Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N-Hydroxy-*N′*-(1-naphthyl)-2-phenylacetamidin-2-one

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Received 4 December 2003
Accepted 12 December 2003
Online 19 December 2003

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.088
Data-to-parameter ratio = 14.6

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

The title compound, $C_{18}H_{14}O_2N_2$, consists of an aminonaphthalene group and a 2-phenyl-1,2-ethanedione 1-oxime group. Intermolecular O—H···N and O—H···O hydrogen bonds [O···N = 2.775 (2) Å and O···O = 3.252 (2) Å], and π–π interactions are highly effective in forming chains, thereby stabilizing the crystal structure. The oxime group has the *E* configuration.

Comment

The oxime (–C═N—OH) moiety is a functional group that is amphiprotic with a slightly basic N atom and a mildly acidic hydroxyl group. As a ligand, the oxime group is potentially ambident with possibilities of coordination through N and/or O atoms. In the vast majority of complexes coordination actually occurs at the N atom. In the solid state, oximes are usually associated *via* O—H···N hydrogen bonds of length ~2.8 Å.

Carbonyl–oximes embrace ligands which contain both carbonyl and oxime groups. The vicinal dioximes are oximes of carbonyl oximes. The complexes of vicinal dioximes have yielded through the decades a continuing series of interesting chemistry. This, in turn, has stimulated research into the coordination chemistry of ligands having other functions in addition to the oxime function. A vast amount of fascinating chemistry has thus accumulated with fundamental bearing on areas such as structure, stability and reactivity of molecules, analytical chemistry and biochemical models (Chakravorty, 1974).

Oxime groups possess stronger hydrogen-bonding capabilities than alcohols, phenols, and carboxylic acids (Marsman *et al.*, 1999), in which intermolecular hydrogen bonding combines moderate strength and directionality (Karle *et al.*, 1996) in linking molecules to form supramolecular structures; this has received considerable attention with respect to directional non-covalent intermolecular interactions (Etter *et al.*, 1990).

Structurally characterized oxime moieties are much less common than carboxylic acids and amides, but from a supramolecular perspective, this functionality does have some unique and desirable features (Aakeröy *et al.*, 2001).

The hydrogen-bond systems in the crystal structures of oximes have been analysed and a correlation between a pattern of hydrogen bonding and N—O bond lengths has been suggested (Bertolasi *et al.*, 1982). The configurational and/or conformational isomers of glyoxime derivatives (dioximes) have also been analysed (Chertanova *et al.*, 1994).

Oxime/oximate metal complexes have been investigated actively in recent years (Kukushkin & Pombeiro, 1999). In general, oxime and dioxime derivatives are very important

DOI: 10.1107/S1600536803028617

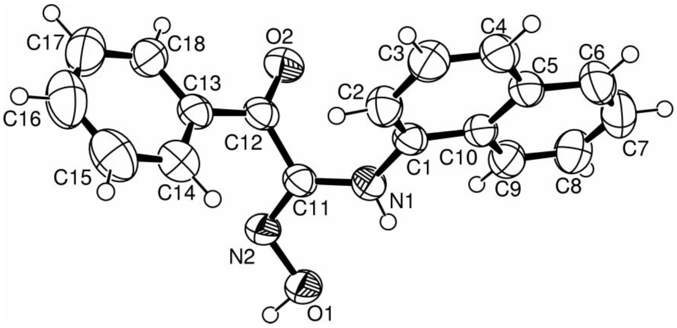
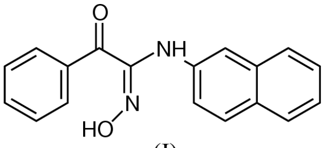


Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of the title molecule with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

compounds in the chemical industry and medicine (Sevagapandian *et al.*, 2000).

The structure determination of the title molecule, (I), was carried out in order to investigate the strength of the hydrogen-bonding capability of the oxime group and to compare the geometry of the oxime moiety with those found in 2,3-dimethylquinoxaline–dimethylglyoxime (1/1), (II) (Hökelek, Batı *et al.*, 2001), 1-(2,6-dimethylphenylamino)-propane-1,2-dione dioxime, (III) (Hökelek, Zülfikaroğlu *et al.*, 2001), *N*-(3,4-dichlorophenyl)-*N*′-hydroxy-2-oxo-2-phenylacetamidine, (IV) (Hökelek *et al.*, 2004) and *N*-hydroxy-2-oxo-2,*N*′-diphenylacetamidine, (V) (Büyükgüngör *et al.*, 2003).



