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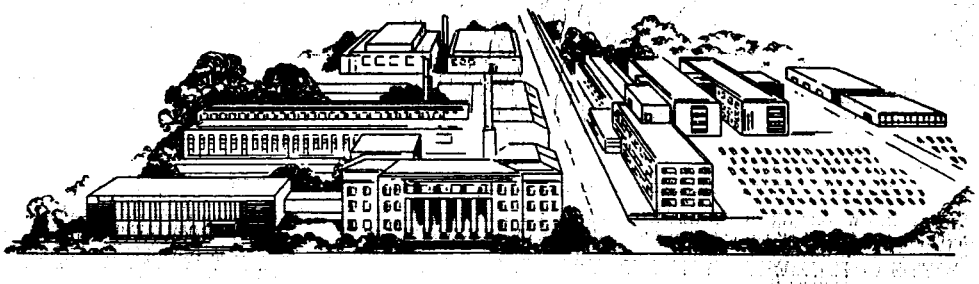

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BATT-4673-Q13

THIRTEENTH QUARTERLY REPORT
(Covering the Period: October 1 through December 31, 1963)

on

ENGINEERING PROPERTIES OF POTASSIUM AND CESIUM

to

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

by

Alexis W. Lemmon, Jr.

January 30, 1964

Contract NAS 5-584

Technical Management
NASA-Lewis Research Center
Nuclear Power Technology Branch

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THIRTEENTH QUARTERLY REPORT

on

ENGINEERING PROPERTIES OF POTASSIUM AND CESIUM

by

Alexis W. Lemmon, Jr.

INTRODUCTION

This program at Battelle is being performed for the National Aeronautics and Space Administration (NASA) to investigate the engineering properties of potassium and cesium. The scope was enlarged only recently to include selected properties of cesium, as well as the work on potassium previously under way. All work is being performed under Contract NAS 5-584, and this report is the thirteenth quarterly report of progress; research done from October 1 through December 31, 1963, is described.

Many of the thermodynamic and transport properties of potassium, both liquid and vapor, have been measured in the temperature range from 900 to 2100 F. Experimental values for vapor pressure, specific heat of liquid, thermal conductivity of liquid, viscosity of liquid, and P-V-T characteristics have been obtained. Currently, the experimental program for measuring the thermal conductivity of potassium vapor is in progress. Experimental techniques for the direct measurement of the specific heat of potassium vapor and for the measurement of the viscosity of the vapor have also been of interest. The latent heat of vaporization, enthalpy, entropy, and specific heat of potassium vapor have been computed from some of the data obtained.

The enthalpy, viscosity, and thermal conductivity of liquid cesium are being measured at present. At a later date the thermal conductivity of cesium vapor also may be determined. It is anticipated that the information derived will be useful in the design, testing, and operation of nuclear electric-power generating systems for use in space, for which either potassium or cesium would be the working fluid.

SUMMARY

Measurements of the viscosity, vapor pressure, thermal conductivity and heat content of liquid potassium have been concluded. In addition, the compressibility of potassium vapor has been measured in the P-V-T apparatus. Also concluded has been the design study of equipment for the direct determination of the specific heat of potassium vapor. The vapor pressure and compressibility data for potassium were used to derive a virial equation of state which, in turn, was used for the computation of enthalpy, entropy, and the specific heat of the vapor. Currently, the effort on potassium

is limited to the operation (and evaluation of the initial results and techniques) of the bare-wire-probe apparatus to obtain values for the thermal conductivity of the vapor. On the cesium phase, the test cells and required auxiliary equipment are being prepared in anticipation of the measurements to be performed during the next 3 months with the ultrapure cesium obtained commercially.

Measurements of the thermal conductivity of nitrogen gas, made at room temperature to assess the reliability of the bare-wire-probe apparatus have given a value of 2.6 ± 0.5 watt cm^{-2} cm C^{-1} as compared with a literature value of 2.7 watt cm^{-2} cm C^{-1}. Minor modifications of the apparatus during the course of the work reduced the scatter found in the early measurements. The results of initial measurements on potassium vapor between 400 and 800 C, however, revealed that it did not respond as nitrogen did to this method of measurement; various possibilities for this anomalous behavior are being explored. Use of the Nb-1Zr cell to 1150 C for extensive evaluation of potassium vapor is planned for the future.

