organic compounds

N,N'-Bis(1-acetylcyclohexyl)-1,8:4,5-naphthalenetetracarboximide

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The title compound, $C_{30}H_{30}N_2O_6$, has crystallographic inversion symmetry with the nitrogen atom and the two oxygen atoms of the naphthalene diimide system deviating by −0.243 (2), 0.109 (3) and 0.247 (2) Å, respectively, from the plane defined by the carbon atoms.

Related literature

For the structure of a related benzene diimide derivative with terminal acetylene groups, see: Gondo *et al.* (2009). For preparative procedures for compounds of this type and for the title compound, see Hamilton *et al.* (1998, 1999); Raehm *et al.* (2002); Ahn *et al.* (1997).
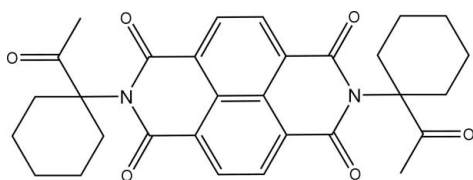
Experimental

Crystal data

$C_{30}H_{30}N_2O_6$	Monoclinic, $P2_1/c$
$M_r = 514.56$	$a = 5.8553$ (2) Å
	$b = 13.6603$ (6) Å
	$c = 15.2820$ (6) Å
	$\beta = 94.001$ (2)°
	$V = 1219.35$ (8) Å^3
	$Z = 2$

Mo $K\alpha$ radiation	
$\mu = 0.10$ mm^{-1}	
$T = 120$ K	
$0.20 \times 0.18 \times 0.06$ mm	

Data collection

Bruker–Nonius 95 mm CCD camera on κ-goniostat diffractometer	11932 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	2397 independent reflections
$T_{min} = 0.981$, $T_{max} = 0.994$	1949 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.063$	174 parameters
$wR(F^2) = 0.150$	H-atom parameters constrained
$S = 1.17$	$\Delta\rho_{max} = 0.60$ e Å^{-3}
2397 reflections	$\Delta\rho_{min} = -0.59$ e Å^{-3}

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: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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supporting information

N,*N*′-Bis(1-acetylcyclohexyl)-1,8:4,5-naphthalenetetracarboximide

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S1. Comment

In a previous paper we presented the structure of a benzene diimide derivative having terminal acetylene groups and solubilizing cyclohexyl substituents (Gondo *et al.*, 2009). This material was prepared for use in oxidative coupling reactions, thereby forming macrocycles as either isolated entities (Hamilton *et al.*, 1999), or as components of molecularly interlocked systems (Hamilton *et al.*, 1998; Raehm *et al.*, 2002). As the corresponding naphthalene diimide analogues of benzene diimide derivatives are known to be generally more powerful electron acceptors, and have therefore been deployed in a variety of supramolecular and materials chemistry contexts, we attempted the preparation of the corresponding naphthalene diimide. However, under all of the standard conditions generally employed in the synthesis of benzene and naphthalene diimides we failed to obtain the desired compound. Only under rather forcing conditions was evidence of ring closure to the imide obtained, but under these conditions adventitious water was also found to have added to the acetylene groups (Ahn *et al.*, 1997). Thus, a low yield of the diketone was the only isolable material obtained from this process and the structure of this compound (I) is reported here.

The title compound has crystallographic inversion symmetry (Fig. 1), (symmetry code: a -*x* + 1, -*y* + 1, -*z* + 1). The nitrogen and the two oxygen atoms of the naphthalene diimide systems deviate by -0.243 (2), 0.109 (3) and 0.247 (2) Å respectively from the plane defined by the carbon atoms.

