

An Adaptation of Pregl's Microcombustion to a Semi-microcombustion Method for the Determination of Nitrogen.

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During the spring of 1925, DOBROVOLNY and one of us (W. M. L.) devised a semi-micro combustion method for the determination of carbon and hydrogen in organic compounds. Since this method proved to be quite serviceable in the hands of others, it was considered advisable to attempt the development of a similar method for nitrogen. Here again, the admirable work of PREGL served as the foundation upon which this semi-micro method was based. A twenty milligram sample was used and the weighings were performed on a high-grade analytical balance sensitive to $\frac{1}{20}$ the mgm. The procedure here described has been found to yield satisfactory results, and the necessary technique is easily acquired.

Description of Apparatus.

The Source of Carbon Dioxide:

Carbon Dioxide was prepared by means of a Kipp Generator in the manner prescribed by PREGL¹).

The Combustion Tube and Filling:

The combustion tube consisted of a 13 mm. (inside diameter) Pyrex tube, 45 cm. in length, sealed to a 5 mm. (inside diameter) tube 8 cm. long. It was attached to the Kipp Generator by means of a capillary tube (A) fitted with a stop cock (not shown in diagram). The combustion tube was filled as follows: A small plug of ignited asbestos was first passed into place in the neck of the tube. An 8—8.5 cm. layer of wire form copper oxide (E) was then introduced. Another plug of asbestos served to hold this copper oxide in place. A roll of copper gauze (D) 4 cm. in length, which had been reduced by means of methyl alcohol was then put into place, followed by a plug of asbestos. A 7 cm. layer of copper

¹) Quantitative Organic Microanalysis, F. PREGL. — Translated by FYLEMAN; J. & A. CHURCHILL, London, p. 79.