Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

1-Methyl-1-phenyl-3-[1-hydroxyimino-2-(succinimido)ethyl]cyclobutane

Muharrem Dinçer,[a]* Namık Özdemir,[a] Ibrahim Yılmaz,[b] Alaaddin Çukurovalı[b] and Orhan Büyükgüngör[a]

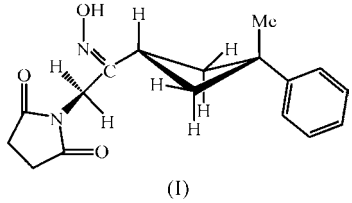
[a]Department of Physics, Arts and Sciences Faculty, Ondokuz Mayıs University, 55139 Samsun, Turkey, and [b]Department of Chemistry, Arts and Sciences Faculty, Fırat University, 23119 Elazığ, Turkey
Correspondence e-mail: mdincer@omu.edu.tr

Received 11 June 2004
Accepted 22 July 2004
Online 21 August 2004

In the title compound, $C_{17}H_{20}N_2O_3$, the cyclobutane ring is puckered, with a dihedral angle of 19.11 (15)°. The 1-phenyl and 3-[1-hydroxyimino-2-(succinimido)ethyl] groups are in *cis* positions. The molecules are linked by O—H···O and C—H···π(benzene) interactions, forming a two-dimensional network.

Comment

3-Substituted cyclobutane carboxylic acid derivatives exhibit anti-inflammatory and antidepressant activities (Dehmlow & Schmidt, 1990), and also liquid crystal properties (Coghi *et al.*, 1976). Oximes show geometric isomerism due to the double bond between the N and C atoms (Mixich & Thiele, 1979; Migrdichian, 1957). Because of significant differences in the physical, chemical and biological properties of these geometric isomers, the determination of the configuration of the isomers is important (Mathison *et al.*, 1989). Oximes and oxime ethers also 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). The oxime group (C=N—OH) possesses stronger hydrogen-bonding capabilities than the alcohol, phenol or carboxylic acid group (Marsman *et al.*, 1999). Hydrogen bonding plays a key role in molecular recognition in chemical engineering (Bertolasi *et al.*, 1982; Gilli *et al.*, 1983; Hökelek *et al.*, 2001). As part of our ongoing study of the relationship between the molecular and crystal structures of cyclobutane and oxime derivatives, a crystal structure determination of the title compound, (I), has been undertaken and the results are presented here.



(I)

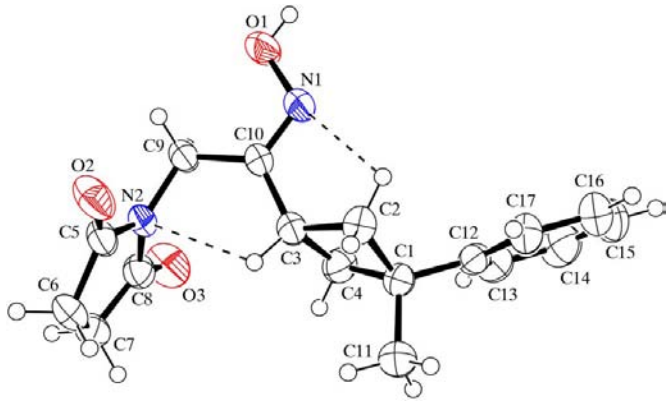


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate the C—H···N interactions.

Previously, we have reported the closely related compound 2-[2-hydroxyimino-2-(3-methyl-3-phenylcyclobutyl)ethyl]isoindole-1,3-dione, (II) (Özdemir *et al.*, 2004). The main aim of the present investigation is to study the types of differences between the structures of (I) and (II), and also to determine the strength of the hydrogen-bonding capabilities of the oxime group.

Fig. 1 shows the molecular structure and conformation of (I), with the atomic numbering scheme. The structure of (I) can be described as being built from planar fragments, *viz*. a cyclobutane ring (C1–C4), an oxime group (C10/N1/O1), a succinimide ring (O2/O3/N2/C5–C8), a benzene ring (C12–C17), and a four-atom bridge (C3/C10/C9/N2) linking the cyclobutane and succinimide rings. The maximum deviation of

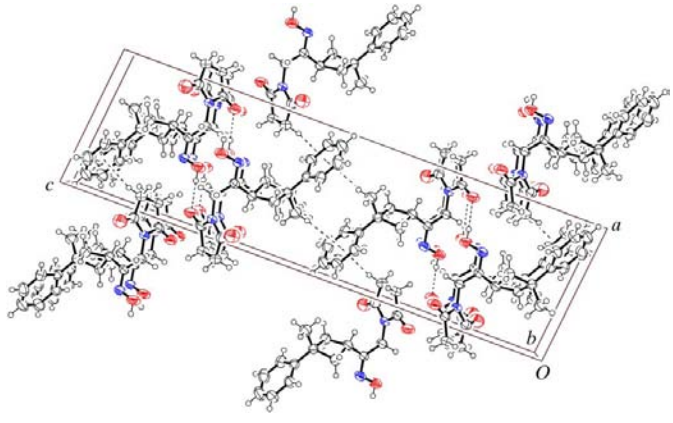


Figure 2
A projection of the crystal structure of (I) along the *b* axis. Dashed lines show the O—H···O and C—H···π interactions.