All the bar stock and tubing of the Nb-1Zr alloy needed for the test cells for the cesium measurements has been received from the suppliers. Metallographic examination of sections and ultrasonic, dye-penetrant, and microscopic examinations of the surface of these materials were judged satisfactory as were the levels of impurities, particularly those of oxygen and nitrogen, reported by the suppliers. Examinations to assure soundness of the material will continue during machining and fabrication.

Cesium in various sized ampoules for loading each of the test cells has been received from the selected supplier. Impurity levels have been judged exceptionally low on the basis of freezing-point curves and spectrographic analyses.

The capsules to contain the cesium for the enthalpy measurements in the Bunsen ice calorimeter are nearly ready for loading. It is expected that the measurements will be made in February.

The pressure vessel and related parts used in measuring thermal conductivity of liquid potassium have been cleaned and reassembled. Also, the new specimen container of the Nb-1Zr alloy is being prepared. Thermal conductivity values needed for data evaluation are being obtained on the Nb-1Zr alloy stock to 900 C, and these will be extended to 1200 C in the apparatus to be used for the cesium measurements prior to the measurements on cesium.

Preparations for the viscosity measurements on liquid cesium are nearly completed. When the new cell is ready, the moment of inertia, period, and residual damping of the empty cylinder will be determined prior to filling with cesium.

EXPERIMENTAL MATERIALS

Joseph F. Walling

All pieces of apparatus which contact cesium will be fabricated from Nb-1Zr alloy. During this quarter all material ordered was received from suppliers and judged acceptable.

General acceptance tests for all Nb-1Zr stock and tubing consisted of a metallographic examination. All material was judged satisfactory by this technique. All solid stock came from the same lot, and chemical analyses were rendered by the supplier. Oxygen and nitrogen levels were well below tolerance limits. Special analyses for nitrogen and oxygen were not run on the tubing. Good previous experience with the same supplier and sufficient confidence in the metallographic examination were deciding factors against doing additional analyses.

Further tests for surface soundness were performed on solid stock marked for use in equipment for measuring the thermal conductivity and heat capacity of liquid cesium. These pieces of material are to be bored out to form rather thin-walled containers. This requires surface soundness in the original stock.

In compliance with instructions, the supplier delivered stock which was treated only with a very light vapor blast or perhaps sharp centerless grind. The supplier performed ultrasonic tests on the material and found no indications which were cause for rejection.

Upon receipt at Battelle, this stock was lightly liquid honed and then examined for surface flaws by means of a dye penetrant. This examination revealed many surface imperfections, some of which were attributed to faults in a swaging die. It is believed that this was good evidence to indicate that no machining operations were performed which might have tended to obscure surface-connected cracks. All imperfections observed were surface pits, not cracks which would have justified rejection. Additional visual observation under a microscope indicated nothing to cause rejection of the original stock.

Although much effort was expended in these additional acceptance tests, they were insufficient to guarantee completely the adequate performance of the material. Indeed, no acceptance tests could be done to provide such a guarantee. Therefore, additional testing will be performed as fabrication of the pieces proceeds. These examinations will be reported in appropriate discussions of Phase IV - Items 2 and 3.

The procurement and analysis of the cesium to be used for the measurements are discussed in a later section of this report.

DETAILS OF INDIVIDUAL PROGRAMS

The experimental and analytical activities on the potassium portion of this program have been concluded with but one exception, the measurement of the thermal conductivity of potassium vapor. The concluded portions are no longer being reported here; those who desire information on the results of the completed work are referred to one or more of the topical reports which are listed in the final section of this report.

