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Structures of 1,4,10,13-Tetrathia-7,16-diazacyclooctadecane, $C_{12}H_{26}N_2S_4$, and 7,16-Bis(4-chlorobenzoyl)-1,4,10,13-tetrathia-7,16-diazacyclooctadecane, $C_{26}H_{32}Cl_2N_2O_2S_4$

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Abstract. 1,4,10,13-Tetrathia-7,16-diazacyclooctadecane (I), $M_r = 326 \cdot 6$, monoclinic, $P2_1/c$, $a = 5 \cdot 200$ (2), $b = 14 \cdot 576$ (7), $c = 11 \cdot 203$ (3) Å, $\beta = 100 \cdot 95$ (2)°, $V = 833 \cdot 7$ Å^3, $Z = 2$, $D_x = 1 \cdot 301$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1 \cdot 5418$ Å, $\mu = 49 \cdot 0$ cm^{-1}, $F(000) = 352$, $T = 295$ K, final $R = 0 \cdot 051$ for 1017 observed reflections. 7,16-Bis(4-chlorobenzoyl)-1,4,10,13-tetrathia-7,16-diazacyclooctadecane (II), $M_r = 603 \cdot 7$, monoclinic, $P2_1/a$, $a = 11 \cdot 8146$ (4), $b = 21 \cdot 6043$ (8), $c = 11 \cdot 5440$ (4) Å, $\beta = 94 \cdot 255$ (6)°, $V = 2938 \cdot 4$ Å^3, $Z = 4$, $D_x = 1 \cdot 364$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1 \cdot 5418$ Å, $\mu = 48 \cdot 6$ cm^{-1}, $F(000) = 1264$, $T = 295$ K, final $R = 0 \cdot 063$ for 2764 observed reflections. A two-atom fragment of the macro-ring in (II) shows positional disorder. The crown-ring conformations in the two structures are significantly different. (I) incorporates a crystallographic inversion center, has no exo- or endodentate S atoms and no anti C—S bonds, while (II) has two exodentate S atoms and three anti C—S bonds.

Introduction. Macrocyclic polyethers ('crown' ethers) contain intramolecular cavities lined with molecular segments that are capable of binding cations. Ammon, Bhattacharjee, Shinkai & Honda (1984) reported the structure of a capped thia-crown, namely 2,2'-azobenzoyl-capped 1,4,10,13-tetrathia-7,16-diazacyclooctadecane (III), the first of the thia-crowns to exhibit the C—S bond in the anti conformation. The present investigation was undertaken to study both the effect of the azobenzene cap or bridge on the structure of the tetrathiadiaza-crown ring, and the effect of the hy-

bridization of the macrocyclic N atoms on the crown structure. Compound (II) contains sp^2-hybridized amide-type N atoms similar to that in the capped molecules, whereas the N atom in (I) is an sp^3-hybridized amino-type.

(I), R = H
(II), R = p-Cl-C₆H₄-C(=O)
(III), R - R = (O=)C-C₆H₄-N=N-C₆H₄-C(=O)
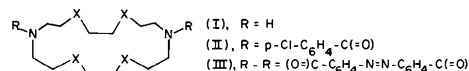
Experimental. Picker FACS-I diffractometer, graphite-monochromatized Cu radiation, cell parameters calculated by least squares from 12/16 reflections automatically centered at $\pm 2\theta$, intensity data collected to $2\theta_{max}$ of 126°, θ–2θ scan, 10 s backgrounds; four standard intensities measured at 100-reflection intervals showed no significant variations. (I) and (II) prepared by SS and YH (procedure unpublished). (I): crystal $0 \cdot 27 \times 0 \cdot 10 \times 0 \cdot 07$ mm, h,k,l range 0–6, 0–16, −13–13, 1643 data measured, 1334 unique excluding systematic absences, 1017 3σ above background; (II): $0 \cdot 47 \times 0 \cdot 13 \times 0 \cdot 10$ mm, 0–13, 0–24, −13–13, 5392 data measured, 4755 unique excluding systematic absences, 2764 3σ above background.

