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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

OCTOBER 1962

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OAK RIDGE NATIONAL LABORATORY

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Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

OCTOBER 1962

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

REACTOR DEVELOPMENT PROGRAM

GAS-COOLED REACTOR PROGRAM

EGCR: Component Fabrication and Testing. — Fabrication work on the EGCR main blower and the vessel coolant compressor continued. The first blower is scheduled for delivery in March 1963 and the second in May 1963.

Fabrication of components for the EGCR experimental loops continued. The lower chase piping is presently being installed in the reactor. All prefabricated subassemblies required for loops TS2, TS3, and TL4 are in place. All stainless steel welding was completed and tested, and approximately 40% of the carbon-steel welding required for the outer piping was completed.

A decision was made to use experimental loop TL-1 for an emergency cooling system for the reactor. Design and procurement will make use of available EGCR loop materials, as feasible.

The test control panel is being fabricated for the control-rod drive test. A fast-response position transmitter, amplifier, and receiver system was ordered for use with the existing encoder and tape readout system for recording rod decelerations during scram tests. Delivery of the rod drive component is expected after January 1, 1963.

Design work is in progress on a facility for determining the reaction rate of steam with full-scale specimens of the graphite sleeves for the EGCR fuel elements and with similarly sized specimens of moderator graphite. The tests will be conducted in the temperature range 1000 to 1800°F and in the pressure range 0 to 365 psia.

EGCR: Graphite Surveillance. — The graphite surveillance program was planned to include tests of the oxidation rate in air, flexural strength, Young's modulus, lattice parameter changes, thermal expansion coefficient, thermal conductivity, dimensional and weight changes, creep, and stored energy. Two methods of temperature measurement in the surveillance channel are being evaluated: (1) temperature-indicating powders and (2) melt wires.

PBRE: Design. — Evaluation of control-rod drives used or proposed for gas-cooled reactors led to selection of a lead-screw drive with a direct-lift magnet as the most suitable arrangement.

A design was prepared in which bellows are used rather than a slip joint to absorb the axial expansion in the inner pipe of the concentric

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ducts connecting the PBRE reactor vessel and heat exchanger. Three identical hinged joints, with one convolution each, are used; one is located near the reactor vessel liner, and the other two are at the steam-generator end. This scheme requires a vertical pipe approximately 3 ft long within the steam-generator vessel.

The feasibility of making the top access nozzle of the reactor vessel large enough for insertion and removal of a 50-in.-diam graphite cylinder was investigated. Nozzle inside diameters of 52 and 56 in. were assumed. Evaluations made in accordance with the Navy code showed that a 4-in. thickness is required in each case, and the vessel thickness must also be 4 in. in the vicinity of the nozzle. Either design is adequate for 2500 pressure and thermal cycles if the inner surface of the nozzle is insulated.

PBRE: Fuel-Handling System. — A mockup facility having components of the type needed for fuel storage and transport in the PBRE was used to test concepts and develop equipment for the fuel-handling system. The facility includes a ball-storage unit in the form of a helical tray with a 48-in. outside diameter and a 6-in. lead between turns (a 16-turn tray would contain 12,000 spheres 1-1/2 in. in diameter in a single layer); a rotary-disk device for removing balls from the bottom of the tray and feeding them single file into the transport piping; a ball-transport pipe having a section in which balls are elevated pneumatically to an upper level and another in which balls dropping to a lower level are retarded with a counterflow of gas to reduce the impact velocity; a ball counter formed from a differential transformer wound about a short length of stainless steel pipe; a broken-ball separator and classifier; and a rotary ball stile. Better performance in discharging spheres from the helical tray was achieved with the rotary feeder than with the vibratory-tray feeder tested previously. The ball stile tested serves adequately as a gas check valve, as well as acting to control the rate of passage of spheres into parts of the system. Satisfactory performance has also been achieved with the ball-transport system, ball counter, classifier, and storage tray.

PBRE: Instrument Development. — An analog-computer study was made of an automatic controller that adjusts the position of a shim regulating rod to maintain the desired PBRE outlet gas temperature. System behavior was studied during transients involving changes in gas flow rate, heat-sink temperature, temperature control set point, and reactivity. Although optimum controller characteristics were not determined, results of the study indicate that a controller of this type could be satisfactorily applied to the PBRE.

Other analog studies were performed to determine transient behavior of the system during various perturbations without control-rod action. Temperature profiles were measured during flow coastdown, blower seizure, and nuclear excursions. The thermal response of the system, as was expected, was extremely slow, especially at low flow rates.

Preliminary investigations were made to determine the suitability of deriving a scram signal from the ratio of flux to coolant flow. These investigations indicated that such a safety signal could provide protection against excessively high temperatures without impairing the ability to operate at various power levels and flow rates within proposed design limits.

PBRE: Heat Transfer and Fluid Flow. – A full-scale mockup of the PBRE core region was assembled and instrumented. Auxiliary components of the mockup include automatic sphere-loading and -discharging devices, probes for determining velocity profiles, and facilities for measuring pressure drop and eddy diffusivities within the bed and the angle of repose of spheres added to the top of the bed.

A preliminary value of 0.401 ± 0.002 was obtained for the overall void fraction of a static bed. This value applies to loose, random packing and is essentially independent of the point of sphere injection (relative to the vessel wall) and of the injection velocity. The axial variation of the void fraction (averaged over increments of four sphere diameters) was within the range 0.457 to 0.382 and showed an initially high value that decreased to a minimum in the region just above the discharge dome and thereafter increased slowly to the top face of the bed.

The initial pressure-drop measurements covered a range of Reynolds numbers from 2500 to 9400. Although the results were consistent with the data of other investigators, additional tests over a wider range of Reynolds numbers will be required to distinguish which of the proposed correlations is applicable to the present situation. Pressure-drop data taken with a 3/8-scale model, using smooth plastic spheres (9/16 in. in diameter), were in agreement with the data derived from the full-scale experiment for velocities below initial levitation.

LITR High-Temperature Irradiation Experiments. – The second high-temperature capsule containing UO_2 pellets was irradiated for a scheduled one week. Extrapolation beyond the range of calibration for W–5% Re vs W–26% Re thermocouples indicated an initial temperature in capsule b of 4420°F. This indicated temperature drifted downward sharply, and after 5 hr of irradiation the central thermocouples of sections a and b indicated, respectively, 3895 and 3945°F. At the end of irradiation, these thermocouples indicated, respectively, 3510 and 3550°F.

ORR Capsule Irradiations. – The design of an assembly for simultaneously irradiating eight fueled-graphite balls was completed. Heat transfer calculations indicate that a satisfactorily uniform temperature can be achieved around the periphery of a ball in the cylindrical geometry of the capsule. This is achieved by utilizing a variable annular gas gap between the graphite structure, in which the ball is located, and the graphite container. Spheroidized powdered graphite is utilized to improve the heat transfer between the ball and the graphite structure.

Irradiation Experiments in GCR-ORR Loop No. 1. – An EGCR-type fuel element (No. 8) is being irradiated that has a roughened surface formed by copper-brazing 0.0063-in.-diam wire with a 0.090-in. pitch on the surface of the stainless steel can. Because of a late design change of the assembly, it was not possible to install thermocouples with the element shroud to measure the outlet gas temperature. It is necessary to rely on thermocouples permanently installed in the loop for this purpose. It appears that these thermocouples may have changed the calibration; therefore it is not possible to make a direct comparison of the performance of this element with that of the previous element, which had a smooth wall and was operated at a similar power level. It will be necessary to irradiate another element with thermocouples installed with the element to provide a calibration point for the loop thermocouples before comparing the data from these experiments.

GCR-ORR Loop No. 2 Experiment Design. — The design layout of the first fueled assembly with graphite balls containing coated uranium carbide particles is nearly complete, and a separately cooled tube for studying fission product deposition in the region immediately downstream of the fuel is being designed. The cell leak test was successfully completed, and preparations are being made for the high-temperature out-of-pile shakedown test of the loop.

Release of Fission Products by In-Pile Burning of Uranium Carbide—Graphite Fuel. — Radiochemical analyses were completed on the experimental assembly in which a uranium carbide—graphite fuel specimen was partially burned in the ORR to study the release of fission products that would occur in a reactor accident. Over 98% of the strontium, zirconium, and barium activities, over 98% of the uranium, and 94% of the cerium activity were retained inside the combustion furnace. Approximately 60 to 75% of the ruthenium, iodine, tellurium, and cesium activities were retained in the furnace. The strontium, barium, cerium, and uranium activities found outside fuel residues were 2 to 15%, as compared with 30 to 50% of the zirconium, ruthenium, iodine, tellurium, and cesium activities. Approximately 58% of the fuel sample was burned. The strontium, iodine, tellurium, barium, cesium, and uranium originally in the unburned portion of the fuel were largely retained, whereas zirconium, ruthenium, and cesium were significantly released from the unburned portion. In interpreting these data, it must be remembered that only a single experiment was conducted, and no statistical measure of reproducibility can yet be made. It must also be borne in mind that the fuel specimen was not homogeneous in UC_2.

On the basis of the information from this experiment, it appears that if combustion of graphite—uranium carbide fuel occurred in a reactor accident under the conditions of this experiment, all but a small fraction of the strontium, zirconium, barium, cerium, and uranium would remain in the vicinity of the fuel, but significant amounts of ruthenium, iodine, tellurium, and cesium would be released. A second experimental assembly is being prepared with the same type of fuel specimen.

Experimental Studies of Fission Product Deposition. — The initial run in a fission product deposition loop at Battelle Memorial Institute was terminated on September 22. The approximate fuel temperature was 1800°F, the maximum gas temperature was 1200°F, the minimum gas temperature was 425°F, and the loop pressure was 225 psig. Preliminary results indicate that I^{131}, Te^{132}, Zr^{95}, Cs^{137}, Ce^{141}, and Ru^{103} were deposited on the loop surfaces.

Gas-Bearing Compressor Development Studies. — A second series of tests was made on the Bristol Siddeley gas-bearing compressor with the thermocouples relocated inside the compressor for more accurate measurement of the temperature of the front journal bearing and the diaphragm area. Three attempts were made to operate the compressor at the design temperature and pressure (600°F and 295 psia), but each attempt resulted in rubbing of the journal bearing as the suction temperature approached 500°F. It is suspected that the heat shields contacted the diaphragm as the temperature increased and caused the diaphragm to distort and subsequently misalign the journal bearing, which is attached to the diaphragm. The heat shields were modified to prevent contact with the diaphragm, and the compressor is being reassembled.

Compatibility Test of Metals and Graphite in Hydrogen. — A test in a hydrogen autoclave containing type AGOT graphite and specimens of types 304L and 310 stainless steel and Inconel was terminated after 1000 hr of operation at 1500°F and an average hydrogen pressure of 850 psig. A steady increase in CH_4 concentration to a level of 19 vol % was noted during the test period.

US-UK Cooperative Program on Irradiation of Stainless Steels. — A cooperative program between the UKAEA and the USAEC was initiated for investigating the in-pile and out-of-pile stress-rupture characteristics of three British stainless steels. The compositions of these steels are Cr-Ni-Nb (20-25-1%), Cr-Ni-Nb (18-8-1%), and Cr-Ni-Nb (25-20-1%). The temperatures of interest are 650, 750, and 850°C in atmospheres of argon plus 1% O_2 for the out-of-pile tests and helium plus 1% O_2 for the in-pile tests.

Metallographic examination of control specimens of the Cr-Ni-Nb (18-8-1%) alloy tested at 650, 750, and 850°C for 500 hr revealed that the microstructure was quite different from that nominally observed in the 18% Cr–8% Ni steels. It has been shown that the British steel undergoes a martensitic transformation upon cooling from a solution-annealing temperature of 1050°C.

Magnetic-susceptibility measurements on specimens quenched to various temperatures indicate that the martensitic transformation begins at about room temperature and ends at −150°C. The steel was also quite sensitive to cold-working, and the magnetic susceptibility was found to increase linearly up to 40% cold-working. Because of the low carbon content (0.065 wt %) of this steel, however, the mechanical properties were not greatly altered by the martensite transformation.

MARITIME REACTORS PROGRAM

Reactor Compartment Filter Tests. — A third series of in-place tests was conducted on board the NS "Savannah" to determine the iodine-removal efficiency of the reactor-compartment emergency ventilation system. Only the emergency system was tested. It was desired to recheck this system because the previous test (ORNL-3351, p 6) had indicated a slight deterioration in efficiency. A further purpose was to try out a new test procedure that uses nonradioactive iodine and activation analysis techniques for determining the iodine content in the sample collectors.

Duplicate tests were made on the system with the same I^{131} tracer procedure employed in previous tests. Tests with the nonradioactive iodine procedure were also performed in duplicate. The results of this third series of 2-hr iodine tests at a system flow rate of 200 cfm are:

	Test 1	Test 2
I^{131} test efficiency, %	>99.99	99.99
I^{127} test efficiency, %	>99.996	>99.991

The system flow rate was taken as that which would exist under the emergency mode of operation with the maximum off-gas dilution.

