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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

NOVEMBER 1963

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ORNL-3543

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

NOVEMBER 1963

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

REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

Steam-Graphite Reaction Rate Experiment. — A second graphite fuel channel sleeve of the type to be used in the EGCR was exposed to superheated steam at a constant gas inlet temperature of 1400°F and pressures varying from 30 to 300 psig in order to study hazard problems associated with a steam-graphite reaction in the EGCR. The generation rate and composition of the gas were measured as a function of steam pressure. The test data indicate that the reaction rate for the range of pressure and temperature indicated above is not a function of pressure. In future tests the effect of inlet temperature on the reaction rate will be determined.

Graphite Oxidation Experiment. — An engineering-scale test was initiated to study the problems involved in maintaining constant trace levels of CO and CO_2 in helium in a simulated reactor coolant system by means of a bypass gas cleanup system. Graphite oxidation reaction rates due to the trace impurities will also be studied.

In the present experiment the helium entering the graphite test section was heated to approximately 1200°F, and the graphite test piece was heated to a maximum temperature of 1825°F. Carbon dioxide was then injected into the loop and allowed to react with the hot graphite until the desired concentrations of approximately 225 ppm CO_2 and 125 ppm CO were established. Operation of the purification system, consisting of a CuO converter and a molecular-sieve bed, was then started to control the CO and CO_2 concentrations at the indicated levels. The system has now operated successfully for 575 hr with the graphite at 1825°F and the CO and CO_2 maintained at 100 to 150 ppm and 200 to 250 ppm respectively. The

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moisture content of the loop gas has steadily decreased over the test period and is now between 1 and 2 ppm.

Pyrolytic Deposition of Low-Density Coatings on Fuel Particles. — Attempts were made to pyrolytically deposit a porous carbon layer on uranium carbide fuel particles. The conditions required for the formation of a porous deposit are a relatively low temperature (< 1400°C) and a high hydrocarbon partial pressure (approaching 1 atm). Since methane, which had been used as the carbon source for coatings applied at 1400°C and above, does not decompose readily at lower temperatures, it was replaced with acetylene for these experiments. Four runs were made under conditions conducive to the formation of low-density deposits. Comparison of initial deposition rates indicated that higher temperatures led to higher deposition rates. In all runs, much soot was formed in or above the fluidized bed, but this did not interfere with the operation. The microstructural appearance of the coatings indicated random crystallite orientation and a rather large amount of porosity. Crushing strength tests indicated that bonding within the coating was good. The strength values were comparable to or greater than those observed for similar thicknesses of coatings deposited from methane at 1400°C. Coating densities will be measured if a technique suitable for porous structures can be developed.

Fission-Gas Release from UO_2. — Tests were completed on a series of UO_2 powders having BET (nitrogen adsorption) surface areas from 0.03 to 0.6 m^2/g. These powders were prepared for evaluation of the relationship between the surface area of a sample and the release of fission gas during low-temperature neutron activation. The results of these tests and tests on other powder samples show a clear linear relation between the BET surface area and the fractional release of fission gas during low-temperature irradiation for BET surface areas from 0.01 to 6.0 m^2/g. This is the type of relation that would be expected if gas escape occurred by a simple recoil mechanism or a diffusion mechanism.

The fractional release of fission gas for a given BET surface area was approximately one order of magnitude lower for the powders than for sintered UO_2 compacts. The slope of the plot of fractional release vs BET surface area was approximately the same for compacts as for powders. The difference in the data for compacts and for powders will be investigated in subsequent tests.

GCR-ORR Loop No. 2 Irradiations. — Loop operation is continuing with the second experimental assembly containing three 6-cm-diam graphite-matrix spheres fueled with coated particles of uranium carbide. Measured central fuel temperatures range up to 1700°F. The total concentration of noble-gas fission products, krypton and xenon, remains below 2000 $\mu\mu c/cm^3$.

ORR Poolside Capsule Irradiations. — Three sweep capsules containing 6-cm-diam graphite spheres fueled with pyrolytic-carbon-coated uranium carbide particles continue to operate at design conditions. Irradiation of these capsules began in August. The indicated central temperature of one capsule has increased from 2500°F to ~2800°F. The estimated burnup (atom % of total heavy metal) is ~1%, and the fission-gas release (R/B) has increased from 1×10^{-4} to 3×10^{-4} during the past two months of irradiation.

REACTOR FUELS AND MATERIALS

Nondestructive Test Development. — We are continuing studies toward improvement and increased utilization of contact microradiographic techniques, which have proved useful for the examination of coated fuel particles being developed for high-temperature gas-cooled reactors. One phase of this program has been the development of reference standards, which are needed to calibrate optically magnified radiographs to guarantee the accuracy of dimensional measurements. For instance, when microradiography is used to measure coating and particle dimensions in coated-fuel particles, variations in technique can change the amount of radiation undercutting around the spherical specimen and hence affect the dimensions of the final images. Also, small errors in the optical magnification can introduce bias. To overcome this, minute tungsten wires have been coated with pyrolytic graphite and very accurately measured. When radiographed simultaneously with the coated particles, these wires provide a positive bench mark that will undergo the same distortion effects as the coated particles. Thus, any necessary correction factor for greater dimensional accuracy can be determined directly from the reference standard.

Mechanical Properties. — Reactor irradiation of stainless steel introduces defects that interfere with the movement of dislocations and hence inhibit plastic deformation under the inevitable stresses that occur in reactor components under normal operation. Normally most metals can absorb large plastic deformations before the fracture stress is reached. However, when metals are irradiated, defects are introduced which inhibit the onset of plastic flow, and the fracture stress is reached with rather low plastic strains. One explanation for this effect is that the release of dislocations at the onset of plastic flow sweeps away the irradiation-induced defects that were inhibiting deformation. Thus the metal suddenly finds that the applied stress is very large compared with the strength in local regions, and the fracture strain is reached locally with only a limited plastic flow for the whole body.

Work is under way to produce in stainless steels a strengthening substructure that will not be lost when an irradiation-hardened piece is

deformed under high stress. The method consists of annealing at a sufficiently high temperature to homogenize the alloy, cold working to introduce dislocations, and then annealing at an intermediate temperature to form the dislocations into a strengthening substructure that cannot be swept out. We have so treated specimens of types 302, 304, 316, and 347 stainless steel under varying conditions. Now, we are testing their mechanical properties and examining their structure by transmission electron microscopy. When conditions for desirable properties and structures are established, treated specimens will be irradiated and tested to evaluate postirradiation mechanical properties.

Zirconium Metallurgy. — Zirconium is the only structural metal that combines a low neutron absorption cross section with adequate strength in the temperature range of power reactors. However, the properties of known zirconium alloys limit the conditions, durations, and media to which they can be exposed. The systematic development of alloys with extended usefulness requires a better knowledge of the fundamental metallurgy of the material, which in turn requires purer and larger single crystals of zirconium. We have successfully zone refined zirconium with repeated passes in a high-vacuum, electron-beam-heated, floating-zone apparatus, with significant purification. After only two passes of the molten zone, we have obtained resistivity ratios ($\rho_{300°K}/\rho_{4°K}$) for zirconium in excess of 400; the highest previously reported value is 260 [Compt. Rend. 256, 5570 (1963)] for material having four zone passes by induction heating. Some contamination of the specimen with carbon and oxygen was known to occur during processing (from back streaming from the oil diffusion pump); this can be prevented by improving the vacuum system. A single zone pass through selected stock has produced the largest known single crystal of α-zirconium, a rod 5/16 in. in diameter by approximately 4 in. long. Having proved the feasibility of zone refining, zone leveling, and single-crystal production in zirconium and its alloys by electron-beam heating, we are now constructing improved equipment to produce specimens as large as 3/4 in. in diameter and about 12 in. long. The materials will be used in studies of crystal growth, transformation behavior, oxidation mechanisms, alloy relationships, and deformation mechanisms, resulting in less ambiguity of the data than is obtained with the usual polycrystalline materials.

Solid Reaction Studies. — The processes that limit the useful life of structural metals, such as creep and corrosion, often depend on rates of diffusion in the metals. We have developed a novel sectioning technique that increases markedly the sensitivity of measuring diffusion in niobium and tantalum. By anodically oxidizing the metal surface and then stripping the oxide film, we can serially section these metals to controlled thicknesses of 100 to 2000 A. Using this technique, we have determined volume diffusion coefficients for Nb^{95} in niobium at temperatures as low as 1000°C. Incomplete data on the diffusion of Nb^{95} in tantalum reveal a quite-pronounced near-surface effect, particularly at low temperatures.

Materials Compatibility Studies. — In liquid alkali-metal systems such as are used to extract heat from high-temperature reactors, corrosion of structural metals is aggravated by the presence of oxygen in the liquid

metal. Yet, no satisfactory method exists for measuring the oxygen content of potassium, the coolant for some proposed space reactors. We are continuing an evaluation of the amalgamation method, which appears to be the most promising method for determining oxygen in potassium. In this method the potassium is absorbed in mercury, leaving a residue that is assumed to be K_2O and to contain the oxygen from the original sample. In previous tests the amalgamation method recovered only 6 to 81% of oxygen that had been added to potassium as K_2O, KO_2, or HgO. Since low recoveries could result if oxygen is lost to the amalgam during analysis, the oxygen concentrations in the mercury reagent before and after amalgamation were determined by a zirconium gettering—vacuum-fusion technique. The amalgam was produced by adding mercury to a potassium sample previously spiked with a large quantity of KO_2. Both triple-distilled mercury and mercury reclaimed by slagging and filtering were examined. Analysis showed that the two grades of mercury and the amalgam each contained approximately 30 ppm of oxygen; so loss to the amalgam did not explain the low recoveries. Analysis recoveries may be low because the oxide species rejected from the amalgam is not stoichiometric K_2O. Accordingly, we are now trying to isolate and determine the species rejected by the amalgam.

POWER REACTOR FUEL PROCESSING

Fluoride Volatility Processing: Aluminum-Alloy Flowsheet Development. — Further study (ORNL-3491, p 8) was made of the phase equilibria in the $KF-ZrF_4$ solvent system, potentially useful for dissolving aluminum-based fuel elements. In an attempt to resolve a discrepancy in previous determinations of liquidus temperatures for compositions close to that of the compound $KF \cdot ZrF_4$, this region of the $KF-ZrF_4$ system was reexamined. Specifically, the effect of oxide additions was evaluated. Thermal analysis showed that the compound $KF \cdot ZrF_4$ sometimes crystallized in a metastable form freezing at 453°C, which, during freezing, often changed exothermally into a more stable form freezing at 478°C. Oxide, in the concentration range 0 to 2500 ppm, raised the visually determined liquidus temperature (the temperature at which a cooling melt became cloudy) above the stable freezing point by approximately 1°C per 20 ppm of oxygen. This effect on the liquidus persisted throughout the concentration range 42 to 55 mole % ZrF_4 but was not noted at 37.5 mole %.

Since the oxygen contaminant in the KF was apparently the reason for the false liquidus point, a procedure was developed to improve the purity of the KF. The usual commercial KF is very hygroscopic and consequently is contaminated with either moisture or KOH, the product of the high-temperature reaction of KF with moisture. Single-crystal and vacuum-distilled KF, the best previously available products, have a KOH content corresponding to about 1200 ppm of oxygen (weight basis). Because KOH in molten KF reacts with various fluorides to precipitate metal oxides as follows:

$$2MF_x + xKOH \rightarrow 2MO_{x/2}\downarrow + xHF\uparrow + xKF ,$$

the production of better quality KF was possible by its vacuum distillation from the remaining molten mixture.

The use of FeF_3 and FeF_2 to precipitate the oxide gave a distilled KF containing 900 ppm of oxygen; the use of UF_4 gave a product containing only 500 ppm. More accurate determinations of the liquidus temperatures in the $KF-ZrF_4-AlF_3$ system were made with the higher quality KF.

Sodium fluoride was added to $KF-ZrF_4-AlF_3$ melts to determine its effect on liquidus temperatures. While this study served as part of the search for a lower-melting solvent with higher AlF_3 solubility, its principal purpose was to determine the effect of adding a part of the NaF in the movable-bed absorber to the fluorinator. This is the normal Volatility Pilot Plant practice while processing Zr-U alloy reactor fuel. The compositions chosen for the study, 52.0-31.0-17.0 and 51.2-23.3-25.5 mole % $KF-ZrF_4-AlF_3$, were approximately those obtained at the end of the volatility process by, respectively, a single-step dissolution of aluminum in 62.5-37.5 mole % $KF-ZrF_4$ solvent and a two-step dissolution with additional KF provided for the second step. The melts were found to dissolve substantial amounts of NaF at 600°C, the process temperature. The liquidus temperatures were lowered to approximately 500°C at NaF concentrations of 10 and 13 mole % for the respective solvents. They were raised to 600°C at 17 and 20 mole % respectively.

