

After the solution had been kept at 0° for ten minutes, excess nitrous acid was removed by the addition of ammonium sulfamate. The volume at this point had increased to 25–30 ml. because of occasional additions of ice. *N*-(2-*N*-Methylanilinoethyl)-acetamide<sup>1</sup> (2.07 g., 0.0108 mole) was added and the mixture was stirred for one hour at 0–5°, after which 1.25 g. of sodium acetate trihydrate was added, and stirring was continued two hours. After the mixture had stood an additional two hours at 0–5° a duplicate portion of sodium acetate in a little water was added, the mixture was stirred a few minutes, then left overnight in the refrigerator. After the slurry had stood at room temperature for one hour the precipitate was filtered off, washed with water and recrystallized from ethanol. The yield was 2.76 g. (86.5%) of orange-red parallelograms exhibiting occasional twinned forms with bifurcated ends: m. p. 177.5–178° (cor.). A solution of the substance in 0.1 *N* hydrochloric acid showed an absorption maximum in the visible spectrum at about 520 m $\mu$ .<sup>2</sup>

(1) J. von Braun, *et al.*, *Ber.*, **70B**, 979 (1937).

(2) Determination by Rachel N. Sclar, Cosmetic Division, U. S. Food and Drug Administration.

*Anal.* Calcd. for C<sub>17</sub>H<sub>20</sub>N<sub>4</sub>O: N, 18.91. Found:<sup>3</sup> N, 18.79.

Attempts to remove the acetyl group by refluxing with 6 *N* hydrochloric acid resulted in deep seated changes in the molecule,<sup>4</sup> whereas heating with 5 *N* aqueous alcoholic alkali for seven hours gave only slight evidence of deacetylation.

In view of results repeatedly recorded for couplings involving *N,N*-disubstituted anilines having free *p*-positions, it is highly probable that the phenylazo group of this compound is in the *p*-position.

(3) By Thomas H. Harris, Food Division, U. S. Food and Drug Administration.

(4) Rowe and Dangerfield, *J. Soc. Dyers Colourists*, **52**, 48 (1936), and Hodgson and Foster, *J. Chem. Soc.*, 755 (1941), have reported on the decomposition of certain azo compounds by mineral acids.

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## COMMUNICATIONS TO THE EDITOR

### DIETHYLSILANEDIOL

Sir:

Although organosilanediols have been known for forty years<sup>1,2</sup> no dialkylsilanediol has been reported. The non-isolation of this type compound was generally attributed to rapidity of intermolecular condensation.<sup>3</sup> Hydrolysis studies on diethyldichlorosilane have previously failed to give the diol.<sup>4</sup>

By careful control of conditions during the hydrolysis and the working up of the product we have prepared diethylsilanediol in good yield from diethyldichlorosilane. Best results were obtained with dilute alkali or saturated sodium bicarbonate solution. However, concentrated sodium chloride solution may also be used. A solution of 30 g. of diethyldichlorosilane in 500 cc. of dry ether was added during five minutes with stirring to the theoretical amount of 1.5 *N* alkali at 0°. Immediate separation of the ether layer and ether extraction of the water layer were followed by drying over 25 g. of potassium carbonate for a short time. The ether solution of the product was then concentrated *in vacuo* to 250 cc. Addition of 250 cc. of pentane followed by cooling gave 15 g., 65% yield, of diethylsilanediol, a white

(1) For references to diphenylsilanediol see Burkhard, *This Journal*, **67**, 2173 (1945).

(2) For the preparation of the phenylethyl, benzylethyl, benzylphenyl and dibenzyl compounds see Robison and Kipping, *J. Chem. Soc.*, **101**, 2148 (1912); Robison and Kipping, *ibid.*, **101**, 2156 (1912).

(3) See for example Krause and Grosse, "Die Chemie der Metallorganischen Verbindungen," Borntraeger Geb., Berlin, 1937, p. 284; Roehow, *Chem. Eng. News*, **23**, 614 (1945).

(4) Martin and Kipping, *J. Chem. Soc.*, **95**, 313 (1909); Hyde and DeLong, *This Journal*, **63**, 1194 (1941).

crystalline solid, m. p. 96° before and after recrystallization and boiling with decomposition at about 140°.

*Anal.* Calcd. for C<sub>4</sub>H<sub>12</sub>SiO<sub>2</sub>: Si, 23.34; C, 39.97; H, 10.07. Found: Si, 23.12; C, 39.91; H, 10.10.

Diethylsilanediol can be stored in ordinary corked bottles. Even exposure to a laboratory atmosphere for long periods of time gives no change. At room temperature it is soluble in water and in oxygen-containing organic solvents, and is sparingly soluble in chlorinated hydrocarbons. The compound is easily recrystallized from ether-pentane, acetone-pentane, chloroform and carbon tetrachloride.

Treatment of diethylsilanediol with thionyl chloride and quinoline gave diethyldichlorosilane. Refluxing with benzene and iodine gave the theoretical amount of water. More extensive data on the preparation and properties of dialkylsilanediols will be reported shortly.

We thank R. N. Walter of this Laboratory for the analyses reported in this Communication.

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### *o*-NITROBENZOYL CHLORIDE AND HAZARD INVOLVED IN ITS DISTILLATION

Sir:

N. C. Cook and F. C. Whitmore have called attention (*Ind. Eng. Chem., News Ed.*, **23**, 2394 (1945)) to the treacherous distillation behavior