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## Structure Reports

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

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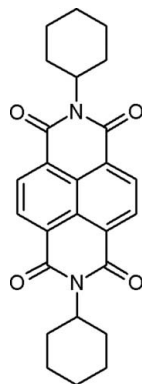
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 Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å;  $R$  factor = 0.067;  $wR$  factor = 0.183; data-to-parameter ratio = 13.5.

The title compound,  $\text{C}_{26}\text{H}_{26}\text{N}_2\text{O}_4$ , 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 mobility *ca*  $6 \text{ cm}^2/\text{Vs}$  [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

$\text{C}_{26}\text{H}_{26}\text{N}_2\text{O}_4$	$V = 1026.19 (6) \text{ \AA}^3$
$M_r = 430.49$	$Z = 2$
Monoclinic, $C2/m$	Mo $K\alpha$ radiation
$a = 8.5410 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 6.6780 (2) \text{ \AA}$	$T = 293 (2) \text{ K}$
$c = 18.4270 (9) \text{ \AA}$	$0.35 \times 0.25 \times 0.17 \text{ mm}$
$\beta = 102.4790 (18)^\circ$	

## Data collection

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

## Refinement

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

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).

We thank Ms Wendy Ahearn and Ms Dianne Meyer of Eastman Kodak Company for material purification and crystal growth *via* sublimation.

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

## References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Chesterfield, R. J., McKeen, J. C., Newman, C. R., Ewbank, P. C., da SilvaFilho, D. A., Brédas, J. L., Miller, L. L., Mann, K. R. & Frisbie, C. D. (2004*a*). *J. Phys. Chem. B*, **108**, 19281–19292.
- Chesterfield, R. J., McKeen, J. C., Newman, C. R., Frisbie, C. D., Ewbank, P. C., Mann, K. R. & Miller, L. L. (2004*b*). *Appl. Phys. Lett.* **95**, 6396–6405.
- Facceti, A., Yoon, M.-H. & Marks, T. J. (2008). *Adv. Mater.* **17**, 1705–1725.
- Jones, B. A., Ahrens, M. J., Yoon, M.-H., Facchetti, A., Marks, T. J. & Wasielewski, M. R. (2004). *Angew. Chem. Int. Ed.* **43**, 6363–6366.
- Katz, H. E., Johnson, J., Lovinger, A. J. & Li, W. (2000*a*). *J. Am. Chem. Soc.* **122**, 7787–7792.
- Katz, H. E., Lovinger, A. J., Johnson, J., Kloc, C., Siegrist, T., Li, W., Lin, Y.-Y. & Dodabalapur, A. (2000*b*). *Nature (London)*, **404**, 478–481.
- Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Shukla, D., Nelson, S. F., Freeman, D. C., Rajeswaran, M., Ahearn, W. G., Meyer, D. M. & Carey, J. T. (2008). *Chem. Mater.* Submitted.
- Westrip, S. P. (2007). *publCIF*. In preparation.

**supplementary materials**

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

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### Comment

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

### 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.  $^1\text{H}$  NMR ( $\text{CD}_2\text{Cl}_2$ , 500.05 MHz):  $\delta$  (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,  $J = 12$  and 11.7 Hz), 2.03 (d, 2H,  $J = 12$  Hz), 1.87 (d, 2H,  $J = 12$  Hz), 1.47 (m, 2H);  $^{13}\text{C}$  ( $\text{CD}_2\text{Cl}_2$ , 500.05 MHz):  $\delta$  = 163.23, 130.74, 127.13, 126.70, 54.85, 29.38, 26.66, 25.52; MS (MALDI-TOF)  $m/z$  calcd. for  $[\text{C}_{26}\text{H}_{26}\text{N}_2\text{O}_4]$  430.5 found: 430.2.

### Refinement

All H-atoms were positioned geometrically using a riding model with  $d(\text{C-H}) = 0.93\text{Å}$ ,  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$  for aromatic 0.97Å,  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$  for  $\text{CH}_2$  atoms.

### Figures

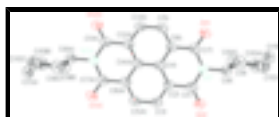


Fig. 1. Structure of the title compound (I), with the atom numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are omitted for clarity.

