A syntesis of β-Phthalimidopropionaldehyde

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Phthaloyl derivatives of amino acids have proved very useful for the further synthesis of compounds in this series[1]). Applying this method and the Rosenmund-Zetsche reduction[2]) E. Radde[3]) prepared α-phthalimidopropionaldehyde from α-phthalimidopropionylchloride. In the same manner we have obtained from β-phthalimidopropionylchloride β-phthalimidopropionaldehyde as a crystalline solid in 90% yield. This aldehyde was prepared first by Moe and Warner[4]) from phthalimide and acrolein in 26% yield. As a new compound we have prepared the semicarbazone of this aldehyde, m. p. 225.5—226°.

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EXPERIMENTAL

β-Phthalimidopropionaldehyde. — β-phthalimidopropionylchloride[5]) (15 g., 0.063 mole) was reduced with hydrogen in boiling xylene (60 ml.) with 2 g. of 5% Pd-$BaSO_4$ as catalyser[6]). The evolved hydrogen chloride was absorbed in water and determined with a 0.5 N sodium hydroxyde solution. After 3 hours, 87% of the theoretical amount of hydrochloric acid was estimated, and the reaction practically ceased. The hot solution was filtered in order to remove the suspended catalyser, and the filtrate left to crystallize in an ice-box. Yield of the crude, colourless β-phthalimidopropionaldehyde was 11.5 g. (90%), m. p. 112—113°. On recrystallization from benzene m. p. 118°. For the analysis the aldehyde was sublimated at 110—115° / 0,016 mm. Colourless needles, m. p. 118.5—119°. Reported[4]), m. p. 119—120°.

Semicarbazone, from β-phthalimidopropionaldehyde and semicarbazide acetate in methanol, m. p. 225.5—226°. White prisms.

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1) S. Gabriel, Ber., **40** (1907) 2647; K. Balenović, Experientia 3 (1947) 369; J. C. Cheenan and V. S. Frank, J. Am. Chem. Soc., **71** (1949) 1856.

2) cf. e. g. E. Mosettig and R. Mozingo, »Organic Reactions«, Sv. IV, New York 1948, pp. 362—377.

3) E. Radde, Ber., **55** (1922) 3174.

4) O. A. Moe and D. T. Warner, J. Am. Chem. Soc., **71** (1949) 1251.

5) S. Gabriel, Ber., **38** (1905) 633.

6) 1. c. (2) pp. 368—369.

8.307 mg substance gave 16.92 mgCO_2, 3.65 mg H_2O
Calc'd for $C_{21}H_{12}O_3N_4$ (260.25): C, 55.38; H, 4.65.
Found: C, 55.58; H, 4.92.

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IZVOD

Sinteza β-ftalimidopropion aldehida

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Redukcijom po Rosenmund-Zetsche-u priređen je β-ftalimidopropionaldehid iz β-ftalimidopropionilklorida u 90% iskorištenju. Novo je opisan semikarbazon toga aldehida sa T. t. 225.5—226°.

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