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METHODS FOR THE ESTIMATION OF CARBON DIOXIDE IN MINERALS AND ROCKS.

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There seems to be two principal methods employed for the estimation of carbon dioxide in a mineral or rock; the one devised by Fresenius and the other by his rival Bunsen. The Fresenius method has been more or less modified by different analysts. In its essential features, however, it is substantially as follows: The flask *K* for the decomposition of the substance has a capacity of 200 to 300 c.c. The flask is closed with a two-hole rubber stopper. The safety tube *a* passes through one hole, and a bulb tube *b* through the other. A funnel is connected with the safety tube at *a* by a rubber tube. Thus the addition of acid can be regulated by the pinch cock at *o*. *d* and *e* contain soda lime and caustic potash respectively. These are connected with the safety tube after the acid has been added and the substance in the flask *K* has been dissolved. The bulb tube *b* serves to condense the steam.

The first U-tube, *f*, contains calcium chloride in its lower portion and the second tube, *g*, is filled with granular calcium chloride. These tubes remove the moisture. To absorb the hydrogen chloride, the tube *h* contains small pieces of pumice stone which have been boiled in a concentrated solution of copper sulphate and afterwards dried in the air bath at 250° to 300° C. A calcium chloride tube, *i*, is connected with *h*. The carbon dioxide is absorbed by the two U-tubes *k* and *l*. They are 11 cm. long and 12 mm. in diameter, and are ⅔ full of coarse soda lime, the remaining space containing calcium chloride. To prevent backward diffusion of carbon dioxide or water, the tube *w* is filled with calcium chloride on the side next the appara-

tus and with soda lime on the other side. The bend of the tube *n* is filled with water which makes it possible to observe the rate of the reaction.

The absorption tubes *k* and *l* are weighed and two or three grams of the substance are placed in the decomposing flask, then the apparatus is tested to learn if all the joints are tight. Dilute hydrochloric acid is slowly added from *c*. When the action has ceased, the funnel *c* is removed and the tube *d* is connected with the funnel tube *a*, and a slow current of air is aspirated through the apparatus, while the flask *K* is heated until the liquid boils. This sweeps the carbon dioxide into the absorption tubes. When the apparatus is thoroughly cooled the tubes *k* and *l* are weighed, the increase representing the amount of carbon dioxide in the specimen. Some recommend the use of only one soda lime tube, but two make the absorption of all the carbon dioxide more certain.

One of the principal difficulties in connection with this method is the possibility of the escape of the carbon dioxide through the numerous rubber connections. Every one who has made an ultimate analysis by combustion understands how constant a source of error the joints are, and how carefully one must guard losses through the walls of rubber tubing.

A much simpler method than the one described was devised by Bunsen, shown in Fig. 2. This method is not usually described in the text-books. A gram of the finely pulverized substance is weighed into the flask, *d*. The bulb *c* is nearly filled with a mixture of one part concentrated hydrochloric acid and three parts water. The bulb *h* contains cotton which assists in condensing the vapor. Attached to the bulb is a small tube filled with fused calcium chloride. The apparatus from *c* to *e* inclusive is carefully weighed. By means of a rubber tube attached to the calcium chloride tube *e*, the dilute acid is started into the flask *d* by suction with the mouth. When all the liquid has passed over, the apparatus is held in the hand and gently warmed, until the powder is dissolved or effervescence ceases. The apparatus is then connected

PLATE XXVIII.

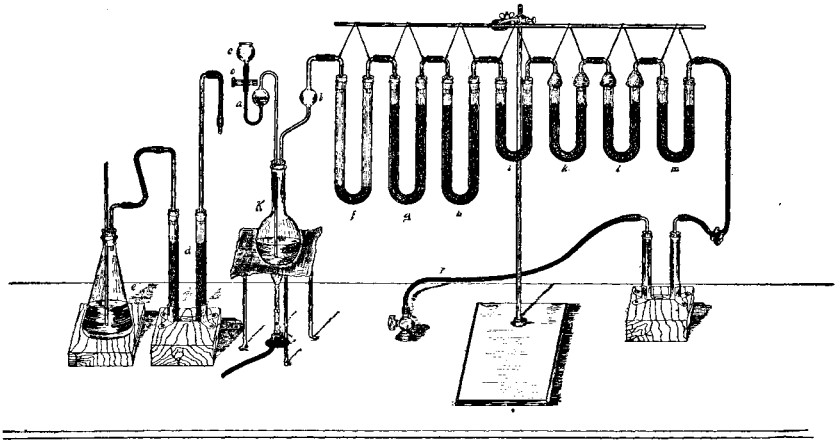

FIG. 1. The method of Fresenius.

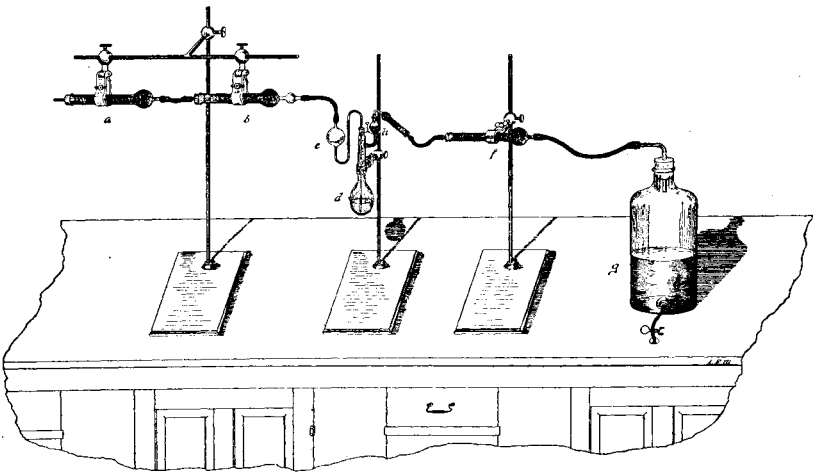

FIG. 2. The Bunsen method.

with two freshly filled calcium chloride tubes on one side and a calcium chloride tube and aspirator on the other side as shown in the figure. Air is drawn through the apparatus for about twenty minutes while the flask *d* is immersed in a beaker of distilled water to cool it. The apparatus is carefully wiped with silk and weighed, the loss, of course, representing the amount of carbon dioxide. The apparatus is gently warmed again, and the air aspirated through as before while the flask is in the beaker of distilled water. A weight that is practically constant is readily attained.

The writer has made scores of determinations by this method, and has supervised hundreds more that have been made by students, especially in Iceland spar, dolomite and siderite. There is not much difficulty in securing a result that differs not more than one-tenth of a per cent from the theoretical. It might seem that there would be a tendency to get too high a result, but such is not the case, providing the rock substance has been sufficiently pulverized, and the calcium chloride in tube *e* is of good quality and is changed sufficiently often to keep it in proper condition. Besides, the acid becomes further diluted as soon as it begins to act on the powder, and at first the action takes place with very little heat.

Where it is desirable to make a carbon dioxide determination in a sulphide like chalcopyrite, it is necessary to prevent the escape of sulphuretted hydrogen as this would give a result too high. This is accomplished by the use of dilute sulphuric acid instead of hydrochloric acid. If there is still an odor of sulphuretted hydrogen, a small quantity of powdered copper sulphate, ferric sulphate, or potassium dichromate is introduced into the bulb with the powdered rock. The carbon dioxide can be easily determined by this means.

After many years of trial, the writer commends this method on account of its simplicity and accuracy. It is a method that even the student with but small quantitative experience can use with success. His ability to secure good results at the outset increases his interest for the further prosecution of his quantitative work.