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MOUND LABORATORY

Operated By

MONSANTO CHEMICAL COMPANY

MIANISBURG, OHIO

M. M. Haring
Laboratory Director

PHYSICS PROGRESS REPORT

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PHYSICS GROUP

R. E. Brocklehurst, T. I. Davenport, T. E. Eyles, L. G. Fauble, E. R. Manring, and H. C. Morgan

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ABSTRACT

1. Measurements on the vapor pressure of polonium and the polonium-lead compound have been deferred until after the move to the Mound Laboratory. The foils containing the activity had some impurities present. Evidence to prove that the impurity is carbon is presented.
2. Tests on the furnace of the vapor-pressure-by-effusion apparatus disclosed that the maximum temperature available was too low. A new furnace designed to reach a higher temperature has been constructed.
3. No work was done this month on the preparation of the X-ray target.
4. The technique devised for the preparation of pure samples to be weighed with the vacuum balance was tested and is deemed unsatisfactory. The quartz tubes containing activity to be weighed in the vacuum balance do not behave like charged dipoles.
5. No work was done directly on the resistivity of polonium.
6. Attempts to make polonium gamma standards failed.
7. The X-ray diffraction analysis of a clogged pinhole in a tantalum sheet indicated that the material deposited in the hole was amorphous and probably carbonaceous. The time study of the decay of polonium has produced a new pattern, perhaps indicating reaction with the Pyrex container.

DETAILED REPORT

The Vapor Pressure of Polonium and Its Compounds with the Quartz-Sickle Gauge - E. R. Manning, 1.

A sample of about 13.9 curies of polonium was prepared for the experiment described in MLM-243.¹ The sample was weighed in the vacuum balance by H. C. Morgan to determine the purity of the sample. The sample and apparatus have been set aside, and the experiment will be continued at Mound Laboratory.

In the preparation of a sample, November 23, 1948, a low vapor-pressure impurity was observed. The sample consisted of two production foils. The foils were placed in a preparation tube and heated to about 250°C. while evacuated. The tube was then sealed off, activity torched from the foils onto walls of the tube, the tube placed in a furnace to drive all activity to one end of the tube, and this portion sealed off. The glass containing the activity was opened and dropped into a second tube. Upon evacuating this second tube, a band of substance, color light brown, was observed on one portion of the tube where the quartz

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surface was crystalline. When this band was heated lightly with a torch to perhaps 400°C., the band became black. Further torching to temperatures well above 800°C. did not affect the band. Air was admitted to the system, and the band disappeared instantly when it was warmed slightly with a torch. It is believed that the impurity was organic, that it adhered to the surface presented by the crystalline quartz although its vapor pressure was low, that heating caused the substance to break down into carbon, and that upon admission of air the carbon oxidized to CO or CO₂.

Vapor Pressure of Polonium by Effusion - L. G. Fauble, 1.

Tests have been run on the furnace described and pictured in MLM-243.¹ These tests have shown that a furnace such as described will not attain the temperatures required for the experiment.

A new type furnace, which it is believed will overcome heat losses encountered in the old furnace, has been designed and constructed. The heating elements in the form of "hairpin" loops pass through the copper block. In addition, a polished aluminum reflector will surround the copper block to help cut down radiation losses.

Work on this problem has been temporarily suspended because of a serious activity spill in the room containing the apparatus. This necessitated destruction of all the glass portion of the apparatus. Since the laboratory is being moved to Mound Laboratory, no additional work on the problem will be carried out at this site.

Preparation of a Target for the Determination of the Primary X-ray Emission Spectrum of Polonium - L. G. Fauble, 0.

All of this month's time was devoted to the vapor-pressure-by-effusion experiment.

Purity of Polonium by the Vacuum-Balance Method - H. C. Morgan, 1.

In MLM-243¹ the factors affecting vacuum-balance operation were discussed. Factor 10, which discussed the possibility that the quartz activity tube might be a charged dipole, has been investigated experimentally. The experimental procedure was quite simple. Since the distribution of the polonium in the tube was not symmetrical, as evidenced by the fact that most of the blue glow was concentrated in one-half of the tube, a weight differential should occur if the tube is charged when the tube is successively hung in two different positions from the pan hook. One set of weighings was made with the active end of the tube hanging down (tube slanting at an angle of about 30 degrees), and a second set of weighings was made with the active end up. If the tube were a charged dipole,

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such a position change would have reversed the induced charges on nearby objects. A change in both the zero point and capsule weight would have then been observed. There was no difference in weight or zero point observed. Therefore the capsule probably is not appreciably charged.

