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DENSITY OF METALLIC POLONIUM

(Information Meeting Paper No. 13)

~~Caution
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Date: November 9, 1948

Prepared by: B. B. Brody

Distributed: DEC 29 1948

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INTRODUCTION

Polonium has been reported to exist in two allotropic forms.¹ Evidence for this was gathered from resistivity measurements and crystal structure studies. X-ray diffraction studies show that the low temperature, α form has a simple cubic structure and a calculated density of 9.32 g./cc. whereas β polonium is a simple rhombohedral crystal with a calculated density of 9.51 g./cc. The density was also determined by measuring the volume which a known mass of metal occupied in a small capillary. The weight of polonium, 1201 ± 6 micrograms was determined calorimetrically. With this weight, a diameter of 0.258 mm. for the capillary and a length of 2.44 mm. for the "ingot", the density of polonium was calculated to be 9.38 g./cc. Only one determination was made because it essentially checked with the more accurate X-ray data.

More recent X-ray data show a density for α polonium equal to 9.16 g./cc. These data also show a coefficient of thermal expansion equal to 20×10^{-6} cm./cm./ $^{\circ}$ C.²

It was felt that further work should be done on this problem to confirm or dispute the values reported.

SUMMARY AND CONCLUSIONS

Four determinations of the density of polonium at 25 $^{\circ}$ C. have been made. These values are 8.69 g./cc., 9.34 g./cc., 9.34 g./cc., and 9.20 g./cc. with a probable experimental error of two per cent, 0.19 g./cc. The material used in the first determination was not subjected to a preliminary purification which makes the value obtained doubtful. In this instance production foils were used directly without a separation from their more volatile constituents. The difference in the reported value from that which appears in the Ad Interim Report³ is due to a recalculation based on the latest information on the physical constants of polonium.⁴

The value of using pure material and knowing the physical properties of polonium are clearly indicated.

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EXPERIMENTAL

The procedure used in making the density determinations was essentially the same in each case with the exception of the one low value reported.

About 25 curies of polonium were placed in a quartz tube which had another tube of about 4 mm. internal diameter sealed to it (Figure 1). The system was evacuated to a pressure of 10^{-5} mm. Hg or better and the portion of the tube containing the polonium was heated with an oxygen torch until the polonium collected on the cooler portions of the tube's walls. The pumps were permitted to continue the evacuating operation during this step in order to pump off any oxygen which might be formed as a decomposition product of the oxide. The tube was then sealed under high vacuum and the polonium concentrated by heating all but the very tip in a furnace at 500°C . A ring of inactive material was visible toward the cooler end of the tube followed very closely by the polonium. The temperature of the furnace was then lowered to 300°C . and the tube placed in the furnace with the larger portion protruding. This temperature was high enough to drive a good bit of the impurity off and low enough to leave a sufficient quantity of polonium behind to be used in the experiment.

The purity of the material finally used in the experiment was not known but should be reasonably high in view of the treatment to which it was subjected.

The tube containing the polonium was sealed off and the activity driven to one end of it. It was then broken open and placed in another tube to which a calibrated capillary was attached (Figure 2). Care was essential at this point to keep small quartz chips from getting into the calibrated space.

This second tube was evacuated and the polonium heated with an oxygen flame in a fashion similar to its preliminary treatment. After it had cooled, the pumps were turned off and the system filled to about 100 mm. pressure with helium from which oxygen was removed by passing the gas through copper turnings at about 400°C . The tube was then sealed off from the rest of the system and placed in a furnace at 500°C . in order to move the polonium past a constriction in the direction of the capillary. In this way a separation from lead which has a vapor pressure of about 10^{-5} mm. at 500°C . compared with about 1 mm. for polonium at 500°C . is

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effectuated.⁵ After cooling, the constriction was closed and the tube again heated at 500°C. to drive the polonium into the smaller bulb to which the capillary was sealed. It was sealed off again and removed to a centrifuge cup specially designed to permit heating the sample just before centrifugation (Figure 3).

