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ENGINEERING

UNITED STATES ATOMIC ENERGY COMMISSION

NATIONAL BUREAU OF STANDARDS PROGRESS REPORT TO THE ATOMIC ENERGY COMMISSION JULY 1, 1950 TO SEPTEMBER 30, 1950

By
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Prepared by Paul L. Howard and Laurel S. Winslow

OBJECTIVE

Radiation detection elements require d-c sources of high voltage capable of furnishing small currents of 10^{-8} to 10^{-11} amperes. The British-made Zamboni pile has been tried in some applications, however, it cannot be specified as a standard component because there is at present no commercial source of supply.

This project has been established for the purpose of developing a Zamboni type pile suitable for the requirements of our equipment. This includes the development of manufacturing techniques so that suitable manufacturers may be set up to supply the units.

DISCUSSION OF REQUIREMENTS

The results of tests made on the British Zamboni pile in present instruments showed much to be desired in the way of battery performance. The resistance of an 800 disc pile was 10^{10} ohms. Figure 1 shows the discharge characteristics of such a pile with an initial current of 0.035 microamperes at 470 volts.

This project was set up with certain desirable requirements to be used as a guide. Most of the characteristics are purposely flexible since equipment requirements are not completely crystallized at this time.

	Type A	Type B
1. Open circuit voltage	800 v	200 v
2. Maximum over-all internal resistance	3×10^9 ohms	10^{10} ohms
3. Maximum current	10^{-8} amperes	10^{-10} amperes
4. Dimensions (cylindrical)	$6'' \times 1''$ (dia)	$2'' \times \frac{1}{4}''$ (dia)
5. Weight	5 oz	2.5 oz

6. Storage life 2 to 4 years for each type
7. After discharge for a period it should recover its open-circuit voltage relatively fast
8. Temperature range −30°C to +45°C

DISCUSSION OF PROGRAM

This project was initiated in June 1950 so this is the first quarterly report. A letter report, dated August 21, 1950, outlined the preliminary phases of the program. This is summarized briefly as follows:

1. A study of the literature pertinent to the Zamboni pile was made in order to ascertain the extent of information available. The most important references are incorporated in Appendix 1.

2. Investigation of the British-type pile was made from the standpoint of determining its composition and voltage characteristics. The cell is a manganese dioxide-tin system using a paper saturated with a 2% zinc chloride solution.

3. In order to establish the reproducibility of the system the first phase involved the processing of cells with our own materials using the British metal negative which was tin.

4. After establishing the fact that we could reproduce the voltage and current characteristics of the British pile the next phase involved the study of using various metals as negatives such as magnesium, aluminum, zinc, and tin (control). This was followed by a study of the variations in types of MnO_2. Work is in progress on the use of various types of conducting carbon mixed with MnO_2 to improve the intercell connection, i.e., reduce over-all internal resistance.

EXPERIMENTAL

The single cell or disc of the British-type Zamboni pile consists of a thin layer of MnO_2 brushed on one side of a wet paper which is coated with an adhesive material such as syrup. A sheet of metal foil is rolled on the other side relying on the same adhesive to hold it to the paper. The discs are cut out of large sheets to form the cells. These are kept at a relative humidity of about 50% throughout the storage and useful life of the cells.

In order to carry out the investigation it was necessary to establish a procedure for measuring the voltage of single cells so tests could be made on various types of cells without introducing intercell contact resistance as an unknown factor. A vibrating reed electrometer (Model 30) was modified so as to measure voltages from 0 to 2.5 volts by means of the the introduction of a back e.m.f. into the feedback circuit. With a shielded measuring unit individual cell voltages could be measured with current drains of not over 10^{-15} amperes.

A study has been made of the various combinations of materials to determine the best use in making a modified copy of the British Zamboni pile. The results of this work are summarized in tables I, II, and III. Rather than use a syrup of unknown composition it was decided to try both "Methocel" or carboxymethylcellulose as the adhesive. These were found to be practical.

There are a number of types of MnO_2, both natural and chemical. Burgess chemical ore, African ore, New Jersey Zinc activated ore, Bright Star chemical and electrolytic ores were tested to determine whether there would be any significant difference in their behavior. These ores were checked against magnesium and aluminum negatives.

In all initial studies distilled water was used along with either the "Methocel" or the carboxymethylcellulose. After obtaining the characteristics of cells where the composition was known the next step was to add the corresponding metal chloride to the water to improve conductivity.

Shelf tests at 90% humidity have been set up on all types of cells. This high humidity was used to accelerate the test. Others were placed on test at 30–40% humidity.

A series of cells were made and put on discharge at 10^6 and 2.2×10^7 ohms. The results of these tests are given in Figs. 2 and 3. One test on the magnesium cell on the 2.2×10^7 ampere discharge is not complete at this time.

DISCUSSION OF RESULTS

In the British pile the cell open-circuit voltage averages 0.7 volts. In Fig. 1 we see that there is a 100 volt drop from open circuit to initial closed-circuit voltage followed by a further gradual drop over the discharge period. Figures 2 and 3 show the improvements obtained during the initial phase of our investigation both from a voltage and capacity standpoint. At the 10^6 drain the British cell is out immediately and at 2.2×10^7 has very little capacity.

Tables I, II, and III show the voltages when using other metal negatives. The results of shelf tests thus far are not as good as we want, however, the results are very encouraging for the beginning.

The first phase of the work is not complete at this time. In order to reduce the over-all resistance of this type of battery it is necessary to reduce the internal resistance of the cells and also the contact resistance between cells. This involves the use of a salt electrolyte to improve the conductivity thus reducing the internal resistance of the cell and a mixture of a good conducting medium in the MnO_2 to reduce the contact resistance. Work on this phase is in progress at the present time.

As soon as a satisfactory pile of this type is developed for manufacture, the second phase of the work will be started. This will be the development of new techniques and materials to obtain a cell of different materials capable of higher drains and streamline production.

Since the work is in a preliminary stage no conclusions can be drawn at this time.