Both structures solved with MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). E map with highest combined figure of merit revealed all C, N and S atoms in (I); H atoms attached to C included in calculated positions with C—H $= 1 \cdot 05$ Å; H linked to N located from difference map [peak height $0 \cdot 46$ (13) e Å^{-3}]. Full-matrix least-squares

minimization of $\sum w(F_o - F_c)^2$, $w = 1/\sigma^2(F)$; C-, N- and S-atom parameters refined with anisotropic temperature factors, H-atom isotropic temperature factors refined; refinement included only those reflections for which $I_c > 3\sigma(I_o)$. Refinement of H coordinates and temperature factors led to some unreasonable positions and is not reported here. For the H linked to N, the refined position was close to the initial, difference-map location. Final $R = 0.051$, $R_w = 0.066$, $S = 2.26$. Av. and max. shift/error 0.15 and 0.16. Max. and min. $\Delta\rho$ in final difference map 0.50 and −0.28 e Å^{-3}.

Best E map for (II) revealed all C, N, O, S and Cl atoms except two C atoms in the crown ring and three in the benzene rings; these positions were approximated. Block-diagonal least-squares structure refinement with anisotropic temperature factors led to $R = 0.089$; temperature factors on S(4) and C(5) were quite large [e.g. S(4), $U_{11} = 0.36$ Å^2; C(5), $U_{33} = 0.52$ Å^2]. Difference maps suggested two sites for each atom, and a four-atom model [S(4), C(5), S(4′), C(5′)] with occupancies of p and p' $(1 - p)$ was used in further structure refinement. Isotropic temperature factors of H atoms, initially placed at calculated positions with C−H = 1.05 Å, were refined in the latter cycles. Final $R = 0.063$, $R_w = 0.061$, $S = 2.97$. Av. and max. shift/error 0.13 and 0.96 [z coordinate of S(4)]. Max. and min. $\Delta\rho$ in final difference map 0.43 and −0.75 e Å^{-3}.

Scattering factors: C, Cl, N, O and S (Cromer & Mann, 1968); H (Stewart, Davidson & Simpson, 1965). *XRAY*76 programs (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976); Univac 1100/82 computer.

Discussion. The atom parameters are listed in Tables 1 and 2.* The positional disorder/high-temperature-factor feature observed in (II) appears to be a common feature in crystal structures of thia-crowns, having been observed in three [(II), (III) and hexathia-18-crown-6, Hartman, Wolf, Foxman & Cooper, 1983] of the four reported structures.

Bond lengths, angles and torsion angles are given in Tables 3 and 4. (I) has crystallographic inversion symmetry; the average C−S and C−C lengths are 1.811 (5) and 1.496 (6) Å respectively. Disorder in (II) leads to some distorted distances; the population-averaged C−S and C−C lengths in the crown ring are 1.792 (7) and 1.502 (9) Å. Both sets of values are close to those reported for similar compounds (e.g. complexes of neutral organic guest molecules with 18-crown-6 and diaza-18-crown-6: Watson, Galloy,

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39579 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Grossie, Vögtle & Müller, 1984; azoarene-capped analogs of 1,4,10,13-tetraoxa-7,16-diaza and 1,4,10,13-tetrathia-7,16-diazacyclooctadecane: Ammon *et al.*, 1984; 18-crown-6: Maverick, Seiler, Schweizer & Dunitz, 1980; complexes of 18-crown-6 with alkali thiocyanates: Dunitz, Dobler, Seiler & Phizackerley, 1974).

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *in* (I)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

The e.s.d. of the last significant digit is given in parentheses.

	x	y	z	U_{eq}
C(2)	0.5208 (8)	0.2818 (3)	0.6043 (3)	0.062 (8)
C(3)	0.2601 (9)	0.2742 (3)	0.5183 (4)	0.057 (7)
C(5)	0.299 (1)	0.4383 (4)	0.3816 (4)	0.093 (6)
C(6)	0.199 (1)	0.4227 (4)	0.2499 (4)	0.079 (4)
C(8)	0.753 (1)	0.4143 (3)	0.8884 (4)	0.063 (5)
C(9)	0.758 (1)	0.3471 (3)	0.7886 (4)	0.065 (8)
N(1)	0.4987 (7)	0.3273 (2)	0.7175 (5)	0.057 (7)
S(4)	0.0992 (2)	0.38212 (7)	0.47655 (8)	0.080 (6)
S(7)	0.5983 (2)	0.52353 (7)	0.84586 (8)	0.080 (6)

Table 2. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2) *in* (II)

pp is the population parameter.

