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2-(2-Hydroxy-5-methylbenzylideneammonio)-benzoate

D. Gayathri,[a] D. Velmurugan,[a]* K. Ravikumar,[b] S. Devaraj[c] and M. Kandaswamy[c]

[a]Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, [b]Laboratory of X-ray Crystallography, Indian Institute of Chemical Technology, Hyderabad 500 007, India, and [c]Department of Inorganic Chemistry, University of Madras, Guindy Campus, Chennai 600 025, India

Correspondence e-mail: d_velu@yahoo.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.053
wR factor = 0.157
Data-to-parameter ratio = 16.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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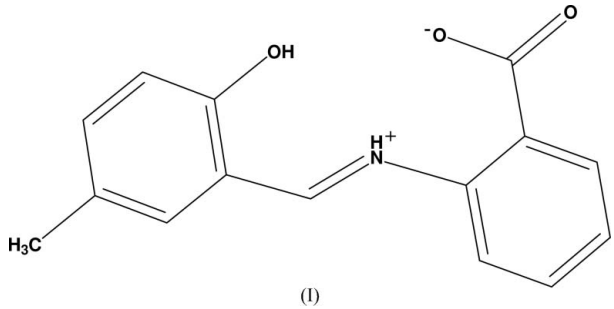
The N atom in the title compound, $C_{15}H_{13}NO_3$, is protonated by the –COOH group. The molecular conformation is stabilized by strong N—H···O hydrogen bonds. The crystal packing is stabilized by O—H···O and C—H···O intermolecular interactions.

Comment

The recognition (Schmidtchen & Berger, 1997) and sensing (Czanik, 1994) of anionic analytes have been considered as a key research theme within the generalized area of supramolecular chemistry. A significant amount of work has been devoted to obtain specific chemosensors that are able to change one or several macroscopic properties, upon complexation with the target guests. In response to the molecular recognition event, changes in colour or optical properties [fluorescence (Bargossi *et al.*, 2000) and absorbance (Löhr & Vögtle, 1985)] are the output signals used in the development of optical chemosensors.

The bond lengths and bond angles of the title compound, (I), are comparable to the literature values (Allen *et al.*, 1987). The N atom is protonated by the –COOH group. The dihedral angle between the two benzene rings in the structure is 5.6 (1)°.



(I)

The molecular structure is stabilized by strong N—H···O interactions in which N1 acts as a bifurcated donor to O1 and O2, generating two *S*(6) motifs. The crystal packing is stabilized by O—H···O and C—H···O intermolecular interactions. Atom O1 acts as donor to atom O2 at $(-x, y, \frac{3}{2} - z)$, generating a dimer of the type $R_2^2(20)$. The packing of molecules in the unit cell viewed down the *a* axis resembles the shape of a wave.

Experimental

2-(2-Hydroxy-5-methylbenzylideneammonio)benzoate was synthesized by Schiff base condensation between 2-aminobenzoic acid and

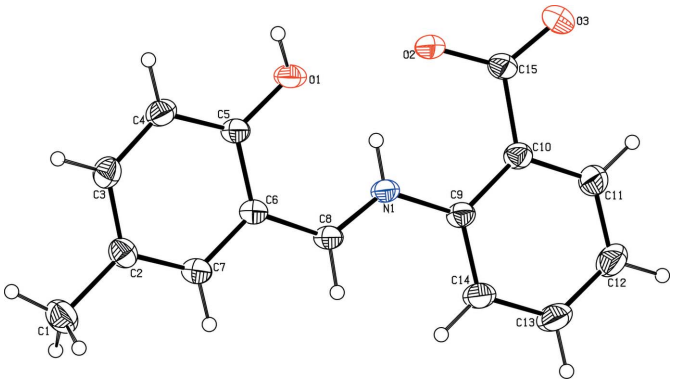


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids.

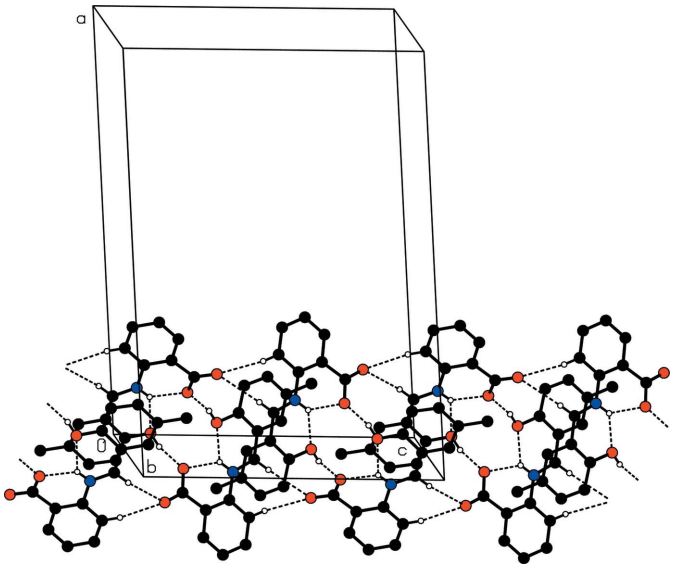


Figure 2
A view of the N—H···O intramolecular and O—H···O and C—H···O intermolecular interactions. For clarity, H atoms that are not involved in hydrogen-bonding have been omitted. Hydrogen bonds are shown as dashed lines.

