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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JULY 1966

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

July 1966

DATE ISSUED

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JULY 1966

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

High-Burnup Coated-Particle Irradiation Experiment. — The purpose of this experiment is to evaluate the performance of loose pyrolytic-carbon-coated fuel particles in a graphite container at high burnup (50 at. % of the heavy metal) and at conditions of interest for high-temperature gas-cooled reactors. Irradiation was completed and the capsule was removed from the reactor on July 14, 1966. The irradiation period extended over a full year, with some interruptions to permit the reactor test space to be used for other experiments. Total irradiation time was 257 days. The estimated heavy-metal burnups are 55 at. % for the General Atomic particles in the lower compartment and 53 at. % for the ORNL particles in the upper compartment. Fission-gas release rates expressed as R/B (ratio of fission-gas release rate to birth rate) were initially about 1×10^{-6} but increased rapidly at about 10 at. % heavy metal burnup and were in the neighborhood of 1×10^{-4} to 5×10^{-4} during most of the test. Fuel temperature, which was about 1400°C at the beginning of the test, decreased to possibly below 1100°C at the end. There is some uncertainty about the fuel temperature during the latter part of the test, because the W–5% Re vs W–26% Re central thermocouple failed at about 45 at. % heavy-metal burnup.

Fast Gas-Cooled Reactor Irradiation Tests. — Irradiation tests are being performed in the ORR poolside facility to support the development of the General Atomic fast gas-cooled reactor design. The fourth capsule (P4B-4) in this series is nearing the end of the planned irradiation period of two ORR cycles and continues to operate satisfactorily. This capsule contains a "flexcan" fuel element and a fuel-supported clad element.

Exposure conditions include 1000 psia external pressure and 750°C cladding temperature. On June 25 a power excursion of the ORR caused the cladding temperature to rise to an estimated 860°C. The capsule appeared to operate normally after the excursion, and a subsequent leak

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test gave no indication of cladding failure. Manual and pressure thermal cycling of the capsule was resumed on July 6, and to date the capsule has been subjected to a total of 48 thermal cycles and 11 pressure cycles.

Fission-Gas Release from Pyrolytic-Carbon-Coated Uranium Oxide Particles During Irradiation. – Capsule B9-30, containing pyrolytic-carbon-coated uranium oxide particles, was irradiated to 1.8 at. % uranium burnup at 1400°C. The fractional release of ^{88}Kr at the beginning of the test was 2.6×10^{-6}. After 159 hr, eight bursts of fission gas were released. Several of the bursts were of sufficient magnitude to drive the exit helium activity monitor off-scale and cause the capsule to be withdrawn from the reactor flux. The purpose of this test was to study the effect of ratio of the thickness of the inner, porous carbon coating to that of the outer, dense coating. The coating thicknesses are listed below:

	OR-491	OR-493	OR-494
Average particle dimensions, μ			
Total diameter	713	703	704
Fuel particle	429	419	412
Porous coating thickness	28	61	93
Total coating thickness	142	142	146
Number of particles irradiated	5	10	570

The OR-491 and OR-493 coated particles were removed from the capsule separately from the OR-494 coated particles. No broken or damaged OR-491 or OR-493 particles were found, but many of the OR-494 particles were damaged. It is estimated that from 5 to 10% of the OR-494 particles have a hole in the coating. The holes appear to be about one-tenth the diameter of the particle, and there are no extending cracks in the coating surfaces around the holes.

All 15 coated particles from batches OR-491 and OR-493 were isolated and examined metallographically. Although none of these coated particles failed, only about half of the thickness of the inner coating remained after irradiation. There was no evidence of carbide formation in any of the particles. The fuel in the particles from batch OR-493 appeared to have moved through the inner porous coatings, but the outer coatings had not been affected.

One hundred particles from batch OR-494 were examined metallographically. The fuel in these coated particles appeared to have operated at a higher temperature than the particles from batches OR-491 and OR-493. This was probably the result of the greater thickness of the inner porous layer. None of the unfailed particles showed evidence of carbide formation, but small crystals of carbide were observed at the inner surface regions of the failed coatings. The fuel in many of these coated particles had moved into large voids that had formed in the coatings, presumably by attack from impurities in the fuel (3500 ppm Al, 200 ppm Ca).

Heat Treatment of Pyrolytic-Carbon-Coated Oxide Particles. — We performed a series of high-temperature heat treatments on several batches of pyrolytic-carbon-coated UO_2, ThO_2, and $(Th,U)O_2$ particles to study the effects of residual carbon in the oxide particles and to determine whether mechanical properties might be deduced from the results. All samples of coated particles had porous inner coatings ($\rho < 1.0$ g/cm^3) and isotropic outer coating layers varying in density from 1.6 to 2.0 g/cm^3 and covering a range of crystallite sizes from approximately 150 A down to approximately 30 A. The results of successive heating for 2 min at 100° intervals from 2200 to 2600°C may be summarized as follows:

1. Coated UO_2 particles showed progressive migration of fuel into the inner coating after heating at 2300°C and above. The pattern of this migration suggests a vapor-phase transport mechanism.
2. All coated UO_2 samples survived heating to 2500°C, at which the internal CO pressure is estimated to be approximately 150 atm, but some failed catastrophically at 2600°C. At 2500°C the stresses from internal pressure caused considerable thermal creep in the coating, which resulted in as much as 25% increase in diameter.
3. The thermal stabilities of coated UO_2 particles having high (6800 ppm) and low (200 ppm) residual carbon were not significantly different.
4. Coated ThO_2 and mixed-oxide particles survived heating to 2600°C with little if any changes in structure or dimensions.

We are conducting additional heat treatments to study the correlation of thermal creep behavior with the physical properties of the coatings.

FUEL-CYCLE STUDIES

Fueled-Graphite Fabrication Development. — In the fuel-cycle work directed toward fuels for gas-cooled reactors, methods are being developed to make ceramic spheres containing thorium and thorium-uranium mixtures in the 100- to 500-μ-diam size range. In conjunction with this effort, studies are being made to develop improved methods for coating these spheres with one or more layers of pyrolytically deposited carbon.

In our 5-in.-diam fluidized-bed coaters, both low- and high-density carbon coats were applied to thoria microspheres. Low-density coats of 1.17, 0.79, and 0.61 g/cm^3 were applied at rates of 7.8, 22.4, and 53 μ/min, respectively, from pyrolyzed acetylene gas. High-density coats of from 1.91 to 2.01 g/cm^3 were applied at rates varying from 1.52 to 2.86 μ/min. The coating with a density of 2.01 g/cm^3 was applied at 2.65 μ/min.

We made two coating runs in our entrained bed coater to see if we could put controlled-thickness coats having a density of 2.0 g/cm^3 on charges of 230-μ-diam thoria spheres. Coating densities of 0.97 g/cm^3 on a 1-kg charge, and 2.0 g/cm^3 on a 1.65-kg charge, were obtained. One goal of the experiment on the 1-kg charge was to deposit a 30-μ-thick coat; a 29.8-μ-thick coat was achieved.

We are attempting to develop an in-line process monitor for rapid determination of the average thickness of the carbon coating on thoria particles. The method we are studying is based on x-ray attenuation in a small sample of coated spheres. Thirty-three samples of 230-μ-diam spheres with carbon coatings ranging in thickness from 22 to 122 μ were examined. Agreement to within ±5 μ of optically measured values was obtained on 27 of 33 samples tested. The coatings of the six samples outside the range cited were found to be nonuniform.

Dicarbide Preparation from Sol-Gel Microspheres. — Two approaches are being studied to make dicarbide microspheres from oxide microspheres prepared by the sol-gel process. One involves heating 1150°C-fired oxide spheres in a rotating bed of lampblack at >2000°C in an argon atmosphere; this approach shows promise of producing theoretically dense dicarbides. In the other, carbon is incorporated in the starting material for the oxide sol. Gel microspheres are formed from the mixed sol, and the microspheres are converted to dicarbide by firing in vacuum or inert gas to about 2000°C; this approach yields a porous product.

The fabrication difficulties reported earlier, when high-carbon-containing UO_2 particles were converted in lampblack and then coated, were found to be caused by coating permeability rather than by the carbon in the spheres. The difficulties were eliminated by using a low-density buffer layer followed by a high-density, isotropic sealer layer, both deposited at low temperatures. Studies with low-carbon (200-ppm) and high-carbon (6800-ppm) UO_2 microspheres now indicate no substantial difference in conversion, fuel migration behavior, or dimensional changes. Irradiation tests of both types of UO_2 spheres are in progress.

Previous results with the oxide-carbon sol approach showed that conversion of ThO_2-C gel shards to ThC_2 gave products of gross porosity (~20 μ), regardless of the firing conditions used. Since microspheres have not shown this problem, they are now being used in the conversion studies. Microspheres of ThC_2 prepared so far show sensitivity to moisture, an indication of porosity. Observations of the shrinkage of these materials also suggest incomplete densification. Such behavior is typical of this preparation approach and is not necessarily undesirable.

Thoria-Urania Sol Preparation. — A laboratory-scale process for making mixed sols of thorium and uranium oxides directly from a solution of thorium-uranyl nitrate has been demonstrated by using a two-stage extraction with long-chain aliphatic amines dissolved in a hydrocarbon diluent. The same amine solution is used in both stages and is regenerated for further use by contacting with sodium carbonate solutions. Most of the work so far has been with the commercially available secondary amine Amberlite LA-2 (n-lauryltrialkylmethylamine). Thoria-urania microspheres (thorium:uranium ratio of 3.42:1) produced from sol prepared this way had a density of 10.1 g/cm^3 and a carbon content of <10 ppm.

We are also preparing mixed sols of thorium and uranium oxides by dispersing UO_3 in boiling thorium nitrate solutions and by coprecipitation of thorium and uranyl hydroxides from thorium-uranyl nitrate solutions. In general, these two approaches yield sols of undesirably high nitrate content, but some satisfactory sols were prepared.

Thorium-Uranium Recycle Facility. — The Thorium-Uranium Recycle Facility is being built to provide adequate shielded space for development of equipment for remote processing and fabrication of Th-^{233}U fuels of various types. Sufficient shielding (5-1/2 ft of normal concrete) is being provided so that highly irradiated fuel assemblies can be accommodated after a three-month decay period.

Construction of the building is approximately 77% complete. All masonry and partition walls in the building, except those between the mechanical and equipment rooms, are now complete. Cell construction has progressed. The ceiling of cell E is being prepared for placement of the roof slab; the liner walls of cell G are at ceiling level; and the walls of cells C and D are up to the manipulator-rail level. The panel walls of the air lock and cell A, as well as cell A door and the air-lock hatch, have been positioned and erected. All equipment furnished under ORNL participation is progressing satisfactorily, except for the radiation shield doors. Door 2 is now installed in preliminary form; door 3 has been delivered and is currently being installed. Door 1 is not yet delivered.

Equipment has been designed and is being constructed for use in development of processes for production of urania and urania-thoria sols. Precipitation-peptization equipment for urania sol preparation is ready for installation in the operating location; laboratory-scale amine-extraction equipment is still being fabricated. Small-engineering-scale amine-extraction equipment is being designed and procured.

^{233}U Storage and Distribution. — ORNL serves as a national storage and distribution point for ^{233}U. The pilot-plant building (Building 3019) has facilities for storing up to 120 kg in solid form and 400 kg as nitrate solution. A glove-box solvent extraction facility, capable of processing 1 kg of ^{233}U per day, was recently installed to complement the existing solvent extraction system, which is capable of processing 6 kg/day.

A geometrically safe, thermo-syphon dissolver with a capacity of 3 kg of ^{233}U (as oxide or metal) per day was installed to augment the existing dissolver, which has a capacity of 0.5 kg/day. In the past report period, 17.2 kg of $^{233}UO_2$ was dissolved. The large solvent extraction system was operated to purify 9.4 kg of this material, and the first shipment (0.5 kg) was transferred to Y-12, where 3.1 kg of oxide and 1.5 kg of metal will be produced for Argonne National Laboratory. Shipments will be made every five days until the commitment is filled. The low ^{232}U content of the material permits several weeks' storage with little radiation hazard from the ^{232}U daughters. In addition, the reduction step, which volatilizes the ^{212}Pb, reduced the radiation considerably. Purification of an additional 10 kg of ^{233}U is scheduled.

An unloading station, capable of emptying 3-liter containers of ^{233}U at a concentration of 375 g/liter, was installed and tested.

Receipts during the month included 30 containers of ThO_2–3% UO_2 (a total of 1500 g of ^{233}U).

Sol-Gel Studies: Urania Sol Preparation. — Methods are being developed for preparing urania sol by two processes: precipitation-peptization and solvent extraction. The first has been developed to the

point that a continuous system using six mixer-settlers with a countercurrent flow of U^{4+}-oxide slurry and wash water has been designed and fabricated for engineering evaluation. In this process, a U^{4+} solution is prepared by catalytic reduction with hydrogen of a 0.5 M $UO_2(NO_3)_2$ solution containing nitric and formic acids. The U^{4+} is precipitated as hydrous oxide by addition of NH_4OH to pH 9.

