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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 1962

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OAK RIDGE NATIONAL LABORATORY

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Oak Ridge, Tennessee
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U.S. ATOMIC ENERGY COMMISSION

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

SEPTEMBER 1962

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

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROGRAM

EGCR: Component Fabrication and Testing. — Fabrication work on the EGCR main and vessel-cooling blowers continued. Assembly of the EGCR control-rod testing facility was completed, except for the rod-drive-mechanism control panel, which is being designed.

A modification of the EGCR prototype top dummy fuel element incorporating 21 circumferential grooves was tested. This modification of the element gave flow characteristics independent of position in the channel and therefore should be satisfactory for EGCR application. An orifice plug for the bottom dummy was designed on the basis of tests run on an adjustable plug. This plug will provide linear flow control over a range of 400 to 2500 lb/hr and will permit more accurate adjustment of fuel-channel flow.

EGCR: Evaluation of Irradiated Fueled Test Capsules. — Two test capsules containing UO_2 pellets of the diameter specified for the EGCR ruptured during irradiation in the ETR. These capsules differed from the EGCR fuel elements in that they contained solid pellets rather than cored pellets, and they were irradiated for a long time at about twice the design power for the EGCR fuel. They were of 0.75-in.-OD, 6-in.-long, type 304 stainless steel tubing with a wall thickness of 0.020 in. The fuel pellets were solid cylinders of normal UO_2. The cladding temperature was controlled at about 1300°F, and the design heat flux was 159,000 Btu hr^{-1} ft^{-2}. The irradiation history indicated, however, that the design power of the elements was exceeded in varying and somewhat indefinite degrees.

Upon removal of the capsules from the irradiation assembly, major cladding ruptures were immediately apparent. Longitudinal fractures were present in the central region of each capsule and were about 3 in. long. Transverse sections through the capsules revealed central voids in the originally solid UO_2 pellets. In the more severe case, the central void was about 0.12 in. in diameter and extended almost the length of the fuel column. A region of columnar grain growth surrounded the central void and extended to a circumferential crack at about 0.15 in. from the outer surface. There was no evidence that the UO_2 had been molten; the central

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void and columnar grain region were formed concurrently by the migration of small voids in the UO_2 toward the hotter (central) region. This behavior is typical of UO_2 heated to a high temperature (> 3000°F) in a steep thermal gradient.

There was a heavy grain-boundary precipitate in the inner surface region of the stainless steel cladding. This precipitate, which appeared to be a nitride, extended into the fracture surfaces. Since a significant fraction of uranium nitride (primarily UN_2) was found in the unirradiated fuel pellets, it is assumed that the nitriding of the cladding resulted from nitrogen release from the fuel.

It appears that there were two primary causes for the cladding failures: (1) the assemblies were overpowered, and, as a result, there was excessive stress on the cladding; and (2) the capsule material lost ductility because of nitride formation and was therefore more susceptible to fracture under stress.

PBRE: Component Fabrication and Testing. — Design work was completed on the Pebble-Bed Reactor Experiment blower-bearing testing unit, and fabrication was started.

PBRE: Fission Product Transport and Deposition Studies. — The analysis of transport and deposition of fission products from a gas stream to the walls of a conduit was extended to provide a more useful treatment for the case of recirculation. The equations were simplified, and experimental investigations for verifying the equations and evaluating physical constants were planned.

PBRE: Experimental Studies of Fission Product Deposition. — A small stainless steel out-of-pile loop developed at BMI is being used for obtaining release rates of long-lived gamma emitters, such as I^{131}, Ba^{140}, Sr^{89}, and Te^{132}, from unclad fuel. The fuel specimen for the first test was irradiated in the BMI reactor.

The neutron-activated UC_2 particles were held in an AGOT graphite bottle at 560°C for 24 hr. Preliminary examination of the deposition tube, which had a temperature gradient varying from 36 to 660°C, and of several traps in the loop indicated that the amount of activity released was very low, except for Xe^{133}.

Two autoclaves containing AGOT graphite plus specimens of types 304L and 310 stainless steel and Inconel have operated for 300 hr at 1500°F with a hydrogen atmosphere at a pressure of 850 psig. Operation is continuing.

GCR-3 Physics. — All calculations of GCR-3 fuel-cycle cost were revised on the basis of the new (July 1962) AEC uranium price schedule and the most up-to-date parameters for the reactor. In particular, the calculations were done for a thermal efficiency of 40% rather than the original value of 36%. The results are summarized below for several values of fuel lifetime, and fuel cycles in which 1/2, 1/3, 1/4, or 1/5 of the core is refueled at each shutdown are compared with the continuous feed cases:

Lifetime (Mwd per metric ton of U)	Fuel-Cycle Cost for Continuous Feed (mills/kwhr)	Fuel-Cycle Cost for Fractional Feed at Shutdown (mills/kwhr)			
		1/5	1/4	1/3	1/2
26,000	1.29	1.38	1.41	1.46	1.54
24,000	1.32	1.41	1.44	1.48	1.56
20,000	1.40	1.49	1.51	1.55	1.62
16,000	1.54	1.62	1.64	1.68	1.74
12,000	1.79	1.85	1.87	1.90	1.97

The feed enrichments required in these cycles are the following:

Lifetime (Mwd per metric ton of U)	Fuel Enrichment for Continuous Feed (wt %)	Fuel Enrichment for Fractional Feed at Shutdown (wt %)			
		1/5	1/4	1/3	1/2
26,000	3.15	3.46	3.57	3.72	3.97
24,000	3.00	3.31	3.40	3.54	3.78
20,000	2.76	3.01	3.09	3.21	3.42
16,000	2.53	2.72	2.78	2.87	3.04
12,000	2.33	2.45	2.49	2.55	2.66

Effects on fuel cost of extending fuel exposure (by increased enrichment), of reduced fabrication-plus-recovery cost, and of reduced neutron capture cross section of the cladding were examined to assist in the appraisal of the long-range potential of reactors of the EGCR type. Results show that exposures of 40,000 to 50,000 Mwd per metric ton are desirable to reduce the fuel cost and the fabrication-plus-recovery costs ($76 per kg of U) assumed for the GCR-3 study to a minimum. The minimum fuel cost for a 9.4-in. lattice pitch would be about 1.2 mills/kwhr, that is, 0.25 mill/kwhr below that for an exposure of 20,000 Mwd per metric ton, despite the higher enrichment required (about 4.5% vs 2.8%). Reducing fabrication-plus-recovery costs by one-half ($38/kg vs $76/kg) would reduce fuel-cycle costs by 0.1 mill/kwhr at 40,000 Mwd per metric ton and by 0.2 mill/kwhr at 20,000 Mwd per metric ton. There is, however, only a modest reduction in the exposure at which minimum fuel cost is achieved (i.e., to about 40,000 Mwd per metric ton). This is true because out-of-pile inventory charges and shipping costs increase with decreasing exposure and exert considerable leverage toward high exposure, despite the lower fabrication costs postulated for low exposure.

A reduced cladding cross section, if not accompanied by increased fabrication cost, would lead to a significant reduction in fuel cost. Relative to the base case, with $76/kg fabrication-plus-recovery cost, the reduction is about 0.22 mill/kwhr, and, under these circumstances, a fuel cost of 1.0 mill/kwhr or less would result. All these calculations were for the reference EGCR lattice pitch of 9.4 in.

Irradiation Experiments in the LITR. — A capsule containing pyrolytic-carbon-coated uranium carbide particles manufactured by the National Carbon Company was removed after six months of irradiation. The estimated burnup was 7.3 at. % of the uranium.

Irradiation Experiments in the ORR Poolside Facility. — Irradiation of the group V capsules was completed, and all capsules were removed for storage prior to hot-cell examination. A new capsule that contains fuel pellets and cladding from early Westinghouse production of EGCR fuel elements was installed as the first unit of the group VI capsules. The remainder of the group VI capsules are scheduled for installation in November. The intervening period will be utilized for preventive maintenance on the capsule irradiation facilities, for installation of argon flux-monitoring devices, and for extension of sweep facilities to enable the monitoring of fission-gas release from six capsules (presently four).

Irradiation Experiments in GCR-ORR Loop No. 1. — Irradiation of the element designated No. 9 at a heat rating of 46,000 Btu hr^{-1} ft^{-1} was completed as scheduled. Operation of the loop at this power density with an indicated element surface temperature of 1575°F represents the most severe conditions to date. The loop was completely stable and provided useful heat transfer information. A new test element, designated No. 8, which has a roughened outer fuel-can surface, was installed in the loop for operation during the next cycle. Test conditions are the same as for the previous experiment, except that it is anticipated that a lower gas-flow velocity will suffice to cool the element and to improve heat transfer.

GCR-ORR Loop No. 2. — Instruments and controls for GCR-ORR loop No. 2 are being checked and calibrated in preparation for the high-temperature out-of-pile shakedown runs. The UO_2-coated steel can that will be used as a fuel specimen in the first in-pile test was fabricated and is being assembled into the fuel test plug.

Plans are being made for later testing of fuel suitable for a pebble-bed reactor. In these tests it will be desirable to maintain an appropriate contact force between the spheres. The use of springs or mechanical linkages would present difficult problems, and such devices might be unreliable for in-pile loop operation. A design was therefore developed for utilizing some of the pressure drop available in the loop to provide suitable contact forces between spheres. The test section is so arranged that it can be rotated over the outlet gas port to vary the coolant flow over the test region independently of the loop flow.

Fission-Gas Release from UO_2. — When testing UO_2 of less than theoretical density for fission-gas release, it is desirable to use specimens for which the geometric surface area is a small part of the total surface area (as measured by the BET method). This condition minimizes the variation in gas release caused by variations in sample geometry.

The fractional release of fission gas during low-temperature neutron activation is expected to be proportional to the surface area per unit volume of the UO_2 sample. Puncture tests of sealed capsules yielded data on a series of samples from a batch of UO_2 pellets having a BET surface area of 8.93 cm^2/g. These data showed that for samples weighing less than 0.5 g, the fractional release is roughly proportional to the surface-area-to-volume ratio of a sphere having the same mass as the sample. This indicates that the geometric surface area is a significant fraction of the total surface area of small samples. Thus the BET surface areas, which are measured on large (approximately 300-g) batches of an oxide, are not valid when used to characterize small samples.

Correlation of Physical Properties of Coated Particles with Their Performance in Irradiation Tests. — Several sets of data on coated particles were cross-plotted to determine whether a correlation exists between measurable properties and in-pile performance. The ratio R/B (ratio of release to rate of birth) for Kr^{88} and the number of cracked coatings resulting from irradiation were plotted against several independent variables. Both of these indicators of in-pile performance showed a dependence on minimum particle-crushing load and on the minimum thickness of the pyrolytic-carbon coating, as determined by measurement of microradiographs. When R/B was plotted against the average crushing load, the dependence was less apparent. This is interpreted to mean that the performance of a given lot of particles is more dependent on the properties of the weakest particles than on the load-carrying ability of an average particle. A similar interpretation appears to apply to the correlation of performance with particle-coating thickness.

