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4-Methylanilinium 2-carboxyacetate

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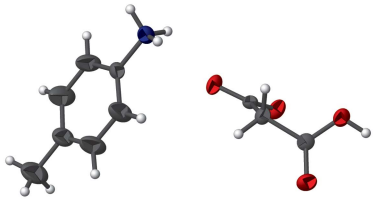
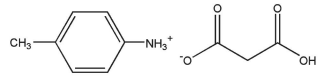
Structural data: full structural data are available from iucrdata.iucr.org

In the crystal of the title salt, $C_7H_{10}N^+\cdot C_3H_3O_4^-$, the cations are linked to the anions via N—H···O and trifurcated N—H···(O,O,O) hydrogen bonds. The anions are linked into [010] chains by O—H···O hydrogen bonds. Taken together, these interactions generate (100) sheets.

3D view



Chemical scheme



Structure description

Organic salts containing strong intermolecular hydrogen bonds have attracted attention as materials that may display ferroelectric–paraelectric phase transitions (Huang *et al.* 1999; Zhang *et al.* 2001). We report herein the synthesis and the crystal structure of the title salt (Fig. 1). Its geometric parameters are comparable with those of reported similar structures (Benali-Cherif *et al.*, 2007, 2009; Kalaiyarasi *et al.*, 2017; Suresh *et al.*, 2017; Wang, 2012).

The asymmetric unit of the title salt (Fig. 1) contains a 4-methylanilinium cation and a carboxyacetate anion linked by an N1—H1*C*···O1 hydrogen bond (Table 1). In the crystal, the cations are linked to the anions via N—H···O and trifurcated N—H···(O,O,O) hydrogen bonds (Fig. 2, Table 1). The anions are linked into [010] chains by O—H···O hydrogen bonds. Taken together, these interactions generate (100) sheets.

Synthesis and crystallization

p-Toluidine (1.33 g) and malonic acid (1.30 g) were taken in a 1:1 ratio and dissolved in water at room temperature. The aqueous solution was stirred continuously for 6 h to obtain a transparent solution which was filtered and kept for slow evaporation. Crystals suitable for X-ray diffraction analysis were obtained after a period of one month.

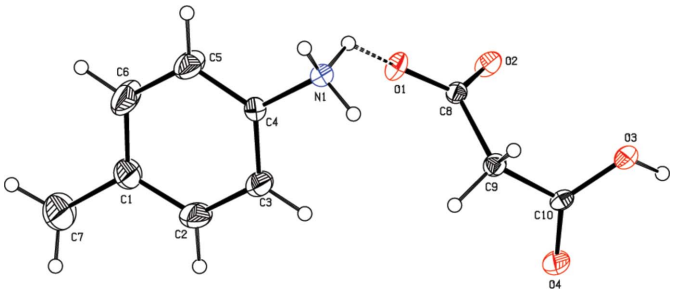


Figure 1
The molecular structure of the title molecular salt, with the atom labelling and 30% probability displacement ellipsoids.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

The authors acknowledge the SAIF, IIT, Madras, for the data collection.

References

Benali-Cherif, N., Boussekine, H., Boutobba, Z. & Dadda, N. (2009). *Acta Cryst.* E**65**, o2744.
Benali-Cherif, N., Kateb, A., Boussekine, H., Boutobba, Z. & Messai, A. (2007). *Acta Cryst.* E**63**, o3251.
Bruker (2004). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Huang, S. D., Xiong, R.-G., Han, J.-D. & Weiner, B. R. (1999). *Inorg. Chim. Acta*, **294**, 95–98.

