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## STATUS AND PROGRESS REPORT

## SEPTEMBER 1957

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# OAK RIDGE NATIONAL LABORATORY

# STATUS AND PROGRESS REPORT

September 1957

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OAK RIDGE NATIONAL LABORATORY
STATUS AND PROGRESS REPORT

September 1957

This status and progress report summarizes somewhat less than one-half the activities of the Laboratory. A few of the activities are reported every month, but most of them are reported on a bimonthly schedule. Program 4700 is reported separately.

## PROGRAM 2000 - SPECIAL NUCLEAR MATERIALS

Waste Metal Recovery - The recovery of 3.6 kg of plutonium from Chalk River solutions, oxide, and scrap, and of 109 g of plutonium from waste solutions accumulated from previous programs was completed. The percentage of plutonium recovered as specification-grade product was 99.8%, with the major loss of 0.11% occurring in the stripping column waste stream of the second plutonium cycle. Of the 3.6 kg of plutonium processed, 1.8 kg was processed by ion exchange only; the remainder required dissolution, one cycle of solvent extraction, and isolation by ion exchange.

The optimum $PuO_2$ dissolution reagent was established as 12 M $HNO_3$ containing 0.02 M HF as catalyst.

Approximately 4.5 tons of Paducah fluorinator ash was processed, with the recovery of 30 g of $Np^{237}$ and 3 tons of uranium. The flowsheet provides for dissolution of ash in 1.8 M $Al(NO_3)_3$—1.0 M $HNO_3$, one cycle of solvent extraction with 15% TBP—85% Amsco, and product concentration by evaporation. Over-all neptunium and uranium losses averaged 1.7 and 0.04%, respectively. The amount of ash dissolved per mole of aluminum nitrate was increased 33% without affecting neptunium recovery. The first batch of neptunium product solution contained 6.4 g of ionium ($Th^{230}$) and 3 kg of $Th^{232}$. (AEC Activity 2350)

Excer Process - In the Excer process, uranium in an ore concentrate is converted by ion exchange to a form suitable for reduction, is reduced, and is precipitated as $UF_4 \cdot 3/4H_2O$. Flowsheets providing for iron reduction, instead of electrolytic reduction used previously, of uranyl salts to U(IV) compounds were designed for ore mills using either the chloride or sulfate system.

In tests on the feasibility of iron reduction, the U(IV) product was separated from the iron by a factor of 3 by continuous anion exchange. Uranium losses in the waste decreased with increasing chloride in the scrub, and were 4 to 5% with an 8 M HCl scrub. Some oxidation of the U(IV) occurred, probably as a result of dissolved oxygen in the solution, and increased with increasing scrub chloride concentration.



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In 11 hr of operation of the continuous precipitation equipment, with electrolytically produced $UCl_4$, $UF_4$ hydrate was formed at an average rate of 1 lb of uranium per hour. The precipitate settled rapidly, and all of it appeared to be the $3/4H_2O$ hydrate, with a bulk tap density of 3.4 g/cc. (AEC Activity 2702)

Fluorox Process - In the Fluorox process, $UF_6$ is prepared by oxidation of $UF_4$ with dry air or oxygen.

In tests on the components of the flame reactor, for oxidation of $UF_4$ to $UF_6$ ($UO_2F_2$ by-product) by means of oxygen, a discontinuous pilot-light assembly was preferable to a continuous assembly, since the latter produced excessive heat. Satisfactory operating conditions appeared to be met by using a fuel gas of 80% CO and a gas flow of 0.3 scfm. The light was ignited by the spark plug approximately ten times per second. Increasing the diameter of the solids nozzle from 11/16 to 3/4 in. decreased the annular area through which the fuel gas flowed. The higher gas velocities made it possible to operate at higher CO concentrations without flash back and thus to produce a longer flame at higher temperature.

In studies of the kinetics of the reaction, the rates in the temperature range 550 to 815°C indicated the reaction to be first-order with respect to the $UF_4$ surface area and to depend on the oxygen partial pressure. At a given temperature the rate constant with air was half that with pure oxygen. Above 750°C, 20% of the $UF_4$ was consumed by the side reaction $UF_4 + UF_6 \rightarrow 2UF_5$.

In kinetic studies on the high-temperature decomposition of $UO_2F_2$, the reaction $3UO_2F_2 \rightarrow 2/3U_3O_8 + UF_6 + 1/3O_2$ was first-order with respect to the $UO_2F_2$ concentration. Rate constants of $7 \times 10^{-4}$ and $24 \times 10^{-4}$ $min^{-1}$ were determined from thermobalance weight-loss curves at 800 and 840°C, respectively, with dry helium as an inert sweep gas. (AEC Activity 2702)

Metallex Process - In the Metallex process, anhydrous $ThCl_4$ is reduced to $ThHg_3$ by sodium amalgam, and the $ThHg_3$ is converted to massive thorium by vacuum distillation of the mercury. In a 36-hr run in the large-scale continuous equipment, 105 lb of thorium, as quasi amalgam, was produced, with about 90% yield. Eight and one-half kilograms of thorium metal of high quality, 0.25 to 0.68% $ThO_2$, was produced from Metallex thorium amalgam by washing, pressure-filtering through porous stainless steel, and vacuum-retorting. The final metal-product density was 7.2 to 9.6 g/cc.

A relation between the gamma activity of thorium amalgams and the average thorium concentration was established for use in monitoring the thorium concentrations of amalgams. The procedure was accurate within 5% for 50-g samples.

In settling-rate and centrifugation studies, the particle diameters of $ThHg_3$, formed by sodium amalgam reduction of $ThCl_4$, in mercury were 0.7 to 16.8 μ, and were increased by aging. Filtration and activity-monitored settling-rate studies roughly confirmed these data.

A button of uranium alloy was produced by reduction of $UF_6$ with sodium vapor. (AEC Activity 2704)

## HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test - After completion of 48 hr of rinsing with 5% nitric acid, the reactor piping was flushed three times with distilled water, and was subjected to a hydrostatic test of 750 psi in the low-pressure system and 3000 psi in the high-pressure system. After a successful retention of the applied pressure, the reactor was emptied to prepare for 300 hr of test operation on water and depleted-uranium solution.

A hydrostatic test of the reactor steam system at 1950 psi was also carried out successfully, and the reactor steam and feedwater flowmeters were calibrated. The fuel and blanket recombiners were reinstalled after the reactor pressure-testing; the fuel recombiner now includes an iodine-removal bed of silver-impregnated ceramic Raschig rings upstream from the recombiner bed.

The reactor was charged with condensate, and startup for run No. 9 was begun on September 18. The run was interrupted the following evening when sample analyses showed high total solids and high pH in the fuel low-pressure system. These solids were subsequently identified as sodium and are thought to have come from the iodine-removal bed. After further washing of the low-pressure system, the test run was restarted on September 23. (AEC Activity 4103.3)

HRT Chemical Pilot Plant - During underwater-maintenance practice in the HRT chemical plant, four components, the high-pressure-system circulating pump and heater, the dissolver, and a low-pressure valve, were removed and replaced, by use of the special tools developed for this purpose. Modifications to increase flexibility in adjoining piping were required before the high-pressure screen could be removed. It is felt that all components designed to be replaceable can be removed and reinstalled under water as proposed.

