The results of the hydrolysis of other 1,1,1-trifluoro-2,2-diarylethanes with alcoholic potassium hydroxide solution are summarized in Table I.

The hydrolysis of 1,1,1-trifluoro-2,2-diphenylethane and of 1,1,1-trifluorodi(*p*-chlorophenyl)ethane with sodium ethoxide in anhydrous ethanol, carried out as described above, gave only ethyl diphenylacetate, m.p. 58°, and ethyl di(*p*-chlorophenyl)acetate, m.p. 89°, respectively. With sodium methoxide in anhydrous methanol, methyl diphenylacetate, m.p. 60°, and methyl di(*p*-chlorophenyl)-acetate, b.p. 173°/2 mm., m.p. 35°, were obtained.

Di(p-chlorophenyl)ketene diethyl acetal. 1,1,1-Trifluoro-2,2-di(*p*-chlorophenyl)ethane (1.6 g.) was refluxed with 25 ml. of 1.5 *N* sodium ethoxide solution in anhydrous ethanol for 2 hours. The solvent then was evaporated under reduced pressure and the dry residue was extracted with anhydrous ether and isolated by evaporation of the solvent *in vacuo*. Thus, 1 g. of di(*p*-chlorophenyl)ketene diethyl acetal was obtained, which melted after recrystallization from ligroin at 102.5–103°. The substance was fluorine-free and decolorized a bromine solution.

Anal. Calc'd for $C_{18}H_{18}Cl_2O_2$: C, 64.1; H, 5.3. Found: C, 64.4; H, 5.3.

Acid hydrolysis of the ketene acetal. A solution of 0.4 g. of the ketene acetal in 3 ml. of 50% ethanol, to which 0.5 ml. of conc'd sulphuric acid had been added, was refluxed for 2 hours, and kept for 24 hours at 0°. The crystals that collected were filtered and extracted with petroleum ether. From the extract, 0.25 g. of ethyl di(*p*-chlorophenyl)acetate was isolated, while the petroleum ether-insoluble residue (0.025 g.) consisted of di(*p*-chlorophenyl)acetic acid.

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The Reduction of Nitrobenzene to Azoxybenzene by Sodium Borohydride[1]

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The versatility of sodium borohydride as a reducing agent has been demonstrated in recent publications.[2,3,4] The work of Brown and Subba Rao[3] and, Brown, Mead, and Subba Rao[4] has been valuable in extending the application of this reagent.

Brown, Mead, and Subba Rao[4] report a vigorous reaction with nitrobenzene if an equimolar amount of LiBr is added. They did not isolate any product. It has been found in this laboratory that nitrobenzene can be reduced to azoxybenzene by sodium borohydride if the reaction is conducted at 90–100° in the diglyme solvent (dimethyl ether of diethylene glycol) used in the work mentioned above.[3,4] Experiments conducted with 1-nitropropane under similar conditions have been unsuccessful.

EXPERIMENTAL

The diglyme solvent was purified by storage over NaOH pellets. Then it was refluxed over $NaBH_4$ and distilled. These operations were carried out in a nitrogen atmosphere.

Nitrobenzene (12 g.) was mixed with 20 ml. of the purified diglyme, and 2.0 g. of $NaBH_4$ were added. The reaction was heated under reflux at 90–100° for six hours. The reaction mixture then was diluted with water, acidified, and steam-distilled. The steam-distillate was discarded. The residue was extracted with *n*-hexane. Removal of the hexane yielded 5.5 g. (58%) of an orange product. Recrystallization yielded 5.2 g. (55%) of a yellow solid melting at 35°. Mixture melting point experiments conducted with Eastman Kodak azoxybenzene did not cause any depression of the melting point.

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[2] Kollonitsch, Fuchs, and Gabor, *Nature*, **173**, 125 (1954).

[3] Brown and Subba Rao, *J. Am. Chem. Soc.*, **77**, 3164 (1955).

[4] Brown, Mead, and Subba Rao, *J. Am. Chem. Soc.*, **77**, 6209 (1955).

1,4-Dibenzoyl-2,3-diphenyl-1,3-butadiene[1]

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During the course of another study it was thought advisable to prepare 1,4-dibenzoyl-2,3-diphenyl-1,3-butadiene (III). Although the substance is unequivocally listed by Bielstein's *Handbuch*, a study of the original literature showed that an element of doubt still exists concerning its structure. In 1898 Wislicenus and and Lehmann[3] reported the condensation of benzil (I) with acetophenone to give *cis*-1,2-dibenzoyl-1-phenylethylene[4] (II) and/or a dibenzoyldiphenylbutadiene which, in view of its preparative method, could be either 1,4-dibenzoyl-2,3-diphenyl-1,3-butadiene (III) or 1,4-dibenzoyl-1,3-diphenyl-1,3-butadiene (IV). This is especially true since the authors also obtained it by the condensation of the dibenzoylphenylethylene (II) with acetophenone. The authors made no attempt to distinguish between the two possibilities, although they favored structure III because one of the several products of chemical reduction was a substance

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[1] Taken from a portion of the Ph.D. dissertation of H. W. B., January, 1955. This work was supported in part by a grant from The University of Texas Research Institute.

[2] To whom requests for reprints should be directed.

[3] Wislicenus and Lehmann, *Ann.*, **302**, 195 (1898).

[4] Prepared earlier in a similar manner by Japp and Miller, *J. Chem. Soc.*, **47**, 35 (1885). Configuration assigned by Japp and Wood, *J. Chem. Soc.*, **87**, 707 (1905).