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N,*N*′-Bis(1-ethynylcyclohexyl)pyromellitic diimide

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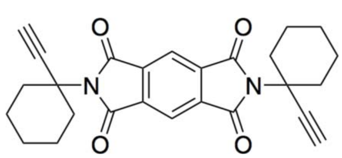
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.065; wR factor = 0.158; data-to-parameter ratio = 13.8.

The title compound, $C_{26}H_{24}N_2O_4$, consists of a symmetrical molecule that lies across a crystallographic inversion centre. The C—C distance in the triple bond is 1.188 (2) Å and there is also an intermolecular C—H···O contact from a terminal acetylene C—H to one of the dimiide O atoms [3.4349 (19) Å].

Related literature

For literature relating to the oxidative coupling of terminal acetylenes, see: Anderson, Anderson & Sanders (1995); Anderson, Walter *et al.* (1995); Hamilton *et al.* (1998); Raehm *et al.* (2002).



Experimental

Crystal data

$C_{26}H_{24}N_2O_4$
M_r = 428.47
Monoclinic, $P2_1/c$
a = 13.1774 (3) Å
b = 7.1519 (1) Å
c = 11.8104 (3) Å
β = 112.495 (1)°
V = 1028.36 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 120 K
0.40 × 0.35 × 0.20 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.963, T_{max} = 0.982
12939 measured reflections
2022 independent reflections
1898 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.158
S = 1.29
2022 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.80 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C14—H12···O2^{i}	0.95	2.52	3.4349 (19)	161

Symmetry code: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON97* (Spek, 2009); 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: ZS2003).

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S1. Comment

The oxidative coupling of terminal acetylenes has proven to be a valuable architectural tool in the preparation of large macrocycles, especially when coupled to a templating mechanism to organize the premacrocycle components (Anderson, Anderson & Sanders, 1995). In this manner some remarkable structures have been assembled with admirable efficiency, given the entropic handicap imposed on the synthesis of large ring macrocycles (Anderson, Walter *et al.*, 1995). One area in which a template greatly favors cyclization, and subsequently forms an integral part of the product structure, is in the synthesis of interlocked molecular compounds (catenanes and rotaxanes, Hamilton *et al.*, 1998). Numerous systems have been reported that rely on the attractive interaction between π-electron deficient aromatic diimides and π-electron rich aromatic diethers to establish the desired templating effect (Raehm *et al.*, 2002). In many of these instances the diimide component was equipped with terminal acetylenes, susbsequent oxidative coupling of which afforded the desired interlocked molecular systems. The title compound was prepared to address a key shortcoming of many acetylenic di-imides of this type, namely their relatively low solubility in most organic solvents, in particular those in which the templating effects, so crucial to macrocycle synthesis, would be most effectively deployed. The presence of cyclohexyl substituents at the junctures of the diimide core with the acetylene substituents engendered high organic solvent solubility while retaining the key structural features required of the diimide unit. Reported here is the structure of the title compound (I), which is a symmetrical molecule that lies across a crystallographic inversion centre (Fig. 1), the asymmetric unit comprising half of the molecule. With such a simple molecule there are very few distinct features to report although it is worth mentioning that the C—C distance in the triple bond is 1.188 (2) Å. There is also an intermolecular C—H···O contact between the terminal acetylene C—H and one of the dimiide O atoms (Table 1).

S2. Experimental

To a stirred solution of 1,2,4,5-benzenetetracarboxylic dianhydride (2.18 g, 10 mmol) in dry THF (20 mL) was added a solution of 1-ethynylcyclohexylamine (2.50 g, 2.74 ml, 20 mmol) in dry THF (10 ml). After 6 h the reaction was evaporated to give a white foam to which was added acetic anhydride (30 ml). After heating at 130° C for 2 h the reaction was cooled to room temperature and poured into vigorously stirred icecold water. The precipitated solids were collected at the pump, washed with cold water, and recrystallized from aqueous DMF to afford pale yellow crystals of the title compound (0.71 g, 17%): m.p. 219–220° C; ^{13}C NMR (100 MHz, $CDCl_3$) δ 166.5, 138.0, 119.0, 83.0, 75.0, 60.0, 36.0, 25.0, 23.0; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (s, 2H), 2.65 (s, 2H), 2.56–2.45, 2.44–2.29, 1.90–1.60, 1.40–1.20 (4 *x* multiplet, 20H). Single crystals of suitable quality for structure determination were grown by vapor diffusion of water into a DMF solution of the title compound.