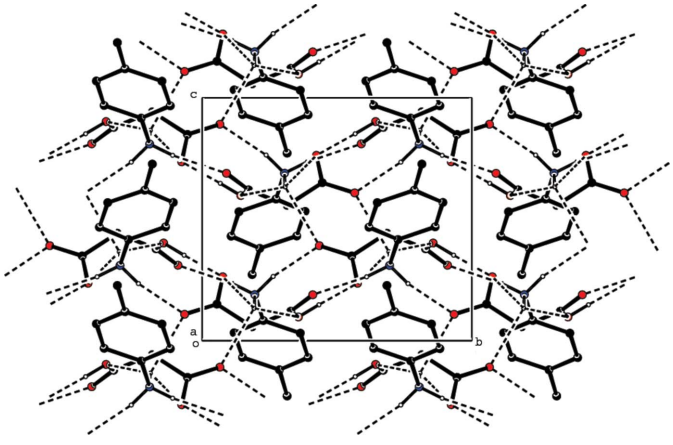


Figure 2
The crystal packing of the title molecular salt viewed along the *a* axis. The hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4^{i}	0.89	1.96	2.846 (2)	172
N1—H1B···O1^{ii}	0.89	1.91	2.789 (2)	169
N1—H1C···O1	0.89	2.40	3.041 (2)	130
N1—H1C···O2^{iii}	0.89	2.47	2.980 (2)	117
N1—H1C···O3^{iii}	0.89	2.21	2.871 (2)	131
O3—H3A···O2^{iv}	0.87 (1)	1.67 (1)	2.5350 (19)	175 (2)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_7H_{10}N^+\cdot C_3H_3O_4^-$
M_r	211.21
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	295
a, b, c (Å)	12.996 (3), 9.2813 (19), 8.665 (2)
β (°)	105.503 (7)
V (Å^3)	1007.1 (4)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.24 × 0.22 × 0.18
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.678, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7352, 1766, 1282
R_{int}	0.031
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.595
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.112, 1.05
No. of reflections	1766
No. of parameters	141
No. of restraints	1
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.19

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SHELXT2016* (Sheldrick, 2015*a*), *SHELXL2016* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2009).

Kalaiyarasi, S., Suresh, S., Akilan, R., Kumar, R. M. & Chakkaravarthi, G. (2017). *IUCrData*, **2**, x170254.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Suresh, S., Pandi, P., Kumar, R. M. & Chakkaravarthi, G. (2017). *IUCrData*, **2**, x171767.
Wang, Y.-C. (2012). *Acta Cryst.* E**68**, o1984.
Zhang, J., Xiong, R.-G., Chen, X.-T., Che, C.-M., Xue, Z.-L. & You, X.-Z. (2001). *Organometallics*, **20**, 4118–4121.

full crystallographic data

IUCrData (2018). **3**, x181714 [https://doi.org/10.1107/S2414314618017145]

4-Methylanilinium 2-carboxyacetate

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4-Methylanilinium 2-carboxyacetate

Crystal data

$C_7H_{10}N^+·C_3H_3O_4^-$
M_r = 211.21
Monoclinic, $P2_1/c$
a = 12.996 (3) Å
b = 9.2813 (19) Å
c = 8.665 (2) Å
β = 105.503 (7)°
V = 1007.1 (4) Å^3
Z = 4
$F(000)$ = 448
D_x = 1.393 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2586 reflections
θ = 2.7–27.1°
μ = 0.11 mm^{-1}
T = 295 K
Block, colourless
0.24 × 0.22 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.678, T_{max} = 0.746
7352 measured reflections
1766 independent reflections
1282 reflections with $I > 2\sigma(I)$
R_{int} = 0.031
θ_{max} = 25.0°, θ_{min} = 2.7°
h = −15→15
k = −11→10
l = −10→10