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

The e.s.d. of the last significant digit is given in parentheses.

	x	y	z	U_{eq}	pp
C(2)	−0.6222 (5)	0.2216 (2)	1.0470 (5)	0.10 (2)	
C(3)	−0.6666 (7)	0.2015 (3)	0.9286 (5)	0.14 (2)	
C(5)	−0.5557 (8)	0.1446 (5)	0.781 (1)	0.17 (1)	0.643 (7)
C(5′)	−0.587 (1)	0.1940 (8)	0.699 (1)	0.14 (2)	0.357
C(6)	−0.5046 (5)	0.1319 (3)	0.6751 (5)	0.11 (2)	
C(8)	−0.2859 (5)	0.1471 (2)	0.7696 (4)	0.11 (2)	
C(9)	−0.2579 (4)	0.0771 (2)	0.7887 (4)	0.08 (2)	
C(11)	−0.2660 (5)	0.0716 (2)	1.0023 (4)	0.12 (3)	
C(12)	−0.2958 (5)	0.0092 (2)	1.0461 (4)	0.09 (3)	
C(14)	−0.3483 (4)	0.0615 (2)	1.2570 (4)	0.07 (3)	
C(15)	−0.4096 (5)	0.0578 (2)	1.3638 (4)	0.11 (3)	
C(17)	−0.5849 (5)	0.1419 (3)	1.2868 (5)	0.09 (3)	
C(18)	−0.6706 (4)	0.1387 (2)	1.1876 (4)	0.08 (3)	
C(19)	−0.7822 (4)	0.2368 (2)	1.1653 (4)	0.07 (2)	
C(20)	−0.8628 (4)	0.2131 (2)	1.2483 (4)	0.07 (2)	
C(21)	−0.8616 (4)	0.2349 (2)	1.3607 (4)	0.05 (3)	
C(22)	−0.9422 (5)	0.2164 (2)	1.4326 (4)	0.07 (3)	
C(23)	−1.0254 (4)	0.1757 (2)	1.3908 (4)	0.08 (3)	
C(24)	−1.0298 (4)	0.1535 (2)	1.2789 (4)	0.06 (3)	
C(25)	−0.9470 (4)	0.1723 (2)	1.2081 (4)	0.05 (2)	
C(26)	−0.0840 (4)	0.0715 (2)	0.9208 (3)	0.06 (2)	
C(27)	−0.0144 (4)	0.0692 (2)	0.8167 (3)	0.06 (2)	
C(28)	−0.0117 (4)	0.0168 (2)	0.7473 (3)	0.08 (2)	
C(29)	0.0558 (4)	0.0154 (2)	0.6538 (4)	0.09 (2)	
C(30)	0.1196 (4)	0.0666 (2)	0.6309 (4)	0.12 (2)	
C(31)	0.1176 (4)	0.1186 (2)	0.6998 (4)	0.08 (2)	
C(32)	0.0513 (4)	0.1190 (2)	0.7916 (4)	0.07 (2)	
Cl(1)	−1.1288 (1)	0.15168 (7)	1.4816 (1)	0.14 (3)	
Cl(2)	0.2036 (1)	0.06426 (8)	0.5137 (1)	0.19 (2)	
N(1)	−0.6961 (3)	0.2005 (2)	1.1353 (3)	0.09 (2)	
N(10)	−0.1974 (3)	0.0677 (2)	0.9025 (3)	0.08 (2)	
O(1)	−0.7961 (3)	0.2884 (1)	1.1232 (4)	0.18 (2)	
O(2)	−0.0355 (3)	0.0757 (2)	1.0179 (2)	0.07 (2)	
S(4)	−0.6035 (3)	0.2190 (1)	0.7998 (2)	0.157 (2)	0.643 (7)
S(4′)	−0.5313 (3)	0.2239 (2)	0.8497 (3)	0.101 (4)	0.357
S(7)	−0.3720 (1)	0.16342 (8)	0.6450 (1)	0.19 (2)	
S(13)	−0.4106 (1)	0.01349 (7)	1.1425 (1)	0.09 (3)	
S(16)	−0.5601 (1)	0.06489 (7)	1.3481 (1)	0.13 (3)	

Table 3. *Bond lengths* (Å), *angles* (°) *and torsion angles* (°) *with e.s.d.'s in* (I)

