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MONSANTO CHEMICAL COMPANY - UNIT III

DAYTON, OHIO

M. M. Haring
Laboratory Director

PHYSICS GROUP PROGRESS REPORT

Date: May 1-31, 1948

Prepared by: H. P. Knauss

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PHYSICS GROUP

R. E. Brocklehurst, L. S. Brooks, T. I. Davenport, M. E. Harvill, H. P. Krause, H. C. Morgan, E. Orban (part time), and L. F. Vassamillet

ABSTRACT

The High Rate Neutron Counter is now in operation, giving one count per pulse received from the counting tube.

A new, improved, weighing procedure is described for the Vacuum Balance Assay Method.

Apparatus is being constructed to remeasure the vapor pressure of postum. A method is proposed to determine the molecular weight of postum vapor.

Samples No. 33 and 42 were run again as part of the long term study by the X-ray laboratory of Q-metal and Q-oxide. The study of single crystals is being continued.

The measurements of vapor pressure of postum by effusion continue to be inconsistent. The troubles may result from loss of activity by leaks.

DETAILED REPORT

Neutronics - T. I. Davenport and M. E. Harvill

Neutron counts were made on eleven sources for the Electrodeposition Group and on forty-six sources for the Neutron Source Group during the period of this report.

The malfunction of the High Rate Neutron Counter, explained in the report for the period, April 1-30, 1948, has largely been corrected. The reported changes in the trigger pair circuits resulted in single counting operation except for a warm up period during which sporadic double counting was observed. After the scaler was in operation for approximately one-half hour, the single counting action became consistent. This warm up effect was greatly reduced when the Electronics Group replaced three 16 MFD filter condensers in the 300 v. unregulated supply and the 5R4 rectifier tube.

Investigation is under way on a new type lead geometry for gamma counting. In this geometry, designed by Dr. H. P. Knauss, the source and the counting tube are separated by lead instead of air. This study has been delayed by repeated failures of the counting tubes.

Purity of Postum (Vacuum Balance Method) - H. C. Morgan

The vacuum balance apparatus has been altered so that a vacuum of 2×10^{-4} mm. of mercury has been obtained. Considerable difficulty has been caused by leaks, but most of them have been sealed. A vacuum of 1×10^{-2} mm. of mercury has been obtained with the Megavac fore pump only, after a twenty-four hour pumping period. A Distillation Products model C M-220 air-cooled oil-diffusion pump, using octoil S as the pumping fluid, reduced the pressure further to 2×10^{-4} mm. of mercury. This diffusion pump without a cold trap or baffles will reach 1×10^{-7} mm. of mercury as an ultimate vacuum. Therefore, some improvement in vacuum perhaps could be obtained, but it would require an excessive amount of work to find all of the minor leaks.

There has been some discussion as to the possibility of losing chips when the tip of the quartz tube holding the activity is broken off. Preliminary tests of the procedure, weighing the tube before the tip is broken and then weighing both tube and tip after the tip is broken off are quite promising. However, chipping does occur fairly often, which could introduce a weighing error if some chips are lost.

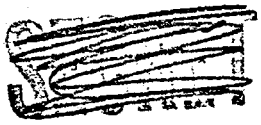
When a quartz tube full of activity (postum) was actually used, some chips resulted when the tip was broken off. A second purity run also resulted in some chips when the tip was broken off. Since it is difficult to collect all chips and then weigh them because of their small size and because the altered micro-balance has no provision to hold chips, an improved technique is needed. A proposed method is discussed under "Future Plans".

Resistivity of Postum - H. C. Morgan

The purity problem is to be solved before further work will be done on the resistivity of postum.

Vapor Pressure of Postum - L. S. Brooks

Apparatus is being constructed to remeasure the vapor pressure of postum with a quartz sickle gauge. A quartz sickle gauge similar to the one employed in April, 1948 has been constructed.



Another copper cylinder to enclose the gauge sample bulb is being machined since the hole along the axis of the copper cylinder used in April, 1948 was slightly off center. Two thermocouples have been calibrated with National Bureau of Standards standard freezing point samples.

X-ray Studies - R. E. Brecklehurst and L. F Vosseniller

A pattern of Sample No. 33, Q-metal, which is now approximately at 3 3/4 half-lives, was run. The UO_2, or Pb-Q compound, is present in diminishing amounts, and Pb is increasing, as expected.

Sample No. 42, Q-oxide, which previously had lost its crystalline structure, still did not produce any evidence of crystallinity.