The closed-loop demineralized-water system was completed and placed in operation. Preparations for "radioactive" operation included replacement of all flange bolts and nuts in the high-pressure system with better grade non-plated bolts and nuts. (AEC Activity 4103.1)

Mathematics and Computation--Homogeneous Reactor Development - Oracle codes were written for determining the economic characteristics of two-region, time-dependent, thorium-breeder reactors and for calculating the heat generation due to gamma-ray absorption in the core shell of two-region, spherical homogeneous reactors. In the latter code the thermal-flux distribution and hence the prompt-gamma source distribution are calculated by using a two-group model; the decay-gamma source is assumed to be uniformly distributed within the core region. The two-group, two-dimensional, three-region reactor code written by Bradshaw was converted to comply with present Oracle equipment (AEC Activity 4103.1)

Reactor Analysis - The nuclear characteristics of HRE-3 type spherical reactors were calculated as a function of operating time. The breeding ratios for reactors fueled with $U^{233}$ are high initially and fall steadily as the core poisons build up. Use of blanket processing retards this decline only slightly, but core processing halts it abruptly. In a typical case, the breeding ratio decreased by about 0.05 in the first three years.

Heat generation rates in the HRE-3 pressure vessel resulting from neutron captures were calculated for various conditions. At a reactor power of 60 thermal Mw, the maximum heat generation rate with a blanket thorium concentration of 1000 g/liter is about 0.015 w/ml, while with a $D_2O$ blanket the maximum rate is about 1.5 w/ml.

The prompt-neutron lifetime and the core temperature coefficient of reactivity for the HRE-3 reactor were calculated to be $5.3 \times 10^{-4}$ sec and $-2.9 \times 10^{-3}$ $\Delta k_e/^{\circ}C$, respectively. For a rate of reactivity addition of 1.5% $\Delta k_e$/sec, the maximum core pressure rise would be about 400 psi if the piping between the core and pressurizer had a 12-in. diameter, a 20-ft length, and if the vapor volume of the pressurizer were 8 $ft^3$. (AEC Activity 4103.1)

Homogeneous Reactor Instrumentation - The pneumatically-powered-bellows valve actuator with a 123-$in.^2$ effective area, being tested for 2-in. control-valve applications, has been cycled 128,000 times to date without failure. Test actuating air pressure has been 80 psi and the stroke 1/2 in.

In a continuation of the evaluation of valve trim materials in the valve test loop, a type 347 stainless steel valve plug flame-plated with tungsten carbide by Linde gave very satisfactory results. A metallographic examination revealed that the plate was intact after the test. One valve plug made from Kennametal K501, a cemented tungsten carbide composition employing a platinum binder, shattered after a few test cycles. A second assembly using composition K96, a similar material employing a cobalt binder, shattered after 50 cycles. A Zircaloy-2 trim set leaked water at the rate of 20 cc/min at 2000 psi after 400 dumps. This is not considered acceptable. However, two more sets will be tested to determine if this material is immune to the ignition reaction encountered with titanium alloys in similar tests. (AEC Activity 4103.1)

HRP Design - The design of facilities for sectioning the HRT core and blanket corrosion-specimen holders preparatory to transport to the metallurgical inspection area was completed.

The HRT pressure-balancing system was redesigned to permit operation without a pressure-balancing valve, leaving only the rupture disks in the line interconnecting the fuel and blanket pressurizers.

Small supplementary recombiners were designed for installation at the vapor outlets of the existing HRT recombiner-condensers. These are necessary when operating at essentially 100% internal recombination in the high-pressure system because of the reduced efficiency of the existing catalytic recombiners when fed with a stream of very low $D_2$-$O_2$ concentration.

Conceptual studies preliminary to design of the HRE-3 continue. Modifications were made to the flowsheets and design-data tabulations to reflect the latest thinking concerning this reactor, and estimates were made of the sizes of major components. Special studies were made of oxygen pressurization and xenon removal. (AEC Activity 4103.1)

HRP Metallurgy - Under at least some conditions, zirconium-niobium alloys have shown greater corrosion resistance to homogeneous reactor conditions than has Zircaloy-2. However, these alloys are complicated and difficult to handle from a metallurgical viewpoint. Work on their morphology and transformation kinetics has shown that at least three major transformations may occur. During quenching from the beta field, a Widmanstätten type of platelet precipitate forms in a retained-beta matrix. This precipitate will redissolve in the matrix on heating to subcritical temperatures. An age-hardening transformation occurs when a beta-quenched alloy is held at temperatures below 550°C. In this transformation the retained-beta matrix, body-centered cubic with $\alpha_o$ = 3.5435 Å, transforms into two body-centered cubic structures, one zirconium-rich and the other niobium-rich. These structures change with aging time and temperature toward the composition Zr—5 wt % Nb and Nb—15 wt % Zr. Both the rate of hardening and the maximum hardness vary with aging time and temperature. To obtain overaging, times of two weeks and temperatures above 500°C are required. The third transformation occurs in beta-quenched alloys held in the temperature range between 550°C and the eutectoid temperature, approximately 615°C. This is a sluggish precipitation of alpha platelets in the grain boundaries, with a consequent enrichment of the neighboring beta phase in niobium, which is then followed by a precipitation of Nb—15 wt % Zr (nominal) between the alpha needles and alpha Zr and Nb—15 wt % Zr (nominal) from the beta matrix.

Small additions of the ternary elements Pd, Pt, or Mo speed up the high-temperature transformation and delay the undesirable hardening transformation; Al and Cu act similarly but are not as effective.

It now appears that by means of small ternary additions (<5%) and suitable heat treatments it will be possible to obtain satisfactory alloys in the Zr-Nb system. These alloys will have strengths considerably higher than the strength of Zircaloy-2, but the elongations will probably be about 10%. (AEC Activity 4103.1)

Laboratory Corrosion Studies - Bromide concentrations of 100 and 200 ppm in boiling and aerated 0.04 m $UO_2SO_4$—0.02 m $H_2SO_4$—0.005 m $CuSO_4$ solution produced severe subsurface attack on stressed and unstressed type 347 stainless steel. The stressed stainless steel was not subject to cracking. In a similar solution containing 50 ppm bromide, subsurface attack was negligible.

When painted on stressed areas of type 347 stainless steel and exposed in oxygenated steam at 300°C, six different marking inks with chloride contents ranging from 3000 to 19,000 ppm produced cracking in every case. Severe pitting on the painted areas also resulted.

Incoloy and Croloy 16-1 stainless steel exposed in pH 2.8 distilled water containing 100 ppm chloride at 300°C were not subject to stress-corrosion cracking after 500 hr. Incoloy was corroded at a rate of 6 mils/year; Croloy 16-1 was corroded at appreciably higher rates, 15 to 45 mils/year.

Preliminary tests with Croloy 16-1 stainless steel in boiling and aerated 0.04 m $UO_2SO_4$—0.02 m $H_2SO_4$—0.005 m $CuSO_4$ solution resulted in corrosion rates around 265 mils/year. The observed corrosion rate in a similar solution at 300°C with oxygen overpressure was 65 mils/year. (AEC Activity 4103.1)

Dynamic Solution Loop Corrosion Tests - A solution containing 0.03 m uranyl carbonate and 0.2 m lithium carbonate, which in previous tests had given low corrosion rates on carbon and stainless steels and which had been phase stable at 250°C, proved to be unstable at 300°C. Under a $CO_2$ partial pressure of 600 psi (at 300°C) only about 0.004 m uranium was soluble. Corrosion rates of carbon steels as well as austenitic stainless steels were 1 to 3 mils/year at 10 to 80 fps in an 890-hr test.

