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3,4-Diethyl-2,5-dihydro-1*H*-pyrrole-2,5-dione

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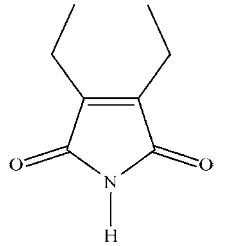
In the crystal structure of the title compound, $C_8H_{11}NO_2$, three distinct molecules are present in the asymmetric unit. The molecules are organized in two different hydrogen-bonded tapes, which form a complex layered structure. A structural comparison with the crystal structures of related maleimide derivatives unravels a stepwise evolution of morphological complexity with increasing molecular complexity for this class of compounds.

Comment

The structure of the title compound, (I), was determined as part of a study on oxidative transformations of artificial tetrapyrroles (Bröring & Hell, 2001; Bröring *et al.*, 2003, 2007). Compound (I) crystallizes in the orthorhombic system (space group $P2_12_12_1$, with $Z = 12$). The asymmetric unit contains three independent molecules (denoted *A*–*C*) in an almost coplanar arrangement and with very similar intramolecular metrics. The C_4N rings of the heterocycles are almost planar, and the peripheral ethyl groups are rotated from the maleimide subunits as quantified by dihedral angles in the range 94.8 (7)–107.2 (6)°. Both ethyl groups of each individual molecule of (I) point towards the same direction (Fig. 1).



(I)

Two N—H···O hydrogen bonds per molecule (Table 1) are present between molecules *A* and *B*, as well as between molecules *C*, resulting in the formation of parallel one-dimensional tapes either of alternating *A*–*B* or of *C* chains, respectively, propagating in the crystallographic *a* direction. Additional weak C—H···O contacts are found in both chains (C3—H3*A*···O1, C14—H14*B*···O5 and C19—H19*A*···O4), which support the tape structure and govern the orientation of one of the ethyl substituents of each molecule (Gdaniec *et al.*, 2002). The C_4N rings of molecules *A* and *B* in the *A*–*B* tapes are not fully coplanar but bent with a dihedral angle of 17.1 (3)°, so that these tapes have a convex and a concave surface, with the methyl groups from the ethyl substituents situated on the concave side. The tapes are arranged edge-to-edge in the *b* direction to form a layer. A second layer of identical tapes is arranged within the van der Waals distance in an inverted propagation direction, *i.e.* rotated around the *b* axis by 180°, so that the convex surfaces form π-stacks on each other. As a result, both concave surfaces of the [(*AB*)(*BA*)] bilayer present a pattern of methyl groups and holes.

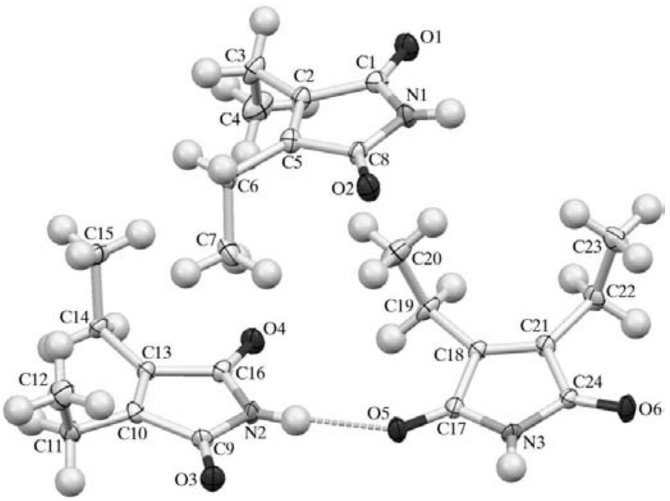


Figure 1
The crystallographically distinct molecules of (I) (N2: molecule *A*; N3: molecule *B*; N1: molecule *C*), showing the atom-numbering scheme. The dashed line indicates the intermolecular N2—H2···O5 hydrogen bond. Displacement ellipsoids are drawn at the 50% probability level.

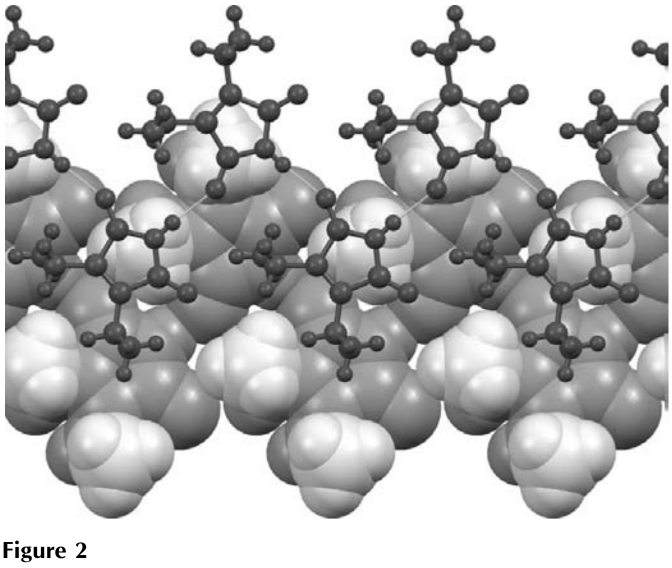


Figure 2
The organization of the one-dimensional chains, showing the stacking of a (*CC*) tape on to the surface of a (*BA*) tape.

