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Redetermination of 1-carboxycyclohexan-1-aminium chloride

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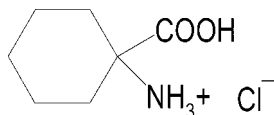
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.033; wR factor = 0.088; data-to-parameter ratio = 9.9.

The crystal structure of the title compound, $\text{C}_7\text{H}_{14}\text{NO}_2^+\cdot\text{Cl}^-$, was reported previously [Chacko, Srinivasan & Zand (1975). *J. Cryst. Mol. Struct.* **5**, 353–357] from Weissenberg photographic data with $R = 0.113$. It has now been redetermined, providing a significant increase in the precision of the derived geometric parameters, viz. mean $\sigma(\text{C}-\text{C}) = 0.003$ Å in the present work compared with 0.021 Å for the previous work. The complete cation is generated by crystallographic mirror symmetry, with three C atoms, two O atoms and the N atom lying on the reflecting plane; the chloride anion also has m site symmetry. The crystal structure is established by a two-dimensional network of $\text{O}-\text{H}\cdots\text{Cl}$ and $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds, generating $C_2^1(4)$ and $C_2^1(7)$ chains, and $R_4^2(8)$ and $R_4^2(14)$ rings.

Related literature

For the earlier structure determination of the title salt, see: Chacko *et al.* (1971, 1975). For related literature, see Rodríguez-Ropero *et al.* (2008). For the crystal structure of the pure amino acid, see: Valle *et al.* (1988). For ring conformation analysis, see: Cremer & Pople (1975). For hydrogen-bond motifs in graph-set notation, see: Etter (1990).



Experimental

Crystal data

$\text{C}_7\text{H}_{14}\text{NO}_2^+\cdot\text{Cl}^-$
 $M_r = 179.64$
 Monoclinic, $P2_1/m$

$a = 7.382$ (3) Å
 $b = 6.357$ (2) Å
 $c = 9.374$ (3) Å

$\beta = 96.239$ (10)°
 $V = 437.3$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation

$\mu = 0.39$ mm⁻¹
 $T = 298$ (2) K
 $0.31 \times 0.27 \times 0.18$ mm

Data collection

Rigaku AFC-7S Mercury diffractometer
 Absorption correction: multi-scan (Jacobson, 1998)
 $T_{\min} = 0.880$, $T_{\max} = 0.930$

4638 measured reflections
 845 independent reflections
 789 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.088$
 $S = 1.01$
 845 reflections
 85 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.18$ e Å⁻³
 $\Delta\rho_{\min} = -0.15$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1B}\cdots\text{Cl}^{\text{i}}$	0.90 (3)	2.34 (3)	3.196 (2)	158 (2)
$\text{N1}-\text{H1A}\cdots\text{Cl}^{\text{i}}$	0.88 (2)	2.58 (2)	3.3816 (13)	152.4 (17)
$\text{O1}-\text{H1}\cdots\text{Cl}^{\text{ii}}$	0.89 (4)	2.15 (4)	3.027 (2)	168 (3)

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

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *PLATON* (Spek, 2003) and *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2212).

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supplementary materials

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Redetermination of 1-carboxycyclohexan-1-aminium chloride

