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# OAK RIDGE NATIONAL LABORATORY

# STATUS AND PROGRESS REPORT

# MARCH 1961

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# OAK RIDGE NATIONAL LABORATORY

# STATUS AND PROGRESS REPORT

# MARCH 1961

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
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U.S. ATOMIC ENERGY COMMISSION

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# OAK RIDGE NATIONAL LABORATORY

## STATUS AND PROGRESS REPORT

## MARCH 1961

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

## REACTOR DEVELOPMENT PROGRAM

### GAS-COOLED REACTOR PROJECT

Operation of EGCR Without Loops. – An analysis of operation of the EGCR core without loops is being made. The specified initial fuel enrichment is 2.46% $U^{235}$, and the effective multiplication factor of the reactor under various conditions will, initially, be the following:

| Core Condition | $k_{eff}$ |
|---|---|
| Hot, equilibrium Xe and Sm | 1.095 |
| Hot, clean | 1.124 |
| Cold, clean | 1.172 |
| Cold, clean, 21 control rods | 0.911 |

If the initial fuel charge were operated without any shifting of fuel, it is estimated that the reactivity lifetime would be 6600 Mwd/metric ton. With one axial inversion of each channel, it is estimated that the reactivity life would be about 8200 Mwd/metric ton. However, it is presently proposed to reposition some of the initial fuel from time to time to effect a smooth transition to an equilibrium fuel cycle; it is estimated that the initial fuel charge, treated in this way, will average ~7000 Mwd/metric ton. On an equilibrium fuel cycle, the feed enrichment necessary to achieve 10,000 Mwd/metric ton burnup will be 2.25%. The compositions of the fuel at discharge for the first

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core (2.46% enrichment) at 7000 Mwd/metric ton and for the equilibrium core (2.25% enrichment) at 10,000 Mwd/metric ton exposure are given below:

| Isotope | Mass at Discharge (kg per metric ton of fuel) | |
|---|---|---|
| | First Core | Equilibrium Core |
| $U^{235}$ | 17.42 | 13.05 |
| $U^{236}$ | 1.28 | 1.66 |
| $Pu^{239}$ | 2.07 | 2.24 |
| $Pu^{240}$ | 0.746 | 1.33 |
| $Pu^{241}$ | 0.0523 | 0.126 |
| $Pu^{242}$ | 0.0120 | 0.0493 |

A program of fuel shifting and replacement is still being sought to achieve a satisfactory compromise among the following objectives: (1) long burnup of initial core, (2) reasonable duty cycle for charge machine, (3) early transition to equilibrium cycle, (4) early attainment of high burnup on some fuel elements. The bank of 21 control rods in the initial, cold reactor is calculated to be worth 0.261 $\Delta k$.

Irradiation Program. – Metallographic examination of the cladding on ORR-irradiated capsule 01-1 showed a precipitate at the inner surface that extended outward along the grain boundaries. The precipitated material was tentatively identified as a nitride or a carbide. The concentration and depth of penetration of the precipitate were uniform.

The plating out of $Cs^{137}$ on the cladding of ORR capsule 08-2 was found to be insignificant. Comparison of data from a leach analysis of the cladding with previously reported data for burnup indicated that less than 0.1% of the $Cs^{137}$ formed had plated out on the cladding.

Two phases other than $UO_2$ were revealed by metallographic examination of the ETR-irradiated capsule E-9 fuel. There are indications that these phases may be $UN_2$ and $U_4O_9$. The fuel showed extensive microcracks, but there was no evidence of grain growth. A grain-boundary precipitate was observed by metallographic examination of the inner surface of the E-9 cladding. The precipitate, probably either a carbide or a nitride, was deposited within grain boundaries to the greatest depths in the vicinity of the dark lines which always occur opposite a pellet-to-pellet interface.

Reactions of Type 304 Stainless Steel with $CO$-$CO_2$ Mixtures. – Data obtained from a 1390-hr test of type 304 stainless steel specimens in a $CO$-$CO_2$ atmosphere have been analyzed. The test temperatures of 16 specimens, which each initially contained 0.068% C, ranged between 725 and 1850°F. The $CO_2$ content of the test atmosphere was maintained at 0.117 vol % and the CO content was 0.175 vol %, which gave a $CO_2$-to-CO ratio of 0.669.

From conclusions based on theoretical principles and prior tests, metal specimens under these conditions should form a nonadherent oxide, and the maximum carburization should occur at an intermediate temperature. In all specimens which showed weight increases greater than 0.40 $mg/cm^2$, the oxide was nonadherent upon cooling. The carbon content reached a maximum of 0.15% at 1560°F, while at 1850°F the carbon content was 0.028%. Carburization was not measurable below about 1200°F.

Reactions of Structural Metals with Impurities Outgassed from Graphite. – A forced-circulation test loop containing type AGOT graphite, metal test specimens, and helium is being operated with a test section temperature of 1400°F and a system pressure of 30 psig. The gaseous impurities in the helium stream, primarily CO and $CO_2$ resulting from the outgassing of the graphite, were finally stabilized after 800 hr of operation by continuous operation of the purification system for $CO_2$ removal and periodic $O_2$ injections for removal of CO. Water is presently being injected into the main test loop at the rate of approximately 0.1 $cm^3$ (liquid) per hour, and the resultant $H_2$, CO, $CO_2$, and $H_2O$ impurity levels are being maintained at, or below, those specified for the EGCR by use of the purification system.

## THERMAL-BREEDER REACTOR PROGRAM

### Homogeneous Reactor Program

Homogeneous Reactor Test. – Reactor startup operations are now in progress. The experimental program will be resumed with the primary objective of studying the behavior of the reactor while operating at full power.

During the maintenance period just completed, the fuel letdown heat exchanger, three high-pressure-system valves, and both fuel feed-pump heads were replaced. In addition, the fuel dump-tank feed line, which contained the leaking fitting (found in February), was equipped with freeze jackets so that the fitting can be isolated by freeze plugs during reactor operation. For additional protection, a clamp was placed on the fitting to make it less likely that the crack will increase in size. All the in-shield maintenance was accomplished in less time than had been estimated.

Thorium Blanket Studies. – Studies of the effects of aging treatments and composition of $ThO_2$ sols on the integrity of the calcined gel particles prepared from them were continued. Aging for 44 hr at 95°C increased the density of the calcined product from 9.6 to 9.8 g/cc, increased the particle strength, decreased cracking, and decreased shrinkage by 15%. Decreasing the nitrate content of the precursor oxide from an $NO_3^-/ThO_2$ mole ratio of 0.4 to 0.23 also increased the integrity and decreased shrinkage by about the same amount. Aging of sols in 2 M acetic and formic acids gave calcined gels of 9.83 and 9.72 g/cc density and with fewer cracks than those prepared from nitrate-dispersed sols.

Reactor Analysis. – Statistical studies of digitized power data from the HRT are being continued with the aid of programs for the IBM 7090 computer. The amounts and rates of reactivity addition required to produce the observed power deviations are being computed. On the average, positive rates and amounts of reactivity addition are larger in absolute value than the negative rates and amounts. The relative standard deviations of both positive and negative reactivity additions are remarkably constant with changes in power level. Salt-trace data obtained from a mockup of the HRT core have also been studied with these programs. So far, a strong correspondence between reactor and mockup data has been observed, supporting the hypothesis that the power fluctuations are hydrodynamic in origin.

Metallurgy and Ceramics. – Three batches of D-40 thorium oxide powders were modified by a method described previously (ORNL-3024) and were used to fabricate round-end pellets on a single-action automatic press. Slight variations in powder densities were found to cause laminations and cracks in the caps of the pellets when this technique was used, making it necessary to readjust the pressing conditions continuously. These difficulties and the poor quality of the resulting pellets dictated the abandonment of this method for large-batch production of pellets.

A more promising method of producing pellets was investigated; it involves the formation of cubes, which are reduced to spheres by tumbling. Cubes were successfully reduced by tumbling both in the unfired condition and after soft firing at 1100 to 1300°C. Unfired cubes were tumbled while dry and with no grinding media; soft-fired cubes were tumbled while wet and both with and without alumina grinding balls. A combination of the two methods appears most desirable, since dry-grinding of the unfired cubes allows for easy recycling of abraded powder and wet-tumbling of fired bodies will produce a smoother finish. The polished spheres are finally fired at 1800°C.

Sintering experiments with slurry-type oxides modified slightly by calcining at 1000°C showed that densities of 97 to 98% of theoretical can be obtained in pressed bodies by heating to 1800°C for 2 hr. These pellets were found to be free of cracks and had an almost glossy texture.

Out-of-Pile Blanket Materials Tests. – As part of the test program for evaluating the relative integrity of experimental $ThO_2$ pellets, the attrition rate of a recent experimental thoria pellet preparation in a well-developed fluidized bed was determined. A superficial water velocity of 0.56 fps in a 2-in.-diam pipe developed a 30% expansion of the 2-in.-high bed of 0.2-in. dome-ended thoria pellets, with moderate pellet agitation, and resulted in a mean (12-hr) weight loss rate of 0.014% per hour. These pellets have shown ball-mill attrition rates of 0.06% per hour (2 hr at 150 rpm, 10 pellets in a 3.75-in.-diam, 4-in.-long water-filled rubber-lined ball mill) and spouted-bed rates of 0.25% per hour (10 pellets, 0.37 fps superficial velocity, 13-fps jet velocity).

In-Pile Blanket Materials Tests. – The inventory concentration of solids in the in-pile slurry loop experiment, as estimated from the original charge and the number of sample and purge removals of predetermined volume, has been compared with the actual sampled concentration

of solids, corrected for rinses and holdup in the sample carrier (but not in other parts of the sampling system). In general, such samples have lower concentrations of solids, possibly implying a circulating concentration below inventory. However, the quantity of $D_2O$ recovered indicated that the carrier sample vessel was frequently not full and may imply retention of some slurry in the sampling system.

Surface-area measurements of samples removed from the loop showed a steady change during the entire period of in-pile operation. The surface area increased from 1.5 $m^2/g$, initially, to more than 40 $m^2/g$ at the end of 2220 hr of total circulation in-pile.

Calculations based on radiochemical analyses of loop samples for $Ce^{144}$ and $Zr^{95}$ indicate a value of average flux based on the total loop contents of $2 \times 10^{12}$ neutrons $cm^{-2}$ $sec^{-1}$ during the in-pile experiment.

Irradiation of autoclave experiment L5Z-155S in the HB-5 facility of the LITR was completed. It was desired to measure the effect of radiation on the degradation of particles gently suspended in a rocking autoclave in comparison with that observed in a slurry pumped in in-pile loop experiment L-2-27S. The Zircaloy-2 autoclave contained a thoria-urania slurry (0.4% U based on thorium) prepared from the same batch (DT-22) used in the in-pile loop experiment. The autoclave contained slurry at a concentration of 1000 g of Th per kg of $D_2O$, 0.014 m Pd, and an oxygen overpressure; it was operated for a total of 770 hr at 280°C. The autoclave was operated largely in the fully inserted position for 453 hr with the LITR at criticality or above. At an assumed flux of $7 \times 10^{12}$ neutrons $cm^{-2}$ $sec^{-1}$, the autoclave had attained the dose ($5 \times 10^{16}$ fissions per gram of solids) absorbed in the in-pile loop experiment.

An over-all corrosion rate of 0.9 mil/yr was observed for the irradiation period. Out-of-pile, a generalized corrosion rate of 4 mils/yr had been observed for the first 48 hr; the rate decreased to 1 mil/yr for the remaining 150 hr before irradiation began.

Based on insertion experiments early and late during the irradiation period, no significant amounts of radiolytic gas were indicated. However, uncertainties in thermocouple readings during much of the irradiation period made it difficult to determine the radiolytic gas present.

## Evaluation Studies

Thorium-Breeder-Reactor Evaluation Studies. – The effect of fissionable-fuel value on fuel costs in the five thorium breeder reactors under study was investigated. Since the use charge for fissionable material was taken as 4%, increasing the value of the fuel lowered the fuel-cycle cost in those reactors having a fuel yield greater than 4%, and increased it otherwise. Increasing the value of $U^{233}$ and $U^{235}$ from \$15/g to \$60/g decreased the minimum fuel cost in the aqueous homogeneous breeder reactor from 0.9 to about 0.5 mill/kwhr and increased the associated fuel yield from 7 to 12%/yr; in all the other systems studied, the minimum fuel cost increased 0.1 to 0.2 mill/kwhr.

