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Crystal structure of 2,5-dimethylanilinium salicylate

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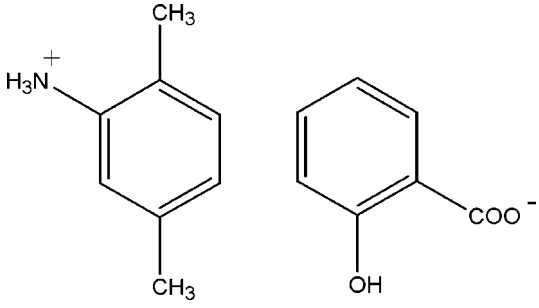
The title molecular salt, $C_8H_{12}N^+\cdot C_7H_5O_3^-$ arose from the proton-transfer reaction between 2,5-xylidine and salicylic acid. In the anion, the dihedral angle between the planes of the aromatic ring and the $-CO_2^-$ group is 11.08 (8)°; this near planarity is consolidated by an intramolecular O—H···O hydrogen bond. In the crystal, the components are connected by N—H···O hydrogen bonds, with all three O atoms in the anion acting as acceptors; the result is a [100] chain. The structure also features weak C—H···O bonds and aromatic π–π stacking [centroid-to-centroid distance = 3.7416 (10) Å] interactions, which lead to a three-dimensional network.

Keywords: crystal structure; hydrogen bonding; aromatic π–π stacking interactions.

CCDC reference: 1415922

1. Related literature

For related structures, see: Fun *et al.* (2011); Mathlouthi *et al.* (2014); Smirani & Rzaigui (2009).



2. Experimental

2.1. Crystal data

$C_8H_{12}N^+\cdot C_7H_5O_3^-$
M_r = 259.30
Monoclinic, $P2_1/c$
a = 6.9645 (5) Å
b = 20.6924 (14) Å
c = 9.2920 (7) Å
β = 95.738 (3)°
V = 1332.38 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 295 K
0.26 × 0.24 × 0.20 mm

2.2. Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.977, T_{max} = 0.982
17007 measured reflections
3384 independent reflections
2339 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.134
S = 1.01
3384 reflections
178 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···O2	0.83 (1)	1.77 (1)	2.5282 (15)	151 (2)
N1—H1A···O1^{i}	0.89	1.80	2.6809 (17)	169
N1—H1B···O2^{ii}	0.89	1.92	2.7998 (16)	168
N1—H1C···O3^{iii}	0.89	2.08	2.9654 (17)	171
C5—H5···O1^{iv}	0.93	2.58	3.237 (2)	128

Symmetry codes: (i) $x-1, -y+\frac{1}{2}, z-\frac{1}{2}$; (ii) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$; (iv) $x-1, y, z$.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7474).

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supporting information

Acta Cryst. (2015). E**71**, o643–o644 [https://doi.org/10.1107/S2056989015014401]

Crystal structure of 2,5-dimethylanilinium salicylate

A. Mani, P. Praveen Kumar and G. Chakkaravarthi

S1. Structural commentary

We herewith report the crystal structure of the title compound, (I), (Fig. 1). The geometric parameters of the title compound (I) (Fig. 1) are comparable with the reported structures [Fun *et al.*, 2011; Mathlouthi *et al.*, 2014; Smirani & Rzaigui,(2009)].

The conformation of the anion is stabilized by a weak O—H···O (Table 1) hydrogen bond. In the crystal structure, the adjacent anions and cations are linked by medium-strength N—H···O (Table 1) hydrogen bonds which link the anions and cations into infinite chain along [100] and these chains are further influenced by C—H···O hydrogen bond (Table 1 & Fig. 2) and π–π [Cg2···Cg2^{i} distance 3.7416 (10)Å; (i) 1-x, -y,1-z; Cg2 is the centroid of (C1—C6) ring] interactions to form a three dimensional network.

S2. Synthesis and crystallization

A mixture of 2,5-xylidine and salicylic acid dissolved in ethanol (molar ratio 1:1:1) was stirred for 3 h and then kept at room temperature. The saturated solution was allowed to evaporating slowly at room temperature. After the evaporation period of three weeks, colourless blocks were recovered.

