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4-Hydroxy-2,2,6,6-tetramethylpiperidinium trichloroacetate

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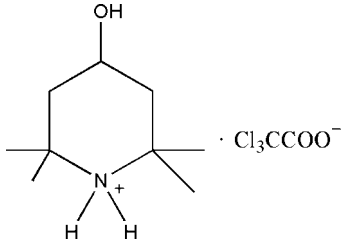
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.024; wR factor = 0.060; data-to-parameter ratio = 16.0.

In the crystal structure of the title compound, $C_9H_{20}NO^+\cdot Cl_3CCOO^-$, the cations and anions are connected via O—H···O, N—H···O, O—H···Cl and N—H···Cl hydrogen bonding. The six-membered ring adopts a chair conformation with the hydroxyl group in an equatorial position.

Related literature

For related literature, see: Borzatta & Carrozza (1991).



Experimental

Crystal data

$C_9H_{20}NO^+\cdot C_2Cl_3O_2^-$
M_r = 320.63
Monoclinic, $P2_1$
a = 6.3468 (13) Å
b = 14.450 (3) Å
c = 8.2175 (16) Å
β = 95.19 (3)°
V = 750.5 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.61 mm^{-1}
T = 113 (2) K
0.12 × 0.10 × 0.08 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.930, T_{max} = 0.953
5459 measured reflections
2858 independent reflections
2636 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.024
$wR(F^2)$ = 0.060
S = 1.06
2858 reflections
179 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Absolute structure: Flack (1983), 996 Friedel pairs
Flack parameter: 0.04 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O3^{i}	0.89 (3)	1.99 (3)	2.8095 (18)	152 (3)
O1—H1···Cl1^{i}	0.89 (3)	2.92 (3)	3.6201 (16)	136 (2)
N1—H1A···O3^{ii}	0.95 (3)	1.87 (3)	2.8085 (19)	170 (2)
N1—H1B···O2^{iii}	0.94 (2)	1.87 (2)	2.796 (2)	165.1 (19)
N1—H1B···Cl2^{iii}	0.94 (2)	2.94 (2)	3.5647 (16)	124.5 (16)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2380).

References

Borzatta, V. & Carrozza, P. (1991). European Patent EP 0 462 069.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, o641 [doi:10.1107/S1600536808005369]

4-Hydroxy-2,2,6,6-tetramethylpiperidinium trichloroacetate

Peng-Wei Zhang, Tong-Yun Zhang, Li Zhang and Yi Deng

S1. Comment

The title compound was obtained as a byproduct in the synthesis of hindered amine light stabilizers preventing the degradation of polyolefins in sunlight, in which 2,2,6,6-tetramethylpiperidin-4-ol is a very important intermediate (Borzatta & Carrozza,1991). We report here the crystal structure 4-hydroxy-2,2,6,6-tetramethylpiperidinium trichloroacetate (Fig. 1). Intermolecular O—H···O, N—H···O, O—H···Cl, N—H···Cl hydrogen bonds are observed which help to establish the crystal packing. The piperidine ring adopts a chair conformation.

S2. Experimental

0.25 g (1.6 mmol) of 2,2,6,6-tetramethylpiperidin-4-ol was dissolved in 3.2 ml of trichloroacetate acid solution (1.6 mmol, 0.26 g). Colorless crystals of the title compound were obtained by slow evaporation of the solvent.

S3. Refinement

All H atoms bound to C atoms were constrained; positioned geometrically (C—H = 0.96–0.98 Å) and refined as riding with U_{iso}(H)=1.2U_{eq}(carrier) or 1.5$_{eq}$(methyl groups). H atoms of O—H and N—H were located from difference maps and then refined freely.

