IUCrData

ISSN 2414-3146

# D-Glutamic acid hydrochloride

Leonardo Y. Fox-Uribe,[a] Javier Hernández-Paredes,[b] Yedith Soberanes,[a] Ignacio Valenzuela-Chavira,[c] Karina D. Garcia-Orozco,[a] Adrian Ochoa-Leyva,[d] Karen Ochoa Lara,[e] Rosa Elena Navarro[e] and Rogerio R. Sotelo-Mundo[a]*

[a]Laboratorio de Estructura Biomolecular, Centro de Investigación en Alimentación y Desarrollo, A. C., Carretetera Gustavo Enrique Astiazarán Rosas No. 46, Col. La Victoria, Hermosillo 83304, Sonora, Mexico, [b]Departamento de Física, Universidad de Sonora (UNISON), Edificio 3R (Planta Alta), Blvd. Luis Encinas J. y Rosales s/n, Col. Centro, Hermosillo 83000, Sonora, Mexico, [c]Departamento de Ciencias Químico Biologicas, Universidad de Sonora (UNISON), Edificio 5A, Blvd. Luis Encinas J. y Rosales s/n, Col. Centro, Hermosillo 83000, Sonora, Mexico, [d]Departamento de Microbiología Molecular, Instituto de Biotecnología, Universidad Nacional Autónoma de México, Av. Universidad 2001, Cuernavaca 62210, Morelos, Mexico, and [e]Departamento de Investigación en Polímeros y Materiales, Universidad de Sonora (UNISON), Edificio 3G, Blvd. Luis Encinas J. y Rosales s/n, Col. Centro, Hermosillo 83000, Sonora, Mexico. *Correspondence e-mail: rrs@ciad.mx

Received 6 December 2018
Accepted 3 April 2019

Edited by S. Bernès, Benemérita Universidad Autónoma de Puebla, México

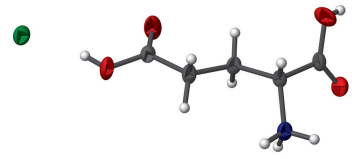
**Keywords:** crystal structure; amino acids; absolute structure; Flack parameter; Hooft parameter.

**CCDC reference**: 1515166

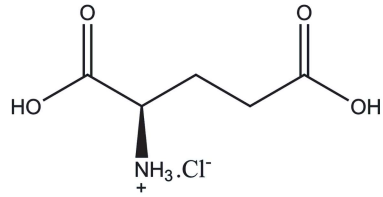
**Structural data**: full structural data are available from iucrdata.iucr.org

The absolute structure of D-glutamic acid hydrochloride [systematic name: (*R*)-1,3-dicarboxypropan-1-aminium chloride], $C_5H_{10}NO_4^+\cdot Cl^-$, has been determined by single-crystal X-ray diffraction at room temperature using Cu $K\alpha$ radiation.

**3D view**



**Chemical scheme**




## Structure description

The asymmetric unit of the title compound (Fig. 1) contains one halide ion ($Cl^-$) and one positively charged molecule of glutamic acid. The molecule of glutamic acid (Glu hereafter) is protonated at the $\gamma$-COOH group. The carbon chain C1—C2—C3—C4—C5 is almost coplanar. The planes of the $\alpha$-COOH and the $\gamma$-COOH groups form angles of 72.4 (16) and 9.3 (10)°, respectively, to the C1–C5 plane.

In the crystal structures of the $\alpha$ and $\beta$ forms of pure L-glutamic acid (L-Glu), molecules of the amino acid that are bound to each other by O—H···O hydrogen bonds, forming infinite chains (Hirayama *et al.*, 1980). In the crystal structure of the title compound reported herein, the $Cl^-$ anion disrupts the characteristic head-to-tail self-assembly between Glu molecules, leading to the formation of an O4—H4···Cl1 hydrogen bond [3.0436 (13) Å]. Furthermore, the $Cl^-$ anion is also bound to two Glu neighbouring molecules by two hydrogen bonds, N1—H1*B*···Cl1 [3.1447 (17) Å] and N1—H1*C*···Cl1