S3. Refinement

H atoms were positioned geometrically and refined using riding model with C—H = 0.93 Å and Uiso(H) = 1.2Ueq(C) for CH, N—H = 0.89Å and Uiso(H) = 1.5Ueq(N) for NH_3, C—H = 0.96Å and Uiso(H) = 1.5Ueq(C) for CH_3. H atom for hydroxyl group was fixed from Fourier map and refined with Uiso(H) = 1.5Ueq(O) and O—H distance was restraint to 0.82 (1)Å.

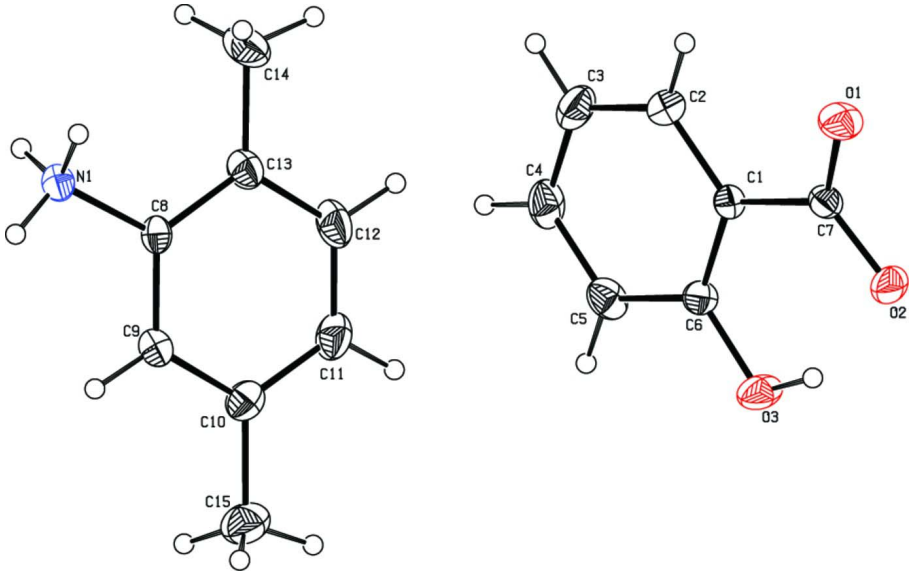

Figure 1

The molecular structure of (I), with 30% probability displacement ellipsoids for non-H atoms.

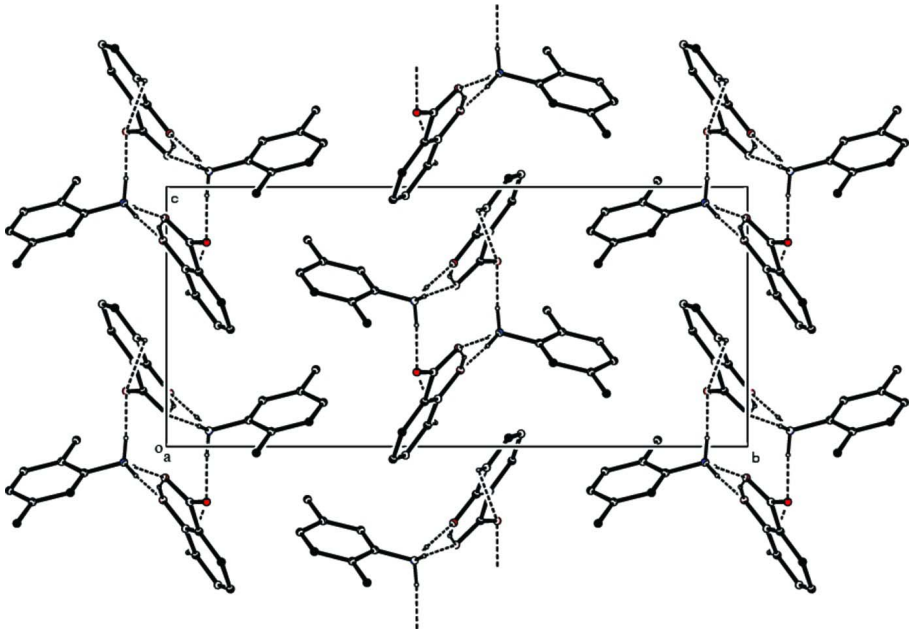

Figure 2

The packing of (I), viewed down *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