A fourth purity run was started according to the procedure outlined in MLM-113.² The polonium was in a sealed quartz tube with one end a thin bubble (Figure 1), and had been weighed and calorimetrored (Steps 1, 2, and 3).

Figure 1

The thin end of the capsule was to have been punctured in a drybox filled with dry nitrogen and supplied with rubber-glove ports so that the operator could hold the quartz tube up to the microflame and allow a hole to be pushed through. Access to the drybox was through an air lock which opened into a hood with an exhaust fan at the top, exhausting the hood air to the roof of the one story laboratory. The dry nitrogen after flowing through the drybox also was exhausted into the top of the hood. Dry nitrogen was allowed to flow through the drybox for about two hours so as to flush out all oxygen.

Step 4 in the assay procedure was then attempted. The thin end of the quartz tube was brought up to the microflame; and the quartz became red and then pushed in some. Careful examination of the tube end through the glass window seemed to indicate that a hole had not been formed. The tube end was then twice heated to white heat, but this only seemed to make the quartz thicker. Finally the quartz was pierced with a sharpened tungsten rod which was in the drybox. The quartz tube containing the polonium was then placed in a corked quartz distillation tube and was passed through the air lock to an operator wearing rubber gloves. After the dry-box operator removed his arms from the glove ports, he went to a Zeuto on a desk six feet away from the air lock and drybox. This Zeuto indicated full-scale deflection on the 1 x scale, or about 80,000 disintegrations per minute. It was thought that this meter was inoperative, since it was not suspected at that time that the air would contain that much activity. The distillation tube was placed on the vacuum line, and the vacuum pump started. The operators left the room to check their clothes for activity. A high level of activity was found, and so a survey crew was called to check the room. The air in the whole room (volume about 5,000 cubic feet) by a rough

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approximation must have contained about 50 millicuries. A calorimetric check of the activity lost revealed a difference of 0.75 curie between before and after the experiment.

It is apparent from the description of the experiment, that the puncturing of the tube with a microflame is a poor technique. Even if the activity is prevented from getting into the same room where the operators are working, the work area will become very hot. This would necessitate frequent cleaning of the area to prevent apparatus contamination.

The only explanation that seems to fit the observed facts is that the thin end of the activity tube broke through and then resealed. The hot gases from the flame washed over the activity and either formed a volatile compound or caused the polonium to disperse in a finely-divided form which could be suspended in the air or as a vapor.

A new assay procedure will be worked out and reported in the near future.

Resistivity of Polonium - H. C. Morgan, O.

Neutronics - T. I. Davenport and T. E. Kyles, 2.

During the period of this report neutron measurements were made of 32 sources for the Neutron Source Group. Eleven measurements were made of gamma emission on sources submitted by various groups.

In order to calibrate the LARC-gamma geometry for the assay of micro-foils and other sources in the activity range of 0.1 to three curies, we obtained 11 microfoils with a total value of approximately three curies. These were transferred to glass vials six centimeters long and 0.7 centimeter in diameter. However, these vials became contaminated and a retransfer was required. We planned to have calorimetry assays made on these foils and then to use them as standards for the calibration and for the evaluation of the precision of the instrument. However, before the calorimetry assay had been completed, the holder containing the vials was dropped. Breakage and subsequent contamination postponed further work.

X-rays - R. E. Brocklehurst, 1.

X-ray diffraction patterns of a pinhole in a tantalum sheet, submitted by W. F. Pesold, indicated that the unknown material which filled the hole could not be crystalline. Comparison with a pattern of a clean pinhole showed definite indications of an amorphous halo which is suggestive of carbonaceous material.

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Patterns of Sample No. 51 show that a compound in addition to PbPo is being formed. This new compound crystallizes in a cubic system with a fairly large unit cell, $\sim 7.44 \text{ \AA}$. This new compound has not been detected in any of the previous samples. Since Sample No. 51 is contained in a Pyrex capillary instead of fused quartz, this compound may be due to a reaction of polonium with some constituent of the glass.

REFERENCES

1. Eichelberger, J. F., Physics Progress Report, MLM-243, Mound Laboratory, Miamisburg, Ohio (November 1-30, 1948).
2. Knauss, H. P., Physics Progress Report, MLM-113, p. 11, Mound Laboratory, Miamisburg, Ohio (May 1-31, 1948).

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