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···O3$^{i}$ | 0.66 | 1.98 | 2.634 (2) | 179 |
| N1—H1$A$···O2$^{ii}$ | 0.88 (2) | 2.11 (2) | 2.8904 (18) | 146.7 (19) |
| N1—H1$B$···Cl1$^{iii}$ | 0.94 (3) | 2.21 (3) | 3.1447 (17) | 172 (2) |
| N1—H1$C$···Cl1$^{iv}$ | 0.88 (3) | 2.32 (3) | 3.1959 (18) | 171 (2) |
| O4—H4···Cl1 | 0.84 (2) | 2.21 (2) | 3.0436 (13) | 169 (2) |

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

**Table 2**
Comparison of parameters for absolute structures.

| | Flack ($x$) | Parsons ($z$) | Hooft ($y$) | Hooft ($G$) |
|---|---|---|---|---|
| D-Glu·HCl | 0.055 (4) | 0.057 (6) | 0.058 (6) | 0.88 (1) |
| L-Glu·HCl | 0.944 (4) | 0.944 (6) | 0.946 (6) | −0.89 (1) |

[3.1959 (18) Å]. It is also observed that all the possible hydrogen-bond donors form interactions with all the available hydrogen-bond acceptors in the crystal (Table 1).

The solution obtained by *SHELXT* (Sheldrick, 2015*a*) was refined to convergence and the resulting absolute structure parameters are listed in Table 2 and *R*-factors are listed in Table 3. The absolute configuration of C2 in this model is *R*, which is that expected based on the D-Glu starting material. To confirm this, the structure was inverted (*i.e.* C2 *S* configuration) corresponding to L-Glu) and re-refined to give a second set of absolute structure parameters (Table 2), which clearly indicate the wrong absolute structure and significantly higher residuals of $R(F)$ = 0.045 and $wR(F^2)$ = 0.1172. We may conclude that this data collection at room temperature has established the absolute structure of the title salt with a high degree of reliability.

## Synthesis and crystallization

All materials were purchased from Sigma–Aldrich (Toluca, Mexico) and used without further purification. 50 mmol of (*R*)-2-aminopentanedioic acid (D-Glu; ≥99% pure) and 50 mmol of $CaCl_2$ were mixed in a final volume of 1 ml ultrapure water (Milli-Q water, Merck Millipore, Mexico).

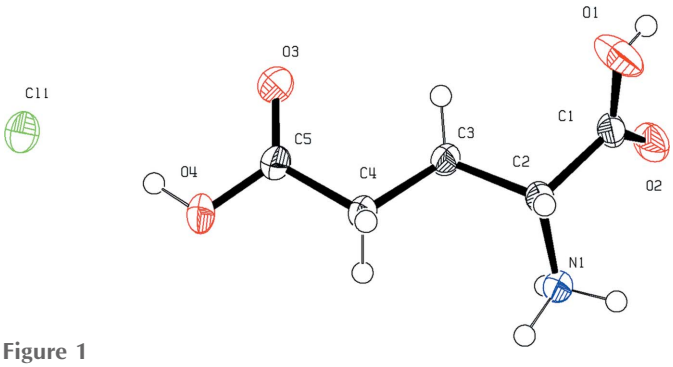



**Figure 1**
*ORTEP* representation of the asymmetric unit of *D*-Glu·HCl. Displacement ellipsoids are drawn at the 50% probability level.

**Table 3**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_5H_{10}NO_4^+\cdot Cl^-$ |
| $M_r$ | 183.59 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 300 |
| $a, b, c$ (Å) | 5.1363 (2), 11.7497 (4), 13.2871 (5) |
| $V$ (Å$^3$) | 801.88 (5) |
| $Z$ | 4 |
| Radiation type | Cu $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 4.03 |
| Crystal size (mm) | 0.25 × 0.10 × 0.03 |
| | |
| Data collection | |
| Diffractometer | Bruker D8 QUEST |
| Absorption correction | Multi-scan (*SADABS*; Bruker, 2012) |
| $T_{min}$, $T_{max}$ | 0.494, 0.754 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 15237, 1630, 1609 |
| $R_{int}$ | 0.034 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.625 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.022, 0.059, 1.07 |
| No. of reflections | 1630 |
| No. of parameters | 114 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.16, −0.23 |
| Absolute structure | See 'Structure description' section |
| Absolute structure parameter | 0.055 (4) |

