$C_8H_{17}NO_2$

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DL-3-Aminoisobutyric Acid Monohydrate

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Abstract

The title acid, 3-amino-2-methylpropanoic acid monohydrate, $C_4H_9NO_2 \cdot H_2O$, crystallized in the centrosymmetric space group *Pbca* in the zwitterionic form. The three H atoms on N, which are involved in hydrogen bonding, are ordered. The three intermolecular N—H$\cdots$O hydrogen bonds have N$\cdots$O distances ranging from 2.758 (2) to 2.809 (2) Å and N—H$\cdots$O angles ranging from 149 (2) to 171 (1)°. The two intermolecular O—H$\cdots$O hydrogen bonds have O$\cdots$O distances 2.739 (2) and 2.755 (2) Å, and O—H$\cdots$O angles 170 (2) and 175 (2)°. Each acid molecule and its associated water molecule are directly hydrogen bonded to five acid molecules and two water molecules; the structure comprises two subsets of molecules which are not cross-linked by these hydrogen bonds. Through basic second-level graphs, approximately two-thirds of the hydrogen-bonding patterns are finite and one-third are chains; there is a single ring pattern, which occurs about a center of symmetry.

Comment

This report on DL-3-aminoisobutyric acid monohydrate is one of a series on hydrogen bonding in amino-substituted carboxylic acids, and follows reports on a novel tetragonal phase of γ-aminobutyric acid and on 8-aminocaprylic acid (Dobson & Gerkin, 1996, 1998). The title acid crystallized in the centrosymmetric space group *Pbca* as a zwitterion, (I), the carboxyl proton having been transferred to the N atom. The refined mol-

$$\overset{+}{H_3N}-CH_2 \cdot CH(CH_3)-COO^- \cdot H_2O$$

(I)
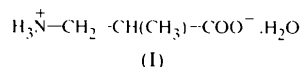
ecule, and the associated water molecule given in the atom list, are shown in Fig. 1, together with the atomic numbering scheme. As a consequence of the proton transfer, cyclic carboxylic acid group hydrogen-bonded dimers are not possible. Each potential acceptor atom (O1, O2, O3) and each potential H-atom participant (H1N, H2N, H3N, H1O, H2O) is involved in hydrogen bonding. In this structure, each acid molecule and its associated water molecule are directly hydrogen bonded to five acid molecules and two water molecules. The structure comprises two subsets of molecules which are not cross-linked by hydrogen bonds: molecules related by symmetry operations 1, 3, 5 and 7, and translations form one subset, while those related by 2, 4, 6 and 8, and translations form the other. The geometric details of the five hydrogen bonds are given in Table 2. The results of hydrogen-bond graph-set analysis (Bernstein *et al.*, 1995), involving the five hydrogen bonds (for this purpose designated *a* to *e*, in the order given in Table 2) for the first- and basic second-level graph sets, are presented in Table 3. This tabulation shows the predominance of finite patterns and chains among the hydrogen-bonding patterns. The five chains propagate along **b** (1) or **c** (4). The nine hydrogen bonds involving the homebase molecule and its associated water molecule are depicted and labeled in Fig. 2. The single ring pattern, which occurs about a center of symmetry, is apparent in Fig. 2.

It should be noted that there are six additional hydrogen bonds having donor$\cdots$acceptor distances very slightly greater than our customary cutoff (3.3 Å), but substantially greater than those included in Table 2, and having angles in the range 89–119°: these are N1—H1N$\cdots$O3^{iii} and N1—H3N$\cdots$O3^{iii}, N1—H1N$\cdots$O2^v and N1—H2N$\cdots$O2^v, and O3—H1O$\cdots$N1^{iii} and O3—H2O$\cdots$N1^{iii} [symmetry codes: (iii) $1-x$, $1-y$, $1-z$; (v) $1-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$]. These bonds make a secondary contribution to the structure.

