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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

OCTOBER 1957

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STATUS AND PROGRESS REPORT

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STATUS AND PROGRESS REPORT

October 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

Distribution and Excretion of Uranium in Man - The basic exposure facility to be used in the study of retention of particulate uranium compounds vs particle size and time has been completed. The initial work of evaluating the equipment and the procedures for the exposure, the sampling, and the analysis has begun.

A device for measurement of the enriched uranium body burden of small live animals has been constructed and tested. The basic purpose of this model is to explore the feasibility of a device for measurement of the uranium body burden of humans. Thirty microcuries of enriched uranium can be detected by an increase in count rate of (7.8 ± 1.3) % of background.

A new mathematical procedure for fitting nonlinear curves has been derived. The procedure, called the "fractionator method", has been successfully tested with a linear combination of exponentials, a function which has been extremely hard to analyze by the available statistical methods. (AEC Activity 2120)

Chemical Plant Criticality Studies - In the program of critical experiments with aqueous solutions of uranium enriched to 93% in U^{235}, a study is being made of the critical dimensions of annular cylindrical volumes in order to investigate more economic processes for the preparation of fissionable materials. In the current experiments both the thickness of the annular space between two aluminum cylinders and the outside diameter of the array are being varied. The inner cylinder is lined with cadmium sheet and filled with water. Measurements with solutions having the concentration that yields minimum critical volumes show that 2.5-in.-thick annuli with outer diameters in the range of 15 to 30 in. will be subcritical at infinite heights when surrounded by water. Arrays having 3.5-in.-thick annuli and 15- to 30-in. outside diameters will be subcritical when unreflected. The effective areas of these vessels are to be compared with presently accepted "safe" cylinder diameters of 5 in. (reflected) and 8 in. (unreflected). (AEC Activity 2712)

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HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test - Test run No. 9 was terminated on September 30 after 155 hr of operation on condensate above 250°C. The fuel dump tanks were thoroughly rinsed to remove any remaining portion of the alkaline material leached from the iodine removal bed in the fuel recombiner. After installation of new diaphragm feed-pump heads of improved design and remachining of the leaking core-blanket rupture-disk holder, a new charge of condensate was added, and the startup for run No. 10 was commenced on October 3. However, on the following day a high pH was again observed in the fuel dump tanks, so it was decided to remove the fuel recombiner assembly. When the run was resumed on the evening of October 4, no further buildup of pH in the system was observed; the chemical separations facility was tied into the reactor, and water operation was continued for two more days.

On October 7, 5.5 kg of depleted uranium was injected into the reactor, and operation above 250°C was maintained for an additional 79 hr. At the end of this period a leaking chemical-plant sample valve, which allowed fuel solution to leak into the low-pressure and off-gas systems of the chemical plant, was replaced, and several minor alterations were made in the reactor system in preparation for a final run with depleted uranium.

The reactor was operated continuously at design temperature and pressure throughout the third week in October. No significant difficulties were experienced during this additional 160-hr period, so operation was terminated to permit preparation for the final leak test of the combined reactor and chemical plant with Kr^{85} tracer gas. (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 the flexibility of the adjoining piping were required before the high-pressure screen could be removed. It is now believed that all components designed to be replaceable can be removed and reinstalled underwater as proposed.

The final test of the hydroclone system was performed in conjunction with the reactor over a 300-hr operating period. Operation was normal except for an incident following a sampling procedure in which both high-pressure sample valves leaked seriously, permitting fuel solution to pass to the low-pressure system. The system was isolated from the reactor, and one leaking valve was replaced.

Holdup tests of the charcoal adsorber beds are in progress to ascertain whether voids exist which might short-circuit active gases. Beds C and D were shown to be adequate; tests on the other two beds, although they appeared to be satisfactory, will be repeated to obtain more accurate data. (AEC Activity 4103.1)

Uranyl Sulfate Blanket Processing - Several experiments were carried out in the P-1 loop in which ZrO_2 or $Zr(SO_4)_2$ was added to 1.4 m UO_2SO_4 circulating at 250°C under 350 to 400 psi of oxygen. The zirconium was substituted for plutonium, which will be added in later experiments. When preformed ZrO_2 was added, the half-time of ZrO_2 disappearance from the circulating solution was 2 to 4 hr either with or without the hydroclone operating. Only 6 to 16% of the ZrO_2 was removed by the hydroclone, and 85 to 90% was removed by adsorption on the loop walls. When the ZrO_2 concentrations were 10 to 100 mg/liter, the calculated hydroclone efficiency was 10 to 20%. When dissolved $Zr(SO_4)_2$ was allowed to hydrolyze in the circulating solution, essentially all the zirconium was adsorbed, and only a trace was removed in the hydroclone.

In the spectrophotometric studies of plutonium solutions, the narrow absorption peak at 835 mμ used for determination of the plutonyl ion was shown to be a nonsymmetrical doublet with a width of 40 Å at half peak height. Both peaks followed the ideal absorption laws. When 770 mg/liter of Pu(IV) in 1.4 m UO_2SO_4 was heated to 90°C, it was partially disproportionated to Pu(III) and Pu(V). Under these conditions, Pu(V) (or PuO^+) is apparently stable. (AEC Activity 4103.1)

Thorium Oxide Blanket Processing - The adsorption of neodymium from dilute neodymium nitrate solution on thorium oxide in aqueous slurries at 250°C decreased as the ThO_2 calcination temperature increased. Thorium oxide calcined at 650°C adsorbed 7400 ppm Nd from 0.006 m $Nd(NO_3)_3$ at 250°C, but that calcined at 1100°C adsorbed only 500 ppm Nd. When PbO was coslurried at 250°C with ThO_2 in dilute solutions of uranium, cerium, or neodymium, >99% of the cations were adsorbed on the PbO, and adsorption on ThO_2 was decreased more than tenfold.

Cerium already sorbed on ThO_2 was 85 to 99% desorbed by coslurrying the ThO_2 with PbO at 250°C and then selectively dissolving the PbO in 4 N HNO_3. When irradiated ThO_2 was treated in this way, 8.4% of the protactinium was removed. (AEC Activity 4103.1)

Thorium Oxide Slurry Development - Sufficient thorium oxalate for conversion to 700 lb of 1-μ ThO_2 was prepared from $Th(NO_3)_4$ and oxalic acid by jet precipitation. Synthetic flocculating agents greatly increased the settling rate of the thorium oxalate during the precipitation but did not affect the chemical or physical properties of the product.

Ultrasonic energy dispersed dry ThO_2 agglomerates to individual 1-μ particles. The oxide prepared by firing thorium hydroxide was gritty and glass-like and composed of large agglomerates. By the gel technique, 3.6 kg of 10- to 20-μ-dia thoria beads was prepared. The beads aggregated on drying.

In irradiated 250, 500, and 700 g Th/Kg H_2O slurries of ThO_2 containing equal ratios of enriched uranium to thorium, gas production and recombination rates, respectively, were directly dependent on power density and slurry concentration. In out-of-pile studies, calcination of thorium-uranium oxides for 16 hr at 1000°C was necessary for satisfactory H_2-O_2 reaction rates (10 moles of hydrogen per hour per liter

at 280°C; hydrogen pressure, 500 psi) with aqueous slurries of this material containing 0.05 m MoO_3. A slurry of 800°C-calcined ThO_2—0.5% U^{235} prepared from the coprecipitated oxalates and containing 20,000 ppm MoO_3 and 3000 ppm sulfate as $Th(SO_4)_2$ was irradiated in the LITR for 343 hr without damage.

Heating 650- and 800°C-fired oxide for 1500 hr in water at 300°C did not affect the crystallite size or specific surface area, but the average particle size of the 650°C-fired oxide was greatly decreased.

