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4-Dodecyl-6-methyl-2-morpholone

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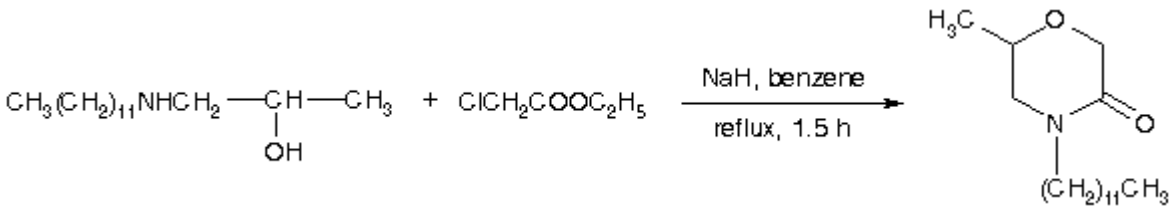

2-Morpholones may be synthesized from the corresponding beta-amino alcohols by heating with an alpha-halo ester [1]. Several alternative methods have also been described [2].

1-Dodecylamino-2-propanol (4.86 g, 20 mmol) was added to a stirred suspension of NaH (0.52 g, 21.5 mmol) in dry benzene (20 ml). The resulting mixture was cooled (ice) and ethyl chloroacetate (2.45 g, 20 mmol) was added during 15 min followed by stirring at room temperature for 1 h and for an additional 1h under reflux. After cooling, ether (50 ml) was added and the mixture was washed with 2 N HCl and water. The organic layer was dried and the solvent evaporated to give a crude product. Recrystallization from hexane afforded the title compound (2.6 g, 46%) as white crystals.

M.p. 37-39 deg.C.

TLC (Hexane/EtOAc 2:1): R_f 0.25.

^{1}H-NMR ($CDCl_3$): 4.18 (ABq, J=16.5 Hz, 2H, H-2); 3.87 (m, J=9.8 and 6.3 and 3.3 Hz, 1H, H-6); 3.40 (m, 1H, C$\underline{H}_2$N); 3.31 (m, 1H, C$\underline{H}_2$N); 3.25 (dd, J=12.0 and 9.8 Hz, 1H, H-5); 3.12 (dd, J=12.0 and 3.3 Hz, 1H, H-5'); 1.54 (m, 2H, C$\underline{H}_2$CH_2N); 1.27 (d, J=6.3 Hz, 3H, CH_3); 1.26 (s, 18H, 9 x CH_2); 0.88 (t, J=6.4 Hz, 3H, CH_3).

^{13}C-NMR ($CDCl_3$): 166.5 (C=O), 69.6 (C-6), 67.8 (C-2), 52.4 (C-5), 46.5 (C$\underline{H}_2$N), 31.9, 29.6, 29.4, 26.9, 22.7 (the other CH_2 groups), 18.3 (CH_3 at C-6), 14.1 (CH_3).

Anal. calc. for $C_{17}H_{33}NO_2$ (283.45): C 72.04, H 11.73, N 4.94; found: C 72.19, H 11.82, N 4.90.

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References and Notes

1. Clarke, F. H. *J. Org. Chem.* **1962**, 27, 3251.
2. Sainsbury, M. in Coffey, S. (Ed.), *Rodd's Chemistry of Carbon Compounds*, Vol. IV, Part H, Elsevier, Amsterdam, 1978, p. 427.

Sample availability: Available from the authors and MDPI, MDPI Reg. No.13732.