One crystallization from benzene and several from methanol elevated the melting point to 181.5–182°.

Anal. Calcd. for $C_{11}H_6Cl_4O_4$: Cl, 41.23. Found: 41.02, 41.18.

DEPARTMENT OF CHEMISTRY
UNIVERSITY OF TENNESSEE
KNOXVILLE 16, TENNESSEE
C. A. BUEHLER
BURTON P. BLOCK

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Derivatives of *sym*-Trialkylbenzenes

2,4,6-Triethylbenzonitrile.—A mixture of 60 g. of 2,4,6-ethylbromobenzene,[1] 5.0 g. of cuprous cyanide and 60 ml. of pyridine was heated under reflux for eleven hours, allowed to cool somewhat and poured into 400 ml. of dilute ammonium hydroxide solution. The nitrile was extracted with a mixture of benzene and ether. The solution was washed with water and with dilute ammonium hydroxide solution, dried over calcium chloride and distilled. The nitrile boiled at 150–151° (24 mm.); n^{20}_{D} 1.5201; d^{25}_{4} 0.9366; yield 64%.

Anal. Calcd. for $C_{13}H_{17}N$: C, 83.37; H, 9.15. Found: C, 83.23; H, 9.12.

Dillingham and Reid[2] made this compound by another method; they reported the boiling point as 108.5° (2 mm.) and the density as d^{25}_{4} 0.9356, but did not include an analysis.

3,5-Dinitro-2,4,6-triethylbenzonitrile.—Two grams of 2,4,6-triethylbenzonitrile was added slowly to 20 ml. of fuming nitric acid which had been cooled to 0°. The mixture was allowed to stand for thirty minutes in an ice-bath and was then poured into 200 ml. of water. The product was recrystallized from methanol; m. p. 69–70.5°; yield 1.5 g.

Anal. Calcd. for $C_{13}H_{15}O_4N_3$: C, 56.30; H, 5.41. Found: C, 56.21; H, 5.57.

1-Ethyl-2,4,6-triisopropylbenzene.—2,4,6-Triisopropylbenzyl chloride was coupled with methylmagnesium iodide according to the method described for 4-*t*-butyl-2,6-dimethylbenzyl chloride.[3] 1,2-Di-(2,4,6-triisopropylphenyl)-ethane[4] was obtained in 63% yield. The alkylation product, 2,4,6-triisopropylethylbenzene, boiled at 101–103° (5 mm.); n^{20}_{D} 1.4927.

Anal. Calcd. for $C_{17}H_{28}$: C, 87.85; H, 12.15. Found: C, 88.21; H, 12.04.

Ethyl 4-*t*-Butyl-2,6-dimethylbenzyl Ether.—This compound was produced in an attempt to reduce 4-*t*-butyl-2,6-dimethylbenzyl chloride. To a solution of 15 g. of sodium hydroxide pellets in 220 ml. of 95% ethanol was added 25 g. of the chloride. Three grams of aluminum powder was added slowly, with stirring, and the mixture heated under reflux for three hours. The ether, isolated in the usual way, boiled at 126–126.5° (7 mm.); n^{20}_{D} 1.5008; yield 78%.

Anal. Calcd. for $C_{15}H_{24}O$: C, 81.75; H, 10.98. Found: C, 82.02; H, 11.03.

Ethyl 2,4,6-Triisopropylbenzyl Ether.—This compound was produced in an attempt to reduce 2,4,6-triisopropylbenzyl chloride by a procedure similar to that used with 4-*t*-butyl-2,6-dimethylbenzyl chloride. The product, isolated in the usual manner, boiled at 132–134° (6 mm.); n^{20}_{D} 1.4928.

Anal. Calcd. for $C_{18}H_{30}O$: C, 82.38; H, 11.52. Found: C, 82.05; H, 11.50.

An attempt to brominate this ether converted it to bromomesitylene.

2,4,6-Triisopropylbenzyl Acetate.—A mixture of 10 g. of silver acetate, 15 g. of 2,4,6-triisopropylbenzyl chloride and 30 ml. of glacial acetic acid was heated for six hours on a steam-bath. The silver chloride was removed by filtration and the solvent evaporated under diminished pressure. The acetate boiled at 161–162° (13 mm.); n^{15}_{D} 1.5033; d^{20}_{4} 0.9536.

