

## 4,5-Diiodo-2-phenyl-1H-imidazole

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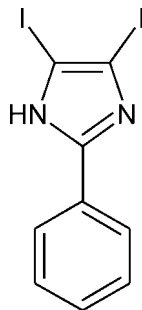
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Key indicators: single-crystal X-ray study;  $T = 150$  K; mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å;  $R$  factor = 0.029;  $wR$  factor = 0.051; data-to-parameter ratio = 20.9.

The structure of the title compound,  $\text{C}_9\text{H}_6\text{I}_2\text{N}_2$ , contains two symmetry-independent molecules. The interplanar angles between the imidazole and phenyl ring planes are 16.35 (3) and 17.48 (6)°. Molecules are connected *via*  $\text{N}-\text{H}\cdots\text{N}$  hydrogen bonds to form zigzag chains along the  $b$  axis. The title compound is the first example of a structurally characterized 4,5-diiodoimidazole with an organic substituent in the 2-position and without protection on the  $\text{N}-\text{H}$  group of imidazole.

## Related literature

For the structures of various related compounds, see: Delest *et al.* (2008); Poverlein *et al.* (2007); Panday *et al.* (2000); Phillips *et al.* (1997); Terinek & Vasella (2003); Mukai & Nishikawa (2010*a,b*); Noland *et al.* (2003); Dou & Weiss (1992); Nagatomo *et al.* (1995). For the use of diiodoimidazoles as starting compounds for ligand synthesis, see: Haruki *et al.* (1965); Ito & Uedaira (2004); Kim *et al.* (1999); Zhang *et al.* (2006). For the synthetic procedure, see: Garden *et al.* (2001); Ishihara & Togo (2006). For typical bond lengths, see: Allen *et al.* (1987).



## Experimental

## Crystal data

$\text{C}_9\text{H}_6\text{I}_2\text{N}_2$	$V = 4367.4$ (5) Å <sup>3</sup>
$M_r = 395.96$	$Z = 16$
Orthorhombic, <i>Abc2</i>	Mo $K\alpha$ radiation
$a = 31.0150$ (6) Å	$\mu = 5.72$ mm <sup>-1</sup>
$b = 17.5010$ (5) Å	$T = 150$ K
$c = 8.0461$ (9) Å	$0.45 \times 0.16 \times 0.07$ mm

## Data collection

Bruker-Nonius KappaCCD area-detector diffractometer	20468 measured reflections
Absorption correction: gaussian (Coppens, 1970)	4914 independent reflections
$T_{\min} = 0.256$ , $T_{\max} = 0.675$	4499 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.058$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$	H-atom parameters constrained
$wR(F^2) = 0.051$	$\Delta\rho_{\text{max}} = 0.63$ e Å <sup>-3</sup>
$S = 1.04$	$\Delta\rho_{\text{min}} = -0.63$ e Å <sup>-3</sup>
4914 reflections	Absolute structure: Flack (1983),
235 parameters	2233 Friedel pairs
1 restraint	Flack parameter: 0.01 (3)

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N}2-\text{H}2\cdots\text{N}3^i$	0.86	2.01	2.845 (5)	165
$\text{N}4-\text{H}4\cdots\text{N}1$	0.86	2.02	2.848 (6)	162

Symmetry code: (i)  $x, y + \frac{1}{2}, z + \frac{1}{2}$ .

Data collection: *COLLECT* (Hooft, 1998) and *DENZO* (Otwinowski & Minor, 1997); cell refinement: *COLLECT* and *DENZO*; data reduction: *COLLECT* and *DENZO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2350).

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans 2*, pp. S1–19.
- Altomare, A., Casciarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Coppens, P. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 255–270. Copenhagen: Munksgaard.
- Delest, B., Nshimyumukiza, P., Fasbender, O., Tinant, B., Marchand-Brynaert, J., Darro, F. & Robiette, R. (2008). *J. Org. Chem.* **73**, 6816–6823.
- Dou, S. & Weiss, A. (1992). *Z. Naturforsch. Teil A*, **47**, 177–181.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Garden, S. J., Torres, J. C., de Souza Melo, S. C., Lima, A. S., Pinto, A. C. & Lima, E. L. S. (2001). *Tetrahedron Lett.* **42**, 2089–2092.
- Haruki, E., Izumita, S. & Imoto, E. (1965). *Nippon Kagaku Zasshi*, **86**, 942–946.
- Hooft, R. W. (1998). *COLLECT*. Enraf-Nonius, Delft, The Netherlands.
- Ishihara, M. & Togo, H. (2006). *Synlett*, pp. 227–230.
- Ito, T. & Uedaira, S. (2004). Jpn. Kokai Tokkyo Koho, JP 2004123550.

