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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JUNE 1957

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

June, 1957

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OAK RIDGE NATIONAL LABORATORY
STATUS AND PROGRESS REPORT

June, 1957

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

June, 1957

This status and progress report summarizes somewhat less than one-half of the Laboratory's activities. A few of the activities are reported every month but most of them are reported on a bimonthly schedule. Program 4400 is reported separately.

PROGRAM 4000 - REACTOR DEVELOPMENT

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Test - Restoration of the HRT to operating condition is continuing satisfactorily, with replacement of the 120 damaged flanges now over 80% complete. The new leak-detector headers are installed, and the tubing bundles penetrating the north wall are welded into position.

The storage recombiner is connected, and both pressurizers are mounted in place, with the blanket pressurizer modified to permit a feed supply from the loop leg as well as from the purge pump. This change should allow the blanket system to be operated without let-down during tests with a heavy-water blanket.

The gamma flux in the stator windings of the fuel circulating pump during power operation was estimated by an experiment which involved filling the pump volute with $Cs_2{}^{137}SO_4$ solution and allowing it to irradiate strips of film placed between the rotor and the stator can. The flux was found to be low enough to preclude damage to the insulation from this source; however, an external shield will be constructed to attenuate the gammas incident from the surrounding cell.

As in May, the bulk of operations effort was devoted to chemical-plant operation and to the preparation of the HRT Operations Manual and testing procedures. (AEC Activity 4103.3)

HRT Chemical Pilot Plant - The testing of the HRT Chemical Plant low-pressure system was continued. Performance of the decay-tank recombiners was satisfactory, but the vent-system recombiners were only about 90% efficient. A pressure method of transfer is being installed in the fuel-charging system because of inconsistent gravity transfer.

The low heat-transfer rates in the dissolver were markedly improved by the addition of mercury in the space between the stainless steel vessel and the tantalum liner. Water testing of the dissolver was completed, and

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three dissolutions of simulated corrosion products were performed. Effective separation of the light- and heavy-water systems was demonstrated with a D_2O-traced solution. (AEC Activity 4103.1)

HRT Fuel Processing - Increasing the acidity of 0.04 m UO_2SO_4 from 0.01 m to 0.02 m decreased the stability of iodate in the presence of Co^{60} radiation at 100°C.

Simulated reactor corrosion-product solids, to which Fe^{55} and Fe^{59} were added as tracers, were recovered quantitatively by rinsing with water and heating to 275°C the stainless steel tubes to which the solids had been added as a dilute slurry. Laboratory studies simulating the operating conditions of the HRT Chemical Plant dissolver indicated that previous unsatisfactory performance of the dissolver was due to insufficient agitation. In three two-cycle dissolutions in the glass-lined dissolver with a segmented jacket to improve the agitation, dissolution was more than 99.5% complete.

The adsorption of krypton on Columbia grade-G charcoal at 25 to 100°C was less than predicted by extrapolation of data obtained at lower temperatures. The presence of small amounts of water markedly reduced the adsorptive capacity of the charcoal for krypton. Unfilled space in a horizontal column permitted channeling which drastically shortened the krypton holdup time in the bed; removal of 3% of the packing from a horizontal bed with 24 theoretical stages and a breakthrough time of 30 min changed the number of theoretical stages to 4 and the breakthrough time to 2.5 and 3.0 min. (AEC Activity 4103.1)

Homogeneous Reactor Thorium Blanket Studies--Chemical Development - A slurry of 800°C-fired thorium-uranium oxide (U/Th mole ratio of 0.005/1), prepared from the coprecipitated oxalates and containing 20,000 ppm of MoO_3 and 3000 ppm of sulfate added as $Th(SO_4)_2$, was irradiated for 247 hr at 300°C in the LITR with no visible damage.

In out-of-pile studies on the catalytic recombination of hydrogen-oxygen mixtures by thorium-uranium oxide slurries (500 g of Th per kg of H_2O) containing 0.05 m MoO_3, slurries of thorium-uranium oxides calcined at 1000°C for 16 and 24 hr and at 1600°C for 24 hr showed high catalytic activities, that is, >>10 moles of H_2 per hour per liter. Slurries of the mixed oxides calcined at 800°C for 24 hr and at 1000°C for 4 hr had low catalytic activities even after treatment with H_2.

Calcination for 12 hr at 1000°, 1200°, 1400°, and 1600°C did not affect the average size of sedimented particles of thorium oxide prepared from the oxalate precipitated at 10°C. The abrasive characteristic, as measured by the penetration rate in the jet impingement slurry-abrasion tester, increased with increasing firing temperature, from 0.15 mil/min for 1000°C to 1.5 mils/min for 1600°C. Only the oxide fired at 1600°C resisted degradation.

