

Hydrogen bonds and C—H···O interactions in *N*-(2-hydroxy-1,1-dimethylethyl)benzamide at 150 K

Roger E. Gerkin

Department of Chemistry, The Ohio State University, Columbus, Ohio 43210, USA
Correspondence e-mail: gerkin@chemistry.ohio-state.edu

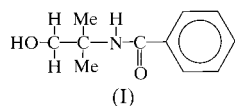
Received 30 March 2000

Accepted 23 May 2000

The title compound, $C_{11}H_{15}NO_2$, crystallized in the centrosymmetric space group $P2_1/n$ with one molecule in the asymmetric unit. There is a single intermolecular hydrogen bond, in which the $N_{\text{donor}} \cdots O_{\text{acceptor}}$ distance is 3.0374 (11) Å and the $N-H \cdots O$ angle is 171.0 (12)°. The single intramolecular hydrogen bond has an $O_{\text{donor}} \cdots O_{\text{acceptor}}$ distance of 2.6279 (11) Å and an $O-H \cdots O$ angle of 161.8 (14)°. The four leading intermolecular C—H···O interactions have H···O distances ranging from 2.52 to 2.65 (2) Å and C—H···O angles ranging from 125.2 (9) to 143°. Chains of interactions form two-dimensional networks.

Comment

This report is one of a series on hydrogen bonding and C—H···O interactions in aromatic compounds. The title compound, (I), crystallized in the centrosymmetric space group $P2_1/n$ with one molecule in the asymmetric unit. The refined molecule and the labeling scheme are given in Fig. 1.



Two hydrogen bonds, one of them intramolecular, and four leading intermolecular C—H···O interactions (Taylor & Kennard, 1982; Steiner & Desiraju, 1998) are present in this structure. The geometric parameters of these are given in Table 2. A central molecule is linked directly to five neighboring molecules by the tabulated interactions, as shown in the stereodiagram (Fig. 2). The results of basic first- and second-level graph-set analysis (Bernstein *et al.*, 1995) involving these interactions, labeled *a–f* for this purpose in the order of their appearance in Table 2, are given in Table 3. Among the 21 patterns 15 are chains, which propagate variously along [101] (8), [010] (5) and [111] (2). Since only two of three such vectors are linearly independent, however, the resulting network of interactions is two-dimensional. Of the five ring patterns, two are disposed about centers of symmetry.

The phenyl ring is nearly planar, the maximum deviation of a ring atom from the best-fit plane describing them being 0.0053 (11) Å. The dihedral angle between this plane and that of the amide group atoms C7, O1 and N1 is 25.77 (12)°. This angle is apparent in Fig. 2.

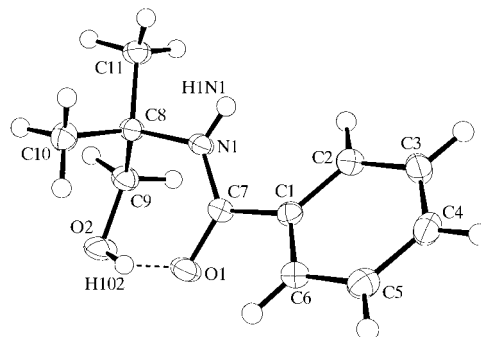


Figure 1

The molecular view of compound (I), showing the atom-labeling scheme, with displacement ellipsoids at the 50% probability level. The dashed line depicts the intramolecular hydrogen bond. H atoms are drawn as spheres of arbitrary radii.

Selected bond distances and angles are given in Table 1. All distances and angles fall within normal limits. For structural comparisons of the aliphatic amide portion of (I), the structure of 4,6-dimethyl-3-(4,4-dimethyl-2-oxazolonyl)-*N*-(2-hydroxy-1,1-dimethylethyl)salicylamide [Inamoto *et al.*, 1996; hereinafter (II)] appears to be the best available, although the s.u.'s are almost ten times those of the present study. For the eight pairs of corresponding non-H interatomic distances in the aliphatic amide substructures of (I) and (II), the greatest difference is just twice the s.u. of that distance given for (II), so these results are in good agreement. The orientation of this group with respect to the attached phenyl ring, however, is quite different in the two molecules, since in (II) the dihedral angle between the best-fit phenyl plane and the amide plane as defined above is 88.4 (9)°. In view of the four additional substituents on the phenyl ring of (II), two of them capable of hydrogen bonding, this difference is not problematic. In (I) the closest intermolecular approaches, excluding pairs of atoms involved in the hydrogen-bonding groups or the tabulated C—H···O interactions, are between H2 and H9Bⁱ [symmetry code: (i) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$] and fall short of the corresponding Bondi (1964) van der Waals radius sum by 0.04 Å.

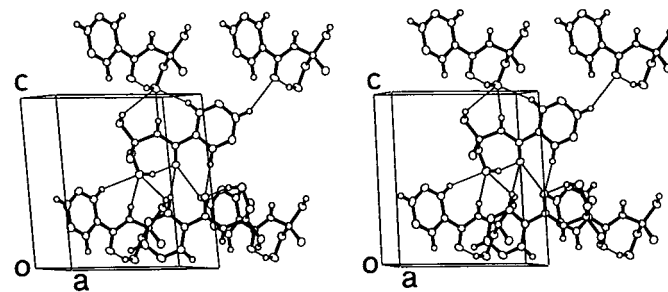


Figure 2

Stereodiagram of (I) viewed nearly down the *a* axis. The finer interatomic lines depict the interactions given in Table 2. For clarity, only those H atoms involved in these interactions are included.

