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ADVANCED RESEARCH AND DEVELOPMENT REPORT

ORNL 903  
Series A  
Progress Report  
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1954

OAK RIDGE NATIONAL LABORATORY  
STATUS AND PROGRESS REPORT  
NOVEMBER, 1950

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PROGRAM 2000 - SOURCE AND FISSIONABLE MATERIALS

TBP Process for Waste Metal Recovery

Construction of the ORNL Metal Recovery Building was completed during this period. Purchase requisitions for the majority of the equipment have now been placed; however, it is expected that slow deliveries on equipment will delay the proposed startup date, March, 1951. Adaptation of equipment that was obtained locally for installation in the building has been started.

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PROGRAM 3000 - WEAPONS

RaLa Development

Chemical and equipment studies are in progress to improve the filtration of the barium sulfate precipitate which in the plant test proved to be unsatisfactory. In the MTR RaLa development program, work was continued on the sulfuric acid flowsheet for the separation of the radioactive barium from the aluminum and uranium.

Special Separations U 236

During the past month a peak U 236 purity of 44%, representing a 236:235 enhancement of 69, has been obtained. The last run of the first stage separation is now under way. Approximately 27 grams of intermediate level (31%) U 236 will than be available for further refinement in the second stage of calutron operation.

Bismuth Separation

Two calutron runs for the separation of isotopes in irradiated bismuth have been completed for the Radiation Laboratory at Berkeley. Collections have been made for masses 208, 209, and 210. Although assay data are not yet available, appreciable enhancements are expected.

MTA Target

A pulsed proton beam is being used to apply a heat flux of 1250 watts per sq in to a uranium target. a 0.05% copper sulphate solution is circulated through the tubular target. The first target ruptured after 21 hours or  $4 \times 10^6$  thermal cycles.

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PROGRAM 4000 - REACTOR DEVELOPMENT

MATERIALS TESTING REACTOR

Mock-up Conversion

The full scale MTR mock-up which has been used for hydraulic tests and critical experiments is now being converted to a low-power reactor for continuous operation. Current effort is directed toward revising the mock-up control system to make it resemble the MTR closely enough for training Arco reactor operating personnel.

In order for the mock-up to duplicate the MTR in all stages of start-up, it is necessary to operate at a power level of several hundred kilowatts. Operation at this level is fairly easy to provide for in the present mock-up by putting concrete blocks in the area from which graphite was removed following completion of the critical experiments.

The conversion is now about 75% complete, and preliminary start-up operations are expected to be undertaken before January 1.

MTR Test and Development

MTR flange mock-up tests are about half completed. The aluminum to stainless steel flange has been tested and found to be satisfactory; testing of aluminum-aluminum flange awaits delivery of stainless steel bolts of the large size required.

25 Process Design

The Idaho Chemical Process area includes four major buildings; the Process Building, the SF Storage Building, the Utility Building, and the Waste Process Building. Concrete has now been poured for the SF Building; future progress on this building is awaiting delivery of the steel. The need for a railroad spur to this building has been eliminated by a change in policy concerning the shipment of the material into the site. For the Process Building, the concrete drain ditch has been poured and the concrete for the deep cells required for waste storage is scheduled to be poured during the coming week. Design specifications for the equipment to be installed in the Utilities Building have been transmitted to

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Foster Wheeler, and the design of this building is under way. The layout of the Waste Process Building has also been transmitted to Foster Wheeler, and its design started.

Design sketches of all vessels and heat exchangers for process equipment have been completed and bids on the sketches that were previously submitted to fabricators have been received. On the basis of these bids, tank fabricators will be chosen during the coming month and the required information supplied them to allow final design drawings on these pieces of equipment to be made. Mill orders have been placed for 750 tons of stainless steel that will be required for vessels and piping. In the Process Building, equipment layouts for the feed makeup area and the dissolver cells have been completed.

The general layout of the liquid waste disposal system, in which there will be three evaporators and seven collection and storage tanks, has been defined. One evaporator will be used for the first cycle waste, a second evaporator for the second and third cycle waste, and the third evaporator will be used to evaporate condensate from the first two evaporators along with radiochemical waste resulting from the remainder of the process. There will be five collection tanks. Three 40,000 gallon capacity tanks and two 20,000 gallon capacity tanks will be used for holding up solutions to be fed to the chemical waste evaporator. Concentrates from the waste evaporators will be sent to two 300,000 gallon stainless steel tanks for permanent storage.

