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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

NOVEMBER 1964

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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DATE ISSUED

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OAK RIDGE NATIONAL LABORATORY
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

NOVEMBER 1964

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

REACTOR DEVELOPMENT

GAS-COOLED REACTOR PROGRAM

The Source-Jerk Experiment. — The source-jerk experiment has been used rather widely as a means of determining the negative reactivity of a subcritical multiplying assembly. In this experiment, a stable neutron population is established with a source present. Following abrupt removal of the source, the rate of decay of the neutron population is a measure of the reactivity. As customarily performed, with a single neutron detector, or with multiple signals interpreted independently, the direct results of the experiment may require substantial corrections to allow for the effects of time modes of the neutron density other than the fundamental mode, and these corrections are usually not well known.

An analysis of this experiment, in connection with the EGCR commissioning program, has shown that we can obtain a rigorous result for the reactivity (independent of any approximations to the neutron transport equation) by using multiple fission detectors appropriately placed to permit a numerical integration of the fission density over the reactor volume. In practice, satisfactory accuracy (better than 1%) can be achieved with a small number of detectors, such as four or five. (This is possible because the reactivity depends primarily on a ratio of two such volume integrals; so a systematic error in performing the integration, arising from the limited number of detectors, tends to cancel out.) In addition, the method has the virtue that the result depends very weakly on the prompt-neutron lifetime, which is often not well known for the subcritical configuration being studied.

GCR-ORR Loop No. 1 Operation. — Irradiation of experimental fuel assembly 14 continued. The fuel is a mixture of $(Th,U)O_2$ and ThO_2 sol-gel particles coated with pyrolytic carbon and is contained in two capsules, one of which is vented to the loop coolant. After two ORR cycles the estimated burnup of the $(Th,U)O_2$ particles is 1.3 at. % of heavy metal (21% of the ^{235}U consumed). The beryllium pieces we added in neighboring core positions have partially compensated for fuel burnup in the assembly, and as a result the central temperature in the vented capsule is being maintained at approximately 2500°F. Fission-gas release from the

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vented capsule appears to be steady, with R/B (ratio of release rate to birth rate) values of approximately 1×10^{-5} to 2×10^{-5} for krypton and xenon isotopes.

LITR Capsule Irradiations. — Capsule L-CP9, which was inserted in LITR position C-42 on July 28, continued to operate satisfactorily at indicated fuel central temperatures of 1720 to 1890°F. The capsule contains carbon-coated UC_2 particles in four separate compartments of ATJ graphite. We estimate that the burnup has now reached 6.4 at. % of the uranium.

ORR Poolside Irradiations. — Two capsules (03A-6 and 04-8) containing EGCR-vendor fuel materials continued to operate at design conditions. The maximum indicated cladding temperatures are 1300 and 1557°F.

Irradiation continued on sweep capsule 05-8, which contains three 6-cm-diam fueled-graphite spheres. We estimate that the burnup has now reached 3.71 at. % of the heavy metal. The indicated fuel central temperature of the top sphere is 2475°F.

AVR capsule 01A-8, which contains three 6-cm-diam spheres supplied by the AVR fuel vendor, has reached an estimated burnup of 1.8 at. % of the heavy metal. The indicated fuel central temperature of the top sphere is 2275°F. Fission-gas release rates have been steady with R/B values of around 10^{-4} for krypton and xenon isotopes.

Eight-Ball Experiment. — We are now irradiating the fifth eight-ball capsule (F1-8B-5). It contains 1-1/2-in.-diam spheres fueled with pyrolytic-carbon-coated UC_2 and $(Th,U)C_2$ particles and specimens of pyrolytic-carbon-coated B_4C. The indicated central temperature of the top sphere is now 1628°F, and the temperature of the B_4C specimens is approximately 1100°F.

BeO-Graphite Compatibility Experiment. — In support of the TARGET reactor concept, we are preparing an irradiation experiment for investigating the compatibility of BeO in contact with graphite at high temperature (1500°C). We redesigned the heater for the electrically heated mockup of the capsule following failure of the original heater in bench tests at 2250°F, and the bench test capsule is now being reassembled to obtain additional heat transfer information on the design concept at higher temperatures. Epoxy injection seals for the in-pile capsule purge-gas lines, necessitated by the need to seal the capsule before removal from the reactor, were developed and tested successfully.

Fission-Gas Release from Coated Particles. — We tested fuel particles manufactured by General Atomic (GA-315) in an instantaneous fission-gas release experiment (B9-23). These particles were made with a buffer isotropic pyrolytic-carbon coating on irregularly shaped porous uranium oxide. The average coated-particle size was 600 to 800 μ. Seven percent burnup was attained at 1400°C before we noted a large increase in fission-gas release. The fission-gas release rate was exceptionally low during most of the irradiation, with an R/B for ^{88}Kr of approximately 10^{-7}, which increased to more than 10^{-3} for ^{88}Kr at the termination of the experiment.

Postirradiation Annealing of Coated Particles. — We annealed pyrolytic-carbon-coated particles from batch OR-201 (duplex coating on UO_2) at three temperatures after having irradiated them to 4.6% burnup at 1370°C. The resulting steady-state release rates of fission products (ppm/hr) are given below:

Annealing Temperature (°C)	Steady-State Fission Product Release Rate (ppm/hr)						
	^{140}Ba	^{144}Ce	^{137}Cs	^{103}Ru	^{106}Ru	^{89}Sr	^{95}Zr
1370	2,000	≦ 35	≦ 290	≦ 5	≦ 75	4,700	≦ 6
1700	14,000	≦ 26	≦ 950	≦ 47	≦ 400	8,000	≦ 24
2000	30,000	≦ 49	≦ 1800	3200	3400	23,000	≦ 11

EGCR Control-Rod Drive Tests. — We tested the scram clutch on the EGCR prototype control-rod drive mechanism through a total of 2134 scrams. During the test, the system operated 155.7 hr in a helium atmosphere and approximately 7 hr in air, for a total of approximately 163 hr. The cycle conditions and the number of cycles are listed below:

Cycle	Number
With clutch as subassembly	20
In air prior to installation in testing facility	50
During shakedown of facility	40
Under simulated reactor conditions	2006
During shutdown of testing facility	18
Total number of cycles	2134

REACTOR FUELS AND MATERIALS

Solid Reaction Studies. — Using the anodizing and stripping technique previously described, we have determined the diffusion coefficient of ^{182}Ta in single crystals of both tantalum and niobium over wide temperature intervals. For each system the absolute value of the diffusion coefficient at any specific temperature was always approximately one-half the corresponding value for ^{95}Nb diffusion. Thus, the correlation factors for ^{182}Ta diffusion in niobium and for ^{95}Nb diffusion in tantalum are temperature independent — a fact that may simplify interpretation of data for self-diffusion in alloys of the niobium-tantalum system.

Nondestructive Test Development. — We have continued to study the feasibility of useful radiography in the presence of a high radiation background. As reported previously (ORNL-3614, pp. 5–6), accumulation of 1 or 2 r of x rays or 32 r of 1.1-Mev gamma rays was tolerated on the radiograph of an aluminum specimen without severe degradation of the image. In the second phase of the program we have studied ways to salvage film that has accumulated more non-image-forming radiation than the above. Photographic reduction using Farmer's reducer proved to be quite

useful; the silver in the image reacted with potassium ferricyanide forming silver ferrocyanide, which then dissolved in hypo. The reducing process allowed useful interpretation of film that had received 16 r of x rays or 380 r of 1.1-Mev gamma rays, an approximately tenfold improvement. Since these are accumulated doses and the primary exposure can generally be kept short, a quite high dose rate can be tolerated. For example, we x-rayed a housing containing a 5-curie source of ^{60}Co. After chemical reduction, excellent detail was observed, although the film had been in a 12,000-r/hr radiation field from the ^{60}Co. The accumulated dose was only about 200 r.

Mechanical Properties. — We are determining the effect of irradiation on the mechanical properties of alloys for fuel cladding and seeking conditions to minimize the effect of subsequent irradiation on mechanical properties. This program involves study of the interaction of radiation-induced defects with the substructure of the alloys, investigation of the effects of irradiation temperature and postirradiation test variables, and neutron dosimetry in the irradiation facilities. First, we are studying type 304 stainless steel. The stabilized stainless steels are also being investigated because of the effects of niobium and molybdenum on the stability of substructures formed by cold working and annealing.

Substructures have been developed in thin sheet specimens of types 304, 302, and 316 stainless steel by cold working and annealing. The specimens have been irradiated, and some of them have been deformed in preparation for examination with the electron microscope.

We have investigated the influence of preirradiation heat treatment on the postirradiation elevated-temperature tensile properties of type 304 stainless steel. Material with a grain size of ASTM 8-10 produced by cold working and annealing at 923°C exhibits at least double the postirradiation ductility of material annealed at normal heat-treating temperatures. An internally consistent effect of cooling rate was observed for the higher-temperature anneals.

The possibility that radiation-induced precipitation caused the low ductility at elevated temperatures in irradiated materials was investigated by irradiating and testing relatively pure copper, nickel, and iron. Since radiation-induced embrittlement occurred in these materials, precipitation effects are not necessary for impairment of ductility.

REACTOR COMPONENTS

Reactor Controls Development. — An experiment was performed to investigate the fluctuations in the current output of an ionization chamber in a radiation field whose average value is constant. These data are necessary for interpreting fluctuation data for nonconstant fields, for example, the fluctuating neutron density in a reactor. For a BF_3-filled ionization chamber and a Pu-Be neutron source, the fluctuation spectrum was constant over the range from 1 to 1000 cps, with a decrease of 6 db per octave at higher frequencies. The break frequency gives an estimate of the velocity of the BF_3 ions as a function of polarizing electric field.

For the same BF_3 chamber in a gamma field (^{60}Co), the fluctuation spectrum was not constant below 1000 cps, but increased toward lower

frequencies at approximately 1 db per octave. Furthermore, the spectrum of a fission chamber in a constant neutron field increased toward higher frequencies, being 2.5 db higher at 3000 cps than at 100 cps. It is tempting to speculate that one is seeing the effect of a correlation between the energy of an event in the detector and its time dependence; further theoretical and experimental investigations are planned.

The results show the need for extreme caution in selecting, calibrating, and using detectors for fluctuation measurements.

POWER REACTOR FUEL PROCESSING

Chloride Volatility Studies with UO_2-PuO_2 Fuel. — Scouting experiments were made on the conversion of high-density UO_2-PuO_2 fuel pellets to U_3O_8-PuO_2 powder with oxygen at 600 to 800°C and subsequent chlorination of the resulting powder with 85% Cl_2–15% CCl_4. Oxidative powdering is a desirable first step, since the unaltered UO_2-PuO_2 pellets chlorinate only very slowly. Pellets containing up to 15% PuO_2 generally oxidized in 3 hr or less at 600°C. However, pellets containing 18 to 35% PuO_2 were not always oxidized, even after exposure to oxygen for 18 to 23 hr at 800°C. Since the pellets have not yet been characterized physically, no correlation between their reactivity in oxygen and their physical properties can be made.

During chlorination of U_3O_8-PuO_2 powders at 600°C, formation of a liquid phase (possibly UO_2Cl_2) has been observed. In experiments at 500°C with a 99% U_3O_8–1% PuO_2 mixture, sintering but no melting was observed. Sublimation of both uranium and plutonium as chlorides was about twice as rapid at 500°C as at 600. About 49% of the plutonium and 97% of the uranium were sublimed as chlorides in 3 hr; about 10^4 moles of chlorine was passed through the system for each gram-atom of plutonium sublimed.

Burn-Leach Process for HTGR Fuel. — One of the potential processes for recovering the uranium from HTGR fuel includes burning the fuel in oxygen at 700 to 750°C in a fluidized bed of alumina, followed by acid leaching to recover the uranium and thorium.

In our first combustion experiment, an HTGR fuel compact (pyrolytic-carbon-coated Th-U dicarbide particles dispersed in a graphite matrix) was burned to produce a 300-g bed containing about 67% Al_2O_3, 25% ThO_2, 6% U_3O_8, and 2% carbon.

Leaching samples of this bed for 6 to 7 hr with boiling 13 M HNO_3–0.05 M HF resulted in good recoveries of uranium and thorium. When sufficient reagent was used to produce a solution 0.5 M in thorium, uranium and thorium losses to the bed were only 0.12 and 0.08% respectively. Little or no alumina dissolved. When the amount of reagent necessary to produce a 1 M thorium solution was used, uranium and thorium losses were 0.5 and 0.73% respectively. In an experiment where a sample of the bed was leached for 7 hr in sufficient boiling 13 M HNO_3 (without fluoride present) to produce a solution 1 M in thorium (if all the ThO_2 had dissolved), only 15% of the uranium and 2% of the thorium were leached. These results show the feasibility of the fluidized-bed burn-leach process for HTGR fuel, provided that fluoride-catalyzed nitric acid is used as the leachant.

