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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

JANUARY 1954

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OAK RIDGE NATIONAL LABORATORY

STATUS AND PROGRESS REPORT

January, 1954

F. T. Howard

and

W. H Sullivan

Date Issued: FEB 5 1954

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January, 1954

The subject material of this report represents approximately half of the Laboratory's program, which is covered with some exceptions on a bimonthly schedule.

PROGRAM 2000 - SOURCE AND FISSIONABLE MATERIALS

Waste Metal Recovery Plant Operation - The metal recovery plant is being modified to permit recovery of plutonium and americium from Hanford metallurgical waste. During the past year, January 2, 1953 to January 23, 1954, a total of 95,550 kg of uranium was recovered from accumulated X-10 wastes. The fission-product beta and gamma activity in the recovered uranium did not exceed 100 percent of the background activity of natural uranium (which is the specification set by K-25). A total of 208.3 g of plutonium was also isolated. (AEC Activity 2344)

Feed Materials - In studies on the production of uranium metal from uranium tetrachloride, 75 percent of the uranium was reduced by a single-stage contact of excess lithium amalgam with uranium tetrachloride dissolved in propylenediamine. The fraction of uranium reduced was not affected by temperature changes in the range 25° to 120°C. If the propylenediamine contained more than 0.1 wt percent water, the uranium tetrachloride hydrolyzed to a nonreactive form of uranium. Centrifugation of the uranium-lithium amalgam phase produced a quasi-solid phase, containing uranium and mercury, which was heated in an inert atmosphere to distil off the mercury, leaving a pyrophoric uranium metal powder.

The use of hydrazine as a reducing agent was studied as a step in a proposed process for the production of uranium tetrafluoride. A 95 percent yield of UF_4 was obtained by treating the double salt, $UO_2F_2 \cdot xNH_4F$, with 42 percent hydrazine hydrate, evaporating it to dryness at 100 to 150°C, and heating the residue at 300°C. Due to simplification of equipment, this process may have economic advantages over the present gaseous process which uses H_2 in HF. The present cost of hydrazine, however, makes this process unattractive, but large-scale commercial production of hydrazine, which is planned shortly, may reduce its cost.

Further studies (see report ORNL-1651) were made on a process, for use at the Feed Materials Production Center (FMPC), to produce low-fluoride uranium from a hydrochloric acid digest of uranium-reduction bomb and liner waste. The uranium from the digest liquor was sorbed on Dowex A-1 anion exchange

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resin, washed with 10 M hydrochloric acid, and converted on the resin bed to uranyl nitrate with calcium nitrate. Elution of the uranyl nitrate with water gave a product containing 30 g of uranium and 24.7 g of calcium per liter, 6 ppm of fluoride, and less than 5 ppm of chloride. FMPC specifications permit up to 500 ppm of fluoride. A scouting run on a second method, in which uranyl chloride was eluted from the resin bed with water and the uranium was precipitated as ammonium diuranate, gave a product with 800 ppm of fluoride and 79,000 ppm of chloride. This second method would require fewer changes than the first in the present operating procedures at FMPC. The uranium capacity of the resin in the scrap digest liquor medium was 65 g of uranium per liter of resin.

Purex Process Development - A method, based on the reaction of easily oxidized compounds with chromyl chloride, was developed for determining the amount of such compounds present in hydrocarbons. This method will simplify preliminary evaluation of hydrocarbons for use in tributyl phosphate solvent extraction processes, where easily oxidized compounds, which cause emulsions in the extraction columns, are undesirable. (AEC Activity 2802)

PROGRAM 3000 - WEAPONS

RaLa Production - A RaLa production run which was begun in November was unsuccessful because of equipment failures and processing difficulties. The stainless steel micrometallic process filters used to filter out the Pb-$BaSO_4$ plugged completely. Backwashing and blowing back with air failed to clear the filter and resulted in leaks at welds and gasketed joints.

The steps used in removing the coating from irradiated slugs and in filtering the uranyl nitrate hexahydrate (UNH) were then re-examined. It was found that, by washing the uranium slugs more vigorously after the coating was removed and by increasing the strength of the HNO_3 used to remove the U-Si-Al bonding alloy, it was possible to obtain a dissolver solution containing less $SiO_2 \cdot H_2O$ and having much better filtration properties. It was found also that the "fines" in the filter aid which had been used in UNH filtration sifted through the Type-G micrometallic filter and interfered with the metathesis and subsequent operations. In a subsequent RaLa production run improved coating-removal procedures were used and the filter aid was eliminated. In this run very smooth and fast operation was obtained, process efficiency was markedly improved over recent runs, and the product yield was the largest yet achieved in the production program. (AEC Activity 3702)

HOMOGENEOUS REACTOR PROJECT

Homogeneous Reactor Experiment - The previously reported (report ORNL-1665) effectiveness of copper, i.e. $CuSO_4$ dissolved in the fuel solution, as a catalyst for the internal recombination of hydrogen and oxygen was further verified by experimental tests. It is planned to use this internal catalyst in future experimental homogeneous reactors.

A total of over 500 megawatt-hours of operation was accumulated during January. The experimental program planned for the HRE is now practically completed. The reactor will be operated continuously at high power for two one-week periods before the final shutdown; the HRE # 1 will then be dismantled to make room for the installation of HRE # 2, which will be known as the Homogeneous Reactor Test (HRT). Dismantled HRE components will be carefully examined in detail to obtain metallurgical and corrosion information. (AEC Activity 4103.1)

HRT Conceptual Design - A homogeneous test reactor (HRT) is being designed to replace the HRE. The new design will provide a reliable experimental reactor for investigating general operational problems of aqueous homogeneous reactors and for testing fuels and novel design concepts that are being considered for large power and production reactors.

