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3-Dodecyl-5-methyl-2-oxazolidinthione

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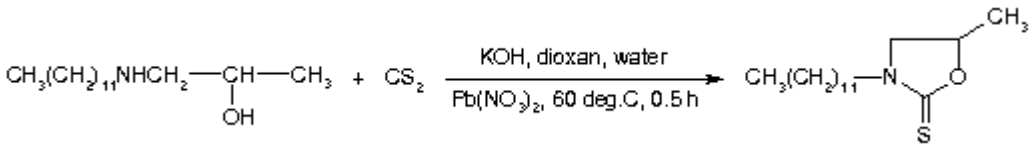

Oxazolidin-2-thiones can be prepared by the cyclization reaction of corresponding beta-amino alcohols with carbon disulfide or thiophosgene [1,2].

A solution of carbon disulfide (1.60 g, 21 mmol) in dioxane (10 ml) was added dropwise to a cold (0deg.C) mixture of 1-dodecylamino-2-propanol (4.87 g, 20 mmol) and KOH (1.12 g, 20 mmol) in water (10 ml) under stirring for 15 min, during which the mixture warmed to room temperature and became yellow and homogeneous. Additional KOH (1.12 g, 20 mmol) in water (20 ml) and lead nitrate (3.65 g, 11 mmol) in water (20 ml) were then added and the suspension was stirred at 60 deg.C for 0.5 h. The orange precipitate was filtered off, washed with MeOH and the solvent evaporated. The product was recrystallized from hexane to give the title compound as white crystals (3.2 g, 56%).

M.p. 37-38 deg.C.

TLC (Hexane/EtOAc 2:1): R_f 0.53.

^{1}H-NMR ($CDCl_3$): 4.84 (m, J=9.6 and 7.8 and 6.3 Hz, 1H, H-5); 3.85 (t, J=9.6 Hz, 1H, H-4); 3.63 (m, 2H, C<u>H</u>$_2$N); 3.33 (dd, J=9.6 and 7.8 Hz, 1H, H-4'); 1.63 (m, 2H, C<u>H</u>$_2$CH$_2$N); 1.49 (d, J=6.3 Hz, 3H, CH_3); 1.33 (bs, 4H, 2 x CH_2); 1.26 (s, 14H, 7 x CH_2); 0.88 (t, J=6.7 Hz, 3H, CH_3).

^{13}C-NMR ($CDCl_3$): 187.2 (C=S), 74.8 (C-5), 54.4 (C-4), 48.1 (<u>C</u>H$_2$N), 31.9, 29.6, 29.4, 29.3, 26.6, and 22.7 (the other CH_2 groups), 20.3 (CH_3 at C-5), 14.1 (CH_3).

Anal. calc. for $C_{16}H_{31}NOS$ (285.49): C 67.31, H 10.94, N 4.91, S 11.23; found: C 67.13, H 10.99, N 4.97, S 11.15.

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References and Notes

1. Ettlinger, M. G. *J. Am. Chem. Soc.* **1950**, *72*, 4792.
2. Sainsbury, M. in Ansell, M. F. (Ed.), *Rodd's Chemistry of Carbon Compounds*, 2nd edn., Vol. IV, Part C, Elsevier, Amsterdam, 1986, p. 303.

Sample availability: Available from the authors and MDPI, MDPI Reg. No.13741.