In laboratory-scale studies, observations of hydrogen evolution during the hydrofluorination dissolution of aluminum metal in molten $KF-ZrF_4$ salts at 600°C, in which only 20 to 30% of the theoretical amount of hydrogen is evolved during the dissolution, indicated that the reaction proceeds not only by an acid-metal reaction but also through reduction of ZrF_4 by the aluminum, with resultant formation of amorphous zirconium. This is subsequently converted to the zirconium hydride by the hydrogen produced in the reaction.

A laboratory test with 61-39 mole % $KF-ZrF_4$, in which the amounts of HF and aluminum were carefully measured along with hydrogen evolution, indicated formation of hydride with an H_2/Zr atom ratio of 1.3/1.

A series of laboratory-scale aluminum dissolution tests was made over the range 60-40 to 70-30 mole % $KF-ZrF_4$ to determine the composition limits for high dissolution rates observed in previous tests. The rates were found to go through a maximum value of 51.2 mils/hr at a composition of 60-40 mole % $KF-ZrF_4$, dropping off rather sharply to minimum rates of about 8 to 9 mils/hr at compositions exceeding either side of the 60-40 $KF-ZrF_4$ mixture by 2 mole %. Further dissolution studies with $KF-ZrF_4$ compositions at or in excess of 65 mole % KF required the addition of AlF_3 in order to reduce the temperature for complete solution of the salt below 600°C. Rates were again observed to go through a maximum, although somewhat lower, value of 25.6 mils/hr, at a base composition of 67-33 mole % $KF-ZrF_4$, dropping off similarly to the 60-40 mole % composition to values of ~8 to 9 mils/hr at 65-35 and 70-30 mole % $KF-ZrF_4$.

In engineering-scale studies, three sections of LITR prototype Al-U alloy elements were dissolved in $KF-ZrF_4$ by use of anhydrous HF between 550 and 610°C in a 5-in.-ID INOR-8 vessel. The element sections were full 18-plate cross sections, 4 and 6 in. long. The 4-in.-long element was completely dissolved at 8 to 12 mils/hr in 66-34 mole % $KF-ZrF_4$. For the 6-in.-long specimen, the dissolution rate was considerably slower, proceeding at only 2.6 mils/hr. For this dissolution, the salt resulting from the first test had been diluted with KF to provide a salt of the following composition: 59-28-13 mole % $KF-ZrF_4-AlF_3$. Again the resulting

salt was diluted with KF, giving an initial composition of 62.5-19.8-17.7 mole % $KF-ZrF_4-AlF_3$. This salt was used for the dissolution of another 6-in. element, and this third dissolution was fairly rapid, proceeding at about 6 mils/hr. The final salt, containing 6200 ppm of uranium, was fluorinated at 600°C, and after 2 hr it contained less than 100 ppm of uranium.

The corrosion of $KF-ZrF_4$ melt, proposed for dissolution of Al-U alloys, was studied on a laboratory scale at Battelle Memorial Institute and reported in their memorandum BMI-X-260 (Oct. 18, 1963). A commercial salt was adjusted with ZrF_4 to a nominal composition of 62.8-37.2 $KF-ZrF_4$ without the usual $HF-H_2$ purification. The salt, as used, analyzed 63.8-36.2 mole % $KF-ZrF_4$ and 131, 29, 766, and 312 ppm of Fe, Cr, Ni, and S respectively. During the first phase of the experiment, aluminum was dissolved with HF at 600°C; the second phase was conducted with the salt from the first phase and new corrosion specimens, but without aluminum dissolution. Corrosion rates were very low. No metal losses were detectable by micrometer measurements on any of the nickel-base alloys exposed during either phase of the experiment. By weight loss, maximum corrosion rates for INOR-8 and HyMu 80 (alloy 79-4) were 0.2 and 0.8 mil/month, respectively, during the first phase, and 0.5 mil/month during the second phase. Unusually severe intergranular attack was noted for Ni—1% Al and Ni—5% Fe specimens during both phases, with lesser amounts in alloy 2 (Ni—10% Mo—5% Fe) and weld filler metal 61.

Fluoride Volatility Processing: Zirconium Dissolution. — Preliminary reports of significantly low corrosion with the $KF-AlF_3-ZrF_4$ salt system have suggested that it might be used advantageously for processing Zr-U alloy fuel. A study of zirconium metal dissolution rates was therefore made in hydrofluorination tests in this system at 600°C. The tests were carried out at five ZrF_4 concentrations on the composition line between 59.5-10.0-30.5 mole % $KF-AlF_3-ZrF_4$ and the ZrF_4 apex on the ternary phase diagram. The measured zirconium dissolution rates, determined gravimetrically, were 7.6, 7.9, 6.5, 5.7, and 4.5 mils/hr, respectively, at 30.5, 36, 41, 46, and 52 mole % ZrF_4. All the tests were for 1 hr and were made in a 1-in.-diam reactor at an HF flow rate of 100 ml/min with approximately 70 to 80 g of salt. For comparison purposes, zirconium metal dissolution rates were measured under approximately the same conditions in the $LiF-NaF-ZrF_4$ system. Dissolution rates of 17.5 and 10.6 mils/hr were obtained, respectively, at 37.5-37.5-25 and 27.5-27.5-45 mole % $LiF-NaF-ZrF_4$. Although dissolution rates in the $KF-AlF_3-ZrF_4$ system are lower than those in the $LiF-NaF-ZrF_4$ system, confirmation of lower corrosion rates in the former may justify some sacrifice in the processing rate.

Fluoride Volatility Processing: Single-Vessel Studies. — Successful engineering-scale dissolutions of Zr-U alloy elements in molten salt with HF and fluorination of the resulting salt were made in the same vessel. An initial salt composition of 35-35-30 mole % $NaF-LiF-ZrF_4$ was used; temperatures were 550 to 575°C. Five dissolution-fluorination cycles and two additional fluorinations were made in a 5-in.-ID alloy 79-4 vessel; the salt recycle scheme was used. Dissolution rates of about 1 mg cm^{-2} min^{-1} were obtained during all dissolutions. Rates were consistent with those from previous dissolutions at similar conditions. No operational difficulties were experienced during any of the dissolutions or fluorinations.

During these and previous single-vessel studies, the effects of cyclic dissolution-fluorination on the corrosion of INOR-8 and alloy 79-4 were observed. The interface corrosion was significantly greater for INOR-8 than for alloy 79-4. A summary of vessel and corrosion-test-bar exposures and corrosion rates, based only on fluorine exposure time, is shown below:

	Test Bars		Vessels	
	INOR-8	79-4	INOR-8	79-4
Exposure, hr				
Molten salt	1060	1060	699	520
HF	142	142	83	79
F_2	66	66	36	34
$Al(NO_3)_3$			78	58
Corrosion rates, mils per hr of F_2 exposure	0.29[a]	0.14[a]	0.87[b]	0.53[b]

[a]By micrometer.

[b]By Vidigage.

Fluoride Volatility Processing: Corrosion. — Laboratory-scale studies were made at Battelle Memorial Institute to determine the corrosivity of 37-52-11 mole % $LiF-NaF-ZrF_4$ with HF at 650°C [see memorandum BMI-X-247 (July 31, 1963)]. This is the melt developed for processing the EBR-1 core 2 meltdown residue in the Volatility Pilot Plant. The corrosion study was made with no dissolution of metal fuel specimens to obtain the worst corrosion conditions. The melt was very highly corrosive, with maximum total corrosion rates (based on micrometer readings, plus intergranular attack) of 84, 86, 54, 83, 84, and 153 mils/month for INOR-8, HyMu 80, alloy 2 (Ni–10% Mo–5% Fe), Ni–1% Al, Ni–5% Fe, and nickel filler metal 61 salt-vapor interface specimens respectively.

Memorandum BMI-X-253 (Aug. 23, 1963) presents the results of a recheck experiment to study the corrosivity of 36.9-27.0-9.1-27.0 mole % $NaF-LiF-BeF_2-ZrF_4$ and HF, with and without dissolution of Zircaloy-2, at 500°C (ORNL-3436, p 13). Corrosion rates for the alloys of most interest were very low. According to weight-loss measurements in the first phase of the experiment with Zircaloy-2 dissolving, all specimens corroded at less than 0.1 mil/month. No attack was measurable by micrometer readings. However, based on sound metal remaining, which reflects intergranular attack, maximum corrosion rates during the first phase were 78 and 23 mils/month for interface specimens of Ni–1% Al and Ni–5% Fe. No intergranular attack was noted for the other alloys. During phase two, without metal dissolution, corrosion rates by weight loss were slightly higher. Maximum

values were 0.14 mil/month for INOR-8 (vapor), 0.27 for HyMu 80 (interface), 0.20 for alloy 2 (vapor), 2.6 for Ni–1% Al (liquid), 3.4 for Ni–5% Fe (interface), and 1.6 for nickel filler 61 (liquid). The only corrosion measurable by micrometer readings was 4.5 mils/month for the Ni–1% Al interface and liquid specimens, and 13.5 and 2.7 mils/month for the Ni–5% Fe interface and liquid specimens respectively. According to the amount of sound metal remaining, Ni–1% Al and Ni–5% Fe had maximum attacks of 32 and 14 mils/month respectively. No intergranular attack was found in the other alloys exposed during phase two.

The average rate of Zircaloy-2 dissolution, based on initial area, was only 0.17 mg cm^{-2} min^{-1}, with a maximum rate of 0.3 mg cm^{-2} min^{-1}. These low rates leave the usefulness of the melt for zirconium dissolution in doubt, pending single-coupon experiments.

Fluoride Volatility Processing: Drop Fluorination Tests. – Falling-drop fused-salt fluorination tests are in progress, with the objectives of determining the feasibility of recovering uranium in a spray type of fluorinator and studying the more important conditions involved. Small droplets of 50-50 mole % $NaF-ZrF_4$ salt, with diameters in the range of 80 to 150 μ, have been obtained in dropping sized powders through a 15-in.-long head furnace on top of the 4-ft fluorination tower. Uranium removal from salt containing 5 mole % UF_4 has been found to depend on fluorination temperature, drop size, and time (of fall) for the drop to pass through the fluorination section. The best removal demonstrated to date has been 99.8% for a drop diameter of 115 μ fluorinated for 3.2 sec at 600°C. The extraction of UF_6 into the fluorocarbon (C_8F_{16}) used to cool and solidify the fluorinated droplets has been observed. This has indicated possibly that diffusion of fluorine into the drop and diffusion of UF_6 from out of the drop are important rate-controlling steps.

Fluoride Volatility Processing: Sorption Studies. – A series of gasometric studies indicated that the retention of the uranium on MgF_2 is due to physical adsorption. The UF_6 is reversibly adsorbed in quantities much smaller than normal for chemical complex formation. When the MgF_2 is heated to 350°C at a pressure of 1 mm Hg, or less, the uranium content is reduced from 1.4 wt % to less than 0.1 wt %.

Two schemes for release of the physically adsorbed UF_6 and isolation from the TcF_6 are suggested. One scheme depends on the preferential desorption of the UF_6 at about 300°C with an inert sweep gas; the other depends on the selective adsorption of the UF_6 by NaF pellets after both the UF_6 and the TcF_6 are desorbed from the MgF_2 pellets with fluorine at 500°C.

Conditions favorable and unfavorable to adsorption of molybdenum and tungsten hexafluorides on sodium fluoride have been determined, and the dissociation (desorption) pressures of these hexafluorides have been measured. This information should be helpful in setting optimum conditions for separation from uranium hexafluoride.

Adsorption of molybdenum hexafluoride on sodium fluoride was unusual in that rapid adsorption occurred at 160 to 182°C, whereas outside this temperature range the adsorption was much slower. In the favorable range, preparations of $NaF_{2.3}/MoF_6$ were attained.

Adsorption of tungsten hexafluoride on sodium fluoride was rapid at all temperatures above 190°C (350° was the maximum tried), and preparations of $NaF_{2.3}/WF_6$ were attained.

The dissociation pressures of the hexafluorides over the complexes with sodium fluoride were:

$$MoF_6\text{-}NaF, \quad \log P_{mm} = 8.44 - 2.94\ (10^3)/T_K\ ,$$

$$WF_6\text{-}NaF, \quad \log P_{mm} = 7.10 - 3.13\ (10^3)\ T_K\ .$$

Also being studied on a larger scale are the effects on NaF pellets of alternately sorbing UF_6 at 100°C from an F_2-UF_6 stream, followed by desorption of UF_6 at 400°C in F_2. The experimental system provides for the transfer of UF_6 between two NaF beds (4 in. in diameter and 6.3 in. deep) which are alternately sorbing and desorbing UF_6. Periodically, NaF samples are taken for obtaining porosimetry data.

Pellets having a surface area of 1 m^2/g and a void fraction of 45% were used in a series of nine sorption-desorption cycles. Each cycle was observed to result in a slight amount of degradation of the pellets. After nine cycles the outer 30% of each pellet was observed to be present as weakly adherent, finely divided sodium fluoride. No increase in bed pressure drop as a result of the pellet disintegration was observed.

