

2,2,6,6-Tetramethyl-4-oxopiperidinium trifluoroacetate

Sven Breitung,^a Hans-Wolfram Lerner^a and Michael Bolte^{b*}^aInstitut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany, and ^bInstitut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

Key indicators

Single-crystal X-ray study

T = 173 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.049

wR factor = 0.141

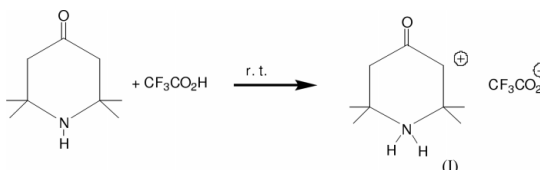
Data-to-parameter ratio = 12.1

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

Both ions of the title compound, $\text{C}_9\text{H}_{18}\text{NO}^+\cdot\text{CF}_3\text{CO}_2^-$, are located on a crystallographic mirror plane. The piperidone ring adopts an almost ideal chair conformation. The different ions form hydrogen-bonded chains, extending parallel to the crystallographic *a* axis.

Comment

Piperidine derivatives have found widespread use as bulky bases in organic chemistry. We report here the reaction of 2,2,6,6-tetramethyl-4-piperidone with trifluoroacetic acid and the X-ray crystal structure analysis of 2,2,6,6-tetramethyl-4-oxopiperidinium trifluoroacetate, (I). The synthesis of (I) was achieved as indicated in the *Scheme* below.



Both ions of (I) are located on a crystallographic mirror plane. Only the methyl groups and the sp^3 C atoms of the piperidone ring and one of the F atoms are located in general positions. Bond lengths and angles are in the usual ranges. The piperidone ring adopts an almost ideal chair conformation, with $Q = 0.525$ (2) Å , $\theta = 176.2$ (3) $^\circ$, $\varphi = 360$ (3) $^\circ$ (Cremer & Pople, 1975). The anions and cations form infinite hydrogen-bonded chains along the crystallographic *a* axis.

Experimental

The title compound was produced from 2 mmol 2,2,6,6-tetramethyl-4-piperidone in 5 ml CHCl_3 , in the presence of 3 mmol trifluoroacetic acid at ambient temperature. Colourless crystals of (I) were grown by storing this solution at room temperature for 2 d. ^1H NMR (CDCl_3 , internal TMS): δ 1.26 (s, 4 \times CH_3), 2.34 (s, 2 \times CH_2), 5.67 (s, br, NH_2). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , internal TMS): δ 30.8 (s, CH_3), 52.7 (s, 2 CH_2), 56.8 (s, 2 \times NCMe), 116.5 (q, CF_3 , $^1J_{\text{FC}} = 292.8$ Hz), 161.7 (q, CO_2 , $^2J_{\text{FC}} = 35.1$ Hz).

Crystal data

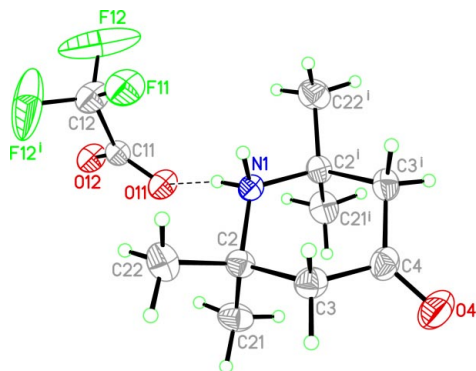
 $\text{C}_9\text{H}_{18}\text{NO}^+\cdot\text{C}_2\text{F}_3\text{O}_2^-$
 $M_r = 269.26$ Monoclinic, $P2_1/m$ $a = 6.6117$ (14) Å $b = 9.1388$ (15) Å $c = 11.3024$ (19) Å $\beta = 100.450$ (17) $^\circ$ $V = 671.6$ (2) Å^3 $Z = 2$ $D_x = 1.332$ Mg m^{-3} Mo $K\alpha$ radiation

Cell parameters from 3675 reflections

 $\theta = 3.7\text{--}25.1^\circ$ $\mu = 0.12$ mm^{-1} $T = 173$ (2) K

Block, colourless

 $0.29 \times 0.18 \times 0.11$ mm


Figure 1

Perspective view of the title compound with the atom numbering; displacement ellipsoids are at the 50% probability level. Symmetry operator for generating equivalent atoms: (i) $x, -y + \frac{3}{2}, z$.

Data collection

Stoe IPDS-II two-circle diffractometer	1215 independent reflections
ω scans	906 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (MULABS; Spek, 1990; Blessing, 1995)	$R_{\text{int}} = 0.041$
$T_{\text{min}} = 0.966, T_{\text{max}} = 0.984$	$\theta_{\text{max}} = 25.2^\circ$
3390 measured reflections	$h = -7 \rightarrow 7$
	$k = -10 \rightarrow 10$
	$l = -12 \rightarrow 13$

Refinement

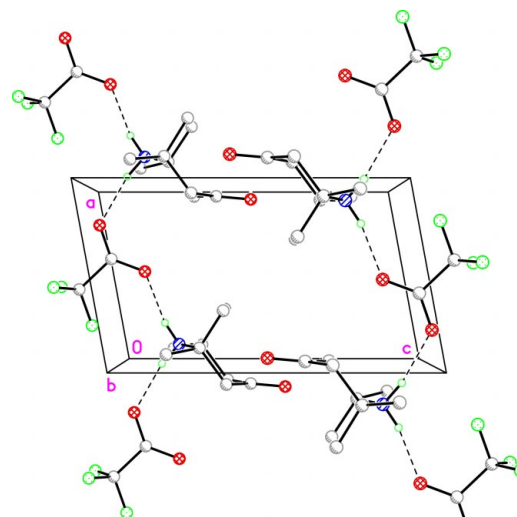
Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0836P)^2 + 0.1272P]$
$R[F^2 > 2\sigma(F^2)] = 0.049$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.141$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.55 \text{ e } \text{\AA}^{-3}$
1215 reflections	$\Delta\rho_{\text{min}} = -0.43 \text{ e } \text{\AA}^{-3}$
100 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Hydrogen-bonding geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A \cdots O11	0.89 (4)	1.91 (4)	2.787 (3)	168 (3)
N1—H1B \cdots O12 ⁱ	0.91 (4)	1.94 (4)	2.840 (3)	168 (3)

Symmetry code: (i) $x - 1, y, z$.


Figure 2

Packing of the title compound. View perpendicular to the ac plane. Only the H atoms bonded to N are shown. Atom codes: C shaded black circles, H small open green circles, F dotted green circles, N blue shaded circles, O cross-hatched red circles.

H atoms bonded to C atoms were refined with fixed individual displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$] using a riding model, with $\text{C}-\text{H} = 0.99 \text{ \AA}$ or methyl $\text{C}-\text{H} = 0.98 \text{ \AA}$. H atoms bonded to N atoms were refined isotropically.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

References

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