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RESEARCH AND DEVELOPMENT

PROGRESS REPORT

FOR THE MONTH OF MARCH 1948

OAK RIDGE NATIONAL LABORATORY

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DIVISION OF
CARBIDE AND CARBON CHEMICALS CORPORATION
OAK RIDGE, TENNESSEE

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BIOLOGY DIVISION

This was a very busy month for the Biology Division since very little time was available to make the necessary arrangements for the Information Meeting in Biology and Medicine. 110 invited persons (out-of-town) attended including 5 Atomic Energy Commission Representatives. Attendance at the meeting on March 25 was between 250 and 300 persons; on March 26 and 27, 200 persons.

Many highly complimentary letters about the success of the Meeting have been received. The general feeling is that such open meetings help very much to build up the scientific reputation of this laboratory.

The papers presented at the open meetings were recorded and are now in the process of being transcribed. It is hoped to publish them in the form of a report.

Radiobiochemistry Section

The radiobiochemistry laboratories, although not completely in operation, have most of the basic facilities installed, and the tempo of the laboratory work is beginning to pick up. Several standard uranium sources have been prepared and calibrated for absolute beta measurements against UX and Ra E. The work on Be analysis is progressing satisfactorily. Separation of Be and Al is currently under investigation. The administration of P32 to plants has progressed to the point where a critical evaluation of the results is in order. The technique for handling P32 plaques has been improved.

Work on the erythrocyte project has been retarded by many delays in establishing necessary facilities in the new laboratory and by the great confusion arising from construction activities both in the laboratory and on the floor above. In spite of conditions which have made research work nearly impossible, investigations of coating agents for glass vessels to be used in blood studies has continued, and a preliminary report describing the gold radiation source has been completed.

Cytogenetics

Work has continued on the induction of micronuclear mutation by beta radiation. Evidence was obtained that the frequency of mutations induced is essentially independent of the time over which the dose was given. This taken together with the non-linear relation to dose and with evidence from further breeding experiments has suggested that the observed reduced vigor after autogamy results from the combined action of several mutant genes. A preliminary experiment has suggested that it will be possible to investigate the induction of gene mutations with ultraviolet radiation. Doses which induce mutation in low frequency have a considerable immediate effect on the animals in contrast with beta radiation which has little if any immediate effect in the doses used. Further improvements have been made in the technique of inactivation of kappa by ultraviolet radiation and further information on dose relations has been obtained.

The study of the low intensity effect of gamma rays on the grasshopper neuroblast is in progress. The standard errors for the 1/4 roentgen series was completed this month. Plans for the 1/16 r group are in progress. The 0.25 Victoreen chamber has been calibrated and a holder is now being built for exposing with this chamber.

The comparative lethal and mutagenic effects of beta and gamma radiation are still being studied. An attempt is being made to establish dosimetry in the thin walled aluminum test tubes which have been used in the preliminary experiments. Dry spores are exposed using several different types of geometry, the spores acting as a biological dosimeter. The most satisfactory geometry is probably obtainable by exposing the spores on the outside of a polystyrene sphere in the center of a cubicle source. This method is now being used. Once the dosimetry is determined, large numbers of beta ray exposures can be made under strictly sterile conditions in the thin walled tubes. Gamma ray exposures await the completion of the new irradiation chamber which should be ready for operation very soon. A system for exposure of dry-spores to different wave-length X-rays as being worked out and the first experiments will be started shortly. The intensity of radiation will be kept constant in all of the experiments with the various radiations.

As was previously reported a new facility for exposure of biological material to a pure flux of thermal neutrons was investigated. A site in the thermal column atop the Clinton reactor was studied and the conditions appear desirable enough to justify the construction of an irradiation chamber, which will accommodate various types of biological material up to the size of a small rabbit. The following figures based on recent measurements show the characteristics of the new location as compared with those of the animal tunnel, where all previous exposures to slow neutrons have been made.