The mockup of the Zircaloy-2—stainless steel transition and expansion joint as used in the HRT reactor vessel was subjected to a fifth test lasting 951 hr, 23 hr with water and 928 hr with fuel solution (0.04 m $UO_2SO_4$, 0.02 m $H_2SO_4$, 0.005 m $CuSO_4$). The test was conducted isothermally at 300°C, and the expansion joint was mechanically deflected 5/8 in. once every 4 hr for a total of 226 deflections. No leakage of fuel solution through the bellows or past the transition joint was detected. However, the pitting attack on the bellows at the edge of the reinforcing rings, which stopped in the absence of mechanical deflections, started again. The average depth of the pits increased roughly 0.003 in. during the test, with three pits now measuring 0.022 to 0.024 in. deep. Total exposure of the mockup has been for 5069 hr, including 4545 hr on fuel solution, 168 thermal cycles, and 374 mechanical deflections. (AEC Activity 4103.1)

Small-Scale Dynamic Slurry Corrosion Tests - In a series of toroid tests at 250°C and 26 fps on oxygenated slurries of thoria produced from jet-precipitated thorium oxalate calcined at 1200, 1400, and 1600°C, attack rates generally increased with increased oxide calcination temperature. Corrosion rates of slurries containing 640 g of thorium per kilogram of water of the 1200, 1400, and 1600°C-fired preparations were, respectively, as follows (in mils per year): type 347 stainless steel, 1, 1, and 1.4; SA-212-B carbon steel, 2, 7, and 10; and Zircaloy-3A, 0, 0.1, and 3. The thoria, of mean particle diameter 0.6 μ, was not degraded. (AEC Activity 4103.1)

Dynamic Slurry Loop Corrosion Tests - Slurry prepared from 800°C-calcined thoria pulverized in a Mikropulverizer to a mean particle diameter of 1 μ was circulated at 300°C with oxygen atmospheres in two 100A pump loop tests at average concentrations of 932 and 641 g of thorium per kilogram of water for periods of 300 and 165 hr, respectively. Specimen corrosion rates were normal; however, system corrosion rates, much of which represented attack on pump parts, were high. Corrosion rates (in mils/year) in the higher concentration

test included the following: loop, 8; austenitic stainless steels at 20 fps, 3 to 4; at 39 to 42 fps, 6 to 19. At the same velocities, corrosion rates for Zircaloy-2 were 0.7 and 3 mils/year. At the lower slurry concentration, attack rates (in mils/year) were as follows: loop, 5; 400-series steels at 20 fps, 1 to 2; at 39 to 42 fps, 2 to 8. Zircaloy-2 displayed rates of 1 to 2 mils/year over the velocity range 20 to 42 fps.

An oxygenated slurry of 1600°C-calcined thoria currently being circulated in a long-term test at an average circulating concentration of 616 g of thorium per kilogram of water has shown a system attack rate of 1.3 mils/year for the first 623 hr of operation. The thoria, which had an initial mean particle diameter of 1.3 μ, has not undergone any detectable degradation during that period of circulation. (AEC Activity 4103.1)

In-Pile Solution Corrosion Loops--LITR - The first all-titanium loop, L-2-14, was dismantled. The loop had been operated at 280°C with a solution of 0.17 m $UO_2SO_4$, 0.4 m $H_2SO_4$, and 0.15 m $CuSO_4$. As in previous experiments, the relationship between corrosion rate of Zircaloy-2 and power density over the range studied was shown to be expressed by an equation of the general form:

$$R = AP(1 - e^{-B/R^{1.5}}),$$

where

R = corrosion rate, mils/year,
P = power density, w/ml,
A and B= constants depending on solution composition and temperature.

Values for the constants A and B have been established from Zircaloy-2 data in this experiment at 0.55 and 25, respectively. In general, the corrosion rate of Zircaloy-2 at a given power density was only about one-half that observed in a previous experiment conducted under similar conditions but with lower excess acid (0.03 m) and recombination catalyst (0.015 m) concentrations. Values for the constants A and B were established from the Zircaloy-2 data in the previous experiment at 1.04 and 95, respectively. The data for crystal-bar zirconium in L-2-14 were in agreement with the Zircaloy-2 data, but the Zr—15% Nb alloy specimens appeared to corrode at rates 2 to 3 times those of Zircaloy-2 at the same power density. (AEC Activity 4103.1)

Autoclave Radiation Corrosion Studies--LITR - For some time a source of concern has been the disagreement, in certain instances, between Zircaloy-2 radiation corrosion data from in-pile autoclave experiments and data from in-pile loop tests. Both systems show marked dependence of corrosion rates on solution power density, temperature, and uranium concentration. In-pile loop tests show no significant variation of corrosion rate in 0.17 m $UO_2SO_4$ solution with excess $H_2SO_4$ concentration in the range 0.008 to 0.04 m. Corrosion in autoclave experiments with 0.04 m excess $H_2SO_4$ has been about 30% less than that with no excess $H_2SO_4$. Similarly, it has been deduced from in-pile loop data that there is no effect on the corrosion of Zircaloy-2 by changing the solvent from $H_2O$ to $D_2O$. An in-pile autoclave which used $D_2O$, however, showed an increase in corrosion rate of about 30% over a similar $H_2O$ test. Another autoclave,

from which atmospheric nitrogen was carefully excluded, showed a corrosion rate about twice that expected. Similar in-pile loop exposures have not been made.

It has been shown recently that if the fission power density to which the Zircaloy-2 surfaces in autoclaves are subjected is presumed to be larger than the power densities in solution by an amount proportional to the percentage of uranium in the scale, then the bomb and loop data are in near agreement. It can reasonably be expected that the discrepancy in the results would be related to the heavy scale retained on bomb surfaces, since this scale is not retained on loop specimens.

The above treatment indicates that no significant change in corrosion rate of Zircaloy-2 was caused by the addition of 0.004 m $CrO_3$, 0.005 m $KTeO_4$, 0.17 m $MgSO_4$, excess acid in the range 0 to 0.04 m, by the replacement of natural water by $D_2O$, or by the careful exclusion of nitrogen.

Excess acid (0.45 m) reduced the corrosion rate by about a factor of 3; the addition of 0.006 m $MoO_3$ seems to have increased the corrosion rate by about a factor of 1.5; and the addition of 0.17 m $Li_2SO_4$ appears to have decreased the rate by a factor of 2.3.

A very recent autoclave exposure included niobium specimens. The corrosion penetration suffered by the niobium was only about one-third that of the Zircaloy-2 specimens. (AEC Activity 4103.1)

## GENERAL REACTOR RESEARCH

Radiation Detector Development - The spectrum of $U^{235}$ fission neutrons was measured with an $Li^6I(Eu)$ scintillation crystal operating at the liquid-nitrogen point as a further check on the usefulness of $Li^6I(Eu)$ as a fast-neutron spectrometer. The fission source was a 5/8-in.-thick by 1-in.-dia slug of $U^{235}$; fission events were initiated by threshold neutrons from the (p,t) reaction. Protons were accelerated in the ORNL 2.5-Mev Van de Graaff generator. The fission neutron spectrum measured in this experiment is in reasonable agreement with previous measurements made elsewhere. The gamma-ray background was entirely negligible. The $Li^6I(Eu)$ technique is now considered to be applicable to problems of interest in fission and shielding studies. (AEC Activity 4202)

Metallurgical Materials and Processing - The effect of nitric acid leaching on magnesium-diffused and heat-treated zirconium specimens was studied. The magnesium-rich grain-boundary phase of zirconium which had been in contact with solid magnesium at 625°C is not significantly attacked by nitric acid. During subsequent heat treatment at 350 and 500°C, some magnesium diffused away from the grain boundaries, and precipitation of a second phase occurred within the grains. Heat treatment did not alter the resistance to nitric acid attack. (AEC Activity 4203)

Power Reactor Fuel Reprocessing: Darex Process - Construction of a loop system for dissolution of stainless-steel-containing fuel elements in aqua regia, stripping of the chloride, and rectification of the nitric acid was completed, and shakedown runs are being made. In dissolver solution adjustment studies, boiling of 14.0 N acid containing 34 g of metal (stainless steel—15% $UO_2$) per liter produced 14.8 to 15.3 N $HNO_3$ vapor as the solution was concentrated to 189 g of metal per liter. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Hermex Process - In the Hermex process, uranium metal is recovered from scrap or fuel elements by dissolution in mercury, and is then separated by filtration, pressing, and distillation operations. The use of a stainless steel dissolver reduced the nonrecoverable uranium loss by a factor of 20 over that in glass, without appreciably increasing the uranium holdup. Recent gross decontamination factors of the quasi amalgam in glass were of the magnitude of about 50, as compared with 18 in earlier runs. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Zircex Process - A 5% Zr—1.5% Nb—93.5% U alloy was hydrochlorinated with gaseous HCl at 545°C, with a total uranium loss of 0.07% and a satisfactory reaction rate of 7.9 $mg \cdot cm^{-2} \cdot min^{-1}$.

