Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Hydroxyanilinium 2-carboxyacetate

Ying-Chun Wang

College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China
Correspondence e-mail: chenxinyuanseu@yahoo.com.cn

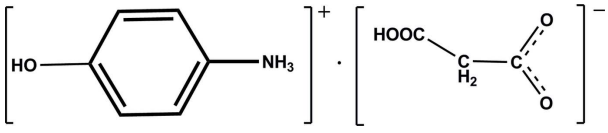
Received 29 April 2012; accepted 24 May 2012

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.133; data-to-parameter ratio = 14.9.

In the title compound, $C_6H_8NO^+\cdot C_3H_3O_4^-$, the amino N atom is protonated, and one of the carboxyl groups is deprotonated to maintain the charge balance. In the crystal, classical N—H···O and O—H···O hydrogen bonds connect the ions into a two-dimensional network parallel to the *ac* plane. In addition, the structure is further stabilized by C—H···O and π–π interactions [centroid–centroid distance = 4.115 (2) Å].

Related literature

For the structures and properties of related compounds, see: Chen *et al.* (2001); Wang *et al.* (2002); Xue *et al.* (2002); Huang *et al.* (1999); Zhang *et al.* (2001); Ye *et al.* (2008).



Experimental

Crystal data

$C_6H_8NO^+\cdot C_3H_3O_4^-$
M_r = 213.19
Monoclinic, $P2_1/c$
a = 5.1416 (1) Å
b = 22.5507 (7) Å
c = 7.8176 (3) Å
β = 97.827 (1)°
V = 897.98 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 173 K
0.10 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.910, T_{max} = 1.000
6380 measured reflections
2040 independent reflections
1611 reflections with $I > 2\sigma(I)$
R_{int} = 0.041

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.133
S = 1.07
2040 reflections
137 parameters
5 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O3^{i}	0.82	1.94	2.745 (2)	168
N1—H1A···O2^{ii}	0.89	2.10	2.989 (2)	177
N1—H1B···O4^{iii}	0.89	2.33	3.090 (2)	144
N1—H1C···O2^{iv}	0.89	1.98	2.836 (2)	160
O1—H1···O4	0.82	1.65	2.450 (2)	165
C3—H3A···O3^{iii}	0.93	2.47	3.398 (3)	175
C8—H8B···O1^{v}	0.97	2.31	3.163 (3)	147

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

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: *SHELXTL*.

This work was supported by a start-up grant from Southeast University, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2357).

References

Chen, Z.-F., Li, B.-Q., Xie, Y.-R., Xiong, R.-G., You, X.-Z. & Feng, X.-L. (2001). *Inorg. Chem. Commun.* **4**, 346–349.
Huang, S.-P.-D., Xiong, R.-G., Han, J.-D. & Weiner, B. R. (1999). *Inorg. Chim. Acta*, **294**, 95–98.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Wang, L.-Z., Wang, X.-S., Li, Y.-H., Bai, Z.-P., Xiong, R.-G., Xiong, M. & Li, G.-W. (2002). *Chin. J. Inorg. Chem.* **18**, 1191–1194.
Xue, X., Abrahams, B. F., Xiong, R.-G. & You, X.-Z. (2002). *Aust. J. Chem.* **55**, 495-497.
Ye, Q., Fu, D.-W., Hang, T., Xiong, R.-G., Chan, P. W. H. & Huang, S.-P.-D. (2008). *Inorg. Chem.* **47**, 772–774.
Zhang, J., Xiong, R.-G., Chen, X.-T., Che, C.-M., Xue, Z.-L. & You, X.-Z. (2001). *Organometallics*, **20**, 4118–4121.

supplementary materials

Acta Cryst. (2012). E**68**, o1937 [doi:10.1107/S160053681202363X]

4-Hydroxyanilinium 2-carboxyacetate

Ying-Chun Wang

Comment

Simple organic salts containing strong intrermolecular H-bonds have attracted an attention as materials which display ferroelectric-paraelectric phase transitions (Chen *et al.*, 2001; Huang *et al.*, 1999; Zhang *et al.*, 2001). With the purpose of obtaining phase transition crystals of organic salts, various organic molecules have been studied and a series of new crystal materials have been elaborated (Wang *et al.*, 2002; Xue *et al.*, 2002; Ye *et al.* ,2008). Herewith, we present the synthesis and crystal structure of the title compound, 4-hydroxyanilinium 2-carboxyacetate.

