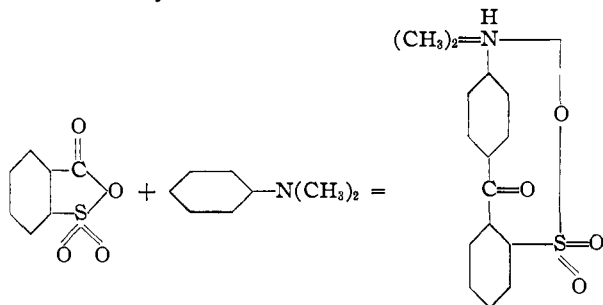


NOTES

4¹-Dimethylaminobenzophenone-2-sulfonic Acid.¹—In attempting to prepare dimethylanilinesulfonephthalein² from dimethylaniline and the anhydride of *o*-sulfobenzoic acid a substance was isolated which proved to be 4¹-dimethylaminobenzophenone-2-sulfonic acid. This compound would be the so-called "intermediate acid" in the preparation of the sulfonephthalein from the anhydride.



The dry acid combines with ammonia, but not with hydrogen chloride. Its barium and ammonium salts were made. Attempts to prepare an acetyl derivative of a γ -lactone form failed. It is isomeric with 4¹-dimethylaminobenzophenone-3-sulfonic acid.³

Dimethylaniline condenses similarly with phthalic acid anhydride, tetrachlorophthalic acid anhydride, 3,4-dichloro- and 3,4-dibromophthalic acid anhydrides.⁴

Experimental Part

One mole (105 g.) of *o*-sulfobenzoic acid anhydride⁵ was dissolved in 3 moles (208 g.) of freshly distilled, warm dimethylaniline. The solution was heated in an oil-bath at 110° for thirty hours. It became green and colorless crystals separated. After cooling, the supernatant liquid was poured off and the crystals were treated with

¹ This work was started at the suggestion of the late Professor W. R. Orndorff, who had hoped to investigate systematically the various amino derivatives in the sulfonephthalein series. As it seems improbable that the present author will continue the problem, this note is published.

² M. D. Sohon, *Am. Chem. J.*, **20**, 127, 257 (1898).

³ R. Willstätter and M. Goldmann, *Ber.*, **39**, 3773 (1906).

⁴ A. Haller and A. Guyot, *Compt. rend.*, **119**, 205 (1894); **126**, 1248, 1544 (1898); **132**, 746 (1901); *Bull. soc. chim.*, [3] **17**, 582 (1893); A. Haller and H. Umbgrove, *ibid.*, [3] **25**, 598, 745 (1901); *Compt. rend.*, **129**, 90 (1899); H. Limpricht and E. König, *Ann.*, **300**, 228 (1898); H. Limpricht and H. Seyler, *ibid.*, **307**, 305 (1899); J. Perard, *Compt. rend.*, **143**, 237 (1906); **146**, 934 (1908); *Ann. chim.*, [9] **7**, 340 (1917); **8**, 22 (1918); G. Cohn, *Pharm. Zentr.*, **55**, 735, 763 (1914); Otto Fischer and Hans Loewe, *J. prakt. Chem.*, [2] **92**, 54 (1915); G. Goldschmiedt, *Monatsh.*, **27**, 849 (1900); Émile Severin, *Compt. rend.*, **130**, 723, 1405 (1900); **142**, 1274 (1906); *Bull. soc. chim.*, [3] **23**, 374 (1900); **25**, 500 (1901).

⁵ E. C. White and S. F. Acree, *THIS JOURNAL*, **41**, 1197 (1919).

boiling 95% ethyl alcohol to remove any adhering oil. A small amount of the compound dissolved and was recovered by concentrating the alcoholic extract. The "intermediate acid" was then purified by recrystallization from water. It is very soluble in hot water and slightly soluble in cold water. It crystallizes from water in colorless plates with two molecules of water of crystallization; yield, 55 g., 43%.

Anal. Subs., 1.3831, 1.4281: loss at 125°, 0.1463, 0.1512. Calcd. for $C_{15}H_{15}O_4NS + 2H_2O$: H_2O , 10.56. Found: H_2O , 10.82, 10.59. Subs., 0.3134, 0.2432: $BaSO_4$, 0.2399, 0.1863. Subs., 0.8400: cc. of 0.1 N H_2SO_4 , 28.42. Calcd. for $C_{15}H_{15}O_4NS$: S, 10.51, N, 4.59. Found: S, 10.51, 10.52; N, 4.74.

Aqueous solutions of the acid change from colorless to a light greenish-yellow at pH 3.4-3.6. The color is too faint for use as an indicator. At 230-235° the substance slowly decomposes forming a green liquid.

Barium Salt.—Five grams of barium carbonate was added to a solution of 5 g. of the acid in 150 cc. of water which was then boiled for two hours. The excess of barium carbonate was filtered off and the filtrate concentrated to crystallization. The following analyses proved the yellow, transparent crystals which separated to be the barium salt of 4¹-dimethylaminobenzophenone-2-sulfonic acid with three molecules of water of crystallization.

Anal. Subs., 0.7378: $BaSO_4$, 0.2150. Subs., 1.0588: loss at 130°, 0.0716. Calcd. for $(C_{15}H_{14}O_4NS)_2Ba + 3H_2O$: Ba, 17.18; H_2O , 6.76. Found: Ba, 17.13; H_2O , 6.76. Subs., 0.9818: $BaSO_4$, 0.3090. Calcd. for $(C_{15}H_{14}O_4NS)_2Ba$: Ba, 18.42. Found: Ba, 18.50.

The barium salt is soluble in acetone, methanol, ethanol; slightly soluble in benzene, chloroform, carbon tetrachloride and insoluble in petroleum ether.

Action of Anhydrous Ammonia.—A sample of the dry acid was exposed to an atmosphere of dry ammonia until it was saturated. On standing in an evacuated desiccator over sulfuric acid for some time the excess ammonia was given off and the stable, yellow ammonium salt remained.

Anal. Subs., 1.2783: NH_3 , 0.0768. Calcd. for $C_{15}H_{15}O_4NS + NH_3$: NH_3 , 5.29. Found: NH_3 , 5.67.

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Note on α,β -Dicyclohexylethylene Glycol.—In a paper on the maximal catalytic reduction of benzoin,¹ a compound provisionally identified as α,β -dicyclohexylethylene glycol was obtained. This compound has now been synthesized and the identification confirmed.

Attempts to condense hexahydromandelonitrile^{2,3} and hexahydromandelamide³ with cyclohexylmagnesium bromide,⁴ with a view to the reduction of the resulting benzoin,⁵ gave only unchanged materials. Monomolecular

¹ Buck and Ide, *THIS JOURNAL*, 53, 3510 (1931).

² Zelinsky, *Ber.*, 41, 2677 (1908).

³ Godchot and Frezouls, *Compt. rend.*, 150, 1248 (1910).

⁴ Wood and Comley, *J. Soc. Chem. Ind.*, 42, T429 (1923).

⁵ Buck and Jenkins, *THIS JOURNAL*, 51, 2163 (1929).