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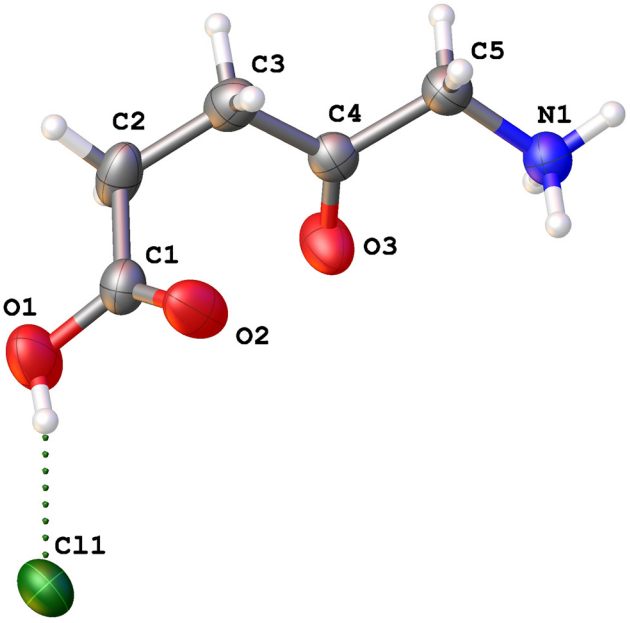
The crystal structure of 4-carboxy-2-oxobutan-1-aminium chloride, $C_5H_{10}ClNO_3$



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Abstract

$C_5H_{10}ClNO_3$, orthorhombic, *Pbca* (no. 61), a = 8.2012(1) Å, b = 11.2215(2) Å, c = 16.8339(3) Å, V = 1549.22(4) Å^3, Z = 8, $R_{gt}(F)$ = 0.0473, w$R_{ref}(F^2)$ = 0.1331, T = 297 K.

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A part of the molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.30 × 0.20 × 0.20 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	4.02 mm^{-1}
Diffractometer, scan mode:	Rigaku Synergy R, ω scans
θ_{max}, completeness:	76.5°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7352, 1555, 0.044
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1,431
$N(param)_{refined}$:	103
Programs:	Rigaku[1], Olex2[2], SHELX[3,4]

1 Source of materials

The compound was obtained commercially (Sigma–Aldrich). Crystals suitable for the diffraction test were taken directly from the provided product.

2 Experimental details

The positions of hydrogen atoms on carbon atoms were calculated geometrically and refined using the riding model. Their coordinates and displacement parameters were constrained to ride on the carrier atom (C–H = 0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) for alkyl H atoms). All the hydrogen atoms on oxygen and nitrogen atoms were located from difference Fourier map inspection and refined with U_{iso}(H) = 1.5U_{eq}(O) or 1.5U_{eq}(N). The distance between the hydrogen atom and the oxygen atom or the nitrogen atoms were refined freely.

3 Comment

5-Amino-4-oxopentanoic acid hydrochloride, more commonly known as aminolevulinic acid (ALA) hydrochloride, serves as a crucial precursor in the synthesis of hemoglobin within the human body.[5] Additionally, it has emerged as a second-generation photosensitizer.[6] In the year 2000, DUSA, an American company, pioneeringly formulated this compound into a photosensitizer drug named Levulan, which subsequently gained marketing approval.[7] Despite an extensive search through the Cambridge Structural Database, no reports on the single crystal

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

	x	*y*	*z*	U_{iso}^*/U_{eq}
Cl1	0.41334 (5)	0.88078 (4)	0.58821 (3)	0.0444 (2)
O1	0.63945 (17)	0.67785 (15)	0.63696 (11)	0.0609 (4)
H1	0.564 (5)	0.731 (3)	0.6246 (18)	0.091*
O2	0.41435 (13)	0.57878 (14)	0.66580 (11)	0.0552 (4)
O3	0.54646 (18)	0.34965 (13)	0.55367 (8)	0.0488 (4)
N1	0.33971 (16)	0.16099 (14)	0.55573 (8)	0.0379 (3)
H1A	0.412 (3)	0.154 (3)	0.5147 (16)	0.057*
H1B	0.256 (3)	0.219 (2)	0.5457 (13)	0.057*
H1C	0.298 (3)	0.088 (3)	0.5603 (14)	0.057*
C1	0.55990 (19)	0.58159 (16)	0.66041 (10)	0.0369 (4)
C2	0.67040 (19)	0.48050 (17)	0.68252 (12)	0.0453 (4)
H2A	0.727836	0.500551	0.731085	0.054*
H2B	0.750938	0.469668	0.640946	0.054*
C3	0.5791 (2)	0.36521 (17)	0.69452 (11)	0.0416 (4)
H3A	0.650080	0.308941	0.721277	0.050*
H3B	0.486462	0.379989	0.728989	0.050*
C4	0.51904 (17)	0.30989 (14)	0.61877 (9)	0.0335 (4)
C5	0.42334 (19)	0.19617 (16)	0.62962 (10)	0.0385 (4)
H5A	0.496634	0.132893	0.645929	0.046*
H5B	0.343278	0.207294	0.671381	0.046*

structure of the ALA molecule were identified. The sole derivative of ALA was a 3-fluorine substituted variant, which crystalized in $P2_1$ space group (CCDC No. 2351622).[8]

The titled compound crystallized in *Pbca* space group with one ALA cation and one chloride anion in its asymmetric unit, forming a 1:1 organic salt. The C–C bond length remains essentially consistent, approximately 1.50 Å. The C–O bonds within the carboxyl group measure 1.322(2) Å and 1.198(2) Å, respectively. It is confirmed that the terminal group is indeed a carboxyl group, rather than a carboxylate group. The carbon chain comprising C2, C3, C4, and C5 exhibits a typical zipper-like configuration. The twist angle between C2–C3–C4–C5 is –178.41(13)°, suggesting that above atoms are nearly coplanar. The torsion angle of C1–C2–C3–C4 is 72.77(19)°, while the torsion angle of C3–C4–C5–N1 is 167.99(13)°. These torsion angle values demonstrate that N1 and the carboxyl group do not reside in the aforementioned plane. Protonated amino groups establish hydrogen bonds with three chloride ions, and the D···A distances of these hydrogen bonds are about 3.2 Å. The terminal carboxyl group also engages in hydrogen bonding with chloride ions, characterized by a D···A distance of 3.0493(17) Å. The chloride ions function as a bonding agent, linking ALA cations together to create a complex three-dimensional network structure.

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Author contribution: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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