- Kim, G., Kang, S., Ryu, Y., Keum, G. & Seo, M. J. (1999). *Synth. Commun.* **29**, 507–512.
- Mukai, T. & Nishikawa, K. (2010a). *Anal. Sci. X-ray Struct. Anal. Online*, **26**, 31–32.
- Mukai, T. & Nishikawa, K. (2010b). *Solid State Sci.* **12**, 783–788.
- Nagatomo, S., Takeda, S., Tamura, H. & Nakamura, N. (1995). *Bull. Chem. Soc. Jpn.* **68**, 2783–2789.
- Noland, W. E., Cole, K. P. & Britton, D. (2003). *Acta Cryst.* **E59**, o458–o460.
- 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.
- Panday, N., Canac, Y. & Vasella, A. (2000). *Helv. Chim. Acta*, **83**, 58–79.
- Phillips, J. G., Fadnis, L. & Williams, D. R. (1997). *Tetrahedron Lett.* **38**, 7835–7838.
- Poverlein, C., Jacobi, N., Mayer, P. & Lindel, T. (2007). *Synthesis*, pp. 3620–3626.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.
- Terinek, M. & Vasella, A. (2003). *Helv. Chim. Acta*, **86**, 3482–3509.
- Zhang, J., Zhang, Y., Schnatter, W. F. K. & Herndon, J. W. (2006). *Organometallics*, **25**, 1279–1284.

**supplementary materials**

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## 4,5-Diiodo-2-phenyl-1*H*-imidazole

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### Comment

Halogenated derivatives of heterocycles are important starting compounds for building new heterocyclic chiral ligands via cross-coupling reactions. In recent times 4,5-diiodimidazole and its derivatives were used for the preparation of various disubstituted 4,5-dicarbaimidazoles (Zhang *et al.*, 2006; Ito & Uedaira, 2004; Kim *et al.*, 1999; Haruki *et al.*, 1965). To the best of our knowledge, there are a couple of structures of dihalogenated imidazoles determined by X-ray diffraction methods, but the 4,5-diido-1*H*-imidazole structure as the most versatile and simplest member of the series is missing. Compound **I** (Scheme 1) was separated as a byproduct from a reaction described by Ishihara (Ishihara & Togo, 2006) but is usually prepared by the method of Garden (Garden *et al.*, 2001). The title compound crystallizes (Fig. 1) in an orthorhombic space group with two independent molecules in the unit cell (Fig. 2). Both molecules reveal similar structural behavior showing two planar conjugated rings – phenyl and imidazolyl. The interplanar angles between imidazolyl and phenyl planes are 16.35 (3)° and 17.48 (6)°, respectively. The formation of infinite chains is provided by the connection of both types of molecules via N–H⋯N hydrogen bridges. The linear chains interact via the iodine atoms at neighbouring imidazole moieties. In contrast to typical C–I bond lengths of ca. 2.095 Å (Allen *et al.*, 1987) which is the same for monoiodoimidazoles, the C–I separations in diiodoimidazoles significantly differ between ca. 2.05 and 2.09 Å (Panday *et al.*, 2000; Terinek & Vasella, 2003). This phenomenon is also seen in one of the molecules of **I**, where the C11–I3 bond length is 2.075 (5) Å and the C12–I4 bond located next to the N–H function is shortened to 2.058 (5) Å. On the other hand, in the first molecule both C–I bond lengths (2.051 (5) and 2.058 (5) Å) are almost identical and thus comparable to the same parameters found in compounds containing the 4,5-diidoimidazolium ion (Mukai & Nishikawa, 2010*a,b*). Only one iodine atom I1 is located in the same plane defined by the imidazole ring, while the others show deviations of 0.07–0.148 Å. Bond lengths between the atoms forming the imidazole rings are comparable to literature values (Allen *et al.*, 1987) except of C1–N2 which is elongated by 0.02 Å and C10–N3 which is slightly shortened.