Anal. Calcd. for $C_{18}H_{28}O_2$: C, 78.19; H, 10.21. Found: C, 78.15; H, 10.44.

Hydrolysis of the acetate yielded 2,4,6-triisopropylbenzyl alcohol, m. p. 83–84°.[4]

REYNOLD C. FUSON
THE NOYES CHEMICAL LABORATORY
UNIVERSITY OF ILLINOIS
URBANA, ILLINOIS
J. WAYNE KNEISLEY
NORMAN RABJOHN
M. L. WARD

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[1] Fuson and Corse, THIS JOURNAL, **60**, 2063 (1938).

[2] Dillingham and Reid, *ibid.*, **60**, 2606 (1938).

[3] Fuson, Denton and Kneisley, *ibid.*, **63**, 2652 (1941).

[4] Fuson, Horning, Ward, Rowland and Marsh, *ibid.*, **64**, 30 (1942).

Derivatives of 8-Chloroquinoline[1]

8-Chloroquinoline Picrate.—A 7.0-g. portion of picric acid (containing 10% water) was dissolved in 90 ml. of benzene. The solution was dried over anhydrous sodium sulfate, filtered, and added to a solution of 4.5 g. of 8-chloroquinoline[2] in 25 ml. of benzene. The yellow precipitate was collected, washed with benzene, and dried. The yield was 10.5 g. (97%); m. p. 178–180°. A sample was recrystallized from ethanol (70 ml./g.) for analysis, m. p. 178–180°.

Anal. Calcd. for $C_{15}H_9O_7N_4Cl$ (392.7): C, 45.88; H, 2.31. Found: C, 45.50; H, 2.11.

Thiourea Addition Compound of 8-Chloroquinoline.—This compound was obtained during an attempted preparation of 8-quinolinethiol from 8-chloroquinoline via the isothiuronium salt. A solution of 1.64 g. of 8-chloroquinoline in 15 ml. of absolute ethanol was boiled under reflux with 0.76 g. of thiourea for fifteen minutes; the thiourea gradually dissolved. On cooling, the clear solution deposited 1.0 g. (38%) of colorless needles, m. p. 131–132°. (The same result was obtained when the reaction mixture was boiled five hours.) The addition compound (0.8 g.) was recrystallized from ethanol for analysis; recovery 0.6 g., colorless needles, m. p. 132–133°. The compound gave positive sodium-fusion tests for nitrogen, halogen and sulfur, but was completely inert to hot alcoholic silver nitrate. This showed that it was not the desired 8-quinolineisothiuronium chloride, but an isomeric addition-compound of 8-chloroquinoline and thiourea.

Anal. Calcd. for $C_{10}H_{10}N_3SCl$ (239.7): C, 50.08; H 4.21. Found: C, 49.78; H, 4.34.

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[1] This work was done under a contract recommended by the Committee on Medical Research between the Office of Scientific Research and Development and the University of Rochester.

[2] Claus and Scholler, *J. prakt. Chem.*, (2) **48**, 140 (1893).

DEPARTMENT OF CHEMISTRY
THE UNIVERSITY OF ROCHESTER
ROCHESTER, NEW YORK
G. E. MCCASLAND

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SOME HYDROXYPEROXIDES

The peroxides shown in Table I have been synthesized[1] in an anhydrous ethereal solution. Hydroxy dicarbethoxymethyl hydroperoxide and di-[hydroxy-dicarbethoxymethyl]-peroxide were prepared from ethyl oxomalonate,[2] while α,α'-dihydroxydiisoamyl peroxide and α,α'-dihydroxy-di-*n*-hexyl peroxide were prepared from isovaleric aldehyde and *n*-hexaldehyde, respectively. α,α'-Dihydroxy-di-*n*-hexyl peroxide was also prepared in good

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[1] Milas, Harris and Panagiotakos, THIS JOURNAL, **61**, 2430 (1939).

[2] Dox, "Organic Syntheses," Coll. Vol. 1, John Wiley and Sons, Inc., New York, 1941, p. 266.