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Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1253). Services for accessing these data are described at the back of the journal.

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2-(Imidazol-1-yl)-1-(2-naphthyl)ethanone Oxime

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Abstract

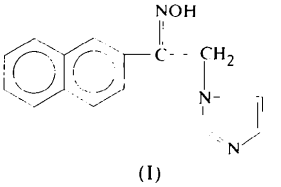
In the title compound, imidazol-1-ylmethyl 2-naphthyl ketone oxime, $C_{15}H_{13}N_3O$, the naphthalene and imidazole rings are essentially planar. The oxime group is twisted by 36.2 (1)° out of the naphthalene plane. The oxime configuration is *Z*. The structure is stabilized by intra- and intermolecular hydrogen bonds.

Comment

Oximes show geometric isomerism due to the double bond between the N and C atoms. The reaction of hydroxylamine hydrochloride with an unsymmetrical ketone may result in either a mixture of two isomeric oximes or only one of the isomers, depending on the structure of the ketone or the reaction conditions (Mixich & Thiele, 1979; Migrdichian, 1957). Because of the great differences in physical, chemical and biological properties of the geometric isomers, determination of the configuration of the isomers is important (Mathison *et al.*, 1989).

Oximes and oxime ethers have a broad pharmacological activity spectrum, encompassing antifungal, antibacterial, antidepressant and insecticidal activities, as well as activity as a nerve-gas antidote, depending on the pharmacophoric group of the molecule (Polak, 1982; Balsamo *et al.*, 1990; Holan *et al.*, 1984; Forman, 1964). An oximino group usually modifies the activity or sometimes is directly responsible for the activity.

In connection with our interest in the anticonvulsant compound nafimidone and antifungal–antibacterial agents with (arylalkyl)azole structures, we have prepared nafimidone oxime (Walker *et al.*, 1981). Since the structure of this oxime is important with respect to the activity and configuration of the O-ether derivatives of this compound that have been prepared in our laboratory, we studied its spectral properties and molecular geometry by UV, IR, ^{1}H NMR, mass spectroscopy, elemental analysis and X-ray crystallography. We report here the structure of nafimidone oxime, (I).



(I)

The naphthalene moiety is essentially planar, with bond lengths and angles in good agreement with those observed in other naphthalene derivatives (Elmalı *et al.*, 1995; Irngartinger *et al.*, 1993). The imidazole ring is also planar [$\Sigma(\Delta/\sigma)^2$ = 1.6]. The dihedral angle between these two planes is 96.98 (8)°. Some significant differences are observed for the bond distances in the imidazole ring compared with the averages derived from the Cambridge Structural Database quoted by Allen *et al.* (1987) [given in square brackets]: N1—C13 1.360 (3) [1.349 (18)], N1—C15 1.335 (2) [1.370 (10)], N2—C14 1.364 (3) [1.376 (11)], N2—C15 1.305 (3) [1.313 (11)] and C13—C14 1.344 (3) Å [1.360 (14) Å]. In two other imidazole oxime derivatives, all the C—N bond distances in the imidazole ring are intermediate between the expected single- and double-bond lengths (Grassi *et al.*, 1993; Bruno *et al.*, 1994). The exocyclic angles around the N1 atom show considerable asymmetry. However,

the sum of the valence angles around N1 is 359.7°, indicating no significant pyramidalization of this atom. The same planar bond configuration at this N atom has been found in (*Z*)-1-benzoyl-5-benzyl-4-methyl-2-phenylimidazole oxime (Bruno *et al.*, 1994) and (*Z*)-3-benzoylimidazol-2-one oxime–(*Z*)-3-*p*-toluoylimidazol-2-one oxime (Grassi *et al.*, 1993). The oxime group is twisted by 36.2 (1)° out of the plane of the naphthalene moiety; conjugation between the oxime group and the naphthalene ring is thus sterically hindered. The oxime configuration is *Z*. The C11—C12 [1.504 (2) Å] and N1—C12 bonds [1.460 (3) Å] are found to have normal single-bond lengths.