In the title compound (Fig. 1), the bond lengths and angles have normal values. The asymmetric unit was composed of one 4-hydroxyanilinium cation and one 2-carboxyacetate anion. The protonated N atom was involved in strong intramolecular N–H···O hydrogen bonds with the N···O distances of N1–H1A···O2^{ii} - 2.989 (2)Å; N1–H1B···O4^{iii} - 3.090 (2)Å and N1–H1C···O2^{iv} - 2.836 (2)Å. The N–H···O and O–H···O H-bonding interactions connected the ion units into a 2D network parallel to the *ac*-plane. The weak non-classical intermolecular C3–H3A···O3^{iii} and C8–H8B···O1^{v} interactions were presented in the crystal structure with C3···O3^{iii} = 3.398 (3)Å and C8···O1^{v} = 3.163 (3)Å, respectively. The crystal packing was further stabilized by aromatic π–π interactions between the benzene rings of the neighbouring cations with the Cg···Cg distances of 4.115 (2)Å (Cg is the centroide of the benzene ring) (Fig. 2 and Table 1). Symmetry codes: (ii) *x*+1, -*y*+1/2, *z*+1/2; (iii) *x*, -*y*+1/2, *z*-1/2; (iv) *x*, -*y*+1/2, *z*+1/2, (v) *x*-1, *y*, *z*.

Experimental

The malonic acid (10 mmol), 4-aminophenol (10 mmol) and ethanol (50 mL) were added into a 100 mL flask. The mixture was stirred at 333 K for 2 h, and then the precipitate was filtrated out. Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of the solution.

Refinement

All the H atoms attached to C atoms were placed into the idealized positions and treated as riding with C–H = 0.93Å (aromatic) and C–H = 0.97Å (methylene) with U_{iso}(H) = 1.2U_{eq}(C). The H atoms based on N and O were placed into the calculated positions with the H–N = 0.89Å and H–O = 0.82Å and refined with U_{iso}(H) = 1.5U_{eq}(N and O).

Computing details

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear* (Rigaku, 2005); data reduction: *CrystalClear* (Rigaku, 2005); 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: *SHELXTL* (Sheldrick, 2008).

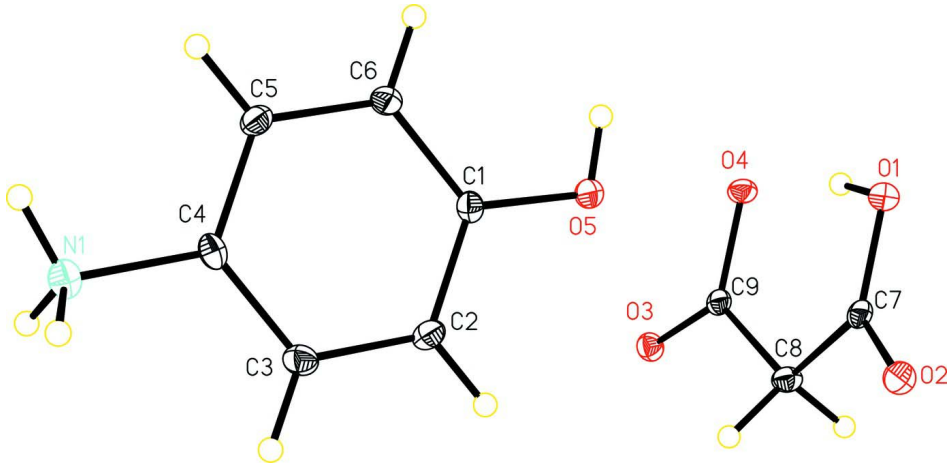


Figure 1

A view of the asymmetric unit with the atomic numbering scheme. The displacement ellipsoids were drawn at the 30% probability level. H atoms are presented as a small spheres of arbitrary radius.

