Argonne National Laboratory
~~Metallurgical Laboratory~~

~~SECRET~~

715961

ANL-WHZ-5
Containing 6 p. and 0 fig.
No. 2 of 3, Series A

T/D 11/29/66
NRD

check with ANL w-2-6 in Preparation

Reading File
DCAA-A13580

Noted
Action
Recd. JUL 27 1946 NH
Ans.
File

July 26, 1946

To: Area Engineer ATTENTION: Private Davis

From: W. H. Zinn

Re: Special Items for the Narrative Report, July, 1946

Administrative. The fact that the Argonne National Laboratory does not have a firmly established permanent site still remains as a source of a number of difficulties in the development of the program. There are indications that the Forest Preserve Commissioners will soon act on the War Department's request for 265 acres at the Argonne Forest stie. If a favorable reply should come to this request some of the difficulties which have been very annoying up to now will have vanished.

Some preliminary investigations have been made of the possibility of the construction of housing for laboratory employees. Here again no further progress is possible without the site matter being settled.

The laboratory is still operating on a 44-hour week because the District has not seen fit as yet to approve all of the wage submission which was made in June.

An invitation has been received to send five people from the Laboratory to a conference at Los Alamos August 19 to 25. Five people have been nominated, namely F. Daniels, N. Hilberry, D. J. Hughes, R. G. Sachs and W. H. Zinn. In the event that some of these should not be able to go A. S. Langsdorf and O. C. Simpson have been nominated as alternates.

The cooperative program with the universities is beginning to function, a fairly large number of requests for information concerning possible participation having been received. A half dozen men from the cooperating universities are on their way. A number of requests for special radiation-survey instruments and for special materials have come from members of the departments of the cooperating universities, and these have been referred to the Director of the Division of Research at Oak Ridge.

Biology Division. A considerable portion of the activity of the division has been devoted to preparation of terminal reports. Several members have only recently returned from the first Bikini test, while others are still in the South Pacific. Research has been mainly directed toward the maintenance, observation and terminal examination of animals exposed to radiostrontium, radium, and plutonium at chronic levels. The data on mammary tumor incidence in rats that received single doses of X rays are now being analyzed and experiments are continuing on the placental and milk transmission of Pu by mice.

~~SECRET~~

Analyses of the radon content of the expired air of a patient who has accumulated radium for ca 35 years have indicated a total-body content of 1.5 micrograms of radium. Measurements were made with the vibrating reed electrometer developed by Dr. Moon, and the radium value assumes that 10^{-12} curies of radon per liter of expired air represent 0.1 microgram of radium in the body. Samples from the same patient are being subjected to biochemical and radiological study, and in anticipation of citrate therapy, methods for the determination of citrate in body fluids are being investigated.

The hematology group has been following the response of the peripheral blood of rats to single doses of X rays. At the LD_{50} (600r), the only level that has progressed sufficiently for analysis, the response is similar to that of rabbits at the same dose level, but with certain essential differences. The heterophils failed to rise at 4 hours and dropped to lower levels than in the rabbit, but followed much the same recovery pattern. Lymphocyte values, after an initial depression, returned to pre-irradiation levels by 30 days, indicating a much more rapid recovery of this element than in rabbits. In both species, an anemia begins at approximately nine days, reaches its maximum at 14-16 days, then begins to recover. In the rabbit, hemoglobin and erythrocyte levels were normal by 23 days, whereas, in the rat a slight degree of anemia has persisted for 66 days.

Further experiments have been initiated testing the effects of X rays and of Sr-89 on goats.

Chemistry Division, Section C-I. The extraction of tritium from the first three cans of lithium fluoride exposed at Hanford was completed. The tritium and helium formed during the 17-day irradiation period remained quantitatively in the lithium fluoride discs until the temperature was increased to 600° C. The yield from the three cans was approximately 7 cc of T_2 with an additional 7 cc of normal H^2, presumably present as an initial impurity. The fact that no appreciable pressure was generated within the cans during irradiation indicates that long irradiations may be safely employed with the consequent production of relatively large volumes of nearly pure tritium.

