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N,*N*′-Dicyclohexylnaphthalene-1,8;4:5-dicarboximide

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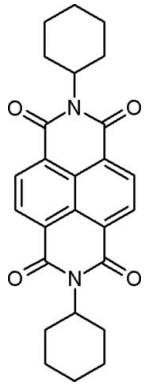
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.067; wR factor = 0.183; data-to-parameter ratio = 13.5.

The title compound, $C_{26}H_{26}N_2O_4$, synthesized by the reaction of naphthalene-1,4,5,8-tetracarboxylic acid anhydride and cyclohexylamine, exhibits good *n*-type semiconducting properties. Accordingly, thin-film transistor devices comprising this compound show *n*-type behavior with high field-effect electron moblity *ca* 6 cm^2/Vs [Shukla, Nelson, Freeman, Rajeswaran, Ahearn, Meyer & Carey(2008). *Chem. Mater.* Submitted]. The asymmetric unit comprises one-quarter of the centrosymmetric molecule in which all but two methylene C atoms of the cyclohexane ring lie on a mirror plane; the point-group symmetry is 2/*m*. The naphthalenediimide unit is strictly planar, and the cyclohexane rings adopt chair conformations with the diimide unit in an equatorial position on each ring.

Related literature

For general background on the semi-conducting properties and use of this class of material in organic thin-film transistor applications, see: Chesterfield *et al.* (2004*a*,*b*); Facceti *et al.* (2008); Jones *et al.* (2004); Katz *et al.* (2000*a*,*b*); Shukla *et al.* (2008).



Experimental

Crystal data

$C_{26}H_{26}N_2O_4$
M_r = 430.49
Monoclinic, $C2/m$
a = 8.5410 (2) Å
b = 6.6780 (2) Å
c = 18.4270 (9) Å
β = 102.4790 (18)°
V = 1026.19 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.35 × 0.25 × 0.17 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: none
3354 measured reflections
1227 independent reflections
787 reflections with $I > 2\sigma(I)$
R_{int} = 0.087

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.182
S = 1.06
1227 reflections
91 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2528).

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N,N′-Dicyclohexylnaphthalene-1,8;4:5-dicarboximide

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S1. Comment

Amongst n-type semiconductors, naphthalene diimide (NDI) and perylene diimide (PDI) based systems have been studied extensively (Chesterfield, *et al.*, 2004*a*; Chesterfield *et al.*, 2004*b*; Facceti *et al.*, 2008; Jones, *et al.*, 2004; Katz, *et al.*, 2000*a*; Katz, *et al.*, 2000*b*). We report here the structure of the title diimide molecule, I, (Fig. 1).

S2. Experimental

The diimide 1 was prepared by direct condensation of 1,4,5,8-naphthalenetetracarboxylic acid anhydride (1.34 g, 5.00 mmol) and cyclohexylamine (30 mmol) in the presence of zinc acetate (50 mg) in 15 mL quinoline. The mixture was heated at 140-150°C for four hours, cooled and diluted with several volumes of methanol. The resulting slurry was filtered, the collected solid washed with methanol and dried in air. The crude product was then purified by train sublimation at 10^{-4} to 10^{-6} torr. ^{1}H NMR (CD_2Cl_2,500.05 MHz): δ (ppm) = 8.76 (s, 4H), 5.10 (t,2H, J = 12 Hz), 2.64 (dt, 2H, J = 12 and 11.7 Hzs), 1.57 (dt, 2H, J = 12 and11.7 Hz), 2.03 (d, 2H, J = 12 Hz), 1.87 (d, 2H, J = 12 Hz), 1.47 (m, 2H); ^{13}C(CD_2Cl_2, 500.05 MHz): d = 163.23, 130.74, 127.13, 126.70,54.85, 29.38, 26.66, 25.52; MS (MALDI-TOF) *m/z* cald. for [$C_{26}H_{26}N_2O_4$]430.5 found: 430.2.

S3. Refinement

All H-atoms were positioned geometrically using a riding model with d(C-H) = 0.93Å, U_{iso}=1.2U_{eq} (C) for aromatic 0.97Å, U_{iso} = 1.2U_{eq} (C) for CH_2 atoms.

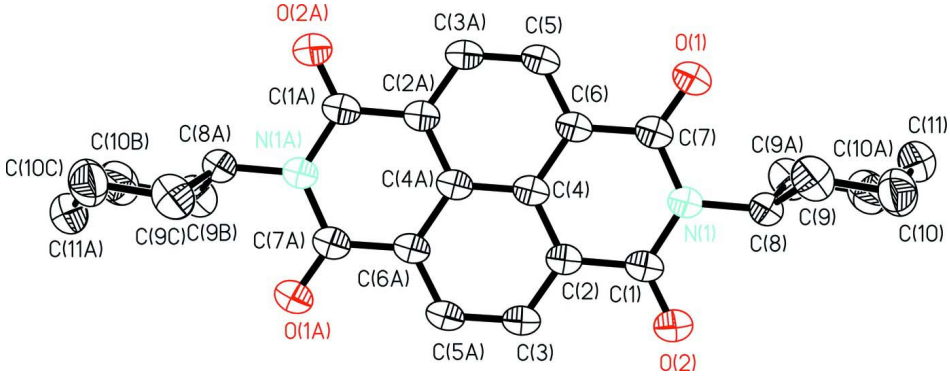

Figure 1

Structure of the title compound (I), with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are omitted for clarity.