The PWR blanket rods were dejacketed at 600 and 725°C with anhydrous HCl in 1.5 and 1 hr, respectively, with uranium losses to the sublimate and to the insoluble residue of less than 0.1% in both casses. Adsorption of chlorine by the unreacted $UO_2$ pellets was approximately 1%. The rate of chlorination of zirconium-alloy fuels by constant-pressure chlorine gas in a closed system was controlled by the rate of heat dissipation from the chlorinator. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Aqueous Dissolution Alternate to Nitric Acid - Stainless steel cladding was removed from $UO_2$ cores and alloys of unirradiated fuel elements by refluxing in 6 M $H_2SO_4$, with solution of 1 to 1.6% of the uranium. The uranium loss to 6 M $H_2SO_4$ was 94% in one experiment with an irradiated APPR (stainless-steel-clad $UO_2$—stainless steel core) fuel element. The reason for the great difference is not known. In a 12-hr continuous electrolytic dissolution, stainless steel dissolved in 3 N $H_2SO_4$ with a current efficiency of 43%, as compared with 90% in 3 N $HNO_3$.

The attack of $HNO_3$-HF mixtures on Zircaloy-2 was decreased sharply by increasing the concentration of Zr(IV) in the acid; 13.1 M $HNO_3$—0.004 M HF did not appear to be a practical dissolvent for Zircaloy-2. Dissolution rates of Zircaloy-2 and U—10% Nb in 9 M HF were 284 and 0.03 $mg \cdot cm^{-2} \cdot min^{-1}$, respectively, indicating the practicability of removing Zircaloy jackets with this reagent.

In electrolytic dissolution of zirconium, HCl was the most promising acid system investigated. Others studied were $HNO_3$ and $H_2SO_4$. About 0.1 M HF had to be present in the HCl to prevent formation of a metallic anode sludge. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Corrosion Studies - Continued tests confirmed previously reported results that titanium can be used for all parts of the Darex-Zircex system except the hydrochlorinator. Haynes 25 and S-816 showed satisfactory resistance in 25 hydrochlorinator-dissolver cycles. In liquid-phase hydrochlorinations, rates of corrosion of nickel and Hastelloy C at 425°C were proportional to the amount of HCl dissolved in molten $AlCl_3 \cdot NH_3$. Both materials corroded too rapidly for use as a container for the molten salt. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Ion Exchange Treatments - Trace amounts of uranium (0.1 g/liter) in aqueous HF solutions of zirconium claddings were sorbed from the $ZrF_4$ colution, made 6 M in chloride, by an anion exchange resin, with 99.8% recovery. The uranium product eluted from the column contained 3 and 0.07 g, respectively, of uranium and zirconium per liter. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Mechanical Processing - Aluminum oxide abrasive allowed a faster abrasive-disk cutting rate and had a lower wear ratio than did silicon carbide. Cutting rates were 0.372 and 0.098 in.$^3$/min for the two abrasives, with wear ratios of 1.14 to 20.6 parts per part of stainless steel. (AEC Activity 4301)

Power Reactor Fuel Reprocessing: Solvent Extraction - A Purex-type solvent extraction flowsheet was designed for recovery and separation of uranium, plutonium, and thorium from the $ThO_2$-$UO_2$ pellets of the Rural Cooperative Reactor fuel. The fuel is dissolved in 8 M $HNO_3$—0.05 M HF. Preliminary batch countercurrent solvent extractions of simulated fuel solution indicated that aluminum nitrate should be added to the solution to complex the fluoride. A third phase, $Th(NO_3)_4 \cdot 4TBP$—TBP—Amsco, was formed. A Thorex-type flowsheet was designed, with which extraction was satisfactory. (AEC Activity 4301)

Fused Salt—Fluoride Volatility Processing - In the fused salt—fluoride volatility process a uranium fuel element is dissolved in HF in a fused fluoride medium, and the $UF_4$ formed is fluorinated to volatile $UF_6$, which is absorbed in NaF traps.

Pilot-plant tests indicated that at the gas flow rates used, 1 scfm, vaporization of $ZrF_4$ is less important than entrainment in carryover of $ZrF_4$ in the gas stream from the fluorinator.

As much as 15 kg of uranium (1.5 flowsheet batches) was absorbed by the pilot-plant NaF absorber before $UF_6$ broke through when the fluorine used was free of HF. An HF trap was therefore installed in the fluorine supply line.

In laboratory-scale equilibrium tests, less than 50 ppm of uranium remained in equimolar NaF-$ZrF_4$ at 600°C after contact with $F_2$ and $UF_6$ gas mixtures up to $UF_6$ partial pressures of 250 mm.

Comparison of results of laboratory tests on desorption of $UF_6$ from the $UF_6 \cdot 3NaF$ complex with a theoretical analysis of the problem indicated that the NaF is not saturated with $UF_6$ at present operating temperatures and flow rates. Results also indicated that the U(V) loss would increase with decreasing temperature of the absorber. (AEC Activity 4302)

Waste Treatment and Disposal - A flowsheet was designed for treating 5000 gal of ORNL waste per day by ion exchange or shale sorption of cesium, strontium, and rare earths and by electrolysis for removal of nitrate and ruthenium. In tests on ion exchange removal of fission products from aqueous waste, Duolite C-3 phenolic cation exchange resin had a distribution coefficient for cesium of ~150 in 0.75 M NaOH—0.1 M $Al(NO_3)_3$. An electrolytic cell removed nitrate and regenerated caustic with a current efficiency of 60 to 80%. Ninety per cent of the ruthenium was plated out. (AEC Activity 4305)

Long-Range Planning Studies - Reactivity lifetimes for U-Th-$D_2O$ reactors were calculated on the basis of nonmoving fuel and on the basis that criticality was controlled by means of a region containing boric acid. Nonuniform fuel burnup and buildup were considered by dividing the core region into three separate regions. For an initial boric acid concentration of 1 g/liter in the control region and 100 g of thorium per liter in the core region, fuel exposures of about 5000 Mwd/ton were calculated for a slab reactor 20 ft thick and for a spherical reactor 20 ft in diameter.

Fuel exposures in heterogeneous Th-U-$D_2O$ reactors have been calculated for continuous-irradiation-type fuel cycles in which fuel rods in adjacent channels move in opposite directions. It was found that the neutron flux had a cosine distribution up to fuel exposures greater than 20,000 Mwd/ton if flux disadvantage factors between adjacent channels were neglected.