2,5-Dimethylanilinium 2-hydroxybenzoate

Crystal data

$C_8H_{12}N^+·C_7H_5O_3^-$
M_r = 259.30
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc

a = 6.9645 (5) Å
b = 20.6924 (14) Å
c = 9.2920 (7) Å
β = 95.738 (3)°

V = 1332.38 (17) Å^3
Z = 4
F(000) = 552
D_x = 1.293 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4550 reflections

θ = 2.4–28.1°
μ = 0.09 mm^{-1}
T = 295 K
Block, colourless
0.26 × 0.24 × 0.20 mm

Data collection

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ω and φ scan
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.977, T_{max} = 0.982

17007 measured reflections
3384 independent reflections
2339 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 28.7°, θ_{min} = 2.0°
h = −8→9
k = −27→27
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.134
S = 1.01
3384 reflections
178 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.3396P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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 > 2sigma(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}
C1	0.52627 (19)	0.05604 (7)	0.67259 (15)	0.0271 (3)
C2	0.5588 (2)	0.09450 (8)	0.55505 (17)	0.0371 (4)
H2	0.6825	0.1100	0.5463	0.045*
C3	0.4109 (3)	0.11016 (9)	0.45114 (18)	0.0474 (4)
H3	0.4352	0.1350	0.3715	0.057*
C4	0.2267 (3)	0.08870 (9)	0.46651 (18)	0.0467 (4)
H4	0.1260	0.1002	0.3979	0.056*
C5	0.1897 (2)	0.05073 (9)	0.58152 (18)	0.0405 (4)
H5	0.0646	0.0369	0.5911	0.049*
C6	0.3394 (2)	0.03300 (7)	0.68349 (16)	0.0303 (3)