The iodine-removal efficiencies measured in the I^{131} tests were slightly higher than in the previous test and in good agreement with earlier measurements. No deterioration of system performance is indicated. The results of the I^{127} tests indicate that a satisfactory testing procedure can be developed by making use of nonradioactive iodine and activation analysis. Additional experience is needed to determine whether there is a significant variation in iodine background with time or location of the ship and to further develop the testing procedures.

ARMY POWER REACTORS

Water Chemistry. — Three recent samples of the water in the ORR pressurized-water loop were analyzed for fission products, and the following data were obtained:

Sampling Date	Nuclear Activity (dis sec^{-1} $liter^{-1}$)				
	Sr^{91}, 9.67-hr	Sr^{92}, 2.6-hr	I^{131}, 8.05-day	I^{133}, 20.8-hr	Np^{239}, 2.33-day
5-29-62	340			1190	
6-3-62				1250	400
6-28-62	870	2200	72	1150	

The Np^{239} activity was measured to verify that the loop water had been exposed to uranium. Fission products in the loop water are now also detectable in the gross gamma spectra of freshly taken water samples as a result of current low levels of corrosion-product activity in the loop water rather than as a result of a recent increase in fission product levels. Typically, the sum of Fe^{59}, Co^{58}, Co^{60}, and Mn^{54} corrosion-product activities is 2000 dis sec^{-1} $liter^{-1}$, which is more than one order of magnitude below activity levels found at the beginning of this water-chemistry study. The fission product levels are a thousandfold lower than those reported for the coolant of the SM-1 reactor, in which microscopic cracks had developed in fuel plates, and are thought to be from traces of uranium impurity in and on the surface exposed to the coolant.

Tests are being conducted to evaluate the effectiveness of magnetite as a decontaminating medium for pressurized-water systems. Samples of loop water withdrawn through a magnetite cartridge at 425 to 427°F provided the following data:

Date	Magnetite Source and Description	Flow Rate (ml/min)	Activity Removed (%)		
			Mn^{56}	Co^{58} + Mn^{54}	Co^{60} + Fe^{59}
7-5-62	Prince Manufacturing Co., +80 mesh	60	+99	+99	95
9-11-62	Mapico black, sintered	80	+99	97	98

These results show decontamination factors ranging from 20 to >100. Thus magnetite appears to be quite effective in removing water-borne activity, most of which, in the present tests, cannot be removed by a 0.45-μ cold filter.

Mapico black is a synthetic magnetite made commerically as a pigment. Like the purest grades of natural magnetite, it is too finely divided for the present purpose. Accordingly, this material was sintered at 1000°C in an inert atmosphere. The −80 to +140 mesh portion of the resulting ground material was used in the second test, as indicated above. The favorable results indicate that this method may be used in sizing high-purity material for loop experiments in which the filter flow will be returned to the loop. Such tests, run over extended periods, would indicate the decontamination capacity of magnetite.

Postirradiation Examination of Fuel Capsules. — The initial phases of the postirradiation examination of 34 highly enriched uranium-type 347 stainless steel cermet samples clad with type 347 stainless steel were completed. These samples were irradiated in the ETR in NaK-cooled capsules prepared by Battelle Memorial Institute. The U^{235} depletion in the samples ranged from 52 to 82%. The operating temperature of the fuel surface was intended to be 600°F, but in some cases it rose to as high as 1290°F. The six samples with fuel loadings of either 34.1 or 38.4 wt % UO_2 failed because of what appears to have been overheating of the remaining 28 samples, which had fuel loadings of either 24 or 26 wt % UO_2; 23 samples were in good physical condition. The five failures in the group with the lower fuel loadings were thought to be caused primarily by overheating during irradiation.

Because many of the samples experienced significant overheating during testing, the only definite conclusion that can be drawn is that cermets with the lower fuel loading (24 to 26 wt % UO_2) can be operated to approximately 70% U^{235} depletion at a surface temperature in the neighborhood of 600°F.

REACTOR FUELS AND MATERIALS DEVELOPMENT

Radiation Effects in Structural Materials. — The decrease in electrical resistivity of Cu-15 at. % Al upon neutron, electron, and gamma irradiation is believed to be due to a short-range ordering reaction. The reaction is stimulated by the increase in atomic diffusivity resulting from the additional concentration of lattice vacancies introduced by the irradiation. A study of the flux dependence of the irradiation-enhanced reaction time has been made. Irradiations at 100°C at various positions in Hole C of the ORNL Graphite Reactor have been carried out, and the resistivity has been measured during irradiation. Also, the neutron flux has been measured as a function of position in the hole by using various thermal, resonance, and threshold detectors. An analysis of the results indicates that the reaction time at constant irradiation temperature is proportional to the inverse square root of the displacement production rate. Theoretical models lead to the conclusion that some interstitial-vacancy recombination takes place during the irradiation.

Repeated irradiations at 100°C following thermal-cycling treatments give rise to the same magnitude of decrease in resistivity as for a virgin sample. However, the reaction time is increased by a factor of about 2.5. This phenomenon is being investigated further.

FUNDAMENTAL HEAT TRANSFER AND FLUID DYNAMICS

Reynolds Stress Measurements. – The linearized-response constant-temperature hot-wire anemometer used for Reynolds stress measurements was replaced by a simpler and more rugged constant-current system. Calculations showed that electronic compensation for the effect of thermal inertia of the hot wire was not necessary for measurements in water. Linearization of the signal with fluid velocity, which is necessary in the experiment and which has heretofore been accomplished in the constant-temperature device, will now be accomplished by use of summing and squaring components of an analog computer. Commercially available hot-wire probes were found to be unsatisfactory and were replaced by homemade probes that consist of 0.0058-mm nickel wire soldered to sewing needles and supported in 1/4-in. tubing.

Boiling Studies. – In an effort to ascertain the inherent uncertainty (randomness) of the critical heat flux in saturated pool boiling, a series of tests is being conducted with distilled, deionized water outside horizontal ac-heated A-nickel tubes in a pool 6 × 6 × 9 in. deep. The maximum experimental uncertainty in the derived critical heat flux is ±5%.

Six tests were conducted with tubes that were taken to physical burnout, and 50 additional tests were made with each of two tubes protected by a burnout-detector circuit that terminates the applied current before the wall temperature exceeds a few hundred degrees Fahrenheit. The critical-flux data may be summarized as follows for saturated water at an average pressure of 14.4 psia:

Test Section	Number of Tests	ϕ_{crit} (Btu hr^{-1} ft^{-2})	
		Range	Average
		$\times 10^6$	$\times 10^6$
Tubes 1–6	6	0.368–0.546	0.464
Tube 7	50	0.334–0.548	0.472
Tube 8	50	0.483–0.596	0.537

For tubes 7 and 8, approximately 56% of the critical fluxes were within the narrow range of 0.500 to 0.550 × 10^6 Btu hr^{-1} ft^{-2}. For the same tubes, the average temperature of the cooled surface at the critical flux was 260 ± 4°F.

Larger critical heat fluxes are observed when a slight stain or dark color appears on the tube surface, and the absence of the color generally

corresponds to smaller critical fluxes. The stain does not affect the measured surface roughness of the tube, which has been at a maximum of 14 ± 3 μin. rms.

It is tentatively concluded that (1) there is considerable inherent uncertainty or scatter in the critical heat flux under saturated-pool-boiling conditions and (2) that solely hydrodynamic theories of burnout do not fully represent the phenomenon, since surface condition appears to be at least a second-order influence.

The experimental investigation of forced-convection saturation boiling with potassium (atmospheric pressure, approximately 1400°F) was continued with the 0.325-in.-diam high-flux boiler previously described. Five determinations of critical heat flux were made, and the values fell in the range from 2.2×10^5 Btu hr^{-1} ft^{-2} at a mass velocity of 7.7×10^4 lb hr^{-1} ft^{-2} to 3.5×10^5 Btu hr^{-1} ft^{-2} at a mass velocity of 1.01×10^5 lb hr^{-1} ft^{-2}. These values for the critical heat flux with potassium agree well with the correlation of Lowdermilk, Lanzo, and Siegel (NACA-TN-4382) for water boiling under similar conditions of mass velocity and boiler geometry.

A correlation of heat flux with temperature difference (the wall inlet temperature minus the saturation temperature) for the high-flux boiler showed reasonable agreement with recent results obtained at General Electric (Evandale) by Brooks and at Atomics International by Noyes. Temperature differences of from 7 to about 55°F were observed that corresponded to fluxes from 6×10^4 to 3.5×10^5 Btu hr^{-1} ft^{-2}. There appears also (ORNL data) to be some variation with quality.

Average heat-transfer coefficients were obtained by combining the heat flux based on the enthalpy change of the fluid with a mean temperature difference derived from the calculated inside wall temperature and the estimated fluid temperature. The boiling coefficients so obtained varied from four to ten times the coefficients that would be obtained without boiling at the same inlet mass flow, as predicted by the equation developed by Johnson, Hartnett, and Clabaugh. This ratio is seen to be a measure of the improvement in heat transfer that accrues from the boiling process.

Pressure-drop data for saturation boiling of potassium are correlated by a plot of the ratio of the measured total two-phase pressure gradient to the frictional pressure gradient for flow of liquid only versus X_{tt}, the Lockhart-Martinelli parameter that includes quality, densities, and viscosities of liquid and vapor.

Two-Phase Hydrodynamics Studies. – A method was developed for determination of liquid film thickness in annular two-phase flow by the principle of electrical capacitance change. This technique makes use of the fact that the capacitance between two parallel electrodes varies inversely as the effective distance between them. For example, if a probe consisting of a small metal electrode is positioned near a conducting surface over which an electrically conducting liquid is flowing, the capacitance between the probe and the surface will increase with the thickness of the film, since the presence of the film decreases the effective distance and hence increases the capacitance. A commercial instrument, called a Proximity Meter (Robertshaw-Fulton Controls Co., model 9501), is used as the

detector. In this instrument the probe forms one arm of an ac bridge. Films of water as thin as 0.001 in. have been measured in contact with air. Tap water is sufficiently conducting for this application.

Because of its high sensitivity and rapid response, a capacitance type of probe may be useful in two-phase flow studies as an indicator of flow regimes and transition. It has been observed, for example, that the amplitude and frequency of the fluctuating output signal from the Proximity Meter can be correlated to some degree with the type of flow observed visually; that is, large low-frequency fluctuations correspond to slug and spray flow and small high-frequency fluctuations correspond to mist flow.

POWER REACTOR FUEL PROCESSING

Fluoride Volatility Processing. – A study of two ternary systems of AlF_3 was initiated in search for molten fluoride mixtures capable of serving as solvents for the dissolution of aluminum-based fuel elements in the fluoride volatility process. Preliminary phase diagrams of the systems $LiF-BeF_2-AlF_3$ and $KF-ZrF_4-AlF_3$ were constructed. The system $LiF-BeF_2-AlF_3$ is divided into two subsystems: $LiF-3LiF\cdot AlF_3-BeF_2$ and $3LiF\cdot AlF_3-AlF_3-BeF_2$. The compounds $3LiF\cdot AlF_3$ and BeF_2 form a quasibinary system having a minimum temperature of 337°C at 60 mole % BeF_2. As solvents for AlF_3 at 600°C, the compounds $2LiF\cdot BeF_2$ and $2KF\cdot ZrF_4$ can retain 31 and ~40 wt % AlF_3, respectively, in liquid solution.

Protactinium Adsorption. – In previous studies, unfired Vycor glass was evaluated as a selective adsorbent for protactinium in Th-U-Pa fuel solutions. In tests made during this report period, silica gel was evaluated as an alternative adsorbent. Specially prepared silica gel was as effective as unfired Vycor glass powder in removing protactinium from thorium nitrate–nitric acid solutions. One gram of silica gel (20 to 40 mesh) in a column 0.4 cm in diameter and 14 cm long removed 95% of the protactinium from 25 ml of a solution that was 0.5 M in thorium, 11 M in HNO_3, 0.1 M in aluminum, plus a trace of fluoride. The Pa^{231} concentration was about 70 mg/liter. Under similar conditions, 20- to 40-mesh commercial silica gel removed only 57% of the protactinium, and 100-mesh fired Vycor glass powder removed only 13%. Previous experiments had shown that unfired Vycor glass removed about 96% under similar conditions.

Solvent Extraction. – The BNL Kilorod solvent extraction process for the removal of Th^{228} from U^{233} was modified so that an acid feed may be used. An acid-deficient aluminum nitrate solution was evaluated as a scrub solution for the prevention of the extraction of acid with the uranium. The decontamination factor of the U^{233} from Th^{228} and its daughters was about 10^4; the U/NO_3 ratio in the product was 2.18.

Hydrolysis of Uranium Carbides. – In order to develop a dissolution process for uranium carbide fuels, the basic chemistry of UC_x hydrolysis is being studied. In continuing tests, uranium dicarbide (nominally $UC_{1.91}$; combined C/U ratio, 1.88; single phase at 1000× magnification)

yielded 43 ml (STP) of gas per gram of carbide when hydrolyzed in water at temperatures between 25 and 99°C. The off-gas consisted of 48% hydrogen, 10% methane, 26% ethane, 5% C_3- to C_8-saturates, 8% olefins, 1% acetylenes, and 2% unidentified unsaturates. Of the total carbon in the sample, 33% was found in the gas phase and 32% in a water-insoluble wax. Temperature had no effect on the products of the reaction. This is in marked contrast to work by Litz, who reported 17% hydrogen at 83°C vs 47% at 100°C.

