angles C(3)—C(1)—C(2)—C(9) of 6·1 (9)° in (I) and 8·6 (9)° in (II) indicate that the C=C double bonds are twisted. All aryl rings are planar and rotated in the same sense from the plane of the central double bond to form a propeller conformation which minimizes the steric interaction between neighbouring aryl groups. Although both molecules adopt similar conformations, the torsional angle C(1)—C(2)—C(16)—C(21) of 65·6 (8)° in molecule (I) is significantly different from that of 60·6 (8)° in molecule (II), which indicates that packing forces affect the molecular conformation to some degree. The Br—C bond lengths of 1·924 (5) and 1·931 (4) Å are significantly longer than the reported mean value of 1·888 Å for Br—C bonds in other olefinic compounds (Kaftory, Apeloig & Rappoport, 1985), but they are similar to those in other triarylvinyl bromides. Other bond lengths and bond angles are in agreement with those reported for other triarylvinyl bromides (Wanigasekera *et al.*, 1984; Kaftory, Biali & Rappoport, 1985).

All aryl rings are also planar in the (*Z*)-isomer and the molecule adopts a conformation similar to that of the (*E*)-isomer. The torsional angles C(2)—C(1)—C(3)—C(8), C(1)—C(2)—C(9)—C(10) and C(1)—C(2)—C(15)—C(20) are 51 (1), 39·1 (9) and 63 (1)° respectively, which are within the range 38·3–79·0° for a series of triarylvinyl propellers reported by Kaftory, Biali & Rappoport (1985). Although the torsional angle equivalent to C(1)—C(2)—C(9)—C(10) has been found to be smaller (38·3°) in (*Z*)-1,2-dimesityl-2-phenylethenol, the torsional angles equivalent to C(2)—C(1)—C(3)—C(8) and C(1)—C(2)—C(15)—C(20) are 79·0 and 74·4° in that compound. The small value of the C(1)—C(2)—C(9)—C(10) torsional angle increases the steric repulsive interaction between the α- and β-aryl groups. Hence the central C=C bond is twisted more severely. The C(3)—C(1)—C(2)—C(9) torsional angle of 13 (1)° is not only larger than that of 1·1° in (*Z*)-1,2-dimesityl-2-phenylethenol but also than in the (*E*)-isomer and in other triarylvinyl compounds (Luo, Barton, Robertson, Lee & Wanigasekera, 1986; Kaftory, Biali & Rappoport, 1985; Biali & Rappoport, 1984). The unusually short C=C bond and the related long Br—C bond may be partly due to a spurious error in the position of atom C(1). It would be necessary to change the coordinates of atom C(1) by approximately three times their estimated standard deviations in order to give C=C and C—Br bond lengths similar to those in the (*E*)-isomer. Other bond lengths and bond angles are normal.

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Structure of a Four-Fused-Ring System Obtained by Intramolecular [2 + 2] Photoaddition

