

A monoclinic polymorph of isoxsuprine hydrochloride

Hemmige S. Yathirajan,^a
 Basavegowda Nagaraj,^a
 Rajenahally S. Narasegowda,^a
 Padmarajaiah Nagaraja^a and
 Michael Bolte^{b*}

^aDepartment of Studies in Chemistry, University of Mysore, Manasagangotri, Mysore 570 006, India, and ^bInstitut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail:
 bolte@chemie.uni-frankfurt.de

Key indicators

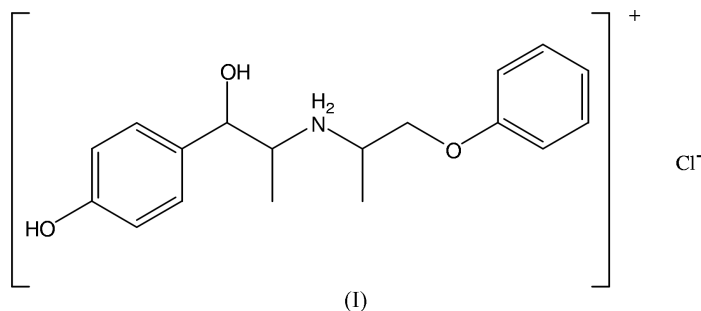
Single-crystal X-ray study
 $T = 173\text{ K}$
 Mean $\sigma(\text{C}—\text{C}) = 0.002\text{ Å}$
 R factor = 0.033
 wR factor = 0.095
 Data-to-parameter ratio = 17.9

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

A monoclinic polymorph of the title compound [systematic name: 2-hydroxy-2-(4-methoxyphenyl)-1-methyl-*N*-(1-phenoxy-2-propyl)ethanaminium chloride], $\text{C}_{18}\text{H}_{24}\text{N}_2\text{O}_3^+\cdot\text{Cl}^-$, has been found in addition to the already known triclinic polymorph. The molecular conformation in both polymorphs is very similar.

Comment

The title compound, (I), is used as a vasodilator and in the treatment of navicular disease. A triclinic polymorph, (II), has already been described (Léger *et al.*, 1981). It crystallizes in $P\bar{1}$ with two molecules in the asymmetric unit. We present here a monoclinic polymorph in $P2_1/c$ with $Z = 4$. A perspective view of (I) is shown in Fig. 1.



The cells of the two polymorphs have nothing in common, except the volume. The unit-cell axes and interaxial angles are completely different. The molecular conformations, on the other hand, are essentially the same. A least-squares fit of (I) with (II) gives r.m.s. deviations of 0.228 and 0.200 Å for the two molecules in the asymmetric unit of (II) (Fig. 2).

The crystal packing is stabilized by several $\text{N}—\text{H}\cdots\text{Cl}$ and $\text{O}—\text{H}\cdots\text{Cl}$ hydrogen bonds (Table 1).

Experimental

The sample of the title compound was obtained as a gift from Jayco Chemical Industries, India. It was used without further purification and recrystallized from methanol.

Crystal data

$\text{C}_{18}\text{H}_{24}\text{NO}_3^+\cdot\text{Cl}^-$
 $M_r = 337.83$
 Monoclinic, $P2_1/c$
 $a = 12.0767\text{ (11) Å}$
 $b = 19.3499\text{ (12) Å}$
 $c = 7.8018\text{ (7) Å}$
 $\beta = 107.482\text{ (7)°}$
 $V = 1738.9\text{ (3) Å}^3$
 $Z = 4$

$D_x = 1.290\text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 24 065 reflections
 $\theta = 3.7\text{--}27.6^\circ$
 $\mu = 0.23\text{ mm}^{-1}$
 $T = 173\text{ (2) K}$
 Block, colourless
 $0.44 \times 0.39 \times 0.35\text{ mm}$

Data collection

Stoe IPDS-II two-circle
diffractometer
 ω scans
Absorption correction: multi-scan
(*MULABS*; Spek, 2003; Blessing,
1995)
 $T_{\min} = 0.904$, $T_{\max} = 0.923$
21 164 measured reflections

4008 independent reflections
3499 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$
 $\theta_{\max} = 27.7^\circ$
 $h = -15 \rightarrow 15$
 $k = -25 \rightarrow 25$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.095$
 $S = 1.07$
4008 reflections
224 parameters
H atoms treated by a mixture of
independent and constrained
refinement

$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.1915P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.26 \text{ e } \text{\AA}^{-3}$

Table 1

Hydrogen-bonding geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O1-H1\cdots Cl1^i$	0.78 (2)	2.36 (2)	3.1285 (11)	168.3 (18)
$N3-H3A\cdots Cl1^{ii}$	0.895 (16)	2.529 (16)	3.3726 (10)	157.2 (13)
$N3-H3B\cdots Cl1$	0.844 (17)	2.344 (17)	3.1852 (11)	174.3 (14)
$O14-H14\cdots Cl1^{iii}$	0.84 (3)	2.33 (3)	3.1730 (11)	178 (2)

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

H atoms bonded to carbon were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$] using a riding model, with $\text{C}-\text{H} = 1.00, 0.99, 0.98$ and 0.95 \AA for tertiary CH, secondary CH, methyl and aromatic CH, respectively. H atoms bonded to nitrogen and oxygen were refined freely.

Data collection: *X-Area* (Stoe & Cie, 2001); cell refinement: *X-Area*; data reduction: *X-Area*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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References

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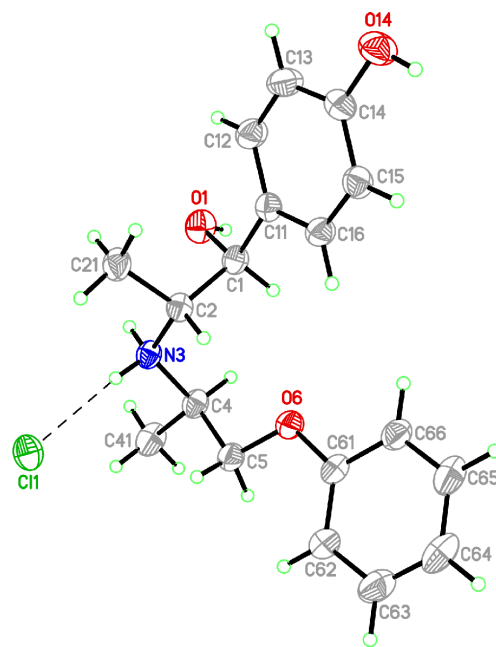


Figure 1

Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level. The dashed line indicates a hydrogen bond.

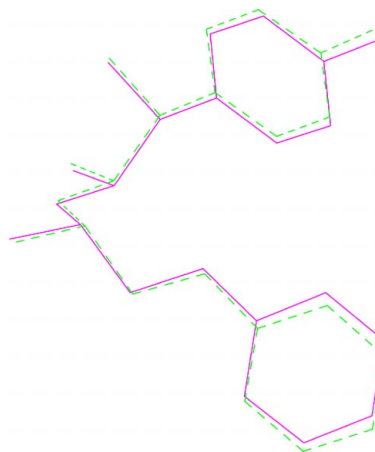


Figure 2

Least-squares fit of (I) (dashed line) with one of the two molecules (solid line) in the asymmetric unit of (II).

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