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4-Methylanilinium 3-carboxy-2-hydroxypropanoate

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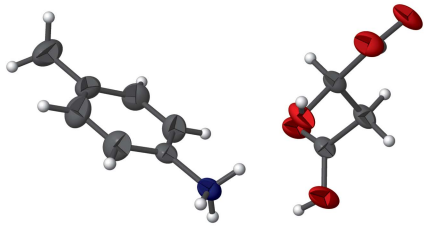
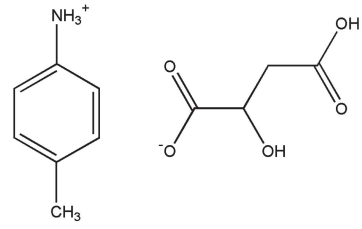
Structural data: full structural data are available from iucrdata.iucr.org

The title molecular salt, $C_7H_{10}N^+\cdot C_4H_5O_5^-$, contains a 4-methylanilinium cation and a 3-carboxy-2-hydroxypropanoate (hydrogen 2-hydroxysuccinate) anion in the asymmetric unit. The cation is protonated at the amine N atom and the anion is deprotonated at one of the hydroxy O atoms of the carboxylic acid groups. An O—H···O hydrogen bond in the anion generates an $S(5)$ graph-set motif. An N—H···O hydrogen bond links the anion and cation in the asymmetric unit. In the crystal, N—H···O and O—H···O hydrogen bonds link adjacent anions and cations, forming a two-dimensional network parallel to the *ac* plane and enclosing $R_2^3(12)$, $R_2^3(14)$ and $R_3^2(10)$ ring motifs.

3D view



Chemical scheme



Structure description

Succinic acid derivatives are mostly used as food and pharmaceutical chemicals (Sauer *et al.*, 2008). We report the synthesis and the crystal structure of the title molecular salt (Fig. 1), which contains a 4-methylanilinium cation and 2-hydroxysuccinate anion in the asymmetric unit. Geometrical parameters are comparable with those for the reported structures of 4-methylanilinium nitrate (Benali-Cherif *et al.*, 2009) and 2-hydroxysuccinate salts (Fleck *et al.*, 2001).

The cation is protonated at amine atom N1 and the anion is deprotonated at hydroxy atom O2. An N1—H1*A*···O5 hydrogen bond links the anion and cation while an O5—H5*A*···O1 hydrogen bond generates an $S(5)$ graph-set motif in the anion (Fig. 1 and Table 1).

In the crystal structure, N—H···O and O—H···O hydrogen bonds link adjacent anions and cations into infinite two-dimensional networks along the *ac* plane (Table 1 and Fig. 2). The inter-ionic N1—H1*B*···O1^{i}, N1—H1*A*···O5^{ii} and O3—H3*A*···O2^{i} hydrogen bonds generate an $R_2^3(12)$ ring motif, the N1—H1*B*···O1^{i}, N1—H1*C*···O2^{ii} and O5—

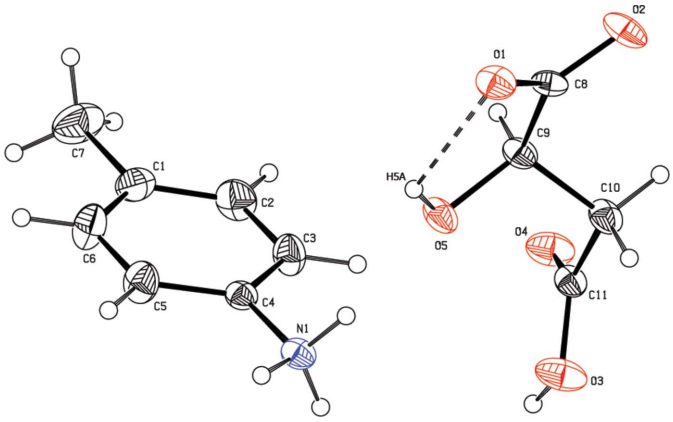


Figure 1
The molecular structure of the title molecular salt, showing the atom labelling and 30% probability displacement ellipsoids. The dashed line represents the intramolecular hydrogen bond.