The second process is in an early stage of development, but appears promising. Here, an aqueous U^{4+} solution containing nitric and formic acids is equilibrated with a primary or a secondary amine dissolved in a hydrocarbon diluent. The nitrate ions and, to a lesser extent, the formate ions are transferred to the organic phase, causing hydrolysis of the U^{4+}. Sols containing up to two moles of uranium per liter have been prepared in this manner. So far, none has been formed into gel microspheres.

MOLTEN-SALT REACTOR PROGRAM

Molten-Salt Reactor Experiment. — Continuous operation at full power (except for two minor shutdowns of 2 hr each) was interrupted after 180 hr by a failure in the electric power supply to a component cooling pump. The standby unit was not started in time to prevent thawing of the drain valves, and both the fuel and coolant loops were drained. The failure occurred at a junction box inside the containment dome for the pump and completely destroyed the junction. As a result, we could not determine the exact cause of the failure. We replaced the power leads, resealed the containment dome, and resumed operation at full power on July 1. The total shutdown time was 99 hr.

Full-power operation continued for another 320 hr before the next interruption occurred. Then, on successive days, two electric power failures, both caused by electrical storms, resulted in control-rod scrams. In both cases, full-power operation was resumed within a short time.

On July 17 the impeller of one of the main blowers failed completely; the blades and the hub to which they were attached disintegrated. We stopped power operation and drained the coolant loop to permit inspection and evaluation of any other damage that might have been caused by the failure. On inspection of the second blower, we found a large circumferential crack in the cast aluminum impeller hub. The causes of the cracking and failure have not been established yet.

The performance of both the primary and secondary salt systems was highly satisfactory during the periods of sustained operation at high power. However, the pressure drop across the main charcoal beds increased slowly, and we had to blow out the obstructions several times to keep the pressure drop below 2 psi. There was no evidence of plugging in the particle trap ahead of the charcoal beds until shortly before the main blower failure. No corrective action had been taken before the shutdown occurred.

The discrepancy between the heat-balance power and that indicated by the nuclear instruments was related to the temperature of the water surrounding the neutron chambers. We installed a cooler to remove the

heat generated by the absorption of nuclear radiation at high powers. Then we repositioned the nuclear detectors to obtain a one-to-one correspondence between the neutronic and heat-balance power measurements at the steady-state water temperature.

The total integrated power at the time of the blower failure was 7822 Mwhr. We had planned to shut down at 10,000 Mwhr to remove graphite and metal corrosion and irradiation specimens from the core vessel. In view of the time required to replace the blowers, we decided to shut down completely and perform the specimen removal and other necessary maintenance at the same time.

Metallurgy. — We have found that Hastelloy N is subject to a high-temperature form of irradiation damage which reduces the time to failure at a given stress and the rupture ductility. We are presently studying this problem from two standpoints. First, the MSRE is constructed mainly of this material, and we are conducting in-reactor studies to predict the safe operating life of this reactor. This work is utilizing the same heats of materials used in constructing the MSRE and will be supplemented by the examination of surveillance specimens taken from the reactor periodically during its operation.

The second area of endeavor is concerned with the use of Hastelloy N in future reactors. We have found in our work on stainless steel that changes in the fabrication history and in the chemical analysis can have a very beneficial effect on the properties of the material in a neutron environment. We are finding that similar improvements can be made in the behavior of Hastelloy N. Studies to date have shown that beneficial effects can be obtained by small additions of titanium, zirconium, and hafnium. Studies are in progress to evaluate the influence of heat treatment and to determine whether other properties of the alloy are affected adversely by the addition of these elements.

Radiation Effects. — Out-of-pile test operation of the molten-salt irradiation assembly was satisfactorily completed, and the assembly was installed in beam hole HN-1 of the ORR during the week of June 6, 1966. The thermal loop contains some 157 g of MSRE solvent salt with the chemical composition of ^{7}LiF-BeF_2-ZrF_4 (64.7-30.1-5.2 mole %).

After installation in the ORR, the loop was brought to operating temperatures normally ranging from about 660°C in the core section to 560–600°C in the cold leg to provide for thermal circulation of the salt. To date (July 20, 1966), the salt has been maintained molten at these temperatures for more than 900 hr. This time has been spent in testing operating functions of the loop assembly and associated auxiliary equipment. The addition of enriched uranium (as ^{7}LiF-UF_4 eutectic) is presently scheduled to be made during the reactor shutdown beginning July 24, 1966. The tests of operating functions have included the following:

1. Calibration of the heat removal capability of the various loop coolers.
2. Reliability of the thermally induced salt circulation. It was found necessary to use argon instead of helium as a cover gas to maintain salt flow (5 to 10 cc/min) for extended periods of time (~16 hr with

helium vs two weeks with argon). We believe that the lower solubility of argon reduces the rate of bubble formation, which is thought to cause loss of flow.

3. Reactor gamma heat measurements with the loop fully inserted and fully retracted, that is, 9 in. from the reactor lattice.

4. Test operation of the salt sampling and addition system. To date, one salt addition has been made to the loop while in-pile.

MSR Processing: Continuous Fluorination of Molten Salt. — We are developing equipment for the continuous removal of UF_4 from the fuel stream of an MSBR by contacting the salt with F_2 in a salt-phase-continuous system. This equipment will be protected from corrosion by freezing a layer of salt on the vessel wall; the heat necessary for maintaining molten salt adjacent to frozen salt will be provided by the decay of fission products in the fuel stream. Present development work consists of two parts: (1) studies in a continuous fluorinator not protected by a frozen wall, and (2) study of a frozen wall system suitable for continuous fluorination but with which an inert gas is used. Experimental work on the nonprotected system is well under way; the protected system is being designed.

The nonprotected system consists of a 1-in.-diam. nickel fluorinator 72 in. long and auxiliary equipment which allows the countercurrent contact of a molten salt with F_2. Experiments can be carried out with molten-salt flow rates of 3 to 50 cm^3/min with fluorinator salt depths of 12 to 54 in. The system is constructed of nickel with the exception of molten-salt transfer lines, which are Hastelloy N. The fluorinator off-gas passes through a 400°C NaF bed for removal of chromium fluorides, a 100°C NaF trap for removal of UF_6, and a soda lime bed for F_2 disposal. Analysis for F_2, UF_6, and N_2 is made ahead of the 100°C NaF bed with a gas chromatograph.

Experiments have been carried out at 600 to 650°C using an NaF-LiF-ZrF_4 mixture containing 0.2 to 0.5 wt % UF_4 and having a melting point of ~550°C. Salt feed rates of 5 to 21 cm^3/min and F_2 rates of 75 to 250 cm^3/min (STP) have been used with molten-salt depths of 40 to 52 in. Uranium removal during one pass through the fluorinator has varied from 95 to 99.6% as determined from salt samples. Equipment operation has been smooth, although several plugs have developed in salt transfer lines and in the fluorinator off-gas.

During the best run to date (CF-10), a molten-salt feed rate of 20.7 cm^3/min and an F_2 feed rate of 250 cm^3/min (STP) were maintained for a 2.5-hr period. The concentration of U in the feed salt was 0.32 wt % and the fluorinator was operated at 650°C with a salt depth of 48 in. The uranium concentration in the salt discharged from the fluorinator during the last hour of operation was 0.0020 wt %, as determined from four salt samples taken at 15-min intervals. Based on inlet and exit uranium concentrations in the salt, 99.4% of the uranium was removed by the fluorinator. The equipment operated smoothly during the run, salt and gas feed rates were constant, and the system appeared to have reached steady state during the last hour of operation.

Continuous fluorination of the fuel stream of an MSBR with equipment of the type studied is considered feasible. Study of continuous fluorination will be continued, and the use of a frozen wall for corrosion protection will be demonstrated.

MSR Processing: Vapor-Liquid Equilibrium Studies in MSBR Salts. — We are attempting to measure the relative volatilities of rare-earth fluorides with respect to LiF in a recirculating Othmer-type equilibrium still. Nine tests with CeF_3, LaF_3, and NdF_3 have been performed at 1000°C and 0.50 mm Hg. Some intermediate results indicate the following relative volatilities:

$$0.0029 > \alpha\text{-}CeF_3\text{-}LiF > 0.0013 ,$$

$$0.00089 > \alpha\text{-}NdF_3\text{-}LiF > 0.00055 ,$$

$$\alpha\text{-}LaF_3\text{-}LiF = 0.0069 .$$

The concentration of each rare-earth fluoride in LiF was 0.02 mole fraction. These values are more than an order of magnitude smaller than previously reported values.

MSBR Processing Studies: Liquid-Metal Extraction of Fuel Salt. — A study has been initiated on liquid-solid equilibria in the system Bi-$LaBe_{13}$ at temperatures in the range 400 to 800°C. The purpose is to gain a better understanding of the extraction of rare earths from molten fluoride carrier salt using liquid metals.

To date, 10 g of $LaBe_{13}$ has been prepared by sintering a powder blend of the elements using an atom ratio 13/1 = Be/La. The charge was contained in a cylindrical tantalum crucible with a close-fitting lid, which in turn was protected by an argon atmosphere contained in a quartz tube. Sintering was done by induction heating, first at 1220°C for 2 hr at 9 to 200 μ pressure, and finally for 1 hr at 1400 to 1500°C under 8 mm of argon. The only crystalline phase identified by x-ray diffraction analysis was $LaBe_{13}$. A few faint unidentified lines were found which indicate a slight impurity, probably relating to a beryllium deficiency. This is supported by the identification of a small amount of $TaBe_{12}$ clinging to the upper part of the crucible wall, which therefore has absorbed some of the beryllium required to satisfy the stoichiometry of $LaBe_{13}$.

Future plans call for equilibration of the beryllide with liquid bismuth, filter sampling of the liquid metal at temperature, and analysis for both La and Be. Analogous beryllides of Ce, Nd, Sm, and Eu will be prepared for similar studies.

Analysis of Radioactive MSRE Fuels. — Although thin-wall liners have been used to contain the MSRE sample ladles during shipment to the High-Radiation-Level Analytical Laboratory, contamination to the inner walls of the transport container and plug containing the liner and sample ladle continued. Recently, a disposable mild-steel plug has been used to contain each sample ladle. Disposal of the highly contaminated plugs has greatly reduced the time required to decontaminate the transport containers.

Twice, difficulties were encountered during the removal of the sample ladle from the transport container. First the latch and then the stainless steel cable were wedged between the threads of the transport container and plug. Though one of the transport containers was damaged beyond reuse, the samples were removed and analyzed.

One sample was received with a silver and a Hastelloy N wire coiled onto the stainless steel cable between the latch and the ladle. The latch, wires, and cable were separated and prepared for radiochemical analyses.

Two fuel samples taken at a reactor power level of 5 Mw were analyzed and found to contain 52 and 47 ppm of oxygen, as compared with concentrations of 49 and 53 ppm determined in samples taken at startup and low power operations.

A 95.6% recovery was obtained in the analysis of an SnO_2 "standard sample" of weight corresponding to about 300 ppm of oxide in a fuel sample. The slight negative bias is attributed to momentary interruptions in the flow of hydrofluorinator effluent gas through the electrolytic moisture monitor cell. It has been found that these flow interruptions occur only when water is present and current is flowing through the cell. This indicates that hydrogen and oxygen evolving from the electrodes form, under the electrolyte of partially dehydrated P_2O_5, bubbles which bridge the capillary and form an obstructing film. Variations in the technique of depositing the electrolyte designed to form a thinner and more uniform P_2O_5 film of lower surface tension have proved unsuccessful in eliminating these interruptions. Current effort is directed toward modifying the flow system to increase its impedance and thus minimize the effect of flow interruptions.

REACTOR FUELS AND MATERIALS

Mechanical Properties Research. — Structural alloys suitable for application at elevated temperatures are being developed for resistance to elevated-temperature irradiation embrittlement. We previously demonstrated that laboratory heats of type 304 stainless steel modified with 0.2% Ti have better postirradiation ductility than unmodified material (ORNL-3896, p. 3). A finely dispersed intragranular precipitate, which prevented accumulation of helium at grain boundaries, caused the improved postirradiation ductility.

Efforts to develop a fine intragranular precipitate in two commercial heats of the modified alloy containing 0.15% Ti with 0.02 and 0.05% C met with limited success. Small strain fields appear when the material is annealed for 1 hr at 900°C; however, continued aging at 900°C or lower temperatures does not cause significant growth of these clusters. The commercial heats now appear to contain sufficient nitrogen (400 to 700 ppm) to combine with nearly all the titanium present, precipitating large Ti(C,N) particles, which are very stable and cannot be taken into solution at temperatures approaching the melting point of the alloy. Thus the amount of titanium available for the formation of fine intragranular precipitates is reduced considerably.

We are now determining (1) how variables such as composition and heat treatment affect the morphology of the precipitates, (2) how precipitate morphology affects the susceptibility to elevated-temperature irradiation embrittlement, and (3) how long-time elevated-temperature exposure affects precipitate structure.