Additional results pertaining to the safety of the ORNL Graphite Reactor have been obtained, assuming enriched fuel elements. The effect of a metal temperature coefficient of reactivity upon permissible reactivity additions was found to be negligible if this coefficient has values between $\pm 5 \times 10^{-5}$ $\Delta k_e/^{\circ}F$. Loss of power to the cooling fans appears to be no problem if a scram is initiated when the fan speed decreases to 80% of its normal value. With the present reactor (natural uranium fuel), it is estimated that the permissible reactivity addition is 1.1%. With enriched fuel and a lag of 300 msec between the time of reactivity addition and the time of control-rod movement, the permissible reactivity addition is about 2.5%. (AEC Activity 4510)

Reactor Operations - A total of 200 special fuel elements, 1 in. in diameter and 8 in. in length, has been received for the ORNL Graphite Reactor. These will be used for fission iodine production and will augment the fuel supply. Modern startup instrumentation, including a fission-chamber log N channel and an ion-chamber log N channel, has been installed. A study on the safety of the enriched Graphite Reactor was completed, and testing of fuel elements continued. A corrugated element and an element with small wires wound on the surface are being compared with a cylindrical element with a smooth surface.

Treatment of a portion of the Laboratory's low-level, large-volume wastes, by a modified lime—soda ash water-softening process, was begun on August 8 in the Process Waste Treatment Plant. During the initial testing period, while adjustments to the process and equipment were being made, a removal of two-thirds of the gross beta activity was observed. These results are only roughly indicative of the efficiency of the plant, since its main purpose is to remove radioactive strontium; more complete analyses are planned for the future.

The Oak Ridge Research Reactor (ORR) - Several items of equipment for the ORR remain to be satisfactorily completed by the building contractor; work continues on these items. The H. K. Ferguson Company is working on building modifications in order to provide an elevator, additional work areas on the second floor, air conditioning, a laboratory and counting room in the basement, a hot cell over the ORR pool, and a helium refrigerator laboratory. This work is progressing satisfactorily and has caused very little interference with the Laboratory's installation program on the reactor.

The installation schedule for the reactor was delayed approximately one month due to difficulties encountered in helium leak testing of portions of the reactor tank. The tank arrived from Boston and was set in place for initial checks on September 20, 1957. Installation work on the reactor tank and on mechanical controls is proceeding, with some overtime being authorized in order to expedite this portion of the work.

Reactor instrumentation and control work is proceeding on schedule.

Fabrication of the large experimental facilities is proceeding slowly at the Paducah plant. One of the large plugs is nearly ready for shipment; work on the second plug is being held up, pending a decision regarding possible modifications which will allow use of this plug in the Pennsylvania Advanced Reactor (PAR) test program.

Hydraulic testing schedules covering the one-month period allotted for this work have been prepared by the Operations Division; tests are scheduled to start on October 15, 1957. Experimental work is in progress to check the proposed method of measuring water velocity through various channels in the reactor core. This method is based on injecting a salt solution into the water stream and measuring the time required for the salt to pass between two electrodes.

Preliminary neutron testing schedules have been prepared by the Operations Division. The schedules are based on testing six lattice configurations and are intended to permit the collection of just enough data to allow operation of the reactor. This work is scheduled to start the latter part of November. The neutron and gamma flux data needed for the experiments now planned are being determined, and, if time permits, the experimenters will be given an opportunity to obtain these data long with tests on the reactor.

PHYSICS

High Voltage Program - By the coulomb excitation of $W^{184}$ it is found that states are excited at 111 and 896 kev. These are interpreted as the first rotational state and the first $2^+$ vibrational state, respectively. The decay rate of the 896-kev state to the ground state is found to be 3 times that expected for an independent particle transition. Similarly, it is found by coulomb excitation measurements that states in $W^{186}$ at 122 and 730 kev are excited. These are interpreted as the first rotational state and the first $2^+$ vibrational state, respectively. Once again the decay rate of the 730-kev state to the ground state is found to be 3 times the independent particle estimate.

A $2^+$ vibrational state has been located in $Os^{188}$ at 633 kev. The decay rate of this state to the ground state is 5 times the independent particle estimate; $2^+$ vibrational states have also been observed in $Os^{190}$ and $Os^{192}$ at 557 and 489 kev, respectively. The observed decay rates of these states to the ground states are approximately 4 times the independent particle estimate. (AEC Activity 5220)

Recoil Spectrometry - The following ions resulting from beta decay in monotritiated methane ($CH_3T$) have been observed with the indicated intensities (%):

| | | | |
|---|---|---|---|
| $H^+$ | 2.5 | $CH_3^+$ | 81.7 |
| $H_2^+$ | 0.14 | $C^{13}H_3^+$, $C^{12}He^3H^+$ | 1.27 |
| $(He^3)^+, H_3^+$ | 0.12 | $(CHe^3H_2)^+$ | 0.06 |
| $(He^3H)^+$ | 0.15 | $(CHe^3H_3)^+$ | 0.06 |
| $C^+$ | 5.0 | $C^{++}$ | 0.08 |
| $CH^+$ | 4.0 | $CH_2^{++}$ | 0.07 |
| $CH_2^+$ | 4.9 | | |

The results indicate that the dominating molecular process that follows the beta decay is one in which the product $He^3$ atom takes its quota of two electrons and leaves the molecule. Less frequently, hydrogens are also shaken off, usually as neutral atoms. There are faint indications that occasionally the $He^3$ may remain bound in the molecule for a sufficient time to pass through the magnetic analyzer.

Three new gamma rays have been found in the decay of $Xe^{133}$ with energies of about 160, 305, and 385 kev. Their intensities relative to 1, for the well-established 81-kev gamma ray, are $10^{-3}$, $10^{-4}$, and $5 \times 10^{-5}$, respectively. The new gamma rays agree nicely in energy with the levels found in coulomb excitation studies of cesium (81 and 163 kev, reported by Temmer and Heydenburg, Phys. Rev. 104, 967, 1956, and 82, 160, 302, and 379 kev, reported by Fagg, Bull. Am. Phys. Soc. 2, 207, 1957). (AEC Activity 5220)

Neutron Diffraction - Neutron diffraction measurements have been obtained on both powdered samples and single crystals of $MnCl_2$ in the liquid-helium temperature region in order to investigate the existence of antiferromagnetic ordering at these low temperatures. The results confirm specific-heat measurements (R. B. Murray, Phys. Rev. 100, 1071, 1955) which exhibit sharp $\lambda$-type anomalies at 1.81 and 1.96°K, by showing transitions to two distinct antiferromagnetic structures at these temperatures. At 1.96°K there is a transition from the paramagnetic state to one antiferromagnetic structure, while in a small temperature region around 1.82°K, there is a reorientation of the atomic magnetic moments into a different type of antiferromagnetic ordering.

Both antiferromagnetic structures are complex and require large unit cells in order to index the observed magnetic reflections. Although $MnCl_2$ crystallizes in the hexagonal layer structure characteristic of $CdCl_2$, it has been impossible to account for the reflections from either antiferromagnetic structure with a magnetic unit cell that is hexagonal. Between the two transition temperatures the results suggest a magnetic structure with an orthorhombic unit cell which grows in antiferromagnetic domains along the hexagonal axes in the base plane of the chemical unit cell. Below 1.82°K a similar domain pattern seems to be required, but in this temperature region a monoclinic magnetic unit cell is necessary in order to explain the observed data. (AEC Activity 5220)

Low-Temperature, Nuclear, and Solid-State Physics - Calculations by use of the angular momentum theory applied to a fissioning aligned nucleus have been carried out and compared with experiments on aligned $U^{233}$ nuclei. When these calculations are specialized to fit the model of A. Bohr, a strong disagreement exists, in that in the Bohr picture the theory predicts a large anisotropy (~15% at 1°K), whereas an almost isotropic distribution was observed.

