AN IMPROVED METHOD FOR THE DETERMINATION OF THE RADIUM CONTENT OF ROCKS.

Thesis

by

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ABSTRACT.

A modified form, developed in this laboratory, of the furnace method for separating radium emanation from rocks originally devised by J. Joly, is described. In the new method the rock is heated with a flux in a nickel crucible by means of a gas flame, the carbon dioxide produced is removed by a sodium hydroxide solution, and the emanation is introduced into an electroscope. The procedure is simpler than that of Joly and gives better results.

Results of tests of this method and of Joly's method are given.

Some results of ~~tests~~ determinations of the radium content of various rocks are given.

Suggestions for further improvement of the method are presented.

INTRODUCTION.

Since the discovery of radioactivity, the activity of rocks has been a matter of great interest, especially since it has been found that practically all rocks contain a small amount of radioactive substances. The determination of the activity of ordinary rocks (not radioactive ores) offers considerable difficulty, however, on account of the very small amounts of substances to be measured.

One of the radioactive elements, radium, has the special advantage that it has a fairly long-lived gaseous active emanation which can be separated from it and led into an electroscope where it can exert to the full its ionizing powers. Hence radium can be measured in extremely small quantities. It is this element alone whose measurement is discussed in this paper.

The greatest difficulty encountered in determining the radium content of rocks is in the removal of the emanation from the rock. Two general methods for accomplishing this have been used: the solution method, in which the rock is put into solution and the emanation removed from the solution by boiling, and the furnace method, in which the emanation is removed directly from the rock by heating. The former is widely used, but is time-consuming and allows many possibilities of error. (A good description of this method will be found in a paper by S. C. Lind.[1])

The attempt to remove the emanation from a rock by heat was made very early. At first the rock was simply heated to a high temperature, generally by means of a carbon resistance furnace or an arc. Methods of this type are described by Strutt[2], Boltwood[3], and Fletcher[4]. They were found to be unsatisfactory because of incomplete and uncertain removal of the emanation.

The first method using a flux to bring the rock into solution while heating was devised by J. Joly[5] in 1911. In two later papers[6,7] he gives some results obtained and some discussion of the method. A complete description of the apparatus and procedure used will be found in Joly's 1911 paper and in a thesis by J. L. Bohn[8]. A short description of the ~~#pp#r######~~ apparatus is given later in this paper, in connection with the results obtained in testing this method.

The chief objectionable features in this method are: the rubber bag used as a gas reservoir probably adsorbs some of the emanation, since rubber is known to be an adsorbent for emanation; the soda lime tube may also take up some emanation, and is a very uncertain agent for removing the carbon dioxide from the gases, especially when it has already been used several times; the electric furnace is troublesome to handle and expensive, requiring a platinum crucible and platinum resistance wire.

With these things in ~~having~~ mind , and having made enough experiments with the Joly apparatus to be convinced that it could be improved, the attempt was made to devise a different method, aimimg at consistency of results, simplicity, portability, and inexpensiveness.

The result, while capable of further improvement, seems to represent an actual advance over the former method, judging from the results obtained in this laboratory.

Another modification of the furnace method has been developed very recently by C. S. Piggot[9,10] at the Geophysical Laboratory, Washington, D.C. He uses a resistance furnace similar to that used by Joly but much larger, and replaces the soda lime and the rubber bag by a sodium hydroxide solution and a mercury displacement gasholder. The results obtained are no better ### than those obtained with the apparatus described in this paper, and the apparatus is much more cumbersome and very expensive. It is possible that with further developement this method will be capable of very great accuracy, but it will hardly come into general use for the reasons given above.

The suggestion has been made that it would be much simpler to determine the radioactivity of rocks by a measurement of the penetrating gamma rays emitted, thus obviating the necessity of removing the emanation from the rocks. This has been tried by Dorsey[11] and Ludewig[12] and has been found to be insufficiently sensitive to detect the very small quantities of radioactive sustances with which one deals in working with ordinary rocks.

The investigation described in this paper has been undertaken primarily for geological reasons. It is thought that the variations in the radium content of the rocks in a region may serve as an indicator for showing the relationships of the various geological formations in the region. Much work will be necessary before it can be determined whether this idea has any value, and it is obviously necessary, in order to carry out this work, to have a simple and quick method for making determinations of the activity of rocks. This may develop into a geological method of great value.

DESCRIPTION OF APPARATUS.

(See Plate 1.)

The furnace is at C, D is a trap to collect water coming from the melt, E is an absorption tube containing a solution of sodium hydroxide, F is a small condenser, G is a gasholder, K and L are drying tubes containing concentrated sulphuric acid and phosphorus pentoxide respectively, and O is an electroscope.

The cycle of operations is, roughly: with stopcocks A and H closed, the melt is heated in the furnace, carbon dioxide being generated and being absorbed in E. Unabsorbed gases collect in G, which is so arranged that the system is kept under reduced pressure. Meanwhile the electroscope has been evacuated through P, which is now closed and N opened. As soon as G is empty, the stopcock on its exit tube is closed, the whole system allowed to stand at low pressure for a while to remove as much gas as possible from the melt, and then A is opened and the electroscope allowed to fill slowly while the solution in E is allowed to boil gently, the steam formed condensing in F. The liquid in D is also boiled. When gas has ceased to enter the electroscope, the stopcock H is turned so as to connect the electroscope to the atmosphere, allowing a little more air to enter.

Then the leaf of the electroscope is charged, allowed to stand 30 minutes, its position read, allowed to stand 60 minutes, and its position read again. The difference between these readings, minus the "zero leak", is the leak for the sample being investigated. The zero leak is determined by filling the electroscope with air, charging, and taking two readings at intervals of 30 minutes, multiplying the resulting difference by two. This is done before every determination, as the zero leak is found to vary somewhat. (It is not necessary to take the zero leak over the full period of one hour because the rate of leak does not vary appreciably with time, as is the case with the leak due to radium emanation.)

