2-Hydroxybenzenaminium acetate

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In the title molecular salt, C₆H₈NO⁺·C₂H₃O₂⁻, the cations and anions are linked by O—H⋯O and N—H⋯O hydrogen bonds, generating a three-dimensional network.

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Keywords: crystal structure; 2-hydroxybenzenaminium cation; acetate anion; hydrogen bond.

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Structural data: full structural data are available from iucrdata.iucr.org
OH bond length (C2—O1) of 1.3520 (9) Å is similar to that observed for structures containing 2-hydroxybenzenaminium as a cation [1.350 (3) Å; Jin & Wang, 2013]. All bond lengths and angles in the 2-hydroxybenzenaminium cation are within normal ranges (Zhao, 2012).

The presence of hydroxyl groups leads to the formation of intermolecular O1—H1···O3 hydrogen bonds. The O1—H1···O3 and N1—H1C···O3 cation–anion hydrogen bonds generate a succession of infinite chains [graph set C12(7)] that propagate in a zigzag manner along the c-axis direction (Fig. 2 and Table 1). The N1—H1A···O2 hydrogen bonds (Table 1) link the chains into corrugated layers parallel to the bc plane, which are formed by a succession of R2(22) rings (Fig. 2). N1—H1B···O2 hydrogen bonds lead to the formation of a three-dimensional network (Fig. 3). No significant π–π stacking interactions were observed, despite the presence of an aromatic ring in the cation.

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

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O1—H1···O3i | 0.894 (14) | 1.709 (14) | 2.6025 (9) | 177.8 (16) |
| N1—H1A···O2 | 0.930 (13) | 1.807 (13) | 2.7251 (9) | 168.7 (12) |
| N1—H1B···O2ii | 0.923 (12) | 1.891 (12) | 2.8019 (9) | 168.8 (11) |
| N1—H1C···O3iii | 0.935 (12) | 1.834 (12) | 2.7531 (8) | 167.2 (12) |
| C6—H6···O3iii | 0.95 | 2.55 | 3.2493 (11) | 131 |

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

### Table 2
Experimental details.

| Crystal data |
|--------------|
| Chemical formula | C6H8NO+·C2H3O2− |
| Mr          | 169.18 |
| Crystal system, space group | Monoclinic, P21/c |
| Temperature (K) | 100 |
| a, b, c (Å) | 9.9150 (2), 7.2523 (2), 11.9573 (3) |
| β (°)  | 98.558 (2) |
| V (Å³) | 850.23 (4) |
| Z      | 4 |
| Radiation type | Mo Kα |
| μ (mm⁻¹) | 0.10 |
| Crystal size (mm) | 0.10 x 0.10 x 0.08 |

| Data collection |
|----------------|
| Diffractometer | Oxford Diffraction Xcalibur |
| Absorption correction | Integration (ABSORB; DeTitta, 1985) |

| Refinement |
|------------|
| No. of reflections | 3105 |
| No. of parameters | 122 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρmax, Δρmin (e Å⁻³) | 0.49, −0.27 |

Computer programs: CrystAlis PRO (Rigaku OD, 2018), SHELXS (Sheldrick, 2008), SHELXL2018/3 (Sheldrick, 2015) and OLEX2 (Dolomanov et al., 2009).

OH bond length (C2—O1) of 1.3520 (9) Å is similar to that observed for structures containing 2-hydroxybenzenaminium as a cation [1.350 (3) Å; Jin & Wang, 2013]. All bond lengths and angles in the 2-hydroxybenzenaminium cation are within normal ranges (Zhao, 2012).