U-230 and U-231, along with U-232, have been produced in sufficient quantities by deuteron bombardment of protactinium to permit a preliminary measurement of thermal neutron fission cross sections. The fission measurements were made in the thermal column of the Argonne heavy water pile. By following the decay of fissionability it was possible to sort out the amount of fission attributable to each of the three uranium isotopes. The U-231 half-life is 4.2 days, the U-230 half-life 20.8 days, and the U-232 half-life 70 years. U-232 was previously prepared by another method and its thermal neutron fission cross section determined as being in the neighborhood of 50 barns. The U-230 cross section calculated from the present measurements is about 25 barns, and that of U-231 in the neighborhood of 300 barns. The last value is accurate only to a factor of 2 or 3 because of unavoidable uncertainties in the U-231 assay (based on X-ray counting, which is very difficult to place on an absolute basis).

Using the techniques of high-temperature microchemistry, all the trivalent americium halides have been prepared and have been found to be isomorphous with the corresponding neptunium and plutonium compounds (X-ray analyses by Dr. Zachariasen). It is interesting to note that the action of a 1:1 $HF-O_2$ mixture on AmO_2 at 500-750° C (strong oxidizing conditions) produced AmF_3 rather than AmF_4. If AmF_4 can be prepared at all it will probably be only by the use of free fluorine.

Separation of americium from plutonium by solvent extraction -- preliminary data show that small amounts of americium (produced by Pu-241 $\xrightarrow{\beta}$ Am-241 long life) may be successfully milked from large quantities of plutonium by a solvent-extraction system using hexone. Excellent recovery of americium and complete decontamination from plutonium is possible. This type of solvent-extraction operation can be made safe for the operating personnel despite the large quantities of plutonium involved. A large-scale set-up for this purpose is therefore being constructed.

On the enriched uranium pile decontamination studies (hexone extraction) dummy column runs have been made simulating suggested first-cycle conditions for the decontamination and recovery of uranium from the proposed Clinton enriched pile. In general the results have been very favorable with uranium losses consistently below 0.1%. Laboratory studies of second-cycle behavior indicate that in the presence of reducing systems (such as Fe II plus N_2H_4) plutonium will remain in the aqueous second-cycle waste under conditions where uranium recovery and decontamination is satisfactory.

In further studies of the Redox process to improve fission-product decontamination in the second cycle, a run was made with tetravalent plutonium using a relatively low concentration (0.5M) of aluminum nitrate as the salting agent. The distribution ratio for Pu(IV) in this concentration range is rather unfavorable so that plutonium recovery in column VIA under these conditions was relatively poor (2% loss). Decontamination, on the other hand, was high, with factors of 7×10^7 for betas and 2.5×10^7 for gammas. Future runs are planned with conditions intermediate between this run and a previous run which gave exceptionally low losses and only moderate decontamination.

Chemistry Division, Section C-II. Values of stored energy in Hanford graphite, as determined by the heat of combustion method, have been obtained on a sample which was irradiated in the water-cooled sidehole of the D pile at approximately 55° C for an exposure equivalent to 836 Mwd/central ton. Data were obtained for samples annealed at various temperatures from the "as received" condition up to 1798° C. A graph of stored energy against annealing temperature is approximately a straight line up to about 1000° C. A table of the results will be included in the monthly report.

Experiments on the release of stored energy by the Sykes method at various heating rates show that the temperature at which maximum stored energy is released increases with the rate of heating.

Preliminary results on the heat of sublimation of graphite have given a value of 204 ± 2 kcal/mole. The error has been calculated by the least squares method and reports only the internal consistency of the data. This value of the heat of sublimation is unexpectedly large and a search is being made for possible systematic errors.