Fluoride Volatility Pilot Plant. — Since the U-Zr alloy processing program in the Fluoride Volatility Pilot Plant has been completed, a "clean break" is desirable prior to starting the next program. This necessitates at least a complete uranium material balance to avoid carrying forward an unmeasured system holdup. In addition to material balance data, wall thicknesses of major vessels should be measured to determine the extent of corrosion.

To achieve these two objectives, a system cleanout program was started. This involves removal of residual uranium and decontamination to gain access to the vessel(s) whose wall thicknesses are to be measured. One complete run was made in which Zircaloy-2 with no uranium was dissolved and the resulting salt fluorinated to recover uranium from NaF beds. This was followed by two barren salt flushes, which removed about 45 g of uranium from the walls of the vessels. The dummy run and the two salt flushes reduced radiation background at the bottom of the cell from 20 r/hr to 2-1/2 r/hr. A significant portion of this background comes from material remaining in the crossover line which carries off-gas from the dissolver to the flash cooler; this line reads 2200 r/hr.

The cold traps in cell 2 are being water flushed. The first flush, which contained a nuclear poison, recovered about 700 g of uranium.

THORIUM UTILIZATION PROGRAM

U^{233} Purification by Solvent Extraction. — An additional 4 kg of U^{233} for use in fabricating fuel rods for zero-power criticality experiments at Brookhaven National Laboratory was purified by solvent extraction from nitrate solution with di(sec-butyl)phenylphosphonate in diethylbenzene. In addition, about 13 kg of U^{233} was purified for fabrication into pellets for use in the Large Power Reactor Physics U^{233} Program at Bettis Atomic Power Division, Westinghouse Electric Corporation. Part of the 13 kg will

be used to make ThO_2–1 wt % $U^{233}O_2$ blanket pellets and part to fabricate ZrO_2–26 wt % $U^{233}O_2$ seed pellets to be clad in Zircaloy-2. All the purified uranium-233 nitrate solutions prepared met the specifications set by the requestor.

Uranium recovery across the solvent extraction units continued to be 99.8% of that fed to the system. The uranyl nitrate product solutions contained about 125 mg of U^{233} per milliliter and had a detectable ionic contamination of 200 ppm of U^{233}. The major amount (75%) of the ionic impurities was derived from the demineralized water used in the process.

Thoria Preparation. – Thorium oxide production by pyrohydrolytic denitration of $Th(NO_3)_4$ solution at temperatures increasing up to a final temperature of 500°C continued at the normal rate of 13 kg of ThO_2 (a batch) per operating day. One 13-kg batch of ThO_2 was rejected because steam was accidentally omitted from the denitration procedure. At present a 546-kg ThO_2 inventory is on hand. This is sufficient to fulfill the future requirements for experiments at Brookhaven National Laboratory. An additional 400 kg of sol-gel grade ThO_2 will be prepared to satisfy needs other than those of the BNL program.

Mixed Thoria-Urania Preparation in the Fuel-Rod Facility. – Four hundred seventy kilograms of mixed ThO_2-$U^{233}O_2$ was used to make 480 fuel rods for the zero-power criticality experiments at Brookhaven National Laboratory. The remainder of the total of 530 kg of mixed oxide produced is in the form of control samples (about 25 kg), system holdup (about 5 kg), and system cleanout (about 30 kg). These data indicate that there is less than 10% recycle of mixed oxide to the reprocessing units. This results from the fact that much of the sample material may be reused to make fuel rods.

The first furnace element failure occurred after 55 firing cycles. A replacement unit failed after only four firing cycles. Examination failed to show the reason for the failure of the second heating element. A faulty heating element or improper operating procedure is the probable explanation.

Radiation exposure doses at various work stations in the sol-gel and rod fabrication cubicle range from 5 to 15 mr/hr of general background in the cubicles to 565 mr/hr inside and at the center of a 5-kg crucible of calcined oxide. The calcined oxide has aged about 30 days since purification of the feed materials. Personnel exposure to ionizing radiation continues to be well below the permissible limits for total-body exposure and exposure to the hands.

Rod Fabrication. – On October 18, 1963, the 360th rod passed all inspections, thus completing the rods for the third shipment to Brookhaven National Laboratory. A material balance after completion of the campaign to prepare the third shipment showed a net loss of 0.15% of the sol-gel-prepared powder. To date, 432 rods have been prepared, of which 192 were prepared this month. The failure of the weld on the blender, reported last month, recurred this month. A replacement shell has been received and is being modified for use.

Thorium-Uranium Fuel Cycle Development Facility (TUFCDF). – Design of the TUFCDF, a facility to be used in the development of processes for

remotely processing and fabricating Th-U^{233} fuel assemblies, has been in progress since January 3, 1963. The title I design effort is complete with the exception of final revisions to drawings by the architect-engineer and final review of these drawings and the written portions of the preliminary proposal in the architect-engineer's offices. Design work is in progress for the construction of a mockup facility in Building 4501 to simulate two modules of the TUFCDF cells. Effort associated with process equipment design has been primarily in the areas of planning and scheduling and development of criteria for various major equipment items. These items include the vertical tube furnace for calcining the sol-gel product, a metering and transfer device for uranyl nitrate solution, a closed-circuit television system for radiographic examination of fuel tube closures, and a means for detecting alpha contamination in a high-gamma radiation field.

Particle Sizing of Sol-Gel Thoria and Thoria-Urania. — Sizing dried gels of ThO_2 and ThO_2-UO_2 is potentially a simpler operation than sizing after calcining the gels to the much more abrasive and refractory fired oxides. Therefore, gel-sizing studies were initiated. Sizing of dried gels before calcination is being tested by using thoria-urania and pure thoria sols with varying compositions, drying conditions, and ball-milling conditions. The results confirm that the size distributions are reproducible for a given sol and that the amount of −16-mesh product may be varied by varying the ball-milling conditions. The product size distribution was significantly changed by varying the NO_3^-/Th ratio. The calcined products contained 8 to 34% of +6-mesh material, which is too large a size for most vibratory compaction uses.

Sol-Gel Microsphere Preparation. — Thoria, thoria-urania, and thoria-carbon microspheres were prepared by drying fluidized sol droplets to gels in columns filled with 2-ethylhexanol plus additives to reduce coalescence and agglomeration. The sol-drop diameters from a two-fluid nozzle can be controlled by controlling the flow rates; the results show agreement with the literature on the breakup of liquid jets. Repeated distillations and reuse, and continuous distillation in a simple system, were demonstrated for the removal of water from the 2-ethylhexanol. About 700 g of microspheres and 10 kg of sol-gel thoria were prepared for evaluations of these materials.

Irradiation of Thorium Oxide Fuels. — Postirradiation examination is in progress on 11 capsules irradiated in the MTR and the NRX at heat ratings of 27,000 to 45,000 Btu hr^{-1} ft^{-1} and burnups of 22,000 to 35,000 Mwd per metric ton of metal. Visual examination indicated no degradation, and metallography revealed no marked effects.

Data from further examination of the MTR-irradiated ThO_2—4.4% UO_2 pellet rods indicate considerably better performance than was reported last month on a preliminary basis. The higher burnup rod released about 15% of the Kr^{85} at a burnup of 102,000 Mwd per metric ton of Th + U, and an average heat rating of 52,000 Btu hr^{-1} ft^{-1}. Fuel appearance was typical of oxide fuels irradiated under similar conditions.

Irradiation of Thorium-Base Alloy Fuels. — Two of the Th-U-Zr capsules are continuing to perform satisfactorily in the General Electric Testing Reactor. Visual examination of the two capsules that were removed showed bowing; also, there was corrosion product on the flow deflector. Both capsules are being disassembled to determine the causes of failure. One of the operating capsules will be examined at the first opportunity to see whether bowing can be related to the radial flux distribution. Another capsule is being prepared for inspection.

Equipment Development for Carbiding and Coating Sol-Gel Spheres. — Plans have been formulated for a pilot line in which sol-gel microspheres can be produced, converted to carbides, and pyrolytically coated with graphite. Preliminary designs for the conversion kiln and the coater are being reviewed.

Permeability of Vibratorily Compacted Fuels. — The permeability of Kilorod fuels and the relationship of this permeability to waterlogging failure are under study. Initial calculations show that the highest practical permeability at a given theoretical density is desirable.

The permeability of vibratorily compacted fuels is of interest because of its relationship to waterlogging. High permeability and low void volume in the compact facilitate the release of water from a waterlogged fuel element during a reactor startup. Present studies are concerned with the quantitative relationship of surface area, permeability, and theoretical density of the fuel to waterlogging susceptibility of Kilorod-type fuel elements.

Literature studies coupled with calculations indicate that values of permeability of about 1 darcy

$$\left| 1 \text{ darcy} = \left(\frac{1 \text{ cm}^3}{\text{sec}}\right) \frac{\text{centipoise}}{\text{cm}^2 \ (\text{atm/cm})} = \left(\frac{\text{flow}}{\text{rate}}\right) \frac{\text{viscosity}}{\text{area} \ (\text{pressure/length})} \right|$$

would be desirable to minimize cladding failure of a fully waterlogged element under rapid startup conditions. The only value obtained to date on a fuel sample shows a permeability of 4×10^{-3} darcy at a packed density of about 87% of theoretical. The surface area for this material is not yet available.

Preparation and Properties of U(IV) Sols and UO_2. — Development of a sol-gel process for the production of dense urania fuels from uranyl nitrate solutions would significantly extend the usefulness of the sol-gel technique. Studies are in progress on the preparation of U(IV) sols by electrolytic reduction—dialysis and also by precipitation and subsequent dispersion by peptization of the hydrous oxide.

In the electrolytic work, carried out in a two-compartment cell with the compartments separated by a cation exchange membrane and using a platinum anode and a mercury cathode, formate-stabilized sols were prepared.

They contained uranium concentrations of 0.05 to 0.07 M and formate-uranium ratios which varied between 0.07 and 0.8. These sols flocculated on being concentrated by heating at 90 to 100°C in argon, and the dried products appeared unsuitable for the production of the desired urania products by subsequent firing. Addition of nitrate to the formate-stabilized sols at a formate-to-nitrate ratio of 2 did not prevent the flocculation of the sols during drying, but the dried products contained a large fraction of the millimeter-size fragments with high-gloss surfaces that typify gels that form high-density UO_2.

In the precipitation-peptization work, a stable 0.4 M urania sol was prepared by the precipitation of the hydrous oxide from uranium(IV) nitrate solution with ammonia, subsequent water-washing, and aging (two weeks). Drying this sol in argon at 95 to 110°C resulted in a large, shiny, black gel product with a small quantity of fragments. This very promising looking material will be carried through a firing step in an effort to produce the desired large-particle-size UO_2 of high density.

Sol-Gel Preparation and Properties of ThC_2 and UC_2-ThC_2. — Spheroids of ThO_2-carbon and ThO_2-UO_2-carbon gels were satisfactorily prepared by methods completely parallel to those used for ThO_2 and ThO_2-UO_2. However, some problems of sol instability still persist and are the subject of current studies.

Undesirable properties in UC_2-ThC_2 preparations were attributed to the formation of chainlike aggregates of carbon or carbon-coated sol particles. The aggregates are thought to be due to dipole properties of the channel-black carbon particles used in the sol. They cause thixotropy in the sol and finally bring about settling, with carbon enrichment of the settled phase. Use of carbon having a lower surface area than that used to date decreased both thixotropy and instability, as did the use of surfactants.

Firing of uranium oxide—thorium oxide gels in 1 atm of argon at 2100°C was compared with firing in vacuum at 1750°C. Both procedures caused complete conversion in 1 hr, but porosities were higher at the lower temperature in vacuum. Porosities were also higher when the rise to maximum temperature was fast.

Physical and Chemical Properties of Molten Chlorides. — Continuous protactinium volatilization from reactor-irradiated, circulating, fused $ThCl_4$-$AlCl_3$-Pa (chloride) is a worthwhile idea to consider for the production of relatively pure U^{233} with good neutron economy. To evaluate this idea, more information is needed on the phase equilibria and chemistry of the fused chloride system. In studies to date it has been found that there is very limited mutual solubility between $AlCl_3$ and $ThCl_4$. In an attempt to "bridge" this large miscibility gap, $ZrCl_4$ has been studied as an additive to the system.

Phase equilibria studies on the $AlCl_3$-$ZrCl_4$ system showed discrepancies which are attributed to supercooling. Mole fractions of $ZrCl_4$ in the $AlCl_3$-$ZrCl_4$ studied include 0.05, 0.14, 0.33, 0.50, 0.67, and 1.00. Phase equilibria studies based on heating and cooling curves on the $AlCl_3$-$ThCl_4$ system were made over a range of mole fractions of $AlCl_3$, including 0.10, 0.25, 0.50, 0.75, 0.9, and 1.0. Differential thermal analyses at 500°C of the $ThCl_4$-$ZrCl_4$ system at a 1:1 mole ratio suggest that free

$ZrCl_4$ was present in the melt. This result is disappointing, since it was hoped that $ZrCl_4$ would be miscible with both $AlCl_3$ and $ThCl_4$ and would be a "bridging solvent."

