organic compounds

3,6-Diiodo-9*H*-carbazole

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In the title compound, $C_{12}H_7I_2N$, the tricyclic aromatic ring system is essentially planar, with an r.m.s. deviation of 0.0272 Å. The two I atoms are marginally out of plane, with the C—I bonds angled at 3.9 (2) and 1.1 (2)° with respect to the planes of their respective benzene rings, above and below the plane of the carbazole ring system. No classical hydrogen bonds are observed in the crystal structure.

Related literature

For the synthesis of the title compound, see: Tucker (1926); Lengvinaite *et al.* (2007). For related compounds, see: Grigalevicius *et al.* (2007); Cui *et al.* (2009); Tian *et al.* (2010); Klejevskaja *et al.* (2007). For their applications, see: Zhang *et al.* (2009); Zhong'an *et al.* (2010); Lu *et al.* (2006); Grigalevicius *et al.* (2006, 2011).
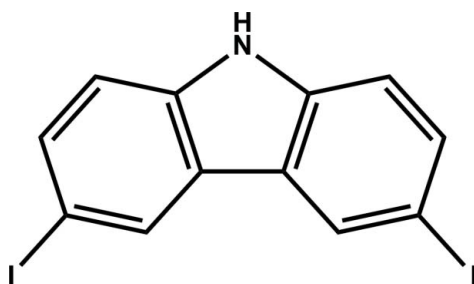
Experimental

Crystal data

$C_{12}H_7I_2N$	Orthorhombic, $Pbca$
$M_r = 418.99$	$a = 11.8823$ (14) Å
$b = 7.8835$ (9) Å	Mo $K\alpha$ radiation
$c = 24.835$ (3) Å	$\mu = 5.37$ mm^{-1}
$V = 2326.4$ (5) Å^3	$T = 293$ K
$Z = 8$	$0.23 \times 0.21 \times 0.18$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	12303 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	2456 independent reflections
$T_{min} = 0.371$, $T_{max} = 0.445$	1879 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.032$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.026$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.052$	
$S = 1.05$	$\Delta\rho_{max} = 0.57$ e Å^{-3}
2456 reflections	$\Delta\rho_{min} = -0.56$ e Å^{-3}
141 parameters	

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT-Plus* (Bruker, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PK2397).

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supporting information

3,6-Diiodo-9*H*-carbazole

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S1. Comment

Carbazole moieties are an important construction block for hole-transporting and electroluminescent materials. 3,6-di-iodo-9*H*-carbazole has been used to design and synthesize carbazole derivatives. We report herein the crystal structure of the title compound. The molecular structure is shown in Fig. 1. All bond lengths and angles are within normal ranges. The tricyclic aromatic ring system is essentially planar with an r.m.s. deviation of 0.0272 Å. There are no classical hydrogen bonds observed in the crystal structure.

S2. Experimental

The title compound was synthesized according to a literature method (Tucker, 1926). Colorless crystals were obtained from a solution in chloroform upon slow evaporation of the solvent.

S3. Refinement

The nitrogen-bound H atom was located in a difference Fourier map and refined freely (N—H = 0.75 (3) Å). Carbon-bound H atoms were positioned geometrically (C—H = 0.93 Å) and refined using a riding model, with $U_{iso}(H) = 1.2$ $U_{eq}(C)$.
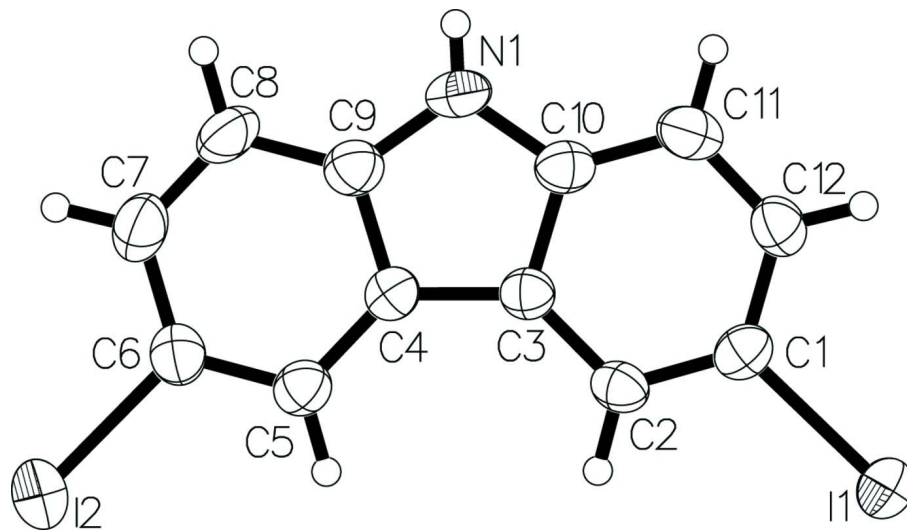

Figure 1

The molecular structure of the title coumpound. Displacement ellipsoids are drawn at 30% probability level..