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Comment

1-Amino-cyclohexanecarboxylic acid is a promising amino acid candidate to serve as basic piece in redesigned protein motifs which constitute the basic modules in synthetic nanoconstructs (Rodríguez-Ropero *et al.*, 2008). Its structure was reported by Valle *et al.* (1988). The title compound, (I), 1-amino-cyclohexanecarboxylic acid hydrochloride, $C_7H_{14}NO_2^+ \cdot Cl^-$, was first reported in the noncentric space group $P2_1$ (Chacko *et al.*, 1971) and later reported in the centrosymmetric space group $P2_1/m$ (Chacko *et al.*, 1975) with $R = 0.113$. The present paper reports a redetermination of the crystal structure of (I), with greater precision and accuracy. Both, the cation and anion are located on a mirror plane, which confirms the space group $P2_1/m$ instead of $P2_1$. In this compound, the cyclohexane ring adopts a chair conformation, with the ammonium and carboxylate groups in axial and equatorial positions, respectively (Cremer & Pople, 1975), while the pure amino acid has an opposite conformation (Valle *et al.*, 1988). In (I), 1-amino-cyclohexanecarboxylic acid is protonated and is linked to the Cl^- anion by a $O-H \cdots Cl$ hydrogen bond (Fig. 1, Table 1). The hydrogen bonds $O1-H1 \cdots Cl1$ ($x, y, z - 1$) and $N1-H1B \cdots Cl1$ ($1 - x, y - 1/2, 1 - z$) form infinite chains running along the $[001]$ direction (Fig. 2) and may be described in graph-set notation as $C^1_2(7)$ (Etter, 1990). The intramolecular hydrogen bonds $N1-H1A \cdots Cl1$ form infinite chains, with graph-set $C^1_2(4)$, running along the b cell axis. The combination of these interactions produces rings with graph-set $R^2_4(8)$ and $R^2_4(14)$, parallel to the bc plane (Fig. 2).

Experimental

1-Amino-cyclohexanecarboxylic acid and hydrochloric acid in equal molar ratio were mixed together with enough water, and heated to a temperature where a clear solution was obtained. Colorless crystals of (I) suitable for X-ray diffraction analysis were grown by slow evaporation of this solution.

Refinement

All H atoms were located in a difference map and their positions were freely refined, with the $U_{iso}(H)$ values set at $1.2U_{eq}(\text{carrier C})$, $1.5U_{eq}(\text{carrier O})$ and $1.5U_{eq}(\text{carrier N})$, respectively.

Figures

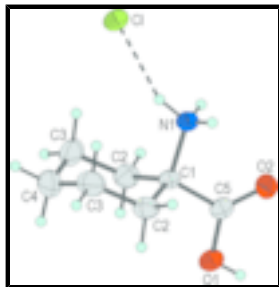


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

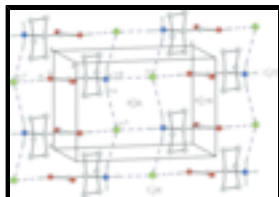


Fig. 2. A partial packing view of (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity. Symmetry codes: (i) $x, y, z - 1$; (ii) $1 - x, y - 1/2, 1 - z$.

1-carboxycyclohexan-1-aminium chloride

Crystal data



$$M_r = 179.64$$

Monoclinic, $P2_1/m$

Hall symbol: $-P\ 2y$

$$a = 7.382\ (3)\ \text{\AA}$$

$$b = 6.357\ (2)\ \text{\AA}$$

$$c = 9.374\ (3)\ \text{\AA}$$

$$\beta = 96.239\ (10)^\circ$$

$$V = 437.3\ (3)\ \text{\AA}^3$$

$$Z = 2$$

$$F_{000} = 192$$

$$D_x = 1.364\ \text{Mg m}^{-3}$$

Mo $K\alpha$ radiation

$$\lambda = 0.71070\ \text{\AA}$$

Cell parameters from 1650 reflections

$$\theta = 2.2\text{--}27.2^\circ$$

$$\mu = 0.39\ \text{mm}^{-1}$$

$$T = 298\ (2)\ \text{K}$$

Block, colourless

$$0.31 \times 0.27 \times 0.18\ \text{mm}$$

Data collection

Rigaku AFC-7S Mercury diffractometer

Radiation source: Normal-focus sealed tube

Monochromator: graphite

Detector resolution: $14.6306\ \text{pixels mm}^{-1}$

$$T = 298\ (2)\ \text{K}$$

ω scans

Absorption correction: multi-scan (Jacobson, 1998)