Dissolution of PuO_2 and PuO_2-UO_2. — Since ceramic PuO_2 and PuO_2-UO_2 mixtures are being proposed as reactor fuels, dissolution tests are being made on unirradiated samples of these materials. The instantaneous dissolution rates of PuO_2 pellets having a density of 10.3 g/cm^3 (about 90% of theoretical density) in 7 and 14 M HNO_3 were 3.5×10^{-4} and 5.6×10^{-3} mg cm^{-2} min^{-1} respectively. The dissolution rate for PuO_2 in mixtures of 14 M HNO_3, HF, and $Al(NO_3)_3$ (molar ratio, Al/F = 3) increased from 7×10^{-3} to 3×10^{-2} mg cm^{-2} min^{-1} as the fluoride concentration was increased from 0.005 to 0.1 M. The corresponding rate increase in 7 M HNO_3 was 5×10^{-4} to 1.3×10^{-3} mg cm^{-2} min^{-1}.

The dissolution rate of 80% UO_2–20% PuO_2 pellets having a density of 9.13 g/cm^3 (about 84% of theoretical density) was 13.5 mg cm^{-2} min^{-1} in 10 M HNO_3–0.5 M HF. After about 2% of a pellet had dissolved, the Pu/U atomic ratio in the solution was found to be the same as that in the pellet, indicating equal dissolution rates.

Sulfex Hot-Cell Tests. — The Sulfex process for the recovery of fuel from stainless-steel-clad UO_2 fuels involves the dissolution of the fuel cladding in sulfuric acid and the subsequent dissolution of the exposed UO_2 in nitric acid. The dissolved UO_2 is then decontaminated by the Purex solvent extraction process. In hot-cell tests, 19 Sulfex decladding experiments were made on type 304 or 316 stainless-steel-clad UO_2 pellets (93 to 96% of theoretical density). The fuel samples had been irradiated to 1545 to 28,200 Mwd/ton. Twenty-one of 32 specimens were initially passive in the 200% excess of 4 M H_2SO_4 used to dissolve the cladding and therefore required initiation of the reaction by wrapping the samples with iron wire. Losses of uranium and plutonium to the decladding reagent ranged from 0.05 to 0.3%. The activity of the decladding solutions was 10^{10} dis min^{-1} ml^{-1} from neutron-activated stainless steel components. The solution was filtered or centrifuged to remove undissolved scale, UO_2 fines, etc. Size distribution of the UO_2 that had been irradiated to between 9700 and 12,900 Mwd/ton averaged 98.9% larger than 10 mesh, 0.7% between 10 and 100 mesh, and 0.4% smaller than 100 mesh.

Solvent extraction runs with the modified Purex flowsheet confirmed earlier results — losses of uranium and plutonium to the raffinate were 0.001 to 0.012% and 0.07 to 0.12% respectively. Adjustment of the feed to 0.1 M in sulfate had almost no effect on losses. Uranium decontamination factors for beta and gamma radiation ranged between 2.1 to 5.4×10^4 and 7.1×10^3 to 1.9×10^4 respectively.

Chemical Analysis of Advanced Reactor Fuels. — Work was completed on two procedures for the determination of U(VI) in dissolver solutions that contain U, Nb, HNO_3, and HF. The first procedure involves the determination

of uranium in the presence of niobium and can be used for fairly pure solutions of niobium and uranium in which the Nb/U weight ratio is 3 or less. In the second procedure, the uranium is separated from the niobium and then titrated; this procedure can be used for solutions in which the Nb/U weight ratio is as high as 8. The separation of uranium from common types of reactor-fuel dissolver solutions by extraction of the uranium into a 0.1 M tri-n-octylphosphine oxide (TOPO) solution in cyclohexane was discussed in previous reports. This separation technique was applied to dissolver solutions containing U, Zr, Al, HNO_3, and HF. The uranium is extracted into the TOPO-cyclohexane solution from a medium 0.5 M in HNO_3 and 0.25 M in NaF, is back-extracted into 3.5 M $(NH_4)_2SO_4$ (pH = 2), and is then titrated. The relative standard deviation of this procedure is 0.2%.

THORIUM UTILIZATION PROGRAM

Thorium Materials Development. — One method of achieving beneficial strengthening of thorium metal is by dispersion hardening, which requires a fine-particle, stable dispersoid. Although theoretically sound, the method is difficult to put into practice. The fabrication technique being attempted consists of hydriding thorium particles, crushing them to micron sizes, mixing them with fine particles of thoria, and then converting the hydride to the metal. When extruded, a metal matrix with dispersions of oxide will result. Milling experiments are in progress to achieve optimum particle size.

Fuel Cycle Development: Fuel Fabrication Studies. — Thoria-urania mixed oxides, supplied by the Chemical Technology Division from the sol-gel process, were used to study various parameters of the vibratory-compaction technique. The effect of particle size distribution on density was investigated. By use of a ternary size distribution, with particles 60% coarse (−10 +16 mesh), 15% medium (−70 +100 mesh), and 25% fine (−200 mesh), rods were prepared with a density of 90% of theoretical. A binary particle distribution is also under study. The percentage of the coarse particles (−4 +16 mesh) was varied from 65 to 80% by using fines of −200 mesh. When the coarse fraction was as low as 65%, only 88% density was achieved. With 70 to 80% coarse fractions, densities ranged from 89.5 to 90.3%.

Fuel Cycle Development: Irradiation Experiments. — Five irradiation capsules (0.460-in. OD, 0.015-in. wall) were fabricated. The 19-in.-long tubes were vibratorily compacted with sol-gel-derived ThO_2—6 wt % UO_2. Three of the tubes were of Zircaloy-2, and two were of type 304 stainless steel.

Ten of the more than thirty other capsules under irradiation have been removed for examination. Even those oxides exposed to the highest heat rating, 44,000 Btu hr^{-1} ft^{-1}, have shown no evidence of sintering. The fission gas release has been lower than expected. No deleterious results have been observed, but the examinations are not complete.

BNL Remote Fabrication Mockup. – Equipment development for the remote fabrication of fuel rods for the Brookhaven National Laboratory Criticality Experiment has been completed. The components were assembled and remotely operated in the Fabrication Laboratory. The equipment works satisfactorily, and soon it will be installed in the Kilorod facility.

Fuel Cycle Development: Fuel Preparation. – In the sol-gel process for the preparation of 3 at. % U–ThO_2 for the Kilorod Program, procedures and process-control measures were developed for the preparation of 7-kg batches. Brookhaven National Laboratory specifications for uranium-content uniformity of 3 ± 0.03 at. % uranium were met. Complete dispersion of steam-denitrator-product ThO_2 in uranyl nitrate–HNO_3 solution was consistently demonstrated by the use of an NO_3^-/ThO_2 mole ratio 10% in excess of that shown by conductimetric titration to be optimum for stable 2 to 6 M ThO_2 sols. Dispersion was accomplished by recirculating the hot suspension through a critically safe tank and pipe loop by means of a centrifugal pump. Uniformity of the U/Th atom ratio in all gel fragments was accomplished, and enrichment of the fines with uranium was prevented by neutralizing the excess nitrate with ammonia.

In experiments involving 50 to 200 g of 3 at. % U–ThO_2 and a gastight furnace, sol-gel oxides calcined in air at 1150°C for 20 min and then reduced and cooled in argon or nitrogen had as low a residual gas content at 1200°C in vacuum (less than 0.005 cc/g) and as high a density as those reduced and cooled in argon–4% hydrogen. In 7-kg charges and a larger furnace, the residual gas content was 10 times higher when no hydrogen was used, but the increase was thought to be due to the reoxidation of the UO_2 by oxygen that leaked in during cooling.

Thorium oxide–carbon gels were prepared by the sol-gel process, and the carbon was uniformly distributed. Gels in which the C/ThO_2 atom ratio was 5.5 and 4.0 and that were heated to 1800 and 2000°C for 2 hr gave products in which 95 and 80% of the oxide were converted to thorium carbide.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. – Design work on final details and on minor revisions to completed designs continued. Emphasis was placed on completion of the electrical design.

The coolant drain tank was completed, and the fuel flush tank is nearing completion. Progress was made in the fabrication of all other INOR-8 components.

The construction of the drain-tank cell was completed, and the cell was made ready for leak testing. The reactor containment vessel is nearing completion. The outside construction work is complete except for checkout of equipment. The building contractor has requested a time extension until November 30. This request is being reviewed.

Reactor Analysis. – Overall temperature distributions in the fuel and graphite of the core were calculated, taking into account detailed distributions of heat generation and fuel velocity. With the reactor operating at 10 Mw and an inlet temperature of 1175°F, the predicted

maxima in the overall temperature distributions in the fuel and graphite were 1262 and 1296°F respectively. Associated average temperatures for the fuel and graphite were calculated from the temperature distributions; the nuclear average temperatures in the fuel and graphite were 1213 and 1257°F, respectively, while the bulk average temperatures in the fuel and graphite were 1198 and 1226°F respectively. Permeation of 0.5% of the graphite volume by fuel salt would increase graphite mean temperatures by less than 2°F and have no effect on the circulating fuel temperatures.

Temperature coefficients of reactivity were calculated for the fuel and graphite regions. With no thorium in the fuel, the temperature coefficient for the fuel salt was -4.45×10^{-5} $(°F)^{-1}$ and was -7.27×10^{-5} $(°F)^{-1}$ for the graphite volume. Power coefficients of reactivity for various modes of operation were also computed; in order to maintain the outlet fluid temperature constant at 1200°F while the reactor power was raised from 0 to 10 Mw, 0.18% reactivity addition would be required. Maintaining the average of the inlet and outlet fluid temperatures constant while increasing the power from 0 to 10 Mw required a 0.47% reactivity addition.

The reactivity worth of the three control rods was calculated for fuel containing no thorium. The total worth was 7.4% reactivity, compared with 6.7% obtained previously with fuel containing 1 mole % ThF_4.

The present shielding of the secondary coolant salt appears adequate with either the proposed $LiF-BeF_2$ salt or the Flinak coolant.

Development of Components and Systems. — The engineering test loop continues to circulate flush salt at 1200°F through the 8-in.-diam graphite core mockup after 1700 hr of operation. The flush salt contains approximately 1500 ppm Zr, which is equivalent to dilution by fuel salt of approximately 1-1/2 wt %. Chemical analyses of samples withdrawn indicate that the zirconium content of the drain-tank salt has decreased. This could indicate settling of ZrO_2, despite the apparent low oxygen content of 200 to 300 ppm.

In the core hydraulic model the flow rate was measured in 77 randomly chosen fuel channels, and the following characteristics were noted.

1. The average flow rate per channel at the outer edge of the core is about 18% less than at the center of the core.

2. Half the fuel channels are perpendicular to the other half, and the flow rate in one half of these channels is about 22% less than in the other half.

3. At a given radius and set of channels, the flow rate per channel may be as much as 15% above or below the average. This spread has been attributed to tolerance variations, which will be considerably reduced in the actual reactor.

Thermal-shock cycling of the drain-tank cooler test has continued for a total of 1027 cycles, 729 of which were from the 1200 to 1400°F temperature range. Two of the nine reactor-grade thermocouples have failed.

An all-metal reflective-type insulation was selected for use on the main reactor piping in the reactor cell. This insulation is equivalent to more conventional insulations in performance and has the added advantages of superior structural strength, lightness, and low neutron activation; it is dust- and crack-free. The insulation selected is a product of the Mirror Insulation Company.

A prototype of the core drain-line freeze valve was tested in the orientation specified for instrumentation of the valve in the reactor cell. The valve did not operate satisfactorily. This was attributed to a gas pocket which formed in the freeze-plug area, and the valve is now being modified to eliminate that difficulty. Instrumentation and controls designed for operation of the freeze valves in the reactor were tested, and they operated satisfactorily.

The sampler-enricher access into the pump bowl was provided with a shield around the capsule guide to reduce the deposition of liquid salt at the access entrance caused by an aerosol resulting from splashing and bursting bubbles on the free surface of the salt.

Work was completed on the construction of a full-scale helium purification mockup, and the test program is in progress. The oxygen removal unit will be studied with respect to purification efficiency, pressure drop, temperature distribution, and integrity under thermal cycling. The oxygen analyzer purchased for this system has performed below the manufacturer's claim but should be adequate under controlled conditions.

MSRE Pump Program. — The prototype fuel pump was rebuilt, and two series of tests were performed with molten-salt $LiF-BeF_2-ZrF_4-ThF_4-UF_4$ (70-23-5-1-1 mole %) at 1200°F for periods of 72 and 5 hr. During the 72-hr series of tests there was considerable noise and vibration present throughout the pump and test system. The pump was removed from the test stand and inspected prior to the second series of tests. The pump was reassembled, and a larger-sized orifice was put into the system. The noise and frequency were considerably reduced; it appears that the orifice plate is a major source of vibration. To further reduce the effects of noise and vibration on the test stand, the system piping (6-in. IPS) will be restrained during steady-state operation.

