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2-Chloro-3-(3-dimethylaminopropylamino)-1,4-naphthoquinone

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.009 Å
Disorder in main residue
R factor = 0.067
wR factor = 0.176
Data-to-parameter ratio = 12.6

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

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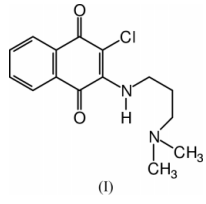
The structure of the title compound, $C_{15}H_{17}ClN_2O_2$, comprises essentially planar molecules slip-stacked along the *a* axis. The propyl chain has sufficient length to allow an intramolecular H-bond between the N—H and $N(CH_3)_2$ groups and the N—H also hydrogen bonds to the adjacent O atom, completing a three-centre association.

Comment

The title compound, (I), was prepared as part of a series of studies investigating the synthesis and structural properties of 2-substituted 3-chloro-1,4-naphthoquinones. Thus far, our structural investigations have concentrated on cyclic nucleophilic substituents such as morpholine (Lynch & McClenaghan, 2000*a*), pyrrolidine (Lynch & McClenaghan, 2000*b*), piperidine (Lynch & McClenaghan, 2001*a*), piperazine (Lynch & McClenaghan, 2001*b*). However, we report here the structure of an *N*-alkyl analogue whose crystallographic parameters typify the difficulties encountered with this series of molecules (see refinement details in *Experimental*).



(I)

The structure of (I) (Fig. 1) comprises essentially planar molecules slip-stacked along the *a* axis. The propyl chain has sufficient length to allow an intramolecular hydrogen bond between the N—H and $N(CH_3)_2$ groups and the N—H also hydrogen bonds to the adjacent O atom (Table 1), completing

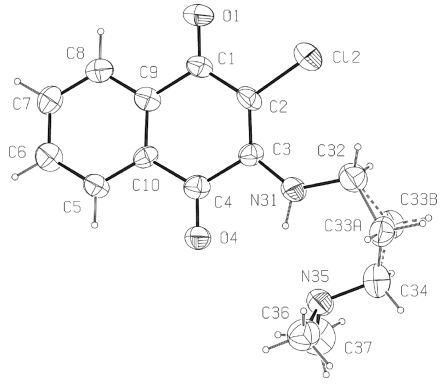


Figure 1
The molecular configuration and atom-numbering scheme for the title compound, showing 50% probability ellipsoids.

a three-centre association. No other intermolecular C—H···O/Cl close contacts are observed. One C atom in the propyl chain (C33) is unequally disordered [0.79/0.21 (2)] over two sites. This disorder may also extend to C34, which has an increased U_{11} of 0.111 (7) Å^2, but not enough to be split.

Experimental

The title compound was obtained from Key Organics Ltd and crystals were grown from an ethanol solution.

Crystal data

$C_{15}H_{17}ClN_2O_2$
M_r = 292.76
Monoclinic, $P2_1/c$
a = 8.0693 (8) Å
b = 13.002 (1) Å
c = 13.931 (2) Å
β = 105.623 (4)°
V = 1407.6 (3) Å^3
Z = 4
D_x = 1.381 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4100 reflections
θ = 2.9–27.5°
μ = 0.27 mm^{-1}
T = 150 (2) K
Needle, red
0.20 × 0.03 × 0.02 mm

Data collection

Bruker–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.947, T_{max} = 0.995
9261 measured reflections
2439 independent reflections
777 reflections with $I > 2\sigma(I)$
R_{int} = 0.277
θ_{max} = 25.0°
$h = -9 \rightarrow 9$
$k = -15 \rightarrow 15$
$l = -15 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.176
S = 0.88
2439 reflections
193 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N31—H31···O4	0.90 (5)	2.13 (5)	2.593 (8)	111 (4)
N31—H31···N35	0.90 (5)	2.01 (5)	2.783 (8)	143 (5)

Crystals in this series of compounds, including those not suitable for data collection, are thin plates with weak high angle data and high R_{int} values have occurred. The crystals of the title compound were extremely thin needles that resulted not only in weak high angle data but also in overall weak data, with the ratio of observed/unique reflections being *ca* 0.32. Hence, R_{int} is very high (0.28) and the data/parameters ratio is low (*ca* 4). In the previous structures, the number of observed data was sufficiently high to generate data/parameters ratios well above 6. All H atoms were included in the refinement at calculated positions, as riding models with C—H set to 0.95 (Ar—H), 0.98 (CH_3) and 0.99 Å (CH_2). The isotropic displacement parameters were set equal to 1.25(U_{eq}) of the carrier atom except for N—H. The latter was located in a difference synthesis, and both the positional and displacement parameters were refined.

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: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON*97 (Spek, 1997); software used to prepare material for publication: *SHELXL*97.

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