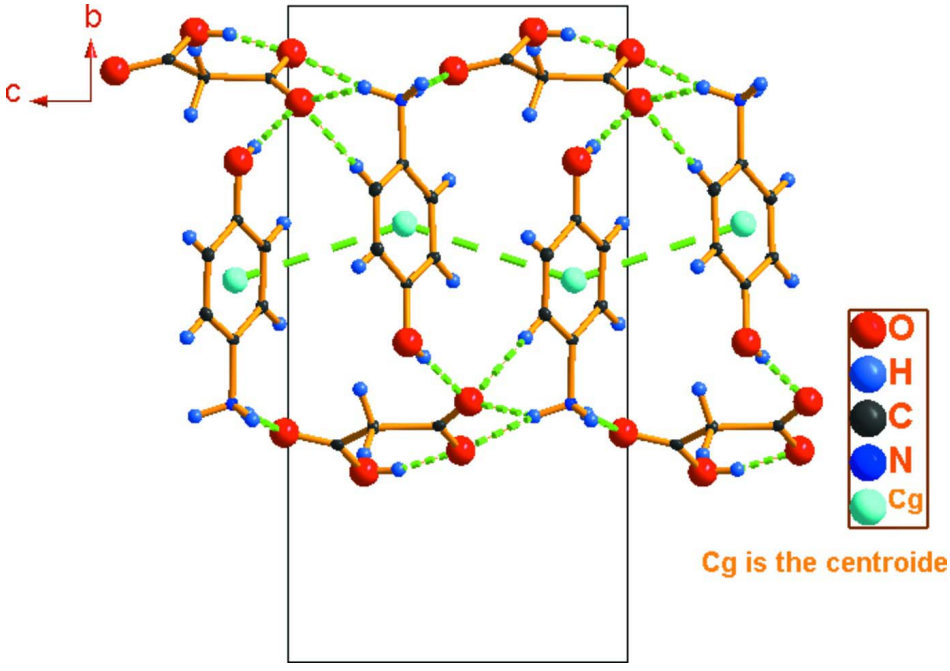


Figure 2

The crystal packing of the title compound viewed along the *a* axis showing the H-bonding and π–π interactions (dotted line).

4-Hydroxyanilinium 2-carboxyacetate

Crystal data

$C_6H_8NO^+·C_3H_3O_4^-$
M_r = 213.19
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 5.1416 (1) Å
b = 22.5507 (7) Å
c = 7.8176 (3) Å
β = 97.827 (1)°
V = 897.98 (5) Å^3
Z = 4

$F(000) = 448$
$D_x = 1.577$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2040 reflections
$\theta = 2.8–27.5°$
$\mu = 0.13$ mm^{-1}
$T = 173$ K
Block, colourless
0.10 × 0.05 × 0.05 mm