The HRT as now conceived combines features of both the HRE and the proposed Intermediate Scale Homogeneous Reactor (ISHR). It is a 5-Mw, two-region reactor fueled with highly enriched uranium, moderated and reflected with D_2O, and operated at 300°C and 2000 psia. The core is a spherical vessel 32 in. in diameter and fabricated of 1/4 in. zirconium. A 10-gm U/kg-D_2O solution is circulated through the core and then through external heat removal equipment at a rate of 400 gpm. The fuel solution contains copper sulfate as a catalyst for the recombination of D_2 and O_2 produced in the core. The core tank is surrounded by a 12 in. D_2O reflector contained by the reactor pressure vessel. The reflector liquid is circulated through heat removal equipment similar to that provided for the core. Uranyl sulfate solutions containing natural, or depleted, uranium or thorium-D_2O slurries can be substituted for D_2O in the reflector to investigate the problems of production of Pu^{239} and U^{233}. (AEC Activity 4109.1)

Reactor Physics - Analysis of the water-boiler kinetic experiments indicates that about 10% of the decomposition products of H_2O appear in the vapor phase within 0.1 second.

Several methods of making calculations for small spherical reactors with heavy water for both moderator and reflector were compared. In particular, the harmonics method converges very rapidly for reactors in which the reflector thickness does not exceed the core radius. The best estimate of the critical mass is obtained from the harmonics method which uses a Gaussian-Yukawa slowing-down kernel. The two-group method gives critical masses which are considerably lower than those obtained by using the Gaussian-

Yukawa kernel. The harmonics method was extended to take into account both multiplication in the reflector and resonance capture. (AEC Activity 4109.2)

In-Pile Loop - Preliminary tests of the loop assembly to be used in the LITR for the study of radiation effects on corrosion were largely completed with generally satisfactory results. The specified temperature, pressure, and flow control seemed to be adequately attainable with the assembly as designed. The design of the operating equipment for the in-pile installation is essentially complete and construction has been started.

Results from the irradiation of samples in the LITR are being correlated with results obtained from out-of-pile tests. At present it appears that those variables (e.g. loop surface-to-volume ratio and previous loop history) which have appeared as second order in the out-of-pile work are materially affecting these correlation studies. (AEC Activity 4109.4)

Dynamic Loop Tests - Corrosion data for 347 stainless steel, exposed to uranyl sulfate solution containing 40-gU/l at 200° and 225°C, were summarized in the November Progress Report (ORNL-1651). These intermediate temperature studies were extended since then to 5-g U/l UO_2SO_4 solutions. The results indicate that, after an initial period of several hundred hours, the corrosion rates at low velocities level out to less than one mil per year. For the higher concentrations the corrosion rate was as much as 29 mils per year.

An all-titanium loop has now been operated for 693 hours at 300°C with 5-g U/l UO_2SO_4 solution containing 0.005-M H_2SO_4. It appears that this amount of acid is not sufficient to prevent the precipitation of the double oxide of titanium and uranium. Such precipitation was observed previously when this system was run at the same sulfate concentration but with no excess acid and at 250°C. (AEC Activity 4109.4)

Engineering Development - A 4000-gpm test loop is being fabricated to operate with uranyl sulfate solutions in the temperature range 250°-300°C at pressures from 1000 to 2000 psi for component performance and corrosion studies.

A 1-gpm Pulsafeeder pump satisfactorily circulated 500 gTh/l thoria slurry against a 1000 psi head. When the pump was started after allowing the slurry to settle overnight, it reached full flow in approximately 10 minutes.

Hydrodynamic studies of an 18-inch model of a new type of reactor core configuration indicate that this arrangement, having both inlet and outlet at the top and no screens or baffles, shows promise for use in reactor systems where a copper catalyst prevents the formation of gas bubbles. The core is of extremely simple construction, and has no problems of differential expansion. (AEC Activity 4109.4)

Instrumentation and Controls - From an instrumentation standpoint the most significant changes in the conceptual design of the HRT reactor as compared with the HRE are the elimination of control rods, the more rigorous conditions of 2000 psi and 300°C in the fuel system, the possible flooding with water for shielding during repair operations, and the emphasis on long-term reliability. The absence of control rods eliminates the principal control function of the radiation detection equipment and will greatly simplify the installation of these instruments.

Operation of valves and pressurizer level controller have been only fairly satisfactory under HRE operating conditions of 1000 psi and 250°C. Very few manufacturers are interested in supplying valves for use at 2000 psi and 300°C. Encouraging progress has already been made at ORNL in redesigning such valves and the liquid level controller.

The use of water for shielding will probably require that a number of measuring and actuating components be able to withstand submersion. This practice will require improved insulation of thermocouple and electrical lines. Pneumatic devices must have weatherproof housings which are equipped with "snorkels" to permit exhausting above water level. (AEC Activity 4109.6)

AIRCRAFT NUCLEAR PROPULSION PROJECT

Aircraft Reactor Experiment - The piping for the sodium and fuel systems was completed, except for some annulus sections. Flushing of the fuel system was competed; as soon as the pump assemblies are installed dynamic tests will be made with water at room temperature. The heaters on all tanks were replaced with tubular units. The reactor is now complete and is in position in the reactor pit. Components for the auxiliary cooling system for the pumps were fabricated and installation is 50% complete. The basic design for the fuel removal system was established and detailing of this design is now in progress. The neutron instrumentation was checked by placing a neutron source in the reactor. The electronic equipment, comprising the counting and monitoring circuits, was found to function properly. (AEC Activity 4401.1)

General Aircraft Reactor Design - A Lucite model of a sump pump, incorporating a centrifuge type of expansion tank, was fabricated and is being tested. Test results are promising for aircraft application in that the pump will deaerate in the speed range below 1400 rpm and it gives only a slow rate of aeration with the shaft horizontal. With the pump upside down, the rate of aeration is roughly 400 in.^{3}/min. It appears that this rate is tolerable, from the control standpoint, if inverted flight is limited to those short, rare periods imposed by gusts in violent storms. The pump is being modified in an attempt to prevent it from shifting to an aerating action at speeds above 1500 rpm.