The piping and structure for one lubrication stand has been assembled, and that for the other unit is nearing completion. The pumps are ready for installation as soon as they pass helium leak tests.

Fabrication of the fuel and coolant pump tanks is about 75 and 60% complete respectively. Fabrication of the two bearing housings is expected to be complete by December 1.

Fabrication of the drive-motor vessels was initiated, and delivery of the motors with vessels is expected in December.

The Inconel centrifugal pump (PKP type) has accumulated 6746 hr of operation in an endurance test with molten-salt $LiF-BeF_2-ThF_4-UF_4$ (65-30-4-1 mole %) at 1225°F, 1950 rpm, and 510 gpm. The test pump with a molten-salt journal bearing is awaiting installation of a larger drive motor prior to startup; the larger drive motor will permit operation at greater speeds and loads on the bearing. A support spool piece is being fabricated to accommodate the larger drive motor. The system salt was analyzed for oxide contamination and found to be acceptable for further use.

Instrumentation Development. — Developmental testing of mechanical methods of attaching mineral-insulated sheathed thermocouples to thin-walled tubes on the MSRE radiator was concluded, and a design for MSRE installations was established. The final design consists of a braze-metal saddle and strap arrangement with the hot junction covered with

1/4 in. of Fiberfrax paper and bonded to the tube with Thermon X-63, a heat-conducting ceramic cement. Measured temperatures obtained with this type of attachment checked to within 5°F of the inside wall temperature at 1000°F with no air flow and to within 10°F with air flow.

Long-term testing of thermocouples of the type specified for use on the MSRE continued. Calibration drift of six prototype surface-mounted couples continues to be less than 2°F after 6000 hr of operation at 1200 to 1250°F in a tube furnace. Eight prototype surface-mounted thermocouples installed on an engineering test facility have accumulated 3000 hr of operation at temperatures between 1040 and 1200°F. These couples continue to agree with their respective reference couples to within 3 to 6°F.

Ten prototype couples were installed in a drain-tank bayonet-cooler test facility to determine whether these couples would withstand the severe thermal shocks to which they will be exposed in similar service in the MSRE. After four weeks of continuous cycling between the temperature limits of 300 and 1400°F, eight couples are still functioning.

Several commercially available, inorganic, sealing and potting compounds are being tested and evaluated in an attempt to develop simpler and better methods of hermetically sealing thermocouple and signal lead termination at disconnects located in high radiation areas within the reactor shield. Compounds which cure and set at room temperature are being tested first. No satisfactory seal has been obtained with Thermostix (a high-temperature inorganic adhesive and potting compound) by the methods tried to date, although this material adheres well to ceramics, lavite, and metals. Several other compounds remain to be tested.

Results of long-term tests of a ball-float-type molten-salt level transmitter and a temperature-scanning system developed for use on the MSRE continue to be satisfactory. The level transmitter has been in operation for nine months. The temperature scanner has operated in excess of 1200 hr.

Development and testing of a conductivity level probe for single-point detection of level in the MSRE storage tanks have continued. Results of tests to date have been satisfactory, and design of the level-probe assemblies for the MSRE is under way.

NUCLEAR SAFETY

Release of Fission Products on Out-of-Pile Melting of Reactor Fuels. — The initial series of five UO_2 melting experiments for the study of fission product release by the cored fuel and center tungsten resistor method was completed. They include those melts of three bare UO_2 PWR-type pellets and one each of the pellets clad in stainless steel and Zircaloy-2. By this method it is believed that a significant improvement in accident simulation is indicated. The melt is observed to puddle at the base, leaving a hollow shell of the original diameter with numerous small melted extrusions and cracks.

The fission product release data are summarized in the table below. A key release number is perhaps illustrated by the rare-gas values, which

Fission Product Release from Single-Element UO_2 Melted in Tungsten Resistor Furnace

Sample: 39 g of UO_2 (PWR type)

Atmosphere: 400 cc of pure helium per minute

Element	Heat Duration (min)	UO_2 Vaporized (%)	Gross Gamma Release (%)	Percentage of Individual Fission Products Released							
				Xe-Kr	I	Te	Cs	Ru	Sr	Ba	Ce/RE
UO_2	5.0	0.8	7.1	63	47	56	44	1.6	1.6	5.3	< 0.6
UO_2	4.0	0.2	5.7	50	30	42	41	0.4	0.9	2.9	< 0.5
UO_2	4.4	0.3	6.9	41	25	29	> 40	0.03	1.2	4.3	0.5
UO_2 (SS clad)	4.7	0.2	5.0	56	52	73	46	0.5	1.0	3.4	0.1
UO_2 (Zr clad)	7.0	0.1	2.6	52	24	1.9	27	0.08	9.8	7.7	0.5
UO_2 + BeO	1.5	20	30	100	90	92	91	61	2.1	4.6	2.2

(Previous results of melting micro-size sample of highly irradiated UO_2 by electric-arc image method)

probably correspond to the approximate percentage of the total sample having melted. When compared with the results previously obtained by melting micro-size samples in a carbon electric-arc image furnace, the only striking difference is in the relatively low ruthenium release. This readily suggests the importance of the trace oxygen in enhancing the volatility of ruthenium, as was discovered in previous work. Other interesting differences are seen between the metal-clad samples. The stainless steel cladding shows little effect, and the results resemble those for the bare sample. On the other hand, the zirconium-clad sample shows a marked suppression not only for ruthenium but also for tellurium. A less-striking suppression is also observed in the iodine release.

Release of Fission Products on In-Pile Melting of Reactor Fuels. — Experiments consisting of the melting of UO_2 in the ORR are being conducted in order to simulate reactor accidents. Experiments 7 and 8 have been performed, and the experimental assemblies were examined in the hot cells. Radiochemical analyses were completed for experiments 6 and 7; the results are as follows:

Material Released by the In-Pile Melting of UO_2 (%)

	Experiment	Sr^{89}	Zr^{95}	Ru^{106}	I^{131}	Te^{132}	Cs^{137}	Ba^{140}	Ce^{144}	UO_2
Release from fuel residues and holder	6	54.6	60.2	46.0	98.9	99.0	99	50.8	48.8	
	7	5.3	1.0	11	96.0		96.5	14.1	2.59	
Release from furnace	6	0.92	0.089	1.29	94.1	53	87	0.46	0.072	0.284
	7	0.87	0.037	2.0	49.7	< 11.1	74.0	0.59	0.323	0.0151

Fission product release in experiment 6 was approximately the same as in previous experiments, although the specimen did not melt completely due to a lower neutron flux. Experiment 7 was performed under different heat transfer conditions at a lower temperature than experiments 2–6, and the specimen formed a hollow cylinder similar to that of experiment 1. Compared with experiments 2–6, experiment 1 showed significantly less release from the fuel residues and holder, but it showed about the same release from the furnace for many of the fission products.

Additional particle size calculations have been made for experiments 2–5 by using the diffusion-channel technique. The following tabulation shows that two particle size groups have been found. One group is centered around an apparent diameter of 22 A, and the other group has an apparent diameter of 30 A. The data for ruthenium and cerium are exceptions; usually these isotopes do not appear among these fine particles, but are carried on larger particles. The remainder of the activity for each isotope, aside from that tabulated, is apparently in the form of larger particles.

Average Percentage of Each Isotope in Exit Gas Carried by Very Fine Particles

Effective Particle Diameter of Group, 22 A				Effective Particle Diameter of Group, 30 A			
I^{131}	39	Cs^{137}	15	Zr^{95}	3	Ce^{144}	13
Sr^{89}	5	Ba^{140}	13	Ru^{106}	10	UO_2	9
Te^{132}	3						

Fission Product Transport Evaluations. — Efforts to develop a method for identifying the chemical form of fission products released in simulated reactor accident experiments led to construction of apparatus designed to permit determination of the heat of vaporization of radioactive materials by a counting technique. It has been tested only with molecular iodine labeled with I^{131}. Samples sealed in a bulb having a 1-mm-ID capillary tube with a break seal near the sealed end are supported in a quartz tube heated by a flexible heating tape. The buildup of iodine activity in a small charcoal trap is measured as it is transported from the sample ampoule by a constant flow of gas at constant temperature. The iodine activity in the charcoal trap is measured by counting apparatus consisting of a crystal detector connected to a linear amplifier, a decade counter, and, through a linear count rate meter, to a recorder.

The data show that it is possible to obtain constant rates of iodine buildup over the temperature range 25 to 115°C, and the rate values obtained can be correlated by an Arrhenius-type plot from which the heat of vaporization of iodine can be calculated. The best values agree quite well with the accepted value for this property of iodine. This demonstrates the validity of the basic assumption on which this method rests, namely, that the rate of increase of iodine activity in the charcoal trap at constant temperature and gas flow rate is directly proportional to the vapor pressure at that temperature.

Versatility of the apparatus was also shown by observing the removal of radioactive iodine fixed on quartz or metal surfaces, either by adsorption or reaction, by interchange with gas-borne normal iodine.

Characterization and Control of Accident-Released Fission Products. — Previous studies employing the particle and vapor characterization technique based on diffusion measurements have indicated that neither high-efficiency particulate filter media nor activated-carbon beds are effective in the removal of ultrafine particles from air streams. Consequently, the experimental work is now being directed toward investigation of less-conventional methods of particle removal.

In order to evaluate one such method, an air stream carrying I^{131}-labeled aluminum oxide particles, with diameters in the 0.002- to 0.007-μ range, was passed through a chamber in which steam was condensing, forming fog. The objective was to enclose the particles in fog droplets to permit more effective filtration. The results, for filtration with and without prior fog formation, indicated removal efficiencies of 99 and 85%

for 0.002-μ particles and 85 and 35% for 0.007-μ particles. These results are encouraging, but in practice considerably greater improvement is required. While optimization of this method might provide a partial solution to the problem of very fine particle containment, it appeared desirable first to study briefly some alternative methods. The use of foam encapsulation for iodine vapor and ≧ 0.074-μ aerosols has been reported by L. Silverman and co-workers at Harvard. In order to determine the effectiveness of this approach for ultrafine particles, a series of tests in which a foam column is utilized has been initiated and is partially completed.

Nuclear Safety Pilot Plant. – Plans are being made in cooperation with the Harvard Air Cleaning Laboratory for a series of foam-suppression tests in the cell in which the nuclear safety pilot plant will be installed early next year upon receipt of the components. In the foam-suppression tests, approximately 10 curies of fission products (primarily iodine from volatilized iodine and mixed fission products from the combustion of irradiated uranium foil) will be released into the cell. Foam generators located in the cell will then be started up to produce a foam that will fill the cell and entrap the fission products. Sketches of the proposed experimental system were completed, and cell preparations are currently under way.

Reactor Containment Handbook. – A draft of Chapter II, "Codes Criteria and Regulations," was reviewed by ORNL personnel, and the comments received are being incorporated into the draft. Chapter II and Chapter IV, "Energy Sources," which was previously completed, are yet to be edited, reproduced, and sent to the AEC for review.

Subcontract negotiations for the preparation of four chapters of the handbook by a vessel manufacturer were completed; the contract was awarded to the Bechtel Corporation. The four chapters, Chapter VII, "Containment Proof Tests," Chapter IX, "Materials Properties," Chapter X, "Design Details," and Chapter XI, "Economics," are scheduled to be completed in draft form by March 1.

WASTE DISPOSAL DEVELOPMENT

Clinch River Study. – The recovery of a calcite-coated current meter from the Clinch River at mile 19.1 during August 1962 provided the first direct evidence of calcium carbonate precipitation from the river water. The meter had remained submerged in the river, approximately 5 ft off the bottom, for 14 months. The calcite coating is 0.004 to 0.005 in. thick and contains significant amounts of Sr^{90} (~10 μμc/g) and Ru^{106} (~124 μμc/g). Such material, if incorporated in river bottom sediments, would add appreciably to the radionuclide content of the sediment.

A laboratory investigation of the effects of power releases from Melton Hill Reservoir on the dispersion of radionuclides in the Clinch River was conducted. The tests were run in a tilting flume, operated by the U.S. Geological Survey, at the National Bureau of Standards in Washington, D.C. The variation of tracer concentration with time and distance was measured for conditions of a steady discharge and of a wave generated by the same magnitude of discharge. Preliminary results indicate that the dispersion characteristics of the wave and of steady flow are similar, with the dispersion coefficient for steady flow being slightly smaller.

Disposal in Deep Wells. – Compositions of two cement mixes for the hydrofracturing experiment have been specified for a simulated waste solution of the following molar composition: $NaNO_3$, 0.317; $Al(NO_3)_3$, 0.022; NaCl, 0.006; NH_4NO_3, 0.025; Na_2SO_4, 0.037; NaOH, 0.219. One of the mixes will tolerate a tenfold increase in waste concentration. The mixes remain pumpable for a minimum of 8 hr and attain a final set within 7 days. Once set, the permeability is less than 0.05 millidarcy. During setting there is no water separation in these mixtures, though there is a density gradient.