Details of apparatus.

<u>The furnace.</u> (See Plate 2.) (Actual size.)

This consists of a nickel crucible C and a steel cover A. The cover is held on by a steel ring B and ten screws. It makes contact with the crucible by means of a spherical surface, ensuring an air-tight joint. A flange is made on the crucible by pressing between two conical dies, after carefully annealing to prevent cracking of the nickel. (The crucible, of 75 cc. capacity, is from the Braun Corporation, Los Angeles.) Two steel tubes screwed into the cover serve as entrance and exit tubes for gases. D is a system of two nickel baffle plates to keep heat from the cover, and is supported by a nickel screw and two nickel bushings; holes are drilled in the plates opposite the entrances of the tubes, to allow free circulation of gases when "sweeping out" the furnace.

The furnace is supported on a tripod. Across this is laid a sheet of asbestos, then two metal bars (shown in cross section), and over these a second sheet of asbestos. The heating is done by a Fisher burner E, which is provided with a ##### compressed-air blast. F is a cylinder of asbestos which serves as a shield against #### heat escape and increases the temperature attainable; it is supported by nichrome wires shown as dashed lines.

Connection is made to the furnace by two pieces of Pyrex tubing which fit over the ends of the steel tubes and are fastened with sealing wax. The cover of the furnace is kept cool by dripping water into the space formed by a circular flange on the upper surface. The stream of cooling water is regulated so that the rate of flow is just equal to the rate of evaporation from the furnace. Distilled water is used to avoid the formation of scale.

<u>The sodium hydroxide tube.</u> (See Plate 3.)

This is made of Pyrex in the shape shown. The little indetations along the top cause bubbles of gas to travel slowly along the tube, making the absorption of carbon dioxide more complete. The bulb on the right end is to prevent the solution from ~~drawin~~ being drawn into the furnace in case there is a back-flow of gas; the enlargement on the left is to facilitate the boiling of the solution, which takes place with a current of gas passing through the tube from right to left. A special funnel, shown in the diagram, is used for putting the solution into the tube. (The dashed line in the diagram shows the level of the solution. It consists of 10 grams of sodium hydroxide dissolved ~~on~~ in 120 cc. of distilled water. A new charge is used for each determination.)

The gasholder. (Plate 3.)

This is sufficiently explained by the drawing. The purpose of the double-walled outer vessel is to decrease the amount of mercury required. The movable part is held upright by a string attached to the upper portion and passing over a pulley to a small counterweight. (A pressure less than atmospheric is automatically maintained in this gasholder because of the mercury displaced by the movable part.)

The drying tubes.

The sulphuric acid in K needs to be renewed occasionally when it gets dark due to organic dust carried into it. The phosphorus pentoxide in in L lasts a very long time. It is important to pack cotton wool in the side of L next to the electroscope to prevent any phosphorus pentoxide from being carried into the latter.

The volume of the gaseous space in the whole system, excluding the electroscope, is 270 cc.

The electroscope.

The electroscope is of a type designed by C. T. R. Wilson, in which the gold leaf is suspended within the emanation chamber. A description will be found in the original paper by Wilson[13] and in the thesis by Bohn mentioned above.

The position of the leaf is determined by a Leitz reading microscope. The leaf is charged to a potential of about 300 volts by means of a charging device consisting of a variable condenser, a 90 volt battery, and a key. The device is operated by charging the condenser and then decreasing its capacity until the leaf reaches the proper potential, then insulating the leaf.

The volume of the electroscope is 1250 cc.

It is necessary that the leaf be charged at least ten minutes before readings are taken, otherwise an abnormally large leak will be observed, on account of "electrical seepage" into the quartz insulation.

With the potential used it will be found that the electroscope will discharge suddenly while it is being evacuated; this occurs at a pressure of about 1 mm. and is due to a discharge through the residual gas.

DETAILS OF PROCEDURE.

Evacuate electroscope, refill with air through cock H, and charge, closing N. Meanwhile weigh out sample, mix with flux in crucible, prepare sodium hydroxide solution and fill absorbing tube, and assemble apparatus. With all air out of

G close cocks H and A and open cock on G. The run can now begin, following this schedule:

0:00- Start heating crucible gently. (Increase the temperature gradually until 0:45, when it should be at its maximum of about 1000° C. ; it should be kept at this temperature until 1:00.)

0:05- Read electroscope.

0:35- Read electroscope. The difference between these readings, multiplied by two, gives the "zero leak". Start evacuating electroscope through P.

0:45- Turn H so as to communicate between G and ~~P~~ O, close P and open N, using the pinchcock M to regulate the flow of air. As soon as the mercury in G comes above the stopcock, close this stopcock. Allow the whole system to remain under reduced pressure for about three minutes, then close M, open A (using B to keep the flow from being too rapid) and, as soon as the system reaches atmospheric pressure, open M slightly. With a slow stream of air passing through the apparatus into the electroscope and water passing through F, boil the solution in E gently with a Bunsen flame. Also boil the water in D ### and drive out any moisture ### in the tube leading to the furnace by means of the flame.

1:00- By this time the filling of the electroscope should be complete. Turn H so as to communicate with the atmosphere, charge the leaf, and then close N. Take apparatus apart and clean E, D, and C thoroughly.

1:30- Read electroscope.

2:30- Read electroscope. The difference between these readings gives the "total leak" for the sample. Clean out electroscope by drawing air through H for a while, then evacuating and refilling.

It is necessary to wait at least three hours between determinations in order to allow the active deposit formed in the electroscope to disintegrate. When only one electroscope is available it is better not to attempt more than two determinations in a day.

(The procedure given above is not necessarily the best possible procedure; perhaps a different time schedule would give better results. The schedule given is simply one that has been tried and found to give satisfactory results. The same is true of the amounts of sample and flux used. An investigation in which the effect of varying these factors is determined would be of some value.)

THE FLUX, SIZE OF SAMPLE, ETC.

