Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,1′-Dibutyl-3,3′-biindolinylidene-2,2′-dione

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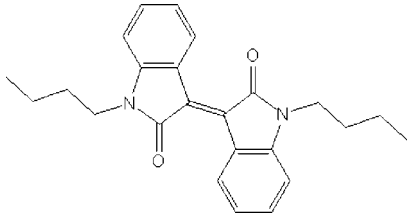
Received 3 September 2007; accepted 11 October 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.057; wR factor = 0.169; data-to-parameter ratio = 14.8.

The title molecule, $C_{24}H_{26}N_2O_2$, has its central C=C double bond placed on an inversion centre, and both indolin-2-one units are coplanar. The three terminal C atoms of the two butyl groups are disordered over two positions; the site occupancy factors are *ca.* 0.54 and 0.46. The central C=C bond exhibits an *E* configuration and is conjugated with the indole heterocycles. This aromatic character is related to the planarity of the isoindigo core and is reminiscent of that observed in stilbene.

Related literature

For the structure of isoindigo, see: von Eller-Pandraud (1960). For the properties of isoindigo derivatives, see: Sassatelli *et al.* (2004).



Experimental

Crystal data

$C_{24}H_{26}N_2O_2$
M_r = 374.47
Monoclinic, $P2_1/n$
a = 9.0301 (2) Å
b = 12.0559 (3) Å
c = 9.8618 (2) Å
β = 110.546 (2)°
V = 1005.32 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 (2) K
0.32 × 0.15 × 0.09 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.82, T_{max} = 0.99
7181 measured reflections
2321 independent reflections
1203 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.170
S = 1.00
2321 reflections
157 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

This work was supported by the National Natural Science Foundation of China (grant Nos. 20472044 and 50673054).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2134).

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  doi:10.1107/S160053680705009X

supplementary materials

Acta Cryst. (2007). E**63**, o4342 [**doi:10.1107/S160053680705009X**]

1,1'-Dibutyl-3,3'-biindolinylidene-2,2'-dione

M.-S. Yuan, Q. Fang, L. Ji and W.-T. Yu

Comment

Isoindigo, which contains a bis-indole framework, can be obtained from various natural sources. Its derivatives are usually known as useful medicines (Sassatelli *et al.*, 2004). Isoindigo may also be used as a precursor for the synthesis of organic two-photon absorption (TPA) compounds, because of its perfect planar π-conjugated structure. When exploring new TPA compounds, we obtained an intermediate compound 1,1'-dibutyl-isoindigo (I), for which we now report the synthesis and structure,

The molecule is centrosymmetric with its inversion centre placed at the midpoint of the C2═C2^{i} bond [symmetry code (*i*): 1 − *x*, 1 − *y*, −*z*], which thus presents an *E* conformation. The asymmetric unit thus contains one-half molecule. The two indole-2-one moieties are coplanar: the maximum displacement from the least-squares plane defined by 20 atoms of the isoindigo core is 0.036 (2) Å for O1 atom.

Both indole-2-one heterocycles are connected by the central C═C double bond, which has a bond length of 1.369 (4) Å, longer than typical C(sp^2)═C(sp^2) double bonds. On the other hand, The C2—C3 bond length, 1.476 (3) Å, is shorter than typical C(sp^3)—C(sp^3) single bonds. This means that the bonding in the fragment C3—C2═C2^{i}—C3^{i} is conjugated, as observed in stilbene. Considering the excellent planarity of (I), the π-conjugation of the title molecule should be better than that of stilbene, which is known as a TPA active compound. The geometry, conformation, and bond characters of (I) are very similar to those of isoindigo (von Eller-Pandraud, 1960). In (I), *n*-butyl groups bonded to the isoindigo core are disordered over two positions (Fig. 1).

There are no significant hydrogen bonds in the crystal structure (Fig. 2). However, the weak intramolecular interaction C4—H4···O1^{i} helps the isoindigo core of the molecule to keep a perfect planar conformation (Fig. 1). This intramolecular contact is characterized by a separation H4···O1^{i} = 2.05 Å and a C4—H4···O1^{i} angle of 137°.

