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A monoclinic form of *N*,*N*,*N*′,*N*′-tetrakis(carboxymethyl)ethylenediammonium dichloride trihydrate

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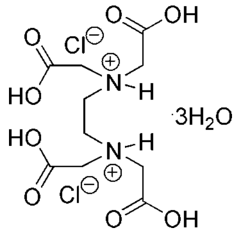
Key indicators: single-crystal X-ray study; *T* = 170 K; mean σ(C–C) = 0.002 Å; *R* factor = 0.034; *wR* factor = 0.075; data-to-parameter ratio = 17.0.

The crystal structure of a monoclinic form of the title compound, $C_{10}H_{18}N_2O_8^{2+}\cdot 2Cl^-\cdot 3H_2O$ or $(H_6edta)Cl_2\cdot 3H_2O$, is reported. The first published form had space group $P\bar{1}$ [Mistryukov, Mikhailov, Sergeev, Zhuravlov, Schelokov, Chernov, Fodorov & Brekhovskikh (1987). *Dokl. Akad. Nauk SSSR*, **295**, 1390–1393], while the structure reported here has *P*2/*c*. A crystallographic twofold rotation axis passes through the mid-point of the central C—C bond of the cation; another twofold axis passes through one water O atom. In the crystal structure, the $(H_6edta)^{2+}$ and Cl^- ions and water molecules are linked *via* hydrogen bonds, forming two-dimensional networks. The NH groups and carbonyl O atoms form trifurcated hydrogen bonds.

Related literature

For related literature, see: Mistryukov *et al.* (1987); Steiner (1999).



Experimental

Crystal data

$C_{10}H_{18}N_2O_8^{2+}\cdot 2Cl^-\cdot 3H_2O$
$M_r = 419.21$
Monoclinic, *P*2/*c*
$a = 11.6817\ (4)$ Å
$b = 5.5845\ (2)$ Å
$c = 16.4627\ (6)$ Å
$\beta = 122.372\ (3)°$
$V = 907.06\ (6)$ Å^3
$Z = 2$
Mo *Kα* radiation
$\mu = 0.42$ mm^{-1}
$T = 170\ (2)$ K
0.4 × 0.05 × 0.05 mm

Data collection

Oxford Diffraction Xcalibur S CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min} = 0.992$, $T_{max} = 1$
10960 measured reflections
2762 independent reflections
1740 reflections with $I > 2\sigma(I)$
$R_{int} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.075$
$S = 0.92$
2762 reflections
162 parameters
All H-atom parameters refined
$\Delta\rho_{max} = 0.50$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5O*A*···O6^{i}	0.89 (3)	1.87 (3)	2.7329 (18)	163 (3)
O5—H5O*B*···Cl1^{ii}	0.98 (3)	2.25 (3)	3.2085 (13)	167 (3)
N1—H1N···O2	0.836 (17)	2.410 (18)	2.6889 (15)	100.3 (14)
N1—H1N···O4	0.836 (17)	2.26 (2)	2.6837 (18)	111.7 (19)
N1—H1N···O4^{iii}	0.836 (17)	2.074 (19)	2.8218 (16)	148.6 (18)
O1—H1O···O5	1.11 (2)	1.46 (2)	2.5278 (16)	158 (2)
O3—H3O···Cl1	0.89 (2)	2.10 (2)	2.9669 (13)	165 (2)
O6—H6O···Cl1	0.84 (2)	2.27 (2)	3.1062 (11)	172 (2)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Version 1.076; Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2016).

References

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Steiner, T. (1999). *Chem. Commun.* pp. 2299–2300.

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supplementary materials

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A monoclinic form of *N*,*N*,*N'*,*N'*-tetrakis(carboxymethyl)ethylenediammonium dichloride trihydrate

G. N. Kaluderovic, S. Gómez-Ruiz, H. Schmidt and **D. Steinborn**

Comment

The title molecule, (I), in this second polymorphic form (Fig. 1) differs from the reported structure (Mistryukov *et al.*, 1987) and possesses twofold crystallographically imposed symmetry axis. The major difference between the polymorphs is found in the N–C–C–N torsion angle. Namely, in the reported structure ethylenediamine part was found to be *antiperiplanar* (torsion angle N–C–C–N = 180.2°) and here it is in *gauche* conformation (torsion angle N–C–C–N = 68.3 (2)°).