2-hydroxy-5-methylbenzaldehyde. 2-Aminobenzoic acid (1 g, 7 mmol) was dissolved in methanol (25 ml), and a solution of 2-hydroxy-5-methylbenzaldehyde (1 g, 7 mmol) in methanol was added with stirring. The resulting mixture was heated at reflux for 1 h and cooled to room temperature. The solid product was collected by filtration and washed with cold methanol. The microcrystalline compound was recrystallized from hot methanol; crystals suitable for X-ray diffraction studies were obtained on slow evaporation. Yield 92%, m.p. 476 K.

Crystal data

$C_{15}H_{13}NO_3$
M_r = 255.26
Monoclinic, $C2/c$
a = 22.5716 (15) Å
b = 7.1742 (4) Å
c = 15.4336 (9) Å
β = 97.093 (2)°
V = 2480.1 (3) Å^3
Z = 8
D_x = 1.367 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
Block, colourless
0.24 × 0.22 × 0.21 mm

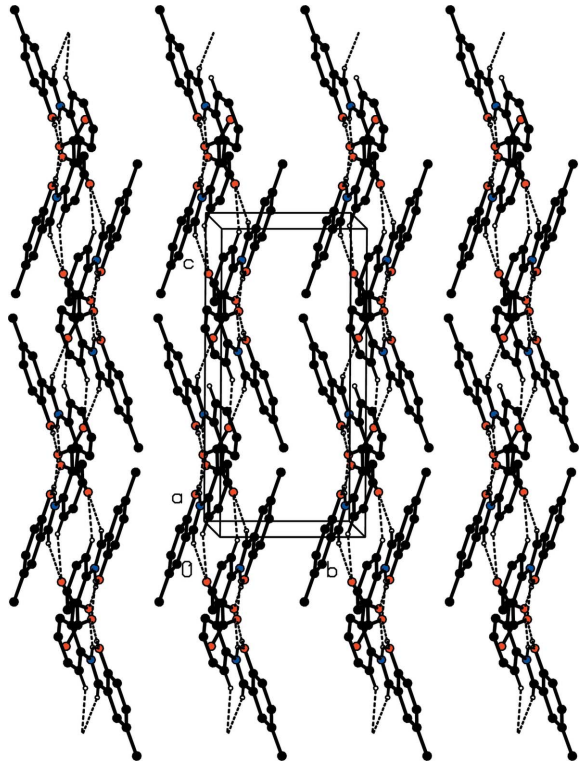


Figure 3
The molecular packing of (I), viewed down the *a* axis. For clarity, H atoms that are not involved in hydrogen bonding have been omitted. Hydrogen bonds shown as dashed lines.

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: none
13716 measured reflections
2941 independent reflections
2471 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 28.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.158
S = 1.05
2941 reflections
174 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0927P)^2 + 0.9763P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1	0.86	2.01	2.663 (2)	132
N1—H1···O2	0.86	1.91	2.592 (2)	135
O1—H1D···O2^{i}	0.82	1.66	2.464 (2)	166
C8—H8···O3^{ii}	0.93	2.56	3.462 (2)	165
C11—H11···O3	0.93	2.47	2.784 (2)	100
C14—H14···O3^{ii}	0.93	2.41	3.282 (2)	156

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

All H atoms were refined using a riding model with C—H = 0.93 Å, U_{iso}(H) = 1.2U_{eq}(C) for aromatic, C—H = 0.96 Å, U_{iso}(H) = 1.5U_{eq}

(C) for CH_3, N—H = 0.86 Å, U_{iso}(H) = 1.2U_{eq} (N) for the NH group and O—H = 0.83 Å, U_{iso}(H) = 1.5U_{eq} (O) for the OH group. In addition, the torsion angles for the rotation of the OH and methyl groups were refined.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PARST* (Nardelli, 1995).

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References

Allen, F. H., Kennard, O., Watson, D., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bargossi, C., Fiorini, M. C., Montalti, M., Prodi, L. & Zaccheroni, N. (2000). *Coord. Chem. Rev.* **208**, 17–32.
Bruker (2001). *SMART* (Version 5.625/NT/2000) and *SAINT* (Version 6.28a). Bruker AXS Inc., Madison, Wisconsin, USA.
Czanik, A. W. (1994). *Acc. Chem. Res.* **27**, 302–308.
Löhr, H. G. & Vögtle, F. (1985). *Acc. Chem. Res.* **18**, 65–72.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Schmidtchen, F. P. & Berger, M. (1997). *Chem. Rev.* **97**, 1609–1646.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.