the separation of radium from barium on a commercial basis could be much more easily effected by this column method than by the current procedure based on fractional crystallization. The method is simple to operate and thus may be easily adapted to remote control. The use of this separation method for the quantitative analysis of radium has not been investigated, but it is probably applicable.

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NEW COMPOUNDS

1-(4-Chloromercuriphenylazo)-naphthol-2

This substance, synthesized in an effort to obtain a colored compound capable of attachment to biological tissue through mercaptan groups only, has been applied successfully in biological investigations.[1,2]

Synthesis.—35.4 g. of *p*-aminophenylmercuric acetate (m. p. 166–167°), prepared by direct mercuration of aniline according to the method of Dimroth,[3] was diazotized at −5° in 500 ml. of 50% acetic acid with 7.0 g. of sodium nitrite, according to the method of Jacobs and Heidelberger.[4] The filtered diazonium salt was coupled to 2-naphthol (15 g. of 2-naphthol, 180 g. of sodium hydroxide, in 2 l. of iced water). After standing a few hours, the precipitate was collected by filtration, washed, dissolved in 200 ml. of glacial acetic acid, filtered, and precipitated by dilution to 2 l. This precipitate was collected, washed, and dissolved by refluxing with 3 l. of 60% ethanol in a water-bath. The hot solution was filtered, the clear filtrate was brought to a boil under reflux, and to it was added 5.8 g. sodium chloride in 150 ml. of 60% ethanol. A cottony red precipitate of 1-(4-chloromercuriphenylazo)-naphthol-2 formed immediately. Refluxing was continued for thirty minutes, the precipitate, 3.6 g. (6.2%), collected by hot filtration and washed several times with boiling 50% ethanol. The precipitate was recrystallized three times from *n*-butyl alcohol (0.9 g. per l. of boiling alcohol) with 95% yield of fine red needle-like crystals which were virtually insoluble in water, but slightly soluble in cold alcohols, chloroform, toluene and decahydronaphthalene, melting with blackening at 291.5–293° (cor.).

Anal. Calcd. for $C_{16}H_{11}ClHgN_2O$: C, 39.76; H, 2.29; Cl, 7.34; Hg, 41.5; N, 5.80. Found: C, 39.36; H, 2.24; Cl, 7.12; Hg, 42.0; N, 6.01.[5]

Degradation.—The product was split by sodium hydrosulfite, yielding 1-amino-2-hydroxynaphthalene.

(1) Bennett, *Anat. Rec.*, **100**, (suppl.) 7, 100 (1948).
(2) Bennett, in press.
(3) Dimroth, *Ber.*, **35**, 2032 (1902).
(4) Jacobs and Heidelberger, *J. Biol. Chem.*, **20**, 513 (1915).
(5) The analyses were preformed by Mr. Nagy of the Microchemical Lab., Massachusetts Institute of Technology.

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β-Glyceryl Phenyl Ether and 1,3-Dichloro-2-phenoxypropane

These compounds were prepared as intermediates in an effort, which proved unsuccessful, to synthesize phenyl cyclopropyl ether by the procedure described by Krantz and Drake[1] for the synthesis of methyl cyclopropyl ether.

β-Glyceryl Phenyl Ether.—The reduction of phenoxymalonic ester[2] by lithium aluminum hydride,[3] with alkaline hydrolysis of the intermediate aluminate, furnished the crude product, m. p. 59–66° in 95% yield. On recrystallization from benzene it was obtained as colorless needles, m. p. 68°. *Anal.*[4] Calcd. for $C_9H_{12}O_3$: C, 64.27; H, 7.19. Found: C, 64.07; H, 7.19.

1,3-Dichloro-2-phenoxypropane. A. From β-Glyceryl Phenyl Ether and Thionyl Chloride.—A solution of 40 g. of β-glyceryl phenyl ether in 40 g. of pyridine, dissolved with the aid of heat, was added dropwise, keeping the temperature below 20°, to 200 g. of thionyl chloride. The flask, with reflux condenser attached, was heated very gently to start the evolution of sulfur dioxide and eventually more strongly until the temperature of the vapor in the flask reached 70°. Excess thionyl chloride was removed under reduced pressure. Water was then cautiously added to the residue, the mixture extracted with ether and the extract washed once with dilute alkali. The final purification was by fractional distillation under reduced pressure, using a 30″ wire-spiral column which resulted in a 75% yield of 1,3-dichloro-2-phenoxypropane, b. p. 103.5–104° (1 mm.), $n^{25}D$ 1.5358. *Anal.* Calcd. for $C_9H_{10}OCl_2$: C, 52.76; H, 4.92; Cl, 34.58. Found: C, 52.67; H, 5.15; Cl, 34.57.

B. From Benzene Diazonium Chloride and 1,3-Dichloropropan-2-ol.—Dry benzene diazonium chloride[5] prepared from 22 g. of aniline hydrochloride was added in small portions over a period of one-half hour, keeping the temperature below 25°, to 200 g. of redistilled 1,3-dichloropropan-2-ol (b. p. 173–175°). Stirring was continued until the evolution of nitrogen and hydrogen chloride ceased (about seventeen hours). Most of the excess dichloropropanol was removed by distillation at 10 mm. pressure and the residue was then fractionated at a lower pressure using the 30″ column. The yield of the desired product was 5.8 g., b. p. 99.5–100° (0.5 mm.), $n^{25}D$ 1.5369. *Anal.* Found: C, 53.20; H, 4.92; Cl, 34.27. The ultraviolet absorption spectrum, exhibiting a maximum at 270 mμ, was virtually identical with that of material prepared by Method A.

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RECEIVED JUNE 14, 1948

(1) Krantz and Drake, U. S. Patent 2,330,979.
(2) Niederl and Roth, THIS JOURNAL, **62**, 1154 (1940).
(3) Nystrom and Brown, *ibid.*, **69**, 1197 (1947).
(4) Performed by Wm. Saschek.
(5) Pray, *J. Phys. Chem.*, **30**, 1478 (1926).
(6) Present address: Department of Chemistry, University of California at Los Angeles.

γ-Chlorocrotylmercaptoacetic Acid and γ-Chlorocrotylmercaptomethylpenicillin

One hundred thirty-five grams of 1,3-dichloro-2-butene (du Pont, Organic Chemicals Department) was added to a solution of 85 g. of sodium hydroxide and 108 g. of mercaptoacetic acid (85%) in 1.0 liter of water over a period of two hours. Rapid mechanical stirring was used and the mixture was held at 45–50° during the addition of the halide and for four hours thereafter. The mixture was extracted with ethylene dichloride and the aqueous layer was acidified with concentrated hydrochloric acid. The resulting oil was extracted with ethylene dichloride. After removal of the solvent the residual liquid was vacuum distilled. The main fraction boiled at 108–111° (0.6 mm.) and was a yellow liquid with a pronounced skunk-like odor; yield 128 g. (77%).

Anal. Calcd. for $C_6H_9ClO_2S$: C, 39.89; H, 5.02; neut. eq., 180.7. Found: C, 39.91; H, 5.04; neut. eq., 180.6.