Phase III. Measurement of Thermal Conductivity of Potassium Vapor

(Joseph Matolich, Jr., and Herbert W. Deem)

The thermal conductivity of potassium vapor is being measured over a temperature range from 480 to 1150 C (900 to 2100 F). The dynamic method with a bare-wire probe, and the apparatus used, were described in the Eleventh Quarterly Report.[1]*

An estimate of the range of parameters expected for potassium vapor indicated that measurements on nitrogen at atmospheric pressure and at room temperature would approximate those for potassium vapor at 400 C. Nitrogen was selected as the gas to be measured in testing the apparatus and method.

Two stainless steel probe and boiler assemblies, as shown in the Eleventh Quarterly Report[1], were made for use in measuring nitrogen and potassium vapor to moderate temperatures. One assembly was supplied with purified nitrogen, and potassium was added to the second assembly.

The measured thermal conductivity of nitrogen at atmospheric pressure and room temperature averaged 2.6 ± 0.5 watt cm^{-2} cm C^{-1} as compared to a literature value of 2.7 watt cm^{-2} cm C^{-1}. Several small modifications in the apparatus made during the course of measurements on nitrogen reduced the scatter of data of the early measurements.

Thermal conductivity measurements have been made on potassium vapor, at various degrees of superheat, from 400 to 800 C. Two marked differences in the results of measurements on nitrogen and potassium vapor were noted. First, there was no clear point in the potassium curve where convection started, a point that was rather clearly defined in the nitrogen curves. This is a point at which the temperature of the probe wire becomes essentially constant as the convection mode of heat transfer is added to those of conduction and radiation. Second, there was a marked dependency of the measured thermal conductivity on power input to the probe. This is not indicated to be the case by the mathematical analysis of the method. A critical evaluation is being made of the mathematical analysis of the method in an effort to understand these two apparent anomalies.

Also, there appears to be at least one reason to suspect the integrity of the stainless steel probe chamber and boiler assembly containing the potassium. The probe wire should be in essentially a good vacuum at room temperature because of the low vapor pressure of potassium. The curves obtained from the potassium probe, at room temperature, are different, however, from curves of the evacuated stainless steel probe. This difference indicates that there may have been a leak in the potassium assembly between the filling, at about 1×10^{-5} mm Hg, and the final closure weld. Also some gas in the probe may have come from outgassing of the chamber and boiler during the measurements. However, radiographs of the boiler before and after the measurements do not show any loss of potassium, and the apparatus, both electrically and mechanically is functioning well.

Parts for the Nb-1Zr probe and boiler assembly for measurements to 1150 C have been machined and are ready for welding.

*See page 10 for references.

If the current study and evaluation of present results of measurements on nitrogen and potassium vapor are successful in resolving the previously mentioned anomalies, work during the next period will include welding the Nb-1Zr assembly, adding potassium to the boiler, and measuring the thermal conductivity of potassium vapor to about 1150 C. Provision is being made to pump out the assembly and then make a final closure in place in the vacuum chamber in which the measurements are to be performed. The probe will be heated during this pumping to reduce the outgassing of the parts and walls during the measurements. It is planned that a vacuum of about 2×10^{-5} mm of mercury will be maintained in the vacuum chamber during the measurements.

Phase IV – Item 1. Preparation and Analysis of Cesium

(Joseph F. Walling)

During the last quarter, cesium was obtained from the supplier in a state of purity and in containers suitable for direct loading into the various pieces of apparatus.

The general loading procedure used successfully for potassium was selected for use with cesium as well. This consisted of breaking a glass ampoule containing the alkali metal under vacuum and delivering the filtered fluid to the desired apparatus. Consequently, measured quantities of cesium were needed in glass ampoules. This proved simple to obtain from suppliers.

The problem of procuring ampoules of cesium of determined purity proved much more difficult. Analysis of samples for foreign-metal content is rather simply carried out by emission spectrography in the visible region. This procedure was also used for potassium. The matter of analyses for carbon, nitrogen, and oxygen is considerably more difficult. However, carbon and nitrogen contents of commercial lots are generally so small as to be of no consequence for present purposes. The oxygen contamination cannot be ignored because of its effect on the container material. Moreover, oxygen is picked up easily from many sources.

