distillation, the residue was dissolved in 150 ml. of benzene and hydrogenated over 3 g. of 5% palladium on charcoal catalyst in a bottle shaken at 50 pounds pressure and room temperature. Hydrogenation was halted after 0.5 hr. and 0.09 mole absorption of hydrogen. After removal of catalyst by filtration, the solution was Claisen-distilled to give 79 g. of crude product, b.p. 65–90° (0.5 mm.). Redistillation through a 10-tray Oldershaw column afforded 34 g. (24% yield) of β-phenylglycidaldehyde, b.p. 70–72° (0.3 mm.); n_D^{20} 1.5448 [lit.[3] values: b.p. 66–68° (0.2 mm.); n_D^{20} 1.5447].

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(5) A sample was added to a mixture of 5 ml. of acetic acid and 50 ml. of 2-propanol; 2 ml. of saturated aqueous sodium iodide was added and the stoppered solution held in the dark for 30 min. at room temperature. The liberated iodine was titrated with 0.1*N* sodium thiosulfate to the disappearance of yellow color.

2,6-Diethyl Homologs of Bromobenzene, Benzonitrile, Benzamide, and Benzoic Acid

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In connection with another project, we had occasion to prepare the heretofore unknown 2,6-diethylbenzoic acid. The conversion of the readily available[1] 2,6-diethylaniline to 2,6-diethylbenzoic acid was carried out by two alternate sequences of reactions. The 2,6-diethyl homologs of bromobenzene, benzonitrile, and benzamide are also unreported in the literature. These intermediates were isolated and their physical properties determined. The hydrolysis of 2,6-diethylbenzonitrile to 2,6-diethylbenzamide rather than 2,6-diethylbenzoic acid, even under the vigorous conditions employed, indicates a considerable steric factor.[2]

EXPERIMENTAL

2,6-Diethylbenzonitrile. 2,6-Diethylaniline was converted into 2,6-diethylbenzonitrile, b.p. 85–86° (1 mm.), n_D^{20} 1.5210; d_4^{27} 0.9614 in a 21% yield *via* the diazonium salt.[3] 2,6-Diethylbenzonitrile was also prepared from 2,6-diethylbromobenzene and cuprous cyanide[4] in an 86% yield.

Anal. Calcd. for $C_{11}H_{13}N$: C, 82.97; H, 8.23. Found: C, 83.02; H, 8.2.

2,6-Diethylbromobenzene. 2,6-Diethylaniline was converted into 2,6-diethylbromobenzene, b.p. 234° (742 mm.); n_D^{20}, 1.5456; d_4^{27}, 1.264 in a 24% yield according to the general direction for the Gatterman reaction.[5]

Anal. Calcd. for $C_{10}H_{13}Br$: C, 56.36; H, 6.15. Found: C, 56.31; H, 6.2.

2,6-Diethylbenzamide. Basic hydrolysis[4] of 2,6-diethylbenzonitrile gave a 91% yield of 2,6-diethylbenzamide, m.p. 136–136.5°, after recrystallization from hexane or water. Hydrolysis of 2,6-diethylbenzonitrile with 90% sulfuric acid gave a 55% yield of 2,6-diethylbenzamide. There was no evidence for the formation of 2,6-diethylbenzoic acid in either the acidic or basic hydrolysis even after an extended reaction time.

Anal. Calcd. for $C_{11}H_{15}NO$: N, 7.90. Found: N, 7.82.

2,6-Diethylbenzoic acid. Eighteen grams (0.1 mole) of 2,6-diethylbenzamide was dissolved in 180 g. of 85% phosphoric acid and heated to 130°. Within 15 min. the clear reaction mixture became opaque and after 1-hr. two layers had formed. The organic layer solidified on cooling and after recrystallization from hexane 2,6-diethylbenzoic acid, m.p. 92–93°, was obtained in a 91% yield.

Anal. Calcd. for $C_{11}H_{14}O_2$: C, 74.13; H, 7.92. Found: C, 74.36; H, 8.1.

2,6-Diethylbenzoic acid was also obtained in a 72% yield from the carbonation of 2,6-diethylphenyllithium, prepared from 2,6-diethylbromobenzene and lithium wire in ethyl ether. The physical and spectroscopic properties of 2,6-diethylbenzoic acid prepared by the two alternate methods were identical.

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The Stereochemistry of the Free Radical Addition of Hydrogen Bromide to 1-Methylcycloheptene[1]

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Since the work of Goering[2] and his group on the stereochemistry of the radical addition of hydrogen bromide to 1-bromocyclohexene and 1-methylcyclohexene, in which stereospecific *trans* addition was observed, the use of cyclic olefins in the study of free radical reactions has become increasingly more important. The effect of a number of factors on radical additions to cyclic olefins has been studied. Of considerable interest has been the influence of ring size on the stereospecificity of the reaction. King Howe[3] reported that the free radical addition of hydrogen bromide to 1-methylcyclopentene afforded at least 94.3% of the *trans* addition product, *cis*-1-methyl-2-bromocyclopentane. Abell and Chiao[4] investigated the radical addition of hydrogen bromide to 1-bromocyclobutane, 1-bromocyclo-

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