tonitrile using sodamide[8] suggested a similar preparation from diphenylacetonitrile. The proposed alkylation was carried out with methyl iodide, using sodium hydride as the alkylating catalyst. The yield on several preparations varied between 80–90%. The product was identified by its physical constants, by analysis, and by hydrolysis to the corresponding amide and acid.

Experimental.—To 32.5 g. (1.35 moles) of sodium hydride and 175 ml. of anhydrous diethyl ether in a three-necked round-bottom flask was added 262 g. (1.35 moles) of diphenylacetonitrile dissolved in 375 ml. of dry benzene and 100 ml. of diethyl ether. Addition was dropwise over a period of 3 hours. The mixture was refluxed for 30 hours. To the cooled reaction mixture was added 192 g. (1.35 moles) of methyl iodide in 100 ml. of benzene dropwise over a period of 6 hours. Sodium iodide was precipitated immediately. After cooling, the mixture was washed with cold 10% acetic acid to destroy any unreacted sodium hydride. The product was extracted with benzene and ether and dried over calcium chloride. Distillation yielded a highly colored fraction boiling constantly at 135° at 1 mm. pressure. This fraction was dissolved in 500 ml. of ether and treated with 80 g. of adsorption alumina, filtered and redistilled. The product boiling constantly at 142° at 2 mm. pressure weighed 227 g. (1.096 moles, 80.7%); $n^{20}D$ 1.5744, d^{20}_4 1.0671; MR_D 64.13 (calcd. MR_D 64.03).

Anal. Calcd. for $C_{15}H_{13}N$: N, 6.76. Found: N, 6.94.

To 14 g. of potassium hydroxide in sufficient absolute ethanol to effect solution was added 5 g. of α,α-diphenylpropionitrile, and the solution refluxed for 168 hours. Slight evolution of ammonia was detected during the hydrolysis. The reaction mixture was poured into 200 ml. of water and 4 g. of crude amide recovered. Recrystallization from *n*-heptane produced a material melting at 103–104°.[9] Acidification of the filtrate from the crude product produced a small amount of α,α-diphenylpropionic acid melting at 173–174°.[10]

(8) H. Adkins and G. M. Whitman, THIS JOURNAL, **64**, 150 (1942).

(9) H. Wieland and E. Dorrer, *Ber.*, **63B**, 407 (1930), report the m.p. as 103–104°. Bateman (ref. 7) gives m.p. as 149°.

(10) Thorner and Zincke (ref. No. 7) report the m.p. as 173–174°.

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Studies in Silico-organic Compounds. XI. Products Resulting from the Action of Benzylmagnesium Chloride on Triethoxysilane[1]

BY ALBERT L. REILLY AND HOWARD W. POST

Incidental to the study of certain other reactions, benzylmagnesium chloride was allowed to react with triethoxysilane in different molar ratios. Benzyldiethoxysilane, dibenzylethoxysilane and tribenzylsilane were formed.

Triethoxysilane (25 g., 0.15 mole) in 150 cc. of anhydrous ether was treated, with stirring, with 147 cc. of a 1.02 *M* solution of benzylmagnesium chloride in anhydrous ether (0.15 mole). After standing overnight at reflux temperature, the solid residue was separated and the ethereal layer was fractionated. Benzyldiethoxysilane was isolated, 3.9 g., 12% yield, b.p. 108–109° (5 mm.), $n^{25}D$ 1.4666, d^{25}_{25} 0.9782.

Anal. Calcd. for $C_{11}H_{18}SiO_2$: Si, 13.3; silane hydrogen (caustic treatment), 0.477; *MR*, 61.06. Found: Si, 12.9; silane hydrogen, 0.454; *MR*, 59.63.

Using double the relative amount of benzylmagnesium chloride, dibenzylethoxysilane was formed, 8 g., 20% yield, b.p. 141–143° (5 mm.), $n^{25}D$ 1.5370, d^{25}_{25} 0.9945.