S3. Refinement

All H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.95 (CH) and 0.99Å (CH_2). The isotropic displacement parameters for all H atoms were set equal to 1.25U_{eq} of the carrier atom. The large maximum and minimum residual electron density peaks [0.60 eÅ^{-3}, 1.45 Å from C13 and -0.80 eÅ^{-3}, 1.30 Å from H1 respectively] are unexplained.

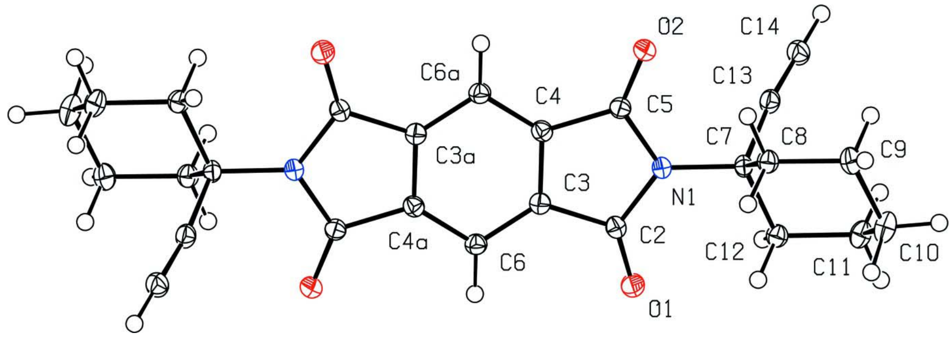

Figure 1

Molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are drawn at the 50% probability level. Symmetry code (a): -*x*, -*y* + 1, -*z* + 1.

N,*N'*-Bis(1-ethynylcyclohexyl)pyromellitic diimide

Crystal data

$C_{26}H_{24}N_2O_4$
M_r = 428.47
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 13.1774 (3) Å
b = 7.1519 (1) Å
c = 11.8104 (3) Å
β = 112.495 (1)°
V = 1028.36 (4) Å^3
Z = 2
$F(000)$ = 452
D_x = 1.384 Mg m^{-3}
Melting point = 492–493 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2528 reflections
θ = 2.9–27.5°
μ = 0.09 mm^{-1}
T = 120 K
Prism, colourless
0.40 × 0.35 × 0.20 mm

Data collection

Bruker–Nonius KappaCCD diffractometer
Radiation source: Bruker Nonius FR591 rotating anode
10 cm confocal mirrors monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ & ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.963, T_{max} = 0.982
12939 measured reflections
2022 independent reflections
1898 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.0°, θ_{min} = 3.3°
h = −16→16
k = −8→8
l = −13→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.158
S = 1.29
2022 reflections
146 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
$w = 1/[\sigma^2(F_o^2) + (0.091P)^2 + 0.2895P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.60$ e Å^{-3}
$\Delta\rho_{min} = -0.80$ e Å^{-3}
Extinction correction: *SHELXL97*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.38 (3)

Special details

Experimental. The minimum and maximum absorption values stated above are those calculated in *SHELXL97* from the given crystal dimensions. The ratio of minimum to maximum apparent transmission was determined experimentally as 0.798007.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.06501 (9)	0.08401 (16)	0.34794 (11)	0.0222 (4)
O2	0.22629 (9)	0.66713 (15)	0.40367 (11)	0.0202 (4)
N1	0.16857 (10)	0.35785 (17)	0.36123 (11)	0.0156 (4)
C2	0.08812 (12)	0.2440 (2)	0.37881 (13)	0.0158 (4)
C3	0.03482 (12)	0.3636 (2)	0.44392 (13)	0.0151 (4)
C4	0.07781 (12)	0.5432 (2)	0.45479 (13)	0.0152 (4)
C5	0.16567 (12)	0.5404 (2)	0.40426 (13)	0.0158 (4)
C6	−0.04412 (12)	0.3126 (2)	0.48923 (13)	0.0162 (4)
H1	−0.0726	0.1893	0.4825	0.020*
C7	0.25590 (11)	0.3002 (2)	0.31631 (13)	0.0150 (4)
C8	0.36865 (12)	0.3129 (2)	0.42496 (14)	0.0169 (4)
H2	0.3812	0.4431	0.4557	0.021*
H3	0.3680	0.2316	0.4925	0.021*
C9	0.46204 (12)	0.2530 (2)	0.38642 (15)	0.0208 (4)
H4	0.4676	0.3424	0.3251	0.026*
H5	0.5323	0.2563	0.4586	0.026*
C10	0.44355 (13)	0.0570 (2)	0.33209 (16)	0.0250 (4)
H6	0.4485	−0.0345	0.3969	0.031*
H7	0.5020	0.0272	0.3017	0.031*
C11	0.33156 (13)	0.0392 (2)	0.22690 (15)	0.0210 (4)
H8	0.3201	−0.0920	0.1979	0.026*
H9	0.3298	0.1191	0.1578	0.026*
C12	0.23911 (12)	0.0975 (2)	0.26781 (13)	0.0169 (4)
H10	0.2376	0.0120	0.3331	0.021*
H11	0.1677	0.0875	0.1977	0.021*
C13	0.25281 (12)	0.4268 (2)	0.21565 (14)	0.0178 (4)
C14	0.25817 (13)	0.5152 (2)	0.13290 (15)	0.0211 (4)
H12	0.2625	0.5858	0.0668	0.026*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0244 (6)	0.0177 (6)	0.0284 (7)	−0.0040 (4)	0.0144 (5)	−0.0057 (5)
O2	0.0223 (6)	0.0163 (6)	0.0262 (6)	−0.0021 (4)	0.0141 (5)	−0.0008 (4)