(I)

Compound (I) (Fig. 1) consists of aminonaphthalene and 2-phenyl-1,2-ethanedione 1-oxime moieties. The dihedral angles between the oxime plane (*A*, atoms O1/N2/C11) and rings *B* (C1–C5/C10), *C* (C5–C10) and *D* (C13–C18) are *A*/*B* = 42.89 (6)°, *A*/*C* = 43.51 (7)° and *A*/*D* = 67.97 (11)°. The naphthalene system itself is not planar, with an angle of 2.32 (5)° between the rings *B* and *C*.

The steric effects of the substituents bonded to the C atom of the oxime group may influence the bond lengths and angles of the oxime moiety (Table 1). Some significant changes in the geometry of the oxime moiety are evident when the bond lengths and angles are compared with the corresponding values in compounds (II)–(V) (Table 2).

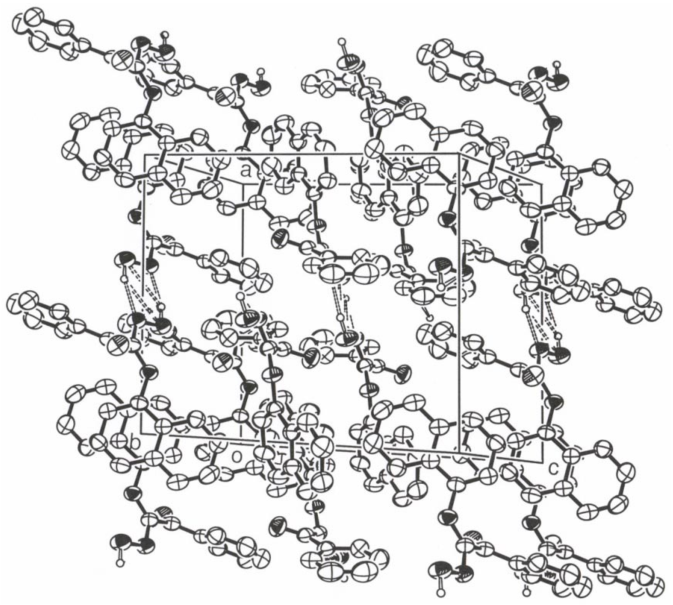


Figure 2
Packing diagram for (I).

The oxime moiety has an *E* configuration [C12—C11—N2—O1 = 172.49 (11)°; Chertanova *et al.*, 1994]. In this configuration, the oxime group is involved as a donor in O1—H2···N2$^{\mathrm{i}}$ intermolecular hydrogen bonding [O1—H2 = 0.94 (2) Å, O1···N2$^{\mathrm{i}}$ = 2.775 (2) Å, H2···N2$^{\mathrm{i}}$ = 1.89 (2) Å and O1—H2···N2$^{\mathrm{i}}$ = 157 (2)°; symmetry code: (i) $1 - x, -y, -z$]. There are also N1—H1···O1 intramolecular [N1—H1 = 0.89 (2) Å, N1···O1 = 2.575 (2) Å, H1···O1 = 2.20 (2) Å and N1—H1···O1 = 105 (1)°] and O1—H2···O1$^{\mathrm{i}}$ intermolecular [O1···O1$^{\mathrm{i}}$ = 3.252 (2) Å, H2···O1$^{\mathrm{i}}$ = 2.63 (2) Å and O1—H2···O1$^{\mathrm{i}}$ = 125 (2)°] hydrogen bonds. These intermolecular hydrogen bonds link the molecules into centrosymmetric dimers. The dimers are interlinked to form chains along the *a* axis by π–π interactions involving ring C1–C5/C10 and that in an inversion-related molecule at $(-x, -y, -z)$; the centroids of these two rings are separated by 3.676 (1) Å. Thus, inter- and intramolecular hydrogen bonds and π–π interactions are highly effective in stabilizing the crystal structure. A view of the molecular packing is shown in Fig. 2.

Experimental

A solution of ω-chloroisonitrosoacetophenone (0.015 mol, 2.75 g) in ethyl alcohol (20 ml) was added dropwise to a solution of 1-naphthylamine (0.015 mol, 4.35 g) in ethyl alcohol (10 ml). The mixture was stirred for 1 h; $NaHCO_3$ (1.10 g) and water (10 ml) were then added. The precipitated product was filtered off, washed with water and then recrystallized from ethyl alcohol.