Experimental

Compound (I) was obtained as a column from a crystalline sample in Dr M. S. Newman's chemical collection. This was cut to provide the experimental sample. One synthesis, among others, is given by Boyd & Hansen (1953).

Crystal data

$C_{11}H_{15}NO_2$	$D_x = 1.282 \text{ Mg m}^{-3}$
$M_r = 193.24$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 22 319 reflections
$a = 10.4286 (2) \text{ \AA}$	$\theta = 1.93\text{--}27.48^\circ$
$b = 9.1195 (2) \text{ \AA}$	$\mu = 0.088 \text{ mm}^{-1}$
$c = 11.3748 (2) \text{ \AA}$	$T = 150 \text{ K}$
$\beta = 112.2995 (11)^\circ$	Cut column, colorless
$V = 1000.88 (3) \text{ \AA}^3$	$0.35 \times 0.27 \times 0.27 \text{ mm}$
$Z = 4$	

Data collection

Nonius KappaCCD diffractometer	$R_{\text{int}} = 0.032$
ω scans with κ offsets	$\theta_{\text{max}} = 27.48^\circ$
22 319 measured reflections	$h = -13 \rightarrow 13$
2293 independent reflections	$k = -11 \rightarrow 11$
1936 reflections with $I > 2\sigma(I)$	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
$R(F) = 0.038$	
$wR(F^2) = 0.098$	
$S = 1.97$	$w = 1/[\sigma_{\text{cs}}^2 + (0.032I)^2]$
2293 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
167 parameters	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

O1—C7	1.2446 (11)	N1—C8	1.4913 (12)
O2—C9	1.4148 (13)	C1—C7	1.5004 (14)
N1—C7	1.3391 (12)		
C7—N1—C8	125.51 (8)	O1—C7—C1	118.81 (9)
C2—C1—C7	123.49 (9)	N1—C7—C1	118.08 (8)
C6—C1—C7	117.36 (8)	O2—C9—C8	115.73 (8)
O1—C7—N1	123.10 (9)		

Table 2

Hydrogen bonds and leading C—H...O interactions (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O2—H1O2...O1	0.89 (2)	1.77 (2)	2.6279 (11)	161.8 (14)
N1—H1N1...O2 ⁱ	0.85 (2)	2.20 (2)	3.0374 (11)	171.0 (12)
C2—H2...O2 ⁱ	0.98	2.52	3.2407 (13)	131
C4—H4...O1 ⁱⁱ	0.98	2.60	3.4305 (12)	143
C6—H6...O1 ⁱⁱⁱ	0.98	2.63	3.4517 (12)	141
C11—H11B...O2 ⁱ	0.97 (2)	2.65 (2)	3.3054 (14)	125.2 (9)

Symmetry codes: (i) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$; (ii) $\frac{1}{2} + x, \frac{5}{2} - y, \frac{1}{2} + z$; (iii) $1 - x, 2 - y, 1 - z$.

Table 3

Basic first- and second-level graph-set descriptors involving interactions designated $a\text{--}f$ in the order given in Table 2.

	a	b	c	d	e	f
a	$S(7)$	$C_2^2(6)$	$C_2^2(7)$	$C_2^1(12)$	$R_4^2(20)$	$C_2^2(8)$
b		$C(5)$	$R_2^1(7)$	$C_2^3(14)$	$C_2^2(12)$	$R_2^1(6)$
c			$C(8)$	$C_2^2(11)$	$C_2^2(11)$	$R_2^1(9)$
d				$C(7)$	$C_2^1(6)$	$C_2^1(16)$
e					$R_2^1(10)$	$C_2^2(14)$
f						$C(5)$

Fourier difference methods were used to locate initial H-atom positions, and these H atoms were refined. Refined C—H distances ranged from 0.96 (1) to 1.05 (1) \AA , with a mean value of 0.99 (3) \AA ; their U_{iso} values ranged from 0.96 to 1.5 times the U_{eq} values of the attached C atoms. The ring H atoms were then made canonical, with C—H = 0.98 \AA and $U_{\text{iso}} = 1.2U_{\text{eq}}$ of the attached C atom.

Data collection: *COLLECT* (Nonius, 1999); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1992–1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN* and *PLATON* (Spek, 1990).

I acknowledge with pleasure my use of the departmental X-ray crystallographic facility, which is supervised by staff crystallographer Dr J. C. Gallucci.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1281). Services for accessing these data are described at the back of the journal.

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
- Boyd, R. N. & Hansen, R. H. (1953). *J. Am. Chem. Soc.* **75**, 5896–5897.
- Inamoto, K., Koikawa, M., Nakashima, M. & Tokii, T. (1996). *Inorg. Chim. Acta*, **249**, 251–254.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Molecular Structure Corporation (1992–1997). *TEXSAN*. Version 1.7. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Nonius (1999). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
- Steiner, T. & Desiraju, G. R. (1998). *J. Chem. Soc. Chem. Commun.* pp. 891–892.
- Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.