The presence of hydroxyl groups leads to the formation of intermolecular O1—H1···O3 hydrogen bonds. The O1—H1···O3 and N1—H1C···O3 cation–anion hydrogen bonds generate a succession of infinite chains [graph set C12(7)] that propagate in a zigzag manner along the c-axis direction (Fig. 2 and Table 1). The N1—H1A···O2 hydrogen bonds (Table 1) link the chains into corrugated layers parallel to the bc plane, which are formed by a succession of R2(22) rings (Fig. 2). N1—H1B···O2 hydrogen bonds lead to the formation of a three-dimensional network (Fig. 3). No significant π–π stacking interactions were observed, despite the presence of an aromatic ring in the cation.
Synthesis and crystallization

The title compound was prepared by heating of a mixture of 2-aminophenol (Alfa Aesar, purity 98%) and acetic acid. This mixture was obtained by dissolution and agitation under reflux for 3 h of 0.5 g of the 2-aminophenol and 0.27 g of acetic acid in a 1:1 stoichiometric ratio in a hot ethanolic solution (20 ml). After warming for a few minutes using a water bath, the solution was cooled and kept at room temperature. Within a few days, yellow needle-like crystals suitable for the X-ray analysis were obtained (yield 60%) by evaporation of the solution.

Refinement

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

Acknowledgements

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full crystallographic data

2-Hydroxybenzenamminium acetate

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2-Hydroxybenzenamminium acetate

Crystal data

\[ \text{C}_6\text{H}_8\text{NO}^+\cdot\text{C}_2\text{H}_3\text{O}_2^- \]

\[ M_r = 169.18 \]

Monoclinic, \( P2_1/c \)

\[ a = 9.9150 (2) \text{ Å} \]

\[ b = 7.2523 (2) \text{ Å} \]

\[ c = 11.9573 (3) \text{ Å} \]

\[ \beta = 98.558 (2)^\circ \]

\[ V = 850.23 (4) \text{ Å}^3 \]

\[ Z = 4 \]

\[ F(000) = 360 \]

\[ D_A = 1.322 \text{ Mg m}^{-3} \]

\[ \text{Mo } K\alpha \text{ radiation, } \lambda = 0.71073 \text{ Å} \]

\[ \theta = 3.3–33.0^\circ \]

\[ \mu = 0.10 \text{ mm}^{-1} \]

\[ T = 100 \text{ K} \]

Prism, yellow

\[ 0.1 \times 0.1 \times 0.08 \text{ mm} \]

Data collection

Oxford Diffraction Xcalibur Sapphire2 CCD diffractometer

\[ \varphi \text{ and } \omega \text{ scans} \]

Absorption correction: integration

(ABSORB; DeTitta, 1985)

\[ T_{\text{min}} = 0.966, T_{\text{max}} = 0.991 \]

52913 measured reflections

3105 independent reflections

2736 reflections with \( I > 2 \sigma(I) \)

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\[ R[F^2 > 2 \sigma(F^2)] = 0.038 \]

\[ wR(F^2) = 0.109 \]

\[ S = 1.05 \]

3105 reflections

122 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: mixed

H atoms treated by a mixture of independent and constrained refinement

\[ w = 1/[\sigma^2(F_c^2) + (0.0542P)^2 + 0.320P] \]

where \( P = (F_c^2 + 2F_s^2)/3 \)

\( (\Delta / \sigma)_{\text{max}} = 0.001 \)

\[ \Delta \rho_{\text{max}} = 0.49 \text{ e Å}^{-3} \]

\[ \Delta \rho_{\text{min}} = -0.27 \text{ e Å}^{-3} \]

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. The hydrogen atoms of the NH\(_3\) and hydroxyl groups were localized in the difference-Fourier map and refined with \( U_{\text{iso}}(\text{H}) \) set to 1.5\( U_{\text{eq}}(\text{O}) \) or 1.2\( U_{\text{eq}}(\text{N}) \). All the other hydrogen atoms were placed in calculated positions with \( C-\text{H} = 0.95 \text{ Å} \) for aromatic CH and \( C-\text{H} = 0.96 \text{ Å} \) for CH\(_3\), and refined using a riding model with fixed isotropic displacement parameters \( [U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C-aromatic}) \text{ and } U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C-methyl})] \).
Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