APPENDIX 1

1. Zamboni, G., Gilbert Annalen, 49, 41 (1815), 51, 182 (1815), 60, 151 (1819).
2. Thompson, S. P. Elementary Lessons in Electricity and Magnetism, p 180 (1901) (Macmillan, London, 1899).
3. Benham, C. E. The Dry Pile (Marshall, London).
4. Anertach, F. Handbuch der Physik, 2nd edition, 4, 185 (Barth, Leipzig 1905).
5. Graetz, L., Handbuch der Elektrizitat und des Magnetism 1, 441 (Barth, Leipzig 1918).

6. Bragg, W. L. Electricity, p 51 (Bell, London, 1936).
7. The Dry Voltaic Pile, Electronic Engineering, 20 (1948) p 317 (October).

Table I—Open-circuit voltages of cells made with 90% MnO_2 (Burgess Chemical) 10% Halo Black mixed with "Methocel" or carboxymethylcellulose and pasted on Munsing paper sized with the corresponding cellulose paste against various metal negatives

Metal negative	Time	Open-circuit voltage of cells pasted with "Methocel"	Open-circuit voltage of cells pasted with Carboxymethylcellulose
Mg	Initial	1.9	1.7
	3 mo (90% humidity)	1.6	1.3
Al	Initial	1.2	1.9
	3 mo (90% humidity)	1.1	1.5
Zn	Initial	1.3	
	3 mo (90% humidity)	1.1	
Sn	Initial	1.0	1.0
	3 mo (90% humidity)	0.7	0.7

Table II—Open-circuit voltages of cells made with various types of MnO_2 using "Methocel" for mixing, Munsing paper sized and Mg and Al as negatives

Type MnO_2	Time	Open-circuit voltage of cells with metal negatives: Mg	Al
1. Burgess Chemical	Initial	1.9	1.1
	2 mo (90% humidity)	1.5	1.0
2. Bright Star Chemical	Initial	1.7	1.1
	2 mo (90% humidity)	1.3	0.9
3. N.J. Zinc Activated	Initial	1.6	1.0
	2 mo (90% humidity)	1.3	0.7

Table II—(Continued)

Type MnO_2	Time	Open-circuit voltage of cells with metal negatives: Mg	Al
4. Bright Star Electrolytic	Initial	1.7	1.1
	2 mo (90% humidity)	1.5	1.0
5. African Natural	Initial	1.6	1.1
	2 mo (90% humidity)	1.4	1.0

Table III—Open-circuit voltages of cells made with various types of MnO_2 using carboxymethylcellulose for mixing, Munsing paper sized and Mg and Al as negatives

Type MnO_2	Time	Open-circuit voltage of cells with metal negatives: Mg	Al
1. Brugess Chemical	Initial	1.6	1.1
	2 mo (90% humidity)	1.3	1.0
2. Bright Star Chemical	Initial	1.4	1.0
	2 mo (90% humidity)	1.3	0.8
3. N.J. Zinc Activated	Initial	1.4	0.85
	2 mo (90% humidity)	1.1	0.8
4. Bright Star Electrolytic	Initial	1.5	1.0
	2 mo (90% humidity)	1.2	0.8
5. African Natural	Initial	1.4	1.0
	2 mo (90% humidity)	1.2	0.9