Tests indicate that essentially none of the filtrate water from these mixes will leak off into the matrix of the shale formation present at ORNL. Other tests show that the concentration of cesium and strontium in the filtrate water is half and one-tenth, respectively, that in the original waste.

A third, less expensive, mix is partially developed.

Disposal in Natural Salt Formations. – The array center heater was operated for 27 days, reaching a steady-state sheath temperature of approximately 430°C and a salt temperature of 183°C 7 in. from the surface of the hole. There was no visible disintegration of salt at the surface of the hole. Failure of heater leads in the peripheral heaters was found to be due to sulfide embrittlement of the nickel wire.

The wall and floor heaters were operated for 33 days. Heater sheath temperature in the floor was 600°C as compared with 560° in the wall, and 250°C at 6 in. out in the salt compared with 230° at the same point in the wall. However, more shattering occurred in the wall than in the floor.

Engineering, Economic, and Safety Evaluation. – An analysis is in progress to ascertain the dose that man may receive as a result of the discharge of radioactive fluids to the Clinch River. A number of avenues are being considered by which man may be exposed to ionizing radiation: (1) consumption of contaminated water and fish, (2) consumption of agricultural produce irrigated with river water, (3) external dose due to contaminated water and bottom sediments during recreational and industrial use of the river, and (4) buildup of radionuclides in water systems utilizing river water. An estimate will be made of the aggregate dose likely to be received by the occupational worker, the population in the vicinity of ORNL, and the average individual of the population at large.

Foam Separation Studies. – The precipitation of calcium and magnesium from ORNL tap water made 0.005 M each in NaOH and Na_2CO_3 and 2 ppm in

Fe^{3+} was tested in 2-, 3-, 4-, and 9-in.-diam sludge columns. With slow (about 10 rpm) rotational agitation of the sludge and at flow rates of 10 to 30 gal ft^{-2} hr^{-1}, hardness was reduced in each column to about 2.6 ppm, as $CaCO_3$, from its initial value of 100 ppm.

In studies of strontium removal by foam separation, wherein decontamination factors of 500 to 5000 were achieved, the feed solution had a composition similar to that of the effluent from a sludge column, and then a surfactant, dodecylbenzene sulfonate, was added. The concentration of the surfactant was 50 ppm.

In continuous, countercurrent foam separation tests, the surfactant was efficiently removed, and its reuse or the avoidance of unnecessary addition of alkylbenzene sulfonates to river water seems workable. The surfactant concentration was easily reduced from 50 to 5 ppm.

PHYSICAL RESEARCH PROGRAM

PHYSICS AND MATHEMATICS RESEARCH

High-Energy Electronuclear Machines: Cyclotron Analogue II. — A new electron source for the analogue of the Mc^2 cyclotron was successfully tested. The optical quality of the beam from the new source is markedly improved over that realized with the earlier filament and accelerating slit. In the new source, electrons from an incandescent filament are first passed through a 180° analyzer system at an energy of 600 ev. From the exit slit of the analyzer section, they are accelerated to a final energy of 3300 ev, corresponding to an orbit radius of 1.75 in. A mechanical linkage permits adjustment of the angle of injection. The salient features of the appearance of the electron beam accelerated from the new source are as follows: (1) The radial width of the beam near the center of the cyclotron is reduced by a factor of 3 and is now approximately equal to the radius gain per turn from acceleration. (2) The beam passes through the difference coupling resonance, $\nu_r - \nu_z = 1$, with only slight axial broadening, even without the geomagnetic compensating coils; previously, a tenfold attenuation of the beam was observed under similar conditions. (3) The sum coupling resonance, $\nu_r + \nu_z = 2$, is barely visible; previously, the axial amplitude of the beam doubled at that point. (4) The integral radial resonance, $\nu_r = 2$, is passed through with little or no attenuation.

Basic Studies of Cryogenic Electromagnets. — A 30- to 50-kilogauss superconducting magnet was designed with a working volume 2 in. in inside diameter by 2 in. long for the region in which the field is within 10% of the central field, and the first of nine sections was constructed.

Some recent results from tests in the high-field solenoid (100 kilogauss) indicate that the critical current for 10-mil Nb-Zr wire is independent of tension up to stresses of 7×10^4 psi for longitudinal magnetic fields. Measurements on the superconductor Ta-Ti in the form of cold-worked wire show that the critical field values are similar to those of Nb-Zr, but the critical currents are lower by an order of magnitude. The critical current and field values of Nb-Zr and Mo-Re wire from European sources were found to be comparable to the properties of U.S. products.

Multiparticle Medium-Energy Nuclear Physics: Heavy-Particle Physics. — Oxygen ions from the tandem Van de Graaff were used to extend the measurements of the excitation function for the reaction $C^{12}(O^{16},\alpha)Mg^{24}$. Measurements were taken at 30 and 150° (laboratory); the range of excitation in the compound nucleus Si^{28} now extends from 23.96 to 30.85 Mev. Fluctuations observed previously in a limited energy region are apparent over the entire measured energy range. The data are now being analyzed for correlations and variations in the width of the resonances. The angular distribution of the transfer reaction $Al^{27}(O^{16},N^{15})Si^{28}$ was measured at 30 Mev (laboratory) for two states in the residual Si^{28} nucleus. Typical transfer reaction curves were obtained with peaks at ~78° for the transfer to the ground state and the excited state. The value of the nuclear radius parameter r_0 deduced from this experiment is 1.75 f.

Multiparticle Medium-Energy Nuclear Physics: The 86-Inch Cyclotron. — Radiations from the decay of 3-hr Ho^{167} suggest that rotational structures are populated in Er^{167}, based on configurations $7/2^+$ [633] at ground, $1/2^-$ [521] at 207.8 kev, and $5/2^-$ [523] at 585.4 kev. The first rotational excitation of the ground state is of M1 + E2 character and of 79.3 kev energy. The energy parameters for the four excited levels of the K = $1/2^-$ sequence (at 207.8, 264.9, 281.5, and 415.1 kev) are $3\hbar^2/\mathcal{J}$ = 67.2 kev, B = +0.001 kev, and a = +0.70. The values of the parameters are characteristic of $1/2^-$ [521] configurations. The 83.4-kev rotational transition ($7/2 \rightarrow 5/2$) in the $5/2^-$ [523] band has a mixing ratio of M1/E2 = 7. A $5/2^-$ [512] level is proposed at 347.7 kev which de-excites by intense E1 radiation to the ground state.

High-Voltage Experimental Program: Elastic Scattering of Alphas by O^{18}. — Differential elastic-scattering cross sections have been measured for 2.4- to 3.5-Mev alphas on O^{18} at center-of-mass angles corresponding to the first four zeros of the Legendre polynomials. Of the eight resonances previously observed in this energy region by the $O^{18}(\alpha,n)Ne^{21}$ reaction, spin and parity assignments have been made to six levels by the combined experimental data.

High-Voltage Experimental Program: Gamma Rays from Neutron Capture in Lead. — Measurements of 30-kev neutron capture spectra [Phys. Letters 1, 262 (1962)] have been extended to allow results to be obtained for a variety of neutron energies. A neutron flight path of 50 cm and a time resolution of 12 nanoseconds (full width at half maximum) make possible the observation of individual resonances in the energy range 10 to 100 kev. A three-dimensional analyzing system with 16 × 128 channels is used for the simultaneous measurement of neutron and gamma-ray energy. Targets of natural and radiogenic lead show resonances in Pb^{206} at 18, 22(?), 27, and 45 kev, as well as the well-known resonance in Pb^{207} near 45 kev. The shape of the latter for the 7.41-Mev ground-state transition in Pb^{208} has been studied, and intensities of three transitions in Pb^{207} have been obtained for each of the other four resonances. These intensities change markedly from resonance to resonance, but the average values cannot be explained in terms of the expected multipolarities unless p-wave capture is important for these resonances.

Electronuclear Machines: The Isochronous Cyclotron (ORIC). — The installation and testing of the power amplifier and other components of the rf system and of the beam deflection and ion-optics systems have continued. The power amplifier was assembled and mounted on the resonator housing, the very extensive control wiring is nearly completed, and components of the system are being tested. The first section of the magnetic channel was designed and is being fabricated; the electrostatic deflector system was redesigned and relocated to accommodate the channel. The No. 1 beam switching magnet for the ion-optics system was fabricated and is ready for installation and testing.

A "library" of current settings for the main magnet, valley, and circular trimming coils is being prepared, along with associated orbit properties. This will aid in setting up the cyclotron for the acceleration of any preselected ion to any specified energy.

Stable Isotopes Development. — Oscilloscope monitoring of collected ion beams was shown to be superior to monitoring by means of milliammeters. The new technique permits instantaneous detection of instabilities and thereby permits early application of corrective adjustments. Current improvement in plutonium sample purity is largely due to the development of the oscilloscope monitoring technique.

The approach planned to achieve increased ion output from calutrons involves a better understanding of the basic ionization processes. Since direct measurements are not practical, several experiments are being performed to obtain the knowledge needed in this field.

A thermal-diffusion cascade operating on a feed gas of CH_4 was successful in enhancing the C^{13} concentration in the gas to 30%. Improved performance of the system was achieved by reducing the hot-wall impurities by activated-carbon traps from the commercial CH_4 feed gas. Further refinements to the system and adjustments in gas feed and draw-off rates are expected to result in increased enrichments of C^{13}.

Special Separations. — Three plutonium runs were completed in the new containment facility which became operational on September 13, 1962. Two receiver pockets containing an estimated 1 g of > 90% Pu^{241} are being processed, and the material is being prepared for analysis.

Low-fired (500°C) plutonium oxide was demonstrated as a satisfactory calutron feed material for internal chlorination by CCl_4. A total of 250 g of plutonium enriched in $Pu^{240-242}$ was separated from americium daughters, converted to carbonate, and heated to low-fired oxide.

Plating of uranium, neptunium, and americium oxides onto various forms has continued and has resulted in shipments of 40 Am^{241} sources, 16 large nickel cylinders and end caps plated with Np^{237}, 32 fission chambers plated with U^{235}, and 7 U^{232} sources plated on nickel. Experimental plating of normal uranium on platinum was completed, and the radioactive plating is being started.

CHEMISTRY RESEARCH

Thorium Oxide Studies. — The thorium oxide preparations irradiated in D_2O (250 to 300°C) in the LITR in experiment C-43-6B were recovered for examination. More than 12 months of irradiation at a neutron flux of 2×10^{13} was achieved. The materials included the P-82 thoria pellets, sintered thoria compacts (prepared by a different method than that for the P-82's), arc-fused thoria pellets, fired sol-gel thoria particles, arc-fused thoria fragments (44 to 74 μ), sol-gel thoria spheres (44 to 74 μ), and 1600°C-fired thoria powder (i.e., classified DT-46).

Visual examination of the materials was hampered by the accumulation of a considerable amount of corrosion products. However, the preparations were freed from the bulk of the corrosion products by stirring them in water in a polyethylene beaker with a Teflon-coated stirring bar. Evidently, only 13 of the 20 P-82 pellets were submerged in water during the irradiation period, and the rest were irradiated in the vapor phase. Those irradiated in water appeared relatively undamaged. The vapor-phase irradiated pellets, however, showed considerable surface damage, and two of the pellets broke during the stirring operation, indicating structural

damage. The interior of the broken pellets showed the presence of a tan-colored surface shell and a black coloration from the shell halfway into the center of the pellet. The other sintered-pellet preparation (a portion of which was also irradiated in the vapor phase) showed considerable damage, but the extent was obscured by extensive corrosion-product accumulation.

The arc-fused pellets and the sol-gel particles appeared virtually undamaged. A fragment knocked from the side of one of the arc-fused pellets during stirring revealed an opaque, glossy-black interior. Before irradiation the arc-fused pellets were light red and translucent.

The ground, arc-fused material appeared visibly unchanged, while the Houdry spheres had absorbed corrosion products and showed some loose agglomeration. The DT-46 material had escaped from its stainless steel container around the fritted disk that formed the enclosure and then had settled on the lower container. It had been formed into a porous plug (readily broken in two) that contained about 2% iron, nickel, and chromium.

Detailed examination of the materials is under way. Material-recovery balances will be obtained, and preparations were made in order to obtain wear tests, metallographic cross sections, and x-ray diffraction patterns of the surfaces and interiors.

Protactinium Chemistry. — The distribution coefficients for protactinium between aqueous sulfuric acid and diethylbenzene solutions of two secondary amines, S-24 and LA-1, are much smaller than for N-benzylheptadecyl amine, but the changes in distribution with amine and sulfuric acid concentration are similar. For all these amines there is a change in the dependence of the distribution coefficient (DC) on sulfuric acid concentration in the range 6 to 9 N, the dependence being higher above this range. For LA-1 and S-24 amines the DC decreases with the fifth power (approximately) of the sulfuric acid concentration in the higher acid range, compared with the eighth power (approximately) for N-benzylheptadecyl amine. There is good indication that these DC's may be correlated much more simply with the concentration of amine sulfate — $(amine)_2H_2SO_4$ — than with the total amine concentration. The amine sulfate concentration is a function of both the amine concentration in the organic phase and the sulfuric acid concentration in the aqueous phase. Since only a small fraction of the amine is in the sulfate form [most is the bisulfate: $(amine)H_2SO_4$] when equilibrated with sulfuric acid in the concentration range of interest, accurate measurements of the amine sulfate concentration are quite difficult. The titration procedure described previously (ORNL-3321, p 25) has been refined to the point that the limit of detection of amine sulfate is about 1% of the total amine.

