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PHYSICS PROGRESS REPORT

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PHYSICS GROUP

R. E. Brocklehurst, L. S. Brooks, T. I. Davenport,
L. G. Fauble*, E. Harvill, H. P. Knauss, E. R. Manning**,
H. C. Morgan, and L. F. Vassamillet

* Transferred to Physics Group June 1, 1948.

** Reported for work June 23, 1948.

ABSTRACT

The problems of the group, whether active, temporarily put aside, or under consideration for the future, are reviewed briefly but comprehensively.

The vacuum box holding the Ainsworth balance has been pumped down by means of a diffusion pump without apparent effect on the smooth action of the knife edges. Radiometer effects have been reduced from 250 micrograms to 10 micrograms, in going from 3×10^{-2} to 4×10^{-4} millimeters of mercury pressure.

Plans are outlined for a redesign of apparatus for measuring the vapor pressure of postum by effusion at comparatively low temperatures.

An X-ray method of measuring the coefficient of expansion, which performed satisfactorily when tried on six other metals in the cubic system, was used to obtain a value of about 20×10^{-6} for α -Q. This is very different from the value of the coefficient obtained by Maxwell and Beamer, $-300 \pm 100 \times 10^{-6}$. It is suggested that an investigation be made of possible phase changes at much lower temperatures and that confirming data be obtained for the coefficient of expansion.

A sample of postum metal has been followed through a period of four half-lives. The lattice constants of both the high and low temperature forms and the theoretical density as reported by Maxwell and Beamer have been confirmed. In addition, a Pb-Q compound resulting from the decay of postum has been identified. It is suggested that another sample of metal be prepared for the purpose of accurately determining the lattice constants through the decay process. Dr. Zachariasen expressed the opinion that the simple cubic structure of α -Q is undoubtedly correct.

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Some information concerning valences, ionic radii, etc., has been obtained from a study of several compounds. These compounds, which include PbQ , ZnQ , QO_2 , QCl_2 , and QCl_4 , have established valences of -2, +2, and +4 with ionic radii of $Q^{--} = 2.07 \text{ \AA}$, and $Q^{+++} = 1.06 \text{ \AA}$. The atomic radius of Q is $1.67 \text{ \AA}$. It is suggested that further work on compounds may yield additional information on the stability of compounds and possibly information on various bond angles.

An investigation of crystal structure, crystal size and crystal orientation of nickel formed by the decomposition of nickel carbonyl has been started, with the hope of correlating physical changes with the conditions of deposition.

A sample of pure tellurium nitrate was found to be in close agreement with the published data for telluric nitrate.

Single crystal studies have been abandoned since the simple cubic structure of α - Q has been accepted.

DETAILED REPORT

Neutronics - T. I. Davenport and E. Harvill

The neutron measurements needed throughout the laboratory include: (a) determination of neutrons emitted by impurities in purified positum; (b) relative efficiencies of neutron sources produced by various means; (c) absolute strength of neutron sources produced for use at other sites; and (d) fast neutron flux as a health hazard.

The standard source used in most of our work is Ra-Be No. 49 (one milligram Ra), which was calibrated against a one gram Ra-Be source No. 38 at Chicago. The probable error is estimated to lie between five and ten per cent. Eventually we hope to attempt an absolute evaluation of a Q-Be source by adapting the method of Siedl and Harris (Rev. Sci. Inst. 18, No. 12, Dec. 1947). They measured the amount of helium produced in boron by the $B(n, \alpha) Li$ reaction, relative to induced gamma activity of manganese mixed with the boron. Preliminary calculations indicate that with the large neutron sources available here, amounts of helium great enough to be measured directly to a precision of one per cent can be collected. It is planned to begin this work after occupying the new laboratory at Unit V.

The measurement of a neutron source strength relative to a standard comprises our immediate study. According to the method of A. O. Hanson, MDDC 110, March 15, 1944, a long counter system is used for the detection of fast neutrons. The problem here is that the response of the counter varies with different energy sources and with the distance, X , separating source and detector. Consequently, the ratio of counting rates of two sources will be, in general, a function of X .

