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(*E*,*E*)-1,4-Bis(2-iodophenyl)-2,3-diaza-1,3-butadiene

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The molecules of the title compound $C_{14}H_{10}I_2N_2$, which are almost planar, lie across centres of inversion. The structure contains no direction-specific intermolecular interactions of any kind.

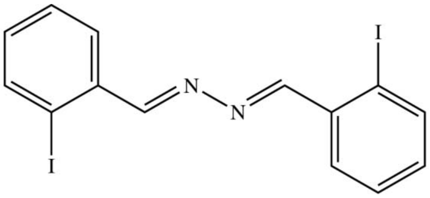
Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.005 Å
R factor = 0.022
wR factor = 0.075
Data-to-parameter ratio = 19.7

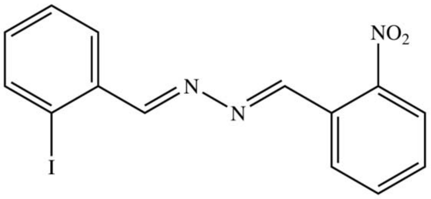
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

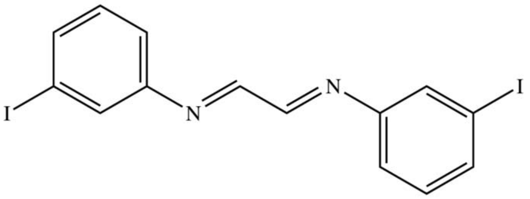
We recently reported that small quantities (*ca* 10%) of the title compound (I) (Fig.1) readily co-crystallized with (*E*,*E*)-1-(2-iodophenyl)-4-(2-nitrophenyl)-2,3-diaza-1,3-butadiene (II), and that the presence of (I) probably arose from some minor reorganization of the substituted aryl groups during either the synthesis or the recrystallization of (II) (Glidewell *et al.*, 2005). In order to assess the relationship between the structures of (I) and (II), we now report the structure of (I).



(I)



(II)



(III)

The molecules of (I) lie across inversion centres in space group $P2_1/n$, and they are almost planar, as shown by the key torsion angles (Table 1). The bond lengths and angles present no unusual features, apart from the exocyclic bond angles at C2, most plausibly associated with the repulsive intramolecular contact between atoms I2 and the H atom bonded to C7. There are no direction-specific interactions of any kind in the crystal structure of (I): C—H$\cdots$N and C—H$\cdots\pi$(arene) hydrogen bonds, aromatic π–π stacking interactions, and short I···I interactions are all absent.

The unit-cell dimensions for compounds (I) and (II) are very different, and these compounds also crystallize in different space groups, with molar volumes which differ by some 5%. It is therefore not clear why compound (I) so readily co-crystallizes with compound (II).

Isomeric with (I) is compound (III) where, again, nearly planar molecules lie across centres of inversion (Cho *et al.*, 2005).

Experimental

A mixture of 2-iodobenzaldehyde (1 mmol) and hydrazine hydrate (0.5 mmol) in methanol (20 cm^3) was heated under reflux for 30 min. The mixture was cooled and the solvent was removed under reduced pressure. The product, (I), was purified by chromatography on alumina, using $CHCl_3$ as eluent: crystals suitable for single-crystal X-ray diffraction were grown by slow evaporation of a solution in ethanol.

Crystal data

$C_{14}H_{10}I_2N_2$
$M_r = 460.04$
Monoclinic, $P2_1/n$
$a = 13.2667\ (4)$ Å
$b = 4.1070\ (2)$ Å
$c = 13.8207\ (4)$ Å
$\beta = 110.3140\ (15)^\circ$
$V = 706.20\ (5)$ Å^3
$Z = 2$

$D_x = 2.163$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1818 reflections
$\theta = 1.0$–27.4°
$\mu = 4.44$ mm^{-1}
$T = 120\ (2)$ K
Lath, yellow
$0.64 \times 0.12 \times 0.08$ mm

Data collection

Bruker–Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.160$, $T_{max} = 0.703$
9545 measured reflections

1614 independent reflections
1455 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5^\circ$
$h = -17 \rightarrow 15$
$k = -5 \rightarrow 5$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.075$
$S = 1.30$
1614 reflections
82 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -1.16$ e Å^{-3}

Table 1
Selected bond and torsion angles (°).

