2-(4-Nitrophenyl)-3-(2-phenethyl)-4-thiazolidine-1-dioxide.—Ten grams of 2-(4-nitrophenyl)-3-(2-phenethyl)-4-thiazolidone was dissolved in hot acetic acid (200 ml.) and cooled. The solution was treated with potassium permanganate as described for the 3-isopropyl compound above. The crude product (10 g.) was recrystallized from isopropyl alcohol, (7 g., 64%), m. p. 147–148° cor.

Anal. Calcd. for $C_{17}H_{16}N_2O_5S$: S, 8.89; N, 7.77. Found: S, 8.98; N, 7.89.

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The Preparation of Methyl *p*-Benzyloxyphenyl Acetate

BY PHILIP WEISS[1]

During the course of an investigation on aryl oxyphenylacetic acids, it was necessary to synthesize methyl *p*-benzyloxyphenyl acetate. The preparation of this compound was effected in the following manner.

***p*-Benzyloxyacetophenone** was prepared according to the method of Priestley and Moness[2] by refluxing *p*-hydroxyacetophenone[3] with benzyl chloride in the presence of sodium ethoxide. This same product was obtained, in lower yields, by effecting the condensation in aqueous alkali. By means of a modified Willgerodt reaction[4,5] using morpholine and sulfur, *p*-benzyloxyacetophenone was converted to *p*-benzyloxyphenylthioacetmorpholide. Hydrolysis with aqueous potassium hydroxide yielded *p*-benzyloxyphenylacetic acid. In this conversion to the acid there was no advantage gained in purifying the crude thioacetmorpholide. Esterification was effected by refluxing *p*-benzyloxyphenylacetic acid in a methanol–chloroform solution using *p*-toluenesulfonic acid as a catalyst. A soxhlet apparatus was used to remove water that was produced in the reaction, thus increasing the yield by shifting the equilibrium to the right.

Experimental[6]

***p*-Benzyloxyphenylthioacetmorpholide.**[4,5]—Twenty-four and nine-tenths grams (0.11 mole) of *p*-benzyloxyacetophenone reacted with 13.9 g. (0.16 mole) of morpholine and 5.1 g. (0.16 mole) of sulfur to yield 27.8 g. of the desired thioacetmorpholide. This was recrystallized from dilute methanol, m. p. 118–119°.

Anal. Calcd. for $C_{19}H_{21}NO_2S$: C, 69.72; H, 6.42; N, 4.28; S, 9.78. Found: C, 69.99; H, 6.65; N, 4.31; S, 9.71.

***p*-Benzyloxyphenylacetic Acid.**[4]—To 250 ml. of 10% potassium hydroxide solution was added 22.5 g. (0.07 mole) of *p*-benzyloxyphenylthioacetmorpholide and the mixture was refluxed for fourteen hours. The product was isolated as described by Schwenk and Bloch[4] for the *o*-isomer. Recrystallization of the crude product from benzene–petroleum ether (30–60°) yielded 6.5 g. of *p*-benzyloxyphenylacetic acid, m. p. 114°.

Anal. Calcd. for $C_{15}H_{14}O_3$: C, 74.38; H, 5.78. Found: C, 74.18; H, 6.03.

Methyl *p*-Benzyloxyphenyl Acetate.—Five grams (0.02 mole) of *p*-benzyloxyphenylacetic acid, 0.2 g. of *p*-toluenesulfonic acid, 25 ml. of methanol, and 25 ml. of chloroform were refluxed in a soxhlet apparatus for six hours. Anhydrous magnesium sulfate was placed in the thimble of the soxhlet apparatus in order to remove the water from the reaction mixture as it was being produced by the reaction. Water was added to the mixture until two distinct layers were formed. The chloroform layer was washed with water, and then extracted twice with a saturated sodium bicarbonate solution to remove any unreacted *p*-benzyloxyphenylacetic acid. The chloroform extract was again washed with water until neutral, and then dried over anhydrous sodium sulfate. After filtering the solution, the chloroform was removed by distillation under diminished pressure. The colorless oily residue solidified on cooling in an ice-bath, and was recrystallized from dilute methanol: m. p. 58.5–59°; yield 4.5 g.

Anal. Calcd. for $C_{16}H_{16}O_3$: C, 75.00; H, 6.25. Found: C, 75.28; H, 6.54.

Saponification of methyl *p*-benzyloxyphenyl acetate by means of alcoholic potassium hydroxide yielded *p*-benzyloxyphenylacetic acid, m. p., 113.5–114°; mixed melting point of this acid with a sample of *p*-benzyloxyphenylacetic acid gave no depression.

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[1] Present address: Wallace and Tiernan Products, Inc., Belleville 9, New Jersey.

[2] Priestley and Moness, *J. Org. Chem.*, **5**, 357 (1940).

[3] Rosenmund and Schnurr, *Ann.*, **460**, 56 (1928).

[4] Schwenk and Bloch, THIS JOURNAL, **64**, 3052 (1942).

[5] Newman, *J. Org. Chem.*, **9**, 521 (1944).

[6] All melting points given are uncorrected.

Preparation of 1,1,1-Trichloro-2,2-bis-(4′-aminophenyl)-ethane

BY MOSHE WEIZMANN, SHALOM ISRAELASHVILI AND RAPHAEL PAPO

Kirkwood and Phillips[1] report the synthesis of 1,1,1-trichloro-2,2-bis-(4′-aminophenyl)-ethane (I) by catalytic reduction of 1,1,1-trichloro-2,2-bis-(4′-nitrophenyl)-ethane (II). Conditions have now been found permitting preparation of (I) by reduction of (II) with stannous chloride and hydrochloric acid (yield 71%). The 4,4′-diamino compound (I) responds to the characteristic reactions of aromatic amines, *i. e.*, diazotization and coupling. The structure of (I) is further proved by its conversion to the known compound 1,1,1-trichloro-2,2-bis-(4′-hydroxyphenyl)-ethane of m. p. 202°,[2] and acetylation to the 1,1,1-trichloro-2,2-di-(4′-acetaminophenyl)-ethane, m. p. 268°.[1]

Experimental

1,1,1-Trichloro-2,2-bis-(4′-aminophenyl)-ethane (I).—Twenty-five grams of (II) was added with stirring over a period of two hours at 0° to a solution of 200 g. of anhydrous stannous chloride in 500 g. of glacial acetic acid saturated with gaseous hydrogen chloride. The resulting precipitate was filtered, washed first with concentrated and then with dilute hydrochloric acid. A dilute solution of sodium hydroxide was added to the suspension of the precipitate in water until *p*H 8 was obtained. The precipitate was washed with water, immediately extracted with acetone and dried over anhydrous sodium sulfate. The diamino compound residue (15 g.), after removal of the solvent, recrystallized from ethanol–water as colorless needles, m.p. 150° (dec.).

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[1] Kirkwood and Phillips, THIS JOURNAL, **69**, 934 (1947).

[2] ter Meer, *Ber.*, **7**, 1201 (1874); Elbs, *J. prakt. Chem.*, **47**, 60 (1874); Stephenson and Waters, *J. Chem. Soc.*, 399 (1946).