Hindered-settling rates for slurries containing 250 g of Th per kg of H_2O decreased with decreasing oxide particle size and increased with increasing slurry temperature in the range from 25° to 300°C. Silicate addition, 5000 to 20,000 ppm SiO_2/ThO_2, slowed the settling rates.

Thoria microspheres with an average diameter less than 10 μ were prepared by Houdry Process Corporation by a gel technique. Operation of equipment to produce ThO_2 fired at 1600°C by flame calcination has not been successful owing to the inability to feed ThO_2 or $Th(C_2O_4)_2$ powders. Special flame calcination equipment, including industrial flame plating guns, was installed in the Building 4501 loop facility area. (AEC Activity 4103.1)

HRP Chemistry--Fuel Studies - The continued studies of aqueous solutions of uranyl sulfate and copper sulfate, saturated with UO_3 and/or CuO at 100°C, have disclosed a slow reaction with either pyrex or silica-glass vessels that produces a uranyl silicate as a solid phase. The x-ray powder pattern for the solid is identical with that for the silicate produced by the action of uranyl fluoride solution on silica glass at high temperature, and also with that for the product of the action of UO_3 slurries on glass at high temperatures. Its composition is thought to be $(UO_2)_2SiO_4 \cdot 3H_2O$. In the sulfate system it is produced most readily from solutions in which the mole ratio of uranium to copper is high. It was not found in any case where this ratio was less than about 1.7. Its formation was not accompanied by any visual evidence of attack on the glass. The importance of these results lies in the serious limitation they place on any further use of glass vessels in experimental work and in the element of doubt placed on the validity of many experimental observations which have previously been reported. (AEC Activity 4103.1)

Slurry Component Development and Slurry Testing - Run 200A-11, in which a sulfated mixed-oxide slurry (U/Th = 0.005) was circulated at 300°C, was concluded after 2300 hr of operation. The maximum concentration that could be circulated without difficulty was 1400 to 1500 g of Th per kg of H_2O. The handling characteristics and generalized attack rate were similar to those experienced with pure thorium oxide slurries. When dismantled, the loop appeared to be free from cake formation.

Runs 200A-12 and -13 were completed, wherein slurries of thorium oxide (batches LO-20 and -23) were circulated at 200°C without additives, in an attempt to manufacture microspheres such as those produced in runs 200A-6, -7, and -8, which were conducted last summer with batches LO-13 and -14 of thorium oxide. The microspheres formed in the same manner as before, indicating that the sphere-forming property under the chosen operating conditions is not characteristic of a particular batch of oxide. The microspheres are to be fired at temperatures up to 1600°C and evaluated as a slurry source material. (AEC Activity 4103.1)

Special Slurry Problems - One hundred pounds of 800°C-calcined thoria was fired at 1600°C and ground in the Mikro-Pulverizer four times. The material will be circulated at temperatures up to 300°C to observe the effect of firing temperature on slurry behavior.

A 30-gpm vertical loop was designed for studying the factors contributing to cake formation during circulation at elevated temperature. (AEC Activity 4103.1)

Slurry Blanket System Development - Major alterations and repairs to the HRT blanket mockup loop were completed. Circulation of water in the system was started so that the system operation can be checked and the gamma scanning equipment can be calibrated before the loop is loaded with thorium oxide. (AEC Activity 4103.1)

Blanket Engineering Research - The design of the vertical-tube viscometer was completed, and parts were ordered. The design of the high-pressure capillary-tube viscometer, as an adjunct to the 200A loop, was completed and construction started. Preliminary tests with thorium oxide were started in the resuspension loop to evaluate the means set up for visual observations. (AEC Activity 4103.1)

Fuel Circulating Equipment - The 400A-1 pump has circulated 0.04 m UO_2SO_4 at 255°C for a total of 10,291 hr without maintenance since the thermal barrier was seal-welded. The 300A-1 pump has operated for 2145 hr in the HRT mockup since it was last repaired.

Mixing tests conducted on the 400A-2 pump labyrinth shaft seal indicated a mixing rate between pump and motor cavity of 2 liters/hr without purge flow, and of 0.2 to 0.4 liter/hr with purge flow in the range 4 to 35 liters/hr.

The stator liner of the 4000-gpm Byron-Jackson pump was repaired. Loop modifications are in progress to permit measurement of heat balances and mixing rates in the pump.

The 20-cfm blower operated without incident for 1463 hr at low pressure, prior to addition of ThO_2 slurry to its hydrostatic bearing fluid as a bearing test.

