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MONSANTO CHEMICAL COMPANY - UNIT III

PROGRESS REPORT

January 16-31, 1948

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PHYSICS GROUP

R. Brocklehurst, L. Brooks, T. Davenport, R. Davis,
E. Harvill, H. P. Knauss, H. Morgan, and L. Vassamillet

ABSTRACT

The sensitivity of the scaler used for neutron counting was found not to be constant, indicating an additional error of approximately 3 per cent on work accomplished since October 22, 1947.

Weighing tests were conducted both with and without a radioactive source in the vacuum balance purity apparatus, to test the accuracy and consistency of repested weighings. A set of weights was calibrated. Tests showed that the presence of a postum sample near one pan disturbs the balance.

A preliminary set of measurements with the effusion apparatus yielded inconsistent results. Modification of the apparatus is necessary.

The vapor pressure of selenium was measured between 0.05 and 290 mm. A table of the numerical results is presented.

X-ray diffraction patterns have been obtained for the purpose of measuring the coefficient of linear expansion of aluminum, copper, lead, and gold. Diffraction patterns were obtained for one sample of Q metal. Although this particular sample was not suitable for obtaining precise values of the coefficient of expansion, early indications are that the values reported by Maxwell and Beamer of Los Alamos are in error both as to magnitude and direction.

DETAILED REPORT

Neutronics - T. Davenport and E. Harvill

On January 25th, it was discovered that the scaler was not operating properly. An intermittent poor connection in the fourth scaling stage was found and corrected. At the same time the sensitivity was checked and found to be 7 mv. At the time of the last previous check, October 22, 1947, the sensitivity was 4 mv. Apparently, the

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sensitivity has not been constant since October 22, 1947. So, a study of the effect of a variable sensitivity on the determination of source strengths was necessary and has been started. The results of the study are presented in Table I.

TABLE I

	Average Counting Rate (c./sec.)	
Sensitivity (mv.)	C-5 (Q-Be)	#49 (Ra - Be)
4	124.29	19.42
7	109.64	16.71

These preliminary results indicate that an error of about 3 per cent is introduced by a change in scaler sensitivity from 4 mv. to 7 mv. This error was calculated from the ratios of the average counting rates for the two sources when the sensitivity was 4 mv. and when it was 7 mv. All other work in progress was interrupted by the scaler repair and related difficulties.

Purity of Postum - R. Davis and H. Morgan

Tests weighings were done on the vacuum balance to see if weights would repeat. It was found that the weighings agreed within about .000005 grams. However, the zero point would change by as much as .000025 grams suddenly. It was thought that the strains on the cast iron vacuum box caused by the air pressure could result in such changes in the zero point. Therefore, a new Wilson seal was inserted in the front of the box about 8 inches to the left of the beam release lever shown in Figure 1, Physics Report of December 16-31, 1947. A system of levers was arranged inside the vacuum balance such that two small auxiliary pans, one on the left balance pan and the other on the right balance pan, could be set on or taken off the balance pans at will without having to let air into the balance box and then opening the front panel. This arrangement, therefore, permitted a balance zero to be obtained as an object was being weighted without breaking vacuum.

The balance weights and riders were then calibrated in the conventional manner, and no further difficulties were encountered.

Radiation shields were then installed between the balance beam and the balance pans so as to prevent heating of the balance beam by direct radiation from the warm radioactive sample. These shields consisted of three 1/8 inch thick aluminum plates, with slots for the pan support and other balance parts, separated 1/4 inch by use of three 10-32 brass bolts. A six case sample of postum in a sealed quartz tube was suspended over the left balance pan to see if the zero point

