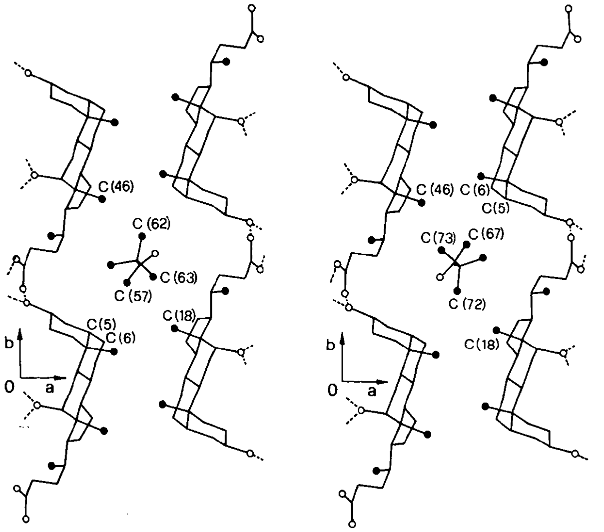

Fig. 3. DCAPNC channel viewed along **c**. Broken lines, filled and open circles represent hydrogen bonds, methyl groups and oxygen atoms, respectively. The two orientations of PNC in the DCA channel are shown. The atoms that interact as summarized in Table 4 are indicated.

The PNC molecule occupies approximately the same position of NBD and QDC in DCANBD and DCA-QDC crystals. The coordinates of the centers of gravity of PNC, considering only the non-H atoms, are (0·248, 0·499, 0·304) and (0·247, 0·503, 0·305), respectively, for the first and second PNC molecules quoted in Table 1, whereas those of NBD and QDC are (0·253, 0·505, 0·331) and (0·248, 0·505, 0·331) respectively. Therefore, since the host structures are nearly equal in DCANBD, DCAQDC and DCAPNC it should be possible to include NBD together with PNC in the channels of DCA. These guest molecules could exchange their positions in the same channel without straining the lattice, so that the lattice-controlled photoinduced reaction NBD→QDC could easily occur.

Table 3. O···O *distances* (Å) *of the hydrogen bonds in a bilayer*

E.s.d.'s are in the range 0·007–0·008 Å.

$O(25)\cdots O(27^{i})$	2·73	$O(26)\cdots O(53^{iii})$	2·72
$O(25)\cdots O(54^{ii})$	2·69	$O(53)\cdots O(55^{i})$	2·78
$O(26)\cdots O(28^{ii})$	2·66	$O(54)\cdots O(56^{iv})$	2·66

Symmetry code: (i) $x, 1+y, z$; (ii) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (iii) $-x, -\frac{1}{2}+y, \frac{1}{2}-z$; (iv) $-x, \frac{1}{2}+y, \frac{3}{2}-z$.

Table 4. *Shortest contacts* (Å) *between PNC and DCA atoms*

E.s.d.'s are in the range 0·013–0·015 Å for methyl–methyl distances.

The numbering of DCA hydrogens is that of the carrier carbons.

$C(62)\cdots C(46^{i})$	3·82	$C(72)\cdots C(18^{ii})$	3·88
$C(63)\cdots C(18^{ii})$	3·71	$C(73)\cdots C(46^{i})$	3·61
$C(57)\cdots H(5)$	3·12	$C(67)\cdots H(33^{iii})$	3·00
$C(57)\cdots Ha(6)$	3·07	$C(67)\cdots Ha(34^{iii})$	2·98

Symmetry code: (i) $x, 1+y, z$; (ii) $\frac{1}{2}-x, -y, \frac{1}{2}+z$; (iii) $\frac{1}{2}-x, 1-y, \frac{1}{2}-z$.

One of the authors (EG) acknowledges financial support from the Consiglio Nazionale delle Ricerche. Drawings by Mr M. Viola.

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Acta Cryst. (1985). C**41**, 232–234

(1*RS*)-15-Thiabicyclo[10.7.0]nonadec-1(12)-ene 15,15-Dioxide, $C_{18}H_{32}O_2S$

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(*Received* 31 *May* 1984; *accepted* 27 *September* 1984)

Abstract. $M_r = 312{\cdot}0$, triclinic, $P\bar{1}$, $a = 8{\cdot}618\ (4)$, $b = 12{\cdot}403\ (3)$, $c = 16{\cdot}792\ (6)$ Å, $\alpha = 88{\cdot}24\ (3)$, $\beta = 79{\cdot}82\ (3)$, $\gamma = 87{\cdot}29\ (3)°$, $V = 1764\ (1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}18$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}8$ cm^{-1}, $F(000) = 688$, $T = 295$ K, final $R = 0{\cdot}067$ over 3033 reflections. Each molecule consists of a 5-thiacyclononene ring (*A*) *trans* fused to a cyclododecene ring (*B*). The flexibility of ring *B* results in