Weldability of Nickel-Bearing Alloys. — We initiated an investigation of the weldability of nickel-bearing alloys such as Inconel 600 and Incoloy 800. The program will entail the preparation and evaluation of a large number of alloys, each of which has been selected in an effort to determine the role of certain elements on the tendency toward microfissuring. Considerable data have been reported pertaining to this subject, mostly secondary information from programs with other objectives. The data are cursory; they have shown trends but have not specifically tied down the problem. The elements that are harmful are known; they are Al, Ti, C, Mo, Si, S, P, Pb, Zr, B, O, N, and H. As can be noted from the list, many of these elements are not residuals but are sometimes added to enhance certain properties. We are determining their threshold values, seeking the mechanisms through which they operate, and developing techniques whereby their effects can be nullified.

The selection of a test for separating alloys of similar weldability is an integral part of this program. The Rensselaer Polytechnic Institute has developed a test that appears to be capable of segregating small differences in weldability by applying a strain of a known magnitude during welding. Their Varestraint Test is being evaluated under subcontract; selected heats of commercial alloys have been submitted to RPI for testing. These alloys are Inconel 600 (high nickel, 72%), Incoloy 800 (high iron, medium nickel, 33%), and Hastelloy X (high nickel, 60%, Cr, Fe, Mo). The results of the first tests have not yet been received.

REACTOR COMPONENTS

Reactor Controls Development: Neutron Fluctuation Analysis. — The recently reported success of two-detector cross-correlation analysis, as applied to the measurement of large shutdown reactivities under adverse experimental conditions via neutron fluctuation observations, has prompted us to investigate more thoroughly both the strengths and limitations inherent in the method. In particular, we wanted to ascertain that (1) the background of uncorrelated events present under high gamma-flux conditions would indeed cancel as anticipated, (2) the statistical precision obtainable would compare favorably with ordinary autocorrelation, and (3) spatial separation of the two detectors would not be a critical factor. Preliminary results from an experiment performed at the PCA verify all three conjectures, with some reservations on the second.

Reactor Controls Development: Reactor Computer Control. — The HFIR control rods must be adjusted to within approximately 1 in. of each other before reactor operation at power levels above 10 Mw is permitted. This operating restriction was imposed to prevent power distribution asymmetry and associated fuel hot spots.

When gross rod unbalance or asymmetry occurs, the operator must manually adjust the rods before the reactor can be returned to full-power operation. The manual adjustment is complicated by the normal action of the servocontroller.

A preliminary study was initiated to determine the merits and feasibility of using the on-line digital computer to perform control-rod trimming function automatically. A program was written to simulate the HFIR control system on the CDC 1604 general-purpose computer. The simulation provides solutions to reactor transient conditions and associated control actions. Several control algorithms for correcting asymmetric rod patterns were investigated. All the simulation investigated to date resulted in improved recovery times.

POWER REACTOR FUEL PROCESSING

Aqueous Process Development: General. — Development is continuing on mechanical, nonaqueous, and aqueous steps for head-end processes designed to convert power reactor fuels into aqueous solutions suitable for fuel decontamination and recovery by solvent extraction. During the past period the major efforts were on (1) further development of the grind-leach process for graphite-base HTGR fuel; (2) hot-cell studies on the burn-leach and grind-leach processes, also for graphite-base HTGR fuel; (3) chemical decladding studies on sintered-aluminum-product-clad (SAP-clad) UC fuel from the HWOCR (Heavy-Water-Moderated Organic-Cooled Reactor); and (4) leaching studies on sheared zirconium-clad ThO_2-UO_2 fuel.

Aqueous Process Development: Burn-Leach Process. — Hot-cell evaluation of the burn-leach process for graphite-base fuels was continued. In this process, HTGR fuel (pyrolytic-carbon-coated UC_2-ThC_2 particles dispersed in a graphite matrix) is crushed to −4 mesh and then burned in a fluidized bed of 60- to 120-mesh alumina using air and/or oxygen at 750°C. After burning, the ThO_2, U_3O_8, and nonvolatile fission product oxides are leached from the alumina bed with fluoride-catalyzed nitric acid to produce a feed solution from which thorium and uranium can be recovered by conventional solvent extraction.

In the current experiments, 90-g batches of coarsely crushed Peach Bottom GAIL III-A fuel irradiated to 8000 Mwd/metric ton were successfully burned in 90 min at 750 ± 25°C by using a nitrogen-air-oxygen mixture to provide controlled burning rates. Of major interest was the volatilization and entrainment of fission products during burning and their removal from the burner off-gas, which was passed through a carbon-precoated 20-μ micrometallic filter inside the top of the burner, then through main and secondary absolute filter packs. In the final burning stage, when only 10% oxygen utilization is being achieved, the off-gas downstream from the micrometallic filter was diverted through a bypass filter to determine whether excessive volatilization of fission products occurred under strong oxidizing conditions.

The results of three runs indicate that the carbon-precoated micrometallic filter performs as a nearly absolute filter for trapping fission product particulates. The following separation factors were calculated for the principal isotopes found on the absolute filter packs downstream from the micrometallic filter: ^{144}Ce, 1.6×10^7; ^{137}Cs, 1.8×10^7; ^{125}Sb, 3.7×10^5; and ^{106}Ru, 3×10^6. The radioactivity measured on the bypass filter pack was 0.5 to 0.05 of that found on the main and secondary filter packs. This indicated that excessive volatilization of fission products does not occur when the last traces of fuel are burned with a large excess of oxygen.

The bed residue, consisting of 60- to 120-mesh alumina plus ThO_2, U_3O_8, and fission product oxides, was leached for 5 hr with refluxing 13 M HNO_3–0.05 M HF, then leached 2 hr with another aliquot of the refluxing dissolvent, and finally washed three times with water. Greater than 99.8% of the uranium and thorium was recovered. Most of the thorium and uranium was found in the first leach, indicating that all the fuel particles and their carbon coatings were burned. About 0.75% of the total gamma activity remained in the alumina bed. This holdup of fission products is probably not excessive, since in plant operation most of the leached alumina will be recycled.

Aqueous Process Development: Grind-Leach Process. – Fine grinding followed by acid leaching is also being evaluated as a method for processing graphite-base HTGR fuels that contain coated fuel particles. Engineering- and laboratory-scale tests and hot-cell experiments are being made with Peach Bottom carbon-coated ThC_2-UC_2 fuel particles in a graphite matrix. The best method found for grinding HTGR fuels is rough crushing to –6 mesh in a hammer mill followed by roll-crushing to reduce the particle size to –140 mesh. Grinding to this size ensures rupture of nearly all the particle coatings. In continuing engineering tests, it was found that the 15- by 8-in. hammer mill, used for rough crushing, ruptured about 30% of the coated particles during the reduction of massive fuel to –6 mesh. Three passes of this material through the 12- by 4-in. double-roll crusher further reduced the fuel to a powder, from which 99.85% of the uranium was consistently recovered by nitric acid leaching and water washing. It was not established whether the 0.15% uranium loss results from a small number of unruptured fuel beads (diameter of ThC_2-UC_2 kernels $\cong 150\ \mu$) or from other causes. When graphite fuel containing still-smaller coated particles (diameter of UC_2 kernels $\cong 75\ \mu$) was crushed, ground, and leached in boiling 13 to 16 M HNO_3, uranium recovery was only 99.0%. This finding suggests that coated-particle fuel containing ~150-μ ThC_2-UC_2 kernels may define the lower limit for fully effective grinding in the 12- by 4-in. double-roll crusher.

Recent laboratory tests were made on the washing of the graphitic residues from leaching. Samples (2.5 g) of crushed fuel were leached for 5 hr with 10 ml of boiling 13 M HNO_3; then the contents of the flask were transferred to a 1-cm-ID filter column, where the product solution was removed and the residue was washed, using pressure filtration. Each residue occupied a volume of about 3 ml (one bed volume); the pressure gradient across the graphite bed was about 2.1 cm Hg per centimeter of bed height. In each case the first three bed volumes of effluent from the column contained the product solution.

Washing with three to four bed volumes of water, either at 25 or at 75°C, resulted in the removal of at least 99.9% of the soluble thorium and uranium. Only trace amounts of thorium and uranium were removed from the bed by washing with an additional 10 to 15 bed volumes of water. Losses of uranium and thorium to the residue after a single washing were about 0.3 and 0.5% respectively.

Washing with 10 M HNO_3 at 25°C gave results similar to those obtained with water, except that the uranium and thorium losses to the residue were 0.1 to 0.3% lower. However, washing with 10 M HNO_3 at 75°C gave markedly different results. After removal of the product solution, the remaining uranium and thorium were eluted only slowly. After three to four bed volumes of acid were passed through the column, 1 to 4% of the soluble uranium and thorium was still retained by the bed. These values were reduced to 0.2 to 1% after washing with seven bed volumes of acid. These results, although inexplicable at present, were corroborated in duplicate experiments.

Hot-cell studies of the grind-leach process were begun by using Peach Bottom GAIL III-B fuel compacts irradiated to 55,000 Mwd/metric ton of uranium plus thorium. These experiments are designed to determine how completely uranium, thorium, and, particularly, fission products can be leached and washed from this fuel. With a modified Waring Blendor, 40 g of −4 mesh fuel was pulverized to −100 mesh in 25 min. The material was sieved to ensure that all the fuel particles were broken. Ten-gram samples were then leached for 5 hr in refluxing 13 M HNO_3–0.05 M HF, leached for an additional 2 hr with a second aliquot of the boiling dissolvent, and washed five times with this same reagent at 25°C. Companion samples were leached and washed in the same manner with 13 M HNO_3 for comparison purposes. Recovery of uranium and thorium was incomplete in all of these tests. Up to 3.29% of the thorium and 2.13% of the uranium were retained in the graphite residue; analyses of subsequent wash liquors indicated that additional washing would not reduce these losses. In previous tests with unirradiated Peach Bottom fuel of approximately the same composition, uranium and thorium retained in the graphite residue averaged 0.01 and 0.002% respectively. Significant amounts of fission products also resisted leaching and washing; from 2.5 to 9% of the total gamma activity, mainly ruthenium, remained in the residues. Additional hot-cell experiments are planned.

Aqueous Process Development: HWOCR Fuel Processing. — Methods for processing fuels for heavy-water-moderated organic-cooled reactors (HWOCR's) are being developed on a laboratory scale. The HWOCR fuel presently receiving the most attention is UC clad in SAP (a sintered dispersion of Al_2O_3 in aluminum). A process that appears feasible for this type of fuel consists in removing the SAP cladding by dissolving it in NaOH solution and then dissolving the hydrous UO_2 residue in HNO_3 in preparation for decontamination and recovery of the uranium by solvent extraction. This method has the advantage that, during the decladding step, the UC is hydrolyzed, releasing most of the carbide carbon as methane. Thus, only traces of organic compounds are found in the solvent extraction feed solution.

Simulated fuel (UC:SAP weight ratio about 10) was digested in boiling 2 M NaOH (initial NaOH:aluminum mole ratio about 4). After 6 hr, 91.2% of the aluminum from the SAP was dissolved; the uranium loss to the solution was only 0.004%; and hydrolysis of the UC was nearly complete. About 8% of the carbide carbon was found as soluble species in the resultant solution. The residue from the decladding step was almost completely dissolved in 5 hr in boiling 5 M HNO_3 to produce a solution that was about 0.5 M in uranium. This solution was slightly cloudy, possibly as a result of a suspension of some undissolved Al_2O_3 from the SAP, and contained only about 1.4% of the carbide carbon as soluble species. Only a trace of unidentified residue remained.

Aqueous Process Development: Shear-Leach Process. — The shear-leach process for multitubular stainless-steel- or Zircaloy-clad UO_2 and ThO_2-UO_2 fuels involves transverse shearing of the fuel bundle into 1- to 2-in. lengths followed by leaching of the exposed fuel oxide in HNO_3. Recent tests were made on the leaching of sheared Zircaloy-2-clad ThO_2-UO_2 fuel. The leachant for this fuel is 13 M HNO_3–0.05 M NaF–0.05 to 0.1 M $Al(NO_3)_3$. The ThO_2-UO_2 is almost completely dissolved in this reagent; however, some of the Zircaloy is also dissolved. The latest tests were made to determine (1) the extent of Zircaloy dissolution; (2) the effect of the dissolved Zircaloy on subsequent feed adjustment and solvent extraction; and (3) the fate of –40 mesh Zircaloy shearing fines, the size range for potential fire and explosion hazards.

Four leaching runs were made with sheared Zircaloy and sol-gel-derived ThO_2-UO_2. Run times and leaching data for thorium and zirconium were as follows:

Run	Leach Time (hr)	Product (g/liter)		Undissolved Th (%)	Dissolved Zr (%)	–40 Mesh Zr in Leached Residue (%)
		Th	Zr			
SG-6	7	318	0.58	2.5	0.43	0.053
SG-7	7	120	1.3	0.17	2.82	0.047
SG-8	7	120	0.78	0.11	1.67	0.052
SG-9	24	264	1.50	0.14	1.43	0.013

Even at this low concentration, the solubilized zirconium is expected to present a problem by precipitating during the conversion of the leachant to an acid-deficient solvent extraction feed. The precipitate would contain highly radioactive zirconium and thus present a potential heat problem. Zircaloy dissolution was suppressed by higher thorium and aluminum concentrations. Only 0.05% of the Zircaloy cladding was present in the potentially hazardous –40 mesh size range after 7 hr of leaching; only 0.01% was present after 24 hr. Thorium losses during a leach time of 7 hr were lower than expected for sol-gel material.

