Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Cyclohexylammonium dichloroacetate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.030
wR factor = 0.078
Data-to-parameter ratio = 21.0

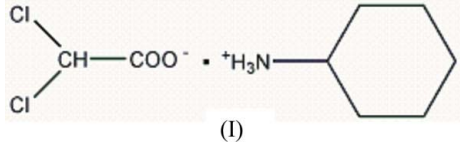
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_6H_{14}N^+\cdot C_2HCl_2O_2^-$, (I), was obtained as a byproduct of the oxidation of the chiral compound 5-(*R*)-menthyloxybutyrolacto[3,4-*b*]-2(*S*)-6(*R*)-1-*N*-cyclohexylaziridine. The structure determination of (I) shows that the cations and anions are connected through weak intermolecular hydrogen bonding, forming a complicated network structure.

Received 15 September 2005
Accepted 28 October 2005
Online 5 November 2005

Comment

Weak non-covalent interactions between molecules, such as hydrogen bonds and π-π stacking effects, can play important roles in determining crystal structures (Desiraju *et al.*, 1999; Mingos, 2004; Ma *et al.*, 2005; Zhu, *et al.*, 2004; Men, *et al.*, 2005; Dimartino *et al.*, 2005; Sontjens *et al.*, 2001; Kim, *et al.*, 2005). Recently, 5-(*R*)-menthyloxybutyrolacto[3,4-*b*]-2(*S*)-6(*R*)-1-*N*-cyclohexylaziridine was oxidized at room temperature when treated with hydrogen peroxide and hydrochloric acid (Guo *et al.*, 2005). However, instead of the expected aziridine product containing two carboxyl groups, the title compound, (I), was obtained. We report here its crystal structure.



(I)

The structure of (I) (Fig. 1) comprises a cyclohexylammonium cation and a dichloroacetate anion. The C1—O1 and C1—O2 bond lengths in the carboxylate group are 1.240 (2) and 1.232 (2) Å, respectively. Hydrogen-bonding interactions play an important role in the structure of the title compound (Fig. 2), with a number of different kinds of

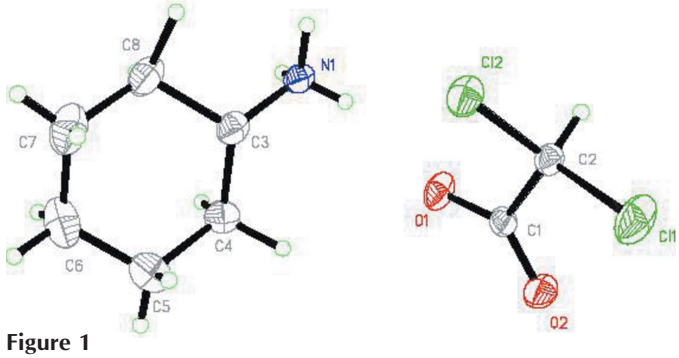


Figure 1
The structure (30% probability displacement ellipsoids) of the title compound.

hydrogen bonding, *viz*. hydrogen bonding between N—H and the O atoms of three dichloroacetate anions, and hydrogen bonding between N—H and the Cl atoms (Table 1). Noteworthy is the presence of a bifurcated hydrogen bond, N1—H1*A*···O1 and N1—H1*A*···Cl2. In addition, there are also a number of weak C—H···Cl and C—H···O hydrogen bonds in the structure.

Experimental

5-(*R*)-Menthyloxybutyrolacto[3,4-*b*]-2(*S*)-6(*R*)-1-*N*-cyclohexylaziridine (0.671 g, 2 mmol) was dissolved in 5 ml acetonitrile. Concentrated hydrochloric acid (0.344 ml, 4 mmol) and excess hydrogen peroxide (2 ml) were then added and the mixture stirred for 24 h at room temperature. The crude product obtained was purified by column chromatography on silica gel to obtain the purified compound (0.411 g). Colourless crystals were grown by slow evaporation of a petroleum–ethanol solution (4:1 *v*/*v*).

Crystal data

$C_6H_{14}N^+\cdot C_2HCl_2O_2^-$
M_r = 228.10
Orthorhombic, $P2_12_12_1$
a = 6.6435 (8) Å
b = 8.5960 (10) Å
c = 19.135 (2) Å
V = 1092.8 (2) Å^3
Z = 4
D_x = 1.380 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5012 reflections
θ = 2.6–28.6°
μ = 0.56 mm^{-1}
T = 293 (2) K
Block, colourless
0.36 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
9724 measured reflections
2497 independent reflections
2308 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.5°
$h = -8 \rightarrow 8$
$k = -11 \rightarrow 11$
$l = -23 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.078
S = 1.08
2497 reflections
119 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0362P)^2 + 0.2076P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Absolute structure: Flack (1983), with 1020 Friedel pairs
Flack parameter: 0.02 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*C*···O1^{i}	0.89	1.94	2.8194 (19)	170
N1—H1*B*···O2^{ii}	0.89	1.89	2.7765 (19)	175
N1—H1*A*···Cl2	0.89	2.90	3.3868 (15)	116
N1—H1*A*···O1	0.89	1.95	2.8237 (19)	166

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

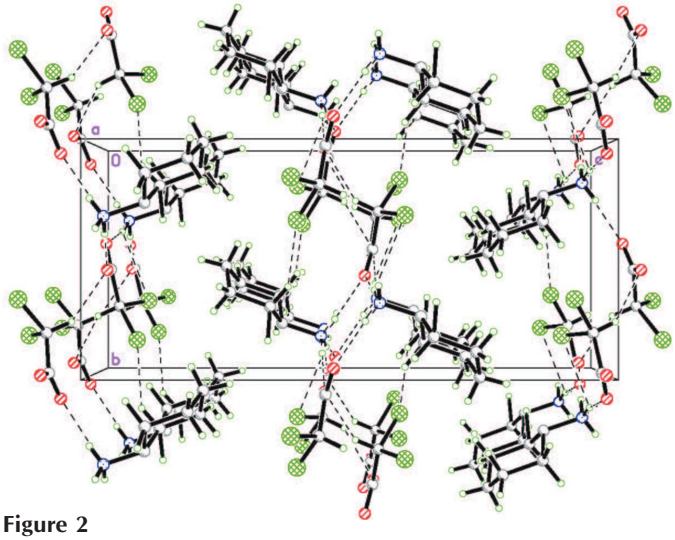


Figure 2
View along the *a* axis of the packing of the title compound, showing the hydrogen-bonding interactions as dashed lines.

H atoms were placed in calculated positions (N–H = 0.89, C—H = 0.97 Å) and refined as riding, with U_{iso}(H) = 1.5U_{eq}(N) and 1.2U_{eq}(C).

Data collection: *APEX2* (Bruker 2004); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker 2004); software used to prepare material for publication: *SHELXTL*.

This work was supported by the National Natural Science Foundation of China (grant No. 29672004).

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