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HOMOGENEOUS REACTOR EXPERIMENT

Experimental and Design Engineering for HRE

All the steel for the HRE Building was delivered during November. The final flowsheet for the HRE has been completed, although minor revisions are being made as needed. Overall design is about 90% completed.

The forged steel pressure vessel has been completed but shipment is delayed because the vendor has not received the high-tensile bolts for use in making hydrostatic tests. Delivery of the pressure vessel to ORNL is expected before January 1.

Phase Relationships of Fuel Media

The solubility of thorium nitrate has been found to be such that one gram of thorium (as metal) per cubic centimeter will be present in the saturated solution at 118°C. (This "saturated solution" is actually crystalline thorium nitrate hexahydrate at room temperatures.) Saturated thorium nitrate solutions decompose at temperatures about 125°C in open systems; however, in a closed system, thorium nitrate appears to reach equilibrium with the vapor phase to keep the salt in solution at temperatures up to about 225°C. This indicates that from the standpoint of concentration and temperature stability, thorium nitrate (with N<sup>15</sup>) might be used in a breeder blanket. Radiation effects have not been studied.

Chemical Processing of Homogeneous Reactor Fuel

The present objective of the laboratory development program is to develop a Homogeneous Reactor chemical process that will require no change in composition of the fuel. Gaseous fission products, xenon and krypton, will probably be separated in the reactor cycle itself. For the chemical processing cycle of the reactor, four methods are now being considered: filtration, adsorption, electrodeposition, and ion exchange. The range of effectiveness of each of these operations is being evaluated.

The Reaction of Hydrogen with Oxygen

Recombination of hydrogen and oxygen at high pressures (700 to 1000 psia) has been achieved using Product 43 as the catalyst.

Platinized alumina has been shown to be nearly as effective as

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Product 43 for the low pressure recombination. The high temperature stability of this material offers a marked advantage over Product 43.

Continued study of poisoning of Product 43 with iodine seems to indicate that such poisoning will not seriously affect the operation of catalytic recombiners.

In-pile tests of Product 43 are scheduled for the week of December 6.

Pilot Tests of Primary Recombiner

Modification of the burner type recombiner to simulate the pertinent sections of the reactor mockup is virtually complete and calibration of the instruments is in progress.

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AIRCRAFT NUCLEAR PROPULSION PROJECT

ANP Central Design

Primary attention has been devoted to developing designs for a liquid fuel reactor using molten fluorides. Study of the controllability has received major emphasis along with the development of a design which would be feasible from the standpoint of fabrication. Work on solid fuel reactors has been slowed somewhat because of the difficulties imposed by the positive or very small negative temperature coefficient calculated for this type of reactor.

ANP Critical Experiments

The uranium allocated for ANP critical experiments is now on hand in Y-12 and is being cast; following this it will be delivered to X-10 to be fabricated into fuel pieces. Delivery of fuel pieces is expected to start by the first of the year. The table on which the halves of the assembly are stacked and the aluminum matrix have been assembled. Both the actuating and monitoring controls have been installed. Overall instrumentation is about 80% complete.

Induced Activities in ANP Pile Coolant

The assembly for circulation of liquid metal coolant during in-pile tests plugged during final testing outside the pile and is now being repaired.

Mechanical Design and Experimental Engineering

The liquid metal centrifugal pump failed during preliminary testing and is now being repaired. As many as 17 thermal convection loops have been in operation with sodium at 1200 - 1500°F. Currently 10 loops are in operation. Visual observation indicates that to date almost all failures have occurred in the welds. The first figure 8 loop is being assembled; others are under construction.

Heat Transfer and Liquid Metals

Equipment for the measurement of local heat transfer coefficients (entrance effects) with sodium is now essentially complete. Current experimental work is devoted to measuring the heat capacity of NaOH.

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Construction of equipment for measuring the heat transfer to boiling potassium will be started in December. Equipment for measuring the viscosity of high temperature liquids is under construction. Bronze pumps now on hand are to be rebuilt into prototypes of the canned rotor, hydraulic bearings, completely sealed liquid metal pump to deliver 100 psi.