N,*N*′-dicyclohexylnaphthalene-1,8;4:5-dicarboximide

Crystal data

$C_{26}H_{26}N_2O_4$
$M_r = 430.49$
Monoclinic, *C*2/*m*
Hall symbol: -C 2y
$a = 8.5410$ (2) Å
$b = 6.6780$ (2) Å
$c = 18.4270$ (9) Å
$\beta = 102.4790$ (18)°
$V = 1026.19$ (6) Å^3
$Z = 2$
$F(000) = 456$
$D_x = 1.393$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 21067 reflections
$\theta = 1.0$–$27.5°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Block, orange
0.35 × 0.25 × 0.17 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
φ and ω scans
3354 measured reflections
1227 independent reflections
787 reflections with $I > 2\sigma(I)$
$R_{int} = 0.087$
$\theta_{max} = 27.4°$, $\theta_{min} = 4.3°$
$h = -10 \rightarrow 10$
$k = -8 \rightarrow 8$
$l = -23 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.067$
$wR(F^2) = 0.182$
$S = 1.06$
1227 reflections
91 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2 + 1.0546P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ is used only for calculating *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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2160 (3)	0.0000	0.28476 (12)	0.0594 (8)
O2	0.7630 (3)	0.0000	0.33332 (13)	0.0647 (8)
N1	0.4894 (3)	0.0000	0.30625 (13)	0.0431 (7)
C1	0.6391 (4)	0.0000	0.35566 (18)	0.0457 (8)
C2	0.6418 (3)	0.0000	0.43622 (17)	0.0422 (8)

C3	0.7854 (4)	0.0000	0.48703 (18)	0.0510 (9)
H3	0.8806	0.0000	0.4703	0.061*
C4	0.4979 (3)	0.0000	0.46157 (16)	0.0384 (7)
C5	0.2086 (4)	0.0000	0.43639 (18)	0.0494 (9)
H5	0.1100	0.0000	0.4030	0.059*
C6	0.3486 (3)	0.0000	0.41020 (17)	0.0410 (7)
C7	0.3427 (4)	0.0000	0.32968 (18)	0.0446 (8)
C8	0.4868 (4)	0.0000	0.22507 (16)	0.0460 (8)
H8	0.5991	0.0000	0.2207	0.055*
C9	0.4125 (3)	0.1896 (4)	0.18665 (13)	0.0591 (7)
H9A	0.4684	0.3060	0.2110	0.071*
H9B	0.3010	0.1986	0.1901	0.071*
C10	0.4238 (3)	0.1862 (5)	0.10529 (14)	0.0708 (9)
H10A	0.3699	0.3031	0.0803	0.085*
H10B	0.5356	0.1926	0.1022	0.085*
C11	0.3488 (5)	0.0000	0.0665 (2)	0.0664 (11)
H11A	0.3624	0.0000	0.0156	0.080*
H11B	0.2348	0.0000	0.0654	0.080*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0365 (12)	0.088 (2)	0.0494 (14)	0.000	0.0005 (9)	0.000
O2	0.0363 (12)	0.105 (2)	0.0535 (14)	0.000	0.0113 (10)	0.000
N1	0.0330 (12)	0.0522 (17)	0.0428 (14)	0.000	0.0052 (10)	0.000
C1	0.0323 (15)	0.051 (2)	0.0514 (19)	0.000	0.0047 (12)	0.000
C2	0.0322 (15)	0.0478 (19)	0.0456 (18)	0.000	0.0066 (12)	0.000
C3	0.0296 (15)	0.071 (2)	0.0513 (19)	0.000	0.0072 (12)	0.000
C4	0.0309 (14)	0.0364 (16)	0.0459 (16)	0.000	0.0040 (11)	0.000
C5	0.0292 (14)	0.065 (2)	0.0503 (19)	0.000	0.0012 (12)	0.000
C6	0.0303 (15)	0.0445 (18)	0.0460 (17)	0.000	0.0033 (12)	0.000
C7	0.0357 (15)	0.0466 (19)	0.0491 (18)	0.000	0.0038 (13)	0.000
C8	0.0379 (15)	0.059 (2)	0.0403 (17)	0.000	0.0059 (12)	0.000
C9	0.0643 (15)	0.0483 (15)	0.0601 (16)	−0.0050 (13)	0.0033 (11)	0.0031 (12)
C10	0.0716 (17)	0.080 (2)	0.0561 (16)	−0.0103 (17)	0.0045 (12)	0.0170 (15)
C11	0.056 (2)	0.094 (3)	0.047 (2)	0.000	0.0059 (16)	0.000

