Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

3,6-Dioxaoctane-1,8-diammonium bis(trichloroacetate)

Mustafa Odabaşoğlu[a]* and Orhan Büyükgüngör[b]

[a]Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Kurupelit Samsun, Turkey, and [b]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Kurupelit Samsun, Turkey

Correspondence e-mail: muodabas@omu.edu.tr

Received 3 January 2006
Accepted 19 January 2006
Online 25 January 2006

In the title compound, $C_6H_{18}N_2O_2^{2+}\cdot 2C_2Cl_3O_2^-$, the cation possesses a twofold rotation axis passing through the mid-point of the central C—C bond. Hydrogen-bonded rings are formed by two cations and two anions. The ions further form a three-dimensional network *via* N—H···O and N—H···Cl hydrogen bonds.

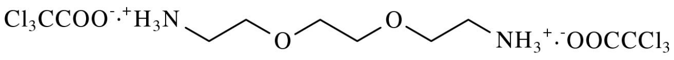
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
R factor = 0.069
wR factor = 0.204
Data-to-parameter ratio = 18.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Intermolecular interactions, especially hydrogen bonds, are probably the most widely used interaction to generate supramolecularly organized organic systems with a variety of novel structural features (Prins *et al.*, 2001; Desiraju, 1995). Hydrogen bonding has been used effectively to predict and design supramolecular assemblies in one and two dimensions (Karle *et al.*, 1997). We have been interested in supramolecularly hydrogen-bonded systems formed by organic amines and carboxylic acids (Odabaşoğlu, Büyükgüngör & Lönnecke, 2003; Odabaşoğlu, Büyükgüngör, Turgut *et al.*, 2003; Odabaşoğlu & Büyükgüngör, 2006). The present work is part of a structural study of compounds of organic ammonium systems with hydrogen-bond donors and we report here the structure of the title compound, (I) (Fig. 1).



(I)

In compound (I), the cation possesses a twofold rotation axis passing through the mid-point of the central C—C bond. In (I), the 3,6-dioxaoctane-1,8-diammonium ions are linked to the trichloroacetate ions through N—H···O and N—H···Cl hydrogen bonds, resulting in the formation of hydrogen-bonded rings (Fig. 2 and Table 2). The hydrogen-bonded rings are formed within a three-dimensional network. It is generally recognized that N—H···O bonds of the type $N^+\cdots O^-$ with matching pK_a values of hydrogen-bond donor and acceptor groups are stronger than N—H···O bonds involving neutral acceptors and donors. These bonds traditionally are considered to be salt bridges and are termed low barrier hydrogen bonds or charge-assisted hydrogen bonds (Hess & Reinhardt, 1999). Accordingly, the strongest N—H···O hydrogen bonds are found in amine–carboxylates which possess linear hydrogen bonds, with an average N···O bond distance of 2.811 Å and an average N···O bond angle of 158°. The title compound has a slightly longer N—H···O bond distance and a slightly smaller N—H···O bond angle (average N···O bond distance = 2.835 Å and average bond angle N—H···O = 151°). In (I), the C3—N1 bonds are equal to a normal Csp^3—Nsp^3

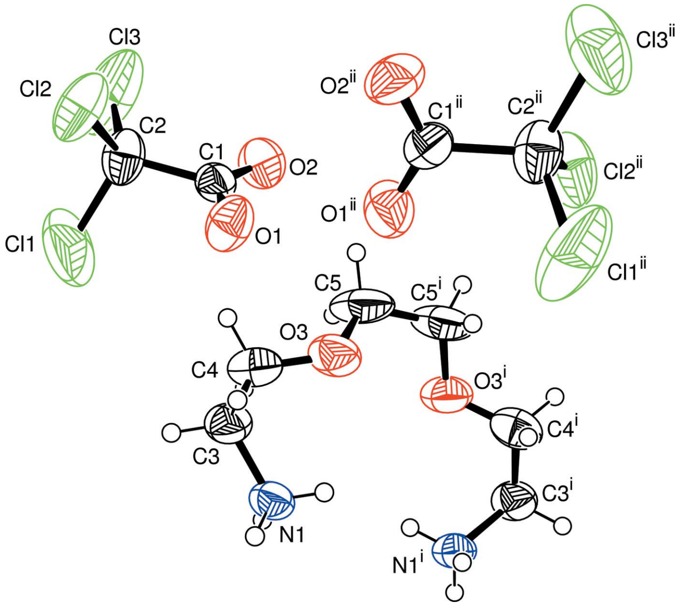


Figure 1
A view of the ions of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x + 2, y, -z + \frac{3}{2}$; (ii) $-x, y, -z + \frac{1}{2}$.]

single-bond length (Vaidhyanathan *et al.*, 2002) but the C1—C2 bond is slightly longer than a normal C—C single bond. This elongation is attributed to the repulsive effect of electrons on the Cl and O atoms.