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.112
S = 1.05
1766 reflections
141 parameters
1 restraint
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.3929P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All H atoms were located in a difference map. The coordinates of the H atom bonded to O were refined with U(H) set to 1.5U_{eq}(O). H atoms bonded to C and N were refined using a riding model with U(H) set to 1.2U_{eq}(C) for the methylene group and 1.5U_{eq}(C,*N*) for the methyl and the NH_3 group. Both of them were allowed to rotate but not to tip.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.02784 (18)	0.2864 (3)	0.8804 (3)	0.0459 (6)
C2	0.05733 (19)	0.1495 (3)	0.9312 (3)	0.0575 (8)
H2	0.009107	0.074666	0.896510	0.069*
C3	0.15679 (18)	0.1185 (2)	1.0327 (3)	0.0526 (7)
H3	0.174453	0.024333	1.065946	0.063*
C4	0.22828 (15)	0.2265 (2)	1.0833 (2)	0.0279 (5)
C5	0.20331 (19)	0.3638 (2)	1.0313 (3)	0.0527 (7)
H5	0.252962	0.437536	1.063048	0.063*
C6	0.1029 (2)	0.3927 (3)	0.9305 (3)	0.0654 (9)
H6	0.085992	0.486688	0.895849	0.078*
C7	−0.0826 (2)	0.3149 (3)	0.7724 (4)	0.0721 (9)
H7A	−0.083796	0.293072	0.663661	0.108*
H7B	−0.100685	0.414418	0.780352	0.108*
H7C	−0.133570	0.255254	0.804694	0.108*
C8	0.40281 (15)	−0.05466 (19)	0.8494 (2)	0.0235 (5)
C9	0.38105 (16)	−0.1849 (2)	0.9427 (2)	0.0268 (5)
H9A	0.424395	−0.176610	1.052432	0.032*
H9B	0.306807	−0.182213	0.944691	0.032*
C10	0.40283 (16)	−0.32853 (19)	0.8784 (2)	0.0253 (5)
N1	0.33326 (13)	0.19495 (17)	1.1900 (2)	0.0309 (4)
H1A	0.331546	0.109900	1.236679	0.046*
H1B	0.350879	0.263146	1.264632	0.046*
H1C	0.381413	0.192567	1.133796	0.046*
O1	0.37677 (11)	0.06550 (14)	0.89126 (17)	0.0345 (4)
O2	0.44594 (12)	−0.07617 (13)	0.73668 (17)	0.0327 (4)
O3	0.50449 (11)	−0.35675 (14)	0.90227 (17)	0.0325 (4)
H3A	0.5173 (18)	−0.4328 (17)	0.852 (2)	0.049*
O4	0.33156 (11)	−0.41000 (15)	0.81073 (19)	0.0395 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0363 (13)	0.0460 (14)	0.0477 (15)	0.0015 (11)	−0.0024 (11)	0.0024 (11)
C2	0.0397 (14)	0.0407 (15)	0.080 (2)	−0.0133 (11)	−0.0052 (14)	0.0067 (13)
C3	0.0361 (13)	0.0300 (12)	0.081 (2)	−0.0041 (10)	−0.0025 (13)	0.0090 (12)
C4	0.0252 (11)	0.0264 (11)	0.0320 (11)	0.0024 (8)	0.0073 (9)	0.0001 (9)
C5	0.0493 (15)	0.0311 (13)	0.0623 (17)	−0.0078 (11)	−0.0119 (13)	0.0033 (12)
C6	0.0616 (17)	0.0336 (14)	0.078 (2)	0.0027 (12)	−0.0207 (16)	0.0105 (13)
C7	0.0498 (17)	0.0668 (19)	0.081 (2)	0.0030 (14)	−0.0155 (16)	0.0079 (16)
C8	0.0224 (10)	0.0206 (10)	0.0247 (10)	−0.0008 (8)	0.0011 (9)	−0.0013 (8)
C9	0.0299 (11)	0.0232 (10)	0.0291 (11)	0.0002 (8)	0.0111 (9)	−0.0007 (8)
C10	0.0309 (11)	0.0205 (10)	0.0255 (11)	−0.0024 (9)	0.0094 (9)	0.0054 (8)
N1	0.0274 (9)	0.0244 (9)	0.0410 (11)	−0.0006 (7)	0.0090 (8)	0.0020 (8)
O1	0.0441 (9)	0.0204 (7)	0.0398 (9)	0.0065 (6)	0.0122 (7)	−0.0011 (6)
O2	0.0465 (9)	0.0232 (7)	0.0331 (8)	−0.0009 (6)	0.0187 (7)	0.0001 (6)

