

2-(Imidazol-1-yl)-1-(2-naphthyl)ethanone O-propyloxime hydrochloride

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Key indicators

Single-crystal X-ray study

$T = 295\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$

R factor = 0.032

wR factor = 0.090

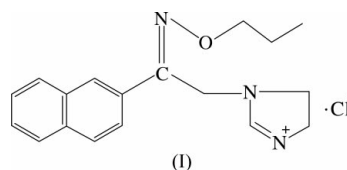
Data-to-parameter ratio = 13.0

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 {systematic name: 1-[2-(2-naphthyl)-2-(propoxyimino)ethyl]-4,5-dihydroimidazol-3-ium chloride}, $\text{C}_{18}\text{H}_{20}\text{N}_3\text{O}^+\cdot\text{Cl}^-$, is stabilized by intermolecular hydrogen bonds. The naphthalene moiety and the imidazole ring are essentially planar. The imidazole N2 atom is protonated and is connected to the Cl^- anion through a hydrogen bond [$\text{Cl}\cdots\text{N}2 = 3.004(2)\text{ \AA}$]. The configuration of the cation is *Z*.

Comment

Oximes and oxime ethers show geometric isomerism because of the double bond between the N and the O atoms in their structure. Depending on the reaction and the reaction conditions, *E*, *Z* or a mixture of *E/Z* isomers in different proportions may be obtained. Reaction of alkyl halides or alkyl sulfates with oximes is one of the widely used methods to prepare oxime ethers, and the configuration of the oxime ethers prepared in this way is usually the same as the configuration of the oxime (March, 1992; Balsamo *et al.*, 1990).



In this study, the title compound, (I), was prepared as a potential anticonvulsant and antimicrobial compound, by *O*-alkylation of nafimidone oxime (*Z*), which was synthesized by the reaction of nafimidone and hydroxylamine hydrochloride (Karakurt *et al.*, 2001; Kendi *et al.*, 1998). The structural elucidation of (I) was achieved by IR, ^1H NMR, MS data and elemental analysis. The compound exhibits both anticonvulsant and antimicrobial activities

As shown in Fig. 1, the present compound is a salt. The configuration of the cation is *Z*. This result is in accordance with the literature, in that oxime ethers obtained by alkylation of oximes with alkyl halides are generally expected to have the same configuration as the oximes (Balsamo *et al.*, 1990). The X-ray crystal structure of nafimidone oxime, which was obtained as a single isomer in high yield, showed that this compound was in the *Z* configuration (Kendi *et al.*, 1998). As expected, the naphthalene moiety and the imidazole ring, are essentially planar [maximum deviations $-0.012(2)\text{ \AA}$ for C7 and $-0.004(2)\text{ \AA}$ for C15]. The dihedral angle between these two planes is $66.91(6)^\circ$. The C–N bond distances in the imidazole ring are not the expected single- and double-bond lengths [$\text{N}2-\text{C}13\ 1.312(2)\text{ \AA}$ and $\text{N}1-\text{C}13\ 1.325(2)\text{ \AA}$], as

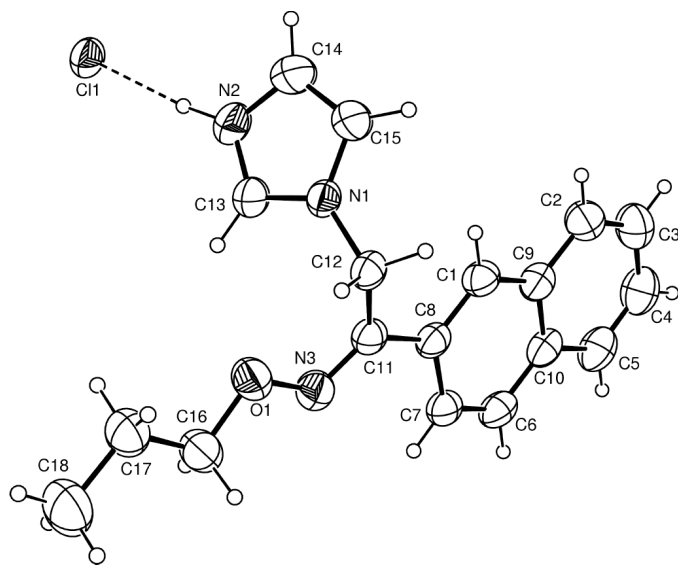


Figure 1

ORTEP (Johnson, 1965) drawing of the asymmetric unit of the title compound, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

reported in other imidazole oxime derivatives (Karakurt *et al.*, 2001; Kendi *et al.*, 1998; Grassi *et al.*, 1993). The positive charge on imidazole atom N2 results from proton transfer from the HCl. A strong hydrogen bond exists between the imidazole NH⁺ group and the Cl⁻ anion and, therefore, the C–N bonds mentioned above have intermediate values between the expected single- and double-bond lengths.

The orientation of the naphthalene system is defined by the torsion angles C8–C11–C12–N1 [91.7 (2)°] for the imidazole ring and C1–C8–C11–N3 [164.6 (1)°] for the oxime. These angles are 91.5 (3) and 162.8 (2)° in 1-(2-naphthyl)-2-(imidazole-1-yl)ethanone *O*-(2-propen)oxime hydrochloride (Karakurt *et al.*, 2001); the oxime in 2-(imidazol-1-yl)-1-(2-naphthyl)ethanone oxime (Kendi *et al.*, 1998) is twisted out of the naphthalene plane by 36.2 (1)°.

