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1-(N,N-Dimethylamino)adamantane

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The title compound, an important antiviral agent [1] was prepared from commercially available 1-aminoadamantane by titanium(IV) isopropoxide mediated reductive amination of formaldehyde [2]. A mixture of 1-aminoadamantane (0.38 g, 2.5 mmol), titanium(IV) isopropoxide (1.42 g, 5 mmol) and paraformaldehyde (0.3 g, 10 mmol) was magnetically stirred at 50 °C for 2 h. The mixture was then cooled to ambient temperature, after which sodium borohydride (0.190 g, 5 mmol) and absolute ethanol (6 ml) were added. The resulting mixture was further stirred at room temperature for 3 h and at 50 °C for 2 h. The reaction was quenched with aqueous ammonia (15 ml, 2 M), the resulting precipitate of TiO₂ was filtered and washed with diethyl ether (2 x 10 ml). The organic layer was separated and the aqueous part was extracted with ether (20 ml x 2). The combined organic extracts were dried (Na₂SO₄) and concentrated to afford 1 as a colorless oil. Flash chromatography of the crude product over neutral alumina using a diethyl ether-hexanes mixture (4:1) furnished analytically pure 1 (0.39 g, 90%) as a colorless liquid.

¹H NMR (300 MHz, CDCl₃):1.55-1.75 (m, 12 H), 2.08 (br s, 3H), 2.26 (s, 6H).

¹³C NMR (75.5 MHz, CDCl₃): 29.6, 37.0, 37.2, 38.1, 53.7, 150.5.

Anal. Calc. for C₁₂H₂₁N: C 80.38, H 11.81, N 7.81. Found: C 80.39, H 12.03, N 7.64.

References

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- 2. Bhattacharyya, S. Tetrahedron Lett. 1994, 35, 2401; Bhattacharyya, S. J. Chem. Soc. Perkin Trans. 1, 1995, 1845.

Sample Availability: Available from the author.

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