The temperature of the cup containing the tube in which the activity was enclosed was raised to about 400°C. The temperature was well above the melting point of the polonium. The centrifuge was permitted to spin at 2000 r.p.m. for about twenty minutes. During this time the polonium was able to collect in the calibrated tip and the cup cooled to below the melting point of the polonium. The length of the metallic thread was measured by means of a traveling microscope. Where bubbles were found in the thread, a measurement of the base and an estimate of the height permitted a correction to be applied.

The weight of polonium was determined calorimetrically.

CALIBRATION OF CAPILLARY

A heavy walled quartz capillary was drawn out by heating a piece of quartz tubing to thicken it and then exerting a fairly uniform but fast pull on either end of the hot portion. This resulted in a slightly tapered capillary which was then cut at a reasonable distance and sealed at the small end. Only those of about 0.2 mm. diameter were used for further work. The tube was weighed empty and with a series of small droplets of mercury on an Ainsworth Type FDJ Microbalance. The droplets were forced individually to the bottom of the capillary by centrifugation and were subsequently measured in length by means of a calibrated traveling microscope. The volumes obtained in this fashion were corrected for the mercury meniscus and plotted against the length of the mercury thread. When a sufficient number of points were obtained, the tube was inverted and centrifuged in order to remove the mercury from the tip.

CALCULATIONS AND ASSUMPTIONS

In making the calculations it was assumed that the temperature of the thread was about 50°C. above the temperature of the surroundings on the basis of an observation that the furnace temperature was only about 200°C. when a mass of polonium melted.⁵ When a thermocouple is placed directly in the metal it is observed to melt at about 254°C.¹ This and

a value of 20×10^{-6} cm./cm./°C. for the coefficient of linear expansion were used to calculate the length of the thread at 250°C., close to the observed melting point of polonium.² The assumption was also made that at the melting point and during centrifugation the molten metal completely filled the end of the capillary. Also, that it behaved like a normal metal in contracting when it solidified and cooled further to the temperature at which the length was measured.

The observed length was corrected back to the length close to the melting point 250°C., and the volume at that temperature was read from the calibration curve for the capillary. This volume and the calorimetric determination of the weight of polonium is necessary to calculate the density at 250°C. This is then corrected to the density at 25°C. The formulas used are:

$$l_{t'} = l_t (1 + \alpha (t' - t))$$

$$D_{t'} = M/V$$

$$D_{25} = \frac{D_{t'}}{(1 - 3\alpha (t' - 25))}$$

l = Length of polonium thread.

t' = 250°C.

t = 75°C.

α = Coefficient of linear expansion.

M = Mass of polonium - gm.

V = Volume of polonium - cc.

D = Density.

POSSIBLE SOURCES OF ERROR

1. Calibration of Capillary

A smooth curve may be drawn through the points obtained with mercury. The deviation from the curve may be used to assign a value for the error in the volume determination. A typical calibration curve is shown in Figure 4.

2. The error in measuring the length of the thread for these experiments is determined by the sensitivity of the traveling microscope.

3. An error may be present in making the temperature correction. If it is assumed that this correction is good to twenty per cent, the error is correspondingly reduced.

4. The error in the calorimetric determination of the weight of polonium used is 0.2 per cent.⁶

5. In a couple of cases one or two bubbles appeared in the thread.

These were assumed to be spherical segments and with the measurement of the base and an estimation of their height, a correction could be applied. It appeared that all the bubbles present were observed, although the small possibility exists that some were present within the thread and escaped observation.

If the polonium sample is essentially 100 per cent pure then the overall error in such a determination should be about two per cent.

RESULTS

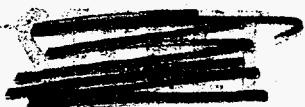
The essential data are listed in the following table:

Length of Thread (mm.)	Volume at 250°C. (cc. x 10 ⁻⁵)	Wt. Polonium (mg.)	Density at 25°C. g./cc.	Possible Error g./cc.
4.273	14.59	1.249	8.69	± 0.19 (known to be impure)
7.734	20.22	1.866	9.34	± 0.14
8.047	35.32	3.253	9.34	± 0.21
9.447	29.42	2.670	9.20	± 0.17

A fairly typical thread of the metal in a capillary is shown in Figure 5.