$$T_{\min} = 0.880, T_{\max} = 0.930$$

4638 measured reflections

845 independent reflections

789 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.023$$

$$\theta_{\max} = 25.0^\circ$$

$$\theta_{\min} = 2.2^\circ$$

$$h = -8 \rightarrow 8$$

$$k = -6 \rightarrow 7$$

$$l = -11 \rightarrow 11$$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.033$	$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.1831P]$
$wR(F^2) = 0.088$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{\max} < 0.001$
845 reflections	$\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$
85 parameters	$\Delta\rho_{\min} = -0.15 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.042 (10)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl	0.41343 (8)	0.2500	0.64377 (6)	0.0414 (3)
O2	0.5446 (2)	0.2500	0.04758 (18)	0.0511 (5)
O1	0.2629 (3)	0.2500	-0.06850 (18)	0.0528 (5)
H1	0.324 (5)	0.2500	-0.146 (4)	0.079*
N1	0.4370 (3)	0.2500	0.3054 (2)	0.0343 (5)
H1A	0.506 (3)	0.138 (3)	0.300 (2)	0.051*
H1B	0.398 (4)	0.2500	0.393 (3)	0.051*
C1	0.2863 (3)	0.2500	0.1837 (2)	0.0292 (5)
C2	0.1715 (2)	0.4484 (3)	0.19289 (19)	0.0408 (5)
H2A	0.251 (3)	0.566 (3)	0.1923 (19)	0.049*
H2B	0.090 (3)	0.452 (3)	0.107 (2)	0.049*
C3	0.0597 (3)	0.4450 (4)	0.3193 (2)	0.0539 (6)
H3A	0.140 (3)	0.449 (4)	0.409 (2)	0.065*
H3B	-0.018 (3)	0.571 (4)	0.315 (2)	0.065*
C4	-0.0557 (4)	0.2500	0.3201 (3)	0.0626 (9)
H4A	-0.145 (5)	0.2500	0.232 (4)	0.075*
H4B	-0.148 (5)	0.2500	0.397 (4)	0.075*
C5	0.3819 (3)	0.2500	0.0475 (2)	0.0358 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl	0.0457 (4)	0.0544 (4)	0.0238 (4)	0.000	0.0025 (2)	0.000
O2	0.0359 (10)	0.0837 (14)	0.0345 (10)	0.000	0.0078 (7)	0.000
O1	0.0443 (11)	0.0932 (15)	0.0205 (9)	0.000	0.0023 (7)	0.000
N1	0.0335 (11)	0.0449 (12)	0.0238 (10)	0.000	-0.0002 (8)	0.000
C1	0.0303 (11)	0.0355 (12)	0.0211 (11)	0.000	-0.0004 (8)	0.000

supplementary materials

C2	0.0439 (10)	0.0402 (10)	0.0373 (10)	0.0074 (8)	-0.0004 (8)	0.0024 (7)
C3	0.0497 (11)	0.0707 (14)	0.0412 (10)	0.0221 (10)	0.0052 (9)	-0.0058 (10)
C4	0.0386 (15)	0.105 (3)	0.0449 (17)	0.000	0.0076 (13)	0.000
C5	0.0385 (13)	0.0425 (13)	0.0262 (12)	0.000	0.0025 (10)	0.000

Geometric parameters (Å, °)

O2—C5	1.201 (3)	C2—C3	1.516 (3)
O1—C5	1.322 (3)	C2—H2A	0.95 (2)
O1—H1	0.89 (4)	C2—H2B	0.95 (2)
N1—C1	1.504 (3)	C3—C4	1.505 (3)
N1—H1A	0.88 (2)	C3—H3A	0.97 (2)
N1—H1B	0.90 (3)	C3—H3B	0.98 (2)
C1—C5	1.524 (3)	C4—H4A	0.99 (3)
C1—C2	1.528 (2)	C4—H4B	1.05 (4)
C5—O1—H1	109 (2)	C4—C3—C2	111.85 (19)
C1—N1—H1A	110.2 (13)	C4—C3—H3A	107.9 (13)
C1—N1—H1B	114.0 (18)	C2—C3—H3A	109.9 (12)
H1A—N1—H1B	107.1 (16)	C4—C3—H3B	110.2 (13)
N1—C1—C5	105.28 (18)	C2—C3—H3B	108.4 (13)
N1—C1—C2	109.04 (12)	H3A—C3—H3B	108.5 (17)
C5—C1—C2	110.97 (12)	C3—C4—H4A	108.6 (10)
C3—C2—C1	112.64 (16)	C3—C4—H4B	114.6 (8)
C3—C2—H2A	113.8 (11)	H4A—C4—H4B	99 (3)
C1—C2—H2A	107.7 (12)	O2—C5—O1	125.2 (2)
C3—C2—H2B	108.3 (11)	O2—C5—C1	123.6 (2)
C1—C2—H2B	105.8 (12)	O1—C5—C1	111.2 (2)
H2A—C2—H2B	108.2 (16)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1B \cdots C1	0.90 (3)	2.34 (3)	3.196 (2)	158 (2)
N1—H1A \cdots C1 ⁱ	0.88 (2)	2.58 (2)	3.3816 (13)	152.4 (17)
O1—H1 \cdots C1 ⁱⁱ	0.89 (4)	2.15 (4)	3.027 (2)	168 (3)