Endurance testing of six diaphragm feed-pump heads in pump loops and in the HRT mockup continued without failures. These heads have been operating for 1060, 1094, 2100, 2100, 3877, and 5129 hr, respectively. Two purge-pump diaphragms failed after 796 and 946 hr of operation, but they were both manufactured from a batch of diaphragm sheet which has given very poor service.

The test of the feed pump with a Stellite piston was discontinued so that the pump could be rebuilt to contain piston rings. A direct water piston drive for a diaphragm pump was severely damaged in 130 hr of operation.

Check-valve tests showed that seats of types 17-4 PH, 347, and Carpenter 20 stainless steel and Kennametal K-501 were superior to seats of Stellite 6 in distilled-water service.

Fatigue tests (conducted at Ohio State University) of type 347 stainless steel sheet in UO_2SO_4 solution showed that the endurance limit was not improved by cold rolling except when the sheet was rolled to the full-hard condition. (AEC Activity 4103.1)

Miscellaneous Equipment - The construction of the thermal-cycle test facility was begun, following receipt of the Besler steam generator and the de-aerator.

Fabrication of a small titanium heat exchanger by Pfaudler and of titanium-lined steel pipe by Crane is progressing. These two contractors were finally able to procure all the required materials (approximately 1-1/2 years behind the original schedule).

A literature survey of reactor remote-maintenance techniques was made. A test program is being initiated to evaluate nongalling bolting materials for internal use in homogeneous-reactor components. (AEC Activity 4103.1)

Heat Removal Equipment - Fabrication of the HRT replacement heat exchanger is about to be started by Babcock and Wilcox. Most of the shop drawings have been approved for construction. Some details of the welding and inspection procedures remain to be resolved. Procurement of tubing and other materials is on schedule.

Calculations of transient temperatures during thermal cycling of the spare heat exchanger are in progress. Specifications for small experimental heat exchangers incorporating advanced features were prepared and sent to vendors. (AEC Activity 4103.1)

Gas Handling Equipment - Reconstruction of the high-pressure recombiner loop, with Inconel and Croloy 16-1 materials being used in regions subject to stress-corrosion cracking, was started. (AEC Activity 4103.1)

Solution Systems Development - The HRT mockup completed a 1500-hr run in which 0.04 m UO_2SO_4 was circulated. The run was terminated because of a tube failure in the Foster Wheeler heat exchanger, which had been in service for 10,165 hr. Disassembly of the heat exchanger showed that some of the tubes had become plugged with solids during a previous 1.4 m UO_2SO_4 run. The failure occurred in one of the plugged tubes.

A bed of silvered alundum pellets preceding the low-pressure recombiner was shown to remove iodine from the system. However, the bed was not large enough to effect complete iodine removal, and as a result the recombiner was partially poisoned. It was found that the platinized catalyst could be regenerated by reducing the steam diluent until the temperature of the H_2-O_2 reaction reached 650°C.

Following the mockup run, 1000 g of solids (about 50% U) was found to be deposited in the horizontal pressurizer header. The solids apparently had been formed during prior 1.4 m UO_2SO_4 operation and had been deposited during the 0.04 m run by some sort of mass-transfer mechanism.

The continued operation of the eight HRT-type ring-joint flanges demonstrated that they could be sealed properly by tightening the bolts to initial loadings of 45,000 psi stress. Both oval and octagonal ring gaskets and both old and new gaskets gave equally good results. Preliminary data indicate that the desired bolt loading can be achieved with the normal torque-wrench procedures used at the HRT. (AEC Activity 4103.1)

PACKAGE POWER REACTOR PROGRAM

Army Package Power Reactor (APPR-1) - The Laboratory continued to furnish technical support to the Army Reactor Branch (AEC) on the 10-Mw pressurized-water reactor being tested at Fort Belvoir, Virginia, by Alco Products, Inc. Alterations in the steam generator were made in order to improve the quality of the steam supplied to the turbine. The 700-hr performance test was started on June 9, 1957.

The results from irradiation of five miniature absorber samples in the MTR indicate that failure can be expected at between 20 to 30% burnup for APPR operating conditions. Eighteen additional samples were shipped to the MTR to be irradiated for a more detailed study of the critical area. A full-scale APPR-1 control-rod absorber section which operated as an MTR shim rod for five MTR cycles showed no defects upon visual examination; it was returned to the Laboratory for further tests. All fuel-sample irradiations were completed, and specimens are now being examined and evaluated. (AEC Activity 4201.1)

Gas-Cooled Reactor Development - Design work continued on the gas-cooled materials testing facility which will be installed in the Oak Ridge Research Reactor. The specifications for all major components except the gas preheater were completed and procurement initiated. Most of the instrumentation items were procured in preparation for assembly of the main control panel. The in-pile test section was redesigned for new lattice positions assigned to the loop. Reactivity effects of the loop upon the ORR were estimated by first-order perturbation theory for various positions and conditions. (AEC Activity 4201.1)

GENERAL REACTOR RESEARCH

Basic Reactor Research - Preliminary analysis of UNIVAC calculations for predicting the critical mass of the proposed BSF UO_2--stainless steel reactor for k = 1 and k = 1.025 resulted in the following values. The uranium is considered to be enriched in U^{235} to 93% by weight. All cases refer to a 5 by 5 element loading infinitely reflected by water.

