

HYDROGENATION OF ALKENYLFURAN-MALEIC ANHYDRIDE ADDUCTS

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Maleic anhydride-alkenylfuran adducts with the double bond 2, 3 to the ring, first prepared by the present authors [1], being available, conditions have been found for selective and exhaustive hydrogenation of one of them in the presence of Pd/BaSO₄. The compounds synthesized are of interest as potential herbicides and plant growth regulators.

Experimental

1 g 3-(3-methylbuten-2-yl)-3, 6-endoxo- Δ^4 -tetrahydrophthalic anhydride [1] was hydrogenated under the usual conditions in 25 ml dry ether containing 0.5 g 5% Pd/BaSO₄. After 4 hr, 188 ml H₂ (99%) had been absorbed, after which absorption ceased. 0.89 g (87%) 3-(3-methylbutyl)-3, 6-endoxohexahydrophthalic acid, mp 171-172° (ex water) was obtained. Found: C 61.19, 60.98; H 8.17, 8.05%, acid value 436.9. Calculated for C₁₃H₂₀O₅: C 60.92; H 7.86%, acid no. 439.8.

1.43 g 3-(3-methylbuten-2-yl)-3, 6-endoxo- Δ^4 -tetrahydrophthalic anhydride was dissolved in 40 ml 5% NaOH, filtered immediately, and added to 1 g 5% Pd/BaSO₄, previously saturated with H₂ while suspended in 10 ml water. In 1 hr 45 min, 139 ml H₂ was absorbed, after which the rate of hydrogenation dropped sharply. The hydrogenation was terminated, the catalyst separated off, and the filtrate acidified with H₂SO₄ to give 1.1 g (67%) 3-(3-methylbuten-2-yl)-3, 6-endoxohexahydrophthalic acid, mp 150°. Found: C 57.35, 57.55; H 7.55, 7.62%, acid no. 411.7, bromine value 69.13. Calculated for C₁₃H₁₈O₅·H₂O: C 57.34; H 7.40%, acid no. 412.4, bromine value 58.90.

REFERENCE

1. Yu. I. Tarnopol'skii and V. N. Belov, ZhOKh, 1, 595, 1965.

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