3,6-Diiodo-9*H*-carbazole

Crystal data

$C_{12}H_7I_2N$	$F(000) = 1536$
$M_r = 418.99$	$D_x = 2.392$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 12303 reflections
$a = 11.8823$ (14) Å	$\theta = 1.6–26.8°$
$b = 7.8835$ (9) Å	$\mu = 5.37$ mm^{-1}
$c = 24.835$ (3) Å	$T = 293$ K
$V = 2326.4$ (5) Å^3	Block, colorless
$Z = 8$	$0.23 \times 0.21 \times 0.18$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer	12303 measured reflections
Radiation source: fine-focus sealed tube	2456 independent reflections
Graphite monochromator	1879 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.032$
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	$\theta_{max} = 26.8°$, $\theta_{min} = 1.6°$
	$h = -8 \rightarrow 15$
	$k = -9 \rightarrow 9$
$T_{min} = 0.371$, $T_{max} = 0.445$	$l = -31 \rightarrow 29$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.026$	
$wR(F^2) = 0.052$	$w = 1/[\sigma^2(F_o^2) + (0.0193P)^2 + 0.8116P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	
2456 reflections	$(\Delta/\sigma)_{max} = 0.002$
141 parameters	$\Delta\rho_{max} = 0.57$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.56$ 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: 0.00021 (4)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C11	0.7583 (3)	0.4740 (4)	0.77946 (15)	0.0445 (9)
H11	0.8290	0.5240	0.7822	0.053*
C12	0.6978 (3)	0.4308 (4)	0.82476 (14)	0.0406 (9)

H12	0.7277	0.4525	0.8587	0.049*
H1N	0.807 (3)	0.511 (4)	0.6716 (13)	0.032 (11)*
I2	0.35650 (2)	0.22435 (4)	0.536173 (10)	0.06040 (11)
I1	0.50690 (2)	0.28678 (4)	0.891514 (9)	0.04915 (10)
N1	0.7525 (3)	0.4684 (4)	0.67876 (13)	0.0460 (8)
C5	0.4904 (3)	0.2882 (4)	0.63769 (13)	0.0387 (8)
H5	0.4264	0.2467	0.6548	0.046*
C4	0.5818 (3)	0.3460 (4)	0.66751 (12)	0.0333 (8)
C6	0.4967 (3)	0.2939 (5)	0.58238 (14)	0.0432 (9)
C9	0.6766 (3)	0.4125 (4)	0.64077 (14)	0.0402 (9)
C1	0.5918 (3)	0.3545 (4)	0.82033 (13)	0.0373 (8)
C2	0.5437 (3)	0.3208 (4)	0.77121 (13)	0.0357 (8)
H2	0.4730	0.2705	0.7690	0.043*
C3	0.6032 (3)	0.3638 (4)	0.72449 (12)	0.0333 (8)
C8	0.6826 (3)	0.4177 (5)	0.58496 (14)	0.0478 (10)
H8	0.7461	0.4603	0.5677	0.057*
C7	0.5931 (3)	0.3587 (5)	0.55598 (14)	0.0492 (10)
H7	0.5954	0.3612	0.5186	0.059*
C10	0.7105 (3)	0.4408 (4)	0.72961 (14)	0.0375 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.033 (2)	0.047 (2)	0.054 (2)	−0.0035 (18)	0.0000 (19)	−0.0083 (18)
C12	0.040 (2)	0.043 (2)	0.0391 (19)	0.0011 (17)	−0.0046 (17)	−0.0074 (16)
I2	0.0586 (2)	0.0815 (2)	0.04106 (16)	−0.00842 (15)	−0.01063 (13)	0.00035 (13)
I1	0.04885 (17)	0.06034 (18)	0.03825 (14)	−0.00679 (13)	0.00108 (12)	0.00815 (11)
N1	0.0340 (19)	0.055 (2)	0.0490 (18)	−0.0110 (17)	0.0093 (18)	0.0011 (16)
C5	0.036 (2)	0.040 (2)	0.039 (2)	0.0034 (17)	0.0027 (17)	0.0040 (16)
C4	0.034 (2)	0.0312 (18)	0.0345 (18)	0.0029 (15)	0.0017 (16)	0.0029 (14)
C6	0.044 (2)	0.048 (2)	0.0375 (19)	−0.0003 (19)	−0.0025 (17)	−0.0026 (17)
C9	0.036 (2)	0.043 (2)	0.042 (2)	0.0021 (17)	0.0030 (17)	0.0004 (16)
C1	0.038 (2)	0.0355 (19)	0.0385 (18)	0.0031 (17)	0.0052 (17)	0.0040 (16)
C2	0.0298 (18)	0.034 (2)	0.0427 (19)	−0.0003 (15)	−0.0044 (16)	0.0050 (15)
C3	0.0312 (19)	0.0328 (19)	0.0358 (18)	0.0010 (15)	0.0002 (15)	0.0010 (14)
C8	0.041 (2)	0.061 (3)	0.041 (2)	0.003 (2)	0.0151 (18)	0.0017 (18)
C7	0.055 (3)	0.059 (3)	0.0341 (19)	0.005 (2)	0.0050 (19)	−0.0020 (17)
C10	0.0331 (19)	0.037 (2)	0.043 (2)	0.0003 (16)	0.0043 (17)	−0.0027 (16)