C3—C2—C1	121.6 (3)	C1—C2—I2	122.2 (2)
C3—C2—I2	116.2 (2)		
C6—C1—C7—N1	−5.2 (5)	C1—C7—N1—N1^i	−179.9 (3)

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

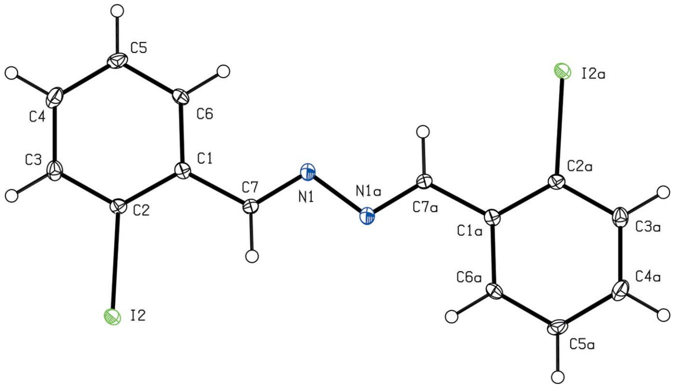


Figure 1
The molecular structure of compound (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level, and atoms labelled with suffix ‘a’ are related by the symmetry operator $(1 - x, 1 - y, 1 - z)$.

All H atoms were located in difference maps and they were then treated as riding atoms with C—H distances 0.95 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The deepest hole is located 0.72 Åfrom atom I2.

Data collection: *COLLECT* (Hooft, 1999); cell refinement: *DENZO* (Otwinowski & Minor, 1997) & *COLLECT*); data reduction: *DENZO* & *COLLECT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PRPKAPPA* (Ferguson, 1999).

X-Ray data were collected at the EPSRC X-Ray Crystallographic Service, University of Southampton, UK; the authors thank the staff of the Service for all their help and advice. JLW thanks CNPq and FAPERJ for financial support.

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$D_x = 2.163$ Mg m^{-3}
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Cell parameters from 1818 reflections
$\theta = 1.0$–27.4°
$\mu = 4.44$ mm^{-1}
$T = 120$ K
Lath, yellow
0.64 × 0.12 × 0.08 mm

Data collection

Bruker-Nonius KappaCCD diffractometer
Radiation source: Bruker-Nonius FR591 rotating anode
Graphite monochromator
Detector resolution: 9.091 pixels mm^{-1}
φ & ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
$T_{min} = 0.160$, $T_{max} = 0.703$
9545 measured reflections
1614 independent reflections
1455 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 27.5°$, $\theta_{min} = 1.8°$
$h = -17 \rightarrow 15$
$k = -5 \rightarrow 5$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.075$
$S = 1.30$
1614 reflections
82 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -1.16$ e Å^{-3}

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.7374 (3)	0.4440 (7)	0.5557 (2)	0.0150 (6)
C2	0.8164 (2)	0.3184 (8)	0.5201 (2)	0.0149 (6)
I2	0.777975 (15)	0.09737 (5)	0.373819 (14)	0.01682 (12)