In spite of these relationships, it is possible that the performance of the particles is primarily dependent on the structure of the coating. Three of the lots tested represented the laminar, columnar, and duplex types of coatings and yielded values of R/B and number of cracked coatings that decreased in the same order. A fourth lot of particles, HTM-2, also represented the columnar structure but gave poorer performance than any of the other three lots. These particles had somewhat thinner coatings, however, which were therefore weaker; this circumstance may have masked the dependence on the structure of the coating. Additional evidence must be obtained before the performance may be definitely attributed to a particular property.

Only a marginal relationship was found when the results of neutron-activation testing (Xe^{133} released) were plotted against particle-surface contamination (α count). All other attempts to correlate performance with a particular property failed to show any relationship.

Experimental Investigation of Graphite Mechanical Properties. — A preliminary outline of the out-of-pile test program for determining the mechanical properties of nuclear-grade graphite was prepared. The objectives are to obtain, on a statistical basis, the strength, the fracture elongations, and the moduli of elasticity. Variations in these quantities as a function of position in graphite bodies will be studied and values representative of a particular graphite determined. The correlations between extrusion size and properties will be studied, and methods for relating nondestructive test results to those from destructive tests will be examined. EGCR-type, needle-coke graphite will be used as the standard.

A statistical examination of the existing data on EGCR-type graphite was used to estimate the number of tests required for reliable sampling.

Jigs and fixtures to be used for studying the rupture characteristics of graphite bodies are being fabricated. Tensile, compressive, and flexural specimens will be tested.

An experimental facility for studying in-pile creep is being constructed. Constant-stress cantilever-beam specimens loaded by pressurized bellows will be ready for insertion in a poolside facility of the ORR during the November shutdown. The primary purpose of the experiment will be to determine the influence of irradiation on the fracture elongation of graphite. The irradiation is scheduled for two reactor cycles, provided the creep rates are large enough to produce significant strains during that period.

Stress Analyses of Perforated Flat Plates. — Tests on perforated flat plates with equilateral triangular-pitch hole patterns were completed at the University of Tennessee. Two plates, both clamped around the outer edge and hydrostatically loaded, were used; one had holes of uniform size on a 1-1/4-in. spacing and the other had holes of both uniform and duplex size on a 5/8-in. spacing. Strain gages and brittle lacquer were used to obtain the stresses and stress patterns respectively. The brittle-lacquer crack pattern indicated that the principal stresses were always normal and tangential to the holes near the hole edges.

The ratio of the maximum deflection of a perforated plate to the maximum deflection of a solid plate was found to vary linearly with the ratio of the areas. The slope appeared to be a function of the hole pitch alone and not the particular hole-diameter combinations. The maximum stresses and deflections were compared with calculated values, using "equivalent solid plate" methods. For ligament efficiencies greater than 40%, an analysis developed by Westinghouse yielded results that compared most favorably with the experimental values.

Stress Analyses of Nozzle-to-Sphere Attachment. — An experimental study of the stresses at a nozzle-to-sphere attachment was initiated. A steel hemisphere, 29-3/8 in. in inside diameter and 3/8 in. in wall thickness, with a single nozzle attached at the apex, will be used to examine the stresses as a function of nozzle diameter, wall thickness, and angle of attachment. The angles will be 0, 15, 30, and 45°. The unit will be loaded with internal pressure, and loads will be applied to the nozzles. The nozzle loads include pure moments and concentrated axial and normal loads. Strain gages are being applied for the first series of studies.

STUDIES AND EVALUATIONS

Reactor Evaluation Studies. — The fuel costs of large, natural-uranium reactors for use in water desalinization plants have been calculated. The reactor concept studied was a low-temperature, low-specific-power, pressure-tube reactor cooled by light water; the moderator was either D_2O, graphite, or a D_2O-graphite mixture. The reactor power was varied from 2500 to 100,000 Mw (thermal); the reactors were assumed to operate on a continuous countercurrent fueling schedule with uranium metal. Assuming

cost bases associated with a municipal plant, total fuel cycle costs of about 0.1 mill/kwhr (thermal) (about 3¢/million Btu) appear attainable so long as fuel exposures of 7000 Mwd/ton are permissible from a radiation-damage viewpoint, fabrication plus chemical processing costs are about $16/kg, and the plutonium credit is about $6/g.

An evaluation of the Babcock and Wilcox consolidated nuclear steam generator was begun. This concept considers a pressurized water reactor designed for maritime applications; boiling is permitted in the core region. With three radial zones of different fuel enrichments, it was found that boiling took place only in the middle fuel zone (this zone was about half the volume of the core). In this region, boiling began after the fluid had passed about two-thirds through the core; when leaving the core region, about 10 vol % vapor was present in fluid leaving the outer middle zone, and about 25 vol % vapor was present in fluid leaving the inner middle zone.

REACTOR FUELS AND MATERIALS DEVELOPMENT

Nondestructive Test Development. — X-ray attenuation techniques are being investigated for application to inhomogeneity determinations in thin fuel plates. A gamma-scintillation system seems to offer the most versatility. Commercial x-ray equipment is being used as the radiation source. Precautions have been taken to control the input voltage to the unit for the most stable operation. The x-ray beam passes through an array of collimators both before and after the fuel-plate specimen. These collimators or masks serve to restrict the area of interrogation on the plate and also prevent scattered irradiation from reaching the sodium iodide crystal used as the detector. The crystal is optically coupled to a photomultiplier tube, the output of which is fed to a modified strip-chart recorder. The fuel plate can be translated both longitudinally and transversely across the collimated beam, allowing complete X-Y scanning of the fuel core section. Several materials are being studied for reference standards to equate fuel concentration changes to x-ray transmission variations. These materials include uranium-aluminum metallic foils, U_3O_8 in aluminum cermet foils, and equivalent attenuation thicknesses of steel and aluminum. The uranium-bearing standards can be used through broad energy ranges, but some difficulty has been experienced in obtaining homogeneous foils. Integration, however, has allowed good attenuation relations to be established. The equivalent attenuation thicknesses of steel and aluminum will be valid only near the reference energies, but the homogeneity and machinability of such materials will allow single-point standardization without integration. These techniques will be applicable for the determination of uranium concentration change in any flat-plate fuel element, such as those for the HFIR, ATR, ORR, MTR, etc.

Mechanical Properties. — Previous studies have shown that type 304 stainless steel is carburized as a result of exposure to flowing CO_2 at elevated temperatures. In an effort to better understand this phenomenon, a series of Fe-Ni-Cr alloys was prepared to study the influence of composition. The alloys were arc-melted and were nominally Fe, Fe—1% Cr, Fe—3% Cr, Fe—10% Cr, Ni, Ni—5% Fe, Ni—15% Cr, and Ni—15% Cr—5% Fe. It was

found that the alloys which did not contain chromium were decarburized after exposure to CO_2 at elevated temperatures. However, the alloys which contained chromium were carburized, the extent of carburization being proportional to the chromium content.

Further studies on a vacuum-melted 18% Cr—8% Ni—Nb stabilized steel have shown that minor impurities may also be important in the carburization of these materials in CO_2. The vacuum-melted steel, which has the same nominal composition as type 304 stainless steel with the exception of the stabilizing element, was considerably more resistant to carburization and oxidation in CO_2 than was type 304 stainless steel. The absence of certain minor impurities in the vacuum-melted steel may result in the formation of a more adherent oxide which inhibits carburization and further oxidation.

Materials Compatibility Studies. — Compatibility studies with the lithium-niobium system have continued. Single-crystal specimens of niobium containing 1200 ppm oxygen were exposed to lithium vapor at 540°C for 0.5 and 30 hr. Maximum attack was 6 mils in the 0.5-hr test and 12 mils in the 30-hr test. Corrosion was only slightly less severe than might be expected if the specimen had been exposed to liquid lithium under similar conditions.

The effect of dislocation density in niobium on corrosion was studied with single crystals having the same orientation but with a factor of 10 difference between their dislocation densities. Specimens were exposed to lithium for 2 hr at 500°C. Metallographic examination showed 11 mils penetration along closely spaced crystallographic planes in the specimen containing the lower dislocation density and 5 mils penetration along more widely spaced planes in the other sample.

These results suggest that the presence of dislocations in niobium may affect the oxygen distribution in the material, which, in turn, can affect the corrosion process. These studies will be continued.

A series of experiments have been conducted in order to determine the effect of heat treatment on the subsequent corrosion resistance of oxygen-contaminated niobium to lithium. Five specimens were prepared from a single crystal in order to eliminate the effect of grain orientation on lithium penetration. The specimens were heat-treated at 20, 370, 540, 815, and 1040°C for 100 hr. Each specimen was then tested in lithium for 20 hr at 370°C. Similar depths of penetration were observed on all specimens, indicating that prior heat treatment does not significantly alter the corrosion behavior of oxygen-contaminated niobium exposed to lithium.

Fuel Element Development. — A novel method has been developed for fabricating dense uranium monocarbide at very low temperatures. Liquid-phase sintering is used to lower fabrication temperature, yet the final structure is free of the weakening, eutectic constituent ordinarily associated with this method of fabrication.

By use of 5 to 10 wt % UAl_2 as a fabrication aid, the aluminum is volatilized out during sintering, while the residual uranium is combined with carbon present in the system. For material fabricated at 1300°C, densities as high as 93% of theoretical are achieved. The microstructure is free of secondary constituents, and chemical analyses show 95% and higher efficiency in aluminum removal.

REACTOR COMPONENTS DEVELOPMENT

Reactor Controls Development. — The flexible pulse preamplifier and fission chamber assembly previously described (ORNL-3211, p 13) was operated successfully under field conditions. The pulse pickup noise from all sources was reduced, by correct circuit design and layout techniques, to the point where no noise pulses larger than one-third the alpha-particle pulse height were observed. The noise pulses are, therefore, much smaller than neutron pulses and cannot interfere with the use of this instrumentation for reactor control. One assembly with its drive mechanism was installed for use in the ORR, and others will be installed so that field experience can be gained.

Development of a new pulse-channel instrument for reactor control use has reached the stage of successful breadboard demonstration of the circuit design. This instrument (comprised of pulse amplifier, discriminator, reactor power and period computers, and servo amplifiers) and the flexible preamplifier and chamber assembly constitute the new wide-range instrument channel [Trans. Am. Nuclear Soc. 3, 454 (1960)], which is the startup system in the "second generation" of ORNL reactor control instrumentation.

A large magnet was developed and tested for use with an experimental scram latch. With a force of 800 lb, holding a load of 400 lb, the release time was 10 msec.

POWER REACTOR FUEL PROCESSING

UO_2 Dissolution. — The instantaneous dissolution rate of UO_2 in boiling nitric acid that contained sodium nitrate or lithium nitrate was the same as that in nitric acid alone of the same nitrate concentration. Mixtures studied varied from 10 to 80% salt and from 2 to 13 N HNO_3. The addition of nitrite did not vary the dissolution rate, probably because the nitrite is lost from the boiling solution. The addition of 0.1 N HF decreased the dissolution rate when the HNO_3 was ≥ 5 N and increased the rate when the HNO_3 was less than 5 N.