Three grams was found to be a good size for the sample, and, in order to keep conditions uniform, this was kept constant in all of the work done. The flux used consists of sodium carbonate, potassium carbonate, and boric acid in the proportions of 2:2:1 by weight. 20 grams of this are used for each sample.

The rock samples are prepared by grinding the rock in an iron mortar and sifting through a 60-mesh screen.

Considerable trouble was experienced with some of the rocks which came from an oil well core, the effect being that the crucible developed cracks when these rocks were used. It was found that these rock were incompletely oxidized and contained traces of sulphides, the sulphur attacking the nickel crucible. This trouble was overcome by adding a small amount (½ gram) of potassium chlorate to the melt when working with these rocks.

THE STANDARDS.

Two standards were used. First, a standard uranium ore (Carnotite) put up by the Denver Fire Clay Co. and containing 1.58% uranium, and second, a standard radium solution from the Radium Institute of Chicago containing 4.87×10^{-11} grams of radium per cc. Since 1 gram of uranium contains in equilibrium with it 3.4×10^{-7} grams of radium, the standard ore contains 5.37×10^{-12} grams of radium per milligram.

These two standards have been compared by Mr. J. L. Bohn and found to agree with one another.

A correction must be made for the "emanating power" of the standard ore, that is, for the fraction of the emanation produced by the ore which is given up to the air and hence lost. The determination of the amount of this correction is described later.

THEORY OF THE METHOD.

The basic assumption of this method of determining the radium content of rocks is that the fraction of the total amount of emanation present ######## in a melt that goes into the electroscope is independent of the source of the emanation. Making this assumption, and finding the leak due to a rock sample and that due to the same amount of the same rock plus a known amount of standard ore, one can calculate the radium content of the rock. Since the whole melt becomes a homogeneous mass, and the amount of material in the standard used (about 10 mg.) is small, this assumption seems to be susceptible to no objections.

The results of calibrating the apparatus as explained above are expressed in the constant k, giving the number of grams of radium in the melt corresponding to one division per hour leak of the electroscope. There is no a priori reason that this should be the same for different rocks, but it was found to be the same for all of the rocks tried, within the limits of error of the calibration. This indicates that the fraction of the emanation removed from the melt is independent of the nature of the melt; in fact, the evidence is that the removal is nearly complete in all cases.

A calibration of the electroscope directly in terms of emanation was made also. (This does not enter directly into the determination of of the activity of rocks, but is necessary in the determination of the emanating power of the standard. A tube, shown in full size on Plate 3, was made of Pyrex. One cc. of the standard radium solution was put into it together with about ten cc. of water, which was boiled gently with a slow current of air passing through, and then both ends were sealed. After a measured time interval the larger end was connected to the system at F, the seals broken, and the electroscope filled through it while the solution was boiled. Thus a known amount of emanation was introduced into the electroscope. The same thing was done connecting the emanation tube at D, boiling for ten minutes while air was drawn through it into G, then boiling the solution in E as when making a determination on a rock. Thus the amount of loss of emanation in E was determined.

Determination of the emanating power of the standard.

A weighed amount of the standard ore was put into into a glass tube, which was sealed and allowed to stand for a measured length of time. Then the seals were broken and a slow stream of air drawn through the tube into the electroscope, and the resulting leak observed. From this and the absolute calibration of the electroscope the amount of emanation given up can be determined, and, since the time of standing is known, the equilibrium amount can be calculated. The emanating power is theh the ratio of this amount to the known total amount of emanation in equilibrium with the standard. The amount which is obtained when a sample of standard is used for calibrating the apparatus is simply the amount occluded in the ore, which is obtained by multiplying the calculated total amount by one minus the emanating power.

RESULTS.

1. Absolute calibration of electroscope.

In each of these determinations 1 cc. of the standard solution was used. 1 and 2 were obtained with one 1 cc. sample, 3 and 4 with another. In 2 and 4 the sodium hydroxide tube was used.

(In giving the results, only the differences between the total and the zero leaks will be given. The zero leak is generally about 0.30 divisions per hour. In the table below, r is the ratio of the number of curies of emanation introduced to the number of divisions leak.)

no.	time of standing	curies emanation	leak	r
1	6d. 21h.	$34.6\text{x}10^{-12}$	3.52	$9.83\text{x}10^{-12}$
2	14d. 2h.	44.9 "	3.99	11.24 "
3	12d. 0h.	43.0 "	4.92	8.75 "
4	10d. 1h.	40.8 "	4.13	9.88 "

ratio r_1/r_2 is 0.875, r_3/r_4 is 0.885, mean 0.880.
mean of r_1 and r_3 is $9.3\text{x}10^{-12}$.
Therefore the absolute value of r #### for the electroscope is $9.3\text{x}10^{-12}$, and the amount of loss in the sodium hydroxide tube is 12%.

The fact that the ratios r_1/r_2 and r_3/r_4 agree better than r_1 and r_3 indicates that the greatest error is in the measuring out of the sample with a 1cc. pipet.

2. Emanating power of standard ore.

Weight of sample 17.0 mg.

time of standing 14 days.

leak 4.86; corrected to infinite time of standing 5.28.

therefore amount of emanation given up is 49.1×10^{-12} curies.

total amount of emanation produced is 91.4×10^{-12} curies.

Therefore the emanating power is 0.533, and #### the correction factor by which the amount of standard used in calibrating the ~~electroscope~~ apparatus should be multiplied is 0.467.

3. Calibration of apparatus under actual operating conditions.

(Weight of rock sample is 3 grams in each case.)

Rock no. 1.

A. rock and flux.

values of leak: 0.48, 0.41, 0.44, 0.42.

mean: 0.44.

B. rock, flux, and standard ore.

mg. standard	leak	leak due to standard	k
18.5	5.60	5.16	9.00×10^{-12}
11.5	4.64	4.20	6.88 "
8.5	3.26	2.82	7.57 "

Mean value of k is 7.82×10^{-12}.