Isotherms for CO_2 adsorption on thoria at 4°C showed an apparent surface saturation of four hydrogen ions and two sulfate ions per surface site. This is tentatively explained as a displacement of two hydroxyl groups per surface site by hydrogen sulfate groups. (AEC Activity 4103.1)

HRP Chemistry: Fuel Studies - Two-liquid-phase transition temperatures were determined for the system $CuSO_4$-UO_2SO_4-H_2SO_4-H_2O. These data indicate maximum temperature and composition limits within which particular homogeneous reactor fuels can be used without the appearance of a solid or a second liquid phase. Briefly, in the region of HRT solution composition of $CuSO_4$, UO_2SO_4, and H_2O, an excess amount of H_2SO_4 is necessary, both to eliminate the formation of basic (as opposed to acidic) uranium and copper solid compounds and to raise the two-liquid-phase transition temperature. (AEC Activity 4103.1)

HRP Analytical Chemistry - A volumetric method by which macro quantities of bismuth are titrated with standard ethylenediaminetetraacetic acid (EDTA) to a visual end point of pyrocatechol violet was adapted for use in the determination of from 20 to 100 μg of bismuth in uranyl sulfate solutions. A spectrophotometric method was used to establish the end point, since the visual end point is indistinct and may be obscured by colored ions.

An improved method was developed for the separation of from 10 to 300 μg of ruthenium as the nitroso salt, chloride, or sulfate from uranyl sulfate; in this method ruthenium is volatilized by pyrolysis at 1000°C in a stream of moist oxygen.

Improved separation of fluoride from stabilizing agents such as clays and related materials was accomplished by a novel technique, namely, fusion of the clays with Na_2WO_4 plus WO_3 and pyrolysis of the fused material at 825°C in a stream of moist oxygen.

An anion exchange technique can be applied to the separation of thorium from cobalt by reaction of the cobalt with 1-(2-pyridylazo)-2-naphthol (PAN). The cobalt-PAN chelate is extracted into a small volume of chloroform, after which the absorbancy of the chloroform extract is measured. From 0.5 to 7 μg of cobalt can be estimated by this procedure with a coefficient of variation of about 5%. (AEC Activity 4103.1)

Slurry Component Development and Slurry Testing - Run 200-A-14 has been continued for over 2100 hr at 300°C, with no apparent change in the physical properties of the slurry or in the appearance of the particles other than a slight rounding-off of the corners. Samples of this 1600°C-fired oxide withdrawn during the first 1500 hr of circulation all showed mean particle diameters of 2.5 μ, indicating no noticeable degradation. (AEC Activity 4103.1)

Slurry Blanket System Development - After run SM-3 the slurry blanket system was overhauled, and a titanium impeller was installed in the pump. Run SM-4 was started as a continuation of SM-3 and has been in progress for 800 hr. Repeated measurements of the gamma-ray transmission through the slurry, with improved techniques, indicate that a region of higher-than-average thorium concentration exists in the upper hemisphere at all flow rates tried. As before, reduction of the flow to about 230 gpm brought about marked differences in the thorium distribution, although there was no evidence of a settled bed at the bottom.

Microscopic examination of the slurry showed the gradual growth of spheroids about 16 μ in diameter in this sulfated slurry made from 800°C-fired oxide. (AEC Activity 4103.1)

Blanket Engineering Research - The high-pressure capillary-tube viscometer has been completed, and shakedown runs were made from 200 to 250°C, the contents of one of the circulating loops being utilized as a supply of slurry.

From pressure-drop flow measurements made with a slurry containing 685 g of Th per kilogram of H_2O, it is apparent that the substitution of the coefficient of rigidity for the viscosity in the calculation of the Reynolds number yields a unique curve for the Fanning friction-factor plot in the turbulent range. This curve lies very nearly on top of the corresponding curve for Newtonian fluids in smooth pipe. (AEC Activity 4103.1)

Core Development - A 4.9-in.^{2} steam-heated disk was constructed for use in determining the inside-wall heat transfer coefficient at various locations on a full-scale low-pressure model of the HRE-3 core tank. Flow rates will be varied up to 6000 gpm. (AEC Activity 4103.1)

Centrifugal Circulating Equipment - The 400A-1 pump and loop is being operated on gas-saturated (O_2 + N_2) 0.04 m fuel solution to determine whether the gas will come out of solution. If it were to do so, it might cause localized impeller attack, gas-binding of the pump, or gas-binding of the motor. The pump has operated for 650 hr with no apparent ill effects.

The 400A-2 pump was operated with the same shaft seal configuration as that installed on the HRT pumps. Mixing rates of 0.48 liter/hr and of less than 0.002 liter/hr were obtained with purge rates of 0 and 10.2 liters/hr, respectively.

The 300A-1 pump has been operated in the HRT mockup for 3109 hr since it was last repaired.

The 4000-gpm pump was shut down because of the failure of the electrical terminals after 114 hr of operation. The pump was repaired and has since run for 500 hr. Mixing experiments similar to those run on the 400A-2 pump are presently being conducted.

The 20-cfm blower was run for approximately 50 hr with ThO_2 slurry in the bearing fluid in an attempt to evaluate a hydrostatic bearing for slurry. With the existing system it was impossible to maintain the slurry in suspension. (AEC Activity 4103.1)

Miscellaneous Equipment - Construction of the thermal-cycle test facility was virtually completed, except for instrumentation.

The experimental stainless-steel-to-titanium transition joint was thermally cycled successfully in the flange test facility. The leakage rates were as low as those observed with all-stainless-steel flanges. Fabrication of an all-titanium letdown heat exchanger for the HRT mockup is in progress, the new transition joint being utilized.

The new HRT flange bolts were "run in" by being loaded three to seven times until their torque-load properties were uniform. A test of 19-9 DL (a high-strength austenitic material) flange bolts indicated that they can be used in joints where differential expansion between the bolting and the stainless steel base metal is undesirable. Preliminary tests of stainless steel flanges assembled with standard steel bolts without ferrules, loaded initially to about 45,000 psi, indicated leak rates approximately the same as those in tests with ferrules. (AEC Activity 4103.1)

Heat Removal Equipment - Fabrication of an all-stainless-steel (type 347) heat exchanger to replace the one which failed on the HRT mockup is under way.

Five small tube bundles are being thermally cycled in boiler water containing 100 ppm Cl and 50 ppm O_2. No failures occurred during the first 1000 hr of the test. (AEC Activity 4103.1)

Gas Handling Equipment - The characteristics of a water-jet gas ejector were determined at pressures up to 1500 psi in order to evaluate the usefulness of such devices in HRE-3 pressurizers. The hydraulic efficiency of the ejector increased from 5% at atmospheric pressure to 26% at 1500 psi; this performance is considered to be promising.

Syracuse University reported that tubing can withstand considerably greater transient (detonation) loading than static loading. The pressures developed during partial and complete explosions of H_2-O_2-steam mixtures were calculated. Measurements of reaction, explosion, and detonation limits are being continued. (AEC Activity 4103.1)

Fuel Systems Development - The HRT mockup Calrod heaters, many of which had burned out, were replaced with strip heaters. A suspected leak of uranyl sulfate (mentioned in the ORNL Status and Progress Report, August 1957, ORNL-2393) was attributed to solids leaking from the Calrod heaters rather than from the system. A new run on the mockup has been started, in which 0.04 m UO_2SO_4 is being circulated. This run has been continued for 1017 hr.

Further iodine removal tests in the mockup low-pressure system gave the following results: silver-alundum bed at 150°C, 98% efficiency; silver-alundum bed at 120°C, 83% efficiency; and silver-plated Yorkmesh bed at 120°C, 97% efficiency.

A small side stream from the high-pressure system of the mockup was boiled in a 1/2-in. sched-80 titanium pipe for 450 hr. A thin scale, composed mainly of an unidentified aluminum compound with some TiO_2 and Fe_2O_3, was found in the boiling section after the test. (AEC Activity 4103.1)

Feed Pump Development - One of six diaphragm feed-pump heads in operation suffered a fatigue failure in its seal weld, probably as a result of improper bolting assembly. No diaphragm or check-valve failures occurred. The heads now being tested have operated for 7847, 6111, 4427, 4427, 2184, and 713 hr, respectively. Two rebuilt purge-pump heads containing 100-mesh screening and experimental diaphragms and valve trim have been under test for 556 hr.

