dine has been studied and optimum conditions for the preparation of aminoguanidine with satisfactory yields by that method are described.

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The Condensation of Certain Aromatic Methyl Ketones

BY DANIEL B. CLAPP AND AVERY A. MORTON

Bernhauer, Müller and Neiser[1] have studied recently the condensation of aromatic methyl ketones by means of potassium pyrosulfate and sulfuric acid. They were successful by this method, in obtaining 1,3,5-tri-*p*-diphenylbenzene from *p*-acetyldiphenyl, but failed in an attempt to prepare 1,3,5-tri-α-naphthylbenzene and 1,3,5-tri-β-naphthylbenzene from α- and β-acetylnaphthalene, respectively. In the course of other work we have prepared the above three compounds by the method of Reddelien,[2] which involves the use of aniline and aniline hydrochloride. This method is more suitable for the preparation of 1,3,5-tri-*p*-diphenylbenzene than that with potassium pyrosulfate, since in the former case purification involves only a few recrystallizations of the crude product.

Experimental

1,3,5-Tri-*p*-diphenylbenzene.—*p*-Acetyldiphenyl (120 g., 0.61 mole), aniline hydrochloride (6.4 g.), and freshly distilled aniline (86 g.) were heated together at 175° under a carbon dioxide atmosphere for three hours. The aniline was removed by distillation, and the residue was poured, while still hot, into a pan, where it quickly set to a brittle, red-brown resin. On grinding the resin in a mortar with ether, and filtering, 43.5 g. of crude product was obtained. Three recrystallizations from toluene gave 25 g. of pure 1,3,5-tri-*p*-diphenylbenzene; yield 23%; white needles; m. p. 230.5–231°; gives no color with cold concentrated sulfuric acid; almost insoluble in ether, alcohol and acetone; slightly soluble in glacial acetic acid; soluble in benzene and toluene.

Anal. Calcd. for $C_{42}H_{30}$: C, 94.34; H, 5.66; mol. wt., 534. Found: C, 94.50, 94.31; H, 5.71, 5.68; mol. wt. (Rast), 536, 538.

1,3,5 - Tri - α - naphthylbenzene.—α - Acetylnaphthalene[3] (26 g., 0.153 mole), aniline (21.4 g.) and aniline hydrochloride (1.6 g.) were heated for one and one-half hours under carbon dioxide at 175°. The aniline was then removed by distillation under atmospheric pressure, and the remaining tar refluxed with 100 cc. of glacial acetic acid for one hour. On cooling, 4 g. of solid separated, which gave 2.8 g. of pure material after four recrystallizations from a mixture of equal parts of glacial acetic acid and acetic anhydride; yield 18%. In preparations in which impure α-acetylnaphthalene (contaminated with the β-isomer) was used, only tars separated from the acetic acid solution on cooling. However, it was found that an attempt to purify the acetic acid solution by boiling with decolorizing carbon caused the adsorption of the 1,3,5-tri-α-naphthylbenzene on the carbon, from which it could be recovered in a relatively pure condition by extraction with a glacial acetic acid–acetic anhydride mixture. The substance crystallizes in white, flat needles with rounded ends; m. p. 190.5–191°; it gives no color with concentrated sulfuric acid.

Anal. Calcd. for $C_{36}H_{24}$: C, 94.69; H, 5.31; mol. wt., 456. Found: C, 94.61; H, 5.40; mol. wt. (Rast), 466, 450.

1,3,5 - Tri - β - naphthylbenzene.—β - Acetylnaphthalene (Eastman Kodak Co.) (19.2 g., 0.113 mole), aniline (15.6 g.) and aniline hydrochloride (1.2 g.) were heated for four and one-half hours under carbon dioxide at 175°. After the reaction mixture had stood in the ice box for one day, 5.5 g. of crystalline material had separated. Three recrystallizations from toluene gave 3.4 g. of pure substance; yield 20%; white needles; m. p. 234–235°; gives no color with cold concentrated sulfuric acid; less soluble than tri-α-naphthylbenzene in common solvents.

Anal. Calcd. for $C_{36}H_{24}$: C, 94.69; H, 5.31; mol. wt., 456. Found: C, 94.58; H, 5.38; mol. wt. (Rast), 462, 468.

Summary

1,3,5-Tri-*p*-diphenylbenzene, 1,3,5-tri-α-naphthylbenzene and 1,3,5-tri-β-naphthylbenzene have been prepared from *p*-acetyldiphenyl, α-acetylnaphthalene and β-acetylnaphthalene, respectively, by the action of aniline and aniline hydrochloride.

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[1] Bernhauer, Müller and Neiser, *J. prakt. Chem.*, **145**, 301 (1936).

[2] Reddelien, *Ann.*, **388**, 194 (1912); *cf.* Vörlander, Fischer and Wille, *Ber.*, **62**, 2836 (1929).

[3] Fröschl, *Monatsh.*, **59**, 275 (1932).