Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Ethyl 4-butylamino-2-chloroquinoline-3-carboxylate

Vyacheslav N. Baumer,[a]* Oleg V. Shishkin,[a] Igor V. Ukrainets,[b] Svetlana G. Taran[b] and Jaradat Nidal Amin[b]

[a]Department of Alkali Halide Crystals, Institute for Single Crystals, National Academy of Sciences of Ukraine, 60 Lenina ave., Kharkiv 61001, Ukraine, and [b]Department of Pharmaceutical Chemistry, Ukrainian National Academy of Pharmacy, 4 Blyukhera str., Kharkiv 61002, Ukraine

Correspondence e-mail: baumer@xray.isc.kharkov.com

Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.072
Data-to-parameter ratio = 20.3

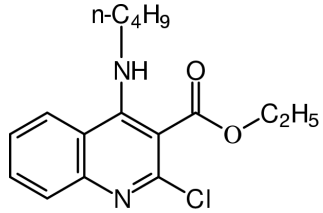
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 December 2000
Accepted 1 February 2001
Online 19 February 2001

The title molecule, $C_{16}H_{19}ClN_2O_2$, contains three planar fragments, *i.e.* the quinoline system (*A*), the butylamino group (*B*) and the carboxyl group (*C*). Angles *A*/*B* and *A*/*C* are 9.1 (2) and 79.08 (4)°, respectively. The alternation of the bond lengths in the quinoline bicycle correlates with literature data.

Comment

During investigation of the reaction of ethyl 2,4-dichloroquinoline-3-carboxylate with nucleophilic reagents, it was found that this compound reacts with sodium acetate giving ethyl chloro-2-oxo-1,2-dihydroquinoline-3-carboxylate, as identified in previous work (Ukrainets *et al.*, 1995). However, X-ray diffraction analysis has shown the product of this reaction to be ethyl 4-butylamino-2-chloroquinoline-3-carboxylate, (I), the structure of which is presented here.



(I)

The quinoline fragment of (I) (Fig. 1) is planar within 0.02 Å. The Cl atom lies in the plane of the quinoline bicycle. The carboxyl group is rotated by 79.08 (4)° with respect to the quinoline fragment [the C2—C3—C11—O1 and C2—C3—C11—O2 torsion angles are 95.7 (1) and −81.7 (1)°, respectively]. The non-H atoms of the butylamine fragment are coplanar within 0.03 Å and this plane is rotated by 9.1 (2)° with respect to the quinoline fragment. This rotation may be caused by steric repulsion between the H5 and H2 atoms, [the distance is 2.05 (2) Å and the sum of the van der Waals radii is 2.32 Å].

A noticeable alternation of the C—C (C—N) bonds is observed within the quinoline fragment, *i.e.* the N1—C2, C3—C4, C5—C6 and C7—C8 bonds are shorter and the remaining bonds are longer than the mean value for aromatic C—C bonds (1.387 Å; Bürgi & Dunitz, 1994). Such a feature is observed also for other quinoline-containing structures (Merlino, 1968; Blackburn *et al.*, 1996; Chiang *et al.*, 1991; Gdaniec *et al.*, 1980; Gdaniec & Dziembowska, 1980; Ahmet *et al.*, 1995).

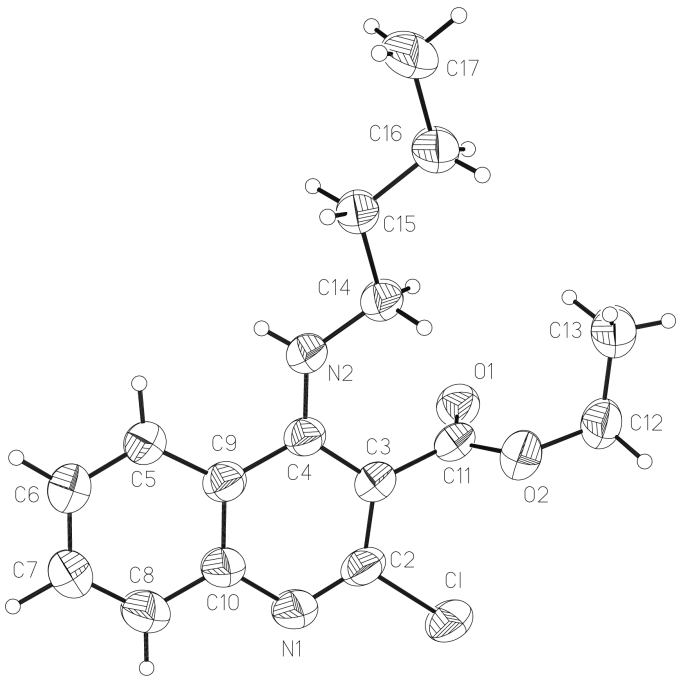


Figure 1
Numbering scheme and displacement ellipsoids for the title compound (50% probability level).

Table 1
Selected geometric parameters (Å, °).

