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2,3,5,6-Tetramethylpyrazinium–trichloroacetate–trichloroacetic acid (1/1/1)

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.006 Å
R factor = 0.063
wR factor = 0.185
Data-to-parameter ratio = 15.4

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

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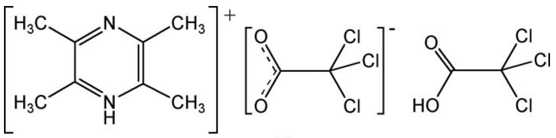
In the title adduct, $C_8H_{13}N_2^+\cdot C_2Cl_3O_2^-\cdot C_2HCl_3O_2$, the tetramethylpyrazine molecule is protonated at one of the N atoms and linked to the trichloroacetate anion *via* an N—H···O hydrogen bond. The trichloroacetate anion is also linked to the trichloroacetic acid molecule *via* an O—H···O hydrogen bond.

Comment

Tetramethylpyrazine is mainly used for the clinical treatment of nephrosis and ischemic cerebrovascular disease, due to its function of anticoagulation and angiectasis. It also has a protective effect against ischemic neuronal damage in the hippocampus (Luo, 1994). In the past, some supramolecular compounds have been synthesized with tetramethylpyrazine (Bailey *et al.*, 1992; Adams *et al.*, 1993; Tian & Yang, 1993; Abourahma *et al.*, 1995; Smyth *et al.*, 1996; Dong *et al.*, 2003). We report here the structure of the title complex, (I).



(I)

Fig. 1 shows the molecular structure of (I) with the atom numbering. In the tetramethylpyrazinium cation (TPM), atom N2 is protonated, atom N1 remains neutral and the C7—N2—C3 bond angle [124.2 (3)°] is larger than C2—N1—C6 [120.3 (3)°]. The left and right halves of TPM are slightly different with regard to their bond lengths and angles; they are related by pseudo-C_2 symmetry. TPM is linked to a trichloroacetate anion *via* an N2—H2···O1 hydrogen bond and is further linked to a trichloroacetic acid molecule *via* an O4—H4···O2 hydrogen bond (Table 2). The bond lengths of O1—C10 [1.217 (5) Å] and O2—C10 [1.210 (5) Å] are consistent with a delocalized carboxylate group, while the bond lengths of O3—C12 [1.178 (5) Å] and O4—C12 [1.264 (6) Å] are consistent with a carboxylic acid group.

Experimental

A aqueous solution (10 ml) of trichloroacetic acid (2 mmol, 0.33 g) was added slowly to an ethanol solution (10 ml) of 2,3,5,6-tetramethylpyrazine (1 mmol, 0.14 g). The mixture was stirred for several minutes and left to stand at room temperature for about two weeks, after which time colorless prismatic crystals were obtained.

Crystal data

$C_8H_{13}N_2^+ \cdot C_2Cl_3O_2^- \cdot C_2HCl_3O_2$
$M_r = 462.95$
Triclinic, $P\overline{1}$
$a = 9.3627\ (12)$ Å
$b = 9.6100\ (13)$ Å
$c = 11.7698\ (15)$ Å
$\alpha = 87.956\ (3)^\circ$
$\beta = 72.496\ (2)^\circ$
$\gamma = 79.561\ (2)^\circ$
$V = 993.0\ (2)$ Å^3
$Z = 2$
$D_x = 1.548$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2421 reflections
$\theta = 2.2$–25.1°
$\mu = 0.88$ mm^{-1}
$T = 298\ (2)$ K
Prism, colorless
$0.47 \times 0.43 \times 0.39$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.667$, $T_{\max} = 0.709$
5308 measured reflections
3520 independent reflections
2898 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.010$
$\theta_{\max} = 25.2^\circ$
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 10$
$l = -8 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.185$
$S = 1.07$
3520 reflections
229 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0967P)^2 + 0.7704P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.76$ e Å^{-3}
$\Delta\rho_{\min} = -0.42$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O3—C12	1.178 (5)	N1—C2	1.333 (5)
O4—C12	1.264 (6)	N1—C6	1.334 (5)
Cl4—C11	1.747 (4)	N2—C7	1.338 (5)
Cl5—C11	1.735 (4)	N2—C3	1.340 (5)
Cl6—C11	1.743 (4)	C1—C2	1.508 (5)
C11—C12	1.535 (5)	C2—C3	1.384 (5)
Cl1—C9	1.757 (3)	C3—C4	1.484 (6)
Cl2—C9	1.755 (3)	C5—C6	1.484 (6)
Cl3—C9	1.762 (4)	C6—C7	1.387 (5)
O1—C10	1.217 (5)	C7—C8	1.498 (5)
O2—C10	1.210 (5)	C9—C10	1.551 (5)
O3—C12—O4	126.9 (4)	N1—C6—C7	120.6 (3)
O3—C12—C11	120.5 (4)	N1—C6—C5	117.8 (4)
C2—N1—C6	120.3 (3)	N2—C7—C6	117.1 (3)
C7—N2—C3	124.2 (3)	N2—C7—C8	118.4 (4)
N1—C2—C3	121.3 (3)	O2—C10—O1	128.7 (4)
N1—C2—C1	117.0 (4)	O2—C10—C9	114.9 (4)
N2—C3—C2	116.5 (3)	O1—C10—C9	116.3 (3)
N2—C3—C4	118.0 (4)		

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

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
N2—H2$\cdots$O1	0.90 (5)	1.80 (5)	2.692 (4)	175 (5)
O4—H4$\cdots$O2	0.85 (8)	1.69 (8)	2.517 (5)	165 (8)

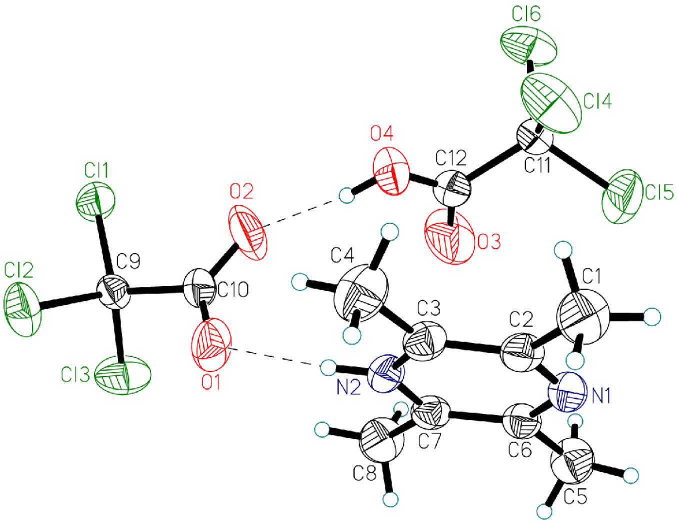


Figure 1
A view of (I), showing 40% probability displacement ellipsoids. Hydrogen bonds are indicated by dashed lines.

H atoms attached to N and O atoms were located in a difference Fourier map and refined isotropically. Other H atoms were placed in calculated positions (C—H = 0.96 Å) and allowed to ride on their parent atoms, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL*97.

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