Hot-Cell Studies. — Following completion of the studies on the processing of irradiated carbide fuels in cell 2, Building 4507, the highly contaminated equipment was stripped from the cell containment box, and preparations were made to remove the box from the cell. Meanwhile, a new containment box for cell 2 is being fitted with equipment for measuring fission product gas to provide data on fission-gas release during shear-leach studies scheduled for January 1965. Small-scale, batch fluidized-bed apparatus is being constructed for installation in cell 1 early next year. This equipment will be used to test the fluidized-bed process with irradiated samples of graphite-base fuels.

Shear-Leach Process. — The development and evaluation of a batch shear-leach process for recovering fissile and fertile material from spent power-reactor fuel assemblies were continued on an engineering scale with unirradiated prototype fuel.

The fabrication of equipment to demonstrate the disassembly of Consolidated Edison core B fuel by multiple withdrawal of tubes from the "egg crate" prior to shearing of the loose tubes is nearing completion. The primary components are an ejector, bumper, hydraulic cylinder, gripper head, and elevating jacks. The fuel assembly, resting on rollers, will be pushed against the ejector, thereby moving one row of tubes (13 or 14) about 3 in. out of the egg crate. The gripper head will then clamp the tubes, and the tubes will be withdrawn from the egg crate by the hydraulic cylinder. The tubes will finally be loaded into an open-top envelope for shearing. Successful shearing tests with 90 and 156 loose tubes have been accomplished.

Modifications to the shear in preparation for shearing tests with unirradiated NPR-1 fuel assemblies are nearly complete. The modifications consist in making semicircular inserts for the gags and fixed blade holder, and a circular envelope. Tests on this fuel will also include particle size distribution measurements, power requirements of the shear, and leaching studies.

The latest inspection of the shear revealed an appreciable increase in the amount of scoring on the type 420 stainless steel gibs, compared with observations made in the previous inspection. About 10,000 cuts have been made on various unirradiated prototype fuels; nearly 3000 have been made since the last inspection. It is estimated that the shear has been stroked an additional 10,000 times for various reasons, resulting in a total of about 20,000 strokes. Part of the scoring is attributed to some misalignment in the shear; that has been corrected. The gibs are being cleaned by grinding. Measurements indicated a hardness of 53 Rockwell C for the Stoody-I liners, and 47 Rockwell C for the gibs. The liners were only moderately scored.

Fluoride Volatility Processing: Aluminum-Alloy Flowsheet Development.— To obtain maximum benefits from $KF-ZrF_4-AlF_3$ equilibrium phase behavior in reprocessing aluminum-matrix fuels, we must have accurate knowledge of the composition limits described by the 600°C isotherm. The phase diagram of the $KF-ZrF_4-AlF_3$ system reporting these limits (ORNL-3594) has been used to develop the reprocessing method currently used in the ORNL pilot plant.

Standard procedures now employed during the process sequentially generate salt mixtures that display 550 and 600°C liquidus temperatures at the following compositions:

Composition (mole %)			Liquidus
KF	ZrF_4	AlF_3	Temperature (°C)
55.0	30.6	14.4	550
51.5	29.0	19.5	600
64.7	21.1	14.2	600
56.9	18.6	24.5	550
51.5	17.0	31.5	600

Because occasional difficulties have been observed in attempting to transfer liquid KF-ZrF_4-AlF_3 mixtures in the pilot plant and because selection of the optimal composition paths for process use requires that the composition region included within the 600°C isotherm be known with maximum accuracy, additional liquidus data were obtained for the key compositions listed above, using the visual polythermal method of obtaining liquid-solid transition data. For these measurements, very pure reagents (distilled KF and sublimed AlF_3 and ZrF_4) were used. The phase-transition data obtained confirmed our previous determinations of the locations of the 550 and 600°C isotherms near these compositions to within 1 and 1.5 mole % respectively. These results, as well as those from our previous observations with oxide-contaminated components, suggest strongly that the solubility of oxides in KF-ZrF_4-AlF_3 melts is quite low. A consequence of low oxide solubility is that liquidus temperatures will rise sharply if substantial amounts of contaminant oxides are present in the reagents used to prepare the starting solvent mixture. If this is indeed the case, pretreatment of the initial melts in the pilot plant to remove oxides may be necessary before liquid transfer operations are performed.

Fluoride Volatility Processing: Fluidized-Bed Method. — A mixture of 40% HF in oxygen is being considered as the decladding reagent for reactor fuel elements clad with stainless steel (ORNL-3614) and also for elements clad with Zircaloy. Design of a proposed pilot plant to demonstrate a fluidized-bed fluoride-volatility process for low-enrichment UO_2 fuels clad in stainless steel or Zircaloy has begun. The facility is being planned for Building 3019. Development work for the facility is being done at ANL, BNL, ORGDP, and ORNL, and the design will be accomplished by ORNL and ORGDP.

Laboratory studies showed that the HF-catalyzed destructive oxidation method for decladding is not equally effective for different types of stainless steel. For type 347 stainless steel, attack rates of 16.7, 14.6, and 16.4 mils/hr were measured with 200 ml/min of 40% HF in O_2 at 600, 650, and 675°C respectively. For type 304 stainless, the rates

were much higher, and a large inverse temperature effect was observed. Rates of 40.7, 39.5, 34.3, and 20.0 mils/hr were obtained, respectively, at 600, 625, 650, and 675°C under the same conditions as above. A similar temperature effect was seen with type 348, where rates of less than 0.1, 4.5, and 0.6 mils/hr were measured at 575, 600, and 650°C. The data clearly indicated that the rate of attack as well as the temperature coefficient is dependent on the metal composition.

Boat hydrofluorination tests with type 304 stainless steel oxide material prepared at 600°C with 40% HF in O_2 demonstrated that a slow conversion to the fluorides occurs. The conversion, however, was much slower than in the case of treatment with fluorine, and not much of the chromium was volatilized. The tests consisted in dividing one batch of oxide into three portions and hydrofluorinating two of these, respectively, for 1.5 and 3 hr with 40% HF in O_2 at 600°C. Analysis showed fluoride contents of 3.8, 5.9, and 8.9% for the original and the 1.5- and 3-hr samples, respectively, while the corresponding chromium analyses were 13.3, 12.4, and 12.1%. As was suspected from thermodynamic calculations, the oxide-hydrofluorination reaction is unfavorable in terms of the equilibrium constant, and the reaction proceeds only by constant removal of the water formed.

The oxide mixture generated by the reaction of stainless steel with 40% HF in O_2 was almost totally converted to fluoride by treatment with fluorine at 600°C for 2 hr. This was illustrated in one test with the oxides of type 347 stainless steel, where the fluoride analyses before and after treatment were, respectively, 3.3 and 49.8%. Much of the chromium was volatilized during the treatment. The original material had a chromium content of 26.9%, based on the iron content, while the final material, after fluorination, had only 15.9% chromium.

Tests were made with Zircaloy-2 oxide to show that the conversion to fluoride with the 40% HF–O_2 reagent depends on the length of treatment. Fluoride contents of 27.1, 35.2, and 35.5% were measured, respectively, after 1-, 2-, and 3-hr treatments at 600°C. The original "oxide" contained only 7.73% fluoride.

Fluoride Volatility Processing: Sorption Rate and Capacity Studies. — The conditions affecting the rate of sorption and the total capacity of NaF pellets when used for collecting UF_6 have been studied to provide design information for sorber beds.

Strikingly superior characteristics have been noted in some experimental NaF pellets prepared at Paducah (batches 6 and 8). Each of the materials was tested through three sorption-desorption cycles in the gasometric system used previously with standard Harshaw pellets (ORNL-3718, p. 9). The Paducah pellets excelled in lack of physical deterioration and in capacity for UF_6.

During the tests, the Paducah pellets showed little evidence of powdering and disintegrating, as compared with Harshaw pellets. During subsequent handling, a small amount of powdered NaF was observed, principally from the pellets of batch 8. These pellets had a more uniform capacity with cycling than the pellets from batch 6 did, but both were much better than the Harshaw pellets, as is shown by the following data:

Pellets	Moles of UF_6 Sorbed per Mole of NaF		
	Cycle 1	Cycle 2	Cycle 3
Harshaw	0.14	0.05	0.05
Batch 6	0.15	0.13	0.10
Batch 8	0.12	0.11	0.11

Fluoride Volatility Processing: Fluoride Volatility Pilot Plant. — The experimental work is designed to (1) gather data on the processing of aluminum-uranium alloy fuel elements and (2) study the volatility processing of even shorter-cooled fuels than were available during the zirconium program. Earlier runs demonstrated that application of the process to aluminum is possible; we are now working toward the second objective above.

The FVPP exceeded its earlier maximum radioactivity level, which was reached with the processing of uranium-zirconium alloy fuel elements cooled six months. One run was successfully completed in which spent ORR fuel elements cooled six months were processed. A higher-radioactivity run now in progress (RA-3) is almost complete; spent ORR fuel elements are being processed 80 days after discharge from the reactor.

There have been no major difficulties. In the first two runs, the fuel elements partially melted before they dissolved completely. But in run RA-3, a slight change in barren-salt composition permitted the use of a lower temperature for dissolving (575 vs 600°C); melting was avoided, resulting in the best dissolving operation thus far in the aluminum program.

Activity releases to the atmosphere have been undetectable except in the off-gas system. Prior to and during the dissolving process, rare gases are released to the stack. Also, during fluorination, additional radioactivity (primarily due to tellurium) is released; preliminary estimates indicate that this amounts to only a fraction of a curie. In addition to tellurium, the off-gas from RA-3 contained some ruthenium and a trace of ^{131}I.

Fluoride Volatility Processing: Corrosion. — A letter report (BMI-X-312) was received that describes studies performed at Battelle Memorial Institute to determine the optimum conditions under which cathodic protection could be applied to reduce corrosion in molten fluoride salt systems in the presence of HF. One of the most corrosive systems encountered in earlier studies, 52-37-11 mole % $NaF-LiF-ZrF_4$, was used.

The study demonstrated that cathodic protection would reduce corrosion of submerged INOR-8 components in fused salt systems. Negligible protection was provided, however, at the interface areas at practical current densities (less than 10 amp/ft^2). In view of this, the technique appears to have severely limited application.

The results also indicated that some cathodic protection would be provided to the container while it is in contact with the Zircaloy fuel element during dissolution, since the Zircaloy is so strongly negative with respect to INOR-8 or Hastelloy B.

As previously reported (ORNL-3680, p. 9), the corrosivity of two slightly different methods for processing Al-U alloy fuels was determined on an engineering scale. Test bars of INOR-8 and alloy 79-4 were exposed to both the hydrofluorination (dissolution by reaction with HF) and fluorination steps in the same vessel. A letter report (BMI-X-315) was received that summarizes the detailed metallographic examinations made of the test bars after exposure by Battelle Memorial Institute. Exposure conditions and results of measurement by micrometry are also included.

Presented in another section of the report are results of a similar examination of three INOR-8 rods exposed to a laboratory-scale simulated hydrofluorination step for the recovery processes for aluminum- and zirconium-based fuel.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — During this period, we heated all salt piping, the reactor, the heat exchanger, and the drain tanks to 1200°F to determine the adequacy of the provisions for preheating and to remove moisture from the salt systems.

During the check-out of the 50 removable heater-insulation boxes in the reactor and drain tank cells, we found that four or five in the drain tank cell would be difficult or impossible to replace remotely unless modifications are made to give better clearances. About half of the heater boxes required minor modifications to eliminate potential short circuits in bead-insulated power leads.

The heating and purging of the fuel system in the reactor cell required ten days. During this time we evacuated the fuel system twice, at 500 and at 800°F, after moisture in the helium purge exceeded 1000 ppm. The moisture content of the helium was reduced to less than 20 ppm before the temperature reached 1200°F. The fuel and coolant circulating pumps were used during this period to circulate helium to assist in removing moisture from the loops.

The heating of the salt systems showed that the heaters are capable, except for a few places on the large circulating lines, of maintaining the vessels and piping at 1200°F while operating at approximately half their rated capacity.

We completed the check-out and repair of heaters in the drain tank cell while the reactor cell was being heated. We then heated the tanks and piping in this cell while purging with helium from the fuel circulating loop, which was already dry and hot.

