

2. The simple physical constants of five dialkyl oxalates are reported.

3. By the action of the calculated amount of ammonia on the dialkyl oxalates, six alkyl oxamates were isolated. Among these, the isopropyl and *n*-butyl esters are new compounds. The methyl, *n*-propyl, and isobutyl derivatives have been mentioned previously, but their melting points are for the first time accurately given.

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NOTE

A Note on a Modification of Pregl's Micro-methoxyl Apparatus.— Considerable experience in this Laboratory with Pregl's¹ micro apparatus for the estimation of methoxyl has shown certain difficulties. The operation requires very close attention on account of the marked bumping of the contents of the decomposition flask and the consequent danger of the sucking back of the washing solution from the washer and that of the silver nitrate solution from the receiver. To avoid these difficulties the following modifications of Pregl's apparatus have been designed and placed in use.

1. This modification (Fig. 1) is a two-piece apparatus in which the two parts are connected by means of a ground-glass joint at G. The upright outlet tube is provided with two bulbs *c, c'* to condense any hydriodic acid carried along by the carbon dioxide stream. The carbon dioxide is led into flask A through the tube B which is sealed on the side of the flask and drawn into a capillary reaching almost to the bottom of the latter. In the procedure the apparatus is charged

through the opening G. The ground joint is then moistened with a drop of hydriodic acid and replaced. Two rubber bands are attached to the

¹ F. Pregl, "Quantitative Organic Microanalysis," second English edition translated from the third German edition by E. Fyleman, P. Blakiston's Son & Co., Inc., Philadelphia, Pa., 1930, p. 181.

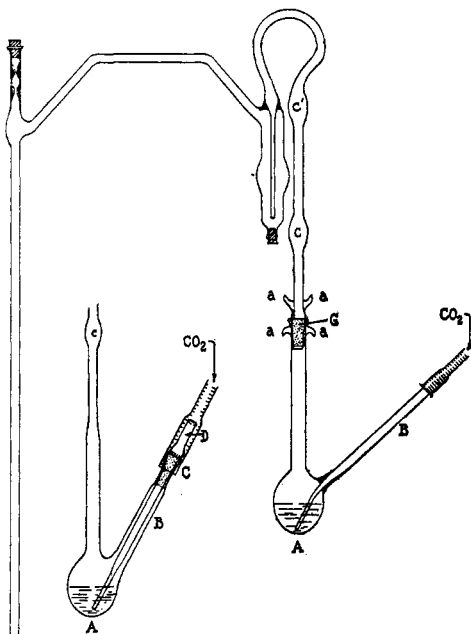


Fig. 2.

Fig. 1.

One-third actual size.

hooks a, in order to hold the two parts tightly and the tube B is connected with the carbon dioxide generator. The rest of the procedure is as described by Pregl.

2. In this one-piece Pregl's apparatus (Fig. 2) the carbon dioxide is led into the decomposition flask A through the tube D drawn into a capillary and entering the side arm B of the flask. This tube fits tightly at the outer end of the side arm by means of a ground-glass joint C. After the apparatus has been charged through the side arm B, the inside of the latter and the ground joint C are moistened with hydriodic acid and the tube D inserted. Then the rubber tube leading to the carbon dioxide generator is slipped over the end of the glass tube D and over the ground-glass joint. There is no retention of alkyl iodide in the side arm on account of the presence of hydriodic acid.

Numerous methoxyl determinations with either modification 1 or 2 have demonstrated that boiling is smooth, bumping does not occur, accurate results are obtained and almost no attention to the operation is necessary after the initial adjustment of the carbon dioxide stream and the microburner.

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COMMUNICATIONS TO THE EDITOR

THE STRUCTURE OF THE FERROCYANIDES

Sir:

A recent contribution by Stephen and Hammerich [*J. prakt. Chem.*, **129**, 285-308 (1931)] on the action of ferrocyanic acid on bicyclic terpenes leads to some interesting conclusions with regard to the structure of the addition compounds.

These investigators have studied the addition of ferrocyanic acid to such compounds as camphene, α -pinene and nopinene, getting compounds corresponding to the formula $(C_{10}H_{17})_2H_2Fe(CN)_6$. Most of the product rearranges on treatment with alkalis but there is a 7% yield of an amine and an alcohol called by the authors isobornylamine and isoborneol. The presence of these two products seems to call for the existence of two forms of the addition product—one completely ionized and the other only partially so.

To obtain isoborneol from the hydrolysis of the α -pinene addition product we may assume complete ionization of the compound $(C_{10}H_{17})_2H_2Fe(CN)_6$, the hydroxyl from water attaching itself to the cation $C_{10}H_{17}$. Potassium formate, ammonia and ferrous hydroxide are the other products.