Protactinium Studies. — Unfired Vycor glass powder was used to remove protactinium from solutions simulating those obtained in the dissolution of fully irradiated, 30-day-decayed, Consolidated Edison Thorium Reactor fuel. This solution was 0.5 M in thorium, 11 M in HNO_3, approximately 0.2 M in aluminum, and 0.04 to 0.1 M in F^-, and the Pa^{231} concentration was 120 mg/liter. More than 99% of the protactinium was adsorbed on passing 130 ml of solution at a flow rate of 1 ml cm^{-2} min^{-1} through a column about 0.6 cm in diameter and 25 cm long, containing 5 mg of 60- to 80-mesh unfired Vycor glass. In a similar column experiment, 10 mg of Pa^{231} was adsorbed on 1 g of unfired Vycor. After washing with 11 M HNO_3 the column was eluted with 0.5 M oxalic acid. More than 99% of the protactinium was recovered at a concentration 12 times the feed concentration; the thorium decontamination factor was 6300.

There was no change in the concentration of a Pa^{231} solution after one week of contact with type 347 stainless steel (2 cm^2 of stainless steel per milliliter of solution). The solution was 0.5 M in thorium, 11 M in

HNO_3, and contained a trace of fluoride; the Pa^{231} concentration was 75 mg/liter.

Shear-Leach Process. — Three shear-leach runs were made with ORNL Mark I fuel elements (unirradiated stainless-steel-clad UO_2) to evaluate the leaching characteristics of fuel sheared to lengths of 1/2, 1, and 1-1/2 in. respectively. Each of the first two runs used eight batches of sheared fuel; each batch contained about 24.4 g-moles of UO_2 and represented 8 in. of fuel assembly. The last run used nine batches; each batch contained about 26.6 g-moles of UO_2 and represented 9 in. One batch was fed to the rotary-screw leacher every hour and contacted countercurrently over a 4-hr period with 7 M HNO_3 at 90 to 95°C at an HNO_3/UO_2 mole ratio of 4.

These runs indicate that apparent steady state is obtained in the leacher in 5 to 6 hr, that 66% of the UO_2 dissolves in 1 hr, and that the remaining UO_2, introduced to the leacher as fines, tends to cake and dissolve very slowly. For fuel sheared to 1/2-, 1-, and 1-1/2-in. lengths, the steady-state dissolution rate was 20.8, 22.6, and 22.9 g-moles of UO_2 per hour, and the UO_2 fines accumulation rate was 3.6, 1.8, and 1.5 g-moles of UO_2 per hour respectively. Since these runs show that UO_2 fines crust or cake, thereby blocking access of the leach acid to the fines beneath the crust, shearing to greater lengths, thus producing less fines, reduces but does not eliminate the amount of undissolved UO_2.

The steady-state uranium concentration in the leach-product solution was 500 to 520 g/liter, and the HNO_3 concentration was 1.4 to 1.5 M. Dilution of this product with the drain liquor from the leacher produced a satisfactory Purex solvent extraction feed.

Stainless-steel-cladding fines, $\leq 1000\ \mu$ in diameter, dissolved completely in the nitric acid; this represents about 0.06% of the cladding entering the leacher.

Hydrolysis of Uranium Carbides. — The heat treatment of uranium-carbon alloys with a C/U gross atom ratio of 2 at 2000°C for 6 hr and at 1100 to 1260°C for 238 hr had little effect on the combined C/U atom ratio (1.84), the x-ray powder pattern, or the hydrolytic properties. Hydrolysis in water at 80°C yielded about 40 ml (STP) of gas per gram of sample; the off-gas contained approximately 40% hydrogen, 16% methane, 27% ethane, 6% C_3- to C_8-saturates, and 9% unsaturates. Of the total carbon in the alloys, about 35% was evolved as gaseous products, and 22% was converted to insoluble wax.

The microstructure of the as-cast UC_2 sample showed a principal dicarbide phase and flakes of graphite; both heat-treated specimens had an additional third phase of parallel plates throughout the dicarbide grains. Since heat treating did not affect the chemical properties, this phase was probably soluble in the dicarbide when quenched from the melt.

Corrosion. — "A" nickel exposed to nitrogen-saturated CCl_4 (at room temperature) at approximately 770°C corroded at rates of 565 to 613 mils/month for 24-hr exposures. "A" nickel is the most corrosion-resistant metallic material thus far tested in this environment.

Pyroceram 9608 reacted with molten aluminum at approximately 700°C. Three of the four specimens fractured during a 24-hr test, and the fourth specimen cracked on application of very slight stress. "A" nickel suffered extensive local attack in contact with molten aluminum, but some

areas became covered with a black tenacious material that resisted further attack.

In glass corrosion studies no boron was leached from glass Raschig rings containing 3.79 wt % boron after five weeks of contact with solutions containing 100 g of uranium per liter, 0.2 to 5 g of thorium per liter, and 0.1 to 0.04 M HNO_3. Samples having 110 cm^2 of surface area were exposed to 150 ml of solution at room temperature, or at room temperature to 65°C. The boron content of the final solutions varied from 0.02 ppm less to 0.04 ppm more than that of control solutions which had not contacted the Raschig rings.

Solvent Extraction. — It was found possible to calculate the distribution coefficient for the extraction of dilute uranium from acid-deficient aluminum nitrate solution into 2.7% di-sec-butyl phenyl phosphonate (DSBPP) in diethylbenzene from pH data on the aqueous feed solution without knowing either the aluminum concentration or the acid deficiency of the solution. This relationship is

$$\log K_d = 7.04 - 2.38 \text{ (pH of feed)} .$$

No other single variable has been found to correlate with the distribution coefficient. These data indicated a first-order dependence of uranium hydrolysis on the hydrogen ion concentration in the systems studied.

The efficiency of sieve-plate pulsed columns for a process using DSBPP in diethylbenzene for the separation of uranium from thorium was (expressed as HETS): 2.1 ft at a pulse frequency of 70 cpm and 4.0 ft at 50 cpm, for uranium extraction; 4.1 ft at 70 cpm and 4.6 ft at 50 cpm, for thorium scrubbing; 4.2 ft at 70 cpm and 6.7 ft at 50 cpm, for uranium stripping.

Fluoride Volatility Processing. — In an extension of tests conducted at Battelle Memorial Institute (BMI) and previously reported (ORNL-3307, p 19), the corrosion of various Ni-Fe-Mo experimental alloys was compared with that of INOR-8 and alloy 79-4 during exposure to HF for 72 hr in 50.5-37.0-12.5 mole % $NaF-LiF-BeF_2$ at 650°C. For an undetermined reason, this melt was even more corrosive than previous ones of the same composition. Metallography showed that INOR-8 and the 85-5-10 wt % and 80-18-2 wt % Ni-Fe-Mo alloys had been attacked the least, in contrast with the excellent showing of alloy 79-4 in the earlier tests:

Alloy	Composition (wt %)			Maximum Rate of Interface Penetration (mils/month)		
	Ni	Fe	Mo	Wt-Loss Data	Micrometer	Metallography
INOR-8	71	5	16[a]	27.5	115	100
79-4	80	16	4	30.0	380	375
2	85	5	10	18.2	60	100
31	85	13	2	18.0	80	125
32	80	18	2	20.9	75	100
33	80	5	15	17.9	110	135
34	75	15	10	19.1	75	135
35	75	20	5	26.2	260	293
36	80	12.5	7.5	20.6	205	185
37	80	16	4	21.3	170	188
38	85	10	5	19.9	105	115

[a]Plus 7% Cr.

In simulation of conditions that would be encountered in the burning and fluorination of graphite-base fuels (e.g., the High-Temperature Gas Reactor and the Pebble-Bed Reactor fuels), various nickel-base alloys were cyclically exposed at BMI for 1 hr to CO-CO_2-O_2 (5-65-30%) at 700°C, and for 1 hr to F_2 at 400°C, with 1-hr helium flushes during temperature transitions. After 20 cycles, maximum penetration rates in mils/month were: "L" nickel, 1.6; Duranickel, 3.8; Monel, 8.2; alloy 79-4, 11.5; and INOR-8, 5.4. Inconel-X and Inconel rates were 31.4 and 14.9 mils/month, respectively, after six cycles.

Fluoride Volatility Pilot Plant. — The system is being decontaminated with aqueous solutions prior to a corrosion check and several plant modifications. The modifications are desirable before short-cooled fuel is processed.

The problem of salt recycle as a means of lowering overall salt cost was studied by a series of dissolutions of Zircaloy-2 with HF in molten NaF-LiF-ZrF_4; an INOR-8 bench-scale dissolver vessel was used. Ten tests were made with about 35 mole % ZrF_4 in the initial charge, with the addition of NaF-LiF to each product salt to bring the composition back to about 35 mole % ZrF_4 for the following test. Sampling of the product salt indicated practically no corrosion-product buildup. Dissolution rates were in the same range that would have been expected with fresh salt.

THORIUM UTILIZATION PROGRAM

Planning and Evaluation Studies. — Optimization of the fuel processing rate with respect to fuel-cycle cost in the Molten Salt Converter Reactor disclosed that the optimum rate was 1.7 ft^3 of fuel salt per day (about 50 kg of Th per day), and the corresponding fuel-cycle cost was about 0.74 mill/kwhr (electrical). Studies of gamma heating in the reactor vessel did not lead to changes in vessel design. Results from Equipoise-3 and 2DGH calculations indicated less than 1.5 w/cc heating in the core hold-down plate and less than 0.6 w/cc in the vessel wall.

In order to determine the magnitude of the fuel-cycle cost in a reactor modeled after the Babcock and Wilcox Spectral Shift Control Reactor, pertinent data were taken from report BAW-1241 and used to develop fuel-cycle costs. Using the bases given in the AEC Cost Handbook, a preliminary fuel-cycle cost of 1.68 mills/kwhr (electrical) was obtained. Preliminary nuclear calculations for a thorium-uranium reactor have yielded results substantially in agreement with those obtained by Babcock and Wilcox for the same reactor.

A machine program is being developed which will enable ready modification of the MODRIC multigroup cross sections; this program provides self-shielding factors for the fast-group cross sections and computes an effective thermal-group cross section based on a thermal spectrum calculation. Programming is complete and the code is ready for machine testing.

Testing of Thoria-Pellet and -Suspension Fuels. — Capillary viscometer tests were made at room temperature on aqueous slurries of several densities prepared from irradiated thoria-urania solids recovered from the first in-pile slurry loop experiment, designated L-2-27S. The material

recovered from the loop dump tank was largely composed of solids drained from the loop at the end of the run, together with a lesser quantity of purges removed from the loop while taking samples at various times during in-pile operation. Viscosities were also determined on slurries prepared from the original unpumped thoria-urania batch (DT-22) used to prepare the load for the in-pile loop experiment.

The irradiated material, which had been exposed to a dose of 1.6×10^{19} nvt and developed 7×10^{16} fissions per g of solids, had an average particle size of 0.76 μ, while the original DT-22 solids had an average particle size of 1.7 μ.

Yield stresses of irradiated and unirradiated samples of various densities generally agreed with and did not exceed the correlation formula $\tau_y = 210\phi^3/D^2$, where τ_y is yield stress in lb_f/ft^2, ϕ is volume fraction of solids, and D is mean particle diameter in microns. No effect of irradiation on yield stress was evident except that associated with particle size changes.

