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Redetermination of α-D-glucosamine hydrochloride: elucidation of the hydrogen-bonding scheme

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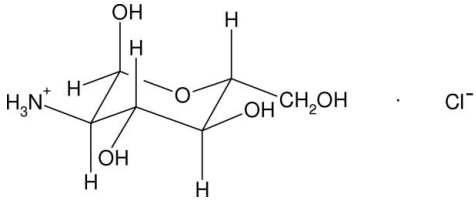
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.029; wR factor = 0.075; data-to-parameter ratio = 18.5.

The previously reported crystal structures [Chu & Jeffery (1965). *Proc. R. Soc. London Ser. A*, **285**, 470–479; Chandrasekharan & Mallikarjunan (1969). *Z. Kristallogr.* **129**, 29] of the title compound, $C_6H_{14}NO_5^+\cdot Cl^-$, have been confirmed to higher precision, and the H atoms located, allowing the elucidaton of the hydrogen-bonding network. A combination of O—H···O, N—H···O, O—H···Cl and N—H···Cl links results in a three-dimensional network. Considered by themselves, the inter-cation O—H···O and N—H···O hydrogen bonds result in undulating (001) layers. The configurations of the chiral C atoms are: C1 *S*, C2 *R*, C3 *R*, C4 *S* and C5 *R*.

Related literature

For an earlier structure determination of the title compound, in which the H atoms were not located, and background literature, see: Chu & Jeffery (1965); Chandrasekharan & Mallikarjunan (1969); Allen *et al.* (1987).



Experimental

Crystal data

$C_6H_{14}NO_5^+\cdot Cl^-$
M_r = 215.63
Monoclinic, $P2_1$
a = 7.1474 (5) Å
b = 9.2140 (6) Å
c = 7.7650 (5) Å
β = 112.884 (1)°
V = 471.12 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 295 (2) K
0.42 × 0.30 × 0.15 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.851, T_{max} = 0.944
3941 measured reflections
2217 independent reflections
2124 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.075
S = 1.07
2217 reflections
120 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}
Absolute structure: Flack (1983), 761 Friedel pairs
Flack parameter: 0.07 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O5^{i}	0.89	1.89	2.7772 (17)	172
N1—H1C···O4^{ii}	0.89	2.15	2.8930 (19)	141
N1—H1D···Cl1^{iii}	0.89	2.38	3.1744 (13)	149
O2—H2···Cl1^{iv}	0.82	2.35	3.1448 (12)	162
O3—H3···Cl1^{v}	0.84	2.35	3.1911 (14)	173
O4—H4···Cl1	0.82	2.35	3.1667 (14)	175
O5—H5···O3^{v}	0.81	1.95	2.7373 (17)	163

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

We thank Strides Arco Labs, Mangalore, India for the gift sample of glucosamine hydrochloride. BKS thanks AICTE, Government of India, New Delhi for financial assistance under the Career Award for Young Teachers scheme.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2375).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1999). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chandrasekharan, R. & Mallikarjunan, M. (1969). *Z. Kristallogr.* **129**, 29.
Chu, S. S. C. & Jeffery, G. A. (1965). *Proc. R. Soc. London Ser. A*, **285**, 470–479.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

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supplementary materials

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Redetermination of α-D-glucosamine hydrochloride: elucidation of the hydrogen-bonding scheme

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Comment

The present study confirms the previous structure determinations (Chu & Jeffery, 1965; Chandrasekharan & Mallikarjunan, 1969) of the title compound, (I), to modern standards of precision. All the geometrical values for (I) (Fig. 1) fall within their expected ranges (Allen *et al.*, 1995) and the six-membered ring is well described as a chair: for atoms C1, C2, C4 and C5, the r.m.s. deviation from their mean plane is 0.021 Å: C3 and O1 deviate from the plane by −0.652 (2)Å and 0.6477 (19) Å, respectively.

Here, the H atoms have been located, allowing the hydrogen-bonding scheme, involving a combination of O—H···O, N—H···O, O—H···Cl and N—H···Cl links, to be elucidated (Table 1). The inter-cation N—H···O and O—H···O bonds result in undulating (001) sheets (Fig. 2).

Experimental

A sample of glucosamine hydrochloride was obtained from Strides Arco labs, Mangalore, India and recrystallized from water to yield colourless chunks of (I). m.p.: 449–451 K.