Fatigue tests of types 347, 316, and AM350 stainless steel sheet in air, water, and fuel solution demonstrated that annealed type 347 stainless steel suffers by far the least loss of endurance limit due to corrosive environment. Extrapolation of data obtained thus far indicates that annealed type 347 stainless steel is the best choice for diaphragm material.

The turbine pump has pumped 15 gpm of water against a 300-psi head for 207 hr. It will begin pumping fuel solution after the shakedown run is completed.

The Stellite-piston pump was fitted with four Haynes alloy No. 25 rings and was placed in operation, pumping 1 gpm of fuel solution against a 500-psi head.

A slurry check-valve-material test loop was constructed and will commence operation with slurry at the completion of a shakedown on water. (AEC Activity 4103.1)

Gaseous-Fission-Product Disposal - Dynamic studies of the adsorption of fission gases on HRT charcoal showed that xenon was adsorbed 16 times as strongly as krypton. In equilibrium adsorption tests, Linde molecular sieve 5A had 25% of the capacity of charcoal for krypton adsorption. (AEC Activity 4103.1)

PACKAGE REACTOR DEVELOPMENT

Army Package Power Reactor (APPR-1) - The Laboratory continued to furnish technical support to the Army Reactors Branch (AEC) on the 10-Mw pressurized water reactor being tested at Fort Belvoir, Virginia, by Alco Products, Inc. The six-month test operation and training program which started July 25 was continued without major difficulties. The accumulated thermal power production is now over 2 Mw-years, or approximately 13% of the anticipated core life.

A control-rod absorber section incorporating Eu_2O_3 in a matrix of iron was fabricated and shipped to Alco Products, Inc., for evaluation in their critical facility. Silicon-free type 304 stainless steel powder is being obtained for fabrication of absorber sections of Eu_2O_3 dispersed in stainless steel. A control-rod fuel assembly of a new type, containing depleted uranium dispersed in type 347 stainless steel and incorporating the Eu_2O_3—elemental stainless steel flux suppressor, was completed.

Two depleted UO_2—type 347 stainless steel stationary fuel elements were found to meet all dimensional requirements after brazing. Work was initiated to develop a method of end-box attachment which eliminates plate warpage encountered with the present procedure.

In the APPR-1 radiation damage program, irradiation of boron absorber samples was continued at the MTR. Analytical procedures are being developed for the determination of boron burnup in irradiated samples. Postirradiation examination and analysis of previously irradiated fuel samples were continued. (AEC Activity 4603.1)

Gas-Cooled Reactor Development - The Laboratory is supplying technical assistance to the Army Reactors Branch (AEC) in its program to develop small closed-cycle gas-cooled reactors for military and civilian applications. A 2-Mw Gas-Cooled Reactor Experiment (GCRE) is being designed and built by Aerojet-General Corp. at Arco, Idaho. The Laboratory reviewed the preliminary GCRE control system and the tentative fuel element design and submitted recommendations. Materials for advanced-concept GCRE fuel elements were selected, and plans were made for the fabrication of test samples. An initial measurement in an experimental study of the escape of fission products from clad fuel elements indicated that a negligible amount gets through a 5-mil stainless steel cladding in one week at 1650°F.

Design and component procurement were continued for the gas-cooled in-pile loop which will be installed in the Oak Ridge Research Reactor. Because efforts to locate a satisfactory outside supplier for one of the major components, the economizer, do not look promising, the Laboratory has undertaken the design of this component. A nuclear mockup of the in-pile section was completed for use in ORR preoperational tests. (AEC Activity 4603.1)

Maritime Ship Reactor (MSR) - As part of its effort to provide technical support to the Maritime Reactors Branch (AEC), the Laboratory began a technical review of drawings and specifications for the 80-Mw, slightly enriched,

pressurized-water reactor being furnished by Babcock and Wilcox Co. for the first nuclear merchant ship. Preliminary specifications of the pressure vessel, pressurizer, and primary water purification system were reviewed, and recommendations were submitted. Recommendations were also made concerning an analog computer being procured by the Maritime Reactors Branch. Other areas in which the Laboratory can support the Maritime program are being explored. (AEC Activity 4603.1)

GENERAL REACTOR RESEARCH

Civilian-Power Advanced-Type Gas-Cooled Reactor (GCR) - Work on the enriched GCR was initiated in September, with the object of achieving a power plant that would give the lowest-cost power obtainable from this type of reactor and would, at the same time, be as safe a system as possible. A survey of the available literature indicated that capital costs for a Calder Hall type of reactor would be roughly six times the fuel costs; hence, fuel enrichment can be used most effectively to cut capital costs by reducing the core size and increasing the temperature level. The latter would greatly simplify the steam system, reduce the capital investment, and increase the over-all thermal efficiency.

The information available on metallic uranium fuel elements indicates that these are barely adequate for Calder Hall temperature levels; however, UO_2 appears to be promising for central fuel element temperatures up to 1800°F. Results of heat transfer, thermal stress, and mechanical design studies, coupled with some experimental data, indicate that the UO_2 will be fractured by thermal stresses at the power densities required for good economy; however, if the UO_2 is contained in an adequate can, such cracks will probably not have serious consequences. If the UO_2 can be fabricated to 94% or more of the theoretical density, the fission-product gas evolution will cause little increase in pressure within a sealed can. All reactor designs to date have been based on the premise that the blowers, heat exchangers, and other items external to the reactor must be readily accessible for maintenance; hence, canning of the UO_2 is required in order to prevent the spread of particulate activity.

A reference design has been prepared as a basis for cost and performance studies. The range of values for the more important parameters is given below:

Gas coolant	He or CO_2
Gas system pressure	100 to 400 psi
Fuel enrichment (maximum)	2%
Fuel element	Canned ceramic
Moderator	Graphite
Reactor heat output	360 Mw
Peak fuel element can temperature	1200°F
Peak helium temperature	1050°F
Helium temperature rise through reactor	400 to 650°F
Steam temperature out of superheater	750, 850, and 950°F
Steam pressure out of superheater	600, 900, and 1200 psia

The effects of variations in these values on performance and costs are being evaluated. Every effort will be made to make the costs directly comparable for the full range of conditions. (AEC Activity 4119)

Molten Salt Reactor Program - A civilian molten salt reactor program has been initiated to take advantage of the know-how developed in the project associated with the Aircraft Reactor Experiments (ARE). The use of molten salts as reactor fuels is expected to provide the benefits of a fluid fuel in an unpressurized system operating at temperatures high enough to generate 1000°F steam for the efficient production of power. The wide range of solubility of uranium and thorium fluorides in the fluoride salts is a unique feature that allows a variety of reactor types of be considered. The considerable chemical, metallurgical, and engineering experience in handling molten salts gives the system some advantage with respect to other high-temperature fluid systems.

Design studies of a reactor plant comprising a two-region homogeneous molten fluoride reactor and generating 240 electrical megawatts were carried to the point of making preliminary cost estimates. Power costs of 8.4 mills/kwhr were estimated, based on a 57-million-dollar plant and a fuel-cycle cost of 2.5 mills/kwhr. The achievement of these costs requires the completion of a development program to demonstrate the long-term corrosion resistance of the container materials selected for this service and to find satisfactory ways of servicing the reactor fuel system remotely.

Two Inconel thermal convection loops operating with a peak temperature of 1250°F and a temperature drop of 170°F have completed 1000-hr tests. Examination revealed no detectable corrosion. The fuel salts used in these tests were mixtures of NaF with either ZrF_4 or BeF_2. One mole per cent of UF_4 was used in each test. These results were more encouraging than was expected with Inconel at these temperatures, and they lead to optimistic expectations that the more resistant nickel-molybdenum alloys now being tested will have good long-term corrosion resistance. Expansion of the corrosion test program is under way.

