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4-Hydroxyanilinium 2-chloroacetate

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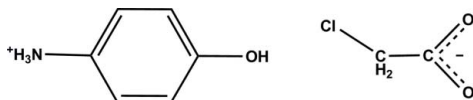
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 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.048; wR factor = 0.104; data-to-parameter ratio = 17.8.

In the crystal of the title salt, $\text{C}_6\text{H}_8\text{NO}^+\cdot\text{C}_2\text{H}_2\text{ClO}_2^-$, the 4-hydroxyanilinium cation links to adjacent chloroacetate anions via $\text{N}-\text{H}\cdots\text{O}$ and $\text{O}-\text{H}\cdots\text{O}$ hydrogen bonds; weak $\text{C}-\text{H}\cdots\text{O}$ interactions also occur between the anions and cations.

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

 $\text{C}_6\text{H}_8\text{NO}^+\cdot\text{C}_2\text{H}_2\text{ClO}_2^-$
 $M_r = 203.62$

 Monoclinic, $P2_1/c$
 $a = 10.702$ (3) Å

 $b = 4.5242$ (10) Å

 $c = 19.357$ (7) Å

 $\beta = 96.825$ (2)°

 $V = 930.6$ (5) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.38$ mm⁻¹
 $T = 173$ K

 $0.10 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2 diffractometer

Absorption correction: multi-scan

 (*CrystalClear*; Rigaku, 2005)

 $T_{\min} = 0.910$, $T_{\max} = 1.000$

6248 measured reflections

2122 independent reflections

 1699 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.045$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.104$
 $S = 1.09$

2122 reflections

119 parameters

4 restraints

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{N1}-\text{H1A}\cdots\text{O3}$ | 0.91 | 1.86 | 2.755 (3) | 166 |
| $\text{N1}-\text{H1B}\cdots\text{O2}^{\text{i}}$ | 0.91 | 1.87 | 2.777 (3) | 171 |
| $\text{N1}-\text{H1C}\cdots\text{O2}^{\text{ii}}$ | 0.91 | 1.90 | 2.762 (2) | 157 |
| $\text{O1}-\text{H1}\cdots\text{O3}^{\text{iii}}$ | 0.82 | 1.87 | 2.683 (2) | 172 |
| $\text{C8}-\text{H8A}\cdots\text{O1}^{\text{iv}}$ | 0.99 | 2.38 | 3.319 (3) | 159 |

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

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: XU5534).

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supplementary materials

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4-Hydroxyanilinium 2-chloroacetate

Ying-Chun Wang

Comment

Simple organic salts containing strong intramolecular 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-chloroacetate.

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-chloroacetate anion. The protonated N atom was involved in strong intramolecular N—H \cdots O hydrogen bonds with the N \cdots O distances of 2.777 (3)Å, 2.762 (2)Å and 2.755 (3)Å, respectively. The N—H \cdots O and O—H \cdots O H-bonding interactions connected the ion units into a 2D network parallel to the *ab*-plane. The weak intermolecular C8—H8A \cdots O1 interaction was presented in the crystal structure with C8 \cdots O1 = 3.319 (3)Å.

Experimental

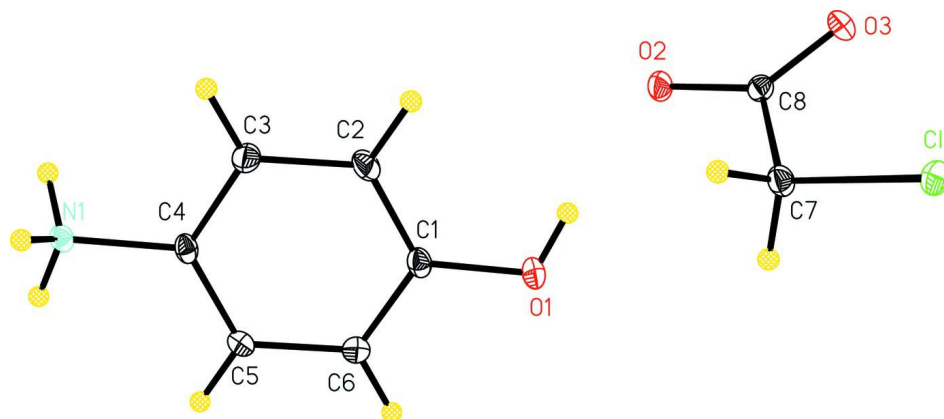
The 2-chloroacetic acid (10 mmol), 4-aminophenol (10 mmol) and ethanol (50 mL) were added into a 100 mL flask. The mixture was stirred at 333 K for 2 h, and then the precipitate was filtered out. Colourless crystals suitable for X-ray diffraction were obtained by slow evaporation of the solution.

Refinement

All the H atoms attached to C atoms were situated into the idealized positions and treated as riding with C—H = 0.95 Å (aromatic) and C—H = 0.99 Å (methylene) with $U_{iso}(H) = 1.2U_{eq}(C)$. The positional parameters of the H atoms bonded to N and O were located in a difference Fourier map and refined with restraints of H—N = 0.91 (2) and H—O = 0.82 (2) Å, $U_{iso}(H) = 1.5U_{eq}(N,O)$.

