

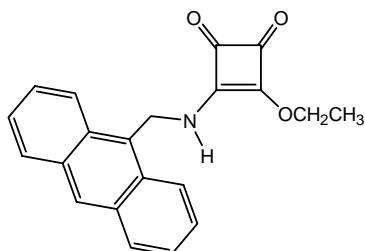
**Preparation, Solid State Characterization and Computational Study of  
a Crown Ether Attached to a Squaramide**

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Selected data for **1**:

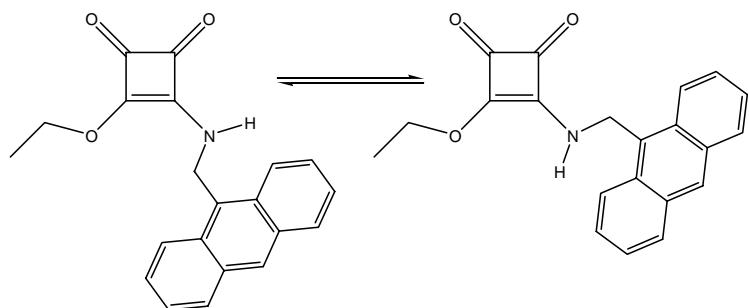
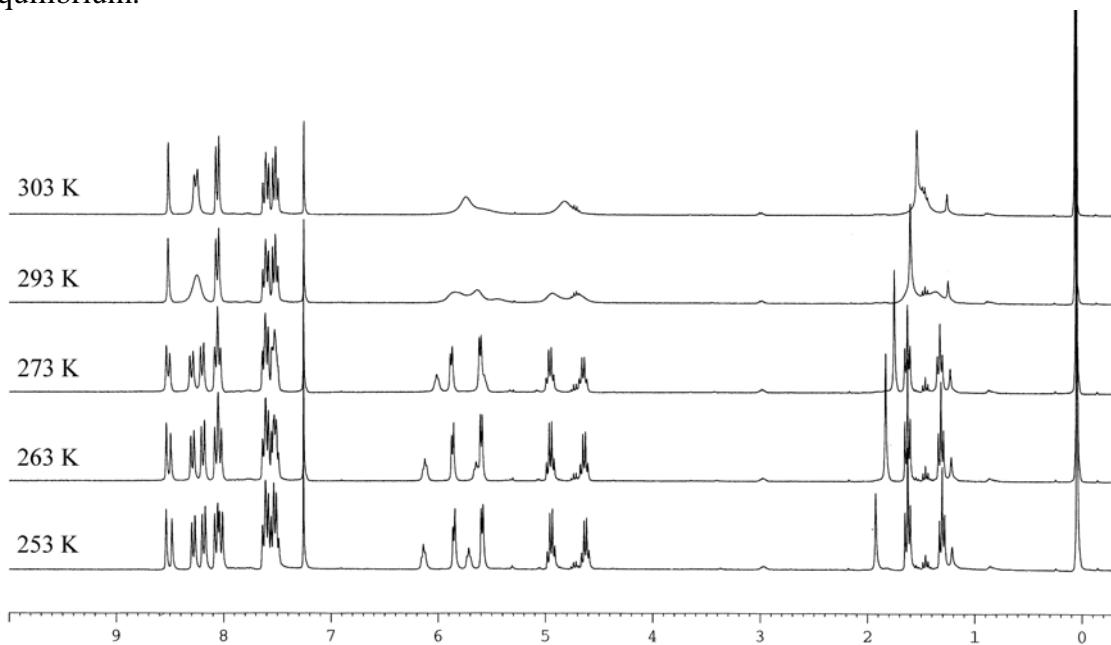
**Yield** 52%. **Anal.** Calc. for  $C_{32}H_{38}N_2O_8$ : C, 66.42; H, 6.62; N, 4.84. Found C, 66.22; H, 6.58; N, 4.70%. **NMR** ( $CDCl_3$ , 293 K):  $^1H$  (300 MHz),  $\delta$  = 8.42 (s, 1H), 8.38 (d,  $J$  = 8.9 Hz, 2H), 7.98 (d,  $J$  = 8.9 Hz, 2H), 7.56 (dd,  $J$  = 6.9 Hz, 7.7 Hz, 2H), 7.46 (dd,  $J$  = 7.7 Hz,  $J$  = 6.9 Hz, 2H), 7.16 (br s, 1H, NH), 6.64 (br s, 1H, NH), 5.99 (dd,  $J$  = 14.3 Hz, 6.3 Hz, 1H), 5.74 (dd,  $J$  = 14.3 Hz, 6.3 Hz, 1H), 3.9 (m, 2H), 3.66-3.33 (m, 23H) ppm.  $^{13}C$  (300 MHz),  $\delta$  = 184.08, 169.09, 167.72, 151.30, 132.24, 130.77, 129.79, 129.42, 128.76, 127.47, 126.00, 124.61, 77.91-69.41, 45.08, 40.96 ppm. **IR** (KBr): 3235, 2896, 1666, 1602, 1538, 1449, 1111, 742  $cm^{-1}$ . **HRMS** (ESI $^+$ ) m/z (%): 601.2 ( $M+Na^+$ , 100).

Selected data for the intermediate:



**Yield** 29%. **Anal.** Calc. for  $C_{21}H_{17}NO_3$ : C, 76.12; H, 5.17; N, 4.23. Found C, 76.64; H, 5.11; N, 4.05%. **NMR** ( $CDCl_3$ , 293 K):  $^1H$  (300 MHz),  $\delta$  = 8.51 (s, 1H), 8.23 (br s, 2H), 8.09 (d,  $J$  = 8 Hz, 2H), 7.63 (dd,  $J$  = 6.7 Hz, 7.8 Hz, 2H), 7.51 (dd,  $J$  = 7.8 Hz, 6.7 Hz, 2H), 5.89 (br s, 1H), 5.60 (br s, 1H), 5.40 (br s, 1H, NH), 4.94 (br s, 1H), 4.61 (br s, 1H), 1.45 (br s, 3H) ppm.  $^{13}C$  (300 MHz),  $\delta$  = 183.99, 178.02, 172.74, 132.23, 130.74, 130.28, 129.90, 128.07, 126.07, 123.55, 70.62, 41.58, 16.55 ppm. **IR** (KBr): 3251, 1698, 1642, 1531, 1379, 1322, 733  $cm^{-1}$ . **HRMS** (ESI $^+$ ) m/z (%): 685.23 ( $2M+Na^+$ , 100).

Several spectra of the intermediate at a range of temperatures (303 K-253 K) follow, the broad signals at room temperature are due two the existence of two rotamers in equilibrium.



$^1\text{H}$  NMR spectra of **1** and **1+AcONa** at 298K in  $\text{CDCl}_3$ :