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — Since UCNC craftsmen were on strike during most of the period covered by this report, little progress was made in the coolant cell, drain-tank cell, and reactor cells. The H. K. Ferguson Company completed the installation of conduit and cable trays and installed most of the electrical service and thermocouple wiring. They also completed about 70% of the auxiliary piping. The Ferguson portion of the work is ~50% complete, and the overall project was ~87% complete on November 1, 1963.

Chemical Development. — In a study of the freezing behavior of MSRE fuel, samples of melts were quenched after equilibrations at intervals of 3°C over a temperature range that included the first formation of crystals (melting point) and complete solidification. Identifications of the crystals that had been in equilibrium with the liquid at each temperature were based on optical properties as observed with a petrographic microscope. An idealized equilibrium crystallization path such as the following for $LiF-BeF_2-ZrF_4-UF_4$ (65-29.1-5-0.9 mole %) is consistent with the results:

Phases in Order of Appearance	Approx Mole % of Fuel Frozen at First Appearance	Temperature of First Appearance (°C)	Final (Approx) Vol %
$2LiF \cdot BeF_2$	0	434	73.5
$2LiF \cdot ZrF_4$	3	431	20.4
$7LiF \cdot 6UF_4$	70	416	3.2
BeF_2		360	2.9
Complete	100	353	100

The fact that the uranium can be concentrated by a factor of no more than about 3 before it begins to precipitate along with the other phases is relatively favorable for avoiding hazardous segregation and is relatively unfavorable for partial freezing as a means of concentration for reprocessing.

Ordinarily, equilibrium does not prevail, and the usual cooling paths involve a glassing of BeF_2-rich liquids that are low melting and thus are the last to freeze; hence crystals of pure BeF_2 are not to be expected, but rather, the last liquid to solidify forms glasses which incorporate variable amounts of the phases listed above with BeF_2.

In addition, the fuel has never been observed to freeze without considerable occlusion of later phases among the earlier crystals; this trapping also tends to give a uniform distribution of uranium in frozen fuel and is very effective for rapidly frozen melts.

Preparation of MSRE Materials. — It was recently discovered that conventional welds in copper might contain large amounts of included oxide, leading to the development of porosity when exposed to the combination of hydrogen, HF, and molten fluorides which will be present in the premelting furnace of the MSRE fuel production facility. Accordingly, the copper liners were removed from three of these furnaces, and nickel liners were substituted. When the furnaces were reassembled, the top interior surfaces were heavily plated with nickel to ensure that no surfaces available to the molten fuel or to its vapor would be of a composition which would give undesirable corrosion products. The furnaces have been reassembled and are ready for use. The copper liners are being examined to see whether the seam welds do contain the oxide inclusions which would explain the presence of corrosion products in previous production operations.

Of the 12,000 lb of beryllium fluoride ordered from the Brush Beryllium Company for use in making the MSRE fuel, flush, and coolant salt mixtures, approximately 5000 lb has been received. Samples of this material are being analyzed to determine whether the impurity concentrations are within specified limits. The rest of the order is expected to be shipped in December.

The large-scale horizontal treatment furnace, formerly used in the preparation of zirconium tetrafluoride by the hydrofluorination of zirconium tetrachloride, was moved from the storage area into the fluoride production facility. It will be used in the heat and HF-dehydration treatment which was found (ORNL-3491, pp 11–12) to be effective for converting the available Li^7F into a free-flowing crystalline material of acceptable bulk density.

Radiation Effects. — As in previous irradiations, the appearance of fuel, graphite, and INOR-8 from six recently sectioned capsules (ORNL-MTR-47-5, greater than 10% burnup) was generally good from the standpoint of structural integrity and lack of visible corrosive effects, even in cases in which relatively large amounts of F_2 had evolved from the frozen fuel. Nothing indicative of significant malfunction of molten fuel was noted. Neither the visual appearance of the components nor a microscopic examination of the fuel crystals gave any clues to the cause of the previously recognized variability in susceptibility of the frozen fuel to radiation damage, as manifested by different efficiencies of fluorine evolution, in some of the capsules.

Peculiar and exceptional features, generally attributable to F_2 evolution, included shiny films, as yet unidentified, on the salt at the metal-salt interface in two capsules in which reduced metal was expected because of the loss of fluorine, and also an example of severe attack of graphite as a result of a six-month exposure to tens of atmospheres of F_2 and the radiation of the decaying fuel. Possibly, but probably not directly, associated with evolution of F_2 was the presence of many gas bubbles at the fuel-metal interface in one of the lower power capsules; the contour of the bubbles indicated that the melt did not wet the metal.

Also a few milligrams of salt apparently escaped from graphite (AGOT) that had been impregnated with some 300 mg of fuel prior to the exposure; distillation may have been the cause, but the case for this explanation remains in doubt.

Irradiation Tests. — Design of the sixth irradiation experiment of the ORNL-MTR-47 series (47-6) is nearing completion. Objectives of the experiment include the study of uranium deposition in graphite under irradiation conditions in which the fuel is maintained molten throughout the test and the measurement of CF_4 evolution at MSRE operating conditions.

Two sealed capsules and two purged capsules will be irradiated. Each capsule assembly consists of a cylindrical INOR-8 capsule with a central graphite core submerged in fuel salt. The capsule is surrounded by a close-fitting copper heater sleeve in which tubular electrical resistance heaters are embedded. The heater, in turn, fits into a cylindrical water jacket for cooling. A 0.010-in. gas gap exists between the copper heater sleeve and the water jacket to maintain a central temperature of about 1300°F at design conditions. The heater will be used during zero or low reactor power to maintain the fuel molten, thereby avoiding conditions (frozen, irradiated fuel) which are known to result in the release of F_2 gas.

Initially, the capsules will be operated at MSRE conditions — that is, 14 w/cc fission power density at 1225°F central temperature. Fission power density can be increased to a maximum of about 52 w/cc by moving the experimental assembly closer to the reactor core, and a range of operating temperatures at a given power is provided by varying the composition of the mixture in the gas gap between 100% helium and 100% nitrogen.

The two purged capsules will be identical except for the graphite cores, one being one-half the length of the other, to provide information on the effect of varying graphite surface area and submersion depth on CF_4 evolution. The two sealed capsules will be identical except for uranium percentage and enrichment in the fuel.

A prototype of the purged capsules, loaded with fuel salt but no graphite, is undergoing tests to determine if the capsule purge tubes will plug with salt transferred by distillation. A bench test for the heaters and the gas-mixture temperature control system is being assembled.

Analysis of Radioactive MSRE Fuels. — The prototype tube cutter was modified and tested in order to open the newly revised copper ladle sampler. This ladle has two slots, 1/4 by 1/2 in., to facilitate the filling and draining of the molten salt. The cutter was fitted with two cutter wheels in parallel so that the one cutter wheel maintained the tension and alignment while the second cutter wheel was passing through the filling and draining slots of the ladle. A motor and clamp were added to hold and revolve the ladle while the tube cutter, held stationary by a rod on the motor mount, cut the ladle. The motorized version functioned satisfactorily but will be improved to facilitate operation.

Spectrophotometric methods for determining iron and molybdenum with the remotely operated filterphotometer were tested in the mockup.

The statistical evaluation of methods of analysis of contaminants in the MSRE fuel has been expanded to include iron and molybdenum. Iron

is determined by the colorimetric bathophenanthroline method and molybdenum by the colorimetric thiocyanate procedure. When the analyses were performed in sulfuric acid solutions of nonirradiated MSRE salts and under simulated hot-cell conditions, a relative standard deviation of 2.0% was obtained for iron and 1.8% for molybdenum.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. — Specifications and drawings for the HFIR fuel assembly were prepared and were issued to possible commercial fabricators for their comments. The present time schedule calls for issuing the actual bid request in January 1964.

All enriched fuel plates for the outer fuel element for critical experiment No. 3 were fabricated and were turned over to the shop for machining. Excluding rejects because of the ±10% tolerance on short-length (approximately 1/2-in.) averages of fuel concentration, the fuel-plate recovery rate was greater than 95%. The determination of the tolerances on averages of fuel concentration must be made on machined plates. Spot-checking representative plates shows that a slight increase in the short-length average tolerance to either ±12 or ±15% should give overall plate recoveries of greater than 90%.

Component Development. — Seventy corrosion assemblies which represent combinations of metals and nonmetals were exposed to pH 5 water at 100°C and then examined to determine compatibility and corrosion rates in the HFIR primary water system. Major findings to date are as follows: (1) aluminum alloy 6061-T6 in contact with stainless steel undergoes moderate pit-type attack, which can be tolerated in structural components; (2) beryllium is compatible with aluminum but not with stainless steel; (3) the aluminum skin on the control-rod plates suffered severe attack in areas contacted by the bearings, and further work is required to evaluate and resolve this problem; (4) an electrolyzed coating on stainless steel threads reduces galling of stainless-to-stainless threaded connections without reducing the corrosion resistance; (5) no conclusive evidence is yet available on the content and solubility of chlorides in several proposed plastics for the control-rod drive seals which will be in contact with austenitic stainless steels.

A full-scale mockup of one quadrant of the HFIR control region has been designed and is being fabricated. This mockup will be installed in the test and will duplicate flow conditions in the control region of the reactor.

Fabrication of reactor components is proceeding, and all parts which have been certified for construction are now being fabricated.

Design and Analysis. — Detail design work on the removable reflector is proceeding. Principal design criteria are: (1) the thermal stress level at 100-Mw operation is not to exceed 12,000 psi in any uncontained beryllium component; (2) an aluminum liner is to be provided at the innermost surface of the removable reflector regardless of the calculated thermal stress levels; (3) the removable reflector is not to serve as a structural or alignment member of the control-rod track assembly.

Eight 1/2-in.-diam vertical irradiation facilities will be provided in the removable reflector. Access to these facilities will be provided through the upper track assembly.

An extensive review of the fuel-element hot-spot analysis is being conducted. Among other things, the analysis is being expanded to include a method of predicting incipient boiling at the hot spot.

The hydraulic loads on the outer control rod have been determined and reported. The asymmetrical load on the upper track structure when one of the outer rods does not scram upon a scram signal is estimated to be less than 150 lb.

Work on determining the velocity and temperature distribution of the control-region coolant has begun. From the results the temperature distribution and the thermal deflection of the outer control rod will be estimated.

An analysis of stresses, bearing loads, and deflections of the control plate and its guidance system is being made. The results to date show that the design is probably adequate if the necessary tolerances can be met.

Machining of the HFIR pressure vessel is nearing completion without major deviations. To date, the machining of the nozzles satisfies their functional requirements.

Corrosion Studies. — The corrosion rate of hot-pressed reactor-grade QMV beryllium was unaffected by velocity in the range 12 to 81 fps when exposed in pH 5.0 water containing 4 to 5 ppm of dissolved oxygen at 100°C. After exposure periods ranging to 10,400 hr, the average corrosion rate of beryllium specimens was 2.0 mils/yr at all velocities within the above range. At flow rates of 2 to 3 fpm in the same water, average corrosion rates were 0.5 mil/yr after exposure times up to 12,500 hr. In both velocity ranges the corrosion rate did not change with exposure time. At all flow rates, pitting was observed, with pit depths up to 6 mils not uncommon. The incidence of pitting was independent of velocity.

Reports. — A draft of the HFIR description report will be issued to a limited number of people for comment early in December. Following a review of the comments, the report will be issued as a formal ORNL report in early 1964.

The safety analysis report is approximately 30% complete. The meteorological calculations, which were done on a unit release basis, have been completed. The fission product inventory was tabulated for various times-in-cycle. All other areas of the report are being worked on.

PHYSICAL RESEARCH

Reactor Operations. — Through November 17 of this year, the ORR and the LITR operated 78.2 and 86.0% of the time respectively.

Increasing evidence of distortion of the beryllium in the ORR has been noted over the past year. One piece was bowed approximately 0.50 in.; this piece, together with several others, has been replaced.

The ORNL Graphite Reactor was shut down November 4 after 20 years' service and has been placed in standby condition. Facilities at the other reactors are being provided for the experimenters displaced by the shutdown.

Installation of the remote console for the LITR in the ORR control room has been completed, and the LITR is now being operated from this console.

Installation of the sprinkler system in the ORR building has been completed.

Waste Disposal. — Work aimed at improving the monitoring of the stack for particulate alpha contamination is continuing. The goal is a system which minimizes false alarms due to natural radon and thoron activity in the air and which provides rapid identification of the contaminating alpha isotope. The proposed system uses alpha-energy spectroscopy as a means of discriminating against natural radon and thoron activity and as a means of identifying the contaminant. At present, work is under way on improving the sample collection system to obtain thin samples, either directly on filter paper or by electrostatic precipitation on a metal tape.