C7	0.6859 (2)	0.04029 (7)	0.78590 (16)	0.0303 (3)
C8	0.01106 (19)	0.35845 (7)	0.60141 (15)	0.0281 (3)
C9	−0.1341 (2)	0.33448 (7)	0.67764 (16)	0.0326 (3)
H9	−0.2289	0.3622	0.7052	0.039*
C10	−0.1397 (2)	0.26951 (8)	0.71351 (18)	0.0384 (4)
C11	0.0036 (3)	0.23006 (8)	0.6699 (2)	0.0471 (4)
H11	0.0023	0.1862	0.6914	0.057*
C12	0.1487 (3)	0.25462 (8)	0.5951 (2)	0.0484 (5)
H12	0.2440	0.2268	0.5686	0.058*
C13	0.1568 (2)	0.31966 (8)	0.55812 (18)	0.0367 (4)
C14	0.3146 (3)	0.34526 (9)	0.4753 (2)	0.0562 (5)
H14A	0.2589	0.3651	0.3874	0.084*
H14B	0.3971	0.3104	0.4524	0.084*
H14C	0.3886	0.3767	0.5329	0.084*
C15	−0.2965 (3)	0.24282 (9)	0.7963 (2)	0.0571 (5)
H15A	−0.2783	0.1971	0.8094	0.086*
H15B	−0.4198	0.2507	0.7433	0.086*
H15C	−0.2917	0.2635	0.8890	0.086*
N1	0.00494 (17)	0.42673 (5)	0.56098 (14)	0.0306 (3)
H1A	−0.0346	0.4304	0.4673	0.046*
H1B	0.1223	0.4438	0.5785	0.046*
H1C	−0.0766	0.4475	0.6126	0.046*
O1	0.84427 (15)	0.06900 (6)	0.78665 (13)	0.0441 (3)
O2	0.65428 (15)	−0.00223 (6)	0.87917 (13)	0.0431 (3)
O3	0.29811 (16)	−0.00571 (6)	0.79399 (13)	0.0469 (3)
H3A	0.402 (2)	−0.0124 (11)	0.844 (2)	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0274 (7)	0.0264 (7)	0.0274 (7)	0.0019 (5)	0.0017 (6)	−0.0008 (5)
C2	0.0388 (8)	0.0367 (8)	0.0365 (8)	−0.0016 (7)	0.0067 (7)	0.0049 (7)
C3	0.0619 (12)	0.0442 (10)	0.0355 (9)	0.0061 (8)	0.0017 (8)	0.0117 (7)
C4	0.0468 (10)	0.0568 (11)	0.0337 (9)	0.0155 (8)	−0.0099 (7)	0.0011 (8)
C5	0.0291 (8)	0.0546 (10)	0.0365 (9)	0.0022 (7)	−0.0037 (7)	−0.0051 (7)
C6	0.0287 (7)	0.0336 (7)	0.0284 (7)	−0.0007 (6)	0.0021 (6)	0.0000 (6)
C7	0.0257 (7)	0.0345 (8)	0.0308 (7)	0.0014 (6)	0.0036 (6)	0.0004 (6)
C8	0.0281 (7)	0.0252 (7)	0.0296 (7)	0.0024 (5)	−0.0032 (6)	−0.0023 (6)
C9	0.0293 (7)	0.0318 (7)	0.0365 (8)	0.0021 (6)	0.0017 (6)	−0.0035 (6)
C10	0.0423 (9)	0.0350 (8)	0.0371 (8)	−0.0039 (7)	0.0005 (7)	0.0021 (7)
C11	0.0547 (11)	0.0282 (8)	0.0579 (11)	0.0044 (7)	0.0025 (9)	0.0050 (8)
C12	0.0450 (10)	0.0346 (9)	0.0659 (12)	0.0127 (7)	0.0066 (9)	−0.0058 (8)
C13	0.0338 (8)	0.0335 (8)	0.0431 (9)	0.0033 (6)	0.0050 (7)	−0.0046 (7)
C14	0.0482 (11)	0.0482 (10)	0.0763 (14)	0.0038 (8)	0.0272 (10)	−0.0080 (10)
C15	0.0622 (12)	0.0484 (11)	0.0629 (12)	−0.0095 (9)	0.0165 (10)	0.0084 (9)
N1	0.0274 (6)	0.0268 (6)	0.0370 (7)	0.0000 (5)	0.0003 (5)	−0.0015 (5)
O1	0.0278 (6)	0.0586 (7)	0.0452 (7)	−0.0091 (5)	−0.0002 (5)	0.0057 (6)
O2	0.0305 (6)	0.0545 (7)	0.0429 (6)	−0.0005 (5)	−0.0028 (5)	0.0203 (5)

O3	0.0300 (6)	0.0659 (8)	0.0436 (7)	−0.0128 (5)	−0.0013 (5)	0.0194 (6)

Geometric parameters (Å, º)