	<u>New Site</u>	<u>Tunnel</u>
Slow neutron flux - - - - -	2×10^9	2×10^8
Cadmium ratio - - - - -	10^2	10^5
Gamma Ray contamination - - - - -	30 r/hour	-5 r/hour

Mouse Genetics Experiment

Further work has been done on the technique of artificial insemination. Most of the equipment has been obtained and experiments are proceeding as rapidly as the multiplication of mice will permit. One of the animal rooms has been set up with inverted night and day lighting in order to control time of ovulation. Various improvements have been made in the animal colony, including the design and construction of cages. Pilot experiments on the treatment and sterilization of mouse boxes have given results that will be valuable in the planning of large scale program. In addition to continuing the genetic work reported earlier, the systematic development of stocks for linkage tests has now begun.

Physiology and Pharmacology

At the suggestion of Dr. Shields Warren, a short-term investigation of the actions of rutin upon the irradiated rat has been undertaken. A rutin dosage of (0.5% at pH 8 - 8.3) 5 mgm was administered subcutaneously, daily. The rats were irradiated with 250 KV X-rays, with no filtration, H.V.L. of 0.4 mm. cu. The exposures were both above and below the L.D. 50% (about 600-650 r). The experiments completed thus far are essentially negative. These include the acute L. D. 50%, the weight curve for 10 days following sublethal exposure, the appearance of hemorrhage in the lymphnodes and the G. I. Tract, and the clotting and prothrombin times. In vitro, rutin has no significant effect upon the blood of normal or irradiated rats, or of the normal man, as measured by clotting, recalcification, and prothrombin times; nor can it antagonize heparin. Work still under way indicates that the bleeding time and the platelet count of the irradiated animal are probably not affected. On the other hand, the incomplete "chronic effect" experiment indicates that rutin may have a beneficial effect. If positive, this result would have to be repeated. No results are yet available on the cutaneous response of the irradiated rat tail (2400 r).

Microbiology

Experiments were conducted with a dried cell preparation of Propionibacterium pentosaceum. In the presence of A.M.P., glycerol, pyruvate and carbonyl-labelled acetate, no labelled propionate appeared. However, the fermentation did yield normal amounts of unlabelled propionate. In view of the previous experiments in which C^{14} from $NaHC^{14}O_3$ is readily incorporated into propionate, it is possible that pyruvate may not be the common intermediate compound which gives rise to propionate, acetate, and CO_2 . This possibility is at present being tested under a variety of conditions and (simultaneously) upon whole non-proliferating cells. 280 microcuries of carbonyl-labelled acetate were prepared. The new gas monitor (flow type ionization chamber coupled to recording potentiometer) was exceedingly helpful in following the course and extent of the carbonation of Grignard reagent. After purification a hydrogen electrode microtitration of 0.3 mg of the synthesized acetate yielded the expected curve and pK value. Part of this acetate is being used for current metabolic experiments, and a portion will be used for the synthesis of carbonyl-labelled pyruvate, needed for the next steps in the propionic acid fermentation studies.

Investigations on the ability of external beta radiation to include biochemical and phage resistant mutations in the "B" strain of Escherichia coli have been continued.



CHEMISTRY DIVISION

In the process for recovering uranium from enriched reactors, the laboratory work is nearing completion with only relatively minor points to be covered. An adequate process seems to be assured and the work in the Chemistry Division is tapering off.

The special U236 sample is in the process of decontamination in the U233 pilot plant.

The preparations for the Xenon experiment seem to be well in hand, although the actual installation of the equipment for the large scale processing is proving to be slow, principally because of difficulties in installing equipment in the limited space in the concrete cell. Preliminary calibration of the assay equipment indicates that the assay can be made with a probable error of less than 5%. Work is now in progress on a method for isolating the relatively large amount of Cs¹³⁵ which will be formed as a daughter in the Xenon experiment.

One hundred and four millicuries of purified barium carbonate containing C¹⁴ has been prepared to use as a standard both for this laboratory and for distribution. It is planned to determine its activity by means of the beta calorimeter already in use.

The cubic phase of beryllium oxide previously described has been found in other samples of beryllium oxide briquettes. Experiments are being continued to determine the conditions of formation of this new phase.

The gas phase fission cloud chamber is now in operation at the reactor, and conditions have been obtained that give over two fission tracks per picture. Statistical studies of the fission process with this instrument should begin shortly.

