Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*S*)-2-(1*H*-Imidazol-1-yl)succinic acid

Jing-Mei Xiao

Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 211189, People's Republic of China
Correspondence e-mail: xjm_cool@163.com

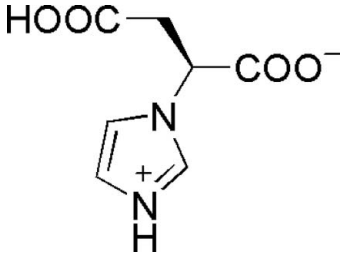
Received 21 December 2008; accepted 23 April 2009

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.050; wR factor = 0.151; data-to-parameter ratio = 9.4.

The title compound, $C_7H_8N_2O_4$, is a zwitterion, [formal name = (*S*)-3-carboxy-2-(imidazol-3-ium-1-yl)propanoate], in which the deprotonated negatively charged carboxylate end shows almost identical C—O bond distances [1.248 (4) and 1.251 (4) Å] due to resonance. The molecules are involved in intermolecular O—H···O and N—H···O hydrogen bonds, which define a tightly bound three-dimensional structure.

Related literature

For the use of imidazol-1-ylalkanoic acids as probes to determine the intracellular and extracellular pH and cell volume by ^{1}H NMR, see: López *et al.*(1996). For the preparation of the title compound, see: Bao *et al.* (2003).



Experimental

Crystal data

$C_7H_8N_2O_4$ M_r = 184.15
Orthorhombic, $P2_12_12_1$
a = 7.3212 (16) Å
b = 7.9193 (16) Å
c = 14.254 (3) Å
V = 826.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.18 mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.97, T_{max} = 0.98
8489 measured reflections
1110 independent reflections
952 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.151
S = 1.12
1110 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	1.91	2.716 (4)	155
O3—H3*C*···O1^{ii}	0.86	1.71	2.572 (3)	177

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

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: *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: BG2234).

References

Bao, W., Wang, Z. & Li, Y. (2003). *J. Org. Chem.* **68**, 591–593.
López, P., Zaderenko, P., Balcazar, J. L., Fonseca, I., Cano, F. H. & Ballesteros, P. (1996). *J. Mol. Struct.* **377**, 105–112.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2009). E**65**, o1157 [doi:10.1107/S1600536809015220]

(*S*)-2-(1*H*-Imidazol-1-yl)succinic acid

Jing-Mei Xiao

S1. Comment

Imidazol-1-ylalkanoic acids are used as new probes to determine the intracellular and extracellular pH and cell volume by ^{1}H NMR. (López *et al.*, 1996). In this report we present the structure of (*S*)-2-(1*H*-imidazol-1-yl)succinic acid. As shown in Fig. 1, the title compound $C_7H_8N_2O_4$ exists in the form of an inner salt where the unprotonated, negatively charged carboxylato end shows almost identical C-O bond distances (1.248 (4) and 1.251 (4)Å respectively) due to resonance.. The molecules are involved in intermolecular O—H···O and N—H···O hydrogen bonds (Table 1) which define a tightly bound 3D structure.

S2. Experimental

The ligand was prepared according to a literature method (Bao *et al.*, 2003). A formaldehyde water solution (36%, 1.67 g) and a glyoxal water solution (32%, 3.62 g) were mixed in a 50 ml, three-necked flask provided with a stirrer and a reflux condenser. While the mixture was heated at 50 °C with stirring, a mixture of *L*-2-aminosuccinic acid (2.66 g, 0.02 mol), ammonia solution (28%, 1.21 g) and sodium hydroxide solution (10%, 8 g) was added in small portions during 0.5 h. After the mixture was stirred for an additional 8 h at 50 °C, the cooled mixture was acidified to pH=3 with concentrated hydrochloric acid. After stirring for 30 min, the suspension was filtered. The resulting solid was washed with H_2O and dried in vacuum over P2O5 at room temperature. Colourless crystals suitable for X-ray diffraction were obtained from a solution of 100 mg in 15 ml H_2O by slow evaporation after one month.

S3. Refinement

Positional parameters of all the H atoms except for H3C were calculated geometrically and the H atoms were set to ride on the C and N atoms to which they are bonded, with Uiso(H) = 1.2Ueq(C or N). The carboxyl H3C was initially refined and subsequently allowed to ride with Uiso(H) = 1.5Ueq(O). Due to the abscence of anomalous diffraction effects, Friedel pairs were merged.

