

3-(5-Bromo-2-hydroxybenzylammonio)propanoate

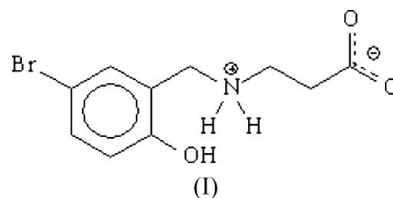
Xiu-Ju Yin,^a Jin-Hua Cai^a and
Seik Weng Ng^{b*}^aDepartment of Chemistry and Life Science,
Hechi University, Yizhou 546300, Guangxi,
People's Republic of China, and ^bDepartment of
Chemistry, University of Malaya, 50603 Kuala
Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.006 \text{ \AA}$
R factor = 0.035
wR factor = 0.082
Data-to-parameter ratio = 14.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{10}\text{H}_{12}\text{BrNO}_3$, exists as a zwitterion in the
solid state; the delocalized $-\text{CO}_2$ unit carries the negative
charge and the secondary N atom carries the positive charge.Received 18 October 2006
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Comment

Schiff bases derived from 5-bromosalicylaldehyde possess
useful antifungal and anticancer properties (Liang *et al.*, 2006;
Zhao & Liu, 2002). The $\text{C}=\text{N}$ double bond in these systems is
rigid, but can be reduced. The reduced compounds are of
chemical interest as they are more flexible (Chen *et al.*, 2003;
Hazell *et al.*, 1997); for example, *N*-(2-hydroxybenzyl)alanine
functions as a chelate to Cu^{II} (Sureshan & Bhattacharya, 1998;
Yang *et al.*, 2001, 2004).The 4-bromo-substituted title compound, (I), exists as a
zwitterion in the solid state (Fig. 1), with a delocalized
carboxylate group at one end of the molecule. The secondary
ammonium group interacts with the O atoms of other zwitter-
ions, forming a linear chain structure. Interestingly, both H
atoms are engaged in bifurcated hydrogen bonds (Table 1).

Experimental

5-Bromosalicylaldehyde (0.20 g, 1 mmol), β -alanine (0.09 g, 1 mmol)
and potassium hydroxide (0.06 g, 1 mmol) were dissolved in 80%
aqueous methanol (20 ml). The mixture was kept cold while stirring,
giving a clear yellow solution. An aqueous solution (5 ml) of sodium
borohydride (0.50 mmol, 0.02 g) was added, followed by addition of 1
M hydrochloric acid until the pH of the reaction mixture reached
5.5. The resulting white solid was collected and washed with ethanol.
The compound was purified by recrystallization from 50% aqueous
methanol (15 ml). Colourless prismatic crystals separated from the
solution after 2 weeks in about 60% yield. Elemental analysis found:
C 43.80, H 4.37, N 5.17%; calculated: C 43.78, H 4.38, N 5.15%.

Crystal data

 $\text{C}_{10}\text{H}_{12}\text{BrNO}_3$
 $M_r = 274.12$
Orthorhombic, $Pna2_1$
 $a = 13.971(4) \text{ \AA}$
 $b = 14.280(4) \text{ \AA}$
 $c = 5.553(2) \text{ \AA}$
 $V = 1107.9(8) \text{ \AA}^3$ $Z = 4$
 $D_x = 1.643 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
 $\mu = 3.70 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
Prism, colourless
 $0.30 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Bruker APEX-II CCD
diffractometer
 φ and ω scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)
 $T_{\min} = 0.366$, $T_{\max} = 0.477$

5103 measured reflections
2177 independent reflections
1687 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\text{max}} = 27.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.082$
 $S = 0.92$
2177 reflections
147 parameters
H atoms treated by a mixture of
independent and constrained
refinement

$w = 1/[\sigma^2(F_o^2) + (0.0466P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.54 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -0.40 \text{ e } \text{Å}^{-3}$
Absolute structure: Flack (1983),
825 Friedel pairs
Flack parameter: 0.04 (1)

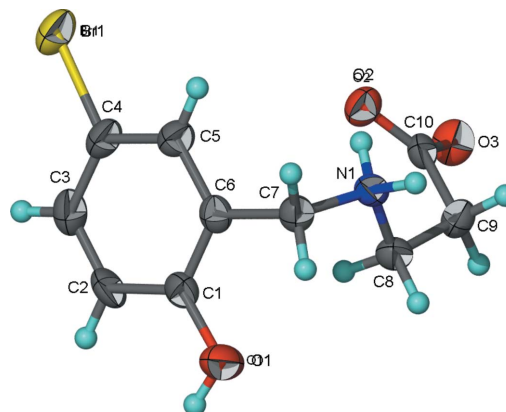


Figure 1
The molecular structure of (I), showing displacement ellipsoids at the 50% probability level and H atoms as spheres of arbitrary radii.

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$O1-H1o \cdots O3^i$	0.85 (1)	1.80 (2)	2.620 (4)	161 (5)
$N1-H1n \cdots O2$	0.85 (1)	2.22 (3)	2.836 (4)	129 (3)
$N1-H1n \cdots O2^{ii}$	0.85 (1)	2.38 (3)	2.929 (4)	122 (3)
$N1-H2n \cdots O2^{iii}$	0.85 (1)	2.28 (3)	2.993 (5)	141 (4)
$N1-H2n \cdots O3^{iii}$	0.85 (1)	2.36 (3)	3.106 (5)	147 (4)

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

Carbon-bound H atoms were placed at calculated positions ($C-H = 0.93-0.97 \text{ Å}$) and were included in the refinement in the riding-model approximation, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C)$. The ammonium and hydroxy H atoms were located in a difference Fourier map, and were refined with distance restraints of $N-H = O-H = 0.85 (1) \text{ Å}$, and with isotropic displacement parameters.

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: SHELXL97.

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