After steady-state temperatures and heater settings were determined, the salt systems were cooled down. Following inspection and minor work, the drain tank cell was reheated in preparation for charging flush salt. While the fuel and coolant loops are cool, we are inspecting them and improving the insulation and/or heating in the problem areas.

Installation of the charcoal beds and the walls and doors of the high-bay area were completed. Work continued on the vapor-condensing system and the fuel reprocessing system.

Metallurgy. – Studies were conducted on a procedure for brazing graphite to INOR-8, a material combination of interest in molten-salt systems. Included in this work were (1) an evaluation of promising braze materials that are compatible with fluoride salts and wet both graphite and metal surfaces, and (2) an evaluation of several materials with suitable thermal expansion coefficients for use as transition pieces. The transition sections are needed to accommodate stresses in the joint caused by the large difference between the expansion coefficients of graphite and INOR-8. Molybdenum, niobium, tantalum, and tungsten were evaluated as transition materials.

Test results indicated that metal combinations with wide differences in coefficient of expansion could be brazed successfully if the brazing alloy used were ductile enough to accommodate the strain imposed. Molybdenum-to-INOR-8 and tungsten-to-INOR-8 combinations were soundly joined using gold-nickel and copper braze alloys. In graphite-to-metal joints, the ductility of the alloy made little difference, and the limiting factor in obtaining a sound joint was the difference in coefficient of expansion between graphite and the metal. Thus, the molybdenum-to-graphite and the tungsten-to-graphite combinations resulted in crack-free joints, whereas much cracking occurred in joints between graphite and niobium or tantalum, whose coefficient is higher.

Chemical Development: Phase Relationships of MSRE Coolant and Fuel Salts. – New determinations of phase transition temperatures over a short composition range of the $LiF-BeF_2$ system, including the composition of the MSRE coolant, were made to a precision of 0.3°C by the interpretation of the temperature-time curves obtained from slowly cooled melts. The peritectic temperature was found to be 457.7°C at a composition between 65.5 and 66.0 mole % LiF. A sample of MSRE coolant, taken from one of the batches loaded into the reactor, showed a phase transition temperature of 457.6°C. This indicated that the material contained at least 65.5 mole % LiF and thus confirmed the composition indicated by the weights of the materials used in its preparation.

A three-dimensional model of the $LiF-BeF_2-ZrF_4$ phase diagram was constructed as a means of affording a simple graphic display of the crystallization behavior of the MSRE fuel and coolant salts. The model is now on display at the MSRE site. Except for the crystallization reaction in which the uranium phase $7LiF \cdot 6UF_4$ is produced, all equilibrium crystallization reactions of the fuel and coolant salts are evident from the model.

Fractional crystallization of the MSRE fuel in the dump tanks could be expected to introduce local heating problems if the concentration of uranium fluoride in the freezing liquids increased sharply. Very slow cooling, such as might occur in the MSRE dump tanks, should enhance liquid-solid phase separation during freezing. An experiment was conducted to reproduce on a laboratory scale the conditions under which the MSRE fuel might be expected to freeze in the dump tanks. Reactor Division values for estimated cooling rates in the interbayonet salt volumes were used. Some 565 g of liquid salt was frozen by cooling from 467 to 339°C at a rate of 0.387°C/hr. The frozen ingot was sectioned vertically and polished. Upon petrographic examination, minute crystals of $7LiF \cdot 6UF_4$ were found to be distributed throughout the frozen ingot; no regions of gross uranium concentration gradients were observed by visual examination.

Chemical Development: Iodine Release from Spilled Salt. – A fourth experiment was performed in the series directed toward establishing the factors which are important in affecting iodine release when molten fuel is suddenly exposed to a large volume of water, as would be the case in one of the postulated accidents to the MSRE. A 10.87-g charge of the MSRE fuel mixture was melted with 11.3 wt % of NaI and discharged into 500 ml of 1% H_2O_2 solution; the hydrogen peroxide was included because it would probably be present in water exposed to the intense radiation field likely to occur during an accident. A stream of helium was passed through the hydrogen peroxide solution continuously and, in turn, into a solution of sodium thiosulfate. Reactions of each of these two solutions were easily observed immediately. Iodide ion was oxidized to molecular iodine rapidly in the H_2O_2 solution and swept out with the helium stream, reacting subsequently with sodium thiosulfate. Chemical analysis showed that no detectable iodine (less than 0.07 %) remained in the undissolved salt.

Preparation of MSRE Materials. – Approximately 5755 lb of the mixture $LiF-BeF_2$ (66-34 mole %) was transferred from 22 salt storage containers to the coolant drain tank in the MSRE. All fill operations were accomplished in a routine manner and without the incurrence of any detectable beryllium contamination of the reactor facility. Subsequent examination of the in-line sintered-nickel filters showed the presence of cracks in the sintered nickel. These are attributed to excessive thermal gradients in the small furnace used for heating the filters at the reactor transfer site. Cracks in filter elements had never been observed previously in the production facility, and the coolant salt had all been filtered during its production. More uniform heating of the filter element has been arranged for the flush-salt loading operation.

Production of the fluoride solvent base $LiF-BeF_2-ZrF_4$ (64.7-30.1-5.2 mole %) for the MSRE fuel was begun. Final approval has been received for the preparation of the highly enriched $^{235}UF_4-^{7}LiF$ MSRE concentrate, and the necessary radiation alarm system has been installed in Building 9201-3 (Y-12).

Radiation Effects. – A completed digest of analyses of gas samples obtained during the last in-pile experiment showed that the rate of production of CF_4, which was too low to detect, was less than 0.1% of the rate of production of xenon. This is lower by a factor of a thousand than the rate at which CF_4 production in the MSRE would become significant.

Graphite that was submerged in MSRE fuel during the in-pile exposure has been analyzed for fuel constituents; only traces were found, and the amount of uranium was about that expected from the extent of contamination with fuel salt. Such results were in marked contrast with those from earlier experiments in which the fuel had been allowed to freeze and to cool to room temperature, and hence to evolve fluorine gas, during reactor shutdowns. In this experiment, heaters kept the fuel molten, and uranium concentrations in the graphite, generally of the order of 10 ppm as analyzed by a method involving the counting of delayed neutrons, were lower by a factor of 100 than that encountered in earlier experiments and are insignificant.

MSR Processing. — Design of the MSRE fuel processing system, including instrumentation, was completed. Installation of process piping in the fuel processing cell was completed, and the electrical and sheet metal work was started. Construction of the gas supply station was completed except for some instrumentation. Fabrication of the instrument control panels was started. The fuel storage tank salt sampler flowsheet was issued for comment, and preparations are being made to modify the fuel sampler-enricher mockup for this purpose.

Analysis of Radioactive MSRE Fuels. — The equipment to be used for handling and analyzing the MSRE salt samples is being set up in Building 2026 and will be ready for use on radioactive MSRE salt samples by the last week of November.

Essentially all the personnel to be responsible for analyzing the MSRE salt samples have been trained on the methods.

An alternative method for the determination of oxide that may be applied to the MSRE fuel is under investigation. Briefly, the fluoride salt sample (approximately 400 g) is heated to 600°C under an inert atmosphere. A carrier gas — H_2 containing about 0.01 atm of anhydrous HF prepared by controlled mixing — is introduced into the melt, then passed through a precision wet test meter to keep a running total of the gas volume that passes through the reaction vessel during the experiment. The oxides in the salt react with the HF to give an equilibrium concentration of water, which is determined by successive Karl Fischer titrations of the off-gas. The HF concentration in the off-gas is monitored by successive KOH titrations. In each series of titrations the flow rates are measured by means of a Bubble-O-Meter. The oxide in the melt, therefore, is directly related to the partial pressures of HF and H_2O determined as a function of the volume of influent gas which passed through the melt during the experiment. To date, only preliminary tests on Li_2BeF_4 salts have been made; the effect of zirconium and uranium in the melt will be investigated.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Metallurgy. — Work with the fuel-element manufacturer is proceeding. The last large item of equipment, the automatic welding machine, has now been received and placed in operation. Some differences have been noted in the enriched oxide received from the present vendor when compared with the oxide used in the development and that received from a second vendor. What effect, if any, this will have on the fuel element has not yet been determined. It does, however, affect the homogeneity scanner and will require new standards.

Physics. — Recent critical experiments associated with the determination of the Eu_2O_3-Ta-Al control-rod worth have been completed. Three sets of experiments were conducted. In the first set the circumferential aluminum gaps between the poison quadrants in the inner control rod (shim-regulating rod) were purposely not aligned azimuthally with the corresponding aluminum-water gaps in the outer rod (four shim-safety rods

constituting a cylinder). This procedure was consistent with all previous control-rod calibrations. Recently, however, there have been strong indications that mechanical considerations will require that the gaps be aligned. Therefore, a second set of experiments was conducted to determine the reduction in shutdown margin associated with the aligned gaps.

After completion of the above experiments, one full-length quadrant in the outer control rod was replaced with an all-aluminum quadrant. This quadrant was then equipped with a variable-length, 0.025-in.-thick cadmium cover and was used in the third set of experiments to simulate the case of one stuck or removed safety rod. These experiments were conducted with the control-rod gaps aligned.

Results from the second and third sets of experiments are shown in the table as applied specifically to the particular reactor cases of interest.

Case	Conditions	Shutdown Margin (dollars)	
		HFIRCE-3[a]	1st Production Core[b]
I	All rods in, NT + V[c]	7.6	>7.6
II	Four safeties in, regulating rod out, T + V	5.3	≅5.3
III	3/4 scram from clean critical, T + V	3.4	>3.4
IV	3/4 scram from clean critical, one safety out, T + NV	2.4	>2.4
V	Four safeties in, regulating rod out, NT + V	3.8	≅3.8

[a]Core loading: HFIRCE-3 fuel elements in HFIRCE-3 facility.

[b]Core loading: fuel elements being fabricated by ORNL, installed in HFIR facility.

[c]NT + V refers to "no target but optimum void in island."

Case III was simulated by covering the Eu_2O_3 "region" and all but 2 in. of the Ta "region" of the outer rod aluminum quadrant with 0.025 in. of cadmium. Because of the relatively high energy of the return neutron current from the reflector, and because of the above windows in the Ta region, it is believed that in the actual case, where Eu_2O_3 and Ta completely cover the octant partially covered by cadmium in the experiment, the shutdown margin will be considerably greater. Experimental results show that with four safeties fully inserted, and with the regulating rod in the symmetrical, clean, critical position, the shutdown margin was 6.5 dollars for case a.

Feasibility studies are being made in connection with power distribution measurements at various points in the fuel cycle. The method being investigated at present involves the measurement of the concentration of a short-half-life fission product in a small punching taken from a removable fuel plate, after operation at a reduced power level (about 50 Mw). Several fission products having half-lives in the range of 5 to 17 hr are being considered. At present, ^{91}Sr and ^{97}Zr appear to be most promising from the standpoint of chemical separation and decay gamma spectrum.

Design and Analysis. – The heat transfer analysis of the fuel element is being expanded to include operation at low power with the pressure vessel open. Under this condition and with 10% of full flow the incipient boiling power level is calculated to be more than a factor of 4 higher than the maximum proposed unpressurized operating power level of 2-1/2 Mw.

Essentially all of the core component and core structural drawings are certified for construction. The drawings of the control-rod inspection and assembly fixtures and the control-rod track assembly fixture were completed.

Instrumentation and Controls. – The design of the instrumentation for the HFIR cladding failure detector, which will detect cladding failures of approximately 1/8 in. diameter or larger, is essentially completed. It incorporates a BF_3 neutron counter, preamplifier, pulse amplifier, and log count rate meter to measure the delayed neutron activity. Details of the preamplifier still remain to be established. Many standard reactor control modules were used in the design. This has minimized engineering time and will result in faster fabrication and assembly. All shop work and drafting has been scheduled, and the procurement of all special components has been completed.

A faulty fuel element detector system has been designed to detect gross releases of fission products into the primary coolant system of the HFIR. One channel of the system has been assembled and is being tested in the ORR. It consists of an ion chamber, a flux amplifier, and a fast trip comparator. The ion chamber is equipped with a rotatable lead shield to permit testing during operation. In the HFIR, three channels will be placed in coincidence and will operate through the reactor safety system. The ion chambers will monitor the exit water line from the reactor at its point of entry into the pipe tunnel approximately 2 sec downstream from the core. At this point, a doubling of the background count will be produced by a 1% meltdown in which the fission products are evenly distributed throughout the primary water system.

