organic compounds

L-Glutamic acid hydrochloride at 153 K

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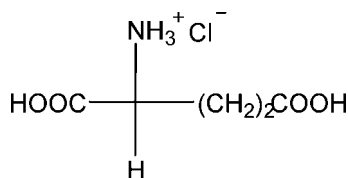Correspondence e-mail: zgdlzg@sina.com

The title compound [systematic name: (*S*)-1,3-dicarboxy-propanaminium chloride], $C_5H_{10}NO_4^+\cdot Cl^-$, has been investigated previously by Dawson [*Acta Cryst.* (1953). **6**, 81–83], with R = 0.106 and without the location of H atoms, and then by Sequeira, Rajagopal & Chidambaram [*Acta Cryst.* (1972). B**28**, 2514–2519] using neutron diffraction with R = 0.043. The present determination at 153 K has R = 0.017 and all the H atoms are located. There are obvious differences in some C—C bond lengths between the present and previous studies. In the present structure, L-glutamic acid is protonated and is linked to the Cl⁻ anion by an O—H···Cl hydrogen bond. The crystal structure is established by a three-dimensional network of O—H···O, N—H···O and N—H···Cl hydrogen bonds.

Related literature

For related literature, see: Delfino *et al.* (1978); Kirfel & Wallrafen (1985). For previous structure determinations, see: Dawson (1953) [Cambridge Structural Database (CSD; Version 5.26; Allen, 2002) refcode LGLUTA01]; Sequeira *et al.* (1972) (refcode LGLUTA).

Experimental

Crystal data

$C_5H_{10}NO_4^+\cdot Cl^-$	V = 786.73 (4) Å³
M_r = 183.59	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
a = 5.1016 (1) Å	μ = 0.45 mm⁻¹
b = 11.6386 (4) Å	T = 153 (2) K
c = 13.2500 (3) Å	0.49 × 0.48 × 0.35 mm

Data collection

Siemens P4 diffractometer	7742 measured reflections
Absorption correction: ψ scan (*SHELXTL*; Bruker, 1998)	1798 independent reflections
T_{min} = 0.808, T_{max} = 0.858	1775 reflections with $I > 2\sigma(I)$
	R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.019	$\Delta\rho_{max}$ = 0.29 e Å⁻³
$wR(F^2)$ = 0.055	$\Delta\rho_{min}$ = −0.25 e Å⁻³
S = 1.15	Absolute structure: Flack (1983),
1798 reflections	725 Friedel pairs
101 parameters	Flack parameter: 0.02 (4)
H-atom parameters constrained	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···Cl1	0.91	2.29	3.1406 (10)	157
N1—H1B···Cl1ⁱ	0.91	2.34	3.1890 (10)	155
N1—H1C···O2ⁱⁱ	0.91	2.04	2.8700 (13)	151
O1—H1···O3ⁱⁱⁱ	0.84	1.79	2.6208 (13)	170
O4—H4···Cl1ⁱᵛ	0.84	2.21	3.0389 (9)	168

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; 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: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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supplementary materials

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Comment

The crystal structure of the title compound has been investigated by Dawson (1953) using the multiple-film technique for *h*0*l* and 0*kl* intensity data collection, then by Sequeira *et al.* (1972) using neutron diffraction. Besides, the *L*-glutamic acid (LGA) halogen acid salts of LGA hydroiodide (Kirfel & Wallrafen, 1985), LGA HBr and LGA HCl HBr (Delfino *et al.*, 1978) have been reported before. Recently we have made a structure determination of the title compound at 153 K and make comparisons with Dawson's and Sequeira's studies, hereby.

The unit-cell parameters and space group of the title compound (see crystal data) and the respective values in Dawson's structure [a = 5.16 (1), b = 11.80 (2), c = 13.30 (2) Å] and Sequeira's structure [a = 5.151 (6), b = 11.789 (19), c = 13.347 (20) Å] demonstrate the high quality of modern X-ray diffractometer and software. The final *R* value of the present case [0.0171] is much smaller than that of Dawson's [0.106] and Sequeira's [0.043]. The hydrogen atoms were not determined in Dawson's study, due to the incompleteness of the estimated intensity data.