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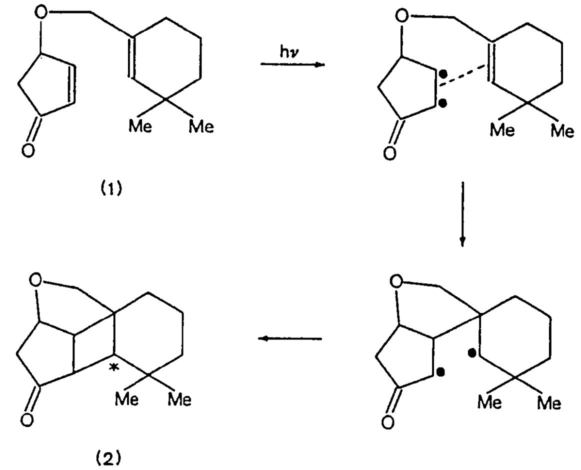
Abstract. 9,9-Dimethyl-3-oxatetracyclo[5.5.1.$0^{1,8}$.$0^{4,13}$]tridecan-6-one, $C_{14}H_{20}O_2$, $M_r = 220{\cdot}31$, monoclinic, $P2_1/c$, $a = 15{\cdot}342$ (2), $b = 6{\cdot}593$ (1), $c = 13{\cdot}085$ (2) Å, $\beta = 113{\cdot}71$ (1)°, $V = 1211{\cdot}8$ (3) Å^3, $Z = 4$, $D_m = 1{\cdot}18$, $D_x = 1{\cdot}208$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}073$ mm^{-1}, $F(000) = 480$, room temperature. The structure was refined to $R = 0{\cdot}076$ for 1796 independent reflections having $I > 0$ [$R = 0{\cdot}046$ for 1246 reflections with $I > 2\sigma(I)$]. The X-ray analysis confirms the identity of the main photoproduct obtained by irradiation of the olefinic enone DL-4-oxo-2-cyclopentenyl (3,3-dimethyl-1-cyclohexenyl)methyl ether. The two five-membered rings, both in a twisted conformation, are *cis*-fused, while the junction of the cyclobutane–cyclohexane system is *trans*. The six-membered ring is in a slightly distorted and flattened chair conformation, and the cyclobutane ring is non-planar with a dihedral angle of ~152°. Deformations of the *trans*-fused rings, mainly resulting from steric interactions, are discussed.

Introduction. Intramolecular [2 + 2] photocycloaddition of olefinic enones is a widely used reaction for the synthesis of a variety of ring systems found amongst natural products. During their work directed towards the total synthesis of tricyclic dicyclopenta[*a*,*d*]cyclooctane terpenes, Gariboldi, Jommi & Sisti (1986) have recently investigated the [2+2] photoaddition of monocyclic and bicyclic diallyl ethers were one of the two double bonds is conjugated with a carbonyl group ('activated' diallyl ethers). In the case of DL-4-oxo-2-cyclopentenyl (3,3-dimethyl-1-cyclohexenyl)methyl ether (1) (Scheme 1), irradiation at 310 nm gave a mixture of two products, in the ratio 3:2, with very high yield (92%). Chromatography on silica gel allowed the separation of the less abundant oily adduct from the major photoproduct, a crystalline solid with melting point 377–378 K. By chemical and spectroscopic methods, the two substances were both identified as tetracyclic compounds (2) differing in the relative configuration at their starred C atoms, hence in the junction between the cyclobutane and the cyclohexane rings. To confirm the identity of the crystalline diastereoisomer, and to determine its conformation, an X-ray structure analysis was performed.



Scheme 1

Experimental. Crystallization from light petroleum–diisopropyl ether at room temperature yielded single crystals as colorless prisms elongated along the crystallographic *b* axis. D_m by flotation in dilute K_2HgI_4 solution; single crystal of approximate dimensions $0{\cdot}28 \times 0{\cdot}25 \times 0{\cdot}15$ mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; lattice parameters from least-squares treatment of 25 reflections in the range $11{\cdot}5 \leq \theta \leq 15{\cdot}3°$. Intensity measurements, up to $\sin\theta/\lambda = 0{\cdot}5947$ Å^{-1}, by variable-rate ω-scan technique; 2114 independent reflections collected ($0 \leq h \leq 18$, $0 \leq k \leq 7$, $-14 \leq l \leq 14$), of which 318 with $I < 0$ were given zero weight; variance $\sigma(F_o^2) = [\sigma^2(I_{\rm raw}) + (pI_{\rm raw})^2]^{1/2}/{\rm Lp}$, where $\sigma(I_{\rm raw})$ was based on counting statistics, and $p = 0{\cdot}02$. Three standard reflections, $21\bar{2}$, 002, and $40\bar{4}$, periodically checked; all three showed the same decay (0·2% h^{-1}) in their intensity, with a total loss of 10·8% in the 55 h of exposure time; linear decay, Lorentz and polarization corrections, no absorption correction. Structure solved by direct methods; H atoms located by difference maps; full-matrix least-squares refinement based on F; C and O atoms anisotropic, H atoms isotropic; 1796 observations and 226 variables, including a secondary-extinction coefficient [final value $= 4{\cdot}5\,(6) \times 10^{-7}$], $w = 4F_o^2/\sigma^2(F_o^2)$; final $R = 0{\cdot}076$, $wR = 0{\cdot}042$, $S = 1{\cdot}30$ [$R = 0{\cdot}046$ and $wR = 0{\cdot}038$ for 1246 reflections with $I > 2\sigma(I)$]; max. $\Delta/\sigma = 0{\cdot}12$ [for B of atom H(14)B]; scattering factors and real and imaginary anomalous-dispersion corrections for neutral O and C atoms from *International Tables for X-ray Crystallography* (1974), for H atoms from Stewart, Davidson & Simpson (1965); no peaks higher than $0{\cdot}24$ e Å^{-3} on the final difference map. Programs: *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), *SDP-Plus* (Frenz, 1983); all computations on a PDP 11/34 computer.