### Experimental

The title compound **I** was isolated in 30% yield as a byproduct from the reaction mixture of 2-phenyl-1*H*-imidazole-4-carbaldehyde with ethan-1,2-diamine, iodine and potassium carbonate in tert-butylalcohol, according to Ishihara (Ishihara & Togo, 2006) in order to prepare 2'-phenyl-4,5-dihydro-1*H*,1'*H*-2,4'-biimidazole. The reaction mixture was separated by the help of column chromatography on silicagel (ethyl acetate, hexane and dichloromethane – 1:3:10). Further crystallization from dichloromethane gave pure **I**. The identity and purity of **I** was confirmed by the same melting point, <sup>1</sup>H NMR and mass spectra patterns as published elsewhere (Garden *et al.*, 2001). Single crystals of **I** were obtained by slow vapour diffusion of hexane into a solution of **I** in dichloromethane.

## Refinement

All hydrogen atoms were discernible in the difference electron density map. However, all hydrogen atoms were placed into idealized positions and refined riding on their parent C or N atoms, with N–H = 0.86 Å, C–H = 0.93 Å for aromatic H atoms, with  $U(H) = 1.2U_{eq}(C/N)$  for the NH group and  $U(H) = 1.5U_{eq}(C/N)$  for other H atoms, respectively.

## Figures

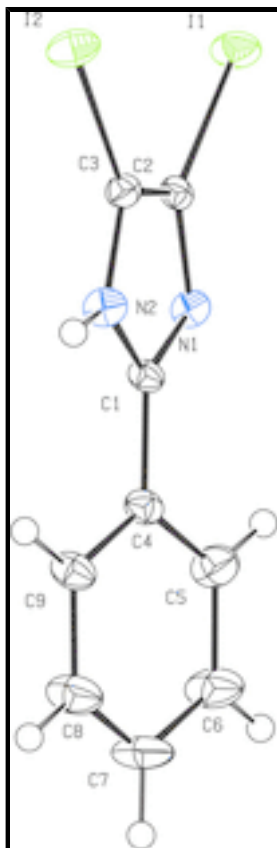


Fig. 1. View of one of the independent molecules of the title compound with the displacement ellipsoids shown at the 50% probability level. H atoms are shown with arbitrary radii.

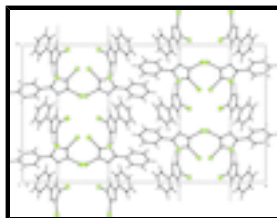


Fig. 2. View of the crystal structure down the *c* axis showing the hydrogen bond interactions.

## 4,5-Diiodo-2-phenyl-1*H*-imidazole

### Crystal data

$C_9H_6I_2N_2$   
 $M_r = 395.96$

$D_x = 2.409 \text{ Mg m}^{-3}$   
Melting point: 472 K

Orthorhombic, *Aba2*  
Hall symbol: A 2 -2ac  
 $a = 31.0150$  (6) Å  
 $b = 17.5010$  (5) Å  
 $c = 8.0461$  (9) Å  
 $V = 4367.4$  (5) Å<sup>3</sup>  
 $Z = 16$   
 $F(000) = 2880$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å  
Cell parameters from 20579 reflections  
 $\theta = 1-27.5^\circ$   
 $\mu = 5.72$  mm<sup>-1</sup>  
 $T = 150$  K  
Needle, colourless  
 $0.45 \times 0.16 \times 0.07$  mm

### Data collection

Bruker–Nonius KappaCCD area-detector diffractometer  
Radiation source: fine-focus sealed tube graphite  
Detector resolution: 9.091 pixels mm<sup>-1</sup>  
 $\phi$  and  $\omega$  scans to fill the Ewald sphere  
Absorption correction: gaussian (Coppens, 1970)  
 $T_{\min} = 0.256$ ,  $T_{\max} = 0.675$   
20468 measured reflections

4914 independent reflections  
4499 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.058$   
 $\theta_{\max} = 27.5^\circ$ ,  $\theta_{\min} = 2.3^\circ$   
 $h = -39 \rightarrow 40$   
 $k = -22 \rightarrow 19$   
 $l = -9 \rightarrow 10$

### Refinement

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.029$   
 $wR(F^2) = 0.051$   
 $S = 1.04$   
4914 reflections  
235 parameters  
1 restraint  
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  
 $w = 1/[\sigma^2(F_o^2) + (0.0196P)^2 + 8.5806P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.63$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.63$  e Å<sup>-3</sup>  
Absolute structure: Flack (1983), 2233 Friedel pairs  
Flack parameter: 0.01 (3)

### Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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.