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.

4-Hydroxyanilinium 2-chloroacetate

Crystal data

$C_6H_8NO^+ \cdot C_2H_2ClO_2^-$

$M_r = 203.62$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 10.702\ (3)\ \text{\AA}$

$b = 4.5242\ (10)\ \text{\AA}$

$c = 19.357\ (7)\ \text{\AA}$

$\beta = 96.825\ (2)^\circ$

$V = 930.6\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 424$

$D_x = 1.453\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2122 reflections

$\theta = 2.7\text{--}27.5^\circ$

$\mu = 0.38\ \text{mm}^{-1}$

$T = 173\ \text{K}$

Block, colorless

$0.10 \times 0.05 \times 0.05\ \text{mm}$

Data collection

Rigaku Mercury2
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $13.6612\ \text{pixels mm}^{-1}$

CCD profile fitting scans

Absorption correction: multi-scan

(*CrystalClear*; Rigaku, 2005)

$T_{\min} = 0.910$, $T_{\max} = 1.000$

6248 measured reflections

2122 independent reflections

1699 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 2.7^\circ$

$h = -12 \rightarrow 13$

$k = -5 \rightarrow 5$

$l = -25 \rightarrow 23$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.048$

$wR(F^2) = 0.104$

$S = 1.09$

2122 reflections

119 parameters

4 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

$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 0.4P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.31\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.31\ \text{e \AA}^{-3}$

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|---------------|--------------|--------------|----------------------------------|
| C11 | 0.41622 (5) | 1.32273 (13) | 0.29181 (3) | 0.02016 (16) |
| O3 | 0.29262 (14) | 0.7514 (4) | 0.42037 (8) | 0.0222 (4) |
| O2 | 0.48355 (14) | 0.9420 (4) | 0.41518 (8) | 0.0188 (4) |
| N1 | 0.37140 (16) | 0.5601 (4) | 0.55354 (10) | 0.0163 (4) |
| H1A | 0.3555 | 0.6038 | 0.5074 | 0.024* |
| H1B | 0.4170 | 0.3902 | 0.5590 | 0.024* |
| H1C | 0.4158 | 0.7104 | 0.5760 | 0.024* |
| O1 | -0.08865 (14) | 0.4356 (4) | 0.66202 (9) | 0.0250 (4) |
| H1 | -0.1461 | 0.3684 | 0.6347 | 0.037* |
| C2 | 0.1616 (2) | 0.3348 (6) | 0.54972 (13) | 0.0223 (5) |
| H2A | 0.1781 | 0.2289 | 0.5094 | 0.027* |
| C7 | 0.3694 (2) | 0.9122 (5) | 0.39246 (11) | 0.0163 (5) |
| C1 | 0.2513 (2) | 0.5212 (5) | 0.58293 (12) | 0.0164 (5) |
| C6 | 0.2298 (2) | 0.6739 (6) | 0.64231 (12) | 0.0204 (5) |
| H6A | 0.2929 | 0.7995 | 0.6651 | 0.025* |
| C4 | 0.0232 (2) | 0.4565 (5) | 0.63473 (12) | 0.0184 (5) |
| C5 | 0.1148 (2) | 0.6413 (6) | 0.66814 (12) | 0.0235 (5) |
| H5A | 0.0989 | 0.7458 | 0.7088 | 0.028* |
| C3 | 0.0466 (2) | 0.3032 (6) | 0.57581 (13) | 0.0231 (5) |
| H3A | -0.0159 | 0.1760 | 0.5531 | 0.028* |
| C8 | 0.3133 (2) | 1.0670 (5) | 0.32664 (12) | 0.0191 (5) |
| H8A | 0.2366 | 1.1738 | 0.3364 | 0.023* |
| H8B | 0.2873 | 0.9156 | 0.2909 | 0.023* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| C11 | 0.0217 (3) | 0.0185 (3) | 0.0211 (3) | -0.0005 (2) | 0.0056 (2) | 0.0027 (2) |
| O3 | 0.0177 (8) | 0.0311 (10) | 0.0180 (8) | -0.0054 (7) | 0.0023 (7) | 0.0060 (7) |
| O2 | 0.0146 (8) | 0.0172 (8) | 0.0236 (9) | -0.0023 (7) | -0.0018 (6) | 0.0011 (7) |
| N1 | 0.0141 (9) | 0.0171 (10) | 0.0177 (10) | -0.0021 (8) | 0.0020 (7) | 0.0020 (8) |
| O1 | 0.0135 (8) | 0.0374 (11) | 0.0245 (9) | -0.0036 (8) | 0.0041 (7) | 0.0002 (8) |
| C2 | 0.0221 (12) | 0.0226 (12) | 0.0232 (12) | -0.0053 (11) | 0.0066 (10) | -0.0074 (10) |
| C7 | 0.0161 (11) | 0.0166 (11) | 0.0165 (11) | 0.0005 (9) | 0.0035 (9) | -0.0015 (9) |
| C1 | 0.0126 (10) | 0.0173 (12) | 0.0193 (12) | -0.0005 (9) | 0.0023 (9) | 0.0059 (9) |
| C6 | 0.0158 (11) | 0.0267 (13) | 0.0181 (11) | -0.0025 (10) | -0.0012 (9) | -0.0015 (10) |
| C4 | 0.0135 (10) | 0.0217 (12) | 0.0197 (12) | 0.0005 (9) | 0.0012 (9) | 0.0069 (10) |