The C4—C5 [1.5066 (15) Å] bond length intermediates Dawson's [1.54 Å] and Sequeira's [1.475 (7) Å] (Table 1). The C2—C3 distance [1.5312 (15) Å] differs from that of Dawson's [1.55 Å], the C1—C2 [1.5156 (16) Å] deviates from that of Sequeira's [1.535 (7) Å]. The C—O, C—N and the other C—C bond lengths of these three cases have little distinctions respectively. Between the present and Dawson's study, the major distinctions of bond angles lie in C1—C2—N1 and C3—C2—N1 [108.02 (9) and 111.94 (9)° for present, 111 and 110° for Dawson's case, respectively]. The torsion angles of the main skeleton are finely coherent with those in Sequeira's study, respectively (Table 1). But, some torsion angles related to H atoms that are involed in hydrogen bonds are deviated greatly from those in Sequeira's study, respectively [H1C—N1—C2—C1 (−60.0, −48.0°), H1B—N1—C2—C1 (60.0, 73.2°) and H1A—N1—C2—C1 (−180.0, −165.1°); H1—O1—C1—C2 (179.8, 168.9°), the former and latter values for the present (Table 1) and Sequeira's study, repectively].

In the title compound, *L*-glutamic acid is protonated and is linked with the Cl⁻ anion by a O—H···Cl hydrogen bond (Fig. 1, Table 2). The crystal structure is established by a three-dimensional network of O—H···O, N—H···O and N—H···Cl classic hydrogen bonds (Fig. 2, Table 2).

Experimental

L-Glutamic acid and hydrochloric acid in equal molar ratio were mixed together with enough water, and heated to a temperatur where clear solution was obtained. The solution were layed in a ventilative place. Colorless single crystals of the title compound were obtained from the solution by slow volatilization of water at room temperature for 7 days.

Refinement

All hydrogen atoms were placed in calculated positions and allowed to ride on their parent atoms at distances of 0.99 (methylene), 1.00 (methine), 0.91Å (N—H), 0.84Å (O—H), with U_{iso}(H) values 1.2–1.5 times U_{eq} of the parent atoms.
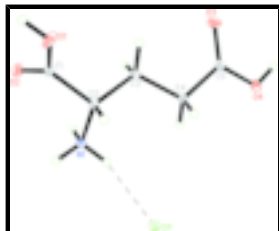
supplementary materials

Figures

Fig. 1. A view of the title compound showing with 40% probability displacement ellipsoids. Hydrogen bonds are indicated by thin lines.
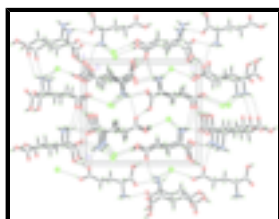

Fig. 2. The packing diagram of the title compound viewed down along the *a* axis. Hydrogen bonds are indicated by thin lines.

(*S*)-1,3-dicarboxypropanaminium chloride

Crystal data

$C_5H_{10}NO_4^+\cdot Cl^-$	$F_{000} = 384$
$M_r = 183.59$	$D_x = 1.550$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 7746 reflections
$a = 5.1016 (1)$ Å	$\theta = 3.1–27.5º$
$b = 11.6386 (4)$ Å	$\mu = 0.45$ mm^{-1}
$c = 13.2500 (3)$ Å	$T = 153 (2)$ K
$V = 786.73 (4)$ Å^3	Block, colorless
$Z = 4$	$0.49 \times 0.48 \times 0.35$ mm

Data collection

Siemens P4 diffractometer	1798 independent reflections
Radiation source: fine-focus sealed tube	1775 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.016$
$T = 153(2)$ K	$\theta_{max} = 27.5º$
ω scans	$\theta_{min} = 3.1º$
Absorption correction: ψ scan (SHELXTL; Bruker, 1998)	$h = -6 \rightarrow 6$
$T_{min} = 0.809$, $T_{max} = 0.858$	$k = -15 \rightarrow 15$
7742 measured reflections	$l = -15 \rightarrow 16$

Refinement

Refinement on F^2	H-atom parameters constrained

Least-squares matrix: full

$w = 1/[\sigma^2(F_o^2) + (0.0313P)^2 + 0.1681P]$
where $P = (F_o^2 + 2F_c^2)/3$

$R[F^2 > 2\sigma(F^2)] = 0.019$

$(\Delta/\sigma)_{max} = 0.001$

$wR(F^2) = 0.055$

$\Delta\rho_{max} = 0.29$ e Å^{-3}

$S = 1.15$

$\Delta\rho_{min} = -0.25$ e Å^{-3}

1798 reflections

Extinction correction: SHELXL97 (Sheldrick, 1997),
$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

101 parameters

Extinction coefficient: 0.058 (4)