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From the way in which these determinations were made the value reported is that for polonium in the β modification provided the previous observations on the transition from β to α polonium are correct.¹

On the basis of the last three determinations one may say that the density of polonium is 9.29 g./cc. with a standard deviation from the mean, $\sigma = \pm 0.07$ g./cc. and a probable error equal to ± 0.06 g./cc. for a single determination.

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1. C. R. Maxwell and W. H. Beamer, Report No. LA-604 Physical Properties and Crystal Structure of Polonium, August 30, 1946.
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3. E. B. Brody, Report No. MLM-103, Density of Metallic Polonium-Ad Interim, May 18, 1948.
4. R. A. Staniforth, Information Report No. 37, Fundamental Constants and Conversion Factors, December 31, 1947.
5. R. Davis and L. Brocks, Physics Group Progress Report, November 16-30, 1946.
6. D. Anger, Calorimetry Research Group Progress Report, October 1-15, 1946.

The following illustrations are the slides used in presenting this paper at the Information Meeting.

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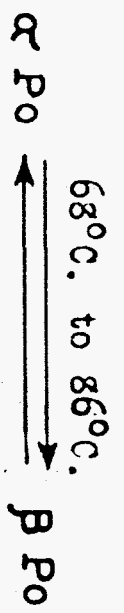
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REPORTED PHYSICAL PROPERTIES

REF 236



DENSITY

LOS ALAMOS

X-RAY DENSITY

9.32 gm./cm.³

9.51 gm./cm.³

DIRECT DENSITY

9.38 ± 0.5 gm./cm.³

MOUND

X-RAY DENSITY

9.16 gm./cm.³

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THERMAL EXPANSION

LOS ALAMOS

- 300 ± 100 x 10⁻⁶ cm./cm./°C.

MOUND

+ 20 x 10⁻⁶ cm./cm./°C.

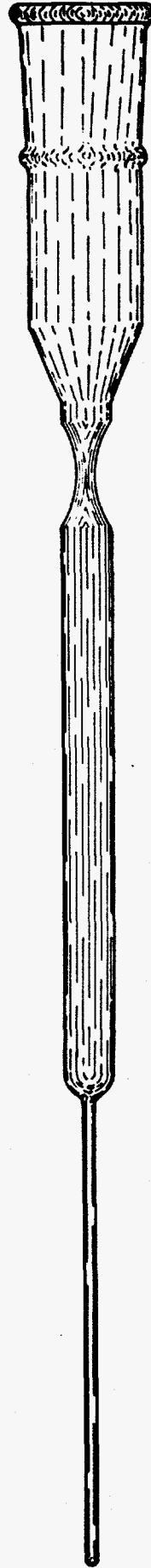
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FIGURE 1