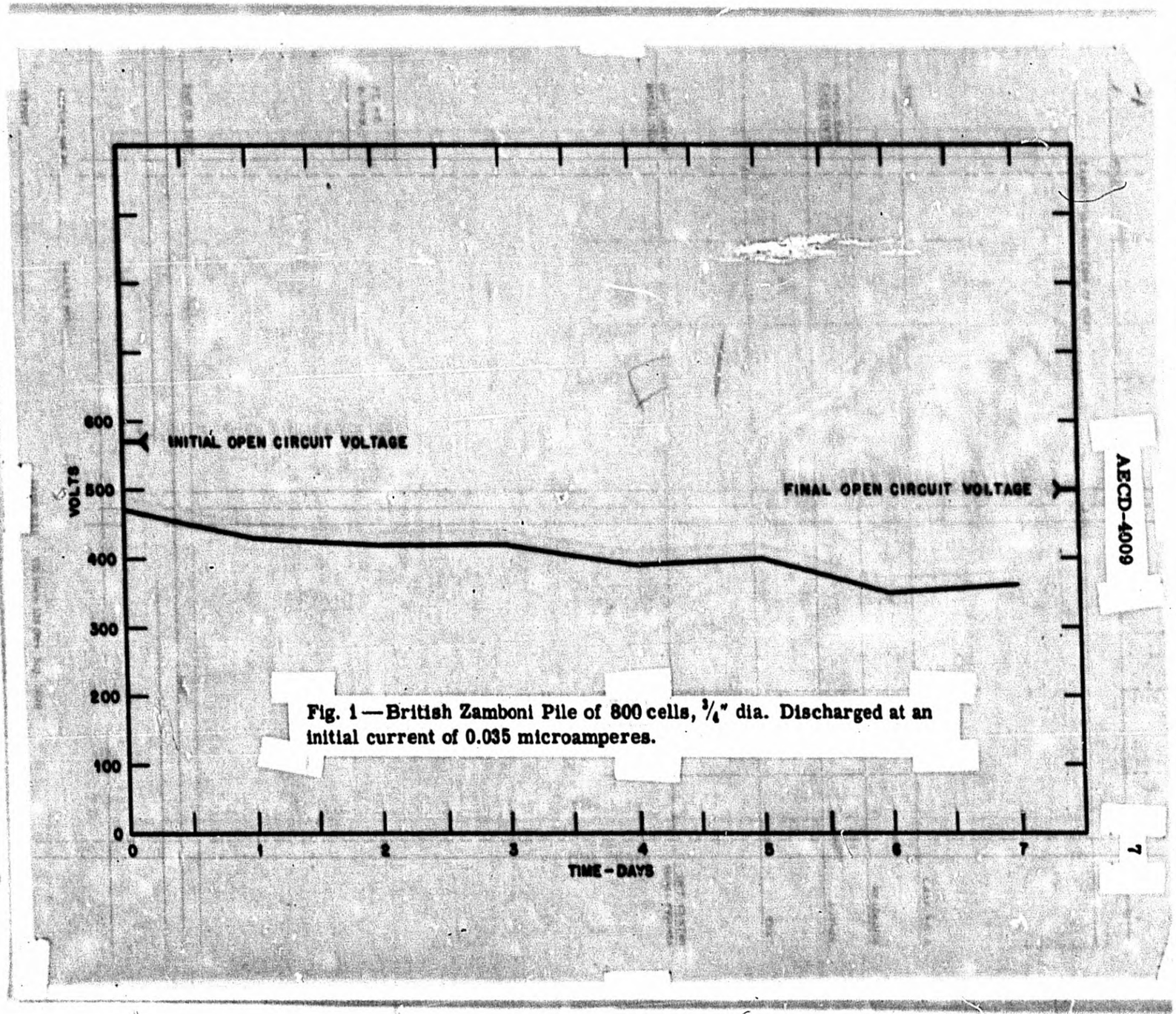

Fig. 1 — British Zamboni Pile of 800 cells, ¾" dia. Discharged at an initial current of 0.035 microamperes.

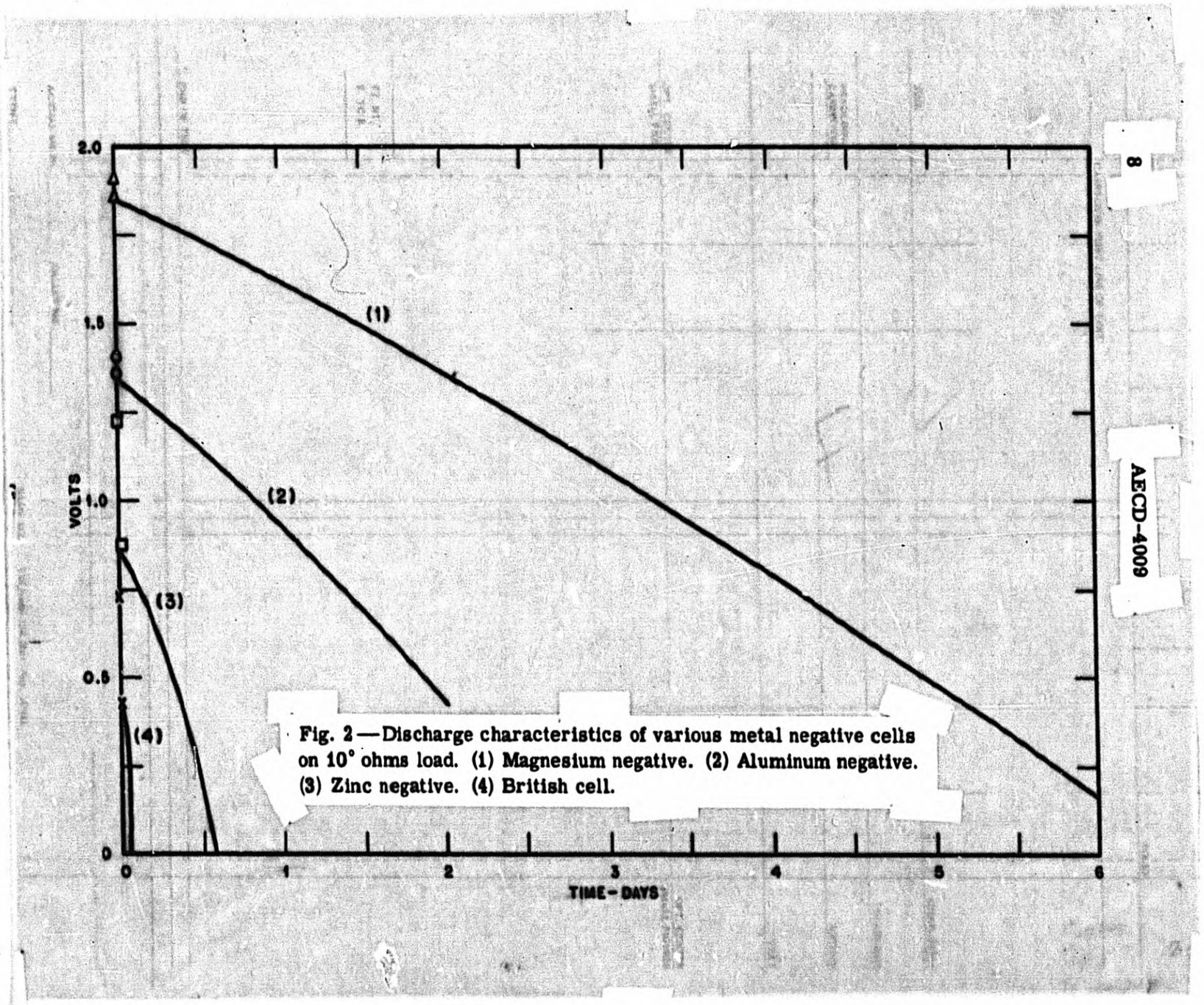

Fig. 2—Discharge characteristics of various metal negative cells on 10° ohms load. (1) Magnesium negative. (2) Aluminum negative. (3) Zinc negative. (4) British cell.