The second in-pile slurry loop, designated 0-1-28S, was partially dismantled. The connector section of the apparatus which joins the loop package to the shield plug was inspected. Two detached and burned wires were found, one in the loop circulating pump circuit and the other in the pressurizer heater circuit. These wires had evidently shorted and then opened in the connector section. It was concluded that the operational failure of the loop components resulted from a malfunction in the connector region rather than any intrinsic design faults in the loop equipment.

Tests to observe the possibility of controlling the fluidization of a thoria pellet bed at elevated temperatures in the core of an in-pile pellet loop have been conducted in an out-of-pile mockup. The unfluidized cylindrical bed of P-140 thoria pellets was 1-1/2 in. in diameter by 1-1/2 in. high. The loop was operated at 280°C. Bed expansion was observed by x-ray photography. Operation of the circulating pump at 80 cycles, corresponding to a superficial flow rate of 3.4 fps, resulted in fluidization to a bed expansion of approximately 25%. The bed appeared unexpanded with pump operation at 60 cycles (flow rate 2.4 fps). This implied that the bed design under consideration was feasible for in-pile application, with respect to operation with the bed alternately fluidized and unfluidized.

Engineering of Thorium Reactor Systems. — Saturated and subcooled pool boiling burnout tests were made with aqueous thoria slurries and with water. Whereas the water results obtained agreed with literature correlations, the presence of 1800 g of ThO_2 per liter reduced the saturated burnout heat fluxes to about two-thirds of the predicted values and also reduced the effectiveness of subcooling in preventing burnout. This confirms the earlier observation that the slurry burnout in forced convection is about one-half that predicted for a pure liquid of similar properties.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — Design of the reactor and auxiliary piping is 90% complete. Remaining design work consists primarily of revisions to existing drawings and incorporation of development information into the design.

All materials are on hand for the fabrication of the MSRE components except the graphite for the moderator. Delivery of this item is expected in November. Component fabrication is progressing according to schedule.

The outside modification of Building 7503 is approximately 95% complete. The interior modification work is approximately 85% complete. Due to delay in the delivery of expansion joints for the containment-cell penetrations, the completion of the work inside the building will be delayed approximately one month (from October 1 to November 1).

Metallurgy. — Two isothermal tests were completed (at 600 and 700°C) to determine the corrosive effects of CF_4 vapor on INOR-8 immersed in molten fluoride salt and on INOR-8 held in the vapor region over the fluoride salt. Test specimens at 600°C, which were removed at 100-hr intervals to 1000 hr, underwent negligible weight changes and exhibited no consistent pattern as to weight loss or gain. Metallographic examination revealed no evidence of surface change.

At 700°C, similar specimens immersed in salt up to 700 hr exhibited some weight gain (1–3 mg/cm^2), while the vapor-phase specimens showed a small weight loss. Metallographic examination revealed some light pitting and possible carburization at the surface.

The results of these and earlier experiments indicate that CF_4 vapor does not react appreciably with INOR-8 at 600°C, but that minor attack may occur at 700°C.

Chemical Development. — The density of $LiF-BeF_2-ZrF_4-UF_4$ (66.85-29.0-4.0-0.15 mole %), a mixture which is under study as a fuel for the MSRE, was determined by measuring the buoyancy of a platinum bob in the range 550 to 800°C. The density in grams per milliliter is $2.21 - 5 \times 10^{-4}T(°C)$ within an estimated accuracy of better than 1%; these measurements are about 3% higher than the values obtained from a method of estimation which relies on the constancy of partial molal volumes at a given temperature.

The manometer vapor pressures of MSRE fuel (tentative top position 66.85-29.0-4.0-0.15 mole % $LiF-BeF_2-ZrF_4-UF_4$) have been measured over the range 1050 to 1300°C. The pressure-temperature data are represented by $\log P(mm\ Hg) = 9.56 - 11{,}400/T(°K)$. The extrapolated vapor pressure at 704°C, the design temperature of the MSRE, is 0.0078 mm; that at 662°C, the operating temperature, is 0.0024 mm. If it is assumed that the 5000 liters/day of off-gas leaving the pump bowl is 10% saturated, approximately 20 mg of fuel solvent would be distilled away each day. This amount is low enough to suggest that deposits that have been noted in vapor regions arise almost entirely from other transport mechanisms, such as mist, due to mechanical entrainment.

Preparation of MSRE Materials. — Structural modifications to the fluoride fuel production facility were completed by September 15; revisions to the process control equipment should be completed by October 15. The production of purified fluoride melts for MSRE operations is now scheduled to begin in November 1962.

The tentative specifications for the purchase of beryllium fluoride were reviewed with commercial vendors. Through a cooperative effort involving the two major sources of supply, samples of BeF_2 from their current and developmental production operations were examined for chemical purity. Both companies have initiated changes in their production processes in hopes of being able to meet MSRE specifications. Final specifications have been submitted to project and purchasing personnel for comment before production requirements for 10,000 lb of BeF_2 are advertised.

Two treatments of oxide-contaminated salt from the Engineering Test Loop with mixtures of anhydrous hydrogen fluoride and hydrogen provided additional opportunities with large equipment to improve techniques for mixing these gases at known concentration ratios and at constant flow rates. Methods for sampling and analyzing the effluent gas from the reaction vessel during hydrofluorination were also improved. These techniques will also be used in the production of fluoride mixtures for the MSRE.

Radiation Effects. — Elemental fluorine at pressures as high as 35 atm was generated in some of the rapidly frozen irradiated capsules (7% burnup at 750°C) that were included in tests of the radiation-induced evolution of CF_4 from MSRE fuel and graphite (test 47-4). The lack of visible evidence of corrosion of the inside walls of the INOR capsule and the presence of the customary UF_4-containing phases in the fuel strengthen the conclusion that the fluorine was formed from radiolysis in the frozen salt after shutdown. The evolved fluorine corresponds to 1% of the fluoride content of the capsule, or to approximately all of the UF_4. If, in some unlikely manner, this fluorine had remained apart from the fuel melt at operating temperatures, the uranium would have been in a highly reduced state, or even altogether absent from the fuel, having been reduced to the metal. Since another capsule, of different configuration, in the same exposure did not generate a measurable amount of elemental fluorine, the undesirable effects may be strongly dependent on some such incidental factor as the freezing history of the capsule. Also, since the fluorine apparently recombines as fast as it forms at elevated temperatures, the accumulation after shutdown could presumably be avoided by merely holding the fuel molten until a substantial portion of the after-heat has decayed.

Irradiation Tests. — The equipment for the irradiation experiment ORNL-MTR-47-5 (see ORNL-3369, to be issued), designed to measure the CF_4 content of the off-gas from fissioning MSRE fuel in contact with submerged graphite, has been fabricated and is at the MTR. The installation of the gas collection system has been in progress since September 4, but the availability of manpower at the MTR has necessitated postponement of the scheduled insertion from September 17 to October 8.

The off-gas collection system is being reviewed from the standpoint of material compatibility, in light of the fluorine gas found during the current postirradiation examinations of the capsules from the preceding experiment 47-4 (see ORNL-3369, to be issued, and ORNL-3282). Fluorine is believed to accumulate when the reactor is shut down and to recombine with the salt on reheating. Stainless steel lines in the high-temperature region of the purge system may be exposed to fluorine for brief periods. The experiment is considered safe, but it may be useful only for obtaining qualitative information about the formation of free F_2. Nickel collection bottles, prepassivated with fluorine, are being prepared to replace the stainless steel bottles. The amount of corrosion to be expected in the stainless steel gas lines in the high-temperature portion of the experiment is being determined by tests on samples obtained from spare capsule parts.

MSR Processing. — The process flowsheet was approved, and detailed design of the fuel processing system was begun. The fuel storage tank will be provided with cooling-water coils to enable oxide removal from

fuel after only a few days' decay. Completeness of oxide removal will be determined by condensing and sampling the $HF-H_2O$ stream. Excess fluorine from the UF_6 volatilization operation will be disposed of by reaction with SO_2 to form SO_2F_2, a relatively inert, nontoxic gas.

Analysis of Radioactive MSRE Fuels. — The following equipment was fabricated and will be assembled in the cell for checkout: a modified mixer mill for use in homogenizing the fuel; a cell for use with the alternative polarographic method of determining uranium; an automatic pipetter, a filter photometer, and other equipment for the determination of reducing normality.

A prototype of the cell equipment is being constructed for use in removing the sample-filled ladle from the transport container and in reloading the transport container with an empty ladle.

Design, procurement, and fabrication of the other analytical equipment necessary for analyzing the sample in the cell are in progress.

An amperometric method for the determination of zirconium with cupferron was evaluated for application to radioactive molten fluoride salt samples.

The titration is performed in a coulometric cell with a stirred mercury pool, which serves as the working electrode. A small platinum wire serves as the "reference" electrode. After deoxygenation of the sample solution, cupferron (previously standardized and deaerated) is fed in through a constant-flow buret. The trace of the current drawn by the polarized mercury electrode shows a right-angular inflection at the end point. The method is particularly suitable for solutions containing fluoride ions.

The procedure is now being adapted to hot-cell use, in which a check will be made of possible radiation effects.

ADVANCED REACTOR DEVELOPMENT

Advanced Test Reactor Corrosion Program

In a continuation of the study of the corrosion of aluminum through which heat is being passed, a series of experiments was conducted to determine the effect of heat flux on the buildup of corrosion products on the aluminum. In these tests the cooling water flow rate and pH were constant at 45 fps and 5.0 (with HNO_3). The aluminum surface temperature at the axial midpoint of the specimen was maintained at 400°F by changing the cooling-water temperature from test to test, and the test time was 142 hr except for the test conducted at 2×10^6 Btu hr^{-1} ft^{-2}, which lasted only 92 hr. Essentially the same corrosion product thickness formed on the specimens subjected to heat fluxes of 1.0, 1.5, and 2.0×10^6 Btu hr^{-1} ft^{-2}, the thicknesses ranging from 1.3 to 1.4 mils. At the 0.5×10^6 Btu hr^{-1} ft^{-2} level only 0.8 mil of oxide developed. Some data, previously obtained over a limited heat flux range, indicated that only surface temperature, not heat flux, determined the rate of corrosion product formation. This series of tests, however, clearly shows that heat flux per se is an important variable and must be investigated more thoroughly.

High Flux Isotope Reactor Design and Development

Metallurgy. — In the recently welded lip-type fuel element, placement of Teflon strips between the composite fuel plates during assembly has proved to be effective in repairing out-of-tolerance spacings. More conclusive evidence of the stability of these repairs will depend on subsequent stress relief of the fuel element.

The concept of assembling plates by sliding them between two grooved tubes now appears most promising. In the first experiment of this type, unfueled plates were assembled with little difficulty in a 369-plate array and joined by circumferential welds at 1-in. intervals along the length of the fuel element. Measurements revealed that all spacings were in tolerance. Shrinkage and warpage due to welding were negligible.

Studies on the compatibility of cadmium compounds as a burnable poison with aluminum have been concluded. A cadmium-containing stabilized glass supplied by Bausch and Lomb proved most compatible throughout the thermal treatments in the manufacture of fuel plates. No significant loss of cadmium was indicated.

