

period of stirring had elapsed, the solution was poured into ten volumes of cold water. The white semi-solid mass was dissolved in carbon tetrachloride and dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure and the solid material was recrystallized several times from ethanol. One and one-half grams of 4-(4-chlorophenyl)-phenyl acetate was isolated from this material.

The same product was therefore obtained as with carbon tetrachloride, showing that the solvent, in this instance, did not affect the point of entry of the chlorine.

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NEW COMPOUNDS

Mixed β -Naphthyl Thioethers

In the continuance of our investigation of sulfonium compounds,¹ we required several mixed sulfides not reported previously.

β -Naphthyl *n*-hexyl sulfide was prepared by mixing 5 g. of thio- β -naphthol with sodium ethylate (0.8 g. of sodium in 50 cc. of alcohol) and slowly adding 5.1 g. of *n*-hexyl bromide. After heating for one hour on the water-bath, the sodium bromide was removed by filtration and the alcoholic solution was distilled. The β -naphthyl *n*-hexyl sulfide is a light yellow oil that boils at 160° (20 mm.). The yield was 4.7 g. or 69%.

Anal. Calcd. for $C_{18}H_{20}S$: S, 13.11. Found: S, 13.53.

β -Naphthyl *n*-heptyl sulfide was prepared similarly from 10 g. of thio- β -naphthol and 11.1 g. of *n*-heptyl bromide. A crystalline substance melting at 34° was obtained. The yield was 13.2 g. or 82%.

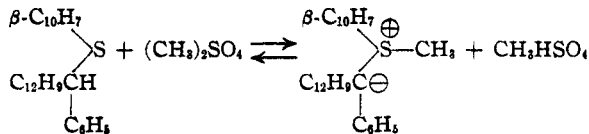
Anal. Calcd. for $C_{17}H_{22}S$: S, 12.40. Found: S, 12.61.

β -Naphthylthiophenylxenylmethane.—Forty-five grams of phenylxenylchloromethane was dissolved in 82 g. of dry thiophene-free benzene. To this was added 30 g. of thio- β -naphthol dissolved in an equal amount of benzene. After refluxing for two hours and standing overnight, a white crystalline compound was obtained. It was recrystallized from glacial acetic acid and white needles were obtained. They melted at 155°. The yield was 60.5 g. or 95%.

Anal. Calcd. for $C_{23}H_{22}S$: S, 7.96. Found: S, 8.01.

Attempted Preparation of β -Naphthylphenylxenylmethanemethylsulfonium Sulfate.—A solution of 10 g. of β -naphthylthiophenylxenylmethane in 30 g. of benzene was mixed with 3 g. of dimethyl sulfate. A light blue color resulted which deepened to a dark purple on refluxing. The color was immediately destroyed by the addition of water and the β -naphthylthiophenylxenylmethane was recovered unchanged.

An application of the theories of Hughes² and Ingold³ would result in the following formulation.



β -Naphthylsulfenylphenylxenylmethane.—This sulfoxide was prepared by treating the sulfide in glacial acetic acid with 30% hydrogen peroxide. After standing for four days the solution was diluted with an equal volume of water and extracted with ether. Recrystallization from acetic acid yielded the sulfoxide melting at 220° in a yield of 90%.

Anal. Calcd. for $C_{23}H_{22}SO$: S, 7.65. Found: S, 7.76.

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Fatty Derivatives of Salicylic Acid and α -Naphthol

Methyl-(2-capryloxy)-benzoate.—This compound was prepared by the reaction of methyl salicylate with capryl chloride at 200–225° until no more hydrogen chloride was evolved (about five hours); b. p. 217–219° (12 mm.), n_D^{25} 1.4888, yield, 71%.

Anal. Calcd. for $C_{18}H_{24}O_4$: C, 70.56; H, 8.55. Found: C, 70.66; H, 8.29.

Methyl (4-Capryl)-salicylate.—Prepared by carrying out a Fries rearrangement on the above ester according to the procedure of Cox¹ except that light petroleum ether was used as a solvent instead of carbon disulfide. The material distilled at 180–190° (1.5 mm.) and after two recrystallizations from light petroleum ether melted at 66.5–67.5°.

Anal. Calcd. for $C_{18}H_{26}O_4$: C, 70.56; H, 8.55. Found: C, 70.42; H, 8.60.

4-Caprylsalicylic Acid.—Hydrolysis of the preceding compound by refluxing with 15% sodium hydroxide solution yielded 4-caprylsalicylic acid which was crystallized to constant melting point from benzene, m. p. 120.5–121.5°.

Anal. Calcd. for $C_{17}H_{24}O_4$: C, 69.84; H, 8.27. Found: C, 69.46; H, 8.24.

α -Naphthylcaprylate.—This compound was prepared according to the procedure used by Stoughton for the lower esters² except that a longer reaction time was necessary (three hours on the steam-bath); yield about 60%. The ester boiled at 156–157° (1 mm.), n_D^{25} 1.5442.

Anal. Calcd. for $C_{18}H_{26}O_2$: C, 79.96; H, 8.20. Found: C, 79.98; H, 8.12.

2-Caprylyl- α -naphthol.—Stoughton's procedure (*loc. cit.*) for the rearrangement of the lower esters was also used for this compound. It was first crystallized from alcohol and then twice from slightly diluted glacial acetic acid, m. p. 68–68.5°.

Anal. Calcd. for $C_{18}H_{26}O_2$: C, 79.96; H, 8.20. Found: C, 79.62; H, 8.17.

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(1) Cox, *This Journal*, **52**, 356 (1930).

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