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N,*N*′-Bis[2-(methoxycarbonyl)ethyl]-ethane-1,2-diammonium dichloride

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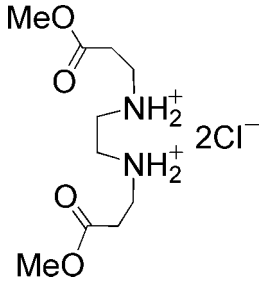
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.037; wR factor = 0.086; data-to-parameter ratio = 11.4.

In the crystal structure of the title compound, $C_{10}H_{22}N_2O_4^{2+}\cdot 2Cl^-$ or $(H_2Me_2eddp)Cl_2$ ($H_2Me_2eddp^{2+}$ is the dimethyl *N*,*N*′-di-3-propanecarboxylatoethane-1,2-diyldiiminium cation), the packing is stabilized by an infinite two-dimensional ···Cl···H—N—H···Cl··· hydrogen-bonding network. In addition, short C—H···Cl contacts are observed.

Related literature

For related literature, see: Aakeröy *et al.* (1999); Bruhn *et al.* (2008); Kaluderović & Sabo (2002); Kaluderović *et al.* (2005, 2007, 2008); Krajčinović *et al.* (2008); Mijatović *et al.* (2005).



Experimental

Crystal data

$C_{10}H_{22}N_2O_4^{2+}\cdot 2Cl^-$
M_r = 305.20
Monoclinic, $P2_1/c$
a = 8.9030 (8) Å
b = 10.3327 (10) Å
c = 8.3269 (10) Å
β = 101.763 (10)°
V = 749.93 (13) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.44 mm^{-1}
T = 293 (2) K
0.42 × 0.12 × 0.10 mm

Data collection

Stoe STADI4 diffractometer
Absorption correction: none
5296 measured reflections
1324 independent reflections
1021 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
2 standard reflections
frequency: 60 min
intensity decay: random variation ±5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.085
S = 1.13
1324 reflections
116 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N—H3···Cl^{i}	0.97 (3)	2.10 (3)	3.064 (2)	171 (2)
N—H4···Cl	0.85 (2)	2.30 (2)	3.092 (2)	156 (2)
C3—H8···Cl^{ii}	0.95 (2)	2.73 (3)	3.619 (3)	156.3 (18)

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

Data collection: *STADI4* (Stoe & Cie, 1996); cell refinement: *STADI4*; data reduction: *STADI4*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 2001); 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: PK2097).

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N,*N*′-Bis[2-(methoxycarbonyl)ethyl]ethane-1,2-diammonium dichloride

Goran N. Kaluderović, Anchan Paethanom, Christoph Wagner, Tibor J. Sabo and Harry Schmidt

S1. Comment

The title compound $(H_2Me_2eddp)Cl_2$ belongs to a class of compounds that have recently been used as ligand precursors in the synthesis of Co(III), Pt(II) and Pt(IV) complexes (Kaluđerović & Sabo, 2002; Kaluđerović *et al.*, 2008). The platinum complexes have been tested against various types of tumor cell lines and some of them have shown promising results in *in vitro* studies (Kaluđerović *et al.*, 2005; Mijatović *et al.*, 2005). There are few crystal structures of these ligand precursors, or indeed of the corresponding platinum complexes, reported in the literature. To date, only four solid state structures of metal complexes containing platinum(IV) (Kaluđerović *et al.*, 2007, 2008; Krajčinović *et al.*, 2008), and only one crystal structure of ligand precursor *O,O′*-diisopropyl-ethylenediammonium-(*S,S*)-di-2-propanoate dichloride, $[(S,S)\text{-}H_2i\text{-}Pr_2eddp]Cl_2$ (Krajčinović *et al.*, 2008) have been described.

Bond lengths and angles for the title compound are in the same range as found for $[(S,S)\text{-}H_2i\text{-}Pr_2eddp]Cl_2$ (Krajčinović *et al.*, 2008). All non H atoms in the $H_2Me_2eddp^{2+}$ cation are essentially co-planar with the largest deviation being for the C1 atom (0.175 (2) Å). The solid-state structure is stabilized by H-bonds. The $H_2Me_2eddp^{2+}$ cations are joined in infinite two-dimensional networks through H-bonds *via* N—H groups and chloride anions (···Cl···H—N—H···Cl···; Figs. 2 and 3). The structural parameters of these two hydrogen bonds (N—H3···Cl = 3.064 (2) Å, N—H3···Cl = 171 (2)°, N—H4···Cl = 3.092 (2) Å, N—H4···Cl = 156 (2)°) are in accord with analogous hydrogen bonds in $[(S,S)\text{-}H_2i\text{-}Pr_2eddp]Cl_2$ (Krajčinović *et al.*, 2008). Furthermore, short C—H···Cl contacts (C—H···Cl = 3.619 (3) Å, C—H···Cl = 156 (2)°) provide additional stabilization to the structure (Aakeröy *et al.*, 1999; Bruhn *et al.*, 2008).