Discussion. Final atomic coordinates are given in Table 1.* Bond distances and angles involving non-H atoms with their e.s.d.'s are listed in Table 2. Fig. 1 shows the atom numbering.

Stereochemistry. The nature of the compound under study as a tetracyclic tridecane derivative (2), hereinafter TT2, is confirmed. The two five-membered rings are *cis*-fused, while the junction between the cyclobutane and the six-membered rings is *trans*. Indirectly, and on the basis of ^{1}H NMR data, the oily diastereoisomer could be assigned a *cis*-fusion between the latter two rings.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43270 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Conformation. The cyclohexane ring shows a slightly distorted and flattened chair conformation, with torsion angles across the six C—C bonds in the range 49·9 (3)–63·8 (2)° (mean absolute value 55·2°). The amount of flattening can be evaluated by comparing the total puckering amplitude Q (Cremer & Pople, 1975) of this ring, 0·562 (2) Å, with the value, 0·625 Å, of an ideal cyclohexane chair with C—C bond lengths of 1·53 Å, the average value found here. The magnitude of the distortion is given by the puckering parameter θ, 4·8 (3)°, not too far from one of the two values (0 and 180°) describing an ideal chair conformation. The distortion occurs in the direction of a twisted conformation, as indicated by the third polar parameter, $\varphi = 91$ (3)°.

Table 1. *Positional parameters and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$(Å^2)
O(1)	0·58299 (9)	0·2040 (3)	0·2708 (1)	4·62 (4)
O(2)	0·6753 (1)	0·4670 (2)	0·5782 (1)	5·12 (4)
C(1)	0·5369 (1)	0·1646 (4)	0·3447 (2)	4·39 (6)
C(2)	0·5396 (2)	0·3470 (4)	0·4161 (2)	4·49 (6)
C(3)	0·6301 (1)	0·3296 (3)	0·5206 (1)	3·70 (5)
C(4)	0·6529 (1)	0·1058 (3)	0·5406 (1)	3·59 (5)
C(5)	0·5975 (1)	0·0039 (3)	0·4257 (2)	3·86 (5)
C(6)	0·6925 (1)	−0·0094 (3)	0·4100 (1)	3·37 (5)
C(7)	0·7342 (2)	−0·1873 (4)	0·3713 (2)	4·89 (6)
C(8)	0·8418 (2)	−0·1518 (4)	0·4150 (2)	5·58 (7)
C(9)	0·8921 (2)	−0·1092 (4)	0·5409 (2)	5·37 (7)
C(10)	0·8500 (1)	0·0675 (4)	0·5849 (2)	4·38 (6)
C(11)	0·7446 (1)	0·0147 (3)	0·5363 (1)	3·53 (5)
C(12)	0·6829 (1)	0·1808 (3)	0·3378 (2)	3·78 (5)
C(13)	0·8756 (2)	0·2719 (4)	0·5497 (2)	5·35 (7)
C(14)	0·8903 (2)	0·0619 (5)	0·7125 (2)	7·11 (8)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *involving the non*-H *atoms, with e.s.d.'s in parentheses*

