Ethyl β-Keto-ζ-phenoxyheptoate.—This compound was prepared by the procedure described for ethyl β-keto-ε-phenoxyhexoate by the use of 6 g. of sodium, 175 cc. of diethyl carbonate, and 50 g. of δ-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 $n^{21}\text{D}$ 1.5009, 1.5020 and 1.5039 were collected. They totalled 28 g. (40%). The middle fraction possessed d^{23}_{23} 1.0650, $M\text{D}$ calcd. (keto form) 71.19, (enol form) 72.23, found 73.10.

Anal. Calcd. for $C_{15}H_{20}O_4$: 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 $C_{16}H_{23}O_4N_3$: 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 δ-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.

DEPARTMENT OF BIOCHEMISTRY
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Di-(β-phenoxyethyl) Oxalate

Incidental to a study of plasticizers, several aryloxyalkyl esters of oxalic acid have been prepared. Among these is di-(β-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 ($^3/_{16}$" 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 β-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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α-Auromercapto-acetanilide

α-Carbamylmercapto-acetanilide was prepared in 60 and 80% yields by the procedures of Jaeger[1] and of Beckurts and Frerichs,[2] respectively. It crystallized from methanol or glacial acetic acid as plates or scales, m. p. 159° (cor.).[3] Heated with dilute ammonium hydroxide, α-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 α-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.[4] Calcd. for C_8H_8NOSAu: 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.

α-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 α-mercapto-acetanilide, $C_6H_5NHCOCH_2SX$, where X = metal, -acyl, -$CONH_2$, $SCH_2CONHC_6H_5$, etc.

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

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

(3) Jaeger[1] gives 148–152° and Beckurts[2] gives 147°.

(4) Microanalyses by Dr. Carl Tiedcke.

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BOOKS RECEIVED

June 10, 1945–July 10, 1945

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