

**SHORT
COMMUNICATIONS**

Synthesis of 1,8-Nonamethylene-3,6-diazahomoadamantan-9-one

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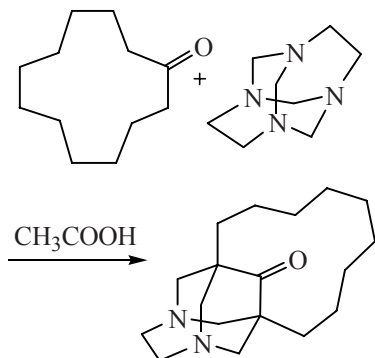
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By ketones condensation with 1,3,6,8-tetraazatricyclo[4.4.1.1^{3,8}]dodecene (tetramethylenediethylenetetramine) in 2-propanol in the presence of acetic acid we formerly obtained 3,6-diazahomoadamantan-9-one and its derivatives with substituents in the nodal positions [1–3]. We report here on the synthesis by similar procedure of 13,16-diazatetracyclo[9.6.1.1^{11,13}.1^{11,16}]eicosan-18-one (1,8-nonamethylene-3,6-diazahomoadamantan-9-one) by the condensation of cyclododecanone with tetramethylenediethylenetetramine.



1,8-Nonamethylene-3,6-diazahomoadamantan-9-one. A solution of 1.82 g (10 mmol) of cyclododecanone, 1.68 g (10 mmol) of tetramethylenediethylenetetramine, and 1.5 g (25 mmol) of acetic acid in 20 ml of 2-propanol was stirred at room temperature and left standing for 7 days. The reaction mixture was concentrated in a vacuum, the viscous residue was extracted with hot heptane (4×30 ml), the extract was passed through a bed of anhydrous aluminum oxide, the solvent was distilled off in a vacuum. The product was purified by crystallization from heptane. Yield 0.6 g (21%), mp 153–154°C.

IR spectrum (KBr), ν , cm^{-1} : 1715 (C=O). ¹H NMR spectrum, (CDCl₃), δ , ppm: 2.45 m (4H, NCH₂CH₂N), 3.25 br.s, 2.25 br.s (2H, NCH₂C), 3.15–1.85 m, 2.10–1.80 m (6H, NCH₂C), 1.70–0.70 m (18H, 9CH₂). ¹³C NMR spectrum (CDCl₃), δ , ppm.: 215.8 (C¹⁸), 62.2, 62.1, 60.9, 60.8, 54.7, 52.9 (C^{12,14,15,17,19,20}), 43.5 (C^{1,11}), 29.7, 25.8, 25.3, 24.4, 22.9, 22.6, 22.4, 22.3, 21.9 (C^{2–10}). Mass spectrum, m/z (I_{rel} , %): 290 (12) [M]⁺, 236 (86), 194 (16), 149 (27), 123 (25), 95 (54), 84 (87), 72 (87), 55(100), 43(59). Results of elemental analysis are in agreement with the calculated values.

IR spectrum of the compound under study was recorded on a spectrophotometer Bruker IFSv. The ¹H and ¹³C NMR spectra were registered on a spectrometer WM-250 (Bruker) (at 250.13 and 62.86 MHz respectively), internal reference TMS. Mass spectrum was measured on Finnigan MAT-90 instrument with a direct sample admission into the ion source, accelerating voltage 5.0 kV; cathode emission current 100 μA , ionizing electrons energy 70 eV, ionization chamber temperature 200°C. Perfluorokerosene was used as standard. The resolution $M/\Delta M$ was 10000.

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