A plastic model of the essential parts of a pump for the fluoride system, the pump volute, plenum chamber region, and core shells, was constructed for flow studies. The tests indicate that, as expected, turning vanes will be required at the impeller discharge for approximately 90 degrees of the periphery ahead of the volute cutoff point. The velocity distribution through the core is fairly good. There are no indications of serious flow separation even though no mixing vanes have been installed, as yet, in the core inlet passage. (AEC Activity 4401.1)

Experimental Engineering - Mechanical Contrivances - The sump pumps for in-pile loops were modified successfully to overcome gassing and yet maintain an ample safety factor in pressure head and flow requirements. These pumps proved satisfactory in mechanical reliability tests at room temperature, and are now being subjected to high-temperature tests. The development and design of a pump for in-pile installation is now being undertaken so that the costly shielding required for the external pumps can be eliminated.

Thin, concave, Inconel disks (Fulton Skinner seals) were tested for 300 hr as retainers for a graphite and molybdenum disulphide packing mixture; the results are good, thus far. Pump shafts with spiral grooves within the packing region designed to aid retention have operated well, have not had incipient failures despite high impregnation of the packing with fluorides, and apparently have high reliability. One seal, operated 3500 hr before a heating failure, caused termination of the experiment. Post-run examination indicated only minor damage to the shaft, and the packing was thoroughly impregnated with fluorides.

In another life test, a shaft, which was sealed with a mixture of graphite and beryllium fluoride, operated for 4500 hr before termination caused by shaft seizure. Post-run inspection showed severe shaft scoring, but the seal had not failed.

No materials other than those containing either graphite or beryllium fluoride, or a combination of both, as the primary seal constituents were used successfully in sealing rotating shafts in fluorides, and these were only successful with shafts under 1-1/2 in. in diameter.

Impellers, fabricated by welding, provide adequate capacity for the sodium pumps and have been chosen because of the difficulty in obtaining sound impellers of cast Inconel. Drilled-port impellers were selected for the fluoride pumps because of simplicity of fabrication and to provide the highest impeller efficiency at specified operating conditions.

Mechanical difficulties encountered in the primary pump seal were corrected by proper hardening of the metal seal faces. The difficulty in the oil-to-air seal appeared to be due to inaccuracies in the flatness of adjacent seal surfaces and to a possible unloading of spring forces because of the 60-psi pressure under which the oil system operates.

In another ARE component test, a d-c motor under load in a helium atmosphere at 140°F (similar to the conditions anticipated in the ARE pits) operated 1150 hr without incident. The brush wear appears to be entirely normal. (AEC Activity 4401.1)

Experimental Engineering - Heat Transfer Research - A sodium-to-fluorides heat exchanger, consisting of six tube bundles each of forty 0.010-in.-ID Inconel tubes, was operated for 1700 hr at various temperatures, for 1300 hrs in the 1200° to 1500°F range. Visual inspection of the heat exchanger surfaces after termination due to a power failure showed them to be very clean and with no sign of damage. (AEC Activity 4401.1)

Mathematics and Computation - A preliminary analysis of the results of a large number of two-region multigroup calculations shows that, as expected, the critical mass of a beryllium-reflected NaOD-moderated fluoride-fuel reactor is less than half that of the similar two-region reactor without hydroxide in the core. This critical mass appears to be about the same as that of the three-region, Be-island, fluoride-fuel-annulus, Be-reflector reactor design. However, if Li^7OD is used instead of NaOD, the critical mass would be reduced by a factor of 2. (AEC Activity 4401.1)

Reactor Chemistry - Tentative agreement was reached with representatives of Pratt and Whitney Aircraft Corporation regarding production of purified $NaZrF_5$ and $NaZrF_5$-UF_4 mixtures for their engineering-scale testing program. The schedule calls for a total of 1000 lb of material to be delivered before April 1; Pratt and Whitney's estimate of their needs, which may be too high, is set at about 9000 lb of material for the calendar year 1954. This quantity of material, superposed on the estimated ORNL needs for 5000 to 6000 lb, can be prepared in equipment which is available or presently being constructed.

Continued study of the NaF-ZrF_4 binary system by several techniques shows the existence of a compound at 40 mole % ZrF_4 (presumably $Na_3Zr_2F_{11}$) which has a mean refractive index of 1.47, a very low birefringence (about 0.004), and a distinctly fibrous habit. Its x-ray diffraction pattern is that of a nearly cubic compound. It melts incongruently at about 510°C to Na_2ZrF_6 and a liquid.

Recent experiments show that uranium metal reacts with pure molten $NaZrF_5$, under an atmosphere of hydrogen at 800°C, to produce UF_3 and zirconium hydride:

$$H_{2(g)} + U_{(c)} + NaZrF_{5(l)} \longrightarrow UF_{3(l)} + ZrH_{2(c)} + NaF_{(l)} .$$

The UF_3 and NaF shown as reaction products are soluble in the large excess of $NaZrF_5$, at least to the extent of 2 mole % of UF_3. Visual, x-ray, and petrographic examination of the resulting solidified melt shows no uranium metal and indicates complete reaction. It is very likely, although not completely certain, that the corresponding reaction

$$U_{(c)} + NaZrF_{5(l)} \longrightarrow UF_{3(l)} + Zr^{o}{}_{(c)} + NaF_{(l)}$$

is complete in the absence of hydrogen.

Experimental study of the reaction of Cr^{o} with FeF_2, by using molten $NaZrF_5$ as the solvent, has shown the reaction to be

$$Cr^{o}{}_{(c)} + FeF_{2(l)} \rightleftharpoons Fe_{(c)} + CrF_{2(l)} .$$

The reaction proceeds so nearly to completion that accurate and reproducible figures for the equilibrium constant are difficult to determine from the analytical data. (AEC Activity 4401.5)

Corrosion Research - Static corrosion tests showed that carburization or decarburization of structural metals is dependent on the carbon content of the fluid in contact with the structural material. Therefore, additional static tests are being made to determine the effects of additions of 0.1, 0.5, and 1% graphite to lithium, sodium, and various fused salts contained in Inconel, Hastelloy B, and types 310, 316, and 430 stainless steel.

A series of Inconel thermal convection loops, operated with fluoride mixtures of varying uranium content (0.5 to 15 wt %), showed gradually increasing depth of attack (up to 2 mils) with increasing uranium content. However, fewer deposits were found in the cold legs of the loops in which the mixtures of higher uranium content were circulated.