Component Development. – Flow tests in the control region quadrant mockup indicate that at the minimum resistance position of the control rods the flow rate through the control region will be about 1112 gpm at the required pressure drop of 7 psi. The maximum deflection measured for the above case on the outer control rod at a location 12 in. above the core midplane was about 35 mils. Based on deflection experiments conducted on this rod prior to its installation in the mockup, the 35-mil deflection corresponds to a load on the rod of 104 lb.

The corrosion test program for reactor component materials has been completed, and a report outlining the results of this program is now being prepared.

Reports. – The Safety Analysis has been reviewed by ORNL management and by ORO and DRD personnel. Minor changes will appear in the final version.

The meteorological radioactivity considerations are being reevaluated and have been programmed for the computer.

THORIUM UTILIZATION PROGRAM

Irradiation of Thorium Oxide Fuels. – Fission-gas samples from the two vibratorily compacted MTR-1 capsules, the four vibratorily compacted ETR-1 rods, and the high-burnup-pellet rod were collected and analyzed. One of the MTR-1 capsules contained arc-fused thoria-urania, and the other contained sol-gel thoria-urania. Both had been irradiated to 75,000 Mwd/metric ton at a linear heat rating of about 45,000 Btu hr^{-1} ft^{-1}. The ETR-1 rods had been irradiated to about 22,000 Mwd/metric ton at a linear heat rating of 100,000 Btu hr^{-1} ft^{-1}. These four capsules contained sol-gel thoria-urania that had been exposed to different calcining atmospheres in three cases, and contained powder of a different particle-size distribution in the fourth. Although precise percentages of gas release during irradiation cannot be assigned until the burnup analyses have been completed, the relative releases from the MTR-1 capsules at approximately 75,000 Mwd per metric ton of metal appear to be roughly the same as those at 40,000; about 4% of the fission gas was released from the arc-fused material and 13% from the sol-gel material.

Preliminary examinations of the as-sectioned portions of the ETR-1 group revealed central voids varying in length from about 1 in. at the peak burnup end to almost the full 11-in. capsule length. Although final evaluation will have to wait for detailed metallography and burnup analyses, it appears that the linear heat rating of 100,000 Btu hr^{-1} ft^{-1} is marginal for central void formation. These samples will be used to help define the temperature ranges for columnar grain growth, sintering, etc.

Additional gamma-spectrometric examinations were made on a high-burnup-pellet rod that had revealed anomalous peaks in the gamma scan. Cesium concentrations near the longitudinal center of two of the 1/2-in.-long pellets at one end were the source of these anomalous peaks. Concurrent ^{60}Co activities from the stainless steel cladding indicate an essentially uniform nvt along the entire 3-1/2-in. length of fuel. Cursory examinations of the sections removed for metallography and burnup showed circumferential as well as radial cracking. Metallography of longitudinal sections may reveal the causes for these cesium concentrations.

Thorium-Uranium Recycle Facility (TURF). – As of October 29, 1964, the Title II design was almost 86% complete. The design features of the remote crane and manipulator system, which affect the building design, were completed by Programmed and Remote Systems Corporation. The initial mockup tests on the remote installation and removal concept for the shielding-window alpha seal indicated that minor modifications in design are required to ensure proper remote alignment and to reduce handling difficulties. These modifications are almost complete, and the second phase of testing will be started immediately in an effort to release the

final design and procurement specifications as soon as possible. The specifications have been revised to permit the separate purchase of the window forms, alpha-seal assemblies, and bulk shielding windows.

Mockup studies for the TURF hot cells will be done on the second floor of Building 4501. Equipment to simulate these cells has been installed, and experimental tests were started.

Title II design of the TURF sol dryer is 90% complete.

A geometrically safe slab tank is again being considered as a storage vessel for ^{233}U solutions. The choice between the use of borated steel plate (fixed-poison) and a geometrically safe design for nuclear safety will be based on economic factors.

Title II design of the remaining sol-gel process equipment is being delayed, pending the establishment of final chemical and equipment flowsheets.

The design of the oxide fabrication equipment is about 18% complete. Design effort on the powder preparation equipment was concentrated on the disk crusher and spiral elevators. The detailed mechanical design of the vibratory compactor was completed and approved and is being estimated. A welding-machine electrode holder is being designed. A flow diagram, piping layouts, purchase specifications, and design of mechanical components are being prepared for the fuel-rod cleaner. A preliminary design for a density scanner has been adopted; it moves a tube past a fixed gamma source and in so doing runs the long fuel tube into the path of the cell crane and manipulator systems. Interlocks will be provided to avoid damage to fuel tubes.

Permeability of Vibratorily Compacted Fuels. — Last month it was reported that the maximum resistance to water-vapor flow was at the end of the test elements. This cast doubt on the value of the permeability measurements. It now appears that the resistance at the ends of the element was due to the method of element preparation. Fine material at each end formed during packing, resulting in a short region of low permeability at the upper end, and partial plugging at the lower end of the retaining plate, which had 20-μ holes. The plugged filter plate was in effect eliminated by inserting a capillary tube into an unused tap just above the filter plate, and using this tube as the simulated cladding defect.

Several experiments were made at various heat inputs, and qualitatively the results were similar to those previously reported. Peak internal pressure always occurred before reaching the critical temperature of water, regardless of the rate of temperature rise. In an extreme case, total pressure within the sample rose to 2800 psi at an observed rate of temperature rise of 70°C/sec on the cladding and 40°C/sec in the fuel. The average rate of temperature rise in the element was between these values.

Coated-Particle Development Laboratory. — Modifications to room 241, Building 4508, to make it into a coated-particle development laboratory are complete. The building-modification directives were changed to include the construction of equipment enclosures, which are being procured by the H. K. Ferguson Company.

Pellet Fabrication. — The effect of the calcining and sintering temperatures on thoria, as well as the effect of the environment used in

these operations, was investigated. It was found that calcining and sintering in hydrogen caused the bodies to fracture, whereas this did not occur in air. An upper limit of 650°C on calcining before pressing and sintering the pellet produced the highest density. No significant difference in the temperature range 400 to 650°C was noted.

Fuel-Rod Homogeneity Scanner. — The fuel-rod homogeneity scanner was received. Its operation is based on measurement of absorption of ^{60}Co or ^{137}Cs gamma rays. The electrical wiring has been finished, and preliminary testing indicates that the system is ready for use.

The drive unit that moves the rods under the gamma scanner consists of a Slo-Syn motor coupled to a rack and pinion. Longitudinal speeds of 2, 4, 8, 12, 16, and 24 in./min are available by simply changing the pinion gears.

The irradiation sources are 2 curies of ^{60}Co and 5 curies of ^{137}Cs, housed in a single container with about 6 in. of lead between the sources.

Safety shields are being made from Plexiglas and will be installed on the scanner to prevent accidental exposure of the operator's hands to the radiation.

Fission-Gas-Release Studies on Sol-Gel ThO_2-UO_2. — Work on the high-vacuum apparatus for determining fission-gas release from sol-gel-prepared fuels was completed, and 12 samples (0.500 g each) were prepared for irradiation. Samples were divided into four groups according to their mesh size; the ranges were −10 +16, −35 +50, −70 +100, and −140 +170. The material contains 0.0466 g of ^{235}U per gram of sol-gel material.

Three samples of different mesh sizes have been irradiated in the LITR. Fission-gas release measurements were made on only one of the samples. The others cannot be examined at this time due to their high gamma radiation. About a week will be needed before the samples will decay to a safe level.

A fractional release of 524 ppm of ^{133}Xe has been calculated from the results of a puncture test on sol-gel sample B1-10-16. A total release of 8130.5 ppm was found after heating the sample at 1300°C for 12 hr.

Fueled-Graphite Equipment Development. — Effort in this program has centered about the key problem of developing satisfactory pyrolytic-carbon-coating equipment. As the size of microspheres increases beyond 400 μ, the cost of coating by fluidized-bed techniques becomes very expensive. Another approach to the conventional fluidized-bed technique would be to coat the microspheres in a rotating drum. This has been studied by a number of investigators and abandoned; however, we decided to reexamine the problem in view of the unusually large particle sizes in which we are interested. Tests with glass models and a mathematical analysis revealed several important problems that must be solved before the concept becomes feasible.

Gas Evolution from Sol-Gel Uranium-Thorium Oxide Fuels. — The adsorbability on ThO_2–3% UO_2 of some of the gases normally evolved upon heating thoria prepared by the sol-gel process was studied under controlled conditions. Whereas N_2 and O_2 showed very little tendency to adsorb on the oxide, CO_2 could be reversibly adsorbed between room temperature and 970°C.

Process Demonstration and ^{233}U Distribution. — The Oak Ridge National Laboratory serves as a national storage and distribution point for ^{233}U. Title II design of an expansion and improvement of the storage facilities is nearing completion.

The new facility will permit the separate storage of solutions containing 400 kg of ^{233}U with various ^{232}U contamination levels. The present single storage vessel has a 60-kg ^{233}U capacity. Five storage tanks, designed to be critically safe, will be installed in a doubly contained section of the Building 3019 (Pilot Plant) pipe tunnel.

In addition, a small-scale cleanup facility will be provided to decontaminate ^{233}U nitrate solutions from ^{232}U decay products.

Engineering Development Studies. — Procedures and equipment are being developed to adapt the sol-gel processes to completely remote operation as required for the TURF. The process for using 2-ethylhexanol as a drying solvent to convert sols into 50- to 1000-μ-diam microspheres of oxides has been developed to permit continuous operation with a variety of sol compositions. Continuous operation at a capacity of over 1 kg of ThO_2 microspheres per day was demonstrated, and kilogram quantities of samples were prepared. Installation of a TURF cell mockup facility and installation and testing of a 3-in.-ID vertical-tube calcining furnace are in progress.

Sol-Gel Microsphere Preparation. — The emphasis for these studies has been on developments necessary for the preparation of kilogram-size special samples of microspheres. Continuous operation of the microsphere column at 1.3 and 2.5 cc/min of sol was further demonstrated. The higher rate corresponds to a rate of ThO_2 production of about 1.25 kg/day, assuming a 4 M thoria sol. The column was operated with several sols other than pure thoria. These tests established the conditions necessary for preparing gel spheres from such sols and gave samples for investigating the drying and calcination conditions required for these compositions. Apparatus for drying the microspheres was installed. A yield of about 85 wt % in a 125- to 175-μ range was obtained for the longest sample-preparation run.

There is a slow accumulation of nitrate ion in the 2-ethylhexanol drying solvent during extended periods of column operation. For some sol compositions, a clustering of microspheres occurs after the nitrate concentration builds up in the 2-ethylhexanol. The clustering can be eliminated by adding fresh 2-ethylhexanol. An anion exchange column was installed in the side-stream distillation system now being used to remove water from the 2-ethylhexanol. This column will be evaluated to determine its effectiveness in removing the nitrate ion from the solvent.

Vertical-Tube Furnace. — The 3-in.-ID vertical-tube calciner was operated with about 1.4 kg/hr of ThO_2–4% UO_2 gel feed to prepare products that were (1) calcined at 1150°C and cooled in Ar–4% H_2, (2) calcined at 1150°C in Ar–4% H_2 and cooled in argon, and (3) calcined at 1150°C

in argon and cooled in argon. The O/U ratios in the calcined products were about 2.015 for the first two conditions, and 2.09 for the third. Mechanical operation was excellent. Other analyses such as gas releases from the calcined product are not yet available. Preparations were completed for the second major period of test operation with gel feed. The prototype of the feeder proposed for remote operation was installed. The experiments during November were aimed at testing this feeder, determining the effects of fines on the furnace and furnace operation, and providing about 50 kg each of products prepared under argon and with Ar–4% H_2 atmospheres.

Sol Encapsulation Studies. – This project was completed, and a final summary report was received [Encapsulation of Sols to Produce Rounded Particles, Final Summary Report for ORNL Subcontract No. 2326, by E. C. Martin (Southwest Research Institute, San Antonio, Texas), October 21, 1964]. One objective of this project was to use the encapsulation of sols in a shell of organic material as an initial step for preparing microspheres. Several polymer systems were developed for the encapsulation of a thoria sol or a thoria-urania sol. In general, the polymers selected have a high water permeability. These polymers were selected to evaluate the feasibility of gelling the sol by diffusion of the water through the shell. In all instances the shells collapsed into misshapen "pods" as water left the sol droplets, decreasing their diameter inside the shells. It is thought that the rate of water diffusion through the shell was greater than the diffusion of air through the shell, creating a void inside the shell, causing it to collapse. This was augmented by the fact that the shell materials are not elastomers and therefore will not contract as the volume of the sol decreases. Suspending the capsules in a liquid that will cause the shell to swell and also extract the water appeared to show some promise; however, no satisfactory system was developed. In several instances the capsules were suspended in liquids that diffused through the shell, extracted the water, and gelled the sol. This approach caused the capsules to deform; in some cases the sol crystallized.