Primary atom site location: structure-invariant direct methods

Absolute structure: Flack (1983), 725 Friedel pairs

Secondary atom site location: difference Fourier map

Flack parameter: 0.02 (4)

Hydrogen site location: inferred from neighbouring sites

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of F^2 > 2sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

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

	x	y	z	$U_{iso}*/U_{eq}$
Cl1	1.05471 (5)	0.93130 (2)	0.26203 (2)	0.01395 (9)
O1	0.4387 (2)	0.53632 (7)	0.07337 (7)	0.0205 (2)
H1	0.3341	0.5102	0.0301	0.031*
O2	0.20701 (18)	0.69846 (7)	0.05162 (7)	0.0161 (2)
O3	0.4214 (2)	0.55251 (8)	0.45585 (6)	0.0204 (2)
O4	0.70041 (18)	0.67059 (8)	0.53439 (6)	0.0167 (2)
H4	0.6454	0.6361	0.5859	0.025*
N1	0.5606 (2)	0.82410 (8)	0.15658 (7)	0.0120 (2)
H1A	0.6701	0.8569	0.2025	0.018*
H1B	0.3939	0.8490	0.1676	0.018*
H1C	0.6117	0.8442	0.0932	0.018*
C1	0.3864 (2)	0.64491 (10)	0.08985 (9)	0.0116 (2)
C2	0.5705 (2)	0.69659 (9)	0.16728 (8)	0.0102 (2)
H2	0.7532	0.6689	0.1547	0.012*
C3	0.4798 (2)	0.65720 (10)	0.27187 (8)	0.0115 (2)
H3A	0.3074	0.6926	0.2863	0.014*
H3B	0.4550	0.5728	0.2705	0.014*
C4	0.6668 (2)	0.68690 (10)	0.35714 (8)	0.0124 (2)
H4A	0.8458	0.6610	0.3395	0.015*

H4B	0.6712	0.7713	0.3666	0.015*
C5	0.5815 (3)	0.63012 (9)	0.45392 (8)	0.0119 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01329 (14)	0.01559 (13)	0.01296 (14)	−0.00183 (11)	−0.00097 (10)	0.00137 (10)
O1	0.0232 (4)	0.0145 (4)	0.0238 (4)	0.0019 (4)	−0.0111 (4)	−0.0082 (3)
O2	0.0166 (4)	0.0164 (4)	0.0153 (4)	0.0004 (3)	−0.0052 (3)	0.0000 (3)
O3	0.0284 (5)	0.0194 (5)	0.0134 (4)	−0.0119 (4)	0.0021 (4)	0.0018 (3)
O4	0.0214 (5)	0.0196 (4)	0.0089 (4)	−0.0053 (4)	−0.0014 (4)	0.0029 (3)
N1	0.0134 (4)	0.0119 (4)	0.0105 (4)	−0.0028 (4)	−0.0012 (4)	0.0014 (3)
C1	0.0119 (6)	0.0148 (5)	0.0081 (5)	−0.0022 (4)	0.0004 (4)	0.0006 (4)
C2	0.0107 (5)	0.0100 (5)	0.0101 (5)	−0.0009 (5)	−0.0006 (5)	−0.0006 (4)
C3	0.0121 (5)	0.0128 (5)	0.0096 (5)	−0.0025 (4)	0.0006 (4)	0.0016 (4)
C4	0.0138 (5)	0.0146 (5)	0.0089 (5)	−0.0039 (5)	0.0011 (4)	0.0014 (4)
C5	0.0139 (6)	0.0114 (5)	0.0105 (5)	0.0002 (4)	0.0007 (5)	0.0005 (4)

Geometric parameters (Å, °)

