TABLE III.—MINIMUM AND MAXIMUM RECORDED TITER TESTS, MELTING POINTS, IODINE NUMBERS, AND REFRACTIVE INDICES OF INSOLUBLE FATTY ACIDS.

	Titer test.		Melting point.		Iodine number.			
Fatty acids.	Min., ° C.	Max., ° C.	Min., ° C.	Max., ° C.	Min.	Max.	Refractive index at 60° C.	
Olive Oil	17.2[1]	27.0[2] 26.4[1]	19.2[5]	31.0[5]	86.1	90.2[5]	1.4528[5] 1.4410[4]	..
Cottonseed Oil	28.5[2]	38.5[2]	34.0[7]	40.0[7]	105	115[7]	1.4460[7]	..
Soya Bean Oil	22.5[2]	25.0[2]	26.0[3]	29.0[3]	122[6]		1.465	27.5°[6]
Cocoanut Oil	21.5[2]	26.0[2]	24.0[3]	27.0[3]	8.3	9.3[8]	1.4295[8]	..
Lard Oil	34.5[2]	39.5[2]	..	..	..	...	....	..
Oleo Stearin	48.0[3]	51.0[3]	..	..	..	...	....	..
Tallow (Beef)	43.0[9]	45.0[9]	43.0[9]	47.0[9]	25.9	41.3[9]	1.4375[9]	..

[1] Lewkowitsch, *loc. cit.*, Vol. I, 517.

[2] Fryer and Weston, "Oils, Fats and Waxes," Vol. I, 70 (1918).

[3] Leach, *loc. cit.*, 528.

[4] Lewkowitsch, *loc. cit.*, 519.

[5] *Ibid.*, Vol. II, 351.

[6] *Ibid.*, 119. [7] *Ibid.*, 197. [8] *Ibid.*, 659. [9] *Ibid.*, 779.

THE PREPARATION OF TRIBROMHYDRIN AND PROPADIENE.*

BY M. W. TAPLEY AND P. M. GIESY.

In the course of the research on anesthetics being carried out by our laboratories it was desired to make sufficient propadiene for physiological investigation. Upon looking up the literature, the most feasible method for our purposes was found to be that of Gustavson and Demjanoff.[1] This method requires 1,2-dibrompropylene which, in turn, is prepared from tribromhydrin (1,2,3-tribromopropane).

Most methods of preparing tribromhydrin require heating at an elevated temperature in a sealed tube, the best method being that of Kronstein.[2] In this method a mixture of propylene dibromide with bromine in the presence of iron wire is heated in a closed tube in a water-bath for two hours. The resulting reaction gives an almost quantitative yield of tribromhydrin. The method has, however, the drawback of requiring the reaction to be carried out in a bomb tube, which was highly undesirable from a laboratory point of view. To avoid this the following modified method was evolved:

A mixture of 200 Gm. of propylene dibromide and 300 Gm. of bromine was placed in a 500-cc. round-bottom reaction flask and a small bundle of iron wire (20 Gm. of card teeth) added. The flask was then gently heated under a reflux condenser with the free flame, the heating being gradually increased until hydrobromic acid was no longer evolved. This required from one to two hours. The resulting mixture was distilled under vacuum and then redistilled at atmospheric pressure. The fraction boiling from 219° to 221° C. was collected. Yield, 78% of the theoretical, based on tribromhydrin.

The conversion of tribromhydrin into 1,2-dibrompropylene was accomplished by the method of Gustavson and Demjanoff,[1] which consists in dropping small

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* Read before Scientific Section, A. PH. A., Des Moines meeting, 1925.

[1] *J. Prakt. Chem.*, [2] 38, 202 (1888).

[2] *Ber.*, 24, 4246 (1891).

portions of tribromhydrin into a flask containing powdered potassium hydroxide. The flask is heated in an oil bath kept at 145° to 150° C., and at this temperature the reaction is complete. The oil is then steam-distilled by cooling the reaction flask to 130° C., adding water, and distilling. Upon redistilling the oil, the fraction boiling between 139° and 141° C. is collected. The yield as given by Gustavson is one-fifth of the original tribromhydrin used. In our work a 28% yield was obtained.

Propadiene is readily made by dropping 1,2-dibrompropylene into a flask containing zinc dust and 80% alcohol. The flask is heated on a water-bath under a reflux condenser. The apparatus used was essentially that described by us in connection with the preparation of methyl acetylene.[1] A 76% yield of pure propadiene was obtained. Twenty Gms. of the dibrompropylene gives about two liters of propadiene.

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THE CHEMISTRY OF NITROSYL CHLORIDE.*

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The remarkable stability of nitrosyl chloride, coupled with the contradictory fact that it easily reacts with numerous substances, makes the subject of its chemical behavior decidedly interesting. There is probably no known compound which provides a greater variety of reactions compared to the simplicity of its structure and to the resistance shown toward decomposition by physical agencies. Furthermore, few organic or inorganic compounds fail to react with nitrosyl chloride in some way or other. We are presenting this short review of the known facts as introductory to an extensive investigation of its general behavior.

Nitrosyl chloride has been prepared in a variety of ways,† all depending upon the reaction of chlorine or of a chloride with an oxide or acid of nitrogen or with some simple nitrogen derivative. Undoubtedly most of these are meritorious and efficient for certain purposes, but the best practical method for general use is the reaction of nitrosyl sulphate upon sodium chloride.[9] The former can easily be produced by saturating sulphuric acid with the vapors from aqua regia or by heating a mixture of nitric and sulphuric acids with sulphur, the latter being a modification of a method by Girard and Pabst.[23]

So manufactured, nitrosyl chloride is a reddish brown gas with a characteristic odor, condensing to a dark red liquid boiling[74] at —5.8° C., and solidifying to blood red crystals[46] melting at[49] —60° to —61° C. The liquid has a specific gravity[29] of 41.165 at —12° C., and a critical temperature[49] of 163–4° C. The absorption spectrum has been investigated by Manganini.[33] Other physical constants are given in the references cited. The vapor pressure agrees with the formula NOCl at all temperatures up to 700° but above this it rapidly becomes dissociated[34,35]

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[1] Tapley and Giesy, JOUR. A. PH. A., February 1926, p. 115.

* Scientific Section, A. PH. A., Des Moines meeting, 1926.

† See references, 2, 3, 4, 5, 7, 9, 12, 15, 23, 26, 27, 28, 29, 33, 41, 47, 50, 53, 55, 59, 60, 61, 63, 64, 69, 73, 75.