Two thermal convection loops of 1/2-in. tubing were fabricated from special, high-purity, low-carbon (0.014%) Inconel cast by the Metallurgy Division. In one loop, titanium (0.3%) was added to tie up any remaining carbon. In both loops, the maximum depth of attack was 3.5 mils in 500 hr of operation with high purity $NaF-ZrF_4-UF_4$. Similar loops, fabricated from commercial tubing, showed attack to 3 mils in most areas, but the attack reached a depth of as much as 5 mils in a few spots. Little, if any, improvement was obtained with the high purity Inconel. In the ranges normally encountered, carbon does not appear to influence the attack sufficiently to be of interest. (AEC Activity 4401.5)

Metallurgy - Fabrication - For use at high temperatures, niobium and molybdenum must be clad with an oxidation-resistant material. Samples of niobium were roll-clad at temperatures of 900°, 1050°, and 1200°C to establish a metallic bond between the niobium and the following cladding materials: nickel, Hastelloy B, Inconel, and types 446 and 310 stainless steel. These experiments showed that the rolling temperature should be less than 1200°C to prevent too much diffusion, which would result in the formation of brittle intermetallic compounds. It was found also that nickel-base alloys exhibit considerable inter-diffusion and the formation of a brittle gray phase.

These results indicate that an iron-base alloy should be used as the cladding metal and that the hot rolling should be done at approximately 900°C. (AEC Activity 4401.5)

Metallurgy - Welding and Brazing - In the past it was thought that most brazing alloys would wet high-temperature alloys only when hydrogen was present as a reducing agent. In recent experiments it was found that Nicrobraz, Ni-P, Au-Ni, and Cr-Ni-Si alloys will wet nickel, Inconel and type 304 stainless steel when brazed in a fast-flowing helium atmosphere. These findings are of importance because in certain applications it is desirable to use a dry neutral helium atmosphere when ceramic or other nonmetallic materials are being used as jigs or fixtures for precision brazing applications. The use of helium has obvious advantages from the standpoint of safety. (AEC Activity 4401.5)

Reactor Physics - Reactor problems are being set up for solution on the Laboratory's new computer, the ORACLE. A method has been devised for obtaining the energy distribution of neutrons slowing down in moderators of constant scattering and 1/v absorption cross sections. (AEC Activity 4401.7)

Shielding Research - Testing of the rear section of the mockup of the shield for the General Electric Company GE-R1 reactor was completed at the Bulk Shielding Facility (BSF). As expected, peaks of radiation intensity occurred beyond the voids in the shield. Measurements are now being made behind the front section of the mockup. After testing of the front section is completed the entire shield assembly will be tested at the Tower Shielding Facility (TSF) to determine the overall performance of the shield design.

Improved statistics resulting from long counting times have resulted in more accurate measurement of the fast-neutron spectrum at the face of the reactor in the BSF. Translation of counting rates into flux values is in progress.

All the detection instruments for the isodose plotter at the TSF have been assembled. Operational checks are now being made. (AEC Activity 4401.7)

ARE Fuel Recovery - Present plans call for extracting the uranium from the ARE fuel with 5% tributyl phosphate and isolating it by ion exchange. The aluminum containers for transporting the ARE fuel to the processing site will be 8.5 in. in diameter and 17 in. high. They will hold as much as 50 kg of fused salt containing 4 kg of U^{235}. Extrapolation of data obtained by completely dissolving cylinders of simulated fuel 5 in. long and 5 in. in diameter in a 50-gal dissolver indicates that 12 hr will be required for dissolution of fuel charges 17 in. long and 8.5 in. in diameter. The 12-hr dissolving time was determined under conditions more like those expected in the plant than the 6- to 8-hr value mentioned in a previous report (ORNL-1651) and is therefore believed to be more accurate. Calculations indicate that 8 in. of lead shielding will be required for the carrier.

In batch countercurrent tests of the proposed one-cycle solvent extraction process (report ORNL-1556), the uranium was decontaminated by a factor of 2×10^5. This decontamination will permit the ion exchange uranium product to meet Y-12 specifications (fission-product beta and gamma activity not exceeding 30 times that of normal uranium). Batch ion exchange tests have been made, and calculations based on the results of these tests indicate that a 3-in.-dia by 6-ft-long Higgins continuous ion exchange contactor will permit a processing rate of 9 kg of uranium per day.

In a scouting experiment on the feasibility of recovering uranium from fused fluoride fuel by fluorination of the molten salt, 99.98% of the uranium was volatilized as uranium hexafluoride when the salt was treated with a 23-fold excess of elemental fluorine for 4 hr at 620°C. A gross beta decontamination factor of 270 was obtained. (AEC Activity 4401.81)

SCHOOL OF REACTOR TECHNOLOGY

School of Reactor Technology - Students in ORSORT have completed their first semester examinations and are now beginning their second semester studies.

Applications are being received for the 1954-55 session of ORSORT, which is to start September 13, 1954. The final date for receipt of such applications is March 15, 1954. (AEC Activity 4841)

GENERAL REACTOR RESEARCH

Idaho Chemical Plant - In further studies (see ORNL-1651) on improving the recovery of U^{235} from fuel assemblies used in the Materials Testing Reactor (MTR), the decontamination of the uranium product from ruthenium was increased from 6×10^4 to 1×10^7 by pretreating the feed to the second and third cycles with sodium nitrite. If this same improvement in decontamination is achieved in the plant, the U^{235} product from 120-day-cooled MTR assemblies would have a ruthenium beta activity of only 10% of that of unirradiated U^{235}. At Y-12, specifications limit the beta activity to 30 times that of U^{235} background.

A solvent-extraction process, in which 1.5% tributyl phosphate is used as the extractant and aluminum nitrate as the salting agent, was developed for separating U^{235} from STR and SIR fuel dissolver solutions. In laboratory studies, the process gave beta decontamination factors greater than 10^6, with a uranium loss of less than 0.1% per cycle. (AEC Activity 10543)

PROGRAM 5000 - PHYSICAL RESEARCH

REACTOR OPERATIONS

Graphite Reactor - Many slugs now in the reactor were never heat treated to transform the uranium to the beta phase. Measurements of several rows of these slugs while still in the pile indicated that the rows had increased in length approximately 6 in. This $\sim$0.143 in. average elongation per slug agrees fairly well with earlier measurements (0.187 in) obtained by measuring individual slugs after they had been irradiated a comparable period (464 days). A careful watch is being kept on all such slugs in the reactor; eventually they will have to be replaced.