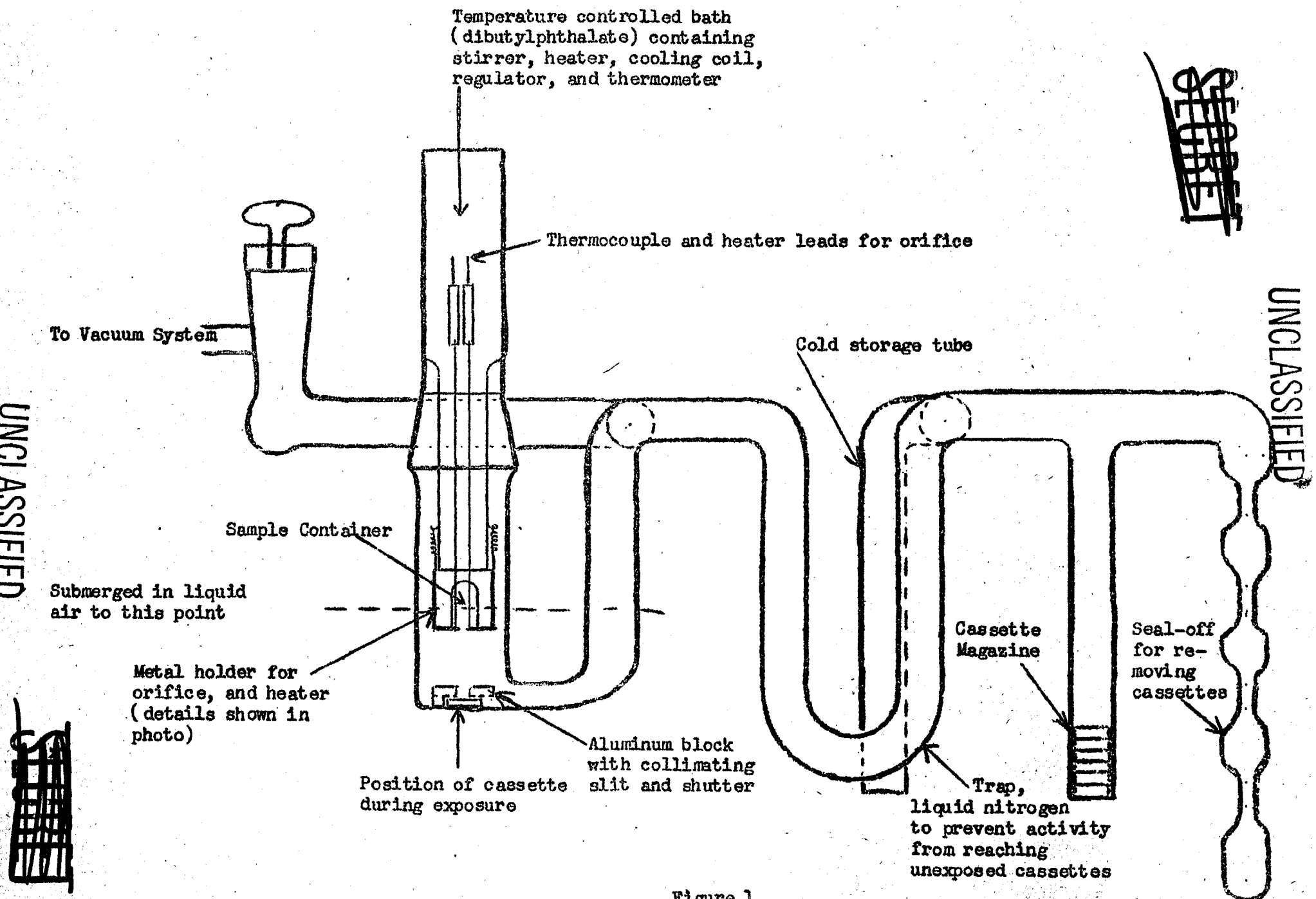

Figure 1

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or the weights of the auxillary pans were affected. No significant weight difference greater than .000010 gm. was observed. However, when a 50 case sample of postum was suspended over the left pan, both the zero point and the auxillary pan weights were changed by as much as .000150 gm. Therefore, the shielding arrangements will have to be further investigated, and the exact causes of the large weight errors will have to be determined.

