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2,4,6-Trimethylanilinium 2-carboxyethanoate

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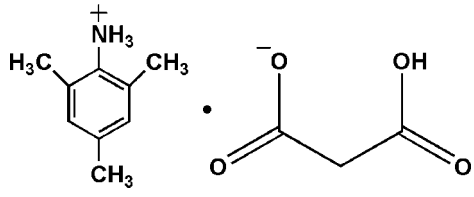
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.065; wR factor = 0.166; data-to-parameter ratio = 17.6.

The anion of the title molecular salt, $C_9H_{14}N^+\cdot C_3H_3O_4^-$, features an intramolecular O—H···O hydrogen bond. In the crystal, intermolecular N—H···O interactions link each cation to three different anions.

Related literature

For general background to ferroelectric organic frameworks, see: Ye *et al.* (2006, 2009); Fu *et al.* (2007). For phase transition of ferroelectric materials, see: Zhang *et al.* (2008); Zhao *et al.* (2008).



Experimental

Crystal data

$C_9H_{14}N^+\cdot C_3H_3O_4^-$
M_r = 239.27
Orthorhombic, *Pbcn*
a = 13.732 (3) Å
b = 7.8522 (16) Å
c = 23.124 (5) Å
V = 2493.3 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.981, T_{max} = 0.981
23089 measured reflections
2856 independent reflections
1643 reflections with $I > 2\sigma(I)$
R_{int} = 0.096

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.166
S = 1.05
2856 reflections
162 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O2	0.99 (4)	1.46 (4)	2.421 (3)	160 (3)
N1—H1A···O4^{i}	0.89	2.02	2.879 (3)	162
N1—H1A···O3^{i}	0.89	2.51	3.248 (3)	140
N1—H1B···O1^{ii}	0.89	2.09	2.826 (3)	140
N1—H1B···O4^{iii}	0.89	2.58	3.075 (3)	116
N1—H1C···O2^{iv}	0.89	1.92	2.798 (3)	167

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iii) $x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (iv) $-x+\frac{1}{2}, -y+\frac{1}{2}, z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 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: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5560).

References

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Fu, D.-W., Song, Y.-M., Wang, G.-X., Ye, Q., Xiong, R.-G., Akutagawa, T., Nakamura, T., Chan, P. W. H. & Huang, S. D. (2007). *J. Am. Chem. Soc.* **129**, 5346–5347.
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S1. Comment

In the crystal structure, one hydrogen –bonding network of N-H···O hydrogen bonds which established between ammonium groups and hydrogen malonateions, and one kind of intramolecular hydrogen bond O3—H···O1 which established between O3 and O1 contribute to the stability of crystal packing.

In the structure, atom N1 is hydrogen bonded to O atoms of hydrogen malonate ions through hydrogen bonds.

The study of ferroelectric materials has received much attention. Some materials have predominantly dielectric-ferroelectric properties. The title compound was studied as part of our work to obtain potential ferroelectric phase-transition materials (Ye *et al.*, 2006; Fu *et al.*, 2007; Zhao *et al.* 2008; Zhang *et al.*, 2008; Ye *et al.*, 2009). Unluckily, the compound has no dielectric anomalies in the temperature range 93–453 K, suggesting that it might be only a paraelectric.

S2. Experimental

For the preparation of the title compound, the Malonate(0.5 g) was added to the ehanol solution of the 2,4,6-trimethyl-aniline, The resulting precipitate was filtered. Colorless crystals suitable for X-ray analysis were formed after several weeks by solw evaporation of the solvent at room temperature.

S3. Refinement

Positional parameters of all the H atoms bonded to C and N atoms were calculated geometrically and were allowed to ride on their parent atoms with U_{iso}(H) = 1.2Ueq(C) or U_{iso}(H) = 1.5Ueq(C,N). The H atom bond to O3 was freely refined.

