

small cloth moistened with glycerine, and finally with a dry cloth.

The rubber tubing which is used to cap the absorption tubes is cleaned with a glass rod moistened with glycerine and then wiped dry with a cloth.

### Detailed Procedure.

"Burning out" the Combustion Tube. The combustion tube is heated by means of two Tirrill burners, which have been provided with fish-tails, while a slow stream of oxygen is passed through the tube. The analyst should satisfy himself that the tube has been completely "burned out" by running a blank determination. The details of the procedure for a blank determination are similar to those of an actual determination and therefore are not enumerated separately.

Weighing Absorption Tubes and Sample. During the time required for "burning out" the combustion tube, the absorption tubes and the sample are weighed. These weighings are performed as follows: The capped absorption tubes are wiped with a cloth moistened with water until they are perfectly clean. They are then wiped with a dry cloth and allowed to stand near the balance for a period of about twenty minutes. A rack fashioned out of aluminum wire serves as a convenient method of support for the absorption tubes. After twenty minutes have elapsed, the caps are removed and the tubes are carefully transferred to the balance pan. A small cradle constructed of aluminum wire supports the tube on the balance pan. The tube is then weighed to the nearest 0.05 mgm.

The sample is weighed into a small boat. (The authors have used a boat constructed of platinum foil. In the case of some substances, e. g. ozonides, a porcelain boat was preferred on account of its lower thermal conductivity). A sample of about twenty milligrams is used. Here again, the weighings are made to the nearest 0.05 mgm.

The Combustion Proper. During the latter part of the procedure described under the weighings the burner nearest the bubble counter is turned off. After the end of the tube has cooled sufficiently the absorption train is connected to the combustion tube, the drying train is disconnected from the combustion tube, the oxidized copper spiral is removed by means of a suitable hook and the boat containing the sample is introduced. The oxi-

dized copper spiral is replaced, the drying train is connected to the combustion tube, and the water is started to flow drop-wise from the Mariotte flask into a graduate. The operations are carried out in this order and without delay. The burner which was turned off is now lighted, after removing fish-tail, and placed under the copper spiral. The burner nearest the absorption train has been allowed to burn continuously during these operations. The heating of the sample is carried out in such a manner as to require about 15 minutes for its oxidation. The fish-tail is then placed on the burner nearest the drying train and the heating continued until the boat has been burned clean, at which time this burner is turned off. Experience soon enables the operator to control the rate of combustion. Any moisture which may have collected in the end of the phosphorus pentoxide tube is removed by grasping this end between the warm prongs of a pair of crucible tongs. About 100 cc. of oxygen is then aspirated through the apparatus to sweep out the last amounts of carbon dioxide and water. The flow of water from the Mariotte flask is then discontinued, and the absorption train is disconnected from the combustion tube.

The experienced analyst will have a second pair of weighed absorption tubes and a second sample available at this time, for the apparatus is now ready for another combustion.

The absorption tubes which were used for the first sample are then attached to a second potassium hydroxide guard tube and Mariotte flask. Air which has been passed through a purifying train containing phosphorus pentoxide and potassium hydroxide is then aspirated through the absorption tubes in order to displace the oxygen. About one hundred cc. of air suffices for this purpose. Instead of this procedure the analyst may find it preferable to fill the absorption tubes with oxygen before their first weight is taken. The tubes are then capped, wiped and finally after standing for about twenty minutes are weighed in the manner previously described.

With a little experience, it is possible to carry out seven or eight analyses in an eight hour day. Furthermore, the results obtained by students, who were unfamiliar with both this method and that of macro-combustion, have convinced the authors that the technique of the method here described is acquired more easily than that of the macro-combustion. This is most probably due to the accurate control of the combustion which is afforded by the

use of the oxygen pressure regulator and the Mariotte flask. The adaptation of these two devices for this purpose is due to the excellent work of PREGL.

### Results.

The following typical analyses were carried out using the above procedure. The substances analyzed, which contained only carbon, hydrogen and oxygen, were materials obtained from different sources and no attempt at further purification was made.

This method can be extended to include compounds which contain other elements, by making suitable changes in the tube filling.

**Table I.**

Substance analyzed	Found		Calc.	
	%H	%C	%H	%C
Vanillin . . . . .	5,3	63,2	5,30	63,13
	5,4	63,5		
	5,3	63,2		
Salicylic Acid . . . . .	4,6	60,7	4,38	60,86
	4,5	60,7		
Benzoic Acid . . . . .	5,0	68,8	4,95	68,82
	5,0	68,7		
Anthraquinone . . . . .	4,0	80,4	3,88	80,75
	3,9	80,5		
	3,8	80,5		
Duroquinone . . . . .	7,4	73,1	7,37	73,12
	7,5	73,2		
	7,5	73,2		
	7,5	72,9		
Resorcinol . . . . .	5,6	65,2	5,50	65,42
Mannitol . . . . .	7,6	39,7	7,75	39,54
	7,8	39,7		
Anisic Acid . . . . .	5,3	62,8	5,30	63,13
	5,4	62,8		
Benzalacetophenone . . . . .	5,9	86,7	5,81	86,50
	5,8	86,5		
Hydroduroquinone . . . . .	8,6	72,2	8,50	72,25