Crystal data

$C_{18}H_{14}N_2O_2$
$M_r = 290.31$
Monoclinic, $P2_1/c$
$a = 12.0073\ (11)$ Å
$b = 8.4530\ (9)$ Å
$c = 15.0167\ (15)$ Å
$\beta = 94.188\ (8)^\circ$
$V = 1520.1\ (3)$ Å^3
$Z = 4$
$D_x = 1.269$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4977 reflections
$\theta = 2.3$–27.9°
$\mu = 0.08$ mm^{-1}
$T = 293\ (2)$ K
Block, yellow
0.30 × 0.26 × 0.19 mm

Data collection

Stoe IPDS-II diffractometer
φ scans
13 837 measured reflections
3713 independent reflections
1641 reflections with $I > 2\sigma(I)$
$R_{\mathrm{int}} = 0.055$
$\theta_{\max} = 28.3^\circ$
$h = -14 \rightarrow 15$
$k = -11 \rightarrow 11$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.088$
$S = 0.86$
3713 reflections
255 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0423P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.11$ e Å^{-3}
$\Delta\rho_{\min} = -0.20$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C11	1.3507 (19)	C12—C11	1.513 (2)
N1—C1	1.4156 (19)	C13—C14	1.383 (2)
N2—C11	1.2890 (18)	C13—C18	1.395 (2)
N2—O1	1.4243 (15)	C9—C8	1.365 (3)
C1—C2	1.363 (2)	C4—C3	1.346 (2)
C1—C10	1.421 (2)	C6—C7	1.349 (3)
O2—C12	1.2147 (17)	C7—C8	1.395 (3)
C12—C13	1.473 (2)	C14—C15	1.384 (3)
C11—N1—C1	125.79 (14)	C13—C12—C11	118.57 (12)
C11—N2—O1	110.59 (12)	C6—C5—C4	121.94 (16)
C2—C1—N1	121.82 (14)	C14—C13—C12	121.81 (14)
N1—C1—C10	117.62 (13)	C18—C13—C12	118.46 (14)
C9—C10—C1	123.70 (14)	N2—C11—N1	125.01 (14)
O2—C12—C13	123.28 (14)	N2—C11—C12	113.23 (13)
O2—C12—C11	118.13 (14)	N1—C11—C12	121.05 (13)
C11—N1—C1—C2	26.6 (2)	O1—N2—C11—C12	172.49 (11)
C11—N1—C1—C10	−156.23 (14)	C1—N1—C11—N2	−160.03 (15)
N1—C1—C10—C9	4.7 (2)	C1—N1—C11—C12	30.2 (2)
N1—C1—C10—C5	−175.49 (13)	O2—C12—C11—N2	−123.78 (16)
C1—C10—C5—C6	177.28 (14)	C13—C12—C11—N2	54.32 (19)
O2—C12—C13—C14	−159.27 (16)	O2—C12—C11—N1	47.1 (2)
O2—C12—C13—C18	17.8 (2)	C13—C12—C11—N1	−134.83 (15)
O1—N2—C11—N1	2.1 (2)	N1—C1—C2—C3	178.50 (14)

Table 2
Comparison of the bond lengths and angles (Å,°) in the oxime moiety of (I) with the corresponding values in the related compounds (II)–(V) (double entries are presented for structures that have two independent molecules).

	(I)	(II)	(III)	(IV)	(V)
N2—O1	1.424 (2)	1.403 (2)/ 1.396 (2)	1.423 (3)/ 1.396 (3)	1.429 (4)	1.417 (1)
N2—C11	1.289 (2)	1.281 (2)/ 1.281 (2)	1.290 (3)/ 1.282 (3)	1.241 (6)	1.290 (1)
C11—C12	1.513 (2)	1.477 (3)/ 1.473 (3)	1.489 (3)	1.551 (7)	1.510 (1)
C12—C11—N2	113.2 (1)	115.2 (2)/ 115.0 (2)	116.6 (2)/ 115.0 (2)	118.3 (5)	114.3 (1)
C11—N2—O1	110.6 (1)	112.4 (1)/ 112.2 (1)	109.4 (2)/ 111.5 (2)	112.2 (4)	110.7 (1)

H atoms were located in a difference map and their positional and isotropic displacement parameters were refined [N—H = 0.89 (2) Å and C—H = 0.94 (2)–1.01 (2) Å].

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS-II diffractometer (purchased under grant F.279 of the University Research Fund).

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