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1-Iodotriptycene

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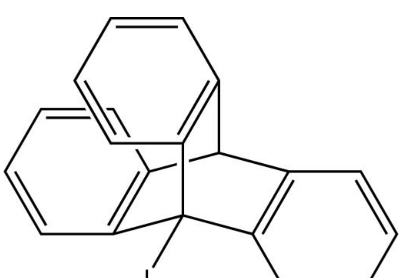
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.003 Å; R factor = 0.021; wR factor = 0.059; data-to-parameter ratio = 18.5.

The title compound, $C_{20}H_{13}I$, is a halogenated derivative of triptycene. The molecule shows crystallographic as well as non-crystallographic C_3 symmetry. The asymmetric unit comprises one third of the molecule. Dispersive I···I contacts [I···I = 3.6389 (3) Å] connect the molecules into dimers. The shortest centroid–centroid distance between two π-systems is 3.8403 (12) Å.

Related literature

For the crystal structures of 1-bromotriptycene, 9,10-dibromotriptycene and 10-bromo-9-triptycyl iodoformate, see: Palmer & Templeton (1968), Abergel & Dinca (2004) and de Wet *et al.* (1978), respectively. For the preparation, see: Bartel *et al.* (1971).



Experimental

Crystal data

$C_{20}H_{13}I$
M_r = 380.20
Hexagonal, $R\bar{3}$
a = 11.8820 (4) Å
c = 17.6800 (5) Å
V = 2161.68 (12) Å^3
Z = 6
Mo $K\alpha$ radiation
μ = 2.21 mm^{-1}
T = 200 K
0.56 × 0.51 × 0.25 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.568, T_{max} = 0.746
4033 measured reflections
1184 independent reflections
1156 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.021
$wR(F^2)$ = 0.059
S = 1.15
1184 reflections
64 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Data collection: *APEX2* (Bruker, 2010); cell refinement: *SAINT* (Bruker, 2010); data reduction: *SAINT*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NK2107).

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supplementary materials

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1-Iodotriptycene

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Comment

The chemistry of molecules featuring double and triple bonds involving elements from the third row of the periodic system of the elements (or below) is affected by the marked tendency of oligo- and polymerization. The introduction of sterically demanding, "bulky" protective groups in proximity to such bonding systems allowed the isolation and characterization of respective compounds on grounds of steric shielding and, as a consequence, markedly decreased rate of polymerization. It seemed of interest for us to study whether the presence of such aforementioned bonding systems has an influence on the metrical parameters of the applied protection groups as well. Therefore, we determined the crystal structure of the title compound. So far, the molecular and crystal stuctures of 1-bromotriptycene (Palmer & Templeton, 1968), 9,10-dibromo-triptycene (Abergel & Dinca, 2004) as well as 10-bromo-9-triptycyl iodoformate (de Wet *et al.*, 1978) are the only examples of structurally characterized triptycene compounds bearing a halogenido substituent on the bridgehead carbon atom present in the literature.

Halogenation took place on one of the bridgehead carbon atoms of the triptycene molecule (Figure 1). The least-squares planes defined by the atoms of the three aromatic moieties enclose angles of 60.03 (4) ° and 60.03 (7), respectively.

In the molecules, dispersive I···I contacts whose range falls by more than 0.3 Å below the sum of van der Waals radii can be observed (Figure 2). These connect two molecules to dimeric units whose I···I vector is pointing along the crystallographic *c* axis. The aromatic moieties of one molecule in such a dimer adopt a staggered conformation towards the aromatic moieties in the other molecule when projected along the I···I axis. The closest intercentroid distance between two π-systems was measured at 3.8403 (12) Å.

Experimental

The compound was formed through the thermolysis of 9-triptycyl iodoformate according to a published procedure (Bartel *et al.*, 1971).

Refinement

Carbon-bound H atoms were placed in calculated positions (C—H 0.95 Å for aromatic carbon atoms, C—H 1.00 Å for the bridgehead carbon atom) and were included in the refinement in the riding model approximation, with U(H) set to 1.2U_{eq}(C).

Figures

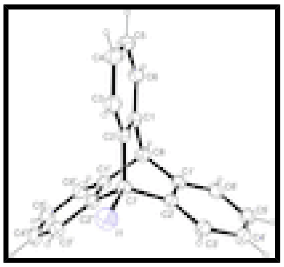

Fig. 1. The molecular structure of the title compound, anisotropic displacement ellipsoids are drawn at 50% probability level. Symmetry operators: [i] *-y*, *x-y*, *z*; [ii] *-x* + *y*, *-x*, *z*.

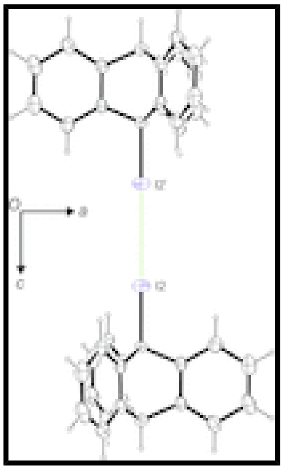

Fig. 2. Intermolecular I···I contact, viewed along [0 - 1 0]. Symmetry operator: [i] *-x*, *-y*, *-z*.