Hot-Cell Operations. — Operations in the hot cells were normal. Eight fueled capsules from the ORR, containing either fueled graphite balls or UO_2, were examined; impact and crushing strength tests were run on several of the fueled graphite balls. Examination of experiment ORNL 41-8, containing about 180 BeO pellets encapsulated in stainless steel, continued. The capsules from MSRE experiment 47-5 were slit; samples of salt and carbon were selected for analysis. About 50 irradiated stainless steel tensile specimens were pulled. Profile measurements were made on several ORR beryllium reflector pieces which had bowed.

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

PHYSICS AND MATHEMATICS

Charge Spectrometry. — The charge states of rare-gas atoms after electronic readjustment to an inner-shell vacancy have been measured with a magnetic mass spectrometer. Vacancies were produced by interaction of x rays with orbital electrons. The wavelengths of the x rays were chosen so as to eject electrons preferentially from one particular principal shell of the atom.

The following table lists the elements investigated, together with the shells in which primary vacancies were formed. Also given are the target and filter materials.

Element	Shell	Target	Filter
Neon	K	Mo, Ti, Al	Be
Argon	K	Mo, Ti	Be
	L	Al	Be
Krypton	K	Mo	Be
		Mo	Be, Zr
	L	Ti	Be
Xenon	M	Al	Be

The so-called $K_{\square}$ spectra of argon and krypton proved very similar to the spectra previously obtained from the corresponding nuclides that undergo electron capture.

In a first approximation the charge spectra of neon and argon can be reproduced from Auger and radiative transition rates (calculated by Rubenstein) and electron shake-off probabilities. More extensive ionization has been observed, however, in the course of the electronic reorganization than can be accounted for by the present theories. This has been particularly evident for the $K_{\square}$ spectrum of neon and the $L_{\square}$ spectrum of argon.

Neutron Diffraction. — Neutron diffraction measurements have been made on a single crystal of neodymium at temperatures below 20°K. The crystal structure is known to be hexagonal with a four-layer stacking sequence along the c axis of type ABAC. Thus, alternate layers have different near-neighbor environments, either hexagonal or face-centered cubic. Between 19 and 8°K, magnetic satellite peaks have been observed which are displaced from the nuclear peaks by reciprocal lattice vectors equal to $\pm\tau b_1 + b_3$ and $\pm\tau b_1 - b_3$. No satellites have been observed along the b_1 direction, indicating that the moments are aligned in this direction. The structure may be described as an antiferromagnetic arrangement of layers along the c axis with a sinusoidal modulation of the moments within each layer. The period of this modulation, described by the quantity τ^{-1}, increases from roughly seven to eight times the a_1 period as the temperature is lowered. Below about 8°K, the number of magnetic peaks doubles. Two closely spaced peaks appear in about the same reciprocal lattice locations as the single peaks in the higher temperature region. The ordered moments giving rise to both sets of satellites are in the b_1 direction, as indicated by the absence of magnetic peaks along this direction. The structure in the low temperature region is similar to that of the higher temperature region except that there are two different values for τ occurring simultaneously. These two sets of satellites may be associated with the two types of sites present in the crystal structure.

Theoretical Physics: Elastic Scattering of Deuterons from Ca^{40}. – The elastic scattering from Ca^{40} of deuterons of 7, 8, 9, 10, 11, 12, and 21 Mev has been subjected to optical-model analysis, as a preliminary to a distorted-waves study of the $Ca^{40}(d,p)$ reaction. Several ambiguities in the optical-model parameters were found, and these have been studied in detail. The inclusion of a polarization potential and of spin-orbit coupling was found to have little effect. Sets of parameters which give the best overall fit at all the energies were found.

Theoretical Physics: Distorted-Waves Theory of Direct Nuclear Reactions, with Spin-Orbit Effects. – A general and flexible formulation has been given of the distorted-waves theory of direct nuclear reactions which takes account of the effects of including spin-orbit coupling in the distortion and which does not make the zero-range approximation. The evaluation of the transition amplitude was shown to involve two distinct steps, one the construction of a form factor which characterizes the particular reaction mode and the particular nuclei, and the other the treatment of the dynamics of the distorted waves, which is similar for all reactions. Expressions were obtained for various differential cross sections and polarizations. As examples, procedures for treating inelastic scattering and stripping and knock-on reactions were formulated. The symmetry properties of the reaction amplitudes were studied.

Low-Energy Neutron Time-of-Flight Spectroscopy: Acceleration of Free Neutrons Under Gravity. – The 1951 Brookhaven measurement of the acceleration due to gravity has been repeated using the ORR 180-m evacuated flight path. Two horizontal collimating slits 8 mils high were located at 0 and ~4 m respectively. Two detector slits were located at 180 m and displaced vertically from one another by 5.75 in. A boral filter allowed all fast neutrons (which did not fall appreciably) to pass through the upper slit. The lower slit was covered by 4 in. of beryllium. Intensity measurements were made as a function of the vertical position of the assembly comprising the slits, the detectors, and the beryllium filter. The transmission edges associated with the (100) and (002) lattice spacings in the beryllium filter were used to define neutrons in the "slow" beam having particular velocities. Assuming the de Broglie relation between wavelength and velocity, the acceleration of free neutrons due to gravity was found to be $g = 976 \pm 3$ cm/sec^2 [(100) planes] and $g = 972 \pm 7$ cm/sec^2 [(002) planes]. These may be compared with the local value, $g = 979.7$ cm/sec^2, and the early result, $g = 935 \pm 70$ cm/sec^2.

Low-Energy Neutron Time-of-Flight Spectroscopy: Capture Gamma-Ray Spectra. – The gamma-ray spectra from neutron capture in individual resonances using samples enriched in the various tin isotopes have been studied with a large (9-in.-diam by 12-in.-long) sodium iodide crystal. A flight path of 5 m results in a neutron energy resolution of about 15% up to 200 ev, which is sufficient to resolve most of the resonances in these enriched samples up to this energy. The capture gamma-ray spectra from many resonances are quite simple, consisting of a few intense gamma rays. The spectra from the target nuclides Sn^{124}, Sn^{122}, Sn^{120}, Sn^{118}, and Sn^{116} show a transition to the ground state or to a level near ground state which comprises 10 to 50% of the gamma-ray spectrum. From the assignments

of the low-lying states and assuming that the resonances are s-wave, these strong gamma rays must be M1 transitions. Based on a total radiation width of 0.11 ev, the partial widths of these strong M1 transitions are 100 to 500 times the values predicted from the formula of Blatt and Weisskopf. Enhanced M1 transitions for this mass region are predicted by Bergqvist et al.

Low-Temperature, Nuclear and Solid State Physics: Multiplication in the Pulse-Height Response of Silicon Surface Barriers to Fission Fragments. – A number of investigators have observed a multiplication phenomenon in the response of silicon surface barriers to fission fragments. In many cases, this multiplication is anomalous in the sense that it occurs at electric field intensities well below that required for avalanche multiplication. One group has suggested that this phenomenon is related to a "threshold depth." However, other results from investigations of the response of surface barriers to fission fragments and heavy ions for a wide range of silicon resistivities and bias voltages have exhibited no systematic evidence to support the concept of a "threshold depth." In the work here, it was demonstrated that multiplication at anomalously low field strengths can be significantly reduced or eliminated by surface treatment after fabrication or before application of the front electrode. A possible multiplication mechanism based on the concept of tunneling injection through the oxide layer, which was first suggested by Gibson and Miller, is consistent with the experimental results.

Low-Temperature, Nuclear and Solid State Physics: Conversion Electrons from the 40-msec Isomeric State in In^{114}. – The square-wave modulated proton beam of the 5.5-Mv Van de Graaff has been used to excite the known 40-msec isomeric state in In^{114} by the reaction $Cd^{114}(p,n)In^{114m}$. The target was a thin enriched (98.5%) Cd^{114} foil located at the entrance aperture of a double-focusing magnetic spectrometer. Pulses from the spectrometer were gated synchronously with the beam modulation in order to avoid prompt background. Two well-separated electron peaks were observed, corresponding to the K and to the L + M + ... conversion lines from a 318 ± 5 kev transition. Preliminary estimates of the ratio of conversion coefficients $\alpha_K/(\alpha_{tot} - \alpha_K)$ give 1.5 ± 0.3. The frequency dependence of the peak counting rate leads to a half-life of 40 ± 4 msec.

High-Voltage Experimental Program: Scattering of Neutrons by Deuterons and the Dineutron. – Recently a calculation has been made [Phys. Rev. 131, 1740 (1963)] which predicts a large threshold effect in the n-d scattering cross section on the assumption that the dineutron exists and that one of two possible sets of scattering lengths, a_2 = 0.7 fermi and a_4 = 6.4 fermis, is the correct set. This effect would manifest itself either as a cusp in the total cross section or as a rounded step of 15 to 23% variation in the integral scattering cross section in the vicinity of the three-body threshold (neutron bombarding energy of ~3.3 Mev in the laboratory system). Its exact location and "width" would depend upon the binding energy of the dineutron, among other factors, and it would be shifted downward from 3.3 Mev by an amount equal to the binding energy.

We have measured both the total cross section and the integral scattering cross section with energy resolution and increments of the order of 20 kev from 2.8 to 3.6 Mev. Statistical standard deviation errors were about ±2% for the total cross section data and ±4% for the integral scattering cross section data. Within these limits our data do not show any deviation from a smooth curve.

High-Voltage Experimental Program: Absence of States in Be^8 at 7.56, 13.91, and 18.00 Mev. – The $Li^7(p,\alpha)\alpha$ and $Li^7(p,\gamma)Be^8$ reactions have been studied in an effort to confirm recently reported states in Be^8 at energies of 7.56, 13.91, and 18.00 Mev [Nucl. Phys. 36, 597 (1962)]. The alpha particles assigned to the state at 7.56 Mev are shown to be attributable to the $O^{18}(p,\alpha_0)N^{15}$ reaction. The alphas from the state at 13.91 Mev are shown to result from the $F^{19}(p,\alpha_0)O^{16}$ reaction. The postulation of a state at 18.00 Mev was based upon the presumed resonant formation of Be^{8*} which decayed by, first, gamma-ray emission to the states at 7.56 and 13.91 Mev, and then alpha-particle breakup of these states. Since both of the alpha-particle groups are assigned to contaminants, this state is also not confirmed. Resonances reported from the $Li^7(p,\gamma_0)Be^8$ and $Li^7(p,\gamma_1)Be^{8*}$ (2.9 Mev) reactions at 0.72 and 0.87 Mev bombarding energy are also not observed.

High-Voltage Experimental Program: Cross Sections for the $Ni^{58}(\alpha,p)$, $Ni^{58}(\alpha,\gamma)$, and $Co^{59}(\alpha,n)$ Reactions. – Thick-target yields have been measured for the $Ni^{58}(\alpha,\gamma)$ reaction from 4.9 to 6.1 Mev, the $Ni^{58}(\alpha,p)$ reaction from 4.9 to 11 Mev, and the $Co^{59}(\alpha,n)$ reaction from 5.8 to 10 Mev. The reactions were identified by measuring the coincident annihilation radiation from the induced positron activities. The absolute thick-target positron yields, which were measured at approximately 100-kev energy intervals, have an accuracy of ±6%. The reaction cross sections were obtained to an accuracy of 15 to 20% by the differentiation of the smooth thick-target yield curves. Over the measured energy range, the cross sections increase from 1 to 12 μb for the $Ni^{58}(\alpha,\gamma)$ reaction, 0.6 μb to 316 mb for the $Ni^{58}(\alpha,p)$ reaction, and 0.06 to 228 mb for the $Co^{59}(\alpha,n)$ reaction. The total reaction cross section for Ni^{58} has been compared with some values predicted by optical models. The $Co^{59}(\alpha,n)$ yields have been compared with those obtained by measuring the neutrons directly with the graphite-sphere detector.

CHEMISTRY

Effects of Radiation on Analytical Methods. – Studies of the effects of gamma radiation on acidic solutions of arsenazo III have been completed; similar studies on solutions of metal complexes of the arsenazo compound are being continued. In the former studies, the rates of radiolytic decomposition of arsenazo III in hydrochloric, sulfuric, phosphoric, and acetic acid solutions have been determined.

The investigations on the effects of gamma radiation on lithium chloride solutions, such as are used in separations of the actinides, have yielded direct evidence of the formation of free chlorine in irradiated acidic lithium chloride solutions. Irradiation of a 10 M LiCl–0.2 M HCl solution with a dose of more than 10^6 rads results in the buildup of free chlorine, which can be detected spectrophotometrically at 330 mμ. A very sensitive absorption peak at 233 mμ, tentatively assigned to HOCl, has been observed in irradiated 10 M LiCl. This peak, not seen in dilute solutions, is at least as sensitive as the o-tolidine test for determining chlorine.

