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3,7-Dichloroquinoline-8-carboxylic acid

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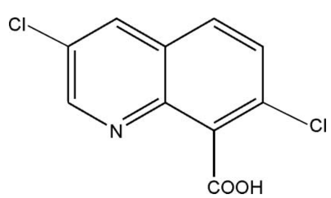
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.006 Å; R factor = 0.064; wR factor = 0.141; data-to-parameter ratio = 13.2.

The title compound (trade name: quinclorac), $C_{10}H_5Cl_2NO_2$, was crystallized from a dimethyl sulfoxide solution. Quinclorac molecules are packed mainly *via* π–π stacking interactions between neighbouring heterocycles (interplanar distance: 3.31 Å) and *via* O—H···N hydrogen bonding.

Related literature

For the use of 3,7-dichloroquinoline-8-carboxylic acid as a herbicide, see: Nuria *et al.* (1997); Pornprom *et al.* (2006); Sunohara & Matsumoto (2004); Tresch & Grossmann (2002). For related complexes, see: Li *et al.* (2008); Turel *et al.* (2004); Zhang *et al.* (2007).



Experimental

Crystal data

$C_{10}H_5Cl_2NO_2$
M_r = 242.05
Triclinic, $P\overline{1}$
a = 7.5002 (12) Å
b = 8.4016 (14) Å
c = 8.732 (3) Å
α = 102.529 (6)°
β = 93.439 (6)°
γ = 116.479 (4)°
V = 472.98 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 173 (2) K
0.26 × 0.22 × 0.20 mm

Data collection

Bruker SMART APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.84, T_{max} = 0.88
5948 measured reflections
1834 independent reflections
1102 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.140
S = 1.01
1834 reflections
139 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N1^i	0.84 (5)	1.91 (5)	2.753 (4)	173 (4)

Symmetry code: (i) $-x+1, -y+2, -z+1$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2136).

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supporting information

Acta Cryst. (2008). E**64**, o1786 [doi:10.1107/S1600536808026238]

3,7-Dichloroquinoline-8-carboxylic acid

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S1. Comment

Quinclorac (3,7-dichloroquinoline-8-carboxylic acid) is one of the most effective herbicides (Nuria *et al.*, 1997; Pornprom *et al.*, 2006; Sunohara & Matsumoto, 2004; Tresch & Grossmann, 2002), and is widely used in agriculture. In addition, as a quinolinecarboxylate derivate, quinclorac could chelate metal ions, forming corresponding complexes (Li *et al.*, 2008; Turel *et al.*, 2004; Zhang *et al.*, 2007). As an extension of these studies, we report herein on the structure of quinclorac.

A quinclorac molecule, which is the asymmetric unit of the structure, is shown in Fig. 1. All the bond distances and bond angles of quinclorac are normal and call for no further comment. Two types of intermolecular interations are easily found in the structure of quinclorac (Fig. 2). There exists a π-π interaction between adjacent quinin cycles with an inversion center located halfway between the aromatic rings, thus forming stacks along the *a* direction. Quinclorac molecules of adjacent chains are joined through H-bonding of O1—H1···N1^{i} (symmetry code: (i) 1 - *x*, 2 - *y*, 1 - *z*) (Table 1) into a triclinic supramolecular architecture (Fig. 2).

S2. Experimental

Quinclorac was obtained from a commercial source and used directly without further purification. Quinclorac (0.5 mmol, 0.121 g) was dissolved in 10 mL DMSO. After ether vapor slowly diffused into the solution at room temperature for several days, colorless prismlike crystals suitable for crystallographic research were obtained.

S3. Refinement

All the non-hydrogen atoms were located from the Fourier maps, and were refined anisotropically. The hydroxyl hydrogen, H1A, was found from the Fourier difference maps and refined isotropically with a fixed O—H bond length. All other H atoms were positioned geometrically. All isotropic vibration parameters of hydrogen atoms were related to the atoms which they are bonded to with U_{iso}(H) = 1.2 U_{eq}(C,O).