Liquid Metals Research

The corrosion program has centered its attention on changing from a three-component system to a two-component system to make the testing conditions more valid. At present stainless steel tubing is being tested for corrosion attack with the following liquid metals; lead, lithium and sodium. Results from these tests are awaiting completion of the metallographic examinations.

The powder metallurgy laboratory should be completed by the end of the year. Basic compatibility tests of fuel element components have been started with existing equipment.

The welding laboratory is now being designed. It will be located in the rolling mill building and is expected to be completed by about March 1.

Radiation Damage

Preliminary data obtained from the creep of a 316 stainless steel cantilever beam indicate that there is at least a transient increase in creep rate when the already creeping beam is subjected to a neutron flux. The test was carried out at 1050 degrees Fahrenheit under a maximum fiber stress of 4000 pounds per square inch. The irradiation took place in the ORNL reactor where the fast flux (greater than 1 mev) is approximately  $4 \times 10^{10}$  neutrons/cm<sup>2</sup>/sec.

Shielding

The Bulk Shielding Facility has suffered setbacks in that the overhead crane has not been delivered and also a large section of the main floor was found to be faulty and must be replaced.

The liquid metal duct experiment progresses but slowly because of the large number of measurements which must be made by the foil technique.

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It now appears that the full length duct with liquid metal core in place cannot be measured with the present technique due to the low intensity of the thermal column source. Either the detection technique must be exchanged for a more sensitive one, e.g. large  $\text{BF}_3$  counters, or the experiment must be completed in one of the other facilities.

In the lid tank a considerable number of attenuation measurements have been made on neutrons through pure  $\text{B}_4\text{C}$ . An interesting comparison of the effectiveness of  $\text{B}_4\text{C}$  and water has become apparent during these experiments. Although  $\text{B}_4\text{C}$  has a high total cross section, the large fraction of this due to scattering makes the buildup factor in this material appreciably greater than that in water. A small fraction of water introduced into the  $\text{B}_4\text{C}$  is much more effective in the region next to the source where neutron collimation has not yet taken place and buildup due to multiple scatterings is therefore serious. The same amount of water at the rear of the sample did not increase the attenuation nearly as much. This is the first clear-cut experiment demonstrating the effect of buildup on fast neutron attenuation.

A mathematical method has been developed for recognizing an optimum arrangement of different materials in a unit shield, using easily obtained experimental measurements. Thus, for example, the lead and water system will have one and only one optimum distribution of lead slabs in the water, for a given aircraft shield design, and this is recognized by a simple criterion. This greatly reduces the total number of experiments required for shield research.

#### Liquid Fuel System for High Temperature Reactor

Low Melting Fluoride System. The demonstrated corrosive properties of fluoride melts containing lithium fluoride has shifted the emphasis to study of the  $\text{NaF}-\text{BeF}_2-\text{UF}_4$  system. Preliminary observations have shown the existence of a ternary eutectic in this system melting at about  $340^\circ\text{C}$ ; this eutectic unfortunately contains less than 0.5 mole percent of  $\text{UF}_4$ . It appears at present that as much as 15 mole percent of  $\text{UF}_4$  may be dissolved at temperature below  $500^\circ\text{C}$ .

Suspensions of Uranium Compounds in NaOH. A considerable amount of data on stability of the suspensions has been accumulated during the past month. It is apparent that suspensions prepared from  $\text{UO}_3$  are superior to those prepared from sodium diuranate and uranyl phosphate. The settling rate of all these suspensions is increased by long (24 hours) digestion at  $700^\circ\text{C}$  prior to testing. It still seems likely, however,

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that little agitation would be required to maintain homogeneity in the system.

ANP Corrosion Tests

A preliminary series of tests measuring the rate of corrosion of several metals and ceramics in lithium fluoride-uranium fluoride eutectic was completed early this month. A second series of tests is now in progress, with the 400 series stainless steels predominant. The series consists of three tests; the lithium fluoride-uranium fluoride eutectic will be employed in the first and third tests, while the sodium-uranium fluoride eutectic will be employed in the second.

ANP Materials Tests

An experiment has been under way for two weeks to check the performance of Type 316 stainless steel flanges and chrome-molybdenum bolts at 1500°F to 1800°F and at 50 pound per square inch internal pressure.