The study of single crystals is continuing with the objective of obtaining all possible information for Laue patterns.

Vapor Pressure by Effusion - Edward Orban

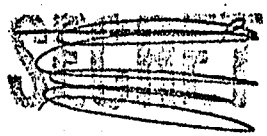
Table I shows the data taken for autoradiographs of selected foils from runs on the vapor pressure apparatus, reproduced in Figures 1 and 2. (See Physics Group Progress Report dated April 1-30, 1948). Column four gives the actual counts obtained on the foil; whereas, column five gives the counts calculated as the correct count if the vapor pressure had fallen on the curve obtained by L. S. Brooks. The counts found at low temperatures were higher than those found by calculation, while the actual counts at high temperature were much lower. Some anomalous effect was occuring at high temperatures which did not take place at low temperatures. This may have been due to the fact that there was not enough activity in the crucible to supply the amount needed to fill it. However, analysis of the quartz tube placed in the crucible showed the presence of 0.15 units in it after the completion of the experiment. No count was made of the material in the crucible.

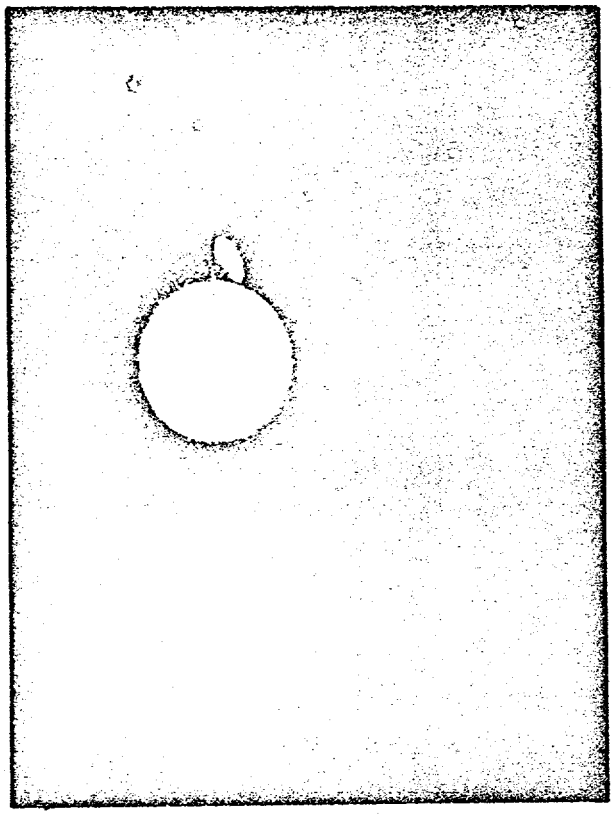
After the instrument had been used for a period of time, apparently pieces of activity began to fall on the foil causing the high counts at low temperatures. It is thought that this was caused by knocking off pieces from the walls when the steel ball, which was used as a shutter, was moved around the inside of the equipment. After the instrument had been in use for several days an appreciable amount of material collected on the walls.