The distribution of protactinium between aqueous sulfuric acid and Dowex 1 resin was dependent upon the total amount of protactinium present. For a given acidity, the amount of protactinium remaining in the aqueous phase was constant, and, as more protactinium was added, it sorbed on the resin. This concentration in the aqueous phase was directly proportional to the square of the sulfuric acid concentration. Such behavior indicates that the protactinium absorbed in the resin has an activity coefficient independent of the concentration.

Surface Chemistry. — A new sample of ThO_2 for adsorption tests was prepared by grinding fused material and classifying the resulting powder to give a fraction with particle diameters between 0.7 and 1.2 μ. Tests of this preparation showed that its adsorption of HCl corresponded to about 5% of the calculated surface sites when the concentration of the HCl solution was 10^{-5} M, and about 12% when the concentration was 10^{-3} M. These values are not significantly different from those previously obtained (ORNL-3242, p 21) with HNO_3 adsorption on a thoria sample prepared from the oxalate.

Pyrolysis and Radiolysis of Organic Liquids. — Calibration factors in terms of the peak heights have been determined for the identified constituents of the gaseous and low-boiling products formed during the pyrolysis of pure biphenyl. These factors were applied to the data obtained from the pyrolysis of 3 g of biphenyl at 425°C for 12 hr, in which about 0.5% decomposition was produced. Consequently, the following quantities of products were found, expressed as moles $\times 10^4$ per mole of biphenyl decomposed: hydrogen, 21; methane, 0.25; ethane, 0.1; ethylene, ~0.1; and propane, ~0.05.

Analysis of Molten Salts by Electrochemical Methods. — An experimental device was designed and fabricated for conducting voltametric studies in molten fluoride salt systems. The melt is contained in a graphite cell about 2 in. in diameter by 4 in. long. The cell is fitted with a graphite thimble as an inner compartment which accommodates an isolated counter electrode. A convenient volume of molten material, about 45 ml, is introduced into the system. The depth of the molten salt in the cell and in the isolated compartment is maintained at the same level. Electrical contact is made through the graphite thimble. To maintain a vacuum or controlled atmosphere, the cell is enclosed in a quartz liner approximately 2-1/2 in. in diameter and 10 in. long which is fitted with a removable cap. In the cap, five 3/16-in. Swagelok fittings are utilized as a means of access to the melt for the placement of three electrodes, a thermocouple, and a helium inlet tube. Connections for the maintenance of vacuum and inert atmospheric conditions are provided by means of a three-way vacuum stopcock. The eutectic mixture LiF-NaF-KF (46.5-11.5-42 mole %) was chosen as the first solvent electrolyte for examination because of the wide potential range which should be available before electrolyzing the melt itself. Current-voltage curves were recorded on the melt at 495°C, utilizing a controlled-potential polarograph and three platinum electrodes. A cathodic wave at −0.5 v and an anodic wave at +1.1 v vs a platinum quasi-reference electrode were noted. It is believed that these waves are due to metallic impurities, since on analysis it was found that the salt contained 200 ppm Fe and 100 ppm Cr. To eliminate these extraneous waves, the melt was electrolyzed at 5 ma for periods totaling about 8 hr. After electrolysis, the diffusion current of the wave at −0.5 v was found to have decreased from about 250 μa to about 50 μa, while the wave at +1.1 v was no longer detectable. It appears from this work that electrolysis at constant current is an effective method for removing reducible metal impurities from melts of this type. It is proposed to eliminate this interference by electrolysis and then to study the effects of known reducible metals in the melt under controlled conditions. Additions of reducible species will be made by the anodic oxidation of the pure metal directly in the molten fluoride salt.

Radiation Chemistry. — When Ce^{4+}—0.8 N H_2SO_4—$NaNO_3$ solutions are subjected to Co^{60} gamma radiolysis, the $G(Ce^{3+})$ value increases with increasing $NaNO_3$ concentration. The rate of increase becomes directly proportional to the solute concentration in the region 2 to 5 M. Since the $G(Ce^{3+})$ values are in excess of those predicted by the radical and molecular product yields, they are attributed to a direct effect of the ionizing radiation on the dissolved solute.

In aqueous $NaNO_3$ solutions, pH = 4 to 5, large amounts of NO_2^- and O_2 are observed that may be attributed to a direct effect. Therefore, O^{18}-labeled water was used in aqueous $NaNO_3$ solutions to determine the origin of the molecular oxygen formed during Co^{60} radiolysis. Mass spectrographic analyses indicated that the molecular oxygen (mass 32), which can come only from the NO_3^-, is formed in direct proportion to the $NaNO_3$ concentration, suggesting the reaction $NO_3^- \rightsquigarrow NO_2^- + 1/2\ O_2$. Evidence for this reaction in the Ce^{4+}—0.8 N H_2SO_4—$NaNO_3$ system is obtained from the quantitative agreement between the molecular oxygen (mass 32) yield and the $G(Ce^{3+})$ that is directly proportional to $NaNO_3$ concentration. For each oxygen molecule, two nitrite ions are formed, each of which reduces two ceric ions. Thus it is concluded that a direct effect on the nitrate ion, directly proportional to the $NaNO_3$ concentration, takes place in agreement with the reaction $NO_3^- \rightsquigarrow NO_2^- + 1/2\ O_2$.

Chemical Physics: Neutron Diffraction. — X-ray diffraction data were obtained from liquid water and interpreted in terms of a model of the molecular arrangement. The model consists of a framework similar to that existing in normal ice, but expanded anisotropically, and contains interstitial molecules within half the cavities formed by the framework. This model conforms to the experimental density of water and reproduces the x-ray diffraction pattern with exceptionally high precision.

The crystal structure of chloral hydrate, $CCl_3CH(OH)_2$, was determined from 2142 independent neutron diffraction reflections. Bond lengths were determined within a standard error of about 0.003_5 A for bonds to hydrogen and to within about 0.002 A for other bonds. The molecule is in the staggered configuration about the C-C axis. Two kinds of hydrogen bonds, O-H···O (rather weak, 2.842 and 2.898 A), join the molecules in layers parallel to (100).

Corrosion Tests for the TRU Facility. — After 1000-hr exposure periods, no hydrogen pickup was found in tantalum exposed in the solution and to the vapor above oxygenated 10 M LiCl—0.1 M $LaCl_3$ solution at 35°C. In a similar test with Zircaloy-2 in the latter environment, a solution-exposed specimen was found to contain 120 ppm of hydrogen, compared with 65 ppm in unexposed material. No pickup occurred in the vapor-exposed specimen in the test.

An increase in corrosion rate was obtained in oxygenated 3 M HCl solution at 43°C when welded Hastelloy C specimens were thermally cycled a number of times between 843 and 427°C prior to the test. The cyclic treatment simulated an operation that would be necessary in the "glassing" of Hastelloy C process equipment. The cycled specimens exhibited an average corrosion rate of 142 mils/yr after 400 hr. Uncycled specimens corroded at an average rate of 95 mils/yr during the same time period. All specimens suffered preferential attack in heat-affected zones adjacent to welds.

Nylon (a polyamide-type plastic) underwent near-negligible dimensional and weight changes after 1000-hr periods at 80°C in 1 M mono-2-ethylhexylphenylphosphonic acid and 30% Alamine 336 chloride solutions. Hycar OR (a synthetic nitrile-type rubber with reportedly good irradiation resistance) behaved very poorly in the two solutions. Specimens underwent appreciable swelling and blistering during 100-hr tests at 80°C. In the vapor above boiling and oxygenated 6 M HCl solution, Hycar OR specimens became quite brittle during a 100-hr test.

Target Preparation. — Several materials prepared in target form for the first time are: WO_2 and WO_3 in a copper matrix, Li^6H and Li^7H in thicknesses of < 100 μg/cm^2 on 25-μg/cm^2 carbon, and various Nb-Zr superconductor alloys rolled to a thickness of 0.1 mil. Changes in the existing vacuum evaporator systems are in progress which should improve target uniformity, purity, and structural strength.

Transuranium Element Production

Transuranium Facility. — The architect-engineer design of the transuranium facility is about 75% complete and is progressing on schedule. Completion is expected by December 15, 1962. Seventy-two drawings and 38 specification sections have been received for ORNL review. Detail drawings of the intercell-conveyor housing were completed and transmitted to the architect-engineer for inclusion in the contract package. The housing to be installed by the lump-sum contractor will be provided by ORNL. The facsimile of the equipment-transfer system is progressing. The hoist and the door lock are being constructed. Drawings of the top assembly are complete and have been issued, as have the drawings of the bottom assembly. The installation and assembly drawings for the cell cubicle were completed, and component drawings for the architect-engineer design package were revised and transmitted.

Chemical Process Development. — In solvent extraction tests with short-cooled materials, it was found that short-lived Ag^{111} and Tc^{99} followed the transuranium elements in the Tramex process. Tellurium-132 appeared to partially extract, and a small fraction followed the products; Mo^{99}, I^{131}, and I^{132} were extracted but did not strip.

The effect of alpha radiation on the amine solvent used in the Tramex process was studied in a batch test with Cm^{242}. At a level of 3 w/liter a slight decrease in curium distribution coefficient, from 3.6 to 3.2, was noted at 60 whr/liter. Lanthanide distribution coefficients remained less than 0.1. After 200 whr/liter some interfacial material formed, but no difficulty in phase disengagement was noted throughout the experiment, which was terminated at 300 whr/liter. The extracted Cm^{242} was readily stripped with 9 M HCl after this extreme exposure.

Nickel from corrosion or target material follows the transplutonic elements through the Tramex process. However, tests indicated that the nickel can be separated from the product by an oxalate precipitation. In a solution that was about 0.1 M in HNO_3 and in the presence of ammonium ion, a separation factor of 250 was obtained when the transplutonic elements were precipitated as oxalates.

Process Equipment Development. — After minor modifications the conveyor automatic port closure was operated through 1000 cycles without difficulty and with little wear. The intercell pass-through was tested, and modifications were recommended. An electric impact wrench with torque-limiting socket-wrench extensions was tested and was recommended for use with manipulators in order to prevent bolt shearing or thread damage.

Four protective coatings withstood 3.4×10^9 r of exposure from a Co^{60} source while submerged in deionized water, and the recommended coating, from results to date, is the Amercoat No. 74 surface with the Fiberglas and No. 66 Seal epoxy system.

Design of Experimental Facilities. — Equipment for testing the complete Tramex process at full activity level and on a pilot scale is being installed in cell 4, Building 4507. Design of this facility is 85% complete, and the piping drawings are complete. Installation of two 200-liter storage tanks with their shielding was completed, and the interior of the cell was painted. The panel board is 90% complete, and the transmitter rack is being installed. Completion of this installation is scheduled for February 1, 1963.

HFIR Target Fabrication Development. — The HFIR irradiation targets will consist of cermet pellets of aluminum powder and transuranium-element oxides enclosed in aluminum tubes. In simulated pellets made from −100 mesh aluminum and UO_2 powders, the aluminum was the continuous phase, as confirmed by metallography. In previous tests with aluminum and Gd_2O_3 powders which had been blended in a rod mill, the oxide was the continuous phase, and thermal conductivity was only about 20% as high as that for aluminum. This was attributed to the very small particle size of the Gd_2O_3 after the milling step.

The welding conditions required for producing good welds on the end caps were: a current of 38 to 40 amp and a tube rotational speed of 9 to 10 rpm. With the plug joint design used, this gave a satisfactory 70% penetration. Difficulty was encountered, however, in tack-welding the hexagonal spacer tube that surrounds each target and mechanically aligns the individual targets in the target assembly. When three full-scale tubes were hydrostatically collapsed onto the pellets, about half the spacer-tube tack welds were sheared.

In a set of experiments to determine the optimum pressure of the tube collapse, it was found that collapse is very nearly complete at 8000 psi, but somewhat higher pressure will be needed to provide the intimate contact required for good heat transfer.

Corrosion Studies. — The Tramex process involves handling both LiCl and HCl solutions over a range of temperatures from about 40°C to the boiling point. At the higher temperatures, only tantalum and Zircaloy-2 have been found to be satisfactorily corrosion resistant. Hastelloy C can be used for applications at lower temperatures.

The possibility of hydrogen pickup in tantalum and Zircaloy-2 was examined in a number of environments in 1000-hr tests. No hydrogen pickup was detected in tantalum exposed in the solution and the vapor of a 10 M LiCl—0.1 M HCl mixture at about 135°C. Similarly, no pickup was found at 35°C in a 10 M HCl—0.0023 M $LaCl_3$ solution that contained H_2O_2. However, tantalum plate on stainless steel base metal was severely embrittled during 300 hr in oxygenated and boiling 6 M HCl solution when the base metal

was attacked through defects in the plating. A solution-exposed Zircaloy-2 specimen exhibited some evidence of hydrogen pickup after 1000 hr at 35°C in a 10 M HCl–0.0023 M $LaCl_3$ solution that contained H_2O_2. The hydrogen content of the specimen was 120 ppm, compared with 65 in the unexposed material. No hydrogen pickup occurred in the vapor-exposed specimen.