N1	0.0160 (6)	0.0157 (7)	0.0173 (7)	0.0003 (5)	0.0089 (5)	−0.0002 (5)
C2	0.0153 (7)	0.0177 (8)	0.0150 (7)	0.0000 (5)	0.0065 (6)	0.0006 (6)
C3	0.0151 (7)	0.0160 (8)	0.0138 (7)	0.0004 (5)	0.0051 (6)	0.0002 (5)
C4	0.0144 (7)	0.0165 (8)	0.0144 (7)	0.0004 (5)	0.0053 (6)	0.0019 (5)
C5	0.0167 (7)	0.0158 (8)	0.0153 (8)	0.0011 (5)	0.0068 (6)	0.0014 (5)
C6	0.0164 (7)	0.0147 (7)	0.0173 (8)	−0.0008 (5)	0.0064 (6)	−0.0003 (6)
C7	0.0150 (7)	0.0165 (8)	0.0159 (7)	0.0017 (5)	0.0085 (6)	0.0004 (6)
C8	0.0175 (8)	0.0182 (8)	0.0154 (8)	0.0004 (5)	0.0069 (6)	−0.0006 (6)
C9	0.0161 (8)	0.0257 (9)	0.0211 (8)	0.0010 (6)	0.0078 (6)	0.0001 (6)
C10	0.0205 (8)	0.0294 (9)	0.0251 (9)	0.0066 (6)	0.0086 (7)	−0.0039 (7)
C11	0.0227 (8)	0.0228 (9)	0.0185 (8)	0.0032 (6)	0.0091 (6)	−0.0038 (6)
C12	0.0181 (8)	0.0174 (8)	0.0159 (8)	−0.0001 (6)	0.0071 (6)	−0.0010 (6)
C13	0.0159 (7)	0.0189 (8)	0.0196 (8)	0.0027 (5)	0.0081 (6)	0.0001 (6)
C14	0.0219 (8)	0.0224 (8)	0.0217 (8)	0.0040 (6)	0.0111 (6)	0.0048 (7)

Geometric parameters (Å, º)

