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Structure of *N*-Methoxycarbonylamino-3,4-bis(4-nitrophenyl)maleimide at 140 K

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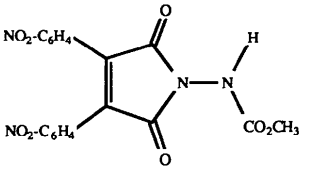
Abstract. Methyl *N*-[3,4-bis(4-nitrophenyl)-2,5-dioxo-2,5-dihydro-1-pyrrolyl]carbamate, $C_{18}H_{12}N_4O_8$, $M_r = 412{\cdot}32$, monoclinic, $P2_1$, $a = 8{\cdot}171\ (3)$, $b = 10{\cdot}351\ (2)$, $c = 10{\cdot}965\ (3)$ Å, $\beta = 97{\cdot}03\ (3)°$, $V = 920{\cdot}4$ Å^3, $Z = 2$, $D_x = 1{\cdot}49$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}112$ mm^{-1}, $F(000) = 424$, $T = 140$ K, $R = 0{\cdot}031$ for 1529 observed reflections [$I > 3\sigma(I)$]. The methoxycarbonylamino group is closely perpendicular [83·1 (1)°] to the almost planar maleimide ring. The dihedral angles between the phenyl rings [C(5)–C(10) and C(11)–C(16)] and the maleimide moiety are 41·0 (1) and 39·8 (1)° respectively. The molecules related by a twofold screw axis are hydrogen bonded through the H atom on the amino N atom to the O atom of one carbonyl group {N(2)···O(1^i) 2·989, H···O(1^i) 2·115 Å, ∠N(2)—H···O(1^i) 164·9° [(i) $-x$, $\frac{1}{2}+y$, $1-z$)]}, forming infinite chains along the **b** direction.

Experimental. Yellow plate-shaped crystals by recrystallization from benzene–ethanol mixture. Suitable crystal (0·48 × 0·43 × 0·12 mm) mounted on an Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; lattice parameters refined by least-squares fitting of 2θ values of 25 reflections in the range 10·3–19·7°. Attempts to solve the structure from intensity data at room temperature were unsuccessful probably because of the high thermal motion. Intensity data recollected at 140 K with maximum Bragg angle $\theta_{max} = 24°$ (corresponding to h 0/9, k 0/12, l −13/13) and ω–2θ scan technique [ω scan width $\Delta\omega = (1{\cdot}00 + 0{\cdot}35\tan\theta)°$, variable scan rate, maximum scan time 60 s per reflection]. Three standard reflections showed a slight decay of 3·3% over 29 h of X-ray exposure time. 1839 measured reflections of which 1710 were unique ($R_{int} = 0{\cdot}023$). Data corrected for previous linear decay, Lorentz–polarization effects but not for abosorption. Among possible space groups $P2_1$ and $P2_1/m$, the former was chosen from intensity statistics and successful refinements. Structure solved by direct methods using the *MULTAN*11/82 series of programs (Main *et al.*, 1982) and refined by full-matrix least squares (on F's) with weights $w = [\sigma^2(F) + 0{\cdot}04F_o^2]^{-1}$. Non-H atoms refined anisotropically and H atoms from a difference Fourier map simply included in structure-factor calculations ($B_{iso} = 4$ Å^2). $R = 0{\cdot}031$, $wR = 0{\cdot}041$, $S = 1{\cdot}740$ for 271 variables and 1529 observed reflections [$I > 3\sigma(I)$]; max. shift/e.s.d. $(\Delta/\sigma) = 0{\cdot}0$, max. height in final difference synthesis 0·25 e Å^{-3}. Atomic scattering factors for neutral atoms and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV).

All calculations performed on PDP 11/60 computer using *SDP*/PDP V3.0 software package (Frenz, 1985). Atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Bond distances and bond angles are in Table 2. Fig. 1 shows a general view of the molecule and Fig. 2 a stereoscopic view of the unit cell. A diagram of the molecule is shown below.



(I)

Related literature. The synthesis of (I) is described by Florac, Baudy-Floc'h & Robert (1988). The present X-ray study was undertaken to verify unambiguously the stereochemistry of the title compound (Florac, 1989). For recent related structures see McPhalen & James (1983) and Loehlin (1985).