S2. Experimental

Under standard conditions for aromatic diimide formation (Hamilton *et al.*, 1998; Hamilton *et al.*, 1999) no evidence for the production of the desired acetylenic diimide could be found. Ring closure accompanied by unwanted addition of water across the acetylene bonds was observed using an alternative protocol (Ahn *et al.*, 1997), giving a very low yield (<5%) of diketone (I) after chromatographic isolation. 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 (ArH), 0.98 (CH$_3$) and 0.99Å (CH$_2$). The isotropic displacement parameters for all H atoms were set equal to 1.25U_{eq} of the carrier atom. A large residual electron density (0.60 eÅ^{-3}) is located 0.57Å from H4.
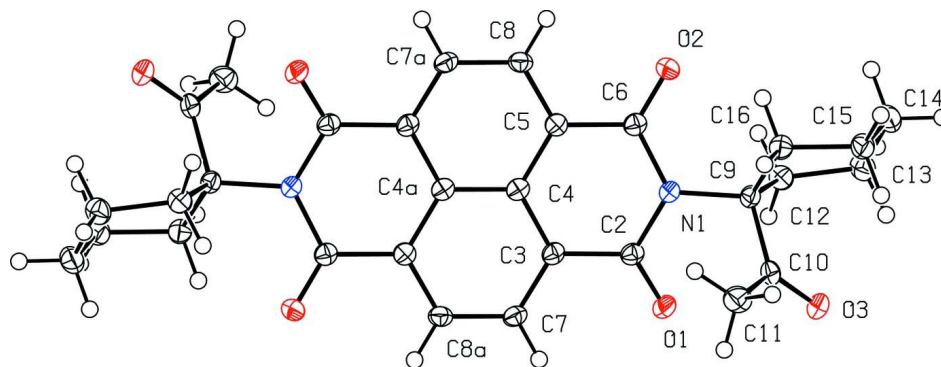
Figure 1

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

N,N'-Bis(1-acetylcyclohexyl)-1,8:4,5-naphthalenetetracarboximide

Crystal data

$C_{30}H_{30}N_2O_6$	$F(000) = 544$
$M_r = 514.56$	$D_x = 1.401$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2881 reflections
$a = 5.8553$ (2) Å	$\theta = 2.9$–27.5°
$b = 13.6603$ (6) Å	$\mu = 0.10$ mm^{-1}
$c = 15.2820$ (6) Å	$T = 120$ K
$\beta = 94.001$ (2)°	Plate, orange
$V = 1219.35$ (8) Å^3	$0.20 \times 0.18 \times 0.06$ mm
$Z = 2$	

Data collection

Bruker–Nonius 95 mm CCD camera on κ-goniostat diffractometer	$T_{min} = 0.981$, $T_{max} = 0.994$
	11932 measured reflections
Radiation source: Bruker Nonius FR591 rotating anode	2397 independent reflections
	1949 reflections with $I > 2\sigma(I)$
10 cm confocal mirrors monochromator	$R_{int} = 0.049$
Detector resolution: 9.091 pixels mm^{-1}	$\theta_{max} = 26.0°$, $\theta_{min} = 3.1°$
φ and ω scans	$h = -7 \rightarrow 7$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$k = -16 \rightarrow 16$
	$l = -18 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.063$	H-atom parameters constrained
$wR(F^2) = 0.150$	$w = 1/[\sigma^2(F_o^2) + (0.0786P)^2 + 0.2672P]$
$S = 1.17$	where $P = (F_o^2 + 2F_c^2)/3$
2397 reflections	$(\Delta/\sigma)_{max} < 0.001$
174 parameters	$\Delta\rho_{max} = 0.60$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.59$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.094 (8)

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 0.770335.

Geometry. Least-squares planes (x,y,z in crystal coordinates) and deviations from them (* indicates atom used to define plane) 3.5634 (0.0035) x - 0.8935 (0.0092) y + 11.4066 (0.0079) z = 7.0465 (0.0048) * -0.0167 (0.0010) C2 * 0.0088 (0.0016) C3 * -0.0098 (0.0013) C4 * -0.0071 (0.0012) C5 * 0.0161 (0.0011) C6 * 0.0087 (0.0011) C7 - 0.0027 (0.0028) C8 - 0.2429 (0.0022) N1 0.1089 (0.0026) O1 0.2471 (0.0023) O2 Rms deviation of fitted atoms = 0.0118