Fluoride Volatility Processing: Treatment of Zircaloy-Clad Fuel. — The two current methods for processing Zircaloy-clad UO_2 have disadvantages. The better of these uses HCl to form volatile $ZrCl_4$. However, this method requires a separate reactor for disposal of the $ZrCl_4$ — a distinct disadvantage in the planned pilot plant, where space is at a premium. The other process uses HF-O_2 mixtures to form ZrO_2. Here the acceptable fuel load is quite small, because of lumping of the UO_2 when it is constricted in the presence of HF-O_2 mixtures. It is estimated that the fuel-to-bed-area ratio should not exceed 0.1. Since neither of these processes is ideal, alternative methods of processing have been investigated.

One such method consists in hydriding zirconium with dilute hydrogen at about 600°C to fracture the cladding, oxidizing at about 450°C to disperse the UO_2 as U_3O_8, completely pulverizing the cladding by treatment at 450°C with HF-O_2 mixtures, and fluorinating at 450 to 550°C to volatilize the uranium and plutonium. This program was carried out in a 0.9-in.-ID fluidized-bed reactor without difficulty. In this test the fuel occupied about 43% of the cross-sectional area of the bed. This is about four times the fuel charge possible with the HF-O_2 process. Only a single reactor is required, since the zirconium remains as a part of the fluidized bed.

The hydriding reaction proceeds readily at temperatures above 560°C in the presence of solid fluorides such as NaF, LiF, or ZrF_4, or if a small amount of HF is used in the initial period. The hydriding reaction is exothermic, and effective penetration rates of 80 to 160 mils/hr are attainable. The density of the Zircaloy is reduced to about 5.65 g/cm^3 after the reaction, and the hydrided material is severely cracked. These cracks allow penetration of oxygen in the second step, so that the UO_2 is oxidized to a fine U_3O_8 powder. This oxidation step has little effect on the zirconium hydride. The reaction of zirconium hydride with HF-O_2 mixtures (cladding pulverization) is very vigorous and exothermic, but can be easily controlled by diluting the gas with nitrogen and by using only about 5 to 10% HF in the mixture. The HF in the mixture does not escape from the fluidized bed, but reacts with the fine U_3O_8 to form UF_4. The final fluorination reaction is very smooth and requires only a relatively small amount of fluorine, since the uranium is already converted to UF_4. The presence of ZrF_4 in the bed does not result in losses of uranium greater than those observed with an alumina bed.

In addition to the reactions involved in the alternative decladding scheme, we also found that HF-H_2O and H_2-H_2O mixtures react with Zircaloy at about 600°C, yielding, primarily, ZrO_2. However, the ability of the steam to pyrohydrolyze the fluorides in a fluoride bed, giving oxides and HF, must be considered in any future use of these mixtures.

The decladding scheme, as described above, appears to be potentially valuable and will be explored further in a short series of additional fluidized-bed tests.

Fluoride Volatility Processing: Corrosion Problems Related to the FBVPP. — More results are available from studies to determine the cause of intergranular modifications sometimes found in nickel after exposure to conditions of the fluidized-bed volatility process. This work is

being done at the Columbus Laboratories of Battelle Memorial Institute under subcontract. As previously reported (ORNL-3968, p. 7), sulfur or other impurities in some brands of alumina, the fluidized-bed medium, seem to be responsible.

Recently, nickel 200 and 201 specimens were exposed, in an evacuated Vycor capsule, to four types of alumina for 200 hr at 520°C. At 25-hr intervals, fresh alumina was added. Earlier tests had included continuous exposures for 25, 50, 100, and 200 hr. For exposures of nickel 201 to three Norton Alundums, Regular, 38, and RR, and to Alcoa T-61 alumina, penetrations (in mils) after 25 and 200 hr were, respectively, 7.5 and 20.0, 4.0 and 19.0, 1.0 and 4.8, and 0.2 and 6.0. Penetrations in nickel 200 specimens were nearly identical. (Modifications are visible when the specimens are examined metallographically after etching with 1:1 HNO_3-CH_3COOH.) Results are consistent with sulfur analyses previously reported, except for the high penetration rates in the presence of Norton 38, advertised and analyzed as a relatively pure alumina.

To determine the degree of harmfulness of the modifications to nickel, small tensile-test specimens were exposed to Norton 38 Alundum in evacuated capsules in two groups (in triplicate for each condition). After 25 hr at 520°C, the specimens of the first group showed modifications to a nominal depth of 5 mils (10 mils, total). The second group was exposed to fresh alumina six times for a total exposure of 350 hr at 520°C. The nominal penetration was 20 mils (a total of 40 mils of the 55-mil-thick specimens). Tensile tests were made at room temperature and at 520°C. Base-line data for specimens not exposed to the alumina were obtained from tensile tests of as-received specimens and after exposure in vacuum at 520°C for 350 hr.

The intergranular modifications significantly affected strength characteristics. For example, specimens exposed for 350 hr and pulled at room temperature showed reductions of 58 and 88%, respectively, for ultimate tensile strength and elongation; corresponding values for specimens pulled at 520°C were 31 and 91%.

Also at BMI, more corrosion tests were made of metals being considered in the construction of a waste-solids leacher for the FBVPP. First results were reported earlier (ORNL-3968, p. 8). Because chromium was highly resistant to boiling 13 M HNO_3–0.1 M HF (a likely leachant composition), Blaw-Knox alloys NA 55 and NA 64 (50% Cr–50% Ni and 60% Cr–40% Ni) were tested in this solution. Unwelded specimens corroded at 5.6 and 8.0 mils/month, respectively, in a 24-hr test. Except for the value of 3.9 mils/month for chromium (not fabricable), lowest values obtained previously were about 17 mils/month for Hanford alloy 20 and Corronel 230. Type 309SCb stainless steel corroded at 80 and 90 mils/month, with knife-line attack at the weld–base metal junction.

In tests using a boiling solution of 13 M HNO_3–0.1 M HF to which 0.1 M $Al(NO_3)_3$ had been added, rates of attack in 24 hr were 1/4 to 1/8 lower than without the $Al(NO_3)_3$. The following corrosion rates (mils/month) were noted: alloys NA 55 and NA 64, 1.7 and 2.0; Hanford alloys 17, 20, and 21, 4 to 5; and type 309SCb stainless steel, 10.9 to 17.4. Tests will be made to determine the fabricability of Blaw-Knox alloys NA 55 and NA 64.

Fluidized-Bed Volatility Pilot Plant. — The FBVPP will be installed in cells 1, 2, and 3 of Building 3019 to study the processing of spent UO_2 power reactor fuel clad in either Zircaloy or stainless steel. The status of the flowsheet was reported previously (ORNL-3968, p. 8).

New percentage-completion figures for process equipment are: primary reactor, 52; pyrohydrolyzer, 95; reactor gas cooler, 18; two NaF traps, 75; NaF trap outlet filter, 90; and two cold traps, 85.

Design of the reactor-gas cooler was modified by removing some of the internal fins to lessen the possibility of fines retention. Based on favorable results from tests of sintered-nickel-fiber filters at ORGDP, the total number of filter tubes necessary for the primary and backup filters needed for the primary reactor and the pyrohydrolyzer was reduced. The smaller number of tubes made it possible to include the primary and secondary filters in the primary filter shell in both cases. Difficulties in replacing filters will, therefore, be halved. Drawings of the revised filters were issued for approval.

Piping drawings for head-end equipment were also issued for approval.

Drawings for one assembly of several in progress for waste-solids handling equipment were issued for approval. Design of the ball valve for controlling flows of waste solids under the primary reactor and pyrohydrolyzer was completed.

Preparation of cell 3 continued slowly. About three months has been required as compared with the one month originally estimated. Drilling of four 12-in., three 18-in., and two 8-in. holes through 5 ft of concrete was finished, after many delays caused by equipment failure, the transition between the cost-plus-fixed-fee contractors, and a brief labor stoppage. Installation of curbing to support shielding at the lowest level has begun.

HIGH FLUX ISOTOPE REACTOR DESIGN AND DEVELOPMENT

Reactor Analysis. — During May 1966 the Advisory Committee on Reactor Safeguards (ACRS) gave its approval for the HFIR to proceed to 100 Mw. Previous to this time the reactor was operating at 20 Mw with the ORNL "first production" fuel loading. Shortly after ACRS approval was received the power level was raised to 50 Mw, and at or from this level various operational tests, such as "flow-coastdown," scram, and vibration (ion chamber output), were conducted. No significant difficulties were encountered. After accumulating ~2230 Mwd, the core expired on June 19; the burnup was 10 to 20% greater than anticipated.

The next fuel loading installed was the Metals and Controls (fuel element commercial fabricator) "second production" fuel loading, which is now operating without difficulty at 75 Mw. It is expected that the burnup of this core will be essentially the same as the previous one, although the M&C core has a little less burnable poison, and the poisons in the beryllium reflector are increasing.

A recent review of the HFIR fuel element heat transfer analysis and results from continuing experiments with fuel plate deflection characteristics indicates that there is no apparent reason for not operating the

HFIR at 100 Mw. The reason for the latest review was associated with uncertainties regarding previous fuel plate deflection experiments, particularly those involving differential thermal expansion. Our latest results indicate that no axial instability exists under HFIR operating conditions; thus the present overall heat removal analysis looks more favorable than the original, which considered some axial buckling of the fuel plate. Experiments will continue for the purpose of discovering the conditions which will produce buckling and thus for establishing our margin of safety.

Development studies associated with longitudinal spacers in the fuel element coolant channels are continuing. Thus far, spacers have been attached to fuel plates (single plates) using an ultrasonic welder, and they have also been installed in a partial element by drawing techniques. A partial element containing the welded spacers and another containing the drawn spacers have been flow tested; no significant deleterious effects were observed. Further partial elements will be tested to determine the effect of clearance between spacer and plate. It is not anticipated at this time that the spacers will be required in the present fuel element design. However, should difficulties arise and/or if the design is modified to permit a longer cycle time, it might be desirable to install the spacers.

Core Transient and Noise Analysis. — Efforts are continuing toward obtaining proper weighted cross sections necessary to describe, with a "small model," the reactivity behavior of the HFIR. Several MODBURN calculations were performed to obtain curves of reactivity vs operating time for each isotope of interest. These curves are now available for ^{135}Xe, ^{149}Sm, ^{235}U, B, and gross fission products. Additional MODBURN runs have been made by others and will be used for transient analysis.

The acoustical background noise of the HFIR at different pressures and flows was recorded on magnetic tape for future analysis. Recordings were made with a microphone placed at three different locations to ascertain the best location for future acoustical measurements.

Three HFIR cores were evaluated (prior to installation in the reactor), using neutron fluctuation techniques, to measure their subcritical reactivity when immersed in water. The results of these tests were in agreement with measurements made using a different technique.

Neutron fluctuation measurements were made at the HFIR during the initial increase of power to 50 Mw to detect any unusual resonances in the frequency spectrum of the neutron density fluctuations. Resonances were present at 11 and 18 cps. These resonances are not unusual, in that similar results have been obtained at other reactors.

Component Development. — The HFIR target tower and target holder assemblies have shown excessive wear on their seating surfaces, due to vibration and rotation of the target holder and the target tower at HFIR design flow rates. This wear apparently resulted from movement of the target tower relative to the target holder at the lower end and relative to the target tower spider at the upper end. The movement has apparently occurred due to the lack of a positive net downward force on the target tower during operation. The target tower spider has been redesigned and equipped with springs to provide a net positive downward force of 200 lb

in addition to the weight of the target tower and spider assemblies. The target holder has been redesigned to incorporate features found to be desirable during remote maintenance practice. The outer shroud flange was redesigned to provide attachments for target tower stabilizers, if required, and to provide more structural integrity for the outer shroud flange orifices and flow diffusers.

Reactor components have been fabricated and tested to determine whether the design of the target tower spider, the target tower, the target holder, and the outer shroud flange is satisfactory. A group of tests have been conducted on the target tower to determine whether vibration and rotation of the tower have been eliminated. Accelerometers were mounted inside the lower end of the target tower and at a point 46 in. above the lower end of the target tower. This installation was designed to measure movement of the target tower in the x, y, and z planes. The equipment was installed in the reactor, and tests were run using various pump combinations and operating pressures that are commonly used during normal reactor operations. The maximum movement recorded during these tests was 0.0015 in., which is quite acceptable. These tests indicated that no improvement in the target tower performance can be expected if stabilizer bars are added, and demonstrated that the redesigned components perform satisfactorily under all expected normal reactor operating conditions.

Reactor Operations. — The reactor has operated very smoothly during this period. The power level was increased to 75 Mw on June 30, and since then the reactor has operated uneventfully at that level with no shutdowns. The total energy accumulated to date is about 4000 Mwd.

Testing of the new 1-1/8-in.-thick charcoal filters in the special building hot exhaust system was completed. The factor for decontamination of all banks for both elemental iodine and methyl iodide was measured as greater than 3000. It is planned to replace the canister-type filters in the off-gas systems with the 1-1/8-in.-thick pleated-type filters.

