Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-Di-*n*-tetradecylpyromellitic diimide

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.005 Å
R factor = 0.059
wR factor = 0.203
Data-to-parameter ratio = 14.5

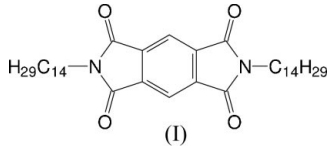
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 February 2004
Accepted 15 March 2004
Online 24 March 2004

The structure of the title compound, $C_{38}H_{60}N_2O_4$, has been determined and is similar to other compounds of this type, being essentially rod-shaped with the packing dominated by the lamellar arrangement of the molecules. The molecule lies on an inversion centre; thus only one alkyl chain, one imide ring and one of the non-bridgehead C atoms in the benzene ring are unique. The diimide moieties are arranged in a classic herring-bone structure, with two close non-hydrogen-atom contacts of 2.874 (5) and 2.946 (5) Å.

Comment

In a previous publication, we investigated the thin-film characteristics of neutral pseudorotaxanes consisting of 1:1 and 1:2 mixtures of bis(1,5-naphtho)-38-crown-10 with *N*-alkyl derivatives of both pyromellitic diimide and 1,4,5,8-naphthalenetetracarboxylic diimide (Lynch *et al.*, 1999). The Langmuir spreading solutions used in this study were subsequently refrigerated for storage while the paper was being refereed and published. These solutions, over a period of several months, eventually evaporated to dryness, yielding crystals of varying quality. From the solution containing a 1:1 molar mixture of the crown and *N*,*N*′-di-*n*-tetradecylpyromellitic diimide, two distinct crystal forms were identified, separated and characterized using single-crystal X-ray techniques. One of those structures was that of (I), reported here, while the other form was that of a second polymorph of the crown (Lynch & Hamilton, 2004*a*). From the solution containing a 1:1 molar mixture of the crown and the naphthalene diimide analogue, crystals were obtained which gave the structure of the diimide; however, the poor quality of the crystals led to poor data and hence a poor structure refinement (Lynch & Hamilton, 2004*b*).



(I)

The structure of (I) is similar to other compounds of this type, being essentially rod-shaped (Fig. 1) with the packing dominated by the lamellar arrangement of the molecules. The diimide moieties arrange in a classic herring-bone structure with two close non-hydrogen-atom contacts: C6···O21($-x$, $-\frac{1}{2}+y$, $\frac{1}{2}-z$) = 2.874 (5) Å and C2···O61(x, $\frac{1}{2}-y$, $-\frac{1}{2}+z$) = 2.946 (5) Å. The chains are inclined at an angle of *ca* 40° to the plane of the diimide ring. The naphthalene diimide analogue similarly resides on an inversion centre, the molecules also pack in layers and the chains in this compound are also

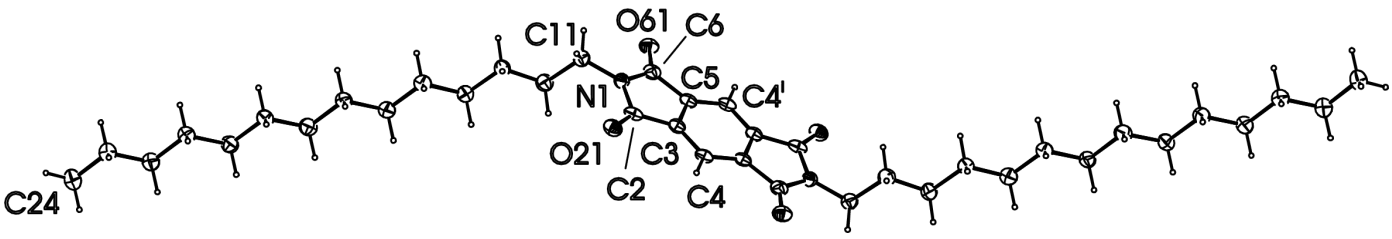


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level. For clarity, only the first and last C atoms of the alkyl chain have been labelled. [Symmetry code (i): $-x, 1-y, 1-z$.]

inclined at an angle of *ca* 40° to the plane of the ring system, but this structure differs from (I) in that the naphthalene moieties are parallel to each other, displaying interplanar distaces of *ca* 3.3–3.4 Å in the overlap regions.