Critical inventories and conversion ratios were computed for the homogeneous 240-Mw (electrical) reactor in which the fuel is UF_4 dissolved in binary mixtures of BeF_2 with Li^7F. The diameters of the spherical cores were varied between 5 and 10 ft; the thorium concentrations in the core were varied from 0 to 1 mole %. The blanket consisted of 25 mole % ThF_4 in Li^7F. The volume assumed for the external fuel circuit, including piping, pumps, and heat exchangers, was 340 ft^3.

In clean systems fueled with U^{235}, a minimum total inventory of about 100 kg was found at a core diameter of 9 ft (with zero thorium concentration); neutron absorption in the blanket yielded a conversion ratio of about 0.3. By adding thorium to the core, conversion ratios up to 0.65 were obtained with critical inventories ranging up to 400 kg. Uranium-233, were it available, would be a much superior fuel. For example, at a given conversion ratio, the critical mass is about one-half that required for U^{235}, and breeding ratios approaching 1.0 can be obtained.

In the near future, the performance of these reactors will be calculated as the fission products and higher uranium isotopes build up during power operation. The effects of these poisons on U^{235}-fueled reactors are expected to be compensated for in large measure by the addition to the core of the U^{233} that has been generated, since it is a much better fuel than U^{235} in these reactors.

A study has been made of two thermodynamic systems for producing power from a molten fluoride reactor capable of producing 600 Mw of heat. The first heat transfer system involves the transferring of heat, in turn, from fuel salt to coolant salt to sodium to water, achieving a turbine steam condition of 1800 psia, 1000°F, and reheat to 980°F. The cycle efficiency is 43.1%, corresponding to a turbine heat rate of 7920 Btu/kwhr. The second system transfers heat from fuel to coolant salt to mercury to water. Mercury vapor at 180 psia and 1000°F expands through a turbine, exhausting at 7 psia and 605°F. The condensing mercury generates steam at 1250 psia, which is superheated to 950 deg by mercury vapor bled from the turbine and direct from the line. The mercury turbine produces 35% of the power. The binary cycle efficiency is 49.3%, corresponding to a turbine heat rate of 6940 Btu/kwhr.

The high cycle efficiency of the mercury system tends to compensate for the high investment required, so that the production costs for the two systems are comparable. The heat transfer areas per electrical megawatt of capacity are nearly the same for both systems. For the sodium system, the heat transfer area per megawatt between the fuel and the steam is one-third to one-fourth that used in a fossil-fuel furnace. (AEC Activity 4201)

Basic Reactor Research: Experimental and Theoretical - A detailed study of the effect of interference between potential scattering and resonance scattering on the resonance absorption of neutrons was completed. Analytical results were obtained for the homogeneous and heterogeneous resonance integrals in (1) the narrow resonance approximation and (2) the infinitely heavy absorber approximation. One of the consequences of this study was the conclusion that, for lumps of heavy elements ($A \sim 240$) with surface-to-mass ratios greater than about 0.1 cm^2/g, the geometric dependence of the resonance integral is given quite well by a formula proposed by Wigner (e.g., L. Dresner, Nuclear Sci. and Eng. 1, 501, 1956). A comparison made by E. Hellstrand (private communication) with some of his own experimental results for U and UO_2 gives excellent agreement.

A measurement of the spectrum of prompt gamma rays having energies between about 0.4 and 2.3 Mev resulting from the fission of U^{235} was made with a Compton two-crystal scintillation spectrometer. During about seven days of operation, approximately 3×10^5 sec of usable data were obtained on the spectrum itself, yielding adequate counting statistics. Appropriate calibration and check data were also obtained to enable correction and analysis of the spectrum. Since even a preliminary analysis is difficult, it is at present impossible to indicate the degree of agreement between this and previous measurements.

The reactivity effect of a uniform distribution of boron carbide (B_4C) in the core of the BSR was determined experimentally by means of the inhour equation. A thin strip of B_4C-impregnated Plexiglas was inserted into each fuel element. As each strip was removed, the reactor period caused by its removal was measured, and the reactivity worth of each B_4C strip was then obtained from the inhour equation. Since the ratio of the nonleakage probability of prompt neutrons to that of delayed neutrons appears in the inhour equation, the experimental results depended on the value chosen for this ratio. Two values were calculated. The total reactivity effect of the distributed B_4C was calculated from the change in thermal utilization. The calculated value of $\Delta C/C$, where C is the criticality constant, was $(1.33 \pm 0.07)\%$; the experimental value was $(1.36 \pm 0.19)\%$. (AEC Activity 4202)

Fuel Element Development - The objective of the present program has been to develop a satisfactory fuel material for aluminum plate-type elements which contains uranium with a 20% enrichment of the U^{235} isotope. The 20% enrichment limitation presents fabrication difficulties because of the higher concentration of uranium required. Investigations are currently being conducted on 48 wt % U—Al alloys and on dispersion-type fuels containing uranium oxides or uranium carbides.

Further investigations to minimize the localized thinning of the cladding in composite aluminum fuel plates containing the 48 wt % U—Al alloy have been confined to increasing the ductility of the fuel alloy. It was found that a 3% addition of silicon completely suppressed formation of the UAl_4 intermetallic compound, with retention of the UAl_3 compound in the phase structure of the alloy. This modification decreases the volume of brittle intermetallic compound present by 18%, with a resultant improvement in fabricability. Mechanical property tests revealed a decrease in room-temperature yield strength from 18,000 psi for the unmodified alloy to 12,000 psi for the alloy containing 3% silicon. The decreased yield strength has been reflected in the reduction of edge cracking which occurs during the rolling of aluminum-uranium alloys containing 40 to 50 wt % U, and it is anticipated that improvements in the fabrication of fuel composites will be obtained by substitution of the 3% silicon—modified alloy for the unmodified 48 wt % U—Al alloy. Minor ternary additions of tin, germanium, zirconium, and titanium also appear to suppress the UAl_4 intermetallic compound in this alloy system.

Efforts have been directed toward preparing a fuel element, containing U_3O_8 enriched in the U^{235} isotope at the 20% level, for irradiation testing in the MTR. Difficulty was experienced with contamination of the inactive plate ends of the fabricated plate by the fuel. Preliminary results revealed that masking the billet frame before insertion of the fuel core prevented the spread of U_3O_8 particles on the surfaces of the aluminum frame. Radiographic examination of plates fabricated from billets prepared by this technique showed containment of the U_3O_8 within acceptable limits.

Composite fuel plates in which the fuel cores consisted of dispersions of 50 wt % UC_2 in aluminum have exhibited a marked propensity to blister

during the heat treatments required in processing. Metallographic examination of the composites has revealed that gas pockets occurred in the fuel material; the pressure created evidently caused localized blisters on the surfaces of the plates. Gas analyses of the blistered portion showed evidence of hydrocarbons. (AEC Activity 4203)

Waste Disposal Research and Engineering: Disposal of High-Level Radioactive Wastes by Sintering - Pilot Pit No. 2, a large-scale test of the sintering method for the fixation of high-level waste, was operated for 48 days. The maximum temperature achieved in the 6-ft-dia by 2-ft-thick cake was approximately 560°C, with the average temperature in the cake being approximately 470°C. The cake temperature ceased to rise when the power input dropped below the fission-product decay curve due to heater failure after 21 days of operation. (AEC Activity 4305)

Radiation Damage: Advanced Engineering and Development - Work has been continued on the in-pile helium liquefier. Tests have been made in order to determine what pressure might be obtainable on the discharge side of the Joule-Thomson expansion valve under operating conditions. The results indicate that a pressure lower than one-third of an atmosphere will be reached, and hence bombardments at temperatures below 4°K may be possible. Additional tests have been made in order to determine whether a 1-hp vacuum pump can be used as a compressor discharging at 30 psig. Mechanically, the results look promising; however, the problem of removing the heat of compression from the helium must be solved.

A design for a radiation calorimeter has been essentially completed, and construction has been started. This calorimeter will be used to study the stored energy released in bombarded metals through the temperature range of 80 to 300°K.

