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MOUND LABORATORY
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Laboratory Director

PHYSICS PROGRESS REPORT

SPECIAL REREVIEW
FINAL DETERMINATION

Date: November 1-30, 1948
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PHYSICAL GROUP
ABSTRACT

1. In the proposed vapor pressure measurement of postum over an extended period of time, fracture of the quartz-sickle gauge has necessitated the substitution of a more direct though less precise method.

2. A sample is being prepared for the measurement of the vapor pressure of postum by effusion.

3. A method of mounting a sample of postum for X-ray emission spectroscopy has been devised. The necessary metal and glass parts have been made. If the sample is prepared successfully, it will be shipped to Oak Ridge for the X-ray analysis.

4. An unexplained discrepancy in results of repeated weighings with the vacuum balance has led to a reassessment of the possible sources of error. Ten possible sources of error are enumerated and discussed.

5. There has been no progress on the resistivity of postum.

6. A gamma counter utilizing the recently devised lead geometry has been constructed, tested, calibrated, and turned over to the Assay Group.

7. A sample of the red postum bromide prepared by reacting postum and HBr was essentially the same as the previous sample prepared by reacting the elements. The latest patterns of Sample No. 51, elemental postum, indicate that the change in lattice constant of the G phase resulting from the substitution of lead atoms is slightly smaller than that obtained from the analyses of Sample No. 33.

DETAILED REPORT

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

The quartz-sickle gauge designed to measure the vapor pressure of postum over an extended period of time fractured because of an internal strain, the cause of which is unknown.

A less exact though more direct method for studying some of the properties of the compound Pb2 has been devised. This consists of placing
a calorimetered sample of pure postum in one end of a long quartz tube
of about 3 mm. base. After the postum has decayed for some time, a mixture
of postum and PbQ should exist. If the vapor pressure of PbQ is appreciably
lower than that of postum, it should be possible to separate the two by vola-
tilization. If the less volatile component is PbQ, then the number of postum
atoms determined by caloriometry should equal the number of lead atoms obtained
by decay. The required quartz apparatus has been constructed, and a sample
of postum is being prepared.

2. Vapor Pressure of Postum by Effusion Method — L. O. Fauble, 0.6.

The large Dejar flasks have been delivered. The apparatus has
been thoroughly checked on a dry run and everything has worked satisfactorily.
Twenty-five units of postum have been delivered and are being purified for the
first run.

3. Preparation of a Target for the Determination of the Primary X-ray
Emission Spectrum of Postum — L. O. Fauble, 0.4.

The problem of preparing an X-ray tube target for the measurement
of the primary spectral emission lines of postum has been undertaken. The
actual exposure will be done at Oak Ridge where the X-ray equipment is
located, only the target being prepared here.

Electrolytic deposition of postum on the target has been tried
by the Electrodeposition Group here; but they have been unable, as yet,
to achieve the necessary case density per unit area.

It is proposed to try achieving this case density per unit area
by a volatilization technique.

The apparatus for this attempt is shown assembled in Figure 1,
and disassembled in Figure 2. To assist in exact placement of the oven
orifice, a universal joint, which allows lateral motion as well as angular
motion, has been constructed from two ball joints as shown in Figure 1 and
Figure 2.

To use the volatilization apparatus, postum will be placed in the
nickel oven, the apparatus assembled, evacuated, the X-ray target chilled
with liquid nitrogen, and the nickel oven heated using a Westinghouse 5 kW
electronic heater. Temperature will be measured by a platinum-platinum,
rhodium thermocouple spot welded to the brass oven support.

After the volatilization is complete, the system will be filled
with dry helium, removed from the vacuum system, placed in a helium-filled
Assembled X-ray Target Preparation Apparatus
Components of X-ray Target Preparation Apparatus

- Liquid Air Cooled Finger
- To Vacuum Pump
- Housekeeper Seal
- Collimator Holder and Shield
- Oven Support
- X-ray Target
- Universal Joint
- Thermocouple Outlet
- Ni Oven

Fig. 2
drybox, and disassembled. The coated X-ray target will be removed and placed on its special support for shipment to Oak Ridge.