The loss of neutrons to xenon in the molten-salt breeder reactor has been evaluated, assuming a graphite porosity of 10%, for xenon diffusion coefficients of $10^{-5}$ and $10^{-7}$ $cm^2/sec$. If the coefficient was $10^{-5}$ and the stripping cycle time was 25 sec (20 cfs), then the loss of neutrons would amount to 0.005 on the breeding gain. This rate of stripping appears reasonable, and the cycle time is appreciably greater than the time required to circulate the fuel salt through the reactor and the heat exchangers, which takes about 3 sec. If the diffusion coefficient was $10^{-7}$, then a cycle time of 50 sec (stripping rate of only 10 cfs) would be required to hold the breeding loss to 0.005. According to a recent report (ORNL CF-61-2-59), graphite of this grade is within the capability of present-day technology. Thus the original assumption of a 0.005 breeding loss due to xenon absorption appears justified.

## MOLTEN-SALT REACTOR PROJECT

MSRE Design. — Drawings of the reactor, drain tanks, and radiator were reviewed and returned to the design group for revision. Drawings for reactor-cell power and thermocouple disconnects were issued for comment. An alternative concept for a flanged reactor vessel was started.

Electrical-distribution drawings were issued for comment. Approvals are being obtained for the drawings covering site preparation, to be done by a cost-plus-fixed-fee contractor. All structural design work is proceeding. The first package of drawings (associated with construction in the reactor, radiator, and containment areas) is scheduled for completion in May; this work will be done by lump-sum contractors.

Prints are complete and out for comment on a 7-ton crane hook, a crane-handled pneumatic hook and hand, and a freeze-flange torquing tool. Drawings for jacks to separate flanges on the heat exchanger are being prepared. Three layouts of manipulator arrangement have been made.

Component Development. — Construction of the engineering test loop was completed, and the preoperational check was begun.

A facility was constructed to permit thermal cycling of large MSRE development flanges in air under closely controlled conditions to aid in the development of a gas seal and to study plastic deformation of the metal.

A freeze-valve test, in which tubular electrical-resistance heaters were used to thaw the salt, was terminated after 100 satisfactory cycles.

Testing of one design of a split-shell insulation package for a pipe heater indicated that there was excessive heat loss along the radial split lines. Redesign to reduce the heat loss is in progress.

The reactor-vessel heater test was put into operation and was found to require 20 to 30% more than the design power. Heat loss due to inleakage of air was also found to be a problem; careful design and construction of the insulation system is required for control of air leakage.

A water mockup of a molten-salt poison tube for reactivity control, with gas being used to raise and lower the fluid in the tube, was tested with some success.

Remote Maintenance. – The salt system of the remote-maintenance demonstration facility was removed; partial mockups of the MSRE pump and that portion of the secondary piping within the containment vessel are now being installed. A mockup of a semiremote-maintenance shield was installed on the remote-manipulator bridge to simulate the MSRE working elevation, and the mockup will be used to develop tools to accomplish known jobs.

Design studies indicate that each pipeline heater section will require special tools for replacement because of variations in pipe supports, freeze-flange equipment, and heater design.

Extreme-range thermal cycling (room temperature to 1400°F) on two gas-buffered integral ring-joint spring-clamp flanges for 1/2-in. lines is proceeding, and satisfactory performance is being achieved.

Work was started on remote furnace-brazing of small pipe using inductance heating and inert-gas purge. A system was demonstrated for taking a section of pipe to braze temperature without excessive oxidation.

MSRE Instrumentation. – Preparation of preliminary drawings for MSRE instrument applications is approximately 75% complete: nine drawings were completed, and six of the nine were issued for comment.

Preliminary thermocouple-location drawings were completed, were issued for comments, and are being revised to include recommended changes in the number and location of thermocouples.

Studies are being made of methods of installing thermocouple and instrument wiring in the reactor and storage-tank areas and of providing vessel penetrations, disconnects, and conduit end seals for this wiring. Preparation of conduit-layout and disconnect-location drawings is in progress.

Several types of thermocouple, instrument, and electrical disconnects were developed. Prototypes of these disconnects are being tested in the remote-maintenance facility.

A program was started for development of devices for measurement of levels in molten-salt systems. A float-type continuous level element and a single-point conductivity-type element are being developed.

Pump Development. – The pump with a molten-salt-lubricated journal bearing has now circulated $LiF-BeF_2-UF_4$ (62-37-1 mole %) at 1225°F for 8088 hr. The Inconel centrifugal pump (PKP type) has now circulated $LiF-BeF_2-ThF_4-UF_4$ (65-30-4-1 mole %) at 1225°F for 4368 hr. Preliminary testing of equipment modifications for this pump began in preparation for the back-diffusion tests with $Kr^{85}$. Evidence indicates that there is no leakage from the lower-shaft oil seal into the pump tank. No trace of oil distillate was found in a liquid-nitrogen cold trap in the off-gas line from the pump tank. No spectrographic evidence of oil was found in off-gas from the pump tank.

Fabrication of the prototype of the MSRE fuel pump awaits delivery of the INOR-8 volute and impeller castings. The founder is making initial tests by casting INOR-8 into some simple configurations. Fabrication of the test stand for the prototype pump was started.

The pump-tank configuration was established from results of analyses of temperature distributions for various configurations and operating conditions. The flow of cooling air for the pump tank was specified.

Chemistry. – Previously described beneficial effects from including $ZrF_4$ in the fuel to scavenge oxide contamination were obtained with simplified fuel mixtures which, for example, did not contain $ThF_4$. Oxide additions have now been made to the nominal MSRE fuel by incremental substitutions of BeO for part of the $BeF_2$ in $LiF-BeF_2-ZrF_4-ThF_4-UF_4$ (70-23-5-1-1 mole %). In accord with earlier results, a $ZrF_4/UF_4$ ratio of 3 or greater was adequate for preventing the formation of $UO_2$, whereas $UO_2$ did precipitate when BeO was present in an amount sufficient to reduce (by conversion of $ZrF_4$ to $ZrO_2$) the $ZrF_4/UF_4$ ratio to 1. An amount of BeO calculated to completely precipitate all the $ZrF_4$ and $UF_4$ did not produce a detectable precipitation of $ThO_2$.

The composition of the compound which precipitates first (at 442°C) as the MSRE fuel is cooled has been found to be $6LiF \cdot BeF_2 \cdot ZrF_4$. This compound melts semicongruently to LiF and liquid at 474 ± 2°C; as an equilibrium phase in the fuel system it does not contain dissolved $ThF_4$ or $UF_4$. Powder diffraction data for the compound are indicative of the hexagonal system. Optically, the crystals are uniaxial negative and have low birefringence; the mean refractive index is 1.418.

Slow freezing of the fuel, in a manner approaching equilibrium crystallization, segregates $ZrF_4$-rich phases in the first solids to deposit, and hence depletes $ZrF_4$ in the last liquid to freeze. Under these unusual circumstances, protection against precipitation of $UO_2$ is lost for the last liquid to freeze, and traces of $UO_2$ can be produced in the MSRE fuel at low temperatures, though not at temperatures above the fuel liquidus.

Metallurgy. – A radiographic technique was developed to determine the distribution of molten salt in graphite subjected to the standard permeation screening test. Nine different grades of graphite were examined after 100-hr exposures to salt at 1300°F under 150 psig pressure. Grade B-1 graphite was found to take up salts only in the region of the surface, to a depth of 0.025 in. No salt was observed in low-permeability pipe (CEY type) tested when the ends were sealed. Some grades of graphite were penetrated more unevenly, sometimes to the center of the specimen. The radiographic technique is expected to be useful for establishing the suitability of MSRE graphite.

Corrosion Tests. – Metallographic examination of loop 9354-3 was recently completed. This loop was operated for 19,942 hr under the following conditions: salt mixture, $NaF-LiF-BeF_2$ (27-35-38 mole %); maximum salt-metal interface temperature, 1200°F; bulk salt temperature, 1150°F; temperature difference, 150°F; flow rate, 2 gpm; Reynolds number, 3000. The examination revealed no evidence of attack of the surfaces exposed to the flowing salt in either the hot- or cold-leg areas of the loop. However, macroscopic examination of specimens removed from the end of the cooler coil did reveal the presence of a small amount of metallic deposit. Analyses of this deposit have not been completed.

Reactor Analysis. – Preliminary nuclear calculations were performed for a molten-salt reactor with 0.040-in.-thick, 3-in.-ID tubes separating the fuel salt from the core graphite. The core was assumed to be a homogeneous mixture of graphite, fuel salt, and INOR-8, and the multigroup diffusion-theory IBM 704 program GNU was used.

A reactor with tubes compares with one without tubes as follows:

| | With Tubes | Without Tubes |
|---|---|---|
| Fuel volume fraction | 0.24 | 0.24 |
| Circulating inventory, kg of $U^{235}$ | 134 | 49 |
| Temperature coefficient of reactivity due to salt expansion | $-2.4 \times 10^{-5}/°F$ | $-3.2 \times 10^{-5}/°F$ |
| Mean core thermal flux at 10 Mw, neutrons $cm^{-2}$ $sec^{-1}$ | $7.4 \times 10^{12}$ | $2.5 \times 10^{13}$ |
| Percentage of total fissions caused by thermal neutrons | 67 | 86 |

The temperature coefficient of reactivity due to graphite expansion is decreased from $-6 \times 10^{-5}/°F$ to $-3 \times 10^{-5}/°F$ by the use of the tubes, and there is an additional positive temperature coefficient of about $4 \times 10^{-6}/°F$ due to INOR-8 expansion, thus changing the isothermal temperature coefficient from $-9 \times 10^{-5}/°F$ with no tubes to $\sim -5 \times 10^{-5}/°F$ with tubes.

## HIGH FLUX ISOTOPE REACTOR

Control and Instrumentation. – Title II drawings and specifications are being prepared for the control and instrumentation of the reactor and process system by ORNL and by Singmaster and Breyer personnel. Biweekly conferences are being held to coordinate these efforts.

A program to establish the most practical way in which to perform the on-the-line tests of the various components of the coincidence safety system has been started. Performance tests will be made in the HFIR hydraulic mockup.

A prototype of the ionization chamber has been assembled.

The design of a collimator to increase the flux at the ionization chamber location has been started. The chamber must operate in a flux between $10^9$ and $10^{10}$ neutrons $cm^{-2}$ $sec^{-1}$.

Corrosion. – Additional tests concerning the effect of pressure on the corrosion of type 6061 aluminum subjected to a heat flux of $2 \times 10^6$ Btu $hr^{-1}$ $ft^{-2}$ and cooled with water at pH 5 have qualitatively confirmed the results previously reported; that is, at low pressures, where boiling might have occurred within the oxide film, the rate of temperature increase in the aluminum and the corrosion of aluminum were less than under the same conditions at pressures sufficiently high to prevent boiling. Further testing is necessary to establish a quantitative relationship between pressure (boiling) and corrosion rate.

Preliminary experiments to determine the effect of pH on the rate of oxide formation and on the corrosion of type 6061 aluminum have indicated that at a heat flux of $2 \times 10^6$ Btu $hr^{-1}$ $ft^{-2}$, the rate of temperature increase was the same when the pH of the coolant was either 5.0 or 5.3; however, at a pH of 5.7 with the other conditions the same, the rate

was significantly higher. For example, at the two lower pH values the maximum rate of temperature increase was approximately 16°F/day, whereas at the highest value the rate of increase was about 30°F/day. The actual amount of metal corroded on each specimen has not been determined at this time.

An isothermal corrosion test conducted in a 100A loop at 100°C with water adjusted to pH 5 with nitric acid was terminated after 2500 hr. Significant corrosion of the aluminum in crevices between aluminum and stainless steel was observed. In addition, specimens of beryllium showed relatively severe pitting. Uncoupled specimens of several aluminum alloys and types 347 and 416 stainless steel showed negligible attack. A similar loop run in which the pH of the water will be adjusted to 5.3 will be started to see whether corrosion is less severe at the lower acid concentration.

Metallurgy. – The fabrication of the fuel element to be used in the critical experiment was completed.

A method for evaluating the $U^{235}$ content in the HFIR alloy core has been statistically judged to be accurate within 4%. This method is based on measuring the density of the extruded slug, and requires correlation of density with the uranium concentration in this alloy.

Two hundred twenty-five plates were fabricated for the inner annulus fuel element. These plates contain a powder-metallurgically-prepared fuel core containing 25 wt % $U_3O_8$ and 0.05 wt % $B_4C$ in aluminum and a filler piece containing 0.05 wt % $B_4C$ in aluminum. The plates were hot rolled at 450°C and reduced 91.5% in thickness. After cold rolling 7% in thickness, the plates were annealed at 500°C, roller leveled, and annealed again. Essentially all plates blistered in the 500°C annealing treatments on the side of the plate which contains the 0.05% $B_4C$–Al insert. Although the exact cause for such massive blistering is unknown, examination of inserts has shown spherical-type porosity, indicative of gas pockets. It is presumed that gas pressure developed from these localized areas caused blistering.

