[CONTRIBUTION FROM THE SHEFFIELD CHEMICAL LABORATORY YALE UNIVERSITY.]

THE PENFIELD TEST FOR CARBON.

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Received January 4, 1917.

The way the late Professor S. L. Penfield tested for carbon and carbonates in minerals is not, to our knowledge, in the literature of analytical chemistry. The method is novel only in the delicate way he used a well-known test. He fused a mixture of substance and lead chromate in a small horizontal, hard glass tube, Fig. 1, containing near the open end a

Fig. 1.

droplet of a solution of barium hydroxide. The appearance of a film of barium carbonate indicated the presence of carbon in the substance. If no film is seen when fusion is effected the open end of the tube may be closed with the finger to keep out carbon dioxide from the air and the tube at once removed from the lamp flame. Then the result may be carefully observed.

The method has been used in this laboratory a number of years, especially for testing metals for carbon. It is so delicate that lead chromate which has been exposed to the air in preparation will react for carbon from the dust in the air. Lead chromate precipitated from the nitrate, washed by decantation and dried in a beaker covered with a watch glass has been found to be free from carbon. Fused and pulverized chromate may be freed from carbon by heating in an atmosphere of oxygen as described later. Glass tubes double the required length may be cleansed by wiping the inside with a wet rag or by a mixture of bichromate and oil of vitriol and then rinsed with distilled water. They should be dried in a horizontal position, as dust might enter if they were inclined. The closed tubes are made in the usual way and if not used at once they should be protected from dust.

In order to determine the delicacy of the method the following experiments were made: The lead chromate used was precipitated from the nitrate, washed, dried, fused and pulverized. It was then placed in a hard glass tube, Fig. 2, and heated as hot as possible without sintering,

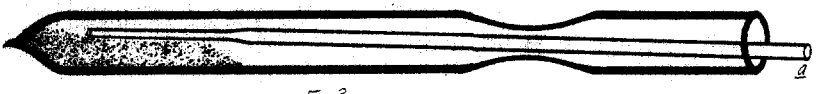

Fig 2.

and oxygen was passed through the tube *a* for fifteen minutes. The chromate was found to give no reaction for carbon. It was kept in the

tube in which it was heated and the open end was closed with a test tube. Silicon carbide was used as a source of carbon as it is not attacked by oxygen gas at high temperatures when burning out carbon from dust. It was quite pure and in the form of an exceedingly fine powder obtained by flotation. Aluminum oxide was made from the hydroxide and heated in oxygen. The mixture used for the tests contained 9.990 g. of aluminum oxide and 0.010 g. of silicon carbide. These were thoroughly mixed in a mortar and then heated an hour in oxygen to burn out all carbon compounds except silicon carbide. The mixture was kept from atmospheric dust in the same way as the lead chromate. The closed tubes were made of hard glass and had a little bulb near the open end as shown in Fig. 3. The bulb *a* contained solid potassium hydroxide to keep out carbon dioxide from the air.

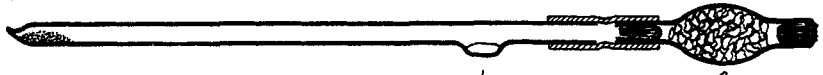

Fig. 3.

For a test a little lead chromate was placed in a closed tube and the tube was weighed, then a little of the Al_2O_3-SiC mixture was added and the tube was weighed again. The difference between the two weights was the amount of the Al_2O_3-SiC mixture taken. The materials were mixed by shaking and then a drop of a saturated solution of barium hydroxide was placed in the bulb *b* by means of a narrow pipet and the potash tube was put on. Finally the mixture was fused in a Bunsen flame.

The following results were obtained with the different weights of the Al_2O_3-SiC mixture:

100	mg. gave an abundant white film.
20	mg. gave a distinct white film.
14	mg. gave a slight but distinct white film.
10	mg. gave a very faint white film.
8	mg. gave a doubtful result.
4	mg. gave absolutely no result.
1.5	mg. gave absolutely no result.

Ten milligrams of the mixture is the smallest amount that gave an unquestionable reaction. This amount corresponds to 0.01 mg. of silicon carbide containing 0.003 mg. of carbon. Undoubtedly smaller amounts of carbon may be detected by using a minute drop of solution of barium hydroxide and observing the result with a microscope.

If there is doubt regarding a faint test as to whether it comes from possible dust in the tube or from the substance tested, it is well to run a blank at the same time for comparison.

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