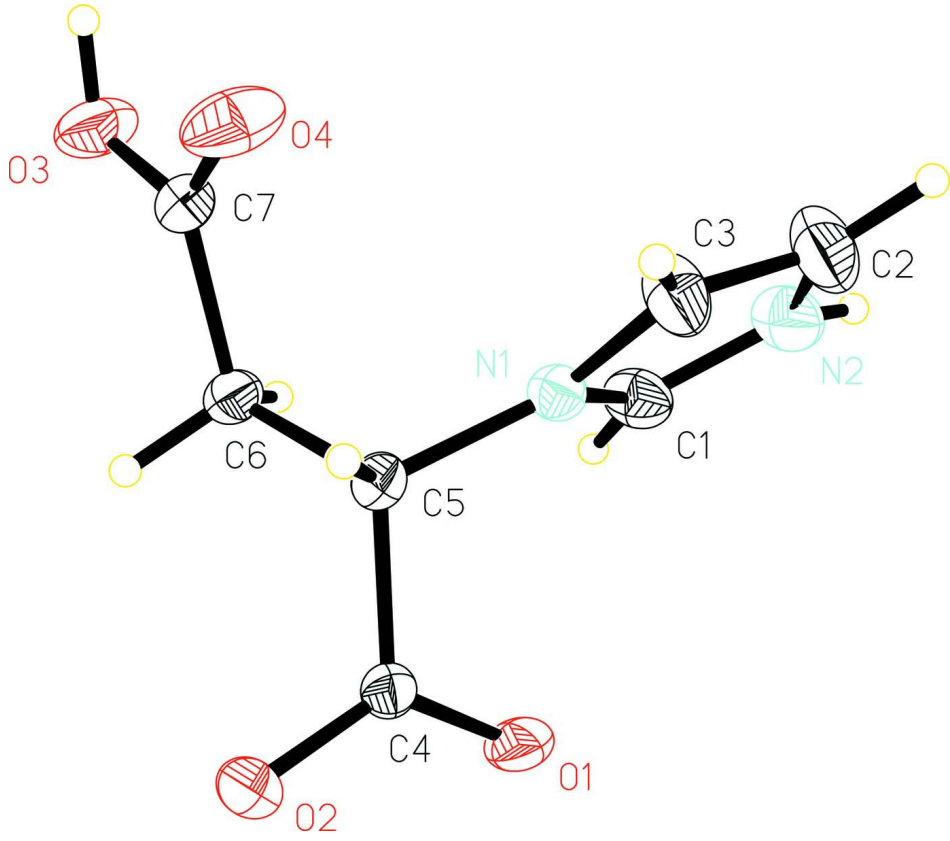


Figure 1

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

(*S*)-3-carboxy-2-(imidazol-3-ium-1-yl)propanoate

Crystal data

$C_7H_8N_2O_4$
$M_r = 184.15$
Orthorhombic, $P2_12_12_1$
Hall symbol: p 2ac 2ab
$a = 7.3212$ (16) Å
$b = 7.9193$ (16) Å
$c = 14.254$ (3) Å
$V = 826.4$ (3) Å^3
$Z = 4$
$F(000) = 384$
$D_x = 1.480$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2123 reflections
$\theta = 2.8$–$27.4°$
$\mu = 0.12$ mm^{-1}
$T = 293$ K
Prism, colorless
0.25 × 0.20 × 0.18 mm