Experimental

Crystals of the title compound were obtained following the total evaporation of an equimolar mixture of bis(1,5-naphtho)-38-crown-10 and (I) in 10 ml chloroform (0.1 mg cm^{-3}) at 277 K.

Crystal data

$C_{38}H_{60}N_2O_4$	D_x = 1.175 Mg m^{-3}
M_r = 608.88	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 6262 reflections
a = 38.939 (3) Å	θ = 1.0–27.5°
b = 4.9902 (3) Å	μ = 0.08 mm^{-1}
c = 8.9040 (5) Å	T = 150 (2) K
β = 95.896 (2)°	Plate, colourless
V = 1721.01 (19) Å^3	0.30 × 0.25 × 0.01 mm
Z = 2	

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer	2896 independent reflections
	1366 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.093
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)	θ_{max} = 25.0°
	$h = -45 \rightarrow 46$
T_{min} = 0.992, T_{max} = 0.999	$k = -5 \rightarrow 5$
6743 measured reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.059	$w = 1/[\sigma^2(F_o^2) + (0.0882P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.203	
S = 0.97	$(\Delta/\sigma)_{max} < 0.001$
2896 reflections	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
200 parameters	$\Delta\rho_{min}$ = −0.27 e Å^{-3}

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.95 (aromatic H atoms), 0.99 (CH_2 H atoms) and 0.98 Å (CH_3 H atoms). The isotropic displacement parameters were set equal to $1.25U_{eq}$ of the carrier atom. The high R_{int} value was the result of weak high-angle data.

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO*, *SCALEPACK* (Otwinowski & Minor, 1997) and *COLLECT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

The authors thank the EPSRC National Crystallography Service (Southampton, England).

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supporting information

Acta Cryst. (2004). E**60**, o597–o598 [https://doi.org/10.1107/S1600536804006038]

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$C_{38}H_{60}N_2O_4$
$M_r = 608.88$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 38.939$ (3) Å
$b = 4.9902$ (3) Å
$c = 8.9040$ (5) Å
$\beta = 95.896$ (2)°
$V = 1721.01$ (19) Å^3
$Z = 2$
$F(000) = 668$
$D_x = 1.175$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6262 reflections
$\theta = 1.0$–$27.5°$
$\mu = 0.08$ mm^{-1}
$T = 150$ K
Plate, colourless
0.30 × 0.25 × 0.01 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SORTAV; Blessing, 1995)
$T_{min} = 0.992$, $T_{max} = 0.999$
6743 measured reflections
2896 independent reflections
1366 reflections with $I > 2\sigma(I)$
$R_{int} = 0.093$
$\theta_{max} = 25.0°$, $\theta_{min} = 1.1°$
$h = -45 \rightarrow 46$
$k = -5 \rightarrow 5$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.203$
$S = 0.97$
2896 reflections
200 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
$w = 1/[\sigma^2(F_o^2) + (0.0882P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.08654 (7)	0.5189 (6)	0.5668 (3)	0.0239 (8)
C2	0.06735 (10)	0.6964 (8)	0.4715 (4)	0.0263 (10)
O21	0.07873 (6)	0.8812 (5)	0.4046 (3)	0.0302 (7)