No. of Plates Per Element	Fuel Plate Thickness* (mils)	Core Temperature (°F)	Uranium Mass (kg)	
			For k = 1	For k = 1.025
17	30	66	5.37	5.82
20	30	66	6.03	6.53
23	30	66	6.74	7.31
17	20	66	4.22	4.55
20	20	66	4.59	4.96
23	20	66	4.99	5.38
25	20	66	5.29	5.71
20	25	66	5.27	5.69
20	30	183	6.07	6.58

*Includes 5-mil cladding on each side.

(AEC Activity 4520)

Thorex Process - Pilot plant run HD-25 was terminated after 610 hr of intermittent operation. The feed preparation equipment was decontaminated, and plant modifications for processing short-decayed irradiated thorium were started. About 1300 kg of thorium product was recovered and 3100 g of U^{233} product was sorbed on ion exchange resin. The solvent extraction losses averaged 2.1 and 1.1% for thorium and uranium, respectively. Gross-gamma decontamination factors were 2.3×10^4 for thorium and 1.2×10^3 for uranium.

The design of the third uranium cycle was completed, and installation is 75% complete. Design of the aqueous waste storage facility was started. The sample conveyor system was completed and initial run-in was started. The continuous interface purge system is 30% complete.

In laboratory process improvement studies, fission-product extraction was decreased by increasing the concentration of thorium in the organic phase. Ruthenium decontamination increased continuously with decreasing acidity. I^{131} was not completely removed by the addition of KI and sulfite to the solvent extraction feed and scrub, but was removed when the KI was added prior to the feed adjustment step.

The flowsheet for the Thorex 1A-column interfacial emulsions and/or solids was made final. Low surface tension of the scrub used in the column was found to cause severe interfacial emulsions. (AEC Activity 4610)

Power Reactor Waste Disposal Engineering Studies - The power requirements, mixing procedure, material balance, and off-gas filtration system for a 30-gal self-sintering experiment were evaluated. Simulated self-sintering, with electrical resistance heaters operating at power levels representative of the

heat available in an acid aluminum nitrate waste solution, showed that the total energy (including heat losses) and time required for sintering at 800°C are 34,000 Btu/gal of clinker and 5 days, respectively. The clinkers produced in these experiments exhibited an average solubility of 10^{-4}% as determined by static soak-leach test. Petrographic thin section study of the clinker material indicated that the sintering process promotes the formation of a compound mellite structure capable of large and varied ionic substitution. (AEC Activity 4630)

PROGRAM 5000 - PHYSICAL RESEARCH

PHYSICS

High Voltage Program - A massive 5-ft-dia sphere of graphite with a re-entrant hole allowing (p,n) reactions to occur at the center of the sphere was used for measurements of the T(p,n) cross section near threshold. Initial neutrons, regardless of energy and direction, are thermalized and diffuse outward, without excessive absorption, to BF_3 counters distributed over the sphere in tangential holes near the surface. Most data points for the T(p,n) cross section from threshold to about 1 Mev above threshold were measured with a Zr-T target whose composition was investigated, but absolute normalization was obtained with a gas target. Absolute detection efficiency was related through an Sb-Be source to the Ra-Be source at the National Bureau of Standards. A systematic ±5% uncertainty is assigned. At the upper energy end of the measurements there is a 15% discrepancy from the results of Jarvis et al. (LA-2014). These measurements are of value for neutron detection when converted by detailed balancing to the inverse He^3(n,p) reaction. The agreement with recent He^3(n,p) data from both Los Alamos (Phys. Rev. 80, 488, 1950) and Harwell (Rev. Sci. Instr. 26, 1037, 1955) is notable in view of the divergence of the T(p,n) cross section from earlier results.