Another objective of the project was to prepare stable, low-viscosity dispersions of carbon blacks in water and in sols. Several nonionic surfactants such as Renex 30 were used with an ultrasonic disperser (Bronson Sonifier) to give stable aqueous dispersions that contained about 100 g of carbon per liter. Such dispersions were possible both in water and in 2 M ThO_2–4% UO_2 sols. A low-viscosity 100-g/liter carbon sol in isopropyl alcohol was obtained by using polyvinylpyrrolidone as the dispersing agent. Attempts to disperse carbon blacks in benzene, toluene, and cyclohexane were unsuccessful.

Gel microspheres were prepared by dispersing sol droplets in hydrocarbons such as hexane and letting the drops fall through the hydrocarbon into diethylenetriamine, or other amines. After repeated washings, the gel spheres could be dried and calcined at 900°C to give oxide microspheres having pycnometric densities of 7.4 to 8.9 g/cc.

PHYSICAL RESEARCH

Reactor Operations. — The ORR operated 89.5% and the LITR 88.1% of the time during September and October 1964. There have been no spurious shim-rod drops since the installation of the new rod drives at the ORR earlier in the year. Instruments for the HFIR, including the heat-power calculator and the wide-range fission chamber channel, are being tested at the ORR.

A large pneumatic tube was installed in a beam hole at the LITR, primarily for irradiations for activation analysis. A section of the underground aluminum reactor inlet water line was replaced because of leaks caused by corrosion on the outside of the pipe. The line had been in service since 1952.

Leaks in the BSF pool persisted, and it was necessary to drill four wells in order to lower the water table enough so that the inside surfaces could be dried for painting. The pool has been painted and is now ready for filling.

Waste Disposal. — The radioactive waste disposal monitoring system was extended to provide new or improved coverage at two additional facilities. The 3020 (Pilot Plant) stack was equipped with a variety of continuous monitors and an in-stack sampler located on a newly constructed balcony at the 50-ft level on the stack. The system essentially duplicated the one in operation at the 3039 stack with improvements in the electronics. Included are beta-gamma and alpha particulate tape monitors and an iodine monitor which read out continuously at the Pilot Plant control panel and at the Waste Monitoring Control Center, Building 3105. Use of the old inventory sampler located at the base of the stack has now been discontinued, and the daily discharge is calculated from analyses of samples taken in-stack.

At Building 2026, the High-Radiation-Level Analytical Laboratory, the stack is now equipped with devices to monitor the stack discharge and process waste effluent. For the stack a beta-gamma particulate tape monitor and an in-stack sampler have been installed. The process waste discharge will pass through a newly constructed manhole containing the conventional flow-measuring, sampling, and activity-measuring instruments. Readout from all these devices will become a part of the total information telemetered from all parts of the waste disposal system to the Monitoring Control Center and will be kept under continuous surveillance at that point.

Hot-Cell Operations. — Operations in the hot cells were normal. Three fueled capsules were disassembled and examined in detail; two were fueled with $(U,Th)C_2$ coated particles and one contained graphite spheres fueled with $(U,Th)C_2$ coated particles. Four UO_2 meltdown experiments for fission-gas release study were disassembled; two UO_2 meltdown experiments were assembled and welded in the ORR cells prior to insertion in the reactor.

Molten-salt experiment MTR-47-6 was received from the MTR; specimens of the carbon, salt, and metal containers are being examined by metallography, x-ray diffraction, and gamma scanning. Detailed examination of seven ThO_2-UO_2 capsules irradiated at the ETR continued. Several Ta-Al absorber plates were visually examined and measured for changes in dimensional measurements and bow.

About 102 bend specimens and 184 tensile specimens were tested. Five ORR poolside boxes containing tensile specimens and tube-burst specimens were disassembled; dimensional measurements were made on the tube-burst specimens. Three fueled capsules from the ETR were partially disassembled and the NaK removed and disposed of in the burial ground. Two capsules containing refractory metals were disassembled.

PHYSICS AND MATHEMATICS

Charge Spectrometry: Charge Spectrum of Neon. — An examination of the charge spectrum of neon resulting from vacancies in the K shell has been made as a function of the energy of the x rays that produce these vacancies. From this study, information has been obtained on two important phenomena related to atomic readjustment.

First, an experimental evaluation has been made of the relationship between the extent of electron shake-off that results from a sudden change in effective charge and the degree of the "suddenness" of this change. In the sudden approximation the criterion for a nonadiabatic change is that the wave function of the system is not altered during a transformation of the Hamiltonian. Since a change in the former is related to $\exp\,[-i(\epsilon_m - \epsilon_n)\tau/\hbar]$, then the exponent must be much less than 1 for the sudden approximation to be valid. In our study, $\epsilon_m - \epsilon_n$ is related to the binding energy of the electron that is shaken off, whereas τ, the time necessary for changing the Hamiltonian, is proportional to the velocity of the outgoing photoelectron. We find from our data that the sudden approximation breaks down when $i(\epsilon_m - \epsilon_n)\tau/\hbar \approx 0.1$.

Second, we have found evidence for two-electron emission in an Auger process. Hitherto, an Auger process has been described solely as one in which only one electron is ejected. By producing K vacancies in neon with energies smaller than that necessary for double ionization, we may interpret the charge spectrum entirely in terms of the Auger process filling the vacancy. We find that about 8% of the processes result in two-electron removal.

Two possible sources of double ionization have been examined theoretically: (1) electron shaking due to changes in the effective charges as the K vacancy is filled and (2) Coster-Kronig transitions in the L shell. Both sources have been deemed negligible, leaving only the explanation that Auger processes may occur by multiple electron ejection.

Charge Spectrometry: Electrostatic Energy Analyzer. — An electrostatic energy analyzer, previously used for recoil studies, has been adapted for energy measurements of electrons that are ejected from atoms under x-ray bombardment. The binding energies of the L electrons in krypton have been determined experimentally for the first time by measuring the energy of the photoelectron. In this experiment, $K\alpha_{1,2}$ x rays of titanium with 4508 ev energy were incident on free krypton atoms. The L_I, L_{II}, and L_{III} energy levels are tentatively given as 1921 ± 6, 1732 ± 7, and 1684 ± 7 ev, respectively, with reference to the argon K level of 3203 ev. In comparison, Cauchois interpolates for the krypton L levels energies of 1904, 1726, and 1675 ev, whereas the tables of Hill et al.

list interpolated values of 1920, 1730, and 1670 ev. The present apparatus will allow a final determination of the krypton L levels with an accuracy of ±0.2% or approximately ±4 ev.

Theoretical Physics: Inelastic Scattering of Deuterons and Protons from ^{46}Ca. — The ^{46}Ca level scheme has been studied by the ^{46}Ca(d,d′) and the ^{46}Ca(p,p′) reactions at a bombarding energy of 7.00 Mev at the MIT-ONR electrostatic generator. Proton spectra were recorded at six angles in the MIT multiple-gap spectrograph with an energy resolution of 10 kev. Seven excited states in ^{46}Ca were identified below 3.8 Mev excitation. The (d,d′) experiment was performed at the Tandem Laboratory at Aldermaston, England, utilizing a 10.005-Mev deuteron beam and the 24-gap spectrograph of Middleton and Hinds [Nucl. Phys. 34, 404 (1962)]. Four excited states in ^{46}Ca below 4.5 Mev excitation were observed. The deuteron angular distributions have been interpreted by means of the distorted-wave theory to yield values of the spin and parities of the excited states. A comparison has been made of the ^{46}Ca level scheme with other $(f_{7/2})^{\pm 2}$ nuclei.

Theoretical Physics: Computer Codes. — A FORTRAN code, Athena, written for an IBM 7090 computer, computes Slater integrals and form factors suitable for inelastic-scattering calculations. It uses single-particle wave functions which are eigenstates of motion in a Woods-Saxon potential well. Two-body forces of Gauss, Yukawa, double-Yukawa ("soft core"), and Coulomb radial dependence may be used. Nonlocal effects on the single-particle wave functions may be included in the local energy approximation.

A FORTRAN code, Overlap, computes radial integrals of a variety of radial operators, using single-particle wave functions which are eigenstates of motion in a Woods-Saxon potential well. The radial operators currently available are r^n, $0 \leq n \leq 5$, and $(d/dx)(e^x + 1)^{-1}$, where $x = (r - R_0)/a_0$. Other functional forms may be added easily.

Low-Energy Neutron Time-of-Flight Spectrometry. — Recent measurements ("High Resolution Neutron Transmission Measurements of the 1148-ev Resonance of Neutrons upon ^{56}Fe," Phys. Letters, in press) show that the 1148-ev resonance of neutrons upon ^{56}Fe is produced by p-wave neutrons. Therefore the intense high-energy gamma-ray transitions to the low-lying negative-parity states in ^{57}Fe cannot be E1 transitions, but are most probably M1 transitions. The large NaI crystal detector at the ORNL fast chopper was employed to measure the capture spectrum from this resonance. Five distinct high-energy gamma rays were observed at 7.64, 7.48 ± 0.08, 6.36 ± 0.04, 4.9 ± 0.1, and 4.4 ± 0.1 Mev with relative strengths of approximately 10, 2, 5, 1, and 1 respectively. The gamma-ray spectrum is dominated by the strong gamma ray at 7.64 Mev, as is the thermal capture spectrum. However, the 6.36-Mev gamma ray is about ten times as strong in resonance capture, and the 5.95-Mev gamma ray seen in thermal capture is less than one-tenth as intense in resonance capture.

High-Voltage Experimental Program: Elastic and Inelastic Scattering of Protons from ^{106}Pd and ^{108}Pd. — Angular distributions for the elastic and inelastic scattering of 12- and 13-Mev protons from ^{108}Pd and of 13-Mev protons from ^{106}Pd have been measured between the angles of 24 and

165°. The targets were 0.5- to 1.0-mg/cm^2 metallic foils of the separated isotopes. The proton groups were detected with a surface-barrier detector with a resolution of about 60 kev. Small differences between the ^{106}Pd and ^{108}Pd cross sections for the elastic scattering of 13-Mev protons were observed. The maximum variation is about 25%. If at 13 Mev the small-angle elastic cross sections are normalized to the Rutherford value, the differential cross sections for excitation of the first 2^+ states in the two nuclei vary from about 8 to 0.7 mb/steradian between the forward and backward angles, with maximums near 68 and 128°; similarly, the cross sections for the 3^- states at 2.06 and 2.03 Mev in ^{106}Pd and ^{108}Pd, respectively, vary from about 1 to 0.2 mb/steradian. Differential cross sections were measured for the two-phonon states observed at 1.13 Mev (2^+ + 0^+) and 1.23 Mev (4^+) in ^{106}Pd, and at 0.94 Mev (2^+) and 1.05 Mev (0^+ + 4^+) in ^{108}Pd.

High-Voltage Experimental Program: Cross Sections for Isobaric Analog Resonances in the ^{117}Sn(p,n) and ^{119}Sn(p,n) Reactions. — Cross sections for the ^{117}Sn(p,n)^{117}Sb and ^{119}Sn(p,n)^{119}Sb reactions were measured for proton energies from 3 to 5.5 Mev. Neutrons produced in targets of separated isotopes were detected by Macklin's 4π flat-response counter, which is a 5-ft sphere of graphite with BF_3 counters embedded near its surface. The cross sections increase smoothly with energy except for pronounced resonances at 4.5 Mev for ^{117}Sn(p,n) and at 4.65 Mev for ^{119}Sn(p,n). The assignment of these resonances to isobaric analogs of the ground states of ^{118}Sn and ^{120}Sn leads to Coulomb separation energies which agree well with work on the tin isotopes by Becker et al. [Bull. Am. Phys. Soc. 9, 107 (1964)]. At the resonances the background cross sections from normal states can be estimated from smoothed curves joining the off-resonance yields; the resulting backgrounds are 6 and 9 mb at the ^{117}Sn and ^{119}Sn resonances respectively. Resonance contributions have been determined thus far for targets whose thickness, 100 to 200 kev, is larger than the natural widths. Preliminary results with these targets give integrated contributions of 0.2 mb-Mev for ^{117}Sn and 0.35 mb-Mev for ^{119}Sn.

