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Classification changed to UNCLASSIFIED
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by Carleton L. Lewis 9/10/20
Reviewed by C.W. Huntington 8/21/79

DETERMINATION OF THE PURITY OF POLONIUM BY MEANS OF THE VACUUM BALANCE

(Ad Interim Report)

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Date: March 8, 1949

Prepared by: H. C. Morgan

Distributed: APR 11 1949

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INTRODUCTION

The assay technique is discussed in detail. The results of three purity determinations are: 94.92 per cent, 100.50 per cent, and 106.9 per cent. The errors present in each determination are stated. The inherent dangers in the present procedure are pointed out, and an improved method is outlined.

SUMMARY AND CONCLUSIONS

It is important in certain research problems to know the purity of the radioactive material used in the experiments with an accuracy commensurate with the requirements of the problems. Three such problems which are of immediate interest are:

1. Vapor pressure of polonium.
2. Resistivity of polonium.
3. X-ray lattice constants.

The purity problem was discussed and the present program was initiated by Dr. Ray Davis, Jr.¹

A technique for assaying the purity of polonium accurate to ± 0.1 per cent or better is needed. Present methods are accurate to about 5 to 10 per cent and are not capable of measuring large quantities of polonium (10-50 curies). Recent half life measurements are precise to 0.02 per cent. The main problem is to devise a method of weighing the sample to 0.1 per cent. Direct weighing of the sample within this limit on a microbalance can be accomplished if the following two effects from self heating of the sample are eliminated:

1. Convection air currents inside the balance case.
2. Temperature gradients in the balance parts (particularly the balance beam).

The first effect can be eliminated by weighing the polonium (sealed in a tube) in a vacuum, and the second can be greatly reduced by radiation shielding.

A technique for weighing radioactive samples in a vacuum to the required accuracy has been worked out by the author² for samples as large as 50 curies weighing 11.1 milligrams.

The initial assay procedure is outlined below:

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1. Place the sample of polonium to be assayed in a quartz tube of the size and shape shown below.

2. Calorimeter the tube for the quantity of polonium, accurate to 0.2 per cent.

3. Scratch the tube at Point A and weigh on a microbalance (Ainsworth) in an evacuated case.

4. Break the tube at Point A (in helium atmosphere) and place the two pieces of the tube inside a quartz tube that can be placed on a vacuum line.

5. Distill the polonium out of the weighing tube onto the walls of a containing tube.

6. Remove the two pieces of the weighing tube and reweigh.

The above procedure, Steps 3 to 6, was first checked with a weighed piece of tellurium. The weight of the sample obtained from this treatment agreed with the initial weight of tellurium to 0.05 per cent. When the tube was broken, care was taken to retain chips of quartz which might have been formed. The tube was broken on a piece of black transfer paper. However, when polonium was used instead of tellurium, difficulties were encountered. A dry-box filled with dry nitrogen in place of helium was used. It was found to be quite difficult to break off the tip, through glove ports, without the formation and possible loss of chips. Two purity runs were made using this technique. On the first run the purity was 94.92 per cent, and on the second run of the same sample the purity was 100.50 per cent. Details of a run with a sample calculation are found at the end of this report.

In retrospect there were several serious sources of errors in these two experiments. Several chips were lost which should decrease the purity, as in the 94.92 per cent result; but the chip loss cannot explain the 100.50 per cent result. These purity runs were done while the errors in the vacuum balance were still being investigated and were done more to check the procedure of purity assay than anything else. It is now known that a serious radiometer effect was present which would introduce an error as large as five per cent. The pressure in the vacuum balance at this time was around 2×10^{-2} mm. of mercury, instead of the 1×10^{-4} mm. of mercury that was found to be necessary.

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It was thought that the possibility of error resulting from chip loss in Step 4 of the assay procedure could be eliminated by a change in procedure and in design of the sample tube. The redesigned tube is shown below:

A microflame could be applied to the thin end of the tube; and the end would fuse and be pushed into the tube since the capsule was evacuated. A hole should result.

Steps 3 and 4 would now read:

3. Weigh tube on an Ainsworth microbalance in an evacuated case.

4. Apply a microflame to the thin end of the tube in a dry nitrogen atmosphere, and allow a hole to be pushed through. Place pierced tube inside a new quartz distillation tube which can be evacuated.

The third and fourth purity runs were attempted using the altered procedure outlined above. The third purity run resulted in a 106.9 per cent calculated purity, and the fourth run was terminated by a serious activity spill. The third experiment used an activity capsule which had been used as a radioactive weighing sample in the vacuum balance testing. About two-thirds atmosphere of helium had built up in the tube from emitted alphas, and the thin end did not push in. The microtorch tip touched the capsule, probably resulting in a material transfer to or from the activity capsule. A pointed tungsten rod was finally used to pierce the tube end. In the fourth purity run, the microflame apparently pierced the tube end and vaporized or combined with some of the polonium in the activity capsule. It is known that three-fourths of a curie of polonium was lost and that because of a faulty exhaust hood over the drybox air lock, about ten millicuries of polonium was distributed in the air in the room ten minutes after the air lock was opened.

The details of the fourth purity run have been discussed in conference, and it was concluded that the microflame piercing method was too dangerous to use. Please refer to suggestions for future work at the end of this report for a recommended new method.

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Calculations

PURITY RUN NO. 1

Weight of Capsule Containing Sample: 3.964241 gms.
Weight of Capsule Without Sample: 3.952046 gms.
Weight Difference (Polonium + Lead + Impurities): 0.012195 gms.

To find weight of polonium + lead in capsule as determined by calorimeter:

Time between distilling polonium into capsule and calorimentering sample:

14 days, 1 1/2 hours.

Fraction polonium remaining after 14 days, 1 1/2 hours:³

0.931988.

Weight of polonium originally in capsule =
Weight of calorimetered value + decay lead:

$$\frac{48.485}{0.931988} \text{ curies} = 52.0232 \text{ curies.}$$

Weight of original polonium:

$$52.0232 \text{ curies} \times 222.517 \times 10^{-6} \text{ gms./curies} = 0.011576 \text{ gms.}$$

We may compute the purity by proportion:

$$\frac{0.012195 \text{ gm.}}{0.011576 \text{ gm.}} = \frac{100\%}{X\%}$$

$$X = 94.92\% \text{ pure.}$$

Suggestion for Future Work

It is obvious that the crux of the problem lies in piercing the activity capsule without losing fragments of the weighing tube or getting dangerous amounts of polonium in the air or on the drybox walls.

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Since there is at present a 7.5 cm. long by about 2.5 cm. diameter restriction on sample tubes which the calorimeters will accept, the proposed quartz weighing tube assembly diagrammed below will have to be separable.

A weighing tube assembly such as this can be cleaned separately, and the inner capsule which contains the polonium can be taken out to be calorim-etered. The inner capsule can be dropped on its frangible end (Point A) and thus opened. The outer tube should contain any fragments during weighing which are formed.

Acknowledgments

The author wishes to express his appreciation of the assistance of Dr. Ray Davis, Jr.¹ in outlining the research program and in the initial experimental work. E. R. Manring, L. G. Fauble, and H. W. Schamp, Jr. were also of great help during various phases of the theoretical and experimental work.

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