Data collection

Rigaku Mercury CCD 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)
$T_{min} = 0.910$, $T_{max} = 1.000$
6380 measured reflections
2040 independent reflections
1611 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.8°$
$h = -5\rightarrow6$
$k = -28\rightarrow27$
$l = -10\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.133$
$S = 1.07$
2040 reflections
137 parameters
5 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
$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2 + 0.3433P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.40$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
O5	0.4476 (3)	0.34202 (6)	0.35078 (19)	0.0191 (3)
H5	0.5845	0.3567	0.3982	0.029*
O3	−0.1396 (3)	0.40201 (6)	0.53276 (17)	0.0187 (3)
O2	0.1261 (3)	0.43371 (6)	−0.01270 (17)	0.0183 (3)
O1	0.3518 (3)	0.47345 (6)	0.22298 (18)	0.0184 (3)
H1	0.3277	0.4710	0.3244	0.028*
O4	0.2284 (3)	0.45183 (6)	0.50832 (17)	0.0182 (3)
C4	0.5512 (4)	0.16064 (8)	0.3365 (2)	0.0155 (4)
C3	0.3222 (4)	0.18648 (10)	0.2564 (3)	0.0193 (4)
H3A	0.1887	0.1630	0.1995	0.023*
N1	0.5772 (4)	0.09560 (7)	0.3326 (2)	0.0193 (4)
H1A	0.7383	0.0853	0.3796	0.029*

H1B	0.5489	0.0830	0.2237	0.029*
H1C	0.4601	0.0792	0.3921	0.029*
C8	−0.0615 (4)	0.42836 (9)	0.2494 (2)	0.0171 (4)
H8A	−0.1252	0.3894	0.2111	0.021*
H8B	−0.2060	0.4559	0.2224	0.021*
C9	0.0095 (4)	0.42644 (8)	0.4436 (2)	0.0152 (4)
C6	0.7210 (4)	0.25601 (9)	0.4274 (3)	0.0177 (4)
H6A	0.8545	0.2793	0.4849	0.021*
C1	0.4915 (4)	0.28237 (8)	0.3480 (2)	0.0151 (4)
C7	0.1530 (4)	0.44583 (9)	0.1445 (2)	0.0154 (4)
C5	0.7508 (4)	0.19480 (9)	0.4208 (3)	0.0179 (4)
H5A	0.9047	0.1770	0.4730	0.021*
C2	0.2936 (4)	0.24722 (9)	0.2616 (2)	0.0184 (4)
H2A	0.1409	0.2648	0.2068	0.022*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O5	0.0184 (7)	0.0138 (7)	0.0239 (7)	0.0005 (6)	−0.0019 (6)	−0.0006 (6)
O3	0.0210 (7)	0.0179 (8)	0.0173 (7)	−0.0017 (6)	0.0030 (6)	0.0005 (5)
O2	0.0196 (7)	0.0195 (8)	0.0159 (7)	−0.0006 (6)	0.0030 (5)	−0.0005 (5)
O1	0.0163 (7)	0.0186 (8)	0.0197 (7)	−0.0017 (6)	0.0003 (5)	0.0003 (6)
O4	0.0172 (7)	0.0171 (7)	0.0187 (7)	−0.0012 (6)	−0.0032 (5)	−0.0031 (6)
C4	0.0208 (10)	0.0125 (10)	0.0146 (9)	−0.0015 (8)	0.0072 (8)	−0.0004 (7)
C3	0.0204 (10)	0.0195 (11)	0.0170 (10)	−0.0026 (8)	−0.0006 (8)	−0.0034 (8)
N1	0.0257 (9)	0.0178 (9)	0.0150 (8)	−0.0002 (8)	0.0047 (7)	0.0004 (7)
C8	0.0152 (9)	0.0210 (11)	0.0146 (9)	−0.0018 (8)	0.0002 (7)	−0.0008 (8)
C9	0.0160 (10)	0.0111 (9)	0.0175 (9)	0.0026 (8)	−0.0006 (7)	−0.0013 (7)
C6	0.0173 (10)	0.0163 (10)	0.0185 (10)	−0.0016 (8)	−0.0012 (8)	−0.0023 (8)
C1	0.0192 (10)	0.0130 (10)	0.0131 (9)	0.0000 (8)	0.0024 (7)	0.0001 (7)
C7	0.0153 (9)	0.0115 (9)	0.0183 (9)	0.0035 (8)	−0.0010 (7)	0.0015 (7)
C5	0.0156 (9)	0.0200 (11)	0.0173 (9)	0.0018 (8)	−0.0005 (7)	0.0024 (8)
C2	0.0165 (9)	0.0206 (11)	0.0169 (10)	0.0023 (8)	−0.0016 (8)	0.0007 (8)