High-Voltage Experimental Program: Resonance Parameters for Neutron Scattering from ^{208}Pb. — Since intermediate states are expected to show up in relatively pure form around closed-shell nuclei, it is of interest to identify the quantum parameters of resonances arising from the scattering of neutrons from such nuclei as ^{208}Pb. At neutron energies in the vicinity of 1.75 Mev there are several resonances which, depending on the ℓ-value assignment, could have reduced widths of the order of 10% of the Wigner limit [Phys. Rev. 127, 2192 (1962)]. A phase-shift analysis of elastic neutron scattering [Bull. Am. Phys. Soc. 8, 82 (1963)] in principle permits an assignment of resonance parameters. Although in cases where a number of phase shifts are involved, several sets of phase shifts often fit the same differential scattering data, nevertheless, one is generally able to limit the selection by arguments of consistency. Such a phase-shift analysis gives the following tentative assignments for several relatively broad resonances in the energy region of interest: 1.701 (5/2 − 0.02), 1.715 (1/2 + 0.09), and 1.749 (7/2 − 0.04). Here the resonant energy in Mev is followed by information in parentheses which includes the J value, the parity, and the reduced width in units of $3\hbar^2/ma^2$.

CHEMISTRY

Effects of Radiation on Analytical Methods. — The pyrolytic graphite electrode (PGE) and the "glassy carbon" electrode (GCE) have been under study for possible utilization as voltammetric indicator electrodes. The physical nature of these electrodes is such that they should be extremely useful as indicator electrodes in the analysis of radioactive solutions. A study was, therefore, carried out to establish the extent of any permanent damage caused by gamma radiation. Prototypes of both the PGE and the GCE were exposed to gamma radiation in various doses up to 10^7 rads. The electrodes were then checked for changes in electrometric behavior. In no case was any detectable change found in the electrode material itself, although the epoxy sheathing was found to be slightly affected. This effect, however, was not severe enough to prevent the use of these electrodes in hot-cell operations.

The thermal stability of ferrous sulfate, ceric sulfate, and oxalic acid dosimetry solutions in sealed tubes up to a temperature of 250°C was investigated for possible means of utilizing some one of them for high-temperature chemical dosimetry. Oxalic acid readily decomposed and was found to be inapplicable. Ceric sulfate in 0.8 N H_2SO_4 hydrolyzed at 250°C and produced a white precipitate. Increasing acid content prevented this hydrolysis; however, ceric ion was reduced to the cerous state. Ferrous sulfate had the highest degree of thermal stability. Air-saturated solutions showed some degree of oxidation, which can be eliminated by degassing before exposure to radiation. Ferrous sulfate under a cover of nitrogen gas was oxidized slightly, an effect that was probably due to a small amount of oxygen impurity.

Mechanism of Aqueous Radiation Corrosion of Reactor Materials. — We have previously shown that for the first 500 to 1000 min the rates of film growth for zirconium and Zircaloy-2 in oxygenated 0.05 m H_2SO_4 at 200 to 300°C can be satisfactorily expressed according to the logarithmic and cubic-rate laws. Also, others have reported cubic kinetics at longer exposure times. We recognized that a hyperbolic sine function relating the rate to film growth, which has foundation in theoretical models, might provide a better and more significant analysis of the data over long exposure periods. Others have argued that the anodic current is a hyperbolic sine function of the field strength,

$$i_+ = A[\exp(BV/x) - \exp(-BV/x)] = 2A \sinh(BV/x) , \qquad (1)$$

where V/x is the field strength, V is the voltage drop across the film, and x is the film thickness. It was shown that the particular rate law relating film thickness to time t will depend on the portion of the range of time studied. For small x and $BV/x > 3$ the growth is a logarithmic function of time. As x increases, and BV/x approaches 1, the film growth approximates a cubic function of time.

We carried out measurements of the anodic current i_+ as a function of time at constant anodic potential (about 1 v anodic to the corrosion

potential) at 199, 230, 230, 253, 255, and 284°C. Values of x were obtained by integration of the anodic current i_+. Values of BV for small x, where Eq. (1) is approximated by

$$i_+ = A \exp (BV/x) , \qquad (2)$$

were obtained from the slope of log i_+ vs 1/x plots. The validity of Eq. (1) over long periods was then determined by using these values. The results satisfactorily fit Eq. (1) over periods extending to 10,000 min for currents ranging over two orders of magnitude and film thicknesses from 600 to 3000 A.

These are the first reported tests of the applicability of the hyperbolic sine function at these low currents and field strengths. The significance of these findings with regard to physical models and theories is being studied.

Chemical Engineering Research. — Current efforts are directed toward proving the ability of the stacked-clone contactor to handle chemical flowsheets other than the one used during the equipment-development phase of the project. A two-phase system, 18% tributyl phosphate—Amsco—$NaNO_3$—H_2O, with uranyl nitrate as the distributing species, has been used almost exclusively. Some variation in physical properties and in contactor behavior was achieved by increasing the salting strength from 1 to 3 M and/or by varying temperatures from 20 to 50°C.

More recently, the machine was duplicated in stainless steel, and tributyl phosphate (TBP) concentrations as high as 100% were used. Flooding rates increased with increased density difference between phases and with decreased viscosity of the aqueous phase. Flooding (all at 40°C and compared at an aqueous to organic flow ratio of 10) has been observed at 4.95 liters/min for 30% TBP and 1 M $NaNO_3$; 4.90 and 2.55 liters/min for 3.54 M $NaNO_3$ and 53 and 100% TBP respectively; 2.42 liters/min for 100% TBP and 1.8 M $NaNO_3$; and 4.53 liters/min for 18% TBP and 1 M $NaNO_3$. Extraction and stripping efficiencies have remained high: 60 to 70%.

Separations Process Development. — The phenol extraction (Phenex) process, originally developed for recovering cesium, was successfully applied to the recovery of rubidium from alkaline ore leach liquors. A higher pH is required than that needed for effective cesium extraction. In a batch countercurrent test, more than 99.5% of the cesium and 97.5% of the rubidium were recovered from an Alkarb (alkali-metal carbonate byproduct of the lithium industry) solution at pH 14 with 1 M 4-sec-butyl-2-(α-methylbenzyl)phenol (BAMBP) in Amsco 125-82 in three extraction and three water scrub stages. Cesium would be separated by selectively stripping rubidium from the scrubbed extract, but this was not demonstrated. Decontamination factors for rubidium were 48 from potassium and 2500 from sodium. Rubidium extraction from pollucite ore leach liquor raffinates (following BAMBP extraction of cesium and upward adjustment of the pH of the liquor) was also demonstrated.

Separations Chemistry Research. — The extraction of certain metal ions by di(2-ethylhexyl)phosphoric acid (HA) from acid aqueous solutions

is markedly slow. A study of the kinetics of extraction of one of these, Fe(III), from perchlorate solutions shows rates conforming to:

$$-d[Fe^{3+}]/dt = k_1[Fe^{3+}][HA]_0^{0.5-1}/[H^+] ,$$

activation energy = 15.1 kcal/mole, at [HA] < 0.6 M,

$$-d[Fe^{3+}]/dt = k_2[Fe^{3+}][HA]_0^2/[H^+]^2 ,$$

activation energy = 10.3 kcal/mole, at [HA] > 0.6 M. Both the slight response to change of stirring rate and the high activation energies (from measurements at 25 to 60°C) indicate that the rate-controlling step is a chemical reaction, with different reactions controlling below and above 0.6 M HA.

Equilibria in the extraction of strontium by HA were previously reported (ORNL-3436). Extraction coefficients of the other alkaline earths (each at 0.001 M) have now been determined as functions of pH and HA concentration in benzene. The order of extraction is: Be ≫ Ca > Mg > Sr > Ba from 4.0 M $NaNO_3$ (Mg ≈ Sr from 0.5 M $NaNO_3$), that is, in inverse order to the atomic weights except that magnesium is less extractable than calcium. The curves of extraction coefficient vs pH are all similar to that previously described for strontium, with a prominent maximum at or fairly near pH 5. The curves of extraction coefficient vs HA concentration at low pH, where the reagent is nearly all in the acid dimer form $(HA)_2$, indicate extract stoichiometries corresponding to $MH_{n-2}A_n$, n varying as shown below, with the higher n values appearing at higher HA concentrations:

Molarity of $NaNO_3$	Be	Mg	Ca	Sr	Ba
4.0	4		5–6	6	6
0.5		4–6	4–5	4–6	4–6

High-Temperature Aqueous Solution Chemistry. – An investigation of the behavior of the osmotic coefficients of aqueous electrolytes at elevated temperatures has been extended to 165°C. As was found previously at 150°C, the osmotic coefficients of the 1-1 electrolytes fall off rapidly with increasing salt concentration, a behavior pattern at variance with that encountered at lower temperatures, where the osmotic coefficients usually rise with increasing salt concentration. For example, at 165°C the osmotic coefficients of 1, 2, 3, 4, and 5 m NaCl are 0.93, 0.939, 0.973, 0.790, and 0.2022 respectively. The values for lithium chloride at the same molalities are 0.932, 0.946, 0.928, 0.762, and 0.197, confirming the results previously obtained at 150°C. At both 150 and 165°C, the osmotic coefficients of LiCl are decidedly lower than those of NaCl for molalities greater than about 2.

A test was made of the question whether the hydrolysis of LiCl, to create HCl molecules which escaped into the vapor phase and falsified the

salt concentration in the sample dish, might account for the observed low osmotic coefficients. The isopiestic experimental conditions were simulated in an all-glass apparatus, strong enough to withstand over 5 atm of pressure and provided with a cold leg into which steam could condense together with any free HCl molecules. The volume of the cold leg was calibrated, so that the concentration of the equilibrating solution could be precisely calculated from its initial concentration. The condensate was tested by adding to it 3 volumes of 1 N $AgNO_3$; turbidity resulting from the precipitation of AgCl was tested by using a Beckman model B spectrophotometer as a nephelometer (sensitive to a Cl^- concentration of 7×10^{-10} equivalent/liter). In a control experiment starting with 0.005 m HCl, transport of HCl to the condensate was demonstrated. Experiments with NaCl and LiCl (starting with 1 m solutions) failed to give any evidence of HCl transport to the condensate. These results confirm the observations made by Appleby, Crawford, and Gordon (J. Chem. Soc. 1934, 1665) when they measured the vapor pressure of saturated LiCl up to a temperature of 180.20°C.

Physical Chemistry of Molten Salts: HF-H_2O Equilibrium with Molten Fluorides. — In continuing studies of the reactions involving H_2O, HF, and molten LiF-BeF_2 mixtures, it has been found that, in addition to the formation of oxide,

$$H_2O(g) + 2F^-(soln) \rightleftharpoons O^{2-}(soln) + 2HF(g) ,$$

a reaction occurs in which hydroxide is formed:

$$H_2O(g) + F^-(soln) \rightleftharpoons OH^-(soln) + HF(g) .$$

The experimental results — the effluent partial pressures of HF and H_2O measured as a function of the volume of a known mixture of HF, H_2O, and H_2 bubbled through a melt — have been used to evaluate the equilibrium quotients associated with the above reactions, namely,

$$Q_O = \frac{P_{HF}^2[O^{2-}]}{P_{H_2O}} \quad \text{and} \quad Q_A = \frac{P_{HF}[OH^-]}{P_{H_2O}} .$$

This evaluation was accomplished by computer solution of a pair of simultaneous differential equations which yielded, from Q_O and Q_A, calculated values of P_{HF} and P_{H_2O} which were compared with the observed values. By a general least-squares procedure, Q_O and Q_A were then adjusted to give the best fit to the data. By combining Q_O values with the results of previous measurements on melts saturated with BeO (ORNL-3663, p. 26), the concentration of dissolved oxide at BeO saturation was also calculated.

Measurements as a function of the mole fraction of BeF_2 between 0.27 and 0.60 and as a function of temperature between 500 and 700° have been completed, and the computations are nearly complete. For the composition 2LiF-BeF_2, the preliminary results are:

	500°	600°	700°
Q_O, atm moles/kg	5.0×10^{-6}	6.0×10^{-5}	7.5×10^{-4}
Q_A, moles/kg	4.5×10^{-3}	1.0×10^{-2}	2.0×10^{-2}
$[O^{2-}]$ at BeO saturation, moles/kg	6.7×10^{-3}	1.1×10^{-2}	3.0×10^{-2}

Physical Chemistry of Molten Salts: Reference Electrodes for Use with Molten Fluoride Solvents. — The electrode reactions $Be = Be^{2+} + 2e$ and $2HF + 2e = H_2 + 2F^-$ were studied in $2LiF\text{-}BeF_2$ at 508°C to determine whether they could be used as thermodynamically defined reference half-cells.