9-iodo-9,10-dihydro-9,10[1',2']-benzenoanthracene

Crystal data

$C_{20}H_{13}I$	D_x = 1.752 Mg m^{-3}
M_r = 380.20	Mo *K*α radiation, λ = 0.71069 Å
Hexagonal, $R\overline{3}$	Cell parameters from 3561 reflections
Hall symbol: -R 3	θ = 4.1–28.3°
a = 11.8820 (4) Å	μ = 2.21 mm^{-1}
c = 17.6800 (5) Å	*T* = 200 K
V = 2161.68 (12) Å^3	Block, colourless
Z = 6	0.56 × 0.51 × 0.25 mm
F(000) = 1116	

Data collection

Bruker APEXII CCD diffractometer	1184 independent reflections
Radiation source: fine-focus sealed tube	1156 reflections with $I > 2\sigma(I)$
graphite	R_{int} = 0.011
φ and ω scans	θ_{max} = 28.3°, θ_{min} = 3.5°
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)	*h* = −15→10

T_{min} = 0.568, T_{max} = 0.746	k = −13→15
4033 measured reflections	l = −20→23

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.021$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.059$	H-atom parameters constrained
S = 1.15	$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 2.801P]$ where $P = (F_o^2 + 2F_c^2)/3$
1184 reflections	$(\Delta/\sigma)_{max} < 0.001$
64 parameters	$\Delta\rho_{max}$ = 1.52 e Å^{-3}
0 restraints	$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
I1	0.0000	0.0000	0.102907 (11)	0.03354 (10)
C1	0.11904 (15)	0.11668 (15)	0.33674 (10)	0.0219 (3)
C2	0.12090 (15)	0.11714 (15)	0.25794 (10)	0.0207 (3)
C3	0.22522 (17)	0.21626 (17)	0.21950 (11)	0.0264 (3)
H3	0.2274	0.2169	0.1658	0.032*
C4	0.32674 (18)	0.31494 (17)	0.26061 (13)	0.0330 (4)
H4	0.3988	0.3826	0.2346	0.040*
C5	0.32397 (18)	0.31565 (18)	0.33846 (14)	0.0337 (4)
H5	0.3933	0.3842	0.3657	0.040*
C6	0.21961 (18)	0.21594 (17)	0.37755 (12)	0.0284 (4)
H6	0.2174	0.2160	0.4313	0.034*
C7	0.0000	0.0000	0.22412 (15)	0.0188 (5)
C8	0.0000	0.0000	0.37075 (17)	0.0224 (5)
H8	0.0000	0.0000	0.4273	0.027*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.04045 (12)	0.04045 (12)	0.01971 (13)	0.02022 (6)	0.000	0.000
C1	0.0206 (7)	0.0206 (7)	0.0262 (8)	0.0115 (6)	−0.0022 (6)	−0.0016 (6)
C2	0.0177 (7)	0.0176 (7)	0.0276 (8)	0.0094 (6)	−0.0006 (6)	−0.0004 (6)
C3	0.0230 (7)	0.0231 (7)	0.0318 (9)	0.0107 (6)	0.0039 (6)	0.0044 (6)
C4	0.0212 (8)	0.0203 (8)	0.0530 (12)	0.0071 (6)	0.0005 (7)	0.0042 (8)
C5	0.0238 (8)	0.0206 (7)	0.0538 (12)	0.0089 (7)	−0.0117 (8)	−0.0056 (8)
C6	0.0280 (8)	0.0254 (8)	0.0349 (9)	0.0157 (7)	−0.0098 (7)	−0.0072 (7)
C7	0.0202 (7)	0.0202 (7)	0.0162 (12)	0.0101 (4)	0.000	0.000
C8	0.0242 (8)	0.0242 (8)	0.0189 (13)	0.0121 (4)	0.000	0.000

Geometric parameters (Å, °)