N(1)—C(2)	1·455 (5)	C(5)—C(6)	1·486 (6)
N(1)—C(9')	1·457 (6)	C(6)—S(7)	1·818 (6)
C(2)—C(3)	1·510 (6)	S(7)—C(8)	1·806 (5)
C(3)—S(4)	1·801 (4)	C(8)—C(9)	1·491 (7)
S(4)—C(5)	1·819 (6)		
C(9')—N(1)—C(2)	110·4 (4)	C(5)—C(6)—S(7)	112·7 (4)
N(1)—C(2)—C(3)	112·3 (4)	C(6)—S(7)—C(8)	104·9 (3)
C(2)—C(3)—S(4)	114·7 (3)	S(7)—C(8)—C(9)	116·8 (3)
C(3)—S(4)—C(5)	104·8 (3)	C(8)—C(9)—N(1')	113·5 (4)
S(4)—C(5)—C(6)	112·3 (4)		
C(9')—N(1)—C(2)—C(3)	−170·4 (3)	C(5)—C(6)—S(7)—C(8)	−95·4 (4)
N(1)—C(2)—C(3)—S(4)	58·4 (4)	C(6)—S(7)—C(8)—C(9)	65·2 (2)
C(2)—C(3)—S(4)—C(5)	69·3 (4)	S(7)—C(8)—C(9)—N(1')	−55·3 (6)
C(3)—S(4)—C(5)—C(6)	95·5 (4)	C(8)—C(9)—N(1')—C(2')	164·9 (4)
S(4)—C(5)—C(6)—S(7)	−178·7 (3)		

Table 4. *Bond lengths* (Å), *angles* (°) *and torsion angles* (°) *with e.s.d.'s in* (II)

N(1)—C(2)	1·463 (7)	S(16)—C(17)	1·823 (6)
N(1)—C(18)	1·487 (6)	C(17)—C(18)	1·473 (7)
N(1)—C(19)	1·351 (6)	C(19)—O(1)	1·221 (6)
C(2)—C(3)	1·492 (6)	C(19)—C(20)	1·491 (7)
C(3)—S(4)	1·754 (7)	C(20)—C(21)	1·380 (6)
C(3)—S(4')	1·959 (9)	C(20)—C(25)	1·384 (6)
S(4)—C(5)	1·723 (11)	C(21)—C(22)	1·368 (7)
S(4')—C(5')	1·929 (13)	C(22)—C(23)	1·379 (7)
C(5)—C(6)	1·426 (13)	C(23)—Cl(1)	1·746 (5)
C(5')—C(6)	1·693 (17)	C(23)—C(24)	1·375 (6)
C(6)—S(7)	1·765 (6)	C(24)—C(25)	1·381 (7)
S(7)—C(8)	1·735 (5)	C(26)—O(2)	1·224 (5)
C(8)—C(9)	1·560 (7)	C(26)—C(27)	1·506 (6)
C(9)—N(10)	1·462 (5)	C(27)—C(28)	1·389 (6)
N(10)—C(11)	1·459 (6)	C(27)—C(32)	1·370 (7)
N(10)—C(26)	1·343 (6)	C(28)—C(29)	1·389 (7)
C(11)—C(12)	1·492 (7)	C(29)—C(30)	1·376 (7)
C(12)—S(13)	1·820 (6)	C(30)—Cl(2)	1·737 (5)
S(13)—C(14)	1·794 (5)	C(30)—C(31)	1·379 (7)
C(14)—C(15)	1·478 (7)	C(31)—C(32)	1·364 (7)
C(15)—S(16)	1·781 (6)		
C(2)—N(1)—C(18)	116·7 (4)	N(1)—C(19)—O(1)	120·6 (5)
C(2)—N(1)—C(19)	119·8 (4)	N(1)—C(19)—C(20)	119·6 (4)
C(18)—N(1)—C(19)	123·5 (4)	O(1)—C(19)—C(20)	119·7 (4)
N(1)—C(2)—C(3)	111·1 (5)	C(19)—C(20)—C(21)	121·6 (4)
C(2)—C(3)—S(4)	124·8 (5)	C(19)—C(20)—C(25)	118·8 (4)
C(2)—C(3)—S(4')	96·3 (5)	C(21)—C(20)—C(25)	119·3 (4)
C(3)—S(4)—C(5)	94·1 (5)	C(20)—C(21)—C(22)	120·7 (4)
C(3)—S(4')—C(5')	95·7 (5)	C(21)—C(22)—C(23)	119·0 (4)
S(4)—C(5)—C(6)	116·9 (8)	C(22)—C(23)—Cl(1)	119·4 (4)
S(4')—C(5')—C(6)	104·4 (7)	C(22)—C(23)—C(24)	121·8 (4)
C(5)—C(6)—S(7)	122·2 (5)	Cl(1)—C(23)—C(24)	118·8 (4)
C(5')—C(6)—S(7)	105·0 (6)	C(23)—C(24)—C(25)	118·4 (4)
C(6)—S(7)—C(8)	103·4 (3)	C(24)—C(25)—C(20)	120·8 (4)
S(7)—C(8)—C(9)	114·8 (3)	N(10)—C(26)—O(2)	122·9 (4)
C(8)—C(9)—N(10)	110·3 (4)	N(10)—C(26)—C(27)	118·0 (3)
C(9)—N(10)—C(11)	116·0 (4)	O(2)—C(26)—C(27)	119·1 (4)
C(9)—N(10)—C(26)	123·3 (4)	C(26)—C(27)—C(28)	121·6 (4)
C(11)—N(10)—C(26)	118·5 (3)	C(26)—C(27)—C(32)	119·6 (4)
N(10)—C(11)—C(12)	111·9 (4)	C(28)—C(27)—C(32)	118·7 (4)
C(11)—C(12)—S(13)	111·5 (4)	C(27)—C(28)—C(29)	120·4 (4)
C(12)—S(13)—C(14)	101·3 (2)	C(28)—C(29)—C(30)	119·0 (4)
S(13)—C(14)—C(15)	112·6 (4)	C(29)—C(30)—Cl(2)	118·6 (4)
C(14)—C(15)—S(16)	117·2 (4)	C(29)—C(30)—C(31)	120·8 (4)
C(15)—S(16)—C(17)	104·4 (3)	Cl(2)—C(30)—C(31)	120·6 (4)
S(16)—C(17)—C(18)	109·9 (4)	C(30)—C(31)—C(32)	119·2 (5)
C(17)—C(18)—N(1)	112·4 (4)	C(31)—C(32)—C(27)	121·8 (4)
C(18)—N(1)—C(2)—C(3)	89·7 (5)	C(9)—N(10)—C(11)—C(12)	100·7 (5)
N(1)—C(2)—C(3)—S(4)	178·7 (4)	N(10)—C(11)—C(12)—S(13)	−164·5 (3)
C(2)—C(3)—S(4)—C(5)	107·9 (6)	C(11)—C(12)—S(13)—C(14)	−61·2 (4)
C(3)—S(4)—C(5)—C(6)	173·8 (7)	C(12)—S(13)—C(14)—C(15)	−163·0 (4)
S(4)—C(5)—C(6)—S(7)	69·0 (8)	S(13)—C(14)—C(15)—S(16)	−47·6 (5)
C(5)—C(6)—S(7)	49·6 (7)	C(14)—C(15)—S(16)—C(17)	−61·5 (5)
C(6)—S(7)—C(8)—C(9)	69·0 (4)	C(15)—S(16)—C(17)—C(18)	132·5 (4)
S(7)—C(8)—C(9)—N(10)	−172·9 (3)	S(16)—C(17)—C(18)—N(1)	178·9 (3)
C(8)—C(9)—N(10)—C(11)	74·9 (5)	C(17)—C(18)—N(1)—C(2)	86·1 (5)