Rock no. 2.

A. values of leak: 0.36, 0.37; mean 0.37.

B.

mg. standard	leak	leak due to standard	k
10.6	3.49	3.12	8.53×10^{-12}
12.2	4.39	4.02	7.62 "

Mean value of k is 8.08×10^{-12}.

Rock no. 3.

A. values of leak: 0.37, 0.40; mean 0.39.

B.

mg. standard	leak	leak due to standard	k
11.8	3.95	3.56	8.31×10^{-12}
10.5	3.39	3.00	8.78 "

Mean value of k is 8.60×10^{-12}.

Rock no. 4.

A. values of leak: 0.49, 0.48; mean 0.49.

B.

mg. standard	leak	leak due to standard	k
9.0	3.10	2.91	7.78×10^{-12}
17.9	6.27	5.78	7.78 "

Mean value of k is 7.78×10^{-12}.

(Petrological descriptions of the rocks will be given later.)

The variation of the results when the standard is present is seen to be greater than that when the rock alone is used. This is probably due to the large value of the emanating power of the standard, considerable variations in the amount of emanation lost being possible when the total loss is so large.

Since the fluctuation among different values of k obtained for a single rock is as large as that among values obtained for different rocks, it is impossible to say whether there is an intrinsic difference in this constant for different rocks; but it can be concluded that such a difference is less than the limit of accuracy at present attained. Hence the mean of all the values of k obtained above will be used in calculting the fadium contents of the rock samples dealt with in this paper. This mean value of k is $\underline{8.07\times10^{-12}}$.

4. Other general results.

To determine the effect of very fine grinding on a rock, some of rock no. 1 was ground in an agate mortar to a very fine powder and the leak due to a 3-gram sample determined. It was found to be 0.40. This is less than that for the rock not finely ground, indicating that there is some loss of emanation in the fine-grinding process.

The leak due to the flux alone was determined by making a run with no rock or standard in the furnace. The resulting leak was less than 0.01, showing that the radioactive contamination of the apparatus and chemicals is negligible. The same was found to be true for the potassium chlorate used.

The addition of ½ gram of potassium chlorate to the flux was found to have no effect on the leak produced by a sample of an igneous rock, but increased the leak produced by the incompletely oxidized sulphur-containing rocks by about 15%, bringing the value of k for these rocks to the same value as that for the igneous rocks. It is possible that a substance is formed by these rocks under reducing conditions which is capable of adsorbing emanation.

Potassium nitrate and potassium bichromate were also tried as oxidizing agents; the former produces nitric oxide which causes trouble in the electroscope, the latter has a tendency to attack the crucible.

Some runs were made with the standard and flux alone, to determine the effect of the presence of the rock. The results were:

mg. standard	leak	k
5.8	1.75	8.32×10^{-12}
12.5	3.91	8.04 "
4.8	1.53	7.87 "

mean value of k is 8.08×10^{-12}.

Since the value of k is the same as that obtained with a rock present, it is concluded that the rock has no important effect on the removal of the emanation from the melt.

In case work is done with very active samples, it may be necessary to decrease the period over which the leak is taken to ½ hour. With this in mind, several determinations of the ratio of the 1-hour leak to the leak during the first half hour of this period were made. The mean ratio given by eleven such determinations is 2.08.

A determination was made of the emanating power of one of the rock samples (no. 4 was chosen because it is a finely-divided shale which might be expected to have a large emanating power.) The result showed that the emanating power was less than 2%, and therefore negligible in comparison with other sources of error.

5. Results with electric furnace.

Before the apparatus described above was developed, the author was working with an electric furnace built by Bohn[8] according to the design of Joly[5]. In this apparatus the melt is heated in an electrical resistance furnace, the carbon dioxide produced being absorbed in a soda-lime tube and the excess gas collecting in a rubber bag. The rest of the procedure is essentially the same, the same electroscope being used. This made method was found to be unsatisfactory, and many changes were tried. The soda-lime tube was especially troublesome. When the sodium hydroxide absorbing tube described above had been made, it was decided to make a final test of the electric furnace using this tube in place of the soda-lime tube. It was impossible to replace the rubber bag with the gasholder because the mercury seal on the furnace will not withstand any but very slight differences of pressure. Only 15 grams of the flux are used with this furnace on account of the small size of the crucible.

Results.

A. Rock (3 gram samples of no. 1) and flux.

values of leak: 0.44, 0.49, 0.44, 0.60, 0.48.

mean: 0.48.

B. Rock, standard, and flux.

mg. standard	leak	leak due to standard	k
5.4	2.55	2.07	6.56×10^{-12}
5.7	2.29	1.81	7.91 "
8.4	3.22	2.74	7.70 "

Mean value of k is 7.39×10^{-12}

Radium content of rock is 1.18×10^{-12} grams Ra/gram rock.

C. Standard and flux.

mg. standard	leak	k
8.5	2.33	9.20x10^{-12}
14.0	4.52	7.79 "
8.2	2.72	7.59 "

mean value of k is 8.19x10^{-12}.

It will be seen that the results with the rock alone do not agree with one another quite as well as corresponding results with the nickel crucible furnace. The value of k obtained when the rock is present is noticeably lower than that obtained with the new furnace, indicating a more complete removal of the emanation (probably because a slightly higher temperature is reached); but this advantage is over balanced by the uncertainty and inconvenience of the method. The result obtained for the radium content of the rock agrees very well with that obtained by the new method.

6. Results of determinations of the radium content of rocks.

Description of samples investigated:

no. 1. Biotite granite. Stream pebble from Arroyo Seco, Pasadena.

no. 2. Pegmatite. Surface rock from region of Arrowhead Lake, California. Collected at site of penetrating radiation observations.

no. 3. Micaceous shale. This and the succeding samples are from the core of an oil well in Southern California. (Well Bolsa 36.) Depth 2052 feet.

no. 4. Micaceous shale. Depth 2248 feet.

no. 5. Micaceous shale. Depth 2459 feet.

no. 6. Sand. Depth 2659 feet.

no. 7. Micaceous shale (foraminiferal). Depth 2852 feet.

