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Disorder in ondansetron hydrochloride dihydrate

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.002 Å
Disorder in main residue
R factor = 0.033
wR factor = 0.087
Data-to-parameter ratio = 15.7

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

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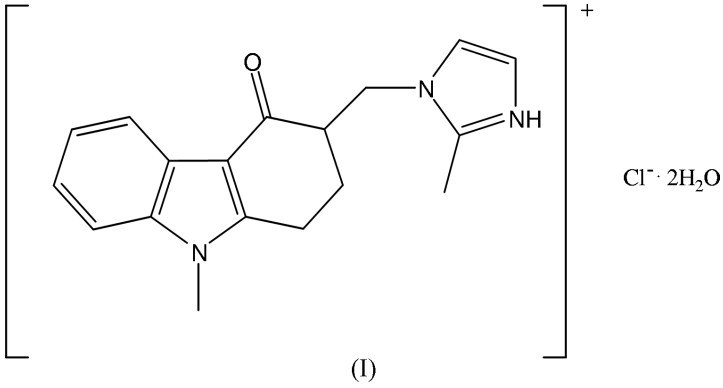
The structure of the title compound, 2-methyl-1-(1,2,3,9-tetrahydro-9-methyl-4-oxo-4*H*-carbazol-3-ylmethyl)-1*H*-imidazol-3-ium chloride dihydrate, $C_{18}H_{24}N_3^+\cdot Cl^-\cdot 2H_2O$, has been reported previously by Collin, Moureau, Quintero, Vercauteren, Evrard & Durant [(1995). *J. Chem. Soc. Perkin Trans.* 2, pp. 77–84] and Chandra Mohan & Ravikumar, [(1995), *Acta Cryst.* C**51**, 2627–2629]. In both determinations, all atoms were refined as clearly ordered. In contrast to this, we present here a redetermination of this structure from new intensity data where two atoms of the cyclohexenone ring are disordered over two sites. Apart from this disorder, our results agree with the already published data.

Comment

The title compound, (I), is an anti-emetic, which has been described by Milne & Heel (1991). It is a highly selective 5-HT3 receptor antagonist (Ye *et al.*, 2001). A perspective view is shown in Fig. 1.



(I)

The structure of (I) has already been determined twice at room temperature by two different research groups (Collin *et al.*, 1995; Chandra Mohan & Ravikumar, 1995). Both of these structures were refined as perfectly ordered. However, we have collected data at low temperature and discovered that two atoms of the cyclohexenone ring are disordered over two sites. In order to check if the crystal had undergone a phase transition upon cooling, a data set was collected on a different crystal at room temperature. This crystal showed the same disorder as the crystal investigated at low temperature. As a result, a phase transition can be ruled out. A closer look at both published ordered structures shows that the displacement ellipsoids of the atoms in question (C2 and C3 in the present structure) are elongated, which suggest disorder. Furthermore, the bond length between these atoms is significantly shortened in the two published structures (1.479 and

1.485 Å) compared with the value [1.536 (3) Å] we found in the major component. This indicates an average of two positions for the two atoms. As a result, the previous structures seem to be disordered as well, but were not refined as such. Apart from this disorder, our results agree with the already published data. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 1.6 plus three updates; *Mogul* Version 1.0; Allen, 2002).

Experimental

The sample of the title compound was obtained as a gift from CIPLA, India. It was used without further purification and recrystallized from methanol to give colourless blocks.

Crystal data

$C_{18}H_{20}N_3O^+\cdot Cl^-\cdot 2H_2O$
M_r = 365.85
Monoclinic, $P2_1/c$
a = 15.0216 (13) Å
b = 9.6464 (8) Å
c = 12.6545 (10) Å
β = 100.708 (6)°
V = 1801.8 (3) Å^3
Z = 4
D_x = 1.349 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 20705 reflections
θ = 3.7–27.6°
μ = 0.23 mm^{-1}
T = 173 (2) K
Block, colourless
0.33 × 0.27 × 0.14 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 1990; Blessing, 1995)
T_{min} = 0.928, T_{max} = 0.967
24157 measured reflections
4181 independent reflections
3172 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 27.7°
$h = -19 \rightarrow 19$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.087
S = 0.96
4181 reflections
267 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0567P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N23—H23···O1W	0.92 (2)	1.75 (2)	2.6663 (16)	178.0 (19)
O1W—H1WB···Cl1	0.82 (2)	2.32 (2)	3.1252 (14)	166 (2)
O2W—H2WB···Cl1	0.89 (3)	2.33 (3)	3.2171 (15)	175 (2)
O1W—H1WA···Cl1^{i}	0.85 (3)	2.32 (3)	3.1334 (14)	161 (2)
O2W—H2WA···Cl1^{ii}	0.82 (3)	2.41 (3)	3.2205 (15)	172 (2)

Symmetry codes: (i) $-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $-x, 1-y, -z$.

The site-occupation factors of the disordered atoms refined to 0.791 (6) and 0.209 (6). In the second crystal examined at room temperature, the ratio of the site-occupation factors refined to 0.759 (9)/0.241 (9). H atoms were refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl})] using a riding model, with C—H = 1.00 Å, C—H = 0.99 Å, C—H = 0.98 and 0.95 Å, for tertiary CH, secondary CH, methyl CH and aromatic CH, respectively. The methyl groups were allowed to rotate but not to tip. H atoms bonded to O and N atoms were refined isotropically.

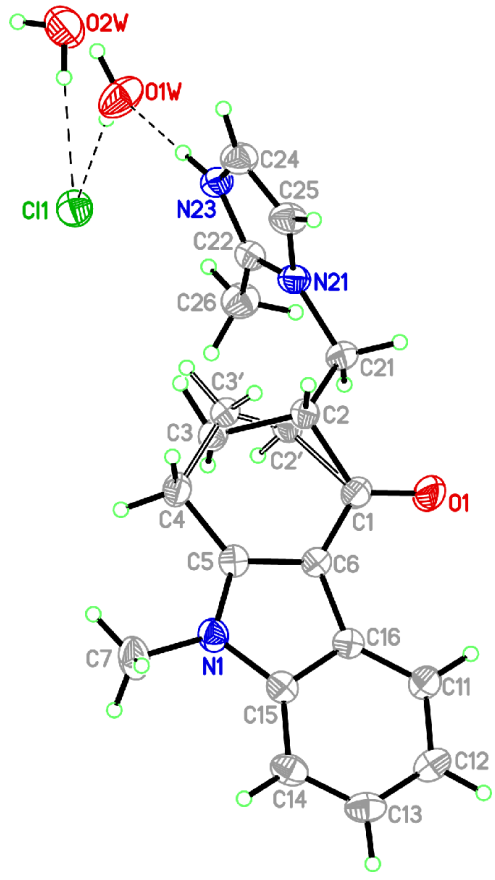


Figure 1
Perspective view of the title compound, with the atom numbering. Displacement ellipsoids are drawn at the 50% probability level.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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