The anisotropy of alpha particles from $U^{234}$ and $U^{235}$ nuclei at low temperatures has been measured in the manner previously reported for $U^{233}$. Uranium-234, being an even-even nucleus, was not expected to become aligned. An isotropic distribution was observed, as expected. Uranium-235 alpha particles, on the other hand, appear from the data to have an angular distribution given by $W(\theta) = 1 - \frac{0.0683}{T} P_2(\cos \theta)$; this is essentially the same result as that previously obtained with $U^{233}$ and is similar to that previously obtained for $Np^{237}$. Thus three examples now exist where a strong disagreement with the Hill-Wheeler picture of alpha emission from deformed nuclei occurs. (AEC Activity 5220)

Thermal Diffusion - Approximately 200 ml of $A^{36}$ has been enhanced from 0.3 to 30% concentration. The argon plant has essentially reached equilibrium and has been shut down. The contents will be stripped of accumulated air, combined, and charged into a single column for final concentration.

The krypton plant contains about 100 ml of $Kr^{86}$ available at 75% concentration. A recent circulating pump failure and a current heater failure have prevented the attaining of the expected 90+% concentration of isotope 86. Along with the $Kr^{86}$ separation, $Kr^{78}$ has been raised from 0.35 to 5%, and $Kr^{80}$ from 2 to 21%.

A very inexpensive, simple, and reliable separation column, with a standard Calrod for a heater, has been constructed and tested with argon gas. With a separation factor comparable with that attained in a hot-wire column, the mass transport is several times greater and equilibrium time is several times shorter than for hot-wire operation.

There has been some demand for $C^{13}$ and for $O^{17}$ and $O^{18}$. Theory indicates that CO should be a good molecule for separation by thermal diffusion, and three columns, with a 5/16 -in.-dia Calrod hot wall and an 11/16-in.-dia cold wall, have been constructed and are ready to start evaluation of CO gas. This molecule, CO, offers the possibility of separating both oxygen and carbon simultaneously in a single plant. (AEC Activity 5250)

Mass Spectrometry Research and Development - In cooperation with the U. S. Department of Agriculture, the $K^{41}/K^{39}$ ratio was determined for 72 potassium samples. The purpose of this work was to evaluate the use of $Rb^{86}$ as a "potassium isotope" in soil chemistry problems.

The degree of precision required for this work dictated the use of a mass spectrometer capable of the simultaneous and direct comparison of the two individual ion currents. Since no such spectrometer was available, a 6-in., 60-deg spectrometer was designed and built that permitted such signal collection. It was possible to do this with very little capital outlay, since with the exception of the ratio recorder, the spectrometer tube itself, and the vacuum system, the other components were salvaged from an earlier spectrometer.

The resulting spectrometer is a very versatile instrument. It permits rapid removal and replacement of source and collector assemblies; all parts of the spectrometer tube itself, together with the vacuum system, are readily accessible for the interchange and testing of the individual component parts; and, in general, it is well-suited as the basic instrument for use in a mass spectrometric research and development program. (AEC Activity 5250)

Mathematical Research—Mathematics and Computations - A program has been written which utilizes the Oracle compiler to assemble a routine for the purpose of examining the behavior of certain statistics and of their estimated asymptotic variances. The routine will take random samples from a given multinomial population, on which a 4 x n classification may be imposed ($n < 33$),

and compute three statistics, $\hat{I}_2^1$, $\hat{I}_3^1$, $\hat{I}_4^1$, together with their estimated asymptotic variances, and store this information on a 2-in. magnetic tape. The $\hat{I}_2^1$, $\hat{I}_3^1$, and $\hat{I}_4^1$ are the estimates of the three marginal probabilities remaining after the 4 x n classification has been reduced to a 3 x n classification. An example of these statistics is the "corrected incidences" of A. W. Kimball's Disease Estimation Subject to Multiple Causes of Death.

This is an empirical sampling routine which will enable the statistician to solve the distribution problems of the statistics. The analytical solution of the distribution problems has so far been elusive.

To use the routine, a parameter item is prepared (on a standard Multilith blank form) which specifies the population to be sampled. This item, together with ten other standard items, is compiled on the Oracle to provide the computer program. The 8-32 floating-point arithmetic is used throughout. (AEC Activity 5260)

## CHEMISTRY

Chemical Engineering Research - A method of steam-pulsing solvent extraction columns, by using an induction-heated element in the pulse line, was devised and used to operate a 2-in.-dia pulsed column satisfactorily.

A liquid-liquid separator with high throughput and low holdup and pressure drop would be desirable for use in a high-speed solvent extraction contactor. An axial-flow hydroclone, in which turbulence and pressure drop should be lower than in a conventional hydroclone, was designed, and a first model performed satisfactorily with a kerosene-water charge.

A method of measuring an organic-aqueous interfacial area was devised that was based on the $(\alpha,n)$ reaction between $Po^{210}$ alpha radiation in an aqueous phase and fluorine in a fluorocarbon organic phase. The $(\alpha,n)$ activity was counted outside the tube containing the liquids, the specific counting rate being $0.116 \pm 0.009$ count/min per square centimeter of interface with an aqueous phase containing 1.1 mc of $Po^{210}$ per milliliter in contact with completely fluorinated cyclooctane. (AEC Activity 5310)

Americium Recovery - A solvent extraction process is being developed to separate approximately 40 g of $Am^{241}$ from 40 kg of lanthanum and cerium. In batch countercurrent extraction runs made under proposed flowsheet conditions, a partitioning column using 15.5 M $HNO_3$ in four scrub stages and 17 M $HNO_3$ in six extraction stages recovered 99.96% of the americium contaminated with 0.93% of the original lanthanum.

Lanthanum and americium distribution coefficients between tributyl phosphate and 7.5 to 17.2 M $HNO_3$ increased from 0.045 to 0.52 and from 0.165 to 5.2 as the acidity increased from 7.5 to 17.2 M. (AEC Activity 5310)

Chemistry of Corrosion - The theory of the non-steady-state behavior of multicomponent electrochemical systems, especially corrosion systems, was developed. Measurements on certain corrosion systems were made with a new technique based on this theory. Positive and negative currents of equal magnitude are alternately applied to an electrode for fixed periods of time such that the change in electrode potential is less than about 10 mv in each period of polarization. The information obtained in this manner, when analyzed on the basis of the theory, yields directly the differential capacity of the electrode-solution interphase system as well as the rate of the corrosion reaction.

The reversible hydrogen potential was measured on platinum in molten hydrated magnesium chloride. The result is of interest in connection with studies on the theory of stress-corrosion cracking.

The effect of change in pH on hydrogen overvoltage on zirconium was determined. Polarizabilities of zirconium in various oxygen-saturated solutions were also measured. It was found that corrosion rates could be calculated from the data obtained above 60°C, provided that certain reasonable assumptions were made. Below 60°C, difficulties were encountered which prevented accurate estimation of the rates, although the results may be of value in determining the nature of a corroding zirconium surface. (AEC Activity 5330)

Ion Exchange Studies - The cesium-ammonium cation exchange equilibrium was studied as a function of exchanger capacity on a series of variable-capacity resins previously described. Again it was found that the selectivity for the preferred ion ($Cs^+$) increased with decreasing capacity. This result was confirmed for the cesium-sodium, hydrogen-sodium, ammonium-sodium, and cesium-ammonium systems on a series of sulfone cross-linked cation exchangers, and for the cesium-sodium and hydrogen-sodium systems on a series of divinyl benzene cross-linked exchangers of variable capacity.

Distribution coefficients for the partition of tracer quantities of zinc(II) between a sulfonium anion exchanger (Stamex S-44) and various concentrations of hydrochloric acid and lithium chloride were measured. The absorption out of hydrochloric acid solutions was similar to that observed on Dowex 1. The absorption of zinc(II) by Stamex out of concentrated lithium chloride solutions proceeded so slowly that equilibrium was not reached even after 60 days. The large apparent difference between the absorption of zinc by Stamex S-44 and Dowex 1 from lithium chloride solutions may therefore be only a reflection of the difference in the equilibration rate of these two exchangers.