  DOI: 10.1107/S0108270104018074

the succinimide ring from planarity is 0.0413 (13) Å for atom C6. The C3—C10—C9—N2 torsion angle is 8.5 (2)°, corresponding to a (+)-synperiplanar configuration, and the plane of the four-atom bridge is almost perpendicular to the succinimide ring (Table 1). The plane of these four atoms makes a dihedral angle of 76.19 (8)° with the mean plane of the cyclobutane ring.

Although close to being planar, the cyclobutane ring in (I) is more puckered than in (II). The C4/C1/C2 plane forms a dihedral angle of 19.26 (17)° with the C2/C3/C4 plane [11.55 (3)° in (II); Özdemir *et al.*, 2004]. The mean plane of the cyclobutane ring forms a dihedral angle of 81.62 (6)° with the plane of the succinimide ring. The oxime moiety has an *E* configuration, with a C3—C10—N1—O1 torsion angle of 175.95 (14)°, which corresponds to a (+)-antiperiplanar configuration. In this configuration, atom O1 acts as hydrogen-bond donor to atom O2 of the succinimide group at $(1-x, \frac{1}{2}+y, \frac{1}{2}-z)$. The O···O distance is 2.7594 (16) Å, which is a little shorter than that in (II) [2.814 (3) Å]. In (I), the plane of the oxime moiety is twisted by 74.95 (13)° out of the mean plane of the cyclobutane ring, but it is almost coplanar with the four-atom bridge, with a dihedral angle of 3.2 (2)°. The bond lengths and angles of the oxime moiety in (I) are close to those in (II).

There are two weak intramolecular C—H···N interactions in (I) (Fig. 1). Each of these interactions forms a five-membered ring. As a point of difference from (II), two intermolecular C—H···π(benzene) interactions are also observed (Fig. 2 and Table 2). The centroid (*Cg*3) of the C12–C17 benzene ring acts as a single acceptor for both these C—H···π interactions. A two-dimensional network is formed by the O—H···O and C—H···π(benzene) interactions. There are no intermolecular π–π interactions in the crystal structure of (I).

Experimental

A mixture of 1-phenyl-1-methyl-3-(2-succinimidoacetyl)cyclobutane (2.853 g, 0.01 mol), synthesized according to the method of Ahmedzade *et al.* (2003), hydroxylamine hydrochloride (0.695 g, 0.01 mol) and pyridine (5 ml) in ethanol (100 ml) was refluxed for 3 h. The solvent was removed by distillation and the resulting solid was filtered off, washed with cold water, dried and recrystallized from ethanol to obtain the title compound (yield 2.8 g, 85%; m.p. 426 K). Elemental analysis calculated for $C_{17}H_{20}N_2O_3$: C 67.98, H 6.71, N 9.33%; found: C 68.02, H 6.84, N 9.45%. IR spectroscopy (KBr pellet, ν, cm^{-1}): 1620 (C═N), 3253 (–OH oxime). ^{1}H NMR ($CDCl_3$, p.p.m.): 7.10–7.30 (*m*, 5H, aromatic), 4.4 (*s*, 2H, CH_2 cyclobutane), 3.5 (*quint*, 1H, J = 8.9 Hz, CH cyclobutane), 1.74–2.75 (*m*, 8H, CH_2 cyclobutane plus succinimide), 1.49 (*s*, 3H, CH_3).

Crystal data

$C_{17}H_{20}N_2O_3$
M_r = 300.35
Monoclinic, $P2_1/c$
a = 8.8356 (7) Å
b = 5.7520 (5) Å
c = 30.803 (2) Å
β = 95.821 (6)°
V = 1557.4 (2) Å^3
Z = 4
D_x = 1.281 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12 629 reflections
θ = 1.3–26.1°
μ = 0.09 mm^{-1}
T = 250 K
Prism, colourless
0.63 × 0.53 × 0.35 mm

Data collection

Stoe IPDS-2 diffractometer
w scans
11 598 measured reflections
2694 independent reflections
2179 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 25.0°
$h = -10 \rightarrow 10$
$k = -6 \rightarrow 6$
$l = -34 \rightarrow 36$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.146
S = 1.02
2694 reflections
200 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.112P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.74 e Å^{-3}
Extinction correction: *SHELXL*97 (Sheldrick, 1997)
Extinction coefficient: 0.027 (6)

Table 1
Selected geometric parameters (Å, °).