QUARTZ TUBE USED IN PRELIMINARY PURIFICATION OF
POLONIUM



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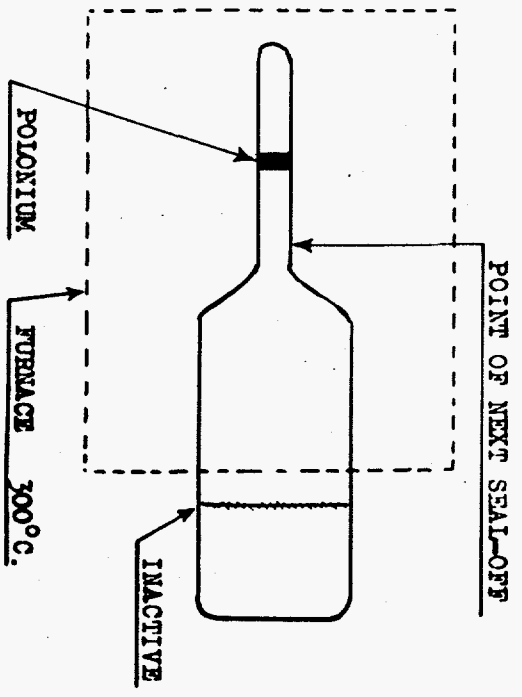
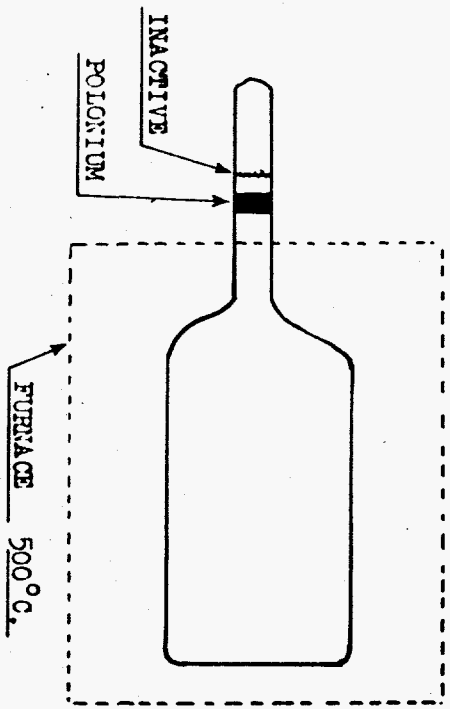
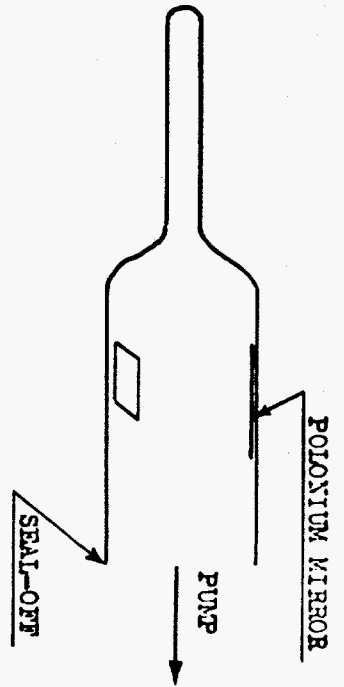
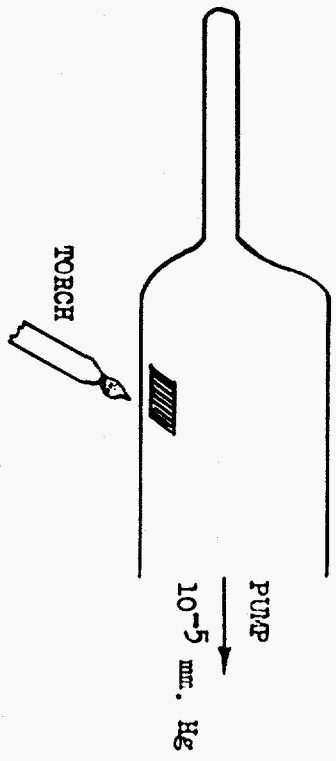
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FINAL POLONIUM PURIFICATION