S2. Experimental

The title compound was obtained as described in literature (Kaluđerović & Sabo, 2002). Colourless single crystals suitable for X-ray structure determination were obtained from mother liquor by slow evaporation at room temperature over several days.

S3. Refinement

The amine and methylene H atoms were found in a difference map and refined while methyl H atoms were positioned geometrically and treated as riding, with C–H bond lengths constrained to 0.96 Å.

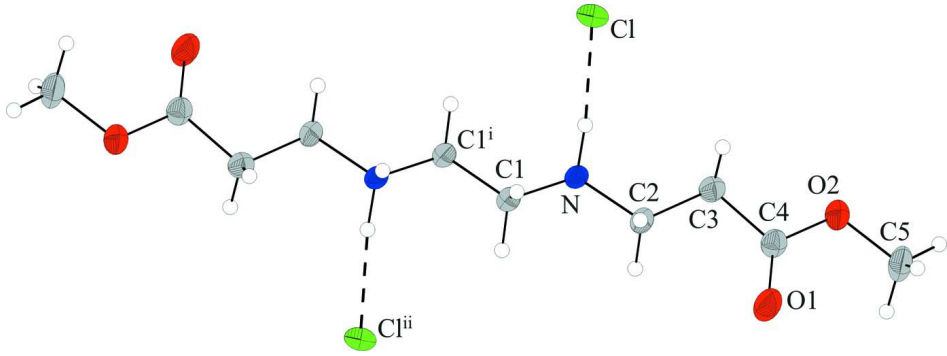

Figure 1

DIAMOND representation of $(H_2Me_2eddp)Cl_2$. [Symmetry code: (i) -*x* + 1, -*y* + 1, -*z* + 1; (ii) 1 - *x*, *y*-1/2, -*z* + 1/2]

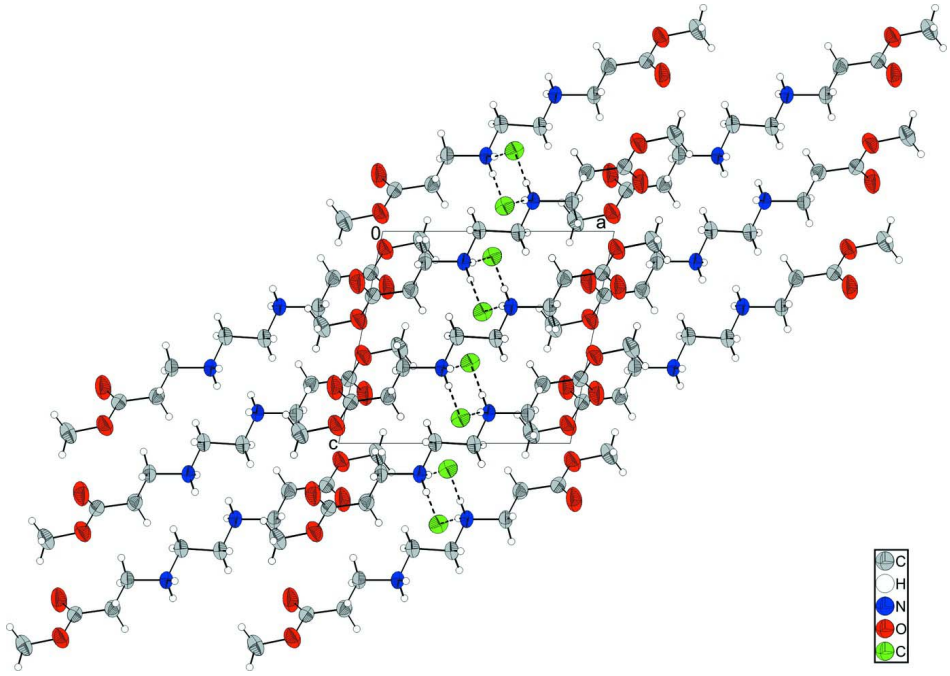

Figure 2

Network of H-bonding viewed along *b*-axis.

