action as well as several others, were tried. The results are summarized in Table I.

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RECEIVED FEBRUARY 21, 1951

# NEW COMPOUNDS

## Certain *n*-Dodecyl Sulfides

**2-*n*-Dodecylmercapto-2-methyl-4-pentanone.**—Following the procedure of Thompson,[1] anhydrous hydrogen chloride was passed through a solution of 17.2 g. (0.085 mole) of *n*-dodecyl mercaptan and 8.6 g. (0.088 mole) of mesityl oxide, initially at room temperature, for one and a half minutes. The exothermic reaction which resulted raised the temperature to 75°. The reaction mixture was heated at about 60° for three hours and, after standing two days, was distilled under reduced pressure. A fraction of 13.3 g. (52%) of pale yellow liquid was obtained, b.p. 176–178° (4 mm.), $n^{20}_{D}$ 1.4694.

*Anal.* Calcd. for $C_{18}H_{36}OS$: C, 71.93; H, 12.08; S, 10.67. Found: C, 71.4; H, 12.1; S, 10.39.

**Methyl 3-*n*-Dodecylmercaptopropionate.**—This compound was prepared by the method of Hurd and Gershbein.[2] 86.1 g. (1.0 mole) of methyl acrylate was slowly added by means of a dropping funnel to a cold stirred mixture of 123 g. (0.608 mole) of *n*-dodecyl mercaptan and 0.5 g. of sodium methoxide. The exothermic reaction ensuing was kept at room temperature by means of an ice-bath. After the addition of the methyl acrylate, the mixture was stirred at room temperature for 16 hours. The filtered reaction mixture was then distilled under reduced pressure. There was obtained 149 g. (85%) of a colorless liquid, b.p. 162–163.5° (0.5 mm.), $n^{20}_{D}$ 1.4660.

*Anal.* Calcd. for $C_{16}H_{32}O_2S$: C, 66.61; H, 11.18; S, 11.12. Found: C, 66.7; H, 11.3; S, 10.99.

***n*-Dodecyl *p*-Nitrobenzyl Sulfide.**—Ninety-four and seven-tenths grams (0.468 mole) of *n*-dodecyl mercaptan was added to a solution of 26.6 g. (approx. 0.468 mole of $NaOCH_3$) of sodium methoxide reagent in 400 ml. of methanol. To this was then added a solution of 101 g. (0.468 mole) 4-nitrobenzyl bromide in 600 ml. of warm methanol. The reaction mixture was refluxed four hours. Excess solvent was removed by distillation and the residue was poured into 800 ml. of cold water. The orange solid forming was filtered off and recrystallized twice from a mixture of methanol and benzene. A total of 96.7 g. (61%) of light yellow crystals was obtained, m.p. 31–33°.

*Anal.* Calcd. for $C_{19}H_{31}O_2SN$: S, 9.50. Found: S, 9.09.

***n*-Dodecyl *p*-Aminobenzyl Sulfide.**—An ethanol solution of 72.7 g. (0.216 mole) of *n*-dodecyl *p*-nitrobenzyl sulfide was refluxed with an excess of tin and hydrochloric acid for 16 hours. The solvent was removed by distillation. The orange residue solidified on cooling. It was taken into solution with methanol. The solution was made basic with sodium methoxide and was then poured into a liter of cold water. The yellow solid which separated was recrystallized from methanol. A pale yellow solid, m.p. 40°, was obtained.

*Anal.* Calcd. for $C_{19}H_{33}SN$: C, 74.21; H, 10.82; S, 10.43. Found: C, 74.7; H, 10.87; S, 10.05.