The feasibility of preparing by powder metallurgy techniques both the poison-bearing filler and the fuel piece as an entity in a single pressing operation has been demonstrated. Examination of the fuel contour in the pressed compact revealed a meniscus effect at the edge of the compact. Measurements of the fuel contour in the fabricated composite plate showed that the contour produced by this single-step process compared very favorably with that produced by the previously developed forging process for making fuel cores and filler pieces.

Several relatively large cores containing 40 vol % tantalum dispersed in aluminum and 40 vol % rare earth dispersed in aluminum have been prepared with no apparent difficulty. These will be subsequently used in the development of alclad plates for the control rods.

The compatibility of 40 vol % Eu_2O_3 in nickel was demonstrated. Metallographic examination of a rolled nickel-clad plate of this material did not reveal any reaction of Eu_2O_3 with nickel. These results indicate that this materials system may be a suitable backup if needed in the control rod.

Complete nondestructive inspection of each HFIR element can be a time-consuming, expensive operation. The program to detect fuel segregation in regions as small as 5/64 in. in diameter has therefore been increased in scope to develop rapid inspection equipment for x-ray attenuation techniques. Foil standards containing U_3O_8 in aluminum have been prepared and examined. Although detection of fuel in the required small areas appears feasible, some difficulty has been experienced in obtaining foils with the desired homogeneity. Development has been limited by the response time of the recorder, and the highest scanning speed achieved is only 16 in./min. It is anticipated that these difficulties will be overcome with additional instrumentation and modification of equipment.

Design and Analysis. — The steady-state hot-spot analysis procedure was used to produce curves showing the effects of operating pressure and core flow rate on steady-state burnout power level and the onset of hot-spot nucleate boiling. These curves will be used in evaluation of the HFIR control and safety systems, particularly in regard to pump coastdown and startup accidents.

As a result of critical experiment data and nuclear studies, the active length of the control rods was reduced from ~6 to ~4 ft. A revised layout drawing of the control-region structural assembly was made, to indicate the benefit that would result from a corresponding decrease in length of this assembly.

Instrumentation. — Analog studies are under way to examine the behavior of the present HFIR model during a fast transient due to insertion of positive δk at design point. In addition, the same model will be examined for safety and controllability when undergoing relatively slow transients, such as pump coastdown and slow rod motion.

Engineering Development. — The HFIR mockup has now been operating intermittently for one month at various flow rates up to 100% of design flow. Tests are being conducted to determine flow distribution through the various reactor coolant flow paths. Concurrently with flow measurements, strain gages placed in strategic locations are being monitored to detect vibration and deflections which may be flow dependent.

Preliminary operation of the mechanical drive system for the inner and outer control rods has been erratic, due to sticking drive rods and failure of the rods to scram satisfactorily. The mockup has been dismantled to determine the cause of these problems, and steps are being taken to correct these conditions before resuming operations.

Critical Experiments. — The two-section control plates have been calibrated for various displacements starting from four delayed-critical positions, which were achieved with different boron concentrations in the moderator. Additional power distributions were measured for each of these critical conditions.

The control plates have now been further changed to include a 4-3/4-in.-long middle section of moderate neutron absorbing power, the so-called grey section. Control plate calibrations and measurements of power distributions with these control plates have been started.

PHYSICAL RESEARCH PROGRAM

Reactor Operations. — During the first 37 weeks of 1962 the ORR was in operation 83% of the time.

An aluminum-cadmium shim rod has been installed in place of one of the fuel-cadmium shim rods at the LITR. If this proves successful in operation, it will eliminate the need for replacing the shim rods.

A demineralizer and filter system was placed in service at the ORNL Graphite Reactor canal. The ion columns remove contamination from the water as it is recirculated. The discharge of radioactive waste water, which previously accounted for a large fraction of the Sr^{90} in the process waste system, is also eliminated.

Waste Disposal. — Construction of trench No. 7 was completed on schedule; the trench is now being pretreated with caustic prior to placing it in service for intermediate-level waste disposal.

The liquid-waste monitoring system installation has been completed. The 3039 stack monitoring system installation is approximately 95% complete but is progressing very slowly for lack of craft assistance.

Hot-Cell Operations. — Operations in the hot cells were routine. Examination of fuel element S-81 from the SM-1 reactor at Fort Belvoir was completed. One standard-UO_2 meltdown and one UC_2-graphite (embedded UC_2) meltdown were disassembled and examined. Several ORR and LITR capsules containing UC_2 and UO_2 fuels were examined. Also, two ORR poolside experiments containing tube-burst specimens were disassembled and examined.

PHYSICS AND MATHEMATICS RESEARCH

Charge Spectrometry. — The effect of recoil energy on electron shake-off following β^- decay is usually negligible. However, the large recoil experienced in the β^- decay of He^6 has made the measurement of this effect possible. A calculation as to the probability of shaking off one electron has been made for comparison with the experimentally determined value. This calculation is based on transition probabilities as first given by Levinger [Phys. Rev. 90, 11 (1953)]:

$$P_{i \rightarrow f} = \left| \int \psi_f^* \, e^{-iKz} \psi_i \, d^3r \right|^2 ,$$

where ψ_i is the initial state of He^6, $\psi_f^* e^{-iKz}$ is the final state of the lithium ion recoiling in the z direction, and K is the wave number of an electron corresponding to the nuclear recoil velocity V. On expanding e^{-iKz}, the first term is unity, which gives the simple overlap integral associated with pure Coulomb shake-off. The remaining terms in the expansion contain powers of K and contribute to recoil shake-off. Calculations of probabilities of transitions to virtual and to continuum

states were made. The probability for losing one electron due to recoil energy is 0.173%. Considering the approximations, necessary in several steps of the calculation, comparison with the experimental value of (0.30 ± 0.05)% is satisfactory.

Neutron Diffraction. – Neutron diffraction measurements were made on polycrystalline samples of a 75% Pd–25% Mn alloy. It was found that long-range positional order could be obtained by appropriate heat treatment and that this order was of the one-dimensional antiphase domain type with a period of 4 cubic unit cells. Below 170°K, antiferromagnetic reflections were observed for this ordered alloy. A magnetic structure is proposed which satisfactorily accounts for the observed magnetic reflection intensities. On the basis of this structure the atomic magnetic moments are 4.0 ± 0.2 μ_B/Mn and 0.2 ± 0.1 μ_B/Pd.

Theoretical Physics. – An analysis of the differential cross sections for $V^{51}(n,d)Ti^{50}$ at 14.4 Mev for the 0^+, 2^+, 4^+, and 6^+ excited states of Ti^{50} has been done by using the distorted-wave Born approximation. The data are consistent with the shell model predictions for $(1\ f_{7/2})^3$ when seniority is a good quantum number. Small amounts of $\ell = 1$ pickup to the 2^+ and 4^+ states cannot be excluded.

Fast-Chopper Time-of-Flight Spectrometer. – Measurements were done in the energy range from 30 to 10,000 ev with a time resolution of 10 nanoseconds/m, using the ORNL fast chopper and the 180-m flight path. The metallic sample thicknesses were 0.20 and 0.15 in. for Sn^{112} and Sn^{114}, respectively, and 1.0 in. for Sn^{122} and Sn^{124}. The sample of Sn^{115} was an oxide powder and had a thickness equivalent to 0.057-in. metal. In this energy range, 12, 7, 4, 6, and 5 resonances in $Sn^{112,114,115,122,124}$, respectively, were observed. Measurements were also made with a 45-m flight path on 4-in.-thick natural tin and all ten enriched isotopes from 50 to 0.2 ev with an energy resolution of 1.5%. Even below 30 ev several new resonances, which had not been previously observed, were found. Detailed corrections for "missed" resonances must be made to obtain average level spacings and strength functions.

Equipment for the study of gamma-ray spectra from low-energy neutron capture was installed at the ORR. The fast chopper, with the high-intensity rotor, is used to provide a neutron beam, and the gamma-ray detector is a heavily shielded 9 × 12 in. NaI crystal. A flight path of 5 m allows the determination of neutron energies with an energy resolution of approximately 15%. Two types of measurement have been made in initial experiments: (1) the study of thermal neutron capture in small quantities of separated isotopes; (2) the study of capture in the resonance region. For example, thermal capture spectra have been observed for nine of the isotopes of tin. Each spectrum shows a number of prominent gamma rays as well as many that are poorly resolved. Capture in copper has been compared for the first two resonances, at 227 ev (Cu^{65}) and 580 ev (Cu^{63}), and for the nonresonant capture at various energies below the resonances. The nonresonant spectra are very similar and differ from the spectrum for the 580-ev resonance, which is similar to that observed at higher neutron energies. These results are of interest for testing the suggestion that direct capture is important in copper.

High-Voltage Experimental Program: Oak Ridge Tandem Accelerator. — The voltage rating of the tandem accelerator has been increased from 6 Mv to 7.5 Mv; thus the proton energy range has been extended from 12 Mev to 15 Mev and data have been taken at 15 Mev. The oxygen ion energy range is increased from 42 Mev to 52.5 Mev. The accelerator has been used to calibrate special fission detectors, with bromine ions as artificial fission fragments with energies ranging up to 100 Mev. For heavy-fragment calibrations, iodine ions will be used.

High-Voltage Experimental Program: Neutron Capture. — Further measurements of 30- and 65-kev neutron capture cross sections have been made with the 1.2-m liquid scintillator. The results are shown below. Cross sections at 30 kev have been measured for Sm, Sr, and Zr isotopes by using the fast Moxon-Rae detector at the ORNL pulsed 3-Mv Van de Graaff. Capture cross sections (from 10 to 55 kev) were measured with Y^{89}, Pb^{206}, Pb^{207}, F^{19}, S, and $Sn^{116-120}$ samples.

Elemental Neutron Capture Cross Sections

Z	Element	σ_c (mb)	
		E_n = 30 ± 7 kev	E_n = 65 ± 20 kev
15	P	7 ± 1	0.5 ± 0.2
17	Cl	11 ± 4	4 ± 2
19	K	16 ± 2	4 ± 1
20	Ca	10 ± 1	3 ± 1
22	Ti	29 ± 3	5 ± 1
25	Mn	22 ± 2	9 ± 1
31	Ga	103 ± 9	56 ± 5
33	As	350 ± 32	175 ± 16
34	Se	94 ± 8	51 ± 5
37	Rb	180 ± 16	89 ± 8
52	Te	97 ± 9	35 ± 3
56	Ba	61 ± 5	33 ± 3
63	Eu	2560 ± 230	1580 ± 140
72	Hf	510 ± 45	330 ± 30
75	Re	900 ± 80	525 ± 50
76	Os	300 ± 27	175 ± 16
77	Ir	795 ± 70	450 ± 40

CHEMISTRY RESEARCH

Effects of Radiation on Analytical Methods. – Work continued on the effect of gamma radiation on analytical methods, utilizing a Co^{60} source of about 2800 curies.

Exploratory tests on the thiocyanate–stannous chloride spectrophotometric method for molybdenum revealed that gamma irradiation produces a fading of the color of the absorbing Mo-CNS complex. In utilizing this method, it will probably be necessary to make direct comparison of irradiated and nonirradiated solutions prepared at the same time.