Two more flux mapping runs were made at ~1 Mw to measure the neutron flux in the target region and in some of the vertical experiment holes.

Eleven fuel elements have been received from the fuel element fabricator.

PHYSICAL RESEARCH

Reactor Operations. — Both the ORR and the LITR operated approximately 90% of the time during May and June.

At the ORR, neutron flux monitors for determining the energy spectrum were placed in several of the irradiation facilities to check their activities against calculated values. The objective of this program is to establish a procedure which will be useful to experimenters in the future.

An experiment for measuring the electric dipole moment of the neutron was installed in horizontal beam hole No. 2 at the LITR.

The Bulk Shielding Reactor operated for approximately 155 hr as required during this period. By the end of June, revisions of the reactor for 2-Mw operation were approximately 59% completed.

Approximately 30% of the fuel in the ORNL Graphite Reactor had been unloaded and shipped at the end of June.

Waste Disposal. — Work has begun on the permanentization of the Hydrofracturing Plant to permit routine disposal of radioactive liquid wastes. The first injection of evaporator concentrated waste is tentatively scheduled for November 1966. The size of the injection will be determined by the capacity of the waste storage tanks located at the plant site. If the present capacity of 40,000 gal cannot be increased in time for the first injection, two 40,000-gal injections will be made. If two surplus tanks, which are now available, can be installed in time, it may be possible to make a single 80,000-gal injection.

Hot-Cell Operations. — Three capsules containing pyrolytic-carbon-coated UO_2 particles were disassembled and examined in detail. Seven capsules containing material being studied as possible high-temperature reactor fuels were examined extensively; examination included gas sampling, gamma scanning, visual examination, photography, physical dimensioning, weighing, density determination, and sampling for metallography and burnup analysis. Five fueled capsules were examined for General Electric.

Two in-pile UO_2 meltdown experiments were examined; NSPP meltdown No. 14 was disassembled, and the filters from this experiment were disassembled and sampled for analysis. Examination of the fuel tubes and fuel plates from an Army PM fuel test was completed. Several isotope source capsules, empty or containing alpha sources, were examined metallographically. About 300 tensile specimens were destructively tested; for this the Instron machine in cell A, Building 3026D, was operated on the 4 to 12 shift about 50% of the time (in addition to full operation on the day shift). The eight in-cell creep-test machines continued in full operation.

A remotely operated mercury pycnometer for accurately determining the volume of small irradiated pellets was developed. The repeatability has been found to be ±0.15% on standards of about 0.008 in.^{3} in size.

PHYSICS AND MATHEMATICS

Theoretical Physics: A Smooth Velocity-Dependent Potential and Nuclear Matter. — A velocity-dependent potential of the form

$$V(r,p) = -V_1(r) + p^2 e^{-s^2 p^2} V_2(r) + V_2(r)\, p^2 e^{-s^2 p^2} ,$$

where $V_1(r)$ and $V_2(r)$ are Gaussians, is used to fit the singlet (0 to 320 Mev) phase shifts for elastic nucleon-nucleon scattering. This exponential velocity dependence replaces the hard core by a short-range repulsion which is much softer than the case of quadratic ($s = 0$) velocity dependence used by Green, Levinger, et al. The two-body scattering problem is solved in momentum space by numerical summation of the Born series; the $^1S_0$ scattering length is calculated separately by a rapid matrix inversion method. The applicability of ordinary many-nucleon perturbation theory for this interaction is tested by calculation of the first-order (P_1) and second-order (P_2) potential energy per particle of nuclear matter. A rapid singlet-even convergence rate of $P_2/P_1 = 4.3\%$ at $k_F = 1.5$ F follows, as a result of the reduced off-energy-shell matrix elements of this two-body interaction. One also finds qualitative agreement with the singlet-state potential energy per particle obtained by Sprung et al. in their complete nuclear matter calculation. The harmonic oscillator matrix elements required for Hartree-Fock calculations of spherical nuclei are evaluated quite simply for this potential. Also, since this potential readily separates into relative x, y, and z coordinates, it is well suited for Hartree-Fock calculations of deformed nuclei.

Low-Energy Neutron Time-of-Flight Spectroscopy. — Angular distribution measurements using a 6-cm^3 lithium-drifted germanium crystal have been made of the high-energy gamma rays from the capture of resonance energy neutrons in samples enriched in ^{118}Sn, ^{122}Sn, and ^{124}Sn. If p-wave neutrons are captured to form a $3/2^-$ state, the angular distribution of the transition to the low-lying $1/2^+$ state will be peaked at 90°, while the transition to the low-lying $3/2^+$ state will have a minimum at 90°. The ratio of the intensities of these two high-energy gamma rays will change by a factor of 2 (assuming pure E-1 transitions) from 90 to 135°. The measured ratios changed by factors of 1.43 ± 0.12 for ^{118}Sn and 1.5 ± 0.3 for ^{122}Sn. Since s-wave capture, and p-wave capture to form a $1/2^-$ state, must be isotropic, there must be resonance capture forming $3/2^-$ states in both ^{118}Sn and ^{122}Sn. For ^{118}Sn about half of the resonance capture is in the 45.8-ev resonance and about half is in the 359-ev resonance, which is s wave. Other measurements using a manganese filter to remove the contribution from the 359-ev resonance yielded a change in the ratio by a factor of 1.59 ± 0.15. Hence the 45.8-ev resonance must be p wave with spin $3/2^-$. For ^{122}Sn, resonance capture is due principally to the 107- and 260-ev resonances, and we can conclude only that one of these resonances is p wave with spin $3/2^-$. For ^{124}Sn the ratio of the two gamma rays did not change with angle, which agrees with results of McNeill, McConnell, and Firk [Can. J. Phys. 43, 2156 (1965)]. Since the resonance capture is mainly from the 62-ev resonance, which is known to be a p-wave resonance, the capturing state must be $1/2^-$.

Low-Temperature, Nuclear and Solid-State Physics. — The ordered alloys AuMn, Au_2Mn, and Au_3Mn have been studied using the Mössbauer effect of ^{197}Au at 4.2°K. In contrast to our previous results with other gold alloys, the Mössbauer isomer shift for the gold nuclei is not linear with composition. The shifts (in mm/sec) as measured with respect to gold are:

AuMn, 4.553 ± 0.014; Au_2Mn, 2.808 ± 0.016; and Au_3Mn, 1.777 ± 0.013. The recoilless radiation spectrum indicates very little, if any, splitting of the ^{197}Au nuclear states for AuMn. The Au_2Mn spectrum corresponds to a spin Hamiltonian for the ^{197}Au ground state (I = 3/2) of

$$H = 2\mu H_x I_x/3 + P[3I_z^2 - I(I + 1)]/2 ,$$

where $H_x = (1.650 \pm 0.01) \times 10^6$ gauss and $P = (9.23 \pm 0.4) \times 10^{-4}$ cm^{-1}. The interaction in Au_3Mn is predominantly magnetic, the field being $(8.50 \pm 0.1) \times 10^5$ gauss. These results may be correlated with the crystal structure of the alloys.

High-Voltage Experimental Program: Differential Scattering of Neutrons from Oxygen and Intermediate States. — The resonance structure of neutron scattering from ^{16}O between 3 and 4 Mev is of particular interest in connection with explanations of intermediate states. There are several resonances of rather large reduced widths [Bull. Am. Phys. Soc. 10, 261 (1965); Lister and Sayre, Phys. Rev. 143, 745 (1966)]. The narrow peak at 3.77 Mev, however, has created a problem in the phase shift analysis of scattering data in this energy region. Fossan et al. [Phys. Rev. 123, 209 (1961)], from total cross-section measurements, found that the height of this peak corresponded to a J = 5/2 resonance of 25-kev width. But in ^{16}O proton scattering, the ^{17}F level whose excitation corresponds closely to this one is a $J = 7/2^-$ resonance [Phys. Rev. 126, 2147 (1962)]. We have remeasured the total cross section in good geometry with 5-kev resolutions in the vicinity of 3.77 Mev. Assuming that the 3.77-Mev peak is a single resonance and correcting for the experimental energy resolution of 5 kev, we find that Γ = 19 kev, which is slightly less than, but consistent with, the 22 kev reported by Walton et al. [Phys. Rev. 107, 1067 (1957)], and the 25 kev found by Fossan et al. With a $J = 5/2^-$ assignment, we got a reasonable fit to an angular distribution at the 3.77-Mev peak [Bull. Am. Phys. Soc. 10, 261 (1965)]. However, as Lister and Sayre have pointed out, we cannot fit angular distributions on either side of the resonance with a consistent set of phase shifts and a $5/2^-$ or $5/2^+$ assignment. In a recent analysis we have arrived at a solution to this problem. There is a narrow, ~3-kev, $J = 3/2^+$ resonance under an ~17-kev $J = 7/2^-$ resonance. The large $d_{3/2}$ background phase shift makes the narrow $d_{3/2}$ resonance show up as a dip, thus reducing the peak height in the total cross section. These assumptions give a satisfactory fit to our angular distributions measured near 3.77 Mev.

High-Voltage Experimental Program: Feasibility Study of a Gamma-Ray Spectrometer for Astronomical Investigations. — We are investigating the possibility of building a heavily shielded gamma-ray spectrometer with an angular aperture of a few degrees, a relatively large sensitive area, and good energy resolution (less than 10 kev). The most promising design, now being actively pursued, uses a matrix of Ge(Li) detectors totaling up to 800 cm^2. The aperture/collimator consists of lead or bismuth (low neutron capture and inelastic scattering cross section) surrounded by a plastic scintillator that gives an anticoincidence signal when struck by energetic particles.

The device should have sufficient sensitivity (if placed in a low earth orbit) to detect line gamma-ray fluxes of $\sim 10^{-5}$ cm^{-1} sec^{-1}, less

than the flux predicted for several radioisotopes remaining in the crab nebula. The detector should also be capable of detecting gamma rays from $H(n,\gamma)$ in solar flares.

One of the major uncertainties to be explored in the immediate future is the feasibility (both technical and economic) of producing the required volume of Ge(Li) detectors and placing them into an array that results in good peak efficiency and resolution.

High-Voltage Experimental Program: Cross Sections for Analog Resonances in ^{75}As, ^{111}Cd, and $^{113}Cd(p,n)$ Reactions. – Total (p,n) cross sections have been measured to an accuracy of ±10% near analog resonances for targets of ^{75}As, ^{111}Cd, and ^{113}Cd; target thicknesses were, respectively, 5, 26, and 13 kev. Resonances observed at bombarding energies of 3059 ± 6 and 3101 ± 6 kev for ^{75}As are interpreted as the analogs of the 2^- ground state and of a previously unreported 41-kev state of ^{76}As. The cross sections at resonance are 14 and 17 mb; the off-resonance cross section is 9 mb. Each resonance has a natural width of 11 kev. Resonances observed at 4022 ± 20 kev for ^{111}Cd and at 4300 ± 8 kev for ^{113}Cd are identified as the analogs of the ground states of ^{112}Cd and ^{114}Cd. The ^{111}Cd resonance has a natural width of 25 kev and a maximum cross section of 4.6 mb; the corresponding values for ^{113}Cd are 28 kev and 10.6 mb. The cross sections at resonance for ^{111}Cd and ^{113}Cd are 1.5 and 1.6 of the nonresonant cross sections. Analogs of the first excited 2^+ states of ^{112}Cd and ^{114}Cd are observed as weak resonances at 4639 ± 20 kev for ^{111}Cd and at 4857 ± 12 kev for ^{113}Cd. These resonances have a width of about 35 kev and are approximately 10% of the nonresonant cross section.

Spectroscopy Research. – As the first phase of a program to calculate energy levels and g values of the spectra of tellurium and polonium, calculations have been performed for the fundamental configuration p^4 of Te I and Po I. The equations given by Condon and Shortley for the configuration p^4 in intermediate coupling were used. By variation of the parameters (F_0, F_2, and ρ), these equations were fitted to the known energy levels; then F_0 was adjusted to make the mean deviation zero, and the root mean square deviation was calculated. This gave 150 cm^{-1} for Te I and 19 cm^{-1} for Po I. Then the g values were calculated, using as the transformation matrix that required to diagonalize the energy matrix. The results are shown in the table:

	Te I	Po I
$3P_2'$	1.466 (1.4618)	1.399 (1.3961)
$3P_1$	1.501 (1.49)	1.501
$1D_2'$	1.035	1.102

For comparison some experimental values are included in parentheses. Those given to five figures are from Axensten et al. as determined by the atomic-beam magnetic resonance method; that given to three figures is from Green, determined by the Zeeman effect. These results cast doubt on the validity of the analysis of Te I, since a preliminary calculation indicated

a root mean square deviation of 35 cm^{-1} for Se I. The deviation for Te I would be expected to be smaller than 150 cm^{-1}.