Refinement

The O-bound hydrogen atoms were located in a difference map and refined as riding in their as-found relative positions with U_{iso}(H) = 1.2U_{eq}(O). The other hydrogen atoms were geometrically placed (C—H = 0.97–0.98 Å, N—H = 0.89 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier). The $-NH_3^+$ group was allowed to rotate, but not to tip, to best fit the electron density.

Figures

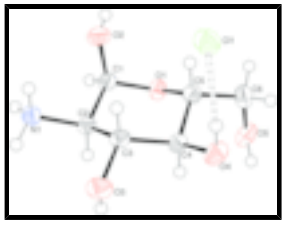

Fig. 1. View of the molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). The hydrogen bond is shown as a double dashed line.

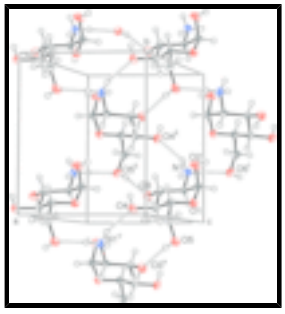

Fig. 2. Part of an (001) sheet of cations in (I) linked by O—H···O and N—H···O hydrogen bonds. Symmetry codes as in Table 1.

α-*D*-glucosamine hydrochloride

Crystal data

$C_6H_{14}NO_5^+ \cdot Cl^-$	$F_{000} = 228$
$M_r = 215.63$	$D_x = 1.520$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2yb	Cell parameters from 2993 reflections
$a = 7.1474$ (5) Å	$\theta = 5.0$–$30.0°$
$b = 9.2140$ (6) Å	$\mu = 0.40$ mm^{-1}
$c = 7.7650$ (5) Å	$T = 295$ (2) K
$\beta = 112.884$ (1)°	Chunk, colourless
$V = 471.12$ (5) Å^3	0.42 × 0.30 × 0.15 mm
$Z = 2$	

Data collection

Bruker SMART1000 CCD diffractometer	2217 independent reflections
Radiation source: fine-focus sealed tube	2124 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.020$
$T = 295(2)$ K	$\theta_{max} = 30.0°$
ω scans	$\theta_{min} = 5.0°$
Absorption correction: multi-scan SADABS (Bruker, 1999)	$h = -10 \rightarrow 10$
$T_{min} = 0.851$, $T_{max} = 0.944$	$k = -9 \rightarrow 12$
3941 measured reflections	$l = -10 \rightarrow 9$

Refinement

Refinement on F^2	Hydrogen site location: difmap (O-H) and geom (others)
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.029$	$w = 1/[\sigma^2(F_o^2) + (0.0452P)^2 + 0.0391P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.075$	$(\Delta/\sigma)_{max} < 0.001$

S = 1.07	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
2217 reflections	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
120 parameters	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1 restraint	Extinction coefficient: 0.042 (7)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 761 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.07 (5)

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 $F^2 > \sigma(F^2)$ 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}
C1	0.0118 (2)	0.23568 (18)	0.9307 (2)	0.0254 (3)
H1A	−0.0990	0.2269	0.9748	0.030*
C2	0.2141 (2)	0.22407 (17)	1.09837 (19)	0.0224 (3)
H2A	0.2115	0.1363	1.1687	0.027*
C3	0.3952 (2)	0.21459 (18)	1.0412 (2)	0.0234 (3)
H3A	0.4058	0.3070	0.9830	0.028*
C4	0.3592 (2)	0.09447 (18)	0.89614 (19)	0.0233 (3)
H4A	0.3528	0.0017	0.9553	0.028*
C5	0.1553 (2)	0.11884 (19)	0.7337 (2)	0.0248 (3)
H5A	0.1577	0.2125	0.6748	0.030*
C6	0.1010 (3)	0.0004 (2)	0.5870 (2)	0.0332 (4)
H6A	−0.0254	0.0255	0.4850	0.040*
H6B	0.2059	−0.0051	0.5371	0.040*
N1	0.23946 (19)	0.35206 (15)	1.22281 (17)	0.0266 (3)
H1B	0.1307	0.3608	1.2511	0.032*
H1C	0.2535	0.4319	1.1644	0.032*
H1D	0.3494	0.3400	1.3272	0.032*
O1	−0.00356 (15)	0.12014 (13)	0.80456 (15)	0.0261 (2)
O2	0.00134 (17)	0.37174 (15)	0.85023 (15)	0.0348 (3)
H2	−0.1164	0.3899	0.7813	0.042*
O3	0.57819 (17)	0.19429 (15)	1.20313 (17)	0.0332 (3)
H3	0.5477	0.1193	1.2483	0.040*
O4	0.52073 (18)	0.08680 (15)	0.83229 (17)	0.0325 (3)
H4	0.5190	0.1640	0.7788	0.039*