## supplementary materials

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### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I3	0.239603 (10)	0.127609 (18)	0.00147 (5)	0.02406 (8)
I2	0.185474 (11)	0.465127 (18)	0.86344 (5)	0.02557 (9)
I1	0.142369 (12)	0.262855 (18)	0.73764 (5)	0.02639 (9)
I4	0.225974 (10)	0.324363 (17)	0.26443 (4)	0.02035 (8)
N1	0.11736 (13)	0.3687 (2)	0.4558 (5)	0.0172 (9)
N3	0.14573 (13)	0.1439 (2)	0.0814 (5)	0.0160 (8)
C14	0.05141 (17)	0.1305 (3)	0.0519 (7)	0.0210 (12)
H14	0.0683	0.1040	-0.0242	0.025*
C9	0.11213 (17)	0.5450 (3)	0.2022 (6)	0.0217 (12)
H9	0.1321	0.5741	0.2610	0.026*
C3	0.15163 (16)	0.4339 (3)	0.6553 (6)	0.0166 (10)
C1	0.11925 (16)	0.4415 (3)	0.4126 (6)	0.0153 (10)
C4	0.10087 (15)	0.4731 (2)	0.2585 (7)	0.0178 (10)
C13	0.07108 (16)	0.1796 (3)	0.1643 (6)	0.0151 (10)
C11	0.18543 (15)	0.1775 (3)	0.1070 (6)	0.0163 (10)
C2	0.13758 (15)	0.3637 (3)	0.6074 (6)	0.0146 (10)
C12	0.18168 (16)	0.2429 (3)	0.1949 (6)	0.0170 (10)
C10	0.11796 (16)	0.1907 (3)	0.1591 (6)	0.0150 (10)
N2	0.13961 (13)	0.4833 (2)	0.5321 (5)	0.0184 (9)
H2	0.1441	0.5318	0.5297	0.022*
C18	0.04545 (16)	0.2180 (3)	0.2794 (7)	0.0219 (11)
H18	0.0582	0.2511	0.3556	0.026*
C5	0.07161 (18)	0.4297 (3)	0.1697 (7)	0.0269 (13)
H5	0.0637	0.3815	0.2079	0.032*
C16	-0.01789 (19)	0.1579 (3)	0.1692 (8)	0.0301 (14)
H16	-0.0476	0.1507	0.1714	0.036*
N4	0.13889 (12)	0.2501 (2)	0.2280 (6)	0.0147 (8)
H4	0.1273	0.2867	0.2834	0.018*
C17	0.00127 (17)	0.2077 (3)	0.2786 (8)	0.0296 (13)
H17	-0.0156	0.2343	0.3548	0.035*
C15	0.00750 (18)	0.1195 (3)	0.0545 (8)	0.0284 (13)
H15	-0.0053	0.0865	-0.0215	0.034*
C8	0.0937 (2)	0.5739 (3)	0.0577 (7)	0.0313 (14)
H8	0.1005	0.6230	0.0224	0.038*
C7	0.0656 (2)	0.5299 (3)	-0.0314 (8)	0.0343 (15)
H7	0.0542	0.5487	-0.1302	0.041*
C6	0.0541 (2)	0.4581 (3)	0.0261 (8)	0.0347 (15)
H6	0.0350	0.4285	-0.0352	0.042*

### Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
I3	0.02447 (18)	0.02458 (16)	0.02313 (18)	0.00572 (12)	0.00691 (17)	-0.00173 (16)
I2	0.03117 (19)	0.02441 (17)	0.02114 (18)	0.00247 (14)	-0.01031 (16)	-0.00504 (16)