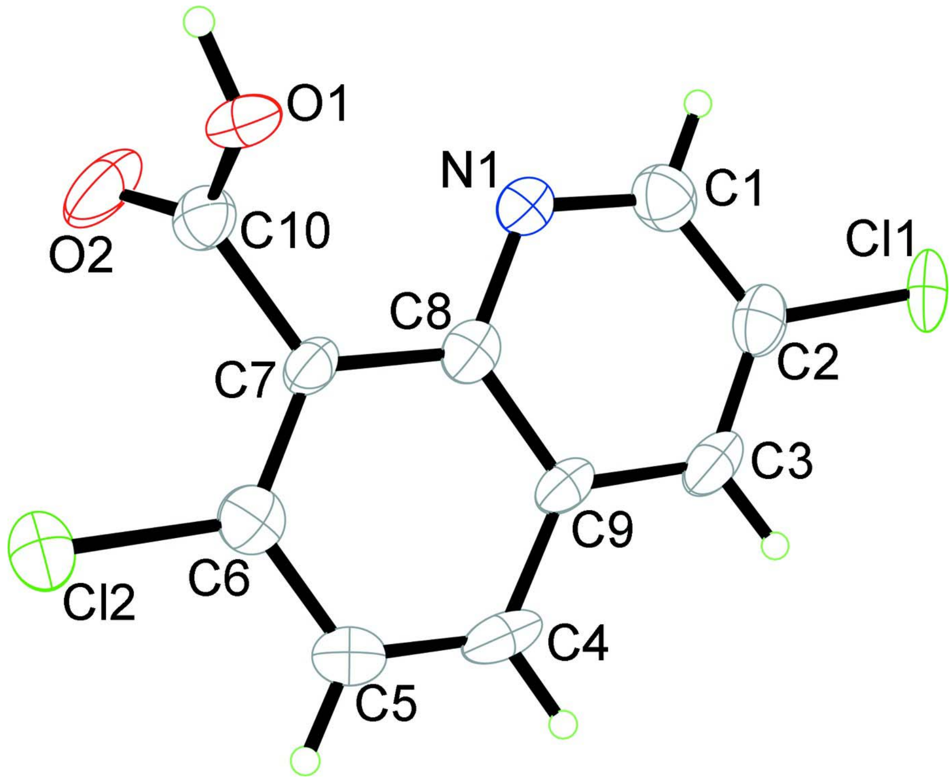

Figure 1

The asymmetric unit of quinclorac with atom labels and 50% probability displacement ellipsoids for non-H atoms.