It is believed that no procedure for determining a meaningful oxygen content of liquid cesium exists now. The best information available to us indicates that mercury amalgamation and butyl bromide techniques, sometimes used for sodium and potassium, fail totally when applied to cesium. The nature of this failure is that quantitative oxygen additions to aliquots cannot be accurately detected. Neutron-activation techniques have not progressed sufficiently to be reliable at low concentrations.

Because of these factors more indirect inference of contamination levels must be made. A well accepted technique for estimating total impurities is that of freezing-point depression. It was possible to obtain cesium from a supplier in large glass ampoules, each fitted with a resistance-thermometer well and a stainless steel agitating device. It was then possible to obtain freezing-point curves during agitation of the metal which had been sealed in this container suitable for our loading into an apparatus. A representative curve is shown in Figure 1.

Freezing points of six ampoules were determined to be 28.52, 28.52, 28.53, 28.52, 28.54, and 28.52 C. The depressions indicated by these freezing points reflect total contamination. But, calculation of the absolute magnitude of contamination is not completely reliable for several reasons.

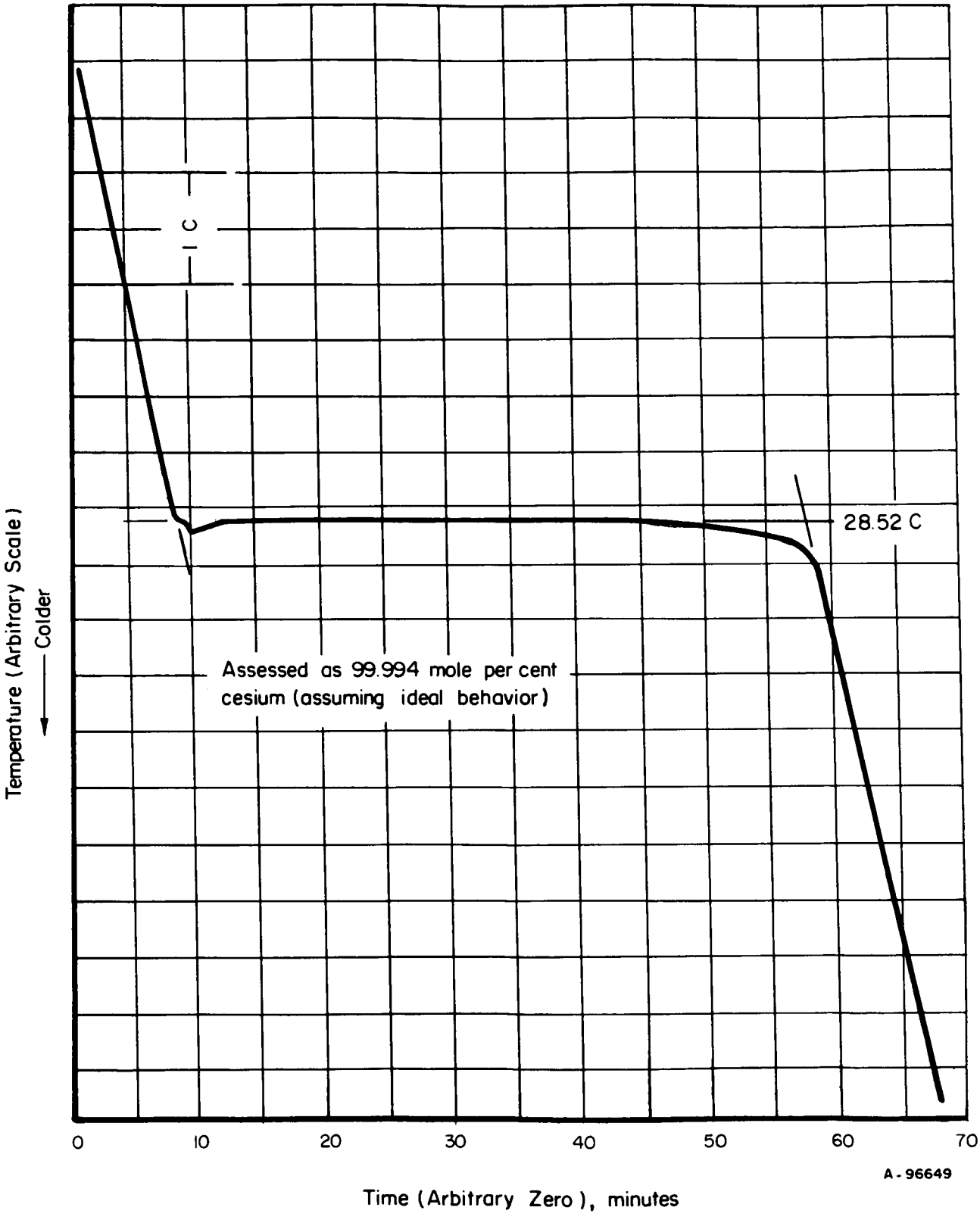

FIGURE 1. FREEZING POINT CURVE OBTAINED WHILE STIRRING THE CESIUM IN LARGE AMPOULE 2

(1) The freezing point of absolutely pure cesium is not known.

(2) The freezing-point-depression constant is therefore unknown.

(3) The correctness of the method generally used to determine freezing point and depression of a freezing point is questionable when applied to cesium.

The method generally used to determine freezing points has been extensively developed by Rossini.(2) It involves determining the freezing point of a sample as varying fractions of it are frozen. It assumes an ideal liquid solution in equilibrium with absolutely pure solid. This assumption is questionable in present applications, but investigation in this area is beyond the program scope. It is nonetheless possible to infer several things in general:

(1) The higher the freezing point the purer the sample.

(2) The smaller the slope of the equilibrium portion of the curve, the purer the sample.