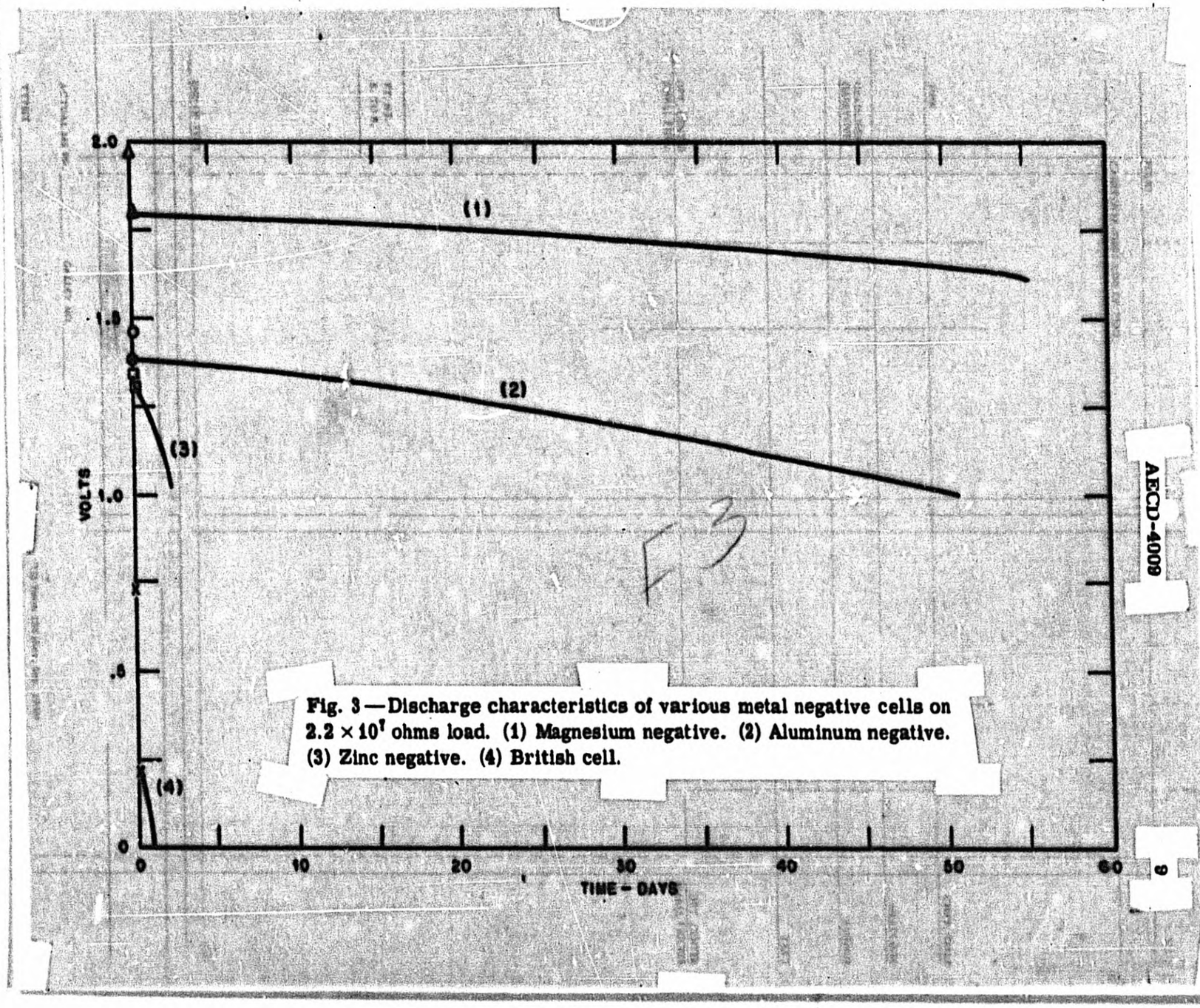

Fig. 3 — Discharge characteristics of various metal negative cells on 2.2×10^7 ohms load. (1) Magnesium negative. (2) Aluminum negative. (3) Zinc negative. (4) British cell.

Report to the Atomic Energy Commission
Division of Heat and Power (3.2)
National Bureau of Standards
July-September, 1950

Heat of Solution, 0° to 200°C (Project 2603)

All of the electric wiring for the heat of reaction vessel has been completed. A change in the design of the "inner" system of the reaction vessel has been made. The original design provided for stirring in the liquid only by convection caused by the heater on the reaction vessel. To provide an additional factor of safety on the stirring, it was decided to provide for some mechanical stirring at the completion of the experiment. For this purpose, a novel device has been designed which not only provides the stirring, but also has a valve which can be used in the experiments where hydrogen gas is evolved (such as the solution of beryllium metal). Some of the parts for this "inner" reaction vessel system have been made and assembled, and the performance of the type valve used has been tested.

In the solution experiments, it is necessary to measure accurately the amount of HF solution which is introduced into the reaction vessel. For this purpose, a special filling apparatus has been designed which uses a platinum-rhodium cylinder as a container for the HF and a piston (using Teflon packing) to push out the required amount of HF, as measured by a screw thread on the outside of the piston.

Heat Capacities from 0° to 900°C (Project 2604)

The chemical analyses of the compositions of the three sodium-potassium alloys were made. These analyses also established and confirmed that the weights of the samples whose heat capacities had been measured were substantially different from those previously assumed. On the basis of these results, the partial molal heat capacities of sodium and potassium in the three alloys (78%, 54%, and 45% K) were calculated and were found to be very close to the heat capacities of the pure elements which had been found earlier in this project.

A request has been made by the Oak Ridge National Laboratory for measurements on the heat capacity of lithium in the range up to 900°C.

Arrangements have been made to have the Knolls Atomic Power Laboratory prepare and seal capsules containing pure lithium in a manner similar to that used for the sodium and potassium samples.

The measurements on the heat capacity of sodium have been published in the July issue of the Journal of Research NBS. The mercury results are now in the process of publication in the same journal. The results on beryllium have been offered for publication in the Journal of the American Chemical Society.

The apparatus for measuring the solubilities of certain materials in liquid sodium has been designed and is now being made in the shop. It will be possible in this apparatus to make more accurate measurements of the melting points of the samples, as well as to determine the solubilities at the melting points.

As part of the program of this Bureau to develop and maintain standards of heat capacity, measurements of heat capacity have been completed on two pure materials. The first material, Al_2O_3 in the form of synthetic sapphire, has been measured up to 900°C, and is now being issued on a limited scale as a convenient standard of heat capacity at high temperatures. Normal heptane has been measured up to 250°C (within 17 degrees of its critical temperature) and is also issued as a standard sample for lower temperatures.

Because of interest expressed through the AEC in three low-temperature phase-equilibrium experimental problems, these were examined for their general feasibility. There were planned in some detail procedures and apparatus which promised to give the order of accuracy requested.