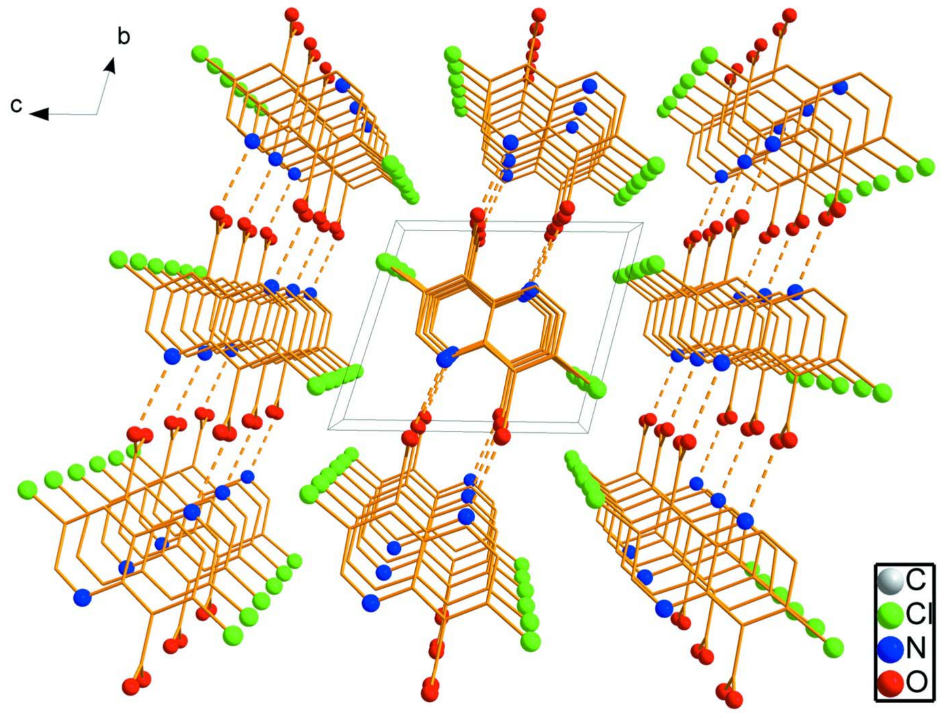

Figure 2

Packing diagram of quinclorac showing the π-π stacks along the *a* direction. Intermolecular H-bonding is indicated via dashed lines.

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Crystal data

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Hall symbol: -P 1
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c = 8.732 (3) Å
α = 102.529 (6)°
β = 93.439 (6)°
γ = 116.479 (4)°
V = 472.98 (17) Å^3
Z = 2
F(000) = 244
D_x = 1.700 Mg m^{-3}
Mo *Kα* radiation, λ = 0.71073 Å
Cell parameters from 958 reflections
θ = 2.1–25.5°
μ = 0.66 mm^{-1}
T = 173 K
Prismlike, colorless
0.26 × 0.22 × 0.20 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.84, T_{max} = 0.88
5948 measured reflections
1834 independent reflections
1102 reflections with $I > 2\sigma(I)$
R_{int} = 0.067
θ_{max} = 26.0°, θ_{min} = 2.4°
$h = -9 \rightarrow 9$
$k = -8 \rightarrow 10$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.140$
$S = 1.01$
1834 reflections
139 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.43$ e Å^{-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 > 2sigma(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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.3476 (6)	0.5471 (6)	0.2465 (5)	0.0354 (10)
H1	0.3861	0.5957	0.1583	0.043*
C2	0.2932 (6)	0.3616 (6)	0.2276 (5)	0.0324 (9)
C3	0.2343 (6)	0.2876 (6)	0.3515 (5)	0.0337 (10)
H3	0.1963	0.1613	0.3413	0.040*
C4	0.1686 (6)	0.3342 (6)	0.6269 (5)	0.0369 (10)
H4	0.1286	0.2086	0.6220	0.044*
C5	0.1655 (7)	0.4475 (6)	0.7605 (5)	0.0363 (10)
H5	0.1215	0.4011	0.8489	0.044*
C6	0.2272 (6)	0.6342 (6)	0.7699 (5)	0.0308 (9)
C7	0.2918 (6)	0.7078 (5)	0.6455 (5)	0.0253 (8)
C8	0.2910 (5)	0.5882 (5)	0.5036 (5)	0.0261 (8)
C9	0.2304 (6)	0.4002 (5)	0.4943 (5)	0.0261 (8)
C10	0.3645 (6)	0.9105 (5)	0.6610 (4)	0.0293 (9)
Cl1	0.29545 (15)	0.22889 (15)	0.04666 (12)	0.0385 (3)
Cl2	0.23077 (17)	0.77619 (16)	0.94953 (12)	0.0422 (3)
N1	0.3496 (5)	0.6591 (4)	0.3774 (4)	0.0283 (7)
O1	0.5586 (4)	0.9997 (4)	0.6659 (3)	0.0302 (6)
H1A	0.597 (7)	1.106 (7)	0.652 (5)	0.036*
O2	0.2510 (5)	0.9766 (4)	0.6634 (5)	0.0519 (9)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.038 (2)	0.041 (3)	0.032 (2)	0.021 (2)	0.0079 (18)	0.012 (2)