C3	0.9252 (3)	0.3381 (8)	0.5790 (3)	0.0204 (7)
C4	0.9564 (3)	0.4865 (10)	0.6753 (3)	0.0243 (7)
C5	0.8802 (3)	0.6146 (7)	0.7125 (3)	0.0217 (8)
C6	0.7720 (3)	0.5935 (7)	0.6531 (3)	0.0183 (7)
C7	0.6223 (3)	0.4242 (7)	0.4961 (3)	0.0173 (7)
N1	0.5529 (2)	0.5194 (8)	0.5345 (2)	0.0220 (6)
H3	0.9776	0.2510	0.5536	0.025*
H4	1.0305	0.5003	0.7160	0.029*
H5	0.9019	0.7163	0.7783	0.026*
H6	0.7202	0.6823	0.6788	0.022*
H7	0.5991	0.3393	0.4279	0.021*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0170 (16)	0.0159 (15)	0.0130 (15)	0.0026 (11)	0.0064 (13)	0.0050 (11)
C2	0.0177 (15)	0.0163 (13)	0.0112 (14)	−0.0006 (12)	0.0056 (12)	0.0030 (12)
I2	0.01818 (16)	0.01792 (16)	0.01565 (16)	0.00262 (6)	0.00751 (11)	−0.00004 (6)
C3	0.0167 (16)	0.0255 (16)	0.0198 (17)	0.0041 (13)	0.0072 (13)	0.0064 (13)
C4	0.0149 (16)	0.0342 (17)	0.0203 (17)	−0.0014 (15)	0.0017 (13)	0.0048 (15)
C5	0.0231 (19)	0.030 (2)	0.0104 (17)	−0.0047 (12)	0.0035 (14)	0.0006 (11)
C6	0.0194 (18)	0.0246 (17)	0.0147 (16)	0.0001 (11)	0.0106 (14)	−0.0001 (11)
C7	0.0148 (16)	0.0243 (17)	0.0116 (16)	0.0018 (11)	0.0033 (13)	−0.0003 (11)
N1	0.0140 (14)	0.0348 (15)	0.0167 (14)	0.0035 (13)	0.0047 (11)	−0.0022 (13)

Geometric parameters (Å, º)

C1—C2	1.402 (4)	C4—H4	0.95
C1—C6	1.403 (5)	C5—C6	1.387 (5)
C1—C7	1.464 (5)	C5—H5	0.95
C2—C3	1.391 (4)	C6—H6	0.95
C2—I2	2.111 (3)	C7—N1	1.273 (5)
C3—C4	1.391 (5)	C7—H7	0.95
C3—H3	0.95	N1—N1^{i}	1.407 (6)
C4—C5	1.387 (6)		

C2—C1—C6	117.5 (3)	C3—C4—H4	119.7
C2—C1—C7	122.9 (3)	C6—C5—C4	119.6 (3)
C6—C1—C7	119.6 (3)	C6—C5—H5	120.2
C3—C2—C1	121.6 (3)	C4—C5—H5	120.2
C3—C2—I2	116.2 (2)	C5—C6—C1	121.5 (3)
C1—C2—I2	122.2 (2)	C5—C6—H6	119.2
C4—C3—C2	119.3 (3)	C1—C6—H6	119.2
C4—C3—H3	120.4	N1—C7—C1	120.8 (3)
C2—C3—H3	120.4	N1—C7—H7	119.6
C5—C4—C3	120.5 (3)	C1—C7—H7	119.6
C5—C4—H4	119.7	C7—N1—N1^{i}	112.3 (4)

C6—C1—C2—C3	0.5 (5)	C3—C4—C5—C6	0.2 (5)
C7—C1—C2—C3	−179.4 (3)	C4—C5—C6—C1	0.2 (5)
C6—C1—C2—I2	−178.3 (2)	C2—C1—C6—C5	−0.5 (5)
C7—C1—C2—I2	1.8 (4)	C7—C1—C6—C5	179.4 (3)
C1—C2—C3—C4	−0.2 (5)	C2—C1—C7—N1	174.7 (3)
I2—C2—C3—C4	178.7 (3)	C6—C1—C7—N1	−5.2 (5)
C2—C3—C4—C5	−0.2 (5)	C1—C7—N1—N1^{i}	−179.9 (3)

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