An overhaul of the refrigerator operating the hole 12 cryostat has been completed. Due to a faulty fume filter which allowed oil carry-over during the first run, additional maintenance work had to be done.

From this first run after the overhaul, some survey-type data on the damage rate and on subsequent annealing of a number of metals were obtained, even though it was not possible to complete the bombardment. These data are being studied at the present time.

The Hall mobility of irradiated single-crystal plates of n-type germanium has been determined from room temperature to 50°K for a series of irradiations by fast neutrons and gamma rays. The effect of additional charge-center scattering on the mobility is being investigated. The role of minority-carrier traps as additional scattering centers is also being examined. Such studies are expected to aid in an understanding of the position and nature of radiation-introduced defect states.

The lifetime of excess carriers injected into germanium by an electrical pulse or a light pulse is dependent on the number of recombination centers present. Irradiation introduces recombination centers, and lifetime measurements aid in an understanding of the process of carrier recombination. The lifetime of holes in n-type germanium has been determined as a function of irradiation by fast neutrons and gamma rays, and the lifetime of electrons in p-type germanium is currently being examined. Information of this type is of special importance to those who must use semiconducting devices in a radiation field. (AEC Activity 4540)

PROGRAM 5000 - PHYSICAL RESEARCH

ISOTOPE PRODUCTION

Radioisotope Production - The 100,000th shipment of radioactive material was made on August 26. This shipment to the University of Iowa consisted of 75 mc of K^{42}, a recently developed processed radioisotope.

Amsco-125 instead of CCl_4, which was tested previously, was used as the carrier solvent in a full-scale production run in the short-lived-fission-products facility. Good phase separation was obtained with the lighter solvent, and no organic matter was noted in the final solution. Separation of rare-earth and alkaline-earth fractions will be handled in the glassware cell, either by oxalate precipitation or by ion exchange instead of by extraction.

A 1-curie promethium tungstate source was made with a volume of less than 60 μl and a weight of 62 mg. The cylindrical source was 4 mm in diameter by 4.7 mm high and was enclosed in stainless steel with a 5-mil stainless steel window. The tungstate compound was made from a Pm^{147} solution containing from 15 to 17 mg of inactive neodymium per curie of promethium. The radiation 3 in. from the surface of the sealed source was 70 mr/hr through the 5-mil window. A qualitative analysis of the x-ray spectrum indicated an x-ray peak at an energy of 30 to 35 kev.

Radioisotope Development - Phase II equipment installation is approximately 95% complete at the Fission Products Pilot Plant.

Promethium-147 was effectively separated from inactive fission neodymium. A mixture of rare earths was adsorbed in a 50-mm column volume of 60- to 100-mesh Nalcite HCR resin. The elutriant was 0.5 M α-hydroxyisobutyrate adjusted to pH 3.0 at 30°C. There was no evidence of neodymium in the elutriant up to 100 column volumes.

The presence of a 0.09 N hydrazine concentration in a synthetic Purex waste solution permitted 94.7% of the Tc^{95} activity to be carried on the iron precipitate when gaseous NH_3 was bubbled into the solution in order to bring the pH to 3.0. When the experiment was repeated with the omission of the hydrazine, no Tc^{95} activity was carried on the iron precipitate. Contacting synthetic Redox solutions with 100% TBP (tributyl phosphate) caused extraction of 92.9% of the Tc^{95} into the TBP. Ninety per cent of the Tc^{95} was back-extracted from 100% TBP with a 1:1 wash of 16.0 N HNO_3.

Tests on shielding attenuation for 2.83 curies of Cs^{134} indicate that a 1000-curie source of Cs^{137} containing 5% Cs^{134} will produce a radiation intensity through 4.5 in. of lead of 2.0 mr/hr due to Cs^{137} gammas and 6.0 mr/hr due to Cs^{134} gammas. An additional 0.8 in. of lead must be added to reduce the radiation due to the combined gammas to 2.0 mr/hr. (AEC Activity 5100)

Stable Isotope Separation - The separation of chromium (masses 50, 52, 53, and 54) has been completed. Monitored ion currents equivalent to 12.3 g of Cr^{54} were collected. Total collection of all isotopes is estimated to be 572 g.

The separation of carbon in order to achieve a high-purity (90%) sample of C^{13} is still in progress. Monitored ion currents equivalent to 0.396 g of C^{13} (47.2g of total carbon) have been collected. Two samples of material obtained from the use of normal CO_2 charge assayed 73.7 and 81.7% C^{13}. Preparations now are being made to use and recycle charges previously enriched in C^{13} content.

The first calutron separation of erbium isotopes (masses 162, 164, 166, 167, 168, and 170) is in progress. Charge feed of $ErCl_3$ is being ionized in the high-temperature type of ion source, and the ions produced are impinging in graphite collector pockets. Monitored currents produced to date are equivalent to a total isotopic collection of 59.8 g. A "ghost" sample is being collected at the mass-172 position.

Chemical processing has been completed on new lots of Cr^{50}, Cr^{53}, Cr^{54}, Ti^{50}, Fe^{54}, Rb^{85} (two samples), Eu^{151} (97.76%), and Dy^{158}. Twenty new lots of isotopes were added to the inventory, five of which were refined during the period, namely, Ti^{50}, Fe^{54}, Rb^{85} (two samples), Eu^{151}, and Dy^{158}. Eighteen lots of returned isotopes, including seven lots of samarium, were processed chemically and returned to the inventory for re-use.

Conversions of inventory forms were made as follows: $Li_2^7CO_3$ to $Li_2^7SO_4$, NH_4Br^{79} to $AgBr^{79}$, $Rb^{87}Cl$ to $Rb^{87}F$; and the oxides of W^{182}, Ti^{48}, Ti^{49}, Pb^{206}, Ni^{58}, Cd^{112}, Cd^{113}, Cd^{114}, Sn^{112}, Sn^{120}, Cu^{63}, Cu^{65}, and Fe^{56} to the elements.

Several inventory lots of $Cu^{65}O$ were combined, dissolved, and reconverted to CuO in order to give a large (24-g) homogeneous sample.

Special targets were fabricated as follows: Cu^{63}, Cu^{65}, and normal Co were electroplated on platinum disks; Zn^{66}, Zn^{68}, Ni^{58}, Cd^{112}, Cd^{113}, and

Cd^{114} were electroplated and stripped as thin foils; Pb^{206} (two lots), Pb^{207}, Pb^{208}, Sn^{112}, and Sn^{120} were rolled into foils; and Ti^{48} and Ti^{49} were pelleted in special forms. Cyclotron targets were prepared from normal calcium and from Mg^{25} by evaporation.

A plating technique has been devised which will produce ductile zinc (or cadmium) and which consequently facilitates the production of very thin unsupported foils. The plating solution contains sodium cyanide in the ratio of 2.5 g of cyanide for each gram of zinc oxide and appears to work best at room temperature with a current density of 75 ma/cm^2. The ratio of cyanide to oxide is slightly higher for cadmium, although the other conditions remain the same.

Stable Isotope Development - The M-16 ion source was modified slightly and then subjected to testing in eight separation runs using an $ErCl_3$ charge. The results, as tabulated below, indicate that its performance is superior to that of the conventional M-14 ion source:

Comparison of M-16 and M-14 Ion-Source Performance

	Collection Rate (mg Er/reception hr)	Consumption Rate (g Er/reception hr)	Process Efficiency (%)	Ion Reception (hr)
M-14	45.2	1.01	4.49	875
M-16	59.4	0.88	6.82	341

The M-12 ion source, modified to utilize two ionization filaments in sequence, has been equipped with a deep arc chamber and has become the accepted source for use in carbon isotope separation.

Enriched C^{13} is available for use as calutron charge material in the form of KCN and $BaCO_3$. Evaluation of these materials as charges has been completed with the use of normal instead of enriched compounds. The ratio of the peak carbon ion output achieved from each, as compared with that achieved from the usual CO_2 charge, is as follows: KCN, 1/10; $CaCO_3$, 1/2; $BaCO_3$, 1/2. If all factors are considered, CO_2 would be the best charge, with $CaCO_3$ being the second choice. During use of enriched C^{13} charge, the exhaust gases will be scrubbed to recover the carbon not ionized in the process.

