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(*S*)-2-(2-Chloroquinolin-3-yl)-2-[(*S*)-α-methylbenzylamino]acetonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.055
wR factor = 0.093
Data-to-parameter ratio = 14.4

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

The title compound, $C_{19}H_{16}ClN_3$, crystallizes with two independent molecules in the asymmetric unit. The structure is stabilized by C—H···N, N—H···N and C—H···Cl hydrogen bonds.

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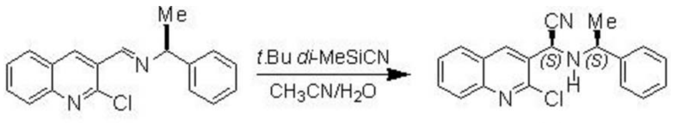
Comment

Quinolines are an important group of heterocyclic compounds. Among these, 2-chloro-3-formylquinolines occupy a prominent position as they are key intermediates for further (β)-annelation of a wide variety of rings and for various functional group interconversions (Meth-Cohn, 1993). Particular interest in quinoline derivatives arises owing to their biological activity, namely as antibiotics (Jackson & Meth-Cohn, 1995; Kansagra *et al.*, 2000), anti-inflammatories (Schroderet, 1989), anti-tumourals (Joseph *et al.*, 2002)), anti-oxidants (Laalaoui *et al.*, 2003) and analgesics (Heide *et al.*, 1986; Solomon, 1970). In the same way, α-aminoacids are of great biological and economic importance (Williams, 1989). The asymmetric Strecker reaction is one of the most important methods for the synthesis of enantiomerically pure α-aminonitrile derivatives, which are useful intermediates for the synthesis of α-aminoacids. The use of (*S*)-(−)-α-methylbenzylamine-derived aldimines has a significant role in the diastereoselective Strecker synthesis (Bhanu-Prasad *et al.*, 2004).

In recent years, we have developed a programme devoted to the synthesis and biological evaluation of quinolyl derivatives (Moussaoui *et al.*, 2002; Kedjadja *et al.*, 2004; Menasra *et al.*, 2004, 2005; Rezig *et al.*, 2000). In a continuation of our efforts in this area, we report here a short and efficient procedure for the preparation of the title α-aminonitrile, (2), containing a quinolyl ring system, and its crystal structure determination.



(1) (2)

The crystallographic asymmetric unit of (2) contains two independent molecules, labelled *a* and *b* (Fig. 1). The analysis shows that atoms C11 and C12 each have an *S* configuration in both independent molecules. The geometric parameters of (2) (Table 1) are in agreement with those of other structures containing similar molecular connectivity (Benali-Cherif, Cherouana *et al.*, 2002; Benali-Cherif, Dokhane & Abdaoui, 2002). The 11 atoms defining the chloroquinolyl planes, *i.e.* N1 and Cl1–C10, have maximum deviations of 0.0037 (3) Å for

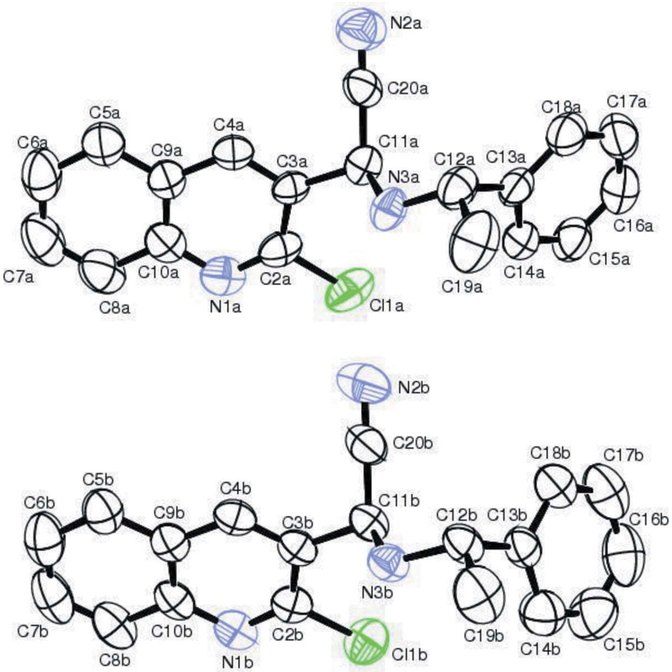


Figure 1
Views of the two independent molecules of (2), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

