NOTE.

The Combustion of Volatil Organic Liquids.—The writer in the course of the last few years has had occasion to make many analyses of volatil organic liquids. The determination of carbon and hydrogen by the familiar method originally devised by Liebig is fraught with serious practical difficulties when the compound to be analyzed has a low boiling point. The usual method in such cases has been to weigh out the liquid in a small bulb tube provided with a capillary, which after filling was sealed to prevent loss during weighing. The bulb was then placed in a boat, the tip of the capillary broken off, the whole then placed in the combustion tube in the furnace. By gradual heating of that part of the tube where the bulb was located, the liquid was slowly volatilized and burned. The carbon dioxide and water resulting from the combustion were collected and weighed in the usual manner.[1]

The disagreeable features of this method are avoided by a new process which has been introduced and is now used in this laboratory. This new method is as follows: A combustion tube is drawn out at one end and a tube bearing a female ground glass joint is sealed on. That part of the tube which is within the heating zone of the furnace is now filled completely with coarse copper oxide held in place at the ends by suitable spirals made of copper gauze. The end bearing the ground glass joint protrudes about four inches from the end of the furnace, while at the other end, the combustion tube protrudes 3 inches or so, and to this end by a rubber stopper the sulfuric acid tube and potash bulb are attached in the usual fashion. The liquid to be analyzed is weighed out in a small U tube, which is fitted at the top of each arm with ground glass stoppers which close the end of the tubes, but which are perforated on one side by a small hole, which can be made coincident with the side outlet tubes simply by turning.

Thus each stopper serves to close the vertical tubes, also acts as a stopcock to each side arm. To one side arm a rubber hose connection is made to a drying apparatus, while the other is fitted with a male ground glass joint, which just fits the ground glass end of the combustion tube.

To weigh out the liquid, the weight of the empty tube having been determined, one stopper is removed, the liquid introduced, best by a small pipet, and the stopper immediately replaced. No vaporized liquid can now escape as the tube is perfectly tight. It is best to previously lubricate the upper third of each stopper with a small amount of soft paraffin, the unlubricated space below wherever pervious is filled with air, forming an effective cushion to prevent absorption of vapor by paraffin, if the former is soluble.

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[1] An excellent account of this process is given in Gattermann, "The Practical Methods of Organic Chemistry."

After weighing, the tube is connected to the combustion tube by the ground glass joint, the external fourth being lubricated by a very small amount of soft paraffin, while to the other side arm of the U tube the drying apparatus is connected. The combustion tube has meanwhile been thoroughly ignited and is now red hot. The two stopcocks of the U tube are now turned so as to permit a stream of pure dry air (from the usual purifying apparatus) to stream over the surface of the liquid to be burned. The vapor, mixed with the excess of air used, passes through the combustion tube, and over the red hot copper oxide, while the resulting water and carbon dioxide are caught in the customary sulfuric acid tube and potash bulb. If the liquid has a low boiling point like ether, it is well to immerse the lower end of the weighing tube in cold water, to prevent the ether from volatilizing too rapidly. With acetone, neither cooling nor warming is necessary. For a liquid boiling at 100 to 125°, it is best to heat the U tube to 50 or 60° by immersing the lower part in a beaker of warm water.

This method is rapid and accurate and since it is not necessary to allow the furnace to cool between each analysis, several combustions can be made in a single day.

Before using this apparatus for the analysis of hydrocarbons, it was tried out with acetone.

The acetone was purified by drying thoroughly with calcium chloride, and subsequently distilling, using a large Le Bel Henniger fractionating column. The sample taken for analysis boiled at 56° under 760 mm.

Subst., 0.3645, 0.1471; CO_2, 0.8260, 0.3361; H_2O, 0.3302, 0.1303.

Calculated for C_3H_6O: C, 62.07; H, 10.34

Found:	I.	II.
C	61.80	62.30
H	10.06	9.85

Analyses were next made of an octane (boiling point, 118.8–119°), and octylene (boiling point, 119.6–120.5°), which had been obtained in a hydrocarbon research.

Subst., 0.3157; CO_2, 0.9868; H_2O, 0.4089.

Calculated for C_8H_{16}:	C, 85.62; H, 14.38.
Found:	C, 85.27; H, 14.37.

Subst., 0.2935; CO_2, 0.9012; H_2O, 0.2935.

Calculated for C_8H_{18}:	C, 84.10; H, 15.90.
Found:	C, 83.74; H, 15.74.

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