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Bis(ethanolaminium) succinate–succinic acid (1/1)

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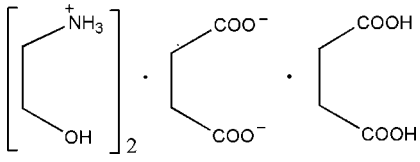
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.106; data-to-parameter ratio = 14.8.

The asymmetric unit of the title compound, $2C_2H_8NO^+\cdot C_4H_4O_4^{2-}\cdot C_4H_6O_4$, consists of half a succinate anion, half a succinic acid molecule and one ethanolaminium cation. The succinate anion and succinic acid molecule, both of which are located on inversion centres, are linked by O—H···O hydrogen bonds, forming a chain along the [2$\bar{1}$0] direction. The chain and the ethanolaminium cation are further connected by O—H···O and N—H···O hydrogen bonds.

Related literature

For related structures of co-crystals and salts of succinic acid, see: Aakeroy *et al.* (1998); Batchelor *et al.* (2001); Borthwick (1980); Braga *et al.* (2003); Bruno *et al.* (2004); Büyükgüngör & Odabasoglu (2002); Flensburg *et al.* (1995); Kuipers *et al.* (1997); Li *et al.* (2003); MacDonald *et al.* (2001); Prasad & Vijayan (1990); Reitz *et al.* (1998); Urbanczyk-Lipkowska (2000).



Experimental

Crystal data

$2C_2H_8NO^+\cdot C_4H_4O_4^{2-}\cdot C_4H_6O_4$
M_r = 358.35
Triclinic, $P\bar{1}$
a = 5.821 (5) Å
b = 8.428 (7) Å
c = 9.077 (7) Å
α = 87.74 (1)°
β = 73.628 (11)°
γ = 80.380 (12)°
V = 421.2 (6) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.42 × 0.34 × 0.30 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.855, T_{max} = 0.898
2330 measured reflections
1628 independent reflections
1517 reflections with $I > 2\sigma(I)$
R_{int} = 0.011

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.106
S = 1.07
1628 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O1^{i}	0.89	2.09	2.976 (2)	171
N1—H1B···O3^{ii}	0.89	1.93	2.810 (2)	167
N1—H1C···O5	0.89	2.55	2.868 (3)	102
N1—H1C···O5^{iii}	0.89	2.31	2.913 (3)	125
O2—H2···O4	0.82	1.66	2.466 (2)	166
O5—H5···O4	0.82	2.01	2.697 (2)	142

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IS2756).

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S1. Comment

There exist three forms of succinic acid in molecular complex, namely, succinate (Kuipers *et al.*, 1997; Urbanczyk-Lipkowska, 2000), succinic acid (Aakeroy *et al.*, 1998; Batchelor *et al.*, 2001), hydrogen succinate (Flensburg *et al.*, 1995; MacDonald *et al.*, 2001). Interestingly, some mixed forms are also available (Prasad & Vijayan, 1990; Reitz *et al.*, 1998; Büyükgüngör & Odabasoglu, 2002; Braga *et al.*, 2003; Bruno *et al.*, 2004). However, no convincing explanation for the formation of any of the complex. Recently, the title complex, (I) (Table 1 & Fig. 1), is synthesized, and the structure is studied hereafter.

As shown in Fig. 1, the asymmetric unit is composed of one ethanolaminium cation, half a succinate anion, and half a succinic acid molecule. The succinate anion and succinic acid molecule are linked by an O2—H2···O4 hydrogen bond (Table 1), then are associated with ethanolaminium cation by an O5—H5···O4 hydrogen bond. The distances of [C1—O1 1.2187 (18) Å and C1—O2 1.3009 (18) Å] indicate a carboxylic group, where C1—O1 stands for the carbonyl C=O bond. The distances of [C3—O3 1.2310 (18) Å and C3—O4 1.2831 (17) Å] indicate a carboxylate anion, where the C—O and C=O bonds are equalized, and no distinct carbonyl C=O bond is observed (Borthwick, 1980). This is mixed mode of succinate and succinic acid, and is similar to those observed in other cases (Prasad & Vijayan, 1990; Li *et al.*, 2003; Bruno *et al.*, 2004).

The succinate anion and the succinic acid molecule are arranged almost in the same layers (Fig. 2), and the ethanolaminium cation are sandwiched between the layers by O—H···O and N—H···O hydrogen bonds (Table 1).

S2. Experimental

Succinic acid (5.1 g) and ethanolamine (2.4 g), in a molar ratio of 1:1, were mixed and dissolved in sufficient ethanol by heating to 373 K, at which point a clear solution resulted. The system was then cooled slowly to room temperature. Crystals of (I) (4.3 g) were formed, collected and washed with ethanol.

