Notes A department for short papers of immediate interest.

3-Hexyl- and 3-Octyladipic Acids[1,2]

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In the course of studies on the physical properties of liquids designed as potential lubricating oils, certain glycol ethers were prepared. The present report describes the preparation of two intermediates for glycols which were desired for these studies. Full details of their conversion to glycols and glycol ethers will be published elsewhere. The hexyl group is "normal" and the octyl group is derived from diisobutylene. Thus the octyl acid is 3-(2′,4′,4′-trimethylpentyl)adipic acid.

EXPERIMENTAL[3,4]

4-Hydroxycaprophenone was prepared from 129 g. (1.37 moles) of phenol, 186 g. (1.40 moles) of aluminum chloride, and 184 g. (1.37 moles) of caproyl chloride. The product distilled to give two fractions: (*1*) 2-hydroxycaprophenone, 98.2 g. (37.5%), b.p. 104–106°/1.0 mm. (reported 142–143°/10 mm.[5]); and (*2*) 4-hydroxycaprophenone, 91.5 g. (35%), b.p. 155–158°/1.0 mm. (reported 207–208°/10 mm.[5]).

4-Hexylphenol. Distillation of the product of Clemmensen reduction of 4-caprophenone afforded 91.5 g. (78%) of water-white 4-hexylphenol, b.p. 110–114°/1.0 mm. (reported 146–147°/10 mm.[5]).

4-Hexylcyclohexanol. The 4-hexylphenol was reduced using Raney nickel[6] at 150° and 100 atm. After an induction period of 15–30 min., the reduction started and proceeded rapidly, being complete in 3 hr. The catalyst was filtered off, and the alcohol (from the catalyst) was distilled out of the filtrate. The crude 4-hexylcyclohexanol was not purified but was converted directly to 3-hexyladipic acid, as described below.

3-Hexyladipic acid. A solution of 50% nitric acid (100 g., 0.80 mole) was heated nearly to boiling and 40 mg. of ammonium vanadate was added. The mixture was stirred and 44.0 g. (0.24 mole) of crude 4-hexylcyclohexanol was added slowly, the temperature being maintained at 60–65° by means of an ice bath. After complete addition the mixture was stirred for an additional hour and then was cooled. The acid formed a solid cake which was filtered off, washed with water to remove nitric acid and then was taken up in ether, washed thoroughly and was distilled at 176–179°/0.2 mm. to give 36.0 g. (65%) of 3-hexyladipic acid. Recrystallization from 70–90° petroleum ether afforded white plates, m.p. 71–72°.

Anal. Calcd. for $C_{12}H_{22}O_4$: C, 62.58; H, 9.62. Found: C, 62.84; H, 9.73.

4-Octylcyclohexanol. A 120 g. batch of 4-octylphenol[7] was hydrogenated at 150° and 1000 p.s.i. using a Raney nickel catalyst. There was obtained 100 g. (81%) of 4-octylcyclohexanol, b.p. 100–104°/0.3 mm.

3-Octyladipic acid. The oxidation of 4-octylcyclohexanol (100 g., 0.472 mole) was carried out as with 4-hexylcyclohexanol using 200 g. of 50% nitric acid and 95 mg. of ammonium vanadate. Upon completion of the reaction the mixture was placed in an ice bath and allowed to stand thus overnight. The nitric acid was decanted from the pasty mass which was then washed several times with water and dissolved in ether. The ethereal solution was thrice washed with water and was dried over magnesium sulfate. Evaporation afforded a yellow oil which rapidly solidified. Recrystallization from benzene yielded 3-octyladipic acid as colorless platelets, m.p. 133–135°, 68.5 g. An additional crop of 8 g. was obtained by concentration of the mother liquor to half its volume; thus the over-all yield was 76.5 g. (63%). Further recrystallization from cyclohexane-ethyl acetate raised the m.p. to 136–137°.

Anal. Calcd. for $C_{14}H_{26}O_4$: C, 65.08; H, 10.14. Found: C, 65.26; H, 10.03.

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[2] Abstracted from a portion of WADC Technical Report 53-45, June 1953. Released for publication by Mr. Harold Rosenberg, Senior Materials Laboratory, Directorate of Research.

[3] Melting points and boiling points are uncorrected.

[4] Microanalyses by Microtech Laboratories, Skokie, Ill.

[5] G. Sandelescu and A. Girard, *Bull. soc. chim.* (4), **47**, 1300 (1930).

[6] A. A. Pavlic and H. Adkins, *J. Am. Chem. Soc.*, **68**, 1471 (1946).

(7) Kindly supplied by Rohm & Haas Co., Philadelphia, Pa. The substance is prepared by alkylating phenol with diisobutylene.

Halogen Derivatives of 8-Aminoquinoline

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In the course of work on the preparation of several new "ferroin" type oxidation-reduction indicators, it became of interest to synthesize certain trihalo-8-aminoquinolines. The specific derivatives desired were those in which all the available benzene ring positions (that is, positions 5, 6, and 7) are substituted by chlorine and/or bromine. Such derivatives have not heretofore been reported. However, it is known that the 5 and 7 positions, being strongly activated by the amino group, are easily halogenated directly.[1,2] Therefore, it was judged that the desired trihalo-8-

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[1] A. Claus and E. Setzer, *J. prakt. Chem.*, (2) **53**, 404 (1896).

[2] R. C. Elderfield and E. F. Claflin, *J. Am. Chem. Soc.*, **74**, 2953 (1952).