

**Ethyl  $\beta$ -Keto- $\gamma$ -phenoxyheptoate.**—This compound was prepared by the procedure described for ethyl  $\beta$ -keto- $\epsilon$ -phenoxyhexoate by the use of 6 g. of sodium, 175 cc. of diethyl carbonate, and 50 g. of  $\delta$ -phenoxybutyl methyl ketone. The distillation of the residue from the concentration of the ether extract yielded 3 cc. of forerun to 158° and 18 g. (26.5%) which distilled at 162–164° at 1 mm. In another run, the product was distilled in a Hickmann vacuum still using a bath temperature of 100–120° at 0.02 mm. pressure. Three fractions possessing  $n_D^{20}$  1.5009, 1.5020 and 1.5039 were collected. They totalled 28 g. (40%). The middle fraction possessed  $d_4^{25}$  1.0650,  $M_D$  calcd. (keto form) 71.19, (enol form) 72.23, found 73.10.

*Anal.* Calcd. for  $C_{15}H_{20}O_5$ : C, 68.12; H 7.63. Found: C, 68.45; H, 7.69.

The semicarbazone, after recrystallization three times from a large volume of water, melted at 212–214°.

*Anal.* Calcd. for  $C_{15}H_{22}O_4N_2$ : N, 13.08. Found: N, 12.99.

The N-phenylpyrazolone was prepared by warming 100 mg. of the keto ester and 50 mg. of phenylhydrazine for fifteen minutes in 5 cc. of 80% ethanol with 0.1 cc. of acetic acid. After recrystallization from 90% ethanol, it melted at 118.5–120°.

*Anal.* Calcd. for  $C_{19}H_{20}O_2N_2$ : N, 9.08. Found: N, 8.95.

Upon longer heating of the keto ester with an excess of phenylhydrazine in alcohol alone, a cleavage of the keto ester occurred and  $\delta$ -phenoxyvaleryl phenyl hydrazide was obtained. After recrystallization from 75% ethanol, it sintered at 240° and melted at 285–287°.

*Anal.* Calcd. for  $C_{17}H_{20}O_2N_2$ : N, 9.85. Found: N, 9.87.

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#### Di-( $\beta$ -phenoxyethyl) Oxalate

Incidental to a study of plasticizers, several aryloxyalkyl esters of oxalic acid have been prepared. Among these is di-( $\beta$ -phenoxyethyl) oxalate. A search of the literature reveals that this particular ester has not been prepared previously.

The apparatus consisted of a 500-ml. 3-necked round-bottom flask equipped with a thermometer and gas leg in one side neck, an air condenser ( $\frac{3}{16}$ " inside diameter, 2 feet in length) in the other side neck and a mercury-sealed mechanical agitator in the middle neck. The flask was electrically heated.

The charge consisted of 63 g. of oxalic acid dihydrate, 138.2 g. of  $\beta$ -phenoxyethanol (phenol "cellosolve") and 2 ml. of a catalyst consisting of 50 g. of 95% sulfuric acid, 100 g. of phosphorous acid and 150 g. of methanol. The mixture was gradually heated under an atmosphere of inert gas, *i. e.*, carbon dioxide or nitrogen, to 150° and held at that temperature for four hours. On cooling, the solution solidified to a white, wax-like solid. The yield of crude product, m. p. 112.5–114.5°, was 156.5 g. or 95%. Crystallization from benzene and/or acetone resulted in a product melting sharply at 114°.

*Anal.* Calcd. for  $C_{18}H_{18}O_6$ : C, 65.5; H, 5.49; sapn. equiv., 165.2. Found: C, 65.7; H, 5.54; sapn. equiv., 164.8.

The ester is a hard, white wax-like solid and may find use as a synthetic wax.

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#### $\alpha$ -Auromercapto-acetanilide

$\alpha$ -Carbamylmercapto-acetanilide was prepared in 60 and 80% yields by the procedures of Jaeger<sup>1</sup> and of Beckurts and Frerichs,<sup>2</sup> respectively. It crystallized from methanol or glacial acetic acid as plates or scales, m. p. 159° (cor.).<sup>3</sup> Heated with dilute ammonium hydroxide,  $\alpha$ -mercapto-acetanilide was formed, m. p., after crystallization from methanol, 113° (cor.).

**Procedure.**—Sulfur dioxide was introduced into a solution of 10 g. of potassium auric bromide in 100 cc. of 95% alcohol. The potassium bromide which formed was removed by filtration and the light yellow solution added to an alcoholic solution of 3 g. of  $\alpha$ -mercapto-acetanilide. A white precipitate formed at once. The mixture was diluted with water, the solid separated by centrifugation, washed repeatedly and successively with water, anhydrous ethanol and pentane, and dried *in vacuo* over phosphorus pentoxide. A yellowish gray powder insoluble in all common organic solvents was thus obtained. Upon heating, the compound darkened at about 230° and decomposed at 254°.

*Anal.*<sup>4</sup> Calcd. for  $C_8H_8NO_3Au$ : C, 26.42; H, 2.23; N, 3.85; S, 8.82; Au, 54.3. Found: C, 26.57; H, 2.52; N, 3.89; S, 8.72; Au, 54.58.

$\alpha$ -Auromercapto-acetanilide, when treated with a 4% solution of  $SeOCl_2$  in concentrated sulfuric acid, gives an intense purplish-red color. This reaction is given by other derivatives of  $\alpha$ -mercapto-acetanilide,  $C_6H_5NHC(=O)CH_2SX$ , where X = metal, -acyl, -CONH<sub>2</sub>,  $SCH_2CONHC_6H_5$ , etc.

(1) Jaeger, *J. prakt. Chem.*, [2] **16**, 18 (1877).

(2) Beckurts and Frerichs, *ibid.*, [2] **66**, 173 (1902).

(3) Jaeger<sup>1</sup> gives 148–152° and Beckurts<sup>2</sup> gives 147°.

(4) Microanalyses by Dr. Carl Tiedcke.

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#### BOOKS RECEIVED

June 10, 1945–July 10, 1945

R. E. BURK and OLIVER GRUMMITT, Editors. "Advances in Nuclear Chemistry and Theoretical Organic Chemistry." (Frontiers in Chemistry. Vol. III.) Interscience Publishers, Inc., 215 Fourth Avenue, New York 13, N. Y. 165 pp. \$3.50.

RENÉ J. DUBOS. "The Bacterial Cell in its Relation to Problems of Virulence, Immunity and Chemotherapy." (Harvard University Monographs in Medicine and Public Health.) Harvard University Press, Cambridge, Mass. 460 pp. \$5.00.

H. H. LOWRY, Chairman. "Chemistry of Coal Utilization." Vols. I and II. Prepared by the Committee on Chemical Utilization of Coal, Division of Chemistry and Chemical Technology, National Research Council. John Wiley and Sons, Inc., 440 Fourth Avenue, New York, N. Y. (London, Chapman and Hall, Ltd.) 920 pp. + 1868 pp. Set of two volumes, \$20.00.

FRANS VERDOORN, Editor. "Plants and Plant Science in Latin America." (Vol. 16 of "A New Series of Plant Science Books.") The Chronica Botanica Co., Waltham, Mass.; G. E. Stechert and Co., New York City, N. Y. 381 pp. \$6.00.

"A.S.T.M. Standards on Rubber Products (with Related Information)." Prepared by A.S.T.M. Committee D-11 on Rubber Products, December, 1944. Published by the American Society for Testing Materials, 260 Broad Street, Philadelphia 2, Pa.