Spectral studies established that Mo(VI) has an absorption maximum at about 217 mμ, whereas Mo(III) absorbs less strongly with a maximum at approximately 295 mμ. The radiolytic reduction of Mo(VI) can be evaluated by comparison of the spectra of irradiated and nonirradiated solutions. When a solution containing 10 μg/ml of Mo(VI) is deaerated with helium and then exposed to 5×10^5 r of irradiation under a helium atmosphere to prevent reoxidation of Mo(III), 95% of the Mo(VI) is reduced. If, however, the irradiation is made with the sample exposed to air, the reduction is much less complete, although some reduction can be detected by absorbance measurements made immediately following the irradiation.

Tests involving the gamma irradiation of neutral and slightly acidic solutions of KCNS indicate that the pure thiocyanate is quite resistant to radiolysis. This observation was recently confirmed by M. Duflo [J. Chim. Phys. 59, 655 (1962)]; consequently, further work on this subject will be curtailed. An investigation, however, is being continued on the effect of radiation on thiocyanate under conditions prevailing during the extraction of molybdenum thiocyanate.

Gamma radiation has been found to have some effect on both the Beckman-type saturated calomel (S.C.E.) and glass electrodes. In carrying out this investigation on high-impedance electrodes, it was necessary to use a vibrating-reed electrometer for the potential measurement rather than the Rubicon-B potentiometer previously used. It was observed that a temporary change in potential is produced on exposure of the S.C.E. to gamma radiation. The potential of the electrode returns to its normal value after removal from the radiation field. The log-log plot of time vs potential is linear; therefore, by extrapolation to the time at which the electrode is removed from the irradiation field, the true change in electrode potential induced by the irradiation can be measured. This change in potential is a function of dosage, although apparently not a linear function. The tests on glass electrodes indicate that radiation damage to this type of electrode is permanent.

Chemical Engineering Research: Foam Separation. – The determination of HTU_x values for stripping of Sr^{89} from a dodecylbenzenesulfonate solution was continued. HTU_x values from 13 to 20 cm, using a single tube on the 6-in.-ID column center line as a liquid feed distributor, show that very poor liquid-foam contact is obtained from 27-cm countercurrent lengths. For 52- and 57-cm countercurrent lengths, the HTU_x values indicate normal liquid-foam contact for the lower half of the countercurrent

region at 100 gal ft^{-2} hr^{-1}, but channeling may persist throughout the entire length at higher flow rates. One liquid distributor gave distinctly lower HTU_x values than other distributors for flows below 120 gal ft^{-2} hr^{-1}, but four distributors all gave HTU_x values of 2 to 4 cm for flows over 140 gal ft^{-2} hr^{-1}. The differences in HTU_x values for the spinnerette gas spargers with 50- and 80-μ-diam holes are small, variable, and probably not significant. When the slurry formed by the addition of 5×10^{-3} M NaOH and 5×10^{-3} M Na_2CO_3 to process water was fed to a foam separation column, the $CaCO_3$ and $Mg(OH)_2$ precipitates were removed from the solution by the foam, and the strontium decontamination factor necessary for low-activity waste (about 200) was possible, depending on flow rate. When 10 ppm of Fab was added to simulate impurities in low-activity waste, precipitation was delayed, and the decontamination factor for strontium was reduced to 10 or less.

Chemical Engineering Research: The $NaF-SbF_5$ Complex. — Some laboratory data on the sorption of SbF_5 on NaF was obtained, because Sb^{125} activity has been observed as a contaminant in UF_6 from the Volatility Pilot Plant. In two tests, NaF/SbF_5 mole ratios of 3.95 and 4.03 were secured at 200°C, using 12- to 20-mesh NaF and a pressure of less than 5 psig. Previous experience with similar complexes indicates that the actual stoichiometric ratio that could be achieved with fine material would probably be 2/1. Preliminary measurements with a helium transpiration method indicated that the dissociation pressure of the $NaF-SbF_5$ complex is less than 0.04 mm Hg in the range 210 to 305°C.

Volatility Studies. — Tests were conducted on the hydrochlorination of Al-U alloy fuel in fluoride salts, and dissolution rates of 5 to 9 mils/hr at 500 to 600°C were obtained. Little reaction appears to occur below the salt surface, possibly due to the low solubility of HCl in the melt employed, 31-24-45 mole % $LiF-NaF-ZrF_4$. The most rapid reaction appeared to occur just above the surface of the salt in tests in which the fuel-element specimen was allowed to float in the melt.

Solvent Extraction Research. — In further testing of alkylbenzenes for possible use as diluents for organophosphorus extractants, several high-boiling-point (higher than 349°C) polymethylbenzenes were shown to be much more resistant than Amsco 125-82 to degradation by boiling 2 M HNO_3. In a test where degraded 1 M TBP-Amsco extracted 4000 counts/sec of $Zr-Nb^{95}$ from 2 M HNO_3, similarly degraded 1 M TBP solutions in hemimellitene (1,2,3-trimethylbenzene), in prehnitene (1,2,3,4-tetramethylbenzene), and in isodurene (1,2,3,5-tetramethylbenzene) extracted only 125, 175, and 1400 counts/sec respectively.

In further studies on the extraction of metal ions from 0.5 to 10 M HCl and LiCl—0.2 M HCl mixtures (0.5 to 10 M total Cl^-), maximum extraction coefficients obtained with 0.1 M amine in diethylbenzene were about 2 for Ti(III), 3 for Ti(IV), 60 for Mn(II), 1000 for Co(II), 1000 for Zn(II), 100 for Cu(II), 2 for As(III) and As(V), 8000 for Ga(III), and 700 for Zr(IV). In general, extraction coefficients increased with the increase in chloride concentration and were higher with Aliquat 336 than with the other amines.

In connection with studies of alkaline-earth extraction from sodium nitrate solutions by benzene solutions of di(2-ethylhexyl)phosphoric acid (HDEHP) and its sodium salt (NaDEHP), precise vapor-pressure-difference (Δp) measurements at 20°C confirmed the dimerization of HDEHP (0.06 to 0.5 M) and indicated increasing polymerization of mixtures of HDEHP and NaDEHP (total reagent concentration = 0.10 M) as the NaDEHP:HDEHP ratio increased. The polymerization of NaDEHP in benzene was indicated by $\Delta p = 55 \pm 5$ μ to be 13 ± 1 monomers per polymer at 0.10 M and varied directly but not linearly with concentration. The strontium salt of HDEHP was also shown to be polymerized.

Batch extraction tests, directed toward explaining and correcting the poor decontamination encountered in the amine extraction of plutonium from Purex 1BP solution, showed that aged 1BP solution does contain a considerable amount of readily extractable gamma activity. Up to about 5% of the gross gamma was extracted in single equal-volume contacts with diethylbenzene, Amsco 125-82, and n-dodecane. Surprisingly, the gamma extraction was about 10 times higher by the hydrocarbon alone than in the presence of alcohol or alcohol plus amine. However, gamma extraction by the amine solution after prescrubbing with diluent was still significant; therefore, prescrubbing will not suffice to correct the poor decontamination.

Ion Exchange Studies. — A study of the effects of gamma radiation on anion exchange resins was initiated, using the hydroxyl form of Dowex 1-X10 (50 to 100 mesh). A sample of the resin was placed in a system of flowing demineralized water (less than 1 micromho/cm) and exposed in a 10,000-curie Co^{60} source to a dose of 4.2×10^8 r (about 1.1 whr per gram of dry resin). The maximum specific conductance of the continuously monitored effluent was 130 micromho/cm; the corresponding maximum pH was 10.7. The effluent solution had a strong odor characteristic of amines, the active groups on the anion resin. At the end of the experiment, about 62% of the original resin volume was recovered. About 10% of the original resin was recovered as particles smaller than 200 mesh; the residual 28% apparently had been converted to water-soluble radiolysis products.

A cursory examination of the continuously recorded pH data indicated that a substantial fraction of the active sites on the resin had been lost to the effluent after an exposure of about 1.3×10^8 r (0.35 whr per gram of dry resin) — about 1/3 of the total dose. During this period, the pH decreased to 8.4; thereafter it slowly decreased to about 7.8 to 8.0.

Mechanisms of Separations Processes. — Partial pressures of tributyl phosphate at 25°C over its HNO_3-H_2O solutions are being measured as part of a study of the thermodynamics of extraction of nitrates by TBP. For this purpose the transpiration technique with P^{32}-labeled TBP is being used. Anhydrous TBP, water-saturated TBP, and TBP in equilibrium with 0.765, 2.29, 4.36, and 5.46 M HNO_3 exhibited partial pressures of 0.558 ± 0.060, 0.374 ± 0.060, 0.275, 0.161 ± 0.012, 0.0763 ± 0.0031, and 0.0456 ± 0.0020 μ respectively. Standard deviations ranged from 0.060 to 0.0020 μ, as indicated above, on 3 to 12 measurements.

From the ratio of pressures of TBP over water-saturated and dry TBP, the rational activity of TBP saturated with water is 0.670 ± 0.085. The corresponding molar activity coefficient, y_T, of TBP in the water-saturated solution is, therefore,

$$y_T = (0.670 \pm 0.085)\,\frac{C_0}{C} = 0.716 \pm 0.091 \ ,$$

where C_0 is the concentration of TBP in the anhydrous state, 3.654 moles/liter, and C is the corresponding water-saturated value, 3.420 moles/liter. Based on this value of the activity coefficient of TBP and a previously reported value of $K_1 y_T = 1.47$, the thermodynamic equilibrium constant, K_1, for the extraction of nitric acid by TBP is 2.05. This value, which has a standard deviation of about 0.25 to 0.3, is based on the activities of the H^+ and NO_3^- ions corresponding to the extraction equation

$$H^+_{aq} + NO_3^-{}_{aq} + TBP_{org} \rightleftharpoons TBP{\cdot}HNO_{3\,org} \ .$$

In terms of the more probable mechanism of extraction represented by the equation

$$HNO_{3\,aq} + TBP_{org} \rightleftharpoons TBP{\cdot}HNO_{3\,org} \ ,$$

the thermodynamic equilibrium constant is $2.05 \times 1.17 \times 10^4 = 2.40 \times 10^4$, where 1.17×10^4 is the thermodynamic equilibrium constant for the dissociation of HNO_3 into its ions.

Nuclear Methods of Chemical Analysis: Neutron Flux Monitoring. — Experiments are being carried out to find combinations of stable nuclides which, when irradiated simultaneously in a high neutron flux, will make it possible to measure the average thermal and resonance neutron flux from the induced radioactivities in the samples. Such measurements have heretofore been made by irradiating one or more neutron flux monitors with and without cadmium covers. In many cases for long irradiations at a high neutron flux, there are a number of objections to the use of cadmium covers, not the least of which is the burnup of the Cd^{113} (about 10 mils/month at a flux of 10^{14} neutrons cm^{-2} sec^{-1}), which progressively decreases the opaqueness of the cadmium cover to thermal neutrons.