Low Intensity Test Reactor (LITR) - The weak point in the LITR is generally believed to be the neoprene gasket in the reactor tank. A sample of this gasket material which was suspended in the tank near the installed gasket was examined after accumulating a dose equivalent to 15% of that accumulated by the actual gasket. The test sample was hard and brittle, but the elongation was only 1% and there was no indication of crumbling. The test is being continued and periodic examinations will be made.

ISOTOPE PRODUCTION

Radioisotope Production - Radioisotope shipments increased from 10,691 in 1952 to 12,036 in 1953, and the amount of activity shipped, excluding Co^{60} sources, increased 50.5%. The largest gains were made in Cs^{137}, Sr^{90}, Ru^{103}, Cr^{51} and Tl^{204}.

The new I^{131} production unit is nearing completion; chemical test runs will be made in February. (AEC Activity 5111)

Radioisotope Research - Physical and chemical studies were made on Cs^{137} for use in preparing intense, concentrated gamma-ray sources for teletherapy machines. $Cs^{137}Cl$ is easily handled as dry powder, is non-hydroscopic, and can be formed into dense pellets at 15,000 psi. A test pellet of pure fission product $Cs^{137}Cl$, 1 cm in diameter and 1.1 cm high, contained 60 curies. The density was 3.37 g/cc and the activity concentration was 20 curies Cs^{137} per gram gross weight.

Crude, compressed off-gases from the Arco dissolver were processed to separate and purify the Xe and Kr fractions. The main krypton isotope present is K^{85}. The xenon fraction, thought to contain only stable isotopes, was separated and purified. To check the purity of this xenon gas mixture, the atomic weight was measured and found to be 131.4. This value was in good agreement with the atomic weight calculated from fission yield data. The very minute amount of activity remaining in the Xe fraction was tentatively identified as Kr^{85}.

The separation of long-lived fission products from wastes, such as Purex LAW, has become an important chemical development problem. In the all-precipitation process being developed, the selective separation of the large amounts of Fe contamination before the carbonate precipitation of alkaline earths has been a major problem. It was found that $Fe_2O_3 \cdot xH_2O$ precipitates brought down by hydrolysis of urea in boiling solutions carry Ru but only minor amounts of the other fission product elements. These results have been obtained on test batches up to 100 curies. (AEC Activity 5131)

PHYSICS

Neutron Diffraction - Neutron diffraction data were obtained for iron at a series of elevated temperatures up to 1000°C in order to establish the magnetic scattering effects through the Curie temperature (T_c) and in the γ-phase region. Most of the change in the diffuse scattering as the temperature was raised to near T_c can be accounted for by thermal scattering in the normal crystal lattice. The excess scattering is very much smaller than the calculated paramagnetic scattering. Pronounced, temperature-dependent small angle scattering was noticed, however, which appeared to be magnetic in origin. This small angle scattering indicates a gradual disruption of the magnetic lattice into smaller domains as the temperature is increased until at T_c only a few unit cells are magnetically coherent. Above T_c the small angle scattering transforms gradually into short-range order scattering and this persists into the γ-phase region. The patterns taken for iron in the γ-phase indicate a normal face-centered cubic lattice with no suggestion of an antiferromagnetic structure. (AEC Activity 5211)

Slow Neutron Physics - Single magnetized crystals of magnetite were used as the neutron beam polarizer and the analyzer in an experiment which demonstrated anew the value of the spin of the neutron. The intensity of neutrons reflected from the analyzer crystal was measured as a function of the amplitude of an oscillating magnetic field located between the polarizer and analyzer. When these measured intensities were compared to those calculated for different assumed values of the neutron spin, agreement could be achieved only for $S = 1/2$. (AEC Activity 5211)

High Voltage Program - The triton yield of Li^6 (d,t) was measured; the cross section was found to rise from 0.05 barn at 0.37 Mev to 0.22 barn at 2.05 Mev. Most of the change occurs below 1 Mev. The targets used allow a measure of the forward and backward yields. In the center-of-mass system, the data show that at $<$ 0.6 Mev an excess of tritons are scattered in the backward direction. Above this energy there is a forward excess. (AEC Activity 5211)

Theoretical Physics - Attention has been given to the problem of the angular correlation of gamma rays and other radiations emitted by a nucleus which has been excited by electrostatic interation with incident protons. When the second or higher excited level of the nucleus is reached in the reaction

there is a cascade of two or more gamma rays. The correlation of these γ rays with the incident beam is a triple correlation process and the primary problem is the determination of the particle parameters. (L. C. Biedenharn and M E. Rose, Rev. Mod. Phys. 25, 729 (1953).) These particle parameters are just the same as in the correlation of one gamma ray with the proton beam (K. Alder and A. Winther, Phys. Rev. 91, 1578 (1953).)

The previously reported extended computation of the Racah coefficients is now complete. The tabulation printed by the UNIVAC is being photographically reproduced for final distribution. A UNIVAC code for a similar (but simpler) tabulation of the Clebsch-Gordan coefficients is being prepared. Work continued on the previously described machine computation of electron scattering from nuclei. The coding of this problem for the ORACLE is now in progress. (AEC Activity 5211)

CHEMISTRY

Waste Treatment Studies - In further radiation damage tests (see report ORNL-1651) on materials for possible use in lining earth basins to contain boiling (230°F) radioactive wastes, a paraffin-filled "P_2O_5-blown" asphalt and two commercially available asphalt membranes (one filled with vegetable fiber and the other with asbestos) were not changed by exposure to 1×10^9 roentgens of gamma radiation. All other materials tested became honeycombed during this treatment and therefore cannot be used when impervious membranes are needed. These asphaltic materials melt at 230°F and, if used, will have to be insulated from the boiling waste. (AEC Activity 5361)