Cl—C2	1.7818 (11)	C4—C9	1.4579 (15)
O1—C11	1.2245 (13)	C5—C6	1.3719 (18)
O2—C11	1.3425 (13)	C5—C9	1.4376 (15)
O2—C12	1.4712 (15)	C6—C7	1.4203 (19)
N1—C2	1.3057 (14)	C7—C8	1.3662 (18)
N1—C10	1.3914 (14)	C8—C10	1.4250 (17)
N2—C4	1.3715 (13)	C9—C10	1.4318 (15)
N2—C14	1.4638 (15)	C12—C13	1.517 (2)
C2—C3	1.4222 (14)	C14—C15	1.5321 (15)
C3—C4	1.4202 (14)	C15—C16	1.5322 (17)
C3—C11	1.5080 (15)	C16—C17	1.527 (2)
C11—O2—C12	117.34 (10)	C7—C8—C10	121.11 (12)
C2—N1—C10	116.14 (9)	C10—C9—C5	117.51 (10)
C4—N2—C14	127.57 (9)	C10—C9—C4	118.84 (9)
N1—C2—C3	128.43 (10)	C5—C9—C4	123.65 (9)
N1—C2—Cl	114.37 (8)	N1—C10—C8	117.95 (10)
C3—C2—Cl	117.13 (9)	N1—C10—C9	122.30 (10)
C4—C3—C2	116.56 (10)	C8—C10—C9	119.75 (10)
C4—C3—C11	124.01 (9)	O1—C11—O2	123.84 (10)
C2—C3—C11	118.59 (9)	O1—C11—C3	122.10 (10)
N2—C4—C3	123.84 (10)	O2—C11—C3	114.01 (9)
N2—C4—C9	118.58 (9)	O2—C12—C13	111.68 (11)
C3—C4—C9	117.57 (9)	N2—C14—C15	110.43 (9)
C6—C5—C9	120.92 (11)	C16—C15—C14	112.80 (9)
C5—C6—C7	120.99 (12)	C17—C16—C15	112.46 (12)
C8—C7—C6	119.68 (13)		

Experimental

Crystals of (I) suitable for X-ray study were grown *via* the slow evaporation of an alcohol solution.

Crystal data

$C_{16}H_{19}ClN_2O_2$
M_r = 306.78
Triclinic, $P\overline{1}$
a = 8.174 (2) Å
b = 8.760 (2) Å
c = 11.793 (2) Å
α = 75.68 (2)°
β = 85.77 (2)°
γ = 84.98 (2)°
V = 813.9 (3) Å^3
Z = 2
D_x = 1.252 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 34 reflections
θ = 13.0–15.0°
μ = 0.24 mm^{-1}
T = 290 (2) K
Elongated prism, colourless
0.45 × 0.40 × 0.25 mm

Data collection

Siemens *P3*/*PC* diffractometer
$2\theta/\theta$ scans
Absorption correction: by integration (*XPREP*; Siemens, 1991)
T_{min} = 0.887, T_{max} = 0.945
5630 measured reflections
5401 independent reflections
2629 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 32.5°
$h = -12 \rightarrow 12$
$k = -12 \rightarrow 13$
$l = 0 \rightarrow 16$
2 standard reflections
every 98 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.072
S = 0.91
5401 reflections
266 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0213P)^2]$ where P $F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.004
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

Data collection: *P*3 (Siemens, 1989); cell refinement: P3; data reduction: *XDISK* (Siemens, 1991); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1991); software used to prepare material for publication: *SHELXL*97.

References

Ahmet, M. T., Miller, J. R., Osborne, A. G. & Warmsley, J. F. (1995). *Acta Cryst.* C**51**, 2105–2108.
Blackburn, A. C., Dobson, A. J. & Gerkin, R. E. (1996). *Acta Cryst.* C**52**, 409–411.
Bürgi, H.-B. & Dunitz, J. D. (1994). Editors. *Structure Correlation*, Vol. 2, p. 771. Weinheim, New York, Basel, Cambridge, Tokyo: Verlag VCH.
Chiang, L. Y, Swirczewski, J. W., Kastrup, R., Hsu, C. S. & Upasani, R. B. (1991). *J. Am. Chem. Soc.* **113**, 6574–6579.
Gdaniec, M. & Dziembowska, T. (1980). *Pol. J. Chem.* **54**, 87–94.
Gdaniec, M., Jaskolski, M. & Kosturkiewicz, Z. (1980). *Pol. J. Chem.* **54**, 1539–1544.
Merlino, S. (1968). *Atti Accad. Naz. Lincei*, **45**, 60–65.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1989). *P*3. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
Siemens (1991). *XDISK*, *XPREP* and *XP*. Siemens Analytical X-ray Instruments Inc., Karlsruhe, Germany.
Ukrainets, I. V., Taran, S. G., Gorokhova, O. V., Marusenko, N. A., Kovalenko, S. N., Turov, A. V., Filimonova, N. I. & Ivkov, S. M. (1995). *Chem. Heterocycl. Compd* (*Engl. Transl.*), **31**, 2, 167–175.