The study of the effect of gamma radiation on the dimethylglyoxime (DMG) spectrophotometric method for nickel has been continued. An infrared study of irradiated DMG solutions has been carried out in which the role of ethanol in "protecting" the DMG solutions has been evaluated. It has been found that DMG is approximately 5 times as resistant to gamma radiation in an ethanolic solution as it is in an aqueous solution.

Separations Chemistry Research. – Continued study of equilibria in the extraction of strontium by di(2-ethylhexyl)phosphoric acid (HA) confirmed a synergistic enhancement of extraction on addition of tributyl phosphate (TBP) or trioctylphosphine oxide. The mole ratios at maximum extraction and the dependence on pH suggest that formation of the compound $SrA_2 \cdot 2HA \cdot 2TBP$ is involved. Diluent effects other than synergism are also involved. For example, it was found that the extraction maximum shifts with respect to pH, without significant change in value of the maximum extraction coefficient, on substituting chloroform, dodecyl alcohol, and dibutyl ether for the hydrocarbon diluent.

A direct method is being studied for measuring activity coefficients of aqueous acids in acid-salt mixtures, by parallel or simultaneous equilibration of the mixed solution and the pure acid solution with an appropriate organic solution of an amine. Preliminary values for sulfuric acid in sodium sulfate solutions are in encouraging agreement with the best literature values as previously collated for bisulfate estimation (ORNL-2281):

$m_{H_2SO_4}$	$m_{Na_2SO_4}$	$\gamma_{\pm}$ H_2SO_4	
		Experimental Data	Literature Data
0.35	1.06	0.092	0.081
0.22	1.04	0.090	0.080
0.056	1.03	0.080	0.076

A particular advantage of this method should be its applicability to multicomponent systems, including those for which no suitable reversible electrode may be available, so long as extraction of all metal ions involved is sufficiently low.

Separations Process Development. — The use of the phenol extraction (Phenex) process for recovering cesium from pollucite ore leach liquors was successfully demonstrated in a continuous mixer-settler run. The feed liquor was prepared by roasting the ore with a mixture of sodium carbonate and sodium chloride at 800°C and leaching with water. More than 98% of the cesium was recovered in four extraction and three water scrub stages with a 1 M solution of 4-sec-butyl-2-(α-methylbenzyl)phenol in kerosene. The extract was stripped with hydrochloric acid to give a pure cesium chloride product solution (375 g of cesium per liter) containing less than 0.02% combined alkali-metal impurities. Overall decontamination factors for cesium in the solvent extraction process were 170 from rubidium, 7000 from potassium, more than 100,000 from sodium, more than 700 from lithium, more than 100,000 from silica, more than 1100 from aluminum, and 30 from iron.

In the TALSPEAK process the lanthanide elements are separated from the trivalent actinides by preferential extraction with a dilute hydrocarbon solution of di(2-ethylhexyl)phosphoric acid (HDEHP) or 2-ethylhexylphenylphosphonic acid (HEH[ΦP]) from aqueous solutions 1 M in lactic acid and containing sodium or ammonium diethylenetriaminepentaacetate at pH 3.0 to 3.5. Subsequently, the actinides are extracted by a more concentrated solution of the extractant and finally stripped by dilute nitric acid. Extraction of the actinides by concentrated extractants from aqueous solutions at pH 3.0 to 3.5 was hindered by low rates of decomposition of the aqueous complexes, requiring equilibration times up to 20 min. The equilibration time was reduced to less than 1 min by increasing the lactic acid concentration to 3 M or preferably by reducing the pH to 1.5 to 2.0 with nitric acid. Distribution coefficients for the extraction of americium were also increased until a pH of 1.5 was reached. Maximum distribution coefficients with 1 M HDEHP in n-dodecane were greater than 100, eliminating the necessity of using the stronger but rare extractant HEH[ΦP].

Volatility Studies. — Extension of the molten-salt fluoride-volatility process to low-enrichment nuclear reactor fuels requires the separation and recovery of plutonium. Studies are therefore being conducted to determine the volatilization rate and recovery for plutonium hexafluoride in this process. Equilibrium constants K for PuF_6 volatilization of 0.43 to 0.50 at 600°C and 0.29 at 500°C have been measured with 31-24-45 mole % $LiF-NaF-ZrF_4$ and F_2. (The constant K is defined as X_g/X_l, where X_g is the mole fraction of plutonium in the vapor and X_l is the mole fraction of plutonium in the liquid salt.) At 650°C a K of 0.25 was obtained with 30-25-45 mole % $LiF-NaF-ZrF_4$, but this inverse temperature effect may be attributable to impurities in the second batch of salt caused by different purification techniques.

These equilibrium constants compare favorably with that of 0.016 at 600°C derived from literature data [J. Phys. Chem. 65, 398 (1961)] for the reaction

$$PuF_4(s) + F_2(g) = PuF_6(g)$$

when account is taken of the activities in the solid phase.

The K at 600°C for PuF_6 volatilization from 54-20-26 mole % KF-ZrF_4-AlF_3, the final salt composition of the proposed aluminum alloy flowsheet, is 0.15.

At 600°C, BrF_5 did not remove plutonium from 30-25-45 mole % LiF-NaF-ZrF_4 to any practical extent.

Plutonium fluoride remained in the liquid salt in a fractional-crystallization experiment in which up to 30 wt % of the salt was frozen out of a salt of starting composition of 33-67 mole % NaF-ZrF_4 and starting liquidus temperature of 670°C.

Radiolysis of Metal Fluorides. – Experiments to determine the rate of evolution of fluorine from solid lithium fluoride under bombardment by fast electrons were conducted using equipment and techniques similar to those employed in previous experiments with MSRE-type fused salt (ORNL-3491, p 19). The lithium fluoride was in the form of crystal particles which passed between 33- and 47-mil sieves. Estimated temperatures in the salt were 25 to 35°C. Total doses of about 5×10^{17} and 10^{18} Mev/g, accumulated at dose rates of about 6×10^{16} and 1.2×10^{17} Mev g^{-1} hr^{-1}, were employed in two experiments. No fluorine evolution was detected.

Under comparable irradiation conditions in the previous experiments, the MSRE-type salt was shown to evolve fluorine at an approximately uniform rate of 175 μg/hr at a dose rate of 6×10^{16} Mev g^{-1} hr^{-1} and after a dose of about 2×10^{17} Mev/g. Consideration of the results obtained with the two different salts, and of the scatter of the experimental data, showed that the rate of fluorine evolution from the lithium fluoride was less than 1% of that from the MSRE-type salt.

Surface Chemistry. – Simple mechanical agitation of powdered ThO_2 samples eliminated the slow portion of the heat of immersion into water; this action had very little effect on the specific surface area of the samples. In order to gain insight into the nature of the surface interaction, samples of ThO_2 were exposed to known relative humidity and the heat of immersion was subsequently measured. These experiments showed that ThO_2 covered with a monolayer of water has a heat of immersion about 20% of that of a dry sample. A vacuum microbalance has been placed in operation to study the adsorption of water vapor on ThO_2 for correlation with the heat of immersion data previously obtained.

The electrical conductivities of aqueous KCl solutions within a capillary of ceramic ThO_2 were measured and compared with the specific conductivities of the bulk solutions. For all KCl concentrations less than 1 N, the apparent surface conductivity of the ThO_2 was greater than that measured earlier by a slightly different procedure.

Electrochemical Kinetics and Its Application to Corrosion. – Experiments were reported previously which showed that the film on passive iron or stainless steel has the properties of an ion exchange material. The kinetic equation for the exchange process suggests that the activation energy increases linearly with the extent of replacement of chromate ions in the system investigated. Subsequent experiments in sulfate, phosphate, or chromate solutions at pH values around 9 showed that all these systems behaved similarly, so that it is presumably the hydroxide ion that replaces chromate ions in the film. This, and other experiments at a pH of about 4, further substantiates the conclusion that the film is at least

partially in a hydroxylated state in alkaline or weakly acidic media. When the exchange reactions are conducted at pH 2.4 with either sulfate or chromate as the electrolyte and stainless steel as the substrate, the data can be best interpreted as arising from a complex process involving at least two approximately successive steps. Experiments with pure iron and pure chromium are in progress in an effort to identify the separate processes.

High-Temperature Aqueous Solution Chemistry. – The investigation of both the concentration and temperature dependence of osmotic coefficients of aqueous solutions has been extended to 150°, or higher, for salt concentrations above 1 m. This particular region is of interest since many thermodynamic properties of aqueous salt solutions (above these temperatures) tend to resemble those found for non-hydrogen-bonded solvents. As the temperature is lowered the behavior of aqueous salt solutions tends to be characterized, with increasing intensity, by structural anomalies; it is this latter factor that has given rise to a profusion of structural models purporting to provide a basis for interpreting the behavior of water and aqueous solutions.

At 151.4°C, the most pronounced feature of the osmotic coefficients (ϕ) is the almost total absence of the sharp rise in the ϕ values normally accompanying increases of salt concentration. For example, the osmotic ratio of LiCl relative to NaCl is found to be practically independent of concentration. In other words, the usually highly anomalous LiCl system now appears to behave like NaCl.

Chemical Separation of Isotopes. – Integrated operation of the O^{17} facility began October 1, 1963, when the water distillation and thermal diffusion cascades were interconnected. Withdrawals of approximately 700 mg/day of oxygen containing 99.5% O^{18} are now being made. Establishment of the steady-state O^{17} gradient in the thermal diffusion cascade has not yet been completed. The O^{17} and O^{18} gradients in the water distillation cascade continue to build up. The present maximum O^{17} and O^{18} concentrations in this cascade are 1.2 and 77% respectively.

TRANSURANIUM-ELEMENT PRODUCTION

Acid Control in Tramex Feed. – The use of methanol in Tramex feed to inhibit the loss of acid by radiolysis appears to be completely satisfactory and does not produce any detrimental side effects in the extraction process. In the most recent test made with 10 ml of feed at an activity level of 10 w/liter, the extraction coefficients for the distribution of curium between 0.6 M Alamine 336·HCl–diethylbenzene and 10 M LiCl–0.03 M HCl–2% CH_3OH did not change over an eight-day period of time. This shows not only that acidity was effectively controlled but also that the products of the radiolysis of methanol do not interfere with the extraction of curium. Methanol was so effective in reducing the radiolysis

of acid and water that almost no radiolytic gas was formed. The use of methanol in Tramex feed caused no gross change in extraction kinetics or fission product behavior in mixer-settler tests made at 50°C with feeds containing tracer curium and fission products.

Tramex Safety. — Additional small-scale tests are being made to determine the fire and explosion hazards of Alamine 336·HNO_3 in diethylbenzene. An argon blanket was not effective in reducing the explosion hazard of a 4% solution of Alamine 336·HNO_3 in diethylbenzene in contact with concentrated nitric acid. However, mild explosions occurred (with or without the argon blanket) only when initiated by an electric spark in the vapor phase after the solution was evaporated almost to dryness and the pot temperature was 147 to 150°C. An explosion without the spark was observed when 8 M $LiNO_3$ (16% of total volume) was added and the above mixture was evaporated to near dryness. The pot temperature at the time of the explosion was about 255°C. All these conditions are much more extreme than those planned for the Curium Recovery Facility.

Americium-Curium Separation at High Activity Levels. — Methods that may be suitable for separating gram amounts of Am^{243} from Cm^{244} or of Am^{241} from Cm^{242} are being evaluated at high activity levels. More than 95% of the americium contaminated with only 0.07% of the original curium was obtained by two successive precipitations of $KAmO_2CO_3$ from 3 M K_2CO_3 at activity levels of about 50 w/liter. Ion exchange at high activity levels also appears to be possible if methanol is added to the process solutions. Satisfactory separations of americium from curium were demonstrated at a concentration equivalent to about 50 w/liter by eluting from cation exchange resin with α-hydroxyisobutyrate solution or from anion exchange resin with lithium nitrate solution when the process streams contained 20 vol % methanol to decrease radiolytic gas formation. Similar runs without methanol could be made only by spring-loading the resin column to prevent radiolytic gas from disrupting the resin bed.

Separation of Trivalent Actinides from Lanthanides by Organophosphorus Extraction from Aminopolyacetic Acids (TALSPEAK Process). — In the TALSPEAK process the lanthanides are preferentially extracted from the actinides by a dilute hydrocarbon solution of di(2-ethylhexyl)phosphoric acid (HDEHP) from a solution containing lactic acid and pentasodium or -ammonium diethylenetriaminepentaacetate [Na_5DTPA or $(NH_4)_5DTPA$]. The trivalent actinides are then extracted by a more concentrated solution of the extractant, and finally the actinides are stripped from the extractant with dilute nitric acid. Preliminary test results showed that the extraction of the actinides was hindered by low rates of decomposition of the aqueous complexes. Later it was found that this difficulty could be overcome by increasing the lactic acid concentration to 3 M. Still more recently a better solution to the problem was found; it involves decreasing the pH of the aqueous solution from about 3 to 1.5–2.0 with nitric acid.

