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# Redetermination of 1-carboxycyclohexan-1-aminium chloride

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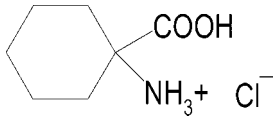
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.003 Å; $R$ factor = 0.033; $wR$ factor = 0.088; data-to-parameter ratio = 9.9.

The crystal structure of the title compound, $C_7H_{14}NO_2^+\cdot Cl^-$, 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 $\sigma$(C—C) = 0.003 Å in the present work compared with 0.021 Å for the previous work. The complete cation is generated by crystallographic mirrror 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 O—H···Cl and N—H···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

$C_7H_{14}NO_2^+\cdot Cl^-$
$M_r$ = 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) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.39 mm$^{-1}$
$T$ = 298 (2) K
0.31 × 0.27 × 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 $I > 2\sigma(I)$
$R_{int}$ = 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···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$B$···Cl | 0.90 (3) | 2.34 (3) | 3.196 (2) | 158 (2) |
| N1—H1$A$···Cl$^{i}$ | 0.88 (2) | 2.58 (2) | 3.3816 (13) | 152.4 (17) |
| O1—H1···Cl$^{ii}$ | 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/MSC, 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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# supporting information

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## Redetermination of 1-carboxycyclohexan-1-aminium chloride

**Lusbely M. Belandria, Gerzon E. Delgado, Asiloé J. Mora, Luis E. Seijas and Teresa González**

### S1. 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). It structure was reported by Valle *et al.* (1988). The title compound, (I), 1-amino-cyclohexanecarboxylic acid hydrochloride, $C_7H_{14}NO_2^+.Cl^-$, 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-cyclohexane-carboxylic acid is protonated and is linked to the $Cl^-$ anion by a O—H···Cl hydrogen bond (Fig. 1, Table 1). The hydrogen bonds O1—H1···Cl1 ($x$, $y$, $z$ - 1) and N1—H1B···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···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).

