for arabinose utilization to the locus for proline biosynthesis. This strain divides by the formation of uninucleate cells from binucleate cells. Logarithmic-phase cultures are inactivated exponentially by uv radiation. Both uv-induced haploidization with recombination, and recombination with retention of the diploid state occur by a conservative

mechanism. Recombination is obligatory to obtain haploidization. Recombination with retention of the diploid state is nonreciprocal. Analysis of recombination data indicates that the chromosome of this strain is replicated sequentially from a fixed point of origin which is between the proline and galactose loci. The arabinose-to-proline region of the chromosome is replicated late in this sequential replication cycle. Data obtained from uv-induced recombination in stationary-phase cultures indicate that either the chromosomes are all duplicated or that all the cells are binucleate. Preliminary experiments indicate that the photoreactivable sector for uv-induced recombination is less than 20%.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — Correlative light and electron microscope studies on germinal centers of the mouse splenic white pulp following antigenic stimulation are being conducted. Germinal centers have been controversial subjects revolving around a proliferative versus degenerative role in the production of lymphocytes. Recent evidence suggested that germinal centers may produce antibody-forming plasma cells.

The ultrastructure of the plasma-cell line is characteristically different from the lymphocyte in the hyperplastic development of plasma-cell endoplasmic reticulum, the cytoplasmic system of paired membranes which implicates plasma cells as antibody producers. With this morphological criterion, plasma-cell development can be followed systematically in splenic white pulp with the electron microscope. All stages of development, including mitotic figures, are found in addition to degeneration of plasma cells and their subsequent removal by macrophages. The data indicate that germinal centers are the sites of formation and destruction of plasma cells or their precursors. Whether germinal centers are the sole sites for the initiation of the immune response and whether plasma-cell production is their only role remain to be answered.

Tissue Culture. — The scoring of periodic samples of peripheral leukocytes from men accidentally irradiated in four different radiation accidents is being continued routinely. Studies of the induction of human somatic chromosome aberrations by neutrons and by protons are in progress. Experiments with 14-Mev neutrons and with 130-Mev protons are complete, and preliminary work was completed for irradiations with 2.5-Mev neutrons and with 22- and 450-Mev protons.

The phenomena of endoreduplication and polyploidy in human leukocytes are being investigated. Microspectrophotometric studies have shown that endoreduplicated nucleic contain twice as much DNA as do diploid nuclei. It has also been shown that both endoreduplication and tetraploidy are induced directly by irradiation of leukocytes, apparently by different mechanisms.

MOLECULAR- AND CELLULAR-LEVEL STUDIES

Biophysics. — Comparisons have been made of the effects of ultraviolet irradiation of uv-resistant and uv-sensitive cells of *E. coli* on the numbers of dimers produced, the photoreactivation (in vivo) of dimers, and the stability of the dimers in cells kept in the dark, and the relation between dimer production and DNA synthesis in bacteria. The dimers are photoreactivable in both sensitive and resistant strains. Preliminary data indicate that they are also split (probably enzymatically) in resistant cells kept in the dark but are not split in sensitive cells. The studies on DNA synthesis are explicable in the above observations if one assumes that a dimer represents a block to DNA synthesis in vivo. Approximately one dimer per DNA strand of 6×10^6 A is sufficient to block DNA synthesis. In sensitive cells the blocks are permanent; in resistant cells they are repaired.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND INSTRUMENTATION

Basic Instrumentation. — Techniques for producing a thick, sensitive volume in silicon by drifting lithium ions were further developed. Good yield and reproducibility were achieved in drifting lithium to thicknesses of up to 1.2 cm in whole ingot sections (23 to 26 mm in diameter). Lithium-compensated silicon-diode detectors 12 mm thick and 23 mm in diameter gave 6.8-kev energy resolution for 122-kev gamma rays from Co^{57}. Such large-volume devices must be operated at reduced temperatures to minimize noise due to thermally generated charge carriers. Dry ice (200°K) is usually adequate. A bias of 200 v or more is required to collect the charge when a 2-μsec amplifier clipping time is used.

Steam-grown oxides were investigated for surface passivation of silicon diodes for radiation detection. Diodes protected with a 3000-A-thick oxide layer showed virtually no measurable response to gas ambient changes. The change in current, capacitance, or shunt resistance was less than 1% as the ambient was changed from vacuum to dry oxygen, room air (50% relative humidity), and ammonia vapor. However, measurements indicated that a strong n-type surface conductivity is produced at the silicon surface by the steam-grown oxide. Reverse currents are several orders of magnitude higher than for an untreated diode.

Compensation of this strong n-type surface conductivity by a gallium prediffusion was investigated. Diodes made by diffusing gallium before the steam oxidation step show that this technique was successful in maintaining a p-type surface under the oxide. Lithium drifting in such a diode produced an exact compensation of the surface states at the oxide-passivated surface. A p-type surface conductivity is necessary in order to compensate the surface with n-type lithium ions during the drift process. Such diodes have much lower reverse current than diodes having only the steam-grown oxide protection, but not as low as unprotected diodes in the proper ambient.

ISOTOPE DEVELOPMENT PROGRAM

Application of Radioisotopes in Analytical Chemistry: Radioactive Tracers in the Determination of Alkaline-Earth Elements. – Experimental work was completed on a study of the use of radioactive tracers in the determination of alkaline-earth elements. In this work emphasis has been placed on the methodology, including simultaneous application of two or more tracers. The gamma-active nuclides Ca^{47}, Sr^{85}, and Ba^{133} were used, and gamma spectrometry was utilized to distinguish the nuclides and to make quantitative measurements.

Application of Radioisotopes in Analytical Chemistry: Liquid Scintillation Counting of C^{14} with a "Balanced Quenching" Technique. – A method was developed for liquid scintillation counting at a constant, known efficiency of C^{14} in quenched samples. This method eliminates the manipulation and calculation problems of yield correction and avoids the radiochemical contamination inherent in the internal standard method. This "balanced quenching" technique is insensitive to differences in activity levels from sample to sample and also to small shifts in instrument operating parameters. The technique is ideally suited to routine counting of similar types of samples in any activity range.

Application of Radioisotopes in Analytical Chemistry: Solvent Extraction Studies. – Radiotracers of more than 50 elements were prepared by neutron activation and used in the evaluation of 4-sec-butyl-2-(α-methylbenzyl) phenol as an extractant in the determination of cesium and rubidium. The extraction characteristics of carrier-free amounts of each of these elements as well as milligram quantities of the alkali and alkaline-earth elements have been established. A method for the separation and determination of tracer levels and milligram amounts of cesium and rubidium was developed as a result of these extraction studies.

Analysis of Gamma-Ray Nuclides by Electronic Spectra Resolution. – The collection of standard spectra for the library of standard response functions was continued. A computer code is being written so that data for all such spectra may be automatically plotted in a standard three-decade, semilogarithmic format. With this addition to the program, it will be possible to distribute standard spectra in a graphical form as well as on punched paper tape.

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