The structure of title compound is stabilized by intramolecular and intermolecular H-bonds (Table 2). The title compound shows a trifurcated intramolecular H-bond involving three carbonyl O atoms (O2, O4 and O4^{i}) and N—H moieties. The formation of the N–H···O intramolecular hydrogen bonds (Fig. 1) leads to the formation of five-membered (with O2 and O4) and eight membered rings (involving O4^{i}) closed by H-bonding. The H_6edta^{2+} cations are bonded in infinite chains through H-bonds *via* water molecules and chloride anions (O1—H···O5—H···Cl1···H—O3; Fig. 2). Long O1—H1O and short H1O···O5 bond lenghts are found within the structure. This is commonly observed in acidic O···H···O bridges (Steiner, 1999). The infinite chains are connected *via* H-bonds between the O6 of one water molecule and O5 atoms of two other water molecules and two chloride anions (Fig. 2).

Experimental

The title compound was obtained unintentionally as the product of an attempt to syntesize [$NiCl_2(Me_4edta)$] by the reaction of $NiCl_2$ (0.5 mmol) and Me_4edta (0.5 mmol) in water (20 ml). The solution was stirred for 3 h at room temperature. By slow evaporation over two weeks colorless crystals of the title compound were afforded due to hydrolysis of starting material Me_4edta.

Refinement

All H atoms were found.

Figures

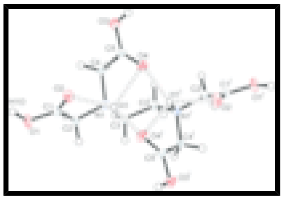

Fig. 1. *ORTEP* representation of H_6edta^{2+} cation in the title compound. Displacement ellipsoids are plotted at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. [Symmetry code: (i) $-x + 1/2, y, -z + 3/2$]

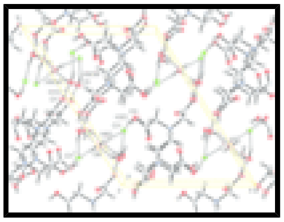

Fig. 2. Network of H-bongind viewed normal to *b*-axis.

N,*N*,*N*′,*N*′-tetrakis(carboxymethyl)ethylenediammonium dichloride trihydrate

Crystal data

$C_{10}H_{18}N_2O_8{}^{2+}\cdot 2Cl^{-}\cdot 3H_2O$	$F_{000} = 440$
$M_r = 419.21$	$D_x = 1.535$ Mg m^{-3}
Monoclinic, *P*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2yc	Cell parameters from 3118 reflections
a = 11.6817 (4) Å	θ = 2.5–32.2°
b = 5.5845 (2) Å	μ = 0.42 mm^{-1}
c = 16.4627 (6) Å	*T* = 170 (2) K
β = 122.372 (3)°	Small needle, colourless
V = 907.06 (6) Å^3	0.4 × 0.05 × 0.05 mm
Z = 2	

Data collection

Oxford Diffraction Xcalibur S CCD diffractometer	2762 independent reflections
Monochromator: graphite	1740 reflections with $I > 2\sigma(I)$
Detector resolution: 16.356 pixels mm^{-1}	$R_{int} = 0.042$
T = 170(2) K	$\theta_{max} = 30.5°$
ω and φ scans	$\theta_{min} = 2.5°$
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)	$h = -16\rightarrow16$
$T_{min} = 0.992$, $T_{max} = 1$	$k = -7\rightarrow7$
10960 measured reflections	$l = -23\rightarrow23$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.034$	All H-atom parameters refined
$wR(F^2) = 0.075$	$w = 1/[\sigma^2(F_o^2) + (0.0347P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.92$	$(\Delta/\sigma)_{max} < 0.001$
2762 reflections	$\Delta\rho_{max} = 0.50$ e Å^{-3}
162 parameters	$\Delta\rho_{min} = -0.25$ e Å^{-3}

Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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}
O3	0.40757 (12)	0.3165 (2)	0.42566 (8)	0.0232 (3)
O4	0.41403 (10)	0.34559 (18)	0.29282 (7)	0.0201 (2)
O5	1.08106 (13)	0.5932 (2)	0.65974 (9)	0.0292 (3)
O6	0	1.1041 (3)	0.25	0.0294 (4)
C3	0.57529 (15)	−0.1579 (3)	0.28642 (10)	0.0157 (3)
C4	0.55585 (15)	0.0606 (3)	0.41261 (10)	0.0163 (3)
C5	0.45072 (14)	0.2554 (3)	0.37004 (10)	0.0159 (3)
H3O	0.347 (2)	0.433 (4)	0.3983 (14)	0.056 (7)*
H4A	0.5124 (15)	−0.091 (3)	0.4094 (10)	0.013 (4)*
H4B	0.6221 (15)	0.099 (3)	0.4786 (11)	0.016 (4)*
H5OA	1.038 (3)	0.689 (5)	0.678 (2)	0.110 (11)*
H5OB	1.145 (3)	0.498 (5)	0.716 (2)	0.105 (10)*
H6O	0.071 (2)	1.021 (4)	0.2792 (16)	0.065 (8)*
H1N	0.6095 (18)	0.172 (3)	0.3266 (13)	0.030 (5)*
H1O	0.988 (2)	0.419 (4)	0.5885 (15)	0.064 (7)*
H2A	0.8135 (16)	0.010 (3)	0.3860 (11)	0.025 (4)*
H2B	0.7973 (16)	−0.096 (3)	0.4646 (12)	0.022 (4)*
H3A	0.5973 (15)	−0.295 (3)	0.3238 (11)	0.015 (4)*
H3B	0.6279 (15)	−0.159 (2)	0.2570 (10)	0.013 (4)*
Cl1	0.24726 (4)	0.76000 (7)	0.33996 (3)	0.02310 (11)
O1	0.95474 (10)	0.2511 (2)	0.54558 (7)	0.0233 (2)
O2	0.75640 (11)	0.44325 (18)	0.45465 (7)	0.0205 (2)
N1	0.62474 (12)	0.0450 (2)	0.35771 (8)	0.0142 (3)
C1	0.82708 (14)	0.2676 (3)	0.47641 (10)	0.0176 (3)
C2	0.77559 (15)	0.0331 (3)	0.42386 (11)	0.0196 (3)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0295 (6)	0.0246 (6)	0.0206 (6)	0.0057 (5)	0.0169 (5)	0.0018 (5)
O4	0.0245 (6)	0.0201 (5)	0.0168 (5)	0.0051 (5)	0.0118 (5)	0.0028 (4)

O5	0.0249 (7)	0.0307 (7)	0.0245 (6)	−0.0011 (6)	0.0083 (5)	−0.0043 (5)
O6	0.0222 (10)	0.0222 (9)	0.0391 (10)	0	0.0133 (8)	0
C3	0.0182 (7)	0.0109 (7)	0.0137 (7)	0.0008 (6)	0.0058 (6)	−0.0006 (6)
C4	0.0178 (7)	0.0173 (8)	0.0129 (7)	−0.0015 (6)	0.0077 (6)	0.0009 (6)
C5	0.0161 (7)	0.0148 (7)	0.0161 (7)	−0.0041 (6)	0.0082 (6)	−0.0036 (6)
Cl1	0.02071 (19)	0.0243 (2)	0.02212 (19)	0.00210 (18)	0.01002 (15)	−0.00208 (17)
O1	0.0145 (5)	0.0230 (6)	0.0207 (6)	0.0005 (5)	0.0017 (4)	−0.0033 (5)
O2	0.0204 (6)	0.0147 (5)	0.0203 (5)	0.0016 (5)	0.0068 (5)	0.0013 (4)
N1	0.0150 (6)	0.0113 (6)	0.0135 (6)	−0.0001 (5)	0.0057 (5)	0.0002 (5)
C1	0.0155 (7)	0.0206 (8)	0.0147 (7)	−0.0016 (7)	0.0068 (6)	0.0007 (6)
C2	0.0146 (8)	0.0172 (8)	0.0207 (8)	0.0022 (6)	0.0052 (6)	0.0014 (7)

Geometric parameters (Å, °)

