9,10-Diiodophenanthrene

Ruri Yokota, Chitoshi Kitamura* and Takeshi Kawase

Department of Materials Science and Chemistry, Graduate School of Engineering, University of Hyogo, 2167 Shosha, Himeji, Hyogo 671-2280, Japan
Correspondence e-mail: kitamura@eng.u-hyogo.ac.jp

The whole molecule of the title compound, $C_{14}H_8I_2$, is generated by crystallographic twofold symmetry. The molecule is planar [maximum deviation = 0.0323 (6) Å] with the I atoms displaced from the mean plane of the phenanthrene ring system by only 0.0254 (5) Å. In the crystal, molecules form face-to-face slipped antiparallel π–π stacking interactions along the c axis with an interplanar distance of 3.499 (7) Å.

Related literature

For the synthesis of the title compound, see: Rodrígeuz-Lojo *et al.* (2012). For a related structure, see: Yokota *et al.* (2012).
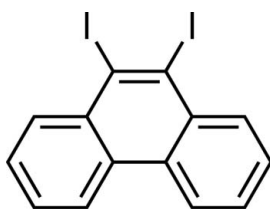
Experimental

Crystal data

$C_{14}H_8I_2$ M_r = 430

Monoclinic, $C2/c$
a = 18.094 (2) Å
b = 9.4557 (14) Å
c = 7.4187 (10) Å
β = 111.953 (3)°
V = 1177.2 (3) Å^3

Z = 4
Mo $K\alpha$ radiation
μ = 5.31 mm^{-1}
T = 223 K
0.52 × 0.08 × 0.05 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: numerical (*NUMABS*; Higashi, 1999)
T_{min} = 0.388, T_{max} = 0.869

5595 measured reflections
1345 independent reflections
1077 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.083
S = 1.12
1345 reflections

73 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.92 e Å^{-3}
$\Delta\rho_{min}$ = −0.92 e Å^{-3}

Data collection: *RAPID-AUTO* (Rigaku, 1999); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1999). *NUMABS*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1999). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rodrígeuz-Lojo, D., Cobas, A., Peña, D., Pérez, D. & Guitián, E. (2012). *Org. Lett.* **14**, 1363–1365.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yokota, R., Kitamura, C. & Kawase, T. (2012). *Acta Cryst.* E**68**, o3174.

9,10-Diiodophenanthrene

Ruri Yokota, Chitoshi Kitamura and Takeshi Kawase

Comment

o-Diiodoarenes are valuable synthetic intermediates. We were able to obtain suitable single crystals of 9,10-diiodo-ophenanthrene, the title compound, by the recently published synthetic method of Rodrígeuz-Lojo *et al.* (2012). We report herein the crystal structure of $C_{14}H_8I_2$, the title compound.

In the molecular structure of the title compound, (I), the mean plane of the arene ring displays a maximum deviation of 0.0323 (6) Å for I1 (Fig. 1). The molecule possesses C_2 symmetry, and half of the formula unit is crystallographically independent. Bonds lengths and angles are in good agreement with the standard values. Crystal packing is stabilized by face-to-face, slipped, antiparrallel, π-π stacking along the direction of the *c* axis with an interplanar distance of 3.499 (7) Å (Fig. 2). Very recently, we have reported the crystal structure of 9,10-dibromophenanthrene (Yokota *et al.*, 2012), the bromine analog of (I), which displays a similar packing arrangement.

Experimental

The title compound was prepared according to the literature method (Rodrígeuz-Lojo *et al.*, 2012) Single crystals suitable for X-ray analysis were obtained from a toluene-hexane solution.

Refinement

All the aromatic H atoms were positioned geometrically and refined using a riding model with C—H = 0.94 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Computing details