S3. Refinement

All H atoms were placed in calculated positions and allowed to ride on their parent atoms with distances of 0.89 Å for the amido, 0.97 Å for the methylene and 0.82 Å for the hydroxyl group, and with isotropic displacement parameters 1.2–1.5 times U_{eq} of the parent atoms.

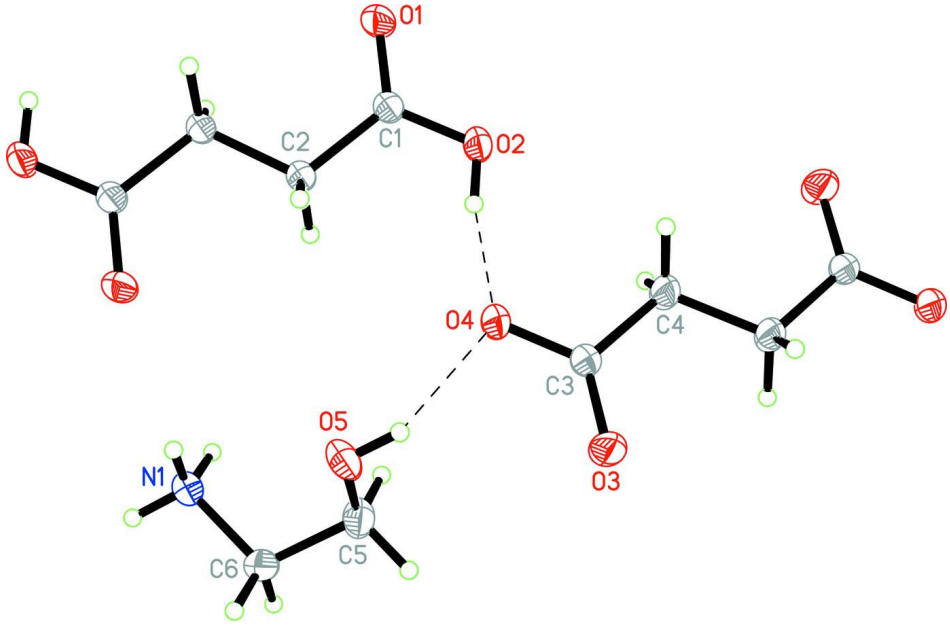

Figure 1

The molecular structure of the title compound. Hydrogen bonds are illustrated as dashed lines.