Chemical Separation of Isotopes - It was demonstrated that the ammonia (gas) vs ammonium nitrate (aqueous) countercurrent system for the enrichment of nitrogen isotopes can be satisfactorily refluxed at the aqueous end by the use of a lime slurry. The lime, used to convert NH_4^+ to NH_3, replaces the more expensive sodium hydroxide in the refluxing step. Three runs were made with an infinite feed of ammonium nitrate. At the bottom of the exchange column the ammonium nitrate was mixed with lime slurry and fed to the stripping column. Analysis of the effluent from the stripper showed no ammonia present to the limit of the analysis ($<$.001M). Isotopic assays on samples taken from the bottom of the exchange column showed a total separation factor of 1.19 in a 7.5 hour run. An approximate extrapolation to equilibrium yielded a total separation factor of 1.26 and a stage length of less than 12 inches. No attempt was made to minimize the stage length in this run.

A program for the enrichment of chlorine isotopes was initiated with a study of the system Cl_2 (gas) vs Cl^-(aqueous). The system is being studied in a 12-ft column, 9 ft of which is used as the exchange section and the remainder serves for refluxing. Chlorine gas, which is used as the infinite reservoir, is passed up the column countercurrent to an acidified aqueous

chloride ion solution. Upon reaching the reflux section the chlorine is reduced to aqueous chloride ion which flows back down the column and is discarded at the bottom. Alkaline sodium nitrite is being used at present as the reducing (reflux) agent. In a preliminary trial a total separation factor of 1.023 was achieved with Cl^{37} concentrating at the top of the exchange section. (AEC Activity 5361)

Instrumentation for Nuclear Chemistry - An electron-beam pulse-amplitude analyzer (report ORNL-1587) which uses a "picket fence" type of grid as a means of sorting pulses into many discrete channels was modified. Instead of using a grid structure to define the position of the electron beam a different type of target was devised and investigated. The target, made of Be-Cu, was activated by heating it to 600°C in a vacuum in order to increase its secondary emission yield to about 3.0 (electrons emitted/incident electron) at an incident electron beam energy of 2 kev. After activation, a metal grid mask having an equal number of spaces and webs was placed over the Be-Cu target and a thin layer of nickel was deposited through the parallel slots in the grid by vacuum evporation. Thus, the face of the target became a series of uniformly spaced bands of nickel and Be-Cu. Since the difference in the secondary emission yield for nickel and activated Be-Cu surfaces is about 2.0, a series of pulses are observed when an electron beam is deflected across such a target. The number of pulses obtained thus shows which discrete channel the initial pulse falls into. (AEC Activity 5361)

Hot Laboratory Research - Approximately 139 mg of high purity Np^{237} was isolated from Chalk River metal recovery wastes for use as a monitor in the development of nuclear weapons. Separation of the Np^{237} from U, Pu, Am, and the lanthanides was accomplished with a chloride system in Dowex-1 anion resin columns. Advantage was taken of the fact that the heavy elements existing in an oxidation state higher than III, form complexes in strong HCl solutions and are readily adsorbed by an anion exchange resin. Rare earths and other non-complexing ions were separated from U, Np and Pu on a 1.2-liter Dowex-1 resin column by using 10 - 12-M HCl. Uranium was removed from the Np and Pu on a 6-ft, 0.8-liter column conditioned with 5-M HCl; the U was more strongly complexed at this acidity. On reducing the Pu to the +3 state with $SnCl_2$, at least 97% of it was removed from the Np^{237} in one pass through a column with 10 - 12 M HCl.

Plutonium contamination of the Np^{237} was determined, by pulse analysis and column separation, to be not more than 0.4 ppm. U^{238} contamination is being determined by activation analysis. (AEC Activity 5361)

Chemistry of Corrosion - In continuation of the study of the electrochemical behavior of stainless steel in dilute sulfuric acid, some experiments were made on the changes of potential associated with the formation and breakdown of oxide films. It was shown that the film formed by oxidation in air at 450°C is about as noble as a platinum electrode under the same experimental

conditions. When the film fails, however, the potential falls by approximately a volt and hydrogen is liberated. The active metal resulting from film breakdown goes into solution at a nearly constant potential. After the metal goes into solution the electrode potential rises abruptly to a new height corresponding to the oxidation of the hydrogen adsorbed or dissolved in the metal at the lower potential. Evidence was also obtained that the stainless steel (type 347) has a characteristic film-forming potential at approximately 240 mv, noble to the normal hydrogen electrode, corresponding to the so-called Flade potential of carbon steel in the neighborhood of 500 mv.

Two specimens of carbon steel in the technetium inhibition experiments have been under observation for over a year. Even with 10 ppm of chloride ion present in one case, no corrosion has been observed. The solutions have remained water clear and the metal bright.

Data obtained from the study of decomposition rates of alkali amalgams indicate that the reaction is probably zero order instead of half order as commonly reported.

A potentiometric method for the determination of the concentration of lithium amalgam from hydrogen electrode measurements was developed. The method is accurate to within 1.0 - 2.0% over the concentration range from 0.8 to 0.1 M and appears capable of refinement to give considerably greater accuracy. (AEC Activity 5361)

THE ORACLE - The installation of the ORACLE was completed and a programmed test routine demonstrating the general condition of the computer was successfully executed. Noise tests on the electrostatic memory are currently in progress. Design work was begun on the installation of the magnetic tape unit. Development projects now in progress include a photographic curve plotter and an odd-even type check for the electrostatic memory and the magnetic tape unit. (AEC Activity 5511)

PROGRAM 6000 - BIOLOGY AND MEDICINE

BIOLOGY

Cytogenetics - In order to obtain evidence on the question as to whether the oxygen concentration at the time of irradiation with rays influences the frequency of chromosome breaks produced or whether it affects the process of rejoining of broken ends, male drosophilae which contained a ring X-chromosome were treated. Irradiations were made in air and nitrogen at dosage levels from 1000 to 4000 r. If the influence of oxygen is on the

breakage it would be expected from such a genetic system that the frequency of loss of the ring chromosome would be much greater in flies treated in high rather than low oxygen concentrations. Losses of the ring were equally frequent following irradiation at low dosages in air and in nitrogen and not significantly more frequent after high dosages in air. These results appear to be irreconcilable with the chromosome breakage hypothesis of oxygen action.