|      |   x    |   y    |   z    | Uiso* (Å²) |
|------|--------|--------|--------|------------|
| O1   | 0.12872 (6) | 0.83288 (9) | 0.65994 (5) | 0.01629 (13) |
| H1   | 0.1381 (14) | 0.928 (2) | 0.6142 (12) | 0.024*       |
| O2   | 0.09592 (7) | 0.29888 (8) | 0.65411 (5) | 0.01686 (13) |
| O3   | 0.15033 (7) | 0.10731 (8) | 0.52349 (5) | 0.01664 (13) |
| N1   | 0.12133 (7) | 0.56570 (9) | 0.81635 (5) | 0.01242 (13) |
| H1A  | 0.1133 (12) | 0.4870 (19) | 0.7543 (10) | 0.015*       |
| H1B  | 0.0461 (12) | 0.6407 (18) | 0.8160 (10) | 0.015*       |
| H1C  | 0.1280 (12) | 0.4909 (18) | 0.8806 (10) | 0.015*       |
| C3   | 0.35594 (9) | 0.93249 (12) | 0.74235 (7) | 0.01774 (16) |
| H3   | 0.358215 | 1.025256 | 0.686567 | 0.021*       |
| C5   | 0.46510 (9) | 0.77468 (13) | 0.90970 (8) | 0.01994 (17) |
| H5   | 0.540747 | 0.760726 | 0.967953 | 0.024*       |
| C4   | 0.46648 (9) | 0.91021 (13) | 0.82754 (8) | 0.02042 (17) |
| H4   | 0.543738 | 0.988327 | 0.829677 | 0.025*       |
| C2   | 0.24139 (8) | 0.81883 (11) | 0.73847 (6) | 0.01328 (14) |
| C6   | 0.35205 (8) | 0.65957 (12) | 0.90596 (7) | 0.01606 (15) |
| H6   | 0.350402 | 0.566114 | 0.961414 | 0.019*       |
| C1   | 0.24197 (8) | 0.68208 (10) | 0.82091 (6) | 0.01217 (14) |
| C8   | 0.13683 (8) | 0.26870 (11) | 0.56096 (6) | 0.01288 (14) |
| C7   | 0.16938 (9) | 0.42886 (12) | 0.48900 (8) | 0.01940 (16) |

Atomic displacement parameters (Å²)

|      | U¹¹ | U¹² | U¹³ | U²² | U²³ | U³³ |
|------|-----|-----|-----|-----|-----|-----|
| O1   | 0.0172 (3) | 0.0157 (3) | 0.0153 (3) | −0.0014 (2) | 0.0001 (2) | 0.0046 (2) |
| O2   | 0.0220 (3) | 0.0142 (3) | 0.0157 (3) | −0.0024 (2) | 0.0073 (2) | −0.0030 (2) |
| O3   | 0.0255 (3) | 0.0124 (3) | 0.0124 (2) | 0.0019 (2) | 0.0041 (2) | −0.00051 (19) |
| N1   | 0.0147 (3) | 0.0109 (3) | 0.0120 (3) | −0.0006 (2) | 0.0030 (2) | 0.0005 (2) |
| C3   | 0.0180 (3) | 0.0161 (3) | 0.0198 (4) | −0.0030 (3) | 0.0053 (3) | 0.0024 (3) |
| C5   | 0.0150 (3) | 0.0222 (4) | 0.0217 (4) | −0.0007 (3) | −0.0003 (3) | 0.0001 (3) |
| C4   | 0.0154 (3) | 0.0212 (4) | 0.0248 (4) | −0.0040 (3) | 0.0036 (3) | 0.0000 (3) |
| C2   | 0.0149 (3) | 0.0121 (3) | 0.0131 (3) | 0.0002 (2) | 0.0032 (2) | 0.0005 (2) |
| C6   | 0.0162 (3) | 0.0160 (3) | 0.0157 (3) | 0.0013 (3) | 0.0015 (3) | 0.0011 (3) |
| C1   | 0.0133 (3) | 0.0110 (3) | 0.0126 (3) | −0.0001 (2) | 0.0033 (2) | 0.0000 (2) |
| C8   | 0.0128 (3) | 0.0123 (3) | 0.0135 (3) | 0.0001 (2) | 0.0019 (2) | 0.0007 (2) |
| C7   | 0.0213 (4) | 0.0154 (3) | 0.0229 (4) | 0.0008 (3) | 0.0078 (3) | 0.0063 (3) |

