A Note on the Preparation of Monomethyldiethylmercaptoglucose.—Monomethyldiethylmercaptoglucose can be obtained by treating the monosodium derivative of diethylmercaptoglucose with methyl iodide. Emil Fischer[1] treated the monosodium diethylmercaptoglucose with methyl iodide in the presence of methyl alcohol, and thought that he regained the original diethylmercaptoglucose. However, some oily disks were formed which he did not investigate. In the present work the sodium diethylmercaptoglucose was prepared according to Fischer's method by treating the diethylmercaptoglucose with sodium ethoxide. The excess of alcohol was evaporated and the methylation was done in absence of alcohol. The solid residue was refluxed with excess methyl iodide until all dissolved. The excess of methyl iodide was evaporated and to the solid residue water was added. From this solution a substance was extracted by means of ether and chloroform which was recrystallized three times from absolute alcohol. It gave a melting point of 155°. The results of micro-analysis for carbon and hydrogen are given in the following table.

TABLE I

ANALYTICAL DATA FOR MONOMETHYLDIETHYLMERCAPTOGLUCOSE

Sample, mg.	Weighed, mg.	Calcd. for, %	Found %
5.020	H_2O, 3.600	H, 8.05	8.03
	CO_2, 8.140	C, 43.97	44.21

The results seem to indicate that the monomethyldiethylmercaptoglucose as prepared by the above method is the same as that isolated by the present author,[2] as a by-product from the preparation of pentamethyldiethylmercaptoglucose.

CONTRIBUTION FROM THE
CHEMICAL LABORATORY OF
WASHINGTON SQUARE COLLEGE
NEW YORK UNIVERSITY
NEW YORK, N. Y.

PHILIPPOS E. PAPADAKIS

RECEIVED MAY 23, 1930
PUBLISHED AUGUST 5, 1930

The Bromination of Hexane.—When the vapor of hexane is passed through bromine water, *p*-dibromobenzene and hexabromobenzene are said to be obtained.[1] F. O. Rice[2] has pointed out that such a transformation of hexane to an aromatic derivative seems doubtful. Since hexane spectroscopically free from benzene was available, it seemed desirable to investigate this question further.

---

[1] E. Fischer, *Ber.*, **27**, 673 (1894).

[2] P. E. Papadakis, THIS JOURNAL, **52**, 2147 (1930).

[1] Blair, Leabury and Wheeler, *J. Soc. Chem. Ind.*, **43**, 298T (1924).

[2] F. O. Rice, "The Mechanism of Homogeneous Organic Reactions from the Physical-Chemical Standpoint," American Chemical Society Monograph, No. 39, The Chemical Catalog Company, Inc., New York.

Pure (99.5%) nitrogen gas was first passed through concentrated sulfuric acid and bubbled through 20 g. of the pure hexane in a distilling flask. The arm of this flask was connected with a bubbler containing water with an excess of bromine. Five hours were required to vaporize the hexane, and examination of the bromine solution revealed no benzene bromides. Repeating with the bromine solution kept below zero and the hexane vaporized in the course of forty hours, no benzene bromides were found. A large amount of the hexane was recovered.

Repeating under the same conditions, but with the bromine solution exposed to a 100-watt Mazda lamp, again no benzene bromides were obtained.

When one gram of benzene was added to the hexane, and the vaporization carried out in the cold and in the dark, some benzene bromides were readily obtained.

The production of the derivatives mentioned in the literature can therefore be traced to hexane slightly contaminated with benzene.

This work was done at the suggestion of Dr. M. S. Kharasch, of the University of Chicago.

CONTRIBUTION FROM THE
DEPARTMENT OF CHEMISTRY
UNIVERSITY OF CHICAGO
CHICAGO, ILLINOIS.
FRANCIS M. PARKER

RECEIVED MAY 23, 1930
PUBLISHED AUGUST 5, 1930

Determination of Halogens in Organic Compounds.—We wish to thank Professor Robertson[1] for calling our attention to his papers, which we overlooked, probably because we were searching for a general method for the determination of chlorine, bromine and iodine and Robertson's method is, as he states, not suitable for iodine compounds.

We have tested his method using samples of about 0.15 g. of bromobenzene and obtained low results, a tendency to which Robertson also calls attention. Duplicates checked less closely than by our method.

The use of sodium peroxide instead of arsenite is obviously optional in either method, but the latter was decided upon after a trial of both reagents.

The time required depends upon whether the gravimetric or volumetric method is used. We needed eighty-five minutes by the gravimetric method with Robertson's apparatus.

The principal advantages of our method[2] are: (1) it is a general method applicable to compounds of chlorine, bromine and iodine.

---

[1] Robertson, THIS JOURNAL, **52**, 3023 (1930).

[2] Thompson and Oakdale, *ibid.*, **52**, 1195 (1930).