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The usable range of the value of X for a given source is determined by background and coincidence considerations. Since the usable ranges for X for various sources, including the standard, do not necessarily overlap, it is necessary to use experimentally determined values of the functions of X for practical intercomparison of sources. A. O. Hanson reports (ibid.) a flat response of the detector when X is large. Therefore, we propose collection of data on the ratios of the counting rates as a function of X of a series of secondary standards (Q mixtures) and extrapolation of these data to infinite distance. Using these extrapolated ratios of the secondary standards to the primary standard as the true ratios for the calibration, we would then compute suitable corrections for data taken at a convenient distance from the detector. Some of this work has been done already; but because of difficulties with the scaler (see below) and inadequate equipment for positioning sources, the studies so far must be considered preliminary.

The design of a device to give precision source positioning was made by the Laboratory Instrument Design Group from specifications set up by this laboratory. However, the machine shop, because of a crowded schedule of priority projects, has not as yet begun the construction.

Much retrogression has been caused by malfunction of the high rate neutron counter, EL-136. This scaler was installed on January 21, 1947 and apparently functioned correctly for some time thereafter. However, on April 1, 1948 it began to count a known standard at a noticeably slower rate. This precipitated an investigation leading to the discovery that the counter was almost consistently recording two counts per pulse received from the counting tube. By checking back over data taken on the standard source, the conclusion was reached that, for at least five months previous to this discovery, the double counting effect was consistent and was considered normal. It is thought that the scaler was undergoing the change from single to double counting operation gradually and probably somewhat erratically since its installation. However, the many geometry, tube and technique changes made during this time obscured the metamorphosis. Much of the data collected in the past with this scaler is prejudiced as is the interpretation of the data insofar as calculated values of precision and coincidence correction are concerned.

Work on the second portion of the main research problem has been held up for three reasons: (1) lack of precision source positioning apparatus; (2) malfunction of the laboratory scaler; (3) studies of an immediate but incidental nature (see below).

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This laboratory does routine counting of neutron sources for other groups. Since a considerable amount of time is required because of the number of such sources and their low counting rate, an auxiliary geometry offering increased handling efficiency and counting efficiency (in terms of counts per neutron) is required. To this end a tub type geometry has been refined and a study of its characteristics completed (Physics Progress Report, March 1-31, 1948). However, this work was also delayed by the scaler difficulties. A report on counting techniques with the tub is scheduled for the immediate future.

Now underway is a study of a new type gamma counting geometry in which the counting tube and source are separated by lead instead of the usual air gap. This geometry was designed by Dr. H. P. Knauss for use by the Assay Group at Unit IV. The study concerns: (1) counting rate for a given source; (2) evaluation of a counting rate per case factor; (3) background; (4) reproducibility of results; (5) coincidence correction; (6) ease of operation.

A one-input model IDL scaler and a Geiger Muller counting tube with suitable amphenol connectors comprise the counting system. Considerable difficulty was encountered in obtaining a tube that would keep its plateau during the investigation. Much data, prejudiced by the faulty tubes, had to be discarded.

This investigation should be completed within one week.

Resistivity of Postum - H. C. Morgan

A complete description of the apparatus and the procedure to be used in the experiment appeared in the Physics Progress Report, August 15-31, 1947.

The apparatus has been built and tested for film condensation with cadmium. The resistivity measuring circuit has also been completed and tested. Further work has been in abeyance since January 1948, until postum of known purity can be obtained.

This work is being done so that the resistivity in the range of -200°C . to 100°C . can be determined. If any phase transformations occur in this temperature range, a break in the resistivity vs. temperature curve is expected. A phase change from an α to a β crystal form has been observed elsewhere. It is our intention to verify this effect, and, if possible, obtain accurate data.

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

A full discussion of the purity problem can be found in the Physics Progress Reports, January 1-15, 1947; February 15-28, 1947;

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August 16-31, 1947. Briefly restating the problem, it was found that evidence of unknown impurities often appeared as bands condensed beside the postum metal band in the quartz tubes during vacuum distillation.

Certain research problems, such as the vapor pressure of postum, the resistivity of postum, the density of postum, and the coefficient of thermal expansion of postum required either postum of known purity or pure postum. The essential first step toward either of these goals is to have an accurate and reliable means of determining the purity. Since it is now possible by calorimetric methods to determine the amount of postum present in a sample tube with an accuracy of .1 per cent, the purity can be found if the weight of the sample can be determined.

