

Table 1. Selected geometric parameters (\AA , $^\circ$)

S1—O5	1.447 (2)	O1A—C11A	1.298 (3)
S1—O6	1.470 (2)	O2A—C11A	1.211 (3)
S1—O7	1.476 (2)	O1B—C11B	1.291 (3)
S1—O8	1.466 (2)	O2B—C11B	1.182 (3)
O5—S1—O6	109.9 (1)	O1A—C11A—O2A	125.2 (2)
O5—S1—O7	109.9 (1)	O1A—C11A—C4A	114.5 (2)
O5—S1—O8	112.2 (1)	O2A—C11A—C4A	120.2 (2)
O6—S1—O7	107.8 (1)	O1B—C11B—O2B	123.5 (3)
O6—S1—O8	109.6 (1)	O1B—C11B—C4B	112.9 (3)
O7—S1—O8	107.3 (1)	O2B—C11B—C4B	123.6 (3)
H1O9—O9—H2O9	112 (3)		

Table 2. Geometry of hydrogen bonds and leading intermolecular C—H...O interactions in (I) (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
O1A—H1O1A...O6	1.16 (4)	1.34 (4)	2.493 (2)	172 (3)
O1B—H1O1B...O9	0.96 (4)	1.62 (4)	2.572 (3)	172 (4)
N1A—H1N1A...O7 ⁱ	1.01 (3)	1.61 (3)	2.592 (3)	163 (3)
N1B—H1N1B...O8 ⁱⁱ	1.00 (3)	1.66 (3)	2.650 (3)	169 (3)
O9—H1O9...O2A ⁱⁱⁱ	0.93 (4)	1.92 (4)	2.846 (3)	170 (4)
O9—H2O9...O5 ^{iv}	0.77 (4)	2.16 (4)	2.892 (3)	159 (4)
O9—H2O9...O7 ^{iv}	0.77 (4)	2.45 (3)	3.072 (3)	139 (3)
C3A—H3A...O2A ⁱⁱⁱ	0.98	2.34	3.255 (3)	154
C2B—H2B...O9 ^v	0.98	2.46	3.293 (4)	142
C2B—H2B...O6 ^{vi}	0.98	2.47	3.210 (3)	133
C8A—H8A...O7 ^{vii}	0.98	2.50	3.191 (3)	127
C8B—H8B...O5 ^{viii}	0.98	2.52	3.435 (4)	155
C7B—H7B...O5 ^{viii}	0.98	2.57	3.290 (3)	130
C6A—H6A...O1A ^{viii}	0.98	2.57	3.514 (3)	163
C7A—H7A...O7 ^{viii}	0.98	2.61	3.461 (3)	146
C2A—H2A...O9	0.98	2.67	3.498 (4)	142

Symmetry codes: (i) $x, y, 1+z$; (ii) $1-x, -y, 1-z$; (iii) $1-x, 1-y, 1-z$; (iv) $-x, 1-y, 1-z$; (v) $1-x, -y, 2-z$; (vi) $x, y, -1, 1+z$; (vii) $1+x, y-1, z$; (viii) $-x, 2-y, 1-z$.

Table 3. Basic first- and second-level graph set descriptors involving conventional hydrogen bonds which are designated a–g in the order given in Table 2

	a	b	c	d	e	f	g
a	D	—	C ₂ ³ (11)	D ₂ ³ (7)	D ₂ ³ (7)	D ₂ ³ (7)	D ₂ ³ (7)
b		D	—	D ₂ ³ (10)	D ₂ ³ (5)	D ₂ ³ (5)	D ₂ ³ (5)
c			D	D ₂ ³ (7)	D ₂ ³ (10)	D ₂ ³ (7)	D ₂ ³ (5)
d				D	—	D ₂ ³ (7)	D ₂ ³ (7)
e					D	D ₂ ³ (5)	D ₂ ³ (5)
f						D	R ₂ ¹ (4)
g							D

H—C) atoms were allowed for as riding atoms; coordinates for H atoms on N and O atoms were determined from difference maps and refined isotropically.

Data collection: *MSCI/AF C Diffraction Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCI/AF C Diffraction Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *TEXSAN* and *PLATON* (Spek, 1990).