Assembling of the outer annulus fuel element and the inner annulus fuel element was completed on February 20 and March 3, respectively. Difficulty was experienced with the outer element when measurements revealed its diameter to be 0.140 in. oversize. The diameter was reduced to 0.030 in. oversize by rolling the assembly down against a fixed stop on the assembly lathe. Final measurements showed that the fuel element was also 0.040 in. out of round. Measurements of the completely assembled inner annulus fuel element revealed it to be 0.010 in. under the specified diameter and 0.030 in. out of round.

Design. – Piping stress analyses of the primary piping system resulted in some rearrangement of the pump and heat exchanger piping.

Technical evaluations of the bid proposals for the primary heat exchanger and circulating pump were completed.

Layout drawings of the reactor component arrangement in the region of the core have been prepared. Sufficient detail is included to complete the analysis of the coolant flow requirements in the region of the target, core, reflector, and beam tubes. Particular attention is being given to the problem of the hydraulic forces encountered in the region of the control rods during operation in the present concept.

The four engineering facility holes have been moved 2 in. into the reflector region.

Drawings and specifications are being prepared for issue on the beam tubes, bellows, and thimbles.

Fabrication drawings of the mechanical control drives are 75% complete. Shop orders have been placed on many parts.

A number of drawings were prepared to show the arrangement of the beam tube and shutter concept, which should provide adequate flexibility to allow adaptation to specific needs in the future. The drawings have been worked out only in sufficient detail to establish that this concept will work and to allow dimensioning of the parts that must be embedded in the concrete.

Critical Experiment. – The apparatus for a second series of critical experiments in support of the design of the HFIR is 90% complete. These experiments will be performed with a much more faithful mockup of the reactor than were the earlier ones, since it utilizes a uranium-aluminum alloy plate-type fuel element, a beryllium reflector, natural water moderation, and two three-section control sleeves. The sections of the sleeves are equal-length cylinders of aluminum, nickel, and a copper-silver alloy – neutron absorbing materials of different intensity – joined into a single cylinder of height about three times that of the fuel element. The control system consists of the two cylinders, concentric with and adjacent to the fuel and independently operable, with the order of the materials in one inverted with respect to the other. In this manner combinations of the three materials may be juxtaposed to the fuel for a wide range of control.

The two 20-in.-long annular sections of the fuel element were constructed separately. The larger, having diameters of 17 and 11 in., contains 6 kg of enriched uranium; the diameters of the smaller are 10.5 and 5.4 in., and it contains about 2.5 kg of uranium. The larger element was submerged in water, in the absence of the beryllium reflector, and found to be very subcritical. It was also subcritical both with the water in the central region removed and with annuli of water, of several thicknesses, adjacent to the inner surface of the element. The experiment will be repeated with the two annular sections assembled. The results will provide firm bases for safe practices during the fabrication, storage, and shipment of elements of this type.

## MARITIME REACTORS PROGRAM

Physics. – Calculations for a cold, clean, 4.2 wt % enriched, Zircaloy egg-crate core for the NS "Savannah" reactor have been completed. Comparison of the results with previous calculations for a 4.2 wt % enriched, stainless steel egg-crate core indicate that the substitution of Zircaloy for stainless steel would increase the cold multiplication factor by 0.075 and reduce the shutdown margin of the core with control rods by 0.008. These results are qualitatively in agreement with the Marty II and Marty III critical experiment results obtained by the Babcock and Wilcox Company for the AEC. The critical experiments indicated that replacement of steel by aluminum in a core of this type causes a greater increase in reactivity than in control-rod worth.

Irradiation Tests. – The experiment No. 1 fuel specimens (six swaged $UO_2$ fuel rods) which were irradiated to an estimated peak burnup of 1700 Mwd per metric ton of $UO_2$ in the ORR pressurized-water loop are under postirradiation examination at the G-E Vallecitos facility. The clusters and individual rods have been visually examined, photographed, and measured. No gross dimensional changes were noted. Gamma scans of the rods showed dips which may indicate fuel separations. Irradiation of experiment No. 3 fuel specimens is continuing. The present estimated peak burnup is 2400 Mwd per metric ton of $UO_2$.

## ARMY PACKAGE POWER REACTORS

Metallurgy. – Fuel element S79, which was exposed in the SM-1 reactor at Fort Belvoir for 16.4 Mwyr (approximately 42% $U^{235}$ burnup), is undergoing detailed postirradiation examination in the ORNL hot cells. A cursory visual inspection revealed no signs of failure.

A boron-gradient absorber section containing 10 g of $B^{10}$ dispersed in a special type 200 austenitic stainless steel matrix and clad with type 304L stainless steel has been manufactured and shipped to Alco Products, Inc., for nuclear-worth measurements. In this absorber the boron is dispersed through the plate thickness in a manner which minimizes surface-concentrated irradiation damage. The boron-gradient absorber will be tested in core II of the SM-1 reactor.

Studies are in progress on the use of borosilicate glasses as burnable poison in composite $UO_2$–stainless steel dispersion fuel plates. Results obtained thus far indicate that boron losses during plate processing are not detectable, whereas similar samples processed with other boron-bearing compounds show erratic and often large boron losses.

## ANP PROGRAM

Shielding Research. – A series of experiments was begun at the Tower Shielding Facility with the TSR-II in the Pratt and Whitney reactor shield mockup in order to check the shield design calculations. Traverse measurements made close to the shield showed that the ratio of the front-of-the-shield dose to the back-of-the-shield dose for both fast neutrons and gamma rays roughly confirmed the design calculations. A reactor power level of 40 w was used during the fast-neutron dose measurements, and the power level for the gamma-ray measurements was 200 w.

Significant progress was made at the Bulk Shielding Facility in the program for determining the energy and angular distribution of the thermal and epithermal neutrons leaking from and within shields. The angular distributions were determined at 4.95 ev, at 1.44 ev, and at subcadmium energies for neutrons leaking from a 4-in.-thick slab of lithium hydride. It is expected that this information can be used to predict the angular distribution of the neutron leakage from lithium hydride at any epithermal energy.

The energy spectra of neutrons leaking within an infinite medium of lithium hydride were calculated by the NDA moments method. Calculations were made for four input neutron energies ranging from 16 kev to 16 Mev at positions in the slab which were 11, 23, and 34 g/cm$^2$ from the source. These calculations lead to the preliminary conclusion that there are no marked differences between the energy spectra of the leakages from 1- and 16-Mev neutrons. More accurate calculations are planned with the NDA Niobe code, which will use a medium of finite thickness and will yield information on angular distribution as well as energy of the leakage.

Preliminary measurements were made at General Atomic which indicated that neutron pulses from targets in the General Atomic electron linear accelerator could be used to measure the energy spectra of the neutron leakage from lithium hydride at one or more angles. This information could be combined with the experimental data on angular distribution to provide a cross check with the calculations.

Columbium Alloy Research. — The apparatus containing the first group of Cb—1% Zr alloy in-pile tube-burst specimens was disassembled. The brightness of the specimens indicated that a fairly good inert atmosphere had been maintained during the irradiation. The assembly containing the second group of specimens, which ruptured at high pressure, is being disassembled. A third in-pile apparatus is under construction for use in an experiment with a lower applied stress. The lower stress should permit a longer period of irradiation prior to failure.

Data were obtained which indicated that oxygen plays an important role in the aging of Cb—1% Zr alloy. The data on wrought material showed that the addition of oxygen to the alloy raised the annealing temperature required for aging to occur. Heat No. S24 WC-4 normally contains 250 ppm of oxygen and undergoes aging at 927°C after being annealed at 1600°C for 2 hr. When specimens of the material were contaminated with oxygen to levels ranging from 460 to 1200 ppm prior to annealing at 1600°C for 2 hr, no aging effects were produced by treatments at 927°C. A 2-hr anneal at 1800°C resulted in aging reactions at 927°C in specimens containing 470 to 700 ppm of oxygen, but no aging was detected in specimens containing 800 to 1200 ppm of oxygen. These data do not rule out the possibility of carbon and/or nitrogen having an effect on the aging of the alloy. However, tests of high-purity Cb—1% Zr alloy containing varying amounts of carbon showed no aging after a 1600°C anneal. Preliminary tests with specimens contaminated with nitrogen also showed no aging.

When normal low-oxygen-content specimens of heat XM-339 of Cb—1% Zr alloy are welded, the welds have good ductility. When this material was contaminated to oxygen levels between 820 and 4200 ppm prior to welding, brittle welds were produced. A 3-hr anneal at 2000°F or above after welding was adequate to eliminate the embrittlement. Annealing carried out at 1900°F was not effective.

Liquid-Metal Technology. — Construction of the stainless steel apparatus for the study of boiling-potassium heat transfer was completed. The apparatus was assembled with the low heat flux test section for the preliminary experiments. The initial charge of potassium was introduced and circulated several hours for loop cleanup. It was apparent that the system required only minor adjustments prior to initiating a program of measurements.

The Haynes alloy No. 25 corrosion loop for circulating boiling and condensing potassium at 1600°F was modified to remove operational instabilities, and similar modifications were made on a recently constructed Inconel loop. A natural-circulation boiling loop test system for circulating potassium at temperatures of 1800 and 2000°F is being designed.

Beryllium Oxide Research. – The sixth experimental assembly for irradiating beryllium oxide specimens was inserted in the ETR. This apparatus provides for the irradiation of six specimens of isostatically pressed BeO, 1.18 in. in diameter and 3 in. long. Some of the specimens are clad with stainless steel and some are unclad. The temperature at the center of the specimens will be 800 to 1000°C.

The "porous collector" method, a technique whereby very fluid eutectic liquids in BeO systems are removed by absorption in a porous BeO collector, was used to study the $BeO-ZrO_2$ systems. The following observations were made: (1) the system is very probably a simple eutectic, (2) the eutectic temperature is 2045 ± 10°C, (3) the eutectic composition is 58.7 ± 2 mole % BeO, (4) no solid solubility of BeO in $ZrO_2$ or of $ZrO_2$ in BeO was detected by x-ray examination of specimens prepared by the porous-collector technique. The limit of detection of these methods is below 1 mole % of the solute.

## NUCLEAR TECHNOLOGY AND GENERAL SUPPORT

Fuel Element Catastrophe Studies. – The initial melting experiments demonstrating fission product release from $UO_2$ in various atmospheres as a function of burnup were completed for air and helium atmospheres. The results conform to the expected pattern. Increased releases were observed over previously reported experiments at tracer level. As in the tracer-level experiments, 5 to 10% of the $UO_2$ was vaporized in air but only a trace was vaporized in helium. The release percentages are compared in the table below, which also contains some average releases observed on melting in $CO_2$. All experiments were performed by the carbon arc—image furnace method.

| Atmosphere | Per Cent of Individual Fission Product Released | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Xe-Kr | I | Te | Cs | Ru | Sr | Ba | Rare Earths |
| | | | | Tracer-Level Experiments | | | | |
| Air | 98.2 | 95.0 | 79.0 | ~40.0* | 66 | 0.2 | 0.5 | 0.5 |
| Helium | 99.7 | 90.0 | 92.0 | 91.0* | 61 | 2.0 | 4.9 | 2.2 |
| $CO_2$ | 80.6 | 76.8 | 71.2 | ~50.0* | 44.9 | 0.2 | 1.1 | 0.85 |
| | | | | ~1600 Mwd/ton Burnup** | | | | |
| Air | 100.0 | 99.7 | 93.6 | 92.5 | 95 | 0.4 | 1.8 | 3.0 |
| Helium | 99.9 | 92.2 | 98.2 | 98.5 | 90.4 | 2.1 | 6.6 | 5.0 |

*Cesium analyses at tracer level subject to further study.
**GCR-test $UO_2$, ~95% of theoretical density; irradiation estimated.

Reactor Evaluation Studies. – The fuel value of plutonium in Dresden-type reactors was studied as a function of the economic ground rules used for uranium. The present cost of enriched uranium was calculated by using the standard relationship involving natural-uranium cost and separative-work cost. Three cases were considered: case 1 assumed "present" uranium costs and 4%/yr inventory charges; case 2 considered the natural-uranium cost to be 75% of present cost, and 5%/yr inventory charges; case 3 considered the natural-uranium cost and also the separative-work cost to be 75% of present costs, and 5%/yr inventory charges. For uniform-batch irradiation and the same fuel fabrication cost, independent of fuel composition, the value of fissile plutonium in the second cycle was $11.40, $9.50, and $7.50 per gram for cases 1–3 respectively. Taking into consideration the change in enriched-uranium value as the economic ground rules were changed, the corresponding value for fissile plutonium when normalized to present cost was $11.40, $10.90, and $10.10 per gram for cases 1–3 respectively. Thus, if the fuel value of plutonium can be specified relative to the present enriched-uranium price schedule, its value relative to possible future price schedules appears to vary in about the same fashion as the $U^{235}$ value.