It was previously noted that concentrated sulphuric acid is rather resistant to reactor radiation. During this month it has been found that the addition of UO₂SO₄ to the acid (5.0 g. U/liter) causes an increase in the gas evolution on irradiation. This gas contains a large excess of oxygen indicating that either the uranium or the sulphate is reduced under these conditions.

PHYSICS DIVISION

Since the quarterly report, which summarizes the current status of all work, has recently appeared, there are rather few additional new developments in the Physics Division which need to be reported at this time. Among the items of interest are the following:

I. High Flux Reactor. The heat production rate in the reflector of a slab reactor surrounded by Be and graphite has been measured by means of ionization chambers placed in the reflector. The results with CO₂ filled and He filled chambers agree well, and indicate that the heat rate at the graphite-Be interface when the reactor runs at 30 MW is about .7 watts/cm³. This rate is about 35% higher than the value originally computed. The effect of hydrogen in the Be reflector has been measured; it is found that a 5 mm layer of lucite at the Be-pile interface and 2% by volume lucite beyond is to reduce the reactivity by about 2 $\frac{1}{2}$ % in $\frac{k}{k}$.

The full scale servo system has been put together and is undergoing frequency response tests.

II. Reactor Physics. The low cross section of Zr has been definitely verified: three independent runs on the oscillator give an average thermal capture cross section (corrected for B impurity) of about 0.35 b.

A 100 neutron Monte Carlo computation of the neutron distribution in H₂O from a plane high energy source has been completed. The distribution, in general, looks reasonable although, because of the rather small sample, there is an asymmetry between the distribution to the right and left of a plane source.

III. Neutron Physics

Studies of order-disorder in Fe-Co alloys have been made by means of neutron diffraction. Extra lines attributable to a super lattice show up in the neutron powder patterns which do not appear at all in X-ray patterns because the X-ray scattering power of Fe and Co are so nearly alike.

IV. Solid State Physics

Measurements have begun on U-Al alloy material which has been irradiated at Hanford for 1 year: this corresponds to about 30 days in the high flux reactor and represents a destruction by fission of every third U atom. The material for the first time appears to be very much more brittle than originally. However, there are still no dimensional changes nor deformation of the slug surface.

RADIOISOTOPES

The following table indicates the number of shipments for February and March, 1948, and a total-to-date figure since August, 1946, the start of the Isotope Distribution Program:

	February 1948	March 1948	TOTAL August, 1946, to March 1948, Inc.
Separated Material			
706 D Area	152	221	2,090
Unseparated Material			
100 Area	51	78	850
TOTAL	203	299	2,940

[REDACTED]

Included in the above figures are eight shipments during February and twenty-three during March to institutions in foreign countries.

The two hundred ninety-nine shipments this month represent an increase of forty-seven percent over February and an increase of thirty-four percent over the previously high of two hundred twenty-three shipments for January, 1948.

Iodine (I¹³¹ - 8a)

Thirty 75-gram cans of irradiated tellurium were processed this month and approximately 33,399 millicuries of I¹³¹ were shipped. All of the product was within specifications.

The dissolver cell was decontaminated during the month to allow the installation of a drain line from the UH transfer tanks to the sump. This will allow the tanks to be washed without sending the wash water to the fissile product column cell.

A nitric acid coating-removal procedure, using mercuric nitrate catalyst, was tried on a batch of ninety-day slugs. The UH solution contained 0.04 mg aluminum per ml, which is considered satisfactory.

Phosphorus (P³² - 14.3d)

Six 2000-gram cans of irradiated sulfur were processed. Approximately 2,943 millicuries of P³² were shipped.

Equipment was installed during the latter part of the month to improve the method of loading sulfur cans into the melter of the pressure process equipment. This new equipment is designed to reduce the radiation hazard. A run has not been made since the installation of this equipment.

Carbon (C¹⁴ - 5100 y)

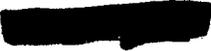
Seven hundred Ca(NO₃)₂ slugs were processed in seven runs. Approximately 480 millicuries were produced with an isotopic ratio averaging 4.54% and ranging from 3.8% to 5.8%.

An attempt to increase the isotopic ratio was made by purging the equipment with nitrogen before processing the Ca(NO₃)₂ slugs. There was no noticeable improvement.

Work continued on the installation of equipment for the Be₃N₂-C¹⁴ process. The installation is about seventy percent completed.

