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2,2′-Methylenebis(isoindoline-1,3-dione)

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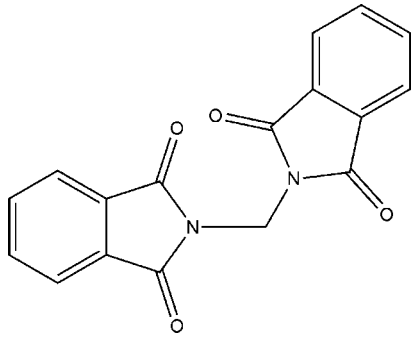
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.124; data-to-parameter ratio = 12.8.

The title compound, $C_{17}H_{10}N_2O_4$, consists of two phthalimide units connected by a methylene bridge. The N—C—N bond angle is 110.64 (12)°. In the crystal structure, the dihedral angle between the two phthalimide units in one molecule is 88.96 (2)°. The crystal packing is stabilized by the π–π overlap of neighboring phthalimide units, with closest interplanar packing distances of 3.470 (1) and 3.626 (7) Å, and by weak C—H···O interactions.

Related literature

For related literature, see: Orzeszko *et al.* (2000).



Experimental

Crystal data

$C_{17}H_{10}N_2O_4$
M_r = 306.27
Triclinic, $P\overline{1}$
a = 7.6660 (9) Å
b = 9.5810 (8) Å
c = 10.2780 (6) Å
α = 104.325 (3)°
β = 99.768 (4)°
γ = 96.030 (3)°
V = 712.23 (11) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.80 × 0.21 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID imaging plate system diffractometer
Absorption correction: none
6295 measured reflections
3181 independent reflections
2604 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.124
S = 1.05
3181 reflections
248 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H10···O1^{i}	0.98 (2)	2.49 (2)	3.273 (2)	136.3 (16)
C9—H5···O2^{ii}	1.001 (16)	2.508 (16)	3.4152 (18)	150.6 (12)
C5—H4···O2^{iii}	1.008 (19)	2.50 (2)	3.3480 (19)	141.6 (14)
C2—H1···O4^{iv}	0.97 (2)	2.58 (2)	3.205 (2)	121.9 (15)

Symmetry codes: (i) $x, y, z-1$; (ii) $-x, -y, -z+1$; (iii) $-x+1, -y, -z+1$; (iv) $-x+1, -y+1, -z+2$.

Data collection: *RAPID-AUTO* (Rigaku, 2006); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BV2068).

References

McArdle, P. (1995). *J. Appl. Cryst.* **28**, 65.
Orzeszko, A., Kaminska, B., Orzesko, G. & Starosciak, J. (2000). *Farmaco*, **55**, 619–623.
Rigaku (2006). *RAPID-AUTO*. Version 3.0. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

supplementary materials

Acta Cryst. (2007). E**63**, o4385 [**doi:10.1107/S1600536807050738**]

2,2'-Methylenebis(isoindoline-1,3-dione)

Z. Jiang, J.-D. Wang, M.-J. Lin, N.-S. Chen and J.-L. Huang

Comment

N-substituted phthalimides possess important biological activity (Orzeszko *et al.*, 2000). The title compound (I) featuring two phthlimide moieties was synthesized by the reaction of phthalimide with polyformaldehyde in concentrated sulfuric acid. The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the two phthalimides planes is 91.876°. In the crystal structure of (I), the molecules pack in columns through π-π interactions between the phthalimide moieties with distances of 3.470 (1)Å and 3.626 (7) Å, and slip distances of 1.47Å and 1.62 Å, respectivly. There are weak C—H···O interactions between these columns (Table 1).

Experimental

Under N_2 atmosphere, cold water bath and stirring, phthalimide (8.8 g) was dissolved in concentrated sulfuric acid (50 ml) followed by the careful addition polyformaldehyde (0.5 g). After the solid disappeared, the solution was warmed to 323 K and stirred for 10 h, then cooled to room temperature, and poured into ice-water with vigorous stirring. The filter cake was washed to be neutral and then dried. The product was purified by chromatography on a silica gel column with alcohol-hexane (V/V=3/7) as eluent. Single crystals of (I) were obtained by slow evaporation from its dichloroethene solution at room temperature.

IR(KBr, ν, cm^{-1}): 3115,3025 (Ar—H), 2968 (C—H), 1731(C=O), 1710 (C=O),1464, 1431, 950, 734. ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 7.860–7.877 (m, 4H), 7.721–7.738(m, 4H), 5.459 (s, 2H).

Refinement

The H atoms were located in an electron-density difference map and refined isotropically.

