mean plane of 1,2,4-triazacyclopentane is 45° compared with 75·3° in (I). The N(2)—C(16) bond length (1·436 Å) is therefore longer than the other N—C(sp^2) bonds in the molecule (1·364–1·403 Å). The differences can be ascribed to interaction of the N lone-pair electrons with the two carbonyl groups [C(14), C(15)] while there is no interaction with the phenyl group. The same difference was also observed by van der Ende, Offereins & Romers (1974); 1·431, 1·357–1·403 Å.

The packing of molecules in the unit cell is shown in Fig. 3. Intermolecular distances shorter than van der Waals contacts have not been found.

The author wishes to thank Professor D. Ginsburg and Dr J. Kalo for providing a sample of the compound.

References

ENDE, C. VAN DER, OFFEREINS, B. & ROMERS, C. (1974). Acta Cryst. B30, 1947–1952.
HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). Acta Cryst. 17, 1040–1044.
JOHNSON, C. K. (1965). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.
KALO, J., VOGEL, E. & GINSBURG, D. (1977). To be published.
KORAT, M., TATARSKY, D. & GINSBURG, D. (1972). Tetrahedron, 28, 2315–2332.
MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data. Univs. York, England, and Louvain, Belgium.

The Monoadduct between 12-Thia[4.4.3]propella-2,4-diene anti-12-Oxide and 4-Phenyl-1,2,4-triazoline-3,5-dione*

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Abstract. $C_{20}H_{21}N_3O_3S$, monoclinic, $P2_1/n$, $a = 7·344$ (2), $b = 27·029$ (6), $c = 9·165$ (2) Å, $\beta = 100·75$ (3)°, $M_r = 383·48$, $Z = 4$, $D_x = 1·306$ g cm^{-3}. The two six-membered rings are in the boat form; the substituted ring is folded away from, the other ring towards, the central five-membered ring. The 4-phenyl-1,2,4-triazoline-3,5-dione ring is syn with respect to the latter. The central five-membered ring has an envelope shape with the S—O(1) bond flapping towards the unsubstituted six-membered ring.

Introduction. The configuration of the title compound is of interest in connexion with stereochemical studies of the Diels–Alder adducts of [4.4.X]propella-2,4-dienes ($X \equiv$ substituted rings) (Korat, Tatarsky & Ginsburg, 1972; Kalo, Vogel & Ginsburg, 1977). Intensities from a colourless crystal, 0·4 × 0·3 × 0·3 mm, were collected on a semi-automatic Stoe–Weissenberg diffractometer with graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0·71069$ Å) with the $\omega/2\theta$ scanning technique; 2295 reflexions were measured, of which 1562 were significantly above background $[F_o > 3\sigma(F_o)]$. The structure was solved by direct methods

with MULTAN 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and refined by least-squares analysis in two blocks. H atoms were located on a difference map. These were included in the refinement at positions calculated on the basis of the heavy-atom positions. A constant temperature factor ($U = 0·076$ Å^2) was assigned to the H atoms; their positional and vibrational parameters were not refined. Scattering factors for S, C, N, O and H were taken from Hanson, Herman, Lea & Skillman (1964). The final R for 1562 reflexions was 0·104. The final positional parameters are listed in Table 1.[†] Corresponding interatomic distances, angles and torsion angles involving S, C, N and O atoms are shown in Fig. 1.

Discussion. The rather high value of R is probably due to systematic errors in the intensity measurements which may also affect the differences between chemically equivalent bond lengths and angles.

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* Configuration of Diels–Alder Adducts. II.

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$$C_{20}H_{71}N_3O_3S$$

Table 1. *Fractional coordinates for non-hydrogen atoms* ($\times 10^4$) *(e.s.d.'s in parentheses) and calculated hydrogen atom positions* ($\times 10^3$)