Sulfur (S³⁵ - 87.1d)

Approximately 650 millicuries of S³⁵ as sulfate were prepared from irradiated KCl during the month.



Fission Products

One run was finished and another is now in progress on the all-column fission product equipment. Approximately five curies of fission product activities were obtained in the various fractions, which were removed for purification on apparatus outside the cell.

Zr-Cb fractions are being purified by extracting the Zr and alpha emitters into TTA-benzene. Alpha activity is removed with dilute acid and the Cb is removed as it grows in by extracting with strong HCl. Various methods are being tried at present to remove the Zr-Cb mixtures in medium suitable for shipment. All outstanding orders for Cb were filled during the month.

Yttrium continued to be produced in small amounts, twenty-five to fifty millicuries per week, which is being used to fill outstanding orders. The method has been improved by eliminating a TTA-benzene extraction step which was used to remove alpha emitters; the purification is now handled entirely on columns by careful adjustment of pH and use of ammonia from Amberlite resin.

Small quantities of fairly pure rare-earth fractions, low in cerium and yttrium, have been obtained by fractionation on resin columns. The alpha contamination has been reduced to a sufficiently low level so that the material may be shipped to fill some outstanding orders. These rare-earth fractions have not yet been further separated to yield pure Pr or Nd.

No attempt is being made at present to purify the fractions containing some of the other fission products, such as Ba, Sr, Ce, Cs, and Ru.

Ruthenium (Ru¹⁰⁶ - 17)

The purification of Ru by distillation from the iron hydroxide precipitate on which it is carried has been improved considerably so that yields of seventy to eighty percent are obtained consistently. Considerable inactive Ru formed in fission makes the specific activity fairly low, about one millicurie Ru¹⁰⁶/milligram Ru. Approximately 110 millicuries of Ru¹⁰⁶ were produced during the month to fill outstanding orders.

Calcium (Ca⁴⁵ - 180d)

About fifty millicuries of Ca⁴⁵ were purified from Hanford irradiated material during the month. Six side hole tubes, each containing ten grams CaCO₃, were prepared and sent to Hanford for irradiation.

Purification of scandium and packing in cadmium-shielded tubes for irradiation in the Hanford reactor continued.

[REDACTED]

Zinc (Zn⁶⁵ - 250d)

Work was resumed on the separation of Zn from copper cyclotron target backings which had been bombarded with deuterons. However, only 0.1 millicurie of Zn⁶⁵ is obtained per backing, which makes production of Zn⁶⁵ impractical unless specially bombarded copper targets can be obtained. The specific activity of the preparation was 0.3 millicurie Zn⁶⁵/milligram Zn and the purity was ninety-nine percent.

No further work on this problem is planned.

Organic Synthesis

Two shipments of C¹⁴ labeled methanol (3.04 mc) were made during the month from material prepared under the direction of the Chemistry Division.

POWER PILE DIVISION

Nuclear Engineering

Navy Water-Cooled Reactor - Analytical work on the nuclear characteristics of the Navy water-cooled reactor was continued along the following lines:

- a. An additional point on the curve of γ as a function of Be-H₂O was determined.
- b. Calculations of critical mass as a function of reactor radius were completed for several conditions of poisoning for the 50% Be-H₂O reactor with water reflector.
- c. As part of an investigation of reflectors for the Be-H₂O reactor, reflector savings were computed as a function of reflector thickness for water reflectors and calculations on beryllium reflectors were started.
- d. Analysis of the inter-relation of energy release in the reactor, boiler, pressure, and load, as a function of time was begun.
- e. Analyses using zirconium in place of beryllium were begun.
- f. Calculations were continued on the control effect of different sizes and patterns of control rods, and the effects of changes in L² and γ on the control rod effectiveness. In addition, an investigation was begun to determine the heating of control rods.

Shielding. Work continued on the general problem of shielding, but the emphasis was shifted to calculations of the shielding required for the ducts and boiler of the water-cooled Navy reactor. Analysis of experimental data obtained on the helical duct through the Clinton Reactor was made and a report is in preparation.

[REDACTED]

Simulator. Work continued on the neutron flux distribution simulator. An analysis is being made to determine a convection factor to apply to the value of the network resistor units to make the network the exact equivalent (insofar as the mesh terminals are concerned) of the distributed electrical analog of a reactor.