Results:

sample no.	leak	mean value of radium content
1.	0.48	1.18x10^{-12} grams Ra/gram rock.
	0.41	
	0.44	
	0.42	
2.	0.36	0.99 " "
	0.37	
3.	0.37	1.04 " "
	0.40	
4.	0.49	1.31 " "
	0.48	
5.	0.35	0.93 " "
	0.34	
6.	0.15	0.43 " "
	0.17	
7.	0.46	1.30 " "
	0.50	

DISCUSSION AND CONCLUSION.

The chief source of error and uncertainty in this experiment is in the standard, as can be seen by a comparison of those results in which the standard is involved with those in which a rock alone is present, the much greater concordance of the latter being evident. The cause of this uncertainty in the standard is its large emanating power; since more than half of its emanation is lost to the air, it is too much to expect that the loss should be constant for all samples of the standard taken. Also, the determination of the amount of emanation lost by the standard involves the use of a subsidiary standard (the standard solution), increasing the chances of error.

This trouble can be overcome by the preparation of a standard of low emanating power. Such a standard has been prepared and will be tested. It should increase the reliability of the results greatly. (It was prepared by fusing a weighed amount of the standard ore with borax glass in the proportions of about 1:20, and coarsely grinding the resulting mixture.)

The furnace and the absorption tube and gasholder behave very satisfactorily, and only minor changes in them are suggested. In spite of the use of potassium chlorate, the crucible still shows some tendency to be disintegrated by the sulphide-containing rocks, so that if determinations are to be made on many such rocks it may be advisable to use a platinum crucible of the same design as the nickel crucible.

The process of boiling the sodium hydroxide solution would be made easier by enlarging the bulbs at the ends of the absorption tube.

Work will be done later on the use of a Wulf quartz fiber electroscope in place of the present instrument. It is hoped that this will increase the sensitivity of the method as well as making the apparatus more portable.

Quartz insulation has been found to be bad for the reason that it becomes a poor insulator unless its surfave is perfectly dry. This condition is impossible to fulfill when air is constantly being drawn into the electroscope, in spite of the use of drying tubes. In future instruments, therefore, amber insulation will be used.

In concluding, I wish to express my obligations to Mr. René Engel, who made the petrological investigations of the rock samples; to Mr. Julius Pearson, who assisted in designing the furnace; and to the Standard Oil Company of California, whose financial assistance has made this research possible.

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(Note added after completion of thesis.)

A new calibration of the apparatus, using the new standard whose preparation is described on p. ~~22~~ 23 of the thesis, was made.

The data concerning this standard are:

Weight of standard ore used	1.025 g.
Weight of mixture after fusion	20.1 g.

Therefore one centigram of the mixture contains 2.79x10^{-12} g. Ra.

Its emanating power, determined by the method described in the thesis, is 2%.

It was allowed to stand over 30 days before use.

Using this standard and rock no. 1, the apparatus was then calibrated. (A determination of the leak produced by the rock alone was made, to test the constancy of the apparatus; this gave the result 0.44, identical with the mean value previously obtained.) The results of the calibration were;

wt. of std.	leak	leak due to std.	k
7.90 cg.	2.99	2.55	8.48x10^{-12}
9.85 "	3.57	3.13	8.60 "
4.90 "	2.00	1.56	8.59 "

The mean value of k is 8.56x10^{-12} g. Ra/division leak.

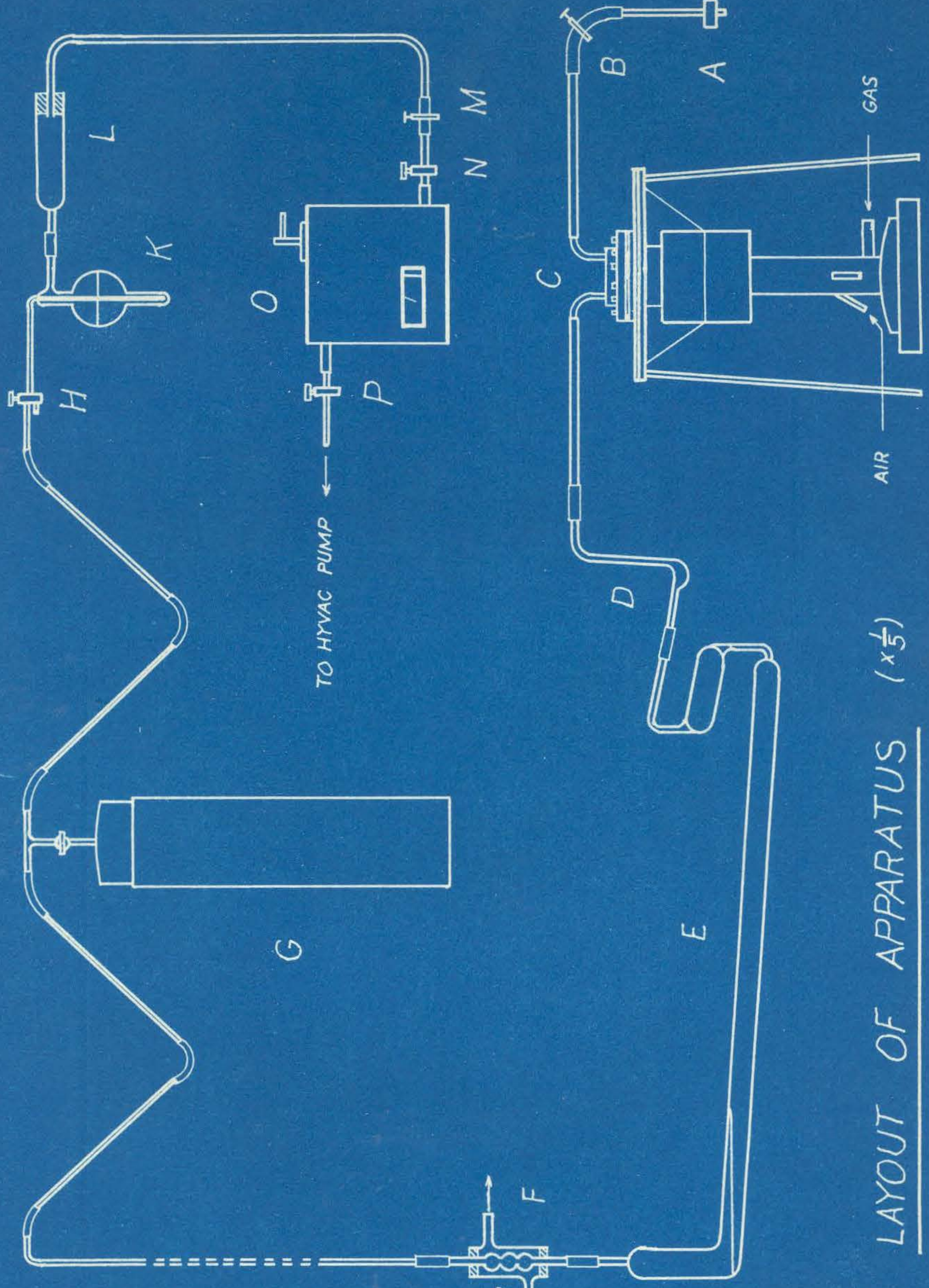
The individual values agree much better with one another than those of the previous calibration using the standard ore directly. The mean differs from that previously obtained (8.07×10^{-12}) by 6%, which is within the range of variation of individual values obtained before.