Table I

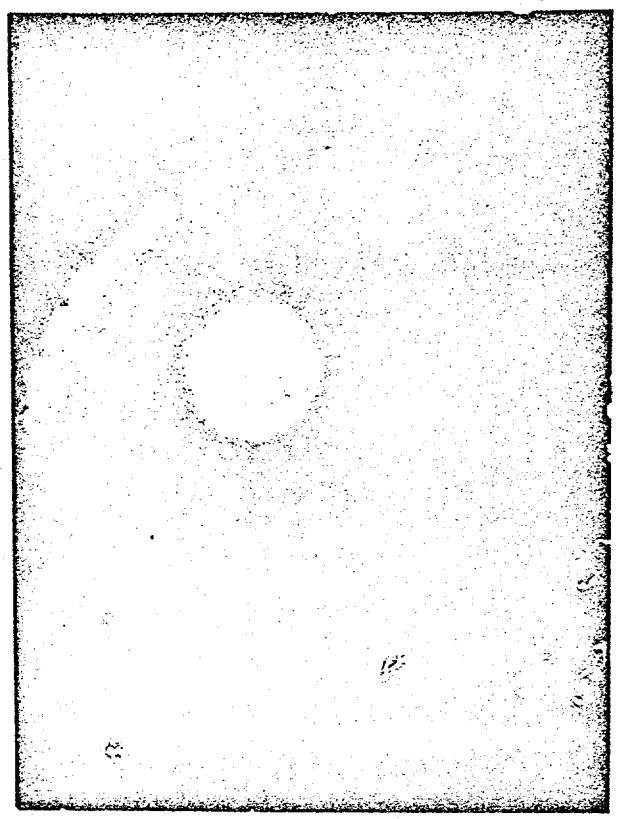
AUTORADIOGRAPH OF FOILS

Run No.	Cassette No.	Temperature °C.	Actual Counts/min.	Theoretical Counts/min.	Photographic Exposure Time	Remarks
3	8	114.1	13,584	10,605	17 hrs.	Good deposit with some very light spots, and a heavy spot on the side of the deposit.
15	25	339.0	41,512	4.92×10^7	7 hrs.	Outline of disc. Heavier circle. Smooth deposit in center.
22	A	152.8	1.00×10^6	1.35×10^4	10 min.	No deposit. Scattered heavy spots on foil.
23	30	154.0	2.77×10^4	1.34×10^4	7 hrs.	No deposit. Scattered heavy spots on foil.
26	23	220.3	4.37×10^5	2.12×10^5	25 min.	No deposit. Scattered spots on on foil.
29	4	263.3	3.44×10^3	4.22×10^6	4 2/3 days	Light deposit. Several scattered heavy spots.
34	?	151.0	2.44×10^4	2.18×10^4	7 hrs.	No deposit. Scattered heavy and light deposits.

