TABLE VI.

Complete Analysis.

	Present.				Reported.				
No.	Hac. %.	HPr. %.	HBu. %.	Total acid. %.	Hac. %.	HPr. %.	HBu. %.	Total acid. %.	Mean error. %.
1	0.720	0.350	0.195	1.255	0.700	0.380	0.190	1.270	4.7
2	0.720	0.340	0.195	1.255	0.760	0.280	0.210	1.250	7.8
3	0.720	0.340	0.195	1.255	0.720	0.340	0.190	1.250	0.7
4	0.720	0.340	0.195	1.255	0.720	0.330	0.200	1.250	1.5
5	0.631	0.590	0.560	1.782	0.623	0.598	0.566	1.787	1.0
6	0.631	0.590	0.560	1.782	0.679	0.546	0.559	1.784	3.8
								Average,	3.25

NOTE.—Acids used in making up standard samples: (1st) glacial acetic acid, (2nd) propionic acid, prepared by saponifying the ethyl ester, fraction used boiling between 139–142°, (3rd) butyric acid, prepared from the ester, boiling at 161–163°.

SAN DIEGO, CALIF.

NOTE.

Rapid Organic Combustion.—We wish to report here on a method of rapid combustion as it is practiced in our laboratory. There is nothing essentially new in the principle of the method as employed by us. To Dennstedt,[1] to Beck[2] and to Miss Marie Reimer[3] belong the credit of building up the theory and practice of the rapid combustion. However, in our laboratory where the demand made on every furnace is very great and where combustions are made continually, there were many occasions when none of the three methods worked satisfactorily, even when carried out exactly under the original conditions given by their respective authors. In the course of years some of the imperfections of the three methods were corrected, and the advantageous parts of all were combined in one, so that finally we have a combustion tube which served for more than three hundred combustions without change of the catalyst.

Cerium dioxide was employed as the catalyst, as suggested by Beck. It was soon found that when the dioxide was prepared in the manner described by the original authors, samples were obtained that differed considerably in their efficiency. Often the catalyst was exhausted after a very few combustions. On other occasions the dioxide was inactive from the start, and at times an inactive dioxide improved after several combustions, but became exhausted after a few additional combustions. As the same source of cerium nitrate gave samples of dioxide of variable

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[1] Dennstedt, M., "Anleitung zur vereinfachten Elementaranalyse," III. Aufl. Hamburg, **1910**, p. 66.

[2] Beck, J., *Ber.*, 46, 2574 (1913).

[3] Reimer, M., THIS JOURNAL, **37**, 1636–38 (1915).

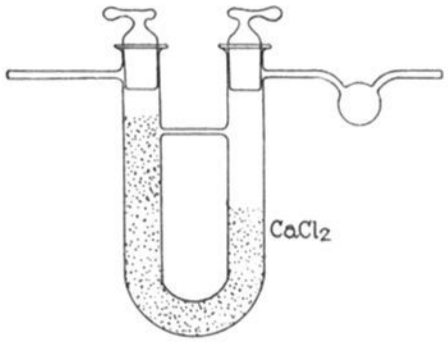
efficiency it was thought that some component of illuminating gas had a deleterious effect on the catalyst. Because of this it was concluded to convert the nitrate into the dioxide in an atmosphere of oxygen. The catalyst prepared in this manner seems to maintain its efficiency indefinitely.

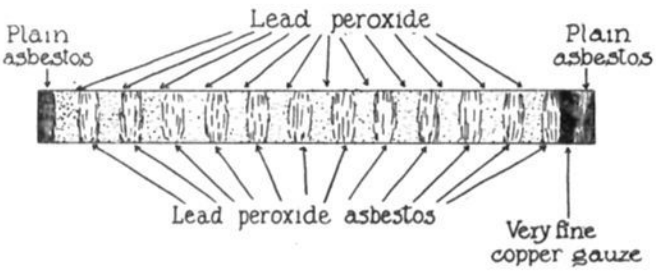
The central part of the tube is filled with copper oxide in wire form. At each end of the copper oxide is placed a charge of cerium dioxide. There is a layer of pure asbestos between the copper oxide and the catalyst, as well as between the catalyst and the neighboring part. An innovation in the tube is a small coil made of very fine platinum gauze. The coil serves as a protection for the boat from copper oxide that might be carried over from the terminal copper coil. Through the presence of this coil it is possible to obtain reliable data, regarding the ash content of a substance.