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The *C* tapes are different in that the individual molecules bind in a conformation with the pairs of ethyl groups alternately oriented to either side of the tape. As before, these tapes pack edge-to-edge and form another layer. This *C*-derived layer is situated on top of the $[(AB)(BA)]$ bilayer so that the extending methyl groups fill the holes in the surface of the $[(AB)(BA)]$ layer (Fig. 2). The whole crystal constitutes $[(AB)(BA)(CC)]$ trilayers stacked on top of one another and alternately rotated by 180° around the crystallographic *c* axis, so that the complete description of repeating layers in the *c* direction is $[(AB)(BA)(CC)(AB)'(BA)'(CC)']$ (Fig. 3).

The structures of two related compounds, *viz.* maleimide (Cox & Parker, 1996) and tetrahydrophthalimide (Kirfel, 1975), have been reported previously. The lower degree of molecular complexity of these compounds with respect to (I) is reflected in their crystal structures. Tetrahydrophthalimide forms one-dimensional chains *via* N—H···O hydrogen bonds between individual molecules, very similar to the tapes formed by (I). The major difference occurs from the orientation of the alkyl groups, which are essentially coplanar for tetrahydrophthalimide and do not produce patterned surfaces of the tapes. In addition, only one crystallographically distinct (disordered) molecule is found, so that as a result only one type of tape is formed and organized into a simple π-stacked layer structure. Maleimide is devoid of alkyl substituents. The asymmetric unit comprises four distinct molecules *A*–*D*, and these form doubly hydrogen-bonded dimers, *viz.* *A*–*B* and *C*–*D*. These dimers then pack mainly by π-stacking interactions.

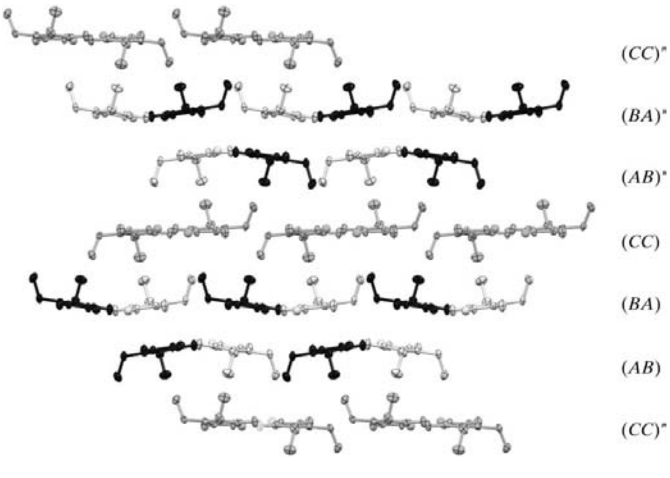

Figure 3
The overall layer structure of (I) viewed along the *a* axis.

Experimental

Compound (I) forms as an oxidation product from porphyrins (Martin *et al.*, 1980; Schaefer *et al.*, 1985; Bonnett & Chaney, 1987; Pancost *et al.*, 2002), bile pigments (Landen *et al.*, 1983) and pyrroles (Quistad & Lightner, 1971; Schoenleber *et al.*, 1984), usually upon photooxidation. ^{1}H NMR (300 MHz, CDCl$_3$): δ 7.07 (*br s*, 1H), 2.41 (*q*, J = 7.6 Hz, 4H), 1.15 (*t*, J = 7.6 Hz, 6H). Crystals suitable for X-ray diffraction were grown by slow evaporation of a solution of (I) in dichloromethane/hexane.

Crystal data

$C_8H_{11}NO_2$
$M_r = 153.18$
Orthorhombic, $P2_12_12_1$
$a = 7.9602\ (13)$ Å
$b = 11.6135\ (18)$ Å
$c = 26.586\ (5)$ Å
$V = 2457.7\ (7)$ Å^3
$Z = 12$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 193\ (2)$ K
0.41 × 0.19 × 0.05 mm

Data collection

Stoe IPDSI diffractometer
Absorption correction: multi-scan (Blessing, 1995)
$T_{\min} = 0.876$, $T_{\max} = 0.998$
8425 measured reflections
2593 independent reflections
1381 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.091$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.108$
$S = 0.80$
2593 reflections
304 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1$^{\rm i}$	0.88	1.98	2.851 (4)	172
N2—H2···O5	0.88	2.03	2.901 (4)	173
N3—H3···O4$^{\rm ii}$	0.88	2.00	2.872 (4)	169

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

All H atoms were placed in idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.98–0.99 Å and N—H distances of 0.88 Å. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *EXPOSE* in *IPDS* (Stoe & Cie, 1999); cell refinement: *CELL* in *IPDS*; data reduction: *INTEGRATE* in *IPDS*; program(s) used to solve structure: *SIR92* (Giacovazzo, 1992); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *MERCURY* (Version 1.4.1; Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV3071). Services for accessing these data are described at the back of the journal.

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