H5*A*···O4^{i} hydrogen bonds generate an $R_2^3(14)$ ring motif and the N1—H1*A*···O5, N1—H1*C*···O2^{ii}, O5—H5*A*···O4^{iii} and O3—H3*A*···O2^{i} hydrogen bonds generate an $R_3^2(10)$ ring motif (Fig. 3; for symmetry codes, see Table 1).

Synthesis and crystallization

The title compound was synthesized from the raw materials *p*-toluidine and DL-malic acid which were taken in 1:1 ratio and dissolved in water at ambient temperature. The aqueous solution was continuously stirred for six h to prepare a homogeneous solution. After a transparent solution was

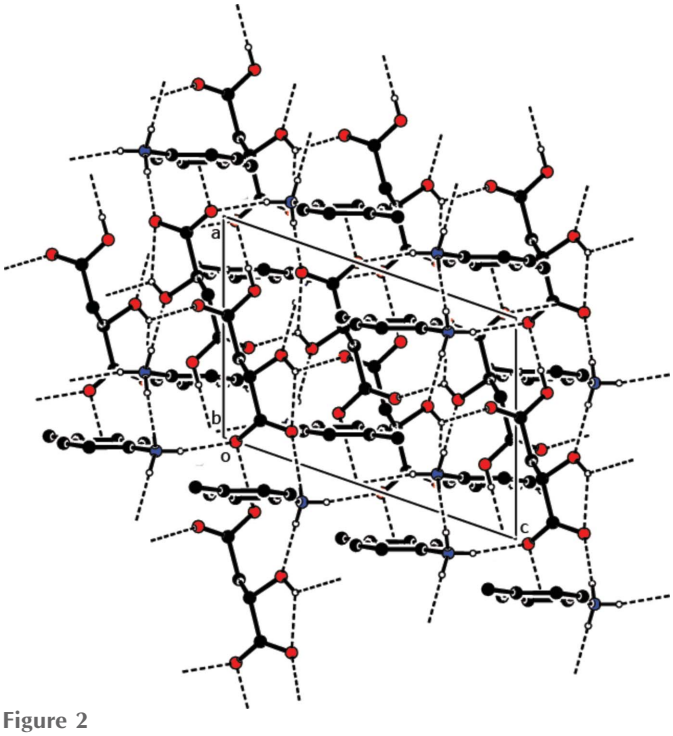


Figure 2
The crystal packing of the title molecular salt, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*A*···O1	0.82 (1)	2.12 (3)	2.6101 (15)	119 (2)
N1—H1*A*···O5	0.87 (1)	1.98 (1)	2.8381 (18)	172 (2)
N1—H1*B*···O1^{i}	0.87 (1)	1.88 (1)	2.7328 (18)	168 (2)
N1—H1*C*···O2^{ii}	0.87 (1)	2.01 (1)	2.8736 (19)	170 (2)
O3—H3*A*···O2^{i}	0.83 (1)	1.75 (1)	2.5830 (16)	179 (3)
O5—H5*A*···O4^{iii}	0.82 (1)	2.26 (2)	2.8088 (18)	125 (2)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_7H_{10}N^+\cdot C_4H_5O_5^-$
M_r	241.24
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	295
a, *b*, *c* (Å)	7.4849 (8), 16.1306 (16), 10.4904 (10)
β (°)	109.234 (3)
V (Å^3)	1195.9 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.26 × 0.22 × 0.20
Data collection	
Diffractometer	Bruker Kappa APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.686, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19297, 3403, 2078
R_{int}	0.047
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.700
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.048, 0.136, 1.03
No. of reflections	3403
No. of parameters	173
No. of restraints	5
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.37, −0.20

Computer programs: *APEX2* and *SAINT* (Bruker, 2004), *SHELXS97* (Sheldrick, 2008), *SHELXL2016* (Sheldrick, 2015) and *PLATON* (Spek, 2009).