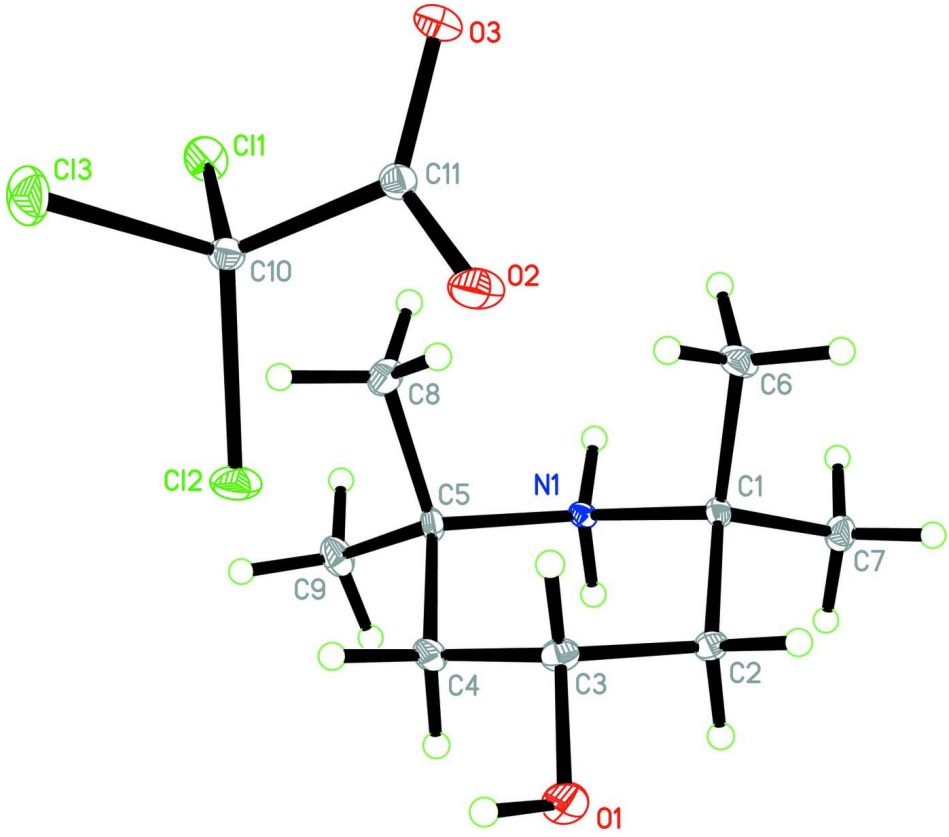


Figure 1

Crystal structure of the title compound with atom labeling and displacement ellipsoids drawn at the 30% probability level. H atoms are shown as small spheres of arbitrary radii.

4-Hydroxy-2,2,6,6-tetramethylpiperidinium trichloroacetate

Crystal data

$C_9H_{20}NO^+\cdot C_2Cl_3O_2^-$
$M_r = 320.63$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.3468\ (13)$ Å
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$c = 8.2175\ (16)$ Å
$\beta = 95.19\ (3)°$
$V = 750.5\ (3)$ Å^3
$Z = 2$
$F(000) = 336$
$D_x = 1.419$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2559 reflections
$\theta = 1.4–27.9°$
$\mu = 0.61$ mm^{-1}
$T = 113$ K
Block, colorless
0.12 × 0.10 × 0.08 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Confocal monochromator
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
$T_{min} = 0.930$, $T_{max} = 0.953$
5459 measured reflections
2858 independent reflections
2636 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.5°$
$h = -8\rightarrow8$
$k = -15\rightarrow19$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.060$
$S = 1.06$
2858 reflections
179 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}
Absolute structure: Flack (1983), 996 Friedel pairs
Absolute structure parameter: 0.04 (4)

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}
Cl1	−0.04464 (7)	0.51471 (3)	0.10942 (5)	0.01980 (10)
Cl2	0.34863 (6)	0.41431 (3)	0.12885 (6)	0.02203 (11)
Cl3	−0.00077 (8)	0.35637 (3)	−0.09864 (5)	0.02572 (11)
O1	0.7973 (2)	0.47566 (9)	0.51619 (17)	0.0227 (3)
H1	0.798 (4)	0.453 (2)	0.415 (4)	0.063 (10)*
O2	0.1032 (2)	0.27147 (9)	0.27469 (18)	0.0247 (3)
O3	−0.20242 (18)	0.34925 (9)	0.25644 (15)	0.0169 (3)
N1	0.4803 (2)	0.71305 (9)	0.64562 (18)	0.0111 (3)
C1	0.4608 (3)	0.62855 (12)	0.75507 (19)	0.0129 (3)
C2	0.6286 (3)	0.55858 (11)	0.7135 (2)	0.0139 (3)
H2A	0.7701	0.5832	0.7515	0.017*
H2B	0.6080	0.5005	0.7739	0.017*
C3	0.6235 (3)	0.53661 (11)	0.5324 (2)	0.0159 (4)
H3	0.4876	0.5051	0.4945	0.019*
C4	0.6475 (3)	0.62493 (13)	0.4346 (2)	0.0164 (3)
H4A	0.6371	0.6090	0.3170	0.020*
H4B	0.7905	0.6506	0.4638	0.020*
C5	0.4836 (3)	0.69993 (12)	0.46158 (19)	0.0135 (3)
C6	0.2366 (3)	0.58770 (13)	0.7353 (2)	0.0198 (4)
H6A	0.1327	0.6377	0.7379	0.030*
H6B	0.2191	0.5443	0.8248	0.030*
H6C	0.2149	0.5550	0.6306	0.030*
C7	0.5076 (3)	0.66393 (13)	0.9293 (2)	0.0189 (4)