O1—N1	1.4030 (18)	C1—C12	1.510 (2)
O2—C5	1.214 (2)	C1—C11	1.525 (2)
O3—C8	1.208 (2)	C1—C4	1.550 (2)
N1—C10	1.274 (2)	C1—C2	1.552 (2)
N2—C5	1.369 (2)	C2—C3	1.547 (2)
N2—C8	1.391 (2)	C3—C10	1.495 (2)
N2—C9	1.4552 (18)	C3—C4	1.556 (2)
C10—N1—O1	110.61 (13)	C1—C4—C3	89.85 (12)
C5—N2—C8	112.71 (13)	O2—C5—N2	124.36 (14)
C5—N2—C9	124.33 (13)	O3—C8—N2	123.25 (15)
C8—N2—C9	122.96 (13)	N2—C9—C10	113.71 (12)
C4—C1—C2	88.39 (12)	N1—C10—C3	117.08 (13)
C3—C2—C1	90.11 (12)	N1—C10—C9	120.40 (14)
C2—C3—C4	88.35 (12)	C3—C10—C9	122.40 (12)
C5—N2—C9—C10	85.44 (19)	C4—C3—C10—N1	−75.92 (19)
C8—N2—C9—C10	−94.48 (18)	C2—C3—C10—C9	−157.32 (15)
O1—N1—C10—C3	175.95 (14)	C4—C3—C10—C9	100.17 (17)
O1—N1—C10—C9	−0.2 (2)	N2—C9—C10—N1	−175.52 (15)
C2—C3—C10—N1	26.6 (2)	N2—C9—C10—C3	8.5 (2)

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

*Cg*3 is the centroid of the C12–C17 benzene ring.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···N2	0.98	2.50	2.9030 (19)	104
C2—H2*B*···N1	0.97	2.51	2.879 (2)	102
O1—H1···O2^{i}	0.82	1.94	2.7594 (16)	177
C7—H7*A*···*Cg*3^{ii}	0.97	2.74	3.68	163
C11—H11*C*···*Cg*3^{iii}	0.96	3.12	3.71	121

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

The oxime H atom was located from a difference density map and the other H atoms were positioned geometrically. All H atoms were treated using a riding model, with an O—H distance of 0.82 Å and C—H distances of 0.93–0.98 Å, and with U_{iso}(H) = 1.2U_{eq} (1.5U_{eq} for methyl) of the parent atom. The maximum electron density is located 0.70 Å from atom C3 and the minimum electron density is 0.59 Å from atom C11.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (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).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OB1188). Services for accessing these data are described at the back of the journal.

References

Ahmedzade, M., Çukurovalı, A. & Koparır, M. (2003). *J. Chem. Soc. Pak.* **25**, 51–55.

Balsamo, A., Macchia, B., Martinelli, A., Orlandini, E., Rossello, A., Macchia, F., Bocelli, G. & Domiano, P. (1990). *Eur. J. Med. Chem.* **25**, 227–233.

Bertolasi, V., Gilli, G. & Veronese, A. C. (1982). *Acta Cryst.* B**38**, 502–511.

Coghi, L., Lanfredi, A. M. M. & Tiripicchio, A. (1976). *J. Chem. Soc. Perkin Trans.* 2, pp. 1808–1810.

Dehmlow, E. V. & Schmidt, S. (1990). *Liebigs Ann. Chem.* p. 411.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Forman, S. E. (1964). *J. Org. Chem.* **29**, 3323–3327.

Gilli, G., Bertolasi, V. & Veronese, A. C. (1983). *Acta Cryst.* B**39**, 450–456.

Hökelek, T., Zülfikaroğlu, A. & Batı, H. (2001). *Acta Cryst.* E**57**, o1247–o1249.

Holan, G., Johnson, W. M. P., Rihs, K. & Virgona, C. T. (1984). *Pestic. Sci.* **15**, 361–368.

Marsman, A. W., Leussing, E. D., Zwikker, J. W. & Jenneskens, L. W. (1999). *Chem. Mater.* **11**, 1484–1491.

Mathison, I. W., Solomons, W. E., Morgan, P. H. & Tidwell, R. R. (1989). *Structural Features and Pharmacologic Activity*. *Principals of Medicinal Chemistry*, edited by W. O. Foye, pp. 49–77. Philadelphia: Lea and Febiger.

Migrdichian, V. (1957). *Open-Chain Saturated Compounds*. *Organic Synthesis*, pp. 703–707. New York: Reinhold.

Mixich, G. V. & Thiele, K. (1979). *Arzneim. Forsch.* (*Drug Res.*), **29**, 1510–1513.

Özdemir, N., Dinçer, M., Yılmaz, İ. & Çukurovalı, A. (2004). *Acta Cryst.* E**60**, o145–o147.

Polak, A. (1982). *Arzneim. Forsch.* (*Drug Res.*), **32**, 17–24.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED*32 (Version 1.04). Stoe & Cie, Darmstadt, Germany.