After the postum sample was sealed into the quartz weighing capillary, the sample was weighed in the vacuum balance. The sample contained 49.293 cases on October 28, 1942. A series of check weighings showed one set of values at 1.655025 gms, and another group at about 1.656050 gms. These weighings were made at pressures below 0.3 microns of mercury. Since these weight variations could not be explained by any known factor, the sources of balance errors were resurveyed and some previously overlooked sources were found possible.

The possible factors affecting the vacuum balance are listed:

1. Heating of balance beam and parts by direct radiation.
2. Direct conduction of heat from pan to beam.
3. Radiometer effect at reduced pressures.
4. Temperature changes in balance from external causes.
5. Sticking of knife edges to agates at reduced pressures.
6. Dust particles falling onto or off from balance parts.
7. Vibration.
8. Charging of various ungrounded balance parts by gamma ray photo-electric effect.
9. Small potentials resulting from contact potentials (work functions).
10. Quartz tube could be a charged dipole.

The listed factors will now be discussed in order:

1. Three 1/3 inch thick aluminum plates five inches by twelve inches were stacked between the balance beams and the weighing pans and separated by a 1/4 inch space. Small holes and slots were cut into the
plates to allow free operation of the balance. This should be a more than adequate provision to stop direct radiation (except gamma) and distribute any heat caused by radiation absorption evenly.

2. R. Davis found that a 30 case sample of postum suspended in a vacuum heated itself to 61°C. This sample in a quartz tube was suspended by a 22 gage platinum wire sling to a hook on the left balance pan. Heat transfer could have occurred by conduction up and through the agate and knife edge to the balance beam.

If the heat output of a 30 case sample were to flow through the balance beam, we can compute what the maximum temperature differential will be and also the maximum zero point shift. The beam is aluminum .3 cm. x .6 cm. x 14 cm. long. The equation for longitudinal heat flow in it is

$$t_2 - t_1 = \frac{HL}{KA}$$

where:

- $H$ = Heat flow in calories/sec.
- $L$ = Length in cm. of beam.
- $A$ = Cross-sectional area in cm.$^2$
- $K$ = Coefficient of thermal conductivity.
- $t_2 - t_1$ = temperature difference in °C. between ends of beam.

Substituting the numerical values, the temperature difference is

$$t_2 - t_1 = \frac{(0.00761 \times 30) 14}{.49 \times .3 \times .6}$$

$$t_2 - t_1 = 36°C.$$

Now we proceed to find the change in weight produced by a temperature change of 36°C. This will be done by finding the period of the balance, and then calculating the effective weight assuming that the beam has no weight.

Period with no weight on pans = 25 sec.

Period with 2.656 gm. on each pan = 26 sec.
The equation for the period, $T$, in terms of the moment of inertia, $I$, and torque constant, $K$, is

$$T = 2\pi \sqrt{\frac{I}{K}},$$  \hspace{1cm} (2)

$$26 = 2\pi \sqrt{\frac{I_1}{K}},$$  \hspace{1cm} (3)

$$25 = 2\pi \sqrt{\frac{I_2}{K}}.$$  \hspace{1cm} (4)

The moments of inertia with and without the weight are:

$$I_1 = \sum m_r^2 + 2.656 \text{ gm. } x 2 \times 6.5 \text{ cm.}^2,$$  \hspace{1cm} (5)

$$I_2 = \sum m_r^2.$$  \hspace{1cm} (6)

Putting equation (6) into equation (5) we have

$$I_1 = I_2 + 2.656 \times 2 \times 6.5^2.$$  

Substituting the value for $I_1$ into equation (3), and solving equation (3) and equation (4) for $I_2$, we get

$$I_2 = 2750 \text{ gm. cm.}^2.$$  \hspace{1cm} (7)

