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7,8-Dichloro-2,3-diphenylquinoxaline

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Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.108
Data-to-parameter ratio = 33.6

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

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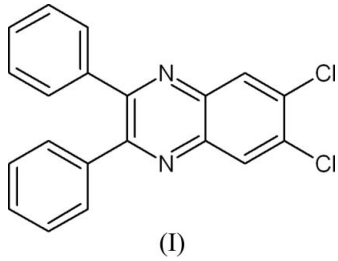
The title compound, $C_{20}H_{12}Cl_2N_2$, was synthesized from the reaction of equimolar amounts of 4,5-dichlorobenzene-1,2-diamine and benzil in acetonitrile, utilizing molecular iodine as the catalyst. The crystal structure was determined at 110 K. The dihedral angle between the two phenyl rings is 57.0 (1)°.

Comment

During the past several years, quinoxalines have been used effectively as building blocks for metal-containing two-dimensional networks (Willett *et al.*, 2001). Our previous investigations have shown that metal halide salts coordinate readily with quinoxalines to form interesting coordination polymers (Dueno *et al.*, 2006). Our current work involves the synthesis of substituted quinoxalines (Brown *et al.*, 2004), which may lead to novel three-dimensional structures upon coordination to copper halides.



(I)

We report here the crystal structure of 6,7-dichloro-2,3-diphenylquinoxaline, (I) (Fig. 1), which exhibits bond distances and angles that are unexceptional, all falling within ranges established in the literature (Stacy *et al.*, 2005). Due to their close proximity, the phenyl substituents are not coplanar with the quinoxaline rings. The torsion angle C1—C2—C15—C20 is −157.6 (1)° and C2—C1—C9—C14 is −127.7 (1)°.The dihedral angle between the two phenyl rings is 57.0 (1)°.

Experimental

A 50 ml round-bottomed flask was charged with 4,5-dichlorobenzene-1,2-diamine (0.250 g, 1.41 mmol), benzil (122 mg, 1.41 mmol), iodine (36 mg, 0.141 mmol), and acetonitrile (15 ml). The reaction was monitored by thin-layer chromatography until complete consumption of the starting materials (20 min). The resulting amber solution was concentrated to dryness under reduced pressure. The dark-purple crude product was then subjected to flash column chromatography using silica gel (eluent: 95:5 hexane–EtOAc) in order to remove residual iodine. The pale-yellow solution was evaporated to dryness under reduced pressure to give (I) (yield 0.426 mg, 86%) as a white powder (m.p. 430 K). This powder was then crystallized from a minimal amount of ethanol, and afforded (I) as pale-yellow needles.

Crystal data

$C_{20}H_{12}Cl_2N_2$
M_r = 351.22
Monoclinic, $C2/c$
a = 25.158 (3) Å
b = 7.8022 (10) Å
c = 18.678 (2) Å
β = 119.697 (6)°
V = 3184.7 (7) Å^3
Z = 8
D_x = 1.465 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 110 K
Fragment cut from a needle, light yellow
0.23 × 0.20 × 0.15 mm

Data collection

Nonius KappaCCD (with an Oxford Cryosystems Cryostream cooler) diffractometer
ω scans with κ offsets
Absorption correction: multi-scan (*DENZO* and *SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.912, T_{max} = 0.941
33023 measured reflections
7301 independent reflections
5571 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 35.6°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.108
S = 1.03
7301 reflections
217 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0445P)^2 + 2.2948P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

H atoms were positioned geometrically and refined as riding on their carrier atoms, with C—H = 0.95 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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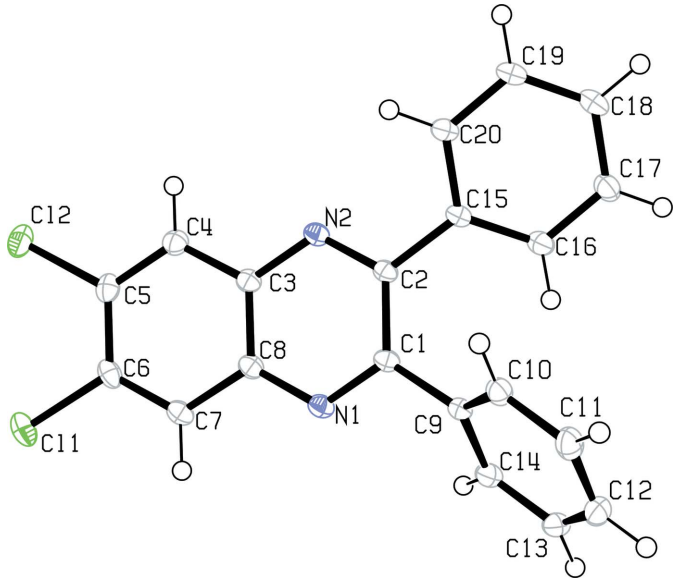

Figure 1
ORTEP-3 view (Farrugia, 1997) of (I). Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radius.

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