Data collection

Rigaku Mercury2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
$T_{min} = 0.97$, $T_{max} = 0.98$
8489 measured reflections
1110 independent reflections
952 reflections with $I > 2\sigma(I)$
$R_{int} = 0.053$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.151$
$S = 1.12$
1110 reflections
118 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
$w = 1/[\sigma^2(F_o^2) + (0.0852P)^2 + 0.2196P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ 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. 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}
C1	0.9679 (4)	0.2336 (4)	0.6099 (2)	0.0299 (7)
C2	0.9073 (4)	0.4110 (4)	0.6415 (2)	0.0309 (7)
H2A	0.9846	0.4450	0.6943	0.037*
C3	0.9360 (5)	0.5376 (4)	0.5624 (2)	0.0367 (7)
H3A	1.0554	0.5197	0.5346	0.044*
H3B	0.8447	0.5193	0.5142	0.044*
C4	0.9226 (5)	0.7177 (4)	0.5977 (2)	0.0378 (7)
C5	0.6549 (5)	0.4474 (5)	0.7626 (3)	0.0493 (10)
H5	0.7259	0.4826	0.8130	0.059*
C6	0.4741 (6)	0.4230 (7)	0.7635 (3)	0.0640 (13)
H6	0.3962	0.4380	0.8144	0.077*
C7	0.5708 (5)	0.3653 (5)	0.6236 (3)	0.0422 (8)
H7	0.5719	0.3333	0.5608	0.051*
N1	0.7172 (3)	0.4110 (3)	0.67364 (18)	0.0314 (6)
N2	0.4268 (4)	0.3725 (4)	0.6766 (2)	0.0507 (8)
H2	0.3175	0.3487	0.6589	0.061*
O1	0.8474 (4)	0.1251 (3)	0.5944 (2)	0.0507 (7)
O2	1.1355 (3)	0.2163 (3)	0.59668 (16)	0.0409 (6)
O3	0.9107 (5)	0.8279 (3)	0.52925 (19)	0.0584 (9)
H3C	0.8908	0.9290	0.5489	0.070*
O4	0.9181 (5)	0.7543 (4)	0.67883 (19)	0.0638 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0279 (14)	0.0283 (15)	0.0334 (15)	0.0011 (12)	−0.0001 (12)	0.0036 (13)
C2	0.0315 (15)	0.0274 (15)	0.0338 (15)	−0.0038 (13)	0.0014 (13)	−0.0015 (12)
C3	0.0455 (18)	0.0265 (15)	0.0382 (16)	0.0006 (15)	0.0092 (16)	0.0019 (12)
C4	0.0396 (17)	0.0287 (15)	0.0452 (18)	0.0001 (15)	0.0057 (15)	0.0020 (14)
C5	0.0390 (19)	0.064 (3)	0.045 (2)	−0.0105 (19)	0.0083 (16)	−0.0169 (19)
C6	0.055 (2)	0.077 (3)	0.061 (2)	−0.014 (2)	0.024 (2)	−0.022 (3)
C7	0.0345 (16)	0.0433 (19)	0.0486 (18)	0.0050 (17)	−0.0069 (16)	−0.0066 (15)
N1	0.0293 (13)	0.0301 (13)	0.0349 (14)	−0.0001 (11)	−0.0025 (11)	−0.0032 (11)
N2	0.0310 (14)	0.0487 (18)	0.072 (2)	−0.0010 (15)	−0.0041 (16)	−0.0126 (16)
O1	0.0411 (13)	0.0241 (12)	0.087 (2)	−0.0010 (10)	0.0034 (14)	−0.0071 (13)
O2	0.0350 (12)	0.0383 (13)	0.0494 (14)	0.0044 (10)	0.0047 (11)	−0.0035 (11)
O3	0.090 (2)	0.0294 (13)	0.0560 (15)	0.0060 (15)	0.0122 (16)	0.0052 (11)
O4	0.106 (3)	0.0381 (14)	0.0472 (15)	0.0013 (17)	−0.0113 (17)	−0.0097 (12)

Geometric parameters (Å, º)

C1—O2	1.249 (4)	C5—C6	1.338 (6)
C1—O1	1.251 (4)	C5—N1	1.378 (4)
C1—C2	1.541 (4)	C5—H5	0.9300
C2—N1	1.465 (4)	C6—N2	1.347 (6)
C2—C3	1.522 (4)	C6—H6	0.9300
C2—H2A	0.9800	C7—N2	1.299 (5)
C3—C4	1.516 (4)	C7—N1	1.338 (4)
C3—H3A	0.9700	C7—H7	0.9300
C3—H3B	0.9700	N2—H2	0.8600
C4—O4	1.193 (4)	O3—H3C	0.8601
C4—O3	1.312 (4)		
O2—C1—O1	126.3 (3)	O3—C4—C3	112.6 (3)
O2—C1—C2	115.3 (3)	C6—C5—N1	107.9 (4)
O1—C1—C2	118.3 (3)	C6—C5—H5	126.1
N1—C2—C3	111.3 (3)	N1—C5—H5	126.1
N1—C2—C1	111.4 (2)	C5—C6—N2	106.7 (3)
C3—C2—C1	110.1 (2)	C5—C6—H6	126.6
N1—C2—H2A	108.0	N2—C6—H6	126.6
C3—C2—H2A	108.0	N2—C7—N1	109.1 (3)
C1—C2—H2A	108.0	N2—C7—H7	125.4
C4—C3—C2	111.4 (3)	N1—C7—H7	125.4
C4—C3—H3A	109.3	C7—N1—C5	106.4 (3)
C2—C3—H3A	109.3	C7—N1—C2	126.5 (3)
C4—C3—H3B	109.3	C5—N1—C2	127.1 (3)
C2—C3—H3B	109.3	C7—N2—C6	109.9 (3)
H3A—C3—H3B	108.0	C7—N2—H2	125.1
O4—C4—O3	123.8 (3)	C6—N2—H2	125.1
O4—C4—C3	123.6 (3)	C4—O3—H3C	112.9

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.86	1.91	2.716 (4)	155
O3—H3*C*···O1^{ii}	0.86	1.71	2.572 (3)	177

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