The heats of exchange of $H^+$, $Li^+$, $K^+$, and $Cs^+$ ions with $Na^+$ ions were measured with a series of variously cross-linked Dowex 50 type cation exchangers. Differential values were estimated from the variation of the heats with resin loading, and enthalpy changes for the hypothetical process MR (a = 1, equilibrium with 0.1 N MCl) + $N^+Cl^-$ (a = 1, aqueous) $\longrightarrow$ NR (a = 1, equilibrium with 0.1 N NCl) + $M^+Cl^-$ (a = 1, aqueous) were obtained by integration.

The differential heat-composition curves parallel (with negative sign) the selectivity coefficient variations observed in equilibrium measurements. Free-energy values were computed from the latter and, by using the integral heats of exchange, entropies of exchange were estimated. Representative values are those for the K-Na system where the free energy, heat, and entropy are -170 cal/mole, -290 cal/mole, and -0.4 $cal \cdot mole^{-1} \cdot deg^{-1}$, respectively, for a 2% DVB (divinyl benzene) resin and -410 cal/mole, -1170 cal/mole, and -2.6 $cal \cdot mole^{-1} \cdot deg^{-1}$ for a 24% DVB resin. (AEC Activity 5330)

Characterization of Short-lived Fission Products - Long-lived rhenium activity was produced by neutron irradiation of $KReO_4$, and the decay properties were studied with scintillation spectrometers. The single-crystal gamma-ray spectrum indicated gamma rays of energy 0.062, 0.111, 0.163(?), 0.218, 0.252, 0.320, 0.385, 0.544, 0.646, 0.800, 0.908, and 1.16 Mev. Additional transitions in very low intensity may exist. Two very weak beta rays with energies of approximately 140 and 290 kev were also measured.

A summary of the gamma-gamma coincidence results follows: the 0.111-Mev peak is coincident with gamma rays of 0.062, 0.111(?), 0.214, 0.252, 0.318, 0.388, 0.450, 0.543, 0.640, 0.797, 0.897, and 1.14 Mev; in coincidence with 0.908 Mev are gamma rays of energy 0.062, 0.111, 0.218, 0.251, and 0.320 Mev, respectively; the 0.800-Mev gamma ray is coincident with transitions of 0.062, 0.111 0.185(?), 0.215, 0.248, and 0.320 Mev, respectively.

Most of the above data are characteristic of 50-day $Re^{184}$, although the lifetimes of some of the gamma rays as well as the gross decay followed with an end-window proportional counter indicate a component with a considerably longer half life. This is perhaps either an isomer of $Re^{184}$ or long-lived $Re^{189}$.

Scintillation spectrometer measurements on 16-sec $Tc^{100}$ were continued. The single-crystal gamma-ray spectrum was resolved into ten gamma rays of energy 0.542 $\pm$ 0.005, 0.600 $\pm$ 0.007, 0.71, 0.81, 0.89, 1.01, 1.14, 1.31, 1.49, and 1.8 Mev. Gamma-gamma coincidence spectrometry established coincidences between the 0.600- and the 0.542-Mev gamma rays.

A single-crystal gamma-ray spectrum of 86-sec $I^{136}$ was analyzed into the following gamma-ray components: 1.32 $\pm$ 0.01, 1.55, 1.89, 2.32, 2.61, 2.84, and 3.18 Mev. Other gamma rays were found at 0.21, 0.27, 0.39, and 0.49 Mev, but their relationship to the $I^{136}$ decay was not established. Gamma rays in coincidence with 1.32 Mev were found at 0.20, 0.28, 0.40, 0.58, 0.90, and 1.35 Mev.

A study of the decay of 52-min $I^{134}$ was begun. The single-crystal beta spectrum was resolvable into components of 1.5-, 2.1-, 2.7-, and 3.5-Mev energy. Twelve gamma rays were disclosed by an analysis of the gamma-ray spectrum: 0.14, 0.42, 0.53, 0.62, 0.84, 0.89, 1.07, 1.14, 1.25, 1.44, 1.61, and 1.78 Mev. Beta-gamma and gamma-gamma coincidence spectrometry investigations are in progress. (AEC Activity 5330)

Chemical Separation of Isotopes - The infrared spectra of $N^{14}N^{15}O^{16}$, $N^{15}N^{14}O^{16}$, and $N^{15}N^{15}O^{16}$ were observed from 2.5 to 20 μ by using a model 112 Perkin-Elmer spectrometer. The frequencies of the three fundamental vibrational bands and 15 overtone and combination bands were tabulated and compared with the frequencies of $N^{14}N^{14}O^{16}$. With the use of known anharmonic constants and isotopic relations, approximate zero-order frequencies were calculated and the values were compared with observed frequencies for all the bands of each species. A set of force constants for infinitesimal displacements was computed for the set of molecules, and the partition function ratios for the various species were calculated from the data:

Partition Function Ratios for $N_2O$

| T (°K) | $\frac{N^{15}N^{14}O^{16}}{N^{14}N^{14}O^{16}}$ | $\frac{N^{14}N^{15}O^{16}}{N^{14}N^{14}O^{16}}$ | $\frac{N^{15}N^{15}O^{16}}{N^{14}N^{15}O^{16}}$ | $\frac{N^{15}N^{15}O^{16}}{N^{15}N^{14}O^{16}}$ |
|---|---|---|---|---|
| 273.16 | 1.0874 | 1.1546 | 1.0884 | 1.1557 |
| 298.16 | 1.0772 | 1.1360 | 1.0783 | 1.1372 |
| 500 | 1.0360 | 1.0628 | 1.0355 | 1.0623 |

(AEC Activity 5340)

## METALLURGY

High-Temperature Reactions of Metals and Ceramics - An electron optical study of oxide films on niobium was undertaken to determine the cause of nonprotective oxide formation on niobium. It was found that the previously observed acceleration of the oxidation rate of niobium could be correlated with the formation of small, blisterlike cracks in the oxide film. An oxidation model was proposed to explain the behavior of niobium. The results of a similar investigation of the oxidation of tantalum supported the oxidation mechanism suggested for niobium.

Comparable studies of the microtopography of oxide films on zirconium and vanadium are now in progress. Under the experimental conditions thus far investigated, small, nodular proturberances rather than blisters were the characteristic features of these oxides. The mode of formation of these surface features is not yet understood, but they are such a prominent part of the oxidation process that they will have an important bearing on any oxidation mechanism proposed for zirconium or vanadium. (AEC Activity 5420)

## BIOLOGY

Cytology and Genetics--Plants and Fungi - Combined neutron and x irradiation in *Vicia* gives an additive yield of chromosome exchanges, whereas the exchange frequency with x rays alone is proportional to the square of the dose. The apparent absence of interaction between the neutron- and x-ray-induced breaks can be explained on the basis of the LET (linear energy transfer) difference, provided that the proximity of broken ends must be of the order of 0.1 μ for rejoining to occur, in contrast with the previously accepted value of about 1.0 μ.