Experimental

1-Butyl-1*H*-indole-2,3-dione (1.5 g) and 1-butyl-1*H*-indole-2-one (1.5 g) were mixed with polyphosphoric acid (15 g), reacted at 333–338 K for 30 min. under N_2, and then heated to 433–443 K with stirring. After 3 h, the mixture was poured into ice water and stirred for 1 h. The solution was extracted in chloroform and dried over Na_2SO_4. After removing the solvent, the crude product was purified by column chromatography on silica gel, eluting with petrol ether, affording the title compound (1.4 g, 47.1%). The compound was dissolved in THF and purple plate crystals of (I) formed on slow evaporatiion, at room temperature, over one week.

Refinement

Atoms C10, C11 and C12 were found to be disordered over two positions. Site occupation factors converged to 0.540 (8) [C10/C11/C12] and 0.460 (8) [C10'/C11'/C12']. Bond lengths C10—C11, C11—C12, C10'—C11', and C11'—C12' were restrained to 1.54 (1) Å. H atoms were positioned geometrically and allowed to ride on their carrier atom. The C—H bond lengths for aromatic, methyl and methylene groups were set to 0.93, 0.96 and 0.97 Å, respectively.

Figures

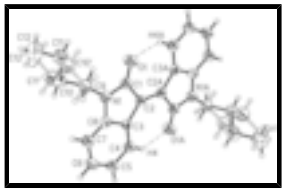

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

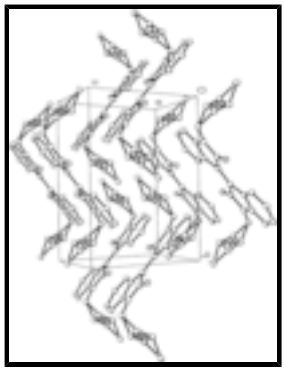

Fig. 2. The molecular packing of crystal (I). All H atoms have been omitted for clarity.

1,1'-Dibutyl-3,3'-biindolinylidene-2,2'-dione

Crystal data

$C_{24}H_{26}N_2O_2$	F_{000} = 400
M_r = 374.47	D_x = 1.237 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 977 reflections
a = 9.0301 (2) Å	θ = 2.8–20.6º
b = 12.0559 (3) Å	μ = 0.08 mm^{-1}
c = 9.8618 (2) Å	T = 293 (2) K
β = 110.546 (2)º	Plate, purple
V = 1005.32 (4) Å^3	0.32 × 0.15 × 0.09 mm
Z = 2	

Data collection

Bruker APEXII CCD area-detector diffractometer	2321 independent reflections

Radiation source: fine-focus sealed tube
Monochromator: graphite
Detector resolution: 10.00 pixels mm^{-1}
T = 293(2) K
φ and ω scans
Absorption correction: multi-scan (APEX2; Bruker, 2005)
T_{min} = 0.82, T_{max} = 0.99
7181 measured reflections
1203 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.6º
θ_{min} = 2.8º
h = −10→11
k = −15→13
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.170
S = 1.00
2321 reflections
157 parameters
4 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
$w = 1/[\sigma^2(F_o^2) + (0.0743P)^2 + 0.1566P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
N1	0.5019 (2)	0.30724 (15)	0.18760 (19)	0.0628 (5)	
C3	0.6752 (2)	0.39265 (16)	0.0975 (2)	0.0512 (5)	
C2	0.5247 (2)	0.45466 (16)	0.04392 (19)	0.0508 (5)	
C8	0.6545 (3)	0.30456 (18)	0.1826 (2)	0.0570 (6)	
O1	0.2828 (2)	0.41384 (15)	0.0981 (2)	0.0947 (7)	
C1	0.4184 (3)	0.39394 (19)	0.1093 (2)	0.0599 (6)	
C6	0.9168 (3)	0.2428 (2)	0.2345 (3)	0.0756 (7)	
H6	0.9986	0.1936	0.2799	0.091*	
C7	0.7717 (3)	0.2290 (2)	0.2498 (3)	0.0709 (7)	
H7	0.7537	0.1707	0.3037	0.085*	
C5	0.9414 (3)	0.3278 (2)	0.1536 (3)	0.0728 (7)	
H5	1.0400	0.3355	0.1446	0.087*	
C4	0.8229 (3)	0.40266 (19)	0.0847 (2)	0.0636 (6)	
H4	0.8422	0.4598	0.0298	0.076*	
C9	0.4387 (3)	0.2311 (2)	0.2693 (3)	0.0799 (8)	
H9A	0.3428	0.2628	0.2761	0.096*	0.540 (8)
H9B	0.5148	0.2246	0.3670	0.096*	0.540 (8)
H9C	0.3906	0.2761	0.3243	0.096*	0.460 (8)
H9D	0.5255	0.1906	0.3384	0.096*	0.460 (8)