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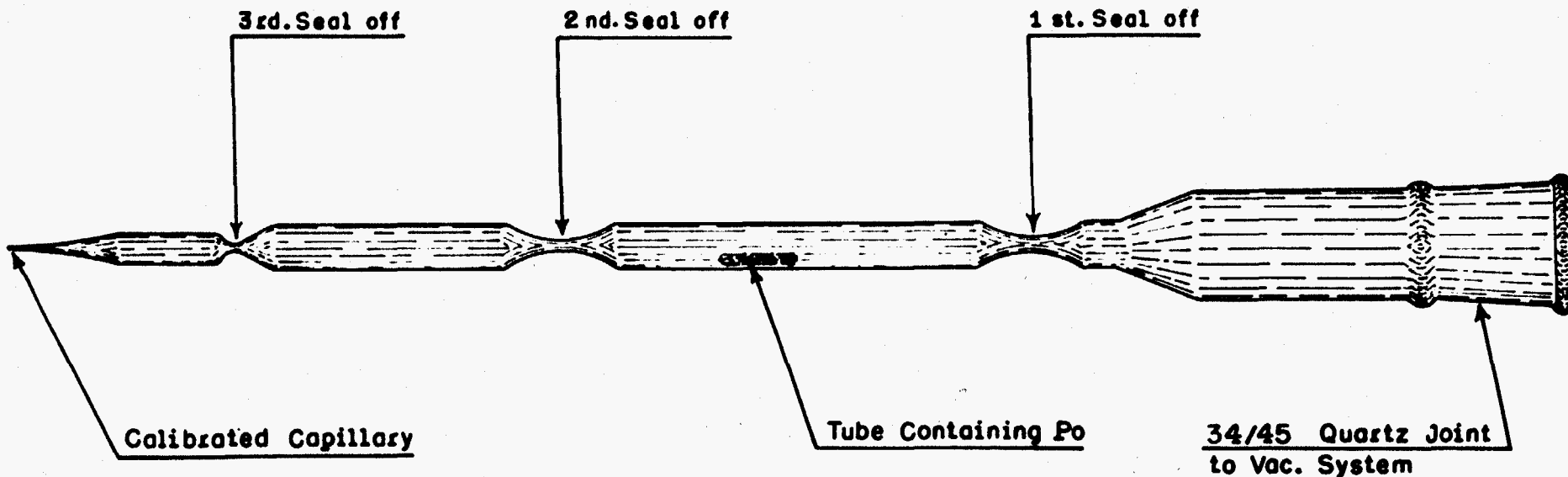
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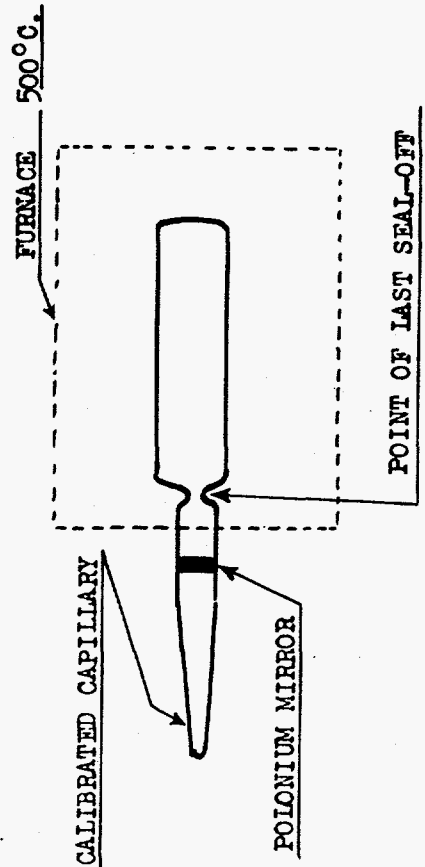
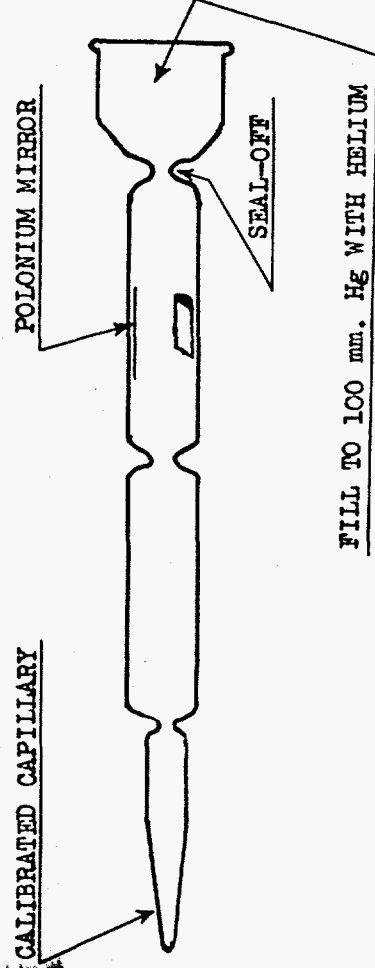
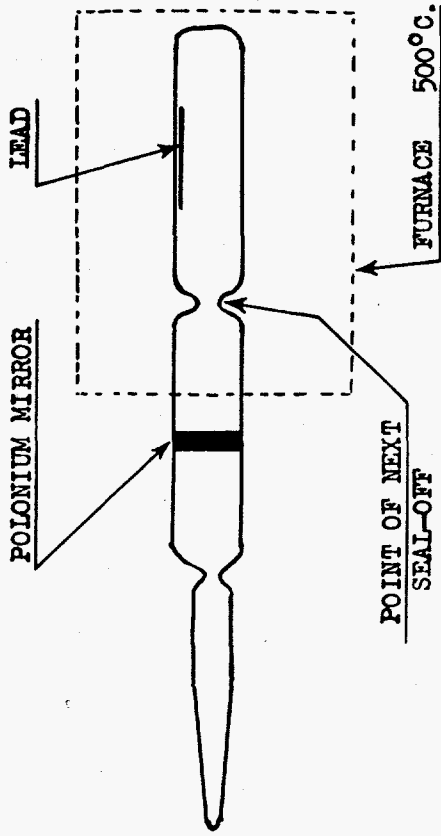
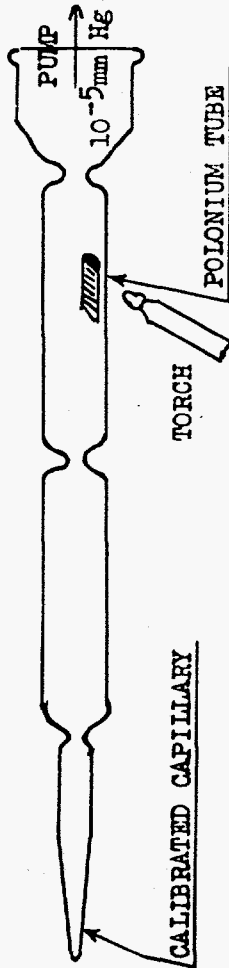
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—■— FIGURE 2 —■—
QUARTZ TUBE USED IN DENSITY DETERMINATION