ORTEP drawings (Johnson, 1976) of (I) and (II) are shown in Figs. 1 and 2. The macrocycles have different shapes: in (I), the ring resembles a figure 8 with C(5) and its inversion mate at the waist, whereas the shape of (II) is more rectangular. In the capped thia-crown (III), the ring is oval-shaped with the N atoms at the far ends of the oval. The N···N transannular distances in (I)–(III) are 6·10 (1), 7·25 (1) and 7·83 (1) Å, respectively. It is now clear that the azobenzoyl cap in (III) is the factor responsible for the stretched oval.

Bond lengths and angles in the chlorobenzoyl segments of the molecule are normal. The six atoms in each amide moiety, C—C(=O)—N—C₂, show average deviations of 0·031 and 0·061 Å from their least-squares planes. The benzene rings are twisted by 47·5 and 39·9° from their respective amide planes.

The conformations of the crown rings can be described by the following terminology: 'g^+' and 'g^-' indicate *gauche* conformations with 0 to +120 and 0 to −120° torsion angles; 'a' indicates the antiperiplanar conformation with an angle of ±120 to 180° (Dale, 1980); an underline, such as '$\underline{a}$' or '$\underline{g}$' refers to the C—N conformation. With the use of these definitions, the
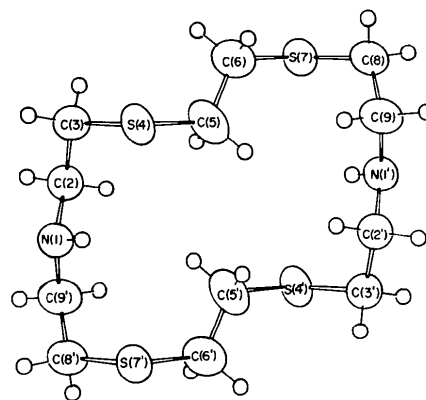

