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2,5-Bis(3,7-dichloroquinolin-8-yl)-1,3,4-oxadiazole

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.003 Å
R factor = 0.038
wR factor = 0.103
Data-to-parameter ratio = 16.5

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

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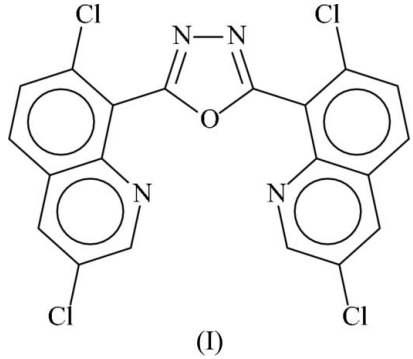
In the title molecular structure, $C_{20}H_8Cl_4N_4O$, the 1,3,4-triazole ring is twisted with an r.m.s. deviation of 0.0035 Å. One of the quinolinyl substituents makes a dihedral angle of 55.8 (1)° with respect to the central ring, while the other is rotated by 71.7 (1)°; these twists are necessary to relieve steric crowding.

Comment

The title compound, (I), is a diaryl-substituted 1,3,4-oxadiazole, an example of which is the previously determined crystal structure of 2,5-diphenyl-1,3,4-triazole (Kuznetsov *et al.*, 1998; Franco *et al.*, 2003). The synthesis of 1,3,4-oxadiazoles is readily achieved by the treatment of aromatic carboxylic acids with hydrazine dihydrochloride in a mixture of ortho-phosphoric acid, phosphorus pentoxide and phosphorus oxychloride (Bentiss & Lagrenee, 1999). Unlike the phenyl derivatives, only a few quinolinyl analogs have been reported (Dabhi *et al.*, 1992; Narayana *et al.*, 2005; Zhang *et al.*, 1989).



(I)

The title 1,3,4-triazole incorporates the quinclorac entity, which is used commercially as a potent herbicide (Grossmann, 1998), into its structure (Fig. 1). The 1,3,4-triazole ring is twisted with an r.m.s. deviation of 0.0035 Å. One of the quinolinyl substituents (containing atom N1) makes a dihedral angle of 55.8 (1)° with respect to the central ring, while the other quinolyl ring (containing atom N4) is rotated by 71.7 (1)°. These large twists arise from steric interactions; in contrast, the diphenyl analog is essentially planar (Kuznetsov *et al.*, 1998; Franco *et al.*, 2003).

Experimental

To a mixture of quinclorac (3,7-dichloro-8-quinolinecarboxylic acid) (24.2 g, 0.1 mol) and hydrazinium sulfate (6.5 g, 0.05 mol), which was dissolved in a mixture of 85% orthophosphoric acid (27 ml), were added phosphorus pentoxide (42.6 g, 0.3mol) and phosphorus

oxychloride (46.0 g, 0.1 mol). The viscous liquid was heated at 413 K for 4 h. The cooled mixture was poured on to crushed ice. Sodium hydroxide was added until the solution registered a neutral pH. The resulting white solid was collected, washed with water and then dried and purified by recrystallization from ethanol in about 80% yield (m.p. 508–509 K).

Crystal data

$C_{20}H_8Cl_4N_4O$	$Z = 8$
$M_r = 462.10$	$D_x = 1.631$ Mg m^{-3}
Orthorhombic, *Pbcn*	Mo $K\alpha$ radiation
$a = 14.1016$ (8) Å	$\mu = 0.65$ mm^{-1}
$b = 9.8811$ (6) Å	$T = 295$ (2) K
$c = 27.016$ (2) Å	Block, colorless
$V = 3764.4$ (4) Å^3	0.27 × 0.17 × 0.16 mm

Data collection

Bruker APEX-II area-detector diffractometer	4317 independent reflections
φ and ω scans	2953 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.037$
21631 measured reflections	$\theta_{max} = 27.5°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0437P)^2 + 1.1566P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.103$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
$S = 1.02$	$\Delta\rho_{min} = -0.30$ e Å^{-3}
4317 reflections	
262 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C11	1.354 (2)	N2—N3	1.420 (2)
O1—C10	1.358 (2)	N3—C11	1.282 (3)
N2—C10	1.280 (3)		
C11—O1—C10	103.0 (2)	N2—C10—O1	112.4 (2)
C10—N2—N3	106.1 (2)	N3—C11—O1	112.7 (2)
C11—N3—N2	105.7 (2)		

H atoms were positioned geometrically (C—H = 0.93 and 0.97Å) and were included in the refinement in the riding-model approximation, with U_{iso}(H) values set at 1.2U_{eq}(C).

Data collection: *SMART* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976) and *SHELXTL* (Bruker, 2004); software used to prepare material for publication: *SHELXL97*.

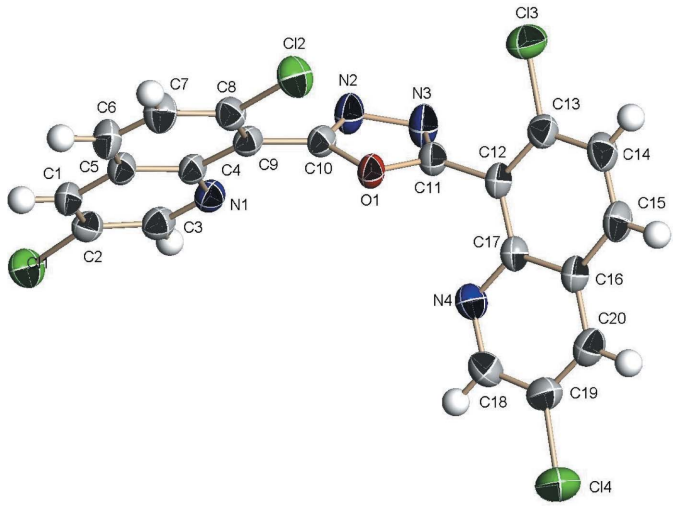


Figure 1
ORTEPII (Johnson, 1976) plot of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are drawn as spheres of arbitrary radii.

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