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Bis(4-maleimidophenyl)methane

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In the crystal structure of the title compound, $C_{21}H_{14}N_2O_4$, the asymmetric unit contains one-half of the molecule, with the other half generated by a crystallographic twofold axis; the central C atom lies on the twofold axis. The dihedral angle between the substituted phenyl and pyrrole rings is 52.1 (1)°. The molecular packing in the crystal is stabilized by π–π-stacking interactions and weak C—H$\cdots\pi$ interactions.

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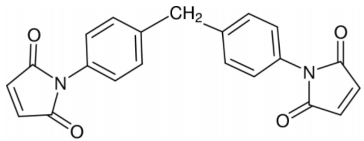
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.091
Data-to-parameter ratio = 9.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The title compound, (I), is an important kind of bismaleimide (BMI). As classical high-performance thermosetting polyimides, BMI resins have been widely studied. BMI resins are attractive because of their high chemical, corrosion and radiation resistance, as well as their attrition-enduring, insulating, and mechanical properties (Jin *et al.*, 2001; Glatz & Mulhaupt, 1993; Gawdzik *et al.*, 2001). However, the most attractive property is that they have excellent hot/wet stability up to 573–623 K (Gawdzik *et al.*, 2001). Therefore, BMI resins have been widely used for advanced composite materials, multi-layered lamination materials, abrasive materials, sealing materials, molding materials, powder coating, adhesives, *etc.* (Jin *et al.*, 2001; Glatz & Mulhaupt, 1993; Gawdzik *et al.*, 2001). We report here the crystal structure of (I).



(I)

The asymmetric unit of (I) contains one-half of the molecule (Fig. 1), with the other half generated by a crystallographic twofold axis passing through atom C11. The bond lengths and angles of the phenylmaleimide moiety are comparable with those observed in *N*-(4-hydroxyphenyl)maleimide (Rodriguez *et al.*, 2002). The dihedral angle between the substituted phenyl and pyrrole rings is 52.1 (1)°. The torsion angles C4—N1—C5—C6 and C1—N1—C5—C10 are −53.4 (3) and −51.4 (3)°, respectively. Atom O1 lies in the pyrrole plane, whereas O2 deviates from it by 0.027 (2) Å. In the molecule, the dihedral angle between the symmetry-related phenyl rings bridged by C11 is 76.59 (4)°. The symmetry-related pyrrole rings are nearly orthogonal, with a dihedral angle of 83.74 (6)°. The end-to-end distance of the molecule is 13.54 Å.

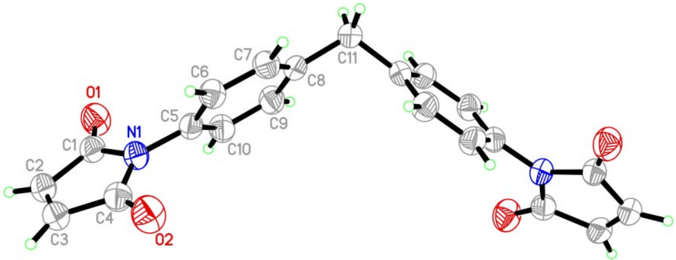


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme for one asymmetric unit. The other half of the molecule is generated by a twofold axis through C11.

The molecular packing in the crystal is stabilized by π–π-stacking interactions and C—H···π interactions. The maleimide moiety and phenyl ring of the symmetry-related molecule at (y, $\frac{1}{2} - x$, $\frac{1}{4} + z$) are stacked with their centroids separated by a distance of 3.484 (1) Å. A weak C—H···π interaction involving C9 and the maleimide moiety is observed, such that H9···*Cg* = 3.01 Å, C9···*Cg* = 3.750 (2) Å and C9—H9···*Cg* = 138°, where *Cg* is the centre of gravity of the pyrrole ring at ($-\frac{1}{2} + x$, $\frac{1}{2} - y$, z).

Experimental

An acetone solution (15 ml) of maleic anhydride (2 mmol, 196 mg) and 4,4′-diaminophenylmethane (1 mmol, 198 mg) was stirred for 1 h, then acetic anhydride (5 ml), anhydrous magnesium acetate (0.2 g) and triethylamine (5 ml) were added. The resultant solution was heated to 353 K and allowed to stand for 1 h. The solution was then cooled to room temperature. On addition of water, shiny yellow crystals were deposited, filtered and washed with water and acetone in turn. Recrystallization was carried out from ethanol; the crystals were dried in a vacuum desiccator over $CaCl_2$ (yield 82%). Analysis calculated for the title complex: C 70.39, H 3.94, N 7.82%; found: C 70.15, H 4.00, N 7.68%.

Crystal data

$C_{21}H_{14}N_2O_4$
$M_r = 358.34$
Tetragonal, $I4_1cd$
$a = 12.9880$ (4) Å
$c = 20.9435$ (12) Å
$V = 3532.9$ (3) Å^3
$Z = 8$
$D_x = 1.347$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 7096 reflections
$\theta = 3.0$–28.3°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.50 × 0.50 × 0.44 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.917$, $T_{max} = 0.959$
10396 measured reflections
1133 independent reflections
1074 reflections with $I > 2\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 28.3°$
$h = -16 \rightarrow 17$
$k = -17 \rightarrow 16$
$l = -27 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.032$
$wR(F^2) = 0.092$
$S = 1.08$
1133 reflections
122 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0615P)^2 + 0.5468P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.203 (2)	C1—C2	1.494 (3)
O2—C4	1.208 (2)	C2—C3	1.311 (3)
N1—C4	1.398 (2)	C3—C4	1.493 (3)
N1—C1	1.405 (2)	C8—C11	1.520 (2)
N1—C5	1.429 (2)		
C4—N1—C1	109.78 (15)	N1—C1—C2	105.70 (16)
C4—N1—C5	125.65 (15)	O2—C4—N1	125.45 (18)
C1—N1—C5	124.56 (16)	O2—C4—C3	128.41 (19)
O1—C1—N1	125.37 (17)	N1—C4—C3	106.14 (15)
O1—C1—C2	128.92 (17)	C8^i—C11—C8	113.04 (19)

Symmetry code: (i) $-x$, $-y$, z.

All H atoms, except for H11 attached to C11, were positioned geometrically and treated as riding atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$. H11 was located from a difference Fourier map and it was allowed to ride on the attached atom during the final cycles of refinement. Due to the absence of any significant anomalous scattering, the 631 Friedel opposites were merged.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 1990).

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