We conclude that the new ### standard actually does allow a greater degree of certainty and accuracy than does the standard ore itself, for reasons given in the thesis (p. ~~22~~ 23).

The results obtained with the rock samples investigated should be corrected to the new calibration, which can be done by multiplying them by 1.06. The corrected results are:

sample no.	radium content	
1.	1.25×10^{-12} grams	Ra/gram rock.
2.	1.05 "	"
3.	1.10 ""	"
4.	1.39 "	"
5.	0.99 "	"
6.	0.46 "	"
7.	1.38 "	"

Plate 1.



LAYOUT OF APPARATUS ($\times\frac{1}{5}$)

Plate 2.

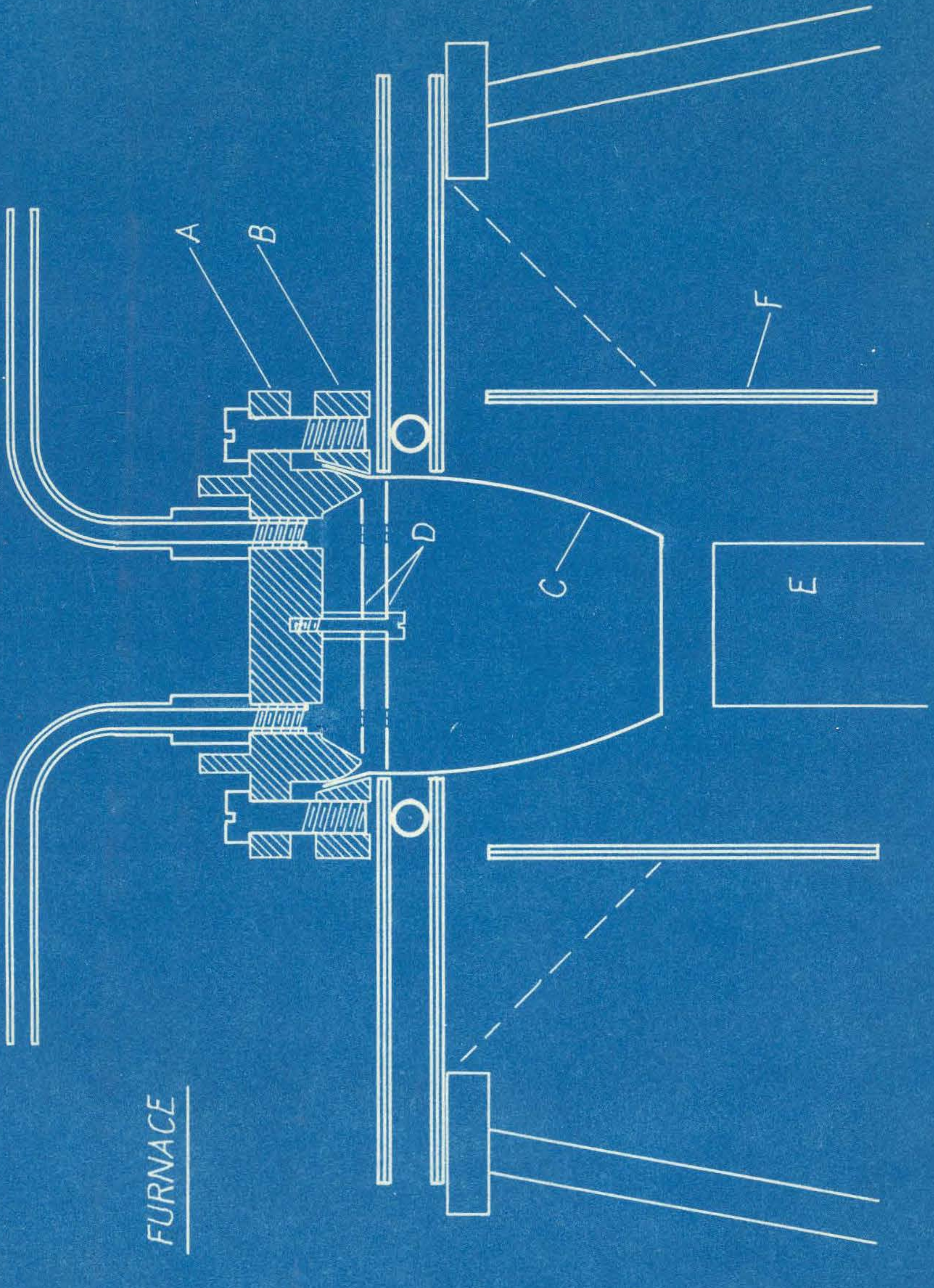


Plate 3.