O5	0.07990 (17)	−0.13730 (14)	0.65823 (17)	0.0354 (3)
H5	0.1895	−0.1737	0.7165	0.042*
Cl1	0.52602 (6)	0.39359 (5)	0.65041 (5)	0.03359 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0204 (6)	0.0265 (8)	0.0268 (6)	0.0012 (5)	0.0064 (5)	−0.0039 (6)
C2	0.0207 (6)	0.0209 (7)	0.0242 (6)	0.0001 (5)	0.0074 (5)	0.0000 (5)
C3	0.0193 (6)	0.0222 (8)	0.0269 (6)	−0.0004 (5)	0.0068 (5)	−0.0002 (5)
C4	0.0230 (6)	0.0210 (8)	0.0271 (6)	0.0000 (5)	0.0111 (5)	0.0003 (5)
C5	0.0255 (6)	0.0244 (8)	0.0252 (6)	−0.0004 (5)	0.0106 (5)	0.0007 (6)
C6	0.0328 (8)	0.0396 (10)	0.0280 (7)	−0.0020 (7)	0.0127 (6)	−0.0073 (7)
N1	0.0261 (6)	0.0271 (7)	0.0252 (5)	0.0003 (5)	0.0085 (4)	−0.0036 (5)
O1	0.0217 (5)	0.0266 (6)	0.0293 (5)	−0.0030 (4)	0.0092 (4)	−0.0067 (4)
O2	0.0322 (5)	0.0282 (8)	0.0344 (5)	0.0054 (5)	0.0025 (4)	0.0018 (5)
O3	0.0213 (5)	0.0321 (7)	0.0366 (6)	−0.0031 (5)	0.0009 (4)	−0.0014 (5)
O4	0.0308 (5)	0.0306 (7)	0.0429 (7)	0.0047 (5)	0.0217 (5)	0.0018 (5)
O5	0.0264 (5)	0.0293 (8)	0.0496 (7)	−0.0001 (5)	0.0137 (5)	−0.0106 (5)
Cl1	0.03269 (18)	0.0357 (2)	0.02785 (16)	−0.00225 (17)	0.00684 (12)	−0.00076 (17)

Geometric parameters (Å, °)

C1—O2	1.390 (2)	C5—O1	1.4413 (17)
C1—O1	1.4219 (19)	C5—C6	1.515 (2)
C1—C2	1.5273 (18)	C5—H5A	0.9800
C1—H1A	0.9800	C6—O5	1.415 (2)
C2—N1	1.4902 (19)	C6—H6A	0.9700
C2—C3	1.5260 (19)	C6—H6B	0.9700
C2—H2A	0.9800	N1—H1B	0.8900
C3—O3	1.4311 (17)	N1—H1C	0.8900
C3—C4	1.528 (2)	N1—H1D	0.8900
C3—H3A	0.9800	O2—H2	0.8199
C4—O4	1.4243 (16)	O3—H3	0.8407
C4—C5	1.5286 (19)	O4—H4	0.8213
C4—H4A	0.9800	O5—H5	0.8094
O2—C1—O1	112.90 (12)	O1—C5—C6	106.48 (12)
O2—C1—C2	108.30 (13)	O1—C5—C4	109.03 (11)
O1—C1—C2	109.18 (12)	C6—C5—C4	113.42 (14)
O2—C1—H1A	108.8	O1—C5—H5A	109.3
O1—C1—H1A	108.8	C6—C5—H5A	109.3
C2—C1—H1A	108.8	C4—C5—H5A	109.3
N1—C2—C3	109.32 (12)	O5—C6—C5	112.77 (12)
N1—C2—C1	109.48 (12)	O5—C6—H6A	109.0
C3—C2—C1	112.65 (11)	C5—C6—H6A	109.0
N1—C2—H2A	108.4	O5—C6—H6B	109.0
C3—C2—H2A	108.4	C5—C6—H6B	109.0
C1—C2—H2A	108.4	H6A—C6—H6B	107.8