The crystal structure of (I) is stabilized by intermolecular hydrogen bonds (Table 2).

Experimental

Nafimidone oxime (0.01 mol) was added to a solution of sodium ethoxide prepared from sodium (0.011 mol) and anhydrous ethanol (25 ml), and the resulting solution was refluxed for 30 min. After ethanol was evaporated under reduced pressure, the residue was dissolved in anhydrous dimethylformamide (DMF, 20 ml) and propyl bromide (0.01 mol) was added. The mixture was refluxed for 3 h at 343 K, then the DMF was evaporated to dryness. The oily residue was dissolved in dry benzene and treated with HCl gas to obtain the hydrochloride salt. The residue was recrystallized from methanol/ethyl acetate. Yield = 84%, m.p. = 443–445 K. UV λ_{\max} (log ϵ): 297.2 (4.14), 286.0 (4.17), 243.6 (4.47), 212.6 (4.38) nm. IR: 3147 (aromatic

C–H stretching), 2962, 2880 (aliphatic C–H stretching), 2412 (N⁺–H stretching), 1032 (C–O deformation), 934 (N–O deformation), 871, 830, 761 (naphthalene C–H deformation) cm⁻¹. ¹H NMR (CDCl₃-*d*, p.p.m.): *d* 1.00 (*t*; 3H; –CH₃), 1.8 (*m*; 2H; CH₂–CH₃), 4.35 (*t*; 2H; CH₂–O), 5.70 (*s*; 2H; CH₂–N), 7.10–8.10 (*m*; 8H; naphthalene H3–8 and imidazole H4, H5), 8.40 (*s*; 1H; naphthalene H1), 9.60 (*s*; 1H; imidazole H2). Mass spectrum (EI, 70 eV): *m/e* 293 (78%, *M*⁺), 262, 234, 207, 195, 170, 153 (100%, base peak), 127, 81, 69 and 43. Elemental analysis, C₁₈H₂₀ClN₃O·0.5H₂O (338.84): calculated C 63.81, H 6.25, N 12.40%; found: C 63.33%, H 5.77%, N 12.21%.

Crystal data

C₁₈H₂₀N₃O⁺·Cl⁻
M_r = 329.82
 Triclinic, *P* $\bar{1}$
a = 8.6832 (16) Å
b = 9.5662 (13) Å
c = 11.3595 (16) Å
 α = 104.967 (11)°
 β = 107.547 (13)°
 γ = 94.120 (14)°
V = 857.6 (2) Å³

Z = 2
D_x = 1.277 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 25 reflections
 θ = 10.3–18.1°
 μ = 0.23 mm⁻¹
T = 295 (2) K
 Prism, colorless
 0.46 × 0.42 × 0.24 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 Non-profiled ω scans
 Absorption correction: ψ scan (North *et al.* 1968)
*T*_{min} = 0.899, *T*_{max} = 0.946
 3444 measured reflections
 3216 independent reflections
 2561 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.007
 θ_{\max} = 26.2°
h = 0 → 10
k = –11 → 11
l = –13 → 13
 3 standard reflections
 frequency: 120 min
 intensity decay: –1%

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.032
wR (*F*²) = 0.090
S = 1.02
 3216 reflections
 248 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0448P)^2 + 0.1717P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.24 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.15 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

O1–N3	1.4040 (17)	N1–C12	1.4749 (19)
O1–C16	1.436 (2)	N2–C13	1.312 (2)
C8–C11	1.476 (2)	N2–C14	1.357 (2)
C11–N3	1.2872 (19)	N2–H1'	1.00 (2)
C11–C12	1.510 (2)	C15–C14	1.341 (3)
N1–C13	1.3249 (19)	C16–C17	1.496 (3)
N1–C15	1.365 (2)	C17–C18	1.515 (3)
N3–O1–C16	109.95 (12)	N2–C13–N1	109.10 (15)
N3–C11–C8	116.87 (13)	N1–C12–C11	112.42 (12)
C8–C11–C12	120.37 (13)	C14–C15–N1	106.93 (16)
C13–N1–C15	108.00 (14)	O1–C16–C17	106.55 (15)
C13–N2–C14	108.40 (15)	C15–C14–N2	107.56 (17)
C13–N2–H1'	126.8 (13)	C16–C17–C18	113.02 (19)
C11–N3–O1	111.29 (12)		

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N2-H1' \cdots Cl1$	1.00 (2)	2.02 (3)	3.004 (2)	172.5 (17)
$C12-H12A \cdots Cl1^i$	0.99 (2)	2.79 (2)	3.748 (2)	163.3 (13)
$C15-H15 \cdots Cl1^i$	1.00 (3)	2.69 (3)	3.623 (2)	155.5 (16)

Symmetry code: (i) $1+x, y, z$.

The H atoms attached to C12, C15, C16, C17 and N2 and were located from a difference map and refined freely. The other H atoms were positioned geometrically at distances of 0.93 and 0.97 Å for aromatic and methyl H atoms, respectively, and refined riding on their parent atoms.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP* (Johnson, 1965); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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2-(Imidazol-1-yl)-1-(2-naphthyl)ethanone O-propyloxime hydrochloride. Erratum

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In the paper by Özbey *et al.* [*Acta Cryst.* (2002), E58, o1269–o1271], the *Scheme* and systematic name are incorrect. The correct *Scheme* is shown below and the correct systematic name is 1-[2-(2-naphthyl)-2-(propoxyimino)ethyl]imidazol-3-ium chloride.

