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3a,4,5,6,7,8,9,9a-Octahydro-3-(4-chlorophenyl)cyclooct[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 cyclooctene 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 VibromixerTM stirrer for 15 seconds. After the addition of all of the NaOCl solution, the test-tube was agitated with a VibromixerTM stirrer for 15 seconds every 5 minutes over the next hour. The reaction mixture was allowed to stand 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 95% ethanol to yield shiny white plates. Yield: 0.51g (75%).

M.p. 124-125°C.

IR (KBr pellet, cm⁻¹): 3085, 3070, 1930, 2909, 2871, 2854, 1593, 1494, 1469, 1402, 1383, 1343, 1091, 919, 899, 838.

¹H-NMR (300MHz, CDCl₃, ppm): 1.2-2.2 (12 H, multiplets), 3.40 (1H, t, J=9.4 Hz), 4.50 (1H, m), 7.38 (2H, m), 7.61 (2H, m).

¹³C-NMR (75.5 MHz, CDCl₃, ppm): 161.9, 136.0, 129.5, 128.6, 128.3, 86.2, 50.5, 30.6, 25.9, 25.8, 25.6, 25.2, 25.0.

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

- 1. Gingrich, H. L.; Pickering, M. J. Chem. Educ. 1991, 68, 614-615.
- 2. 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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1 von 1 05.05.2009 15:50