

SHORT  
COMMUNICATIONS

## Synthesis of 1,8-Dimethyl-3,6,10,13-tetraazatetracyclo[8.4.1.18,13.02,7]hexadeca-2,6-diene

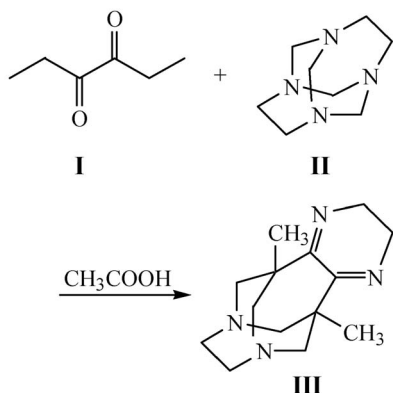
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We previously developed a convenient procedure for condensation of ketones with 1,3,6,8-tetraazatricyclo[4.4.1.13,8]dodecane (**II**) in the presence of acetic acid [1, 2]. Following a similar procedure, we were the first to effect condensation of 3,4-hexanedione (**I**) with compound **II** and obtain 1,8-dimethyl-3,6,10,13-tetraazatetracyclo[8.4.1.18,13.02,7]hexadeca-2,6-diene (**III**).



**1,8-Dimethyl-3,6,10,13-tetraazatetracyclo[8.4.1.18,13.02,7]hexadeca-2,6-diene (III).** A solution of 2.28 g (20 mmol) of 3,4-hexanedione (**I**), 3.36 g (20 mmol) of 1,3,6,8-tetraazatricyclo[4.4.1.13,8]-dodecane (**II**), and 3.00 g (50 mmol) of acetic acid in 100 ml of isopropyl alcohol was stirred for 2 h at room temperature. The mixture was then concentrated under reduced pressure, the viscous residue was treated with hot petroleum ether (bp 70–100°C; 4 × 100 ml), and the

extract was passed through a layer of anhydrous aluminum oxide. The solvent was removed under reduced pressure, and the product was purified by sublimation. Yield 0.45 g (20%), mp 129–130°C. IR spectrum (KBr):  $\nu$  1600 cm<sup>-1</sup> (C=N). <sup>1</sup>H NMR spectrum (CDCl<sub>3</sub>),  $\delta$ , ppm: 0.93 s (3H, CH<sub>3</sub>), 2.7–3.0 m (4H, NCH<sub>2</sub>CH<sub>2</sub>N), 3.07 s and 3.22 s (8H, NCH<sub>2</sub>C). <sup>13</sup>C NMR spectrum (CDCl<sub>3</sub>),  $\delta$ <sub>C</sub>, ppm: 63.4 (C<sup>4</sup>, C<sup>5</sup>), 57.8 (C<sup>11</sup>, C<sup>12</sup>), 45.6 (C<sup>9</sup>, C<sup>14</sup>, C<sup>15</sup>, C<sup>16</sup>), 22.7 (CH<sub>3</sub>). Mass spectrum,  $m/z$  ( $I_{\text{rel}}$ , %): 246 (93) [M]<sup>+</sup>, 231 (35), 203 (48), 202 (43), 190 (31), 174 (22), 161 (100), 153 (30), 149 (45), 147 (54).

The IR spectrum was recorded in KBr on a Bruker IFS spectrometer. The <sup>1</sup>H and <sup>13</sup>C NMR spectra were obtained on a Bruker WM-250 instrument at 250.13 and 62.86 MHz, respectively. The chemical shifts were measured relative to tetramethylsilane as internal reference. The mass spectrum was recorded on a Finnigan MAT-90 mass spectrometer with direct sample admission into the ion source; accelerating voltage 5 kV, cathode emission current 100  $\mu$ A, energy of ionizing electrons 70 eV, ion source temperature 200°C; perfluoro-kerosene was used as reference; resolution  $M/\Delta M = 10\,000$ .

### REFERENCES

1. Kuznetsov, A.I. and Vladimirova, I.A., *Khim. Geterotsikl. Soedin.*, 1988, p. 1700.
2. Kuznetsov, A.I., Vladimirova, I.A., Basargin, E.B., *et al.*, *Khim. Geterotsikl. Soedin.*, 1990, p. 675.