Experimental

The title compound was prepared by mixing 2-[2-(2-aminoethoxy)ethoxy]ethanamine and trichloroacetic acid in a 1:2 molar ratio in water at 353 K. Crystals of (I) were obtained by slow evaporation of the solvent (m.p. 380–381 K).

Crystal data

$C_6H_{18}N_2O_2^{2+}\cdot 2C_2Cl_3O_2^-$
$M_r = 474.96$
Monoclinic, $C2/c$
$a = 22.1995$ (19) Å
$b = 7.5606$ (10) Å
$c = 11.9320$ (12) Å
$\beta = 91.188$ (7)°
$V = 2002.3$ (4) Å^3
$Z = 4$
$D_x = 1.576$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9773 reflections
$\theta = 2.5$–27.8°
$\mu = 0.89$ mm^{-1}
$T = 296$ (2) K
Plate, colourless
0.67 × 0.45 × 0.11 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.611$, $T_{max} = 0.898$
9773 measured reflections
1976 independent reflections
1352 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 26.0°$
$h = -27 \rightarrow 27$
$k = -9 \rightarrow 9$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.069$
$wR(F^2) = 0.204$
$S = 1.07$
1976 reflections
109 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.091P)^2 + 4.5621P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.71$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

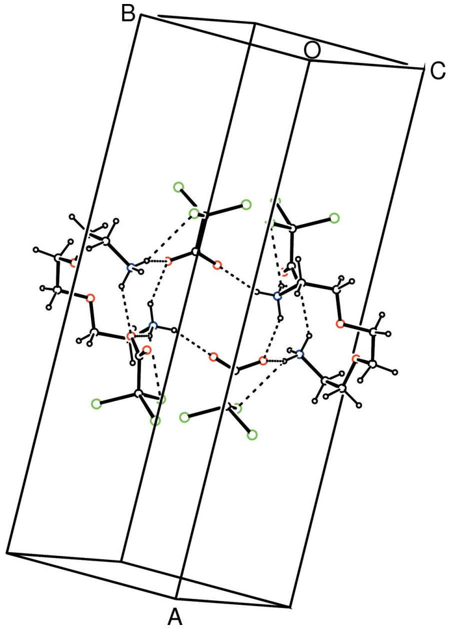


Figure 2
A partial packing diagram of the title compound, showing the hydrogen-bonding scheme (dashed lines).

Table 1
Selected geometric parameters (Å, °).

C1—O2	1.216 (5)	C2—Cl2	1.765 (5)
C1—O1	1.219 (5)	C3—N1	1.484 (5)
C1—C2	1.565 (6)	C3—C4	1.486 (7)
C2—Cl3	1.750 (5)	C5—C5^{i}	1.461 (12)
C2—Cl1	1.757 (6)		
O2—C1—O1	128.7 (4)	C1—C2—Cl1	107.2 (3)
O2—C1—C2	116.6 (4)	Cl3—C2—Cl1	109.6 (3)
C1—C2—Cl3	113.5 (3)		

Symmetry code: (i) $-x + 2, y, -z + \frac{3}{2}$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H5C···O2^{iii}	0.89	1.95	2.788 (5)	157
N1—H5D···O1^{iv}	0.89	2.11	2.878 (5)	143
N1—H5E···O1^{v}	0.89	2.02	2.838 (5)	152
N1—H5E···Cl2^{v}	0.89	2.69	3.343 (4)	131

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

All H atoms were refined using a riding model, with C—H = 0.97 Å [$U_{iso}(H) = 1.2U_{eq}$(parent atom)] for methylene C atoms and N—H = 0.89 Å [$U_{iso}(H) = 1.5U_{eq}$(parent atom)] for ammonium N atoms.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

Desiraju, G. R. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 2311–2327.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Hess, R. A. & Reinhardt, L. A. J. (1999). *J. Am. Chem. Soc.* **121**, 9867–9870.

Karle, I. L., Ranganathan, D. & Haridas, V. (1997). *J. Am. Chem. Soc.* **119**, 2777–2783.

Odabaşoğlu, M. & Büyükgüngör, O. (2006). *Acta Cryst.* E**62**, o236–o238.

Odabaşoğlu, M., Büyükgüngör, O. & Lönnecke, P. (2003). *Acta Cryst.* C**59**, o51–o52.

Odabaşoğlu, M., Büyükgüngör, O., Turgut, G., Karadağ, A., Bulak, E. & Lönnecke, P. (2003). *J. Mol. Struct.* **648**, 133–138.

Prins, L. J., Timmerman, P. & Reinhoudt, D. N. (2001). *J. Am. Chem. Soc.* **123**, 10153–10163.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL97.* University of Göttingen, Germany.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.

Vaidhyanathan, R., Natarajan, S. & Rao, C. N. R. (2002). *J. Mol. Struct.* **608**, 123–133.