I1	0.0322 (2)	0.01704 (15)	0.0299 (2)	-0.00235 (14)	-0.00951 (16)	0.00849 (16)
I4	0.01828 (16)	0.01997 (15)	0.02279 (17)	-0.00410 (12)	-0.00100 (14)	-0.00314 (15)
N1	0.019 (2)	0.012 (2)	0.020 (2)	0.0002 (16)	-0.0024 (17)	-0.0020 (17)
N3	0.022 (2)	0.0113 (19)	0.014 (2)	0.0012 (16)	-0.0021 (17)	-0.0004 (17)
C14	0.023 (3)	0.019 (3)	0.021 (3)	0.003 (2)	-0.003 (2)	-0.002 (2)
C9	0.030 (3)	0.018 (3)	0.017 (3)	-0.003 (2)	-0.001 (2)	0.000 (2)
C3	0.016 (2)	0.017 (2)	0.017 (3)	-0.0009 (19)	-0.002 (2)	0.001 (2)
C1	0.019 (3)	0.013 (2)	0.014 (3)	-0.0019 (19)	-0.0007 (19)	0.0012 (19)
C4	0.020 (2)	0.019 (2)	0.014 (3)	0.0032 (19)	0.002 (2)	0.000 (2)
C13	0.021 (3)	0.011 (2)	0.014 (3)	0.0038 (18)	-0.006 (2)	0.0051 (19)
C11	0.019 (2)	0.013 (2)	0.017 (3)	0.0060 (18)	0.004 (2)	-0.001 (2)
C2	0.015 (2)	0.015 (2)	0.014 (3)	0.0024 (18)	0.003 (2)	0.0008 (19)
C12	0.018 (3)	0.014 (2)	0.019 (3)	-0.0025 (19)	0.001 (2)	0.001 (2)
C10	0.024 (3)	0.010 (2)	0.012 (2)	-0.0031 (18)	-0.001 (2)	-0.0004 (18)
N2	0.024 (2)	0.0103 (19)	0.021 (3)	0.0011 (16)	-0.0028 (18)	-0.0033 (17)
C18	0.022 (3)	0.018 (2)	0.027 (3)	0.0016 (19)	-0.001 (2)	-0.001 (2)
C5	0.028 (3)	0.019 (3)	0.034 (3)	-0.003 (2)	-0.008 (2)	0.005 (2)
C16	0.020 (3)	0.037 (3)	0.033 (4)	-0.008 (2)	-0.002 (3)	0.001 (3)
N4	0.017 (2)	0.0127 (18)	0.014 (2)	0.0015 (15)	-0.0053 (17)	-0.0043 (17)
C17	0.017 (3)	0.034 (3)	0.038 (4)	-0.001 (2)	0.002 (3)	0.002 (3)
C15	0.023 (3)	0.031 (3)	0.031 (3)	-0.006 (2)	-0.010 (2)	0.000 (2)
C8	0.045 (4)	0.024 (3)	0.026 (3)	-0.006 (2)	-0.009 (3)	0.007 (2)
C7	0.047 (4)	0.030 (3)	0.026 (4)	0.003 (3)	-0.015 (3)	0.009 (3)
C6	0.044 (4)	0.030 (3)	0.030 (4)	-0.013 (2)	-0.020 (3)	0.004 (3)

*Geometric parameters (Å, °)*

I3—C11	2.075 (5)	C13—C10	1.467 (7)
I2—C3	2.051 (5)	C11—C12	1.350 (7)
I1—C2	2.058 (5)	C12—N4	1.360 (6)
I4—C12	2.058 (5)	C10—N4	1.345 (6)
N1—C1	1.323 (6)	N2—H2	0.8600
N1—C2	1.374 (6)	C18—C17	1.382 (7)
N3—C10	1.343 (6)	C18—H18	0.9300
N3—C11	1.379 (6)	C5—C6	1.370 (8)
C14—C15	1.376 (8)	C5—H5	0.9302
C14—C13	1.388 (7)	C16—C17	1.374 (8)
C14—H14	0.9301	C16—C15	1.388 (9)
C9—C4	1.381 (7)	C16—H16	0.9299
C9—C8	1.391 (8)	N4—H4	0.8600
C9—H9	0.9301	C17—H17	0.9300
C3—C2	1.360 (7)	C15—H15	0.9299
C3—N2	1.366 (6)	C8—C7	1.367 (8)
C1—N2	1.363 (6)	C8—H8	0.9301
C1—C4	1.472 (7)	C7—C6	1.385 (8)
C4—C5	1.383 (7)	C7—H7	0.9299
C13—C18	1.393 (7)	C6—H6	0.9299
C1—N1—C2	105.9 (4)	N4—C10—C13	124.7 (4)
C10—N3—C11	104.1 (4)	C1—N2—C3	107.4 (4)



