and corresponded to one mole of hydrogen. The alkaline solution was filtered to remove the nickel catalyst, diluted to 500 cc. with 25% sodium hydroxide solution. To this solution there was added 50 cc. of dimethyl sulfate and the methylation was carried out as usual. The alkaline solution was then extracted with two 100-cc. portions of ether and the combined ether extracts washed and dried. After removing the ether, the residue was distilled; yield 12.7 g. (72%), b. p. 135–139° (1 mm.), m. p. 75–77°. The semicarbazone, prepared in the usual manner, melted at 235.5–236.5° after recrystallization from aqueous alcohol.

Anal. Calcd. for $C_{12}H_{15}O_2N_3$: N, 18.01. Found: N, 18.06.

Reduction with Platinum Oxide Catalyst.—To 16.0 g. (0.1 mole) of 1,6-dihydroxynaphthalene in 150 cc. of acetic acid, there was added 200 mg. of Adams platinum oxide catalyst. The hydrogenation was carried out as described for the nickel catalyst and at the end of approximately three hours one mole of hydrogen had been absorbed. After filtering the platinum catalyst, the acetic acid was removed *in vacuo* and the residue dissolved in 300 cc. of 10% sodium hydroxide. Methylation of the crude hydroxy tetralone and isolation of the methylated product was carried out as described above. In this case, distillation yielded 6.8 g. of a pale yellow liquid, b. p. 130–144° (1 mm.) from which no crystalline material could be isolated. One gram of the distilled product was treated with semicarbazide hydrochloride and 0.16 g. of semicarbazone was isolated. The semicarbazone, after recrystallization from aqueous alcohol, melted at 230–232° and showed no depression on admixture with the product obtained by the nickel catalyst reduction. We are continuing our studies of this reduction procedure in order to establish the nature of the non-ketonic material.

CHEMICAL RESEARCH DIVISION
SCHERING CORPORATION
BLOOMFIELD, NEW JERSEY RECEIVED JUNE 22, 1949

Sulfanilamido-quinoxalines

BY BERTIE C. PLATT AND THOMAS M. SHARP

A paper by Wolf, Pfister, Beutel, Wilson, Robinson and Stevens[1] has appeared almost simultaneously with one by us[2] on sulfonamides derived from substituted quinoxalines. It is possible by inspection of the melting points quoted by Wolf,[1] *et al.*, to identify some of the compounds which were not fully identified by them.

In Table III[1] two 2-amino-6(or 7)-methylquinoxalines are described (a) m. p. 178–180° and (b) m. p. 171–173°. The former must be 2-amino-6-methylquinoxaline since it was prepared from 2-chloro-6-methyl-quinoxaline identified by Platt[3] by an unambiguous synthesis of 2-hydroxy-6-methylquinoxaline from 4-nitro-*m*-tolylglycine and conversion to the corresponding chloro and amino derivatives. The compound (b) m. p. 171–173° appears to be a mixture of 2-amino-6-methyl and 2-amino-7-methylquinoxalines since it was prepared from an impure 2-chloro-7-methylquinoxaline m. p. 56–57°. Pure 2-chloro-7-methylquinoxaline has m. p. 76° (Platt[3]). Platt[3] found 2-amino-6-methylquinoxaline, prepared by a method which could yield only one isomer, to have m. p. 181–182°, and 2-amino-7-methylquinoxaline, prepared in a similar manner to have m. p. 178–180°. A mixture of the two in approximately equal proportions had m. p. 172–174°. It is well known that mixtures of isomers in the quinoxaline series are very difficult to separate.

2-Chloro-5(or 8)-methylquinoxaline, m. p. 92–93°, of Table II[1] is identified as 2-chloro-5-methylquinoxaline which we[3] have synthesized rationally (m. p. 95°) and converted to 2-amino-5-methylquinoxaline (m. p. 201–2°). 2-Amino-5(or 8)-methylquinoxaline (m. p. 202–3°) of Table III[1] is therefore the 5-methyl isomer. (The isomeric 2-amino-8-methylquinoxaline we find to melt at 129°). The corresponding N^4-acetylsulfanilamide, m. p. 228–229°, and the N^1-sulfanilamide, m. p. 205–206° (Tables IV and V[1]) accordingly have the methyl groups in the 5-positions.

THE WELLCOME LABORATORIES OF TROPICAL MEDICINE
LONDON, N. W. 1, ENGLAND RECEIVED APRIL 26, 1949

---

[1] Wolf, Pfister, Beutel, Wilson, Robinson and Stevens, THIS JOURNAL, **71**, 6 (1949).

[2] *J. Chem. Soc.*, 2129 (1948).

[3] Platt, *ibid.*, 1310 (1948). This was recognized by Wolf, *et al.*, but by an unfortunate misprint they say ambiguous instead of unambiguous.

[CONTRIBUTION FROM THE EASTERN REGIONAL RESEARCH LABORATORY,[1] PHILADELPHIA 18, PENNSYLVANIA]

2-(2-Chloroethoxy)-ethyl Acetate and 2-Chloroethyl Vinyl Ether

BY C. E. REHBERG

Dioxane is the principal impurity in the crude 2-chloroethyl vinyl ether prepared by the method of Cretcher.[2] Cretcher considered that the two formed an azeotrope which boiled at 107°. This azeotrope appeared remarkable in that its boiling point was between those of the two components of the azeotropic mixture.

In the work reported here, chloroethyl vinyl ether was prepared in 60% yield by Cretcher's method. When the crude product was distilled through a column having 60 theoretical plates, dioxane was obtained at 101–102°, a mixture of dioxane and ether at 102–108°, and finally, pure ether at 108°. Since both pure dioxane and pure ether were distilled from the mixture, it is evident that no azeotrope was formed.

The ether was also distilled at reduced pressure (120 mm.). Dioxane distilled at 52–53°, and chloroethyl vinyl ether at 59°; a mixture of variable composition was obtained between the pure components.

The following properties were observed with chloroethyl vinyl ether[3]: b. p., 108°, 59° (120 mm.); $n^{20}{}_D$ 1.4378; $d^{20}{}_4$ 1.0475.

(2-Chloroethoxy)-ethyl Acetate.—An effort was made to produce chloroethyl vinyl ether by

---

[1] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture. Article not copyrighted.

[2] Cretcher, Koch and Pittenger, THIS JOURNAL, **47**, 1173 (1925).

[3] Cretcher reported b. p., 109° (740 mm.); $d^{15}{}_{15}$ 1.0525; W. Chalmers reported b. p. 108°, $n^{20}{}_D$ 1.4362; $d^{20}{}_4$ 1.044 (*Can. J. Research*, **7**, 464 (1932)).