In the final difference Fourier map, we found a residual charge of 0.461 e Å^{-3} located at 1.021 Å from atom N3. Since the N3—C11 length [1.287 (2) Å] has the normal C_{sp^2}=N value (1.28 Å), this peak should not be an H atom attached to N3, even though its location could suggest this. In *syn*- and *anti*-*p*-chlorobenzaldoxime (Jersley, 1957), it could not be decided whether the intermolecular O···N hydrogen bonds are of the O—H···N or N—H···O type. In our case, there is no doubt that an H atom is bound to O1 and that it contributes to an O1—H1···N2^{i} intermolecular hydrogen bond (geometric details of the hydrogen bonds are given in Table 2).

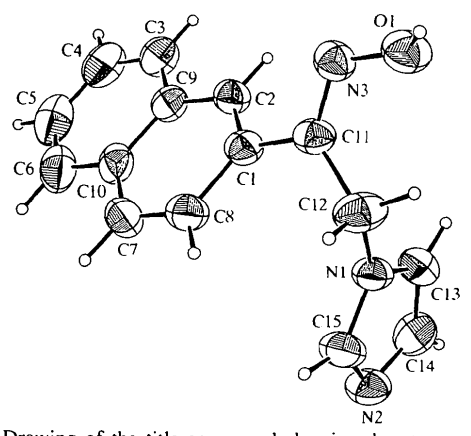


Fig. 1. Drawing of the title compound showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles with arbitrary radii.

Experimental

Nafimidone [2-(imidazol-1-yl)-1-(2-naphthyl)ethanone hydrochloride] (0.03 mol) and hydroxylamine hydrochloride (0.06 mol) were dissolved in ethanol (75 ml) and the pH of the solution was adjusted to 11 using a 15 *N* sodium hydroxide solution. After the solution was refluxed for 3 h, the solvent was evaporated. The residue was dissolved in water and acidified with hydrochloric acid to pH 5. The filtered precipitate was recrystallized from methanol (yield 82%; m.p. 466–469 K). UV λ_{max} (nm) (logε): 283.4 (3.76), 240.4 (4.18); IR (cm^{-1}) (KBr): 3124 (aromatic C—H stretch), 2607 (oxime O—H stretch), 931 (N—O deformation), 864, 822, 755 (naphthalene C—H deformation); ^{1}H NMR (p.p.m.) (DMSO-d_6): δ 5.50 (2H, *s*, CH_2), 6.80–8.30 (10H, *m*, aromatic protons), 12.15 (1H, *s*, OH); MS (EI; 70 eV) *m/e*: 235 (base peak 100%), 207, 195, 180, 154, 139, 127, 109, 82, 54 and 41; elemental analysis ($C_{15}H_{13}N_3O$, 251.29): calculated C 71.70, H 5.21, N 16.72%; found C 71.87, H 5.00, N 16.49%.

Crystal data

$C_{15}H_{13}N_3O$
M_r = 251.29
Monoclinic
$P2_1/a$
a = 11.301 (2) Å
b = 8.665 (1) Å
c = 13.422 (1) Å
β = 107.77 (1)°
V = 1251.7 (2) Å^3
Z = 4
D_x = 1.333 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 10.05–18.30°
μ = 0.087 mm^{-1}
T = 295 K
Prismatic
0.62 × 0.54 × 0.32 mm
Light brown

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2847 measured reflections
2532 independent reflections
1943 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 26.3°
h = 0 → 14
k = 0 → 10
l = −16 → 15
3 standard reflections
frequency: 120 min
intensity decay: 1.5%

Refinement

Refinement on F
R = 0.045
wR = 0.053
S = 1.08
1943 reflections
181 parameters
H atoms riding (see below)
$w = 1/[\sigma(F^2) + (0.02F)^2 + 1]$
or w = 0 if $F^2 < 2\sigma(F^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.461 e Å^{-3}
$\Delta\rho_{min}$ = −0.267 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