	Uncorrected	Corrected*		Uncorrected	Corrected*
O(1)—C(1)	1·433 (3)	1·441	C(6)—C(7)	1·517 (3)	1·524
O(1)—C(12)	1·436 (2)	1·443	C(6)—C(11)	1·527 (2)	1·537
O(2)—C(3)	1·204 (2)	1·211	C(6)—C(12)	1·541 (3)	1·551
C(1)—C(2)	1·513 (4)	1·521	C(7)—C(8)	1·532 (4)	1·540
C(1)—C(5)	1·522 (3)	1·531	C(8)—C(9)	1·538 (3)	1·547
C(2)—C(3)	1·511 (2)	1·520	C(9)—C(10)	1·551 (4)	1·558
C(3)—C(4)	1·515 (3)	1·523	C(10)—C(11)	1·521 (2)	1·529
C(4)—C(5)	1·551 (2)	1·561	C(10)—C(13)	1·526 (4)	1·536
C(4)—C(11)	1·551 (2)	1·558	C(10)—C(14)	1·530 (3)	1·539
C(5)—C(6)	1·554 (3)	1·561			

C(1)—O(1)—C(12)	105·3 (1)	O(1)—C(1)—C(2)	112·0 (2)
O(1)—C(1)—C(5)	105·5 (1)	C(2)—C(1)—C(5)	105·4 (2)
C(1)—C(2)—C(3)	106·6 (2)	O(2)—C(3)—C(2)	126·7 (2)
O(2)—C(3)—C(4)	126·2 (1)	C(2)—C(3)—C(4)	107·1 (1)
C(3)—C(4)—C(5)	104·8 (1)	C(3)—C(4)—C(11)	121·4 (1)
C(5)—C(4)—C(11)	87·1 (1)	C(1)—C(5)—C(4)	108·4 (2)
C(1)—C(5)—C(6)	106·8 (2)	C(4)—C(5)—C(6)	88·6 (1)
C(5)—C(6)—C(7)	129·2 (2)	C(5)—C(6)—C(11)	87·8 (1)
C(5)—C(6)—C(12)	99·9 (1)	C(7)—C(6)—C(11)	109·5 (2)
C(7)—C(6)—C(12)	111·9 (2)	C(11)—C(6)—C(12)	117·3 (1)
C(6)—C(7)—C(8)	107·0 (2)	C(7)—C(8)—C(9)	115·4 (2)
C(8)—C(9)—C(10)	115·5 (2)	C(9)—C(10)—C(11)	101·8 (2)
C(9)—C(10)—C(13)	110·8 (2)	C(9)—C(10)—C(14)	109·9 (2)
C(11)—C(10)—C(13)	115·8 (2)	C(11)—C(10)—C(14)	110·2 (2)
C(13)—C(10)—C(14)	108·1 (2)	C(4)—C(11)—C(6)	89·5 (1)
C(4)—C(11)—C(10)	136·3 (2)	C(6)—C(11)—C(10)	118·8 (1)
O(1)—C(12)—C(6)	106·0 (1)		

* After rigid-body correction.

The furanoid ring exhibits an almost perfect twisted conformation with the twist axis through atom C(5). Puckering parameters are $q_2 = 0{\cdot}376(2)$ Å and $\varphi_2 = 90{\cdot}8$ (4)°, the latter quantity being only marginally different from 90°, the value appropriate to one of the ten possible T forms of a five-membered ring.

The T conformation of the substituted cyclopentanone ring appears more distorted, where the puckering amplitude is $q_2 = 0{\cdot}266$ (3) Å; the twist axis is again through atom C(5), but the phase angle φ_2 is 96·9 (4)° (to be compared with 90 and 108°, the closest values for pure twist and envelope forms, respectively).