However, weighing the radioactive sample on a balance is made difficult by the air currents set up by the heat emitted by the sample. Some work was begun by another group, under the direction of Dr. E. A. Staniforth and Dr. E. A. Rembold, on reducing this error the counteracting evolution of a like amount of heat by a heating coil on the weight pan side of a beam balance. The same physical conditions such as size of heat source, shape of heat source, and similarity of objects surrounding the heat source are established on both sides of the balance.

This present research is endeavoring to eliminate the heat error by weighing the sample in a vacuum.

Initial weighing tests at a pressure of about 3×10^{-2} mm. of mercury showed an error of about 10 to 20 per cent in weighing a sample of about ten milligrams of postum. Heavy radiation shields made of 1/8 inch thick aluminum were then installed to prevent heating of the balance beam by radiation, but most of the error remained. It was then determined by moving a heat source to different positions near the pan that the radiometric effect was exerting a pressure on the balance pan. A shield over the pan eliminated most of the former ten to twenty per cent error, but a maximum of about 2.5 per cent still was present under certain circumstances.

It was thought that if most of the remaining error was still due to the radiometric effect, a better vacuum would reduce the error to a minor amount. Therefore, an oil diffusion pump was installed. This reduced the pressure from 3×10^{-2} to about 4×10^{-4} mm. of mercury, and reduced the weighing error to about 0.1 per cent.

The balance has been checked without activity present and found to be accurate to within five micrograms when evacuated. Some trouble with the agate knife edges sticking to the agate flats had been expected, but so far the balance action has been very smooth.

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Two purity determinations have been made, but these weighings were made before the diffusion pump was added. The purity calculated was 94.5 per cent and 100.5 per cent, approximately, on the same sample. These runs were primarily made to check the procedure of handling the activity, and as a result an improved procedure was outlined in the Physics Progress Report, May 1-21, 1948. Preparations have been completed for a run using the new procedure.

Vapor Pressure of Selenium - L. S. Brooks

The vapor pressure of selenium was measured with the quartz sickle gauge apparatus in order to eliminate any errors in the experimental procedure before a similar apparatus was used to measure the vapor pressure of postum. Selenium was chosen to check the procedure for the following reasons:

1. Selenium and postum are members of the same group in the periodic table.
2. Values of the vapor pressure of selenium and its boiling point are in the literature.
3. The vapor pressure of selenium can be measured in the temperature range in which the vapor pressure of postum lies between 0.1 and 100 mm., according to preliminary experiments.

The main problems with the quartz sickle gauge apparatus as in other methods of measuring vapor pressures, include measuring the temperature which controls the vapor pressure, insuring that the observed pressure is the same as the vapor pressure of the material, and guarding against foreign materials. Preliminary experiments last year revealed certain errors associated with these difficulties, which were attacked as noted below:

1. The surroundings of the sample bulb where the lowest temperature controlling the vapor pressure is measured did not have a uniform and stable temperature. We designed and built a massive copper block which was heated with a furnace with non-uniform windings designed to establish the desired temperature distribution around the sample bulb.
2. The temperature dependence of the quartz sickle gauges was not known. Observations have been made to check their behavior as pressure indicators when subject to deforming pressures at high temperatures.

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3. Pressure indications were not reproducible, so the magnification of the telescope used to observe the sickle gauge deflections was increased and the mercury manometer arm diameters were increased and a more accurate cathetometer to observe the heights of the mercury columns was obtained.

4. What appeared to be a partial pressure from a source other than the selenium metal was detected last year. Chemically purified selenium used in the later measurements did not show this effect below 600°C. To eliminate gas which might be retained in the walls of the quartz tubing in spite of outgassing for two hours, an outgassing period of forty-six hours was adopted for a subsequent postum experiment in which no indication of inadequate outgassing was detected.