C3	0.03053 (9)	0.6150 (7)	0.4746 (4)	0.0226 (9)
C4	0.00077 (9)	0.7297 (7)	0.4040 (4)	0.0239 (10)
H4	0.0013	0.8826	0.3407	0.030*
C5	0.02980 (9)	0.3927 (7)	0.5685 (4)	0.0222 (9)
C6	0.06626 (10)	0.3262 (8)	0.6263 (4)	0.0267 (10)
O61	0.07640 (6)	0.1404 (5)	0.7067 (3)	0.0306 (7)
C11	0.12372 (9)	0.5474 (7)	0.6005 (4)	0.0274 (10)
H111	0.1311	0.4528	0.6961	0.034*
H112	0.1294	0.7396	0.6153	0.034*
C12	0.14350 (9)	0.4365 (8)	0.4766 (4)	0.0303 (10)
H121	0.1442	0.2387	0.4847	0.038*
H122	0.1310	0.4827	0.3776	0.038*
C13	0.18006 (9)	0.5406 (7)	0.4817 (4)	0.0284 (10)
H131	0.1928	0.4905	0.5795	0.035*
H132	0.1795	0.7387	0.4757	0.035*
C14	0.19926 (9)	0.4317 (7)	0.3539 (4)	0.0300 (10)
H141	0.2017	0.2352	0.3661	0.038*
H142	0.1851	0.4654	0.2569	0.038*
C15	0.23474 (9)	0.5511 (8)	0.3456 (4)	0.0320 (10)
H151	0.2491	0.5157	0.4418	0.040*
H152	0.2325	0.7477	0.3336	0.040*
C16	0.25292 (9)	0.4385 (8)	0.2156 (4)	0.0318 (11)
H161	0.2554	0.2422	0.2288	0.040*
H162	0.2382	0.4707	0.1199	0.040*
C17	0.28833 (9)	0.5584 (8)	0.2031 (4)	0.0314 (10)
H171	0.3030	0.5278	0.2991	0.039*
H172	0.2859	0.7544	0.1886	0.039*
C18	0.30640 (9)	0.4416 (7)	0.0735 (4)	0.0322 (10)
H181	0.3089	0.2457	0.0885	0.040*
H182	0.2916	0.4712	−0.0223	0.040*
C19	0.34161 (9)	0.5604 (8)	0.0592 (4)	0.0323 (11)
H191	0.3564	0.5318	0.1551	0.040*
H192	0.3391	0.7561	0.0434	0.040*
C20	0.35951 (9)	0.4418 (8)	−0.0694 (4)	0.0311 (10)
H201	0.3618	0.2459	−0.0541	0.039*
H202	0.3448	0.4716	−0.1654	0.039*
C21	0.39494 (9)	0.5594 (8)	−0.0830 (4)	0.0326 (10)
H211	0.4097	0.5281	0.0127	0.041*
H212	0.3926	0.7556	−0.0973	0.041*
C22	0.41288 (9)	0.4422 (8)	−0.2132 (4)	0.0341 (11)
H221	0.4150	0.2459	−0.1994	0.043*
H222	0.3982	0.4749	−0.3090	0.043*
C23	0.44843 (10)	0.5581 (8)	−0.2261 (5)	0.0392 (11)
H231	0.4633	0.5217	−0.1313	0.049*
H232	0.4464	0.7549	−0.2379	0.049*
C24	0.46567 (10)	0.4442 (9)	−0.3582 (5)	0.0474 (13)
H241	0.4690	0.2507	−0.3444	0.059*
H242	0.4881	0.5306	−0.3626	0.059*

H243	0.4510	0.4779	−0.4525	0.059*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0243 (18)	0.0257 (19)	0.0217 (18)	−0.0017 (16)	0.0020 (15)	0.0002 (15)
C2	0.040 (3)	0.025 (3)	0.014 (2)	0.003 (2)	0.0037 (18)	−0.001 (2)
O21	0.0390 (17)	0.0257 (17)	0.0263 (16)	−0.0053 (14)	0.0055 (13)	0.0019 (14)
C3	0.031 (2)	0.020 (2)	0.018 (2)	−0.0004 (19)	0.0040 (17)	−0.0051 (18)
C4	0.036 (2)	0.020 (2)	0.015 (2)	0.000 (2)	0.0035 (18)	−0.0022 (18)
C5	0.032 (2)	0.018 (2)	0.017 (2)	−0.0004 (19)	0.0037 (17)	−0.0047 (18)
C6	0.033 (2)	0.027 (3)	0.021 (2)	0.002 (2)	0.0024 (18)	−0.008 (2)
O61	0.0390 (17)	0.0273 (17)	0.0249 (16)	0.0039 (14)	0.0004 (12)	0.0017 (14)
C11	0.027 (2)	0.029 (2)	0.026 (2)	0.0013 (19)	0.0013 (18)	0.001 (2)
C12	0.031 (2)	0.031 (3)	0.029 (2)	0.0023 (19)	0.0027 (18)	−0.003 (2)
C13	0.028 (2)	0.030 (2)	0.028 (2)	0.0021 (19)	0.0040 (18)	−0.0026 (19)
C14	0.028 (2)	0.035 (3)	0.027 (2)	−0.0014 (19)	0.0035 (18)	0.000 (2)
C15	0.032 (2)	0.035 (3)	0.029 (2)	0.001 (2)	0.0033 (19)	0.000 (2)
C16	0.030 (2)	0.038 (3)	0.026 (2)	0.002 (2)	0.0027 (18)	−0.001 (2)
C17	0.030 (2)	0.038 (3)	0.027 (2)	0.006 (2)	0.0043 (18)	0.001 (2)
C18	0.035 (2)	0.033 (3)	0.029 (2)	0.003 (2)	0.0040 (19)	0.001 (2)
C19	0.031 (2)	0.039 (3)	0.027 (2)	−0.002 (2)	0.0059 (18)	−0.003 (2)
C20	0.033 (2)	0.032 (3)	0.028 (2)	−0.001 (2)	0.0042 (18)	0.000 (2)
C21	0.032 (2)	0.036 (3)	0.030 (2)	0.001 (2)	0.0055 (18)	0.000 (2)
C22	0.032 (2)	0.037 (3)	0.033 (3)	0.002 (2)	0.0049 (19)	0.000 (2)
C23	0.033 (3)	0.048 (3)	0.037 (3)	0.002 (2)	0.007 (2)	0.001 (2)
C24	0.036 (3)	0.069 (4)	0.039 (3)	0.002 (2)	0.012 (2)	−0.004 (3)