The freezing points reported here are as high as or higher than any reliable reported measurements previously observed. Slopes of the equilibrium portion of all curves are virtually undetectable. A temperature difference of 0.05 C could be seen with ease. On the basis of ideal solution behavior, the supplier has estimated all samples to be 99.994 per cent cesium or better. Thus, all indications are of acceptable material in the six large ampoules.

It was not possible to determine the freezing points of the eight small ampoules. However, it is reasonable to infer their acceptability. Contamination can come from three sources:

(1) The batch material and delivery-tube system.

(2) The atmosphere contacting the metal.

(3) Surface material in each individual ampoule.

In filling, all ampoules were fabricated and transferred while hot to an inert-gas-filled dry box which housed a cesium reservoir. Thus, the atmosphere was common to all ampoules. All ampoules were filled from the same cesium reservoir-delivery tube system. It is, therefore, reasonable to expect similar effects in all ampoules from these two sources of contamination. Significant problems with contamination by surfaces in individual ampoules would be reflected in erratic freezing points of the large ampoules. This is clearly not the case. Therefore, the similar handling of all ampoules makes it reasonable to infer approximate uniformity of the material supplied.

Foreign-metal content has also been assessed by spectrographic techniques. A batch analysis from the supplier and results of a sample analyzed at Battelle are tabulated in Table 1.

TABLE 1. FOREIGN METAL CONTENT OF CESIUM

Element	Amount, ppm Supplier, Batch	Amount, ppm Battelle	Element	Amount, ppm Supplier, Batch	Amount, ppm Battelle
Al	<2	2	Ni	<2	5
Ag	--	1	Pb	<2	--
Ba	8	--	Si	<4	10
B	<18	--	Sn	8	--
Ca	18	3	Sr	<2	--
Cu	<2	10	Ti	<2	--
Cr	<2	--	Tl	<2	--
Fe	<14	2	Li	18	--
Mg	5	1	Na	<16	20
Mn	5	--	K	7	15
Mo	--	1	Rb	35	20

Differences between these analyses are not important for the current experimental program. Sampling error in the one sample analyzed at Battelle could probably explain many discrepancies. However, foreign-metal content is certainly at a satisfactorily low level. Therefore, the cesium is acceptable by all standards imposed.

All cesium has been delivered and is ready for loading into various apparatuses as they are prepared.