Analysis by Spectroscopy (Project 2612)

The NBS has loaned to the AEC a spectrometer and the services of Dr. Herbert P. Broida as required by the AEC to develop spectroscopic means for quantitative determination of the concentration of certain isotopes.

Stedman Packing (Project 2620)

A still consisting of a 1" × 12" Stedman rectifying section was designed and built for operations at liquid hydrogen temperature. Boilup rates and holdup were determined for several heat inputs for hydrogen and for deuterium. Two fractional distillations were carried out on

mixtures of hydrogen and hydrogen deuteride. Excellent separation was obtained at the lower rate; the higher boilup rate gave less efficient separation. Analyses of portions from these distillations were made by the mass spectrometry section. The results of the work were reported under date of September 12.

Work is now in progress on the measurement of entrainment of liquid hydrogen at fixed vapor velocity. It is planned to make experiments at two or more pressures. Construction of the apparatus is about 50% complete.

/s/ Defoe P. Ginning

September 27, 1950

Report No. 19

From: Lauriston S. Taylor

To: Dr. Huntoon
Coordinator of Atomic Energy Commission Projects

Subject: Atomic Energy Commission Quarterly Report of the Radiation Physics Laboratory
National Bureau of Standards for
July 1, 1950, through September 30, 1950

2900. Travel for the National Committee on Radiation Protection. Travel was provided for persons discussing the work of the National Committee on Radiation Protection with related work being performed by scientists in other countries. Travel was also provided for the Chairman of the Subcommittee on Waste Disposal and Decontamination to discuss the activity of this subcommittee at the meeting of the American Chemical Society in Chicago.

2908. Assay of Ores and Sludges. 43 ores and 20 sludges were assayed and reported during the quarter. In addition, there were a number of assays made on standards used by the section of the chemistry division that do the solution work on ores and sludges from the Atomic Energy Commission.

There were eight samples (6 regular samples and 2 standards) cross checked with the AEC New Brunswick Laboratory. Two solutions of each of the samples were prepared by Mrs. Richmond, in the chemistry division, and aliquots of each solution assayed by the Bureau's laboratory and the New Brunswick Laboratory on the same day. Of these eight samples the results of the separated assays checked within 5% except for two samples on which the deviations were 7% and 8% respectively. The latter, we believe, should be disregarded since the values obtained by the AEC Laboratory on the two solutions of this sample varied by over 7%; whereas, the two values obtained by this laboratory checked to exactly 1.5%.

Undoubtedly, because of the discrepancies in these results, other cross checks will be made in the near future. The details of such cross checks and their necessity are determined by Mr. Bright, of the Chemistry Division working in conjunction with Mr. Butler of AEC.

It is hoped that changes in arrangement of present equipment and introduction of new equipment already on hand will be completed within the next quarter. This will greatly increase the over-all efficiency of the laboratory.

2909. Radiation Attenuation Studies. It has been found that since large fluctuations occur in the output of the betatron it is impossible to use the electrometer to measure the current directly. A monitor has been set up to automatically compensate for output fluctuation but this arrangement has not been perfected. In the meantime a Victoreen r-chamber is being used to determine the variation of ionization with wall thickness. The broad peaks on these curves at 40 and 50 Mev would seem to indicate that a single thickness is satisfactory over quite a large range of Mev.

2910. Emission Constants of Radioactive Isotopes. No work has been performed on this project during the quarter, because of lack of personnel. It is expected that the work will be resumed during the next quarter.

2911. 500 Kv Standard Ionization Chamber. An attempt has been made to determine the optimum dimensions required of this chamber to satisfy the definition of the roentgen. The plate separation vs ionization does not have the plateau expected, and the slope of these curves changes with X-ray energy. This data together with the measured ionization/cm of plate, would indicate that the guard plates or/and guard wires are ineffective in producing a uniform field over the full length of the collector plate. A review of this work is being made to determine the possible causes of these discrepancies and the necessary steps to be taken to qualify this chamber as a standard.

2912. 500 to 1400 Kv Electron Studies. In this quarter we have made dosage density measurements on Dupont 652 insensitive film and Minimax film using the G. B. electron accelerator. Measurements were made in the energy interval 600–1500 kv. Preliminary analysis of our data indicates that the density-dosage relationship is independent of energy.

No attempt has been made to study all the systematic or random errors in the experiment, to reduce the dosage data to standard conditions of temperature and pressure, or to make background corrections. Hence, the results are to be understood as preliminary results.

2913. Positive Ion Tube. Techniques have been worked out for electrophoretically coating thoria on ribbon strip cathodes of molybdenum and tungsten. In tests these have given results much superior to the usual oxide-coated nickel cathodes previously used, requiring less outgassing during activation, permitting a higher emission current density in the arc, and permitting reuse after exposure to atmospheric pressure.

The vacuum and accelerating systems for two sections have been completed and tested. With the ion source running, the pressure in the lower section is 5×10^{-6} mm. Accelerated ion currents of 0.4 milliamperes have been measured at the tube base after acceleration through 40 kv in the first and up to 120 kv in the second gap. Field emission from the second gap itself produces at this voltage X-rays of 200 mr per hour at about 2 meters distance.

A magnetic analyzer has now been completed and will be used to separate ion components and determine beam composition from the ion source. The first results using a new design of ion source having shaped graphite electrodes shows a very well focussed beam, but a substantial component of heavy ions essentially undeflected by the magnetic field.

A new ion source block of copper with improved cooling providing a minimum of residual gasses is being constructed.

2917. Low Energy X-ray Measurements. Because of lack of personnel, no work has been done on soft X-ray measurement studies during the last three months except a few minor mechanical adjustments.

2921. Radiation Instrument Calibrations. Studies are being made to determine the operating ranges and quality-dependence of various G-M counter tubes having chrome-iron electrodes and halogen fillings. The studies have indicated that it appears to be feasible to place tubes in parallel that are responsive to various exposure

rates and thus cover measurements with intensity ranges from below permissible level up to 500 r/hr. It furthermore appears that the addition of a tantalum shield having a thickness of a few thousandths of an inch is sufficient to reduce the quality-dependence in the range 0.080–1.0 mev to 10 per cent or less.