H7A	0.6495	0.6912	0.9419	0.028*
H7B	0.5003	0.6124	1.0061	0.028*
H7C	0.4029	0.7110	0.9519	0.028*
C8	0.2629 (3)	0.67658 (13)	0.3825 (2)	0.0198 (4)
H8A	0.1595	0.7193	0.4228	0.030*
H8B	0.2266	0.6130	0.4104	0.030*
H8C	0.2609	0.6825	0.2636	0.030*
C9	0.5521 (3)	0.79290 (13)	0.3950 (2)	0.0213 (4)
H9A	0.4450	0.8399	0.4123	0.032*
H9B	0.5676	0.7871	0.2779	0.032*
H9C	0.6878	0.8113	0.4523	0.032*
C10	0.0700 (3)	0.40324 (12)	0.09850 (19)	0.0130 (3)
C11	−0.0172 (3)	0.33454 (11)	0.2249 (2)	0.0133 (3)
H1A	0.373 (4)	0.7546 (18)	0.674 (3)	0.037 (7)*
H1B	0.610 (4)	0.7410 (15)	0.683 (3)	0.025 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0227 (2)	0.01408 (19)	0.0232 (2)	0.00440 (16)	0.00505 (17)	0.00388 (16)
Cl2	0.01100 (19)	0.0235 (2)	0.0318 (3)	−0.00221 (15)	0.00291 (17)	0.00920 (19)
Cl3	0.0321 (3)	0.0304 (3)	0.0149 (2)	−0.0025 (2)	0.00289 (18)	−0.00596 (17)
O1	0.0249 (7)	0.0194 (7)	0.0241 (7)	0.0108 (5)	0.0033 (6)	−0.0052 (6)
O2	0.0147 (6)	0.0220 (7)	0.0377 (8)	0.0023 (5)	0.0044 (6)	0.0151 (6)
O3	0.0125 (6)	0.0160 (6)	0.0230 (7)	−0.0007 (5)	0.0056 (5)	−0.0007 (5)
N1	0.0114 (7)	0.0098 (7)	0.0124 (7)	0.0007 (5)	0.0033 (6)	−0.0002 (5)
C1	0.0127 (8)	0.0130 (8)	0.0133 (8)	0.0010 (6)	0.0032 (6)	0.0029 (6)
C2	0.0137 (8)	0.0124 (8)	0.0156 (9)	0.0027 (6)	0.0014 (7)	0.0001 (6)
C3	0.0152 (8)	0.0131 (8)	0.0193 (9)	0.0035 (6)	0.0010 (7)	−0.0028 (6)
C4	0.0164 (9)	0.0187 (8)	0.0149 (8)	0.0020 (6)	0.0049 (7)	−0.0023 (7)
C5	0.0167 (8)	0.0153 (8)	0.0089 (7)	0.0010 (6)	0.0031 (6)	0.0010 (6)
C6	0.0152 (9)	0.0174 (9)	0.0276 (10)	−0.0007 (7)	0.0059 (8)	0.0059 (7)
C7	0.0228 (9)	0.0215 (10)	0.0127 (8)	0.0047 (7)	0.0028 (7)	0.0000 (7)
C8	0.0196 (9)	0.0215 (9)	0.0176 (9)	−0.0002 (7)	−0.0023 (7)	0.0013 (7)
C9	0.0272 (11)	0.0164 (9)	0.0214 (10)	−0.0001 (7)	0.0090 (8)	0.0043 (7)
C10	0.0118 (8)	0.0134 (8)	0.0141 (8)	0.0009 (6)	0.0021 (6)	0.0010 (6)
C11	0.0124 (8)	0.0145 (8)	0.0130 (8)	−0.0019 (6)	0.0010 (6)	−0.0006 (6)

Geometric parameters (Å, º)

Cl1—C10	1.7729 (17)	C4—C5	1.532 (2)
Cl2—C10	1.7710 (17)	C4—H4A	0.9900
Cl3—C10	1.7756 (17)	C4—H4B	0.9900
O1—C3	1.427 (2)	C5—C8	1.528 (2)
O1—H1	0.89 (3)	C5—C9	1.529 (3)
O2—C11	1.235 (2)	C6—H6A	0.9800
O3—C11	1.245 (2)	C6—H6B	0.9800
N1—C5	1.526 (2)	C6—H6C	0.9800