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.9857 (2)	0.37034 (9)	0.34838 (9)	0.0236 (4)
O2	0.4959 (2)	0.19444 (9)	0.49973 (9)	0.0223 (4)
O3	1.0165 (2)	0.19678 (10)	0.23338 (9)	0.0266 (4)
N1	0.6940 (3)	0.28341 (11)	0.40186 (10)	0.0170 (4)
C2	0.8167 (3)	0.37028 (13)	0.39014 (12)	0.0181 (4)
C3	0.7265 (3)	0.46151 (13)	0.42772 (11)	0.0173 (4)
C4	0.5438 (3)	0.45653 (13)	0.48277 (11)	0.0164 (4)
C5	0.4497 (3)	0.36554 (13)	0.50527 (12)	0.0171 (4)
C6	0.5449 (3)	0.27368 (13)	0.47037 (12)	0.0177 (4)
C7	0.8165 (3)	0.55098 (13)	0.40659 (12)	0.0190 (4)
H1	0.9401	0.5540	0.3695	0.024*
C8	0.2735 (3)	0.36217 (13)	0.56043 (12)	0.0190 (4)
H2	0.2125	0.3007	0.5763	0.024*
C9	0.7659 (3)	0.19434 (13)	0.35194 (12)	0.0173 (4)
C10	0.8453 (3)	0.22945 (13)	0.26254 (12)	0.0204 (5)
C11	0.6832 (4)	0.29318 (15)	0.20654 (13)	0.0273 (5)
H3	0.7698	0.3451	0.1795	0.034*
H4	0.5707	0.3226	0.2432	0.034*
H5	0.6039	0.2533	0.1606	0.034*
C12	0.9518 (3)	0.13813 (13)	0.40723 (13)	0.0210 (5)
H6	0.9015	0.1301	0.4673	0.026*
H7	1.0936	0.1778	0.4115	0.026*
C13	1.0070 (3)	0.03672 (14)	0.37068 (14)	0.0245 (5)
H8	1.0946	0.0450	0.3180	0.031*
H9	1.1052	0.0007	0.4151	0.031*
C14	0.7927 (3)	−0.02394 (15)	0.34620 (15)	0.0278 (5)
H10	0.8365	−0.0859	0.3182	0.035*
H11	0.7153	−0.0404	0.3999	0.035*
C15	0.6295 (3)	0.03341 (14)	0.28330 (13)	0.0234 (5)
H12	0.4928	−0.0069	0.2668	0.029*
H13	0.7062	0.0489	0.2292	0.029*
C16	0.5565 (3)	0.12816 (13)	0.32632 (13)	0.0202 (5)
H14	0.4763	0.1124	0.3795	0.025*
H15	0.4483	0.1640	0.2852	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0219 (7)	0.0232 (7)	0.0269 (8)	−0.0025 (5)	0.0105 (6)	−0.0044 (6)
O2	0.0292 (8)	0.0171 (7)	0.0213 (7)	−0.0026 (6)	0.0057 (6)	0.0012 (5)
O3	0.0294 (8)	0.0235 (7)	0.0284 (8)	0.0017 (6)	0.0124 (6)	−0.0047 (6)
N1	0.0176 (8)	0.0171 (8)	0.0166 (8)	0.0003 (6)	0.0036 (6)	−0.0009 (6)
C2	0.0186 (9)	0.0210 (10)	0.0146 (9)	−0.0016 (7)	0.0007 (7)	−0.0005 (7)
C3	0.0182 (9)	0.0209 (10)	0.0125 (9)	−0.0015 (7)	−0.0003 (7)	−0.0020 (7)
C4	0.0149 (9)	0.0206 (10)	0.0132 (9)	−0.0001 (7)	−0.0016 (7)	−0.0001 (7)
C5	0.0184 (9)	0.0185 (10)	0.0140 (9)	−0.0003 (7)	−0.0013 (7)	−0.0001 (7)
C6	0.0178 (9)	0.0204 (10)	0.0147 (9)	−0.0015 (8)	−0.0003 (7)	−0.0010 (7)
C7	0.0197 (9)	0.0223 (10)	0.0153 (9)	−0.0015 (8)	0.0042 (7)	0.0004 (7)
C8	0.0218 (10)	0.0194 (9)	0.0160 (10)	−0.0039 (8)	0.0024 (7)	0.0016 (7)
C9	0.0163 (9)	0.0167 (9)	0.0192 (10)	0.0011 (7)	0.0032 (7)	−0.0016 (7)
C10	0.0240 (10)	0.0152 (9)	0.0223 (10)	−0.0024 (8)	0.0032 (8)	−0.0051 (7)
C11	0.0334 (12)	0.0279 (11)	0.0202 (10)	−0.0019 (9)	−0.0006 (9)	0.0019 (8)
C12	0.0189 (10)	0.0208 (10)	0.0232 (10)	0.0003 (8)	0.0007 (8)	0.0005 (8)
C13	0.0209 (10)	0.0213 (10)	0.0312 (11)	0.0041 (8)	0.0017 (8)	0.0028 (8)
C14	0.0257 (11)	0.0188 (10)	0.0397 (12)	0.0005 (8)	0.0074 (9)	−0.0013 (9)
C15	0.0212 (10)	0.0214 (10)	0.0280 (11)	−0.0043 (8)	0.0042 (8)	−0.0057 (8)
C16	0.0174 (9)	0.0210 (10)	0.0221 (10)	−0.0008 (8)	0.0013 (7)	−0.0020 (8)