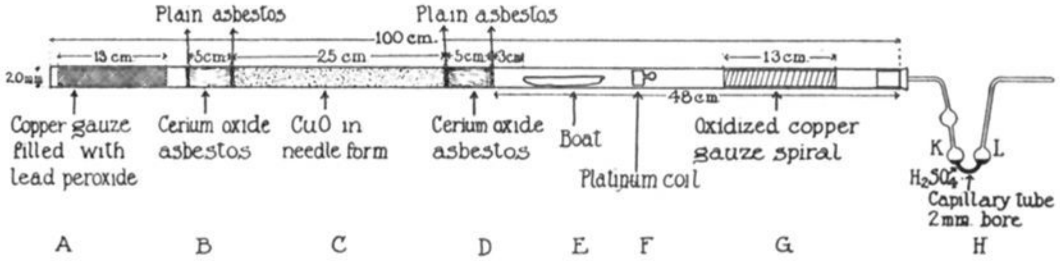
The manner of filling the tube is best seen from the accompanying diagrams.

The Combustion Tubes should be 100 cm. long and 20 mm. in diameter. Where the demand on the furnace is considerable, a tube of transparent quartz was found economical. The combustion tube is connected to any conventional drying train by means of part H. This consists of a capillary tube of 2 mm. bore and of three bulbs, 15 mm. in diameter. The distance between K and L is 25 mm. The section is filled with sulfuric acid.

Absorption Apparatus.—For the absorption of water a U-tube, provided with a bulb for retaining the condensed water as given on the drawing, is recommended. For the absorption of carbon dioxide two usual U-tubes filled with soda lime are employed.







The Lead Peroxide is kept in position in a copper gauze wrapper. Layers of pure peroxide alternate with layers of asbestos impregnated with the reagent. For this purpose, the asbestos is mixed with an equal volume of dry peroxide.

Catalyst.—Purified asbestos fiber is suspended in a saturated solution of cerium nitrate, and the mixture is placed on a boiling water bath to remove the greater part of the water. The still moist asbestos is then transferred to a glass tube and is heated in a stream of oxygen. One end of the tube is drawn to a narrow point which is connected with the oxygen supply. The opposite end is provided with a stopper containing a glass tube, which dips into a flask containing dilute alkali.

Rules for Combustion.—The burners under Section A of the tube are regulated so that the temperature in the center of the lead peroxide is kept constant at 300 to 320°. This temperature should be regulated when all the burners are lighted so as to give the maximum heat required by the combustion. Once so adjusted, the burners remain fixed and should not be disturbed so long as the same charge of the tube remains in service. Before beginning the combustion, the absorption apparatus is attached to the tube which is then burned out in a current of oxygen for thirty minutes. At the end of this time the absorption tubes are weighed and again attached to the tube.

The right end of the tube up to two burners to the left of Section D is then allowed to cool to the temperature of the room. At this moment the boat is inserted into the tube. As soon as the boat is in position, the burners are lighted under the right end of Section G and under Section D. It is recommended to cover the furnace over Section D with asbestos board or with brick, to insure maximum heat of the catalyst. As soon as the requisite temperature of the catalyst is developed, it turns light yellow in color. At this stage the burners are lighted under the right end of the furnace close to the right end of Section E. Following that moment the flames are regulated approximately in the same manner as in the Dennstedt combustion. One has to adapt himself to the nature of the substance.

The combustion is terminated when all the carbon in the boat or on the tube in the region of the boat is burned off. On burning usual substances the combustion lasts about 10–15 minutes from the moment when the catalyst reaches the requisite temperature until the end of the operation.

After the combustion is completed the flames corresponding to Section D and to the right of it are turned off and oxygen is passed for 20 minutes. The absorption tubes are weighed immediately. Generally a combustion consumed 45 minutes, including the two weighings.

P. A. LEVENE AND F. W. BIEBER.

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