Geometric parameters (Å, º)

O5—C1	1.365 (2)	N1—H1B	0.8900
O5—H5	0.8200	N1—H1C	0.8900
O3—C9	1.234 (2)	C8—C7	1.513 (3)
O2—C7	1.248 (2)	C8—C9	1.513 (3)
O1—C7	1.280 (2)	C8—H8A	0.9700
O1—H1	0.8207	C8—H8B	0.9700
O4—C9	1.302 (2)	C6—C1	1.390 (3)
C4—C5	1.378 (3)	C6—C5	1.391 (3)
C4—C3	1.384 (3)	C6—H6A	0.9300
C4—N1	1.473 (2)	C1—C2	1.390 (3)
C3—C2	1.379 (3)	C5—H5A	0.9300
C3—H3A	0.9300	C2—H2A	0.9300
N1—H1A	0.8900		

C1—O5—H5	105.8	H8A—C8—H8B	107.2
C7—O1—H1	102.4	O3—C9—O4	123.22 (17)
C5—C4—C3	120.89 (18)	O3—C9—C8	119.73 (17)
C5—C4—N1	120.19 (18)	O4—C9—C8	117.06 (17)
C3—C4—N1	118.91 (17)	C1—C6—C5	119.96 (18)
C2—C3—C4	119.50 (18)	C1—C6—H6A	120.0
C2—C3—H3A	120.3	C5—C6—H6A	120.0
C4—C3—H3A	120.3	O5—C1—C6	123.16 (17)
C4—N1—H1A	109.5	O5—C1—C2	117.24 (17)
C4—N1—H1B	109.5	C6—C1—C2	119.59 (18)
H1A—N1—H1B	109.5	O2—C7—O1	123.55 (18)
C4—N1—H1C	109.5	O2—C7—C8	119.03 (17)
H1A—N1—H1C	109.5	O1—C7—C8	117.41 (17)
H1B—N1—H1C	109.5	C4—C5—C6	119.60 (18)
C7—C8—C9	117.20 (16)	C4—C5—H5A	120.2
C7—C8—H8A	108.0	C6—C5—H5A	120.2
C9—C8—H8A	108.0	C3—C2—C1	120.44 (18)
C7—C8—H8B	108.0	C3—C2—H2A	119.8
C9—C8—H8B	108.0	C1—C2—H2A	119.8
C5—C4—C3—C2	−0.2 (3)	C9—C8—C7—O1	−19.3 (3)
N1—C4—C3—C2	178.95 (17)	C3—C4—C5—C6	0.8 (3)
C7—C8—C9—O3	−166.29 (18)	N1—C4—C5—C6	−178.33 (17)
C7—C8—C9—O4	14.4 (3)	C1—C6—C5—C4	−0.5 (3)
C5—C6—C1—O5	179.28 (18)	C4—C3—C2—C1	−0.7 (3)
C5—C6—C1—C2	−0.3 (3)	O5—C1—C2—C3	−178.69 (18)
C9—C8—C7—O2	161.91 (18)	C6—C1—C2—C3	1.0 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O3^{i}	0.82	1.94	2.745 (2)	168
N1—H1*A*···O2^{ii}	0.89	2.10	2.989 (2)	177
N1—H1*B*···O4^{iii}	0.89	2.33	3.090 (2)	144
N1—H1*C*···O2^{iv}	0.89	1.98	2.836 (2)	160
O1—H1···O4	0.82	1.65	2.450 (2)	165
C3—H3*A*···O3^{iii}	0.93	2.47	3.398 (3)	175
C8—H8*B*···O1^{v}	0.97	2.31	3.163 (3)	147

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