Distances and angles of special interest in the title molecule are given in Table 1. Among the most comparable molecules for structural comparisons are γ-aminobutyric acid (Dobson & Gerkin, 1996), 8-aminocaprylic acid (Dobson & Gerkin, 1998) and 7-oxo-7-(phenylamino)heptanoic acid (Feeder & Jones, 1994). Whereas γ-aminobutyric acid and the title acid are partially folded and zwitterionic, the 8-aminocaprylic acid is extended and zwitterionic, while the substituted hep-
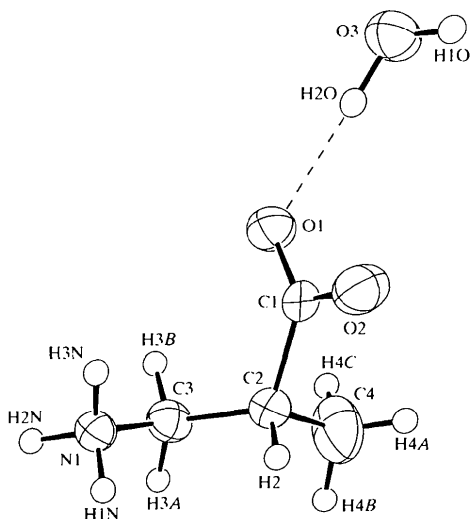
Fig. 1. *ORTEP*II (Johnson, 1976) drawing of DL-3-aminoisobutyric acid monohydrate showing the atomic numbering scheme. Displacement ellipsoids are drawn for 50% probability for all atoms except H atoms, for which they have been set artificially small.
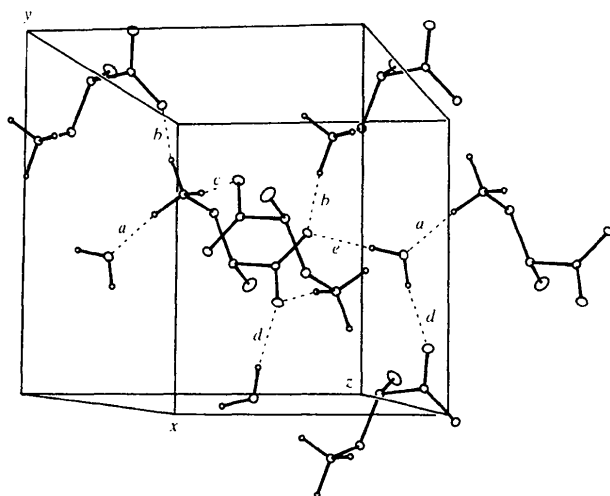

Fig. 2. *ORTEP*II (Johnson, 1976) diagram of a central DL-3-amino-isobutyric acid molecule with its associated water molecule, and the five acid molecules and two water molecules to which they are directly hydrogen bonded. For clarity, displacement ellipsoids are drawn artificially small for all atoms and all H atoms not involved in hydrogen bonding have been omitted. Intermolecular hydrogen bonds for the central acid molecule and its associated water molecule are shown as dashed lines labeled *a–e* in the order given in Table 2.

tanoic acid is extended but not zwitterionic. The observed values of the C1—C2 (present numbering) bond length are 1.483 (3) Å for the substituted heptanoic acid, but 1.519 (4), 1.520 (2) and 1.520 (2) Å for the butyric acid, 8-aminocaprylic acid and the title acid, respectively. Since in the heptanoic acid a neutral carboxyl group is present, while in the butyric acid, 8-amino-caprylic acid and the title acid a carboxylate ion is

present, the observed discrepancy in this bond length may well be systematic. Extensive comparison of hydrogen bonding in the title structure with that in γ-amino-butyric acid, for example, is precluded by the presence of water and its substantial involvement in hydrogen bonding in the title structure. It may be noted, however, that the geometric parameters of the N—H$\cdots$O$_{carboxyl}$ hydrogen bonds in γ-aminobutyric acid are quite similar to those of the N—H$\cdots$O$_{carboxyl}$ hydrogen bonds in the title structure. Thus, in (tetragonal) γ-amino-butyric acid, the three strongest N—H$\cdots$O$_{carboxyl}$ hydrogen bonds have N$\cdots$O distances ranging from 2.764 (3) to 2.797 (3) Å, and N—H$\cdots$O$_{carboxyl}$ angles ranging from 169 (3) to 178 (4)°, while in the present structure, the two N—H$\cdots$O$_{carboxyl}$ hydrogen bonds have N$\cdots$O$_{carboxyl}$ distances of 2.758 (2) and 2.799 (2) Å, and N—H$\cdots$O$_{carboxyl}$ angles of 166 (1) and 177 (1)°.

The closest intermolecular approaches in this structure, excluding pairs of atoms in groups hydrogen bonded to each other, occur between O3 and C3^{vi} [symmetry code: (vi) x, y, $1 + z$], and are 0.03 Å less than the corresponding Bondi (1964) van der Waals radius sum. No other close-approach distance is deficient with respect to the corresponding Bondi (1964) radius sum by as much as its s.u.

Experimental

DL-3-Aminoisobutyric acid was obtained as a white crystalline powder from the Aldrich Chemical Company. This solid was dissolved in ethanol at room temperature and the solution was filtered. Room-temperature evaporation of the filtrate produced chunks, one of which was cut to provide the experimental sample.