Geometric parameters (Å, º)

O1—C7	1.212 (3)	C5—H5	0.9300
O2—C1	1.216 (4)	C6—C7	1.474 (4)
N1—C1	1.401 (4)	C8—C9^{ii}	1.521 (3)
N1—C7	1.411 (4)	C8—C9	1.521 (3)
N1—C8	1.491 (4)	C8—H8	0.9800
C1—C2	1.480 (4)	C9—C10	1.523 (3)
C2—C3	1.373 (4)	C9—H9A	0.9700
C2—C4	1.406 (4)	C9—H9B	0.9700
C3—C5^{i}	1.401 (4)	C10—C11	1.506 (4)

C3—H3	0.9300	C10—H10A	0.9700
C4—C4^{i}	1.409 (6)	C10—H10B	0.9700
C4—C6	1.415 (4)	C11—C10^{ii}	1.506 (4)
C5—C6	1.382 (4)	C11—H11A	0.9700
C5—C3^{i}	1.401 (4)	C11—H11B	0.9700
C1—N1—C7	123.2 (3)	N1—C8—C9^{ii}	112.42 (17)
C1—N1—C8	117.8 (3)	N1—C8—C9	112.42 (17)
C7—N1—C8	119.0 (2)	C9^{ii}—C8—C9	112.7 (3)
O2—C1—N1	121.3 (3)	N1—C8—H8	106.2
O2—C1—C2	120.9 (3)	C9^{ii}—C8—H8	106.2
N1—C1—C2	117.8 (3)	C9—C8—H8	106.2
C3—C2—C4	119.3 (3)	C8—C9—C10	109.7 (2)
C3—C2—C1	120.1 (3)	C8—C9—H9A	109.7
C4—C2—C1	120.5 (3)	C10—C9—H9A	109.7
C2—C3—C5^{i}	121.3 (3)	C8—C9—H9B	109.7
C2—C3—H3	119.3	C10—C9—H9B	109.7
C5^{i}—C3—H3	119.3	H9A—C9—H9B	108.2
C2—C4—C4^{i}	120.0 (3)	C11—C10—C9	111.6 (3)
C2—C4—C6	120.3 (3)	C11—C10—H10A	109.3
C4^{i}—C4—C6	119.7 (3)	C9—C10—H10A	109.3
C6—C5—C3^{i}	120.4 (3)	C11—C10—H10B	109.3
C6—C5—H5	119.8	C9—C10—H10B	109.3
C3^{i}—C5—H5	119.8	H10A—C10—H10B	108.0
C5—C6—C4	119.3 (3)	C10—C11—C10^{ii}	111.3 (3)
C5—C6—C7	120.5 (3)	C10—C11—H11A	109.4
C4—C6—C7	120.2 (3)	C10^{ii}—C11—H11A	109.4
O1—C7—N1	120.8 (3)	C10—C11—H11B	109.4
O1—C7—C6	121.2 (3)	C10^{ii}—C11—H11B	109.4
N1—C7—C6	118.0 (2)	H11A—C11—H11B	108.0
C7—N1—C1—O2	180.0	C2—C4—C6—C7	0.0
C8—N1—C1—O2	0.0	C4^{i}—C4—C6—C7	180.0
C7—N1—C1—C2	0.0	C1—N1—C7—O1	180.0
C8—N1—C1—C2	180.0	C8—N1—C7—O1	0.0
O2—C1—C2—C3	0.0	C1—N1—C7—C6	0.0
N1—C1—C2—C3	180.0	C8—N1—C7—C6	180.0
O2—C1—C2—C4	180.0	C5—C6—C7—O1	0.0
N1—C1—C2—C4	0.0	C4—C6—C7—O1	180.0
C4—C2—C3—C5^{i}	0.000 (1)	C5—C6—C7—N1	180.0
C1—C2—C3—C5^{i}	180.0	C4—C6—C7—N1	0.0
C3—C2—C4—C4^{i}	0.0	C1—N1—C8—C9^{ii}	115.76 (19)
C1—C2—C4—C4^{i}	180.0	C7—N1—C8—C9^{ii}	−64.24 (19)
C3—C2—C4—C6	180.0	C1—N1—C8—C9	−115.76 (19)
C1—C2—C4—C6	0.0	C7—N1—C8—C9	64.24 (19)
C3^{i}—C5—C6—C4	0.000 (1)	N1—C8—C9—C10	176.2 (2)
C3^{i}—C5—C6—C7	180.0	C9^{ii}—C8—C9—C10	−55.5 (4)

C2—C4—C6—C5	180.0	C8—C9—C10—C11	55.2 (3)
C4^{i}—C4—C6—C5	0.000 (1)	C9—C10—C11—C10^{ii}	−56.5 (4)

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