By using the 5-ft-dia graphite sphere, the Li^7(p,n) reaction near threshold was investigated. For this reaction, a Breit-Wigner fit indicates a 2^- level near threshold, in agreement with Newson (Phys. Rev. 100, 960A, 1955). The fit required neutron and proton reduced widths differing by a factor of 5.2. When the contribution of the threshold level is subtracted from the resonance, it is clear that the peak must be attributed to p-wave neutrons forming a J = 3 state, in agreement with Adair (Phys. Rev. 96, 709, 1954). However, the fact that the observed peak cross section is 15% higher than the value (Phys. Rev. 74, 373, 1948) used by Adair and the fact that the observed s-wave contribution is somewhat lower imply that the proton and neutron widths are nearly equal. In turn, since the proton and neutron barriers are

much different, the reduced widths must differ unless in some manner the barriers can be made equal. Thus, in contrast with Adair, it is assumed that the reduced widths for proton and neutron emission are not completely charge independent but must differ by at least a factor of 3. Nuclear coulomb effects may be able to account for the difference in reduced widths. As the reduced widths for the 2^- level near threshold are also unequal, we have taken for simplicity the same ratio of reduced widths for both levels. A small incoherent p-wave addition was required to give the upper part of the curve. (AEC Activity 5220)

The 86-Inch Cyclotron, Nuclear Physics - The energy distribution of the alpha particles from fission was remeasured; the results were in agreement with earlier, less accurate measurements. The fission-fragment spectrograph was disassembled, and the magnet was incorporated in a reaction-product analyzer for use in nuclear reaction studies with the 86-inch cyclotron. Anomalous inelastic proton scattering was measured in targets previously not studied. The large peak at ~ 25 Mev starts in Zn^{64} (where it is as large as in Zn^{68}, and an order of magnitude larger than in nickel, at 60 deg); it continues strong up to atomic number 53, where it gradually begins to decrease. By Z = 56, it has decreased very appreciably; there is little change then up to Z = 60. (AEC Activity 5220)

The 86-Inch Cyclotron, Applied Physics - The isotopes Be^7, Fe^{55}, Co^{55}, Zn^{65}, As^{74}, Sr^{85}, Cd^{109}, and Au^{194} were produced in irradiations for outside customers. A successful technique for the production of Kr^{79} was developed. It involves fusing KBr in the target capsule; under these conditions the Kr^{79} does not diffuse from the crystalline lattice and escape. This isotope, together with Nb^{90} and Tc^{95}, was produced for the ORNL Physics Division. A target for the bombardment of 50- to 100-mg samples of soluble materials was developed. (AEC Activity 5220)

The 63-Inch Cyclotron, Physics - Measurements are continuing on the light-particle spectra from the nitrogen bombardment of aluminum. The angular distribution of protons was found to be isotropic from 0 to 90 deg in the laboratory system at all proton energies. Thin oxygen targets were prepared for the study of light particles from the nitrogen bombardment of this nucleus. Work was begun on a scattering chamber for N-Be scattering. (AEC Activity 5220)

Spectroscopy Research Laboratory - During infrared studies of tritiated ammonia, absorption bands were observed from NT_4Cl formed on the AgCl absorption cell windows. That this was attributable to the beta activity of the tritium was verified through synthesis of NH_4Cl by exposure of an AgCl window and ammonia to the 20-kv excitation of a Tesla discharge. Frequencies in cm^{-1} observed for the various ammonium ion species are given below; calculated frequencies were obtained for ν_3 and ν_4 on the assumption of simple valence forces, the observed frequencies for the NH_4^+ ion, and spectroscopic masses, the latter to correct for molecular anharmonicities.

INFRARED FREQUENCIES OF AMMONIUM ION (22°C)

		NH_4^+	NT_4^+
ν_3	Observed	3146	2022
	Calculated		2033
ν_4	Observed	1404	913
	Calculated		908
$\nu_2 + \nu_4$	Observed	3053	1889

(AEC Activity 5230)

Electronuclear Machines - Experimental studies of beam behavior in the Cyclotron Analogue I were continued. It was found that a distinct beam blowup could be induced at the 4/3 resonance by certain adjustments of the operating conditions. The 4/3 resonance appears qualitatively to act as a critical filter on the radial betatron amplitudes; particles with amplitude less than the critical value pass through the resonance undisturbed, particles with greater than critical amplitude blow up very rapidly. This explanation agrees with theoretical predictions and is qualitatively substantiated by manipulating starting conditions in the Analogue in such a way as to vary the initial radial amplitudes. Various searches for the 3/2 resonance in the Analogue have still revealed no detectable beam disturbance ascribable to that resonance. This fact is of great interest, since the 3/2 resonance is theoretically the most difficult of the resonances which must be passed in the proposed 900-Mev cyclotron.