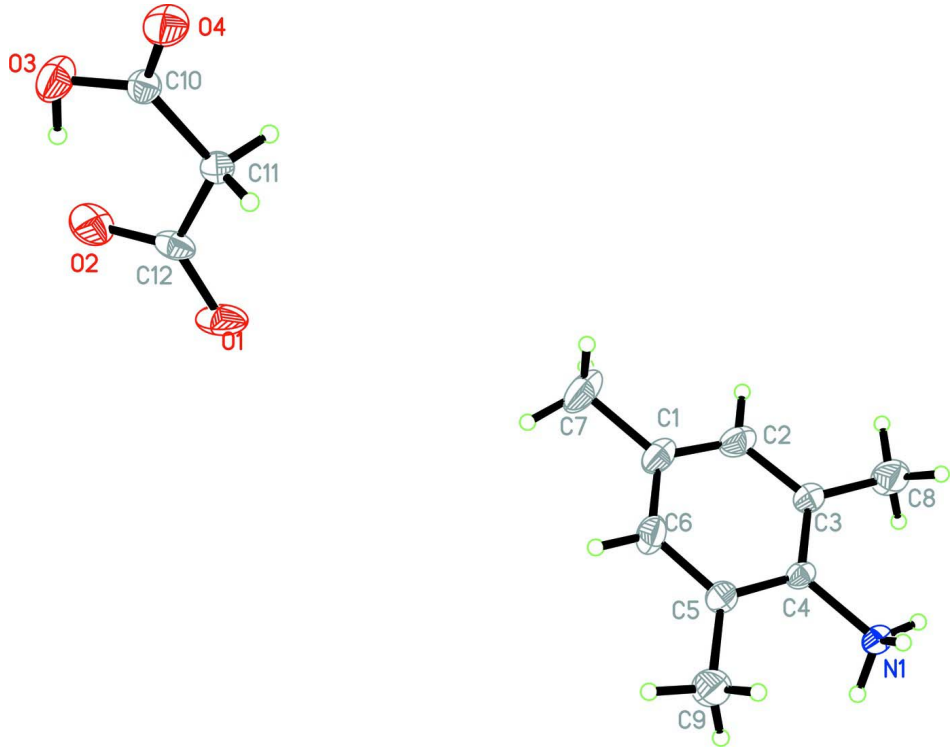

Figure 1

The molecular structure of the title compound, with the atomic numbering scheme.Displacement ellipsoids are drawn at the 30%

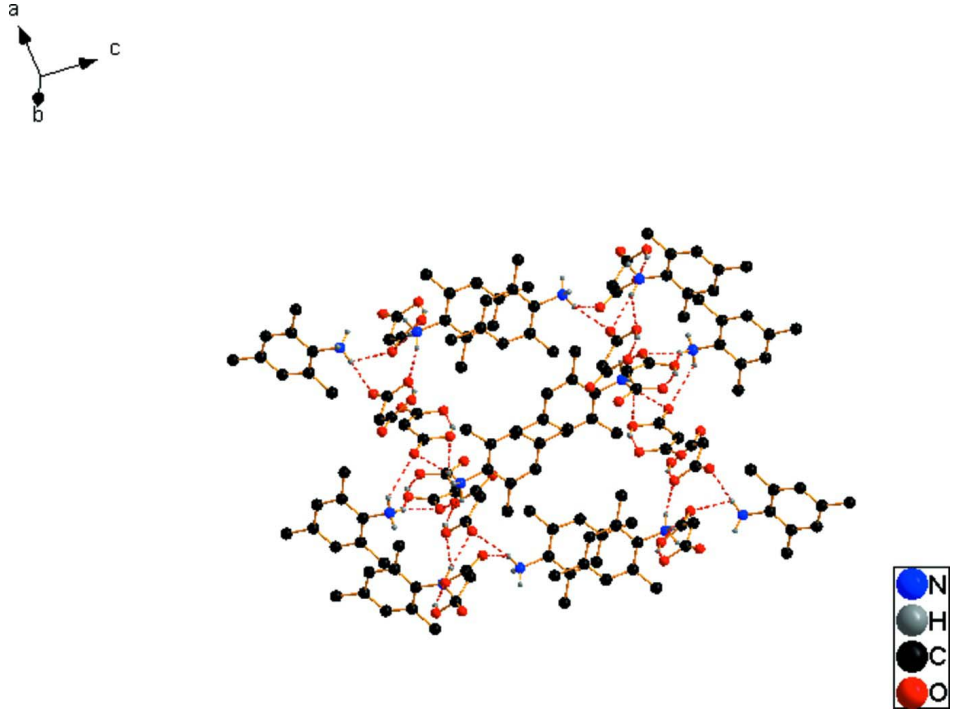

Figure 2

A view of the packing of the title compound, stacking along the *b* axis. Dashed lines indicate hydrogen bonds.