Computer programs: *APEX2* and *SAINT* (Bruker, 2012), *SHELXT2014/4* (Sheldrick, 2015*a*), *SHELXL2014/7* (Sheldrick, 2015*b*) and *ORTEP-3 for Windows*and *WinGX* (Farrugia, 2012).

The solution was left to evaporate at 298 K in a 5 ml glass vial. Good quality crystals of D-Glu·HCl were obtained after four weeks.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3.

## Funding information

Funding for this research was provided by: Consejo Nacional de Ciencia y Tecnología (scholarship to L. Y. Fox-Uribe; grant No. CB-2014–237963 to R. R. Sotelo-Mundo; grant No. INFR-2014–01-225455); Red Temática de Química Supramolecular (grant No. 294810); Instituto de Biotecnología-CIC-UNAM and CIAD ; Secretaría de Educación Pública (PRODEP) (grant No. 511–6/18–8537 to J. Hernández-Paredes; grant No. 35986 to I. Valenzuela-Chavira).

## References

Bruker (2012). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc, Madison, Wisconsin, USA.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Hirayama, N., Shirahata, K., Ohashi, Y. & Sasada, Y. (1980). *Bull. Chem. Soc. Jpn*, **53**, 30–35.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

# full crystallographic data

*IUCrData* (2019). **4**, x190458 [https://doi.org/10.1107/S2414314619004589]

## D-Glutamic acid hydrochloride

**Leonardo Y. Fox-Uribe, Javier Hernández-Paredes, Yedith Soberanes, Ignacio Valenzuela-Chavira, Karina D. Garcia-Orozco, Adrian Ochoa-Leyva, Karen Ochoa Lara, Rosa Elena Navarro and Rogerio R. Sotelo-Mundo**

**(*R*)-1,3-Dicarboxypropan-1-aminium chloride**

*Crystal data*

$C_5H_{10}NO_4^+·Cl^-$
$M_r$ = 183.59
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a$ = 5.1363 (2) Å
$b$ = 11.7497 (4) Å
$c$ = 13.2871 (5) Å
$V$ = 801.88 (5) Å$^3$
$Z$ = 4
$F(000)$ = 384
$D_x$ = 1.512 Mg m$^{-3}$
Cu $K\alpha$ radiation, $\lambda$ = 1.54178 Å
Cell parameters from 9897 reflections
$\theta$ = 5.0–74.4°
$\mu$ = 4.03 mm$^{-1}$
$T$ = 300 K
Plate, colourless
0.25 × 0.10 × 0.03 mm

*Data collection*

Bruker D8 QUEST diffractometer
Radiation source: Microfocus sealed tube, Incoatec I$\mu$S HB
Multiyaler mirrors monochromator
Detector resolution: 102.4 pixels mm$^{-1}$
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (SADABS; Bruker, 2012)
$T_{min}$ = 0.494, $T_{max}$ = 0.754
15237 measured reflections
1630 independent reflections
1609 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034
$\theta_{max}$ = 74.4°, $\theta_{min}$ = 5.0°
$h$ = −6→6
$k$ = −14→14
$l$ = −16→16

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.022
$wR(F^2)$ = 0.059
$S$ = 1.07
1630 reflections
114 parameters
0 restraints
0 constraints
Primary atom site location: dual
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.040P)^2 + 0.0618P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.23 e Å$^{-3}$
Absolute structure: See 'Structure description' section
Absolute structure parameter: 0.055 (4)