Figures

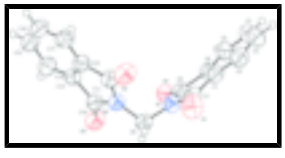


Fig. 1. The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

2,2'-Methylenebis(isoindoline-1,3-dione)

Crystal data

$C_{17}H_{10}N_2O_4$	$Z = 2$
$M_r = 306.27$	$F_{000} = 316$

Triclinic, $P\bar{1}$	D_x = 1.428 Mg m^{-3}
Hall symbol: -P 1	Melting point: 501.6-502.1 K
a = 7.6660 (9) Å	Mo $K\alpha$ radiation λ = 0.71069 Å
b = 9.5810 (8) Å	Cell parameters from 2784 reflections
c = 10.2780 (6) Å	θ = 5.2–54.6º
α = 104.325 (3)º	μ = 0.10 mm^{-1}
β = 99.768 (4)º	T = 298 (2) K
γ = 96.030 (3)º	Prism, colourless
V = 712.23 (11) Å^3	0.80 × 0.21 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID imaging plate system diffractometer	3181 independent reflections
Radiation source: Rigaku rotating anode generator	2604 reflections with $I > 2\sigma(I)$
Monochromator: Graphite Monochromator	R_{int} = 0.026
Detector resolution: 14.6306 pixels mm^{-1}	θ_{max} = 27.5º
T = 298(2) K	θ_{min} = 2.2º
ω scans	h = −9→9
Absorption correction: none	k = 0→12
6295 measured reflections	l = −13→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.046	All H-atom parameters refined
$wR(F^2)$ = 0.124	$w = 1/[\sigma^2(F_o^2) + (0.0625P)^2 + 0.1105P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.05	$(\Delta/\sigma)_{max}$ = 0.003
3181 reflections	$\Delta\rho_{max}$ = 0.14 e Å^{-3}
248 parameters	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-

ing *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.22177 (16)	0.16328 (12)	0.73250 (11)	0.0486 (3)
N2	0.11889 (15)	0.29986 (12)	0.57720 (11)	0.0485 (3)
O1	0.2051 (2)	0.28638 (15)	0.95249 (12)	0.0804 (4)
O2	0.30930 (14)	0.01722 (12)	0.54975 (9)	0.0583 (3)
O3	0.02017 (17)	0.14751 (12)	0.35746 (11)	0.0655 (3)
O4	0.24441 (19)	0.49922 (13)	0.75855 (12)	0.0765 (4)
H1	0.557 (3)	0.256 (2)	1.104 (2)	0.084 (6)*
H2	0.833 (3)	0.151 (2)	1.097 (2)	0.104 (7)*
H3	0.877 (3)	0.000 (2)	0.896 (2)	0.091 (6)*
H4	0.662 (2)	−0.039 (2)	0.683 (2)	0.073 (5)*
H5	−0.011 (2)	0.1138 (17)	0.5916 (16)	0.056 (4)*
H6	0.001 (2)	0.2565 (16)	0.7257 (16)	0.051 (4)*
H7	0.374 (3)	0.715 (2)	0.615 (2)	0.089 (6)*
H8	0.395 (3)	0.753 (2)	0.400 (2)	0.091 (6)*
H9	0.300 (3)	0.573 (2)	0.198 (2)	0.088 (6)*
H10	0.153 (3)	0.339 (2)	0.191 (2)	0.085 (6)*
C1	0.4607 (2)	0.16588 (16)	0.90265 (14)	0.0541 (4)
C2	0.5866 (3)	0.1948 (2)	1.02293 (17)	0.0709 (5)
C3	0.7425 (3)	0.1341 (2)	1.0166 (2)	0.0809 (6)
C4	0.7714 (3)	0.0484 (2)	0.8956 (2)	0.0766 (5)
C5	0.6438 (2)	0.0193 (2)	0.77442 (17)	0.0626 (4)
C6	0.49014 (19)	0.08017 (15)	0.78128 (13)	0.0497 (3)
C7	0.2845 (2)	0.21708 (16)	0.87470 (14)	0.0559 (4)
C8	0.33557 (18)	0.07717 (14)	0.67038 (13)	0.0465 (3)
C9	0.0657 (2)	0.20282 (16)	0.65709 (15)	0.0508 (3)
C10	0.09578 (19)	0.26223 (15)	0.43474 (14)	0.0495 (3)
C11	0.2083 (2)	0.44257 (15)	0.63763 (15)	0.0522 (3)
C12	0.24512 (19)	0.50100 (15)	0.52299 (15)	0.0520 (3)
C13	0.18072 (19)	0.39313 (16)	0.40180 (15)	0.0512 (3)
C14	0.1987 (2)	0.4151 (2)	0.27754 (18)	0.0660 (4)
C15	0.2840 (3)	0.5515 (2)	0.2794 (2)	0.0780 (5)
C16	0.3456 (3)	0.6596 (2)	0.4003 (2)	0.0762 (5)
C17	0.3282 (2)	0.63641 (18)	0.5248 (2)	0.0666 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0567 (6)	0.0478 (6)	0.0386 (5)	0.0036 (5)	0.0095 (5)	0.0085 (5)
N2	0.0555 (6)	0.0429 (6)	0.0433 (6)	0.0021 (5)	0.0080 (5)	0.0086 (5)
O1	0.1052 (10)	0.0882 (9)	0.0477 (6)	0.0289 (8)	0.0253 (6)	0.0058 (6)
O2	0.0602 (6)	0.0692 (7)	0.0375 (5)	0.0045 (5)	0.0081 (4)	0.0030 (4)
O3	0.0829 (8)	0.0533 (6)	0.0487 (6)	−0.0064 (5)	0.0062 (5)	0.0048 (5)