Polarization tests between two beryllium metal electrodes and between two $H_2/HF/Pd$ electrodes showed both systems to be stable and reversible for current densities up to 2 ma/cm^2. With a beryllium and an $H_2/HF/Pd$ electrode couple, a plot of the zero-current potential vs log (P_{H_2}/P^2_{HF}) yielded the reversible (Nernst) slope for HF partial pressures from 0.04 to 0.80 atm.

The potential of the cell

$$Be(s) + 2HF(g, 1\ atm) = H_2(g, 1\ atm) + BeF_2(d)$$

was +1.916 ± 0.001 v. This corresponds to $\Delta F_{cell} = -88.37$ kcal. An independent study (ORNL-3663, p. 29) provides an estimate of the activity of BeF_2 in this solvent and at this temperature, $a_{BeF_2} = 0.049$. Subtraction of RT $\ell n\ a_{BeF_2}$ (−4.67 kcal) from ΔF_{cell} gives $\Delta F^\circ_{cell} = -83.70$ kcal. The JANAF data for HF and pure liquid BeF_2 give $\Delta F^\circ_{cell} = -81.73$ kcal. These two cell values are within the uncertainty of the free energies of formation of BeF_2 and HF. Thus both electrodes appear satisfactory for use as reference electrodes.

Nuclear Methods of Chemical Analysis. — Calculations related to activation analysis with ^3He particles for elements of low atomic number contained in thick and thin targets have been completed. Neutron yields from ^3He irradiations, as measured by a BF_3 long counter and by threshold monitors, have also been obtained.

Isotope Chemistry. — Ion exchange studies were made of alkali-metal ions in anhydrous ethylenediamine (EDA), using the cation exchanger Dowex 50. The effective diffusion coefficients (D^i) and selectivity coefficients ($K^{tracer^+}_{M^+}$) for various ion pairs and resin cross-linkings are given in the table. The tabulated selectivity coefficients are, in general, smaller than those obtained with Amberlite 200 in anhydrous systems, but are 2 to 5 times those for aqueous Dowex 50 systems. The rates of diffusion in anhydrous ethylenediamine are from one-fifth to one-tenth of those for corresponding water systems.

System	D^i (cm^2/sec)	$K_{M^+}^{tracer^+}$
Dowex 50-X1-Li^+–^{134}Cs vs 0.1 N LiSCN–EDA	3.6×10^{-7}	16.1
Dowex 50-X8-Li^+–^{134}Cs vs 0.1 N LiSCN–EDA	3.7×10^{-7}	58.5
Dowex 50-X16-Li^+–^{134}Cs vs 0.1 N LiSCN–EDA	2.6×10^{-7}	400
Dowex 50-X1-Na^+–^{134}Cs vs 0.1 N NaSCN–EDA	2.1×10^{-7}	11.0
Dowex 50-X8-Na^+–^{134}Cs vs 0.1 N NaSCN–EDA	1.6×10^{-7}	27.9
Dowex 50-X16-Na^+–^{134}Cs vs 0.1 N NaSCN–EDA	8.6×10^{-8}	~200
Dowex 50-X1-K^+–^{134}Cs vs 0.1 N KSCN–EDA	1.2×10^{-9}	1.4
Dowex 50-X8-K^+–^{134}Cs vs 0.1 N KSCN–EDA	5.0×10^{-10}	2.5
Dowex 50-X16-K^+–^{134}Cs vs 0.1 N KSCN–EDA	1.5×10^{-10}	3.5
Dowex 50-X1-Cs^+–^{134}Cs vs 0.1 N CsSCN–EDA	2.7×10^{-9}	
Dowex 50-X8-Cs^+–^{134}Cs vs 0.1 N CsSCN–EDA	1.3×10^{-9}	
Dowex 50-X16-Cs^+–^{134}Cs vs 0.1 N CsSCN–EDA	4.3×10^{-10}	
Dowex 50-X1-Li^+–^{22}Na vs 0.1 N LiSCN–EDA	3.4×10^{-8}	1.1
Dowex 50-X8-Li^+–^{22}Na vs 0.1 N LiSCN–EDA	5.8×10^{-9}	1.6
Dowex 50-X16-Li^+–^{22}Na vs 0.1 N LiSCN–EDA	5.2×10^{-10}	9.7

The marked dependence of the selectivity coefficients on type of solvent offers interesting possibilities for practical application in separation processes.

METALLURGY AND MATERIALS

Direct Observation of Lattice Defects. – We have shown that dislocation configurations in deformed body-centered cubic metals and alloys depend on the deformation temperature, the rate of straining, and the applied stress at which the initial mobile dislocations are created. We feel that this latter effect, which has not been considered before, is important in determining twinning and fracture behavior of these materials. Dislocations probably multiply by a multiple cross-slip mechanism; when a jog on a screw dislocation exceeds some critical size, the applied stress can force the newly formed edge components by one another and enable the jog to act as a single-ended source. This critical size is inversely proportional to the applied stress. If the initial dislocations are created at high stress levels, they multiply without extensive cross slip and form densely populated narrow dislocation bands. These act as stress concentrations, which can induce twinning or fracture. Which of these processes occurs appears to depend on the grain boundaries. Under conditions in which the boundary is hardened by impurity segregation, we

believe separation at the boundary and fracture predominate. If the boundary is able to emit twinning partial dislocations, twinning will occur, without fracture. We are currently studying the nature of the grain-boundary structure in an attempt to understand this behavior.

Physical Property Studies. — We have recently modified our longitudinal heat flow apparatus to study the influence of irradiation on the thermal conductivity of small specimens of polycrystalline BeO, MgO, and Al_2O_3. Contrary to expectation, specimens of unirradiated BeO whose densities were between 89 and 97% of theoretical were found to have a thermal resistance that varied linearly between 30 and 120°C. Applying a simple density correction to the values for specimens of different density gave different values for the thermal conductivity of BeO having the theoretical density, indicating subtle specimen differences. We are measuring irradiated BeO specimens.

We have completed measurements on the thermal conductivity of Inconel 702 in the range 20 to 950°C, using our radial heat flow apparatus. Although our thermal-conductivity values are within ±2% of those of other laboratories, we are hesitant to recommend this material as a standard. We have observed changes of 1 to 2% in the thermal conductivity with time at temperature; this behavior is certainly not desirable in a standard. More important, we observed a serious chemical reaction between the Inconel 702, alumina, and the platinum vs platinum-rhodium thermocouples, which became contaminated and were rendered useless. This reaction places doubt on the results of any test in which these materials have been in intimate contact. We are currently installing a specimen of tungsten in the apparatus for measurements to 1000°C. We plan to obtain data on this specimen to much higher temperatures in the direct-heating apparatus.

We have analyzed our measurements to 1000°C of the thermal conductivity, electrical resistivity, and Seebeck coefficient of Armco iron and a higher-purity iron. The latter had a greater thermal conductivity than the Armco iron; impurities changed only the electronic contribution, not the lattice contribution. From this result, the thermal conductivity of any reasonably pure iron can be predicted to ±1.5% up to 1000°C from measurements of electrical resistivity alone.

Sintering Studies. — Our investigation of the high-temperature deformation properties of thoria has recently yielded an interesting result. Solid cylindrical specimens of ThO_2 subjected to a stress of 12,000 psi at constant temperature (a standard compressive creep test) deformed slowly with time as expected. When the reduction in height reached 2%, the specimen had a slightly enlarged diameter and on examination at 30× showed no sign of cracking or impending failure. Observed at 100× or greater magnification, however, the microstructure of the specimen revealed many void regions that were elongated in the direction of the applied stress and were apparently located at grain boundaries. These voids appeared to be largest in the regions of maximum shear stress and were present in significant concentrations at a deformation of 2%. With further deformation the voids continued to enlarge, ultimately forming macroscopic cracks. The specimen, therefore, appears to contain many fine cracks at a very early stage of deformation. Careful examination of the structure suggests that alignment of porosity causes the elongated voids.

Structure of Metals. – Grain-boundary migration plays a fundamental role in many metallurgical phenomena, such as recrystallization, grain growth, and creep. Minute amounts of impurities may greatly reduce the mobility of grain boundaries in pure metals. The basic premise of theories to describe this effect is that the interaction with an impurity atmosphere creates a drag on the moving boundary. Recently, a refined impurity drag theory by Cahn [Acta Met. 10, 789 (1962)] and by Lucke and Stuwe (Recovery and Recrystallization of Metals, p. 171, Interscience, New York, 1963) has removed the shortcomings of earlier approaches. Analyzing our grain-boundary migration rate data on zone-refined aluminum alloys containing copper in terms of this refined treatment, we have demonstrated the usefulness of this theory in interpreting the experimental results and shown for the first time that an excellent quantitative correlation exists between the impurity drag theory and experiment.

Our interpretation indicates a grain-boundary interaction energy of 4000 to 5000 cal/mole for copper impurity in aluminum. We also conclude that the region over which the grain boundary and the impurity atoms interact is large relative to the narrow disordered region in which the diffusivity of the impurity atom is high. In our interpretation most of the drag on the boundary comes from a region away from the center of the boundary.

To compare the relative effects of different impurities in a single solvent, we are presently investigating the effect of addition of gold to zone-refined aluminum.

CONTROLLED THERMONUCLEAR RESEARCH

The DCX-1 Injection-Accumulation Experiment. – Studies of the anomalous proton losses experienced with Lorentz-force trapping at very low pressures have continued. As described in the September report (ORNL-3718), radial losses of protons were indicated by a gridded radial current probe, biased to accept energetic ions (above 900 ev) and mounted in the median plane as the plasma radial limiter. These losses were coincident with azimuthal current fluctuations that produced magnetic probe signals near harmonics of the proton cyclotron frequency.

A foil-covered Faraday cup detector with an energy cutoff at 150 kev has also been used as a radial limiter to investigate these losses. The signals observed were like those seen with the gridded probe; so the radial losses are of very energetic particles. Experiments in which both detectors were employed and one was "shadowed" by moving the other to a slightly smaller radius have shown that the net radial velocity of the protons involved in these losses is very small compared with the precessional velocity.

Only occasional bursts of cyclotron rf signals have associated radial losses. High-resolution time-resolved displays of the frequency composition of these signals in the vicinity of the cyclotron fundamental have shown that the usual frequencies are appropriate for the formation of unstable "clusters" near the stable orbit for 300-kev protons. However, those signals with associated radial losses have additional frequencies,

somewhat lower, that are appropriate for clusters precessing so as to barely miss the radial limiter. It appears that the proton losses are driven by these precessing clusters.

Some modifications of the ion source and injector system are now in progress. The electrostatic lens is being changed to include an electric field to trap electrons beneath the lens in an effort to reduce space-charge spreading of the injected beam. A magnetic quadrupole lens in the 600-kev portion of the beam is being replaced with a solenoidal lens.

Properties of Plasmas Created by Electron Cyclotron Heating. — Preliminary experiments have been made with a 20-kev neutral hydrogen beam injected into the EPA. A fraction of the neutral beam is ionized by the electron cyclotron plasma (ECP) and trapped for a time determined by charge exchange. The observed charge-exchange decay time is about 30 times as long as that observed with no plasma for the same gage pressure. This measurement indicates that the neutral population is reduced by a factor of 30 by the ECP and hence is probably composed of 5-ev neutral hydrogen atoms formed by the dissociation of H_2^+. Because of questions regarding detector sensitivity and collimation, the measurements are not sufficiently precise to give a trapped-ion density.

A detailed study of the hot-electron density and temperature in the ECP has been made. The temperature appears to be a very weak function of microwave power and gas pressure. The temperature is between 100 and 120 kev for power levels from 5 to 30 kw and with gas pressures from 8×10^{-6} to 5×10^{-5} torr. The hot-electron density rises slowly as the microwave power is increased from 5 to 30 kw. However, the density drops sharply as the pressure is raised.

The Elmo facility is nearing completion. The power supplies and fast protection circuitry for the 8-mm microwave power sources are completed and ready for check-out. The coil tests have been completed. Currently the facility is being used with a deuterium arc. The first experiment with microwave power will use the 8-mm power sources. Originally, the initial experiments were to have involved 3-cm heating power and the folded-cusp configuration. The objective of performing the 8-mm power experiments first is to find a scaling law for calculating density, temperature, and volume for plasmas produced by short-wavelength microwave power.