N1—C1	1.528 (2)	C7—H7A	0.9800
N1—H1A	0.95 (3)	C7—H7B	0.9800
N1—H1B	0.94 (2)	C7—H7C	0.9800
C1—C7	1.524 (2)	C8—H8A	0.9800
C1—C2	1.529 (2)	C8—H8B	0.9800
C1—C6	1.535 (2)	C8—H8C	0.9800
C2—C3	1.519 (2)	C9—H9A	0.9800
C2—H2A	0.9900	C9—H9B	0.9800
C2—H2B	0.9900	C9—H9C	0.9800
C3—C4	1.523 (2)	C10—C11	1.573 (2)
C3—H3	1.0000		

C3—O1—H1	112.3 (19)	C8—C5—C4	113.00 (15)
C5—N1—C1	119.53 (13)	C9—C5—C4	110.55 (15)
C5—N1—H1A	113.1 (15)	C1—C6—H6A	109.5
C1—N1—H1A	105.4 (15)	C1—C6—H6B	109.5
C5—N1—H1B	106.5 (14)	H6A—C6—H6B	109.5
C1—N1—H1B	105.4 (13)	C1—C6—H6C	109.5
H1A—N1—H1B	106 (2)	H6A—C6—H6C	109.5
C7—C1—N1	105.40 (13)	H6B—C6—H6C	109.5
C7—C1—C2	110.56 (14)	C1—C7—H7A	109.5
N1—C1—C2	107.56 (13)	C1—C7—H7B	109.5
C7—C1—C6	109.16 (14)	H7A—C7—H7B	109.5
N1—C1—C6	111.62 (14)	C1—C7—H7C	109.5
C2—C1—C6	112.31 (14)	H7A—C7—H7C	109.5
C3—C2—C1	113.80 (14)	H7B—C7—H7C	109.5
C3—C2—H2A	108.8	C5—C8—H8A	109.5
C1—C2—H2A	108.8	C5—C8—H8B	109.5
C3—C2—H2B	108.8	H8A—C8—H8B	109.5
C1—C2—H2B	108.8	C5—C8—H8C	109.5
H2A—C2—H2B	107.7	H8A—C8—H8C	109.5
O1—C3—C2	105.80 (14)	H8B—C8—H8C	109.5
O1—C3—C4	110.67 (14)	C5—C9—H9A	109.5
C2—C3—C4	110.33 (14)	C5—C9—H9B	109.5
O1—C3—H3	110.0	H9A—C9—H9B	109.5
C2—C3—H3	110.0	C5—C9—H9C	109.5
C4—C3—H3	110.0	H9A—C9—H9C	109.5
C3—C4—C5	114.51 (14)	H9B—C9—H9C	109.5
C3—C4—H4A	108.6	C11—C10—Cl2	111.74 (11)
C5—C4—H4A	108.6	C11—C10—Cl1	111.69 (11)
C3—C4—H4B	108.6	Cl2—C10—Cl1	108.65 (9)
C5—C4—H4B	108.6	C11—C10—Cl3	106.67 (11)
H4A—C4—H4B	107.6	Cl2—C10—Cl3	109.25 (9)
N1—C5—C8	110.77 (14)	Cl1—C10—Cl3	108.77 (9)
N1—C5—C9	105.97 (14)	O2—C11—O3	128.65 (16)
C8—C5—C9	108.77 (14)	O2—C11—C10	116.15 (14)
N1—C5—C4	107.55 (13)	O3—C11—C10	115.14 (14)

C5—N1—C1—C7	168.77 (14)	C1—N1—C5—C9	−167.95 (14)
C5—N1—C1—C2	50.78 (19)	C1—N1—C5—C4	−49.69 (19)
C5—N1—C1—C6	−72.85 (19)	C3—C4—C5—N1	50.25 (19)
C7—C1—C2—C3	−166.72 (14)	C3—C4—C5—C8	−72.32 (19)
N1—C1—C2—C3	−52.12 (18)	C3—C4—C5—C9	165.51 (16)
C6—C1—C2—C3	71.08 (18)	Cl2—C10—C11—O2	−29.86 (19)
C1—C2—C3—O1	176.40 (14)	Cl1—C10—C11—O2	−151.81 (14)
C1—C2—C3—C4	56.67 (19)	Cl3—C10—C11—O2	89.47 (17)
O1—C3—C4—C5	−172.59 (14)	Cl2—C10—C11—O3	152.80 (13)
C2—C3—C4—C5	−55.84 (19)	Cl1—C10—C11—O3	30.85 (18)
C1—N1—C5—C8	74.25 (18)	Cl3—C10—C11—O3	−87.87 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O3^{i}	0.89 (3)	1.99 (3)	2.8095 (18)	152 (3)
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N1—H1*B*···O2^{iii}	0.94 (2)	1.87 (2)	2.796 (2)	165.1 (19)
N1—H1*B*···Cl2^{iii}	0.94 (2)	2.94 (2)	3.5647 (16)	124.5 (16)

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