C1—C2	1.388 (2)	C9—H9	0.9300
C1—C6	1.399 (2)	C10—C11	1.381 (2)
C1—C7	1.4886 (19)	C10—C15	1.502 (3)
C2—C3	1.378 (2)	C11—C12	1.379 (3)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.379 (3)	C12—C13	1.392 (2)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.371 (2)	C13—C14	1.500 (2)
C4—H4	0.9300	C14—H14A	0.9600
C5—C6	1.386 (2)	C14—H14B	0.9600
C5—H5	0.9300	C14—H14C	0.9600
C6—O3	1.3556 (18)	C15—H15A	0.9600
C7—O1	1.2524 (17)	C15—H15B	0.9600
C7—O2	1.2696 (18)	C15—H15C	0.9600
C8—C9	1.383 (2)	N1—H1A	0.8900
C8—C13	1.385 (2)	N1—H1B	0.8900
C8—N1	1.4615 (18)	N1—H1C	0.8900
C9—C10	1.387 (2)	O3—H3A	0.829 (10)
C2—C1—C6	118.59 (13)	C9—C10—C15	121.27 (15)
C2—C1—C7	120.85 (13)	C12—C11—C10	121.15 (15)
C6—C1—C7	120.55 (13)	C12—C11—H11	119.4
C3—C2—C1	121.10 (15)	C10—C11—H11	119.4
C3—C2—H2	119.4	C11—C12—C13	122.07 (15)
C1—C2—H2	119.4	C11—C12—H12	119.0
C2—C3—C4	119.33 (16)	C13—C12—H12	119.0
C2—C3—H3	120.3	C8—C13—C12	116.06 (15)
C4—C3—H3	120.3	C8—C13—C14	122.70 (14)
C5—C4—C3	120.96 (15)	C12—C13—C14	121.23 (15)
C5—C4—H4	119.5	C13—C14—H14A	109.5
C3—C4—H4	119.5	C13—C14—H14B	109.5
C4—C5—C6	119.82 (15)	H14A—C14—H14B	109.5
C4—C5—H5	120.1	C13—C14—H14C	109.5
C6—C5—H5	120.1	H14A—C14—H14C	109.5
O3—C6—C5	118.17 (13)	H14B—C14—H14C	109.5
O3—C6—C1	121.70 (12)	C10—C15—H15A	109.5
C5—C6—C1	120.12 (14)	C10—C15—H15B	109.5
O1—C7—O2	122.51 (13)	H15A—C15—H15B	109.5
O1—C7—C1	119.68 (13)	C10—C15—H15C	109.5
O2—C7—C1	117.81 (12)	H15A—C15—H15C	109.5
C9—C8—C13	122.38 (14)	H15B—C15—H15C	109.5
C9—C8—N1	118.31 (12)	C8—N1—H1A	109.5
C13—C8—N1	119.27 (13)	C8—N1—H1B	109.5
C8—C9—C10	120.71 (14)	H1A—N1—H1B	109.5

C8—C9—H9	119.6	C8—N1—H1C	109.5
C10—C9—H9	119.6	H1A—N1—H1C	109.5
C11—C10—C9	117.62 (15)	H1B—N1—H1C	109.5
C11—C10—C15	121.10 (15)	C6—O3—H3A	106.5 (16)
C6—C1—C2—C3	0.3 (2)	C6—C1—C7—O2	10.7 (2)
C7—C1—C2—C3	−178.85 (15)	C13—C8—C9—C10	−0.4 (2)
C1—C2—C3—C4	1.8 (3)	N1—C8—C9—C10	177.30 (13)
C2—C3—C4—C5	−1.7 (3)	C8—C9—C10—C11	−0.2 (2)
C3—C4—C5—C6	−0.6 (3)	C8—C9—C10—C15	−179.99 (15)
C4—C5—C6—O3	−178.60 (15)	C9—C10—C11—C12	0.9 (3)
C4—C5—C6—C1	2.7 (2)	C15—C10—C11—C12	−179.39 (17)
C2—C1—C6—O3	178.81 (14)	C10—C11—C12—C13	−0.9 (3)
C7—C1—C6—O3	−2.0 (2)	C9—C8—C13—C12	0.4 (2)
C2—C1—C6—C5	−2.6 (2)	N1—C8—C13—C12	−177.30 (14)
C7—C1—C6—C5	176.58 (14)	C9—C8—C13—C14	179.91 (15)
C2—C1—C7—O1	10.0 (2)	N1—C8—C13—C14	2.3 (2)
C6—C1—C7—O1	−169.13 (14)	C11—C12—C13—C8	0.3 (3)
C2—C1—C7—O2	−170.18 (14)	C11—C12—C13—C14	−179.29 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3*A*···O2	0.83 (1)	1.77 (1)	2.5282 (15)	151 (2)
N1—H1*A*···O1^{i}	0.89	1.80	2.6809 (17)	169
N1—H1*B*···O2^{ii}	0.89	1.92	2.7998 (16)	168
N1—H1*C*···O3^{iii}	0.89	2.08	2.9654 (17)	171
C5—H5···O1^{iv}	0.93	2.58	3.237 (2)	128

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