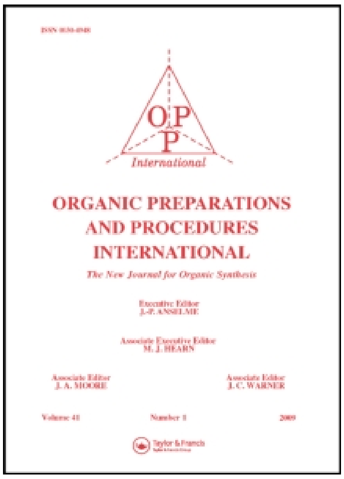

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A FACILE REDUCTION OF 1,8-NAPHTHALIMIDE TO 1,2,3,4-TETRAHYDRO-1,8-NAPHTHALIMIDE

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A FACILE REDUCTION OF 1,8-NAPHTHALIMIDE TO 1,2,3,4-TETRAHYDRO-1,8-NAPHTHALIMIDE

Submitted by
(03/20/89)

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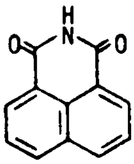
In connection with the synthesis of several targeted heterocyclic compounds for biological evaluation, a large quantity of 1,2,3,4-tetrahydro-1,8-naphthalimide (3a,4,5,6-Tetrahydro-1H-benz[d,e]isoquinoline-1,3[2H]-dione, 2) was needed as an intermediate. A search of the literature revealed only one preparative method of 2 by reduction of 1,8-naphthalimide (1) at high pressure (50 kg/cm^2 or 711 psi) and elevated temperature (80°) over palladium-on-carbon.[1] Although this method of preparation gave a good yield (77%) of 2, it required equipment capable of withstanding rather high pressure and relatively high temperature. These investigators also studied the hydrogenation process with other catalysts, including the use of platinum-on-carbon and rhodium-on-carbon, but again found that only at higher temperature (100°) and high pressure could the compound be reduced, and when rubidium-on-carbon was used, no reduction took place even at elevated temperature and pressure.[1]



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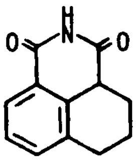

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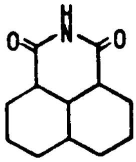

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The catalytic reduction conditions were reinvestigated in our laboratory in order to uncover mild reaction conditions for general use. Since the original report[1] indicated that reduction did not take place in neutral solvents (e.g., ethanol) even at high temperature and could only be accomplished in acidic solvents (e.g., acetic acid), an investigation using a variety of catalysts with different supports was conducted. The following describes for the first time reduction of 1 to 2 under mild conditions suitable for the Parr hydrogenation apparatus by use of rhodium-on-alumina in basic ethanol. The reaction conditions used here may be useful for the preparation of heterocyclic compounds containing partially ring saturated naphthalene derivatives[2] since at elevated temperature the formation of undesired decalin-1,8-dicarboximide (3) has also been reported.[1]

EXPERIMENTAL SECTION

1,2,3,4-Tetrahydro-1,8-naphthalimide.- 1,8-Naphthalimide (8 g) was dissolved in ethanol (40 ml) and water (160 ml) containing sodium hydroxide (1.48 g). The catalyst, 5% Rh/alumina (1.1 g), was added and the mixture was hydrogenated at 25 psi in a Parr hydrogenator at room temperature. After hydrogen uptake ceased (ca. 6.5 hrs.), the catalyst was removed by filtration and the filtrate acidified with conc. hydrochloric acid to give, after recrystallization from 200 ml of ethanol, 6.7 g (82% yield) of 1,2,3,4-tetrahydro-1,8-naphthalimide (2) as a white solid, mp. 198-200°, lit.[1] mp. 199-202°. MS: (m/e) 201 (M^+).

Anal. Calcd. for $C_{12}H_{11}NO_2$: C, 71.63; H, 5.51; N, 6.96. Found: C, 71.90; H, 5.40; N, 6.98

Use of 0.6 g of 5% Rh/alumina at 31 psi of hydrogen at room temperature gave a 74% yield of 2 after 20 hrs. from same amount of starting material.

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