* Lists of structure factors, anisotropic thermal parameters, atomic parameters for H atoms, bond lengths and bond angles involving H atoms, torsion angles and least-squares-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53557 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Non*-H *atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0·0778 (2)	0·499	0·4943 (2)	2·53 (3)
O(2)	0·3472 (2)	0·8829 (2)	0·5022 (2)	2·87 (4)
O(3)	0·3706 (3)	0·0376 (2)	0·1112 (2)	3·97 (5)
O(4)	0·4261 (4)	0·1707 (3)	−0·0277 (2)	5·87 (7)
O(5)	0·9752 (3)	0·9620 (3)	0·1649 (3)	5·57 (6)
O(6)	1·0926 (3)	0·7802 (3)	0·2151 (2)	5·80 (6)
O(7)	0·0810 (2)	0·8396 (2)	0·7825 (2)	3·39 (4)
O(8)	0·2873 (2)	0·7020 (2)	0·7553 (2)	3·08 (4)
N(1)	0·1813 (3)	0·7060 (2)	0·5137 (2)	2·49 (4)
N(2)	0·0872 (2)	0·7547 (2)	0·5997 (2)	2·41 (4)
N(3)	0·3843 (3)	0·1468 (3)	0·0730 (2)	3·23 (5)
N(4)	0·9744 (3)	0·8529 (3)	0·2080 (2)	3·74 (6)
C(1)	0·1742 (3)	0·5807 (3)	0·4703 (2)	2·03 (4)
C(2)	0·3077 (3)	0·5714 (3)	0·3869 (2)	2·01 (4)
C(3)	0·3919 (3)	0·6831 (3)	0·3926 (2)	2·09 (5)
C(4)	0·3127 (3)	0·7743 (3)	0·4745 (2)	2·20 (5)
C(5)	0·3217 (3)	0·4594 (3)	0·3073 (2)	1·95 (4)
C(6)	0·3037 (3)	0·3335 (3)	0·3479 (2)	2·14 (5)
C(7)	0·3203 (3)	0·2307 (3)	0·2711 (2)	2·18 (5)
C(8)	0·3556 (3)	0·2557 (3)	0·1534 (2)	2·46 (5)
C(9)	0·3706 (3)	0·3785 (3)	0·1092 (2)	2·75 (5)
C(10)	0·3533 (3)	0·4814 (3)	0·1861 (2)	2·50 (5)
C(11)	0·5394 (3)	0·7235 (3)	0·3384 (2)	2·06 (4)
C(12)	0·5506 (3)	0·8491 (3)	0·2954 (2)	2·51 (5)
C(13)	0·6930 (3)	0·8915 (3)	0·2516 (3)	2·72 (5)
C(14)	0·8222 (3)	0·8069 (3)	0·2510 (2)	2·69 (5)
C(15)	0·8133 (3)	0·6805 (3)	0·2903 (2)	2·76 (5)
C(16)	0·6716 (3)	0·6389 (3)	0·3338 (2)	2·56 (5)
C(17)	0·1656 (3)	0·7602 (3)	0·7181 (2)	2·34 (5)
C(18)	0·1398 (5)	0·8480 (5)	0·9116 (3)	5·9 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

O(1)—C(1)	1·206 (3)	C(2)—C(3)	1·342 (3)
O(2)—C(4)	1·189 (3)	C(2)—C(5)	1·464 (3)
O(3)—N(3)	1·216 (3)	C(3)—C(4)	1·502 (3)
O(4)—N(3)	1·220 (3)	C(3)—C(11)	1·468 (3)
O(5)—N(4)	1·224 (3)	C(5)—C(6)	1·391 (3)
O(6)—N(4)	1·220 (3)	C(5)—C(10)	1·403 (3)
O(7)—C(17)	1·331 (3)	C(6)—C(7)	1·374 (3)
O(7)—C(18)	1·441 (3)	C(7)—C(8)	1·381 (3)
O(8)—C(17)	1·191 (3)	C(8)—C(9)	1·372 (4)
N(1)—N(2)	1·383 (2)	C(9)—C(10)	1·376 (3)
N(1)—C(1)	1·380 (3)	C(11)—C(12)	1·391 (3)
N(1)—C(4)	1·396 (3)	C(11)—C(16)	1·395 (3)
N(2)—C(17)	1·376 (3)	C(12)—C(13)	1·383 (3)
N(3)—C(8)	1·467 (3)	C(13)—C(14)	1·372 (3)
N(4)—C(14)	1·463 (3)	C(14)—C(15)	1·381 (4)
C(1)—C(2)	1·510 (3)	C(15)—C(16)	1·375 (3)