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GENERAL REACTOR DEVELOPMENT

Radioactive Waste Treatment

The Cottrell electrostatic precipitator is now in operation on the off-gas system from the iodine production unit. In preliminary tests, the removal efficiency of this unit has been about 90%. A study of the unit will be continued to improve its efficiency.

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PROGRAM 5000 - PHYSICAL RESEARCH

PRODUCTION OF RADIOISOTOPES

Radioisotope Production, Development and Operations

The total accumulated KWH for pile operation during November was 1,970,603 averaging 3096 KW per operating hour. Pile down time was 11.6% as compared with 12.28% during October (for both months down time was larger than normal because of the installation of new fans). One ruptured slug was located and discharged without difficulty.

Both new pile cooling air fans have now been installed and found to pull about 10% more air through the pile than the old ones did. Because of this the normal pile operating level has been raised to approximately 3800 KW.

The total number of radioisotope shipments during November was 661. Although the iodine 131 processing equipment was shut down for repairs until November 8, all iodine 131 commitments for the month of November were met.

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STABLE ISOTOPE SEPARATIONS

Calutron Operation and Process Development

Primary attention was given to development of a filament changer, platinum exploratory operations, evaluation of rare gas exploratories, and collections of potassium and vanadium. The filament changer was tested in laboratory-scale equipment and MgO proved successful as an insulating material. Recovery results obtained from stainless steel targets which had been bombarded with neon ions indicated qualitatively that neon was retained. Platinum exploratory runs yielded a peak ion current of 1.5 ma; chlorine arc support gas was superior to nitrogen.

Potassium collections in the alpha channels yielded 85 grams of isotopes, while 6.6 grams of vanadium were collected in the Beta channels. Test runs were under way to evaluate a 4-arc alpha II unit for potassium operations.

Isotope Chemistry

Sample Summary

Samples, Chemical Refinement Completed	19
Samples being refined, November 30	26
Samples awaiting refinement, November 30	17
Samples submitted for mass analysis	12
Mass analyses reported by Assay Laboratory	9
Samples being prepared for Assay Laboratory	57
Isotope samples shipped	57

Potassium and vanadium were supplied for calutron operations and the isotopes of carbon, neodymium, indium, chromium, sulfur, hafnium, calcium, rubidium and potassium were undergoing chemical refinement.

In the separation of rare earths for charge material, it has been found that mandelic acid precipitates the rare earths with sufficient selectivity to make good separations. Also, some separation of rare earths has been obtained by selective reaction of ammonium chloride with hot rare earth oxides.

The reduction of lithium salts to the metal is receiving intensive investigation.

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PHYSICS

High Voltage Program

Manufacturer's tests of the 5 MV electrostatic generator have shown that it delivers a steady beam of 60 microamperes (unanalyzed) at well over five million volts. This means that it is the best Van de Graaff machine which has so far been built. Delivery of components will start in December and assembly at the Y-12 site will continue through January.

Work on the  $B^{11} (p \gamma) C^{12}$  reaction continues with the angular distributions of the 12 Mev and 4 Mev gamma rays now under study.

Conversion of the 2 MV electron Van de Graaff machine to a positive ion accelerator is now actively in progress.

Neutron Diffraction

The degree of polarization in the (220) reflection from a magnetized  $Fe_3O_4$  crystal has been determined and found to be 100% within the experimental uncertainty of perhaps 5%. This appears to be a useful method for producing polarized neutron beams of high intensity, since approximately  $10^7$  neutrons per second can be obtained in such a reflection with collimated pile radiation incident upon the crystal.

Physical Electronics

The single-channel pulse analyzer has been redesigned to require less space and to have only a very small variation in channel width as the pulse height is changed.

Low Temperatures and Nuclear Alignment

Current effort is devoted to establishing ideal conditions for the polarization of nuclei and for polarization of neutrons to maximize the effects of nuclear polarization.

Fundamental Solids Physics

An ion source is being developed for producing ions of atomic weight greater than 4 for use with the Van de Graaff and Cockroft-Walton accelerators. In recent tests, steady beam strengths as high as 800 microamperes (unanalyzed) of sodium ions have been obtained. It is planned to use these ions to study rate of energy loss of the ions in gases and solids.

