Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Redetermination of L-valinium chloride monohydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.036
wR factor = 0.083
Data-to-parameter ratio = 13.6

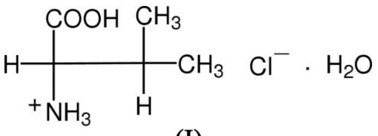
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 4 April 2006
Accepted 4 May 2006

The crystal structure of the title compound, $C_5H_{12}NO_2^+\cdot Cl^-\cdot H_2O$, has been reported previously by Rao [*Z. Kristallogr.* (1969), **128**, 339–341], with an R value of 0.11, without locating the H atoms. The present redetermination shows better agreement of bond lengths with expected values.

Comment

The crystal structure of the title compound, (I), was reported previously by Rao (1969) in the space group $P2_12_12_1$ (a = 6.85, b = 6.17, c = 21.22 Å and R = 0.11), without H atoms. Besides, as valine is a very important amino acid, the crystal structures of L-valine hydrochloride (Ando *et al.*, 1967), DL-valine hydrochloride (Blasio *et al.*, 1977) and L-valine L-valinium perchlorate monohydrate (Pandiarajan *et al.*, 2001) have also been reported. We now report a redetermination of (I) (Fig. 1) with higher precision, and the results are reported here.



(I)

The C—O bond lengths in the present determination (Table 1) are indicative of a typical carboxylic acid, but these lengths observed in the previous determination (1.303 and 1.273 Å) are close to those of a carboxylate group (Borthwick, 1980). The C—C_{methyl} bond lengths in the previous determination deviate significantly from the normal value of 1.530 Å (1.583 and 1.493 Å; Rao, 1969), whereas the present values agree with it. Analysis of the previously published structure indicate that the coordinates of the valinium residue deposited (CSD refcode VALHCL10) in the Cambridge Structural Database (CSD; Version 5.26; Allen, 2002) correspond to the D configuration.

In the crystal structure, the L-valinium residue is linked to the water molecule and the Cl^- anion by N—H···O, N—H···Cl and O—H···Cl hydrogen bonds, forming layers parallel to the ab plane (Fig. 2 and Table 2). No significant interaction is observed between adjacent layers.

Experimental

L-Valine and hydrochloric acid in equal molar ratio were mixed together with enough water, and heated to a temperature at which a clear solution resulted. Colourless single crystals of (I) were obtained from the solution by slow evaporation of water at room temperature for 5 d.

Crystal data

$C_5H_{12}NO_2^+ \cdot Cl^- \cdot H_2O$
$M_r = 171.62$
Orthorhombic, $P2_12_12_1$
$a = 6.155$ (1) Å
$b = 6.828$ (1) Å
$c = 21.099$ (3) Å
$V = 886.7$ (2) Å^3
$Z = 4$
$D_x = 1.286$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.39$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.48 × 0.36 × 0.32 mm

Data collection

Siemens P4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.845$, $T_{\max} = 0.883$
2442 measured reflections
2020 independent reflections
1704 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.023$
$\theta_{\max} = 27.5°$
3 standard reflections every 97 reflections intensity decay: 1.6%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.083$
$S = 0.97$
2020 reflections
148 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.30$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.065 (5)
Absolute structure: Flack (1983), 815 Friedel pairs
Flack parameter: 0.01 (8)

Table 1
Selected bond lengths (Å).

O1—C5	1.315 (2)	C2—C3	1.522 (3)
O2—C5	1.190 (3)	C3—C4	1.537 (3)
N1—C4	1.490 (3)	C4—C5	1.514 (3)
C1—C3	1.523 (4)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1⋯Cl1$^{\rm i}$	0.81 (3)	2.20 (3)	3.003 (2)	171 (3)
O3—H3B⋯Cl1$^{\rm ii}$	0.83 (4)	2.34 (4)	3.171 (2)	178 (4)
O3—H3A⋯Cl1$^{\rm iii}$	0.85 (5)	2.38 (5)	3.187 (2)	159 (5)
N1—H1A⋯O2	0.85 (3)	2.35 (3)	2.677 (3)	103 (2)
N1—H1A⋯O3	0.85 (3)	2.03 (3)	2.866 (3)	167 (3)
N1—H1B⋯O2$^{\rm iv}$	0.94 (3)	2.44 (3)	2.920 (3)	111 (2)
N1—H1B⋯O3$^{\rm iv}$	0.94 (3)	1.96 (3)	2.876 (3)	163 (3)
N1—H1C⋯Cl1	0.86 (2)	2.44 (2)	3.246 (2)	156 (2)
C3—H3⋯O1	0.97 (2)	2.51 (2)	2.915 (3)	105 (2)

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

All H atoms were located in a difference Fourier map and refined freely.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

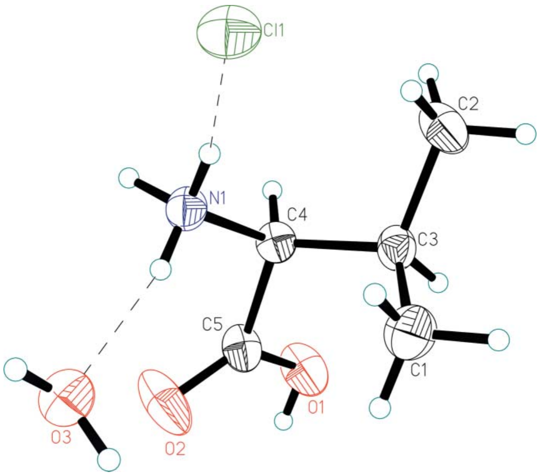


Figure 1
The asymmetric unit of (I), showing 40% probability displacement ellipsoids. Hydrogen bonds are indicated by dashed lines.

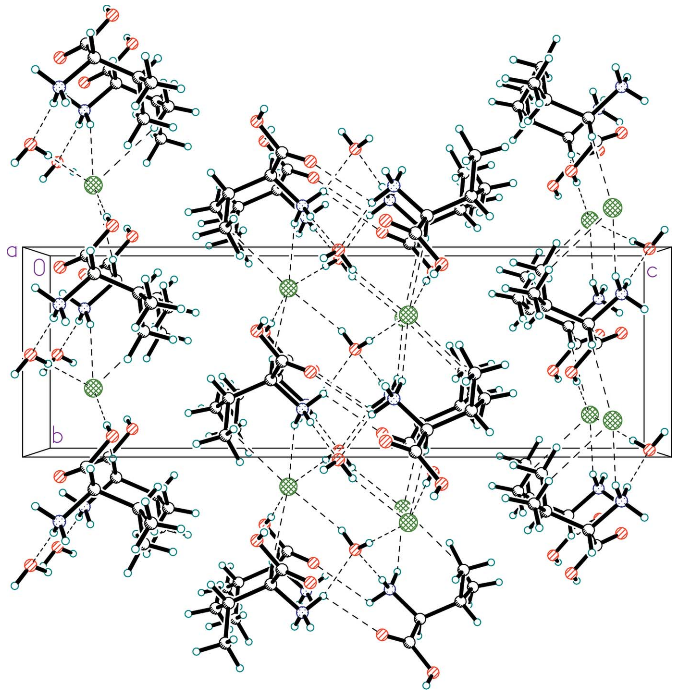


Figure 2
The crystal packing of (I), viewed along the *a* axis. Hydrogen bonds are indicated by dashed lines.

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o2236 Li *et al.* • $C_5H_{12}NO_2^+ \cdot Cl^- \cdot H_2O$ *Acta Cryst.* (2006). E**62**, o2235–o2236