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Crystal data

$C_9H_{14}N^+·C_3H_3O_4^-$
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Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
a = 13.732 (3) Å
b = 7.8522 (16) Å
c = 23.124 (5) Å
V = 2493.3 (9) Å^3
Z = 8
F(000) = 1024
D_x = 1.275 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 2856 reflections
θ = 3.0–27.5°
μ = 0.10 mm^{-1}
T = 293 K
Prism, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.981, T_{max} = 0.981
23089 measured reflections
2856 independent reflections
1643 reflections with $I > 2\sigma(I)$
R_{int} = 0.096
θ_{max} = 27.5°, θ_{min} = 3.0°
h = −17→17
k = −10→10
l = −30→30

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.065
$wR(F^2)$ = 0.166
S = 1.05
2856 reflections
162 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0663P)^2 + 0.8663P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.006
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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}
C1	0.4717 (2)	0.2519 (3)	0.49208 (11)	0.0488 (7)

C2	0.5594 (2)	0.2379 (3)	0.52056 (11)	0.0504 (7)
H2	0.6161	0.2628	0.5003	0.060*
C3	0.56763 (18)	0.1886 (3)	0.57795 (10)	0.0390 (6)
C4	0.48162 (17)	0.1522 (3)	0.60667 (9)	0.0309 (5)
C5	0.39102 (18)	0.1613 (3)	0.58021 (10)	0.0385 (6)
C6	0.3885 (2)	0.2122 (4)	0.52297 (11)	0.0507 (7)
H6	0.3285	0.2200	0.5045	0.061*
C7	0.4666 (3)	0.3072 (4)	0.42944 (12)	0.0738 (10)
H7A	0.4639	0.4292	0.4275	0.111*
H7B	0.4093	0.2601	0.4118	0.111*
H7C	0.5233	0.2673	0.4093	0.111*
C8	0.6659 (2)	0.1771 (5)	0.60677 (13)	0.0646 (9)
H8A	0.7156	0.2095	0.5798	0.097*
H8B	0.6770	0.0623	0.6194	0.097*
H8C	0.6675	0.2523	0.6395	0.097*
C9	0.2980 (2)	0.1207 (4)	0.61237 (13)	0.0593 (8)
H9A	0.3004	0.0055	0.6262	0.089*
H9B	0.2434	0.1337	0.5868	0.089*
H9C	0.2910	0.1971	0.6445	0.089*
C10	0.11880 (17)	1.0449 (3)	0.23443 (10)	0.0344 (6)
C11	0.15426 (18)	0.9020 (3)	0.27175 (10)	0.0364 (6)
H11A	0.1284	0.9190	0.3104	0.044*
H11B	0.2246	0.9107	0.2744	0.044*
C12	0.12942 (17)	0.7225 (3)	0.25311 (13)	0.0401 (6)
H3	0.092 (3)	0.881 (5)	0.1813 (14)	0.093 (12)*
N1	0.48497 (14)	0.1025 (2)	0.66800 (8)	0.0331 (5)
H1A	0.4588	0.1847	0.6895	0.050*
H1B	0.5466	0.0862	0.6786	0.050*
H1C	0.4515	0.0065	0.6730	0.050*
O1	0.13547 (14)	0.6067 (2)	0.28835 (10)	0.0624 (6)
O2	0.10416 (16)	0.7039 (2)	0.19982 (9)	0.0600 (6)
O3	0.08869 (16)	1.0069 (3)	0.18287 (8)	0.0581 (6)
O4	0.11846 (14)	1.1927 (2)	0.25102 (8)	0.0512 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.070 (2)	0.0448 (16)	0.0310 (15)	0.0047 (15)	0.0011 (13)	0.0036 (12)
C2	0.0576 (18)	0.0562 (18)	0.0374 (16)	−0.0033 (15)	0.0122 (13)	0.0065 (13)
C3	0.0417 (14)	0.0415 (15)	0.0338 (14)	−0.0005 (12)	0.0037 (11)	0.0027 (11)
C4	0.0416 (14)	0.0245 (12)	0.0267 (12)	0.0015 (10)	0.0033 (10)	0.0006 (9)
C5	0.0404 (15)	0.0372 (14)	0.0379 (15)	0.0017 (12)	−0.0008 (11)	0.0031 (11)
C6	0.0525 (17)	0.0608 (18)	0.0387 (16)	0.0050 (14)	−0.0126 (13)	0.0068 (13)
C7	0.112 (3)	0.078 (2)	0.0314 (16)	0.006 (2)	−0.0023 (16)	0.0119 (16)
C8	0.0426 (16)	0.096 (3)	0.0556 (19)	−0.0059 (17)	0.0061 (14)	0.0155 (17)
C9	0.0401 (16)	0.079 (2)	0.0588 (19)	−0.0012 (16)	0.0002 (13)	0.0134 (16)
C10	0.0336 (13)	0.0334 (14)	0.0363 (14)	0.0009 (11)	0.0002 (11)	0.0007 (11)
C11	0.0395 (13)	0.0313 (13)	0.0386 (14)	−0.0022 (11)	−0.0035 (11)	0.0027 (11)

