

bath for 3.5 hr, poured into ice, and extracted with Et₂O. The extracts were washed and distd to give 20.5 g (63.5%) of an oil; bp 130–133° (1 mm); *n*_D²⁵ 1.5548; ir, strong band at 5.85 μ.

2,3-Diethyl-6-methoxyindan-1-one (6).—To 70 g of NaOMe was added with stirring 80 g (0.42 mole) of **5**. With cooling 400 g of EtI was added rapidly and the mixture was stirred for 30 min and then heated on the steam bath for 3 hr. Excess EtI was removed by distn, H₂O added, and the mixture extracted (Et₂O). The solvent was evapd after drying (Na₂SO₄) and the residue was distd; yield 74.5 g (81%); bp 155–160° (1 mm); *n*_D²⁵ 1.5393. *Anal.* (C₁₄H₁₈O₂) C, H.

Pyridyllithium Reactions. 2-(*p*-Methoxyphenyl)-1-(2-pyridyl)-cyclohexanol.—To an Et₂O soln (400 ml) of BuLi prepared under N₂ at –10° from 4.1 g (0.6 mole) of Li and 41.1 g (0.3 mole) of BuBr was added at –40°, 47.4 g (0.3 mole) of 2-bromopyridine in 200 ml of Et₂O. After 1 hr, a soln of 30.6 g (0.15 mole) of 2-(*p*-methoxyphenyl)cyclohexanol²⁰ in 500 ml of Et₂O was added dropwise with stirring and the mixture was allowed to warm to room temp. Stirring was continued for 6 hr. H₂O was cautiously added, the organic layer was sepd and combined with an additional Et₂O extract. The combined Et₂O soln were extracted with 10% HCl and, after preliminary washing (Et₂O), the acid soln was basified (NH₄OH) and extracted (CHCl₃). The CHCl₃ soln was washed (H₂O) and coned on the steam bath to an oil which was triturated with pet ether (bp 30–60°) and recrystd from hexane; yield 24.7 g (58%); mp 74–75°. The ir spectrum showed a typical OH band at 3 μ. *Anal.* (C₁₅H₁₉NO₂) C, H, N.

1-(2-Pyridyl)-2,3-diethyl-6-methoxy-indan-1-ol was prepared by a similar procedure; yield 74%; bp 183–189° (1 mm); *n*_D²⁵ 1.5722; strong OH in ir at 3.0 μ. *Anal.* (C₁₉H₂₃NO₂) C, H, N.

1-(3-Pyridyl)-2,3-diethyl-6-methoxy-1-indene (8b).—This

compound was obtained from 3-bromopyridine by the above procedure in 61% yield; bp 180–185° (1 mm); *n*_D²⁵ 1.5923; log *ε*_{235 mμ} 4.05. *Anal.* (C₁₉H₂₁NO) C, H, N.

Dehydration Procedure (Mixture 4).—A mixture of 10 g (0.035 mole) of 2-(*p*-methoxyphenyl)-1-(2-pyridyl)cyclohexanol and 40 g of powdered potassium pyrosulfate was placed in a bath at 240° and the temp raised to 240–260° with manual stirring until the fusion was completed and held at this temp for 1 min. The mixture was allowed to cool somewhat and poured into ice, made basic (NH₄OH), and extracted (CHCl₃), washed, and distd; bp 172–175° (2.5 mm); yield 5.8 g (63%); *n*_D²⁵ 1.6063. *Anal.* (C₁₈H₁₉NO) C, H, N.

1-(2-Pyridyl)-2,3-diethyl-6-methoxy-1-indene (8a).—A mixture of the 2-pyridyl carbinol (10 g) and 7 ml of 85% H₃PO₄ was heated under reflux for 6 hr and poured into ice. The solution was made basic (NaOH) and extracted (CHCl₃). The solvent was removed and the residue was distd; yield 7 g (76%); bp 173–178° (1 mm); *n*_D²⁵ 1.5838; log *ε*_{235 mμ} 4.08. *Anal.* (C₁₉H₂₁NO) C, H, N.

***p*-Methoxyphenyl-2-(2-pyridyl)cyclohexane (2).**—A soln of 5.0 g (0.019 mole) of mixture **4** in 250 ml of EtOH was hydrogenated in a Parr hydrogenator in presence of 0.5 g of PtO₂. The reduction required 20–22 hr. The catalyst was filtered and the residue after removal of the solvent was distd; yield 4.3 g (85%); bp 190–192° (3 mm); *n*_D²⁵ 1.5766. *Anal.* (C₁₇H₂₃NO) C, H, N.

1-(2-Pyridyl)-2,3-diethyl-6-methoxyindane (3).—The indene **8a** (5.5 g, 0.02 mole) in 150 ml of EtOH was reduced for 20 hr in a Parr hydrogenator using Raney Ni catalyst. The catalyst was removed and the product was distd; yield 3.8 g (83%); bp 185–190° (1 mm); *n*_D²⁵ 1.5696. *Anal.* (C₁₈H₂₃NO) HN, calcd: C, 81.10; found: C, 80.68.

New Compounds

1-Dodecylpyridinium Dodecyl Sulfate

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When a mixture of 1-decanol and *N*-bromoacetamide in pyridine is treated with SO₂ under the conditions described for the dehydration of certain steroid alcohols¹ an excellent yield of 1-dodecylpyridinium dodecyl sulfate is obtained. The same compound is obtained when didodecyl sulfate is reacted with pyridine. Evidently this fact had been observed some years ago by Sementsov, *et al.*,² but their "S-containing salt of pyridine" had not been characterized.

Experimental Section³

A solution of 18.6 g of 1-dodecanol and 27.6 g of *N*-bromoacetamide (NBA) in 160 ml of pyridine was treated with SO₂ at about 25° until all of the NBA had been destroyed. Upon pouring the solution into an ice-water slurry, 20.23 g of 1-dodecylpyridinium dodecyl sulfate, mp 88–90°, precipitated. Re-

crystallization from EtOAc gave an analytical sample, mp 90–90.5°. Ir and nmr spectra supported the structure. *Anal.* (C₂₂H₅₀NO₄S) C, H, N, S.

The sample prepared by dissolving didodecyl sulfate in pyridine, followed by addition to H₂O, had mp 91–92° and spectral properties identical with those of the material prepared by the other route.

SH Analog of the Estrogen Hexestrol

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The synthetic estrogen *meso*-hexestrol (I) is frequently used in the clinic. A great number of analogous compounds have been prepared.¹ The thiophenol isostere II should at least be useful in making decisions about bonding forces in estrogen-receptor complexes² and could be expected to show some interesting biological properties. The synthesis of II by a method similar to one previously described³ is reported.

Biological Activity.—The thiophenol analog II was

(1) See L. F. Fieser and M. Fieser, "Reagents for Organic Synthesis," Wiley, New York, N. Y., 1967, p 75.

(2) A. Sementsov, R. J. Kiesel, M. E. McGreal, and W. P. Hart, *J. Org. Chem.*, **23**, 2020 (1958).

(3) Melting points are uncorrected. Where analyses are indicated only by the symbols for the elements, analytical results obtained for those elements were within ±0.3% of the theoretical values.

(1) U. V. Solrussen, *Chem. Rec.*, **37**, 481 (1945); J. Grundy, *ibid.*, **57**, 281 (1957).

(2) H. G. Maotner, *Physiol. Rev.*, **19**, 107 (1967).

(3) S. F. Torf and N. V. Khromov-Borisov, *Zh. Obshch. Khim.*, **31**, 2102 (1961). They report II to have mp 155–157°.