Low doses of radiation cause slowing of the rate of cell division of grasshopper neuroblasts by retarding the middle prophase stage of the mitotic cycle; the well formed chromosomes of this stage revert to the diffuse form of the previous interphase. It was shown that exposure of cells to hypertonic culture medium (which draws water from the cell) causes the chromosomes of middle prophase to shorten (advance) immediately to the form characteristic of late prophase. It was shown experimentally that subjecting the cells to the hypertonic medium right after irradiation reduces the rate of reversion of the middle prophase and therefore reduces radiation-induced mitotic inhibition.

Previous work has shown that H_2O_2 is not an effective mutagen for paramecia and thus cannot be used to explain the oxygen effect and other features of X-ray mutagenesis. This conclusion was further strengthened by measurements of the catalase content and permeability to H_2O_2 of paramecia and by a repetition of certain critical experiments in which cyanide was present to inactive catalase and insure good penetration by the H_2O_2.

A comparison of the behavior of gamete nuclei with incomplete chromosome sets derived by meiosis in haploids with that of gamete nuclei from heavily X-irradiated diploids suggests that the abnormal behavior of the latter is due to loss of parts of the chromosome set. Such losses must act autonomously on the nuclei which contain them since other nuclei in the same cells have complete sets of chromosomes.

The presence of B^{10} in the spores of neurospora was found to enhance the lethal effect of slow-neutron irradiation. Survival of cells produced on a boron-free medium was 80% after a 48-hour exposure to a flux of 1.2×10^9 n/cm^2-sec, whereas survival of cells from a medium containing 16 ppm of B^{10} was less than 3.7×10^{-5}. On the other hand, no difference in sensitivity was found between the cells containing natural nitrogen and those containing 65 at-% N^{15}. (AEC Activity 6400)

Mammalian Genetics - Very little information is available on the induction of any type of genetic effect in female mammals by ionizing radiation. In the work reported here the rate of induction of dominant lethals in mouse oocytes was measured by exposing females to 400 r of X rays and shortly after irradiation mating exposed and control females to unexposed males. The uterine contents of 76 females were examined for dead embryos and resorption sites 13-1/2 - 17-1/2 days after conception. At the same time, the number of corpora lutea in the ovaries of each of these females was

recorded. Survival of embryos in the experimental group was 71% of that in the controls. This is approximately equal to the dominant lethal rate obtained in earlier work with similarly irradiated males. (AEC Activity 6400)

Microbiology - A cyanide- and azide-resistant peroxidation of $DPNH_2$ has been demonstrated (Dolin, Arch. Biochem. 46, 483 (1953) in extracts of Streptococcus faecalis, an organism which contains no catalase or demonstrable cytochrome components. Additional properties of the purified peroxidase were investigated. The enzyme does not utilize $TPNH_2$ (reduced triphosphonucleotide). The K_m's for H_2O_2 and $DPNH_2$ (reduced diphosphonucleotide) are 2.4×10^{-4} and 5×10^{-6} M, respectively. Other oxidizing agents, including organic and inorganic sulfhydryl-oxidizing reagents or cytochrome-c, do not substitute for H_2O_2; however, there is present, possibly as a contaminant, an enzyme which oxidizes $DPNH_2$ with 2,6-dichlorophenolindophenol or ferricyanide, but not with methylene blue, brilliant cresyl blue, or oxygen. Typical substrates for hematin peroxidases, including reduced cytochrome-c, are not attacked. Incubation of the enzyme with $DPNH_2$ leads to an inactivation which is not reversible by dialysis or by incubation with known co-factors or crude Koch-safts. The purified enzyme contains FAD (flavine adenine nucleotide) and small amounts of lipoic acid; however, there is no conclusive evidence for the function of these compounds in the reaction. There is no inhibition by versene or 8-hydroxyquinoline or by exposure of the enzyme to strong ultraviolet light (365 mμ). In a search for other peroxidation systems, it was found that cytochrome-c catalyzed the peroxidation of $DPNH_2$; the latter reaction has the same pH optimum as the S. faecalis enzyme, however, it is heat stable, whereas the bacterial enzyme is extremely heat labile. $CPNH_2$ peroxidation is catalyzed to some extent by crystalline hemin, but not by ferric iron or by ferrous iron at a neutral pH. (AEC Activity 6400)

Radiation Protection - In a comparative study of the protective ability of β-mercaptoethylamine and mercaptoethanol, it was found that the former protects in relatively low concentrations, but comes to a relatively low plateau in protective ability. In contrast to this, mercaptoethanol requires higher concentrations and reaches its plateau at a considerably higher level of protection. There are many differences in the mechanism of protection by these compounds in spite of their close structural relation.

In an investigation of the material released by X-irradiated Escherichia coli cells which were subsequently incubated in a phosphate buffer, glucose solution at 37°C, it was determined that a relatively specific release of cellular constituents is brought about. There is no release of free amino acids or peptide material above that found excreted by unirradiated cells under similar treatment. It appears that nucleic acid fragments, from polynucleotides down to free bases, are released in markedly increased quantity by the exposed cells when fed a metabolite in the presence of phosphate buffer. The appearance of these nucleic acid fragments in the suspending fluid correlates with a breakdown of nucleic-acid-containing components of such cells (AEC Activity 6400)

Biochemistry - The barium and brucine salts of ribose-2-phosphate and ribose-3-phosphate were prepared by ion exchange for final characterization in gram amounts and in very pure form. The specific rotations of the brucine salts in pyridine-water solution are -27.5° and -35.0°C, respectively. The classic value of Levene and Harris of -33.3°C indicates that they had a mixture of the two isomers in the approximate ratio of 23:77, which is not far from the ratio of 30:70 in which these isomers are produced in nucleotide hydrolysis. Since the brucine salt of the ribose-3-phosphate is the less soluble of the pair, some such degree of enrichment is to be expected from their crystallization procedure.