Radiation Detector Development: Gamma-Ray Spectrometer. – Investigations designed to optimize the performance of the model IV gamma-ray spectrometer of the Bulk Shielding Facility have been outlined previously (ORNL-3016, p 264). The experimental measurements are now nearly completed, and analysis of data is in progress. The work to date has indicated significant progress in the identification and separation of radiation backgrounds attendant to the use of the model IV spectrometer. A most encouraging result of the work already completed has been the exemplary performance of the 9-in.-diam, 12-in.-long NaI(Tℓ) composite crystal. Resolution of the order of 12% has been obtained, and no spurious peaks or other unusual effects have been observed.

Radiation Detector Development: Neutron Spectrometer. – The feasibility of the use of the solid-state neutron spectrometer (ORNL CF-60-5-121) in the measurement of neutron energy spectra has been demonstrated by a recent experiment at the Tower Shielding Facility. The leakage spectrum of the Tower Shielding Reactor II was observed over an energy range from ~1 to 10 Mev. Over this range the ratio of foreground to background was 5:1. Other tests have been successfully run with monoenergetic neutrons of energies as high as 8 Mev.

Radiation Detector Development: Analysis of Flux Data. – The Fortran coding system for the IBM 704 and 7090 data processing machines has recently been employed for the construction of two codes designed to facilitate analysis of gold foil data obtained at the Lid Tank Shielding Facility (LTSF). The first code evaluates the saturated activity per unit mass of gold foils exposed to the LTSF thermal flux. The output of the code consists of the weighted mean of the saturated activity per unit mass and the standard error of the weighted mean. Also given as output are the saturated activity per unit mass for each foil count and the variance associated with this value. The second code is essentially a curve-fitting routine. A function, which must at least be linear, but which may be as detailed as a tenth-order polynomial, is fitted to the

data by the method of least squares. The output gives the coefficients of the polynomial and the variance-covariance matrix of the coefficients, as well as the sum of the squares of the residuals. The output also evaluates the sum of the squares of the residuals divided by the number of degrees of freedom, a quantity which approaches unity as the fit is improved. Finally, the output evaluates the polynomial at selected points as directed by the input, giving also the variance-covariance matrix for these values. Running time and costs involved in the use of these codes are very small, representing considerable savings over even the most economical methods of analysis and calculation by hand.

Chemical Behavior of Thermoelectric Materials. — In studies of the thermoelectric characteristics of nearly stoichiometric $UO_2$, it is considered important to be able to correlate the composition of the material with some physical property. Moreover, it is desirable that this property be one which can be determined with reasonable precision in many different laboratories. The x-ray parameter $a_0$ was chosen for initial tests, and the Debye-Scherrer powder technique was used. (The precision routinely available by this technique is considered adequate, although it could be improved substantially by taking very great care in the measurements or by using single-crystal techniques.) The uranium dioxide measured had been prepared by hydrolytic precipitation from molten fluorides and analyzed for oxygen content by the ORNL-Kubota method. The $a_0$ value established for $UO_{2.00\pm0.003}$ was 5.472 ± 0.002 A.

Zirconium Metallurgy: Melting of Technetium. — The world's largest known melt of technetium was made by the arc melting of a 5-g pellet pressed from technetium metal powder. Press forging and rolling of the arc-melted button to more than 50% reduction resulted in longitudinal cracking of the strip. The metal is soft and inherently ductile, the cracking apparently resulting from the very coarse columnar grain structure that existed in the button. It is believed that the solution to the problem of cracking during fabrication can be readily found. Larger melts and fabrication studies are planned in the near future.

Radiation safety procedures are being developed and examined as the metallurgical work proceeds. The results to date indicate that strict containment in remote facilities is not required, that ordinary glovedbox techniques are adequate, and that the consolidated metal can be handled without a dusting problem. The contamination of the rolling mill resulting from rolling the bare metal is not serious and is easily and readily cleaned up. The use of tweezers, tongs, and rubber gloves for handling the consolidated metal adequately limits personnel exposure.

Solid State Reactions. — The reaction between hot-pressed beryllium and arc-cast uranium monocarbide was studied. Heat treatments of 185, 162, 122, and 90 hr at 700, 800, 900, and 1000°C, respectively, resulted in reaction layers 7, 115, 150, and 176 μ thick. A powder sample of this product was obtained with the ultrasonic microchisel and identified by x-ray diffraction as $UBe_{13}$.

Fuel Element Development. — Beryllium oxide bodies containing 30 vol % fuel as $UO_2$ and as a $UO_2$-$ThO_2$ mixture, uniformly dispersed as submicron

particles, have been fabricated by cold-pressing and sintering to 95% of theoretical density and to close dimensional specifications for irradiation tests. The problem of cracking of the body, which was encountered initially, was solved by adjusting the shrinkage rate of the $UO_2$ powder through control of the O/U ratio during sintering.

Beryllium oxide bodies containing 30 vol % $UO_2$ incorporated as the more desirable large particles, both 100 to 150 μ and 150 to 250 μ, were fabricated to 95% of theoretical density by preshrinking the $UO_2$ so that its residual shrinkage approximated that of the BeO; however, occasional microcracks were present.

At a slight sacrifice in density, to 93%, it was possible, by adjusting the heating rate during sintering, to fabricate crack-free bodies from the 100- to 150-μ $UO_2$. Nevertheless, cracking was still found occasionally with the large-size grains.

Single crystals of calcium beryllate ($Ca_2Be_3O_5$) were successfully grown from supercooled melts of BeO-CaO mixtures.

A technique employing the very fluid nature of eutectic liquids in BeO systems was used to establish the eutectic points in three high-temperature, two-component BeO systems. The eutectic temperature and composition were found to be 1860 ± 10°C and 69 ± 2 mole % BeO in the MgO-BeO system, 1890 ± 20°C and 63 ± 3 mole % BeO in the $CeO_2$-BeO system, and 2085 ± 10°C and 58.7 ± 2 mole % BeO in the $ZrO_2$-BeO system.

Nondestructive Test Development. — A new electrode-potential test has been investigated for the evaluation of the integrity of oxidation-resistant coatings on graphite. A galvanic cell is established with the coating as one electrode, a suitable electrolyte, and a secondary electrode. In one such test cell for an Si-SiC coating, a dilute solution of chromic and sulfuric acids was used as the electrolyte and zinc as the secondary electrode. The generated emf is the sum of the oxidation potentials of the two electrode materials. The voltage observed for the Si-SiC/Zn cell was about 0.75 v. On the other hand, the voltage generated for a graphite/zinc cell containing type 901S graphite was about 2.1 v. Tests were conducted on graphite coatings which contained purposely made voids to expose the underlying graphite to varying degrees. Within limits it appears that the generated emf is a function of the exposed area. For example, for a 1/16-in.-diam exposed area a voltage of 1.2 v was noted, whereas for diameters of 5/32, 11/32, and 15/32 in. voltages of 1.6, 1.9, and 2.1 v, respectively, were noted.

It has been demonstrated that the generated voltage can readily be displayed on a strip-chart recorder. Thus, with appropriate mechanical scanning this test technique should be applicable to coating-integrity evaluation for many graphite components. A probable use will be for testing the coating on the graphite sleeve for the EGCR fuel assembly.

Mechanical Properties Research. — A program is being conducted to evaluate the effects of hydrogen on the mechanical properties of various structural materials at elevated temperatures. The materials under study include commercial Inconel, nickel, several Ni-Fe-Cr alloys, types 347 and 304 stainless steel, and columbium. The following significant observations have been made to date.

1. The secondary creep rate of each of the nickel-base alloys listed above is greater in hydrogen than in argon. The rupture life is

less in a hydrogen environment than in an argon environment. The largest effect was noted in commercial Inconel.

2. The magnitude of the creep acceleration observed in commercial Inconel appears to be sensitive to strain rate, the effect decreasing as the strain rate is increased.

3. Although the creep rate of the above nickel-base alloys is accelerated by the presence of hydrogen, no reduction in ductility is observed.

4. Types 304 and 347 stainless steel show similar creep behavior in argon and in hydrogen in tests that have been run for as long as 2000 hr.

5. Hydrogen increases the creep rate of columbium at elevated temperatures, although no reduction in ductility is observed.

It is felt that the high-temperature hydrogen effect that has been observed in these studies is not the same as the low-temperature hydrogen embrittlement that has been observed in steels.

Materials Compatibility Studies. – Type 310 stainless steel and Inconel have been exposed to refluxing potassium in capsule tests for 1000 hr at a boiler temperature of 1600°F. Machined sleeve-type inserts lined the entire length (18 in.) of the test capsules in order that both metallographic and weight-change data could be obtained on the various regions of the test system. The type 310 stainless steel inserts showed slightly larger weight losses in the condenser region and larger weight gains in the boiler (liquid) region than the Inconel inserts did.

Fuel Cycle Technology. – Two 1-kg lots of $UO_2$-$ThO_2$, containing 4.4% enriched uranium, were prepared by sol-gel processes for irradiation testing in the NRX, one by steam denitration of thorium nitrate and the other from calcined thorium oxalate. The particle size of both products ranged from 10 to –200 mesh, with 80% > 16 mesh. Particle densities were 9.94 and 9.92 g/cc, both greater than 99% of theoretical. After grinding and sizing, the oxides were compacted into 12 tubes, 1/4 in. ID by 10 in. long, to a bulk density of 8.7 ± 0.05 g/cc.

Hydrous urania gels prepared from uranous formate solution and calcined at 1100°C in hydrogen gave fragments of 0.8 mm average size, 96.7% of theoretical density. This indicates that it may be possible to prepare dense, large particles of $UO_2$ by the sol-gel process.

Power Reactor Fuel Processing: Mechanical Processing. – Performance testing of the SRE dejacketing equipment with NaK-filled prototype fuel rods resulted in (1) trouble in starting up equipment that had been idle for some time, (2) a dejacketing rate of ~1 rod per hour (10 kg/hr), (3) difficulty in the collection of NaK, and (4) satisfactory over-all performance of equipment and procedures at the end of the tests. The three batches of spent SRE core 1 fuel are scheduled for shipment from the SRE site on April 3, May 1, and June 1. In a fire extinguishment test, burning NaK was extinguished by submerging in Bayol-D.

In back-mixing tests on the rotary screw conveyor tilted to 20° and run at 2 rpm, 40% back-mixing was observed. A total of 13 revolutions vs 10 theoretical is required to empty the conveyor.

A 1/16-scale nine-stage leacher model run with PWR blanket $UO_2$ pellets on a 4-hr dissolution and using 7 M $HNO_3$ at a 20.6 ml/min feed rate

indicated that steady state was attained in 4 to 5 stages. The product composition at apparent steady state varied from 500 g of U per liter and 1.0 M $H^+$ to 375 g of U per liter and 2.1 M $H^+$.

A single-stage dissolution of crushed $UO_2$ pellets in 7 M $HNO_3$ resulted in a peak uranium loading of 500 g/liter and 1.4 M $H^+$ as compared with 375 g/liter and 2.8 M $H^+$ for a similar run with whole $UO_2$ pellets. Instantaneous dissolution rates for these $UO_2$ pellets under simulated inclined-drum rotary leacher conditions in boiling dissolvents analyzing 2 to 8 M $HNO_3$–0 M U and 0.5 to 2.5 M $HNO_3$–2 M U were correlated by

$$\log R = -0.28 + 0.13\ (\underline{M}\ U) + [0.18 + 0.62\ (\underline{M}\ U)^2](\underline{M}\ H^+)\ ,$$

where R is the rate in mg $cm^{-2}$ $min^{-1}$ based on the superficial surface area of right-cylindrical pellets.

Power Reactor Fuel Processing: Beryllia- and Alumina-Containing Fuel. – Addition of 0.1 M $NaH_2PO_4$ to boiling 8 M $HNO_3$ did not increase the rate of dissolution of sintered 70% $UO_2$–30% BeO fuel pellets. High-fired beryllia dissolved at rates of 0.05 and 0.46 mg $cm^{-2}$ $min^{-1}$ in glacial acetic acid solutions containing 1 and 5 M $NH_4HF_2$ respectively.

Uranium losses from "heterogeneous" $UO_2$-$Al_2O_3$ fuel pellets leached for 4 hr with boiling 10 M $HNO_3$ increased from less than 0.2% to about 48% when the $Al_2O_3$ content of the pellets was increased from 10 to 75%. Under identical processing conditions, losses from "homogeneous" pellets were about twice as high as those from "heterogeneous" pellets. The "homogeneous" pellets contained 10-μ $UO_2$ grains dispersed in $Al_2O_3$; the $UO_2$ particle size in the "heterogeneous" pellets was about 150 μ.