In the cyclobutane ring, the four C atoms are displaced by ±0·135 (1) Å from the mean plane, hence the single puckering parameter is $q = \pm 0{\cdot}270$ (1) Å. Alternatively, the conformation of this ring can be described by the dihedral angle between the plane defined by atoms C(5), C(6) and C(11), and that defined by atoms C(5), C(4) and C(11). The value found for this angle in TT2, 151·9 (1)°, is well within the range of values, 145–160°, reported for non-planar substituted cyclobutanes (Adman & Margulis, 1968; Benedetti, Corradini & Pedone, 1970).

Molecular geometry and packing. Packing indices for TT2, computed following the method of Gavezzotti (1985), are in the normal range for compounds of this kind, although the packing coefficient C_k is rather low, 0·687 *vs* 0·704 (Gavezzotti, 1982). There are no intermolecular contacts shorter than the sum of van der Waals radii, with the possible exception of O(1)···H(2)B (at $1-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$), 2·53 (1) Å.

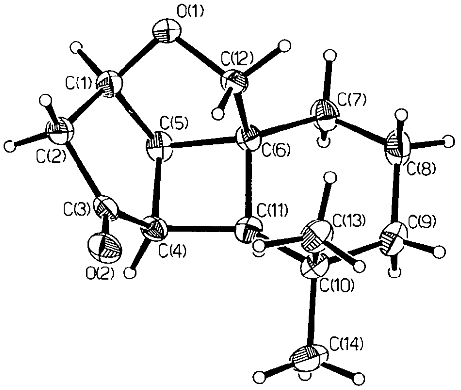

Fig. 1. Perspective view of the molecule with numbering scheme. Non-labeled H atoms were numbered according to the C atom to which they are bonded. The thermal ellipsoids are drawn (*ORTEP*, Johnson, 1970) at a 20% probability level; H atoms are on an arbitrary scale.

Table 3. *Some short intramolecular contacts* (Å)

C(2)···C(12)	2·983 (4)	C(8)···C(12)	3·129 (3)
C(2)···H(12)*B*	2·74 (2)	C(9)···H(11)	2·42 (2)
C(3)···C(6)	3·020 (3)	C(12)···C(13)	3·193 (3)
C(3)···C(12)	2·983 (3)	C(12)···H(13)*C*	2·67 (1)
C(3)···H(12)*B*	2·47 (2)	C(13)···H(12)*B*	2·64 (1)
C(4)···C(12)	2·912 (3)	H(4)···H(11)	2·19 (2)
C(7)···H(11)	2·48 (1)	H(12)*B*···H(13)*C*	2·21 (2)

As expected for a cage molecule like TT2, there are several close intramolecular contacts: as seen in Table 3, most of them involve atoms of the methylene group at C(12), but none can be classified as exceptionally short. Steric hindrance is also documented by other molecular dimensions, especially by the values of the bond angles. A quantity that can be used to gauge overcrowding is the molecular free surface S_M (Gavezzotti, 1985). The difference between the S_M value of TT2 predicted by group increments, 245·9 Å^2, and that actually computed, 237·7 Å^2, seems to indicate a rather small steric hindrance: for highly overcrowded molecules much higher differences, up to 10–11%, have been calculated (Gavezzotti, 1985). However, in the present case crowding effects, that imply a free area smaller than predicted, are partially cancelled by ring-strain effects, which produce a significant increase of the molecular free surface. [For example, in the non-substituted cyclobutane the value of S_M is 95·6 Å^2 for the actual molecule, that obtained by group increments is 83·6 Å^2.] Therefore, the overall reduction of S_M in TT2, although apparently small, qualifies this molecule as remarkably overcrowded.