C10	0.4018 (9)	0.1163 (7)	0.2045 (8)	0.088 (2)	0.540 (8)
H10A	0.4922	0.0888	0.1833	0.106*	0.540 (8)
H10B	0.3842	0.0669	0.2750	0.106*	0.540 (8)
C11	0.2597 (10)	0.1148 (7)	0.0693 (8)	0.112 (3)	0.540 (8)
H11A	0.2804	0.1587	−0.0046	0.134*	0.540 (8)
H11B	0.1709	0.1478	0.0881	0.134*	0.540 (8)
C12	0.217 (3)	−0.0054 (14)	0.014 (2)	0.183 (8)	0.540 (8)
H12A	0.1180	−0.0055	−0.0654	0.274*	0.540 (8)
H12B	0.2095	−0.0510	0.0906	0.274*	0.540 (8)
H12C	0.2984	−0.0341	−0.0188	0.274*	0.540 (8)
C10'	0.3178 (15)	0.1506 (8)	0.1785 (13)	0.101 (3)	0.460 (8)
H10C	0.2780	0.1067	0.2408	0.121*	0.460 (8)
H10D	0.2296	0.1914	0.1119	0.121*	0.460 (8)
C11'	0.3837 (11)	0.0751 (6)	0.0947 (11)	0.105 (3)	0.460 (8)
H11C	0.4727	0.0344	0.1605	0.125*	0.460 (8)
H11D	0.4211	0.1182	0.0301	0.125*	0.460 (8)
C12'	0.254 (3)	−0.0077 (13)	0.0052 (18)	0.126 (6)	0.460 (8)
H12D	0.2979	−0.0570	−0.0473	0.190*	0.460 (8)
H12E	0.1675	0.0327	−0.0617	0.190*	0.460 (8)
H12F	0.2169	−0.0499	0.0694	0.190*	0.460 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0559 (12)	0.0657 (12)	0.0699 (11)	0.0018 (10)	0.0258 (9)	0.0148 (10)
C3	0.0481 (12)	0.0534 (12)	0.0515 (11)	−0.0018 (10)	0.0168 (9)	−0.0030 (9)
C2	0.0484 (13)	0.0523 (12)	0.0547 (12)	−0.0042 (10)	0.0217 (10)	−0.0047 (9)
C8	0.0519 (14)	0.0619 (14)	0.0559 (12)	−0.0017 (11)	0.0173 (10)	−0.0004 (10)
O1	0.0688 (12)	0.0964 (14)	0.1388 (16)	0.0190 (10)	0.0611 (11)	0.0470 (12)
C1	0.0544 (14)	0.0634 (14)	0.0666 (13)	0.0014 (12)	0.0270 (11)	0.0055 (11)
C6	0.0585 (16)	0.0811 (17)	0.0828 (16)	0.0139 (13)	0.0192 (13)	0.0164 (14)
C7	0.0652 (16)	0.0703 (16)	0.0761 (15)	0.0069 (13)	0.0236 (12)	0.0185 (13)
C5	0.0507 (14)	0.0808 (17)	0.0896 (17)	0.0049 (13)	0.0279 (13)	0.0079 (14)
C4	0.0561 (14)	0.0657 (15)	0.0713 (14)	0.0005 (12)	0.0253 (12)	0.0058 (11)
C9	0.0768 (19)	0.0812 (18)	0.0902 (17)	−0.0015 (15)	0.0400 (15)	0.0249 (15)
C10	0.076 (5)	0.078 (5)	0.114 (6)	−0.001 (4)	0.038 (4)	0.024 (4)
C11	0.133 (7)	0.100 (6)	0.095 (5)	0.017 (5)	0.030 (5)	−0.005 (4)
C12	0.169 (13)	0.141 (15)	0.241 (18)	0.001 (10)	0.074 (11)	0.012 (11)
C10'	0.090 (8)	0.101 (7)	0.119 (9)	0.004 (6)	0.044 (7)	0.031 (6)
C11'	0.106 (7)	0.108 (6)	0.106 (7)	0.001 (5)	0.045 (5)	0.007 (5)
C12'	0.174 (15)	0.098 (10)	0.098 (7)	−0.067 (9)	0.037 (8)	−0.033 (7)