Characteristics of Victoreen high-range pocket chambers have been studied. The quality-dependence in particular is found to be within 15 per cent. Also, the insulator leakage of the 100-r and 200-r chambers after full-scale exposures are given has been observed to be within reason. However, the leakage of the 600-r chamber is intolerable, and because of it the chamber is being completely redesigned.

10-r and 50-r dosimeters built by the Kelekot Mfg. Company have been calibrated and studies of their leakages have been made. These dosimeters have generally been observed to indicate dosage of million-volt radiation accurate within ±10 per cent. However, their response to 100-kv radiation is more than double the correct response. The leakage of the dosimeters is tolerable, being 2 per cent or less 24 hours after full-scale dosages are given.

Lauriston S. Taylor
Chief
Radiation Physics Laboratory

Quarterly Report
to
Atomic Energy Commission
Quarter Ending 9/30/50
Project No. 3208

The Synthesis of Isotopically Labeled Sugars and Related Compounds

In accordance with the general plan outlined in our contract, work was begun on the project July 1, 1950. Those engaged either full or part time on the project are: Dr. H. S. Isbell, leader; Dr. H. L. Frush, Dr. J. V. Karabinos, and Nancy B. Holt.

Improvement of the cyanhydrin synthesis and adaptation to a millimole basis. Heretofore, the synthesis of labeled glucose by the cyanhydrin method has been reported with yields of only 5 to 10 percent. The general aim of our work this quarter has been to raise this yield by careful investigation of each step in the process. For the preparation of glucose and mannose the process is:

$$\text{D-arabinose} \xrightarrow[\text{(quant.)}]{\text{cyanide}} \text{cyanhydrin} \xrightarrow[\text{(quant.)}]{\text{hydrolysis}} \left\{\begin{matrix}\text{gluconic acid}\\ \text{mannonic acid}\end{matrix}\right\}$$

(proportions can be varied 1:3 to 3:1)

$$\rightarrow \underset{(80-90\%)}{\text{separation}} \text{ mannonic lactone barium gluconate} \xrightarrow[(90\%)]{} \text{gluconic lactone}$$

$$\text{Lactone} \xrightarrow[(75\%)]{\text{reduction}} \text{sugar} \quad \text{isolation (?)}$$

Yields in parentheses are those so far obtained in this laboratory.

The following progress has been made:

1. By a titrimetric procedure, studied the condensation of D-arabinose with cyanide, and ascertained conditions under which the reaction is substantially quantitative.

2. Studied the saponification of the nitrile, by determination of the ammonia liberated, and ascertained conditions under which the step is substantially quantitative.

3. Devised a convenient method for estimating the composition of the hydrolyzed product formed from D-arabinose and cyanide.

4. Found that the proportions of gluconic and mannonic acid can be varied by changing the conditions of the condensation. This enables one to obtain predominantly either gluconic or mannonic acid.

5. Developed a technique for the separation of crystalline mannonic lactone, and crystalline barium gluconate on a millimole basis in yields corresponding to 80 percent of the amounts estimated by the method of 3 above.

6. Developed a technique for the conversion of barium gluconate to crystalline gluconic delta lactone on a millimole basis in 90 percent yield.

7. Developed a technique for the reduction of lactones of sugar acids on a millimole basis in a closed system with yields of approximately 75 percent by analysis.

Work is now in progress on the separation of the crystalline sugars. A detailed description of the work outlined here will be included in a future report.

/s/ Horace S. Isbell
Project Leader

Quarterly Progress Report
to
Atomic Energy Commission
NBS Section 5.4
Project No. 3200
Chemical Analysis

September 29, 1950

ANALYSIS

(a) for New York Operation Office, U.S.A.E.C.

Number of Samples

Type of Materials	Analyzed for U_3O_8	Analyzed for Loss	Prepared for Ra Detn.	On hand for future treatment U_3O_8	On hand for future treatment Loss	On hand for future treatment Ra	On hand for future treatment Pt
K-65			19			19	
Q-11	28	28	49	12	12	38	
D-28	4	4		2	2		
MGX	17	17		7	7		
Waste Prod. #43					1		1
Total	49	49	68	21	21	57	1

(b) for Colorado Raw Materials Operations, U.S.A.E.C.

Type of Material	Analyzed for V_2O_5	Analyzed for Loss	On hand for future analysis
Fused vanadium pentoxide	12	12	30

INVESTIGATION

Studies have been made on the following analytical problems: (1) qualitative analysis of K-65 residues; (2) behavior of MGX material

during drying tests; and (3) purity and behavior of the NH_4VO_3 used as the standard in vanadium determinations.

COOPERATIVE STUDIES

(a) With the New Brunswick Laboratory of the U.S.A.E.C.– Continuing the study of variations in radium results, M. S. Richmond took portions of twelve N.B.S. solutions of K-65 and Q-11 samples to the New Brunswick Laboratory for comparative measurements. Results have been exchanged on these solutions, but results for similar solutions that were to be prepared at New Brunswick have not been received. Dr. J. E. Hudgens, Chief of the Radiochemical Branch and Dr. J. J. Tregoning, Chief of the Analytical Branch of the New Brunswick Laboratory have been consulted on problems in both radium and uranium measurements.

(b) With R. M. Fowler of the Union Carbide & Carbon Research Laboratories, Inc.–We have analyzed the U.C.&C. standard sample of fused vanadium oxide once, but are to analyze it again under other drying conditions when our work load permits.

/s/ H. A. Bright, Chief
H. A. Bright, Chief
Analytical Chemistry Section

September 29, 1950

To: Dr. R. D. Huntoon, Coordinator of U. S. Atomic Energy Commission Project at the National Bureau of Standards

From: E. J. Prosen, Chief, Section 5.9

Subject: Quarterly Report, 1st Quarter, Fiscal year 1951.
AEC Project No. 3203

Series A: 1 thru 8, R. D. Huntoon, NBS
9 thru 11, E. J. Prosen, NBS

Report on the work for the U.S. Atomic Energy Commission by Section 5.9 (Thermochemistry) for the Quarter ending 9/30/50
AEC Project No. 3203

TECHNICAL SUMMARY

Stored energy in irradiated graphite. Stored energy determinations have been made on 27 samples of irradiated graphite supplied by the General Electric Pile Technology Division, Hanford Works. These determinations were made by the heat of combustion method to study the effect on stored energy of pile irradiation and subsequent annealing at temperatures up to 2,000°C at 100°C intervals. Two reports on this work (NBS-EJP-1, NBS-EJP-5) have been sent to the Hanford Works, Attn: Paul H. Reinker.

