

2-Chloro-3-[4-(ethoxycarbonyl)-1-piperidyl]-1,4-naphthoquinone

Daniel E. Lynch,^{a*} Ian McClenaghan^{b†} and Simon J. Coles^c^aSchool of Natural and Environmental Sciences, Coventry University, Coventry CV1 5FB, England, ^bSpa Contract Synthesis, School of Natural and Environmental Sciences, Coventry University, Coventry CV1 5FB, England, and ^cDepartment of Chemistry, University of Southampton, Southampton SO17 1BJ, England

† E-mail: 106355.1670@compuserve.com.

Correspondence e-mail: apx106@coventry.ac.uk

Key indicators

Single-crystal X-ray study

T = 298 K

Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$

Disorder in main residue

R factor = 0.055

wR factor = 0.154

Data-to-parameter ratio = 11.8

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

The structure of the title compound, $\text{C}_{18}\text{H}_{18}\text{ClNO}_4$, (I), comprises twisted molecules that associate by an intermolecular $\text{C}-\text{H} \cdots \text{O}$ close contact to one of the naphthoquinone O atoms. The other naphthoquinone O atom and the Cl atom are involved in intramolecular $\text{C}-\text{H}$ close contacts, while no similar interactions are observed to the carbethoxy O atoms. The ethyl C atoms are disordered over two sites.

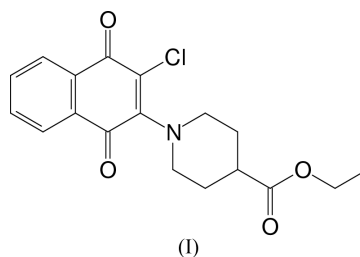
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Experimental

The title compound, (I), was prepared by Spa Contract Synthesis. Crystals of (I) were grown from a methanol solution.



Crystal data

 $\text{C}_{18}\text{H}_{18}\text{ClNO}_4$ $M_r = 347.78$ Monoclinic, $P2_1/c$ $a = 12.780 (3) \text{ \AA}$ $b = 16.931 (3) \text{ \AA}$ $c = 7.8532 (16) \text{ \AA}$ $\beta = 101.85 (3)^\circ$ $V = 1663.1 (6) \text{ \AA}^3$ $Z = 4$ $D_x = 1.389 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation

Cell parameters from 1591

reflections

 $\theta = 1.0\text{--}27.5^\circ$ $\mu = 0.25 \text{ mm}^{-1}$ $T = 298 (2) \text{ K}$

Needle, orange

 $0.45 \times 0.02 \times 0.01 \text{ mm}$

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer

 φ and ω scans

Absorption correction: multi-scan (SORTAV; Blessing, 1995)

 $T_{\min} = 0.895$, $T_{\max} = 0.998$

9954 measured reflections

2802 independent reflections

1040 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.118$ $\theta_{\max} = 25.0^\circ$ $h = -14 \rightarrow 15$ $k = -20 \rightarrow 19$ $l = -9 \rightarrow 9$

Refinement

Refinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.055$ $wR(F^2) = 0.154$ $S = 0.92$

2802 reflections

238 parameters

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0586P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.19 \text{ e \AA}^{-3}$ $\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$

Extinction correction: SHELXL97

Extinction coefficient: 0.0073 (15)

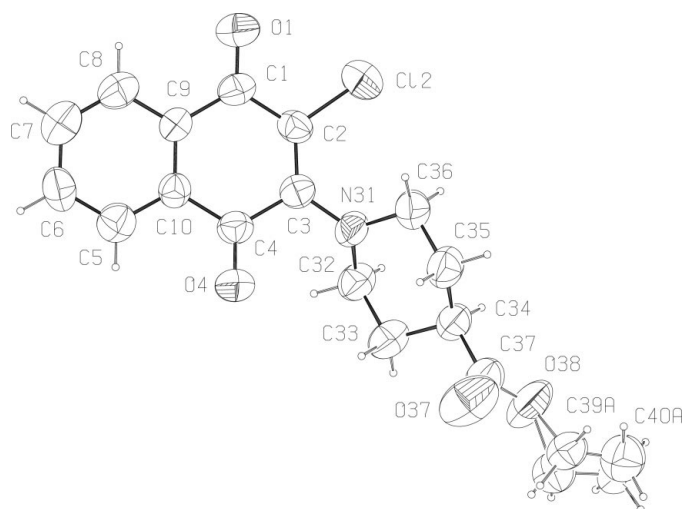


Figure 1
The molecular configuration and atom numbering scheme for (I), showing 50% probability ellipsoids. There is positional disorder of the ethyl group, C39 and C40.

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C8-H8 \cdots O1^i$	0.93	2.51	3.285 (6)	141
$C32-H322 \cdots O4$	0.97	2.31	2.852 (6)	115
$C36-H361 \cdots Cl2$	0.97	2.70	3.026 (5)	100

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

X-ray intensity data were measured with the maximum possible exposure time per frame (120 s) without overloading the detector due

to background scatter. In addition, due to the very small size of the crystal, the rotating anode generator was operated at full power and equipped with a capillary lined collimator in order to increase incident X-ray intensity at the crystal position. However, even with this configuration the number of observed data was low and the resultant data(observed)/parameter ratio is 4.75. The large R_{int} value (> 0.10) may be due to a slight misalignment of the crystal in the X-ray beam. There is a positional disorder of the ethyl C atoms. A split-site model was applicable and it was assumed that there are two possible sets of positions C39A/C40A and C39B/C40B with 50% probability each. The geometry of O38–C39A–C40A and O38–C39B–C40B was restrained to be similar by the *SAME* instruction, and the displacement parameters of C39 and C40 (both *A* and *B*) were restrained to be nearly isotropic by the *ISOR* instruction of *SHELXL97* (Sheldrick, 1997). All H atoms were included in the refinement at calculated positions as riding models with C–H set to 0.93 (Ar-H), 0.96 (CH₃), 0.97 Å (CH₂) and 0.98 Å (CH).

Data collection: *DENZO* (Otwinowski & Minor, 1997) and *COLLECT* (Hooft, 1998); cell refinement: *DENZO* and *COLLECT*; data reduction: *DENZO* and *COLLECT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

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