The relation of *Tradescantia* RBE (relative biological effectiveness) to average LET, extending over the range from 0.26 kev/μ (gamma rays) to 220 kev/μ (heavy particles), has been derived from additional data and estimates from this laboratory, and from data from an alpha-particle experiment (Kotval and Gray, J. Genet. 48, 135— 154 (1947)). The RBE increases smoothly with increasing LET from gamma rays through x rays and the fast neutron range, peaks somewhere between 50 to 70 kev/μ (between lower energy fast neutrons and alpha particles), then decreases to the LET of alpha particles, and varies little if any at greater LET's. (AEC Activity 6130)

Cytology and Genetics--Insects - Circumstantial evidence obtained in several organisms has suggested that the nucleolus controls to some unknown extent the division of a cell. This idea has been tested in living grasshopper neuroblasts by focusing on the nucleolus a microbeam of high-intensity ultraviolet radiation (less than 3 μ in diameter at the focal point). The source was a 1000-w water-cooled high-pressure capillary mercury arc (G-E AH6), and the flux of radiation at the focal point was about 0.15 erg/$\mu^{-2}$/$sec^{-1}$. Middle and late prophase cells were irradiated: in some the microbeam was focused on a nucleolus and in others on a nonnucleolar portion of the nucleus. These cells and unirradiated cells were observed until they divided, or for at least 24 hr. Results obtained to date reveal that irradiation of one or both nucleoli in late prophase with large doses of ultraviolet retards mitotic rate but does not prevent cell division. In four out of every five middle prophase cells, however, irradiation of one nucleolus for 2 or 3 sec is sufficient to prevent cell division, while irradiation of a nonnucleolar portion causes retardation but does not prevent division. Thus it appears that up to a certain point in the mitotic cycle the nucleolus is important in cell division. (AEC Activity 6130)

Pathology and Physiology - Systematic investigation of the induction of leukemia in mice by x radiation has disclosed marked and unexpected age differences in susceptibility. Newborn mice were found to be refractory to the induction of myeloid leukemia, despite maximal susceptibility to the induction of lymphoid leukemia, whereas in aging mice the reverse was true. These differences point to age-dependent changes in the blood-forming tissues of fundamental importance in their response to irradiation. Review of the literature concerning radiation-induced leukemia in human beings suggests the existence of corresponding age-dependent differences in the susceptibility of man. (AEC Activity 6130)

Mammalian Recovery - Studies were made in order to compare the ratios of $P^{32}$-$C^{14}$ incorporation in spleens from normal mice and from irradiated mice injected with rat bone marrow. Preliminary results indicate good incorporation of these isotopes in the deoxyribonucleic acids of normal mouse spleen.

The spleen and lymph nodes of lethally irradiated mice given isologous spleen treatment intravenously showed a remarkable repopulation of lymphatic tissue elements within 24 hr after exposure. These findings suggest that lymphocytes from the white pulp of the spleen go directly to homologous sites in the lethally irradiated mouse. (AEC Activity 6230)

Radiation Immunology - A study of the relative effects of splenic and bone marrow cells on lethally irradiated mice was undertaken. Circulating antibodies were not detectable among lethally irradiated mice treated with isologous bone marrow and rat red blood cells (RBC) antigen but were detectable among 950-r mice treated with isologous spleen and rat RBC. Approximately 33% of mice exposed to 950 r survived the 60-day period of observation when treated after irradiation with homologous bone marrow, but 100% of those treated with homologous spleen died in 36 days.

A study of the effects of the temporal relation of x irradiation and antigen injection on antibody production in mice was undertaken. Mice were injected with sheep RBC antigen at intervals ranging from 10 days before to 45 days after 710 r of x rays. Suppression of antibody formation was observed in both the pre- and postirradiation antigen groups, with maximum inhibition occurring with antigen injection on 0, 1/2, or 1 day postirradiation. Converse to the general inhibitory effect of x irradiation, mice injected with antigen 5 days before x ray showed a mean peak and mean total agglutinin titer significantly higher than the normal control. During the time interval of study the immune mechanism of irradiated mice never returned to a completely normal status. (AEC Activity 6230)

## PROGRAM 8000 - WORK FOR OTHERS

Oak Ridge School of Reactor Technology - On August 19 - 20, 1957, ORSORT held an Information Meeting at which the students reported on their summer design studies. On August 26, the last full-year session of ORSORT ended with 111 students successfully completing the course. During August and September, 51 students reported to the six cooperating universities for the second session of the ORSORT-University Cooperative Program. On September 9, 76 students reported to ORSORT for the second half of the first session.

More than 7000 brochures were mailed to some 2600 industrial organizations, government agencies, and ORSORT alumni in preparation for the third session of the program. This session will begin in the spring of 1958, and the deadline for receipt of applications is November 18, 1957. (AEC Activity 8712)

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## VISITORS

FOREIGN VISITORS:

Allan, D. L., United Kingdom AEA, Great Britain
Allen, K. W., United Kingdom AEA, Great Britain
Barclay, Fred J., United Kingdom AEA, Great Britain
Batchelor, R., United Kingdom AEA, Great Britain
Bauer, F. L., Technischen Hochschule, Germany
Beckurts, K. H., Max Planck Institute, Germany
Boon, E. F., Reactor Centre, Netherlands
Bottenbruch, H. H. F. W., Technischen Hochschule, Germany
Carman, P. C., South African Council for Scientific and Industrial Research
Coltas, E. K., Atomic Energy of Canada
Cox, B., AERE, Harwell, Great Britain
Dawson, J. K., AERE, Harwell, Great Britain
Deuchars, W. M., United Kingdom AEA, Great Britain
Emendorfer, Dieter, Technische Hochschule, Germany
du Toit, S. J., Atomic Energy Board of South Africa
Endo, Y., Japan Atomic Energy Research Institute, Japan
Ferguson, A. T. G., United Kingdom AEA, Great Britain
Freeman, J. M., United Kingdom AEA, Great Britain
Funk, T., Hochtief Aktiengesellschaft, Germany
Groth, W., University of Bonn, Germany
Groven, L., Belgium
Hamaker, J., Reactor Centre, Netherlands
Hannerz, A. K., ASEA Co., Sweden
Hocker, K. H., Technische Hochschule, Germany
Jones, J. S. M., Humphreys and Glasgow, Great Britain
Komaniya, Y., Japanese Government (Harvard University)
Latif, R. A., Iraqi Government, Iraq
Leopold, Former Ruler of Belgium
Liden, Kurt, University of Lund, Sweden
Lindner, H. R., Chalmers University of Technology, Sweden
Lynn, J. E., United Kingdom AEA, Great Britain
Mallison, W. T., Asian-African Branch, Division of International Affairs
Marcus, Y., Massachusetts Institute of Technology (Citizen of Israel)
McEndhill, John J., United Kingdom AEA, Great Britain
McNally, T. J., Atomic Energy of Canada
Miyauchi, T., University of California and University of Tokyo, Japan
Nel, R. J., South African Embassy
Nordemann, J. G., Dekker and Nordemann, Amsterdam, Holland
Olsen, J. L., Eidg Technische Hochschule, Germany (Citizen of Great Britain)
Osborn, V. H., Atomic Energy Board of South Africa
Porter, G., University of Sheffield, Great Britain
Rinderer, L., University of Louisiana, Switzerland (Citizen of Austria)
Roux, A. J. A., Atomic Energy Board of South Africa
Sago, M., University of Michigan (Citizen of Japan)
Schonland, B. F. J., AERE, Harwell, Great Britain

VISITORS (Continued)

Schutte, W. H. W., Technischen Hochschule, Germany
Sivararmakrishman, V. M., ORINS (Citizen of India)
Skjoldsbrand, R. H., AB Atomenergi, Sweden
Solzay, J. E., Belgium
Sumita, K., Japan Atomic Energy Research Institute, Japan
Takahashi, S., Mitshuhushi Metal Mining Company, Japan
Trumpy, G., JENER Co., Norway
Van Arkel, A. E., University of Leiden, Holland
Van Dorsser, A. H. de H., Reactor Centre, Netherlands
Van Rhijn, A. J. R., Atomic Energy Board of South Africa
Venter, J. A., Atomic Energy Board of South Africa
Yasuda, K., Daiichi Bussan Company, Japan

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