Pertinent molecular dimensions of TT2 can be compared with those found in *trans*-bicyclo[4.2.0]oct-1-yl 3,5-dinitrobenzoate [*trans* (4.2.0)] (Barnett & Davis, 1970). It is seen that the insertion of the two fused five-membered rings into the bicyclic system, and the interaction of the axial methyl group at C(13) with the added molecular fragment give rise to the following effects: (i) in the cyclohexane ring, the two C—C bonds adjacent to the fused bond are lengthened by more than 0·02 Å; nevertheless, they are still the shortest C—C bonds in the bicyclic system. In the cyclobutane moiety, the three bonds that in TT2 are shared by two rings virtually maintain their length, while the non-fused bond is elongated by about 0·01 Å; (ii) four out the six torsion angles of the cyclohexane ring change by less than 1·1°, while the other two, namely those across the C(7)—C(8) and C(8)—C(9) bonds, are increased by about 3°; (iii) the four-membered ring becomes flatter, the dihedral angle (see above) being 147·4 (2)° in *trans* (4.2.0); (iv) in the whole framework common to the two molecules, only five corresponding bond angles differ by less than 1°: they are the three angles internal to the cyclobutane ring at C(4), C(5) and C(11), and the two bond angles at C(7) and C(8) (the numbering is that of TT2, and bond angles involving H atoms are not included in the comparison). The largest angular deformations occur at the quaternary C atom C(6), to which an O atom, in place of atom C(12), is bonded in *trans* (4.2.0). Indeed, in the latter molecule, the angles corresponding to C(5)—C(6)—C(7) and C(5)—C(6)—C(12) are larger by 3·6 (2) and 6·2 (2)°, respectively, while that corresponding to C(11)—C(6)—C(12) is smaller by 12·3 (2)°. Clearly, this large variation is dictated by the need of avoiding too short contacts of the methylene group at C(12) with other atoms. To the same reason can be ascribed the large value of C(3)—C(4)—C(11), 121·4 (1)°. Finally, we note that the bond angle C(4)—C(11)—C(10) is increased from 133·8 (2)° in *trans* (4.2.0) to 136·3 (2)° in TT2, the latter being, to our knowledge, one of the largest values ever found for CCC bond angles external to a cyclobutane ring.

Thermal-motion analysis. Hirshfeld's rigid-bond test (Hirshfeld, 1976; Rosenfield, Trueblood & Dunitz, 1978) indicates that in TT2 the differences $\Delta_{A,B}$ between the mean square vibrational amplitudes in the AB direction are, on the average, not significantly larger for pairs of non-bonded atoms (21×10^{-4} Å^2) than for connected atoms (20×10^{-4} Å^2); all individual values of $\Delta_{A,B}$ fall in the range 12–29 $\times 10^{-4}$ Å^2. Therefore, the rigid-body model was considered appropriate in the analysis of the thermal motion of TT2, and the tensors **T, L, S** were computed following the least-squares treatment proposed by Schomaker & Trueblood (1968). All 16 non-H atoms were included in the calculations, and a Cartesian coordinate system defined by the principal moments of inertia of the molecule was employed. No account was taken of the correlations between different components of the atomic vibration tensors **U**, and unit-weight matrices were used. The RMS residue $\Delta U_{\rm RMS} = \langle (U_{\rm obs} - U_{\rm calc})^2 \rangle^{1/2}$ obtained from the analysis, 28×10^{-4} Å^2, is more than twice as large as $\sigma_{\rm RMS}(U_{\rm obs})$, 12×10^{-4} Å^2; this seems to suggest, in contrast with the results of the rigid-bond test, that TT2 behaves as a rigid body only to a first approximation, but a possible relevant underestimate of the U_{ij} e.s.d.'s is to be taken into account.

The translational motion exhibits only a slight anisotropy, the eigenvalues of the tensor **T** being (in Å $\times$ 10^2) 4·6 (1), 4·1 (1), and 3·3 (2). The lengths of principal axes of the **L** tensor are 27(1), 16(1), and 12·3(8)°^{2}, and implied corrections to the interatomic distances (see Table 2) are in the range 0·007–0·010 Å.