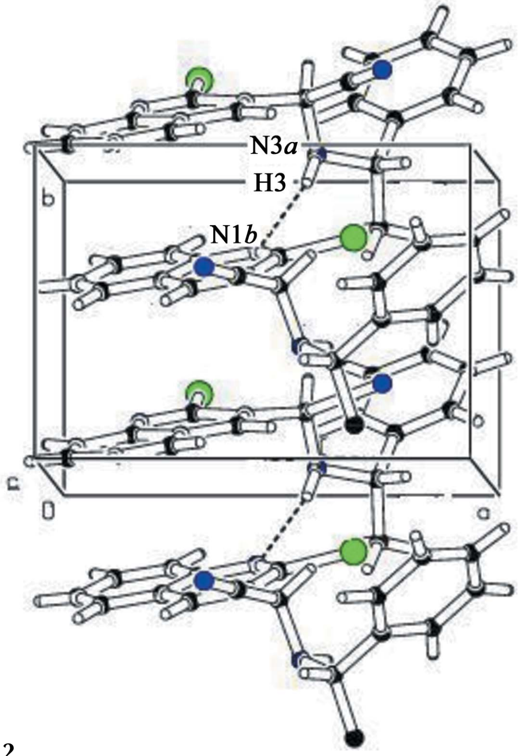


Figure 2
The unit-cell contents of (2), highlighting the N—H···N hydrogen bonding (dashed lines).

atom C7*a* and 0.0035 (3) Å for atom C7*b* for the two independent molecules. In the crystal structure, these planes are almost parallel, forming a dihedral angle of 7.40 (4)°. The clear difference between molecules *a* and *b* is noted in the dihedral angles formed between the chloroquinolyl and phenyl groups of 27.83 (6) and 50.53 (8)°, respectively.

The three-dimensional crystal structure of (2) is stabilized *via* a variety of hydrogen-bonding interactions of the types C—H···N, N—H···N and C—H···Cl (Steiner, 1996), as analysed by *PARST* (Nardelli, 1995) and summarized in Fig. 2 and Table 2.

Experimental

Chiral imine (1) was prepared by condensation of optically active (*S*)-(−)-α-methylbenzylamine with 2-chloro-3-formylquinoline, according to the literature procedure of Meth-Cohn *et al.* (1981). Treatment of (1) with *tert*-butyldimethylsilyl cyanide at room temperature in CH_3CN solution with a few drops of water provided a mixture of two diastereoisomers as a yellow solid (yield 84%). Crystals of (2) were obtained by fractional crystallization from a hexane–CH_2Cl_2 (9:1) solution of this mixture. The isomeric ratio of (2) (63%) was determined from the ^{1}H NMR spectrum of the crude product (m.p. 383 K).

Crystal data

$C_{19}H_{16}ClN_3$
$M_r = 321.8$
Monoclinic, $P2_1$
$a = 9.8540\ (1)$ Å
$b = 7.1090\ (1)$ Å
$c = 23.9330\ (3)$ Å
$\beta = 91.590\ (2)^\circ$
$V = 1675.91\ (4)$ Å^3
$Z = 4$
$D_x = 1.275$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 2548 reflections
$\theta = 1.7$–28.0°
$\mu = 0.23$ mm^{-1}
$T = 293\ (2)$ K
Prism, yellow
0.2 × 0.15 × 0.1 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: none
35588 measured reflections
7662 independent reflections
4482 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.065$
$\theta_{\rm max} = 28.0^\circ$
$h = -13 \rightarrow 12$
$k = -8 \rightarrow 9$
$l = -31 \rightarrow 31$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.093$
$S = 1.03$
7662 reflections
531 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0312P)^2 + 0.1354P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.002$
$\Delta\rho_{\rm max} = 0.15$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.15$ e Å^{-3}
Absolute structure: Flack (1983), 3303 Friedel pairs
Flack parameter: −0.01 (5)

Table 1
Selected geometric parameters (Å, °).

N2*a*—C20*a*	1.113 (3)	N2*b*—C20*b*	1.134 (3)
C17*a*—C18*a*	1.374 (4)	C17*b*—C18*b*	1.394 (5)
N3*a*—C11*a*—C20*a*	114.4 (2)	N3*b*—C11*b*—C20*b*	109.6 (2)
C11*a*—N3*a*—C12*a*	117.1 (2)	C11*b*—N3*b*—C12*b*	114.8 (2)
C3*a*—C11*a*—N3*a*—C12*a*	−169.6 (2)	C3*b*—C11*b*—N3*b*—C12*b*	178.9 (2)
C4*a*—C3*a*—C11*a*—N3*a*	100.8 (3)	C4*b*—C3*b*—C11*b*—N3*b*	89.2 (3)
C19*a*—C12*a*—C13*a*—C18*a*	−99.7 (3)	C19*b*—C12*b*—C13*b*—C18*b*	−125.7 (3)

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Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3*a*—H3···N1*b*^{i}	0.90 (3)	2.31 (3)	3.189 (3)	165.6 (2)
N3*b*—H3′···N2*a*	0.87 (2)	2.68 (2)	3.508 (3)	159.6 (19)
C11*b*—H11′···Cl1*b*	0.98 (2)	2.641 (19)	3.067 (3)	106.3 (13)
C11*a*—H11···Cl1*a*	1.00 (3)	2.76 (2)	3.044 (3)	97.4 (14)

Symmetry code: (i) *x*, *y* − 1, *z*.

All H atoms were freely refined except for the methyl H atoms bonded to atom C19, for which C—H = 0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm iso}$(C).

Data collection: *KappaCCD Server Software* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *enCIFer* (Allen *et al.*, 2004).

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