Fig. 1. An *ORTEP* drawing of (I) normal to the crown-ring least-squares plane with C,N and S atoms depicted as 50% probability boundary ellipses and H atoms as 0·1 Å radius circles.
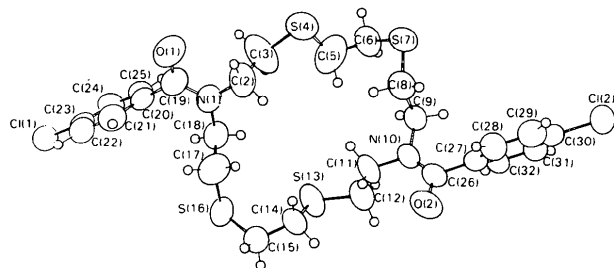

Fig. 2. An *ORTEP* drawing of (II) normal to the crown-ring least-squares plane with C,Cl,N,O and S atoms depicted as 50% probability boundary ellipses and H atoms as 0·1 Å radius circles. S(4') and C(5') are not shown.

conformations of (I)–(III) are $(\underline{a}g^+g^+, g^+ag^-, g^+g^-\underline{a})_2$, $(g^+ag^+, ag^+g^+, g^+ag^+)(g^+ag^-, ag^-g^-, aag^+)$ and $(\underline{g}^+g^+a, aag^+, aag^-)(\underline{a}g^-g^-, ag^-g^-, g^+a\underline{g}^+)$ (the g/a designations on either side of the commas give the C—S bond conformations). In an earlier paper (Ammon *et al.*, 1984) (III) had been reported to have four of eight *anti* C—S conformations, and we find three of eight examples of the *anti* conformation in (II). A major factor for the different ring shapes in (I) and (II) undoubtedly is the hybridization at N which is translated into different C—N conformations in the molecules. In (I), four of four C—N bond conformations are *anti*, whereas four of four in (II) and three of four in (III) are *gauche*. Two of the S atoms in (II) adopt the exodentate (point away from the ring center) position, whereas the other two S atoms as well as all four S atoms in (I) are neither exo- nor endodentate (*i.e.* the C—S—C triangle is more or less perpendicular to the macrocycle plane).

There are no short intermolecular contacts in either (I) or (II). This seemed a bit unusual in the case of (I) since the N—H groups could function as both hydrogen-bond donors and acceptors. Moreover, the position of the amino H atom (see Fig. 1), which points toward the center of the macrocycle, was difficult to rationalize in the absence of an intermolecular effect which favored the inside over the outside position. The explanation for the inside location, however, can be obtained by considering an intramolecular interaction between the amino H and its nearest two S atom neighbors, S(4) and S(7'). The observed H⋯S(4) and H⋯S(7') distances of 2·7 (1) and 2·8 (1) Å respectively [2·6 (1) and 2·7 (1) Å with the N—H bond stretched to 1·0 Å] are close to the van der Waals distance (2·85 Å) for this interaction (Pauling, 1960).

We further note that if the H atom pointed outside, the N atom's nonbonded electron pair would point inside toward the S atoms' nonbonded electrons, resulting in an unfavorable interaction. We cannot, of course, determine from the structural data which of these two effects is the more important.

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α- and β-Epoxides from Monocrotaline, a Pyrrolizidine Alkaloid: 1,2-Dihydro-1α,2α-epoxymonocrotaline, $C_{16}H_{23}NO_7$, and 1,2-Dihydro-1β,2β-epoxymonocrotaline Monohydrate, $C_{16}H_{23}NO_7.H_2O$

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Abstract. 1,2-Dihydro-1α,2α-epoxymonocrotaline: $M_r = 341\cdot4$, orthorhombic, $P2_12_12_1$, $a = 10\cdot140$ (1), $b = 11\cdot691$ (1), $c = 13\cdot681$ (1) Å, $U = 1621\cdot8$ (2) Å³, $Z = 4$, $D_x = 1\cdot398$ Mg m⁻³, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0\cdot83$ mm⁻¹, $F(000) = 728$, $T = 290$ (1) K. 1,2-Dihydro-1β,2β-epoxymonocrotaline monohydrate: $M_r = 359\cdot4$, orthorhombic, $P2_12_12_1$, $a = 9\cdot621$ (1), $b = 12\cdot416$ (1), $c = 14\cdot174$ (1) Å, $U = 1693\cdot1$ (2) Å³, Z