The radionuclides produced in stable Co^{59}, Cr^{50}, and Fe^{58} can serve as the thermal-neutron-sensitive, resonance-neutron-insensitive monitors, whereas certain of the heavier nuclides — those of high A — such as In^{113}, Th^{232}, and Cs^{133} are sensitive to resonance neutrons but relatively insensitive to thermal neutrons. In seeking a monitor for resonance neutrons, preliminary experiments have been made, using Co^{59}-In^{113} and Co^{59}-Th^{232} combinations; it has been demonstrated that, for the reaction $In^{114m}(n,\gamma)In^{115}$, the thermal-neutron cross section is 1000 barns or greater.

Nuclear Methods of Chemical Analysis: Surface Area Studies. — Studies are being made of a method for measuring surface area based on the exchange of a radionuclide in a liquid with atoms of a stable isotope (of the same element) on the surface of a solid in contact with the liquid. Experiments have been made by using Ca^{45} to exchange with calcium atoms on CaF_2 powder dispersed in dilute nitric acid. Also, U^{237} was used to exchange with uranium atoms on powdered UO_2 dispersed in 10^{-3} M sodium pyrophosphate solution. These tests have shown that under suitable conditions surface areas can be measured with a precision of about ±20%.

Nuclear Chemistry: Neutron Cross Sections. — For determination of the thermal neutron capture cross section and of the resonance integral of Pa^{233}, samples of ThO_2 were irradiated at thermal fluxes of about 10^{14} for periods of 3 to 5 days with cadmium and gadolinium filters. Following a short decay period, the uranium fractions were separated and the U^{234}/U^{233} ratios were determined mass spectrographically. Values of 45 ± 5 and 920 ± 90 barns were found for $\sigma_{2200}(Pa^{233})$ and $\int_{0.55} \sigma(Pa^{233})dE/E$ respectively, which may be compared with 39 ± 5 and 930 ± 135 barns reported by Eastwood and Werner. A comparison of the activation produced in the gadolinium- and cadmium-filtered irradiations showed no evidence of a resonance for neutrons between 0.2 and 0.55 ev.

Chemical Physics: Microwave and Radio-Frequency Spectroscopy. — A species believed to be NO_3 has been studied in gamma-irradiated crystals of KNO_3 by use of a 100-kc/sec-modulated spectrometer operating at about 9250 Mc/sec. It was produced by irradiation at 77°K and was observed to disappear after several hours at 77°K. The spectrum consists of three sharp, closely spaced lines arising from the hyperfine coupling with N^{14}. The g-tensor and hyperfine coupling tensor, A, are axially symmetric with respect to the pseudohexagonal axis of KNO_3. The principal g-values and hyperfine values are:

$$g_{\parallel} = 2.0031 \pm 0.0004 \qquad A_{\parallel} = (12.08 \pm 0.15)\ \text{Mc}$$
$$g_{\perp} = 2.0232 \pm 0.0004 \qquad A_{\perp} = (9.80 \pm 0.15)\ \text{Mc}$$

The identification of the species as NO_3 is supported by the axial symmetry and by the very small hyperfine coupling to N.

Physical Chemistry of Molten Salts. — Molar volumes in molten LiF-ThF_4 solutions were measured at 20 mole % composition intervals over the temperature range 800 to 1200°C by the Archimedes immersion method. This system was chosen to ascertain whether the volume behavior was that of an ideal solution; other thermodynamic properties in alkali fluoride—ThF_4 systems have shown large negative deviations from ideality. Within the precision of the measurements (1%), molar volumes were observed to be ideal. The ideality of volume behavior exhibited by this system is consistent with results of density measurements in other fused fluoride solutions and probably means that the volume contribution of ThF_4 in any fluoride melt is equal (or very close) to the volume of pure liquid ThF_4 multiplied by its mole fraction.

Mechanisms of Radiation Corrosion of Zirconium and Zircaloy-2 and Electrochemistry of High-Temperature Aqueous Corrosion of Metals. – Additional electrochemical measurements were made on Zircaloy-2 in oxygenated 0.05 m H_2SO_4 at 200 to 300°C and at potentials of –1.400 to +1.500 v vs Pt at 100°C. Mechanically polished specimens made from the highest-quality Zircaloy-2 plate available were employed. Previous measurements performed on chemically polished Zircaloy-2 of poor quality were questioned because of the appearance of white oxide over some small portions of the electrode surface. The results of these recent measurements confirmed many of the previous results as follows:

1. The open-circuit potential rapidly attained a constant value, –0.850 to –0.950 v vs Pt, in about 2 hr. The corrosion rate continued to decrease for a longer time, but eventually reached an apparent steady state of 0.8 to 1.0 $\mu a/cm^2$.

2. The rate of the anodic partial process at 293°C was also 0.8 to 1.0 $\mu a/cm^2$ and was independent of potential.

3. The activation energy for the anodic partial process was 31 to 32 kcal/mole and was essentially independent of potential and temperature.

The results differed as follows:

1. At anodic potentials, steady state was attained in 2 to 3 hr for the recent measurements, while several days were required in the previous ones.

2. At cathodic potentials, steady-state behavior could not be obtained. The parameters for the cathodic process were determined by using rapid galvanostatic techniques. The results gave an activation energy of 5.4 kcal/mole with no potential or temperature dependence. With the previous sample, cathodic polarization measurements could be made. The apparent activation energy changed with potential and temperature and was in agreement with the present value at –1.2 v vs Pt at all temperatures and at –1.4 v vs Pt below 220°C. These differences have not been explained.

The present measurements have, in addition, demonstrated the following:

1. The rate at 293°C is an exponential function of film thickness, leveling off to an apparent steady-state rate at large film thickness.

2. The activation energy for the overall corrosion reaction, $Zr + O_2 \rightarrow ZrO_2$, at open circuit, is about 32 kcal/mole and, thus, approximately equal to that for the anodic partial process.

Chemical Separation of Isotopes. – The rate of exchange of nitrogen between 1 M solutions of NO_2^- and $N^{15}O_3^-$ at 0°C has been measured. The exchange was observed to occur very slowly in solutions containing less H^+ than NO_2^-. A solution 1.5 M in H^+ exchanged almost completely in 4 hr and was used for single-stage separation factor (α) measurements. The measured α's were 1.057 ± 0.001 and 1.055 ± 0.004 at 0 and 10°C, with N^{15} enriching in the NO_3^-.

Oxygen-17 Pilot Plant. – The water distillation cascade has performed well since startup. After 50 days of operation the O^{17} and O^{18} concentrations are 0.3 and 13.5% respectively.

Chemical Applications of Nuclear Explosions. – Studies of the thermal decomposition of $MgSO_3$ and of the reaction between MgO and SO_2 in the 750 to 800°C range showed that the following reactions occur:

$$MgSO_3 \rightarrow MgO + SO_2 \ ,$$

$$MgO + 1.5SO_2 \rightarrow MgSO_4 + 0.25S_2 \ .$$

The solid reaction product of $MgSO_3$ decomposition contained more than 95% MgO, and the balance was $MgSO_4$. The reaction between MgO and SO_2 is very slow, even at 800°C. Only 9% conversion of MgO to $MgSO_4$ was achieved after 1 hr, using a 1.2-g MgO sample and an SO_2 flow rate of 142 cc/min (mole ratio $SO_2/MgO = 38$). The absence of MgS in the reaction product indicates that any MgS formed is consumed by the following reaction:

$$MgS + 2SO_2 \rightarrow MgSO_4 + 0.5S_2 \ .$$

If thermal decomposition products of $MgSO_3$ were analogous to those of $CaSO_3$, the formation of MgS by the following reaction would be expected:

$$MgO + SO_2 \rightarrow 0.75MgSO_4 + 0.25MgS \ .$$

METALLURGY AND MATERIALS RESEARCH

Ceramics Research: Sintering Studies. – It was reported previously that small single crystals of CaF_2 sintered to the cleaved surface of a large CaF_2 crystal have clusters of dislocations surrounding the weld neck (ORNL-3256, p 25). That the effect resulted from plastic flow was confirmed by further studies, which also indicated that the dislocations were generated early in the heat-treatment period. The concentration of dislocations depends on the extent of plastic flow, which, in turn, depends on the geometry of the contact between the sintering bodies. Where the geometry is favorable, massive concentrations of dislocations surround the weld neck after sintering for 30 min at 1250°C. With less favorable geometry, the dislocations remaining after the 30-min treatment have an arrangement indicating extensive annealing after plastic flow. In some cases dislocations apparently completely anneal out during the 30-min heat treatment. Other tests show that concentrations of dislocations at punch marks anneal out during treatment at 1250°C for 30 min.

Physical Properties Studies. – The vacuum in the total hemispherical emittance apparatus was improved to 5×10^{-7} torr for testing of Cb–1% Zr with less oxidation. Tests were extended to 1400°C; however, all the Pt vs Pt–10% Rh thermocouples came off the specimen at this temperature. Examination of the specimen-thermocouple junctions indicated that a liquid had been present, and this suggests that a eutectic exists between Pt and Cb near 1400°C.

Equipment for a hot-probe technique of determination of the thermal conductivity of powders was assembled. In this technique a cylindrical probe with a large length-to-diameter ratio, made from a material of high

thermal conductivity, is inserted in the powder to be measured and the probe is heated by a constant electrical current. The temperature rise of the probe is related to the thermal conductivity of the surrounding medium. The temperature of the powder can be changed by a special tube furnace. The furnace was uniformly bifilar wound for 24 cm with bare and insulated Nichrome wire in a grooved refractory tube. Critical placement of shaped external radiation shields at each end of the furnace yielded a 15-cm length within ±2.5° of 800°C. This uniformity is better at lower temperatures and is at least 10 times that of an unshielded furnace.

The thermal conductivity of 93.4% dense UO_2 was measured to 1300°C in the radial heat-flow apparatus on the fourth heating cycle. The data to 900°C can be fitted to ±1% by $K = \rho/[4.505 + 0.02363T\ (°K)]$, where K is the thermal conductivity in w cm^{-1} $(°C)^{-1}$, and ρ is the fraction of theoretical density. As observed in the third heating cycle, below 1100°C a plot of 1/K vs temperature is linear to ±1%, but above 1100°C possesses a negative deviation from linearity. This indicates that lattice conduction is being supplemented by another mechanism of heat transfer above 1100°C, possibly that of infrared transmission. It is noteworthy that there were no thermocouple failures during the fourth heating cycle, and this indicates the feasibility of routinely operating the radial heat-flow apparatus to at least 1400°C.

CONTROLLED THERMONUCLEAR RESEARCH

DCX-1 Facility. — The measurements of slow plasma currents to insulated end walls positioned outside the throats of the mirror coils were continued, with emphasis on low-pressure operation. The experiments were extended to include simultaneous measurements of these currents, the intensity of rf activity, the plasma potential (by the lithium beam technique), and signals to the 12 unit neutral particle detectors (NPD). Coincident with bursts of rf activity there were fluctuations in electron current out of the mirrors, increases in plasma potential (by as much as several hundred volts), and sharp increases in signals to the NPD. At the cessation of the rf activity, the current fluctuations ceased, the plasma potential dropped, and the NPD signals gradually returned to the steady-state values. A qualitative explanation for these observations is that the heating rate for electrons in the plasma increases with rf activity, the plasma potential then increases in order to balance the loss rates of slow ions and electrons, the energy loss rate of the fast circulating protons increases, and these additional losses are reflected in a higher charge exchange loss rate by virtue of the E^{-4} dependence of the charge exchange cross section. Quantitative checks of this explanation are in progress.