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

### Crystal data

$\text{C}_{26}\text{H}_{26}\text{N}_2\text{O}_4$

$M_r = 430.49$

Monoclinic,  $C2/m$

Hall symbol:  $-C 2y$

$F_{000} = 456$

$D_x = 1.393 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation

$\lambda = 0.71073 \text{ Å}$

Cell parameters from 21067 reflections

## supplementary materials

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$a = 8.5410 (2) \text{ \AA}$	$\theta = 1.0\text{--}27.5^\circ$
$b = 6.6780 (2) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$c = 18.4270 (9) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 102.4790 (18)^\circ$	Block, orange
$V = 1026.19 (6) \text{ \AA}^3$	$0.35 \times 0.25 \times 0.17 \text{ mm}$
$Z = 2$	

### Data collection

Nonius KappaCCD diffractometer	1227 independent reflections
Radiation source: fine-focus sealed tube	787 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.087$
Detector resolution: 9 pixels $\text{mm}^{-1}$	$\theta_{\text{max}} = 27.4^\circ$
$T = 293(2) \text{ K}$	$\theta_{\text{min}} = 4.3^\circ$
$\varphi$ and $\omega$ scans	$h = -10 \rightarrow 10$
Absorption correction: none	$k = -8 \rightarrow 8$
3354 measured reflections	$l = -23 \rightarrow 20$

### Refinement

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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 ( $\text{\AA}$ ,  $^\circ$ )

O1—C7	1.212 (3)	C5—H5	0.9300
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## supplementary materials

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O2—C1	1.216 (4)	C6—C7	1.474 (4)
N1—C1	1.401 (4)	C8—C9 <sup>ii</sup>	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 <sup>i</sup>	1.401 (4)	C10—C11	1.506 (4)
C3—H3	0.9300	C10—H10A	0.9700
C4—C4 <sup>i</sup>	1.409 (6)	C10—H10B	0.9700
C4—C6	1.415 (4)	C11—C10 <sup>ii</sup>	1.506 (4)
C5—C6	1.382 (4)	C11—H11A	0.9700
C5—C3 <sup>i</sup>	1.401 (4)	C11—H11B	0.9700
C1—N1—C7	123.2 (3)	N1—C8—C9 <sup>ii</sup>	112.42 (17)
C1—N1—C8	117.8 (3)	N1—C8—C9	112.42 (17)
C7—N1—C8	119.0 (2)	C9 <sup>ii</sup> —C8—C9	112.7 (3)
O2—C1—N1	121.3 (3)	N1—C8—H8	106.2
O2—C1—C2	120.9 (3)	C9 <sup>ii</sup> —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 <sup>i</sup>	121.3 (3)	C8—C9—H9B	109.7
C2—C3—H3	119.3	C10—C9—H9B	109.7
C5 <sup>i</sup> —C3—H3	119.3	H9A—C9—H9B	108.2
C2—C4—C4 <sup>i</sup>	120.0 (3)	C11—C10—C9	111.6 (3)
C2—C4—C6	120.3 (3)	C11—C10—H10A	109.3
C4 <sup>i</sup> —C4—C6	119.7 (3)	C9—C10—H10A	109.3
C6—C5—C3 <sup>i</sup>	120.4 (3)	C11—C10—H10B	109.3
C6—C5—H5	119.8	C9—C10—H10B	109.3
C3 <sup>i</sup> —C5—H5	119.8	H10A—C10—H10B	108.0
C5—C6—C4	119.3 (3)	C10—C11—C10 <sup>ii</sup>	111.3 (3)
C5—C6—C7	120.5 (3)	C10—C11—H11A	109.4
C4—C6—C7	120.2 (3)	C10 <sup>ii</sup> —C11—H11A	109.4
O1—C7—N1	120.8 (3)	C10—C11—H11B	109.4
O1—C7—C6	121.2 (3)	C10 <sup>ii</sup> —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 <sup>i</sup> —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 <sup>i</sup>	0.000 (1)	C5—C6—C7—N1	180.0
C1—C2—C3—C5 <sup>i</sup>	180.0	C4—C6—C7—N1	0.0
C3—C2—C4—C4 <sup>i</sup>	0.0	C1—N1—C8—C9 <sup>ii</sup>	115.76 (19)
C1—C2—C4—C4 <sup>i</sup>	180.0	C7—N1—C8—C9 <sup>ii</sup>	-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 <sup>i</sup> —C5—C6—C4	0.000 (1)	N1—C8—C9—C10	176.2 (2)
C3 <sup>i</sup> —C5—C6—C7	180.0	C9 <sup>ii</sup> —C8—C9—C10	-55.5 (4)
C2—C4—C6—C5	180.0	C8—C9—C10—C11	55.2 (3)
C4 <sup>i</sup> —C4—C6—C5	0.000 (1)	C9—C10—C11—C10 <sup>ii</sup>	-56.5 (4)

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

Fig. 1