TRANSFER TO CAPILLARY



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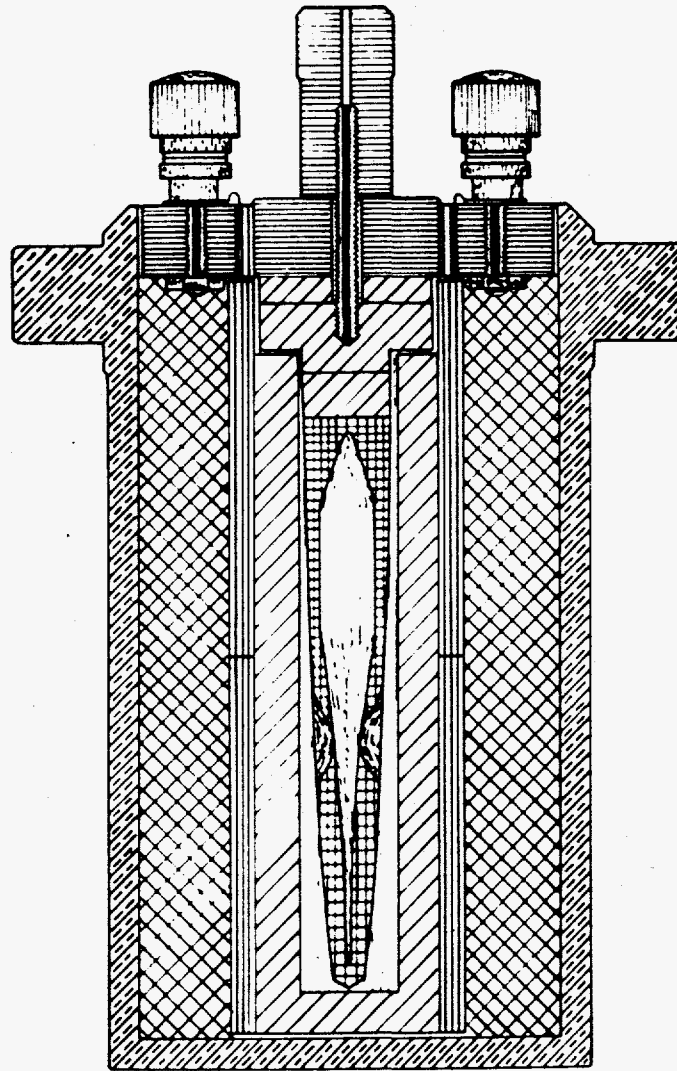
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— **FIGURE 3** —







