

4 α ,14 α ,Dimethyl-5 α -cholest-8-en-3-one thiadiazoline

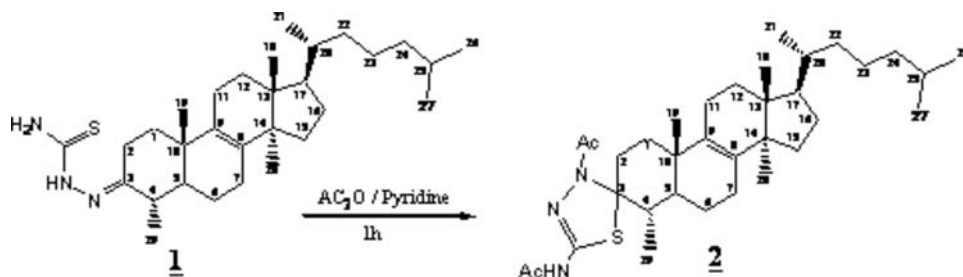
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The compound **2** (0.21g, 0.25mmol) was dissolved in 1ml of pyridine and 1ml of acetic anhydride^{1,2}. The mixture was heated at reflux during 1h with magnetic stirring, and then evaporated under reduced pressure. The residue was purified on silica gel column using hexane: ethyl acetate (80:20) as eluent led to **2** (91.04 mg, 0.16 mmol) in 64 % yield.

Melting point: 185-186 °C (Hexane)

MS (EI, 70eV): 569 (M^+).

¹H NMR (300 MHz, CDCl₃) d(ppm): 8.75 (NH, s); 2.21, 2.25 (COCH₃, 2s); 0.74 (3H-18, s); 1.15 (3H-19, s); 0.87 (3H-21, d, J = 6 Hz); 0.84 (3H-26, d, J = 2 Hz); 0.85 (3H-27, d, J = 2 Hz); 0.87 (3H-28, s); 1.10 (3H-29, d, J = 6 Hz).

¹³C NMR (75 MHz, CDCl₃) d (ppm): 36.42 (C-1); 37.36 (C-2); 100.04 (C-3); 51.29 (C-4); 50.74 (C-5); 21.80 (C-6); 28.30 (C-7); 129.05 (C-8); 131.13 (C-9); 36.31 (C-10); 22.00 (C-11); 25.50 (C-12); 44.89 (C-13); 49.53 (C-14); 30.84 (C-15); 29.85 (C-16); 46.46 (C-17); 15.80 (C-18); 18.20 (C-19); 36.01 (C-20); 18.71 (C-21); 35.86 (C-22); 24.32 (C-23); 32.45 (C-24); 34.60 (C-25); 21.75 (C-26); 21.85 (C-27); 24.25 (C-28); 14.25 (C-29); 143.17 (C=N); 170.15, 171.00 (COCH₃); 22.50, 24.25 (COCH₃).

MS (m/z): 569 (10%), 428 (42%), 315 (85%).

Acknowledgments

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References

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