O1—N3	1.391 (2)	N2—C15	1.305 (3)
N1—C12	1.460 (3)	N3—C11	1.287 (2)
N1—C13	1.360 (3)	C1—C11	1.481 (2)
N1—C15	1.335 (2)	C11—C12	1.504 (2)
N2—C14	1.364 (3)	C13—C14	1.344 (3)
C12—N1—C13	127.8 (2)	N2—C14—C13	110.1 (2)
C12—N1—C15	125.5 (2)	N3—C11—C1	116.1 (1)
C13—N1—C15	106.4 (2)	N3—C11—C12	122.8 (1)
C14—N2—C15	104.7 (2)	C1—C11—C12	121.0 (2)
O1—N3—C11	113.1 (1)	N1—C12—C11	114.8 (2)
N1—C13—C14	106.3 (2)	N1—C15—N2	112.4 (2)
C2—C1—C11—N3	36.0 (2)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···N3	0.95	2.60	2.835 (2)	94
C12—H121···O1	0.90 (2)	2.24 (3)	2.649 (2)	108 (2)
O1—H1···N2'	1.07 (3)	1.66 (3)	2.719 (2)	173 (2)

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

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically, 0.95 Å from their parent atoms; the H atoms of O1 and C12 were refined for a few cycles. For all H atoms except H1, H121 and H122, a riding model was used with $U_{eq}(H) = 1.3U_{eq}(C)$.

Data collection and cell refinement were carried out with *CAD-4 EXPRESS* (Enraf–Nonius, 1993). *MolEN* (Fair, 1990) was used for data reduction, structure solution, structure refinement, molecular graphics and to prepare material for publication. Hydrogen bonds were calculated with *PARST* (Nardelli, 1995).

The authors acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1359). Services for accessing these data are described at the back of the journal.

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Tenfold Interpenetration of Giant Hexagonal $R_{12}^{12}(126)$ Nets in the Hydrogen-Bonded Structure of 1,1,1-Tris(4-hydroxyphenyl)-ethane–4,4′-Bipyridyl (2/3)

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Abstract

In the 2:3 adduct of 1,1,1-tris(4-hydroxyphenyl)ethane [or ethane-1,1,1-triyltris(4-phenol)] and 4,4′-bipyridyl, $2C_{20}H_{18}O_3.3C_{10}H_8N_2$, the components are linked by O—H···N hydrogen bonds [N···O 2.780 (2), 2.745 (2) and 2.731 (2) Å] into puckered two-dimensional nets built from giant hexagons, each involving six units of each component. There are ten such independent nets, all multiply interwoven, within the structure.

Comment

The hydrogen-bonded adducts of 1,3,5-trihydroxybenzene with 4,4′-bipyridyl (Coupar *et al.*, 1996) and with hexamethylenetetramine (Coupar, Glidewell & Ferguson, 1997) have stoichiometry $(triol)_2.(amine)_3$, and in both adducts the structural motif is that of a 'chain-of-rings' (Bernstein *et al.*, 1995). Each ring is formed from two molecules of the triol and two molecules of the amine; these four-component rings are linked into chains by the third amine molecule. In each structure, the triol acts as a triple donor and the amine as a double acceptor of hydrogen bonds, all of which are of the O—H···N type.

In the larger tris-phenol 1,1,1-tris(4-hydroxyphenyl)ethane, $CH_3C(C_6H_4OH)_3$, the hydrogen-bond-donor hydroxy groups are separated by *ca* 9.4 Å in a rather rigid triangle. Thus, in an adduct of this tris-phenol with a diamine such as 4,4′-bipyridyl, in which the hydrogen-bond acceptors are separated by *ca* 7.2 Å at opposite ends of a rigid and effectively linear framework, small-ring formation, as observed in the adducts of 1,3,5-trihydroxybenzene, is precluded by the fixed disposition of the hydrogen-bond donor and acceptor sites in the