A low-temperature (-50°C) cooling system is being designed for use in the 24-in.-radius calutrons. Functionally, the system will permit the cooling of any or all calutron components as required, in order to reduce the background of neutral particles present during high-purity separations.

The system of baffles used in the 24-in. calutrons has been redesigned. The source and receiver regions have been isolated more completely, and the pumping speed from the over-all system has been increased. The new assembly will be used primarily in high-purity separations.

A receiver containing a single collector pocket has been designed for use in a single-pass high-purity U^{236} collection run. All parts have been fabricated, and final assembly is awaiting purification of graphite components in the carbon tube furnace.

A water-cooled copper plate was inserted in the C^{13} pocket during a short portion of the carbon run in order to test the possibility of preparing thin layers of target material directly in the calutron. Two pieces have been submitted to possible users for evaluation.

Work on the possibility of chemically enhancing the abundance of calcium isotopes is being continued. Both di(2-ethylhexyl) phosphoric acid for liquid-liquid extraction and Retardion 11A8 for ion exchange are being investigated.

A sample of lepidolite (a silicate ore which is quite old geologically and rich in alkali metals, especially rubidium) was processed in order to concentrate a small percentage (approximately 200 ppm) of radiogenic Sr^{87}. Mass spectrometric data indicate that the strontium is at least 90% Sr^{87}, whereas the best calutron-produced Sr^{87} is 60.2% pure. Plans are being made for processing some 70 lb of the ore in order to place a substantial quantity of high-purity Sr^{87} in the inventory. (AEC Activity 5100)

PHYSICS

High Voltage Program - The total neutron cross section for C^{13} was measured in a standard transmission experiment for neutron energies from 200 kev to 3 Mev. A rotating, metallic lithium target evaporated in place and bombarded by protons from the 5.5-Mv Van de Graaff served as a neutron source. The detector was a 1-atm propane recoil counter. Ten grams of elemental carbon enriched to 62% in C^{13} and placed in a 10-mil-wall nickel container served as the scattering sample. Background was subtracted experimentally by substituting for the scattering sample a similar nickel container with an amount of C^{12} equal to that in the C^{13} sample. Resonances were observed in the total cross section of C^{13} at E_n = 1.75, 2.43, and 2.45 Mev, corresponding to levels in C^{14} at excitation energies of 9.79, 10.42, and 10.44 Mev.

The total neutron cross section of neon was measured for neutron energies from 0.8 to 1.9 Mev in a transmission experiment. The $Li^7(p,n)Be^7$ neutrons were produced at a lithium rotating target with protons from the 5.5-Mv Van de Graaff. The detector was a 1-atm propane recoil counter. A 1-m-long by 7/8-in.-dia thin-walled (1/16 in.) stainless steel tube filled to 1200 psi with neon gas served as the scattering sample. A similar tube, but evacuated, was used to subtract experimentally the effect due to neutrons other than those scattered by the neon. The resulting cross-section curve, corrected for the second-group neutrons from the $Li^7(p,n)Be^7$ reaction, showed resonances at 0.91, 1.27, 1.31, 1.37, 1.62, and 1.68 Mev, as previously reported (Phys. Rev. 99, 1625, 1955), and a new resonance at 1.85 Mev. (AEC Activity 5220)

Scintillation Spectrometry - The fluorescent yield in the L shell for elements with Z = 82, 81, 80, 72, 70, and 65 has been determined by use of a new technique. An L-shell vacancy is produced in the emission of a K_α x ray, for example, following internal conversion. Coincidences are taken between K and L x rays (with scintillation spectrometers), and use is made of K_α/K_β ratios from x-ray work. The probability of observing an L x ray in coincidence with a K x ray, multiplied by $K_\alpha + K_\beta/K_\alpha$, gives the L-shell fluorescent yield. The results are in agreement with average L-shell fluorescent yields determined by H. Lay in 1934. (AEC Activity 5220)

The 86-Inch Cyclotron: Nuclear Physics - A survey was made of anomalous inelastic proton scattering in about 20 elements by using magnetic analysis. The anomalous peak has considerable structure, different for each element, although there are strong similarities among neighboring elements. Lower lying levels were also found in all elements. Isotopic effects were studied.

The (p,α) reactions in several elements were studied by measuring the alpha particles. Their energies are much lower than those predicted by theory. (AEC Activity 5220)

The 86-Inch Cyclotron: Applied Physics - The isotopes Be^7, Co^{57}, As^{74}, and Sr^{85} were made in routine production, and Au^{194} and Au^{196} were produced in service irradiations for outside customers. The Co^{57} production run averaged 1750 μa for 2 hr on a nickel-plated copper target at a radius of 31 3/4 in. (corresponding to an energy of 21 Mev for the incident protons). The two As^{74} production runs were made by using enriched Ge^{74} as the target material. The expected increase in yield was not obtained. Technetium-95m was made for the Chemistry Division. One successful run on a flat-plate calcium target was made with a Ca^{47} yield of $(4.6 \pm 1.5) \times 10^{-2}$ mc/hr from 200 ± 40 mg of calcium. (AEC Activity 5220)

Heavy-Particle Physics - The energy and the angular distributions of alpha particles from the nitrogen bombardment of oxygen were measured. It was found that, as in the case of aluminum, the alpha particles are peaked in a forward direction. A few runs were made by bombarding sulfur with nitrogen ions, with the subsequent separation of radioactive Ti, Sc, and V. An existing reaction chamber was modified for the investigation of elastic scattering of nitrogen from beryllium. (AEC Activity 5220)

Spectroscopy Research Laboratory - The infrared spectra of the molecules PH_2D, PHD_2, and PD_3 have been observed by the four-cell compensation technique previously employed on the isotopic ammonia species. From these spectra, obtained at NaCl and LiF prism resolution, the fundamental frequencies have been assigned, including the previously unreported ν_3 vibration of PD_3. Observed frequencies are in good agreement with the values predicted with the assumption of a valence-bond force field:

Observed Frequencies (cm^{-1})

		PH_3 (a)	PH_2D	PHD_2	PD_3
ν_1		2323	2332	1683 ± 2	1680
ν_2		990	890	767	727
ν_3	(asym)		----	1691	
		2328			1691
	(sym)		1687	2310	
ν_4	(asym)		975	980	
		1121			804
	(sym)		1093	909	

(a) V. M. McConaghie and H. H. Nielsen, Proc. Natl. Acad. Sci. U.S. 34, 455 (1948). (AEC Activity 5230)

Electronuclear Machines - Cyclotron Analogue II is being designed as an electron model of the proposed 900-Mev azimuthally varying field (AVF) proton cyclotron. The number of sectors in the magnetic field has been fixed at 8, and an operating point path has been chosen along the $\nu_z = 0.2$ line. A coil design satisfying the smooth approximation criterion has been found; the preparation of codes for carrying through high-precision coil design calculations is nearing completion.

Experimental studies of axial focusing problems were continued in the ORNL 44-Inch Cyclotron. Grid-type structures are placed in the central region of the magnetic field in such an arrangement as to provide strong electric focusing forces. By carefully placing the grids and baffling the out-of-phase beam it has been found possible to eliminate beam attenuation in the region where grids were used.

Studies of problems involved in the design and fabrication of high-flux AVF magnets were continued. Pole tip windings were found to be surprisingly effective in enhancing the azimuthal variation of fields, and also in boosting the average field. Average fields of 19.1 kilogauss have been obtained with azimuthal variations having an effective amplitude of ±35%. (AEC Activity 5240)

CHEMISTRY

Research Participation and Traveling Lecture Program - The Research Participants who spent the summer at the Laboratory have completed their assignments and have returned to their home universities. Of the 53 participants representing 44 universities, 41 were engaged in physical-science research.