*Special details*

**Refinement**. Hydrogen atoms for CH and $CH_2$ groups were placed in geometrically calculated positions using the riding model. Hydrogen atom H1 has setting as an idealized OH group, with C1—O1—H1 angle tetrahedral. Hydrogen atoms involved in *X*—H···Cl interactions (H1A, H1B, H1C and H4) have been located from a difference map and refined with free coordinates. All H atoms were refined with calculated isotropic displacement parameters.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.55328 (8) | 0.57267 (4) | 0.26144 (3) | 0.03036 (14) |
| O1 | 0.4381 (4) | 0.53718 (11) | 0.92637 (12) | 0.0451 (4) |
| H1 | 0.349 (6) | 0.5155 (14) | 0.956 (3) | 0.068* |
| N1 | 0.5594 (4) | 0.82112 (12) | 0.84223 (10) | 0.0251 (3) |
| C1 | 0.3874 (3) | 0.64434 (14) | 0.91039 (11) | 0.0245 (4) |
| H1A | 0.572 (5) | 0.8424 (17) | 0.9058 (16) | 0.029* |
| H1B | 0.696 (5) | 0.8561 (19) | 0.8065 (16) | 0.029* |
| H1C | 0.414 (5) | 0.8528 (19) | 0.8208 (16) | 0.029* |
| O2 | 0.2122 (3) | 0.69746 (11) | 0.94905 (10) | 0.0347 (3) |
| C2 | 0.5687 (4) | 0.69496 (13) | 0.83245 (11) | 0.0213 (3) |
| H2 | 0.7466 | 0.6683 | 0.8448 | 0.026* |
| O3 | 0.4199 (4) | 0.55187 (12) | 0.54442 (9) | 0.0440 (4) |
| C3 | 0.4791 (3) | 0.65493 (14) | 0.72847 (11) | 0.0237 (3) |
| H3A | 0.4571 | 0.573 | 0.7302 | 0.028* |
| H3B | 0.3101 | 0.6883 | 0.7145 | 0.028* |
| O4 | 0.6959 (3) | 0.66860 (12) | 0.46696 (9) | 0.0362 (3) |
| C4 | 0.6617 (4) | 0.68448 (15) | 0.64312 (12) | 0.0261 (4) |
| H4A | 0.6646 | 0.7664 | 0.6338 | 0.031* |
| H4B | 0.8367 | 0.66 | 0.6601 | 0.031* |
| H4 | 0.648 (5) | 0.6340 (19) | 0.4144 (17) | 0.031* |
| C5 | 0.5778 (4) | 0.62819 (14) | 0.54680 (11) | 0.0257 (4) |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0287 (2) | 0.0363 (2) | 0.0260 (2) | 0.00446 (18) | −0.00205 (16) | −0.00294 (15) |
| O1 | 0.0502 (9) | 0.0344 (7) | 0.0507 (8) | 0.0051 (7) | 0.0244 (8) | 0.0169 (6) |
| N1 | 0.0279 (7) | 0.0276 (7) | 0.0200 (6) | −0.0064 (7) | 0.0007 (7) | −0.0023 (5) |
| C1 | 0.0256 (9) | 0.0307 (8) | 0.0171 (7) | −0.0042 (7) | 0.0011 (6) | 0.0009 (6) |
| O2 | 0.0369 (8) | 0.0361 (7) | 0.0310 (6) | 0.0010 (6) | 0.0142 (6) | 0.0010 (5) |
| C2 | 0.0208 (8) | 0.0250 (7) | 0.0180 (6) | −0.0021 (7) | 0.0008 (7) | 0.0013 (5) |
| O3 | 0.0610 (10) | 0.0453 (8) | 0.0256 (6) | −0.0267 (8) | −0.0041 (7) | −0.0047 (5) |