Geometric parameters (Å, º)

N1—C6	1.384 (5)	C16—C17	1.518 (5)
N1—C2	1.390 (4)	C16—H161	0.99
N1—C11	1.455 (4)	C16—H162	0.99
C2—O21	1.207 (4)	C17—C18	1.527 (5)
C2—C3	1.493 (5)	C17—H171	0.99
C3—C4	1.384 (4)	C17—H172	0.99
C3—C5	1.391 (5)	C18—C19	1.511 (5)
C4—C5^{i}	1.382 (5)	C18—H181	0.99
C4—H4	0.95	C18—H182	0.99
C5—C4^{i}	1.382 (5)	C19—C20	1.520 (5)
C5—C6	1.497 (5)	C19—H191	0.99
C6—O61	1.213 (4)	C19—H192	0.99
C11—C12	1.514 (5)	C20—C21	1.516 (5)
C11—H111	0.99	C20—H201	0.99
C11—H112	0.99	C20—H202	0.99
C12—C13	1.512 (5)	C21—C22	1.530 (5)
C12—H121	0.99	C21—H211	0.99
C12—H122	0.99	C21—H212	0.99

C13—C14	1.525 (5)	C22—C23	1.515 (5)
C13—H131	0.99	C22—H221	0.99
C13—H132	0.99	C22—H222	0.99
C14—C15	1.513 (4)	C23—C24	1.523 (5)
C14—H141	0.99	C23—H231	0.99
C14—H142	0.99	C23—H232	0.99
C15—C16	1.524 (5)	C24—H241	0.98
C15—H151	0.99	C24—H242	0.98
C15—H152	0.99	C24—H243	0.98