Stored energy in irradiated diamond. Stored energy determinations were made by the heat of combustion method on two samples of diamond and two samples of graphite from the Argonne National Laboratory. This study is being made to compare stored energies in graphite and diamond which have been irradiated simultaneously. One report on this work (NBS-EJP-2) has been sent to the Argonne National Laboratory, ATTN: W. Primak.

Stored energy in beryllia and silicon carbide. A calorimetric vessel has been designed for the determination of stored energy in substances for which the heat of combustion method is not suitable or possible. This vessel should permit direct stored energy determinations to be made. Two samples of unirradiated SiC and two samples of irradiated BeO (hot-pressed) have been received from the Argonne National Laboratory for study.

Heat of formation of diamond. Preparation is being made to determine the heat of formation of unirradiated diamond, the heat of transition of graphite to diamond, and the influence of the degree of graphitization of carbon on its energy content.

For the Director,
by

/s/ Edward J. Prosen
Edward J. Prosen, Chief
Thermochemistry Section

AECD-4009

QUARTERLY REPORT
on Project No. 3205
ATOMIC ENERGY COMMISSION WORK
of the
SPECTROCHEMISTRY SECTION, DIVISION 5, SECTION 10
for
JULY 1 TO SEPTEMBER 30, 1950

TECHNICAL SUMMARY

Research activities were continued on the development of analytical methods for the analysis of project materials. Preliminary work was carried out on the direct spectrometric determination of isotope ratios in uranium samples. The analysis of project materials especially the analysis of uranium-beryllium alloys was continued.

1. Determination of Isotope Ratios in Uranium (Scribner, Ballinger)

The purpose of this task is to investigate the possibilities of applying the Baird Direct-Reading Spectrometer or other direct photoelectric measuring devices to the determination of the ratios of isotopes in ordinary and enriched uranium samples. Prior to the receipt of known isotopic mixtures, preliminary work was carried out in the modification of equipment to be applied to the problem. The illuminating system of the Baird spectrometer has been changed to increase the light efficiency by a factor of about two by incorporating a mirror in the projection system and by relocating the electrode holder near the slit. This modification has resulted in improvement in reproducibility of measurements, particularly with non-uniform sources such as a point-to-plane electrode combination. Reproducibility tests on steel samples show average deviations from the mean amounting to about one percent for most of the elements and as low as 0.5 percent in some cases.

In anticipating work on this and similar problems, a ratio-recorder had been ordered from the Leeds and Northrup Company and delivery is expected at the end of October. This instrument will permit the study of scanning techniques which appear to offer greater promise in isotopic analysis than the fixed-slit system now incorporated in the Baird spectrometer. In this connection also, a visit was made to the Leeds and Northrup Company to examine a new self-contained photoelectric scanning spectrometer. The instrument offers some interesting

possibilities in isotopic analysis and arrangements are being considered for loan of an instrument of this type to the Bureau for test.

Five samples of known mixtures of uranium isotopes were received from Carbide and Carbon Chemicals, Oak Ridge, Tennessee, at the end of this quarter. These samples will serve for calibration purposes in these studies of isotopic analysis.

2. Determination of Beryllium in Uranium (M. E. Mayo and B. F. Scribner)

The results of spectrochemical determinations of beryllium in beryllium-uranium alloys were reported for 14 samples submitted by division 8, section 2. The lower concentration range for these determinations was extended to 0.03% Be with an estimated limit of detection of less than 0.005% Be. Results are now being reported to three significant figures. Minute particles of nitric acid insoluble material observed in the first samples tested were shown to be insignificant to the Be content but as the alloy composition has changed toward very low beryllium values, increasing insoluble material has been noted. Results on the last samples tested indicate that such samples pick up beryllia from the crucible during melting and that enough goes into solution in HNO_3 to give values higher than the calculated addition of Be.

3. Spectra of Elements 43 and 61

In cooperation with Dr. W. F. Meggers of the Spectroscopy Section, the emission spectra of technecium (43) and promethium (61) has been observed in our laboratory and the spectrograms were measured in the Spectroscopy Section. In addition the absorption spectrum of promethium nitrate had been measured in our laboratory. The preparation of the following papers covering this work, for publication in the Journal of Research NBS, was completed during the quarter:

1. The Arc and Spark Spectra of Technecium, W. F. Meggers and B. F. Scribner.
2. The Absorption and Emission Spectra of Promethium, W. F. Meggers, B. F. Scribner, and W. R. Bozman.

4. Miscellaneous Analyses (E. M. Krumrine and M. E. Mayo)

The application of micro methods to uranium-ore-residue sample analysis and the use of gallium oxide as a testing matrix are being investigated. These methods are expected to provide semi-quantitative and quantitative values instead of the general qualitative system now used. One sample of gold irradiated in the Oak Ridge pile was examined spectrochemically for division 5, section 4. Spots on the metal, after a vacuum distillation, were shown to contain mercury as the major metallic impurity.

TESTS

Analyses were made of 47 samples involving 625 determinations. Analytical reports for the quarter totalled 11.

Respectfully submitted,

/s/ B. F. Scribner
B. F. Scribner, Chief
Spectrochemistry Section

AEC Project 8.3
NBS Project 4100

Report of Atomic Energy Commission Projects First Quarter, Fiscal 1951 – Metallurgy Division

September 29, 1950

Equilibrium Diagrams of Binary Alloys, Uranium-Beryllium System

Metallographic study of uranium-beryllium alloys containing less than about 5 weight percent beryllium gave results that were not in accord with the composition as determined by chemical analysis and threw doubt on the accuracy of the chemical determinations of small amounts of beryllium in the presence of large amounts of uranium. As an eutectic and probably solid solution occur in this section of the diagram, accurate analyses are essential to its establishment. At the request of the Metallurgy Division, the Spectrochemistry Section of the Chemistry Division has developed a procedure for the determination of small amounts of beryllium in uranium, giving the desired accuracy and has prepared standards for use in the analysis. All of the low beryllium alloys have been re-analyzed by this spectrochemical method and investigation of the uranium-rich end of the diagram is proceeding.