Anal. Calcd. for $C_{16}H_{20}SiO$: Si, 11.9: silane hydrogen, 0.390; *MR*, 80.08. Found: Si, 11.6; silane hydrogen, 0.380; *MR*, 80.38.

When 0.042 mole of triethoxysilane was allowed to react with 0.153 mole of benzylmagnesium chloride as above, tribenzylsilane was formed, 3 g., 23% yield, m.p. found 90–91°, literature[2,3] 91°.

Anal. Calcd. for $C_{21}H_{22}Si$: Si, 9.3; silane hydrogen, 0.331. Found: Si, 9.1; silane hydrogen, 0.320.

A greater degree of purity could not be attained for benzyldiethoxysilane in spite of careful fractionation and careful attention to the purity of reagents. Triethoxysilane was prepared in accordance with a method already in use.[4]

(1) This work was made possible by a grant from the Office of Naval Research, under contract.

(2) Evison and Kipping, *J. Chem. Soc.*, 2830 (1931).

(3) Jenkins and Post, *J. Org. Chem.*, **15**, 552 (1950).

(4) Havill, Joffe and Post, *ibid.*, **13**, 280 (1948).

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1,1-Bis-(*p*-methoxyphenyl)-1-propene

BY KURT RORIG

From the aluminum chloride catalyzed reaction of propionyl chloride with anisole in carbon disulfide solvent, Gattermann[1,2] has isolated a by-product melting at 100 to 101° in addition to the expected *p*-methoxypropiophenone. Because this by-product gave *p,p'*-dimethoxybenzophenone upon chromic oxide oxidation, Gattermann considered it to be 1,1-bis-(*p*-methoxyphenyl)-1-propene. Gattermann rationalized its formation by assuming that the first-formed *p*-methoxypropiophenone condensed with a second molecule of anisole to give 1,1-bis-(*p*-methoxyphenyl)-1-propene and a molecule of water.

Much later, Skraup and Freundlich[3] prepared 1,1-bis-(*p*-methoxyphenyl)-1-propene by an unequivocal synthesis from *p,p'*-dimethoxybenzophenone and ethylmagnesium bromide. They found that the propene so prepared also melted at 101. However this propene differed in all other respects from the product they obtained by a repetition of Gattermann's experiment. Therefore they concluded that Gattermann had been in error.

By a repetition of Gattermann's work we were able to show that he was indeed correct in formulating the Friedel–Crafts product $C_{17}H_{18}O_2$, m. p. 100–101°, as 1,1-bis-(*p*-methoxyphenyl)-1-propene.

Experimental

Sixty grams of anhydrous aluminum chloride was added, as rapidly as the exothermic reaction allowed, to a solution of 50 g. of anisole and 50 g. of propionyl chloride in 100 ml. of carbon disulfide. After allowing the mixture to stand one-half hour it was carefully decomposed with water and extracted with ether. Upon evaporation of the dried ether extract 62 g. of yellow, semi-crystalline material remained. After two crystallizations from 95% ethanol this gave 23.5 g. of faintly yellowish plates melting 99.5–100.5°.

Anal. Calcd. for $C_{17}H_{18}O_2$: C, 80.28; H, 7.13; OCH_3, 24.41. Found: C, 80.40; H, 7.22; OCH_3, 24.50.

This product gave a deep-red color when dissolved in concentrated sulfuric acid. The melting point of a mixture of this product, m.p. 99.5–100.5°, with some of the 1,1-bis-(*p*-methoxyphenyl)-1-propene, m.p. 99–100°, prepared by Skraup's Grignard synthesis[3] was 99.5–100.5°.

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(1) L. Gattermann, *Ber.*, **22**, 1129 (1889).

(2) L. Gattermann, R. Ehrhardt and H. Maisch, *ibid.*, **23**, 1199 (1890).

(3) S. Skraup and L. Freundlich, *Ann.*, **431**, 269 (1923).