Solving for the effective mass $M_B$ at the end of a 6.5 cm. beam radius:

$$M_B = \frac{2750}{6.5^2} = 65 \text{ gm.}$$  \hspace{1cm} (8)

or

$$\frac{1}{2} M_B = 32.5 \text{ gm.} \text{ (each side of balance).}$$

Setting up the equations of a simple balance:

$$M_1L_1 = M_2L_2,$$  \hspace{1cm} (9)
where \( M_1 \) is mass at radius \( L_1 \) left from pivot,

\( M_2 \) is mass at radius \( L_2 \) right from pivot.

We must now find the ratio of \( L_1 \) to \( L_2 \) when one end of the beam (left) is 36\(^\circ\)C hotter than the other, a linear temperature distribution being assumed.

Let \( L_0 = \) Original length.

\( t = \) Temperature increase.

\( k = \) Coefficient of thermal expansion.

Then \( L_t = L_0 (1 + kt) \) \( (10) \)

which is the new length of the beam at temperature \( t \).

Since the average temperature rise of the left and right beam is 27\(^\circ\)C. and 9\(^\circ\)C. respectively, the beam lengths become

\[
L_{27} = 6.5 \text{ cm.} + 24 \times 10^{-6} \times 27^\circ\text{C.} \times 6.5 \text{ cm.} = 6.5042 \text{ cm.} \quad \text{(left beam),} \quad (11 \text{ a})
\]

\[
L_9 = 6.5 \text{ cm.} + 24 \times 10^{-6} \times 9^\circ\text{C.} \times 6.5 \text{ cm.} = 6.5014 \text{ cm.} \quad \text{(right beam).} \quad (11 \text{ b})
\]

Setting up the balance equation (9) and solving for the weight needed on the right pan when the left end of the beam is 36\(^\circ\)C. hotter than the right,

\[
M_2 = \frac{M_1 \cdot L_{27}}{L_9} = \frac{32.5 \text{ cm.} \times 6.5042 \text{ cm.}}{6.5014 \text{ cm.}} = 32.513 \text{ gms.} \quad (12)
\]

with 2.656 gm. activity capsule.

\[
M_2 = \frac{(32.5 \text{ gms.} + 2.656 \text{ gms.}) \times 6.5042 \text{ cm.}}{6.5014 \text{ cm.}} = 35.170 \text{ gms.} \quad (13)
\]

If we subtract the weight on the left side of the balance from that needed to balance it found in equation (12) and (13), we will have the weight change due to the 36\(^\circ\)C. temperature gradient:

Zero Point \( 32.513 \text{ gms.} - 32.5 \text{ gms.} = .013 \text{ gms. shift/36\(^\circ\)C.} \)

Capsule \( 35.170 \text{ gms.} - 35.156 \text{ gms.} = .014 \text{ gms. shift/36\(^\circ\)C.} \)
These calculations show that a serious error would occur if appreciable heat conduction is present. Since the zero point should shift about 12/14 as much as the capsule weight should shift, and since no significant zero point shift does occur between times when the beam should be hot and times when the beam should be cold, it is believed that no significant heating of the beam is present.

3. It was found that a radiometer effect did exist and was quite large at pressures above one micron of mercury. However, a linear relationship between absolute pressure and weight was found to exist and a small correction factor applied (less than 10 micrograms).

4. Room temperature changes do affect a microbalance in air, but if a zero point is taken at the same time this error is compensated.

5. It might be expected that in a vacuum there might be sticking of the knife edges. This would be due to loss of surface films, or gas evolution from the agate or alloy knife edges. However, in six months of operation there has been no evidence whatever of sticking.

6. Dust will affect any balance, and there is no reason to believe that this should be any more a source of error than in ordinary microbalance operation.

7. The vacuum case is a heavy cast-iron box mounted on a cement balance pier set in a vibration absorbing sand pit. There is no evidence of vibration in the balance or balance pier.