Analyses of ingots from the freezing point determinations show that segregation is much less pronounced in ingots having a uranium matrix than in ingots having a beryllium matrix. Segregation was negligible in ingots having less than 5 weight percent beryllium. This permits use of the low freezing point ingots of the low beryllium alloys for metallographic and X-ray examination, eliminating the necessity for preparation of a series of duplicate ingots.

It was observed that frequently a pick-up of beryllium occurred during preparation of the alloys by melting in beryllia crucibles. This pick-up does not interfere seriously with the preparation of alloys with beryllium contents above about 0.5 weight percent but has hindered the preparation of the series of low beryllium alloys.

There is evidence that the amount of beryllium pick-up is related to the procedure used in firing the beryllia crucibles and is usually negligible for crucibles fired to 1800°C or higher in an oxidizing atmosphere as contrasted with the substantial pick-up from crucibles

fired to the same temperature in the Arsem (graphite resistor) furnace. An oxy-propane fired furnace has been constructed in which to fire the beryllia crucibles to temperatures of 1900°C or higher.

Ten uranium-rich alloys were homogenized for 3 weeks at 1050°C in Argon metallographic studies indicate that 3 weeks is sufficient for homogenizing alloys with 5 weight percent beryllium or less but as the composition approaches that of the compound, UBe_{13}, longer heating periods are necessary for homogenizing.

Thermal analyses have been made on alloys in the range from 0 to 27 percent beryllium although difficulty with oxidation of the specimens is still encountered. Non-porous refractories of the mullite type are being prepared to replace the K-30 refractories at the top and bottom of the thermal analysis furnace. This should reduce the quantity of gas evolved from refractories during heating.

Freezing point values of from 1126° to 1128°C were obtained in the thermal analysis apparatus on the uranium used in preparing the alloys. This is in satisfactory agreement with other freezing point determinations on uranium of similar purity, indicating that temperature measurements in the thermal analysis apparatus are sufficiently accurate. Determinations on alloys near the eutectic composition indicate the solidus to be approximately 1060°C. This will be checked by metallographic methods.

Uranium-Titanium

A preliminary survey of the uranium-rich end of the uranium-titanium has been started. Pick-up of beryllium resulted from melting uranium-titanium alloys in Arsem fired beryllia crucibles. Results of spectrochemical analyses of an ingot from a crucible fired in an oxidizing atmosphere are awaited to determine if the change in firing procedure is effective in reducing the pick-up. The ingots are being studied to determine also the extent of segregation. The thermal analysis curves of ingots containing about 3 atomic percent titanium (composition of the charge) gave arrests at 1222°C, 1147°C, and 1121°C.

/s/ H. E. Cleaves
H. E. Cleaves
Chief, Chemical Metallurgy

Project Number: 4422
Project Title: Production of Large BeO Crystals
Report for Quarter Ending Sept. 30, 1950
Prepared by Ernest M. Levin

Summary of Activities

Mr. O. F. Rynders, formerly of the Glass Section, Mineral Products Division, was obtained for work on the project.

After a review of literature on the subject and a visit to the crystal growing section of the Naval Research Laboratory, it was decided to use the vapor-transport method for the first attempt at producing BeO crystals. This method shows promise because of the volatility of BeO at temperatures above 1,000°C in the presence of water vapor. It is also relatively simple and can be tested easily. The necessary equipment has been obtained or ordered, and a Pt-Rh wound tube furnace is being constructed.

September 29, 1950

MEMORANDUM TO: Dr. R. D. Huntoon

AEC CERAMIC PROJECT
Refractory Porcelains and Applications
in
Nuclear Heat-Generating Devices
Quarterly report for period ending September 30, 1950
NBS Project 9.1-4401

9.1.1 (NBS Project 9.1/31) — Ceramic Survey

A modified and declassified version of Ceramic Survey TID 80, Part II, will be published in the forthcoming October issue of the magazine NUCLEONICS.

9.1.2 (NBS Project 9.1/32) — Metal-Ceramic Compositions

During July we employed the services of J. Wesley Cable, a consulting electrical engineer, so that we might discuss with him our furnace problems involving induction heating and radiation shielding.

Since the radiation shields are in the inductive field, a modified shield design was sought which would not pick up energy from the field. Our consultant's suggested shield design has shown enough promise, when tested with a low-temperature model of stainless steel, to warrant its trial in the furnace. A ceramic cylinder has been cast and matured and now awaits the arrival of molybdenum strips for its completion. The ceramic cylinder serves two purposes — (1) it is, itself, a shielding device, and (2) it supports and positions molybdenum strips which act as reflecting shields without absorbing energy from the inductive field.

A double succeptor has been prepared as a result of Mr. Cable's suggestion. This design permits the current induced in the large outer cylinder to be channeled through a smaller concentric cylinder which results in the latter being heated very effectively. In addition, the outer part which is relatively cool acts as a radiation shield. Space is also provided between the two succeptors for additional shielding. Temperatures of approximately 1800°C have been attained with only a part of

the available power. These temperatures have been held for only short periods of time until the stainless steel strip shield can be replaced with a more complete one of molybdenum.

A vernier dial for power supply adjustment has been obtained and installed. This provides for exact duplication of settings and for almost stepless temperature control.

Various metallic coatings have been investigated to determine their suitability as reflecting shields when applied to the inner surface of the fused silica vacuum furnace chamber. Platinum showed the most promise from the group tested, including gold, chromium, nickel, aluminum, palladium and platinum. Most of these metals, including platinum, can be vapor deposited from a tungsten heater in vacuum.

R. F. Geller
Chief, Porcelain & Pottery Section