The Traveling Lecture Program for the academic year 1957-1958 has begun, with 48 lectures already scheduled, 34 under Program 5000. (AEC Activity 5310)

Raw Materials Chemistry - Strontium-sodium cation exchange between organic solutions of sodium di(2-ethylhexyl)phosphate and alkaline aqueous nitrate solutions showed preferential extraction of strontium over sodium into the organic phase. The separation factors $SF_{Na}^{Sr} = (Sr/Na)_o/(Sr/Na)_a$ were near 30 in extractions from 0.5 to 2 M sodium nitrate solutions containing strontium nitrate at tracer levels or at 0.1 M, and varied little if any with pH (9 to 12) or organic reagent concentration (0.1 to 0.4 M). Cesium-sodium cation exchange showed preferential extraction of sodium over cesium from similar solutions, $SF_{Na}^{Cs} \cong 0.4$.

Plutonium(IV) was extracted from 5 M sulfuric acid solutions with extraction coefficients (o/a) near 10 by 0.3 M tri-n-octylphosphine oxide and by 0.4 M di(2-ethylhexyl)phosphoric acid. The extraction by the latter was decreased slightly on addition of ~ 0.1 M tri-n-octylphosphine oxide and was decreased more (to $E_a^o \cong 1$ to 4) on addition of TBP (tributyl phosphate) or decanol. Qualitatively, this effect of phosphine oxide and of TBP on Pu(IV) extraction by dialkylphosphoric acid is intermediate between the effects on Th(IV) (severe impairment) and on U(IV) (little effect).

Scaleup of gravity-type settlers for the Dapex uranium extraction process was demonstrated in 6-, 22-, and 48-in.-dia tanks. The nominal flow capacity based on flow rate per unit cross-sectional area was virtually independent of settler diameter when wall effects in the small settler were minimized. For water-in-oil dispersions the nominal aqueous flow capacity was 1.1 gpm/ft^2, and for oil-in-water dispersions the aqueous flow ranged from 1.4 at a phase ratio (aq/org) of 1/1 to 4.4 gpm/ft^2 at a phase ratio of 4/1.

A mixer-settler system with 80 separation stages, loaded with rare earths in 14 N HNO_3 and TBP, was operated at total reflux until equilibrium was attained. After 150 hr, analyses made of the aqueous phase at ten-stage intervals showed the following distribution:

Stage No.	Gd	Dy	Ho	Er	Y	Tm	Yb	Lu
1	3.1	3.5	0.8	4.5	85	2.2	0.4	
11				2.6	91	5.2	1.7	
21					20	12	78	
31					T	1.9	98	T
41							99	1
51							97	2.9
61							92	8.2
71							76	24
81							43	57

T = Trace

(AEC Activity 5310)

Chemical Physics - An x-ray diffraction pattern from aqueous bismuth oxyperchlorate solution suggests the existence of hexameric species such as $Bi_6(OH)_{12}^{6+}$ with an octahedral arrangement of bismuth ions. (AEC Activity 5330)

High-Temperature and Structural Chemistry - As predicted in earlier reports, the rubidium chloride—rubidium metal system was found to resemble the corresponding iodide system but to differ from the bromide system in exhibiting a miscibility gap. This gap is the smallest found as yet in any of the alkali metal—halide systems. The compositions of the coexisting liquids at the monotectic temperature of 696°C are 12 and 50 mole % metal. The consolute temperature lies less than 10 deg higher, at 705°C, or 12 deg below the melting point of the pure salt, and the critical composition is approximately 33 mole % metal. (AEC Activity 5330)

Radiation Chemistry - Moist air of varying absolute humidity was irradiated with 1- to 2-Mev electrons, and the pressure change and the infrared spectrum of the system were determined at intervals. Nitrous oxide was formed at a rate independent of the concentration of water vapor present. Nitric acid was produced up to the maximum theoretical concentration as calculated from the initial amount of water present. The nitric acid then disappeared with the stoichiometric production of nitrogen dioxide and water. Subsequent to the appearance of nitrogen dioxide in the system, no more nitric acid was formed — only nitrous oxide and nitrogen dioxide. From a practical point of view, it is noteworthy that for an equal energy input, several times more nitrogen is fixed before than after the appearance of nitrogen dioxide. In order to avoid corrosion in air-cooled reactors the air should be dry. To fix nitrogen in high yield by reactor irradiation the air should be humid. (AEC Activity 5330)

Chemistry of Fused Salts - The self-diffusion coefficients D of the ions in molten $LiNO_3$ were measured over the temperature range 265 to 320°C with Li^6 and N^{15} used as tracers. The results may be expressed in the form of a rate equation $D = Ae^{-\Delta H/RT}$, where ΔH is the heat of activation. For Li^+ the values of A and ΔH respectively are 2.47×10^{-3} cm^2/sec and 5.5 kcal. The value of ΔH is approximately the same as that found for other alkali nitrates previously reported. The values for $D \times 10^5$ for the cations in $LiNO_3$, $NaNO_3$, KNO_3, $CsNO_3$, and $AgNO_3$ respectively are 2.93, 2.33, 1.52, 1.22, and 2.40 cm^2/sec. The self-diffusion coefficients of the nitrate ion in $AgNO_3$ and $LiNO_3$ will be calculated upon completion of the mass spectrometric analysis for N^{15}. (AEC Activity 5330)

Molten Salt Thermodynamics - In connection with investigations of activities in molten fluoride solutions, the vapor pressures of pure molten alkali fluorides were studied by a method which measures the pressure directly. The total pressures thus obtained are made up of the partial pressures of the vapor species, which include dimer and even trimer molecules; for this reason the vapor pressures do not agree with literature values obtained from transpiration methods in which the previously accepted assumption of a monomeric vapor was made.

The experimentally determined total pressures (mm Hg) can be expressed as a function of the absolute temperature by means of the Kirchhoff equations listed below:

Vapor Pressures of Pure Alkali Fluorides

Salt	Equation	Experimental Ranges Temp (°K)	Pressure (mm Hg)
LiF	$\log P = -13{,}890/T - 4.021 \log T + 23.165$	1328-1575	1.3-30.8
NaF	$\log P = -14{,}609/T - 3.518 \log T + 21.863$	1381-1592	1.7-26.6
KF	$\log P = -11{,}910/T - 3.518 \log T + 21.002$	1199-1505	1.8-81.4
RbF	$\log P = -11{,}373/T - 3.518 \log T + 20.995$	1154-1451	2.2-106
CsF	$\log P = -9{,}845/T - 3.518 \log T + 20.556$	1009-1379	1.7-242

(AEC Activity 5330)

Analytical Chemistry Research - The solvent extraction of thorium from acidic solutions with tri-n-octylphosphine oxide (TOPO) was investigated as part of the continuing program on the utilization of organo-phosphorus compounds for the extraction and separation of cationic substances. For all practical purposes it is apparent that thorium is extracted completely (>99%) in a single equilibration from either 1 M HNO_3 or 7 M HCl. If, however, the concentration of nitric acid is increased or the concentration of hydrochloric acid is reduced, the degree of extraction is significantly less. The extraction of thorium from perchloric acid solutions follows essentially the same pattern as that of nitric acid systems, except that the degree of extraction is not as high, whereas from a sulfuric acid medium, extraction is virtually insignificant, regardless of the acid concentration. As a matter of further interest it is to be noted that the equilibration period which is required for the saturation of TOPO with thorium is apparently greater than that which has been reported for zirconium (ORNL-2355, p 18, June 1957).

Development work is nearing completion on the x-ray fluorescence method for determining Nb and Zr in Nb-Zr-U alloys. The sample is dissolved (to avoid segregation errors), U is extracted with TBP, and Zr and Nb are counted by means of x-ray fluorescence, using Rb as an internal standard. These elements are determined with a precision of ± 1% by this technique.

METALLURGY

Ceramics Research - A considerable number of calcinations of thorium oxide have been made at temperatures from 1000 to 1900°C for different departments of ORNL. At temperatures up to 1600°C, the usual procedure was to heat the material in platinum vessels in an electrical resistor type of furnace. At higher temperatures, alumina containers and a gas-fired furnace were generally employed. The firing operation is periodic and is followed by a pulverization procedure.