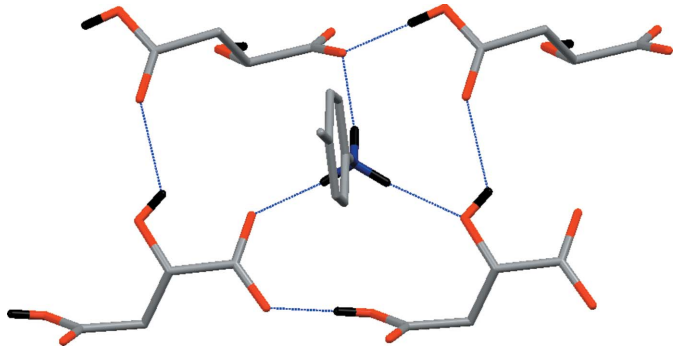


Figure 3
A partial view of the crystal packing showing the ring motifs. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

obtained, it was filtered and kept for slow evaporation. Crystals suitable for X-ray diffraction were obtained after a period of five weeks.

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161525 [doi:10.1107/S241431461601525X]

4-Methylanilinium 3-carboxy-2-hydroxypropanoate

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4-Methylanilinium 3-carboxy-2-hydroxypropanoate

Crystal data

$C_7H_{10}N^+·C_4H_5O_5^-$
M_r = 241.24
Monoclinic, $P2_1/c$
a = 7.4849 (8) Å
b = 16.1306 (16) Å
c = 10.4904 (10) Å
β = 109.234 (3)°
V = 1195.9 (2) Å^3
Z = 4
$F(000)$ = 512
D_x = 1.340 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5534 reflections
θ = 2.4–29.6°
μ = 0.11 mm^{-1}
T = 295 K
Block, colourless
0.26 × 0.22 × 0.20 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
ω and φ scan
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.686, T_{max} = 0.746
19297 measured reflections
3403 independent reflections
2078 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 29.9°, θ_{min} = 2.4°
h = −10→10
k = −22→22
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.136
S = 1.03
3403 reflections
173 parameters
5 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 0.3504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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.

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.2423 (3)	0.50050 (12)	0.9996 (2)	0.0447 (5)