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The 22" Test Cyclotron

A beam deflection system for the 22" test cyclotron has been improved to permit deflecting 20% of a 400 microampere beam.

The 86" Cyclotron

Initial operation of the 86" proton cyclotron was achieved on November 11. Currents to the target of over 100 ma have been measured at energies up to 7 Mev. The pattern of radioactivity on the copper target indicates a well-focussed beam. The dee-to-dee potential was held to 90 kv and no attempt was made to increase the beam current, the immediate problem being stable operation of the grid bias system.

Preliminary design calculations have been completed for several types of production targets; stationary, grazing incidence, rotating disc, liquid metal, and slurry.

Isotope Physics and Spectroscopy

Atomic Spectroscopy

The classification of Sm II (doubly ionized samarium) is being completely checked using new wavelength data and numerous isotope shift data which have been obtained recently. Wavelength data on more than 5000 spectrum lines are included with isotope shifts on about 400 of the strongest lines. It is expected that the analysis of the spectra of Pu will be aided considerably by more complete information on those of Sm. Both atoms are fifth in their respective rare earth series and are expected to have very similar electronic structures.

Nuclear Resonance

A visit was made to the Nuclear Resonance Laboratory at Harvard University and some new improvements in the circuit for the lithium spectrometer were obtained from Dr. R. V. Pound.

Miscellaneous

A calculation of the natural abundance of the Sm 146 nuclide gives a value of  $\leq 0.00002\%$  which compared with  $\leq 0.002\%$  by Ingram, Hess, and Hayden and  $\leq 0.001\%$  by Love and Bell.

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Source, Filament, and Vacuum Laboratory

Principal emphasis was placed on the analyses of four samples of mixtures of the trichlorides of Nd, Sm, and Gd. Mass spectrometer analyses of nickel carbonyl indicate that it is a promising source of  $Ni^+$  ions for the calutron.

A paper entitled, "A Versatile All-Metal  $60^\circ$  Mass Spectrometer" was presented before the American Physical Society meeting in Chicago.

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METALLURGY

Materials Laboratory

Zirconium Reduction

Zirconium corrosion tests are now in progress on the 4 per cent tin-zirconium alloy.

Hafnium Reduction

Thirteen reductions of hafnium tetrafluoride to hafnium metal, totalling about 10 pounds, have been made, part of which will be converted to crystal bar hafnium metal. The latter will be used for physical property research.

Thorium Research

A limited amount of thorium of the highest available purity, iodide thorium, has been obtained for determination of properties. The iodide thorium has been melted in a vacuum arc furnace and rolled into bar stock for making tensile specimens. The tensile properties of this thorium are to be compared with previously determined properties of Ames produced thorium.

It has been found that Ames produced thorium shows a yield point similar to that found in low carbon steels. Apparently, this yield point phenomenon is removed by small additions of titanium to the thorium.

A torsion machine has been constructed and used for torsion testing thorium bars. The values of shear modulus obtained from the torsion test compare favorably with that calculated from tensile test data.

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CHEMISTRY

Chemical Physics

A new 810 Kev beta group and a 720 Kev gamma have been found in low intensity in 8-day iodine 131 decay.

Instrumentation for Nuclear Chemistry

A portable gamma scintillation counter was designed and built for measurement applications which require a high order of sensitivity. The circuit is composed of an electronic high voltage supply, an amplifier and a rate meter. Stability and reasonable speed were stressed at the expense of weight; however, the non-engineered model weighs under 15 pounds. The circuit is capable of handling  $2 \times 10^5$  counts per minute with low resolution losses. The overall stability of the circuit is of the order of 8% over the battery life while measuring a disadvantageous pulse distribution.

With a thallium activated NaI crystal, the lower energy detection limit is of the order of 10-15 Kv. The instrument has been used successfully in finding 25 gms. of U 235 deliberately hidden in a location where the background is of the order of 5000 counts per minute. The 25 gram source was easily detectable from one foot away even through 1/2 inch of steel; without shielding it read 30% over background at 10 feet.

Purex Process

The pilot plant phase of the Purex Process Development has started in the 1.5 inch diameter column in 706-HB. In this work, decontamination factors of  $10^3$  to  $10^4$  for uranium and plutonium through the first extraction were obtained at full Clinton activity level. The startup of the larger pilot plant equipment in Building 205 is scheduled for the coming month. Major work in the laboratories is now concerned with the two-cycle flowsheet. In the Unit Operations Section, development problems associated with the fumeless dissolver and with recovery of the nitric acid from the waste are being considered.