Geometric parameters (Å, °)

N1—C1	1.360 (3)	C9—H9C	0.9700
N1—C8	1.397 (3)	C9—H9D	0.9701
N1—C9	1.463 (3)	C10—C11	1.492 (8)
C3—C4	1.389 (3)	C10—H10A	0.9700
C3—C8	1.406 (3)	C10—H10B	0.9700

C3—C2	1.476 (3)	C11—C12	1.549 (9)
C2—C2^{i}	1.369 (4)	C11—H11A	0.9700
C2—C1	1.519 (3)	C11—H11B	0.9700
C8—C7	1.377 (3)	C12—H12A	0.9600
O1—C1	1.214 (2)	C12—H12B	0.9600
C6—C5	1.364 (3)	C12—H12C	0.9600
C6—C7	1.380 (3)	C10'—C11'	1.486 (9)
C6—H6	0.9300	C10'—H10C	0.9700
C7—H7	0.9300	C10'—H10D	0.9700
C5—C4	1.382 (3)	C11'—C12'	1.556 (9)
C5—H5	0.9300	C11'—H11C	0.9700
C4—H4	0.9300	C11'—H11D	0.9700
C9—C10'	1.500 (12)	C12'—H12D	0.9600
C9—C10	1.511 (9)	C12'—H12E	0.9600
C9—H9A	0.9700	C12'—H12F	0.9600
C9—H9B	0.9700		
C1—N1—C8	110.78 (17)	N1—C9—H9D	109.1
C1—N1—C9	123.35 (18)	C10'—C9—H9D	109.5
C8—N1—C9	125.83 (19)	C10—C9—H9D	80.8
C4—C3—C8	116.91 (19)	H9A—C9—H9D	132.2
C4—C3—C2	135.73 (19)	H9C—C9—H9D	107.4
C8—C3—C2	107.34 (17)	C11—C10—C9	112.5 (7)
C2^{i}—C2—C3	133.3 (2)	C11—C10—H10A	109.1
C2^{i}—C2—C1	122.6 (2)	C9—C10—H10A	109.1
C3—C2—C1	104.10 (17)	C11—C10—H10B	109.1
C7—C8—N1	126.8 (2)	C9—C10—H10B	109.1
C7—C8—C3	123.1 (2)	H10A—C10—H10B	107.8
N1—C8—C3	110.06 (18)	C10—C11—C12	110.9 (11)
O1—C1—N1	122.95 (19)	C10—C11—H11A	109.5
O1—C1—C2	129.3 (2)	C12—C11—H11A	109.5
N1—C1—C2	107.71 (18)	C10—C11—H11B	109.5
C5—C6—C7	120.6 (2)	C12—C11—H11B	109.5
C5—C6—H6	119.7	H11A—C11—H11B	108.1
C7—C6—H6	119.7	C11'—C10'—C9	112.2 (9)
C8—C7—C6	117.8 (2)	C11'—C10'—H10C	109.2
C8—C7—H7	121.1	C9—C10'—H10C	109.2
C6—C7—H7	121.1	C11'—C10'—H10D	109.2
C6—C5—C4	121.4 (2)	C9—C10'—H10D	109.2
C6—C5—H5	119.3	H10C—C10'—H10D	107.9
C4—C5—H5	119.3	C10'—C11'—C12'	109.9 (11)
C5—C4—C3	120.1 (2)	C10'—C11'—H11C	109.7
C5—C4—H4	120.0	C12'—C11'—H11C	109.7
C3—C4—H4	120.0	C10'—C11'—H11D	109.7
N1—C9—C10'	114.8 (4)	C12'—C11'—H11D	109.7
N1—C9—C10	114.3 (3)	H11C—C11'—H11D	108.2
N1—C9—H9A	108.7	C11'—C12'—H12D	109.5
C10—C9—H9A	108.7	C11'—C12'—H12E	109.5
N1—C9—H9B	108.7	H12D—C12'—H12E	109.5