Design work on Analogue II is now receiving special attention; preliminary calculations of coil shapes are in progress. This model will have a spiral-ridged field configuration. Various efforts were made to wind sample segment coils with small wire and small ribbon. Difficulties were encountered because of the curvature of the segments. An effort is now being made to manipulate the coil shape in such a fashion as to allow fabrication from a single turn of a large conductor. (AEC Activity 5240)

Mass Spectrometry and Related Techniques - Tests of the differential pumping system of the 6-in.-radius research mass spectrometer, RS1, showed that there is less than 1% dilution of the ionization gas by the gas in the analyzer chamber when the pressure in this chamber is increased from a reference pressure of 1×10^{-8} mm Hg to 1×10^{-5} mm Hg. Likewise, the pressure in the ionization chamber can be varied by three orders of magnitude without diluting the gas in the analyzer by as much as 1%. Thus the cross-contamination of gases in the two chambers is negligible, a condition which must be met in the study of the interaction of positive ions and molecules. (AEC Activity 5250)

CHEMISTRY

Raw Materials Chemistry - Synergistic enhancement of uranium extraction (in addition to stabilization of the extractant during alkaline stripping) by addition of neutral organophosphorus compounds to di(2-ethylhexyl) phosphoric acid (D2EHPA) in kerosene was described previously (ORNL-1995). The combination of TBP with D2EHPA is now used in several uranium mills. Commercial availability of another synergistic additive, dihexyl hexanephosphonate (DHHP), was recently announced at prices which appear feasible for process use. The uranium extraction coefficient from 0.5 M sulfate solution, pH 1, in combination with 0.1 M D2EHPA, is fourfold greater with DHHP than with TBP. Similar concentrations serve to maintain miscibility of sodium di(2-ethylhexyl) phosphate in the kerosene solution, and a smaller amount of DHHP than of TBP is lost by distribution to the aqueous phase.

	Minimum Concentration to Maintain Miscibility	$E_a^o(U)$ with 0.1 M Additive	Equilibrium Concentration in Aq. Phase
No additive		130	
TBP	0.08 M	500	~25 mg/liter
DHHP	0.06 M	2000	<5 mg/liter

Physical entrainment of solvent in the aqueous raffinate from either the Dapex or Amex processes has been effectively controlled by controlling the type of mixed emulsion in the contactors. With organic continuous mixing, entrainments of <0.2 gal of organic per 1000 gal of aqueous have been obtained after a 15-min settling period. With aqueous continuous mixing under otherwise similar conditions, the entrainment was tenfold greater. Low entrainments after aqueous continuous mixing were achieved only after extended settling times.

In continued study of amines and amine salts in organic solution, measurement of light scattering gave the average molecular weight of di-n-decylamine sulfate as ~ 27,000 (i.e., aggregates containing about 40 amine sulfates) from 0.0025 to 0.05 M in benzene, and that of tri-n-octylamine sulfate as 800 (i.e., no aggregation) up to at least 0.025 M in benzene. This is in general agreement with the results previously obtained by analysis of acid extraction equilibria, which indicated that tri-n-octylamine sulfate in benzene was aggregated only at concentrations above ~ 0.02 M, while di-n-decylamine sulfate was aggregated at all concentrations above ~ 0.001 M.

Uranium extraction from acid sulfate solutions by a benzene solution of the sulfate and bisulfate salts of either amine indicated complexing of the extracted uranyl sulfate with normal amine sulfate only, and without change of the bisulfate:normal sulfate ratio in that fraction which remained uncomplexed. Correlation of the uranyl extractions with aqueous sulfate concentrations permitted estimation of the aqueous uranyl sulfate complex formation constants at unit ionic strength:

$$K_1 = [UO_2SO_4]/[UO_2^{++}][SO_4^{--}] \approx 35,$$

$$K_2 = [UO_2(SO_4)_2^{--}]/[UO_2^{++}][SO_4^{--}]^2 \approx 200,$$

which are somewhat lower than, but still in good agreement with, the values 50 and 350 reported by Ahrland (Acta Chem. Scand. 5, 1151, 1951) from potentiometric measurements. However, the present results indicate little if any formation of $UO_2(SO_4)_3^{4-}$ in solutions up to 1 M in sulfate, $K_3 \ll \sim 100$, in contrast with $K_3 \approx 2500$ (ibid.). (AEC Activity 5310)

Chemical Physics - Various concentrations of solid sulfuric and perchloric acids at liquid-nitrogen temperature were irradiated with gamma rays, and the yield of hydrogen gas (and oxygen) was measured after melting to see whether there is a correlation with the atomic-hydrogen yield at low temperatures as measured by paramagnetic resonance. The gas yields and the atomic-hydrogen yield were found to have about the same concentration dependence in the more concentrated acids (over 5 mole % acid). Some of the more concentrated acids can be frozen either to glasses or crystalline solids, and in the case of sulfuric acid the yield of gas is some thirtyfold higher in the glassy material. The atomic-hydrogen yield shows, within the present accuracy of measurement, identical behavior. Moreover, a crude measure of the absolute atomic-hydrogen yield in sulfuric acid correlates with the molecular-gas yield. This demonstrates a correspondence between the directly measured intermediate (atomic hydrogen) and the final product (hydrogen gas) and is probably the first such case in radiation chemistry. (AEC Activity 5330)