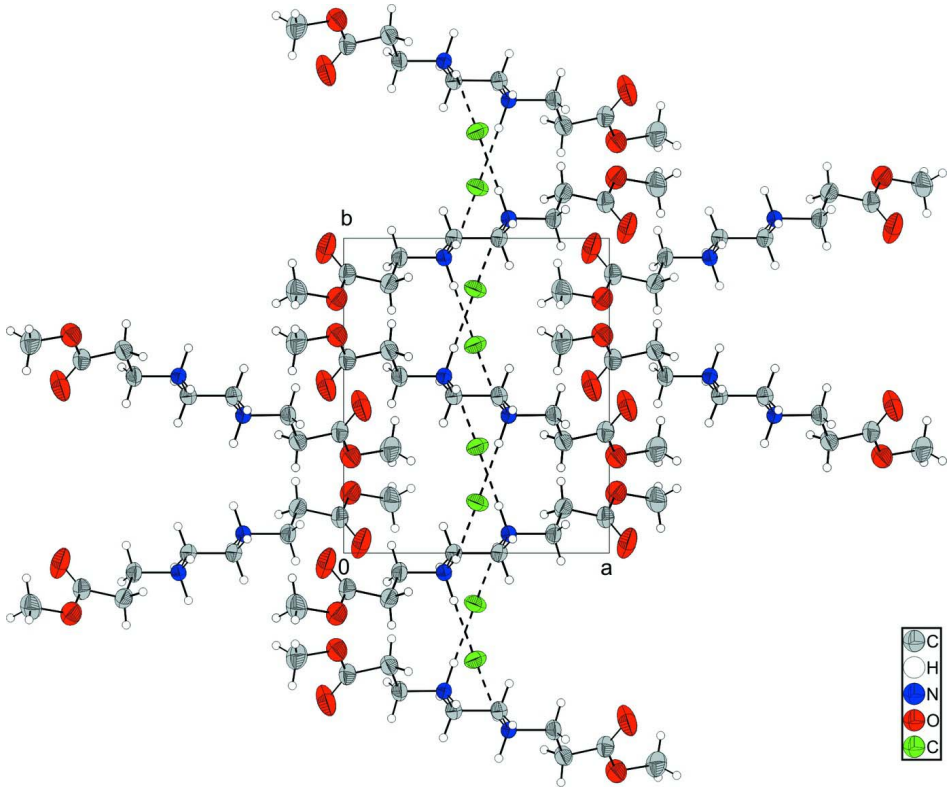

Figure 3

Network of H-bonding viewed along *c*-axis.

N,N′-Bis[2-(methoxycarbonyl)ethyl]ethane-1,2-diammonium dichloride

Crystal data

$C_{10}H_{22}N_2O_4^{2+}\cdot 2Cl^-$
$M_r = 305.20$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.9030\ (8)$ Å
$b = 10.3327\ (10)$ Å
$c = 8.3269\ (10)$ Å
$\beta = 101.763\ (10)°$
$V = 749.93\ (13)$ Å^3
$Z = 2$

$F(000) = 324$
$D_x = 1.352$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 26 reflections
$\theta = 7.7–12.2°$
$\mu = 0.44$ mm^{-1}
$T = 293$ K
Needle, colourless
0.42 × 0.12 × 0.10 mm

Data collection

Stoe STADI4
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
5296 measured reflections
1324 independent reflections
1021 reflections with $I > 2\sigma(I)$

$R_{int} = 0.057$
$\theta_{max} = 25.1°$, $\theta_{min} = 2.3°$
$h = -10\rightarrow10$
$k = -12\rightarrow12$
$l = -9\rightarrow9$
2 standard reflections every 60 min
intensity decay: random variation +−5%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.085$
$S = 1.13$
1324 reflections
116 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0263P)^2 + 0.2601P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.012 (3)