Argonne National Laboratory
~~Metallurgical Laboratory~~

A Chromel-Alumel thermocouple after bombardment in the Hanford pile for 24 days has been checked against a standard couple. No significant change occurred in the thermal EMF properties due to the neutron irradiation.

The thermal conductivity of the pure BeO rod decreased approximately 20% due to neutron bombardment of 24 days in the Hanford pile. This decrease was not annealed out after heating to 620° C.

The thermal conductivity of 90% BeO-10% UO_2 samples decreased approximately 500% after neutron bombardment of 24 days in the Hanford pile. This decrease may not have been the result of neutron bombardment but may have been due to oxidation of samples by the residual air in the irradiation cans. Tests are being carried out in an attempt to separate these effects.

The crushing strength of high-density 90% BeO-10% UO_2 samples decreased approximately 30% after the neutron bombardment. Similar results were obtained on high-density 2% UO_2 samples. The crushing strength of low-density material (both 10% and 2%) decreased approximately 6%. Again, these decreases are not necessarily due to the neutron bombardment but may be due to some oxidation during bombardment.

The linear dimensions of pure BeO samples did not change by a measurable amount after neutron bombardment. The linear dimensions of BeO-UO_2 samples (10% and 2%) increased approximately 0.5% after neutron bombardment.

Tests on the thermal crack-up of BeO tubes subjected to a radial temperature gradient show that thermal crack-up takes place at the thermal stresses predicted by theory using the known physical properties of beryllia. Addition of 2% SiO_2, 2% Al_2O_3, or 2% CaO did not improve the strength of the beryllia tubes toward thermal cracking.

The spectral and total emissivities of white beryllia have been measured over a temperature range from 900° C to 1600° C. The spectral emissivity at 0.65 microns is approximately 0.22 and the total emissivity ranges from approximately 0125 at 900° C to 0.32 at 1600° C.

The elastic modulus of neutron-irradiated beryllia suffered no change within experimental error.

The diffusion constant for xenon from high-density 90% BeO-10% UO_2 is measured to be 2.3×10^{-8} cm^2/min at 1450° C. In the steady state, approximately 5 curies/liter of radioactive xenon will be present in the high-temperature pile coolant if operated at 1450° C. Tests are under way to determine the rate of diffusion as a function of temperature.

A fuel-rod sample containing 1% SiO_2, obtained from Battelle, erroneously reported to have lost no uranium upon heating in oxygen at 1500° C, was found to have lost 35% of the uranium after heating for 7.8 hours under these conditions.

A successful method has been developed to impregnate graphite with uranium oxide up to concentrations of 20 g of uranium/cm^3 of graphite.

1100262

Hazard Evaluations Division. (Formerly Health-Physics). Two serious spills occurred this month at the West Stands. One on June 18 involved 20 liters of fission-product solutions spread over an area of about 600 sq. ft. in the main corridor on the first floor. Beta activity from 20-100 mr/hr at 3" was found and was immediately cleaned up by the chemists with 20% HNO_3. Air samples were well below tolerable levels during the procedure.

The second spill occurred on July 3, on the stairway at the south end of the West Stands. Several liters of solution containing 100 mg of Pu boiled over and spread onto the hood, stairs, wall, and the floor below. The area was partially cleaned up by the chemists and the decontamination squad completed the job. Adequate safety precautions were taken by all involved. The hood used was very poorly constructed and is being removed completely.

Two extraordinarily high alpha air samples were obtained at New Chemistry. One, in Room 31, on July 29, was 1400 times the Pu tolerance concentration. The decontamination squad had been painting some active apparatus there prior to removal for burial and had not thought it necessary to wear gas masks. Most of the room had already been decontaminated, but no high air activity had been observed. Urine and feces samples are being taken of the men involved, but as yet the results have not been determined. On July 3, in Room 11, decontamination procedures again caused a very high air level which was about 200 times tolerance. This time, however, assault masks were worn. These measurements illustrate the seriousness of the air hazard involved in such work and further emphasize the necessity for wearing masks at all times during such activitie .