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CENTRIFUGE CUP EQUIPPED WITH FURNACE

— SCALE: FULL SIZE —



— LEGEND —

-  Brass Centrifuge Cup
-  Asbestos Insulation
-  Nichrome Wire Furnace in Thermocouple Insulators
-  Iron Tube and Cover
-  Transite Insulator
-  Modified Pyrex Centrifuge Cone to hold Quartz Capillary

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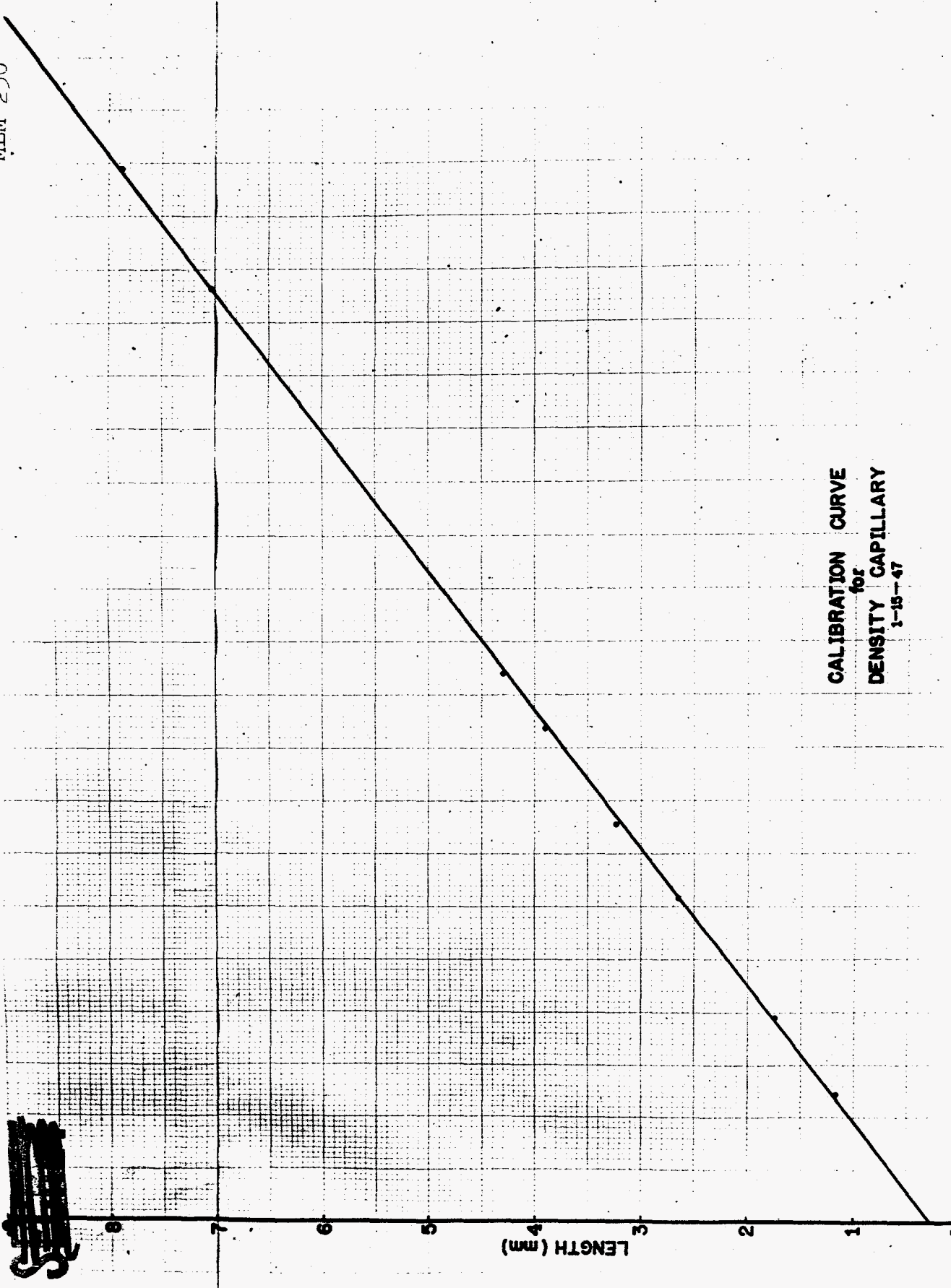
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CALIBRATION CURVE
for
DENSITY CAPILLARY
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10