Programming Research: A Velocity-Dependent Potential for Nuclear Matter Calculations. — To avoid the difficulties in nuclear matter calculations with potentials having a "hard core" infinite repulsion near the origin, Green [Nucl. Phys. 33, 218 (1962)] has used a velocity-dependent potential of the form

$$V_{VD}(r) = W(r)\, p^2 + p^2\, W(r) \ ,$$

where p is the relative momentum operator and W(r) is a central repulsive potential which is everywhere finite. A series of programs has been written in FORTRAN-63 for the CDC 1604 computer to obtain the parameters of potentials containing a velocity-dependent potential in addition to central, spin-orbit, and tensor components. Potentials have been obtained which give fairly good fits to recent two-body scattering data [Arndt and MacGregor, Phys. Rev. 141, 873 (1966); Thorndike et al., University of Rochester report UR-875-126, Contributions to the Williamsburg Conference on Intermediate Energy Physics, Feb. 25, 1966] and the binding energy and quadrupole moment of the deuteron.

Programming Research: Power of Tests for Small Samples from Multinomial Distributions. — The exact power of the likelihood-ratio test criterion ($-2 \log \Lambda$) and Pearson's χ^2 statistic for a simple hypothesis (1/3, 1/3, 1/3) against a simple alternative (0.3, 0.5, 0.2) up to samples of size 190 have been computed. These were compared with the asymptotic power based on the noncentral chi-square distribution in order to study the rapidity of convergence.

Schaffer [Mitt. Math. Statist. 8, 27 (1957)] compared the power of these two tests for a fixed sample size n = 10 for a simple hypothesis against a simple alternative in 15 cases. Based on these examples he concluded that the minimum power function of the likelihood-ratio test is always greater than the minimum power of the chi-square method for a fixed hypothesis against varying simple alternatives. General evidence of this conjecture was not confirmed in the present study.

In his paper on the asymptotically optimal tests for multinomial distributions, Hoeffding [Ann. Math. Statist. 36, 369 (1965)] studied the set of conditions under which the likelihood-ratio test is more powerful than the chi-square test when n is sufficiently large, and indicated the need for small-sample studies. Such small-sample studies are being carried out for trinomial and quadrinomial distributions.

Programming Research: FORTRAN for the IBM 360/75. — The first phase of development on a FORTRAN compiler for the IBM 360/75 has been completed. The compiler will essentially accept statements written in either Control Data FORTRANs or IBM FORTRANs or a mixture of these. Correct object decks are produced. The second phase of development will be to "optimize" the object deck output with a view to improving the present rate of gain over the Control Data 1604. At present, computer-bound programs on the 360/75 run approximately six times as fast as the same programs running on the Control Data 1604. It is expected that this factor will be improved to 10 during the second phase.

Programming Research: Computer Operations. — The ORNL Computing Center provides computation service for the Laboratory through the use of two large-scale digital computer systems, the Control Data 1604-A and the IBM System/360, model 75. The Control Data 1604-A is operated around the clock seven days a week. Over 8400 jobs, requiring 610 hr of computing time, were processed on this computer during the month of June 1966. The IBM System/360 model 75 computer system was turned over to ORNL on March 10, 1966, and passed its acceptance test on May 9, 1966. Productive work was begun almost immediately using a FORTRAN compiler written by the ORNL Systems Programming Group. The work load on this computer increased very quickly, to the extent that during the month of June 1966 over 1800 jobs, requiring more than 500 hr, were processed.

CHEMISTRY

Nuclear Chemistry: Neutron Cross Sections. — Cross-section measurements have been carried out on the 36-hr fission product ^{105}Rh. By irradiating samples of ^{104}Ru using cadmium filters and mass spectrographically examining the ^{105}Pd and ^{106}Pd formed, it was possible to obtain a measure of the capture cross section of ^{105}Rh. A thermal cross section of 14.4 ± 1.5 kilobarns and a resonance integral of 17 ± 3 kilobarns were found.

A measurement has been made of the cross section of the 7.5-day ^{111}Ag by examining the 3.2-hr ^{112}Ag formed during neutron irradiation, using a cadmium filter technique. A thermal cross section of 3.2 ± 2 barns and a resonance integral of 105 ± 20 barns were obtained.

Isotopically enriched samples of ^{110}Cd, ^{111}Cd, and ^{112}Cd have been irradiated to an integrated flux-time of $\sim 3 \times 10^{20}$ neutrons/cm^2 in a position where the ratio of thermal flux to resonance flux per unit lethargy was 8.6. By mass spectrographically examining the cadmium product following irradiation, effective cross sections of 11.0 ± 1 barns for ^{110}Cd, 24.3 ± 3 barns for ^{111}Cd, and 2.2 ± 0.5 barns for ^{112}Cd can be reported.

Nuclear Chemistry: Fission Studies and Characterization of Low-Energy Properties of Nuclei. — The energies of the two gamma rays emitted in the decay of ^{98}Tc were determined with a Ge(Li) detector to be 652.3 ± 0.4 and 745.6 ± 0.7 kev. The two gamma rays were observed to have equal intensities and to be in coincidence with each other. The two gamma rays also were found to occur in coincidence with the same beta group of end-point energy 397 ± 22 kev and absolute intensity 100%.

The half-life of ^{98}Tc was redetermined; the weight of a mass-analyzed sample of technetium was determined spectrochemically, and the activity was measured by gamma-ray spectrometry. The new value of the half-life so determined is $(4.2 \pm 0.3) \times 10^6$ years.

A determination was made of the ^{98}Tc formed in large samples of fission product ^{99}Tc. The ^{98}Tc content was corrected for the estimated yield arising from the ^{99}Tc(n,2n)^{98}Tc reaction. The value of the independent fission yield of ^{98}Tc, corrected for the (n,2n) contribution, was found to be $(8.6 \pm 1.2) \times 10^{-7}$%. This result leads to a fractional chain yield of ^{98}Tc in thermal-neutron fission of $(1.5 \pm 0.2) \times 10^{-7}$.

Crystal Chemistry of Transuranic Fluoride Compounds. — The tetravalent state of protactinium is expected to be the stable form of this element when it is produced by the interaction of neutrons with ^{232}Th in a molten fluoride breeder blanket mixture. It is important, therefore, to determine the crystal properties of PaF_4 and of complexes that may be formed by this compound in fluoride mixtures.

A 17 M HF solution containing approximately 90 mg of ^{231}Pa, purified by passage through an ion exchange column, was evaporated to dryness in a platinum crucible. The weight of the dry residue obtained corresponded very closely to the weight calculated for the compound Pa_2OF_8, but the identity of the phase was not verified by other means. The crucible was placed in a flanged nickel container and heated at a maximum temperature of 500°C in a mixture of hydrogen and anhydrous HF. The brown hygroscopic material produced by this treatment weighed 119 mg (theoretical for PaF_4 = 120 mg). A small amount of material of high refractive index was observed in this preparation, indicating the probable presence of an oxide impurity. Consequently, this material was heated with ammonium bifluoride, and, after this compound decomposed and evaporated, the crucible and its contents were heated in an HF-H_2 mixture at 600°C. Microscopic examination of the product, which is presumed to be PaF_4, revealed it to be single-phase material with an average refractive index higher than that of the isomorphous compound UF_4, but the crystal size was too small to permit a precise determination of its optical properties.

Organic Chemistry: Organic Compounds in Nuclear Technology. — Continued study of 2-ethylhexanehydroxamic acid as an extraction solvent has revealed a high distribution coefficient in favor of the organic phase (0.2 M hydroxamic acid in chloroform) for Fe(III). Equilibrium measurements can be represented by the equation

$$Fe^{3+}(aq) + 3HA(org) \rightleftharpoons FeA_3(org) + 3H^+(aq) \ .$$

Studies are being continued with this interesting complexing agent.

Electrochemical Kinetics and Its Application to Corrosion. — A complete interpretation of the kinetics of the dissolution of zirconium metal in aqueous solutions of HF requires a knowledge of the value of the diffusion coefficient of HF. Because reliable values of this diffusion coefficient were not available in the literature, measurements were undertaken by use of the open-ended capillary method. This method was chosen because it is the most accurate method available in which the apparatus can easily be constructed of plastics which are resistant to attack by HF. The average of eight determinations gave a value of $D = 2.17 \times 10^{-5}$ cm^2/sec for 0.1 N HF at 25°C. The average deviation from this value was 0.07×10^{-5}.

Oxygen-17 Pilot Plant. — The Oxygen-17 Pilot Plant performed satisfactorily during the first six months of 1966. The Water Distillation Cascade remained on stream continuously during this period. The peak concentrations of ^{17}O and ^{18}O in this system are currently 1.4 and 81.1% respectively. Operation of the Thermal Diffusion Cascade was also normal. The peak concentrations of ^{17}O and ^{18}O in the Thermal Diffusion Cascade reached 24.0 and 99.8% respectively. More than 700 g of water, enriched

in ^{18}O from 10 to 96%, was transferred to the Isotopes Division inventory for distribution to prospective users. Also transferred were 9.2 g of oxygen gas containing 99.6% ^{18}O, 16.5 g of water containing 10% ^{17}O, and 45.6 g of high-purity ^{16}O gas containing 10 to 40 ppm ^{17}O and ^{18}O. These products were valued at more than $100,000. All commitments to customers were met.

A number of operating improvements have been made. Several of the more important ones are (1) installation of a separate cooling system which increased the separative work capacity of the Thermal Diffusion Cascade ~25%, (2) installation of a constant-voltage transformer to provide a more constant power supply and longer life for the filaments in the Thermal Diffusion Cascade, and (3) installation of new product collection equipment which minimized handling losses and maintained high purities in the collected products.

Effects of Radiation on Analytical Methods. — One of the more pressing problems encountered in carrying out separations under radiation conditions is that of acid depletion and the consequent undesirable precipitation of different substances. In an attempt to prevent acid depletion from occurring during such processes, the addition of "radiation buffers" has been proposed. One of these is chloral hydrate. This compound, on exposure to gamma radiation, produces an acid (presumably HCl). The acid yield is concentration dependent and is constant for any given concentration of an air-saturated solution. The G value of acid production in 1 M air-saturated solution is about 22 molecules per 100 ev. Since this rate of acid production is a function of the concentration, it is a fairly simple matter to calculate the concentration of this reagent required for any system undergoing acid depletion once the rate of this depletion has been established.

The analysis of the gaseous products from this radiolysis reveals that CO_2 is the major component (76%). Other products had masses ranging between 35 and 164 as determined by mass spectrometry; however, there was no single large concentration of any specific mass; thus it seems likely that CO_2 is one of the products of the primary radiolytic reaction in this system along with the acid. Attempts to determine the G value for CO_2 production have been unsuccessful thus far. It is hoped that the determination of this value will help to elucidate the radiolytic mechanism.

The production of CO_2 as one of the products of the primary radiolytic process is encouraging, because there should be no great buildup of undesirable polymeric species or large molecular aggregates which could constitute possible interferences to desired chemical processes in the system to which chloral hydrate is added to counteract this acid depletion.

The electrometric study on the Alizarin Red-S (AlRS)-Zr complex, initiated for the purpose of establishing a voltammetric method for zirconium, less susceptible to radiation than is the colorimetric measurement of the complex, has been essentially completed. The various parameters such as change in acid concentration, AlRS concentration, etc., have been studied. It has been found possible to determine zirconium at the 0.1-ppm level using the anodic wave of the Zr-AlRS complex. Only F^- and PO_4^{3-} of all the common ions studied interfere, and by using the method of sample preparation recommended, the F^- interference is eliminated. Further work is being carried out to elucidate the reaction mechanism.

Chemical Engineering Research: Stacked-Clone Contactor. — The new prototype stacked-clone solvent extraction contactor was subjected to additional tests by using the system 1 M $NaNO_3$–18% tributyl phosphate in Amsco to extract uranyl nitrate. This contactor, which is applicable to the processing of radioactive materials, features 14 extraction and 4 polishing stages, with all stage pumps driven by a common shaft. Solution throughputs and mass transfer efficiencies average 3.02 liters/min (aqueous:organic ratio of 3) and 66%, respectively, for the system described above. This performance is equivalent to that obtained with the 11-stage experimental device reported previously. The residence time of the solution at these conditions averages about 3 sec per physical stage. The contactor has now operated for more than 250 hr. A prototype pump of design similar to that used in the contactor has operated for more than 12,000 hr without appreciable wear of the bearings.

Separations Chemistry Research: Solvent Extraction. — In the continuing study aimed at resolving apparent anomalies in extraction equilibria with alkylamines, the hypothesis that the aggregation of tri-n-octylamine sulfate (TOAS) in benzene solution might be critically sensitive to water content was previously ruled out with respect to water activities, a_w, up to 0.9. The possibility of a significant change on further increase to the slightly higher water activities existing during typical extractions is now ruled out by demonstration of the same anomalous behavior in uranium extraction from sodium sulfate solution at saturation, $a_w = 0.86$, as at 1 M, $a_w = 0.96$. Isopiestic tests showed that water concentrations deviate even more from Henry's law in undiluted TOAS than they do in an undiluted sulfate-bisulfate mixture (ORNL-3939); there still is no discontinuity or region of critical sensitivity.

Didodecylnaphthalenesulfonic acid (ORNL-3968) appears to exist as a hydrated tetramer in 0.1 M benzene solution. It is a much weaker extractant than di(2-ethylhexyl)phosphoric acid (HDEHP) for strontium (E = 0.075 vs 0.70) and especially for europium (E = 0.05 vs 1000) (0.05 M sodium salt of extractant). In contrast to the HDEHP extractions, there is no maximum of E vs pH. This suggests that, in metal extraction, coordination with the sulfonic oxygens is not as important as coordination with the phosphoric oxygens. This fact is consistent with the lower extraction power for strontium and europium and with the large synergistic enhancement of many extractions previously observed when neutral organophosphorus compounds were added to the homologous dinonylnaphthalenesulfonic acid.