C2	0.027 (2)	0.032 (2)	0.039 (2)	0.0174 (19)	0.0036 (17)	0.0030 (19)
C3	0.033 (2)	0.021 (2)	0.049 (2)	0.016 (2)	0.0051 (19)	0.008 (2)
C4	0.037 (2)	0.025 (2)	0.053 (3)	0.014 (2)	0.006 (2)	0.022 (2)
C5	0.043 (2)	0.035 (3)	0.036 (2)	0.019 (2)	0.0106 (19)	0.017 (2)
C6	0.0262 (19)	0.035 (2)	0.033 (2)	0.0165 (18)	0.0041 (16)	0.0098 (19)
C7	0.028 (2)	0.020 (2)	0.032 (2)	0.0138 (17)	0.0062 (16)	0.0073 (17)
C8	0.0180 (18)	0.024 (2)	0.034 (2)	0.0095 (16)	0.0014 (15)	0.0050 (17)
C9	0.0258 (18)	0.020 (2)	0.036 (2)	0.0126 (16)	0.0030 (16)	0.0091 (17)
C10	0.034 (2)	0.025 (2)	0.0246 (19)	0.0119 (18)	0.0010 (15)	0.0038 (18)
Cl1	0.0372 (6)	0.0432 (7)	0.0423 (6)	0.0303 (5)	0.0101 (5)	−0.0006 (5)
Cl2	0.0548 (7)	0.0433 (7)	0.0343 (6)	0.0280 (6)	0.0144 (5)	0.0089 (5)
N1	0.0318 (18)	0.0235 (18)	0.0295 (17)	0.0130 (15)	0.0066 (13)	0.0069 (15)
O1	0.0319 (16)	0.0189 (15)	0.0364 (16)	0.0071 (13)	0.0056 (12)	0.0120 (13)
O2	0.048 (2)	0.0257 (17)	0.092 (3)	0.0240 (16)	0.0204 (18)	0.0171 (18)

Geometric parameters (Å, º)

C1—N1	1.308 (5)	C5—H5	0.9500
C1—C2	1.391 (6)	C6—C7	1.373 (6)
C1—H1	0.9500	C6—Cl2	1.743 (4)
C2—C3	1.362 (6)	C7—C8	1.414 (5)
C2—Cl1	1.731 (4)	C7—C10	1.510 (5)
C3—C9	1.403 (6)	C8—N1	1.369 (5)
C3—H3	0.9500	C8—C9	1.417 (5)
C4—C5	1.345 (6)	C10—O2	1.206 (5)
C4—C9	1.405 (5)	C10—O1	1.299 (5)
C4—H4	0.9500	O1—H1A	0.84 (5)
C5—C6	1.405 (6)		
N1—C1—C2	124.6 (4)	C7—C6—Cl2	119.9 (3)
N1—C1—H1	117.7	C5—C6—Cl2	118.0 (3)
C2—C1—H1	117.7	C6—C7—C8	117.8 (4)
C3—C2—C1	118.9 (4)	C6—C7—C10	121.0 (3)
C3—C2—Cl1	121.5 (3)	C8—C7—C10	121.2 (3)
C1—C2—Cl1	119.6 (3)	N1—C8—C7	118.2 (4)
C2—C3—C9	119.2 (4)	N1—C8—C9	121.6 (3)
C2—C3—H3	120.4	C7—C8—C9	120.2 (4)
C9—C3—H3	120.4	C3—C9—C4	122.9 (4)
C5—C4—C9	120.5 (4)	C3—C9—C8	118.0 (4)
C5—C4—H4	119.7	C4—C9—C8	119.1 (3)
C9—C4—H4	119.7	O2—C10—O1	125.4 (4)
C4—C5—C6	120.2 (4)	O2—C10—C7	122.5 (3)
C4—C5—H5	119.9	O1—C10—C7	112.1 (3)
C6—C5—H5	119.9	C1—N1—C8	117.7 (3)
C7—C6—C5	122.1 (4)	C10—O1—H1A	113 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···N1[i]	0.84 (5)	1.91 (5)	2.753 (4)	173 (4)

Symmetry code: (i) −*x*+1, −*y*+2, −*z*+1.