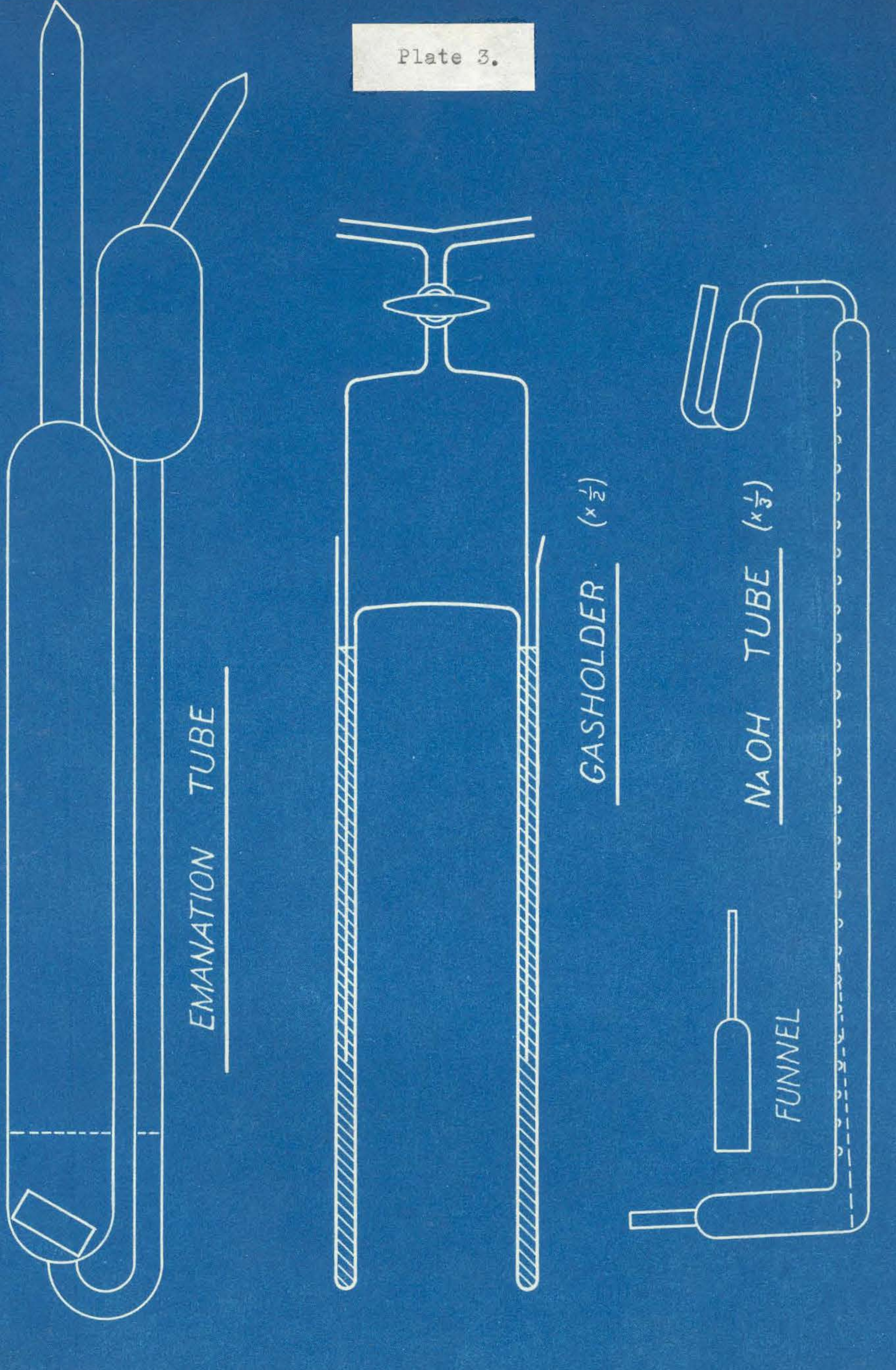
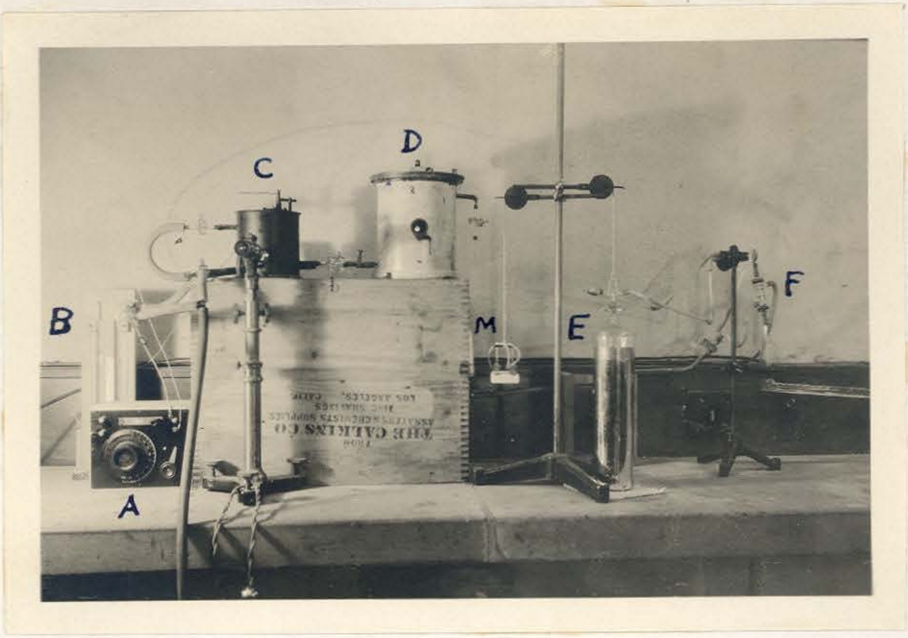
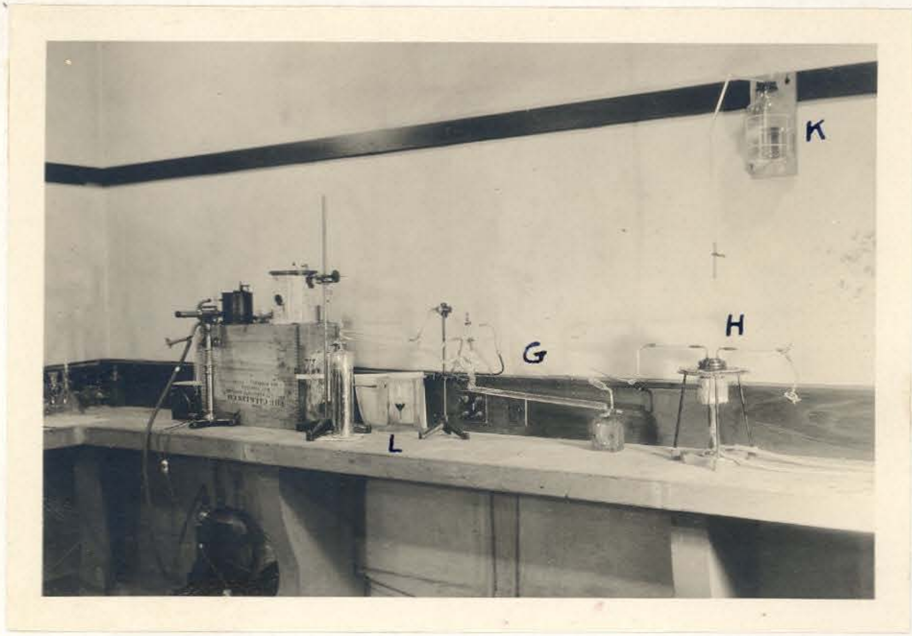