Symmetry codes: (i) $-x+1, y-1/2, -z+1$; (ii) $x, y, z-1$.

Fig. 1

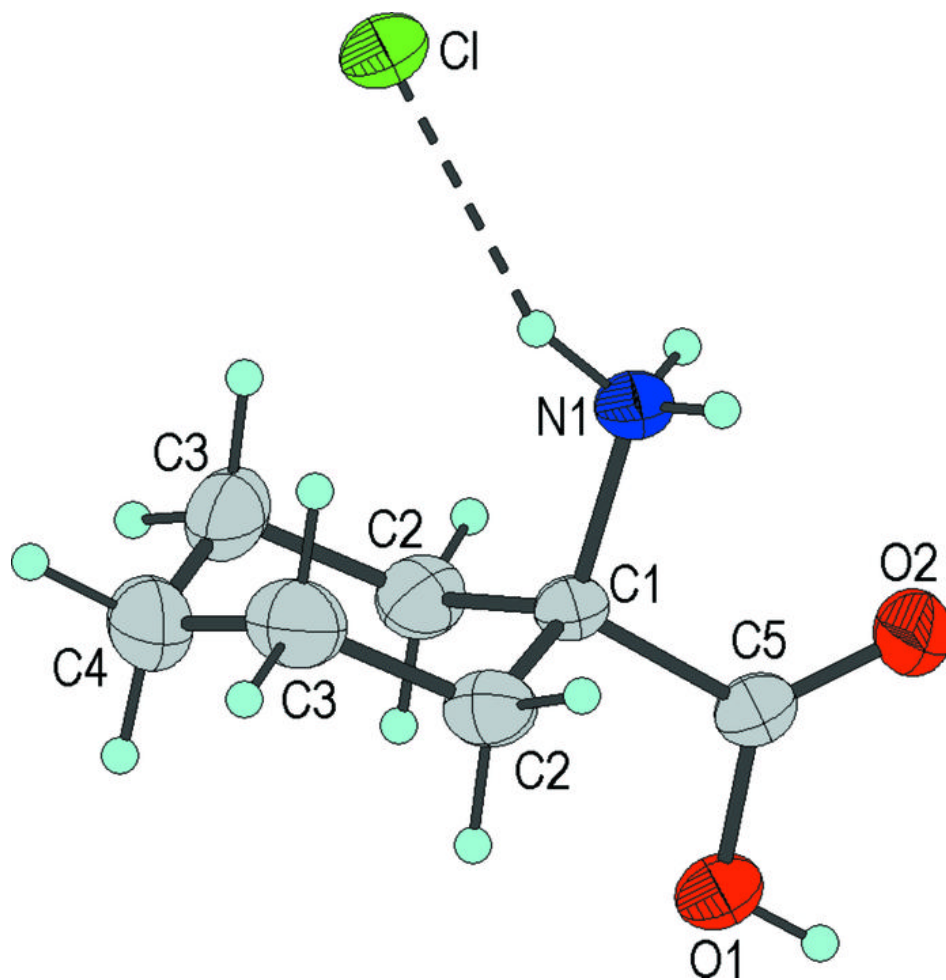


Fig. 2

