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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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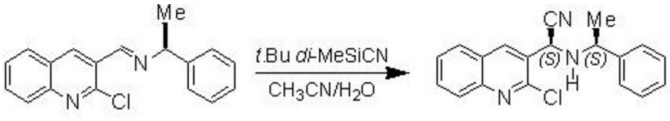
Comment

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

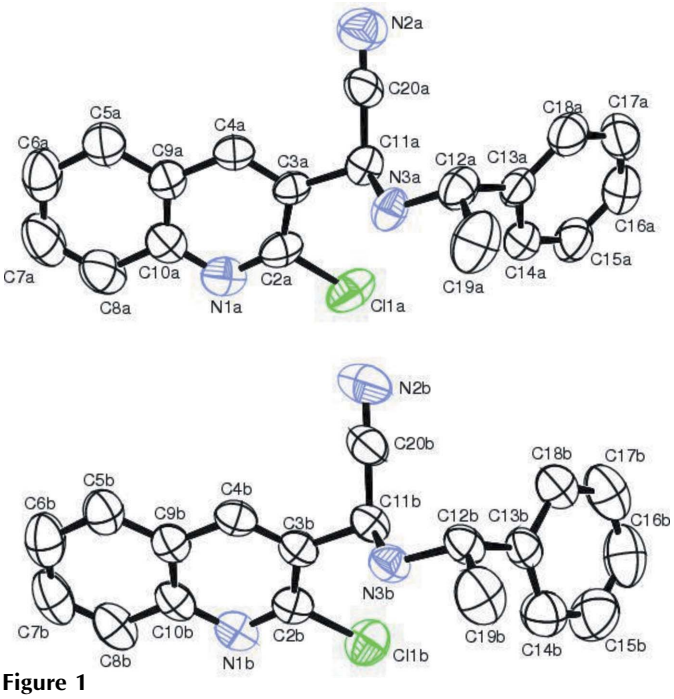


Figure 1
Views of the two independent molecules of (2), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted for clarity.

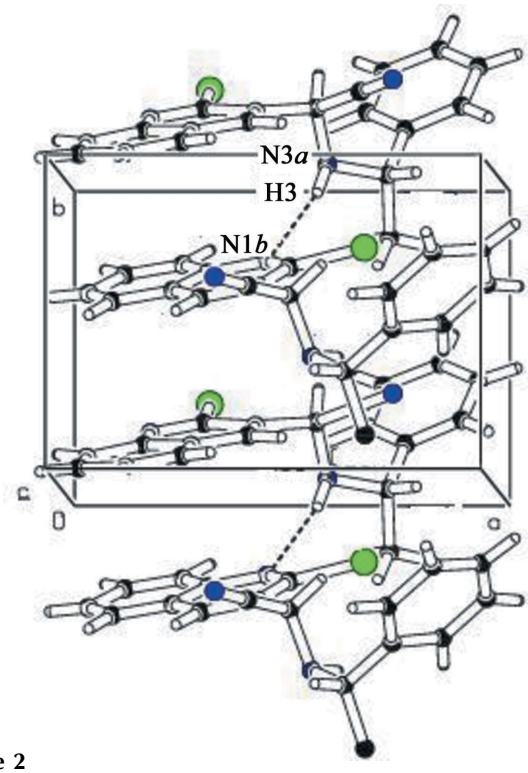


Figure 2
The unit-cell contents of (2), highlighting the N—H···N hydrogen bonding (dashed lines).

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
β = 91.590 (2)°
V = 1675.91 (4) Å^3
Z = 4
D_x = 1.275 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2548 reflections
θ = 1.7–28.0°
μ = 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_{int} = 0.065
θ_{max} = 28.0°
$h = -13 \rightarrow 12$
$k = -8 \rightarrow 9$
$l = -31 \rightarrow 31$

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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −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)

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

D—H···A	D—H	H···A	D···A	D—H···A
N3a—H3···N1b^{i}	0.90 (3)	2.31 (3)	3.189 (3)	165.6 (2)
N3b—H3′···N2a	0.87 (2)	2.68 (2)	3.508 (3)	159.6 (19)
C11b—H11′···Cl1b	0.98 (2)	2.641 (19)	3.067 (3)	106.3 (13)
C11a—H11···Cl1a	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_{iso}(H) = 1.5U_{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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