Properties of Plasmas Created by Beam-Plasma Interaction. — An analysis of the energy distribution of ions near the intersection of the midplane with the anode wall in burnout IV has been made. A velocity-selecting channel parallel to the cylindrical anode with a radial electrical field was used. We observed an energy peak at 65 to 75 ev. Assuming isotropic distribution and calculating the selector aperture we obtained a density of 10^{12} ions/cm^3 at the selector entrance slit (3 in. from the axis). The energy corresponds to the long-lived energy "pocket" between scattering losses which decrease with increased energy and charge-exchange losses, which for nonsymmetrical reactions such as deuterium approach zero at finite energy [Phys. Fluids 6, 450 (1963)]. This is a direct verification of a prediction from scattering and charge-exchange cross sections which had only been observed as a longer decay time in plasmas with unsymmetrical charge-exchange reactions. We also observed an energy peak at 20 to 25 kev as expected but with an indicated density of about 10^{11} ions/cm^3.

The mirror throat openings in burnout V are being reduced to permit a greater pressure gradient and therefore greater power input. Intense sputtering in the midplane-liner intersection area at moderate power input has prompted this change.

Ion Source and Ion Beam Technology. — The 100-kv test stand has been used to study the extraction of ions from large plasma surfaces. The profile of the plasma being emitted from the anode aperture of a duoplasmatron source was determined as a function of the source operating parameters. Using this information a series of "cup" geometries were designed and tested. The beam was extracted at energies of 40 to 100 kev, neutralized by a set of accel-decel electrodes, and then observed as it traveled through a 14- to 16-in. drift space. Some anodes were designed that delivered large beams (up to 500 ma) with apparently small divergence. When these anodes were used on the 600-kv test stand, however, it was found that less current could be focused through a 1-5/8-in. aperture 100 in. from the lens than with the conventional "small cup" geometry. Studies are in progress to use these sources with a solenoid lens to form hydrogen ion beams having a high intensity (hundreds of milliamperes) and a low energy (20 to 80 kev).

Theoretical Plasma Physics. — Ion energy diffusion by microinstabilities in DCX-1 and DCX-2 has been analyzed thermodynamically. The expected spread is $\Delta E = K(E_0 D_E \tau)^{1/2}$, where $K \approx 2$ to 4, E_0 is the injection energy, and τ is the ion lifetime. An upper limit on the diffusion coefficient is estimated to be $D_E \leq E_0 Q^{3/2} \omega_{pi}$, where ω_{pi} is the ion plasma frequency, and Q is the ratio of microinstability electric field energy to the plasma kinetic energy. Energy spread in DCX-2 indicates $Q \approx 10^{-4}$. This is the order of the thermodynamic estimate, neglecting the large energy obtained, if instability destroys the very large temperature anisotropy and pushes ions to low energies. Though not yet explained, this is consistent with the experimental fact that the anisotropy and the hole in the energy spectrum at low energies persist. For DCX-1, the indicated value of Q is even smaller, $Q \approx 10^{-5}$. It is predicted that ΔE increases if the cold electrons are heated, as seems to be the case when electron cyclotron heating is applied to DCX-1. The parametric dependence of the above estimate seems to be borne out in previous DCX-1 data. The above formula indicates $\Delta E \propto n^{1/4} \tau^{1/2}$, n being the density. However, at low densities, where all instability modes are not yet active, the thermodynamic limit is probably not achieved. Then ΔE may vary as predicted through a range, but may increase rather abruptly when new modes become activated. This may be the case in DCX-1, where the low Q suggests all modes are not active. The fact that presently an increase of current does not increase the density beyond a limiting value may signify the onset of new modes. Indeed, with very much higher trapped currents (arc dissociation), densities higher than the present limit with Lorentz dissociation were achieved.

Atomic Cross Sections. — Measurements have been completed on the production of excited hydrogen atoms resulting from the dissociation of 500-kev H_2^+ by a carbon arc. The optimum ratio of H^0/H_2^+ for arc production was 0.20, as compared with 0.14 for production by argon gas. The ratio of H^0 in principal quantum numbers $n > 8$ to the total neutral yield

was 0.0046, which may be compared with a fraction of 0.0127 produced by argon gas collisions.

Attempts to measure resonances in the cross section for electron capture into excited states by passage of protons through atomic hydrogen have been temporarily unsuccessful because of mechanical failure of the tungsten oven used to produce atomic hydrogen. By passing protons through molecular hydrogen and helium, the fraction of H^0 with principal quantum number n = 12 or greater has been determined in the energy range 16 to 34 kev. The fraction increases monotonically with energy, with values at 34 kev of 0.002 and 0.0014 for hydrogen and helium respectively.

BIOLOGY AND MEDICINE

SOMATIC EFFECTS OF RADIATION

Statistical Services and Research: Resolution of Mixed-Frequency Distributions of Ploidy Classes. — Megakaryocytes develop by division of the nucleus but without division of the cell through five stages. The problem is to estimate the number of cells in each stage in a normal mouse. The numbers in each stage vary due to length of time in each stage or due to failure to reach the fourth or fifth stages. The stage of a given cell can be estimated by the DNA content since the amount of DNA doubles from stage to stage. However, some cells are in transition, so that frequency distributions of DNA content are suggestive of series of overlapping Gaussian distributions. Estimation of the parameters in a series of four Gaussian distributions is being conducted by nonlinear search methods.

Statistical Services and Research: A Branching Process in Drosophila. — It has been conjectured that certain mutations observed in offspring of females of *Drosophila melanogaster* result from alkylations on the DNA strands in the zygote which are induced by radiation or by chemicals. These alkylations remain with the strand on which they appear through each replication, that is, through each nuclear division of the zygote. It is known that after eight or nine divisions, some of the nuclei cluster at one pole, and that some of these pole cells develop into gonadal cells. If the gonadal cells do in fact follow this pattern of development, then the observed proportion of mutants in the offspring should be related in some way to the number of mutations which result from alkylation-induced misreplications in the first eight or nine divisions of the zygote. It is important therefore to know, initially, the frequency distribution and the expected number of mutant cells in the zygote after the first n divisions. Preliminary study of this question with the following set of definitions has already yielded some interesting results: (1) Generation zero represents the zygote. Thereafter, generation r consists of those nuclei which result from the rth division (r = 1, 2, ..., n). Thus, there will be 2^r nuclei in the rth generation. (2) A DNA strand with an alkylation may or may not misreplicate. If misreplication occurs in generation r, one hybrid will appear in generation r − 1, and one mutant will result

in generation r + 2. The implication is that a misreplication in generation n − 1 will not produce a mutant in generation n. One hybrid will appear in generation n, but we do not propose to call it a mutant. (3) Define q as the probability of a misreplication at any division ($0 \leqq q \leqq 1$; $p = 1 - q$), due to an alkylation on one of the two DNA strands. If an alkylation appears on each of the two DNA strands in the zygote, then designate the probabilities of misreplication as q_1 and q_2 respectively ($0 \leqq q_1, q_2 \leqq 1$). With these definitions, the expected number of mutations after n divisions is $(2^{n-1} - 1)q$, for an alkylation on a single DNA strand, and $(2^{n-1} - 1)(q_1 + q_2)$, for an alkylation on each of two DNA strands.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. – In the past two months the scope of the animal testing program has greatly increased. We are now receiving wild Kansas cottontail rabbits that will be used in the Co-Carcinogenesis Program. These animals are tested for external and internal parasites, coccidiosis, tularemia, brucellosis, and Rocky Mountain spotted fever. Animals found positive for tularemia, brucellosis, or Rocky Mountain spotted fever are sacrificed and discarded. Animals infected with parasites or coccidia are treated and released from quarantine when they are found to be free of infection. Domestic rabbits receive the same attention except that they are not tested for zoonotic infections. Mice to be used in the Co-Carcinogenesis Program are being tested for *Mycoplasma pulmonis* (PPLO), *Salmonella sp*, *Pseudomonas sp*, and external and internal parasites. One hundred twenty object samples are taken each week from the Co-Carcinogenesis Laboratory and Animal Facility building, and the results are returned to the investigators involved with the exact location and the genus or organism found. Air samples are taken in Co-Carcinogenesis laboratories and animal facilities to monitor the levels of *Pseudomonas* and other bacteria in these areas.

Continued efforts to improve the animal health and testing program in the General Experimental Animal Facility are being made. Weekly object samples are being taken throughout the facility. These areas are also air monitored weekly. Object samples are taken in the General Experimental Animal Facility and receiving and quarantine buildings weekly. All animals purchased come through the receiving and quarantine area and are tested for *Salmonella sp*, *Pseudomonas sp*, and external and internal parasites. The positive animals are sacrificed at the receiving building to prevent contamination of the general animal facility. To determine the effectiveness of our screening program we routinely sample animals from the General Experimental Animal Facility.

There is a great need throughout the Biology Division in general and especially the Co-Carcinogenesis Program for a virus testing laboratory for latent mouse viruses. We have been studying the techniques and methods for setting up such a service.

MOLECULAR- AND CELLULAR-LEVEL STUDIES

Biophysics: Use of Multisection ESR Cavities. — Study of multisection electron spin resonance (ESR) cavity operation has been continued. Successful modification of the magnet has been made to increase the volume of homogeneous field. The Fieldial magnet control circuit and the cross-over point of ESR spectra were used as a relative fluxmeter with reproducibility of about 1 part in 10^5. Algebraic additivity of signals from samples in two cavity sections has been demonstrated within the experimental error of about 1%. From the overlapping spectra of different free radicals in irradiated glycine the spectra of radicals decaying during storage at elevated temperatures have been demonstrated by measuring difference signals. During this work the spectrometer was moved to a new laboratory without apparent change in instrument characteristics.

Biophysics: Beta-Particle Spectroscopy with an NaI(Tl) Crystal Detector. — The pulse-height response of a thallium-doped sodium iodide scintillation crystal to incident beta-, bremsstrahlung-, and K x-ray fluorescence radiations has been studied for the beta emitters ^{14}C, ^{32}P, and ^{89}Sr, using the multichannel analyzer. Conventionally, the use of NaI(Tl) has been restricted to the spectroscopy of photons, and anthracene has been used for beta particles. The pulse height for beta particles is of the same order of magnitude as for photons of equal energy, but the energy dependence is somewhat different. In the energy range 0.6 to 1.7 Mev, the efficiency (counts per incident event) for electrons is high. Comparison of the apparent absorption of continuous absorbers vs multilayer absorbers with air gaps between indicates greater absorption in the latter case.

Biophysics: Experiments in UV Photochemistry of Escherichia coli. — The ultraviolet action spectra for killing of *E. coli* B/r and *E. coli* B/s have been compared. The slopes of the straight-line portions of the survival curves have been used as parameters defining sensitivity. With this definition the action spectra of both strains turned out to be identical within experimental error, B/s being approximately a hundred times as sensitive as B/r at all wavelengths tested (2300, 2380, 2480, 2650, 2805, 2900, and 2967 A). All the survival curves of B/r had a considerable shoulder. The extrapolation numbers of these survival curves varied bimodally with wavelength. Work has been started on the determination of ultraviolet photoproducts produced in vivo in *E. coli* 15 T^-, in the DNA of which thymidine has been partially replaced by 5-bromo-deoxyuridine (BUdR). Tritiated thymidine and ^{14}C-BUdR were used. No evidence has been found for the formation in vivo of a compound similar to the one described by Haug [Z. Naturforsch. 19b, 143 (1964)] for the in vitro irradiation of TpBU (a type of dinucleotide).

ISOTOPE DEVELOPMENT

Application of Radioisotopes in Analytical Chemistry: Radioisotopes in Common Analytical Methods. — Evaluations of the phosphomolybdate and

magnesium ammonium phosphate methods for the determination of phosphorus have been continued with the help of ^{32}P radiotracer. The major difficulty encountered in an attempt to develop a rapid radiotracer method has been the inability to reproduce a standard counting geometry. The counting characteristics of both types of precipitates containing 0.5 to 50 mg of phosphorus are being investigated. Techniques are likewise being studied to achieve a reproducible solid geometry after the removal of siliceous impurities from the phosphate precipitates, so that the sensitivity of the method can be extended to the concentration range of most steel samples (0.01 to 0.1%). The possibilities of counting solutions of the precipitates with a beta proportional counter or suspensions of the precipitates in a liquid scintillator system are also being looked into.

Application of Radioisotopes in Analytical Chemistry: Activation Analysis in Forensic Science. — In a study of the activation analysis of bond papers, the measurement of elements that yield short-lived radionuclides has been completed. Most of the papers contain so much aluminum and titanium that other short-lived activities (of a few minutes half-life) cannot be measured nondestructively. These two elements are very uniformly distributed throughout each sheet, but they differ appreciably in concentration among brands. Preliminary results indicate that a rather large number of long-lived activities are formed in the paper after long periods of irradiation. All results will be reported after the long-lived activities are measured.

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