Power Reactor Fuel Processing: Corrosion Studies. – In 10% fuming $HNO_3$–90% fuming $H_2SO_4$ at 50°C, specimens of LCNA and type 309SCb stainless steel suffered extensive vapor-phase weld decay and intergranular attack. Over-all rates were 0.90 and 2.89 mils/month, respectively, for 336 hr of exposure.

Titanium 45A and Hastelloy F exposed to spent $UO_2$ core dissolution solutions containing 0.01 M $Zr^{4+}$ and 0.05 M NaF were corroded at maximum over-all rates in the vapor phase of 9.01 and 0.29 mils/month, respectively, in 336 hr of exposure. Addition of 0.05 M $Al(NO_3)_3$ decreased the over-all rates to 3.55 and 0.12 mils/month, but titanium specimens in the vapor phase were heavily pitted.

Power Reactor Fuel Processing: Solvent Extraction. – From a synthetic $U^{233}$ product solution 0.24 N in $HNO_3$ and containing 25 g of U per liter and 0.24 g of Th per liter, 99% of the thorium was removed by passing the solution through Dowex 50W resin at 0.7 ml $min^{-1}$ $cm^{-2}$. Only 1% of the $U^{233}$ was sorbed. About 8 g of uranium passed through the column for each milliliter of resin.

The distribution coefficient of $Nb^{95}$ between purified 30% tributyl phosphate in dodecane and aqueous 4 M $HNO_3$ ranged from 0.05 to 0.8, indicating that more than one species of niobium was present.

Zirconium-niobium-95 tracer extracted by 30% TBP in Amsco degraded with 2 to 6 N $HNO_3$ at room temperature was successfully scrubbed from the solvent with three equal-volume aliquots of 2 N $HNO_3$. Tracer extracted by solvent degraded at 106°C was not removed by this treatment.

Power Reactor Fuel Processing: Graphited Fuel. — In laboratory experiments at 60°C with unirradiated fuel specimens containing 3% uranium, the uranium loss to the graphite residue after a single 8-hr leach with 90% $HNO_3$ was 3.5%; losses were less than 0.1% after four 2-hr leaches, both with and without intermittent water washing.

In engineering-scale tests, neutron activation analysis of graphite—3% U fuel that had been leached with 90% $HNO_3$ at 80°C indicated the undissolved uranium content to be ~600 and 200 ppm for particles less than 175 and 15 μ respectively. The average uranium concentration at 24 hr was not significantly less than at 3 hr.

An alternative process for graphited fuel now under study is the burning of fuel and leaching of the residual ash. Combustion, in oxygen at 900°C, of slightly irradiated (~0.001% burnup) fuel specimens containing 3 and 10.9% uranium resulted in the volatilization of 60 and 40% of the $Ru^{106}$ respectively. All the $Zr^{95}$, $Ce^{144}$, and $Cs^{137}$ remained with the combustion ash. A preliminary cost comparison of oxygen combustion vs fuming nitric acid disintegration was begun; separation of $Kr^{85}$ and other volatilized fission products from flue gases containing substantial concentrations of $CO_2$ appears to be a major problem.

Uranium monocarbide reacted with 4.38 M $HNO_3$ to produce NO, $CO_2$, and a little $N_2O$ as the principal gaseous products. No hydrocarbons were detected in the off-gas. In one experiment the resulting nitric acid—uranyl nitrate solution was dark red and contained 1 N $H^+$ and probably about 50% of the original carbon. In the other experiment the solution was orange-red and contained 4 N $H^+$ and probably about 35% of the original carbon.

Power Reactor Fuel Processing: Zirflex Process. — Laboratory tests indicated that in modified Zirflex dissolutions of zirconium-containing fuel in 6.5 M $NH_4F$—0.33 M $NH_4NO_3$—0.01 M $H_2O_2$, the hydrogen peroxide should be added continuously, rather than in batches, and its concentration maintained below 0.01 M to ensure minimum peroxide loss. The first-order decomposition rate constant for $H_2O_2$ was measured as 0.03 $hr^{-1}$. Three batch dissolutions were made in engineering-scale equipment in which 1450- to 1850-g sections of unirradiated U-Zr-Sn multiplate assemblies were treated with 17 to 20 liters of dissolvent to which $H_2O_2$ was added continuously. No elemental Sn or $UF_4$ precipitation was observed at $H_2O_2$ addition rates of $0.5 \times 10^{-5}$, $0.4 \times 10^{-5}$, and $0.75 \times 10^{-5}$ mole $min^{-1}$ $cm^{-2}$ for fuel alloy containing 1.24% U—0% Sn, 2.73% U—0.26% Sn, and 2.6% U—1.6% Sn respectively. The off-gas was principally hydrogen, and its rate of evolution was inversely proportional to the amount of zirconium oxide coating on the metal surfaces.

Corrosion tests on welded specimens of LCNA and Hastelloy F showed over-all rates of 3.09 and 1.15 mils/month in 504 hr exposure in flowing 5.4 M $NH_4F$—0.33 M $NH_4NO_3$—0.003 M $H_2O_2$ at 99°C. The Hastelloy F showed extensive weld decay, while the LCNA showed relatively mild grain-boundary attack.

Fluoride Volatility Processing. — In four complete flowsheet runs in the Volatility Pilot Plant with nonirradiated enriched uranium fuel elements, an HF feed rate of 150 g/min was set as the tentative standard. An instrument dip pipe was substituted for the distribution plate at the HF inlet with no apparent differences in results, and a dip pipe was added for use when necessitated by distribution-plate plugging.

A copper Yorkmesh filter removed 90% of the (simulated) solids from hydrofluorinator off-gas. The solids that passed the filter were of extremely small size. The pressure drop across the filter was a function of total loading but was independent of the solids concentration in the gas stream. A loading of 200 to 255 g of solids on a 3-in.-diam × 6-in.-deep filter resulted in a pressure drop of 3.5 to 3.9 psi with an air flow of 110 std $ft^3/hr$.

A movable-bed sodium fluoride absorber appeared satisfactory in initial tests. It has four temperature zones and two outlets: a trap for complexable radioactive products operated at 400°C, a transition zone for cooling to 100°C for absorption, a first absorber section with an outlet for use in absorption, and a second absorber section with an outlet to the cold traps for use in desorption. After each run, part of the 400°C bed is discharged to waste and the remainder advances.

In tests on $UF_6$ absorption on deep beds of sodium fluoride, at constant HF concentration and total gas flow, the absorption rate decreased with increasing temperature. The rate was increased by $F_2$ pretreatment of the sodium fluoride pellets; a weight loss in the pellets indicates that some impurities are volatilized in the $F_2$ treatment.

In initial tests on the volatilization of $PuF_6$ from fused fluoride salts, with 2 ppm Pu in 31-24-45 mole % $LiF-NaF-ZrF_4$, > 99% volatilized at 600°C. Results were comparable with a 50-50 mole % $NaF-ZrF_4$ melt. The rate of $PuF_6$ volatilization was closely proportional to the fluorine flow rate.

## PHYSICAL RESEARCH PROGRAM

Reactor Operations. — Work is continuing on the ORR control rods, which have required considerable mechanical maintenance recently. Hardened plungers have been installed in the ball-release mechanisms and appear to be an improvement.

Preliminary engineering is almost complete on the proposal to increase the ORR power to 40 Mw.

Waste Disposal. — The second seepage trench is expected to be built by July, at which time it will be possible to discontinue operation of the open waste pits.

Recent monitoring in the White Oak Creek system has permitted a better identification of sources of radioactivity releases than has been possible in the past. Practically no strontium is being released from the waste pits, although considerable amounts of ruthenium are released.

Hot-Cell Operations. — All the Physical Examination Hot Cells, Building 3025, are now in operation. Work in the ORR and the 3026D cells has been exceptionally heavy recently. Several overtime shifts have been worked in recent weeks.

Cold runs in the 3026D dejacketing experiment are being made and are reasonably successful. It is expected that the first shipment of SRE fuel elements will be received about the middle of April.

## PHYSICS AND MATHEMATICS RESEARCH

Charge Spectrometry. — Measurements have been completed on the energy and charge spectra of the recoil $Li^6$ ions from the beta decay of $He^6$ (ORNL-2991). The accuracy of the measurements has been improved, particular emphasis being given to a study of the detection efficiency of the electron multiplier. Recoil energy spectra have been obtained under a variety of experimental conditions for each of the $Li^6$ charge species. The relative intensities of the species are $(89.4 \pm 0.3)\%$ $Li^+$, $(10.5 \pm 0.3)\%$ $Li^{2+}$, and $(0.050 \pm 0.005)\%$ $Li^{3+}$; the production of the multiply charged ions has some dependence on recoil energy. These results are consistent with theoretical expectations. The total recoil spectrum obtained by summing the charge species shows good agreement with predictions for the axial-vector interaction. An evaluation of experimental uncertainties is in progress. At the present stage of analysis the angular correlation coefficient, $\lambda$, is $-0.335 \pm 0.010$ (axial-vector interaction, $\lambda = -1/3$). An absolute determination of the end point for the spectrum yields $3.507 \pm 0.004$ Mev for the decay energy of $He^6$.

Neutron Diffraction. — In a continuing program of investigation of the magnetic properties of rare-earth metals, a series of experiments designed to study the magnetization process in metallic holmium has been carried out. At 4.2°K, a temperature at which the magnetic structure of holmium in the virgin state is a somewhat distorted screw structure, the application of a magnetic field normal to the c axis produces a transformation directly to the ferromagnetic state, in which there is an

extremely large basal phase anisotropy. The $b_1$ directions, 30° from the crystallographic $a_1$ directions, were found to be the easy directions of magnetization. At 64°K the magnetization process is somewhat more complicated, and at least one intermediate state, in which the magnetizations of the individual layers oscillate about the field direction, is observed.

Fast-Chopper Time-of-Flight Spectrometer. — A cerium-activated, $Li^6$-loaded glass scintillator of improved scintillation efficiency and increased $Li^6$ content has been developed and has been used for detecting neutrons with energies from 1 ev to 80 kev by time-of-flight techniques. The scintillator was found to have the following properties: (1) an efficiency of 30% for detecting 1-kev neutrons, (2) a resolution of 18% for the $Li^6(n,\alpha)T$ peak, and (3) a time resolution of $5 \times 10^{-9}$ sec or better when used with a "fast-slow" coincidence system. The scintillator was used with the ORNL pulsed Van de Graaff and the associated nanosecond time-of-flight equipment to measure the neutron spectrum from the reaction $Li^7(p,n)Be^7$ for neutron energies from 5 to 80 kev. The detector was used with a fast-chopper neutron spectrometer to obtain total neutron cross sections up to 5 kev. Lithium- and boron-loaded glass scintillators were compared, and it was concluded that lithium glass is, in general, more suitable for neutron detection.

High-Voltage Experimental Program: Fission Fragment Angular Distributions. — Fragment angular distributions have been measured for the fast-neutron-induced fission of $U^{234}$ and $U^{235}$ for neutron energies up to 3600 kev. The $U^{235}$ distribution shows a slowly increasing amount of forward peaking with increasing neutron energy up to about 1200 kev, after which there is very little change, the ratio of the fragment intensity at 0° to the intensity at 90° remaining very close to 1.13.

The $U^{234}$ distribution exhibits strong sidewise peaking at low neutron energies, becomes isotropic at about 650 kev, and then more and more strongly forward peaked until an energy of 845 kev is reached, after which the extent of the anisotropy decreases. Least-squares fits to the data have been found for three energies for each isotope. In the case of $U^{234}$ near threshold, fission is expected to take place via a relatively small number of levels. More detailed calculations made at the 845-kev point indicate that fission occurs through the K = 1/2 rotational band. It is possible to fit the angular distribution in the following way:

$$W(\theta) = (1.441 \pm 0.051)W_{1/2}^{1/2} + (0.747 \pm 0.048)W_{1/2}^{3/2} + (0.234 \pm 0.039)W_{1/2}^{5/2}.$$

If the distribution is fitted with functions pertaining to the K = 3/2 band, a negative coefficient occurs for the $W_{3/2}^{5/2}$ term, which signifies an unreal situation, since contributions from the different levels add incoherently.

The drop in cross section and asymmetry between 845 and 1050 kev is believed to be due to competition from inelastic scattering by emission of an $\ell = 0$ neutron from the (1/2, 3/2, -) level to a $1^-$ level at 788 kev in $U^{234}$.

High-Voltage Experimental Program: Nanosecond Beam Pulsing. — A pulser has been built to sweep a 10-ma focused beam of protons from a

duoplasmatron ion source across an aperture to produce short bursts of protons for use in a Van de Graaff accelerator. The performance of such systems is intimately linked with the quality of the focus: the smaller this focus is, the shorter the pulses that can be made. Special low-aberration lenses are being used.