Geometric parameters (Å, °)

|      | C5—H5 | C5—C4 | C5—C6 | 0.9500 |
|------|-------|-------|-------|--------|
| O1—H1 | 0.892 (14) | 1.3520 (9) | 1.2600 (9) | 1.3912 (13) |
| O1—C2 | 1.3520 (9) | 1.3912 (13) | 1.3930 (12) | 1.3930 (12) |
O3—C8  1.2675 (9)  C4—H4  0.9500
N1—H1A  0.930 (13)  C2—C1  1.3978 (11)
N1—H1B  0.922 (13)  C6—H6  0.9500
N1—H1C  0.935 (12)  C6—C1  1.3863 (11)
N1—C1  1.4583 (10)  C8—C7  1.5087 (11)
C3—H3  0.9500  C7—H7A  0.9800
C3—C4  1.3903 (12)  C7—H7B  0.9800
C3—C2  1.3985 (11)  C7—H7C  0.9800

C2—O1—H1  109.4 (9)  O1—C2—C1  117.36 (7)
H1A—N1—H1B  112.7 (11)  C1—C2—C3  118.48 (7)
H1A—N1—H1C  106.7 (11)  C5—C6—H6  120.2
H1B—N1—H1C  107.7 (10)  C1—C6—C5  119.66 (8)
C1—N1—H1A  110.9 (8)  C1—C6—H6  120.2
C1—N1—H1B  108.5 (8)  C2—C1—N1  117.84 (7)
C1—N1—H1C  110.3 (7)  C6—C1—N1  120.74 (7)
C4—C3—H3  119.9  C1—C2—C3  118.84 (7)
C4—C3—C2  120.24 (8)  C5—C6—C1  119.48 (7)
C2—C3—H3  119.9  C6—C1—C2  121.40 (7)
C4—C5—H5  120.2  C2—C3—C4  122.53 (7)
C4—C5—C6  119.55 (8)  O2—C8—O3  117.81 (7)
C6—C5—H5  120.2  C8—C7—H7A  109.5
C3—C4—H4  119.7  C8—C7—H7B  109.5
C3—C4—C5  120.66 (8)  C8—C7—H7C  109.5
C5—C4—H4  119.7  O2—C8—O3  117.81 (7)
C5—C4—C3  124.17 (7)  O3—C8—O3  117.81 (7)
O1—C2—C1—N1  −0.22 (10)  C4—C3—C2—O1  178.84 (8)
O1—C2—C1—C6  −178.85 (7)  C4—C3—C2—C1  −0.83 (12)
C3—C2—C1—N1  179.48 (7)  C4—C5—C6—C1  −0.39 (13)
C3—C2—C1—C6  0.85 (12)  C2—C3—C4—C5  0.22 (13)
C5—C6—C1—C2  −178.83 (7)  C6—C5—C4—C3  0.40 (14)
C5—C6—C1—C2  −0.24 (12)

Hydrogen-bond geometry (Å, °)

| D—H···A  | D—H | H···A  | D···A  | D—H···A  |
|----------|------|--------|--------|----------|
| O1—H1···O3 | 0.894 (14) | 1.709 (14) | 2.6025 (9) | 177.8 (16) |
| N1—H1A···O2 | 0.930 (13) | 1.807 (13) | 2.7251 (9) | 168.7 (12) |
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| N1—H1C···O3iii | 0.935 (12) | 1.834 (12) | 2.7531 (8) | 167.2 (12) |
| C6—H6···O3iii | 0.95 | 2.55 | 3.2493 (11) | 131 |

Symmetry codes: (i) x, y+1, z; (ii) −x, y+1/2, −z+3/2; (iii) x, −y+1/2, z+1/2.