O1—C1	1.3101 (15)	C1—C2	1.5156 (16)
O1—H1	0.8400	C2—C3	1.5312 (15)
O2—C1	1.2176 (15)	C2—H2	1.0000
O3—C5	1.2183 (15)	C3—C4	1.5184 (16)
O4—C5	1.3139 (14)	C3—H3A	0.9900
O4—H4	0.8400	C3—H3B	0.9900
N1—C2	1.4916 (14)	C4—C5	1.5066 (15)
N1—H1A	0.9100	C4—H4A	0.9900
N1—H1B	0.9100	C4—H4B	0.9900
N1—H1C	0.9100		
C1—O1—H1	109.5	C3—C2—H2	109.7
C5—O4—H4	109.5	C4—C3—C2	114.55 (9)
C2—N1—H1A	109.5	C4—C3—H3A	108.6
C2—N1—H1B	109.5	C2—C3—H3A	108.6
H1A—N1—H1B	109.5	C4—C3—H3B	108.6
C2—N1—H1C	109.5	C2—C3—H3B	108.6
H1A—N1—H1C	109.5	H3A—C3—H3B	107.6
H1B—N1—H1C	109.5	C5—C4—C3	110.63 (10)
O2—C1—O1	125.29 (11)	C5—C4—H4A	109.5
O2—C1—C2	122.99 (10)	C3—C4—H4A	109.5
O1—C1—C2	111.67 (10)	C5—C4—H4B	109.5
N1—C2—C1	108.02 (9)	C3—C4—H4B	109.5
N1—C2—C3	111.94 (9)	H4A—C4—H4B	108.1
C1—C2—C3	107.85 (9)	O3—C5—O4	123.93 (10)
N1—C2—H2	109.7	O3—C5—C4	122.46 (10)
C1—C2—H2	109.7	O4—C5—C4	113.59 (10)
O2—C1—C2—N1	−21.37 (15)	C3—C4—C5—O3	14.84 (16)
O1—C1—C2—N1	161.21 (10)	C3—C4—C5—O4	−166.61 (10)

O2—C1—C2—C3	99.80 (13)	H1C—N1—C2—C1	−60.0
O1—C1—C2—C3	−77.62 (12)	H1B—N1—C2—C1	60.0
N1—C2—C3—C4	−69.92 (13)	H1A—N1—C2—C1	−180.0
C1—C2—C3—C4	171.39 (9)	H1—O1—C1—C2	179.8
C2—C3—C4—C5	−171.71 (9)		

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N1—H1A⋯Cl1	0.91	2.29	3.1406 (10)	157
N1—H1B⋯Cl1[i]	0.91	2.34	3.1890 (10)	155
N1—H1C⋯O2[ii]	0.91	2.04	2.8700 (13)	151
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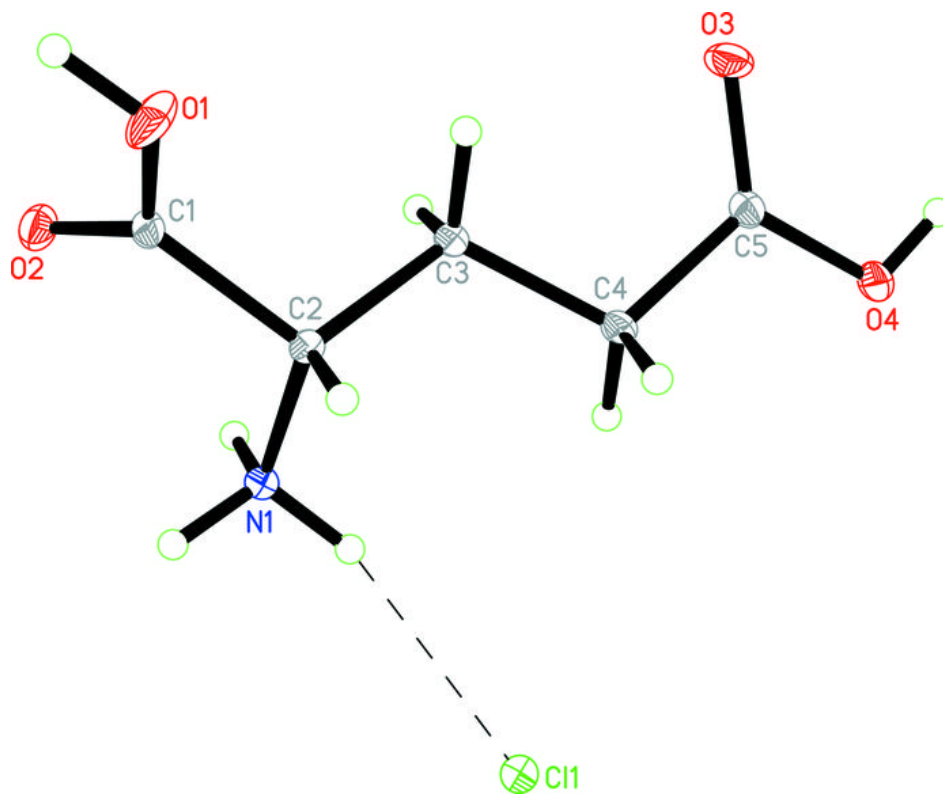
Fig. 1

Fig. 2