O3—C5	1.3049 (16)	O1—H1O	1.11 (2)
O4—C5	1.2124 (16)	O3—H3O	0.89 (2)
C3—N1	1.5066 (18)	N1—H1N	0.836 (17)
C3—C3^{i}	1.508 (3)	C2—H2A	0.947 (19)
C4—N1	1.5003 (18)	C2—H2B	0.923 (17)
C4—C5	1.504 (2)	C3—H3A	0.928 (16)
O1—C1	1.3080 (17)	C3—H3B	0.965 (19)
O2—C1	1.2063 (17)	C4—H4A	0.974 (17)
N1—C2	1.4968 (19)	C4—H4B	0.962 (16)
C1—C2	1.506 (2)		
N1—C3—C3^{i}	114.64 (11)	C4—N1—H1N	108.2 (15)
N1—C4—C5	109.19 (12)	N1—C2—H2A	108.1 (11)
O4—C5—O3	125.08 (14)	N1—C2—H2B	108.8 (13)
O4—C5—C4	122.08 (12)	C1—C2—H2A	108.2 (11)
O3—C5—C4	112.82 (12)	C1—C2—H2B	113.1 (10)
C2—N1—C4	111.51 (11)	H2A—C2—H2B	109.2 (16)
C2—N1—C3	110.14 (11)	N1—C3—H3A	104.4 (10)
C4—N1—C3	114.03 (11)	N1—C3—H3B	108.3 (8)
O2—C1—O1	126.59 (14)	H3A—C3—H3B	107.6 (14)
O2—C1—C2	122.48 (13)	H3A—C3—C3^{i}	108.6 (12)
O1—C1—C2	110.93 (13)	H3B—C3—C3^{i}	112.7 (9)
N1—C2—C1	109.38 (12)	N1—C4—H4A	110.4 (11)
C1—O1—H1O	110.0 (12)	N1—C4—H4B	109.1 (12)
C5—O3—H3O	109.8 (15)	C5—C4—H4A	109.4 (11)
C2—N1—H1N	105.4 (15)	C5—C4—H4B	109.1 (11)
C3—N1—H1N	107.1 (12)	H4A—C4—H4B	109.6 (13)

Symmetry codes: (i) $-x+1, y, -z+1/2$.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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O6—H6O···Cl1	0.84 (2)	2.27 (2)	3.1062 (11)	172 (2)

Symmetry codes: (ii) $-x+1$, $-y+2$, $-z+1$; (iii) $x+1$, $-y+1$, $z+1/2$; (i) $-x+1$, y, $-z+1/2$.

Fig. 1

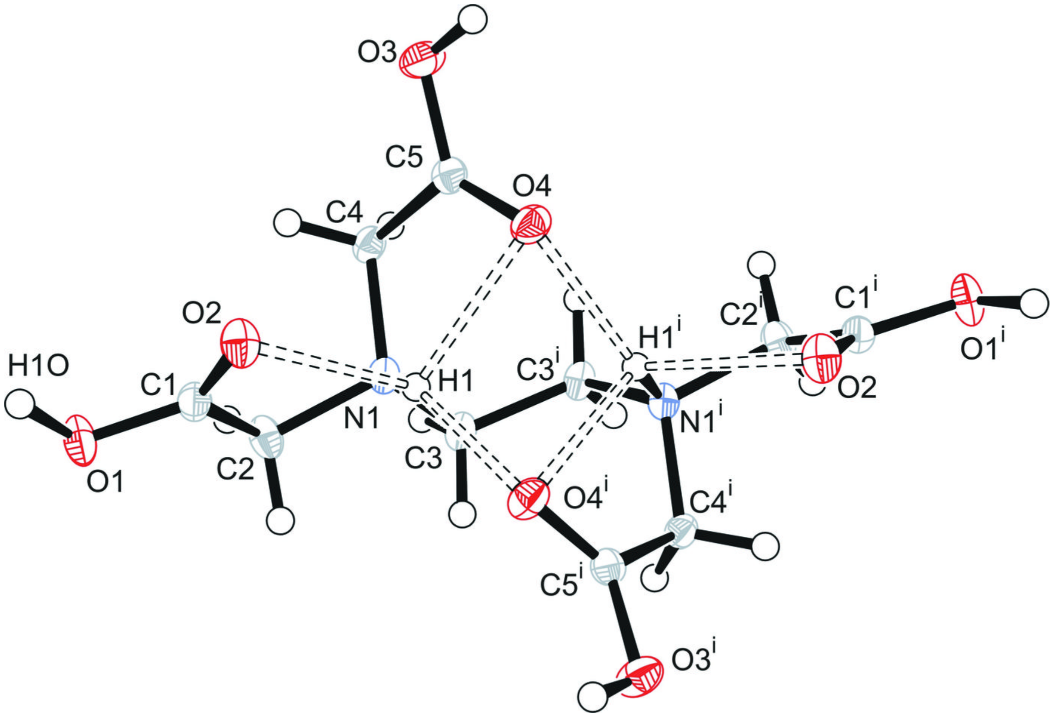
O3
C5
O4
C4
O2
H1O
C1
N1
H1
C3^i
H1^i
C2^i
C1^i
O1^i
O2
N1^i
O1
C2
C3
O4^i
C4^i
C5^i
O3^i

Fig. 2