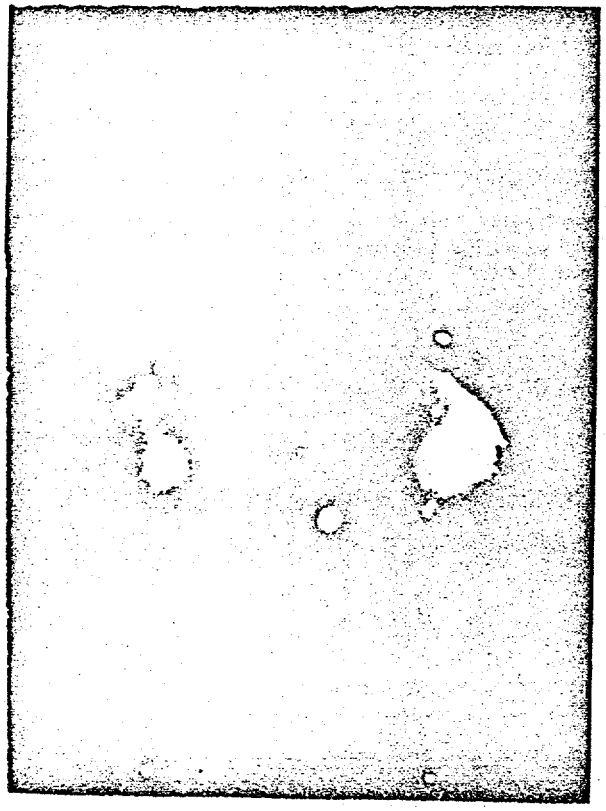




—RUN 3—



RUN 15



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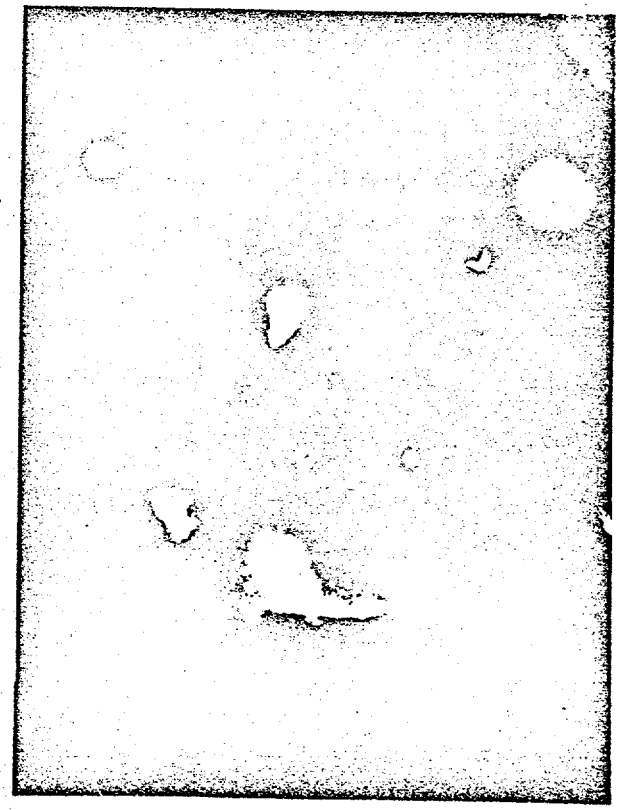
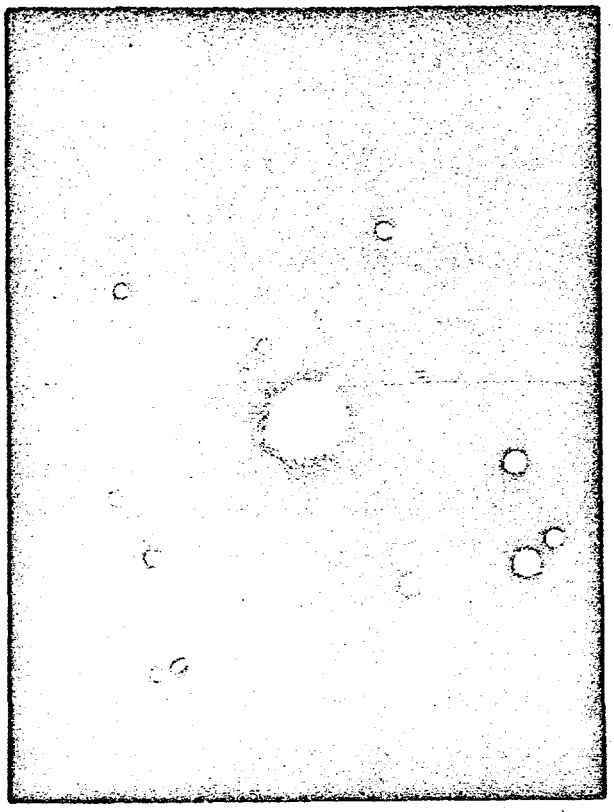


—RUN 23—

FIGURE 1



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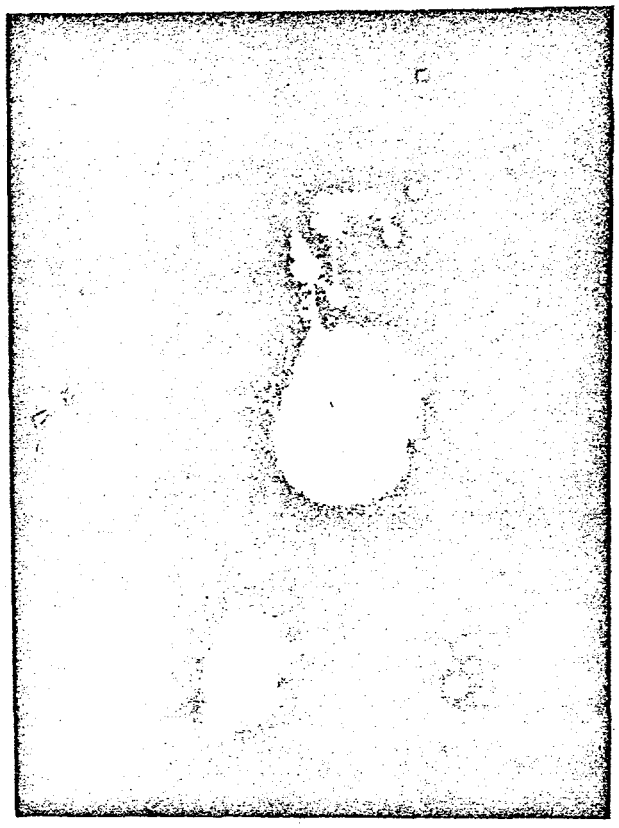

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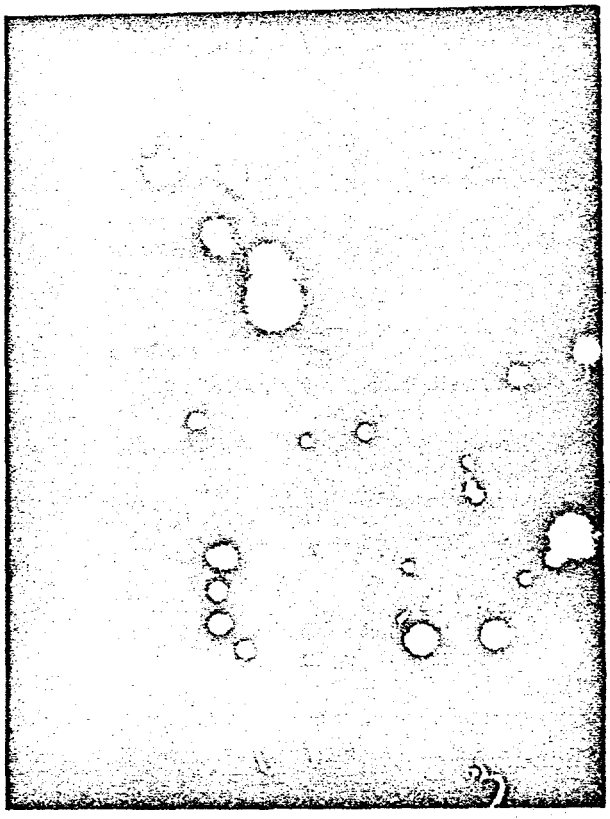