Phase IV - Item 2. Measurement of Specific Heat of Liquid Cesium

(Edward A. Eldridge and Herbert W. Deem)

The enthalpy and specific heat of liquid cesium are to be measured to 1150 C. Enthalpy will be measured in the Bunsen ice calorimeter previously described.[3]

The Nb-1Zr alloy has been received; the capsules have been machined and are now being welded. The capsules were designed similarly to the capsules used for potassium.[3]

During the next period, the capsules will be heat treated, loaded with cesium, and enthalpy measurements made to 1150 C.

Part IV - Item 3. Measurement of Thermal Conductivity of Liquid Cesium

(Joseph Matolich, Jr., and Herbert W. Deem)

The thermal conductivity of liquid cesium is to be measured from 100 to 1150 C (212 to 2100 F). The method, apparatus, and techniques will be the same as those used for liquid potassium, as previously described.[4]

The pressure vessel and related parts used in making thermal-conductivity measurements on liquid potassium[4] have been cleaned and reassembled. Because of potassium leakage during the measurements, it was necessary to replace the titanium guard cylinder, including heaters and cooling coils. This guard cylinder is assembled and ready for attaching heaters and cooling coils.

Dye-penetrant and microscopic examinations of Nb-1Zr alloy specimen containers have continued at intervals during the various stages of machining. The material appears to be sound. The wall of the specimen container was made about 0.040 inch thick, which is about twice the thickness of the chamber wall used to contain the potassium. It was decided to use a thicker wall after microscopic examinations of the potassium container showed areas of porosity with evidence of potassium penetration. This wall leakage is now thought to be responsible for the presence of potassium outside the specimen container, which forced a termination of the potassium measurements because of thermocouple failures.

Thermal-conductivity measurements are in progress on a specimen taken from the bar of Nb-1Zr alloy used for the specimen container. Measurements are being made to about 900 C by a well proven method and apparatus. Thermal-conductivity data for the alloy are needed for use in determining the total heat flow as well as that fraction through the container walls during the thermal-conductivity measurements on cesium.

During the next period, thermal-conductivity measurements on the Nb-1Zr alloy will be extended to about 1200 C in the cesium apparatus, the specimen container will be loaded with cesium, the apparatus assembled, and thermal conductivity of liquid cesium measured to about 1150 C.

Phase IV – Item 4. Measurement of Viscosity of Liquid Cesium

(Elton H. Hall and John M. Blocher, Jr.)

The oscillating-cylinder method is to be used to measure the viscosity of liquid cesium. This technique was described in detail in a previous report.[5]

The apparatus used in the potassium work will be used for the cesium study. A new cylinder for containing the specimen has been fabricated from Nb-1Zr alloy. Final welding has been completed, and the cylinder leak checked with a mass spectrometer. This assembly will now be vacuum annealed at 2200 F for one hour. The measurements of the moment of inertia, period, and residual damping of the empty cylinder will then be determined prior to filling with cesium.

Some experiments have been conducted in which oscillation of the cylinder was initiated electromagnetically by means of the interaction of a pair of Helmholtz coils with the soft iron rod which is attached to the suspension. No improvement was noted over the initiation of oscillation by means of hand-held permanent magnets, as used in the potassium study.

During the next period, the cesium will be loaded into the cylinder and the measurements made.

Part IV - Item 5. Measurement of Thermal Conductivity of Cesium Vapor

(Joseph Matolich, Jr., and Herbert W. Deem)

These measurements are being postponed pending the outcome of the corresponding measurements for potassium vapor.

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(4) Matolich, J., Jr., and Deem, H. W., "The Thermal Conductivity and Electrical Resistivity of Liquid Potassium and the Alloy Niobium-1 Zirconium", Topical Report BATT-4673-T6, Battelle Memorial Institute (April 30, 1963).

(5) Hall, Elton H., and Blocher, John M., Jr., "The Viscosity of Saturated Liquid Potassium from 70 to 1150 C by the Oscillating-Cylinder Method", Topical Report BATT-4673-T1, Battelle Memorial Institute (August 31, 1962).

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(January 30, 1964)

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(a) First Quarterly Report (Covering the Period: October 1 through December 31, 1960), January 30, 1961.

(b) Second Quarterly Report (Covering the Period: January 1 through March 31, 1961), April 30, 1961.

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