Data collection: *RAPID-AUTO* (Rigaku, 1999); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *PROCESS-AUTO* (Rigaku, 1998); program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).
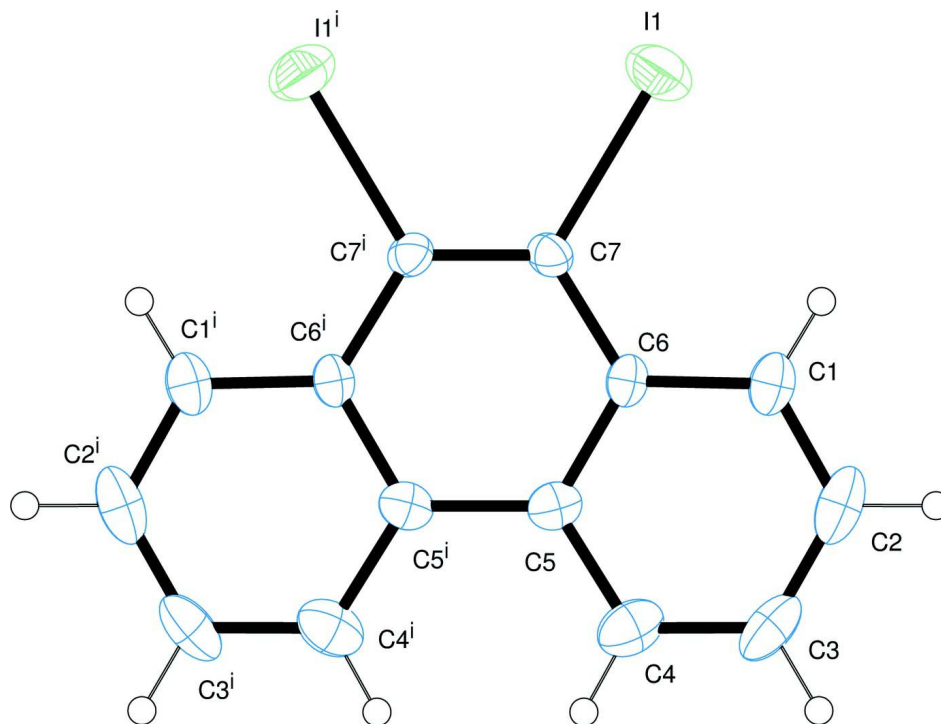
Figure 1

The molecular structure of the title compound, showing the atomic numbering and 40% probability displacement ellipsoids. Symmetry code: (i) $-x + 1$, y, $-z + 1.5$.
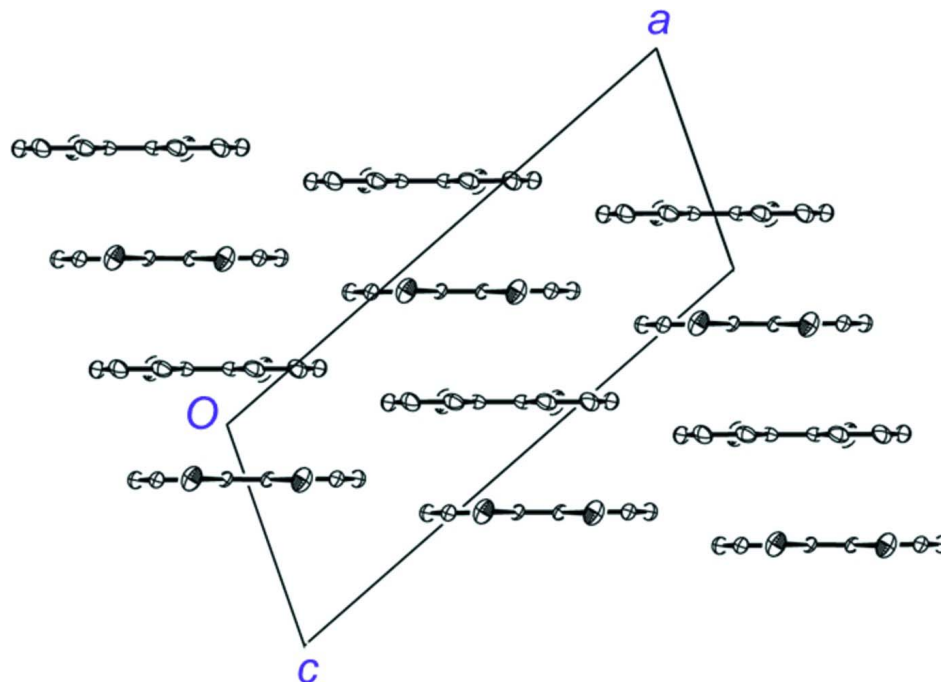
Figure 2

The packing diagram of the title compound viewed along the b axis. Hydrogen atoms have been omitted for clarity.

9,10-diiodophenanthrene

Crystal data

$C_{14}H_8I_2$	$F(000) = 792$
$M_r = 430$	$D_x = 2.426$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 3465 reflections
$a = 18.094$ (2) Å	$\theta = 3.1–27.5°$
$b = 9.4557$ (14) Å	$\mu = 5.31$ mm^{-1}
$c = 7.4187$ (10) Å	$T = 223$ K
$\beta = 111.953$ (3)°	Needle, colorless
$V = 1177.2$ (3) Å^3	$0.52 \times 0.08 \times 0.05$ mm
$Z = 4$	