Geometric parameters (Å, º)

O1—C2	1.214 (2)	C9—C16	1.552 (3)
O2—C6	1.214 (2)	C10—C11	1.509 (3)
O3—C10	1.210 (2)	C11—H3	0.98
N1—C2	1.405 (2)	C11—H4	0.98
N1—C6	1.415 (2)	C11—H5	0.98
N1—C9	1.512 (2)	C12—C13	1.537 (3)
C2—C3	1.485 (3)	C12—H6	0.99
C3—C7	1.378 (3)	C12—H7	0.99
C3—C4	1.408 (3)	C13—C14	1.528 (3)
C4—C4^{i}	1.410 (3)	C13—H8	0.99
C4—C5	1.412 (3)	C13—H9	0.99
C5—C8	1.378 (3)	C14—C15	1.524 (3)
C5—C6	1.487 (3)	C14—H10	0.99
C7—C8^{i}	1.406 (3)	C14—H11	0.99
C7—H1	0.95	C15—C16	1.526 (3)
C8—C7^{i}	1.406 (3)	C15—H12	0.99
C8—H2	0.95	C15—H13	0.99
C9—C12	1.536 (3)	C16—H14	0.99
C9—C10	1.550 (3)	C16—H15	0.99
C2—N1—C6	121.39 (15)	H3—C11—H4	109.5
C2—N1—C9	116.90 (14)	C10—C11—H5	109.5
C6—N1—C9	120.37 (14)	H3—C11—H5	109.5