A continuous multistage test was made of a flowsheet which includes the pH adjustment in the actinide extraction cycle. Eight mixer-settler stages were used in each of the three contactors, and the flow rates were such as to allow mixing-chamber holdups of only 1 min. The feed to the

first contactor was 1 M lactic acid containing irradiated Am^{241}, Cm^{242}, and the fission products from the irradiation. The scrub was 1 M lactic acid–0.1 M $(NH_4)_5DTPA$ at pH 3. The extractant was 0.5 M HDEHP in diisopropylbenzene. Relative flow rates of feed, scrub, and extractant were 1, 2, and 3. The raffinate from the first contactor was adjusted to pH 1.3 and was used as feed for the second contactor. The scrub was 1 M lactic acid–0.05 M $(NH_4)_5DTPA$ at pH 2 and the extractant was 1 M HDEHP in Amsco 125-82. Flow ratios were: feed, 3; scrub, 1; and extractant, 2. The solvent was stripped with 1 M HNO_3 which was scrubbed with 1 M HDEHP in Amsco 125-82. The flow ratios were: feed, 2; strip, 1; and scrub, 0.5. The results of the test were as follows: In the first contactor, 99.9% of the fission product rare earths were removed with a Cm^{242} loss of less than 0.1%; in the second contactor, 99.96% of the Cm^{242} was extracted; and in the third contactor, 99.9% of the Cm^{242} was stripped. No fission products were detected in the product, indicating a decontamination factor greater than 1000.

In tests of the effect of temperature on extraction by solutions of di(2-ethylhexyl)phosphoric acid from lactic acid–Na_5DTPA solutions, extraction at 25°C gave europium/americium separation factors twice as large as extraction at 50°C. The europium distribution coefficients were decreased by a factor of 3 and americium coefficients by a factor of only 1.5 when the temperature was increased from 25° to 50°.

Cell Mockup. – The in-cell handling (by means of the Central Research Laboratory master-slave manipulators and the transfer-case hoist) necessary for replacement of an "alpha" window was demonstrated in the cubicle mockup with a 300-lb steel plate as a stand-in for the window. The complete replacement procedure will be tested later with a complete window assembly. The equipment transfer case for replacement of equipment within the cubicle now appears to be operable. The entire equipment rack is accessible to the glove ports in the transfer case if special tools are used.

Pulsed-Column Studies. – Testing of the phosphonate extraction process, which separates americium and curium from the heavy actinides, in 3/4-in.-diam pulsed columns was continued; rare-earth tracers were used as stand-ins for the actinides. Increasing the temperature from 25° to 50°C reduced the stage height for europium extraction from 9.3 to 7.6 in., and for europium stripping, from 16 to 10 in. Introduction of air into the bottom of the column at 1 ml/min to simulate radiolytic gas release had no effect on stage heights.

HFIR Target Fabrication Development. – Laboratory development work resulted in improvements in the design of several items of equipment for the plutonium target fabrication line. It was learned that if the target tube is partially evacuated while the end closure welds are made, the chronic problem of porosity in the weld zone is minimized. The present method of collapsing the target tubes onto the cermet pellets leads to unacceptable distortions; additional development is required to solve this problem. The design of equipment for the Transuranium Facility is progressing satisfactorily. A number of the equipment items have been fabricated and are undergoing tests. All appear to have achieved the design criteria.

Transuranium Facility Design. – Construction of the Transuranium Processing Plant and design and procurement of equipment for the facility continue with no major problems. The installation of below-grade concrete and underground piping is continuing on schedule. All grade beams except the south wall, the floors of the pipe tunnel and the cell ventilation pit, and the lower walls of the waste-tank pit have been poured. The contract for the intercell conveyor assembly was let. Detailed design of the modified conveyor port closure, dolly canister, and position indicator was completed.

Design work for the chemical process equipment was concentrated on the in-cubicle equipment racks. To date, the design has at least reached the check-print stage on six of the ten racks required. Procurement of disconnect ferrules for process-line connections (numbering 5785) was started.

Corrosion. – Laboratory tests are being run to determine the effect of ferric ion on the corrosion of welded Zircaloy-2 by boiling and oxygenated 6 M HCl. The ferric ion concentrations under study include 5, 10, 15, 25, and 100 ppm. In a previous test with 50 ppm of ferric ion in the 6 M HCl, a marked acceleration in the attack rate was obtained, compared with the observed rate in the absence of ferric ion. However, in the present tests at all ferric ion concentrations under consideration, preliminary results show only a very mild increase in the normal attack rate. Specimens will be analyzed for hydrogen pickup upon completion of the tests.

METALLURGY AND MATERIALS

Ceramics Research: Sintering Studies. – As increasingly high operating temperatures are sought for nuclear and other systems, the need increases for high-quality ceramics, which are normally fabricated by sintering. We are seeking improved understanding of the rate of sintering and the factors governing it, particularly in materials useful in nuclear reactors. Previously we had shown that the initial sintering of thorium oxide occurs primarily by plastic flow. We have now measured the densification kinetics of fine-particle UO_{2+x} in the temperature range 900 to 1400°C and at oxygen-to-uranium ratios from 2.03 to 2.25. The sintering rate increases with temperature much more than the diffusion rate, suggesting that here also densification is not diffusion-controlled. We did not find a minimum in the densification rate of uranium oxide as a function of temperature, as we had previously in thorium oxide. However, we have found that low concentrations of impurities in the thorium oxide were responsible for this minimum.

Physical Properties Studies. – Many modern technical developments, particularly in the field of nuclear energy, involve the transfer of heat at high temperatures and the design of heat transfer elements. Consequently, knowledge of heat transport properties is needed for many materials at temperatures well above those for which reliable data now exist. Therefore, we are engaged in the development of improved apparatus to enable more accurate and more extensive measurement of the heat

transport properties of nuclear materials and concomitant more nearly unequivocal theoretical interpretation. We have reviewed and analyzed the thermal conductivity measurements made on UO_2 and iron in our radial heat flow apparatus and found good theoretical correlation with specific heat and other properties. In the linear expression A + BT (°K) which describes the thermal resistance of UO_2 between 200 and 1000°C, we find that B is an intrinsic property of UO_2 and equal to 0.0223 ± 0.0001 cm/w. On the other hand, A is sensitive to impurity content and varies ±10% around 5 cm(°K)/w. From the value of B we obtain a Debye characteristic temperature for UO_2 of 216°K, which compares favorably with 188°K from x-ray scattering and 160°K from low-temperature specific heats. Our data on iron, which is commonly used as a thermal conductivity reference standard, not only confirm the accuracy of our method but also have provided new information on iron. After correction for thermal expansion, the thermal conductivity of iron may be represented by:

$$K = 0.7273 - 6.260 \times 10^{-4}t \,, \qquad 100 \leqq t \leqq 436°C \,;$$
$$K = 0.6554 - 4.609 \times 10^{-4}t \,, \qquad 436 \leqq t \leqq 786°C \,;$$
$$K = 0.2703 + 2.854 \times 10^{-5}t \,, \qquad 786 \leqq t \leqq 910°C \,;$$
$$K = 0.1736 + 1.211 \times 10^{-4}t \,, \qquad 910 \leqq t \leqq 1000°C \,.$$

The thermal conductivity of iron decreased 4% at the alpha-to-gamma phase transformation (910°C). Electrical conductivity measurements indicate that this decrease is due to a 30% decrease in the lattice portion of the thermal conductivity. This is the first direct observation of this decrease, although a 7% decrease had been indicated previously by one set of thermal diffusivity measurements. The temperature dependence of the thermal conductivity of iron is controlled by the electronic portion, which can be calculated from the electrical conductivity. Analyzing existing data on UC, US, and UN, we find that the temperature dependence of the total thermal conductivity of these materials is also largely controlled by the electronic portion. The remaining lattice portion is as great as 50% of the total thermal conductivity, and is unexpectedly temperature-independent. We have achieved considerable success during the past two months with devices to measure the electrical resistivity, Seebeck coefficient, and thermal conductivity on 1/4-in.-diam × 1/4-in.-high specimens.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. – Experiments with electron-cyclotron heating at modest microwave power levels (up to 20 w) were continued. The effects of central-zone heating (26.6 kMc) and end-zone heating (35 kMc) were investigated, and operation with long plasma containment times (5 to 20 sec) was emphasized. The results with end-zone heating were not very interesting; again only minor changes in plasma properties were observed. Central-zone heating consistently produced a suppression of the rf signals

that can be associated with longitudinal plasma currents, a suppression of the burstlike ejections of plasma electrons out of the mirrors, and a significant reduction in plasma potential. The effects on the spatial distribution of the trapped fast protons were less pronounced and not entirely consistent. On some occasions the addition of the central-zone heating resulted in a reduction of the Z extent of the plasma and in increases in the central proton density of about 10% while the total number of trapped protons remained the same. At other times, this heating appeared to leave the spatial distribution unchanged, but to reduce the total number of trapped protons by about 30%. The specific operating conditions that determine which of these modes is obtained are not yet known. Measurements of the various plasma parameters as a function of the microwave power level are continuing, and these measurements have been extended to include studies of the proton energy distributions.

Measurements of the azimuthal distribution of slow plasma currents out of the mirrors have shown the first low-frequency rotational phenomenon to be found in the DCX-1. There is an intermittent slight modulation of the electron component of this current sometimes at 2 to 4 kc, sometimes at 8 to 12 kc, and at other times at both frequencies. The disturbance appears to be rotating in the direction of ion rotation in the field at the lower frequency. A possible explanation is that this disturbance is due to the formation of electron clumps which are then driven by the radial component of the electric field.

More experiments with Lorentz-force trapping at low pressures have been performed. The data from these have not been completely analyzed.

Beam-Plasma Interaction. — Further evaluation of the hot-electron plasma for stability reveals that it is stable over a wide range of electron temperatures. Measurement of the e-folding time, τ, in the decay of the photon flux after beam turnoff shows a 3/2 power dependence on electron temperature. The e-folding time, τ, has the correct magnitude for multiple scattering of the electrons by the atomic nuclei. A maximum temperature of 130 kev and a τ of 70 msec have been observed for plasma contained between magnetic mirrors. Addition of cusps with axis perpendicular (the Ioffe system) and with axis parallel (the split cusp) to the axis of the mirrors reduced the heating mechanism and provided measurement of τ at electron temperatures as low as 25 kev. All τ's were within experimental error of being on the 3/2 power curve.

Recent beam-plasma interaction studies in the uniform magnetic field of the beta tank facility confirm that the electron temperatures obtained are about equal to the beam voltage, from 20 to 30 kv. Radio-frequency probe observations vary markedly with gas feed into the localized high-pressure region. Two dominant operational modes have been obtained: (1) low-intensity x-ray output with a strong rf signal in the 80-to-120-kc range resulting from self-pulsing of the electron beam; (2) no low-frequency rf except noise continuum but a high-intensity x-ray output which is strongly dependent upon electrode geometry. Severe erosion of the end walls of the high-pressure region has prompted the initiation of an experiment to study ion diffusion and related effects.

Theory. — With the advent of "minimum-B" machines, in which plasma is confined at a local minimum in magnetic-field intensity, there is now

a long list of magnetic-field configurations expected to be stable against "macroinstabilities," whereby large portions of the plasma flow to the confining walls. There remains the localized, rapidly fluctuating turbulence, the "microinstabilities," expected to enhance plasma thermalization and diffusion across the magnetic field. However, if this diffusion is sufficiently slow, a macroscopically stable, toroidal configuration, wherein particles can escape only by crossing the magnetic field, should provide adequate confinement in practice. Using techniques reported previously (ORNL-3410, p 28), we have obtained a lower bound on the diffusion time. Though deliberately chosen pessimistically, the bound is typically tens of milliseconds or more so long as ions are not made to drift along the field at a speed exceeding their thermal speed. For example, the toroidal multipole under construction at the University of Wisconsin is expected to be free of such drifts. We conclude that the multipole, designed to be macroscopically stable, also should not be limited by microinstabilities. Our bound, obtained from conservation laws, is independent of details of geometry, etc.

Radio-Frequency Heating. – Further measurements have been made of the bremsstrahlung from the electron-cyclotron plasma in the EPA facility operated with 25 kw of 10.6-kMc microwave power. A 3 × 3 in. NaI(Tl) crystal mounted on a multiplier phototube viewed ~40 cm^3 of plasma through a 1/4-in.-diam pinhole. The fractional solid angle subtended by the crystal was $\sim 6.5 \times 10^{-7}$. From a comparison of the observed spectrum to the theory developed by Heitler (The Quantum Theory of Radiation, 3d ed., p 252, Clarendon Press, Oxford, England, 1960), a plasma "temperature" of ~120 kev was determined. By measuring the counting rate of photons above 120 kev, a plasma density of $\sim 5 \times 10^{11}$ electrons/cm^3 was inferred. These numbers give an energy density of $\sim 5 \times 10^{16}$ ev/cm^3 or $\sim 8 \times 10^4$ ergs/cm^3.