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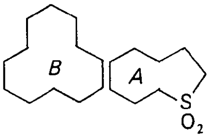
an apparent disorder such that three distinct conformers can be resolved in the unit cell. Molecular mechanics calculations support the near degeneracy of these structures. The conformation of ring *A* is similar in all three model structures, but sulfone oxygen atoms prevent the cyclononene ring from adopting the expected pseudo-twofold symmetry.

Introduction. The class of organic compounds known as 'betweenanenes', bis(*trans*-polymethylene)ethylenes (Marshall & Lewellyn, 1977; Marshall & Chung, 1979), offer unique challenges to synthetic, physical and structural chemists alike. Fava and his co-workers have focused on S-heterocyclic analogs (Calderoni, Cerè, Pollicino, Sandri, Fava & Guerra, 1980); the bicyclosulfone (I) was synthesized (Cerè, Paolucci, Pollicino, Sandri & Fava, 1981) and supplied by Professor Fava.



(I)

Experimental. Colorless crystal, m.p. 374–375 K, 0·48 × 0·84 × 0·16 mm, glued to glass fiber; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; preliminary survey revealed triclinic crystal system; lattice parameters derived from least-squares refinement of 25 well centered reflections ($25 > 2\theta > 20°$); 4097 intensity data measured, $\omega/2\theta$ method in four octants: $2 \leq 2\theta \leq 46°$, $0 \leq h \leq 9$, $-13 \leq k \leq 13$, $-18 \leq l \leq 18$; no systematic variation in three periodically remeasured intensities; 3033 unique observed reflections [$I > 2{\cdot}5\sigma(I)$]. All C, O and S atoms in two independent molecules located by combined heavy-atom and direct methods; H atoms placed in calculated positions and constrained to 'ride' during full-matrix weighted least squares. Owing to disorder two constrained groups of H-atom isotropic temperature factors were refined; all non-hydrogen atoms refined anisotropically; $\sum w(\Delta F)^2$ minimized; $w = [\sigma^2(|F_o|) + 0{\cdot}012|F_o|^2]^{-1}$. Final model with 390 variables yielded $R = 0{\cdot}067$, $wR = 0{\cdot}071$. In final cycle, $(\Delta/\sigma)_{\max} = 0{\cdot}10$, max. residual electron density 0·4 e Å^{-3}. Non-H scattering factors from Cromer & Mann (1968), those for H from Stewart, Davidson & Simpson (1965). Computer programs used: *SHELX* (Sheldrick, 1976), *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), *MM*2 (Allinger, 1977) and the *PROPHET* computing system (Raub, 1974).

Table 1. *Atomic positional parameters* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å$^2 \times 10^3$)