## supplementary materials

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C15—C14—C13	120.8 (5)	C1—N2—H2	126.0
C15—C14—H14	119.8	C3—N2—H2	126.6
C13—C14—H14	119.3	C17—C18—C13	120.0 (5)
C4—C9—C8	120.1 (5)	C17—C18—H18	120.3
C4—C9—H9	119.9	C13—C18—H18	119.8
C8—C9—H9	120.0	C6—C5—C4	119.8 (5)
C2—C3—N2	106.2 (4)	C6—C5—H5	120.3
C2—C3—I2	129.5 (4)	C4—C5—H5	120.0
N2—C3—I2	124.3 (3)	C17—C16—C15	119.2 (5)
N1—C1—N2	110.6 (4)	C17—C16—H16	120.2
N1—C1—C4	124.5 (4)	C15—C16—H16	120.6
N2—C1—C4	124.9 (4)	C10—N4—C12	108.6 (4)
C9—C4—C5	119.8 (5)	C10—N4—H4	126.0
C9—C4—C1	121.4 (4)	C12—N4—H4	125.4
C5—C4—C1	118.9 (4)	C16—C17—C18	121.0 (5)
C14—C13—C18	118.7 (5)	C16—C17—H17	119.8
C14—C13—C10	119.9 (4)	C18—C17—H17	119.2
C18—C13—C10	121.4 (4)	C14—C15—C16	120.2 (5)
C12—C11—N3	111.2 (4)	C14—C15—H15	120.0
C12—C11—I3	129.8 (4)	C16—C15—H15	119.7
N3—C11—I3	118.9 (3)	C7—C8—C9	119.8 (5)
C3—C2—N1	109.9 (4)	C7—C8—H8	120.4
C3—C2—I1	127.4 (4)	C9—C8—H8	119.9
N1—C2—I1	122.6 (3)	C8—C7—C6	119.9 (5)
C11—C12—N4	105.4 (4)	C8—C7—H7	119.4
C11—C12—I4	132.2 (4)	C6—C7—H7	120.6
N4—C12—I4	122.3 (3)	C5—C6—C7	120.6 (5)
N3—C10—N4	110.7 (4)	C5—C6—H6	119.9
N3—C10—C13	124.6 (4)	C7—C6—H6	119.4

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 $\cdots$ N3 <sup>i</sup>	0.86	2.01	2.845 (5)	165.
N4—H4 $\cdots$ N1	0.86	2.02	2.848 (6)	162.

Symmetry codes: (i)  $x, y+1/2, z+1/2$ .

Fig. 1

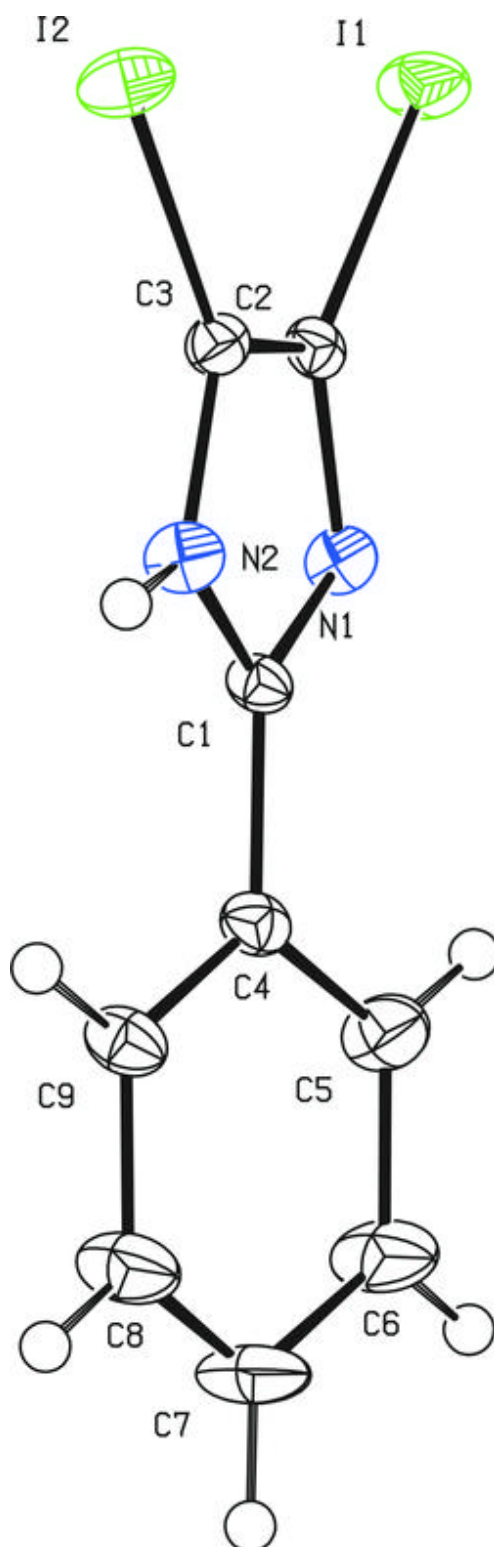


Fig. 2