The thermodynamic properties established for the complexes between α-chymotrypsin and its substrates indicate the possibility that carbon-carbon bonds, if sufficiently activated, and connecting groups of the necessary configurations (e.g., β-keto esters and β-keto nitriles) might be hydrolyzed by this enzyme. To test this hypothesis, ethyl-3-keto-5-phenyl valerate and ethyl 3-keto-5-(p-hydroxyphenyl) valerate were synthesized and subjected to enzymatic action. The first compound was hydrolyzed slowly, presumably to phenylpropionic acid and ethyl acetate. The second compound, being more nearly like a tyrosine derivative, was hydrolyzed rapidly at a zero-order rate for the first three hours to ethyl acetate and p-hydroxyphenylpropionic acid. This acid was isolated from a 70% hydrolyzed mixture, establishing the point of scission at the activated bond between the carbonyl and methylene ester carbons. Thus, a normal proteolytic enzyme can hydrolyze a sufficiently activated carbon-carbon bond. (AEC Activity 6400)

Biophysics - A silver target bombarded with deuterons to produce neutrons by the "drive-in" method produced a neutron yield only about one-tenth that expected from deuterated paraffin or heavy ice targets.

Studies of chromosome aberrations induced in tradescantia by ultraviolet irradiation were continued. Several recent experiments demonstrated that photorecovery following treatment with visible light does not occur in pollen which has been subjected to ultraviolet-produced chromosomal damage. The previous results showing no difference in the air/nitrogen breakage frequencies, as well as the time/intensity relation, have been confirmed. In addition, the "action spectrum" for chromosome breakage has been extended to an upper wave-length limit of 3600 A instead of the 3130 A cutoff reported previously. (AEC Activity 6400)

Pathology and Physiology - In a cooperative project with the Air Force, dosimetry studies, performed with the ORNL 86-Inch Cyclotron, resolved earlier discrepancies between dose measurements obtained with Victoreen chambers, Hurst-type neutron counters, fission detectors, and Failla-Rossi tissue-equivalent chambers. The energy spectrum of neutrons in the biological facility at the cyclotron was characterized experimentally and found to agree closely with that postulated theoretically. Improvements were also made in the dose-monitoring equipment at the cyclotron, including installation of a new proportional counter and scaler. The uptake of Fe^{59} by

erythrocytes in X-irradiated mice is being determined preparatory to obtaining the relative biological efficiency of fast neutrons for depression of erythropoiesis.

An approximate electric analog for circulatory mixing was established which involves a ring-connected set of unit-gain amplifiers. This analog may be of interest in the study of indicator dilution in large mammals. (AEC Activity 6400)

Enzyme Chemistry - Experiments designed to identify the early products containing C^{14} from incubation of bone marrow or chick embryo with C^{14}-formate are under way. Control, irradiated, and aminopterin-treated materials are being studied, Hypoxanthine, adenine, and guanine were identified in bone-marrow extracts and hypoxanthine was found in the acid soluble material from chick embryos. Serine accounts for approximately 45-55% of the activity in bone-marrow acid solubles but C^{14}-serine makes up only 10% of the activity in similar material from chick embryos. Aminopterin inhibits serine and deoxyribonucleic-acid thymine formation about equally, while low concentrations of this radiomimetic agent either enhance or fail to depress labeling in the purines of the ribonucleic acids.

In a continuation of the study of the production of the delayed light by green plants, it was shown that the substance responsible for the delayed light can be stored at liquid nitrogen temperatures. If a photosynthetically active green leaf is illuminated while being plunged into liquid nitrogen and then placed in the dark for several hours at liquid nitrogen temperatures, the delayed light effect is observed when the leaf is immersed in a water bath at 40°C. (AEC Activity 6400)

RADIOISOTOPE SALES AND COSTS

Type of Transaction	December	FY to Date
Domestic Sales	$ 83,325	$ 477,003
Foreign Sales	3,108	24,590
Project-Cash Sales	5,888	52,975
Project-Transfer Credits	77	575
Technical Cooperation Program Credits	686	5,234
Plant Credits	2,178	69,892
Civilian Defense Credits	---	390
Cancer Program Credits	18,860	108,728
*Miscellaneous Income	8	219
Total Income	$ 114,130	$ 709,606
**Income - No production costs incurred	1,583	16,968
Radioisotope Income	$ 112,547	$ 692,638
Radioisotope Costs	$ 82,574	$ 552,542
Radioisotope Shipments	1,026	6,005

*Miscellaneous income - D_2O, Freight, Missing parts, TCP.

**Income from H^3, He^3, B^{10}, and miscellaneous items in excess of handling cost.

GROSS OPERATING COSTS

	Cost for December	FY 1954 Cost to Date
Programmatic Operating Cost - Net	$ 2,367,580	$14,291,718
Plant and Equipment Cost	277,073	1,896,003
Construction Program "H"	3,870	22,229
Work for Other Parties - Transfer	36,434	262,626
Inventory Changes	111	82,595*
Reimbursable Work for Other Parties	135,200	768,328
Deferred Charges	923	5,589
Total Laboratory Cost - Net	$2,821,191	$17,163,898
Estimated Cost for Next Month - Net	$2,900,000	$20,063,898

*Credit

[redacted]

PERSONNEL SUMMARY

	Number of Employees January, 1954	New Hires January	Terminations January
Administration	64	0	0
Operations*	115	0	1
Engineering, Shops and Mechanical	735	11	4
Laboratory and Research	1665	15	11
Protection	160	0	3
Service	391	1	12
Total	3130	27	31

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

A total of 646 Laboratory personnel are located in the Y-12 Area.

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Previous reports in this series:

January 1953	ORNL-1486
February 1953	ORNL-1516
March 1953	ORNL-1529
April 1953	ORNL-1545
May 1953	ORNL-1562
June 1953	ORNL-1576
July 1953	ORNL-1600
August 1953	ORNL-1612
September 1953	ORNL-1622
October 1953	ORNL-1643
November 1953	ORNL-1651
December 1953	ORNL-1665

[redacted]