	x	y	z	U_{eq}†
S(1)	7135 (2)	9021 (1)	4560 (1)	67 (1)
O(1)	6885 (6)	8599 (4)	5380 (3)	101 (2)
O(2)	5877 (5)	8978 (3)	4111 (3)	85 (2)
C(1)	7626 (7)	10411 (4)	4615 (4)	71 (2)
C(2)	7229 (7)	11243 (4)	3994 (4)	74 (2)
C(3)	8634 (8)	11844 (4)	3535 (4)	81 (2)
C(4)	9454 (8)	11349 (4)	2740 (4)	76 (2)
C(5)	9901 (6)	10173 (4)	2883 (3)	58 (2)
C(6)	8963 (6)	9402 (4)	2756 (3)	58 (2)
C(7)	9123 (8)	8279 (4)	3141 (4)	72 (2)
C(8)	8817 (8)	8256 (4)	4067 (4)	78 (2)
C(9)	11386 (6)	9966 (5)	3232 (4)	74 (2)
C(10)	12874 (9)	10412 (7)	2723 (5)	112 (4)
C(11)	13105 (9)	10125 (6)	1822 (5)	104 (3)
C(12)	13326 (11)	8964 (8)	1674 (6)	127 (4)
C(13)	13258 (16)	8603 (9)	767 (7)	166 (6)
C(14)	11740 (21)	8823 (9)	489 (6)	180 (8)
C(15)	10303 (19)	8150 (8)	1040 (7)	158 (6)
C(16)	8762 (28)	8647 (11)	991 (10)	257 (13)
C(17)	8068 (13)	9514 (12)	1401 (6)	158 (6)
C(18)	7643 (7)	9550 (5)	2274 (4)	76 (2)
S(1′)	7455 (2)	5667 (1)	5295 (1)	73 (1)
O(1′)	7183 (7)	5577 (4)	4464 (3)	111 (2)
O(2′)	8848 (5)	6260 (3)	5447 (3)	82 (2)
C(1′)	5725 (7)	6267 (5)	5834 (4)	78 (2)
C(2′)	5846 (8)	6929 (4)	6608 (4)	80 (2)
C(3′)	4650 (7)	6578 (5)	7343 (4)	80 (2)
C(4′)	5291 (7)	5830 (4)	7900 (4)	69 (2)
C(5′)	6001 (6)	4868 (4)	7410 (3)	51 (2)
C(6′)	7536 (6)	4858 (3)	7087 (3)	48 (2)
C(7′)	8137 (7)	4109 (4)	6380 (3)	62 (2)
C(8′)	7479 (7)	4308 (4)	5608 (3)	69 (2)
C(9′)	4818 (6)	3968 (4)	7245 (4)	66 (2)
C(10′*A*)	3901 (15)	3430 (12)	8009 (8)	78 (5)
C(10′*B*)	4840 (21)	2928 (11)	7747 (10)	92 (6)
C(11′)	4611 (17)	2914 (8)	8562 (7)	170 (7)
C(12′)	5365 (11)	3612 (7)	9162 (5)	111 (3)
C(13′)	6672 (11)	3107 (7)	9436 (6)	127 (4)
C(14′)	7867 (12)	3839 (8)	9689 (6)	132 (4)
C(15′)	8631 (10)	4695 (6)	9090 (4)	102 (3)
C(16′)	9471 (8)	4200 (6)	8307 (5)	92 (3)
C(17′)	10087 (7)	5014 (5)	7650 (4)	82 (2)
C(18′)	8793 (6)	5601 (4)	7332 (3)	61 (2)

† $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a^*_i a^*_j \mathbf{a}_i.\mathbf{a}_j$.

Discussion. Atomic coordinates of all non-H atoms in molecules 1 and 2 are shown in Table 1, and intraannular torsion angles are shown in Table 2.* The *trans*-thiacyclononene rings (1*A* and 2*A*) have nearly identical conformations: over the nine pairs of torsion angles, $\Delta\omega$(r.m.s.) = 2·7 (3)°, and total ring-puckering parameters (Cremer & Pople, 1975) Q = 1·09 (1) (1*A*) and 1·10 (1) (2*A*) Å. This conformation is not one of low energy discussed by Dale (1978) or by Burkert & Allinger (1982). There are highly distorted mirror elements through C(2) [$\overline{\Delta C_s} = 62\ (1)°$] and C(3) [$\overline{\Delta C_s} = 43\ (1)°$], and a twofold axis through C(7) [$\overline{\Delta C_2} = 53\ (1)°$] (Duax & Norton, 1975). *MM*2 force-field calculations (Allinger, 1977) resulted in minimum-energy conformers for 1*A* and 2*A* with $\overline{\Delta\omega}$(r.m.s., obs–calc) = 9·0 (5)° and significant van der Waals interactions involving the oxygen atoms and several extra-annular hydrogen atoms.

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* Lists of structure factors, anisotropic thermal parameters, interatomic distances and angles and two diagrams have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39786 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Intraannular torsion angles* (°)

Thiacyclononene ring	1*A*(obs)	1*A*(calc)	2*A*(obs)	2*A*(calc)
S(1)C(1)C(2)C(3)	−122·9 (5)	−105·9	−126·6 (5)	−114·4
C(1)C(2)C(3)C(4)	91·8 (6)	97·1	97·7 (6)	99·4
C(2)C(3)C(4)C(5)	−53·1 (7)	−59·1	−54·1 (6)	−52·5
C(3)C(4)C(5)C(6)	95·2 (6)	97·9	93·1 (6)	92·4
C(4)C(5)C(6)C(7)	−160·6 (5)	−162·7	−158·5 (5)	−163·4
C(5)C(6)C(7)C(8)	61·6 (7)	62·3	62·0 (6)	61·0
C(6)C(7)C(8)S(1)	45·1 (6)	46·1	46·0 (6)	47·0
C(7)C(8)S(1)C(1)	−83·3 (5)	−80·6	−83·8 (5)	−76·2
C(8)S(1)C(1)C(2)	97·2 (5)	76·2	95·1 (5)	74·6