West Stands contamination has been steadily increasing in intensity and area. During the week of July 8 a general clean-up was started. Already appreciable reduction in the levels has been noted. An attempt is being made to do likewise in many of the hooter laboratories at New Chemistry. No significant changes took place at the Argonne or Site B.

Several developments have been made in the way of facilitating the use of hazard evaluation instruments. A nomograph has been devised to eliminate all calculations in using the "Filter Queen" to determine the percent of the tolerable concentration of Pu in air. One series of uranium standards has been prepared for attachment to each survey meter to enable a check on its sensitivity in the field and another set has been made for keeping a daily check on the hand and foot counters.

During the month 2628 pocket meters were read with 3 overexposures. Total film developed was 1731 with 5 overexposures. Possible overexposure to the hands was represented in 3 of these readings. The 2 remaining readings represented possible overexposure to the body and both were window readings only.

Health Division. The situation in West Stands has been reviewed with the Chemistry Division. A broad program of improvement of existing facilities and installation of new facilities is being work out in cooperation with the division.

The level of activity in the New Chemistry building, filtered air section, leaves much to be desired from the health-protection point of view. This is also being discussed with Chemistry Division personnel.

Survey of the amount of Pu excreted with the stools of persons handling Pu is continuing. The persons working in the filtered air section of New Chem tend to show more Pu in stools than those in West Stands or the south end of New Chem.

[redacted]

Investigation of the beryllium toxicity problem indicates that this material must be handled with extreme precaution as both acute and possibly chronic lung lesions may develop.

Metallurgy Division. NaK corrosion work was delayed this month because of failure of the 120 KVA transformer and difficulties in preparing and counting radioiron. Indications of previous test are that weight changes in 1010 steel specimens do not give a true picture since the weight change can be attributed to decarburization. The electroplated radioiron on Armco iron should eliminate the confusion since the change of rate of counting of the layer of radioiron should indicate the amount of corrosion. Specimens from the test just completed are being counted. Another test using electroplated radioiron is in progress using a radioiron whose purity is better known and having a half-life of 47 days.

Other dynamic systems are being designed, several similar to the one in use and one convection-type circulating system.

Dynamic air-corrosion tests on uranium-columbium-zirconium alloy show weight gain rates varying from 0.0006 to 0.0018 mg/cm^2/hr. Two aluminum-coated samples, one appearing to have a porous coating and the other appearing to have a sound coating, show identical weight gains. In all probability both coatings were porous. Further work is being conducted on improving the aluminum coating and nickel-plating the alloy.

Physics Division. In the crystal structure section during the past month further X-ray diffraction studies of irradiated graphite from Hanford were carried out.

One additional americium compound, AmI_3, has been identified and its crystal structure determined.

Most of the time of the mass spectroscopy group has been spent in report writing. A slug from Hanford was examined and found to be pure lead and not the lead-cadmium alloy requested.

Instrument Division. The specifications for the vibrating-reed electrometer have been completed and are now in the possession of the District Office at Oak Ridge. The District promises to get the bids from industrial companies for the production of this instrument as quickly as possible. The wiring of the amplifiers for the eight units being built in the laboratory has been started.

The machine shop has almost all of the parts for Sigmion completed, except for the cabinets. The instrument will be ready for production, and production should be well underway in the coming month.

The secondary emission chambers have proved to be entirely satisfactory. Thus far they have been used only as integration meters for measurement of radiation dosage. The chief advantage of these chambers will probably lie in the fact that the time of collection of the electrons in a vacuum will be extremely short as compared to collection of ions in a gas-filled chamber. Plans are being made to construct a fast amplifier to be used with this chamber in analysis of high-intensity pulses, such as one obtains from the betatron.

Requests for the development of instruments from this and other sites have increased appreciably this past past month. It is regrettable that we have lost so many of our more qualified personnel, especially when it is so difficult to replace them. A major part of our effort will consist of trying to improve the personnel in the Instrument Division.