TABLE 1

Analysis of Pure Compounds

	Trinitro Toluene			Cyclohexane			Oxalic Acid			Phthalic Acid			Acetone		
	Theoretical	I	II	Theoretical	I	II	Theoretical	I	II	Theoretical	I	II	Theoretical	I	II
Mg. sample	......	27.3	31.5	......	20.5	22.0	......	29.5	27.3	......	26.1	30.4	......	21.6	24.2
Mg. Co_2 absorbed	......	34.4	39.1	......	64.2	69.0	......	27.9	26.6	......	54.5	64.1	......	49.2	54.9
Mg. H_2O absorbed	......	3.2	4.0	......	2.7	2.8	......	6.3	5.8	......	8.9	10.4	......	20.2	22.9
% C	33.7	34.2	33.8	85.7	85.4	85.6	26.7	25.8	26.6	57.8	56.9	57.5	62.1	62.1	61.9
% H	1.4	1.3	1.4	14.3	14.6	14.4	2.2	2.4	2.3	3.6	3.8	3.8	10.3	10.4	10.5

The absorption vessels used are illustrated in Figure 1. They have a removable base connected to the body by means of a ground glass joint. The absorption towers are interconnected by ground glass joints, and ground glass joint plugs and caps are provided to stopper the vessels before and after a determination and during weighing. To charge the absorption towers, they are inverted, the base is removed, and small glass wool plugs are inserted into inlet and outlet tubes. The carbon dioxide absorption vessel is filled first with a small amount of anhydrous $CaCl_2$ which absorbs water generated by the soda lime, followed by a main layer of soda lime. After replacing the base, the tower will stand on a balance pan without wire attachments. The water absorption vessel is charged with the same agent used to dry the oxygen stream.

The usual precautions have to be taken while charging the organic substance to be burned and during combustion. If the substance is liquid and very volatile, as in the case of diethyl ether or carbon tetrachloride, it is advantageously filled into a thin glass bulb which is sealed and inserted into the combustion tube. There the capillary bulb is crushed with a glass rod after the layer of oxidizing agent has been heated red hot. The combustion is carried out in a very slow oxygen stream, controlled by bubble counters at the beginning and end of the train. Depending on the type of compound to be analyzed, an oxygen rate of 0.5 to 2 liters per hour is maintained.

Substances which have a tendency to crack and form difficult-to-burn graphite are mixed in a finely pulverized state with an excess of the oxidizing agent (copper oxide or lead chromate) prior to charging them to the combustion tube. Liquids boiling above 120° C. are dropped on fine copper oxide in a pear-shaped weighing bottle, weighed and poured into the combustion tube. The weighing bottle is then repeatedly washed into the combustion tube with fine copper oxide.

The time consumed in carrying out one determination, including weighings, does not as a rule exceed 90 minutes. It can be reduced by operating several combustion units simultaneously. The chemicals in the combustion tube have to be renewed after about 20 determinations.

The method described was used in carbon and hydrogen determinations of organic substances varying in character from pure compounds to digested and hydrogenated plant products containing considerable inorganic impurities.

Table 1, representing check runs of five samples of pure compounds, indicates that dependable results can be obtained.

Table 2 compares results obtained by semimicro, micro, and macro combustion.

TABLE 2

Analysis of a Tar by Semimicro, Micro, and Macro Combustion

	Semimicro		Micro		Macro	
	I	II	I	II	I	II
Mg. sample	24.3	27.1	6.13	8.01	541.2	468.3
% C	86.2	85.6	86.0	86.7	85.9	86.0
% H	7.5	7.6	7.8	7.6	7.5	7.4
% O (by difference)	6.3	6.8	6.2	5.7	6.4	6.4

Table 3 gives a number of results and checks for samples of different volatility, consistency, and ash content.

Table 3

Semimicro Analysis of Different Materials

	Sample 35 Tar		Sample 111 Plant Mat.		Sample 307 Liquid	
	I	II	I	II	I	II
Mg. sample	35.1	23.5	37.2	31.2	20.5	22.0
% C	76.1	75.9	88.0	87.6	85.4	85.6
% H	9.6	10.0	7.9	8.2	14.6	14.4
% O	13.7	14.1	4.1	4.2	0	0
% Ash	0	0	29.8	29.3	0	0

Summary

A semimicro method for the determination of carbon and hydrogen in organic substances of varying character, consistency, and volatility is discussed. An ordinary analytical balance is used for the weighings. Determinations of high accuracy are made on samples containing 15-20 mg. of carbon in less than 90 minutes.

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CORRECTION

Legends for Figures 2 and 3 in the article, "Phase Investigations of Fats. I. Apparatus and Techniques for Fat-Solvent Systems at Low Temperatures (pp. 201-204) were transposed in the June issue of Oil & Soap. Figure 2 refers to the thermoregulator, and Figure 3 to the equilibration-filtration apparatus.