|	x	y	z		x	y	z
S	5823 (4)	1935 (1)	660 (4)	C(18)	10508 (15)	−1448 (4)	1862 (13)
O(1)	4881 (11)	2420 (3)	833 (10)	C(19)	8981 (18)	−1425 (4)	2565 (12)
O(2)	4712 (8)	−270 (2)	1428 (7)	C(20)	8042 (13)	−976 (3)	2698 (11)
O(3)	10434 (10)	337 (2)	3526 (9)	H(2)	299	61	220
N(1)	5821 (9)	525 (2)	2054 (8)	H(3)	375	41	484
N(2)	7825 (10)	−88 (2)	2265 (8)	H(4)	689	65	609
N(3)	7584 (9)	709 (2)	2764 (8)	H(5)	862	113	467
C(1)	4334 (12)	1309 (3)	2546 (10)	H(71)	726	197	520
C(2)	4224 (13)	732 (3)	2696 (11)	H(72)	561	161	554
C(3)	4651 (14)	588 (3)	4325 (11)	H(81)	518	249	374
C(4)	6369 (14)	731 (3)	5025 (11)	H(82)	436	240	526
C(5)	7409 (11)	1027 (3)	4077 (10)	H(91)	264	224	243
C(6)	6279 (13)	1491 (3)	3447 (11)	H(92)	179	218	395
C(7)	6026 (15)	1822 (4)	4773 (13)	H(101)	152	144	245
C(8)	4619 (16)	2241 (4)	4332 (15)	H(102)	266	135	414
C(9)	2802 (16)	2075 (4)	3416 (14)	H(111)	292	155	45
C(10)	2691 (13)	1517 (4)	3167 (12)	H(112)	452	114	34
C(11)	4206 (13)	1436 (4)	873 (11)	H(121)	837	155	216
C(12)	7376 (14)	1766 (4)	2388 (14)	H(122)	793	207	289
C(13)	6028 (13)	11 (3)	1881 (10)	H(16)	1060	−27	93
C(14)	8828 (11)	322 (3)	2941 (10)	H(17)	1224	−104	75
C(15)	8735 (12)	−555 (3)	2101 (10)	H(18)	1115	−177	180
C(16)	10195 (14)	−579 (4)	1376 (12)	H(19)	853	−173	298
C(17)	11146 (14)	−1024 (4)	1265 (13)	H(20)	695	−96	320
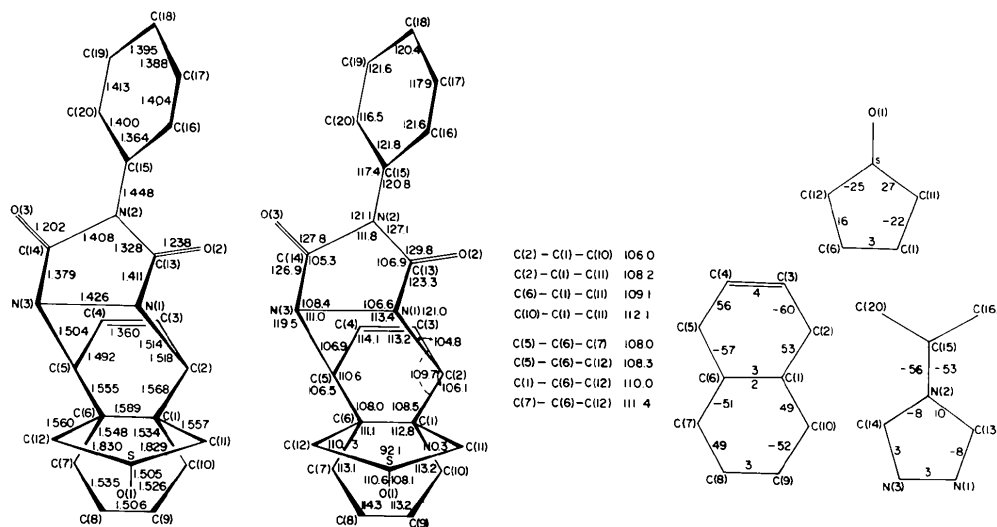
Fig. 1. Bond distances (Å), bond angles and torsion angles (°).

The tetrahydrothiophene part has an envelope shape with the S atom flapping towards the unsubstituted six-membered ring (Fig. 2). A mean-plane calculation shows that S and O(1) are displaced by 0·55 and 2·06 Å respectively from the mean plane through C(1), C(6), C(11), C(12).

