

## The Diadduct between 1,6-Methano[10]annulene, Maleic Anhydride and 4-Methyl-1,2,4-triazoline-3,5-dione\*

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**Abstract.**  $C_{18}H_{15}N_3O_5$ , orthorhombic,  $Pna2_1$ ,  $a = 9.862$  (6),  $b = 15.336$  (7),  $c = 10.023$  (3) Å,  $M_r = 353.34$ ,  $Z = 4$ ,  $D_x = 1.549$  g cm $^{-3}$ . The two six-membered rings are in the boat form, both folded towards the cyclopropane. The 4-methyl-1,2,4-triazoline-3,5-dione and the anhydride rings are *syn* with respect to each other but both are *anti* to the cyclopropane ring.

**Introduction.** The configuration of the title compound is of interest in connexion with stereochemical studies of Diels–Alder adducts of [10]annulenes (Ashkenazi, Vogel & Ginsburg, 1977).

Intensities from a colourless crystal  $0.4 \times 0.3 \times 0.3$  mm were collected on a semi-automatic Stoe–Weissenberg diffractometer with graphite-monochromatized Mo  $K\alpha$  radiation ( $\lambda = 0.71069$  Å) with the  $\omega/2\theta$  scan technique; 1625 reflexions were measured including 31 reflexions with zero net intensity. 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 atoms were included in the refinement at positions calculated on the basis of the heavy-atom positions. A constant temperature factor ( $U = 0.07$  Å $^2$ ) was assigned to the H atoms. The atomic parameters of the H atoms were not refined. Scattering factors for C, N, O and H were taken from Hanson, Herman, Lea & Skillman (1964). The final  $R$  for 1594 non-zero reflexions was 0.080. Final positional parameters are listed in Table 1.† Corresponding interatomic distances, angles and torsion angles involving C, N and O atoms are shown in Fig. 1.

**Discussion.** The two six-membered rings are in the boat form, both folded towards the cyclopropane ring. The

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32935 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

\* Configuration of Diels–Alder Adducts. III.

Table 1. Atomic 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
O(1)	2088 (6)	9069 (4)	2184 (5)	C(14)	3981 (11)	7589 (5)	1908 (7)
O(2)	5271 (7)	7560 (3)	4545 (6)	C(15)	1557 (7)	8367 (3)	7287 (6)
O(3)	925 (8)	7381 (4)	9096 (6)	C(16)	626 (8)	7869 (4)	8225 (8)
O(4)	−686 (6)	8034 (3)	7938 (6)	C(17)	−812 (9)	8652 (4)	6922 (8)
O(5)	−1912 (7)	8872 (4)	6553 (7)	C(18)	567 (7)	8921 (4)	6472 (6)
N(1)	2655 (6)	9064 (3)	4427 (5)	H(2)	216	1043	424
N(2)	3648 (6)	8108 (3)	3080 (6)	H(3)	448	1073	394
N(3)	3617 (5)	8566 (3)	5170 (5)	H(4)	610	989	526
C(1)	2324 (6)	10066 (2)	6264 (6)	H(5)	508	880	669
C(2)	2750 (7)	10023 (3)	4786 (6)	H(7)	320	862	864
C(3)	4193 (7)	10260 (4)	4575 (7)	H(8)	170	956	990
C(4)	5101 (8)	9801 (4)	5291 (7)	H(9)	12	1042	865
C(5)	4390 (6)	9120 (4)	6146 (6)	H(10)	37	1016	593
C(6)	3338 (6)	9523 (3)	7068 (6)	H(111)	300	1078	804
C(7)	2541 (6)	8957 (3)	8089 (6)	H(112)	410	1085	683
C(8)	1651 (7)	9537 (4)	8911 (6)	H(141)	337	775	116
C(9)	782 (7)	10019 (4)	8221 (6)	H(142)	386	696	213
C(10)	878 (7)	9907 (3)	6715 (6)	H(143)	494	770	165
C(11)	3329 (7)	10502 (4)	7207 (7)	H(15)	214	797	674
C(12)	2764 (7)	8794 (4)	3088 (6)	H(18)	66	883	550
C(13)	4344 (7)	8025 (4)	4305 (7)				



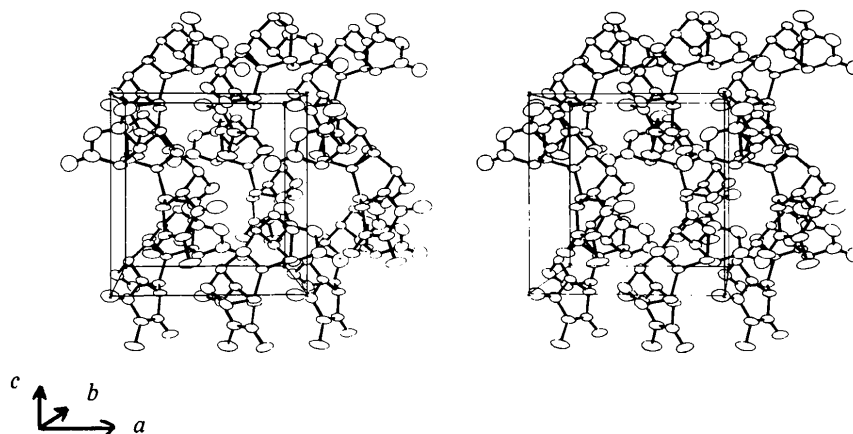


Fig. 3. Packing of molecules in the unit cell. The origin is at the lower left-hand corner of the unit cell.

The puckering of the two six-membered rings, as can be seen from bond angles (Fig. 1), is due to the strain imposed by the two substituents on C(2)···C(5) and C(7)···C(10).

The discrepancies in chemically equivalent bond lengths and angles might be attributed to systematic errors in intensity measurements.

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.

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## 1,4-Dimethyl-9-isobutylthioxanthene 10,10-Dioxide

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**Abstract.**  $C_{19}H_{22}O_2S$ , triclinic,  $P\bar{1}$ ,  $Z = 4$ ,  $M_r = 314.43$ ,  $a = 9.860$  (1),  $b = 18.294$  (1),  $c = 14.197$  (1) Å,  $\alpha = 108.71$  (1),  $\beta = 111.49$  (1),  $\gamma = 116.48$  (1)°,  $V = 1726.81$  Å<sup>3</sup>,  $D_x = 1.209$ ,  $D_m = 1.22$  g cm<sup>-3</sup> (by flotation),  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu(\text{Cu } K\alpha) = 16.48$  cm<sup>-1</sup>. Final residual  $R = 0.057$ . The interactions

between the *meso* and *para* substituents cause the differences in bond lengths and bond angles involving the *meso* atoms.

**Introduction.** Single crystals of the title compound (I) were obtained through the courtesy of Dr A. L. Ternay