O4	0.1027 (10)	0.0600 (7)	0.0521 (6)	−0.0079 (6)	0.0126 (6)	−0.0015 (5)
C1	0.0666 (9)	0.0507 (8)	0.0398 (6)	−0.0039 (6)	0.0053 (6)	0.0116 (5)
C2	0.0866 (12)	0.0694 (11)	0.0434 (8)	−0.0049 (9)	−0.0025 (8)	0.0085 (7)
C3	0.0744 (12)	0.0933 (14)	0.0611 (10)	−0.0078 (10)	−0.0166 (9)	0.0241 (10)
C4	0.0614 (10)	0.0969 (14)	0.0711 (11)	0.0071 (9)	0.0022 (9)	0.0315 (10)
C5	0.0600 (9)	0.0741 (10)	0.0542 (9)	0.0072 (7)	0.0099 (7)	0.0210 (8)
C6	0.0543 (7)	0.0512 (7)	0.0410 (7)	−0.0017 (6)	0.0075 (6)	0.0136 (5)
C7	0.0742 (10)	0.0518 (8)	0.0384 (7)	0.0030 (7)	0.0134 (7)	0.0078 (6)
C8	0.0519 (7)	0.0458 (7)	0.0389 (6)	−0.0031 (5)	0.0102 (6)	0.0101 (5)
C9	0.0533 (8)	0.0502 (8)	0.0485 (7)	0.0041 (6)	0.0126 (6)	0.0129 (6)
C10	0.0516 (7)	0.0490 (7)	0.0453 (7)	0.0057 (6)	0.0089 (6)	0.0092 (6)
C11	0.0563 (8)	0.0433 (7)	0.0522 (8)	0.0050 (6)	0.0093 (6)	0.0062 (6)
C12	0.0499 (7)	0.0465 (7)	0.0599 (8)	0.0085 (6)	0.0119 (6)	0.0142 (6)
C13	0.0498 (7)	0.0531 (8)	0.0527 (8)	0.0089 (6)	0.0119 (6)	0.0167 (6)
C14	0.0701 (10)	0.0752 (11)	0.0583 (9)	0.0095 (8)	0.0183 (8)	0.0255 (8)
C15	0.0787 (12)	0.0930 (14)	0.0835 (13)	0.0173 (10)	0.0309 (11)	0.0513 (12)
C16	0.0712 (11)	0.0650 (11)	0.1051 (15)	0.0064 (8)	0.0282 (11)	0.0411 (11)
C17	0.0631 (9)	0.0516 (9)	0.0849 (12)	0.0021 (7)	0.0155 (9)	0.0206 (8)

Geometric parameters (Å, °)

N1—C8	1.3889 (18)	C4—H3	0.97 (2)
N1—C7	1.4035 (17)	C5—C6	1.373 (2)
N1—C9	1.4496 (19)	C5—H4	1.008 (19)
N2—C10	1.3938 (17)	C6—C8	1.491 (2)
N2—C11	1.4011 (18)	C9—H5	1.001 (16)
N2—C9	1.4549 (18)	C9—H6	0.999 (16)
O1—C7	1.1991 (19)	C10—C13	1.492 (2)
O2—C8	1.2025 (16)	C11—C12	1.479 (2)
O3—C10	1.2053 (17)	C12—C17	1.378 (2)
O4—C11	1.1974 (18)	C12—C13	1.382 (2)
C1—C2	1.381 (2)	C13—C14	1.372 (2)
C1—C6	1.382 (2)	C14—C15	1.393 (3)
C1—C7	1.491 (2)	C14—H10	0.98 (2)
C2—C3	1.387 (3)	C15—C16	1.379 (3)
C2—H1	0.97 (2)	C15—H9	0.93 (2)
C3—C4	1.378 (3)	C16—C17	1.377 (3)
C3—H2	0.95 (2)	C16—H8	0.93 (2)
C4—C5	1.394 (2)	C17—H7	1.01 (2)
C8—N1—C7	111.95 (12)	N1—C9—N2	110.66 (11)
C8—N1—C9	123.56 (11)	N1—C9—H5	110.2 (9)
C7—N1—C9	124.28 (12)	N2—C9—H5	107.6 (9)
C10—N2—C11	112.04 (12)	N1—C9—H6	107.4 (9)
C10—N2—C9	125.16 (11)	N2—C9—H6	108.1 (9)
C11—N2—C9	122.74 (12)	H5—C9—H6	112.8 (13)
C2—C1—C6	120.95 (16)	O3—C10—N2	125.80 (13)
C2—C1—C7	130.36 (15)	O3—C10—C13	128.80 (13)
C6—C1—C7	108.67 (12)	N2—C10—C13	105.39 (11)
C1—C2—C3	117.31 (17)	O4—C11—N2	124.40 (14)