O1—C2—N1	120.69 (16)	H4—C11—H5	109.5
O1—C2—C3	121.85 (16)	C9—C12—C13	114.19 (16)
N1—C2—C3	117.42 (16)	C9—C12—H6	108.7
C7—C3—C4	120.12 (16)	C13—C12—H6	108.7
C7—C3—C2	120.09 (16)	C9—C12—H7	108.7
C4—C3—C2	119.76 (16)	C13—C12—H7	108.7
C3—C4—C4ⁱ	119.6 (2)	H6—C12—H7	107.6
C3—C4—C5	120.91 (16)	C14—C13—C12	112.81 (16)
C4ⁱ—C4—C5	119.5 (2)	C14—C13—H8	109.0
C8—C5—C4	120.00 (16)	C12—C13—H8	109.0
C8—C5—C6	120.44 (16)	C14—C13—H9	109.0
C4—C5—C6	119.55 (16)	C12—C13—H9	109.0
O2—C6—N1	122.08 (16)	H8—C13—H9	107.8
O2—C6—C5	121.05 (16)	C15—C14—C13	110.10 (16)
N1—C6—C5	116.87 (15)	C15—C14—H10	109.6
C3—C7—C8ⁱ	120.33 (17)	C13—C14—H10	109.6
C3—C7—H1	119.8	C15—C14—H11	109.6
C8ⁱ—C7—H1	119.8	C13—C14—H11	109.6
C5—C8—C7ⁱ	120.45 (17)	H10—C14—H11	108.2
C5—C8—H2	119.8	C14—C15—C16	110.27 (16)
C7ⁱ—C8—H2	119.8	C14—C15—H12	109.6
N1—C9—C12	109.66 (14)	C16—C15—H12	109.6
N1—C9—C10	107.88 (14)	C14—C15—H13	109.6
C12—C9—C10	113.28 (15)	C16—C15—H13	109.6
N1—C9—C16	110.69 (14)	H12—C15—H13	108.1
C12—C9—C16	111.39 (15)	C15—C16—C9	111.25 (15)
C10—C9—C16	103.78 (15)	C15—C16—H14	109.4
O3—C10—C11	120.53 (17)	C9—C16—H14	109.4
O3—C10—C9	121.16 (17)	C15—C16—H15	109.4
C11—C10—C9	117.60 (16)	C9—C16—H15	109.4
C10—C11—H3	109.5	H14—C16—H15	108.0
C10—C11—H4	109.5		
C6—N1—C2—O1	160.09 (17)	C2—C3—C7—C8ⁱ	177.80 (17)
C9—N1—C2—O1	−6.8 (2)	C4—C5—C8—C7ⁱ	1.1 (3)
C6—N1—C2—C3	−22.2 (2)	C6—C5—C8—C7ⁱ	−179.98 (17)
C9—N1—C2—C3	170.95 (15)	C2—N1—C9—C12	89.38 (18)
O1—C2—C3—C7	8.2 (3)	C6—N1—C9—C12	−77.62 (19)
N1—C2—C3—C7	−169.49 (17)	C2—N1—C9—C10	−34.4 (2)
O1—C2—C3—C4	−173.83 (17)	C6—N1—C9—C10	158.59 (15)
N1—C2—C3—C4	8.5 (2)	C2—N1—C9—C16	−147.33 (16)
C7—C3—C4—C4ⁱ	−1.0 (3)	C6—N1—C9—C16	45.7 (2)
C2—C3—C4—C4ⁱ	−179.01 (19)	N1—C9—C10—O3	136.51 (17)
C7—C3—C4—C5	−179.86 (17)	C12—C9—C10—O3	14.9 (2)
C2—C3—C4—C5	2.2 (3)	C16—C9—C10—O3	−106.01 (19)
C3—C4—C5—C8	178.90 (16)	N1—C9—C10—C11	−53.1 (2)
C4ⁱ—C4—C5—C8	0.1 (3)	C12—C9—C10—C11	−174.65 (15)
C3—C4—C5—C6	0.0 (3)	C16—C9—C10—C11	64.39 (19)

C4[i]—C4—C5—C6	−178.82 (19)	N1—C9—C12—C13	169.46 (15)
C2—N1—C6—O2	−155.80 (17)	C10—C9—C12—C13	−70.0 (2)
C9—N1—C6—O2	10.6 (3)	C16—C9—C12—C13	46.6 (2)
C2—N1—C6—C5	24.2 (2)	C9—C12—C13—C14	−48.2 (2)
C9—N1—C6—C5	−169.37 (15)	C12—C13—C14—C15	54.2 (2)
C8—C5—C6—O2	−11.5 (3)	C13—C14—C15—C16	−60.5 (2)
C4—C5—C6—O2	167.34 (17)	C14—C15—C16—C9	60.2 (2)
C8—C5—C6—N1	168.43 (16)	N1—C9—C16—C15	−174.79 (15)
C4—C5—C6—N1	−12.7 (2)	C12—C9—C16—C15	−52.5 (2)
C4—C3—C7—C8[i]	−0.2 (3)	C10—C9—C16—C15	69.71 (18)

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