We acknowledge with pleasure our use of the departmental X-ray crystallographic facility, which is supervised by staff crystallographer Dr J. C. Gallucci. The diffractometer was purchased with funds provided in part by an NIH grant.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1225). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 2145–2147

Dicyclohexylammonium trifluoroacetate

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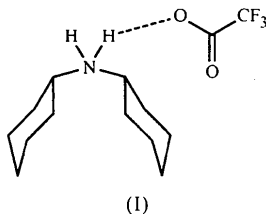
(Received 6 July 1999; accepted 20 August 1999)

Abstract

Two hydrogen bonds [N...O = 2.785 (2) and 2.794 (2) \AA] link the cations and anions of dicyclohexylammonium trifluoroacetate, C₁₂H₂₄N⁺·C₂F₃O₂⁻, into a zigzag chain.

Comment

The wide range of bond dimensions that has been observed in the structures of compounds possessing the trifluoroacetato anion can be attributed to the difficulty of locating the F atoms. The $-CF_3$ group is commonly freely rotating, and the problem of free rotation is sometimes compounded by heavy atoms that reduce the contribution of the rotating $-CF_3$ group to the measured intensities (Gleghorn & Small, 1995). In the structure of trifluoroacetic acid itself, the group does not show free rotation; the structure consists of pairs of molecules of the acid hydrogen-bonded across a centre of symmetry (Nahringbauer *et al.*, 1979). The $-CF_3$ group in the title compound, (I), is freely rotating, a feature that is in agreement with the absence of any strong hydrogen-bonding interactions between the ammonium-H atoms and the F atoms. Two hydrogen bonds [$N \cdots O = 2.785(2), 2.794(2)$ Å] link the cations and anions into a zigzag chain running parallel to the *c* axis. A similar pair of hydrogen bonds [$N \cdots O = 2.733(2), 2.790(2)$ Å] in 1-methylhydrazinium trifluoroacetate link the cation (which is isoelectronic with the dimethylammonium cation) to the anion to form a zigzag chain (Shklover & Struchkov, 1985). In the 1,3-diphenylguanidinium salt, as both O atoms are engaged in twin hydrogen bonds, the



chain-forming, hydrogen-bonding interactions [$N \cdots O = 2.852(3), 3.111(3)$ Å] are somewhat longer (Paixão *et al.*, 1998). With an R_3NH^+ salt, for which only one hydrogen bond is possible, the carboxyl $-CO_2$ portion of the anion should display unambiguous single and double C—O bonds; this expectation is only partially realised in the pyridinium salt, which crystallizes as three independent ion-pairs. Only one $-CO_2$ group displays C—O distances of standard single/double bonds; the C—O distances in the other two $-CO_2$ groups are in reasonable agreement with the average distance of 1.254 Å calculated (from *ab initio* molecular orbital theory) for the carboxyl fragment in the trifluoroacetato ion (Gleghorn & Small, 1995). In the pyridinium salt, the hydrogen bonds are much shorter [$N \cdots O = 2.664(4), 2.681(4), 2.698(4)$ Å; Palmore & McBride-Wieser, 1997].

The dicyclohexylammonium cation has been used as the counterion in a number of *sec*-ammonium carboxylates (Ng *et al.*, 1991; Ng, 1992, 1993, 1994, 1995*a,b*, 1996, 1997). The C—N—C angles in the salts all exceed the 109.5° expected for an sp^2 -hybridized N atom, and

are typified by the 117.4(1)° angle found in the present trifluoroacetate [$N—C = 1.505(2), 1.507(2)$ Å]. The bond dimensions are in excellent agreement with the results of molecular mechanics calculations [$N—C = 1.508, 1.508$ Å, $C—N—C = 117.5^\circ$] performed with the *MM3+* basis set (Hypercube Inc., 1999).

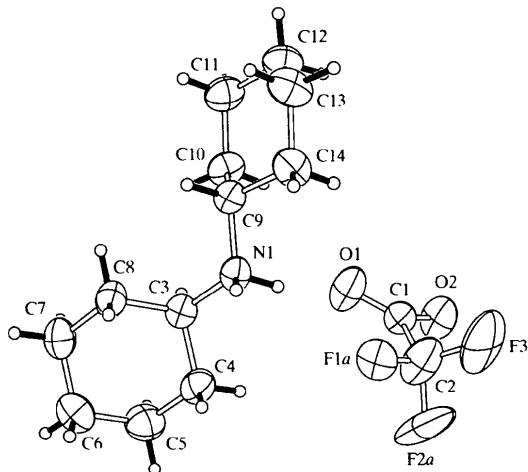
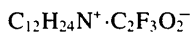


Fig. 1. ORTEP (Johnson, 1976) plot of dicyclohexylammonium trifluoroacetate at the 50% probability level. H atoms are drawn as circles of an arbitrary radius. For clarity only one set of partially occupied F atom sites is shown (see text).