Geometric parameters (Å, º)

C11—C12	1.378 (5)	C5—H5	0.9300
C11—C10	1.387 (5)	C4—C9	1.409 (5)
C11—H11	0.9300	C4—C3	1.445 (4)
C12—C1	1.400 (5)	C6—C7	1.415 (5)
C12—H12	0.9300	C9—C8	1.389 (5)
I2—C6	2.096 (4)	C1—C2	1.373 (4)
I1—C1	2.104 (3)	C2—C3	1.400 (4)

N1—C10	1.375 (4)	C2—H2	0.9300
N1—C9	1.378 (5)	C3—C10	1.418 (5)
N1—H1N	0.75 (3)	C8—C7	1.366 (5)
C5—C6	1.376 (5)	C8—H8	0.9300
C5—C4	1.391 (5)	C7—H7	0.9300
C12—C11—C10	117.9 (3)	C8—C9—C4	121.5 (3)
C12—C11—H11	121.0	C2—C1—C12	121.8 (3)
C10—C11—H11	121.0	C2—C1—I1	119.8 (3)
C11—C12—C1	120.8 (3)	C12—C1—I1	118.3 (3)
C11—C12—H12	119.6	C1—C2—C3	118.6 (3)
C1—C12—H12	119.6	C1—C2—H2	120.7
C10—N1—C9	109.9 (3)	C3—C2—H2	120.7
C10—N1—H1N	127 (3)	C2—C3—C10	118.9 (3)
C9—N1—H1N	123 (3)	C2—C3—C4	134.4 (3)
C6—C5—C4	118.5 (3)	C10—C3—C4	106.7 (3)
C6—C5—H5	120.7	C7—C8—C9	118.4 (4)
C4—C5—H5	120.7	C7—C8—H8	120.8
C5—C4—C9	119.7 (3)	C9—C8—H8	120.8
C5—C4—C3	133.7 (3)	C8—C7—C6	120.6 (3)
C9—C4—C3	106.6 (3)	C8—C7—H7	119.7
C5—C6—C7	121.2 (3)	C6—C7—H7	119.7
C5—C6—I2	119.6 (3)	N1—C10—C11	129.9 (3)
C7—C6—I2	118.9 (3)	N1—C10—C3	108.2 (3)
N1—C9—C8	129.8 (3)	C11—C10—C3	121.9 (3)
N1—C9—C4	108.7 (3)		
C10—C11—C12—C1	0.5 (5)	C5—C4—C3—C2	4.6 (7)
C6—C5—C4—C9	1.7 (5)	C9—C4—C3—C2	−179.0 (4)
C6—C5—C4—C3	177.7 (4)	C5—C4—C3—C10	−176.4 (4)
C4—C5—C6—C7	−0.8 (5)	C9—C4—C3—C10	0.1 (4)
C4—C5—C6—I2	−175.7 (2)	N1—C9—C8—C7	−177.6 (4)
C10—N1—C9—C8	178.7 (4)	C4—C9—C8—C7	1.0 (5)
C10—N1—C9—C4	0.0 (4)	C9—C8—C7—C6	−0.1 (6)
C5—C4—C9—N1	177.0 (3)	C5—C6—C7—C8	0.0 (6)
C3—C4—C9—N1	0.0 (4)	I2—C6—C7—C8	174.9 (3)
C5—C4—C9—C8	−1.8 (5)	C9—N1—C10—C11	178.8 (4)
C3—C4—C9—C8	−178.8 (3)	C9—N1—C10—C3	0.0 (4)
C11—C12—C1—C2	−0.4 (5)	C12—C11—C10—N1	−179.0 (4)
C11—C12—C1—I1	178.5 (3)	C12—C11—C10—C3	−0.4 (5)
C12—C1—C2—C3	0.3 (5)	C2—C3—C10—N1	179.1 (3)
I1—C1—C2—C3	−178.6 (2)	C4—C3—C10—N1	−0.1 (4)
C1—C2—C3—C10	−0.2 (5)	C2—C3—C10—C11	0.3 (5)
C1—C2—C3—C4	178.7 (4)	C4—C3—C10—C11	−178.9 (3)