C2	0.2804 (3)	0.58051 (13)	1.04649 (19)	0.0498 (5)
H2	0.322596	0.590187	1.139054	0.060*
C3	0.2574 (3)	0.64649 (12)	0.95917 (17)	0.0436 (4)
H3	0.284307	0.700093	0.992660	0.052*
C4	0.1946 (2)	0.63270 (10)	0.82288 (16)	0.0297 (4)
C5	0.1539 (3)	0.55398 (12)	0.77248 (19)	0.0463 (5)
H5	0.110417	0.544643	0.679829	0.056*
C6	0.1787 (3)	0.48874 (13)	0.8621 (2)	0.0544 (5)
H6	0.151451	0.435215	0.828325	0.065*
C7	0.2706 (3)	0.42870 (15)	1.0955 (3)	0.0683 (7)
H7A	0.267825	0.448194	1.181262	0.102*
H7B	0.390753	0.403169	1.106808	0.102*
H7C	0.171438	0.388864	1.059646	0.102*
C8	0.8664 (2)	0.81546 (11)	0.87375 (15)	0.0300 (4)
C9	0.6854 (2)	0.80756 (11)	0.91036 (16)	0.0329 (4)
H9	0.711761	0.770807	0.988796	0.039*
C10	0.6193 (2)	0.88973 (11)	0.94679 (19)	0.0372 (4)
H10A	0.591569	0.927381	0.870639	0.045*
H10B	0.717729	0.914278	1.022132	0.045*
C11	0.4433 (2)	0.87581 (10)	0.98457 (16)	0.0307 (4)
N1	0.16966 (19)	0.70315 (10)	0.73151 (15)	0.0324 (3)
O1	0.86759 (15)	0.78284 (8)	0.76746 (12)	0.0398 (3)
O2	1.00180 (15)	0.85307 (9)	0.95801 (11)	0.0443 (3)
O3	0.28940 (16)	0.90265 (10)	0.89417 (13)	0.0509 (4)
O4	0.44432 (17)	0.84089 (9)	1.08584 (13)	0.0495 (4)
O5	0.53901 (15)	0.77145 (8)	0.80248 (12)	0.0405 (3)
H1A	0.2777 (18)	0.7287 (12)	0.753 (2)	0.052 (6)*
H1B	0.080 (2)	0.7348 (11)	0.739 (2)	0.050 (6)*
H1C	0.131 (3)	0.6877 (13)	0.6475 (11)	0.058 (6)*
H3A	0.197 (3)	0.8863 (16)	0.915 (3)	0.087*
H5A	0.589 (3)	0.7479 (16)	0.754 (2)	0.087*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0391 (10)	0.0430 (11)	0.0527 (12)	0.0069 (8)	0.0160 (8)	0.0088 (9)
C2	0.0566 (12)	0.0563 (13)	0.0313 (9)	−0.0025 (10)	0.0073 (8)	0.0025 (9)
C3	0.0539 (11)	0.0403 (11)	0.0338 (9)	−0.0078 (8)	0.0109 (8)	−0.0075 (8)
C4	0.0236 (7)	0.0348 (9)	0.0322 (8)	0.0010 (6)	0.0111 (6)	−0.0015 (7)
C5	0.0604 (12)	0.0433 (11)	0.0345 (9)	−0.0009 (9)	0.0146 (8)	−0.0094 (8)
C6	0.0715 (14)	0.0345 (11)	0.0581 (13)	−0.0012 (10)	0.0226 (11)	−0.0069 (9)
C7	0.0657 (14)	0.0611 (15)	0.0812 (17)	0.0157 (12)	0.0284 (13)	0.0325 (13)
C8	0.0191 (7)	0.0424 (10)	0.0295 (8)	0.0041 (6)	0.0091 (6)	0.0049 (7)
C9	0.0231 (7)	0.0430 (10)	0.0347 (8)	−0.0027 (7)	0.0123 (6)	−0.0018 (7)
C10	0.0259 (8)	0.0443 (10)	0.0453 (10)	−0.0067 (7)	0.0169 (7)	−0.0082 (8)
C11	0.0255 (7)	0.0378 (9)	0.0317 (8)	−0.0023 (6)	0.0133 (6)	−0.0064 (7)
N1	0.0261 (7)	0.0411 (9)	0.0323 (8)	−0.0005 (6)	0.0129 (6)	−0.0015 (6)
O1	0.0312 (6)	0.0538 (8)	0.0394 (7)	0.0036 (5)	0.0184 (5)	−0.0043 (6)

O2	0.0219 (5)	0.0758 (10)	0.0357 (7)	−0.0091 (6)	0.0101 (5)	−0.0050 (6)
O3	0.0270 (6)	0.0840 (11)	0.0446 (7)	0.0028 (6)	0.0156 (5)	0.0187 (7)
O4	0.0343 (7)	0.0718 (10)	0.0437 (7)	0.0051 (6)	0.0145 (5)	0.0159 (7)
O5	0.0247 (6)	0.0522 (8)	0.0472 (7)	−0.0080 (5)	0.0152 (5)	−0.0180 (6)

Geometric parameters (Å, º)