—RUN 34—

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—FIGURE 2—

In most cases the "hot" particles which fell on the foil were enough to cause the photographic emulsion to be reduced, but the deposit itself was too light to be observed.

Run 15 in Figure 1 shows the outline of the disc, which was caused by the background in the lower chamber; the smaller circle, caused by background from the upper chamber; and the deposit in the center.

Calculation showed that only 1/160 of the material present would be required to fill the crucible at the highest temperature. There was a brownish deposit all over the inside of the crucible indicating that the material had diffused out of the quartz tube. The orifice appeared to be open, but there was evidence that the crucible had had a leak other than the orifice. It is possible that the material leaked out before the measurements at high temperature could be made; thus, giving the low results at high temperatures and the more nearly correct results at low temperatures.

Experimental work on this problem will be resumed by L. G. Feuble.

FUTURE PLANS

Molecular Weight of Postum - L. S. Brooks

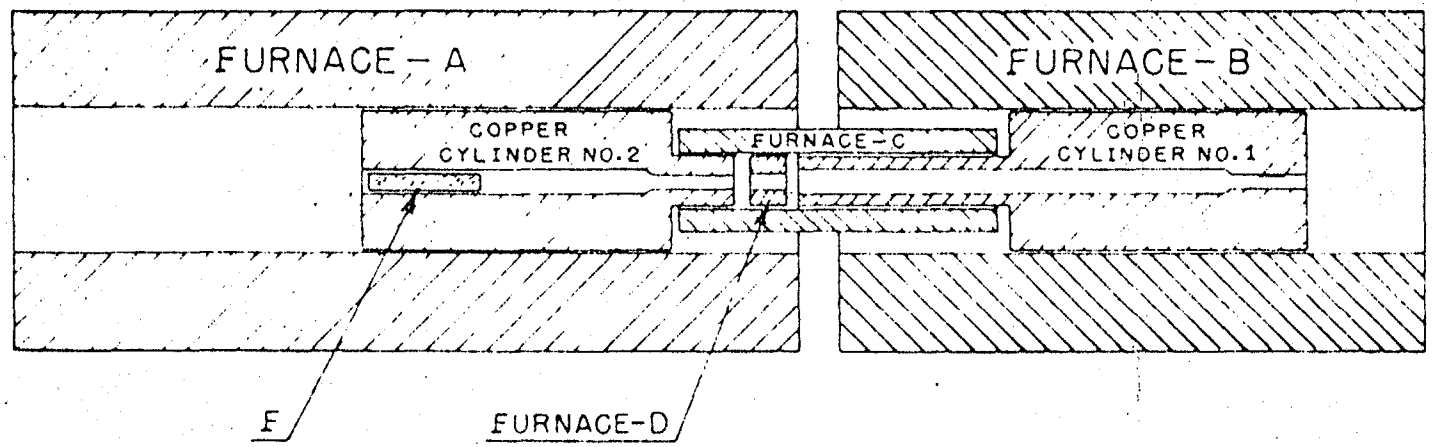
By using the results of the study of the vapor pressure of postum and a slightly modified version of the quartz stickle gauge apparatus, it appears possible to determine the molecular weight of postum gas.

The proposed apparatus is shown in Figure 3. The temperature control apparatus would be the same as that used for the vapor pressure measurement except for the addition of copper cylinder No. 2 and the seal-off furnace D. The proposed quartz vessel AE in Figure 3 consists of a chamber AB, with a measured internal volume to collect a sample of the gas. This is connected by a capillary tube BD to a sample bulb DE for containing the postum metal in the gaseous and liquid states. The quartz vessel fits inside the temperature control apparatus shown in Figure 3. The copper plug F is removeable so that the quartz vessel can be slid into the copper cylinders after they are positioned in the furnaces.