Biltz and Müller¹ in "Über Gasabgabe aus heißen Quarzgeräten" reported that quantities of gases which would be objectionable in our vapor pressure measurements are evolved from heated quartz vessels even though they had been outgassed by heating in a vacuum for a day. They found that this evolution of gases from the quartz could be minimized by heating the quartz alternately in a vacuum and in an oxygen atmosphere. Since Biltz and Müller found that this evolution of gases would, for example, give 6×10^{-4} mm. of pressure in four hours at 635°C., and .055 mm. in twelve minutes at 930°C., it will be necessary to observe the precaution of treating the quartz apparatus in oxygen when measurements are taken at temperatures above 600°C. This outgassing procedure is being tried in a present experiment with selenium. At the same time more information on the temperature dependence of the quartz sickle gauges and the vapor pressure of selenium between 600°C. and 700°C. will be obtained.

Vapor Pressure of Postum - L. S. Brooks

The vapor pressure of postum was measured with a quartz sickle gauge between 0.3 and 900 mm. of mercury pressure at temperatures between 430°C. and 745°C. This work was reported in MLM-85 and at the A. E. C. Information Meeting at the Brookhaven National Laboratory on April 27, 1948. The least squares representation of the data is the following:

$$\log p = - \frac{5377.8}{T} + 7.2345$$

where p is the pressure in mm. of mercury and T is the absolute temperature.

¹W. Biltz and H. Müller, Z. anorg. allgem. Chem. 163, 297-301 (1927).

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From this least squares equation, the extrapolated boiling point is 962.1°C . and the latent heat of vaporization is 24,597 calories per mole. The above equation represents the data taken on the first two days, after which the vapor pressure was lower each day. Since the lowering of the observed vapor pressure with time may be due to the influence of lead and can be measured as a function of the quantity of lead present, it is essential to eliminate as much as possible other influences which would be masked and more difficult to detect. Some of the other influences, part of which are also present in the selenium measurements but are complicated by associated problems unique to the postum measurements, and their preventions or controls are listed below:

1. The vapor pressure measurements are very sensitive to impurities, especially since we measure the total pressure in the system. Therefore, the sample of postum should be assayed as accurately as possible. In the last measurement it was necessary to rely on the effectiveness of several distillations. If the vacuum balance method of assay proves to be satisfactory, the next postum sample will be assayed by that method.

2. The effect of the helium produced by the postum will be complicated by any temperature dependence or deforming of the quartz sickle gauges. During the last postum experiment the helium appeared to escape through the thin quartz gauge at 750°C . The influence of temperature and deforming pressures is being studied and will be minimized by keeping the gauge temperature as low as possible at the beginning and increasing it when necessary for measurements at higher temperatures and by keeping the deforming pressures at a minimum.

3. The evolution of gases from the heated quartz will be more probable when we attempt measurements at higher temperatures. Its control has been proposed in the discussion of the selenium measurements in this report. This problem can also be minimized by keeping the quartz at the lowest possible temperature.

4. The self heating of the postum may cause temperature gradients along the sample bulb which can not be eliminated by the uniform temperature of the surroundings. Temperatures of the surroundings will be adjusted so that the measured temperature represents as nearly as possible the lowest temperature in the sample bulb even though it is higher than the temperature of the copper block.

Two quartz sickle gauge apparatus have been constructed and will be used for postum as soon as samples can be assayed. In the next measurements the following will be attempted.

1. Measure the vapor pressure of sample of postum with a definite purity assay.

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2. Check the present vapor pressure data.
3. Study the effect of lead on the vapor pressure if the effect is indicated again.
4. Measure the vapor pressure at higher temperatures.

X-ray Laboratory - R. E. Brocklehurst and L. F. Vassamillet

Problem 1 - What is the coefficient of expansion of the α -form of Q-metal?

A study of the coefficient of expansion of Q-metal in the α -phase has been carried out to confirm or contest the results on this physical property as reported by Maxwell and Beamer of Los Alamos (L. A. Report #624).

A set of preliminary experiments was made using six other metals, also cubic in structure, to establish the accuracy of the technique. Results of this work have been reported in full. (Measurement of the Coefficient of Expansion of Microgram Samples of Metals. Presented at the Ohio Section of the American Physical Society, March 13, 1948.)

X-ray patterns obtained using samples of Q-metal showed that the coefficient of expansion was positive and of the order of 20×10^{-6} . This is in disagreement with Maxwell and Beamer report of $-300 \pm 100 \times 10^{-6}$.