During these experiments it was not possible to reproduce clearly the second type of fluctuation in slow plasma current previously observed and reported, that of equal and in-phase variations in the flows of ion and electron currents coincident with rf activity at pressures somewhat above base.

Considerable progress has been made on developing rf probes and techniques for isolating various field components. The observations at high pressure do not contradict the explanation previously proposed, that of rotating charge clusters. At near base pressure, however, a distinctly different phenomenon has been detected. This is rf activity having purely longitudinal currents with a wide range of nearly pure frequencies not necessarily commensurate with the proton cyclotron frequency. Some observations have been made while injecting an annular current of 400- to 500-v electrons along field lines into the plasma. A few milliamperes of electron current quenches completely the longitudinal activity but has no discernible effect on azimuthal activity. At pressures of about 5×10^{-9} torr, a current of several hundred milliamperes of electrons induces very strong, almost steady-state, longitudinal activity, again with a wide range of possible frequencies (3-1/2 to 30 Mc/sec) not necessarily commensurate with ω_c. A tentative explanation (by C. E. Nielsen, consultant from Ohio State University) for these longitudinal phenomena proposes that they are two stream instabilities.

Intensive studies of the rf field components have not been made simultaneously with those observations of plasma characteristics affected by the electron heating rate, so it is not presently known just what sort of rf activity is most clearly associated with this heating rate.

High-Current Injection. — The accelerator tube with epoxy insulating skirts has been operated at 650 kv with no beam, and with hydrogen ion currents up to 170 ma at 600 kv. There was no indication that x-ray production caused external breakdown as the current was increased. This appeared to be one of the current limiting factors with the previous tube. Skirted sections have been prepared for the DCX-2 accelerator and will be available for installation in the next two weeks.

The 600-kv beam-analyzing equipment has been operated with ion beams up to 150 ma. The power accountability is consistently 10 to 15% low. The reason for this discrepancy has not been determined.

Theory. — The stability of a spatially uniform ion beam circulating in a magnetic field in the presence of cold electrons has been analyzed with the Vlasov equation. The ions are permitted to have a finite temperature in a frame of reference rotating with the ions. Except for a Doppler shift in normal mode frequencies as a consequence of the rotation, the dispersion relation has the same form as that for the previously analyzed case of ions of the same temperature but with zero average velocity in the rest frame. Thus, like the latter, the circulating beam exhibits the Harris instability when the electron plasma frequency exceeds the ion cyclotron frequency.

The effect of ion and electron temperature on the Harris instability has been examined. Whichever species is hot, new but weak instabilities are introduced at low density, a regime which was stable for cold electrons and cold circulating ions. On the other hand, the stronger instabilities occurring at higher electron density at zero temperature are stabilized for short wavelengths, hot ions being more effective stabilizers than are equally hot electrons. Ion temperature causes Doppler broadening of the ion cyclotron frequency, which, in turn, weakens the

resonance-like Harris instability. Long wavelengths are unaffected, because, for a given temperature, Doppler broadening vanishes as the wavelength increases.

BIOLOGY AND MEDICINE PROGRAM

RADIATION EFFECTS

Genetic Control of Protein Synthesis. — The higher incidence of secondary deaths in lethally irradiated (C57BL × 101)F_1 recipients of C57BL marrow compared with deaths of similar recipients of 101 marrow might be attributed to a greater immunological capacity of C57BL marrow. In the same strain combinations, however, a large percentage of C57BL grafts regress, whereas 101 marrow grafts are retained. The purpose of this study was to determine the relative ability of C57BL and 101 marrow to repopulate the hemopoietic system of lethally irradiated F_1 hybrids. Differences in growth potential of parental marrows in F_1 hybrids may help to explain strain differences in the incidence of secondary deaths and in the frequency of graft regressions. The two parental marrows were mixed in various ratios and injected into irradiated recipients, 20×10^6 cells per recipient. The type of marrow that repopulated the F_1 recipient was determined by assay of the circulating erythrocytes in chimeras at 30, 60, and 90 days after treatment. A total of 171 chimeras were tested. Recipients of mixtures of 14×10^6 C57BL cells and 6×10^6 101 cells, or other inocula containing fewer C57BL and more 101 cells, were 101 chimeras at 60 days. The majority (15 of 18) of recipients of 16×10^6 C57BL and 4×10^6 101 cells became 101 chimeras. An equal number of C57BL and 101 grafts were found among 12 survivors of 18×10^6 C57BL and 2×10^6 101 marrow cells, and 6 of 17 recipients of 1×10^6 101 and 19×10^6 C57BL cells were repopulated by 101 marrow. Similar results were obtained with mixtures of C57BL and BALB/c marrow in (BALB/c × C57BL)F_1 recipients. The data indicate that 101 and BALB/c marrows when injected in a mixture with C57BL marrow are approximately nine times more effective than the latter in repopulating the hemopoietic system of their respective lethally irradiated F_1 hybrids. Immunological and physiological factors of the donor marrows and of the irradiated recipients that may account for such a marked difference in the efficiency of these parental marrows are being studied.

RADIATION GENETICS

Mammalian Genetic Cytology. — 5-Bromodeoxyuridine (5-BUDR) has been shown by a number of workers to have been incorporated, with concomitant thymidine replacement, into the deoxyribonucleic acid (DNA) of bacteria, bacteriophage, and mammalian cells and to have induced chromosome breaks.

It was found here that a 24-hr treatment of Chinese hamster cells with a low concentration (3 μg/ml) of 5-BUDR, while capable of inducing some chromosome aberrations, did not interfere too much with cell growth and reproduction. When the 5-BUDR-treated cells were exposed to various wavelengths of monochromatic ultraviolet (UV) light, an enhancement of induced frequencies of chromatid aberrations and general modification of action spectrum were observed. Particularly clear are the much higher aberration frequencies induced at 2250 and 3130 A. It was known earlier that UV at these wavelengths induced only a few aberrations. This study suggests that DNA is probably the primary site of UV injury which leads to chromosome aberrations. It was found earlier in this laboratory that the presence of a low concentration of cobalt chloride in the culture medium makes the nucleolus of the mammalian cell continue to persist through mitotic metaphase. Investigations show that ribonucleic acid (RNA) synthesis completely ceases during mitosis. It would be interesting, then, to find whether RNA synthesis still goes on in the cobalt-retained nucleolus. Experiments were done by use of pulse-labeling of H^3-uridine, both before and after the cobalt treatment. Preliminary examination indicates that the presence of cobalt salt did not interfere with the migration of the manufactured RNA or RNA precursors to cytoplasm and that the cobalt-retained nucleolus at metaphase had essentially no RNA synthetic activity.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — During the past year considerable evidence has been published suggesting that the thymus before or shortly after birth disburses its cells to lymph nodes, spleen, Peyer's patches, and other lymphatic tissues, where they become the functional cells of the immune mechanism. These findings led to attempts to use injected fetal thymus cells to influence the mortality from secondary disease that occurs after lethally irradiated mice are treated with foreign bone marrow.

It was found that the fetal thymus cells lacked the ability to behave immunologically in the same manner as adult thymus cells. There was no significant effect on the mortality from secondary disease.

Tissue Culture. — The effect of irradiation restricted to either the nucleus or the cytoplasm on cell multiplication is being investigated by comparing survival curves for H^3-thymidine- and H^3-uridine-treated mammalian cells. H^3-Thymidine is much more effective, indicating that reproductive death in this system is mainly caused by irradiation of the nucleus. To study the cytological features of x-ray-induced reproductive death, single mammalian cells are being followed by means of lapse-time cinematography for several days following irradiation. The abnormalities occurring in the first few divisions are recorded on a genealogical basis. Material has been obtained and scored from some of the men irradiated in the Puerto Rico Nuclear Center accident of July 1962. Leukocyte chromosome aberrations were seen shortly after irradiation, even in men estimated to have received only 15 to 19 r of gamma rays. By one month after

irradiation the aberration frequency had dropped, and cells with dicentric chromosomes no longer contained fragments, indicating that the leukocytes had been replaced by cell division. Biological estimates of integrated whole-body dose have been made for three of the men.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND INSTRUMENTATION

Basic Instrumentation. — Further tests on the conversion of p-type silicon to n-type by neutron irradiation demonstrated the feasibility of this method. Residual bulk radioactivity was undetectable after three days. Minority carrier lifetime was restored to an acceptable level by a brief anneal, and conversion to n-type conductivity was verified by Hall coefficient measurements.

TRAINING, EDUCATION, AND INFORMATION PROGRAM

International Reactor Programs at ORNL. — Sixty-two students completed courses at the Reactor School during the 1961–62 session, which terminated on September 14, 1962. The enrollment was as follows:

Program	Number of Students
Reactor Hazards Evaluation	6
Reactor Operations Supervision	9
AEC Trainee Program	8
TVA Trainee Program	9
ORSORT for Carbide employees	
Full-time	6
Part-time	21
Special students	3
	62

The committee on admissions met in August to select qualified applicants for the fifth session of International Programs starting November 5, 1962. Fifteen students representing seven different countries have been selected and notified of their acceptance by the Division of International Affairs, AEC.

At present, approximately fifty students are participating in the pre-ORSORT program; this program terminates the first week in November, when regular programs begin.

ASEE-AEC Summer Institute. — Twenty participants in the 1962 ASEE-AEC Summer Institute, conducted by North Carolina State College, concluded

the last week of their program with experiments at ORSORT. The Reactor School work consisted in a criticality experiment at the Pool Critical Assembly, a reactor kinetics experiment at the ORNL Graphite Reactor, an analog computer experiment, tours, lectures, and the preparation of reports covering the experiments.

ISOTOPE DEVELOPMENT PROGRAM

Application of Radioisotopes in Analytical Chemistry: Determination of Beryllium by the Photoneutron Method. — Fabrication of the apparatus for the determination of beryllium by a photoneutron method was completed. Initial tests were carried out with an Am^{241}-Be neutron source. These tests indicate that the apparatus is operating satisfactorily with an efficiency of about 5% for neutrons having an average energy of 4.5 Mev. Tests with an Sb^{124} gamma source with a dose rate of 200 mr/hr reveal that the interference due to the counting rate of gamma radiation can be eliminated with only a small loss in the neutron counting rate by increasing the pulse-height discriminator voltage. A 400-mc Sb^{124} source is being prepared by the activation of enriched Sb^{123} and is expected to be ready for use by the end of October.

Application of Radioisotopes in Analytical Chemistry: Determination of Alkaline Earths. — Isotope dilution methods for use in the determination of alkaline-earth elements are under study. Low concentrations are determined by flame spectrophotometry; high concentrations by titrimetry. Radioactivity yield corrections are established by the use of Ca^{47}, Sr^{85}, and Ba^{133}. The method of initial separation is now being examined. In making the separation of Ba and Ca, the method of treatment prior to the determination is based on the carrier precipitation of Ba on $PbSO_4$, dissolution in ethylenediaminetetraacetic acid, followed by the decomposition of the Ba complex by acetic acid. For the separation of Ba and Sr, carrier precipitation is carried out on $PbCrO_4$ instead of $PbSO_4$.

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