C(17)—O(7)—C(18)	115·2 (2)	C(2)—C(5)—C(6)	122·2 (2)
N(2)—N(1)—C(1)	125·0 (2)	C(2)—C(5)—C(10)	118·2 (2)
N(2)—N(1)—C(4)	122·6 (2)	C(6)—C(5)—C(10)	118·2 (2)
C(1)—N(1)—C(4)	111·8 (2)	C(5)—C(6)—C(7)	120·5 (2)
N(1)—N(2)—C(17)	115·2 (2)	C(6)—C(7)—C(8)	118·4 (2)
O(3)—N(3)—O(4)	123·2 (2)	N(3)—C(8)—C(7)	119·0 (2)
O(3)—N(3)—C(8)	118·6 (2)	N(3)—C(8)—C(9)	118·1 (2)
O(4)—N(3)—C(8)	118·2 (2)	C(7)—C(8)—C(9)	122·8 (2)
O(5)—N(4)—O(6)	123·5 (2)	C(8)—C(9)—C(10)	118·7 (2)
O(5)—N(4)—C(14)	118·0 (2)	C(5)—C(10)—C(9)	120·0 (2)
O(6)—N(4)—C(14)	118·5 (3)	C(3)—C(11)—C(12)	119·7 (2)
O(1)—C(1)—N(1)	125·7 (2)	C(3)—C(11)—C(16)	120·9 (2)
O(1)—C(1)—C(2)	128·9 (2)	C(12)—C(11)—C(16)	119·4 (2)
N(1)—C(1)—C(2)	105·4 (2)	C(11)—C(12)—C(13)	120·5 (2)
C(1)—C(2)—C(3)	108·7 (2)	C(12)—C(13)—C(14)	118·7 (2)
C(1)—C(2)—C(5)	121·7 (2)	N(4)—C(14)—C(13)	118·6 (2)
C(3)—C(2)—C(5)	129·3 (2)	N(4)—C(14)—C(15)	119·2 (2)
C(2)—C(3)—C(4)	108·3 (2)	C(13)—C(14)—C(15)	122·2 (2)
C(2)—C(3)—C(11)	131·9 (2)	C(14)—C(15)—C(16)	118·9 (2)
C(4)—C(3)—C(11)	119·8 (2)	C(11)—C(16)—C(15)	120·3 (2)
O(2)—C(4)—N(1)	124·5 (2)	O(7)—C(17)—O(8)	126·6 (2)
O(2)—C(4)—C(3)	129·9 (2)	O(7)—C(17)—N(2)	108·3 (2)
N(1)—C(4)—C(3)	105·6 (2)	O(8)—C(17)—N(2)	125·1 (2)

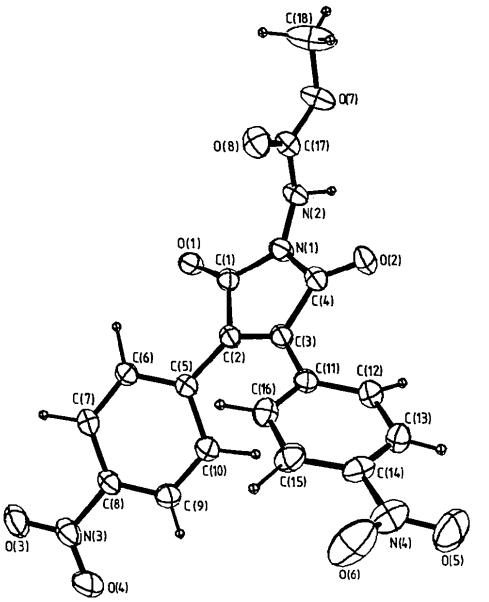

Fig. 1. Perspective view of the molecule with the labeling of the atoms (*ORTEP*II; Johnson, 1976). Thermal ellipsoids are drawn at the 50% probability level for C, N, O atoms; H atoms are spheres of arbitrary radii.

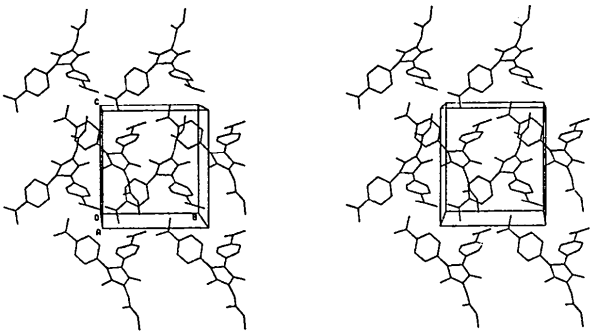

Fig. 2. Stereographic drawing (*PLUTO*; Motherwell & Clegg, 1978) of the molecular packing along the *a* axis; the H atoms have been omitted.

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