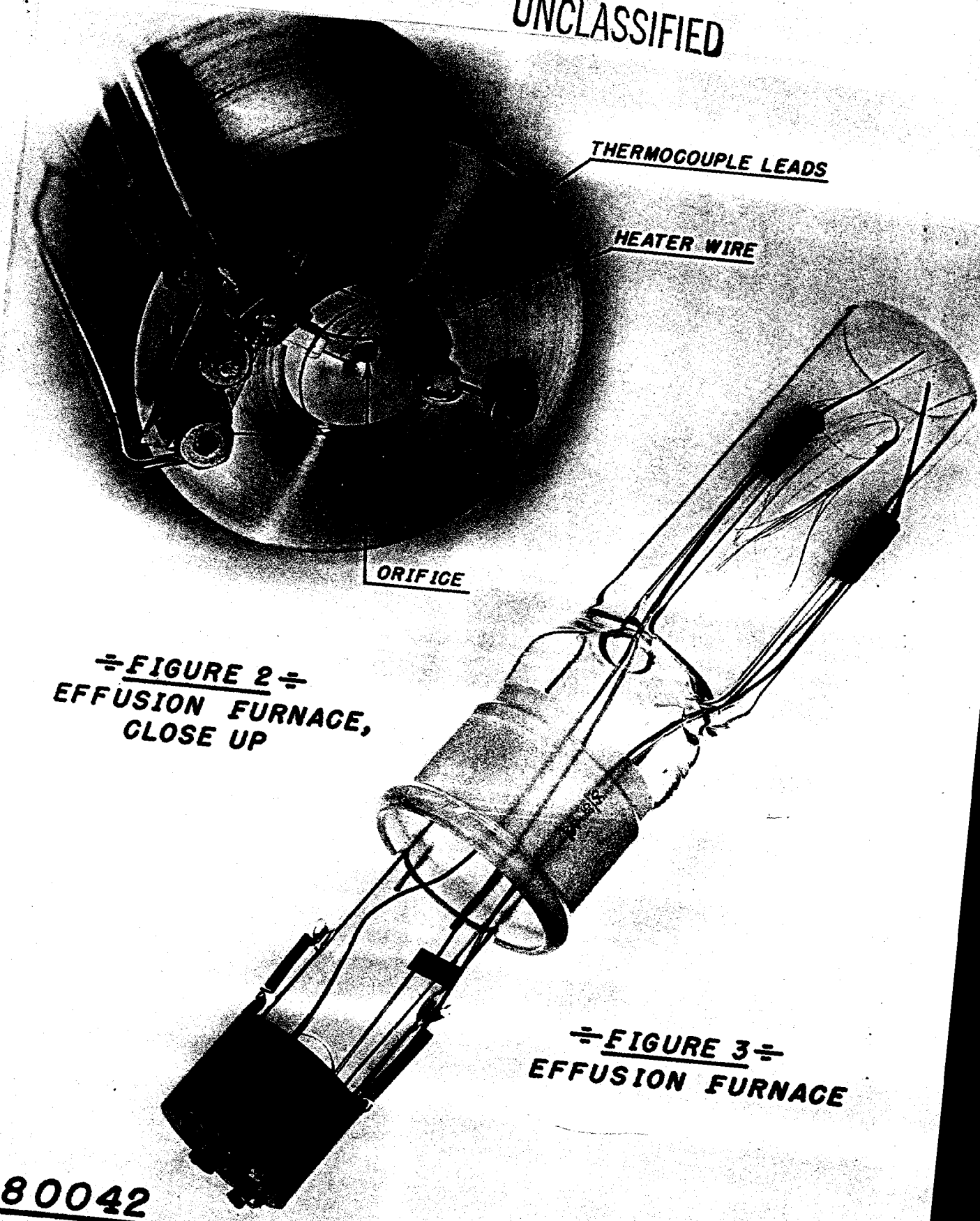
Vapor Pressure of Postum by the Effusion Method - R. Davis

The apparatus for measuring the vapor pressure of postum was completed, and one series of measurements made. The results were very inconsistent, making it necessary to redesign the apparatus before a second series of measurements could be made. This Progress Report will be devoted to a brief description of the apparatus, the results obtained, and finally a discussion of the factors leading to discordant results.

The apparatus used is shown in Figure 1. The sample was contained in a quartz tube placed in the recess at the end of a tube containing a temperature regulated dibutyphthalate bath. A platinum foil containing an orifice (0.03 cm^2.), see Figure 2, was held over the tip of the tube by a steel support held onto the tube by springs, as shown in Figure 3. It was found in preliminary experiments that the orifice was cooled below the temperature of the bath, therefore a tungsten heater was added to heat the orifice to a few degrees above the temperature of the oil bath. A copper-constantan thermocouple was spot welded to the orifice plate so that the temperature of the orifice could be measured at any time during the experiment. The details of the sample holder and heater are shown in Figures 2 and 3.