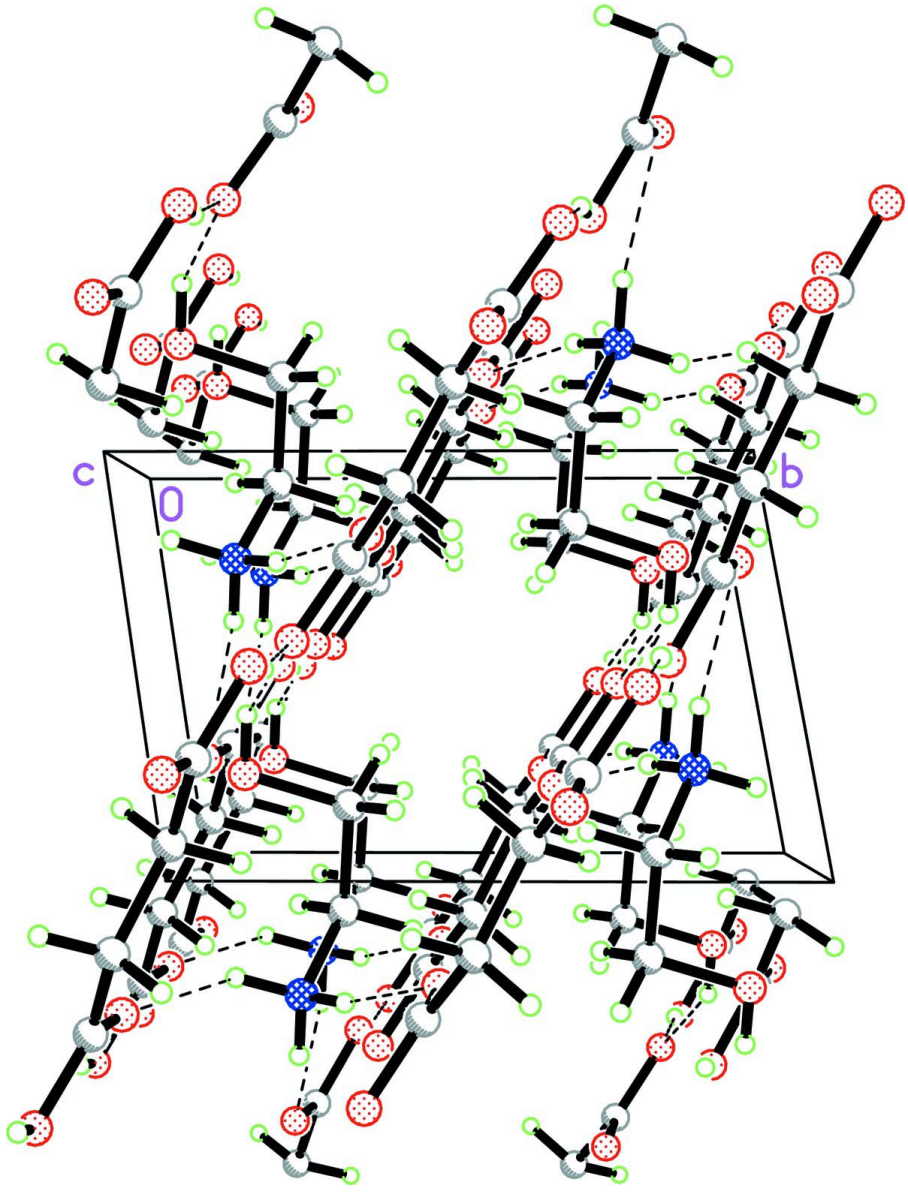

Figure 2

The crystal packing of the title compound, viewed down the *c* axis. Hydrogen bonds are drawn as dashed lines.

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Crystal data

$2C_2H_8NO^+·C_4H_4O_4^{2-}·C_4H_6O_4$
M_r = 358.35
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 5.821 (5) Å
b = 8.428 (7) Å
c = 9.077 (7) Å
α = 87.74 (1)°
β = 73.628 (11)°
γ = 80.380 (12)°
V = 421.2 (6) Å^3
Z = 1
F(000) = 192.0
D_x = 1.413 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1360 reflections
θ = 2.1–17.8°
μ = 0.12 mm^{-1}

T = 293 K
Prism, colorless

0.42 × 0.34 × 0.30 mm

Data collection

Bruker APEX area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.855, T_{max} = 0.898

2330 measured reflections
1628 independent reflections
1517 reflections with $I > 2\sigma(I)$
R_{int} = 0.011
θ_{max} = 26.0°, θ_{min} = 2.3°
$h = -4 \rightarrow 7$
$k = -9 \rightarrow 10$
$l = -11 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.106
S = 1.07
1628 reflections
110 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0582P)^2 + 0.1261P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.160 (15)

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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.27229 (18)	0.92694 (13)	0.23761 (10)	0.0366 (3)
O2	0.49374 (19)	0.79510 (15)	0.37464 (11)	0.0455 (3)
H2	0.4875	0.7808	0.4655	0.068*
O3	0.7920 (2)	0.61078 (13)	0.76185 (11)	0.0410 (3)
O4	0.54895 (19)	0.74043 (13)	0.63248 (11)	0.0401 (3)
O5	0.24811 (17)	0.85165 (11)	0.90544 (11)	0.0341 (3)
H5	0.3825	0.8324	0.8434	0.051*
N1	−0.25612 (19)	0.82179 (14)	1.00467 (12)	0.0293 (3)
H1A	−0.4002	0.8428	1.0740	0.044*
H1B	−0.2650	0.7570	0.9321	0.044*
H1C	−0.2131	0.9133	0.9624	0.044*
C1	0.2989 (2)	0.89023 (16)	0.36369 (14)	0.0263 (3)