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Volume $\times 10^3$ cc.

-16-

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CALCULATION OF DENSITY

ASSUMPTIONS:

1. POLONIUM THREAD 50°C. ABOVE SELECTED STANDARD TEMPERATURE.
2. COEFFICIENT OF LINEAR EXPANSION $+20 \times 10^{-6}$ cm./cm./°C.
3. MELTING POINT OF 250°C. FOR POLONIUM.

FORMULAS:

$$1. l_{t'} = l_t [1 + \alpha (t' - t)]$$

$$2. D_{t'} = \frac{M}{V}$$

$$3. D_{25} = \frac{D_{t'}}{[1 - 3\alpha(t' - 25)]}$$

l = LENGTH OF POLONIUM THREAD

t' = 250°C.

t = 75°C.

α = COEFFICIENT OF LINEAR
EXPANSION

M = MASS OF POLONIUM

V = VOLUME OF POLONIUM

D = DENSITY OF POLONIUM

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CALCULATION OF DENSITY

PROCEDURE:

MASS

- 1. DIRECT CALORIMETRIC DETERMINATION.

VOLUME

- 1. MEASURED LENGTH CORRECTED TO LENGTH AT 250°C.
- 2. VOLUME AT 250°C. READ FROM CALIBRATION CURVE OF CAPILLARY.

DENSITY

- 1. DENSITY CALCULATED AT 250°C.
- 2. DENSITY CORRECTED TO 25°C.

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POSSIBLE SOURCES OF ERROR

1. CALIBRATION OF CAPILLARY.
2. MEASUREMENT OF POLONIUM THREAD LENGTH.
3. ASSIGNMENT OF TEMPERATURES.
4. CALORIMETRIC DETERMINATION OF MASS.
5. "BUBBLE" CORRECTIONS.

IF THE POLONIUM SAMPLE IS ESSENTIALLY 100 PER CENT PURE, THEN THE
OVERALL ERROR IN SUCH A DETERMINATION SHOULD BE ABOUT 2 PER CENT.

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RESULTS

THREAD LENGTH (mm.)	VOLUME 250°C. ($\text{cm.}^3 \times 10^{-5}$)	WEIGHT (mg.)	DENSITY 25°C.	ESTIMATED ERROR
7.734	20.22	1.866	9.34	± 0.14
8.047	35.32	3.253	9.34	± 0.21
9.447	29.42	2.670	9.20	± 0.17
AVERAGE				± 0.10

PRECISION INDEX --- PROBABLE ERROR ± 0.06