The cassettes were stored in the cassette magazine (Figure 1) and were moved by an Alnico magnet over to a position under the aluminum block that contains the collimator (0.6 cm. dia., and 4.1 cm. from the orifice) and shutter. They were exposed to the beam by moving the shutter aside for a given period of time. Finally the exposed cassettes were moved to the seal-off tube (liquid nitrogen cooled) and stored until a desired number were exposed. The tube was then sealed off with a torch while the cassettes themselves were kept cool by liquid nitrogen, and the cassettes were removed. The palladium foils containing the deposit were counted using the appropriate counting instrument.

In this apparatus it is important that the molecular beam be completely condensed. This was accomplished by just submerging the portion of the apparatus under the orifice in liquid nitrogen, and by pre-cooling the cassettes in liquid air. A trap was placed between the hot side of the apparatus and the cold side to prevent activity from reaching unexposed cassettes. In order to condense the beam itself it is necessary that the number of molecules striking the surface from the beam greatly exceed the vapor pressure of postum at the temperature of the condensing surface. One would expect that adequate condensation



÷FIGURE 2÷
EFFUSION FURNACE,
CLOSE UP

÷FIGURE 3÷
EFFUSION FURNACE

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would be accomplished at liquid air temperatures, but on the other hand it is difficult to keep the temperature of the parts of the apparatus down to liquid air temperatures. The cassettes were cooled from 2 to 10 hours in liquid air before exposure, and movement of the cassette through the apparatus was made as rapidly as possible.

A series of measurements was made at 50°C. exposing the cassettes for 9, 62, and 602 seconds in order to determine the order of magnitude of the vapor pressure. Based on these results a series of measurements was made at 11, 23, and 33°C. It was found that the background count was rather high (5 to 10 per cent of the exposed foils), and the number of counts per minute per second exposure to the beam did not increase with temperature as expected. It was believed that the reason for these results was that the entire apparatus was not cold enough to condense the beam on the cassettes completely. With this in view a second run was made in which the cassettes were cooled in liquid nitrogen at least 10 hours before exposure, and care was taken to keep the apparatus as cold as possible. The results obtained are shown in Table II.

TABLE II

Temperature	Time of Exposure (sec.)	Type of Sample	c./min.	c./min. sec. exposure corrected for background
12.0	1826	Blank	11,600	-
12.0	1815	Exposed	1,200,000	657
21.2	418	Blank	19,400	-
21.2	404	Exposed	133,000	282
30.3	123	Blank	9,370	-
30.3	103	Exposed	7,800,000	75,700
40.8	~50	Blank	14,900	-
40.9	48.5	Exposed	66,500	1,060
40.9	52.0	Exposed	28,600	263
50.1	~30	Blank	12,900	-
50.1	28.9	Exposed	26,500	472
62.0	~11	Blank	19,800	-
62.1	16.7	Exposed	23,904	245
71.7	~10	Blank	20,800	-
72.0	6.1	Exposed	30,000	1,530
-	-	Removed directly from cassette magazine	3,770	-

The samples marked blank were moved over to the position of exposure, but the shutter was not opened, and in all cases an attempt was made to keep the time that the blank was held in position under the aluminum about equal to the time of exposure of the sample. At the end of the table the results are shown on a sample that was removed directly from the magazine giving the extent of contamination of the cassettes before exposure.

From the results listed in the table one may make the following observations: (1) The cassettes in the magazine are only slightly contaminated, (2) the blanks are always lower than the exposed foils, and (3) there is no appreciable increase in counts per minute per second exposure with temperature. One would expect an increase in the order of one thousand for the temperature interval 12 to 72°C. The reason for these discordant results is not entirely clear but probably the main difficulty is that the beam was not condensed completely. The heater and sample holder are quite close to the aluminum block and tend to heat it by radiation. This heat is removed only by radiation at its base, hence the resultant temperature is probably too high for condensation. This leads to a general high level of contamination in the apparatus, so that marks the expected increase in the number of molecules effusing with increasing temperature. Another possible difficulty is that the postum, if properly condensed, may re-evaporate before the cassette is removed for counting. At just it was hoped that a counter could be built into the apparatus so that the cassette could be counted immediately after exposure and the possibility of evaporation could be tested by counting the foil after standing a while in the apparatus. Unfortunately counting tubes suitable for this purpose were not available and in view of other factors it was decided to proceed with the experiment and use the seal-off technique. To minimize the possibility of re-evaporation, palladium foil was used in the cassettes, because it has been found that activity sticks to palladium better than it does to platinum. There are other possible factors that would also lead to the above obtained results, but there is no way of telling for certain whether they are occurring or not. Perhaps the activity has reacted with the quartz or with other contaminants (mercury vapor) so that it is no longer in the metallic form, and therefore would not exhibit a high vapor pressure. The outside of the sample tube may be heavily contaminated, but this is rather doubtful.

