132.2. Found: C, 54.50; H, 9.06; neutral equiv., 133.6.

Ethyl *t*-butylglycolate: b. p. 53° at 5 mm.; $n^{25}D$ 1.4210; d^{25} 0.9661. Calcd. for $C_8H_{16}O_3$: C, 59.97; H, 10.07. Found: C, 59.26; H, 10.03.

Ethyl di-*t*-butylglycolate: b. p. 70° at 3.5 mm.; $n^{25}D$ 1.4431; d^{25} 0.9525. Calcd. for $C_{12}H_{24}O_3$: C, 66.63; H, 11.18; $-OC_2H_5$, 20.84. Found: C, 66.51; H, 11.05; $-OC_2H_5$, 18.73.

Di-*t*-butylpivaloylcarbinol (?): m. p. 113–113.5°. Calcd. for $C_{14}H_{28}O_2$: C, 73.63; H, 12.36. Found: C, 73.48; H, 12.56.

Ethyl *t*-butylglycolate failed to react with semicarbazide, as expected, but did yield a derivative, m. p. 166°, with 2,4-dinitrophenylhydrazine, probably due to oxidation at the *alpha* carbon atom.

The analyses for carbon and hydrogen were carried out by Mr. Charles W. Beazley, Micro-Tech Laboratories, Skokie, Ill.

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Some New 1,1,1-Trichloro-2,2-bis-(*p*-alkylphenyl)-ethanes

BY G. F. HENNION AND JOSEPH G. WALSH

The condensations of chloral hydrate with toluene,[1] two of the xylenes,[2] *t*-butylbenzene[3] and benzene itself[4] are known to yield crystalline derivatives structurally analogous to DDT. We have recently extended the reaction to several other alkylbenzenes to determine whether it may be used to prepare solid derivatives useful for identification purposes. Furthermore, an assortment of 1,1,1-trichloro-2,2-bis-(*p*-alkylphenyl)-ethanes (I) was desired to explore the possibility of preparing 4,4′-dialkyldiphenylacetylenes (III) from them by elimination of hydrogen chloride and chlorine.

$$\underset{\text{I}}{(R-C_6H_4)_2CH-CCl_3} \xrightarrow{\text{KOH}} \underset{\text{II}}{(R-C_6H_4)_2C{=}CCl_2} \xrightarrow{\text{2Na}} \underset{\text{III}}{R-C_6H_4-C{\equiv}C-C_6H_4-R}$$

The reactions of the alkylbenzenes with chloral hydrate were carried out in concentrated sulfuric acid (96.9%) in the ordinary manner. Toluene and *t*-butylbenzene gave solid products identical with those previously described.[1,3] Ethylbenzene, isopropylbenzene, *n*-butylbenzene, *s*-butylbenzene and 2-*s*-amylbenzene, however, yielded viscous oils which could not be induced to crystallize. The isolation and purification of these products proved somewhat troublesome because dilution of the crude reaction products with water gave emulsions, probably due to appreciable sulfonation as a side reaction. The emulsions were broken with ether and salt and the ethereal extracts finally distilled three times *in vacuo*. The physical constants, yields after the first distillation, and analyses for these compounds are given in Table I.

TABLE I

1,1,1-TRICHLORO-2,2-*bis*-(*p*-ALKYLPHENYL)-ETHANES

Alkyl group	B. p., °C.	Press., mm.	Yield, %	$n^{25}D$	Chlorine, % Calcd.	Chlorine, % Found
Ethyl[a]	183	0.8	51	1.5780	31.2	31.1
Isopropyl	199	1.4	40	1.5672	28.8	28.5
n-Butyl	203	0.4	42	1.5611	26.8	25.6
s-Butyl	204	0.4	36	1.5597	26.8	25.7
2-*s*-Amyl	210	0.4	27	1.5515	25.0	24.2

[a] Wild, *Helv. Chim. Acta*, 29, 497 (1946), reported the boiling point to be 180° at 0.2 mm. The refractive index was not given.

Each of the seven products (I) was treated with alcoholic potassium hydroxide to prepare the corresponding dichloroethylenes (II). Again the toluene and *t*-butylbenzene derivatives gave solids, as previously reported, while all the other yielded oils. In all cases the dichloroethylenes were insoluble in liquid ammonia and failed to react well with sodium in this medium. An ethereal solution of the di-*p*-tolyl compound, for example, decolorized only a fraction of the theoretical amount of sodium in liquid ammonia and yielded an insoluble, infusible, polymeric substance containing chlorine. All attempts to prepare acetylenes (III) in this manner failed.

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[1] Fischer, *Ber.*, **7**, 1190 (1874).

[2] Elbs, *J. prakt. Chem.*, [2] **39**, 300 (1889).

[3] Cristol, Hayes and Haller, THIS JOURNAL, **68**, 913 (1946).

[4] Baeyer, *Ber.*, **5**, 25 (1872).

Nitration of (*p*-Bromophenyl)-cyclopentane

BY RICHARD D. KLEENE

It was desired to prepare an acetamino derivative of (*p*-bromophenyl)-cyclopentane by the method of Ipatieff and Schmerling[1] for alkyl benzenes. The product obtained was free of halogen as described below.

(3,5-Diacetaminophenyl)-cyclopentane.—One gram of the bromo compound[2] was nitrated with 5 ml. of a mixture of one part of 70% nitric acid and one part of 98% sulfuric acid by volume. The red oil so obtained was dissolved in alcohol and reduced with tin and 36% hydrochloric acid. The amine was then acetylated with acetic anhydride and the resulting solid was recrystallized three times from dilute alcohol. Very fine hair-like needles were formed which melted at 233–234° (uncor.); yield 50%. Analysis showed the complete absence of bromine.

Anal. Calcd. for $C_{15}H_{20}O_2N_2$: C, 69.2; H, 7.70; N, 10.8. Found: C, 68.4; H, 7.57; N, 10.4.

A mixture of this new compound with (2,4-diacetaminophenyl)-cyclopentane[1] (m. p. 228°) melted at 220–225°. The two compounds also appeared dissimilar under the microscope. The removal of the bromine atom indicates that it is probably activated by both nitro groups. This reduces the possibility that the new compound is the 2,5- or the 2,6-diacetamino isomer.

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[1] V. N. Ipatieff and L. Schmerling, THIS JOURNAL, **59**, 1056 (1937); **60**, 1476 (1938).

[2] R. D. Kleene, *ibid.*, **62**, 2883 (1940).