EMANATION TUBE
GASHOLDER $(\times\frac{1}{2})$
NaOH TUBE $(\times\frac{1}{3})$
FUNNEL

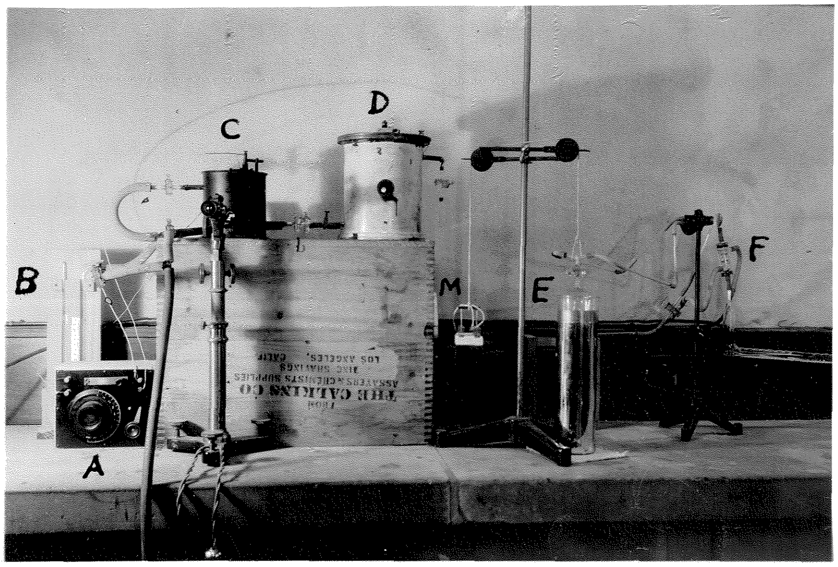
Plate 4.

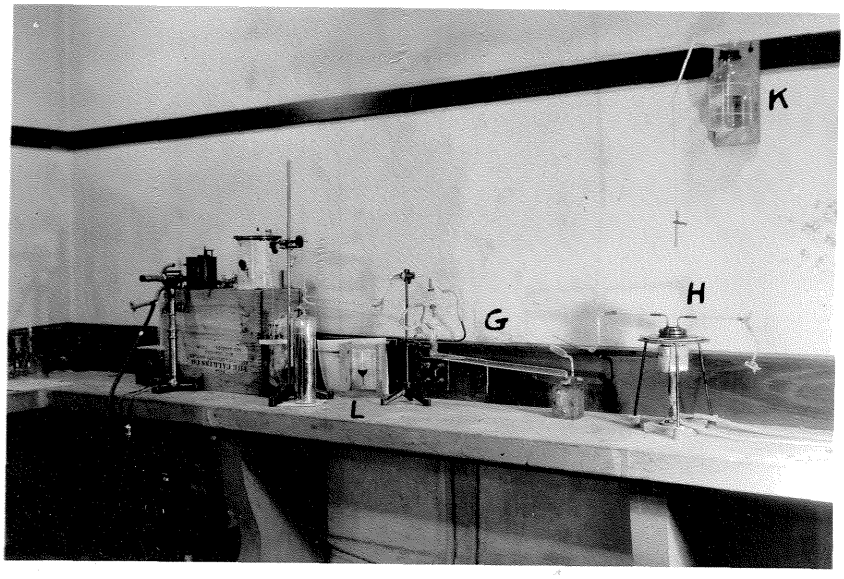




A- Charging device.
B- Mercury manometer for determining pressure in electroscope.
C- Electroscope.
D- Wulf electroscope (not used).
E- Gasholder.
F- Condenser.
G- Sodium Hydroxide tube.
H- Furnace.
K- Cooling water reservoir.
L- Sulphuric acid bulb.
M- Phosphorus pentoxide tube.

Plate 4.





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