Engineering Analysis

Part-time study has been underway on the thermodynamics of a gas-cooled reactor using a gas turbine in a closed system, and it is expected that a report covering this work will be issued in the near future.

A compilation of the physical properties of water (of use in heat transfer calculations) was completed for the range up to 1000°F and 6000 psi.

A survey of the information available concerning the effects of radiations on water was completed and issued. This information was needed especially for guidance in establishing reasonable conditions for the corrosion experiments with zirconium.

A preliminary study was made of flow distribution in parallel channels. It was concluded that mal-distribution would probably occur under local boiling conditions; however, these conditions are not amenable to theoretical analysis but require experimental investigations. Obviously, it is feasible to operate at lower heat flux densities below this unstable region.

Reactor Design

Possible designs for the internal construction of the water-cooled Navy reactor have been under study. The aim is to produce a design capable of fabrication by employing conventional techniques. It is expected that a preliminary design may be completed in the near future in sufficient detail to guide the fabrication studies on beryllium and zirconium in the direction most likely to be of application. The control rod drive study is nearing a close with attention at this point directed toward emergency operation of the rods and the possibility of simplifying the current design.

Coolant System Design

Navy Reactor Study. The study of plant layout for the Navy reactor was continued far enough to outline the requirements for the surge tank and the degassification system. A brief investigation of the generating equipment was made based on information received regarding the sizes of equipment. The sizes of the equipment are such that a side-by-side arrangement is out of the question within 15' 10" hull. Although a tandem arrangement is obviously possible, a study layout will be initiated in the future to investigate the possibility of reducing the space requirements by staggering the two units.

[REDACTED]

Materials Development and Experimental Work

Recovery of Uranium from Graphite Fuel Units. At the time of the last quarterly report of the Division, three recovery runs had been made using the electrolytic process for graphite disintegration developed by this laboratory. An additional run has been made without refluxing either during or after the passage of the current in order to determine whether the uranium will dissolve without lengthy extraction. Experimentation is now underway on the problem of adapting the electrolytic recovery process for large-scale operation.

Corrosion. A few simple tests on corrosion of Al, Zr, and Cb in water were initiated. The metal samples were placed in stainless steel bombs along with distilled H₂O, and heated in a muffle furnace at 350°C for periods ranging from two to six hours. The Al showed a weight gain of .0053 gm/cm² for the six hour test, while the Zr and Cb showed no gain in weight.

These results are in contrast to those published by Goldowski wherein it was stated that aluminum disintegrated in the presence of water at a temperature of 275°C. Goldowski's samples were maintained in pyrex vessels. To simulate these conditions a pyrex tube was placed in the bomb but still corrosion was not noticeable.

Fabrication of Beryllium Oxide. 28-1" I.D. x 1/2" O.D. x 4" long test cylinders were fabricated from coarse grain BeO bodies. Hot-pressed BeO hexagons, crushed and separated into selected ranges of screen sizes, were used as the grain. The study of the fabrication of coarse grain BeO bodies was continued with the substitution of ball milled BeO powder for the "as received" BeO powder. Well bonded BeO grain bodies were obtained by firing at 1550°C and 1650°C in a gas and an oil kiln respectively. Other BeO grain bodies fired at 1900°C in a graphite resistor furnace have a slightly higher value of bulk specific gravity, but they appear to be not as well bonded as those fired in gas and oil kilns.

Thermal Conductivity of Irradiated Graphite and BeO. The reactor experiments on the fast neutron and fission fragment damage to graphite and BeO operating at high temperatures were continued. The preliminary measurements of thermal conductivity at room temperature made on reactor control samples were perfected by using thermopiles in place of thermocouples and a much higher water flow. These changes gave better reproducibility.

Impregnation of BeO and Graphite. The impregnation of low density BeO was satisfactorily developed to give good uniformity up to the desired percentage impregnation and work was started on high density graphite along with intermediate percentage impregnation of the low density graphite heretofore not attempted.

Miscellaneous. The thermal expansion apparatus was worked over and improved so that better reproducibility was obtained on the MgO water crystal supplied by the Bureau of Standards.