C10—C9—H9B	108.7	C11'—C12'—H12F	109.5
H9A—C9—H9B	107.6	H12D—C12'—H12F	109.5
N1—C9—H9C	107.1	H12E—C12'—H12F	109.5
C10'—C9—H9C	108.7		
C4—C3—C2—C2^{i}	2.3 (5)	C3—C2—C1—N1	−1.0 (2)
C8—C3—C2—C2^{i}	−178.7 (3)	N1—C8—C7—C6	−178.2 (2)
C4—C3—C2—C1	−178.0 (2)	C3—C8—C7—C6	1.4 (4)
C8—C3—C2—C1	0.9 (2)	C5—C6—C7—C8	−0.9 (4)
C1—N1—C8—C7	179.5 (2)	C7—C6—C5—C4	0.1 (4)
C9—N1—C8—C7	1.7 (4)	C6—C5—C4—C3	0.3 (3)
C1—N1—C8—C3	−0.1 (2)	C8—C3—C4—C5	0.2 (3)
C9—N1—C8—C3	−177.9 (2)	C2—C3—C4—C5	179.0 (2)
C4—C3—C8—C7	−1.0 (3)	C1—N1—C9—C10'	73.5 (5)
C2—C3—C8—C7	179.8 (2)	C8—N1—C9—C10'	−108.9 (5)
C4—C3—C8—N1	178.60 (18)	C1—N1—C9—C10	108.4 (4)
C2—C3—C8—N1	−0.6 (2)	C8—N1—C9—C10	−74.0 (4)
C8—N1—C1—O1	−179.6 (2)	N1—C9—C10—C11	−71.8 (8)
C9—N1—C1—O1	−1.7 (4)	C10'—C9—C10—C11	26.5 (8)
C8—N1—C1—C2	0.7 (2)	C9—C10—C11—C12	−175.1 (12)
C9—N1—C1—C2	178.54 (19)	N1—C9—C10'—C11'	62.3 (10)
C2^{i}—C2—C1—O1	−1.0 (4)	C10—C9—C10'—C11'	−34.3 (7)
C3—C2—C1—O1	179.3 (2)	C9—C10'—C11'—C12'	178.9 (10)
C2^{i}—C2—C1—N1	178.7 (2)		

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

Fig. 1

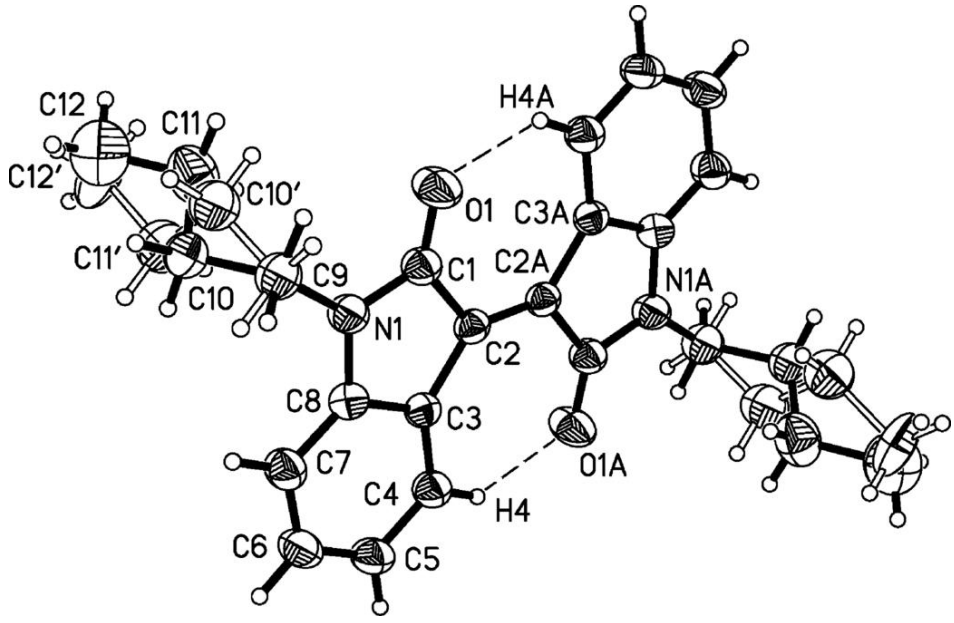
C12
C11
C12′
C10′
C11′
C10
C9
N1
C8
C7
C6
C5
C4
C3
C2
C1
O1
H4A
C3A
C2A
N1A
O1A
H4

Fig. 2