An extensive set of calculations led to the conclusion that focus diameters were larger than predicted because of the coarseness of the grids being used. New fine-mesh grids were installed, and, as predicted, a factor of 10 improvement in focus current density was obtained. With smaller foci, it has been possible to reduce the size of the sweep aperture, with the result that pulse durations of 1 nanosecond are now available at high current values.

Erosion of the lens grids aroused considerable concern. It was found that the sweep deflector plates can be placed above the lens so that the beam goes through the grids only 1% of the time. This has reduced grid erosion to a completely safe level.

Spectroscopy Research Laboratory. — Studies of solid solutions of polyatomic inorganic ions in alkali halides have shown that many of the vibrational absorption bands are extremely narrow (less than 1 $cm^{-1}$ half-width). This characteristic has suggested that these lines may provide a satisfactory frequency calibration standard in the infrared region. In the search for proper absorption frequencies, over 100 different inorganic salts were fused in alkali halides and quenched to solid solutions, yielding approximately 45 sharp absorption bands and a roughly equal number of broad bands. Several new ionic species have been observed in these spectra, but have not yet been identified. The bandwidths can be qualitatively explained by the relationships between the ionic and lattice dimensions, and between the symmetries of the ions and the lattice sites which they occupy. Sixteen sharp bands have been chosen to provide calibration points spaced throughout the region 2200 to 700 $cm^{-1}$, associated with the use of a sodium chloride prism. Preliminary studies indicate that these bands will be satisfactory also for the measurement of slit widths.

## CHEMISTRY RESEARCH

Solvent Extraction Research. — Improved separation of uranium from Zr-Nb and Ru during tributyl phosphate extraction has been noted previously when the aqueous feed is treated with nitrous acid and acetone. A plausible explanation, the formation of isonitrosoacetone, a keto oxime, which strongly complexes Zr-Nb and Ru in the aqueous phase, was supported by finding that diacetyl monoxime, 0.05 M in aqueous 2 M $HNO_3$, depressed the Zr-Nb extraction coefficient of 1 M TBP by a factor of 30 without affecting the uranium coefficient.

Diisopropylbenzene was the most satisfactory diluent found in a study of the amine extraction process for separation of transplutonium elements from fission product rare earths. It has a flash point of 170°F, and a 1 M solution of triisooctylammonium chloride in it was used to extract americium from 8 N LiCl–2 N $AlCl_3$ containing europium. The

extraction coefficient for americium was ~10, and the separation factor with respect to europium was ~100. Phase separation problems appeared no more difficult than with other diluents.

In a program aimed at obtaining activity coefficients for extractants in organic diluents, an apparatus capable of differential vapor pressure measurements to within ±1 μ Hg was constructed. Measurements on the system tri-n-octylamine (TOA)—benzene ($H_2O$ saturated) vs benzene ($H_2O$ saturated) showed pressure differences, at amine concentrations up to 0.2 M, within a few microns of those given by the Raoult's law expression

$$\Delta p = p_0 \frac{X_1^2}{X_1 + n_1/n_2} ,$$

where $p_0$ is the vapor pressure of the solvent, $X_1$ the mole fraction of benzene in the solvent, and $n_1$ and $n_2$ the moles of benzene and amine, respectively, in the solution. This amine-solvent system is apparently ideal in this concentration range, in confirmation of the hypothesis used previously in deriving equilibrium constants for the system TOA-$C_6H_6$:$H_2O$-$H_2SO_4$.

Ion Exchange Studies. — In a head-end precipitation prior to using ion exchange for the recovery of strontium from Purex waste solution, the amount of precipitated strontium increased as the waste was diluted with increasing amounts of fuming nitric acid. More than 90% remained in solution at ~60% $HNO_3$ and almost 90% precipitated at ~80% $HNO_3$. More than 50% of the iron and 5 to 10% of the aluminum, the chief cationic impurities in the waste, precipitated as sulfates with the strontium product in the $HNO_3$ concentration range tested.

Dowex 50W-X8 (20—50 mesh) resin, in a system of flowing demineralized water, was exposed in a 10,000-curie $Co^{60}$ source. The rate of resin degradation was observed to be a nonlinear function of radiation dose, total product losses being 20 and 85 vol % after exposures of $1.1 \times 10^9$ and $5.1 \times 10^9$ r respectively. The most pronounced physical changes included darkening of the resin bead color, the formation of strongly bonded aggregates of resin beads, and bead fractionation.

Mechanisms of Separations Processes. — Nitric acid, 0 to 5 M, was extracted from aqueous solutions with tributyl phosphate—Amsco 125-82 solutions ranging from 5 to 100 vol % TBP. The data are well correlated by

$$\log \frac{K_1 y_{TBP\ org}}{y_{TBP \cdot H_2O\ org}} = \log \frac{[HNO_3]_{org}}{[HNO_3]_{aq} \{(TBP_s)_{org} - (HNO_3)_{org}\}} = a + b \frac{(HNO_3)_{org}}{(TBP_s)_{org}} ,$$

where brackets refer to activities, parentheses to concentrations, y to molar activity coefficients, and $TBP_s$ to total TBP. The $K_1$ is an equilibrium constant for the extraction, and a and b are constants that vary with the TBP/Amsco ratio. From the density and composition analyses, additional calculations showed that: (1) in diluent-free TBP the volume of water is 17.48 ± 0.16 ml/mole at the 95% confidence level; (2) the volume of nitric acid in TBP-Amsco is 40 to 43 ml/mole in the range 5 to

100 vol % TBP; (3) the maximum deviation of the molar volumes of TBP–Amsco 125-82 solutions from ideality, which occurs at ~50 mole % TBP, is ~0.75%.

In preliminary studies of the use of foam separation in decontaminating ORNL low-activity waste, ~85% of the 27 ppm of calcium present was fixed in solution by the chelating agent ethylenediaminetetraacetic acid while strontium was foamed out of solution as the laurylsulfate. At pH 6 to 9 the foam distribution coefficient, $\Gamma/c$, for strontium was 6 to 7 $\times 10^{-4}$ cm. The addition of illite clay to the waste to sorb cesium, and removal of the clay by foaming gave decontamination factors from cesium and strontium of 40–50 and ~2, respectively, when sodium laurylsulfate was used as the surfactant.

The surface tensions of the surfactant RWA-100, an aryl sodium sulfonate, in nitric acid solutions of pH ranging from 7 to 0.2 were qualitatively consistent with an interpretation that as the acidity increases, the surface active form changes from $RSO_3Na$ to $RSO_3H$. At pH 0.6, approximately 90% of the surfactant is in the acid form. This interpretation is also consistent with the sharp decrease in separation of strontium and rare earths from an aqueous solution at pH 1.

High-Temperature Aqueous Solution Chemistry: Electrolytic Conductance. – Cells and auxiliary apparatus have been constructed for the measurement of electrolytic conductance of aqueous solutions and supercritical fluids at high pressure and high temperature. Preliminary measurements on a 0.01 m KCl solution were made at 800°C and at pressures up to 4000 atm. This work represents a new record for reproducible measurements at extremes of pressure and temperature.

High-Temperature Aqueous Solution Chemistry: Solubility of $NiSO_4$. – The solubility of $NiSO_4 \cdot H_2O$ at 325 to 350°C was determined in solutions of $H_2SO_4$ and $H_2O$ which varied from 0.1 to 1.5 m in free $H_2SO_4$. In parallel studies the solubility of $NiSO_4 \cdot D_2O$ was determined at 200, 250, 300, 325, and 350°C. At 325°C the solubility of $NiSO_4 \cdot H_2O$ was found to increase almost linearly with increasing free $H_2SO_4$ – from 0.040 m $NiSO_4$ in 0.1 m $H_2SO_4$ to 0.66 m $NiSO_4$ in 1.5 m $H_2SO_4$. At 350°C the nearly linear increase with free $H_2SO_4$ gave a solubility of 0.025 m $NiSO_4$ in 0.1 m $H_2SO_4$ and 0.55 m $NiSO_4$ in 1.5 m $H_2SO_4$. These data, together with previously determined solubilities at 200, 225, 250, 275, and 300°C for $H_2O$ solutions, are self-consistent to ±1% and show a reasonable extrapolation to the previously reported solubilities of $NiSO_4$ in the supercritical fluid (ORNL-3048, p 30).

High-Temperature Aqueous Solution Chemistry: Liquid-Liquid Immiscibility. – Compositional boundaries of the region of unsaturated solutions between 300 and 400°C were determined for two sets of solutions containing $UO_2SO_4$, $CuSO_4$, $D_2SO_4$, and $D_2O$, and for two sets containing $UO_2SO_4$, $NiSO_4$, $D_2SO_4$, and $D_2O$. The molal ratio, $\underline{m}_{Cu^{2+}}:\underline{m}_{UO_2^{2+}}$, was kept at 1 in the first set containing $CuSO_4$ and at 0.33 in the second set. Similar ratios were maintained for the two sets containing $NiSO_4$. In each set the temperature of liquid-liquid immiscibility was determined for a series of compositions in which the total sulfate was held constant and the molal ratio, $\Sigma(\underline{m}_{cations}):\underline{m}_{SO_4^{2-}}$, was varied from

1.0 to 0.5. Determinations for other series in the same set were made at sulfate concentrations between 0.05 and 1.0 m. For a given sulfate concentration, results showed a nearly linear relationship of increasing temperature with decreasing cation-to-sulfate molal ratio. Some representative composition-temperature boundaries for solutions, 0.6 m in sulfate, are given below:

Temperature of Liquid-Liquid Immiscibility (°C)

| $\Sigma(m_{cations}):m_{SO_4^{2-}}$ | 0.8 | 0.65 | 0.5 |
|---|---|---|---|
| $m_{Cu^{2+}}:m_{UO_2^{2+}} = 1$ | 306 | 331 | 358 |
| $m_{Cu^{2+}}:m_{UO_2^{2+}} = 0.33$ | 303 | 325 | 352 |
| $m_{Ni^{2+}}:m_{UO_2^{2+}} = 1$ | 301 | 327 | 360 |
| $m_{Ni^{2+}}:m_{UO_2^{2+}} = 0.33$ | 300 | 326 | 360 |

High-Temperature Aqueous Solution Chemistry: Uranium(VI) Hydrolysis. — The study of uranium(VI) hydrolysis at 94°C, using the glass electrode concentration cell (ORNL-2874, -2991), as well as a study at 25°C, using a glass electrode—calomel electrode cell, has been completed. The principal hydrolysis steps appear to be

$$UO_2^{2+} + H_2O \rightleftharpoons UO_2OH^+ + H^+ , \qquad (1)$$

$$2UO_2^{2+} + H_2O \rightleftharpoons U_2O_5^{2+} + 2H^+ , \qquad (2)$$

$$3UO_2^{2+} + 5H_2O \rightleftharpoons (UO_2)_3(OH)_5^+ + 5H^+ , \qquad (3)$$

for which the equilibrium quotients are as follows:

| | 25°C | 94°C |
|---|---|---|
| $Q_1$ | $(2.5 \pm 1) \times 10^{-6}$ | $(7 \pm 1) \times 10^{-5}$ |
| $Q_2$ | $(1.1 \pm 0.1) \times 10^{-6}$ | $(3.2 \pm 0.2) \times 10^{-5}$ |
| $Q_3$ | $(5.5 \pm 1) \times 10^{-17}$ | $(2 \pm 1) \times 10^{-13}$ |

From the equilibrium quotients at the two temperatures the following estimates of enthalpy, free energy, and entropy of reaction are obtained:

| Reaction | $\Delta H$ (kcal) | $\Delta F_{25}$ (kcal) | $\Delta S_{25}$ (eu) |
|---|---|---|---|
| (1) | 10.5 ± 1.5 | 7.6 ± 0.2 | 10 ± 5 |
| (2) | 10.6 ± 0.5 | 8.13 ± 0.05 | 8.4 ± 2 |
| (3) | 26 ± 2 | 22.2 ± 0.1 | 13 ± 6 |

The temperature dependence of $Q_1$, $Q_2$, and $Q_3$ indicates not only the increase of over-all hydrolysis with increasing temperature but also the increased importance of the mononuclear $UO_2OH^+$ in the process; for example, it is found that stoichiometric 0.01 m $UO_2(NO_3)_2$ (where only $UO_2OH^+$ and $U_2O_5^{2+}$ are present in appreciable amounts) is 6% hydrolyzed at 25°C, 19% at 100°C, and, by extrapolation, ~50% at 200°C, while the corresponding amounts of $UO_2OH^+$ are 0.5, 4, and ~20% of the total uranium.