C2	0.1142 (2)	0.95017 (16)	0.51200 (14)	0.0275 (3)
H2A	0.1871	1.0147	0.5668	0.033*
H2B	0.0709	0.8586	0.5756	0.033*
C3	0.7343 (2)	0.63837 (16)	0.64176 (14)	0.0275 (3)
C4	0.8844 (2)	0.55193 (17)	0.49388 (15)	0.0328 (3)
H4A	0.7865	0.4850	0.4619	0.039*
H4B	0.9250	0.6314	0.4146	0.039*
C5	0.1738 (2)	0.70523 (16)	0.96716 (17)	0.0336 (3)
H5A	0.2895	0.6486	1.0177	0.040*
H5B	0.1672	0.6370	0.8854	0.040*
C6	−0.0726 (2)	0.74220 (17)	1.08064 (15)	0.0301 (3)
H6A	−0.1207	0.6431	1.1278	0.036*
H6B	−0.0653	0.8120	1.1610	0.036*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0321 (5)	0.0531 (7)	0.0204 (5)	0.0042 (4)	−0.0067 (4)	0.0002 (4)
O2	0.0322 (6)	0.0685 (8)	0.0230 (5)	0.0212 (5)	−0.0038 (4)	−0.0017 (5)
O3	0.0455 (6)	0.0490 (6)	0.0238 (5)	0.0142 (5)	−0.0135 (4)	−0.0085 (4)
O4	0.0341 (6)	0.0510 (6)	0.0240 (5)	0.0185 (5)	−0.0041 (4)	−0.0040 (4)
O5	0.0315 (5)	0.0336 (5)	0.0291 (5)	−0.0030 (4)	0.0042 (4)	−0.0051 (4)
N1	0.0265 (6)	0.0322 (6)	0.0259 (6)	−0.0004 (4)	−0.0040 (4)	−0.0022 (4)
C1	0.0226 (6)	0.0327 (7)	0.0218 (6)	−0.0008 (5)	−0.0052 (5)	−0.0003 (5)
C2	0.0227 (7)	0.0359 (7)	0.0204 (6)	0.0027 (5)	−0.0047 (5)	0.0002 (5)
C3	0.0268 (6)	0.0303 (7)	0.0221 (6)	0.0030 (5)	−0.0057 (5)	−0.0021 (5)
C4	0.0305 (7)	0.0401 (8)	0.0240 (7)	0.0103 (6)	−0.0096 (6)	−0.0077 (6)
C5	0.0284 (7)	0.0299 (7)	0.0387 (8)	0.0014 (5)	−0.0066 (6)	−0.0008 (6)
C6	0.0296 (7)	0.0349 (7)	0.0250 (6)	−0.0038 (5)	−0.0075 (5)	0.0030 (5)

Geometric parameters (Å, º)

O1—C1	1.2187 (18)	C2—C2^{i}	1.516 (2)
O2—C1	1.3009 (18)	C2—H2A	0.9700
O2—H2	0.8200	C2—H2B	0.9700
O3—C3	1.2310 (18)	C3—C4	1.5162 (19)
O4—C3	1.2831 (17)	C4—C4^{ii}	1.508 (3)
O5—C5	1.4181 (19)	C4—H4A	0.9700
O5—H5	0.8200	C4—H4B	0.9700
N1—C6	1.4851 (18)	C5—C6	1.503 (2)
N1—H1A	0.8900	C5—H5A	0.9700
N1—H1B	0.8900	C5—H5B	0.9700
N1—H1C	0.8900	C6—H6A	0.9700
C1—C2	1.5097 (19)	C6—H6B	0.9700
C1—O2—H2	109.5	O4—C3—C4	116.06 (11)
C5—O5—H5	109.5	C4^{ii}—C4—C3	114.11 (14)
C6—N1—H1A	109.5	C4^{ii}—C4—H4A	108.7

C6—N1—H1B	109.5	C3—C4—H4A	108.7
H1A—N1—H1B	109.5	C4^{ii}—C4—H4B	108.7
C6—N1—H1C	109.5	C3—C4—H4B	108.7
H1A—N1—H1C	109.5	H4A—C4—H4B	107.6
H1B—N1—H1C	109.5	O5—C5—C6	108.92 (11)
O1—C1—O2	119.92 (12)	O5—C5—H5A	109.9
O1—C1—C2	123.11 (12)	C6—C5—H5A	109.9
O2—C1—C2	116.97 (11)	O5—C5—H5B	109.9
C1—C2—C2^{i}	113.14 (13)	C6—C5—H5B	109.9
C1—C2—H2A	109.0	H5A—C5—H5B	108.3
C2^{i}—C2—H2A	109.0	N1—C6—C5	111.06 (12)
C1—C2—H2B	109.0	N1—C6—H6A	109.4
C2^{i}—C2—H2B	109.0	C5—C6—H6A	109.4
H2A—C2—H2B	107.8	N1—C6—H6B	109.4
O3—C3—O4	123.24 (12)	C5—C6—H6B	109.4
O3—C3—C4	120.70 (12)	H6A—C6—H6B	108.0

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{iii}	0.89	2.09	2.976 (2)	171
N1—H1*B*···O3^{iv}	0.89	1.93	2.810 (2)	167
N1—H1*C*···O5	0.89	2.55	2.868 (3)	102
N1—H1*C*···O5^{v}	0.89	2.31	2.913 (3)	125
O2—H2···O4	0.82	1.66	2.466 (2)	166
O5—H5···O4	0.82	2.01	2.697 (2)	142

Symmetry codes: (iii) *x*−1, *y*, *z*+1; (iv) *x*−1, *y*, *z*; (v) −*x*, −*y*+2, −*z*+2.