Crystal data

$C_4H_9NO_2.H_2O$	Mo $K\alpha$ radiation
$M_r = 121.14$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 25
$Pbca$	reflections
$a = 17.964 (2)$ Å	$\theta = 11.5–16.2°$
$b = 8.716 (1)$ Å	$\mu = 0.106$ mm^{-1}
$c = 8.172 (1)$ Å	$T = 296$ K
$V = 1279.5 (3)$ Å^3	Cut chunk
$Z = 8$	$0.35 \times 0.27 \times 0.23$ mm
$D_x = 1.258$ Mg m^{-3}	Colorless
D_m not measured	

Data collection

AFC-5S diffractometer	$h = 0 \rightarrow 23$
ω-2θ scans	$k = 0 \rightarrow 11$
Absorption correction: none	$l = -10 \rightarrow 10$
3259 measured reflections	6 standard reflections
1482 independent reflections	every 150 reflections
902 reflections with	intensity variation: $\pm 2.4\%$
$I > 2\sigma(I)$	(average maximum
$R_{int} = 0.031$	relative intensity)
$\theta_{max} = 27.56°$	

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.36$ e Å^{-3}
$R(F) = 0.040$	$\Delta\rho_{min} = -0.27$ e Å^{-3}
$wR(F^2) = 0.056$	Extinction correction:
$S = 1.87$	Zachariasen (1963, 1968)
1482 reflections	Extinction coefficient:
118 parameters	5.4 (8) $\times$ 10^{-7}
All H atoms refined	Scattering factors from
$w = 1/\sigma^2(F^2)$	Stewart et al. (1965) (H)
$(\Delta/\sigma)_{max} < 0.01$	and Creagh & McAuley
	(1992) (C, N, O)

Table 1. Selected geometric parameters (Å, °)

O1—C1	1.247(2)	C1—C2	1.520(2)
O2—C1	1.251(2)	C2—C3	1.522(2)
N1—C3	1.484(2)	C2—C4	1.528(3)
H1O—O3—H2O	114(1)	O2—C1—C2	117.6(1)
O1—C1—O2	124.3(1)	N1—C3—C2	112.3(1)
O1—C1—C2	118.0(1)		

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N1—H1N$\cdots$O3^i	0.96 (2)	1.95 (2)	2.809 (2)	149 (2)
N1—H2N$\cdots$O1^{ii}	1.05 (2)	1.73 (2)	2.758 (2)	166 (1)
N1—H3N$\cdots$O2^{iii}	0.97 (2)	1.83 (2)	2.799 (2)	171 (1)
O3—H1O$\cdots$O2^{iv}	0.86 (2)	1.90 (2)	2.755 (2)	170 (2)
O3—H2O$\cdots$O1	0.90 (2)	1.84 (2)	2.739 (2)	175 (2)

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

Table 3. First- and basic second-level graph-set descriptors involving hydrogen bonds, designated a–e in the order given in Table 2

	a	b	c	d	e
a	D	$D_2^2(5)$	$D_2^2(5)$	$C_2^2(8)$	$C_2^2(8)$
b		$C(6)$	$C_2^2(6)$	$D_3^3(5)$	$D_2^1(3)$
c			$R_2^2(12)$	$D_2^1(3)$	$D_2^2(5)$
d				D	$C_2^2(6)$
e					D

Scan widths were $(1.50 + 0.35\tan\theta)°$ in ω, with a background/scan time-ratio of 0.5. The data were corrected for Lorentz and polarization effects. The Laue group assignment, systematic absences and centrosymmetric intensity statistics indicated space group $Pbca$ (No. 61); since refinement proceeded well it was adopted. Fourier difference methods were used to locate the initial H-atom positions and the H atoms were then refined isotropically. The refined C—H distances ranged from 0.97 (2) to 1.06 (2) Å with a mean value 1.01 (3) Å; refined N—H and O—H distances are given in Table 2. The maximum effect of extinction is 2.3% of F_o for 200. The maximum positive residual peak is located ~0.3 Å from O1 and 1.1 Å from C1; the maximum negative peak is located ~0.7 Å from O3.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

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N-(3,5-Dichlorophenyl)naphthaldimine

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Abstract

Molecules of the title compound [1-(3,5-dichlorophenyliminomethyl)-2-naphthol, $C_{17}H_{11}Cl_2NO$] are nearly planar. Each contains a strong intramolecular N$\cdots$H—O hydrogen bond between the imine and hydroxyl groups [2.570 (3) Å].