

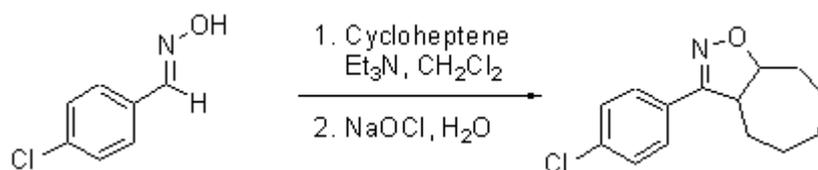
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3a,4,5,6,7,8,8a-Heptahydro-3-(4-chlorophenyl)cyclohept[d]isoxazole

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To a solution of 4-chlorobenzaldehyde oxime (0.40g) in CH₂Cl₂ (10 mL) in a large test-tube were added 1.0 mL of cycloheptene and 3 drops of triethylamine. The solution was cooled to 5°C, then 7.0 mL of 5% NaOCl solution in water was added in small portions. After each portion was added, the test-tube was agitated with a Vibromixer™ stirrer for 15 seconds. After the addition of all of the NaOCl solution, the test-tube was agitated with a Vibromixer™ stirrer for 15 seconds every 5 minutes over the next hour. The reaction mixture was allowed to stand at least overnight. The layers were separated, and the aqueous layer was extracted with CH₂Cl₂ (5 mL). The combined CH₂Cl₂ layers were evaporated to yield an off-white solid. The solid was recrystallized from methanol to yield shiny white plates. Yield: 0.26g (37%).

M.p. 97-97.5°C.

IR (KBr pellet, cm⁻¹): 3085, 3070, 2930, 2909, 2871, 2854, 1593, 1494, 1469, 1402, 1383, 1343, 1091, 919, 899, 838.

¹H-NMR (300MHz, CDCl₃, ppm): 1.35-2.1 (10 H, multiplets), 3.68 (1H, m), 4.87 (1H, m), 7.36 (2H, m), 7.59 (2H, m).

¹³C-NMR (75.5 MHz, CDCl₃, ppm): 159.7, 136.0, 129.4, 128.7, 128.3, 85.6, 51.7, 31.5, 30.5, 28.6, 27.4, 24.1.

GC-MS (ion trap, m/e, in order of decreasing peak size): 137 (100%), 102, 50, 75, 139, 51, no molecular ion detected.

References and Notes

- Gingrich, H. L.; Pickering, M. *J. Chem. Educ.* **1991**, *68*, 614-615.
- Bianchi, G.; De Micheli, C.; Gandolfi, R. *J. Chem. Soc., Perkin Trans. 1* **1972**, 1711-1714.

Sample Availability: available from MDPI.

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