

LABORATORY NOTES

LABORATORIJSKE BILJEŠKE

Note on the Preparation of Ethyl Phenylmalonate*D. Dvornik and P. Hammes**Research Department, »Pliva« Pharmaceutical and Chemical Products, Zagreb, Croatia*

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The procedure based on the method of Wislicenus¹, as reported by Levene and Meyer², was simplified in modifying the process described for the preparation of the methyl ester³.

To a mixture of ethyl phenylacetate (164 g, 1,0 mole) and ethyl oxalate (146 g, 1,0 mole) a warm solution (50°C) of sodium ethoxide (prepared from 34,5 g, 1,5 gatom sodium and 500 ml anhydrous ethanol) was gradually added with occasional stirring, giving soon a nearly solid paste of the sodium derivative. After standing overnight the paste was treated with dilute hydrochloric acid, the phenyloxaloacetic ester isolated in the usual manner and pyrolyzed for one hour at 170° under reduced pressure. Distillation of the pyrolyzed residue gave 196–205 g (83–87%) of ethyl phenylmalonate, boiling at 103–105°/0,2 mm Hg (135°/1–1,5 mm⁴), $D^{15} : 1,101$ ($D_4^{20} : 1,0950^5$), $n_D^{20} : 1,4920$ ($n_D^{20} : 1,4977^5$).

REFERENCES

1. W. Wislicenus, *Ber.* **27** (1894) 1091.
2. *Org. Synth. Coll. Vol. II*, 1946, 288.
3. *B.I.O.S. Final Report No. 1404*, Item 22 & 24, p. 85.
4. B. Flürscheim, E. L. Holmes, *J. Chem. Soc.* **1928**, 1613.
5. H. Scheibler, E. Marhenkel, D. Bassanoff, *Ber.* **58** (1925) 1203.

IZVOD

Bilješka o pripremi etil fenilmalonata*D. Dvornik i P. Hammes*

Modifikacijom postupka za pripremu metil fenilmalonata³, pojednostavljena je Leven-Meyer-ova razrada² Wislicenus-ove metode za pripremu etil fenilmalonata kondenzacijom etil fenilacetata i etil oksalata uz natrijski etoksid i naknadnom pirolizom intermedijernog etoksalil-derivata¹.

ISTRAŽIVAČKI ODJEL,
»PLIVA« TVORNICA LIJEKOVA
ZAGREB

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