Further work needs to be done to determine " α " more accurately for the low temperature form of the metal. Also a study should be planned to prove the existence or non-existence of a possible third phase, at very low temperatures. Such studies, however, entail the use of large samples of the metal under rather dangerous conditions and will, therefore, have to be postponed until the X-ray laboratory has moved to Unit V.

Problem 2 - How can the crystalline forms of Q be best explained?

A time study of the metal to learn the effect of decay upon its lattice constants and crystal structure has been made.

The sample studied was originally prepared to be a compound of Hg-Q, but showed by X-rays to be pure Q as reported by Maxwell and Beamer. This sample was made in January 1947, and since that time seventeen patterns have been run on it, following phase changes as the temperature has been changed and the changes in its crystalline form as it has decayed into lead.

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The following information is known at this time:

- a. Low temperature or α -Q: simple cubic; $a_0 = 3.345 \text{ \AA}$.
- b. High temperature or β -Q: rhombohedral; $a_0 = 3.359 \text{ \AA}$, $\beta = 98^\circ 13'$.
- c. Transition point indicated by study of electrical conductivity, 100°C . (Maxwell and Beamer L. A. Report #624).
- d. Density of α -Q: $\sigma_\alpha = 9.16$.
- e. Existence of a Pb-Q compound: cubic; $a_0 = 6.60 \text{ \AA}$.

According to Zachariasen at the Argonne National Laboratory the information at present is best explained by the following bi-metal phase diagram which shows the change in the distance, d , between atoms as Q decays to Pb. This diagram is hypothetical and schematic.

At this time Sample No. 33 is nearly four half-lives old, i. e., it contains about 6.5 per cent Q. Another metal sample is to be prepared for the specific purpose of detecting, if possible, the changes in the distance "d" as shown in the phase diagram. This new sample will be photographed on a logarithmic time scale so that the values of "d" may be compared against the atomic per cent of Q and lead. This new sample should be studied for eighteen to twenty-four months as was Sample No. 33.

Note: For a long time there was considerable reluctance to accept the fact that Q-metal crystallized in the simple cubic form. This was due to the fact that we expected it to act as a metal. But as a non-metal or semi-conductor the simple cubic structure is considerably more plausible. In the course of our visit to Dr. Zachariasen's laboratory on June 9, 1948, he expressed the opinion that the supporting evidence is sufficient to place the simple cubic structure beyond question.

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Problem 3 - What can be learned from a study of the compounds of Q concerning its ionic and atomic properties?

A study of the crystal structure of as many of the compounds of Q as can be formed will lead to the accumulation of information concerning its valences, ionic radii, metallic or non-metallic properties, etc.

Up to the present the following compounds of Q have been prepared and identified:

- a. Pb-Q; Sample No. 33
- b. Zn-Q; Sample No. 38
- c. QO_2 ; Sample No. 39 and 42
- d. QCl_2 ;
- e. QCl_4 .

Attempts to prepare Hg-Q, $QOCl$, and QO have been unsuccessful.

For convenience the vital information obtained from these compounds is shown below in tabular form.

<u>Composition</u>	<u>Type</u>	<u>Close Associates</u>	<u>Structure</u>	<u>Lattice Structure</u>	<u>Ionic Radii</u>
Pb-Q	NaCl	PbS, PbSe, PbTe	Cubic	$a_o = 6.60$	2.09
Zn-Q	ZnS	ZnSe, ZnTe	Cubic	$a_o = 6.27$	2.05
QO_2	CaF_2		Cubic	$a_o = 5.595$	1.06
	TeO_2	PbO_2	Tetr.	$a_o = 5.765$ $c' = 0.94$	
QCl_2	$PbCl_2$	$PbBr_2, BbF_2$	Rhodr.	$a_o = 4.331$	
				$b_o = 8.944$	
				$c_o = 7.292$	
QCl_4			Mono.		

We see that Q exhibits both metallic and non-metallic qualities and has been shown to have the valences of +4, +2, 0, -2.

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In each case it seems to conform fairly well to the family with which it is temporarily associated. The two crystal structures for QO_2 were accepted as plausible by Dr. Zachariassen, along with the breakdown of both structures with time. (See Progress Reports for September 15-30, 1947 and March 1-31, 1948.) Zachariassen suggested that thorium would be a good "stand in" for Q on the basis of its crystal size. At present the values for ionic radii are rough estimates and will need to be confirmed by values from other compounds.