The 4-phenyl-1,2,4-triazoline-3,5-dione ring is *syn* with respect to the central five-membered ring; in a similar compound with cyclopentane as the central ring

(Kaftory, 1978) it was found to be *anti*. The angle between the two mean planes described by atoms N(1), N(3), C(13), C(14) and N(1), N(3), C(2), C(5) is 134·2° (present work) compared with 139·5° found by Kaftory (1978) and 149·3° found by van der Ende, Offereins & Romers (1974). The dihedral angle between the phenyl group and the triazoline ring in the three compounds is 54, 45 and 75·3° respectively.

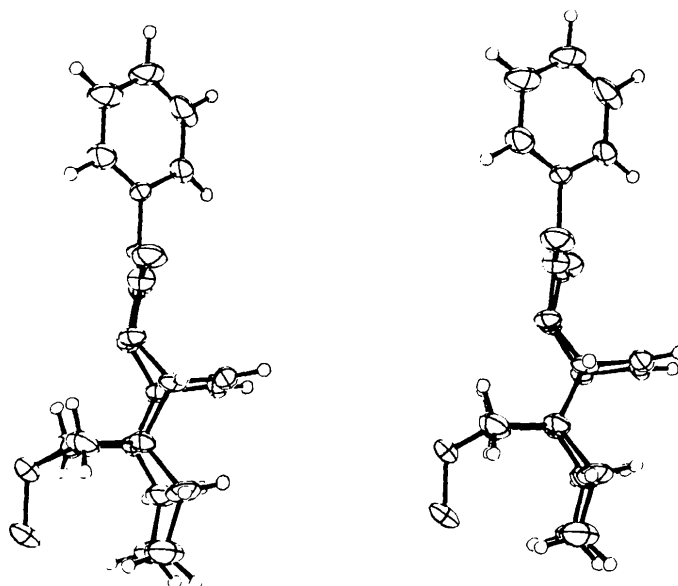The packing of molecules in the unit cell is shown in

Fig. 2. Stereoscopic view of the molecule, showing vibration ellipsoids at the 50% probability level (Johnson, 1965).
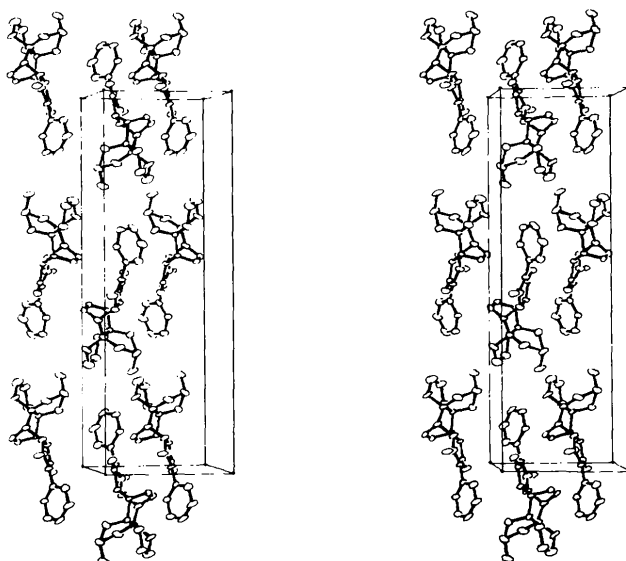

Fig. 3. Packing of molecules in the unit cell.

Fig. 3. Intermolecular distances shorter than van der Waals contacts have not been found.

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References

ENDE, C. VAN DER, OFFEREINS, B. & ROMERS, C. (1974). Acta Cryst. B30, 1947–1952.

HANSON, H. P., HERMAN, F., LEA, J. D. & SKILLMAN, S. (1964). Acta Cryst. 17, 1040–1044.

JOHNSON, C. K. (1965). ORTEP. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

KAFTORY, M. (1978). Acta Cryst. B34, 300–303.

KALO, J., VOGEL, E. & GINSBURG, D. (1977). To be published.

KORAT, M., TATARSKY, D. & GINSBURG, D. (1972). Tetrahedron, 28, 2315–2332.

MAIN, P., WOOLFSON, M. M., LESSINGER, L., GERMAIN, G. & DECLERCQ, J. P. (1974). MULTAN 74. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data. Univs. York, England, and Louvain, Belgium.