The Preparation of Pure Stearic Acid

BY J. P. KASS AND L. S. KEYSER

The preparation of pure stearic acid is complicated by the difficulty of removing final traces of the contaminating palmitic acid, since the separation by physical means is tedious and never unquestionably complete. In a recent study, Guy and Smith[1] considered it necessary to subject "pure" stearic acid to twenty-four recrystallizations from various solvents and to three fractional distillations to obtain a 3.8% yield of final product, m. p. 69.62°, the homogeneity of which was still not beyond question. During the course of our investigations of the chemistry of the fatty acids, we have found it convenient to prepare pure stearic acid totally free from palmitic acid by the catalytic reduction of the readily purifiable octadecenoic acids. Elaidic acid,[2] and especially the α- or β-eleostearic acids,[3] are easily available and may be brought to a high state of purity by a few recrystallizations of the free acids, while pure linoleic acid[4] of theoretical iodine value may be obtained from the crystallizable α-tetrabromostearic acid.[5] Quantitative reduction was effected by shaking the acetic acid solutions of the unsaturated acids for three hours in an atmosphere of hydrogen at room temperature and 45 lb. (3 atm.) pressure in the presence of platinum oxide catalyst. One recrystallization of the product from acetic acid or 85% alcohol yielded a stearic acid melting in a capillary tube at 69.6–70.2° (corr.) and dissolving in concentrated sulfuric acid at 70° without discoloration. A similar procedure was used by Francis, Collins and Piper[6] for the preparation of behenic acid from erucic acid.

(1) Guy and Smith, *J. Chem. Soc.*, 616 (1939).

(2) Rankoff, *Ber.*, **64**, 619 (1931); Smith, *J. Chem. Soc.*, 976 (1939).

(3) Wan and Chen, THIS JOURNAL, **61**, 2283 (1939).

(4) Rollet, *Z. physiol. Chem.*, **62**, 410 (1909).

(5) McCutcheon, *Can. J. Research*, **16**, 158 (1938).

(6) Francis, Collins and Piper, *Proc. Roy. Soc.* (London), **A158**, 707 (1937).

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The Preparation of *m*-Halogenophenols

BY HERBERT H. HODGSON

The author's attention has been drawn to a recent paper by S. Natelson and S. P. Gottfried[1] in which the preparation of *m*-bromophenol is described in language which implies originality, *viz.*: "Since this paper[1] was written, Smith and Haller[2] have published a method for obtaining *m*-bromophenol in good yield .... Their over-all yield is not as good as that obtained from the procedure described herein[1] nor is their procedure as simple."

It is of interest to note that from January to May of this present year (1939) the preparation of *m*-bromophenol has been described no fewer than three times,[1,2,3] and in only one of them[3] has the work of the present writer been mentioned.

The method apparently claimed as new by Natelson and Gottfried[1] was patented by the author[4] in 1923, and C. F. Koelsch[3] has stated recently that this patent of seventeen years ago is substantially true and that yields of 75–80% may be obtained thereby. Similar confirmations of the patent claim, however, have already been made to the writer by numerous chemists during the past seventeen years, who have found yields of the order of 80–90%.

Yields of over 90% of *m*-chlorophenol from *m*-chloroaniline have been obtained by the patented method[4] in large scale (kilo.) preparations. Subsequent work proved that excellent yields of *m*-fluorophenol[5] could also be obtained from *m*-fluoroaniline, while even the non-steam volatile *m*-hydroxybenzaldehyde[6] was prepared in excellent yield by the process.

The patented method[4] is substantially the same in detail as that described at length by Koelsch,[3] and is probably the best process yet devised for the preparation of steam-volatile phenols, since it is of universal application.

(1) Natelson and Gottfried, THIS JOURNAL, **61**, 1001 (1939).

(2) Smith and Haller, *ibid.*, **61**, 143 (1939).

(3) C. F. Koelsch, *ibid.*, **61**, 969 (1939).

(4) H. H. Hodgson and The British Dyestuffs Corporation, English Patent 200,714 (1923); *B. C. A.*, [i] 1005 (1923).

(5) H. H. Hodgson and J. Nixon, *J. Chem. Soc.*, 1879 (1928).

(6) H. H. Hodgson and H. G. Beard, *J. Soc. Chem. Ind.*, **45**, 91T (1926).

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The Freezing Points of Pure High Polymers

BY EDWIN L. LOVELL AND HAROLD HIBBERT

In a recent publication[1] it was shown that the setting points of the polymer-homologous series of polyoxyethylene glycols $HO(CH_2CH_2O)_pH$ followed very exactly the *empirical* equation

$$t_s = P/(a + bP) \qquad (1)$$

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(1) Lovell and Hibbert, THIS JOURNAL, **61**, 1916 (1939).