

ing current (1.72%) and 0.24%, which is the temperature coefficient of the product  $m^{2/3}t^{1/6}$ .<sup>4</sup> The corresponding figure calculated from Lingane and Kolthoff's data for zinc at 25° is 3.42.

(4) D. Ilkovic, *Coll. Czech. Chem. Comm.*, **10**, 249 (1938).

DIVISION OF SOILS, COUNCIL FOR  
SCIENTIFIC AND INDUSTRIAL RESEARCH  
WAITE INSTITUTE, ADELAIDE, SOUTH AUSTRALIA  
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## NEW COMPOUNDS

### Semicarbazone of the Methyl Ester of Azelaic Half-aldehyde

After splitting methyl 9,10-dihydroxystearate with lead tetraacetate,<sup>1</sup> the aldehydic methyl ester was separated from pelargonic aldehyde by fractional distillation, b. p. 159–164° (26 mm.). By the usual procedure, the semicarbazone of the ester could be prepared easily. From a benzene–ligroin mixture prisms are obtained, m. p. 107°.

*Anal.* Calcd. for C<sub>11</sub>H<sub>21</sub>O<sub>3</sub>N<sub>3</sub>: C, 54.3; H, 8.6; N, 17.3. Found: C, 54.3; H, 8.7; N, 16.8.

Scanlan and Swern<sup>2</sup> could not isolate the semicarbazone of the corresponding ethyl ester in a crystalline form.

(1) Hsing and Chuang, *This Journal*, **61**, 3589 (1939).

(2) Scanlan and Swern, *ibid.*, **62**, 2305 (1940).

DANIEL SIEFF RESEARCH INSTITUTE  
REHOVOTH, PALESTINE

F. BERGMANN

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### Quinoline Derivatives

**1,2-Dimethyl-1,2,3,4-tetrahydroquinoline zinci-hydrochloride** was prepared from 1,2-dimethyl-1,2,3,4-tetrahydroquinoline by the method of Emerson and Davis.<sup>1</sup> After recrystallization from glacial acetic acid, filtration, and washing free of acetic acid with ether, the compound melted at 152–154°.

*Anal.* Calcd. for 2C<sub>11</sub>H<sub>15</sub>N·2HCl·ZnCl<sub>2</sub>: Cl, 26.7. Found: Cl, 26.4.

**1,2-Dimethyl-1,2,3,4-tetrahydroquinoline hydriodide** was prepared by mixing equal volumes of 1,2-dimethyl-1,2,3,4-tetrahydroquinoline and hydriodic acid of sp. gr. 1.50. After crystallization had been induced by adding ethyl acetate, the product was collected on a filter and crystallized from absolute alcohol, m. p. 138.5–140.0°.

*Anal.* Calcd. for C<sub>11</sub>H<sub>15</sub>NI: I, 44.0. Found: I, 43.7.

**2,6,8-Trimethylquinoline zinci-hydrochloride** was also prepared from 2,6,8-trimethylquinoline by the procedure of Emerson and Davis.<sup>1</sup> It was purified in the usual way, m. p. ca. 200°.

*Anal.* Calcd. for 2C<sub>12</sub>H<sub>15</sub>N·2HCl·ZnCl<sub>2</sub>: Cl, 25.8. Found: Cl, 25.7.

NOVES CHEMICAL LABORATORY  
UNIVERSITY OF ILLINOIS  
URBANA, ILLINOIS

WILLIAM S. EMERSON  
J. W. DAVIS

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(1) Emerson and Davis, *This Journal*, **61**, 2830 (1939).

### 6-Bromo-3-methoxybenzyl Alcohol and Some Derivatives

**6-Bromo-3-methoxybenzyl Alcohol.** A.—A mixture of 25 g. of 3-methoxy-6-bromobenzaldehyde<sup>1</sup> and 4 g. of aluminum ethoxide in 100 cc. of absolute ethyl alcohol was allowed to stand at room temperature for ten days. The ethyl alcohol and volatile products were then distilled from an oil-bath heated to 120°. The residue was treated with dilute hydrochloric acid and extracted twice with ether. The ether extracts were dried over anhydrous sodium sulfate and the ether evaporated. The crude 6-bromo-3-methoxybenzyl alcohol remained as a slightly oily, crystalline mass which was sufficiently pure for use, yield, 22–25 g. (88–100%).

B.—To a solution of 24 g. of 3-methoxy-6-bromobenzaldehyde in 150 cc. of hot alcohol was added 0.1750 g. of Adams platinum oxide catalyst<sup>2</sup> and 0.013 g. of ferrous chloride. The mixture was shaken with hydrogen until absorption ceased. The platinum was coagulated by the addition of 0.5 cc. of 0.5 *N* sodium hydroxide and filtered out. The filtrate was distilled to remove the alcohol and the brown oily residue was dissolved in ether and dried over anhydrous sodium sulfate. The ether solution was evaporated to about 50 cc., petroleum ether added to turbidity and the solution cooled. Small colorless plates were deposited which were filtered out, washed with a little petroleum ether and dried; yield, 20.5 g. (83%). A portion was crystallized from alcohol, m. p. 49°.

*Anal.* Calcd. for C<sub>8</sub>H<sub>9</sub>O<sub>2</sub>Br: Br, 36.83. Found: Br, 35.63, 35.76.

**6-Bromo-3-methoxybenzyl Chloride.**—To a solution of 25 g. of 6-bromo-3-methoxybenzyl alcohol in 100 cc. of chloroform there was added 12.5 g. of phosphorus trichloride. The mixture was allowed to stand for twenty-four hours and was then poured onto crushed ice to decompose the unreacted phosphorus trichloride. The mixture was extracted with ether, the ether solution washed with sodium bicarbonate solution and dried over anhydrous sodium sulfate. Evaporation of the ether gave 18 g. (66%) of the crude product. A portion was recrystallized from alcohol, m. p. 75.4–76°.

*Anal.* Calcd. for C<sub>8</sub>H<sub>9</sub>OClBr: Cl, 15.06. Found Cl, 14.98.

**6-Bromo-3-methoxybenzyl Methyl Ether.**—To 5.5 g. of sodium in 125 cc. of absolute methyl alcohol there was added 47 g. of 6-bromo-3-methoxybenzyl chloride. The mixture was boiled for two hours. It was allowed to cool and sufficient water was added to dissolve the precipitated salt. The solution was extracted with ether. The ether extract was dried over anhydrous sodium sulfate and the ether distilled off. The residue was distilled in a vacuum, yield, 33 g. (72%), b. p. (9 mm.) 126–129°,  $n_D^{25}$  1.5515,  $d_4^{25}$  1.4259.

*Anal.* Calcd. for C<sub>9</sub>H<sub>11</sub>O<sub>2</sub>Br: Br, 34.60. Found: Br, 34.55, 34.52.

CHEMICAL LABORATORY  
WASHINGTON UNIVERSITY  
ST. LOUIS, MISSOURI

JOHN H. GARDNER  
THOMAS F. McDONNELL

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(1) Pschorr, *Ann.*, **391**, 26 (1912).

(2) Adams, Voorhees and Shriner, "Organic Syntheses," Collective Volume I, 452 (1932).