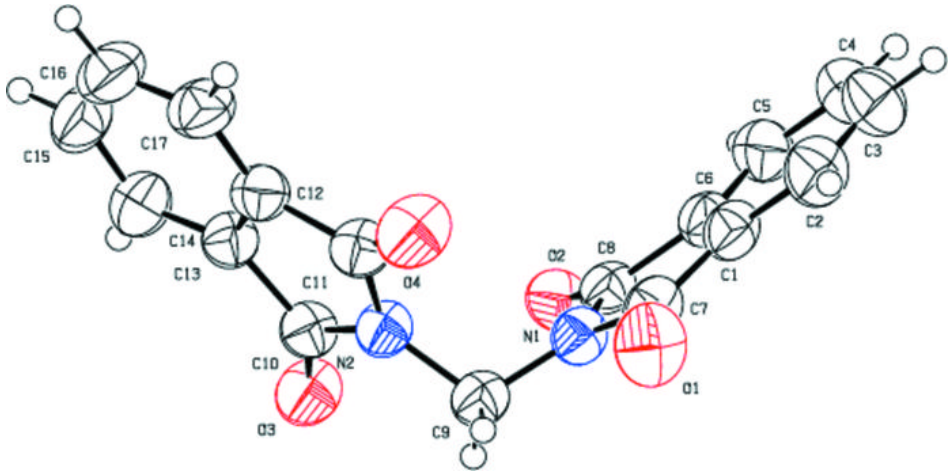
C1—C2—H1	116.6 (12)	O4—C11—C12	129.76 (14)
C3—C2—H1	126.1 (12)	N2—C11—C12	105.85 (12)
C4—C3—C2	121.65 (17)	C17—C12—C13	121.72 (15)
C4—C3—H2	117.8 (14)	C17—C12—C11	129.99 (14)
C2—C3—H2	120.5 (14)	C13—C12—C11	108.29 (12)
C3—C4—C5	120.85 (19)	C14—C13—C12	121.53 (15)
C3—C4—H3	119.8 (13)	C14—C13—C10	130.06 (14)
C5—C4—H3	119.1 (13)	C12—C13—C10	108.41 (13)
C6—C5—C4	117.20 (17)	C13—C14—C15	116.79 (18)
C6—C5—H4	119.5 (11)	C13—C14—H10	121.5 (12)
C4—C5—H4	123.2 (11)	C15—C14—H10	121.7 (12)
C5—C6—C1	122.04 (14)	C16—C15—C14	121.50 (18)
C5—C6—C8	130.12 (13)	C16—C15—H9	117.8 (13)
C1—C6—C8	107.77 (13)	C14—C15—H9	120.7 (13)
O1—C7—N1	124.82 (16)	C17—C16—C15	121.38 (18)
O1—C7—C1	129.90 (14)	C17—C16—H8	117.5 (14)
N1—C7—C1	105.26 (12)	C15—C16—H8	121.0 (13)
O2—C8—N1	124.37 (13)	C16—C17—C12	117.07 (17)
O2—C8—C6	129.37 (14)	C16—C17—H7	122.1 (12)
N1—C8—C6	106.24 (11)	C12—C17—H7	120.8 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H10···O1^{i}	0.98 (2)	2.49 (2)	3.273 (2)	136.3 (16)
C9—H5···O2^{ii}	1.001 (16)	2.508 (16)	3.4152 (18)	150.6 (12)
C5—H4···O2^{iii}	1.008 (19)	2.50 (2)	3.3480 (19)	141.6 (14)
C2—H1···O4^{iv}	0.97 (2)	2.58 (2)	3.205 (2)	121.9 (15)

Symmetry codes: (i) *x*, *y*, *z*−1; (ii) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+2.

Fig. 1


C16
C15
C17
C12
C14
C13
C11
O4
C10
N2
O3
C9
O2
C8
N1
C7
O1
C1
C6
C5
C4
C3
C2