This measurement checks well with a measurement of the energy density computed from the voltage induced in an external coil when the klystron power is turned off. The voltage induced in this coil is related simply to the total magnetic moment of the plasma. Taking a plasma radius and length estimated from Hall-probe measurements and pinhole-camera x-ray photographs, the energy density is computed. In either case the total stored energy in the plasma is several hundred joules.

BIOLOGY AND MEDICINE

RADIATION EFFECTS

Statistical Services and Research. – Mutants of bacteriophage T4D have been isolated and characterized by the fact that they can propagate at low temperatures. Using two-factor cross data for mutations which are uniformly distributed over a large portion of the genome, attempts were made to construct a map which bears a close relation to the "physical map." In principle, one wishes a mapping function which permits conversion of recombination values to map distances. In an effort to arrive at estimates of some of the map distances, 17 observations involving 8

intervals on a small segment of the genome were examined statistically. The data, representing recombination distances, were regarded as 17 independent measurements on 8 unknown intervals. Then estimates of the sizes of the eight intervals were achieved by the method of least squares. The results are being evaluated for possible extensions to the entire genome.

Studies have been made of the action spectra for ultraviolet-induced chromosome aberrations in Chinese hamster cells in vitro, pretreated with 5-bromodeoxyuridine (BUdR) or 5-iododeoxyuridine (IUdR). There is reason to believe that the incorporation of thymidine analogs into the cells will modify the action spectrum. Cells exposed to different wavelengths of ultraviolet radiation in the presence of IUdR or BUdR or neither were scored for numbers of terminal deletions and exchanges. The number of aberrations per cell was estimated at each wavelength. Then, assuming that the numbers of aberrations were Poisson-distributed and that the number of cells scored was fixed, it was possible to estimate the ratio of aberrations per cell in cells treated with BUdR or IUdR to cells not treated in this way, for all levels of ultraviolet irradiation. At the same time, confidence limits were calculated for all the ratios and for each of the estimated mean numbers of aberrations per cell.

Pathology and Physiology. — A primary post-x-irradiation granulocytic leukemia of the RF/Up mouse has been serially transmitted by inoculation of RF/Up recipient mice with leukemic spleen cells, cell-free spleen extract, or plasma. Its transmissibility by cell-free filtrates containing virus particles has been demonstrated by electron microscopy. The similarity of the clinicopathologic features of this disease with those of human chronic granulocytic leukemia and the regular occurrence of the Ph^1 chromosome in patients with the latter disease prompted a search for a similarly characteristic chromosomal abnormality in mice with this leukemia. Cytogenetic analyses were performed on bone marrow cells from 11 mice — five inoculated with spleen cells, five with spleen extract, and one with plasma — who subsequently developed granulocytic leukemia. The majority of the cells analyzed showed a shift in modal number to 41 chromosomes. Also, the majority of the modal cells, as well as some nonmodal cells, had one abnormal-appearing marker chromosome. Both the increase in percentage of 41-chromosome cells and the apparently identical marker were present in all 11 cases of leukemia. A second myeloid leukemia passage line, derived independently from a primary neutron-irradiated RF/Up female leukemic mouse, is being investigated. Preliminary results in 14 recipient leukemic mice indicate the presence of the same marker and modal shift. These findings suggest an etiologic process incorporating features of both viral and somatic mutation concepts of leukemogenesis.

Recovery from Somatic Effects of Radiation. — The ability of transplanted mouse bone marrow cells to colonize the spleen of an irradiated recipient is not dependent on the number of erythroblasts and myeloblasts present in the inoculum, but rather on more primitive precursors. The following evidence suggests that the precursor cell in question may be related to the small "marrow lymphocyte." $(BALB/c \times A/He)F_1$ female mice were exposed to 850 r of x rays and received 5×10^5 marrow cells. Marrow

was retransplanted at 35-day intervals into secondary, tertiary, and quaternary recipients. The latter received, in addition, 10^7 fresh lymph node cells. The proliferative capacity of the marrow was determined by estimating its ability to promote uptake of iododeoxyuridine-I^{131} into the DNA of recipient spleens, and was correlated with its cellular constitution as revealed by differential cell counts. The lymphocyte content appeared to be directly correlated with the proliferative capacity of the marrow throughout the experiment, and both were persistently decreased after the first transplant. With serial transplantation, the lymphocyte deficiency and the inability to proliferate were accentuated. Injection of fresh lymph node cells did not correct these marrow deficiencies, although it improved the survival of the recipients. Changes in the erythroblast and myeloblast content of the transplanted marrow did not appear to be correlated with its competence for continued proliferation. The results reconfirm previous observations suggesting the involvement of small, mononucleated, lymphocyte-like marrow cells in hemopoietic cell production. They also suggest that such a cell is not derived from lymph node lymphoblasts.

Physiology of Blood Platelets. – Megakaryocytes, from which blood platelets are derived, are being studied by microphotospectrometry to determine whether there is a correlation between DNA content and the stage of development or maturation of the cell. Rat bone marrow smears were fixed in Carnoy's fluid and stained by the Feulgen technique. The nuclei were measured on a microphotospectrometer for total DNA content, using the two-wavelength method of Pätau. In the nuclei measured thus far, the amount of DNA present has been found to be correlated with the number of lobes present in the nucleus. For example, a nucleus having one lobe had a relative number DNA content of 0.068 and a nucleus having two lobes, 0.119. The method, therefore, may be useful in studying the megakaryocyte maturation cycle and perturbations of this cycle produced by radiation and other influences.

Medium(Low)-Level Long-Term Effects of Radiation. – Female RF mice were exposed to whole-body irradiation [250-kvp x rays, Co^{60} gamma rays, fast neutrons from a cyclotron (~1 Mev mean energy), and fast neutrons from a polonium-beryllium source (~5 Mev mean energy)] beginning at ten weeks of age. Doses of < 1.0 to > 5000 rads were accumulated at dose rates ranging from ~100 rads/min to less than 0.00007 rad/min (0.1 rad in 23 hr), and the resulting effects on survival and on the incidence of various diseases (neoplasms, lens opacities, and senile and other degenerative changes) were recorded. The life expectancy of 30-day survivors was shortened ~0.83 day per rad of fast neutrons, irrespective of the dose rate, whereas the life-shortening effectiveness of x rays and gamma rays was ~0.24 day/rad at the highest dose rate and only ~0.10 day/rad at the lowest dose rate. The effectiveness of gamma rays was similarly dependent on dose rate for other late somatic effects; hence, the relative biological effectiveness (RBE) of fast neutrons generally increased with decreasing dose rate for these effects. Only in cataract induction did the RBE exceed 10. In the induction of ovarian tumors, it was unusually low (less than 1.0). Our results imply that "multi-hit" radiobiological events are important in the pathogenesis of most late effects of radiation and that protection guides for human exposure to

radiation must be interpreted in the light of time-intensity variations in the effectiveness of x rays and gamma rays.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. – While chemical protection against ionizing radiation has been achieved readily for cells from a wide variety of mammalian tissues, we have at present no means for repairing radiation damage of these cells. Clues from experiments with plant cells and protozoans have recently provided a more optimistic basis for a continued effort in repair of radiation-injured mammalian cells. A major difficulty is a lack of information about the basic nature of the cell lesion or lesions induced by radiation. However, certain obvious factors, such as chromosome abnormalities, faulty synthesis of nucleic acids, and aberrations in cell division, provide a working point of departure for further investigation. We are using an assay system whereby potential repair of mouse bone marrow cells irradiated in vitro can be detected. Repair is measured in terms of the ability of these cells to function and grow in irradiated mice. Red-cell formation is determined by the use of radioactive iron incorporation in the spleen, an organ that contains red-cell-forming tissue. Assuming that repair of radiation injury requires at least a short period, we have investigated several postirradiation cell culture conditions for the bone marrow cells. Results suggest that the extent of damage is not appreciably dependent on culture media insofar as the usual standard preparations are concerned. Similarly, postirradiation temperature and pH do not seem to modify the extent of radiation injury. These factors were investigated because it has been shown that they, in part, determine radiation sensitivity of lower organisms. Alteration of media composition by the addition of agents not normally present in standard culture preparations is being investigated. Adenosine triphosphate, a compound of fundamental importance in biological exchanges of energy, has been tested as a recovery agent. Its failure to modify injury suggests that insufficiency of high-energy compounds is not a key factor in the inability of cells to divide after radiation.

Tissue Culture. – Present experiments indicate a dose-rate fractionation effect on one-hit chromatid aberrations in the corneal epithelium of the Chinese hamster. The effects of the dose rate of x rays on one-hit aberrations and mitotic delay are being further studied in both mammalian and plant cells. In addition, an attempt is being made, by mitotic synchronization, to determine an accurate and complete G_2 sensitivity curve for *Vicia faba* and Chinese hamster cells.

Additional exposures of human leukocytes to 2.5-Mev d-d neutrons have been made, as well as a series of exposures to fission-spectrum neutrons from the HPRR. Material from both series is being scored for chromosomal aberrations. Preliminary results indicate that the relative biological effectiveness (RBE) for fission-spectrum neutrons is approximately 6. As part of the NASA–Biology Division proton RBE study, human leukocytes have also been exposed to 50- and 100-Mev protons from the Harvard cyclotron and to 450-Mev protons from the Chicago cyclotron. Scoring of this material is in progress.

In collaboration with the ORINS Medical Division, a study of chromosomal aberration yields in the peripheral leukocytes of whole-body Cs^{137}-irradiated spider monkeys is in progress.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND RADIATION INSTRUMENTATION

Basic Instrumentation. – The application of field-effect transistors in charge-sensitive preamplifiers was continued. The noise vs frequency spectra (equivalent noise resistance as a function of frequency) of several field-effect transistors were measured with narrow-band filters. Equations for theoretically predicting the preamplifier resolutions were derived. Resolutions below 2 kev full width at half maximum were obtained by cooling the input field-effect transistor with liquid nitrogen.

CIVILIAN APPLICATIONS OF NUCLEAR EXPLOSIVES

Chemical Applications of Nuclear Explosions. – Work here in this program is centered about the laboratory development required for Project Coach, a planned underground detonation of a 5- to 10-kiloton nuclear device designed to produce maximum neutron fluxes in a bedded salt formation near Carlsbad, New Mexico. The purpose of the program is to produce milligram or larger amounts of transcurium elements. Recovery of the isotopes will consist in mining thousands of tons of debris and chemically reclaiming the isotopes.

Countercurrent leaching (three stages) of the water-insoluble residue with 30 ml of 1 N HCl per gram of residue showed that the percentage of rare-earth beta activity or gross beta activity was increased 10 to 20% over a single batch leaching of a gram of the residue with 30 ml of acid. Only a small increase for countercurrent leaching is to be expected, since dissolution of the water-insoluble residue is necessary to remove the activity, and the residue has a limited solubility in the dissolvent. We believe that the 10 to 20% increase with countercurrent leaching is due to more complete decomposition of the silicates, as evidenced by the fact that releaching of the acid-insoluble residue with fresh acid removed an additional 10 to 20% of the radioactivity. Also, a leaching mixture 13 M in HNO_3 and 0.04 M in HF was about three times as effective as 13 M HNO_3 alone, further indicating the necessity for decomposing the silicates in order to leach out the isotopes.

Direct leaching of samples from the Project Gnome event with sulfuric acid without first leaching with water in order remove the salt was ineffective. The acid consumption by several water-insoluble residues varied from 5 to 15 meq/g. No correlation between the amount of acid consumed and the solubility of the residue was observed. However, in general, the greater the radioactivity of the sample, the greater was its acid consumption.

The addition of Sepran filter aid to acid leach liquors prior to the separation of the acid-insoluble fractions improved filtering and settling.

Volumes as small as 10 ml per gram of water-insoluble residue were easily separated by using 0.1 g of Sepran per liter.

Measurement of the distribution coefficients for Ce^{3+}, Tb^{3+}, Eu^{3+}, Am^{3+}, and 14 possible contaminant ions for extraction from a 1 N HCl solution with di-tetramethylbutylphenylphosphonate showed that this solvent could separate rare earths and actinides from the leach liquors. The distribution coefficients for the actinides and rare earths were 3 to 10, and the separation factors from the main contaminant ions were 50 to 1000 per stage. The distribution coefficients had a third-power dependence on the extractant concentration and were inversely proportional to the third power of the hydrogen ion concentration. We believe that extractions from acid solutions are preferable to those from neutral solution because gel formation in leach liquor of low acidity has been observed with several Gnome samples.

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