Further information may be gotten from a study of the rest of the halides. Also it is possible that the angle between the chloride ions may be determined from a study of the relative intensities of the lines in the QCl_2 pattern. It is suggested that a further attempt be made to prepare the monoxide of the metal.

Problem 4 - What is the effect on the physical properties of nickel films of rate of deposition and temperature of decomposition of nickel carbonyl? Are there any other factors influencing these properties?

The appearance of nickel films deposited from nickel carbonyl seems quite sensitive to slight changes in deposition conditions. It may be possible to correlate these physical changes with X-ray information on crystal structure, crystal size, and crystal orientation.

Some twelve samples chosen because they showed the different types of films produced have been examined by X-rays and the patterns show that in all cases the crystal structure is the same, but there is considerable difference as to the particle size and particle size distribution and as to the degree of preferred orientation. These samples were submitted by W. F. Pesold.

Samples are to be prepared under known conditions and an attempt will be made to correlate particle size to these conditions. An attempt also will be made to find any impurities if they are present.

Miscellaneous Comments

A sample of pure tellurium nitrate was examined, and the pattern placed on file for reference. It is in very close agreement with the published data for telluric nitrate. (See Progress Report dated March 1-31, 1948.)

Laue pattern studies have been discontinued because of the acceptance of the simple cubic structure for α -Q.

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FUTURE PLANS

Determination of the Vapor Pressure of Postum by the Effusion Method - L. G. Fauble

Accurate data on the volatility of postum, particularly in the low temperature range, would be useful for the study of a wide variety of problems. For example:

1. To aid in the development of a method of purification of postum.
2. To give information on the thermal stability of compounds of postum.
3. To obtain fundamental thermodynamic properties of the metal and its compounds such as heats of sublimation, heats of fusion, heats of vaporization, melting points, boiling points, and entropies.
4. To aid in separating the metal from other products in its purification in production.
5. To find methods useful in the preparation of thin films of the metal in intricate shapes.

Proposed Method of Attack - At the present time the vapor pressure of postum is moderately well established in the temperature range 430°C. to 735°C. by direct measurement using a quartz spiral gauge. (MM-85).

The temperature range 0°C. to 400°C. has not been conclusively investigated and it is the proposed plan of attack of this problem which will be discussed here.

The vapor pressure of plutonium has been investigated by Phipps, Seifert, and Simpson (CN-3223) (GC) using a modified Knudsen type effusion apparatus.

Dr. R. Davis, Jr., using a modified Knudsen type of apparatus attempted to measure the vapor pressure of postum. (Physics Progress Reports January 1-15, 1947 to MM-85, April 1-30, 1948.)

In MM-65 he has listed the following possible errors in the three modifications of apparatus he used:

1. The postum used may have been impure.
2. The postum may have reacted with impurities present in the crucible or with the platinum itself.

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3. The orifice of the crucible may have become clogged.
4. The beam may not be condensed on the cassettes.
5. The film deposited may evaporate from the cassettes.
6. The orifice may be colder than the rest of the crucible.

Examinations showed that numbers 3, 4, and 5 could be eliminated, but that the others remained.

In redesigning the effusion apparatus the present author has endeavored to avoid some of the troubles experienced by Dr. Davis. Major changes in the apparatus are as follows:

1. In all of the above set ups, Dr. Davis had his crucible above the cassettes with the vapor stream pointed down. In the present design, the crucible is below and the collimator and the cassette holder above.

2. An endeavor will be made to heat the crucible by means of a resistance heater instead of by an induction heater.

3. The collimator will be made of metal instead of glass in order to provide better heat conduction to the cassette.

4. Three thermocouples will be used as follows: (1) at the orifice (2) midway between top and bottom of the crucible and (3) at the bottom of the crucible.

5. An all-tantalum crucible will be used together with the sharp edged inner cups described by Phipps, Seifert, and Simpson to discourage creeping.

6. A carefully prepared sample of postum will be used.

7. A mechanical shutter is being designed which will more effectively seal the collimator opening.

Work has begun on designing and constructing the above apparatus. It is believed that eight to twelve months may be required for satisfactory completion of the above program.

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