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Key indicators

Single-crystal X-ray study
 T = 291 K
 Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
 R factor = 0.032
 wR factor = 0.069
 Data-to-parameter ratio = 24.2

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

Ethyl 5-bromo-9-ethoxy-2-oxo-2H-pyrano-[2,3-g]quinoline-8-carboxylate

The title compound, $\text{C}_{17}\text{H}_{14}\text{BrNO}_5$, was studied as part of a study on the biological properties of pyranoquinoline derivatives. Alkylation of the parent pyranoquinoline was shown to have occurred at the carbonyl rather than the amino site. The molecules are linked by a $\text{C}-\text{H} \cdots \text{O}$ hydrogen bond forming $C(5)$ chains along [001].

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Comment

As part of a study on the synthesis and biological activities of pyranoquinoline derivatives (da Matta *et al.*, 2000; de Oliveira, 2003), alkylation of compound (1) (see scheme) using EtBr was carried out. Compound (1) contains two potential reaction sites, *viz.* the carbonyl and amino sites. While NMR spectroscopy strongly indicated that bromination had occurred predominantly at the carbonyl site to give the pyranoquinoline derivative (2), confirmation was sought using X-ray crystallography.



Structural analysis confirmed that alkylation had indeed occurred at the carbonyl site (Fig. 1). The pyranoquinoline ring system in (2) was confirmed.

Hydrogen bonding occurs *via* $\text{C9}-\text{H9} \cdots \text{O11}^i$ [$\text{C9} \cdots \text{O11}^i = 3.367(4) \text{ \AA}$ and $\text{C9}-\text{H9} \cdots \text{O11}^i = 148^\circ$; symmetry code: (i)

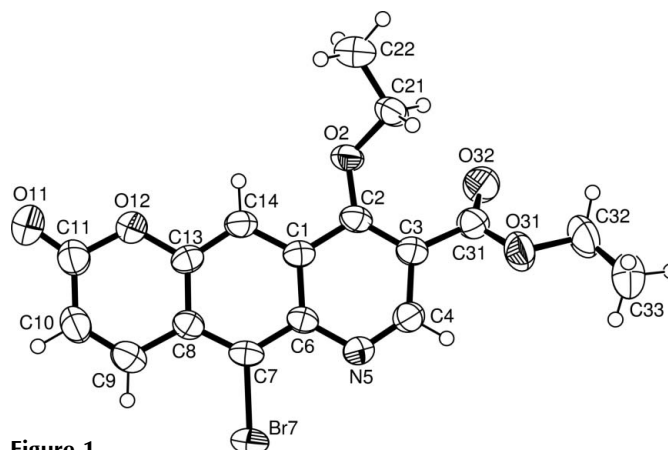


Figure 1

The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

$1 - x, -y, z - \frac{1}{2}$], leading to $C(5)$ chains (Bernstein *et al.*, 1995) along $[001]$ (Fig. 2).

The compound is isostructural with the chloro analogue (de Oliveira *et al.*, 2006).

Experimental

The title compound was obtained from the reaction between EtBr and (1) in DMF solution in the presence of K_2CO_3 (de Oliveira, 2003). Pure product (2) was obtained from the reaction mixture by column chromatography using hexane–ethyl acetate as the eluent (gradient 1:4 to 1:1). Crystals suitable for X-ray crystallography were grown from ethyl acetate (67% yield; m.p. 409–410 K).

Crystal data

$C_{17}H_{14}BrNO_5$

$M_r = 392.20$

Orthorhombic, $Pna2_1$

$a = 7.2542$ (8) Å

$b = 19.542$ (2) Å

$c = 11.2855$ (11) Å

$V = 1599.9$ (3) Å³

$Z = 4$

$D_x = 1.628$ Mg m⁻³

Mo $K\alpha$ radiation

Cell parameters from 4263

reflections

$\theta = 3.5$ – 25.3°

$\mu = 2.60$ mm⁻¹

$T = 291$ (2) K

Prism, pale yellow

$0.42 \times 0.22 \times 0.11$ mm

Data collection

Bruker SMART 1000 CCD area-detector diffractometer

ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2000)

$T_{\min} = 0.549$, $T_{\max} = 0.752$

15681 measured reflections

5296 independent reflections

2737 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.041$

$\theta_{\text{max}} = 32.5^\circ$

$h = -10 \rightarrow 10$

$k = -27 \rightarrow 29$

$l = -17 \rightarrow 14$

Refinement

Refinement on F^2

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.069$

$S = 0.80$

5296 reflections

219 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.034P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.35$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.29$ e Å⁻³

Absolute structure: Flack (1983)

Flack parameter: 0.013 (7)

All H atoms were located in difference maps and then treated as riding atoms, with C–H distances of 0.95 (aromatic) or 0.96 Å (methyl) and $U_{\text{iso}}(\text{H})$ values of $1.2U_{\text{eq}}(\text{C})$ (aromatic) or $1.5U_{\text{eq}}(\text{C})$ (methyl). *PLATON* (Spek, 2003) was used for the hydrogen-bonding analysis.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *OSCAIL* (McArdle, 2003) and *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *OSCAIL* and *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CIFTAB* (Sheldrick, 1997).

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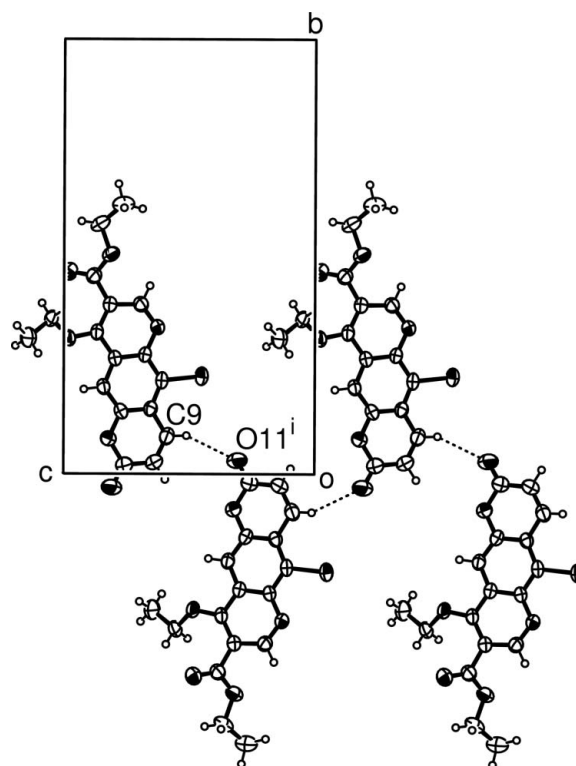


Figure 2

Part of the crystal structure of the title compound, showing the formation of a $C(5)$ chain (hydrogen bonds as dashed lines) along $[001]$. [Symmetry code: (i) $1 - x, -y, z - \frac{1}{2}$]

EPSRC's Chemical Database Service at Daresbury Laboratory (Fletcher *et al.*, 1996).

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