METALLURGY AND MATERIALS RESEARCH

Research and Development on Pure Materials. — Bismuth selenide exists as a single phase over a range of composition through the inclusion of substitutional point defects. The electrical properties of the crystal can be regulated by controlling the temperature and the partial pressure of selenium over the crystal. From measurements of the electrical behavior, it has been found that the crystal maintains solid-vapor equilibrium by diffusion of bismuth atoms within the solid. The dependence of the diffusion coefficient on temperature is expressed by

$$D_{Bi \rightarrow Bi_2Se_3} = 3.4 \exp\ (3 \times 10^4/RT)\ cm^2/sec\ .$$

Excess selenium atoms occupy selenium sites, but some bismuth sites are vacant. Bismuth vacancies are acceptor type.

Preparation of Pure Materials. — Spectroscopic and activation analyses of a large (278-g) crystal of LiF (98.06% Li^7) grown in a Stockbarger furnace (ORNL-3351, p 27), were completed. Cation contaminant concentrations at two locations in the crystal were measured. Results are tabulated below:

Element	Amount (ppm)	
	Bottom	Top
Ag	< 1	
Al		< 20
Fe	< 1	
Mg	< 10	< 10
Mn	2 (1.4 by neutron activation)	20
Na	5	< 50
Ti		< 20

Infrared absorption spectra of this crystal, as with earlier ones, show no OH^- contamination. The bottom portion of this crystal consists of the purest LiF known. A portion from the interior of the LiF crystal (99.99% Li^7) produced in April 1962 (ORNL-3321, p 29) was found to contain 1.5 ppm Mn by neutron activation analysis.

Investigations of systems (ORNL-3321) in which MgO exhibits an extensive primary phase field, potentially useful for crystal growth, have been extended to higher temperatures (1500°C) and to new solvents. Investigations of phase equilibria in the system $MgO-B_2O_3$, in which MgO displays high solubility, and in the system CaF_2-MgO, which provides a large temperature coefficient of solubility of MgO, have shown that these systems behave essentially as described in the literature and that both nickel and graphite can be used as crucible materials in helium atmospheres. Nickel thermal convection loops containing alkali-fluoride solvents produced the largest (6 × 3 × 2-1/2 mm) MgO crystals to date but failed after a few days on developing stress cracks. An Inconel loop, however, has been operated in excess of four weeks.

Crystal Physics. — Several iron-bearing silicate phases, not previously reported, have been synthesized. These phases were formed under supercritical conditions in an iron-lined autoclave during studies of the general system alkali hydroxide—silica—water. Analysis of the chemical and x-ray data indicates that the iron is in tetrahedral coordination with oxygen in lattice positions analogous to those occupied by aluminum in naturally occurring aluminosilicate structures.

A cesium iron silicate, analog to the natural cesium aluminum silicate, pollucite, has been assigned to the cubic space group Ia3d. An unambiguous space group assignment for pollucite has not been possible because the x-ray atomic scattering factors for aluminum and silicon are nearly the same. For iron and silicon, the scattering factors are significantly different, allowing a more detailed structural analysis. Thus by inference, pollucite belongs to the same space group. The physical density, index of refraction, and hardness were determined and were compared with those of pollucite.

CONTROLLED THERMONUCLEAR RESEARCH

Plasma Physics and Arc Research. — The study of the decay of a random deuterium plasma formed between magnetic mirrors by the pressure-gradient arc (mode II) has continued. The region between the magnetic mirrors has been enclosed by a microwave cavity which in the TM 010 mode resonates at 763 Mc without a plasma. A modulated carrier of radio-frequency energy near the fundamental frequency of the empty cavity was fed into the system, and its transmission characteristics were observed. By detecting the output and by time analyzing the amplitude of the modulation as the plasma decayed, another decay constant was measured. The decay constant, by this new method, could be observed simultaneously with the previously used plasma sweeper method. Both methods gave the same value for the decay constant for varying values of the decay constant.

The longer decay constant is presently thought to be due to a decrease in the ion cooling rate which is controlled by the temperature of the plasma electrons. Additional proof that the plasma electrons are hotter than without the cavity is indicated by the copious quantities ($\sim 10^8$/sec) of x radiation emanating from the apparatus.

DCX-1 Facility. — The previous report (ORNL-3368, p 29) described measurements of slow plasma currents to insulated end walls positioned outside the throats of the mirror coils. A qualitative explanation was proposed that involved an increase in the plasma electron heating rate coincident with the bursts of rf activity at low operating pressures. Several quantitative checks have now been completed, and results are in accord with the proposed explanation.

Cyclotron heating of electrons in the plasma region has been coupled with hot ion injection into the DCX-1. These first experiments have used about 5 w of microwave power at 21 kMc, a frequency which limited the H_2^+ injection energy to 410 kev. There was evidence for electron heating; x-ray counts increased outside the machine, and at sufficiently high pressures the heating zones glowed; however, the application of microwave power did not increase the fast-proton density by as much as a factor of 2. On order is a higher-frequency tube that will permit heating of electrons in the plasma region when operation is with the normal field value and 600 kev injection energy.

It had been previously reported that in some cases of low-pressure operation, raising the magnetic field 5 to 10% above the normal value markedly decreased the repetition rate of the low-pressure rf bursts (by as much as a factor of 10) and reduced the plasma volume (by factors of about 3). It now appears that use of the higher magnetic field changes the H_2^+ beam orbit in such a way as to increase the disorder with which protons are initially trapped. This additional disorder, then, to some extent quenches the electrostatic instability that is apparently responsible for the rf bursts and the anomalous spreading of the plasma at low pressures. A more thorough investigation of the effects of changing the H_2^+ beam orbit is now in progress.

DCX-2 Facility. — The DCX-2 has been operated with currents of injected 600-kev ions up to 40 ma. Complex plasma behavior is observed which limits the density of the energetic plasma. Upon beam turnoff, currents of neutral particles are observed emanating from some regions of the machine which reflect a trapped ion density $\sim 10^7$ to $10^8/cm^3$ with a decay mean life of $\sim$8 msec. The mean life of energetic protons in other regions of the machine must be considerably shorter with the beam on, since only very small charge exchange currents are seen from these regions after beam turnoff. Intense atomic-ion cyclotron frequency radiation and its harmonics as high as the sixth are observed. Molecular-ion cyclotron frequency radiation is much weaker.

Ion pumping consistent with the above density is observed when the pressure in the machine is raised. Cold-plasma currents flowing through the mirrors have been studied, and neither ion nor electron energies greater than a few hundred ev have been observed.

Vacuum. — Based on the study of nude-gage calibrations, a potentially satisfactory gage has been designed and is being constructed.

The test facility for initial study of diffusion-pumping mechanisms has been operated to determine if adsorption or solution of gases in the falling oil film on the wall of the pump is involved in pump operation. No pumping in the molecular flow region was observed which could be attributed to either of these mechanisms. Net gas transport was observed, as anticipated, at pressures greater than 50 μ.

BIOLOGY AND MEDICINE PROGRAM

TERRESTRIAL AND FRESH-WATER ECOLOGY

Gamma-Emitting Nuclides in Trees Bordering Waste Pit No. 5. — Significant concentrations of four gamma-emitting radionuclides have been found in pine trees collected around the newly operational Waste Pit No. 5. These nuclides were Ru^{106}, Zr^{95}-Nb^{95}, Cs^{137}, and Ce^{144}. Concentration of Ce^{144} was between 100 and 200 picocuries/g, which is the same as fallout levels. Cesium-137 concentration was uniform in all trees at 30 to 70 picocuries/g. This amount could be attributed to blowout from three open pits which are due west of Waste Pit No. 5. Ruthenium-106 concentration varied from 0.02 microcurie/g to fallout levels, which is 40 to 70 picocuries/g. Twenty-three of the seventy-three trees collected showed a concentration of Ru^{106} greater than that which could be attributed to fallout or to blowout from the open pits. This higher concentration is attributed to seepage (and subsequent uptake by the tree) from Waste Pit No. 5. Five trees showed a concentration of Zr^{95}-Nb^{95} which was greater than fallout levels and also greater than Ru^{106} levels. The highest concentration of Zr^{95}-Nb^{95} was 771 picocuries/g. Concentration of all the isotopes except Zr^{95}-Nb^{95} tended to be greater in needle material than in twig material.

The distribution of "hot" trees indicates that there are three fingers of seepage away from the center of pit 5. One finger extends approximately 110 ft to the southwest of well 7. Finger 2 extends approximately 75 ft to the northwest around well 6. The third finger extends 75 ft to the northeast around wells 2 and 3.

Estimates of total tree burden of Ru^{106} and Zr^{95}-Nb^{95} were made for the trees which showed high concentration of Ru^{106} and/or Zr^{95}-Nb^{95}. The highest tree burden of Ru^{106} was 72.37 μc; 18.37 μc was the highest tree burden of Zr^{95}-Nb^{95}. Most trees showed a burden of less than 1 μc.

Vegetation Studies Around the HPRR. — The Health Physics Research Reactor is surrounded by forest of the pine-hardwood type at distances varying from 30 to 300 ft. Preliminary estimates of reactor operation indicate that adjacent areas of vegetation will receive up to 2300 rads/yr of neutron and gamma radiation at a distance of 25 m from the source. This distance represents the edge of the forest, but scattered hardwood saplings nearer the reactor will receive up to approximately 60,000 rads/yr. Radiation-induced changes in growth rate and phenological behavior of some plant species are anticipated, particularly in the more radiosensitive conifers. Moreover, changes in the structure of the plant community may result from the differential radiosensitivity of species.

A grid reference system consisting of 10- by 10-m grids has been laid out around the reactor. Woody, perennial species in these grids, consisting of 2400 m^2, have been mapped and records taken of their size and general vigor. Dominant trees in the overstory are shortleaf and scrub pine which have stem diameters up to 11 in. at breast height. The understory is composed mainly of hardwoods up to 6 in. in diameter. Pines represent 14.6% of the total number of trees; sassafras, 32.6%; dogwood, 21.0%; persimmon, 14.5%; black gum, 5.8%; red maple, 5.1%; sourwood, 3.6%; tulip poplar, 1.4%; and sumac, 1.4%. Approximately 71% of the total basal area (61.5 ft^2/acre) in this forest is made up of pines.

Measurements of pine branch growth are being made for the current year to determine normal growth patterns in this species prior to reactor operation. October measurements indicated a terminal branch growth of 7.0 ± 0.5 cm for shortleaf pine and 7.9 ± 0.9 cm for scrub pine. Lateral branch growth was less, with 3.6 ± 0.2 cm for shortleaf and 3.3 ± 0.2 cm for scrub pine.

Use of Radioactive Tags to Evaluate Movements of Wild Mammals. — Pine mice (*Microtus pinetorum*) were subcutaneously tagged with Co^{60}, Ta^{182}, and Au^{198} wires and their subsequent movements traced with a Geiger counter in a field under consideration to be intentionally contaminated with radioisotopes for radiation ecology studies. The objectives of the tagging study were (1) to contribute to the knowledge of the ecology of pine mice and (2) to determine the optimum in-soil depth of sheet-metal walls which would prevent escape of burrowing pine mice.

The periods of most activity occurred between 7–10 PM and 2–6 AM, thus indicating both nocturnal and crepuscular activity periods. Diurnal hours were usually spent in underground nesting chambers (5 to 7 in. below soil surface) which were seldom vacated. The vertical stratification of movements was as follows: above ground, 2%; 0 to 2.9 in., 1%; 3 to 5.9 in., 30%; 6 to 8.9 in., 65%; 9 to 12 in., 2%. Besides the obvious photoperiod-activity relationship, evidence for a soil temperature-moisture influence on vertical subterranean movements was also apparent but not intensively investigated. If this field is fenced with sheet metal extending 12 in. into the soil, almost 100% retention of burrowing pine mice would be expected.

Extrapolation from Laboratory to Field in Radioisotope Accumulation by Insects. — A beetle species (*Chrysomela knabi*) was used to test accuracy in the use of laboratory-derived elimination rates to predict equilibrium concentrations of radioisotopes in insects under field conditions. Both elimination rates for Cs^{137} and feeding rates were measured in laboratory experiments, and from these data a predicted equilibrium concentration for Cs^{137} in beetles was derived. The predicted equilibrium value was found to compare satisfactorily with measurements of Cs^{137} in beetles feeding upon plants (willow leaves) growing on White Oak Lake bed.