Volatility Studies. — A desirable step in the volatility process would be the use of a solid sorbent to selectively and reversibly sorb PuF_6, in much the same way that NaF is used in the UF_6-NaF system to recover and decontaminate UF_6. The PuF_6-LiF system, which was previously described (ORNL-3968, p. 22), is not an exact analog of the UF_6-NaF system but may be adequate; it therefore has been more completely evaluated.

Retention of plutonium during the sorption step appears satisfactory. In duplicate tests with two LiF sorbent traps in series (each containing 1.7 g of LiF in a 7.5-mm-ID tube to a depth of about 5 cm), the disposition of the plutonium (in milligrams) was as follows: in the first traps, 66 and 67; in the second traps, 3.0 and 4.7; and in the backup traps, 0.28 and 0.41. During the tests, which lasted 40 min, the PuF_6 was swept into

the train of sorbent beds by fluorine (flow rate, 30 ml/min). In another test with LiF sorbent (7.8 g of LiF contained in a 10.5-mm-ID tube to a depth of about 11.5 cm) and the same fluorine conditions, the entire 100 mg of plutonium was sorbed (limit of detection, 1 μg).

Desorption of plutonium from the LiF as PuF_6 is slow; 4 to 9 hr are required for 98% desorption at 400 to 500°C with the fluorine conditions described above. (However, a fluorine flow rate of 2 ml/min appears to be as effective as 30 ml/min after several hours of induction.) After five cycles of sorption-desorption, the residual plutonium did not increase above that noted after one cycle; it appears to be desorbable with sufficient treatment.

The loading of PuF_6 onto LiF appears comparable to the loading of UF_6 onto NaF. In the test with 7.8 g of LiF, 0.76 g of plutonium was introduced into the bed in 7.5 hr; 0.71 g was sorbed. The maximum loading observed was 0.1225 g of plutonium per gram of LiF.

Separation of PuF_6 from UF_6 was also achieved in the duplicate retention tests described above. Within the limits of detection (0.02 mg), no uranium was found in the LiF sorbent beds; 4.9 and 17.6 mg were found in the respective NaF backup traps.

Mechanisms of Separations Processes. — The electrolysis of water to produce hydrogen is being studied as a means of utilizing the large blocks of cheap electrical power that would be available from proposed dual-purpose desalination reactors. Investigations are continuing with an electrolysis cell consisting of two porous nickel electrodes (85% void, mean pore size of 12 μ, 0.03 in. thick) with a 0.010-in. asbestos membrane saturated with 6 M KOH, the ion-transport medium.

Initial studies using pulsed direct current (half-wave rectified ac) or the normal constant dc to power an electrolysis cell showed that about an 8% decrease in cell voltage was observed with the former when measurements were compared at equivalent current densities ranging from 2000 to 2400 amp/ft^2. (Average current was measured by using a shunt and an integrating digital voltmeter for pulsed dc.) This suggested an apparent substantial improvement in cell efficiency with pulsed current; however, the power ($I \times V$) expenditure had to be determined and compared with constant dc power.

A Hall-effect generator (a magnetosensitive semiconductor in the gap of a ferrite core with two field coils; its voltage output is proportional to the product of the magnetic field and the current) was obtained. Measurements of Hall output voltages with pulsed-current input to the electrolysis cell were made and referenced to the Hall output with constant dc to the cell. The results showed that 70.6 w was expended at an average pulsed current of 24.06 amp, while only 59.7 w was used at a constant dc of 24.04 amp. These results were verified within 1% by an approximate method using oscilloscope measurements of peak voltage and current followed by integration of the data to obtain power. These data, coupled with measurements of current efficiency, indicated a power expenditure of 3.49 kwhr per pound of hydrogen with pulsed current, compared with 29.6 kwhr per pound of hydrogen with constant dc, at 80°C and at a current density of 2230 amp/ft^2. Thus, if this 18% increase in power usage is considered, the use of pulsed current in an electrolysis plant will not be feasible.

Possible reductions in oxygen overvoltage because of reported catalytic effects of weak beta emitters were investigated. The isotope ^{63}Ni (0.067-Mev beta ray, $t_{1/2}$ = 92 years) was electrolytically deposited on a porous nickel substrate at two different activity levels, 26 $\mu c/cm^2$ and 2 mc/cm^2. The IR-free voltages for a cell containing a normal porous nickel cathode and an anode with 26 $\mu c/cm^2$ of ^{63}Ni appeared to be decreased by about 50 mv; however, a control electrode was not prepared. For the second series of measurements, nonradioactive nickel (equivalent by weight to about 1 mc/cm^2 of ^{63}Ni) was deposited on an electrode to be used as a control. The cell voltages measured with the high-activity-level ^{63}Ni (2 mc/cm^2) were about the same as those measured with the control anode, suggesting the absence of any catalytic effect. However, to ensure that any change in surface area caused by nickel deposition is not overshadowing any possible beneficial effect of the radiation, these results will be rechecked at a lower level of activity.

Foam Separation Studies. — As part of a basic study of mechanisms of foam separation, the effects of foam reflux and gas:liquid volume ratio on volume reduction and on decontamination factors for radioactive strontium are being investigated. The apparatus used in these experiments is a 38-mm-ID by 100-cm-high (exclusive of the 9-cm-diam by 17-cm-high foam drainage head) glass column equipped with a basket-type centrifuge foam breaker. A swinging funnel and timer device for splitting the foam condensate stream permits any chosen fraction of the foamate to be returned to the countercurrent (liquid vs air) foam separation column. Thus the effects of foam reflux and gas:liquid volume ratio can be studied as process variables in the removal of strontium from aqueous dodecylbenzenesulfonate solutions. The average total surfactant concentration in the feed stream (actually about one-sixth is added to the 40-cm-deep liquid pool at the bottom of the foam column) is 90 to 100 ppm, while the feed throughput rate is about 40 gal ft^{-2} hr^{-1}.

Data reported up to now (ORNL-3968, p. 22), showing that high volume reduction and high strontium decontamination can be achieved simultaneously, were obtained with pure distilled water as feed. The new data reported below, however, were obtained with distilled water containing 0.005 M NaOH and 0.005 M Na_2CO_3. These low concentrations of caustic and carbonate simulate a typical low-radioactivity-level process waste water that has had the "hardness" removed in a head-end precipitation step (ORNL-3811). The pure gamma emitter ^{85}Sr, in the presence of 10^{-6} M Sr^{2+}, was used as a tracer in all these studies because it facilitates determination of decontamination factors by counting with a single-channel gamma-ray spectrometer.

A kinetic study of the caustic-carbonate system, in which observations were made over a period of 6 hr under the above-described run conditions, revealed that at 95% foam reflux and a gas:liquid volume ratio of 12 ± 1.5, steady-state operation is reached in less than 2 hr.

Volume reduction and strontium decontamination factors obtained from the kinetic run were used in combination with data from two other runs that were identical except that the percent of foam reflux was varied in order to evaluate its effects. The results showed that at 0, 95, and 97.5% foam reflux, under the above-described run conditions, the respective volume reduction factors were 39, 425, and 731, with corresponding strontium decontamination factors of roughly 800, 500, and 220. Again,

as with previously reported (ORNL-3968, p. 22) pure distilled water runs, the volume reduction factor increased with increasing reflux ratio, whereas the strontium decontamination factor decreased. Recycling the collapsed foam to the countercurrent foam separation column enhances strontium removal by this method, as is shown by the great improvement in the volume reduction factor, accompanied by only a moderate decrease in strontium decontamination, as the amount of foam reflux is increased from 0 to 95%. In general, corresponding volume reduction factors obtained under similar conditions using pure distilled water as feed (i.e., no caustic or carbonate present) were about 15% higher, while the strontium decontamination factors were higher by about an order of magnitude.

In addition to the laboratory work, a computer method for filing and retrieving foam-separation information and documents was set up. Several recent requests for information pointed up the desirability of having an easy-to-keep and up-to-date literature file. Therefore, a fairly complete bibliography (over 100 references) of journal articles and papers, project literature, books, and patents pertaining to foam separation, foams, and surface-active agents was assembled. The titles, authors, sources, etc., were punched on IBM cards for use with a short program that was written to produce a computer listing of the documents. In addition, a rudimentary sorting system (by author and by author and site for project literature) was incorporated into the punched-card data. Further work is being done on the sorting system to generalize it to include categorizing of the information into such subjects as areas of application, type of apparatus, and type of surfactant. The computer method is cheap and will permit any desired number of printouts of the bibliography to be made. It will also enable us to keep the literature list current with a minimum of effort.

TRANSURANIUM-ELEMENT PROCESSING

TRU Operations. — The Transuranium Processing Plant officially went "hot" on June 1, when a target irradiated at Savannah River was transferred into the cell bank for inspection. This was one of 18 targets that will undergo additional irradiation in the HFIR in the coming year. No damage was evident except at the fins, where, in some cases, half a fin had been worn away by a vibration-induced fretting-type corrosion. A dummy flow-test element that had been operated in the HFIR for several months was also inspected; it showed the beginnings of similar wear. Spacers will be installed to reposition the rod in the center of the hex spacer can before the irradiation in the HFIR is continued.

Essentially all equipment has now been installed, calibrated, and checked out for operation. An integrated "cold" run was begun but was terminated when gross discrepancies developed in material balances in the dissolution—fluoride removal step. This was traced to precipitation of trivalent rare earths (as the fluorides), and a flowsheet change was made (addition of boric acid to complex fluoride) to correct the situation. Another flowsheet change involved the substitution of the existing centrifuge for a filter to collect the precipitates formed during the process of removing fluoride by hydroxide precipitation.

Rather than repeat the entire cold run, we decided to begin operation with radioactive tracers in the form of 1 g of ^{242}Pu, 1 mg of ^{244}Cm, and approximately 1 curie of cerium. This run has just started.

Process Development. — In continued studies on the cation exchange separation of actinide elements, the first of a series of experiments was completed with micro quantities of ^{241}Am and ^{242}Cm in cell 3 of Building 4507.

A 1-in.-diam, 28-in.-long column packed with 274 ml of ammonium-form Dowex 50W C-12 resin was loaded with approximately 125 mg of ^{242}Cm and an equal amount of ^{241}Am. Chromium and iron (total of about 2 g), ^{106}Ru, ^{85}Zr, and several rare earths (trace quantities) were present as impurities.

Resin degradation at these activity levels (about 1200 w/liter) was not severe, and physical performance of the column throughout the run was satisfactory. Both the stable and radioactive impurities were eluted with ammonium α-hydroxyisobutyrate from the column ahead of the americium-curium fraction, and good separations of the individual rare-earth impurities were noted. Complete analyses of the amercium and curium fractions are not yet available; however, gamma scans of these fractions indicate that partial separation of these elements was obtained.

METALLURGY AND MATERIALS

Physical Ceramics. — We are continuing our efforts to understand the progressive geometric changes that occur in a sintering mass and to apply topological parameters to characterize these changes. Copper powder with a relatively large particle size is being used to provide a sample system in which the void-solid interface can be readily observed. Thus far, we have observed that as sintering progressed the structure generated rapidly achieved a condition independent of the shape of the original particle. The local sintering behavior, however, was heavily dependent on the broader structure that contains it. We also measured the variation of surface area per unit volume as a function of density, and found that in the latter stages of sintering this relationship becomes linear. The relative amounts of interconnected and isolated porosity as sintering progresses are currently being studied.

Diffusion Studies. — The measurement of diffusion of ^{44}Ti and ^{48}V in titanium-vanadium alloys at 10 wt % increments over the entire composition range was completed. Arrhenius-type plots of ln D vs 1/T appeared in most cases to be fitted best by a straight line up to 1300 to 1400°C and by a curved line from there to near the melting point. Adding 40% Ti to vanadium changes the diffusion coefficient of either species at a given fraction of the absolute melting point by a factor of only 4 to 5, but adding 40% V to titanium gives a factor of 100 to 200 change.

CONTROLLED THERMONUCLEAR RESEARCH

Single-Pass Injection-Accumulation Experiments (DCX-1, DCX-3). — During this report period, work has been carried out on design considerations

and certain measurements relating to the neutral injection experiment (DCX-3). Design computations and conceptual mechanical layouts for the DCX-3 magnet system (mirror coils plus quadrupole windings) are proceeding. Two alternative designs are being pursued in parallel, one using water-cooled copper ribbon coils and one using superconducting coils.

A high-resolution energy analyzer has been developed and tested. Using a silicon-barrier detector and an FET preamplifier at 80°K, resolution of 3.0 kev at 10 kev has been obtained for protons. This is more than sufficient to resolve the 20-kev particles leaving the DCX-3 experiment.

Work on the production of neutral beams of 20-kev H^0 has been carried out in conjunction with the Ion Source Group here, and 82 ma of H^0 with energy spreads of 30% have been obtained in geometry approximating that anticipated for DCX-3. This source is being assembled on the DCX-1 apparatus to allow further tests on vacuum problems, trapping rates, and excitation rates into states appropriate for Lorentz ionization (cascade experiments).