Radio-Organic Chemistry - Absolute rates of decarbonylation of formic acid and formic-d acid in 96.2% sulfuric acid were determined at 0 and at 25°C. The calculated isotope effect ratios, k_H/k_D, are 1.49 at 25°C and 1.72 at 0°C. In terms of the reaction mechanism suggested by Hammett, these results, as well as the previously reported C^{14} isotope effect, can be tentatively explained as being due to alteration of the carbon-hydrogen and carbon-oxygen bonds during activation of the protonated formic acid molecules.

Evidence was found in the study of the mass-spectrometric cracking patterns of formic acid, formic-d acid, and formic acid-d that the mass-45 peak in the formic acid pattern may arise from two sources: (1) loss of hydrogen from the carbon atom to form $COOH^+$ and (2) loss of hydrogen from an oxygen atom to form $HCOO^+$. In the cracking patterns of formic-d acid and formic acid-d, hydrogen isotope effects appear to be affecting the relative abundance of several fragments.

Continued testing of thorio- and urano-organic derivatives led to the following conclusions: (1) carboxylate salts of both metals (such as benzoate) are insoluble in molten diphenyl up to 200°C; (2) chelates with acetyl acetone or dibenzoyl methane have an appreciable solubility in molten diphenyl but decompose above 200°C (evidenced by darkening). (AEC Activity 5330)

Radiation Chemistry - A large quantity (about 150 mg) of the solid polymer resulting from the alpha irradiation of CO was prepared. In the past only fractions of a milligram had been produced in this way. (AEC Activity 5330)

Molten Salt Thermodynamics - In a continuing investigation of the effect of composition and temperature on activities in molten salts, the $KCl-FeCl_2$ and $LiCl-FeCl_2$ systems have been studied by means of vapor-pressure measurements. Due to association in the vapor phase, it was necessary to combine total pressure and transpiration data in order to obtain the partial pressures of the vapor species. Pronounced negative deviations from Raoult's law were associated with a positive excess entropy of solution rather than with a negative heat of solution.

The vapors in equilibrium with a KCl melt were found to contain about 23% dimer molecules between 950° and 1150°C. In the case of LiCl, if the associated species is assumed to be all dimer, the apparent composition of the equilibrium vapor is about 67% dimer between 950 and 1050°C. For $FeCl_2$ the amount of dimer increases from 10% at 700°C to 25% at 900°C. Both the $KCl-FeCl_2$ and the $LiCl-FeCl_2$ mixtures gave 1:1 compounds in the vapor. For example, the vapor compound $KFeCl_3$ was the predominant species in equilibrium with the $KCl-FeCl_2$ melts containing 20 to 40% $FeCl_2$, accounting for 80% of the vapor molecules at a liquid composition of 35 mole % $FeCl_2$ at 1000°C. (AEC Activity 5330)

Analytical Chemistry Research - A rapid and sensitive method was developed for the flame photometric determination of lanthanum. Lanthanum is extracted away from many interferences with 0.1 M thenoyltrifluoroacetone (TTA) in 4-methyl-2-pentanone from 1 M acetate, buffered at a pH of 5. Only titanium and aluminum interfere when they are present in amounts greater than lanthanum, while fluoride and phosphate prevent the extraction of lanthanum with TTA. The oxide band at 741 mμ is the most suitable for the determination of lanthanum in the presence of interfering substances. The organic medium enhances the flame emission of lanthanum 100-fold; however, if excess quantities of TTA are present, erratic flame conditions result. It is necessary, therefore, to extract thorium, uranium, copper, and iron, when they are major components of the sample, at a pH of 1.5 before extracting the lanthanum, in order to maintain the TTA concentration at the appropriate level. By aspirating the organic solution of the lanthanum directly into the flame, individual determinations are completed in 30 sec with a coefficient of variation of 1%.

The solvent extraction of zirconium with 0.1 M tri-n-octylphosphine oxide (TOPO) in cyclohexane was shown to be best in acidic chloride and nitrate media. As much as 20 mg of zirconium is extracted quantitatively in a single equilibration with 0.5 millimole of TOPO from 7 M HCl. Under the same conditions 14 mg of zirconium is extracted from 7 M HNO_3. Addition of either chloride or nitrate to sulfate and phosphate solutions of zirconium greatly enhances the extraction coefficient; however, extraction of zirconium from fluoride solutions is essentially nil even when chloride or nitrate is added.