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Structure of 1-Tosylimino-1,5-dithiacyclooctane

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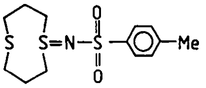
Abstract. $C_{13}H_{19}NO_2S_3$, $M_r = 317{\cdot}48$, triclinic, $P\bar{1}$, $a = 9{\cdot}2622\,(8)$, $b = 9{\cdot}5582\,(6)$, $c = 9{\cdot}1289\,(8)$ Å, $\alpha = 87{\cdot}199\,(8)$, $\beta = 111{\cdot}953\,(6)$, $\gamma = 105{\cdot}050\,(7)°$, $V = 722{\cdot}8\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}459$ Mg m^{-3}, $\lambda(\mathrm{Cu}\,K\alpha_1) = 1{\cdot}54051$ Å, $\mu = 4{\cdot}600$ mm^{-1}, $F(000) = 336$, 295 K, $R = 0{\cdot}034$ for 2361 observed reflexions. The 1,5-dithiacyclooctane ring has a boat–chair conformation with a short transannular $S^{II}\cdots S^{IV}$ contact of 3·143 Å. The $S^{II}\cdots S^{IV}$—N angle is 177·8°. The configuration about the S^{IV} atom is a distorted trigonal bipyramid with two apical S^{IV}—N and $S^{II}\cdots S^{IV}$ contacts. The lengths S^{IV}—N and S^{VI}—N are 1·641 and 1·594 Å, which are normal for *N*-sulfonylsulfilimines. One of the O atoms of the sulfonyl group is *cis* to S^{IV} about the S^{VI}—N bond with a torsion angle of 3·6°. Associated with the *cis* conformation, another short intramolecular contact is observed between S^{IV} and O (2·893 Å).

Introduction. Structures of several *N*-sulfonylsulfilimines have been determined by X-ray analyses (Kálmán, Duffin & Kucsman, 1971; Kálmán & Sasvári, 1972; Cameron, Hair & Morris, 1973; Kálmán, Párkányi & Kucsman, 1980). The S^{IV}=N—S^{VI} bond systems of these compounds show very similar features with long S^{IV}=N and short S^{VI}—N bonds. In the present compound a transannular effect of S^{II} of the 1,5-dithiacyclooctane ring on such a S^{IV}=N—S^{VI} system was expected. An X-ray analysis for the title compound was undertaken to investigate the interaction between the sulfonylsulfilimido group and the 1,5-dithiacyclooctane ring.



Experimental. Colorless plate, 0·45 × 0·30 × 0·15 mm, unit-cell parameters by least squares from 23 reflexions ($70 < 2\theta < 74°$), Rigaku AFC4 diffractometer, graphite monochromated, $2 \leq 2\theta \leq 130°$, $h = 0 \to 10$, $k = -11 \to 11$, $l = -10 \to 10$, ω–2θ scan, scan range $\Delta\omega = 1{\cdot}3° + 0{\cdot}5°\tan\theta$, scan speed 4° min^{-1} in 2θ, three reflexions monitored ($3\bar{2}\bar{1}$, 030, 003) every 50 reflexions, variations within 2%. 2777 reflexions measured, 2444 unique, $R_{\mathrm{int}} = 0{\cdot}03$, 2361 observed, $|F_o| \geq 3\sigma(F)$. Structure was solved by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). 11 non-H atoms were obtained from the *E* map and the remaining non-H atoms were located from the Fourier synthesis. H atoms were found from the difference map. Block-diagonal least squares with anisotropic temperature factors for non-H, isotropic for H. $\sum w(|F_c| - k^{-1}|F_o|)^2$ was minimized. $w = 1/(0{\cdot}36055 - 0{\cdot}02935|F_o| + 0{\cdot}00132|F_o|^2)$. Absorption correction was applied numerically; range of transmission

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