Experimental

Equimolar quantities of dicyclohexylamine and trifluoroacetic acid when heated in a small volume of ethanol precipitated dicyclohexylammonium trifluoroacetate when the solution was cooled.

Crystal data



$M_r = 295.34$

Monoclinic

$P2_1/c$

$a = 9.8336(1)$ Å

$b = 16.7110(3)$ Å

$c = 9.8352(1)$ Å

$\beta = 104.223(1)^\circ$

$V = 1566.67(4)$ Å³

$Z = 4$

$D_x = 1.252$ Mg m⁻³

D_m not measured

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 7857 reflections

$\theta = 2.44\text{--}28.28^\circ$

$\mu = 0.106$ mm⁻¹

$T = 298(2)$ K

Block

$0.48 \times 0.48 \times 0.46$ mm

Colourless

Data collection

Siemens SMART CCD area-detector diffractometer

ω scans

Absorption correction:

empirical (*SADABS*;

Sheldrick, 1996)

$T_{min} = 0.951, T_{max} = 0.953$

10 465 measured reflections

3845 independent reflections

2924 reflections with

$I > 2\sigma(I)$

$R_{int} = 0.030$

$\theta_{max} = 28.28^\circ$

$h = -13 \rightarrow 12$

$k = -17 \rightarrow 22$

$l = -12 \rightarrow 13$

RefinementRefinement on F^2 $R[F^2 > 2\sigma(F^2)] = 0.047$ $wR(F^2) = 0.152$ $S = 1.064$

3845 reflections

297 parameters

H atoms treated by a

mixture of independent
and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0841P)^2 + 0.132P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.27 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$$

Extinction correction: none

Scattering factors from

*International Tables for
Crystallography* (Vol. C)Table 1. *Hydrogen-bonding geometry* (\AA , $^\circ$)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H1A...O1	0.95 (2)	1.86 (2)	2.785 (2)	165 (2)
N1—H1B...O2'	0.91 (2)	1.90 (2)	2.794 (2)	169 (2)

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

The H atoms were refined as riding with $U(\text{H}) = 1.5U_{\text{eq}}(\text{C})$. The ammonium-H atoms were located and refined. The CF_3 group is freely rotating in the structure, and this behaviour was initially modelled as three CF_3 groups sharing a common C atom, subject to $\text{C—F} = 1.29 \pm 0.01 \text{ \AA}$ and $\text{F} \cdots \text{F} = 2.11 \pm 0.02 \text{ \AA}$. The C—F distance of 1.29 \AA is the mean from a number of measurements of trifluoroacetates (Gleghorn & Small, 1995). For a free CF_3CO_2^- anion, a sixfold barrier to rotation about the C—C bond is expected. As the barrier is low, of the order of $10\text{--}20 \text{ J mol}^{-1}$, the most appropriate model would be one involving a continuous, uniform distribution of the F electrons. As an alternative, the electrons were modelled as six sets of F atoms. Each set was given the same displacement parameters by an EADP instruction, and the 18 F atoms were restrained into an approximate circle by a 'FLAT \$F' instruction. For this model, one of the sets had an unacceptably large displacement parameter, and the anion was modelled instead over five sets of F atoms. All F atoms were refined anisotropically, but an 'ISOR \$F 0.01' restraint had to be used.

Data collection: *SMART* (Siemens, 1996a). Cell refinement: *SAINT* (Siemens, 1996b). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL97*.

We thank the National Science Council for R&D, Malaysia (IRPA 09-02-03-0371 and IRPA 190-4609-2801) for supporting this work.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1050). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). **C55**, 2147–2149

1,4-Bis(2-hydroxy-5-methylbenzyl)-piperazine

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(Received 10 May 1999; accepted 9 August 1999)

Abstract

The title compound, $\text{C}_{20}\text{H}_{26}\text{N}_2\text{O}_2$, lies across a crystallographic inversion centre. The piperazine ring adopts a chair conformation. The molecules are stabilized by intramolecular $\text{O—H} \cdots \text{N}$ -type hydrogen bonds in addition to van der Waals forces.

Comment

Piperazine salts are very active against roundworm and pinworm infections in both humans and animals. These drugs are absorbed from the gastrointestinal