Reactor Neutron Cross Sections. – The measurement of the effective neutron capture cross section, $\sigma_{eff}$, the thermal cross section, $\sigma_{th}$, and the resonance capture integral, $I_0$, of 285-day $Ce^{144}$ by the activation method was completed. The values are $\sigma_{eff} = 1.16 \pm 0.30$, $\sigma_{th} = 0.93 \pm 0.23$, and $I_0 = 2.74 \pm 0.27$ barns.

Irradiations of thin samples of $U^{233}$ with and without cadmium filters were carried out in the ORR to compare thermal and epithermal capture cross sections. The $U^{234}$ formed was determined mass spectrographically, and measurements of flux monitors were made. A preliminary value of 150 barns for the capture resonance integral of $U^{233}$, including the 1/v tail, was obtained for neutrons above 0.5 ev.

The partial radiative capture cross section of $Po^{210}$ for the formation of the 25-sec isomer of $Po^{211}$ was found to be less than 0.1 mb. This low cross section may be related to the closed shell of 126 neutrons in $Po^{210}$.

Chemistry of Technetium. – Crystalline $K_2ReF_6$ and $K_2TcF_6$ were prepared, and $H_2TcF_6$ was obtained in aqueous solution. The rate of hydrolysis of $K_2ReF_6$(aq) to $ReO_4^-$ in the presence of air is 0.17% per day at 25°C and 6.2% at 60°C; for $K_2TcF_6$(aq) the rate of hydrolysis to $TcO_4^-$ is 0.015% per day at 25°C and 1.6% at 60°C. The absorption spectrum of $ReF_6^{2-}$(aq) between 2000 and 13,800 A exhibits vibrational fine structure and has principal absorption maxima at the following wavelengths (in angstroms), the molar absorbancy indices being given in parentheses: 11,000 (0.55), 9900 (0.62), 9170 (1.38), 8770 (1.03), 5655 (0.68), 5280 (0.39), and 3050 (8.43). The corresponding data for $TcF_6^{2-}$(aq) (2000 to 11,500 A) are 9090 (0.194), 5680 (0.418), 3520 (16.1), and 2900 (13.9). These are crystal field bands. The much more intense charge-transfer bands lie below 2100 A for both compounds. The acid $H_2TcF_6$ exists only in solution; attempts to isolate the pure acid resulted in decomposition to $TcO_2 \cdot xH_2O$.

Thermochemistry and Calorimetry. – A final determination of the heat of fusion of $Li_3ThF_7$ yielded a value of 13.94 ± 0.05 kcal/mole. The impurity content of the sample, calculated from the premelting, is 0.4 mole %. In the premelting region, equilibration of the sample in the furnace for 6 hr was required. Two previously reported determinations were in error because of lack of attainment of equilibrium in this temperature region.

The standard heat of solution of $KReO_4$ was found to be 13.8 ± 0.1 kcal/mole. The large uncertainty results from the long time (30 to 40 min) needed for dissolving the sample.

Chemistry of the Heavy Elements. – The second prototype high-temperature aqueous spectrophotometric cell, designed to allow gas-liquid equilibration, was tested with favorable results. In an initial experiment

with a $UO_2SO_4$-$CuSO_4$-$D_2SO_4$-$D_2O$ solution, the course of reduction in an $H_2$ atmosphere and reoxidation in an $O_2$ atmosphere for U(VI) and Cu(II) was followed individually and quantitatively as a function of time, temperature, and pressure. Eighty per cent of the copper and an estimated 15% of the uranium were reduced and subsequently reoxidized.

Nuclear Chemistry: Characterization of Short-Lived Fission Products. — The level structure of $Ge^{74}$ was studied by means of the radiations from $As^{74}$. After subtraction of the positron annihilation-in-flight continuum from the singles spectrum, the following gamma-ray peaks (and relative intensities) were resolved: 0.60 (100), 0.635 (30), 1.20 (0.4), 1.6 (0.02), 2.18 (0.04), and 2.53 (0.01) Mev. By gating on the 0.60-Mev gamma ray, coincidence peaks were extracted from the continuum at 0.60, 0.70, 0.88, 0.99, 1.11, and 1.59 Mev. A measurement of the directional correlation of the 0.60-0.60 Mev cascade yielded coefficients $A_2 = -0.248 \pm 0.044$ and $A_4 = 0.251 \pm 0.070$, which are consistent with a 2-2-0 spin sequence and a value of approximately 3 for $\delta$, the mixing coefficient, in agreement with the Davidov-Filippov prediction of 2.7. These data and those obtained from a study of the decay of $Ga^{74}$ lead to energy levels in $Ge^{74}$ at 0.598 ($2^+$), 1.20 ($2^+$), 1.47 ($4^+$), 1.71, 2.18, 2.53, 2.95, 3.05, 3.17, 3.33, 3.41, 3.57, and 3.83 Mev. A new value, 17.74 ± 0.05 days, was obtained for the half-life of $As^{74}$.

The ranges of 2.736-Mev tritons in various substances were measured as follows (values in milligrams per square centimeter): aluminum, 10.10; nickel, 15.15; polystyrene, 6.12; nitrogen, 7.26; argon, 11.40; krypton, 17.83; xenon, 20.36; and air, 7.34. Values of energy loss were determined between 0.2 and 2.7 Mev. The tritons were produced with a reactor neutron beam by the reaction $Li^6(n,\alpha)H^3$. A silicon surface-barrier detector and a multichannel pulse-height analyzer were used to measure the triton energy as a function of absorber thickness. Background effects were eliminated by demanding a coincidence between the tritons and the accompanying alpha particles (correlated at 180°), which were detected in a second surface-barrier detector. From these data, range and energy-loss curves were constructed and empirical range-energy relations were derived.

Chemical Separation of Isotopes. — The rates of exchange between $Li^+$ and $K^+$ were measured for systems containing one of these ions in solution and the other on Dowex 50 or IRC 50 ion exchange resin. In each case the initial resin was anhydrous and was wet only with the solvent used for the liquid phase. The concentration of the salt in the liquid phase was 0.1 to 0.2 N. The times of half exchange, $T_{1/2}$, effective diffusion coefficients of the two exchanging ions inside the resin particles, $D^i$, and selectivity coefficients, $K_K^{Li}$, are presented below:

| System | $T_{1/2}$ (min) | $D^i$ ($cm^2$/sec) | $K_K^{Li}$ |
|---|---|---|---|
| IRC $50 \cdot K^+$ vs LiBr in $HCONH_2$ | 0.58 | | |
| IRC $50 \cdot K^+$ vs LiBr in $CH_3OH$ | 9.4 | $7.5 \times 10^{-8}$ | 1.27 |
| IRC $50 \cdot Li^+$ vs KCl in $H_2O$ | 0.58 | $1.3 \times 10^{-6}$ | 1.14 |
| Dowex 50-X8$\cdot K^+$ vs LiBr in $CH_3OH$ | 45 | $1.4 \times 10^{-9}$ | 0.066 |
| Dowex 50-X8$\cdot K^+$ vs LiCl in $HCONH_2$ | 11 | $5.1 \times 10^{-9}$ | 0.44 |
| Dowex 50-X8$\cdot K^+$ vs LiBr in $C_2H_4(NH_2)_2$ | 52 | $1.4 \times 10^{-9}$ | 0.030 |
| Dowex 50-X8$\cdot Li^+$ vs KCl in $H_2O$ | 0.15 | $4.0 \times 10^{-7}$ | 0.46 |

Chemical Applications of Nuclear Explosions. — Further studies on the kinetics and thermodynamics of tritium exchange in the hydrogen-water system showed an apparent activation energy of 9.2 kcal for the exchange of tritium between HTO and $H_2$ over beds of $CaSO_4$ under flow conditions at steady state. At 600°C the steady-state exchange rate increased from $3.02 \times 10^{-5}$ to $5.99 \times 10^{-5}$ mole of tritium per minute when the weight of the $CaSO_4$ bed was increased from 1.00 to 3.00 g and the HTO/$H_2$ ratio was held constant at 0.07. The volume flow rate of HTO was $4.5 \times 10^{-4}$ mole/min and the residence time was 5 sec. The results suggest that bed diffusion is the rate-controlling process and that the gas mixture does not have free access to the total $CaSO_4$ surface when it is flowing over the $CaSO_4$ bed. Thermodynamic calculations were completed on the extent of exchange for seven isotopic reactions in the hydrogen-water system, in which the species considered were $T_2$, HT, $T_2O$, HTO, $H_2$, and $H_2O$. Significant is the fact that the exchange of tritium in the equilibration of $T_2$ and $H_2O$ is 75 to 90% of the initial $T_2$ from 20 to 800°C according to the equation

$$2T_2 + 2H_2O \rightleftharpoons T_2O + HTO + HT + H_2; \quad \Delta H_0^\circ = -976 \text{ cal.}$$

## METALLURGY AND MATERIALS RESEARCH

Ceramics Research. — The sintering rate of thoria compacts as a function of time was measured from 600 to 1600°C at heating rates of 2.7, 4.0, and 8.0°C per second. The sintering rate attained a peak value at 980°C and passed through a minimum at 1200°C; the initial peak occurred at an increase in density of 15%. The temperature and degree of densification associated with the initial sintering rate peak were the same for each heating rate. The presence of a discontinuity in the sintering rate vs temperature curve suggests the influence of more than one material transport mechanism. The initial peak could indicate densification by a low-temperature mechanism which is then succeeded by another at higher temperature.

Structure of Metals. — Additions of oxygen to high-purity niobium inhibited the formation of mechanical twins at 77°K. In single crystals with clean surfaces, the frequency of twinning for a sample containing 2000 ppm oxygen was about 10% of that for a sample containing 50 ppm oxygen. The presence of a thin oxide layer on the surface completely suppressed twinning. This and earlier observations indicate that

nucleation of mechanical twins in the body-centered cubic metals occurs at free surfaces.

## CONTROLLED THERMONUCLEAR RESEARCH

Cross-Section Studies. — The crossed-beam apparatus for measuring the cross section for the dissociation of $H_2^+$ by $H^+$ is ready to be installed in the DCX. The source of $H^+$ (von Ardenne ion source) delivers at present 10 ma of analyzed $H^+$ at 40 kev through a 1-in. defining hole 38 in. away from the einzel lens focusing system. The large diameter of the beam is chiefly attributed to space-charge blowup at this long distance from the source. The beam will be reduced in size when the source is installed on the DCX-1 by the axial magnetic field focusing. The $H^+$ beam has been pulsed at 10 cps and possesses a 25% 120-cps modulation due to the high-voltage power supply. Thus, all the requirements are separately available to measure the cross section in the DCX-1 — a 10-cps modulated 10-ma $H^+$ beam, a 10-ma $H_2^+$ beam, and a vacuum of $1 \times 10^{-7}$ mm.

The crossed-beam apparatus for measuring the cross section for the dissociation of $H_2^+$ by electrons has been operating successfully for several months. The 100-μa, 20-kev $H_2^+$ beam is crossed by a modulated 10-ma, 50-ev electron beam, and a modulated signal of $H^+$ is sought. Such signals have readily been obtained, but there appears to be a streaming of electrons directly into the detector. Confinement of the electrons by shielding did not alleviate the situation, and confinement by a 50-gauss longitudinal magnetic field is being attempted. This streaming must be prevented before a cross-section measurement will be possible.

DCX-1 Facility. — Investigations of the divergence of the undissociated portion of the $H_2^+$ beam as it emerges from the inner vacuum region have continued. This divergence is a function of the spatial extent of the incoming $H_2^+$ beam, which is determined by the entrance aperture to the inner region. Changing this aperture from a 3/4-in.-diam circle to a 1/4- by 3/4-in. rectangle, with the shorter dimension in the median plane, reduced the spread in the undissociated beam, measured near the bottom of the tank assembly, by a factor of approximately 1/3.

An electric quadrupole lens is being fabricated in order to provide additional control of the $H_2^+$ beam focus. It is believed that proper adjustment of this lens will provide an astigmatic focus that will allow a large portion of the accelerated current to be injected through the rectangular aperture into the inner vacuum region.

A rotating water-cooled copper drum is now used to stop the undissociated beam near the aperture through which this beam emerges from the inner region. Pumping is provided by continuous evaporation of titanium onto the drum. Difficulty has been encountered in the operation of this device in that with an $H_2^+$ beam incident upon the drum, the pressure oscillates at the frequency of rotation of the drum. The cause of this phenomenon is at present unknown.

Continued tests with evaporative titanium pumping in the DCX indicate that base pressures of $1 \times 10^{-8}$ mm Hg and pumping speeds of 30,000 to 60,000 liters/sec are usually obtainable.