Data collection

Rigaku R-AXIS RAPID diffractometer	5595 measured reflections
Radiation source: fine-focus sealed x-ray tube	1345 independent reflections
Graphite monochromator	1077 reflections with $I > 2\sigma(I)$
Detector resolution: 10 pixels mm^{-1}	$R_{int} = 0.030$
ω scans	$\theta_{max} = 27.5°$, $\theta_{min} = 3.5°$
Absorption correction: numerical (*NUMABS*; Higashi, 1999)	$h = -23\rightarrow22$
	$k = -12\rightarrow12$
$T_{min} = 0.388$, $T_{max} = 0.869$	$l = -8\rightarrow9$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.033$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.083$	
$S = 1.12$	H-atom parameters constrained
1345 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0077P)^2 + 13.1956P]$
73 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
	$\Delta\rho_{max} = 0.92$ e Å^{-3}
	$\Delta\rho_{min} = -0.92$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
C1	0.3399 (3)	0.4417 (6)	0.4793 (7)	0.0368 (12)
H1	0.3127	0.3564	0.4332	0.044*
C2	0.3020 (3)	0.5667 (8)	0.4155 (8)	0.0470 (15)
H2	0.2488	0.5669	0.3275	0.056*

C3	0.3408 (3)	0.6927 (7)	0.4781 (9)	0.0467 (15)
H3	0.3146	0.7786	0.4306	0.056*
C4	0.4171 (4)	0.6931 (6)	0.6089 (8)	0.0424 (13)
H4	0.4426	0.78	0.6529	0.051*
C5	0.4587 (3)	0.5675 (5)	0.6798 (7)	0.0289 (10)
C6	0.4192 (3)	0.4383 (5)	0.6137 (7)	0.0261 (9)
C7	0.4618 (3)	0.3079 (5)	0.6846 (7)	0.0281 (10)
I1	0.40244 (3)	0.11933 (4)	0.58098 (7)	0.05783 (18)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.029 (2)	0.051 (3)	0.029 (3)	0.003 (2)	0.008 (2)	0.001 (2)
C2	0.026 (3)	0.078 (4)	0.034 (3)	0.012 (3)	0.007 (2)	0.010 (3)
C3	0.038 (3)	0.057 (4)	0.048 (3)	0.024 (3)	0.019 (3)	0.020 (3)
C4	0.048 (3)	0.040 (3)	0.048 (3)	0.012 (2)	0.028 (3)	0.008 (3)
C5	0.030 (2)	0.030 (2)	0.031 (3)	0.0021 (19)	0.017 (2)	0.0012 (19)
C6	0.023 (2)	0.035 (2)	0.024 (2)	0.0010 (18)	0.0126 (18)	0.0001 (18)
C7	0.033 (2)	0.026 (2)	0.025 (2)	−0.0029 (18)	0.010 (2)	−0.0020 (18)
I1	0.0670 (3)	0.0402 (2)	0.0526 (3)	−0.01527 (19)	0.0067 (2)	−0.00726 (18)

Geometric parameters (Å, º)

C1—C2	1.359 (8)	C4—C5	1.399 (7)
C1—C6	1.409 (7)	C4—H4	0.94
C1—H1	0.94	C5—C6	1.408 (7)
C2—C3	1.372 (9)	C5—C5ⁱ	1.467 (10)
C2—H2	0.94	C6—C7	1.445 (6)
C3—C4	1.359 (8)	C7—C7ⁱ	1.360 (9)
C3—H3	0.94	C7—I1	2.075 (5)
C2—C1—C6	120.9 (5)	C5—C4—H4	119.1
C2—C1—H1	119.6	C4—C5—C6	118.2 (5)
C6—C1—H1	119.6	C4—C5—C5ⁱ	121.9 (3)
C1—C2—C3	120.7 (5)	C6—C5—C5ⁱ	119.8 (3)
C1—C2—H2	119.7	C5—C6—C1	118.5 (5)
C3—C2—H2	119.7	C5—C6—C7	118.7 (4)
C4—C3—C2	119.9 (5)	C1—C6—C7	122.8 (5)
C4—C3—H3	120.1	C7ⁱ—C7—C6	121.5 (3)
C2—C3—H3	120.1	C7ⁱ—C7—I1	120.77 (13)
C3—C4—C5	121.8 (6)	C6—C7—I1	117.8 (3)
C3—C4—H4	119.1		
C6—C1—C2—C3	1.1 (8)	C5ⁱ—C5—C6—C7	−0.9 (8)
C1—C2—C3—C4	−1.6 (9)	C2—C1—C6—C5	−0.4 (7)
C2—C3—C4—C5	1.4 (9)	C2—C1—C6—C7	−179.6 (5)
C3—C4—C5—C6	−0.7 (8)	C5—C6—C7—C7ⁱ	1.2 (8)
C3—C4—C5—C5ⁱ	179.7 (6)	C1—C6—C7—C7ⁱ	−179.6 (6)
C4—C5—C6—C1	0.2 (7)	C5—C6—C7—I1	−179.0 (3)

| C5i—C5—C6—C1 | 179.8 (5) | C1—C6—C7—I1 | 0.2 (6) |
| C4—C5—C6—C7 | 179.5 (5) | | |

Symmetry code: (i) −*x*+1, *y*, −*z*+3/2.