Laboratory measurements of feeding and elimination rates were made for second- and third-instar larvae, new adults, and year-old adults. Field collections yielded numbers of only third-instar larvae sufficient for measurement of Cs^{137} content, however. The elimination constant for third-instar larvae, based on a mean biological half-life for Cs^{137} of 8.6 hr, was 1.94 $days^{-1}$. The feeding rate for third-instar larvae was estimated as: (6.5 mg of dry wt per day) (37.8 μμc per g of willow leaves) = 0.25 μμc/day. The equilibrium concentration predicted from (feeding rate/elimination rate) is thus 0.13 μμc/larva. Average whole-body concentration

in 122 third-instar larvae from White Oak Lake bed was 0.20 ± 0.04 μμc/larva, which does not differ significantly from the predicted value. This agreement confirms the validity of extrapolations from laboratory data to field estimates of feeding rates for insect populations.

Contaminated Forest. – During May 20–24, 1962, a 20 × 25 m forest plot was tagged by introducing 467 mc of Cs^{137} into 35 tree boles to study the rate and extent of transfer of chemical elements from contaminated trees into other compartments of the ecological system under natural forest influences. The greatest initial transfers of cesium from the contaminated trees were through rainfall leaching of leaves and woody tissue of the tagged trees. Rainfall collectors were placed underneath the tree canopy and around the tree stems to measure the extent of this initial transfer.

Results show that detectable amounts of activity may be removed from tagged trees through rainfall leaching within 72 hr after inoculation. Leachability of cesium from tree canopies with rainfall was found to coincide with the active growth of vegetation. Four months after inoculation, 5% (23.4 mc) of the original quantity used was leached out of the tree canopies through rainfall, and 0.06% (268 μc) was removed by stem flow.

Measurements of Litter Decomposition. – The increased use of nylon mesh bags as a device to measure the rate of litter decomposition necessitated a comparison of weight loss of confined and unconfined leaves. Unconfined leaves, whose petioles were inserted into nylon strings, were exposed on top and under the litter layer alongside confined leaves during the period March–December 1961. The weight loss of unconfined leaves was almost twice that of bagged leaves, even though the latter had a higher moisture content (by a factor of 2), which would favor growth of microbiota in the bags. These results indicate that larger organisms, such as millipedes and earthworms, which are physically excluded from the bags, play an important role in the decomposition of leaves. Average weight loss and percent moisture were, respectively, 65% and 49% for unconfined leaves on top of the litter layer and 76% and 57% for unconfined leaves on the bottom of the litter layer. These data indicate the importance of proper placement of experimental bags within the litter layer.

Strontium/Calcium Uptake by Snails. – Strontium-85 was used as a tracer to measure the effect of stable strontium and calcium on the uptake of strontium by aquatic snails under laboratory conditions. The calcium concentration in the solutions ranged from 1 to 200 ppm. Strontium concentrations depended on strontium contamination of the $CaCO_3$ used to prepare the solutions, plus the carrier strontium in the Sr^{85} solution. Very low stable-strontium concentrations were obtained by using $Ca^{40}CO_3$ (8 ppm Sr) prepared from calcium separated in a mass spectrometer. High strontium concentrations were obtained by using reagent-grade $CaCO_3$ which contained 200 ppm Sr. All quantities were converted to atom concentrations, and a multiple regression analysis was used to interpret the relative effect of calcium and strontium in solution on strontium uptake by the snails. On the basis of atom concentrations, rather than ppm or μμc, the results suggested that calcium and strontium compete for the same depositional site in the snail shell. Strontium uptake was directly proportional to the strontium concentration in solution and inversely proportional to the calcium concentration.

Radionuclides in Scales of Smallmouth Buffalo. — Scales of fish taken from White Oak Creek, when examined autoradiographically, exhibited a distinctive pattern of concentric rings because of deposited radionuclides. These rings presumably are the result of growth of the fish when they lived in areas contaminated by radioactive wastes. Gamma scans showed the presence of Cs^{137}, Co^{60}, Ru^{106}, Zn^{65}, and Zr^{95}. Scales of 1257 smallmouth buffalo (*Ictiobus bubalus*) from the Clinch and Tennessee Rivers have been surveyed to determine either gross beta or gross gamma activity. Of 136 Clinch River buffalo, 67% of the scales contained gross beta activity ranging from above background to 400 counts/min. Seventeen percent of 548 Tennessee River buffalo contained gross gamma activity, and in another sample of 571 scales, 17.3% contained gross beta activity. From these fish scales surveyed for gross activity, 342 specimens were selected for autoradiographic analysis, which is in progress. In conjunction with age and growth studies, it is anticipated that it will be possible to determine movements of fish into and out of areas contaminated with radioactive wastes on the basis of location and width of radioactive rings.

Radiation Effects on Aquatic Insects. — Larvae of *Chironomus tentans* were collected from May to December 1961 from White Oak Creek, which is contaminated with radioactive waste from ORNL. In observing the salivary-gland chromosomes for aberrations, it was found that a short inversion in the right arm of chromosome No. 1 appeared frequently. The inversion homozygote was found and 244 slides were scored. A chi-square test was used to determine if the observed three chromosomal arrangements are consistent with those expected on the basis of the Hardy-Weinberg Law:

	Standard Arrangement	Inversion Heterozygote	Inversion Homozygote
Observed	179	50	15
Expected	170.6	66.8	6.6
$p = 0.8361$	$q = 0.1639$	$X^2 = 11.98$	Probability < 0.005

This basic principle of population genetics is being tested on a natural population of organisms exposed to chronic low-level irradiation.

The significant difference found by the chi-square test indicates that the inversion is not at equilibrium in the population and that some factor is affecting the genetic equilibrium.

Evaluation of Fission Product Distribution and Movement in White Oak Drainage System. — Analyses of a series of core samples taken along transects in the bed of White Oak Lake indicate that: (1) most of the Cs^{137} in the lake bed is associated with sediments deposited during the time of the 1943–55 impoundment; (2) within the lake sediments Cs^{137} concentrations vary from 550×10^{-4} to 20×10^{-4} μc/g, with the highest concentrations generally occurring in the lower part of the core; (3) there has been little migration of the Cs^{137} through the lacustrine sediment and/or into the underlying alluvium.

RADIOLOGICAL PHYSICS, HEALTH PHYSICS, AND INSTRUMENTATION

Internal Dose Estimation. — Maximum permissible single intakes of 240 radionuclides were calculated from the data appearing in the ICRP Committee II report. For intake by ingestion, there are 110 single intake values that are in excess of 1 mg of material; the highest single intake value is 5×10^{10} μg (~110 lb). For intake by inhalation, there are 80 single intake values that are in excess of 1 mg; the highest single intake value is 2×10^{11} μg (440 lb).

Preliminary gamma-ray scintillation counting studies were performed in preparation for a forthcoming human-patient uranium-injection study. This program is to be carried out in cooperation with the Argonne Cancer Research Hospital. A collimated 4 × 4 in. NaI crystal for gamma-ray scintillation counting of a phantom which contained a 68-mg source of U^{235} located in the kidneys yielded a crude estimate of the lower limit of U^{235} detection in human kidneys. From these measurements it is estimated that for a counting time of 100 min, not less than ~100 μg of U^{235} can be detected in 300 g of human kidneys with a limit of error of ±100%.

Health Physics Research Reactor Studies. — The Health Physics Research Reactor, HPRR, has been returned to ORNL from the Nevada Test Site for installation in a permanent facility, DOSAR. Installation should be completed by the end of November 1962. During Operation BREN, the reactor was operated a total of 170 kwhr; the highest steady power level utilized was 1450 w, and the largest pulse generated was 4.3×10^{16} fissions.

NUCLEAR ENERGY CIVIL EFFECTS

Dosimetry Applications. — The major advance in the Ichiban Program was the completion of the experiments in Operation BREN. Analyses of BREN data are in progress, and most will be completed within three months. Auxiliary experiments in gamma-radiation spectroscopy will make use of the Health Physics Research Reactor and density scaling of the atmosphere. The computational program for determining the "radiation yields" of the weapons fired over Hiroshima and Nagasaki, Japan, has been resumed.

ISOTOPE DEVELOPMENT PROGRAM

Neutron Activation Analysis Research and Development: Use of a 14-Mev Neutron Generator. — Several improvements have been made to the physical installation of the 14-Mev neutron generator: increased concrete shielding to reduce the radiation leakage to the operating console and to the building exterior; changes in the beam-switching circuit to improve reliability; procurement of both a well-type NaI(Tl) crystal to increase counting efficiency of the low-energy gamma rays and an anthracene crystal for the measurement of beta spectra. An investigation of the elements of the periodic chart has been started. So far, work has been initiated with oxygen, boron, and fluorine. A number of discrepancies between reported half-lives and emitted particle energies have been observed. These discrepancies are being verified by making variations in the irradiation periods, in the use of the two radiation detectors, and in the use of separated stable isotopes. As the reactions and decay schemes are clarified,

it will be possible to determine reaction cross sections of 14-Mev neutrons and to establish the analysis sensitivity of the system. A few incidental samples originating within the laboratory such as uranium oxides, metal sulfates, and a radiolytically produced mixture of the oxide and sulfate of silver have been analyzed satisfactorily for oxygen contents of the order of several percent.

Neutron Activation Analysis Research and Development: Comprehensive Methods. — A program is under way to apply the methods of activation analysis to the determination of the greatest possible number of elemental constituents in various matrices. A tentative method has been developed which involves the activation of a sample with reactor neutrons, followed by the gamma-spectrometric measurement of the induced activities. Eleven short-lived isotopes can be determined by direct, nondestructive methods of measurements, while as many as 56 additional elements after having been separated chemically into seven resolvable groups have been determined in matrices of beryllium or aluminum.

Neutron Activation Analysis Research and Development: Identification and Comparison of Physical Evidence for Law-Enforcement Purposes. — A preliminary study has been made to determine the feasibility of utilizing the methods of radioactivation analysis in "fingerprinting" typed or written documents by identifying trace elements in writing inks, typewriter-ribbon inks, and carbon-paper inks. It is possible to differentiate typewriter print and carbon paper, but because of the large amount of sodium present in inks, no differentiation is possible without some type of separation.

Neutron Activation Analysis Research and Development: Enforcement of the Narcotic Laws. — Neutron activation analysis is being applied in criminal investigations under the federal narcotic laws, specifically on the problem of identifying the source of drugs seized in the alleged illegal traffic of such materials. A number of samples of raw opium have been received; these samples are documented as to country and district of origin and year of growth. The constancy of the uptake of trace elements as a function of time will be evaluated, as well as any patterns in similarity of the trace constituents in samples as a function of certain areas.

Radioisotope Research and Development. — The properties of technetium compounds and elemental technetium were further studied. The solid NH_4TcO_4 salt shows signs of decomposition as evidenced by color changes after storage periods of a few months. It is known that this compound is decomposed readily by heat to form volatile Tc_2O_7. A suitable, safe compound of technetium for storage and shipment is TcO_2. This salt is formed by heating NH_4TcO_4 to 225°C in a hydrogen atmosphere. Technetium metal results from the H_2 reduction if the temperature is raised to 700–800°C, but it has not been possible to obtain the metal free of O_2 by this process. Approximately 5 wt % O_2 is included in the powdered metal, which is readily dissolved in NaOH-hypochlorite, HCl, and HNO_3 solutions. In contrast, vacuum melting of the powdered metal at 2300°C removes the O_2 as TcO_2 and forms massive technetium metal which is inert to the reagents mentioned above.

A calorimeter designed to measure the heat output of Am^{241} targets after irradiation in the ORR was used to measure the Cm^{242} content of the target as a function of total neutron exposure. Calorimetric decay measurements on such targets clearly showed the heat contribution of induced fission products in addition to the Cm^{242} and confirmed the reported half-life of 162.5 days for Cm^{242}. With this instrument, it was also proved that at least 40 wt % of the Am^{241} could be converted to Cm^{242} in a thermal flux of $\sim 3 \times 10^{14}$ neutrons cm^{-2} sec^{-1}.

Radioisotope and Stable Isotope Production. — Ion currents equivalent to 4180 g of separated isotopes were monitored. Isotopes of Ca, Fe, Te, Cu, Br, and Ta were separated and collected, utilizing 28 calutrons.

Centigram to multikilogram quantities of charge materials were supplied to 24 beta and 2 alpha calutrons for the separation of the isotopes of Te, Br, Ca, Cu, Fe, and Yb. Smaller amounts of $SiCl_4$, SiS_2, Sr metal, Ce_2O_3, $PbCl_2$, and a mixture of Ta_2O_5-HfO_2 were supplied for developmental purposes.

A total of 10 kg of commercial Yb_2O_3 was dissolved and reprocessed to produce low-fired (500°C) Yb_2O_3, which is more reactive with CCl_4, the chlorinating agent being used in the calutron; 23 kg of Er_2O_3 was similarly processed.

Chemical recovery and refinement were completed on nine lots of the isotopes of Si, Zn, Gd, and Er. A total of 251 isotope receiver pockets were processed and are in various stages of refinement. Of this total, 126 were processed for mass analysis, spectrochemical analysis, or retention studies. Sputtering information was derived from striking pads contained in eight Sr^{88} and nine Cu^{65} collector pockets.

Special conversions were made on 25 lots of B, Cd, Cr, Fe, Hg, In, Li, Mo, Ni, Pb, and Sn.

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