**Bis-(2-*n*-dodecylmercaptoethyl) Ether.**—To 57 g. (1 mole of $NaOCH_3$) of sodium methoxide reagent in 300 ml. of methanol was added 202 g. (1 mole) of *n*-dodecyl mercaptan. To the resultant mercaptide solution was added slowly 71.5 g. (0.5 mole) of $\beta,\beta'$-dichlorodiethyl ether in such a manner as to produce gentle refluxing of the methanol solvent from the heat of the reaction. After the addition, the reaction mixture was refluxed 20 minutes and then was poured into 500 ml. of cold water. The white solid that formed was filtered off and recrystallized from a mixture of methanol and isopropyl alcohol. A total of 130 g. (55%) of a white waxy solid was obtained, m.p. 31–32°.

*Anal.* Calcd. for $C_{28}H_{58}OS_2$: C, 70.82; H, 12.31; S, 13.50. Found: C, 70.6; H, 12.4; S, 13.2.

***n*-Dodecyl $\beta$-Hydroxyethyl Sulfide.**—To 28.2 g. (0.495 mole of $NaOCH_3$) of sodium methoxide reagent in 300 ml. of methanol was added 100 g. (0.495 mole) of *n*-dodecyl mercaptan. The resultant mercaptide solution was cooled to 5° with an ice–salt-bath. About 28 g. (0.65 mole) of ethylene oxide, previously cooled to 0°, was then added to the mechanically stirred solution over a period of 45 minutes, the reaction temperature being kept below 10°. Another 3 g. of ethylene oxide was added but there no longer was any exothermic reaction. The mixture was stirred an additional 15 minutes and was then poured into a concentrated hydrochloric acid–ice mixture. The upper organic layer was separated, combined with the ether extract of the aqueous layer, and dried over anhydrous sodium sulfate. The solvent was removed and the product was distilled under reduced pressure. There was obtained 55.8 g. (46%) of a colorless liquid, b.p. 168–175° (3 mm.). Redistillation gave a middle fraction, b.p. 145° (1.2 mm.), which solidified on standing; m.p. 28.5°.

*Anal.* Calcd. for $C_{14}H_{30}OS$: C, 68.22; H, 12.27; S, 13.01. Found: C, 68.1; H, 12.2; S, 12.8.

**Bis-(*n*-dodecylmercapto)-methane.**—Anhydrous hydrogen chloride was passed at a moderate rate into a mixture of 7.5 g. (0.25 mole $CH_2O$) of trioxane and 101 g. (0.5 mole) of *n*-dodecyl mercaptan at room temperature for a period of 90 minutes. The contents of the flask solidified as a white mass. This was allowed to stand overnight after which time it was washed thoroughly with water and dried. The yield of the mercaptal was nearly quantitative. It was readily recrystallized from isopropyl alcohol, giving white plate-like crystals, m.p. 42.5°.

*Anal.* Calcd. for $C_{25}H_{52}S_2$: C, 72.04; H, 12.58; S, 15.39. Found: C, 71.8; H, 12.6; S, 15.2.

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RECEIVED APRIL 25, 1951

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(1) R. B. Thompson, U. S. Patent 2,492,334 (1949).

(2) C. D. Hurd and L. L. Gershbein, THIS JOURNAL, **69**, 2332 (1947).

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## Derivatives of Cyclohexanecarboxylic Acid

Two derivatives of cyclohexanecarboxylic acid have been prepared.

**S-Benzylthiuronium cyclohexanecarboxylate** was prepared in the usual fashion, from acid obtained from the Eastman Kodak Company. The melting points were determined with Anschütz thermometers in a Hershberg melting-point apparatus. A sample recrystallized from ethanol for analysis melted at 155–156°.

*Anal.* Calcd. for $C_{15}H_{22}N_2O_2S$ (mol. wt. 294.41): C, 61.19; H, 7.53; N, 9.52. Found: C, 61.19; H, 7.21; N, 9.67.

***p*-Bromophenacyl cyclohexanecarboxylate** was prepared in the usual fashion. A sample recrystallized from ethanol for analysis melted at 90–91°.

*Anal.* Calcd. for $C_{15}H_{17}BrO_3$ (mol. wt. 325.21): C, 55.42; H, 5.27. Found: C, 55.55; H, 5.37.

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RECEIVED APRIL 19, 1951