In view of the many possible difficulties, a new apparatus is being built that incorporates several features designed to avoid these difficulties. The new apparatus will have a platinum lined copper crucible heated by induction heating making it unnecessary to use a filament to heat the orifice. A shutter will be used that plugs the collimating orifice and prevent contamination of the portion of the apparatus where the cassette is held for exposure. The walls of the

apparatus are used to condense the beam, and they will be cooled with liquid air, giving a very cold surface for condensation. This apparatus is now being constructed.

Resistivity of Postum - H. Morgan

The resistivity apparatus is ready for the initial run with cadmium. However, this work was halted in order that the vacuum balance be put into operation. It is necessary to have the vacuum balance in operation to assay a postum sample for purity before the resistivity measurement of postum can be done.

Vapor Pressure of Selenium - L. Brooks

The vapor pressure of selenium was measured between 0.06 and 290 mm. with the quartz Bourdon gauge described in the Physics Group Progress Report for September 1-15, 1947. The measurements in this period were taken at these lower pressures, since there is less possibility of breaking the fragile sickle gauge and the measurements of pressure are more accurate when only low pressures are measured. The numerical results of these measurements are presented in Table III.

When these results are plotted on a graph of the log of the pressure vs. the reciprocal of the absolute temperature, the values lie as close to a straight line as expected with the estimated experimental error. They define a straight line with less deviations than our previous vapor pressure measurements. Since the measurements are being continued, an equation and graph including the present results will be presented with a detailed discussion when the measurements are completed at higher pressures.

X-ray Studies - R. Brocklehurst and L. Vassamillet

Diffraction patterns for the determination of coefficients of expansion have been obtained for aluminum, copper, lead, and gold. For these four metals a total of 18 diffraction patterns, each requiring at least 8 hours of operation of the diffraction equipment, were taken. Similar patterns have been obtained for silver (Progress Report for December 1-15, 1947) and are being obtained for iron.

Three diffraction patterns of a sample of Q metal were made. The sample did not contain enough metal to produce a pattern which could be accurately measured; however, comparison of the patterns indicate that the coefficient of expansion, α_6, is in the range of $+10\text{-}20 \times 10^{-6}$ instead of $-300 \pm 100 \times 10^{-6}$ as reported by Maxwell and Beamer of Los Alamos.

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TABLE III

VAPOR PRESSURE OF SELENIUM

Date	Temperature in °C.	Pressure in mm. of Hg
Jan. 22	269.5	.056
Jan. 22	273.9	.083
Jan. 21	285.5	.139
Jan. 20	312.1	.361
Jan. 21	314.3	.334
Jan. 6	339.5	.834
Jan. 20	340.5	.834
Jan. 20	346.2	1.001
Jan. 20	360.4	1.557
Jan. 7	373.7	2.252
Jan. 19	384.0	2.959
Jan. 19	395.3	3.989
Jan. 23	411.6	6.210
Jan. 7	441.9	12.60
Jan. 23	471.5	23.60
Jan. 8	481.8	29.85
Jan. 8	507.2	49.21
Jan. 14	521.1	62.85
Jan. 14	533.2	78.88
Jan. 9	553.3	113.16
Jan. 9	614.2	288.94

FUTURE PLANS

The vacuum balance apparatus will be modified in an effort to eliminate the discrepancies observed in the presence of large radioactive samples.

Another apparatus to measure the vapor pressure of postum by the effusion method will be constructed with necessary modifications.

Another sample of Q metal for the X-ray diffraction apparatus will be prepared and its coefficient of expansion accurately determined.

Ray Davis, Jr.

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