O3	0.0338 (8)	0.0205 (7)	0.0441 (9)	−0.0009 (6)	0.0121 (7)	−0.0032 (6)
O4	0.0361 (9)	0.0257 (8)	0.0530 (10)	−0.0061 (7)	0.0056 (8)	−0.0073 (7)

Geometric parameters (Å, º)

C1—C2	1.366 (3)	C7—H7C	0.9600
C1—C6	1.373 (3)	C8—O1	1.247 (2)
C1—C7	1.513 (3)	C8—O2	1.265 (2)
C2—C3	1.385 (3)	C8—C9	1.522 (3)
C2—H2	0.9300	C9—C10	1.501 (3)
C3—C4	1.357 (3)	C9—H9A	0.9700
C3—H3	0.9300	C9—H9B	0.9700
C4—C5	1.362 (3)	C10—O4	1.218 (2)
C4—N1	1.459 (2)	C10—O3	1.308 (2)
C5—C6	1.389 (3)	N1—H1A	0.8900
C5—H5	0.9300	N1—H1B	0.8900
C6—H6	0.9300	N1—H1C	0.8900
C7—H7A	0.9600	O3—H3A	0.868 (10)
C7—H7B	0.9600		
C2—C1—C6	116.8 (2)	H7A—C7—H7C	109.5
C2—C1—C7	120.0 (2)	H7B—C7—H7C	109.5
C6—C1—C7	123.1 (2)	O1—C8—O2	125.19 (18)
C1—C2—C3	122.1 (2)	O1—C8—C9	116.89 (18)
C1—C2—H2	118.9	O2—C8—C9	117.92 (16)
C3—C2—H2	118.9	C10—C9—C8	115.34 (16)
C4—C3—C2	119.6 (2)	C10—C9—H9A	108.4
C4—C3—H3	120.2	C8—C9—H9A	108.4
C2—C3—H3	120.2	C10—C9—H9B	108.4
C3—C4—C5	120.2 (2)	C8—C9—H9B	108.4
C3—C4—N1	120.01 (18)	H9A—C9—H9B	107.5
C5—C4—N1	119.81 (18)	O4—C10—O3	123.91 (18)
C4—C5—C6	119.3 (2)	O4—C10—C9	122.39 (18)
C4—C5—H5	120.4	O3—C10—C9	113.70 (16)
C6—C5—H5	120.4	C4—N1—H1A	109.5
C1—C6—C5	122.0 (2)	C4—N1—H1B	109.5
C1—C6—H6	119.0	H1A—N1—H1B	109.5
C5—C6—H6	119.0	C4—N1—H1C	109.5
C1—C7—H7A	109.5	H1A—N1—H1C	109.5
C1—C7—H7B	109.5	H1B—N1—H1C	109.5
H7A—C7—H7B	109.5	C10—O3—H3A	113.8 (16)
C1—C7—H7C	109.5		
C6—C1—C2—C3	2.1 (4)	C2—C1—C6—C5	−1.6 (5)
C7—C1—C2—C3	−178.7 (3)	C7—C1—C6—C5	179.2 (3)
C1—C2—C3—C4	−0.5 (5)	C4—C5—C6—C1	−0.4 (5)
C2—C3—C4—C5	−1.6 (4)	O1—C8—C9—C10	173.92 (16)
C2—C3—C4—N1	−179.9 (2)	O2—C8—C9—C10	−6.4 (2)

C3—C4—C5—C6	2.1 (4)	C8—C9—C10—O4	−107.8 (2)
N1—C4—C5—C6	−179.6 (2)	C8—C9—C10—O3	72.3 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4^{i}	0.89	1.96	2.846 (2)	172
N1—H1*B*···O1^{ii}	0.89	1.91	2.789 (2)	169
N1—H1*C*···O1	0.89	2.40	3.041 (2)	130
N1—H1*C*···O2^{iii}	0.89	2.47	2.980 (2)	117
N1—H1*C*···O3^{iii}	0.89	2.21	2.871 (2)	131
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Symmetry codes: (i) *x*, −*y*−1/2, *z*+1/2; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*+1, −*y*, −*z*+2; (iv) −*x*+1, *y*−1/2, −*z*+3/2.