| | | | | | | |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| C5 | 0.0191 (11) | 0.0338 (15) | 0.0175 (12) | -0.0009 (11) | 0.0014 (9) | -0.0015 (10) |
| C3 | 0.0183 (11) | 0.0225 (12) | 0.0285 (13) | -0.0089 (10) | 0.0027 (10) | -0.0057 (11) |
| C8 | 0.0169 (11) | 0.0197 (12) | 0.0205 (12) | -0.0036 (10) | 0.0012 (9) | 0.0031 (10) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-------------|-------------|--------------|-------------|
| C11—C8 | 1.784 (2) | C2—H2A | 0.9500 |
| O3—C7 | 1.265 (3) | C7—C8 | 1.514 (3) |
| O2—C7 | 1.256 (3) | C1—C6 | 1.384 (3) |
| N1—C1 | 1.477 (3) | C6—C5 | 1.390 (3) |
| N1—H1A | 0.9100 | C6—H6A | 0.9500 |
| N1—H1B | 0.9100 | C4—C3 | 1.383 (3) |
| N1—H1C | 0.9100 | C4—C5 | 1.389 (3) |
| O1—C4 | 1.368 (3) | C5—H5A | 0.9500 |
| O1—H1 | 0.8203 | C3—H3A | 0.9500 |
| C2—C1 | 1.378 (3) | C8—H8A | 0.9900 |
| C2—C3 | 1.392 (3) | C8—H8B | 0.9900 |
| C1—N1—H1A | 109.5 | C1—C6—H6A | 120.4 |
| C1—N1—H1B | 109.5 | C5—C6—H6A | 120.4 |
| H1A—N1—H1B | 109.5 | O1—C4—C3 | 122.4 (2) |
| C1—N1—H1C | 109.5 | O1—C4—C5 | 117.6 (2) |
| H1A—N1—H1C | 109.5 | C3—C4—C5 | 119.9 (2) |
| H1B—N1—H1C | 109.5 | C4—C5—C6 | 120.2 (2) |
| C4—O1—H1 | 113.6 | C4—C5—H5A | 119.9 |
| C1—C2—C3 | 119.4 (2) | C6—C5—H5A | 119.9 |
| C1—C2—H2A | 120.3 | C4—C3—C2 | 120.1 (2) |
| C3—C2—H2A | 120.3 | C4—C3—H3A | 119.9 |
| O2—C7—O3 | 124.5 (2) | C2—C3—H3A | 119.9 |
| O2—C7—C8 | 121.14 (19) | C7—C8—C11 | 114.65 (16) |
| O3—C7—C8 | 114.38 (19) | C7—C8—H8A | 108.6 |
| C2—C1—C6 | 121.2 (2) | C11—C8—H8A | 108.6 |
| C2—C1—N1 | 118.9 (2) | C7—C8—H8B | 108.6 |
| C6—C1—N1 | 119.9 (2) | C11—C8—H8B | 108.6 |
| C1—C6—C5 | 119.2 (2) | H8A—C8—H8B | 107.6 |
| C3—C2—C1—C6 | 1.0 (4) | C1—C6—C5—C4 | 0.3 (4) |
| C3—C2—C1—N1 | -178.1 (2) | O1—C4—C3—C2 | 179.3 (2) |
| C2—C1—C6—C5 | -1.0 (4) | C5—C4—C3—C2 | -0.3 (4) |
| N1—C1—C6—C5 | 178.2 (2) | C1—C2—C3—C4 | -0.4 (4) |
| O1—C4—C5—C6 | -179.3 (2) | O2—C7—C8—C11 | -7.3 (3) |
| C3—C4—C5—C6 | 0.3 (4) | O3—C7—C8—C11 | 173.97 (17) |

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|----------------------------------|-------|-------------|-------------|---------------|
| N1—H1A \cdots O3 | 0.91 | 1.86 | 2.755 (3) | 166 |
| N1—H1B \cdots O2 ⁱ | 0.91 | 1.87 | 2.777 (3) | 171 |
| N1—H1C \cdots O2 ⁱⁱ | 0.91 | 1.90 | 2.762 (2) | 157 |

| | | | | |
|---------------------------|------|------|-----------|-----|
| O1—H1...O3 ⁱⁱⁱ | 0.82 | 1.87 | 2.683 (2) | 172 |
| C8—H8A...O1 ^{iv} | 0.99 | 2.38 | 3.319 (3) | 159 |

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