Chemistry of Uranium Raw Materials

A report is being issued, Y-663, covering studies of the precipitation of yellow cake, a sodium uranyl vanadate resembling carnotite,

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from carbonate leach liquors, and including tests of several methods of precipitating vanadium from these solutions.

New Projects which have been started during the month include (1) studies of the reactions incurred during a high temperature roast of carnotite ores with various sodium salts, and (2) studies of complex ion formation between  $UO_2^{++}$  and  $F^-$  ions.

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PROGRAM 6000 - BIOLOGY AND MEDICINE

HEALTH PHYSICS

Radioactive Waste Disposal Research and Development

Preliminary data on a small water decontamination unit have indicated that water contaminated with a mixture of fission products to the extent of one microcurie per cubic centimeter can be decontaminated to  $10^{-4}$  microcuries per cc in a single pass through the unit. This unit was developed principally for use in the Civilian Defense Program as an emergency treatment unit for contaminated sources of drinking water.

The starting solution on which the unit was tested was selected as being typical of the maximum contamination to be expected in a water supply after a bomb burst. The resultant treated water was well below the level of mixed fission product contamination believed to be permissible for temporary emergency use.

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BIOLOGY

Cytogenetics

Additional compounds have been found which protect exceedingly well against radiation damage. Two of these are several times as effective as cystiene, the compound which until now has been considered the best protector against radiation damage.

Physiology and Pathology

The work on the effects of X-rays and neutrons on cataract formation in mice has given significant results which demonstrate that exposures to even small intensities of slow neutrons will give a appreciably high percentage of cataracts appearing rather late.

Biophysics and Biochemistry

The possibility of studying at ORNL the animals which survive the test of Operations Greenhouse is being discussed. This is a large scale experiment which involves the study of cataract formation and appearance of tumors during the entire life span of exposed and control animals.

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PROGRAM 1000 - RESEARCH FACILITIES

Research Laboratory Building

Foundation walls are being erected for the basement section; about 30% of the concrete pouring for the foundation has been completed.

Isotope Research and Semi-Works Building

About 40% of the foundation and grade-beam work has been completed.

Physics of Solids Building

This building has been completed and will be turned to the Laboratory for use early in December.

Instrument Laboratory Building

The completion date has been extended to December 15, 1950, and is expected to be met. The storeroom has been turned over to the Laboratory and is now being stocked.

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PERSONNEL SUMMARY

	<u>Number of Employees</u> <u>November 30, 1950</u>	<u>New Hires</u> <u>November</u>	<u>Terminations</u> <u>November</u>
Administration	75	1	3
Operations*	109	4	2
Engineering, Shops, and Mechanical	887	6	7
Laboratory and Research	1121	4	5
Protection	167	1	1
Service	344	9	2
TOTAL	2703	25	20

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

RADIOISOTOPE SALES

<u>Sales</u>	<u>September, 1950</u>	<u>October, 1950</u>
Transfer within AEC	\$ 77.00	365.10
Off Project	33,539.43	40,369.19
Foreign	1,284.20	581.85
Cancer Program (Free)	26,902.14	29,122.96
Technical Cooperation Plan	3,812.81	-0-
Total Sales and Transfers to Date	1,287,454.79	1,333,605.43
Total Cancer Program (Free) to Date	594,124.72	623,247.68
Technical Cooperation Plan (Shipments to Date)	6,739.26	6,739.26

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GROSS OPERATING COSTS

(Including X-10 and Y-12)

(a) Actual Cost for Oct., 1950	1,852,809	
Construction - Program "H"	95,628	
	<hr/>	
Total Operating & Constr. Costs		1,948,437
(b) Estimated Operating Costs for Nov., 1950		1,925,000
(c) Actual Accumulative FY 1951 Operating Cost through Oct., 1950	6,952,683	
Actual Accumulative FY 1951 Constr. Cost through Oct., 1950	227,494	
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Total Accumulative FY 1951 Constr. & Operating Cost through Nov., 1950		7,180,177
(d) Estimated Operating Cost FY 1951 through November, 1950		9,105,177