The spectrophotometric determination of zirconium in the TOPO-cyclohexane extract was shown to be feasible. Two reagents, alizarin and pyrocatechol violet, are satisfactory chromogenic compounds for this purpose. The colored complexes that are formed in the nonaqueous media are apparently identical with those formed in aqueous systems.

In the controlled-potential coulometric determination of uranium in HRT fuel previously reported, it was determined that molybdenum is the only fission product causing serious interference. This interference can be eliminated by extracting the molybdenum with a chloroform solution of alpha-benzoinoxime. (AEC Activity 5330)

METALLURGY

Ceramics Research - Counts on leach water activities of 80 sintered clay mixtures, made with 29,000-cpm Purex solution, indicate that for three of the ten clays represented the rate of leaching decreases markedly during the first month. Five of the clays showed better retention of activity by the 50-g clay + 250-ml Purex slurry than by the 100-g clay + 250 ml Purex solution slurry. Ball clays and attapulgites are the most promising ones tested for retention of activity. Waste solutions tagged to 200,000 cpm were utilized in several clay mixes; higher temperatures are necessary to fix these higher activities.

Reactions between samarium oxide and gadolinium oxide with excess silicon were studied. Two silicides of each of the rare earths were identified. Structure studies are incomplete, but the phases are related structurally to the corresponding uranium and thorium silicide. (AEC Activity 5420)

Fundamental Physico-Metallurgical Research - Development of fiber texture as a function of the amount of deformation was studied in an aluminum rod extruded at 1.8 fpm and a temperature below $0^{\circ}C$. Back-reflection x-ray patterns showed that the as-cast billet contained a ⟨001⟩ texture component. With increasing amounts of deformation, the axis density for the ⟨001⟩ direction at first decreased to that corresponding to a random texture and then increased with greater amounts of deformation. Metallographic studies indicated that the increase in the amount of ⟨001⟩ texture was accompanied by the appearance of recrystallized grains. Thus, contrary to existing theories of deformation texture formation, the initial deformation appears to cause grains having ⟨001⟩ directions parallel to the flow direction to move away from this orientation. After a relatively small amount of deformation, recrystallization is initiated which causes the ⟨001⟩ directions in the new grains to be oriented parallel to the rod axis.

Recent results in the study of binary-phase diagrams of zirconium are as follows: (1) The slightly rising α/β phase boundaries of the Zr-Cd system were verified within $\pm 2^{\circ}C$ by a determination of the vapor pressure of cadmium in a 4 at. % Cd alloy. The activity of the beta phase of this alloy, relative to liquid cadmium, was 0.10 at $1250^{\circ}K$, indicating a positive deviation from Raoult's law. Apparatus to contain this alloy at $1100^{\circ}C$ must withstand cadmium partial pressures of 4 atm. (2) A new investigation of the specific heat of pure zirconium shows that the specific heat corresponds to a $(\gamma T + \beta T^3)$ relationship within 1% over the temperature range 1.8 to $4.5^{\circ}K$. (AEC Activity 5420)

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RADIOISOTOPE SALES AND COSTS

Type of Transaction	May 1957	FY 1957 to Date
Domestic Sales	$125,833	$1,752,423
Foreign Sales	9,702	162,791
Project-Transfer	1,256	13,531
Project-Cash Sales	7,989	63,180
Technical Co-operation Program Credits	0	3,395
Plant Credits	4,354	35,075
AEC Credits	19,155	191,297
Total Radioisotope Income	$168,289	$2,221,692
Total Radioisotope Costs	$153,255	$1,393,484
Total Radioisotope Shipments	1,190	12,567
Helium		
Income	$4,845	$43,362
Costs	2,996	6,258
Shipments	3	35

GROSS OPERATING COSTS

	Cost for May	FY 1957 Cost to Date
Programmic Operating Cost - Net	$4,268,118	$47,107,946
Equipment	232,010	2,382,933
Construction	183,997	1,798,475
Work for Others - Transfers	18,488	582,869
Inventory Changes	(112,169)	(169,968)
Reimbursable Work for Others	70,242	767,484
Deferred Charges	27,454	(227,901)
Total Laboratory Cost - Net	$4,688,140	$52,241,838
Estimated Cost for Next Month	$6,000,000	$58,241,838

Credit ()

PERSONNEL SUMMARY

	Number of Employees June, 1957	New Hires June	Terminations June
Administration	70	0	0
Operations*	134	2	1
Engineering, Shops, and Mechanical	868	11	7
Laboratory and Research	2360	182	19
Protection	116	0	0
Service	399	33	4
	3947	228	31

*Includes Electrical Distribution and Steam Plant as well as the Operations Division.

933 Laboratory employees are working in the Y-12 Area.

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April 1957	ORNL-2316
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