| C3 | 0.0240 (8) | 0.0272 (7) | 0.0198 (7) | −0.0051 (6) | −0.0003 (7) | −0.0032 (6) |
| O4 | 0.0476 (9) | 0.0432 (8) | 0.0177 (5) | −0.0119 (7) | 0.0048 (6) | −0.0061 (5) |
| C4 | 0.0290 (9) | 0.0317 (8) | 0.0176 (7) | −0.0086 (7) | −0.0008 (7) | −0.0026 (6) |
| C5 | 0.0314 (10) | 0.0262 (8) | 0.0196 (7) | −0.0017 (8) | −0.0013 (7) | −0.0020 (6) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.303 (2) | O3—C5 | 1.209 (2) |
| O1—H1 | 0.66 (4) | C3—C4 | 1.512 (2) |
| N1—C2 | 1.489 (2) | C3—H3A | 0.97 |
| N1—H1A | 0.88 (2) | C3—H3B | 0.97 |
| N1—H1B | 0.94 (3) | O4—C5 | 1.311 (2) |
| N1—H1C | 0.88 (3) | O4—H4 | 0.84 (2) |
| C1—O2 | 1.210 (2) | C4—C5 | 1.504 (2) |
| C1—C2 | 1.514 (2) | C4—H4A | 0.97 |
| C2—C3 | 1.530 (2) | C4—H4B | 0.97 |
| C2—H2 | 0.98 | | |
| | | | |
| C1—O1—H1 | 109.5 | C4—C3—C2 | 114.83 (14) |
| C2—N1—H1A | 111.3 (13) | C4—C3—H3A | 108.6 |
| C2—N1—H1B | 111.6 (14) | C2—C3—H3A | 108.6 |
| H1A—N1—H1B | 108 (2) | C4—C3—H3B | 108.6 |
| C2—N1—H1C | 114.7 (14) | C2—C3—H3B | 108.6 |
| H1A—N1—H1C | 105 (2) | H3A—C3—H3B | 107.5 |
| H1B—N1—H1C | 106.5 (18) | C5—O4—H4 | 111.0 (16) |
| O2—C1—O1 | 125.29 (16) | C5—C4—C3 | 111.06 (14) |
| O2—C1—C2 | 123.04 (15) | C5—C4—H4A | 109.4 |
| O1—C1—C2 | 111.61 (15) | C3—C4—H4A | 109.4 |
| N1—C2—C1 | 108.17 (13) | C5—C4—H4B | 109.4 |
| N1—C2—C3 | 112.04 (13) | C3—C4—H4B | 109.4 |
| C1—C2—C3 | 108.18 (14) | H4A—C4—H4B | 108 |
| N1—C2—H2 | 109.5 | O3—C5—O4 | 123.88 (15) |
| C1—C2—H2 | 109.5 | O3—C5—C4 | 122.73 (15) |
| C3—C2—H2 | 109.5 | O4—C5—C4 | 113.38 (15) |
| | | | |
| O2—C1—C2—N1 | 20.8 (2) | C1—C2—C3—C4 | −171.82 (14) |
| O1—C1—C2—N1 | −161.84 (16) | C2—C3—C4—C5 | 172.09 (14) |
| O2—C1—C2—C3 | −100.72 (18) | C3—C4—C5—O3 | −14.3 (2) |
| O1—C1—C2—C3 | 76.60 (19) | C3—C4—C5—O4 | 166.94 (16) |
| N1—C2—C3—C4 | 69.0 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···O3$^{i}$ | 0.66 | 1.98 | 2.634 (2) | 179 |
| N1—H1*A*···O2$^{ii}$ | 0.88 (2) | 2.11 (2) | 2.8904 (18) | 146.7 (19) |
| N1—H1*A*···O3$^{iii}$ | 0.88 (2) | 2.55 (2) | 3.1033 (19) | 121.6 (16) |
| N1—H1*B*···Cl1$^{iv}$ | 0.94 (3) | 2.21 (3) | 3.1447 (17) | 172 (2) |
| N1—H1*C*···Cl1$^{v}$ | 0.88 (3) | 2.32 (3) | 3.1959 (18) | 171 (2) |
| O4—H4···Cl1 | 0.84 (2) | 2.21 (2) | 3.0436 (13) | 169 (2) |

Symmetry codes: (i) −*x*+1/2, −*y*+1, *z*+1/2; (ii) *x*+1/2, −*y*+3/2, −*z*+2; (iii) −*x*+1, *y*+1/2, −*z*+3/2; (iv) *x*+1/2, −*y*+3/2, −*z*+1; (v) *x*−1/2, −*y*+3/2, −*z*+1.