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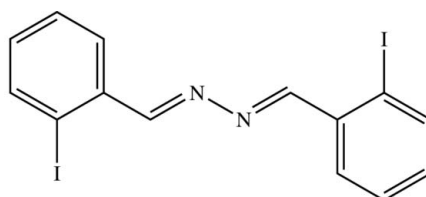
Key indicators

Single-crystal X-ray study
 $T = 120$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
 R factor = 0.022
 wR factor = 0.075
Data-to-parameter ratio = 19.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(*E,E*)-1,4-Bis(2-iodophenyl)-2,3-diaza-1,3-butadiene**The molecules of the title compound $\text{C}_{14}\text{H}_{10}\text{I}_2\text{N}_2$, which are almost planar, lie across centres of inversion. The structure contains no direction-specific intermolecular interactions of any kind.

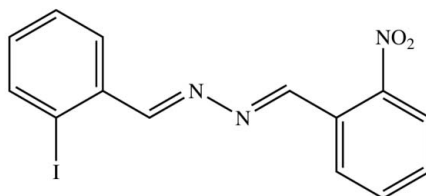
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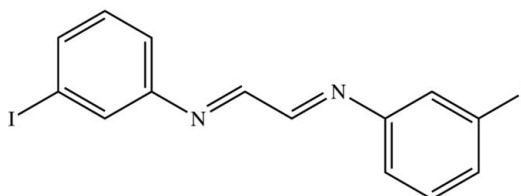
Comment

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): $\text{C}-\text{H}\cdots\text{N}$ and $\text{C}-\text{H}\cdots\pi(\text{arene})$

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 cm³) 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 CHCl₃ 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 ₂ N ₂	$D_x = 2.163 \text{ Mg m}^{-3}$
$M_r = 460.04$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 1818 reflections
$a = 13.2667(4) \text{ \AA}$	$\theta = 1.0\text{--}27.4^\circ$
$b = 4.1070(2) \text{ \AA}$	$\mu = 4.44 \text{ mm}^{-1}$
$c = 13.8207(4) \text{ \AA}$	$T = 120(2) \text{ K}$
$\beta = 110.3140(15)^\circ$	Lath, yellow
$V = 706.20(5) \text{ \AA}^3$	$0.64 \times 0.12 \times 0.08 \text{ mm}$
$Z = 2$	

Data collection

Bruker-Nonius KappaCCD diffractometer	1614 independent reflections
φ and ω scans	1455 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	$R_{\text{int}} = 0.027$
$T_{\text{min}} = 0.160$, $T_{\text{max}} = 0.703$	$\theta_{\text{max}} = 27.5^\circ$
9545 measured reflections	$h = -17 \rightarrow 15$
	$k = -5 \rightarrow 5$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.022$	$w = 1/[\sigma^2(F_o^2) + (0.0439P)^2]$
$wR(F^2) = 0.075$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.30$	$(\Delta/\sigma)_{\text{max}} = 0.001$
1614 reflections	$\Delta\rho_{\text{max}} = 0.64 \text{ e \AA}^{-3}$
82 parameters	$\Delta\rho_{\text{min}} = -1.16 \text{ e \AA}^{-3}$

Table 1

Selected bond and torsion angles ($^\circ$).

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) $-x + 1, -y + 1, -z + 1$.

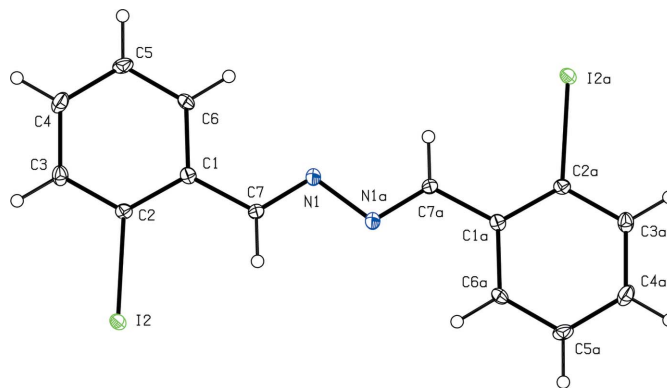


Figure 1

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 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The deepest hole is located 0.72 \AA from 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.

References

- Cho, H. M., Moore, J. S. & Wilson, S. R. (2005). *Acta Cryst.* E61, o3773–o3774.
- Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
- Glidewell, C., Low, J. N., Skakle, J. M. S. & Wardell, J. L. (2005). *Acta Cryst.* C61, o312–o316.
- Hooft, R. W. W. (1999). *COLLECT*. Nonius B. V., Delft, The Netherlands.
- McArdle, P. (2003). *OSCAIL for Windows*. Version 10, Crystallography Centre, Chemistry Department, NUI Galway, Ireland.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.
- Spek, A. L. (2003). *J. Appl. Cryst.* 36, 7–13.