

## NOTE

Note on the Preparation of Pure Methane from Natural Gas<sup>1</sup>

BY H. H. STORCH AND P. L. GOLDEN

A convenient method of removing higher hydrocarbons from natural gas is to pass the gas through a layer of active charcoal. This method has been successfully used during the past year for the preparation of about 1000 cubic feet of methane containing less than 0.05% ethane and no higher hydrocarbons, from Pittsburgh natural gas which contains 8–15% of ethane and 1–3% of hydrocarbons higher than ethane. Because of the large number of inquiries concerning the preparation of pure methane, it was thought desirable to present briefly our procedure and results.

The charcoal unit consists of a section of 15-cm. pipe, 117 cm. long, fitted with reducing caps and 6.4-mm. valves at each end. On the outer surface of the 15-cm. pipe is a tight-fitting, closely wound coil of 8-mm. copper tubing. The whole unit is mounted vertically and insulated with commercial asbestos and magnesia pipe covering. About 20 pounds of coconut charcoal (8–14 mesh, forty minutes service time by accelerated chloropicrin test, obtained from Barnebey and Cheney of Columbus, Ohio) was used. Before admitting the natural gas the char was heated at 110° (high-pressure steam) for about twelve hours, the top valve being open. At the end of this period, a laboratory vacuum oil pump was connected to the char container and the gases were pumped off for about four hours. Natural gas was then passed through an oil gas meter, three drying units (two of which contained calcium chloride and the third sodium hydroxide pellets), and then through the charcoal (cooled to atmospheric temperature) at the rate of 5 cubic feet per hour. Experience showed that the 20 pounds of char would reduce the content of ethane and higher hydrocarbons to less than 0.05% in 100 cubic feet of Pittsburgh natural gas, even during the summer months when the ethane content is highest. The gas obtained in the initial part of a run is rather low in methane—that is, about 96% methane and 4% nitrogen—whereas that obtained subsequently is about 98% methane and 2% nitrogen. About 0.2% of oxygen is a constant impurity. The content of ethane and higher hydrocarbons was determined by low-temperature fractional distillation. For precise work the methane was condensed by using a bath of liquid nitrogen, the non-condensable gases were pumped off, and the methane repeatedly (about twelve times is necessary) melted and frozen with intermittent pumping off of the trapped gases which are thus released. Finally, the initial third and last third of the methane vapor obtained upon warming the apparatus were discarded.

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After passage of 100 cubic feet of natural gas through the 20 pounds of char, the latter was again heated at 110° and evacuated as described above.

PHYSICAL CHEMISTRY SECTION  
PITTSBURGH EXPERIMENT STATION  
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PITTSBURGH, PENNSYLVANIA

RECEIVED JUNE 20, 1930  
PUBLISHED DECEMBER 13, 1932

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF MOUNT HOLYOKE COLLEGE]

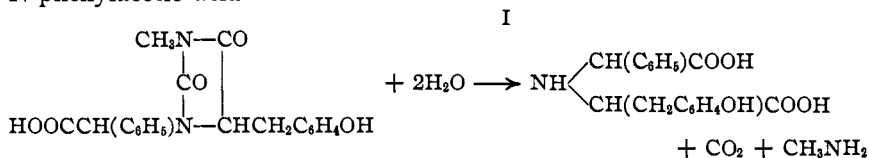
**TYROSINE-N-ACETIC ACID: INTRODUCING A NEW GENERAL METHOD FOR PREPARING SYMMETRICAL AND ASYMMETRICAL IMINO DIBASIC ACIDS**

BY DOROTHY A. HAHN AND ANNE LITZINGER

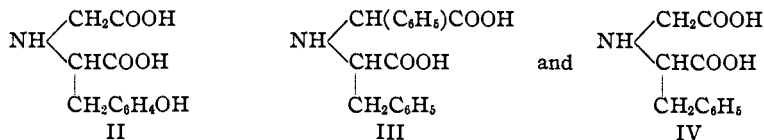
RECEIVED MAY 19, 1932

PUBLISHED DECEMBER 13, 1932

It has been shown that the N-3-methyl-5-tyrosylhydantoin-N-1-phenylacetic acid, on hydrolysis with barium hydroxide, breaks down to tyrosine-N-phenylacetic acid<sup>1</sup>



This reaction has now been found applicable as a new general method for preparing  $\alpha$ -imino dibasic acids of the diacetic acid type,  $\text{HOOCCH}_2\text{NH}-\text{CH}_2\text{COOH}$ , in which the methylene hydrogen atoms are replaced by different hydrocarbon residues. Previous to this time substances belonging to this class have been prepared almost exclusively by hydrolysis of the corresponding dinitriles.<sup>2</sup> In addition to the acid referred to above (I), three others have now been synthesized in this Laboratory, namely, tyrosine-N-acetic acid (II),  $\beta$ -phenylalanine-N-phenylacetic acid (III) and  $\beta$ -phenylalanine-N-acetic acid (IV)



A detailed study of the intermediate products involved in the synthesis of the hydantoin used in the preparation of III and IV is still in progress. The present report is, therefore, limited to a brief description of these acids at the end of this paper preceded by a detailed consideration of *tyrosine-N-acetic acid* and its salts.

A crystalline diethyl ester and a barium salt of the latter acid (II) have

<sup>1</sup> Hahn and Dyer, *THIS JOURNAL*, **52**, 2495 (1930).

<sup>2</sup> Jongkees, *Rec. trav. chim.*, **27**, 287-326 (1908).