

LABORATORY NOTES

LABORATORIJSKE BILJEŠKE

Note on the Preparation of Diethyl Malonate*D. Dvornik and P. Hammes*

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Chlorsulfonic acid was successfully used as a catalyst in some esterifications¹⁻⁶. It was found that in the presence of chlorosulfonic acid an ethanolic solution of cyanoacetic acid is simultaneously hydrolyzed and esterified to give diethyl malonate with 82—84% yield, based on chloroacetic acid.

The solution of cyanoacetic acid, obtained from chloroacetic acid (25 g, 0,265 mole)⁷ after acidification with diluted sulfuric acid, was evaporated to dryness under reduced pressure. The residue was suspended in 98,8% ethanol (110 ml) and chlorosulfonic acid (43,3 g, 0,38 mole) was added with stirring and cooling. After refluxing for seven hours the mixture was poured into water and the ester isolated in the usual manner. Thus, 34,8—35,7 g (82—84%) of practically nitrogen-free diethyl malonate was obtained (b. p. 86—89°C/18 mm.).

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IZVOD

Bilješka o pripremi dietil malonata*D. Dvornik i P. Hammes*

Istodobnom hidrolizom i esterifikacijom cijanoctene kiseline u etanolu uz klor-sulfonsku kiselinu dobiven je dietil malonat (82—84% obz. na kloroctenu kiselinu). Rezultati su objavljeni dozvolom Glavnog inženjera.

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