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

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Key indicators

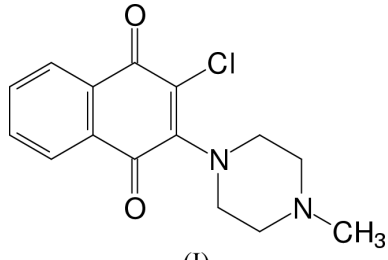
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.116
Data-to-parameter ratio = 16.9

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

In the title molecule, $C_{15}H_{15}ClN_2O_2$, the piperazine ring adopts a chair conformation and the mean plane through that ring forms a dihedral angle of 43.66 (6)° with the planar naphthoquinone moiety. The structure contains two intramolecular C—H···X short contacts to the Cl atom and one of the O atoms, and one intermolecular short contact to the same O atom.

Comment

The title compound, (I), is a derivative of 2,3-dichloro-1,4-naphthoquinone (Dichlone; Metras, 1961; Ikemoto *et al.*, 1977). In Dichlone, both Cl atoms can equally be displaced by nucleophiles, but once one Cl is replaced then the substituent deactivates the second Cl, thus additional replacement by a second nucleophile is much slower. Dichlone and its derivatives display both herbicidal and pesticidal activity (Merck Index, 1996), for which reasons we have instigated a programme of research to investigate the biological properties of a range of new Dichlone derivatives. A search of the Cambridge Structural Database (Allen & Kennard, 1993; Fletcher *et al.*, 1996) indicated that there are nine previously reported monosubstituted derivatives of Dichlone, of which six were prepared by nucleophilic replacement of one Cl atom. We report here the single-crystal structure of the 4-methylpiperazino analogue, (I), which is in addition to our recently reported structures of the morpholino (Lynch & McClenaghan, 2000*a*), pyrrolidin-1-yl (Lynch & McClenaghan, 2000*b*) and 4-(ethoxycarbonyl)-1-piperidyl (Lynch & McClenaghan, 2001) derivatives of Dichlone.



(I)

Experimental

The title compound, (I), was prepared by Spa Contract Synthesis. Crystals of (I) formed when the reaction solution *N*-methylpyrrolidone was poured into excess water.

Crystal data

$C_{15}H_{15}ClN_2O_2$
M_r = 290.74
Monoclinic, $P2_1/n$
a = 12.379 (3) Å
b = 9.389 (2) Å
c = 12.995 (3) Å
β = 116.60 (3)°
V = 1350.6 (5) Å^3
Z = 4
D_x = 1.430 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7161 reflections
θ = 2.9–27.5°
μ = 0.29 mm^{-1}
T = 150 (2) K
Prism, red
0.20 × 0.14 × 0.14 mm

Data collection

Enraf–Nonius KappaCCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)
T_{min} = 0.945, T_{max} = 0.961
11 054 measured reflections
3067 independent reflections
2322 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 27.5°
$h = -15 \rightarrow 14$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.116
S = 1.03
3067 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0685P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C32—H321···O4^{i}	0.99	2.50	3.369 (2)	146
C32—H322···Cl2	0.99	2.69	3.038 (2)	101
C36—H361···O4	0.99	2.30	2.851 (2)	114

Symmetry code: (i) $\frac{5}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$.

All H atoms were included in the refinement at calculated positions as riding models, with C—H distances of 0.95 (aryl H), 0.98 Å (CH_3) and 0.99 Å (CH_2).

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.

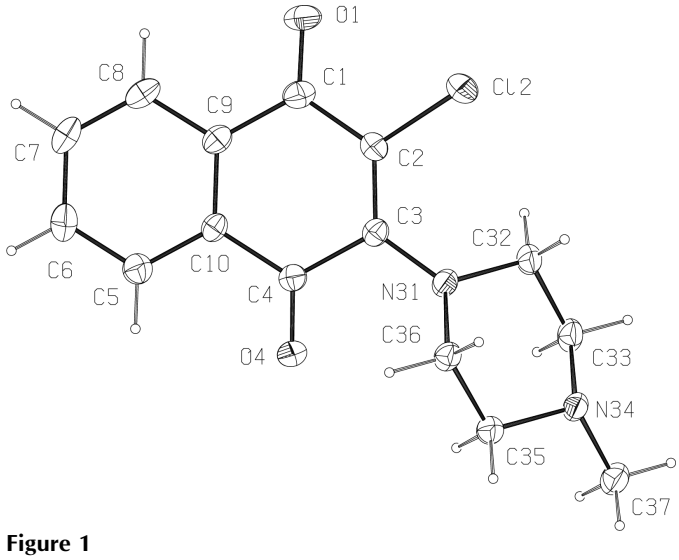


Figure 1
The molecular configuration and atom-numbering scheme for (I). Displacement ellipsoids are shown at the 50% probability level.

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