O1—C2	1.2042 (19)	C8—H2	0.99
O2—C5	1.2100 (18)	C8—H3	0.99
N1—C5	1.4066 (19)	C9—C10	1.522 (2)
N1—C2	1.4136 (19)	C9—H4	0.99
N1—C7	1.4976 (18)	C9—H5	0.99
C2—C3	1.493 (2)	C10—C11	1.528 (2)
C3—C6	1.388 (2)	C10—H6	0.99
C3—C4	1.389 (2)	C10—H7	0.99
C4—C6^{i}	1.387 (2)	C11—C12	1.530 (2)
C4—C5	1.492 (2)	C11—H8	0.99
C6—C4^{i}	1.387 (2)	C11—H9	0.99
C6—H1	0.95	C12—H10	0.99
C7—C13	1.482 (2)	C12—H11	0.99
C7—C12	1.543 (2)	C13—C14	1.188 (2)
C7—C8	1.550 (2)	C14—H12	0.95
C8—C9	1.528 (2)		
C5—N1—C2	110.85 (12)	C7—C8—H3	109.4
C5—N1—C7	120.94 (12)	H2—C8—H3	108.0
C2—N1—C7	127.92 (12)	C10—C9—C8	111.50 (13)
O1—C2—N1	128.31 (14)	C10—C9—H4	109.3
O1—C2—C3	125.88 (13)	C8—C9—H4	109.3
N1—C2—C3	105.81 (12)	C10—C9—H5	109.3
C6—C3—C4	123.13 (14)	C8—C9—H5	109.3
C6—C3—C2	128.11 (14)	H4—C9—H5	108.0
C4—C3—C2	108.76 (13)	C9—C10—C11	111.64 (13)
C3—C4—C6^{i}	122.54 (14)	C9—C10—H6	109.3
C3—C4—C5	107.58 (13)	C11—C10—H6	109.3
C6^{i}—C4—C5	129.77 (14)	C9—C10—H7	109.3
O2—C5—N1	125.73 (14)	C11—C10—H7	109.3
O2—C5—C4	127.39 (14)	H6—C10—H7	108.0

N1—C5—C4	106.83 (12)	C10—C11—C12	111.00 (12)
C3—C6—C4^{i}	114.32 (14)	C10—C11—H8	109.4
C3—C6—H1	122.8	C12—C11—H8	109.4
C4^{i}—C6—H1	122.8	C10—C11—H9	109.4
C13—C7—N1	109.08 (12)	C12—C11—H9	109.4
C13—C7—C12	108.67 (12)	H8—C11—H9	108.0
N1—C7—C12	111.69 (12)	C11—C12—C7	110.79 (12)
C13—C7—C8	110.59 (12)	C11—C12—H10	109.5
N1—C7—C8	108.28 (11)	C7—C12—H10	109.5
C12—C7—C8	108.54 (12)	C11—C12—H11	109.5
C9—C8—C7	111.30 (12)	C7—C12—H11	109.5
C9—C8—H2	109.4	H10—C12—H11	108.1
C7—C8—H2	109.4	C14—C13—C7	172.85 (16)
C9—C8—H3	109.4	C13—C14—H12	180.0
C5—N1—C2—O1	176.47 (15)	C6^{i}—C4—C5—N1	178.17 (14)
C7—N1—C2—O1	−9.7 (2)	C4—C3—C6—C4^{i}	0.8 (2)
C5—N1—C2—C3	−3.03 (16)	C2—C3—C6—C4^{i}	−179.81 (14)
C7—N1—C2—C3	170.78 (13)	C5—N1—C7—C13	−57.01 (17)
O1—C2—C3—C6	5.3 (3)	C2—N1—C7—C13	129.74 (15)
N1—C2—C3—C6	−175.23 (14)	C5—N1—C7—C12	−177.14 (12)
O1—C2—C3—C4	−175.27 (14)	C2—N1—C7—C12	9.6 (2)
N1—C2—C3—C4	4.25 (16)	C5—N1—C7—C8	63.40 (17)
C6—C3—C4—C6^{i}	−0.8 (3)	C2—N1—C7—C8	−109.86 (16)
C2—C3—C4—C6^{i}	179.65 (13)	C13—C7—C8—C9	−61.41 (16)
C6—C3—C4—C5	175.74 (13)	N1—C7—C8—C9	179.13 (12)
C2—C3—C4—C5	−3.77 (16)	C12—C7—C8—C9	57.70 (16)
C2—N1—C5—O2	178.38 (14)	C7—C8—C9—C10	−56.05 (17)
C7—N1—C5—O2	4.1 (2)	C8—C9—C10—C11	54.10 (18)
C2—N1—C5—C4	0.82 (16)	C9—C10—C11—C12	−54.98 (18)
C7—N1—C5—C4	−173.49 (12)	C10—C11—C12—C7	57.87 (17)
C3—C4—C5—O2	−175.58 (15)	C13—C7—C12—C11	61.74 (15)
C6^{i}—C4—C5—O2	0.7 (3)	N1—C7—C12—C11	−177.88 (11)
C3—C4—C5—N1	1.92 (16)	C8—C7—C12—C11	−58.57 (15)

Symmetry code: (i) $-x, -y+1, -z+1$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C14—H12···O2^{ii}	0.95	2.52	3.4349 (19)	161

Symmetry code: (ii) $x, -y+3/2, z-1/2$.