C12	0.0257 (12)	0.0301 (13)	0.0644 (19)	−0.0020 (11)	0.0089 (12)	0.0039 (13)
N1	0.0402 (11)	0.0301 (10)	0.0288 (11)	0.0024 (9)	0.0028 (8)	0.0020 (9)
O1	0.0539 (13)	0.0328 (11)	0.1006 (17)	0.0015 (9)	0.0110 (11)	0.0256 (11)
O2	0.0772 (15)	0.0394 (11)	0.0633 (14)	−0.0112 (10)	−0.0046 (11)	−0.0167 (10)
O3	0.0844 (15)	0.0489 (13)	0.0410 (12)	0.0060 (11)	−0.0167 (10)	0.0007 (9)
O4	0.0643 (13)	0.0296 (10)	0.0596 (12)	0.0075 (9)	−0.0051 (9)	−0.0011 (9)

Geometric parameters (Å, º)

C1—C2	1.376 (4)	C8—H8C	0.9600
C1—C6	1.384 (4)	C9—H9A	0.9600
C1—C7	1.514 (4)	C9—H9B	0.9600
C2—C3	1.387 (3)	C9—H9C	0.9600
C2—H2	0.9300	C10—O4	1.222 (3)
C3—C4	1.385 (3)	C10—O3	1.297 (3)
C3—C8	1.507 (4)	C10—C11	1.497 (3)
C4—C5	1.388 (3)	C11—C12	1.513 (3)
C4—N1	1.472 (3)	C11—H11A	0.9700
C5—C6	1.383 (3)	C11—H11B	0.9700
C5—C9	1.512 (4)	C12—O1	1.224 (3)
C6—H6	0.9300	C12—O2	1.289 (3)
C7—H7A	0.9600	N1—H1A	0.8900
C7—H7B	0.9600	N1—H1B	0.8900
C7—H7C	0.9600	N1—H1C	0.8900
C8—H8A	0.9600	O3—H3	0.99 (4)
C8—H8B	0.9600		
C2—C1—C6	117.2 (2)	H8A—C8—H8C	109.5
C2—C1—C7	121.4 (3)	H8B—C8—H8C	109.5
C6—C1—C7	121.3 (3)	C5—C9—H9A	109.5
C1—C2—C3	123.5 (3)	C5—C9—H9B	109.5
C1—C2—H2	118.3	H9A—C9—H9B	109.5
C3—C2—H2	118.3	C5—C9—H9C	109.5
C4—C3—C2	116.5 (2)	H9A—C9—H9C	109.5
C4—C3—C8	122.6 (2)	H9B—C9—H9C	109.5
C2—C3—C8	120.9 (2)	O4—C10—O3	120.4 (2)
C3—C4—C5	122.8 (2)	O4—C10—C11	122.1 (2)
C3—C4—N1	119.4 (2)	O3—C10—C11	117.5 (2)
C5—C4—N1	117.8 (2)	C10—C11—C12	117.4 (2)
C6—C5—C4	117.3 (2)	C10—C11—H11A	107.9
C6—C5—C9	120.7 (2)	C12—C11—H11A	107.9
C4—C5—C9	122.0 (2)	C10—C11—H11B	107.9
C5—C6—C1	122.6 (2)	C12—C11—H11B	107.9
C5—C6—H6	118.7	H11A—C11—H11B	107.2
C1—C6—H6	118.7	O1—C12—O2	124.8 (3)
C1—C7—H7A	109.5	O1—C12—C11	119.2 (3)
C1—C7—H7B	109.5	O2—C12—C11	116.0 (2)
H7A—C7—H7B	109.5	C4—N1—H1A	109.5

C1—C7—H7C	109.5	C4—N1—H1B	109.5
H7A—C7—H7C	109.5	H1A—N1—H1B	109.5
H7B—C7—H7C	109.5	C4—N1—H1C	109.5
C3—C8—H8A	109.5	H1A—N1—H1C	109.5
C3—C8—H8B	109.5	H1B—N1—H1C	109.5
H8A—C8—H8B	109.5	C10—O3—H3	105 (2)
C3—C8—H8C	109.5		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
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