Cyclododecene ring	1*B*(obs)	1*B*(calc)	2*B*(obs)	2*B*(calc)	2*B*′(obs)	2*B*′(calc)
C(18)C(6)C(5)C(9)	−168·4 (5)	−173·3	−168·8 (5)	−173·5	−168·8 (5)	−173·4
C(6)C(5)C(9)C(10)	125·8 (6)	126·8	123·4 (8)	122·9	79·0 (11)	76·5
C(5)C(9)C(10)C(11)	−48·6 (8)	−45·3	−59·2 (14)	−60·8	57·1 (16)	74·0
C(9)C(10)C(11)C(12)	−63·4 (9)	−66·2	77·9 (15)	80·2	−49·6 (22)	- 76·1
C(10)C(11)C(12)C(13)	169·4 (8)	167·7	−152·1 (11)	−165·5	−97·7 (16)	−70·8
C(11)C(12)C(13)C(14)	−63·1 (13)	−63·7	152·9 (9)	158·9	152·9 (9)	164·1
C(12)C(13)C(14)C(15)	−62·8 (14)	−64·5	−53·9 (12)	−53·8	−53·9 (12)	−64·6
C(13)C(14)C(15)C(16)	158·0 (12)	165·3	−60·4 (10)	−51·5	−60·4 (10)	−63·1
C(14)C(15)C(16)C(17)	−80·0 (20)	−69·8	173·1 (6)	171·6	173·1 (6)	170·7
C(15)C(16)C(17)C(18)	−62·5 (23)	−77·0	−65·6 (8)	−71·1	−65·6 (8)	−68·7
C(16)C(17)C(18)C(6)	61·6 (16)	72·3	−50·7 (7)	−46·4	−50·7 (7)	−41·6
C(17)C(18)C(6)C(5)	81·0 (9)	75·6	120·0 (6)	120·9	120·0 (6)	121·0

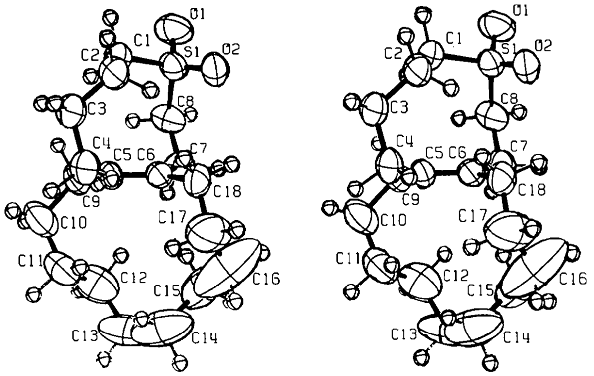

Fig. 1. Stereodiagram (50% ellipsoids) of conformation 1.

The flexible *trans*-cyclododecene rings show disorder in the C(10) and, to a lesser extent, the C(16) regions; the C(10′) disorder was resolved into two atomic positions. These observations may be interpreted as resulting from the superposition of at least two distinct conformers on molecule 2; all have identical nine-membered rings but three different 12-membered rings (1*B*, 2*B* and 2*B*′, Fig. 1).

Ring 1*B* shows highly distorted mirror symmetry [next to C(9) and C(15), $\Delta C_s = 52\ (1)$°] and a total puckering parameter $Q = 1{\cdot}55\ (2)$ Å. Ring 2*B* is of less distorted twofold symmetry [next to C(9′) and C(15′), $\Delta C_2 = 26\ (1)$°] and is less puckered [$Q = 1{\cdot}40\ (2)$ Å]. Ring 2*B*′ has essentially the same conformation as 1*B* but is more puckered [$Q = 1{\cdot}63\ (2)$ Å]; 1*B* and 2*B*′ are related by a pseudorotation such that the 'mirror plane' [$\Delta C_s = 53\ (1)$°] passes next to C(11′) and C(17′). Atoms C(6), C(5), C(9), …, C(18) of ring 1*B* are transformed into C(5′), C(6′), C(18′), …, C(9′) of ring 2*B*′, and comparison of the twelve pairs of equivalent torsion angles yields $\Delta\omega$(r.m.s.) = 8·4 (4)°. *MM*2 calculations reveal three minimum-energy conformers, all within 4 kJ of one another, similar to 1*B*, 2*B* and 2*B*′, with $\Delta\omega$(r.m.s., obs−calc) = 6·6 (3), 5·6 (3) and 12·8 (4)° respectively. The last value probably results from the poor definition of 2*B*′ from the diffraction data. All bond lengths and bond angles, except where poorly defined in the disordered regions, are normal.

We thank the National Institutes of Health, Division of Research Resources, for support of *PROPHET*.

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