O3—C3—C2	109.91 (12)	C2—N1—H1B	109.5
O3—C3—C4	112.80 (13)	C2—N1—H1C	109.5
C2—C3—C4	109.71 (12)	H1B—N1—H1C	109.5
O3—C3—H3A	108.1	C2—N1—H1D	109.5
C2—C3—H3A	108.1	H1B—N1—H1D	109.5
C4—C3—H3A	108.1	H1C—N1—H1D	109.5
O4—C4—C3	111.44 (12)	C1—O1—C5	114.09 (11)
O4—C4—C5	111.22 (11)	C1—O2—H2	109.8
C3—C4—C5	109.58 (12)	C3—O3—H3	100.2
O4—C4—H4A	108.2	C4—O4—H4	106.1
C3—C4—H4A	108.2	C6—O5—H5	111.2
C5—C4—H4A	108.2		
O2—C1—C2—N1	51.67 (15)	C2—C3—C4—C5	53.63 (15)
O1—C1—C2—N1	174.97 (12)	O4—C4—C5—O1	177.79 (13)
O2—C1—C2—C3	−70.19 (16)	C3—C4—C5—O1	−58.55 (16)
O1—C1—C2—C3	53.11 (18)	O4—C4—C5—C6	59.34 (18)
N1—C2—C3—O3	61.87 (16)	C3—C4—C5—C6	−177.00 (13)
C1—C2—C3—O3	−176.18 (13)	O1—C5—C6—O5	−57.79 (16)
N1—C2—C3—C4	−173.55 (11)	C4—C5—C6—O5	62.12 (17)
C1—C2—C3—C4	−51.60 (17)	O2—C1—O1—C5	60.77 (16)
O3—C3—C4—O4	−59.95 (16)	C2—C1—O1—C5	−59.75 (16)
C2—C3—C4—O4	177.16 (12)	C6—C5—O1—C1	−173.77 (13)
O3—C3—C4—C5	176.52 (12)	C4—C5—O1—C1	63.51 (16)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1B···O5^{i}	0.89	1.89	2.7772 (17)	172
N1—H1C···O4^{ii}	0.89	2.15	2.8930 (19)	141
N1—H1D···Cl1^{iii}	0.89	2.38	3.1744 (13)	149
O2—H2···Cl1^{iv}	0.82	2.35	3.1448 (12)	162
O3—H3···Cl1^{v}	0.84	2.35	3.1911 (14)	173
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O5—H5···O3^{v}	0.81	1.95	2.7373 (17)	163

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

Fig. 1

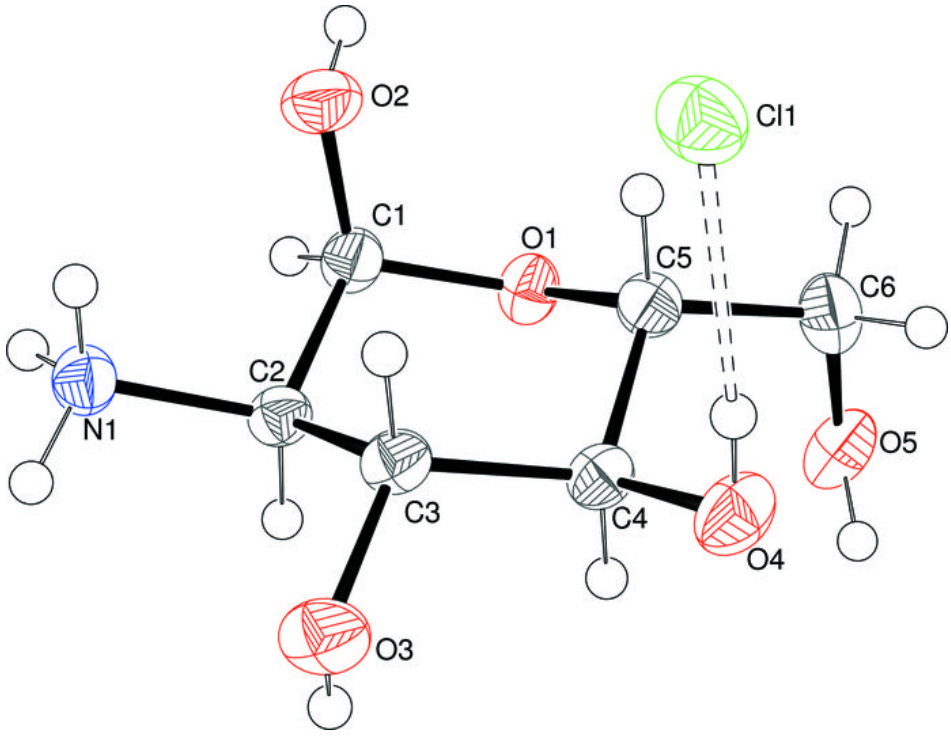

Fig. 2