The DCX-1 mirror field will be used for two groups of experiments in the next few months. One group will utilize 600-kev H_2^+ injection to examine the effects of inductive end walls on the negative-mass instability (as predicted by the theory of Briggs and Niel). The other group will utilize 20-kev H^0 injection to examine the trapping for states n = 13 to 15, the threshold for flute instability (expected at approximately 3×10^7 cm^{-3}), and properties of microinstabilities in this plasma.

Properties of Plasmas Created by Electron-Cyclotron Heating. — The ELMO facility is being operated in a folded-cusp magnetic geometry with up to 5 kw continuous wave of 10.6-Gc microwave heating power. The purpose of the experiment is to compare the properties of hot-electron plasmas produced in positive-gradient magnetic wells (having finite or zero minima) with the properties of the plasmas trapped in negative-gradient magnetic mirrors. One of the diagnostics employed in these preliminary experiments is an x-ray pinhole camera. The pinhole photographs show a plasma that is largely confined to the region bounded by the closed heating surface in the folded cusp. The photographs show evidence of the plasma extending into the ring cusps, but no radial losses to the walls are seen.

This is to be contrasted with the behavior of the plasma when the facility is operated as a magnetic mirror. Pinhole photographs show a plasma confined between the mirror coils, but also show x rays emanating from radial walls.

Properties of Plasmas Created by Beam-Plasma Interaction. — Burnout V was modified during this report period. Its liner diameter was approximately doubled at the midplane, and the magnetic axis was rotated 90°. The axis is now parallel to the long dimension of the vacuum chamber and permits the beam reflecting electrodes to be spaced 10 in. farther from the magnetic mirrors. Both modifications are aimed at reducing impurities and permitting higher power densities in the plasma.

The smaller Burnout IV device was used to develop a technique for monitoring the 3-Mev protons from the D-D reaction. A cesium iodide crystal, light pipe, photomultiplier, and multichannel analyzer were used.

The 3-Mev protons do indeed originate in the midplane region of Burnout IV and at the rate estimated from previous neutron measurements. Since a proton trajectory in the magnetic field can be determined quite accurately, we now believe we can locate not only the reaction region but differentiate between wall and plasma reactions in the larger Burnout V device.

Another measurement of the intermediate energy group in the hot electron blanket has been made. Two probes monitoring ion saturation current, located in the midplane, are separated by 180° and are each positioned radially to intercept a portion of the plasma. When the arc is turned off rapidly, an abrupt discontinuity is generated in the blanket density. The resulting current change in each probe is photographically recorded from an oscilloscope trace which shows several revolutions of the discontinuity. From the known time scale, the drift velocity of the discontinuity is determined which then yields the electron energy, assuming the electron drift is due to the existing magnetic field gradient. The result is that the electron energy is 4.6 kev, in good agreement with other experiments.

We have replaced the iron shielding in the anode channel region with a field bucking coil. Preliminary tests with this coil have permitted generation of the hot electron blanket at a midplane magnetic field of 5.5 kilogauss — greater than a factor of 3 increase over the field attainable without magnetic field reduction in the anode channel. It appears that the electron density is inversely proportional to the magnetic field strength.

Ion Source and Ion Beam Technology. — Work on the injection systems for DCX-3 and INTEREM is continuing. An injection system using a magnesium cell has operated for 5 hr continuously. It has provided a maximum current of 82 ma of 20-kev neutral hydrogen atoms through a 1-1/4-in. aperture 92 in. from the lens. Variations in extraction geometry are being made to determine the optimum configurations for INTEREM. Various arrangements of electrical leads and epoxy insulators are being tested as part of a program of development leading to the design of a new shorter and more stable 600-kev accelerating system.

Theoretical Plasma Physics. — Marginal stability analysis, leading to the identification of stable and unstable regions in some multidimensioned parameter space, has often served as a guide for predicting possibly stable experimental operating regimes. Recently, a highly anisotropic plasma of density $\cong 10^8$ ions/cm^3 has been produced in DCX-2 and observed to be stable in apparent contradiction with such theoretical predictions. These results illustrate the need for more information than marginal stability analysis yields, namely, the propagation characteristics and growth rates of the instability. General techniques for determining propagation properties of unstable waves have existed for some time. However, application of these techniques to any but the simplest dispersion relations has been held up pending the development of suitable computer codes and shortcut procedures necessary to eliminate prohibitively long computing times. Our recently developed computer codes have enabled us to accomplish these objectives. Results show that the plasma is indeed unstable at $\cong 10^8$ ions/cm^3, but that the instability is convective; that is, it propagates out of the system faster than it

grows so that at any point the wave eventually dies out. Such a wave could reach large amplitudes only if the ratio of the machine length L divided by the growth length λ were sufficiently large, or if there were sufficient reflection at the ends of the machine. Although little work has been done on transmission, reflection, and absorption of unstable waves at a plasma boundary, it is suspected that reflection may not be important here. The quotient L/λ turns out to be small, so one should not expect to observe an instability at this density. However, an unexpected theoretical result was that the instability changes character and becomes absolute at higher densities. Since an absolute instability grows in time everywhere in space, machine dimensions play no role, and one would expect to observe the instability. Instabilities are seen in DCX-2 when the anisotropic plasma is formed at higher densities ($\cong 10^9$ ions/cm^3) by breakup on the lithium arc. The predicted growth time is long (~50 μsec), and the observed instability is not strong. The tentative agreement between theory and experiment is encouraging and suggests the importance of a similar examination of other instabilities in order to assess their effects on plasmas.

BIOLOGY AND MEDICINE

Macromolecular Separations: Heterogeneity of E. coli tRNA's. — Differences in the chromatographic position and the number of specific tRNA's from different species, for example, *E. coli* and rat liver, have been demonstrated by other investigators, and it is now generally assumed that specific tRNA's from different species may vary in composition and conformation. The question of differences in specific tRNA's within strains of the same organism has not been previously investigated. A series of experiments was performed, using reversed-phase chromatographic columns to investigate the heterogeneity of the leucine, the phenylalanine, and the valine tRNA's from a variety of *E. coli* sources. The results are tabulated below:

E. coli Strain	Source	Number of Chromatographic Peaks		
		Leucine	Phenylalanine	Valine
B	General Biochemicals	3	1	2
B	Schwarz Bio Research	3	1	2
W	General Biochemicals	3	1	2
W	ORNL	3	3	3
K-12	General Biochemicals	3	1	2
A-19	ORNL	4	2	1

Two strains, *E. coli* W (ORNL) and *E. coli* A-19, showed a greater degree of heterogeneity, as indicated by the occurrence of additional chromatographic peaks. The biological significance of this increased heterogeneity

is not yet clear; from a chemical standpoint, however, additional complications are introduced into the separation and purification of specific tRNA's. It cannot be assumed, then, that a specific tRNA will be identical in different strains of E. coli, or that a separation method that has given satisfactory resolution of a specific tRNA from its chromatographic neighbors in one case will be applicable to the tRNA from a different strain of E. coli.

Macromolecular Separations: Engineering Experiments. — A process for separating and isolating several transfer ribonucleic acids (tRNA's) from a mixture of approximately 20 is being scaled up. The process consists of feeding a charge of mixed tRNA's to a column of diatomaceous earth that has been loaded with a quaternary amine. The mixed tRNA's are then eluted from the column with a solution of sodium chloride saturated with isoamyl acetate. The concentration of the sodium chloride in the solution is steadily increased during elution. The various tRNA's are eluted at slightly different sodium chloride concentrations, and, if column conditions are exactly right, separation will be complete.

Packing stability continued to be a serious problem; the quaternary amine on the column packing was still being washed off by the gradient solution. The packing was finally found to be unstable at low sodium chloride concentrations but quite stable at higher concentrations. Subsequent runs were made with gradient solutions at least 0.45 M in sodium chloride, and no further evidence of instability was seen.

After this problem was solved, an attempt was made to reproduce earlier runs that had achieved complete separation of phenylalanine tRNA and leucine tRNA. It was unsuccessful; the resolution obtained was good but the separation was poor. The difficulty was finally traced to the 0.001 M Versene added to the gradient solution to complex contaminating metal ions. In some way it was preventing the separation of phenylalanine tRNA and leucine tRNA. Fresh solutions were made up without Versene, and a new series of runs was made in which nearly complete separation was achieved.

Two phenylalanine tRNA's were observed in chromatograms of E. coli, strain W. These two tRNA's overlap in the chromatogram and would probably be very difficult to separate. The biologists prefer to work with pure material when possible; hence interest has revived in producing large quantities of E. coli, strain B, which has only a single phenylalanine tRNA. A series of runs was made to grow strain B for processing; each run ended after 2 to 12 hr when the culture became infected. The fermenter was repiped to reduce the chance of an infection, and another series of continuous runs was planned to grow a large quantity of strain B. If continuous operations prove impractical, a batch-type operation will be used.

SOMATIC EFFECTS OF RADIATION

Statistical Services and Research: Confidence Regions for Mean Measurements of Human Chromosomes. — Recent advances in human cytogenetics

have permitted a closer study of the human karyotype with an eye toward the detection of gross anomalies. Among these anomalies the most obvious is the aneuploid. The number of chromosomes in such a nucleus is different from the normal complement of 46. Such a condition in the genotype of a human being may be characterized by a corresponding phenotypic abnormality if the deficiency or superfluity occurs among chromosomes of a certain type. Thus it becomes important to identify the specific chromosomes in the human karyotype as well as to count them.

The present techniques for displaying a human karyotype are tedious at best and require the skills of a highly trained biologist. In spite of this there is a sufficient degree of objectivity in the techniques to encourage the development of scanning devices which, in the company of digital computers, might accomplish the same tasks automatically and with great speed. To achieve such a goal the new devices must be "taught" to measure and assess various bits of information in the chromosome complement so that the decisions which are made are at least as trustworthy as those of a human technician performing the same tasks. Inherent in such a stringent requirement is the need for some knowledge of the natural variability between chromosomes of the same pair, among chromosome pairs, among cells in the same individual, and among individuals.

A statistical study of the errors of measurement of the lengths of chromosome arms is being carried out. The data represent the lengths of the long and short chromosome arms from seven cells in the same individual. A bivariate vector of means is computed, and simultaneous, two-sided confidence regions are estimated for each chromosome pair. These regions take the form of ellipses in the plane of the long-arm and short-arm means, with center at the point specified by the mean lengths of the long and short arms. When all the ellipses are plotted on the same grid, the overlap gives some indication of the inability (using present techniques) to tell one chromosome from another solely on the basis of length measurements.

RADIATION GENETICS

Human Cytogenetics. — Approval for an augmented S-4 experiment, in which both human blood cells and *Neurospora* spores will be irradiated in separate experimental devices during the Gemini-XI manned spaceflight, has been obtained. All required qualification, predelivery, and preinstallation testing of the experimental hardware has been completed. S-4 flight equipment damaged by the spacecraft contractor during and after installation has been replaced. Fabrication of the experimental hardware to be installed just prior to launch is virtually complete.

Two complete mockup experiments with both the blood and the *Neurospora* devices, and following the anticipated Gemini-XI mission profile, have been successfully carried out. The analysis of the blood material has been completed, and the *Neurospora* analyses for the second mockup are now in progress.

Cytological analysis of the material from the Gemini-III vibration and acceleration mockup experiment carried out earlier has been completed.

Although the final statistical analysis is not yet complete, spacecraft vibration and acceleration do not appear to account for the effect demonstrated by the Gemini-III S-4 experiment.

COMBATING DETRIMENTAL EFFECTS OF RADIATION

Mammalian Recovery. — Constant monitoring of the health status of experimental animals forms the cornerstone of disease control. Without this control, ruinous epizootics, not to mention more subtle disease problems, can occur in dense animal populations. This may cause investigators years of work in addition to monetary expense. Besides the dramatic epizootic the less dramatic, but perhaps more pernicious, enzootic diseases may introduce unwanted variables resulting in unreliable or unreproducible results. Inevitably it falls to the pathologist and his ancillary services to study and define the disease problems in any population. In the case of laboratory animals this means that all spontaneously dead animals must be subjected to postmortem examination. Also such categories as sick animals, retired breeders, supernumeraries, and occasional deliberate sacrifices must be put through the diagnostic regimen. In so doing a fund of knowledge of the "normal" pathology of colonies can be established. A pathology monitoring system is now being instituted in the Animal Testing Laboratory, employing routine gross and histologic procedures. This will complement the existing bacteriology and parasitology services. The field of clinical pathology of small laboratory animals remains largely unchallenged. Much work needs to be done to establish base lines and determine strain and species variations. Studies on small animals are now proposed using micro and ultramicro techniques. Sufficient evidence exists to show that significant variation occurs in hemograms of inbred strains of mice. Further study is warranted in this area as well as in clinical chemistry. Through the collection and recording of data by employing the above methods, epidemiological patterns, prevalence rates, and normal values can be established. To be useful this data must be available for periodical analysis, and in large laboratories this means the use of a data computer storing system. Present objectives are to employ such a system.

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