C6—N1—C2	112.6 (3)	C17—C16—H162	108.8
C6—N1—C11	125.7 (3)	C15—C16—H162	108.8
C2—N1—C11	121.7 (3)	H161—C16—H162	107.6
O21—C2—N1	125.9 (4)	C16—C17—C18	113.4 (3)
O21—C2—C3	128.2 (3)	C16—C17—H171	108.9
N1—C2—C3	105.8 (3)	C18—C17—H171	108.9
C4—C3—C5	122.3 (3)	C16—C17—H172	108.9
C4—C3—C2	129.8 (3)	C18—C17—H172	108.9
C5—C3—C2	107.9 (3)	H171—C17—H172	107.7
C5^{i}—C4—C3	115.6 (3)	C19—C18—C17	114.0 (3)
C5^{i}—C4—H4	122.2	C19—C18—H181	108.8
C3—C4—H4	122.2	C17—C18—H181	108.8
C4^{i}—C5—C3	122.0 (3)	C19—C18—H182	108.8
C4^{i}—C5—C6	130.2 (3)	C17—C18—H182	108.8
C3—C5—C6	107.8 (3)	H181—C18—H182	107.6
O61—C6—N1	126.4 (4)	C18—C19—C20	113.6 (3)
O61—C6—C5	127.8 (4)	C18—C19—H191	108.8
N1—C6—C5	105.8 (3)	C20—C19—H191	108.8
N1—C11—C12	112.6 (3)	C18—C19—H192	108.8
N1—C11—H111	109.1	C20—C19—H192	108.8
C12—C11—H111	109.1	H191—C19—H192	107.7
N1—C11—H112	109.1	C21—C20—C19	113.6 (3)
C12—C11—H112	109.1	C21—C20—H201	108.8
H111—C11—H112	107.8	C19—C20—H201	108.8
C13—C12—C11	113.6 (3)	C21—C20—H202	108.8
C13—C12—H121	108.8	C19—C20—H202	108.8
C11—C12—H121	108.8	H201—C20—H202	107.7
C13—C12—H122	108.8	C20—C21—C22	113.8 (3)
C11—C12—H122	108.8	C20—C21—H211	108.8
H121—C12—H122	107.7	C22—C21—H211	108.8
C12—C13—C14	112.8 (3)	C20—C21—H212	108.8
C12—C13—H131	109.0	C22—C21—H212	108.8
C14—C13—H131	109.0	H211—C21—H212	107.7
C12—C13—H132	109.0	C23—C22—C21	113.8 (3)
C14—C13—H132	109.0	C23—C22—H221	108.8
H131—C13—H132	107.8	C21—C22—H221	108.8
C15—C14—C13	114.6 (3)	C23—C22—H222	108.8
C15—C14—H141	108.6	C21—C22—H222	108.8

C13—C14—H141	108.6	H221—C22—H222	107.7
C15—C14—H142	108.6	C22—C23—C24	113.2 (3)
C13—C14—H142	108.6	C22—C23—H231	108.9
H141—C14—H142	107.6	C24—C23—H231	108.9
C14—C15—C16	112.9 (3)	C22—C23—H232	108.9
C14—C15—H151	109.0	C24—C23—H232	108.9
C16—C15—H151	109.0	H231—C23—H232	107.7
C14—C15—H152	109.0	C23—C24—H241	109.5
C16—C15—H152	109.0	C23—C24—H242	109.5
H151—C15—H152	107.8	H241—C24—H242	109.5
C17—C16—C15	114.0 (3)	C23—C24—H243	109.5
C17—C16—H161	108.8	H241—C24—H243	109.5
C15—C16—H161	108.8	H242—C24—H243	109.5

C6—N1—C2—O21	−179.5 (3)	C4^{i}—C5—C6—O61	2.9 (6)
C11—N1—C2—O21	2.5 (5)	C3—C5—C6—O61	−177.1 (3)
C6—N1—C2—C3	2.3 (4)	C4^{i}—C5—C6—N1	−178.1 (3)
C11—N1—C2—C3	−175.7 (3)	C3—C5—C6—N1	1.9 (4)
O21—C2—C3—C4	−0.6 (6)	C6—N1—C11—C12	101.9 (4)
N1—C2—C3—C4	177.6 (3)	C2—N1—C11—C12	−80.4 (4)
O21—C2—C3—C5	−179.2 (4)	N1—C11—C12—C13	161.0 (3)
N1—C2—C3—C5	−1.0 (4)	C11—C12—C13—C14	−178.7 (3)
C5—C3—C4—C5^{i}	−0.7 (5)	C12—C13—C14—C15	174.4 (3)
C2—C3—C4—C5^{i}	−179.1 (3)	C13—C14—C15—C16	−179.5 (3)
C4—C3—C5—C4^{i}	0.7 (6)	C14—C15—C16—C17	179.0 (3)
C2—C3—C5—C4^{i}	179.4 (3)	C15—C16—C17—C18	179.4 (3)
C4—C3—C5—C6	−179.2 (3)	C16—C17—C18—C19	179.7 (3)
C2—C3—C5—C6	−0.5 (4)	C17—C18—C19—C20	179.6 (3)
C2—N1—C6—O61	176.4 (3)	C18—C19—C20—C21	−179.6 (3)
C11—N1—C6—O61	−5.6 (6)	C19—C20—C21—C22	−179.5 (3)
C2—N1—C6—C5	−2.6 (4)	C20—C21—C22—C23	−179.5 (3)
C11—N1—C6—C5	175.3 (3)	C21—C22—C23—C24	−178.8 (3)

Symmetry code: (i) −*x*, −*y*+1, −*z*+1.