I1—C7	2.143 (3)	C4—H4	0.9500
C1—C6	1.389 (2)	C5—C6	1.396 (3)
C1—C2	1.393 (2)	C5—H5	0.9500
C1—C8	1.524 (2)	C6—H6	0.9500
C2—C3	1.388 (2)	C7—C2^{i}	1.5359 (19)
C2—C7	1.5359 (19)	C7—C2^{ii}	1.5359 (19)
C3—C4	1.394 (3)	C8—C1^{i}	1.524 (2)
C3—H3	0.9500	C8—C1^{ii}	1.524 (2)
C4—C5	1.377 (4)	C8—H8	1.0000
C6—C1—C2	120.71 (16)	C1—C6—C5	119.03 (19)
C6—C1—C8	125.48 (18)	C1—C6—H6	120.5
C2—C1—C8	113.81 (16)	C5—C6—H6	120.5
C3—C2—C1	119.92 (16)	C2—C7—C2^{i}	105.82 (13)
C3—C2—C7	127.75 (17)	C2—C7—C2^{ii}	105.82 (13)
C1—C2—C7	112.33 (16)	C2^{i}—C7—C2^{ii}	105.82 (13)
C2—C3—C4	119.24 (18)	C2—C7—I1	112.92 (11)
C2—C3—H3	120.4	C2^{i}—C7—I1	112.92 (11)
C4—C3—H3	120.4	C2^{ii}—C7—I1	112.92 (11)
C5—C4—C3	120.87 (17)	C1^{i}—C8—C1^{ii}	105.46 (14)
C5—C4—H4	119.6	C1^{i}—C8—C1	105.46 (14)
C3—C4—H4	119.6	C1^{ii}—C8—C1	105.46 (14)
C4—C5—C6	120.22 (17)	C1^{i}—C8—H8	113.2
C4—C5—H5	119.9	C1^{ii}—C8—H8	113.2
C6—C5—H5	119.9	C1—C8—H8	113.2
C6—C1—C2—C3	1.2 (2)	C3—C2—C7—C2^{i}	122.9 (2)
C8—C1—C2—C3	−178.53 (13)	C1—C2—C7—C2^{i}	−56.63 (15)
C6—C1—C2—C7	−179.19 (13)	C3—C2—C7—C2^{ii}	−125.1 (2)
C8—C1—C2—C7	1.07 (16)	C1—C2—C7—C2^{ii}	55.38 (16)
C1—C2—C3—C4	−0.4 (2)	C3—C2—C7—I1	−1.06 (17)
C7—C2—C3—C4	−179.98 (14)	C1—C2—C7—I1	179.37 (9)
C2—C3—C4—C5	−0.6 (3)	C6—C1—C8—C1^{i}	−124.7 (2)
C3—C4—C5—C6	0.9 (3)	C2—C1—C8—C1^{i}	55.02 (16)
C2—C1—C6—C5	−0.9 (2)	C6—C1—C8—C1^{ii}	124.0 (2)
C8—C1—C6—C5	178.79 (14)	C2—C1—C8—C1^{ii}	−56.30 (16)
C4—C5—C6—C1	−0.1 (3)		

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

Fig. 1

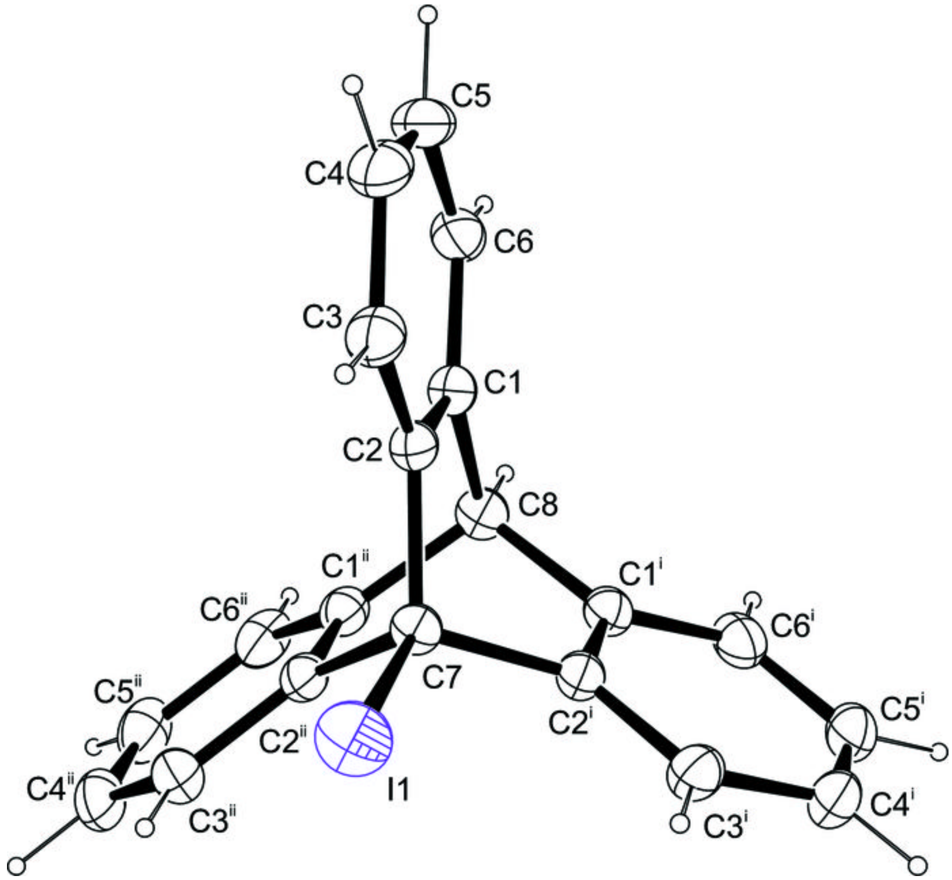
C5
C4
C6
C3
C1
C2
C8
C1ii
C1i
C6ii
C6i
C7
C5ii
C5i
C2ii
C2i
I1
C4ii
C3ii
C3i
C4i

Fig. 2