### S2. 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.

### S3. Refinement

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

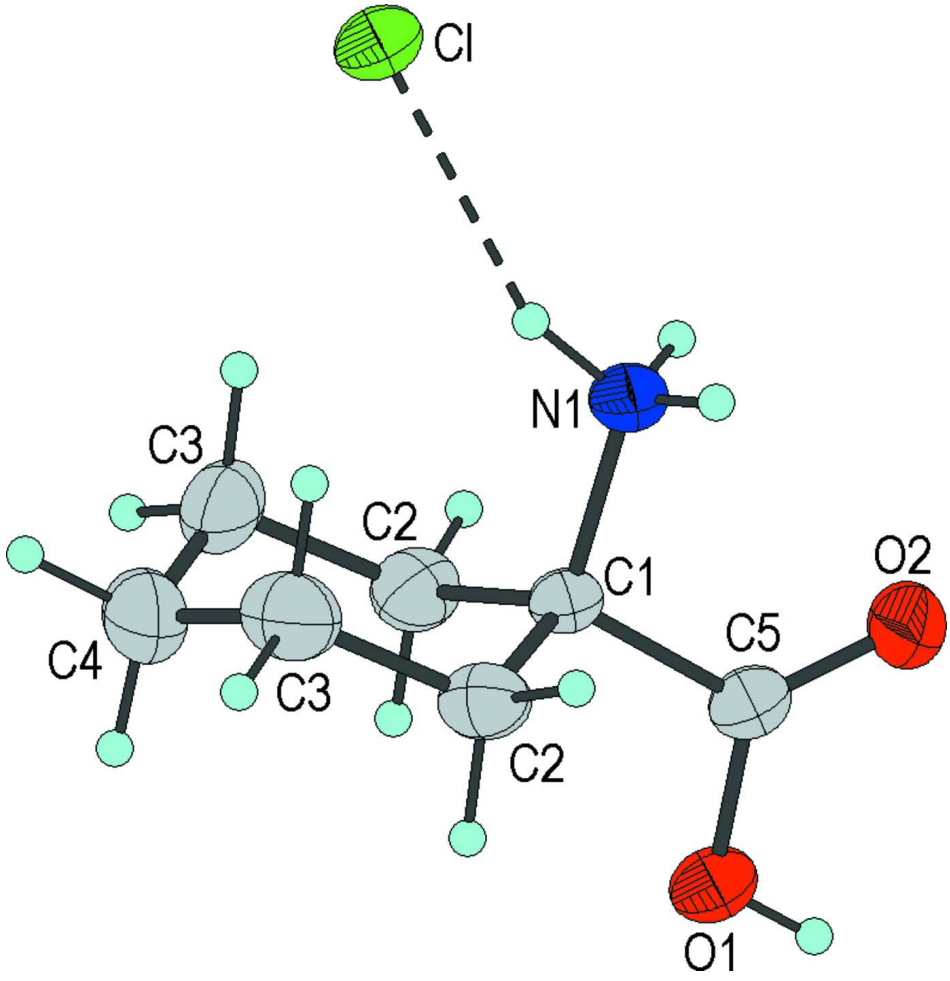


**Figure 1**

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

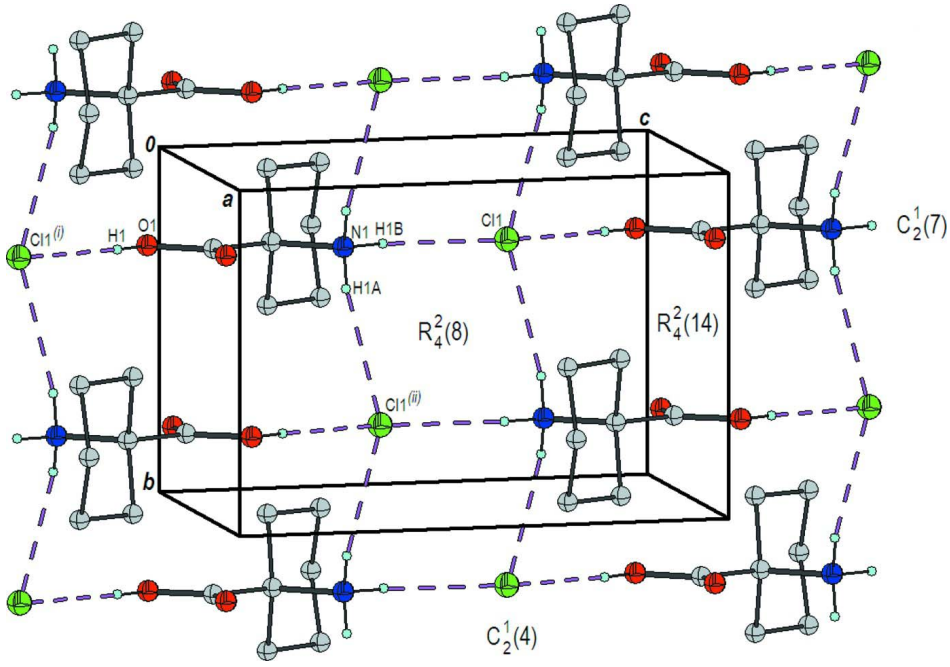


**Figure 2**

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*

$C_7H_{14}NO_2^+ \cdot Cl^-$
$M_r$ = 179.64
Monoclinic, $P2_1/m$
Hall symbol: -P 2yb
$a$ = 7.382 (3) Å
$b$ = 6.357 (2) Å
$c$ = 9.374 (3) Å
$\beta$ = 96.239 (10)°
$V$ = 437.3 (3) Å$^3$
$Z$ = 2
$F(000)$ = 192
$D_x$ = 1.364 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71070 Å
Cell parameters from 1650 reflections
$\theta$ = 2.2–27.2°
$\mu$ = 0.39 mm$^{-1}$
$T$ = 298 K
Block, colourless
0.31 × 0.27 × 0.18 mm

*Data collection*

Rigaku AFC-7S Mercury diffractometer
Radiation source: Normal-focus sealed tube
Graphite monochromator
Detector resolution: 14.6306 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (Jacobson, 1998)
$T_{min}$ = 0.880, $T_{max}$ = 0.930
4638 measured reflections
845 independent reflections
789 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.023
$\theta_{max}$ = 25.0°, $\theta_{min}$ = 2.2°
$h$ = −8→8
$k$ = −6→7
$l$ = −11→11

*Refinement*

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

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| 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 (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 |
| 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, º)*

| | | | |
|---|---|---|---|
| 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, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1*B*···Cl | 0.90 (3) | 2.34 (3) | 3.196 (2) | 158 (2) |
| N1—H1*A*···Cl$^{i}$ | 0.88 (2) | 2.58 (2) | 3.3816 (13) | 152.4 (17) |
| O1—H1···Cl$^{ii}$ | 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.