The need for calcination of ThO_2 at temperatures above 1500°C in air entailed a consideration of possible container materials for the operation. Aluminum oxide containers are available and have favorable ceramic properties. Accordingly, a brief investigation was made of the possibility of contamination of powdered ThO_2 by the alumina containers. For this purpose, pressed compacts of ThO_2 were placed on flat surfaces of commercial alumina containers and were then fired for 4 hr at 1600°C. Spectrographic analyses of the products showed no increase in Al. It was concluded that contamination of the ThO_2 from the alumina containers other than by mechanical abrasion should be rather low. A CF memo covering these tests is in preparation.

It has been thought that calcined particles with desirable characteristics for use in a slurry blanket might be produced from the ThO_2 as received in one operation by a brief exposure to a very high temperature (3000°C). Among the hoped-for properties are a very fine size (1 μ or less) and rounded particle edges which should reduce the abrading action.

The heat sources considered for this purpose were the oxyacetylene torch, the electric arc, and the induction furnace. Preliminary tests of the first two methods were made.

For the oxyacetylene calcination, a metallizing gun (Metco Flame Spray), which employs a gravity feed, was found to be best. However, ThO_2 (from the oxalate process) tended to pack and was passed through the gun with difficulty, even when the gun was vibrated strongly. On the second pass there was much less difficulty.

Thorium oxalate and thorium formate offered less difficulty than the oxide. A thorium compound treated with acetylacetone to produce a thorium oxide—acetylacetone complex was unsatisfactory in the oxyacetylene flame-spray gun, apparently because it has a low melting temperature. Under the given conditions three successive passes through the gun seemed necessary for complete conversion of the oxalate and formate to cubic ThO_2. The product from the oxide, formate, or oxalate consisted of fine grains which, in part, clung together in agglomerates approximately 0.5 mm in diameter; these were easily crushed to a powder. In the case of thorium oxalate and formate there was a notable volume decrease during the first passage through the gun.

Samples of flame-spray-calcined thorium oxide, formate, and oxalate have been submitted to the HRP Blanket Development Group for tests (in toroids) of abrasive properties and ease of particle degradation.

The arc-calcining apparatus consisted of graphite electrodes 1/2 in. in diameter extending into the center of a vertical refractory tube 5 in. in diameter. The powdered materials were fed through the arc by means of a vertical zircon tube which was kept in vibration.

The product of one passage through the arc of either the oxide, formate, or oxalate contained some rather perfect spheres of ThO_2, 0.1 to 0.3 mm in

diameter; many of these were transparent, iridescent, hollow bubbles which floated on water and were carried upward by air currents; they were very fragile. Many solid opaque spheres of about the same size were formed also and appeared to be agglomerates of finer particles; they were resistant to crushing.

After three passes through the arc the product of each of the three compounds consisted mostly of spheres and was made up of about equal proportions of the transparent hollow type and the solid opaque type.

It was concluded that the arc process produced, under the given conditions, an unsuitable product for the HRT blanket because of the comparatively large size of the spheres produced. It is conceivable that refinements in the apparatus might lead to the production of the submicron-sized rounded grains desired. Uses for the transparent bubbles of ThO_2 are being sought.

Spheres, both solid and hollow, of zirconia, alumina, hafnia, and zircon, as well as of thoria (as detailed above), have been produced by arc calcination. The spheres formed were mostly under 0.3 mm in diameter in all cases. Apparently the proportion of spheres and their size and strength can be controlled, if necessary, by compacting the powder, then breaking it into agglomerates in controlled sizes before it is arc-calcined, or by regulating the time period during which the ThO_2 is actually in the hot zone of the arc. (AEC Activity 5420)

Fundamental Physico Metallurgical Research - Investigations of the cadmium-zirconium alloys showed that the beta-phase solid solubility is at least 16 at. % Cd at 1000°C. The vapor pressures of an alloy containing 7.5 at. % Cd were measured between 950 and 1050°C. The partial molar properties of cadmium, with respect to liquid cadmium, were found to be:

$$\Delta F = -5600 \text{ cal/mole},$$
$$\Delta H = -4200 \text{ cal/mole},$$
$$\Delta S = +1.1 \text{ cal/mole-deg}.$$

The heat capacity of a 4-g specimen of zirconium containing a graphite resistance thermometer and a Manganin heater was measured from 1.6 to 4.4°K. The heat capacity of the specimen over this range was found to be

$$C = (1186 \pm 4)\ T + (62.23 \pm 0.38)\ T^3 \text{ ergs/deg}.$$

This specimen will be used as the thermometer- and heater-containing part of the alloy specimens, and the measurement above permits correction of the specific heats of the alloys for the heat capacity of the heater and the thermometer.

A zirconium rod was refined by the use of induction heating to form a liquid zone while the ingot rested upon a water-cooled copper hearth. Purification ratios of approximately 10 to 1 were found for both iron and copper. (AEC Activity 5420)

BIOPHYSICS

Radiation Dosimetry Programs - The spectrum of x rays from the Health Physics 250-kv x-ray machine has been measured with a scintillation spectrometer. As an example, the 250-kv beam, after passing through a filter consisting of 2.1 mm of lead, 1.2 mm of tin, 1.1 mm of copper, and 1.0 mm of aluminum, gives an approximately symmetric number distribution which has a peak at 205 kev and which is 70 kev wide at half height. The effect of the finite resolution of the spectrometer is taken into account by use of the iterative method of Freedman, Novey, Porter, and Wagner, and the corrected number distribution differs only slightly from the experimental distribution mentioned above. The average energy in the intensity distribution obtained from the corrected number distribution was 201 kev, as compared with 201 and 197 kev from copper and tin absorption curves, respectively. The intensity distribution agreed with a spectrum calculated from the theory of Kramers for the filters used, within the experimental accuracy of about 3%. The calibration of the spectrometer and the line profile shapes were obtained from the gamma spectra of Ce^{141}, Hg^{203}, and Tm^{170}. A complete knowledge of the output of the x-ray machine is essential for accurate dosimeter calibration. (AEC Activity 6140)

Internal-Dose Studies - Analyses were made for studying the distribution of the elements in standard man. Representative samples of tissue from 25 different human body organs obtained in each of four American cities have been analyzed for sodium and potassium content by flame photometry. Samples of liver and kidney obtained in Saint Louis from the autopsies of ten stillborn infants have been analyzed spectrographically for 37 elements. Spectrographic analysis of liver, kidney, lung, and bone has been completed also for samples obtained from autopsies performed in Bangkok, Kyoto, and Tokyo.

Tentative biological data for 98 elements have been collected. Proposed values for the effective energy and for the maximum permissible levels of body burden, air concentration, and water concentration for 229 radioisotopes were computed from those data. The results have been sent to the Subcommittees on Internal Dose of the National Committee on Radiation Protection and the International Commission on Radiological Protection.

It is apparent that one of the most important disagreements among experts concerns not the radiation exposure caused by bomb tests, but the biological consequences likely to be produced by this exposure. Since Cs^{137} is one of the most prevalent components of fallout and since Cs^{137} has been found to have a high concentration in the gonads, it is felt that it is of prime importance to reconsider the hazard of this nuclide with respect to reproduction. Therefore, studies have been initiated in this laboratory to ascertain the effects of continuous ingestion of Cs^{137} on the reproduction of sexually mature female and male mice and their offspring. (AEC Activity 6440)

PROGRAM 8000 - WORK FOR OTHERS

ASEE-AEC Nuclear Energy Institute - The ASEE-AEC Nuclear Energy Institute was completed on August 23, 1957. Plans are now being made for a similar program for the summer of 1958. (AEC Activity 8736.3)

Training Program for University Faculty Members - Three university faculty members who attended the 1956-57 session of ORSORT completed their assignment in September. One member of the faculty of Pennsylvania State University joined the ORSORT staff in September for a six-month assignment. (AEC Activity 8736.4)

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