Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

2,2-Dimethyl-1,4-dihydro-2*H*-3,1-benzoxazine

Mustafa Odabaşoğlu,[a]* Özgür Özdamar[a] and Orhan Büyükgüngör[b]

[a]Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Samsun, Turkey, and [b]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Samsun, Turkey

Correspondence e-mail: muodabas@omu.edu.tr

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.061
Data-to-parameter ratio = 15.3

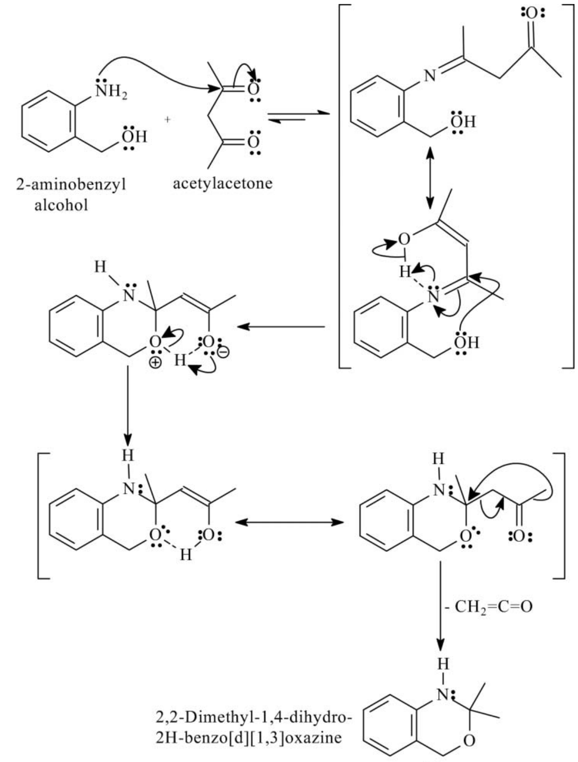
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 3 January 2006
Accepted 31 January 2006

The title compound, $C_{10}H_{13}NO$, crystallizes with two independent but essentially identical molecules in the asymmetric unit. The molecules are linked by two N—H···O and two C—H···O hydrogen bonds into chains of edge-fused rings in which $R_3^3(10)$ and $R_8^8(33)$ rings alternate.

Comment

1,3-Oxazines have generated interest as antipsychotic agents and as possible effectors for serotonin and dopamine receptors (Gentles *et al.*, 1991; Petterson *et al.*, 1990; Peglion *et al.*, 1997). In addition, benzoxazines have been evaluated as antimalarial agents (Ren *et al.*, 2001). The title compound, (I), was prepared by reaction of acetylacetone and 2-aminobenzyl alcohol. This is a new method for synthesizing oxazabenzocyclohexanes (see scheme). The steps proposed in the scheme are based on the more stable character of the reaction products with respect to the intermediate products. The structure of (I) has been determined in order to establish both the geometry of the oxazahetero ring and the nature of the supramolecular interactions.



(I)

Compound (I) (Fig. 1) crystallizes in space group $P2_12_12_1$, with two independent molecules in the asymmetric unit

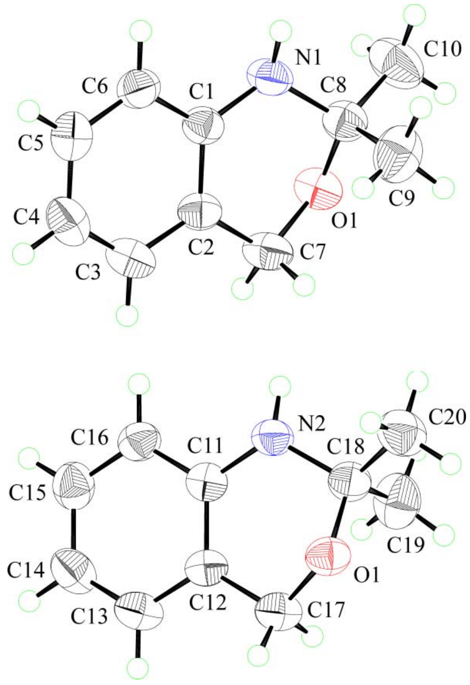


Figure 1
Views of the independent molecules in (I), showing the atom-numbering schemes and 50% probability displacement ellipsoids.

(Fig. 1). The intramolecular dimensions are very similar in the two molecules and the bond distances show no unusual features. The angles at the ring atoms N1 and N2 show considerable expansion from 109° (Table 1).

Molecules of (I) are linked into a chain of edge-fused rings by the combination of two nearly linear N—H···O hydrogen bonds and two rather weak C—H···O hydrogen bonds; geometric parameters are listed in Table 2. Within the asymmetric unit, atom N1 in the first independent molecule acts as hydrogen-bond donor to O2 in the second molecule and, in a similar way, atom N2 in the second independent molecule acts as hydrogen-bond donor to O4 in the symmetry-related 'second' molecule. These hydrogen bonds generate, by translation, a *C*(5) chain (Bernstein *et al.*, 1995) that runs parallel to the [100] direction, as illustrated in Fig. 2.