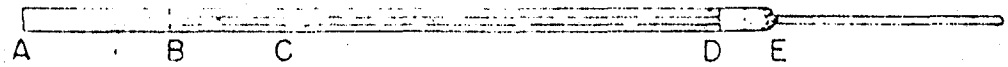
Determination of the molecular weight of postum gas will be limited to temperatures above the lowest temperatures at which the vapor pressures are known. The experimental procedure will be outlined with reference to Figure 3. After the postum metal is admitted

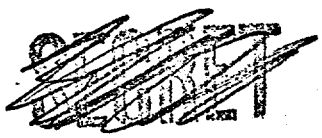
FIGURE 3

TEMPERATURE CONTROL APPARATUS FOR MOLECULAR WEIGHT DETERMINATION



QUARTZ VESSEL FOR MOLECULAR WEIGHT DETERMINATION





to tube AE through a side tube on tubing CD and the side tube is sealed off, tube AE will be slid into the temperature control apparatus through the opening for the copper plug F, and the copper plug will be replaced. The temperatures of the furnaces will be adjusted so that the liquid and gaseous postum in the sample bulb EE will be at a temperature for which the vapor pressure has been measured, the gas in the tube AE will be at some temperature about 50°C. or 100°C. above the temperature of the sample bulb, and the tubing ED will be at a temperature above that of the sample bulb. With the tube AE in copper cylinder No. 2 at a uniform temperature along its length, the quartz tubing at C will be sealed off with the seal-off furnace D. The volume of capillary tubing in the vicinity of the seal-off position C will be small in relation to the volume of the tube AE so that the sealing off operation will not change appreciably the temperature of the main volume of gas in tube AE. Then the quantity of postum sealed in tube AE can be measured with a calorimeter. As shown in the following list there will be enough experimental data to determine the molecular weight of gas from the equation of state:

P = vapor pressure corresponding to the temperature measured in the sample bulb.

V = internal volume of tube AE which was calibrated.

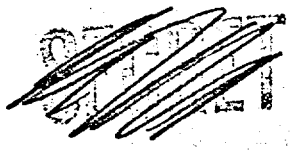
T = temperature of the tube AE at the time the capillary tubing was sealed off at C.

M = mass of postum present in tube AE as determined by the calorimetry.

Weighing Procedure for Macroassay - E. C. Morgan

The following procedure will be tried for macroassay of purity. A quartz tube with a thin walled bulb on one end will be made, evacuated, filled with postum, and sealed off. When the time comes to distill the postum out of the quartz tube, a micro flame will be applied to the bulb. Since the tube is evacuated, a hole will be pulled through the bulb from which the activity can then be distilled. Most of the quartz in the area fused by the micro flame will be in a collar around the hole. Any thin quartz section broken off from the fused area should be carried well within the tube by the inrush of air.

The procedure is summarized:



1. Place postum to be assayed in a quartz tube of the size and shape as shown in Figure 4.

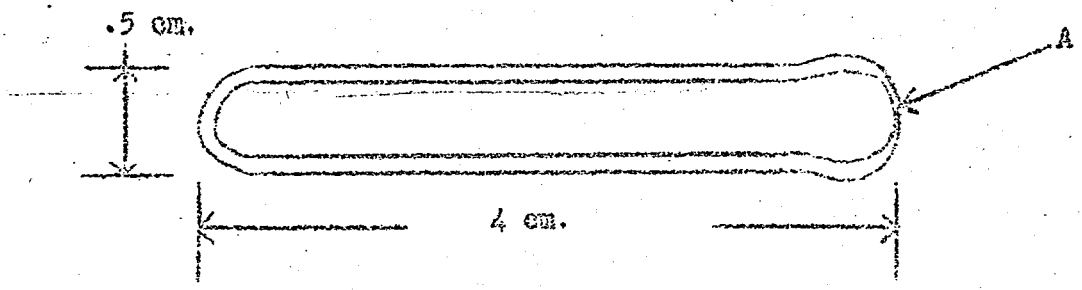


Figure 4

2. Calorimeter the tube for the quantity of postum accurate to .1 per cent.

3. Weigh tube on micro balance (Ainsworth) in an evacuated case.

4. Apply a micro flame at point A in a dry nitrogen atmosphere and allow a small hole to be pulled through bulb.

5. Place weighing tube inside a quartz tube that can be placed on vacuum line, then evacuate and vacuum-distill postum out of weighing tube onto the wall of the containing tube.

6. Remove weighing tube and reweigh in the evacuated balance.

7. Reseal and recalorimeter the weighing tube to check the efficiency of distillation.

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