8. There is a possibility that photoelectrons ejected by the absorption of gamma rays by the aluminum radiation shields and balance parts will leave insulated and ungrounded parts charged. These stray charges could affect both the zero point and the capsule weight similarly, or could affect either one separately. An error of about 25 micrograms, which was thought to be caused by these charges, was observed between successive weighings in the early November tests. All balance parts were grounded and the weight variations were no longer observed.

Of a total of nine weighings at a pressure of about $1 \times 10^{-4}$ mm. of Hg., the average weight was 2.656025 ± 0.000004 gms. The parts were ungrounded and a second series of nine weighings were made at $1 \times 10^{-4}$ mm. of Hg. and the average weight was 2.656020 ± 0.000004 gms. The previous error of about 25 micrograms was not observed during these last weighings with the balance parts ungrounded. There is a possibility that the part being charged is now
grounded even though all ground wires have been removed. This factor will have to be checked further. The time between any two successive weighings was not less than one hour nor greater than 60 hours so as to let the balance come to an equilibrium state.

9. Small potential fields existing between balance parts because of contact potentials should be similar in effect to the heat conduction error. The zero point and the capsule weight should both be shifted about the same amount and in the same direction. There is the possibility of a small constant error, but any intermittent error would show up in the data.

10. If there is a concentration of activity in one end of the quartz capillary, some of the alpha rays emitted may be discharged in the opposite end of the tube. This would leave the capsule a charged dipole which by induction could be attracted to nearby balance parts. The capsule can be tested to see if it is a charged dipole by suspending it in air or a vacuum between two charged plates and observing the deflection, if any.

Factors 9, 9, and 10 will be investigated so as to eliminate them as errors in the vacuum balance operation.

5. Resistivity of Postum — H. C. Morgan, O.

No progress.


During the period of this report, measurements of neutron flux have been made on forty-three sources for the Neutron Source Group. Gamma measurements of eleven sources were made for various groups.

A research model of our newly designed geometry for gamma counting has been completed. The new features of the model are a compensating lead attenuator and a rotating source holder. We have given to the geometry the designation LARC — Y for lead attenuated, rotated counter for gamma detection.

The design incorporates two devices which compensate, within limits, for non-homogeneous activity distribution of unknown samples in comparison with a known standard. They are:

1. A continuously rotating source holder to eliminate errors due to non-uniformity perpendicular to the counting tube axis.

2. A figured lead shape inserted between the sample holder and tube portions of the geometry which causes a flat response regardless of activity.
distribution parallel to the counting tube axis.

For the purpose of assaying K-can samples, a gamma counting geometry has been designed, built by the Machine Shop according to our specifications, and calibrated by this laboratory. It is similar to the LARC-Y except that the compensating lead shape was not included in its design since only one type of sample with a rather well defined activity distribution is to be measured with this geometry. The response of this model using a standard Geiger-Müller tube and the I.D.L. scaler is 12.51 counts/sec./case with a percentage probable error approximately twice the percentage probable error in the counting rate. This counter will be put in use by the Assay Group.

Future Plans

Continue the design of a gamma counter to cover the range of 0.1 to 50 cases of postum which will be independent of the activity distribution.

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

1. Sample No. 53, red postum bromide, was submitted by F. Joy. Most of the pattern was the same as that obtained from the red part of Sample No. 46, also postum bromide. The only difference was that a series of weak lines in Sample No. 46 was absent in Sample No. 53 indicating a higher purity of the compound in Sample No. 53. Sample No. 53 was prepared by reacting postum and HBr while Sample No. 46 was prepared by reacting the elements postum and bromine.

2. The change in lattice constant of the alpha phase of postum due to the substitution of lead atoms is apparently only about 0.01 Å. This is about 60 per cent of the change found by the less accurate measurements that were made with Sample No. 33.

REFERENCE


JPL/1997