Two such chains pass through each unit cell and these two chains are weakly linked by C—H···O hydrogen bonds (H···O < 3.00 Å and C—H···O > 110°) (Jeffrey, 1997). The aryl C6 atom in the first independent molecule acts as a hydrogen-bond donor to atom O1, thereby generating two $R_3^3(10)$ rings and one $R_8^8(33)$ ring. Propagation by translation of the $R_3^3(10)$ motif linking antiparallel *C*(4) chains then generates an [010] chain of edge-fused rings, with two $R_3^3(10)$ rings alternating with $R_8^8(33)$ rings. There are no direction-specific aromatic π–π interactions between adjacent chains of rings in (I).

Experimental

Compound (I) was prepared by combining an *n*-butanol solution (50 ml) containing acetylacetone (1.00 g, 10 mmol) with an *n*-butanol (50 ml) solution containing 2-aminobenzyl alcohol (1.23 g, 10 mmol). The reaction mixture was stirred for 2 h under reflux. Solid (I) was obtained by allowing the clear reaction mixture to stand overnight. Crystals of (I) suitable for single-crystal X-ray diffraction were grown by slow evaporation of a tetrahydrofuran solution of (I) (yield 75%, m.p. 390–392 K).

Crystal data

$C_{10}H_{13}NO$
M_r = 163.21
Orthorhombic, $P2_12_12_1$
a = 8.2844 (9) Å
b = 8.4743 (10) Å
c = 25.388 (3) Å
V = 1782.4 (4) Å^3
Z = 8
D_x = 1.216 Mg m^{-3}

Mo *Kα* radiation
Cell parameters from 9459 reflections
θ = 1.6–26.0°
μ = 0.08 mm^{-1}
T = 296 K
Block, colourless
0.44 × 0.34 × 0.19 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.969, T_{max} = 0.986
9973 measured reflections
3493 independent reflections

2027 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 26.0°
h = −10 → 10
k = −10 → 10
l = −31 → 27

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.061
S = 0.81
3493 reflections
229 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0275P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.398 (2)	C11—N2	1.394 (2)
C7—O1	1.439 (2)	C17—O2	1.430 (2)
C8—O1	1.436 (2)	C18—O2	1.438 (2)
C8—N1	1.447 (2)	C18—N2	1.460 (3)
C7—O1—C8	113.33 (14)	O1—C8—C9	110.92 (15)
C17—O2—C18	113.63 (14)	O1—C8—C10	104.84 (17)
C1—N1—C8	117.64 (16)	N1—C8—C9	112.72 (17)
C11—N2—C18	117.91 (17)	N1—C8—C10	109.30 (18)
O1—C7—C2	111.53 (16)	C9—C8—C10	111.20 (19)
O1—C8—N1	107.51 (16)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.882 (19)	2.25 (2)	3.131 (2)	176.5 (17)
N2—H2*N*···O2^{ii}	0.88 (2)	2.20 (2)	3.082 (2)	177 (2)
C20—H20*C*···O1^{ii}	0.96	2.86	3.707 (3)	148
C6—H6···O2^{iii}	0.93	2.68	3.308 (2)	125

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+\frac{1}{2}, -y+1, z-\frac{1}{2}$.

In the absence of significant anomalous dispersion effects, 1460 Friedel pairs were averaged. All C-bound H atoms were refined using the riding-model approximation, with C—H = 0.93 Å for aromatic, 0.97 Å for methylene and 0.96 Å for methyl H atoms, and with

Acta Cryst. (2006). E**62**, o908–o910 Odabaşoğlu *et al.* • $C_{10}H_{13}NO$ **o909**

U_{iso}(H) = 1.2U_{eq}(C) for aromatic and methylene H atoms, and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms. N-bound H atoms were refined freely; see Table 2 for geometric parameters.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

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

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

Gentles, R. G., Middlemiss, D., Proctor, G. R. & Sneddon, A. H. (1991). *J. Chem. Soc. Perkin Trans. 1*, pp. 1423–1431.

Jeffrey, A. G. (1997). *An Introduction to Hydrogen Bonding*, pp. 85-87. Oxford University Press.

Peglion, J. L., Vian, J., Gourment, B., Despaux, N., Audinot, V. & Millan, M. (1997). *Bioorg. Med. Chem. Lett.* **7**, 881–886.

Petterson, I., Liljefors, T. & Bodeso, K. (1990). *J. Med. Chem.* **33**, 2197–2204.

Ren, H., Grady, S., Gamenara, D., Heinzen, H., Moyna, P., Croft, S., Kendrick, H., Yardley, V. & Moyna, G. (2001). *Bioorg. Med. Chem. Lett.* **11**, 1851–1854.

Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

o910 Odabaşoğlu *et al.* • $C_{10}H_{13}NO$ *Acta Cryst.* (2006). E**62**, o908–o910