With a base pressure of $4 \times 10^{-8}$ mm Hg, injecting a 1-ma $H_2^+$ beam into the inner region caused a pressure rise of $5 \times 10^{-9}$ mm Hg (uncorrected ion gage reading). At this pressure, gas dissociation data indicate exponential decay time constants of 150 msec. The general behavior of the decay time constants upon the admission of helium or hydrogen is like that previously obtained with base pressures of $1 \times 10^{-6}$ mm Hg. At the higher pressures, when the leak gas is presumably the major gas constituent in the plasma region, the ratio of the measured decay time constant to the calculated time constant is 1/2 in the case of a helium leak and 1/4 in the case of a hydrogen leak.

DCX-EP-A. — All new installations on the DCX-EP-A have been put on a hold basis. Construction of the 100-kev neutralized beam accelerator and isolation transformer tower are about 75% complete. Testing and improvements of components in the Sherwood Test Facility are continuing.

Recent experiments in the Sherwood Test Facility on a low-energy prototype of the 100-kev neutralized ion beam accelerator have produced encouraging results. Thermal power measurements of the beam output show a power increase by a factor of 2 over that obtained without electron injection at an acceleration voltage of 1 kv and by a factor of 10 at 7 kv. Power dissipation on the accelerating electrode was also measured thermally. At 1 kv accelerating voltage it was down by a factor of 4, and at 7 kv it was up by a factor of 2.

High-Current Injection. — Modification of the 600-kv beam test facility has been completed, and preliminary tests have been conducted up to 550 kv. The corona is reduced significantly with the new high-voltage lead arrangement. The major problem has been in conditioning the surfaces of the source anode and extractor electrode to hold 150 kv with 0.35 in. spacing. Such a spacing would be required for beam currents of 350 to 400 ma with the present source geometry.

The solenoid lens for the 100-kv test facility has been installed, and preliminary tests indicate that it will perform as expected.

Theory. — A tentative solution of the Fokker-Planck equations for a plasma in a uniform magnetic field has been obtained. The complete self-consistency of the solution has not yet been demonstrated. In addition to single-particle radiation terms, there are collective contributions which are of a different form from those calculated by others.

The numerical Fokker-Planck code is operating satisfactorily and is being used for studies of energy degradation in the DCX-2.

## BIOLOGY AND MEDICINE PROGRAM

### RADIATION EFFECTS ON BIOLOGICAL SYSTEMS

Cell Growth and Reproduction. — The ribonucleic acid of mammalian tissue culture cells is rapidly labeled when the cells are incubated in a medium containing $H^3$-uridine. The initial appearance of radioactivity

in ribonucleic acid is localized in the nucleus; this is consistent with the thesis that the cell nucleus is the source of all of the cell's ribonucleic acid. Synthesis of ribonucleic acid continues during the earliest stage of mitosis, but it stops as the chromosomes become heavily condensed during middle prophase. Ribonucleic acid is probably the principal product of the dispersed interphase chromosome, and thus it is reasonable that its production would cease when the chromosomes are in the highly compacted mitotic form. Protein synthesis continues during mitosis but is measurably depressed.

Pathology and Physiology. — Evidence that $F_1$ hybrid mice may have immunological specificities interfering with acceptance of grafted parental cells was obtained. Hybrid mice from inbred parents differing at the H-2 locus were sensitized to parental antigens by intraperitoneal inoculation of spleen cells. The sensitized animals were used as recipients or as donors in two experiments. In the first, parental marrow was injected into lethally irradiated sensitized recipients to determine whether sensitization would interfere with its transplantation. In the second experiment, sublethally irradiated $F_1$ recipients inoculated with parental antibody-forming cells were concomitantly injected with isologous $F_1$ marrow cells from sensitized or nonsensitized donors, to determine whether the latter cells would reduce the deleterious effects of the former. The results of both experiments demonstrate that acceptance of grafted parental cells may be prevented by immunologically competent $F_1$ hybrid cells if immunogenetic differences exist between the parental strains. Hence, it is suggested that (1) parental histocompatibility genes are not necessarily expressed in the hybrid as codominants, and (2) in parent-to-hybrid radiation chimeras, a host-vs-graft immunological reaction may be a component of "homologous disease."

Biophysics: Magnetic Resonance Studies. — Studies of the modification of x-ray-induced electron spin resonance signals by posttreatments with ultraviolet radiation have been continued. Previous results indicating that the signal is markedly decreased by ultraviolet posttreatment have been confirmed. In the case of glycine, the resonance signals decrease exponentially with applied ultraviolet dose, in accordance with one-hit inactivation or destruction of the x-ray-induced radicals by ultraviolet quanta.

Qualitative studies of the variation of concentration of electron spin resonances in crystalline amino acids as a function of dose of ionizing radiation, postirradiation time, and storage temperature are being conducted. Analyses are being made in terms of kinetic theories of recombination. Preliminary measurements suggest that decay rates are so accelerated at 100°C that one day's studies at this temperature may yield as much information as one year at room temperature. If recombination coefficients can be determined at several temperatures, an Arrhenius plot may yield the energy required for the formation of these resonances. Qualitatively, the notion of recombination with coefficients increased by heat, moisture, irradiation (free electrons and excited states are more common during irradiation), exposure to ultraviolet, and linear energy transfer agrees with a large body of data collected in this laboratory over several years.

Biophysics: Ultraviolet Studies. — Ultraviolet inactivation at 2537 A of the streptomycin marker of transforming principle for *Hemophilus influenzae* has been carried out in air and in oxygen-free nitrogen, with doses up to 3200 ergs/mm$^2$. There is no difference between the curves, within the experimental error of only a few per cent. This indicates that the effects of oxygen on the viscosity and light scattering (i.e., chain breaks) of deoxyribonucleic acid, observed by others at very high doses of ultraviolet, are unimportant at "biological" doses, and hence that chain breaks are probably not involved in the inactivation of deoxyribonucleic acid with respect to its biological activity. The same transforming principle shows no photoprotection. This is evidence against photoprotection being a direct effect on deoxyribonucleic acid.

Any quantitative analysis of the effects of different wavelengths of ultraviolet light on the nucleic acid polymers of biological systems requires a knowledge of the effects of different wavelengths on the constituent bases of polynucleotides. Uridine has been irradiated with ultraviolet at different wavelengths, and the quantum yield for the photolysis has been determined. Within the experimental error of ±5% the quantum yield is constant (and equal to 0.017) between 2800 and 2350 A. At 2300 A it is 0.022 and at 2250 A it is 0.038. At the higher wavelengths, the effects of ultraviolet are reversible by heat; at the lower wavelengths, they are not.

It is known that the spherical plant viruses may be photoreactivated after being inactivated by ultraviolet light. Tobacco mosaic virus shows no photoreactivation, but if the extracted ribonucleic acid of tobacco mosaic virus is irradiated it shows photoreactivation. A simple interpretation of these data is that the only ultraviolet damage that can be photoreactivated is damage to contiguous polynucleotide chains. (In the spherical viruses the nucleic acid is rolled up into a ball, in tobacco mosaic virus the ribonucleic acid is in the form of a single chain surrounded by protein, and the isolated ribonucleic acid of tobacco mosaic virus is partially helical with intrachain hydrogen bonds.) Other relatively simple polynucleotide systems support this interpretation.

Chemical Protection and Enzyme Catalysis. — The walls of blood platelets were found to contain most of the amino acids found in other proteins. The amino acid compositions of the soluble dinitrophenyl protein, insoluble dinitrophenyl protein, and the whole platelet wall are qualitatively identical, although a few quantitative differences were observed.

Malic dehydrogenase from bovine heart has been hydrolyzed in saturated barium hydroxide solution for 20 hr in order to determine the exact tryptophan content. The complete absence of tryptophan has been demonstrated from use of the automatic amino acid analyzer. This should make malic dehydrogenase a convenient protein to study structurally, since special methods are necessary when tryptophan is involved.

COMBATING RADIATION DETRIMENTAL EFFECTS

Radiation Microbiology. — Experiments reported earlier on the strain $B_s$ (Hill) of the bacterium *Escherichia coli* indicated a very small protective effect with 2-mercaptoethylamine (MEA) and an almost negligible oxygen effect (dose reduction factor, 1.0 to 1.2). In order to obtain the maximum oxygen effect with this strain, purified nitrogen was bubbled through the suspensions previous to and during irradiation, whereas the controls had oxygen bubbled through them for the same period. Although the oxygen effect was slightly larger by this procedure, the dose reduction factor was still considerably smaller (1.33 to 1.41) than that found for *E. coli* B/r ORNL (3.13 to 4.15). The protective effect of MEA above that achieved by nitrogen bubbling is approximately the same for both strains.

Mammalian Radiation Recovery. — Radiation protection by S,2-aminoethylisothiourea (AET) of the antibody-forming cells responsible for the homograft reaction is being studied. Preliminary results show that the killing effect of parental spleen cells injected into sublethally irradiated $F_1$ recipient mice is eliminated by a lethal dose of radiation given to the parental donor. Treatment of the donor with AET before irradiation did not protect the cells responsible for the killing of the recipients, for mortality was the same as in the groups receiving parental cells irradiated with no pretreatment.

Isoimmune cytotoxic sera have been used to identify mouse lymphoid cells from radiation bone marrow chimeras that have been injected with blood of a type different from the marrow donor. Preliminary results indicate that lymphoid cells in such chimeras are derived by repopulation from nucleated cells present in the donated blood.

Radiation Immunology. — Preliminary data obtained with the "double cell transfer" method show that the homograft-rejecting capacity of isolated mouse spleen cells is less sensitive to radiation than their capacity for synthesizing titratable antibody proteins. Thus, for example, 200 r (in vitro) destroyed 75% of the former capacity and 98% of the latter.

It has been shown by the in vivo culture method that the clonal selection theory of Burnet is not compatible with the experimental data. Briefly, according to this theory, the expected relative antibody-forming activity of mouse spleen cells toward non-cross-reacting sheep and rat antigens is 100:1 when these cells are heavily stimulated with sheep antigen and 1:100 when stimulated with rat antigen. Instead, the relative activity in both cases was 1:1.

Previously, the first site of antibody formation was reported to be in the nucleus. This site has now been localized in the pyronin-positive nucleolus. Thus, contrary to the common belief that the nucleolus is involved in transfer of "information" to the ribosomes of the cytoplasm, the protein-synthetic mechanism itself commences here.

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## FOREIGN VISITORS

| Name | Affiliation | Citizen of |
|---|---|---|
| Amelinckx, S. | Centre de'Etude de l'Energie Nucleaire | Belgium |
| Anahara, R. | Japanese Nuclear Material Test Reactor Team | Japan |
| Bailly, H. P. | Commissariat a l'Energie Atomique | France |
| Bletzinger, P. | Wright Air Development Center | Germany |
| Borsch-Supan, W. G. | National Bureau of Standards | Germany |
| Caillat, R. | French Atomic Energy Commission | France |
| Cibollini, E. | Duke University | Italy |
| Evans, C. C. | United Kingdom Atomic Energy Authority | United Kingdom |
| Gauthron, M. | Commissariat a l'Energie Atomique | France |
| Griboval, P. | University of Grenoble | France |
| Grove, W. P. | United Kingdom Atomic Energy Authority | United Kingdom |
| Guinier, A. | University of Paris | France |
| Heal, T. J. | United Kingdom Atomic Energy Authority | United Kingdom |
| Hugo, J. P. | South African Atomic Energy Board | South Africa |
| Ikuchi, Y. | Japanese Nuclear Material Test Reactor Team | Japan |
| Inoue, Y. | Japanese Nuclear Material Test Reactor Team | Japan |

| Name | Affiliation | Citizen of |
|---|---|---|
| James, D. W. | Rensselaer Polytechnic Institute | New Zealand |
| Joedoatmodjo, S. | Institute of Atomic Energy, Indonesia | Indonesia |
| Kusono, T. | Japanese Nuclear Material Test Reactor Team | Japan |
| Lawton, H. | AGR Engineering Design Team | Great Britain |
| Linacre, J. K. | United Kingdom Atomic Energy Authority | United Kingdom |
| Neethling, A. C. | South African Atomic Energy Board | South Africa |
| Niizaki, M. | Japanese Nuclear Material Test Reactor Team | Japan |
| Nilsson, R. | University of Uppsala | Sweden |
| Pfender, E. | University of Stuttgart | Germany |
| Pilling, F. D. | United Kingdom Atomic Energy Authority | Great Britain |
| Ringot, C. | Commissariat a l'Energie Atomique | France |
| Salesse, M. | French Atomic Energy Commission | France |
| Seki, Y. | Japanese Nuclear Material Test Reactor Team | Japan |
| Shima, S. | Japanese Nuclear Material Test Reactor Team | Japan |
| Small, S. H. | International Atomic Energy Agency | United Kingdom |
| Stam, E. | Virginia Polytechnic Institute | Israel |
| Taniguchi, N. | Japanese Nuclear Material Test Reactor Team | Japan |
| Tsuzuki, T. | Japanese Nuclear Material Test Reactor Team | Japan |
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