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Key indicators

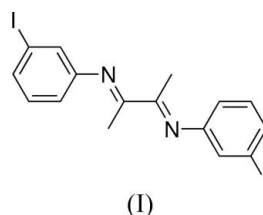
Single-crystal X-ray study
 $T = 193\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.026
 wR factor = 0.068
Data-to-parameter ratio = 16.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.(3-Iodophenyl)[2-(3-iodophenylimino)-1-methyl-
propylidene]amineIn the crystal structure of the title compound, $\text{C}_{16}\text{H}_{14}\text{I}_2\text{N}_2$, the molecule lies on a crystallographic inversion center and hence the two imine groups are mutually *trans*.

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Comment

Molecules containing the 1,4-diaza-1,3-butadiene skeleton are interesting because of their versatile coordination behavior and the properties of their metal complexes (van Koten & Vrieze, 1982). The two imine groups of the title compound, (I), are planar. The angle between the planes of the diimine group and each benzene ring is $89.3(2)^\circ$.

Experimental

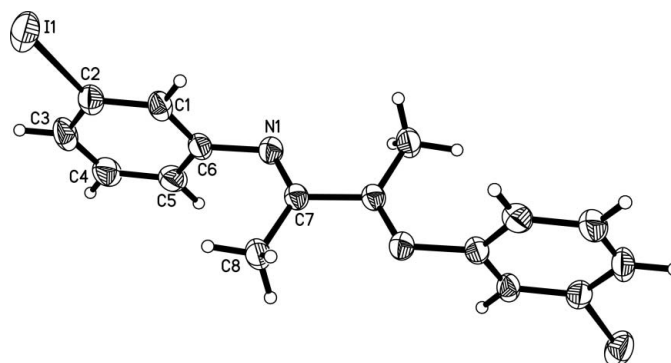
The title compound was prepared by the reaction of 2,3-butanedione with 2 equivalents of 3-iodoaniline in the presence of *p*-toluenesulfonic acid in toluene solvent using Dean–Stark apparatus (Hell-dörfer *et al.*, 2003). The product was separated by silica gel column chromatography (ethyl acetate/*n*-hexane, 1:20) with 3% triethylamine and was recrystallized from diethyl ether at room temperature. Single crystals suitable for X-ray diffraction were grown at room temperature by evaporation of a diethyl ether solution. ^1H NMR (500 MHz, $\text{THF}-d_8$): δ 7.45 (*m*, 2H), 7.20 (*t*, 2H), 7.12 (*t*, 2H), 6.78 (*m*, 2H), 2.11 (*s*, 6H). ^{13}C NMR (126 MHz, $\text{THF}-d_8$): δ 169.4, 153.4, 133.5, 131.4, 128.4, 118.9, 95.0, 15.3. HRMS calculated for $\text{C}_{16}\text{H}_{14}\text{I}_2\text{N}_2$: 487.9247; found: 487.9247.

Figure 1
SHELXTL (Bruker, 2001) plot showing 35% probability ellipsoids for non-H atoms and circles of arbitrary size for H atoms. The unlabeled atoms are related by the symmetry operator $(2 - x, -y, -z)$.

Crystal data

C₁₆H₁₄I₂N₂
M_r = 488.09
 Orthorhombic, *Pbca*
a = 11.481 (2) Å
b = 9.2619 (19) Å
c = 15.723 (3) Å
V = 1672.0 (6) Å³
Z = 4
D_x = 1.939 Mg m⁻³

Data collection

Siemens SMART/Platform CCD
 diffractometer
 ω scans
 Absorption correction: integration
 (*SHELXTL/XPREP*; Bruker,
 2001)
T_{min} = 0.496, *T_{max}* = 0.754
 13409 measured reflections

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.026
wR(*F*²) = 0.068
S = 0.99
 1530 reflections
 92 parameters
 H-atom parameters constrained

Mo *K*α radiation
 Cell parameters from 950
 reflections
 θ = 3.1–25.9°
 μ = 3.75 mm⁻¹
T = 193 (2) K
 Tabletr, yellow
 0.22 × 0.20 × 0.08 mm

1530 independent reflections
 1067 reflections with *I* > 2σ(*I*)
R_{int} = 0.059
 θ_{max} = 25.4°
h = -13 → 13
k = -10 → 11
l = -18 → 18

$$w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 0.4421P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.51 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.68 \text{ e } \text{Å}^{-3}$

Methyl H-atom positions, *R*-CH₃, were optimized by rotation about the *R*-C bonds with idealized C-H (0.98 Å). The remaining H atoms were included as riding (C-H = 0.95 Å). Methyl H atom *U_{iso}* values were assigned as 1.5 times *U_{eq}* of the carrier atom; the remaining *U_{iso}*(H) values were assigned as 1.2 times *U_{eq}*(carrier).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *XCIF* (Bruker, 2001).

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References

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