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	0.5853 (3)	0.4998 (2)	0.5051 (3)	0.0387 (6)
H2	0.624 (3)	0.586 (2)	0.507 (3)	0.046 (7)*
H1	0.636 (3)	0.454 (2)	0.596 (3)	0.051 (7)*
C2	0.7898 (3)	0.4385 (3)	0.3579 (3)	0.0401 (6)
H5	0.835 (3)	0.409 (2)	0.462 (3)	0.052 (8)*
H6	0.818 (3)	0.525 (3)	0.344 (3)	0.046 (7)*
C3	0.8277 (3)	0.3565 (3)	0.2221 (3)	0.0421 (6)
H8	0.756 (3)	0.376 (2)	0.126 (3)	0.047 (7)*
H7	0.819 (3)	0.272 (2)	0.248 (3)	0.045 (7)*
C4	0.9857 (3)	0.3853 (2)	0.1953 (3)	0.0417 (6)
C5	1.1771 (3)	0.3294 (3)	0.0491 (3)	0.0609 (8)
H9	1.1942	0.4207	0.0408	0.091*
H11	1.1832	0.2882	−0.0528	0.091*
H10	1.2539	0.2937	0.1356	0.091*
N	0.6217 (2)	0.43799 (19)	0.3563 (3)	0.0345 (5)
H3	0.585 (3)	0.349 (3)	0.352 (3)	0.051 (7)*
H4	0.577 (3)	0.480 (2)	0.271 (3)	0.047 (8)*
O1	1.0669 (2)	0.4681 (2)	0.2636 (3)	0.0799 (7)
O2	1.02593 (19)	0.30760 (17)	0.0849 (2)	0.0566 (5)
Cl	0.49596 (7)	0.66139 (5)	0.11984 (7)	0.0475 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0311 (12)	0.0375 (13)	0.0491 (15)	−0.0017 (10)	0.0122 (11)	−0.0005 (11)
C2	0.0277 (12)	0.0450 (15)	0.0488 (15)	−0.0014 (10)	0.0104 (11)	−0.0051 (12)
C3	0.0363 (13)	0.0453 (16)	0.0444 (14)	−0.0044 (11)	0.0076 (11)	−0.0059 (12)
C4	0.0340 (13)	0.0505 (14)	0.0390 (13)	0.0011 (11)	0.0038 (10)	−0.0028 (11)
C5	0.0503 (16)	0.0728 (19)	0.0679 (18)	0.0073 (14)	0.0312 (14)	−0.0037 (16)
N	0.0272 (10)	0.0305 (10)	0.0461 (12)	0.0013 (8)	0.0080 (8)	0.0056 (9)
O1	0.0440 (11)	0.1084 (17)	0.0928 (16)	−0.0259 (12)	0.0270 (11)	−0.0537 (14)
O2	0.0499 (11)	0.0618 (12)	0.0648 (12)	−0.0047 (9)	0.0271 (9)	−0.0190 (9)
Cl	0.0542 (4)	0.0336 (3)	0.0529 (4)	0.0092 (3)	0.0064 (3)	0.0026 (3)

Geometric parameters (Å, º)

C1—N	1.487 (3)	C3—H7	0.91 (2)
C1—C1^{i}	1.505 (4)	C4—O1	1.188 (3)
C1—H2	0.95 (2)	C4—O2	1.324 (3)
C1—H1	0.93 (3)	C5—O2	1.454 (3)
C2—N	1.494 (3)	C5—H9	0.9600
C2—C3	1.506 (3)	C5—H11	0.9600
C2—H5	0.93 (2)	C5—H10	0.9600
C2—H6	0.94 (3)	N—H3	0.97 (3)
C3—C4	1.498 (3)	N—H4	0.85 (3)
C3—H8	0.94 (2)		
N—C1—C1^{i}	110.0 (3)	H8—C3—H7	109 (2)
N—C1—H2	105.7 (14)	O1—C4—O2	123.0 (2)
C1^{i}—C1—H2	111.0 (14)	O1—C4—C3	124.8 (2)
N—C1—H1	107.8 (15)	O2—C4—C3	112.2 (2)
C1^{i}—C1—H1	111.5 (15)	O2—C5—H9	109.5
H2—C1—H1	111 (2)	O2—C5—H11	109.5
N—C2—C3	111.63 (19)	H9—C5—H11	109.5
N—C2—H5	104.7 (15)	O2—C5—H10	109.5
C3—C2—H5	113.1 (16)	H9—C5—H10	109.5
N—C2—H6	107.3 (15)	H11—C5—H10	109.5
C3—C2—H6	109.6 (15)	C1—N—C2	112.25 (18)
H5—C2—H6	110 (2)	C1—N—H3	108.0 (14)
C4—C3—C2	111.1 (2)	C2—N—H3	109.2 (14)
C4—C3—H8	108.8 (14)	C1—N—H4	109.2 (17)
C2—C3—H8	107.9 (15)	C2—N—H4	107.4 (16)
C4—C3—H7	110.7 (15)	H3—N—H4	111 (2)
C2—C3—H7	109.0 (15)	C4—O2—C5	116.20 (19)
N—C2—C3—C4	164.5 (2)	C3—C2—N—C1	170.4 (2)
C2—C3—C4—O1	−5.0 (4)	O1—C4—O2—C5	−0.5 (4)

C2—C3—C4—O2	175.3 (2)	C3—C4—O2—C5	179.2 (2)
C1^{i}—C1—N—C2	178.3 (3)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—H3···Cl^{ii}	0.97 (3)	2.10 (3)	3.064 (2)	171 (2)
N—H4···Cl	0.85 (2)	2.30 (2)	3.092 (2)	156 (2)
C3—H8···Cl^{iii}	0.95 (2)	2.73 (3)	3.619 (3)	156.3 (18)

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