C1—C6	1.375 (3)	C8—O2	1.2596 (19)
C1—C2	1.377 (3)	C8—C9	1.530 (2)
C1—C7	1.503 (3)	C9—O5	1.4155 (19)
C2—C3	1.378 (3)	C9—C10	1.508 (2)
C2—H2	0.9300	C9—H9	0.9800
C3—C4	1.368 (2)	C10—C11	1.512 (2)
C3—H3	0.9300	C10—H10A	0.9700
C4—C5	1.371 (3)	C10—H10B	0.9700
C4—N1	1.458 (2)	C11—O4	1.200 (2)
C5—C6	1.382 (3)	C11—O3	1.302 (2)
C5—H5	0.9300	N1—H1A	0.868 (9)
C6—H6	0.9300	N1—H1B	0.868 (9)
C7—H7A	0.9600	N1—H1C	0.869 (9)
C7—H7B	0.9600	O3—H3A	0.831 (10)
C7—H7C	0.9600	O5—H5A	0.816 (10)
C8—O1	1.2357 (19)		
C6—C1—C2	117.62 (18)	O1—C8—C9	117.47 (14)
C6—C1—C7	121.3 (2)	O2—C8—C9	116.00 (14)
C2—C1—C7	121.06 (19)	O5—C9—C10	109.40 (13)
C1—C2—C3	121.37 (18)	O5—C9—C8	110.47 (13)
C1—C2—H2	119.3	C10—C9—C8	112.47 (14)
C3—C2—H2	119.3	O5—C9—H9	108.1
C4—C3—C2	119.60 (17)	C10—C9—H9	108.1
C4—C3—H3	120.2	C8—C9—H9	108.1
C2—C3—H3	120.2	C9—C10—C11	108.84 (14)
C3—C4—C5	120.66 (17)	C9—C10—H10A	109.9
C3—C4—N1	119.05 (15)	C11—C10—H10A	109.9
C5—C4—N1	120.28 (15)	C9—C10—H10B	109.9
C4—C5—C6	118.69 (17)	C11—C10—H10B	109.9
C4—C5—H5	120.7	H10A—C10—H10B	108.3
C6—C5—H5	120.7	O4—C11—O3	123.16 (14)
C1—C6—C5	122.06 (19)	O4—C11—C10	123.35 (15)
C1—C6—H6	119.0	O3—C11—C10	113.45 (15)
C5—C6—H6	119.0	C4—N1—H1A	107.0 (14)
C1—C7—H7A	109.5	C4—N1—H1B	109.3 (14)
C1—C7—H7B	109.5	H1A—N1—H1B	112 (2)
H7A—C7—H7B	109.5	C4—N1—H1C	111.8 (15)
C1—C7—H7C	109.5	H1A—N1—H1C	111 (2)
H7A—C7—H7C	109.5	H1B—N1—H1C	105.1 (19)
H7B—C7—H7C	109.5	C11—O3—H3A	108.4 (19)

O1—C8—O2	126.49 (14)	C9—O5—H5A	107 (2)
C6—C1—C2—C3	−0.6 (3)	C4—C5—C6—C1	0.1 (3)
C7—C1—C2—C3	179.1 (2)	O1—C8—C9—O5	−7.5 (2)
C1—C2—C3—C4	0.3 (3)	O2—C8—C9—O5	174.38 (14)
C2—C3—C4—C5	0.2 (3)	O1—C8—C9—C10	−130.05 (16)
C2—C3—C4—N1	179.61 (17)	O2—C8—C9—C10	51.8 (2)
C3—C4—C5—C6	−0.4 (3)	O5—C9—C10—C11	58.19 (18)
N1—C4—C5—C6	−179.80 (17)	C8—C9—C10—C11	−178.66 (13)
C2—C1—C6—C5	0.3 (3)	C9—C10—C11—O4	69.8 (2)
C7—C1—C6—C5	−179.3 (2)	C9—C10—C11—O3	−107.77 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*A*···O1	0.82 (1)	2.12 (3)	2.6101 (15)	119 (2)
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N1—H1*B*···O1^{i}	0.87 (1)	1.88 (1)	2.7328 (18)	168 (2)
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O5—H5*A*···O4^{iii}	0.82 (1)	2.26 (2)	2.8088 (18)	125 (2)

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