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3-[(*E*)-(5-Bromo-2-hydroxyphenyl)methylideneaminomethyl]cyclohexanecarboxylic acid

Shaukat Shuja,[a] Saqib Ali,[a] Nasir Khalid[b] and Masood Parvez[c]*

[a]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, [b]Nuclear Chemistry Division, Pakistan Institute of Nuclear Science and Technology, PO Nilore, Islamabad, Pakistan, and [c]Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4

Correspondence e-mail: parvez@ucalgary.ca

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.032
wR factor = 0.084
Data-to-parameter ratio = 18.4

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

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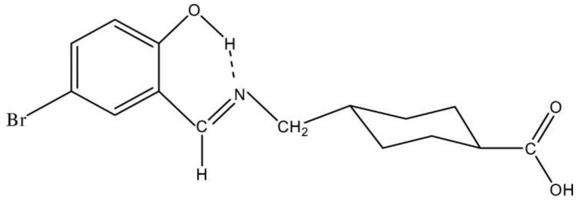
The title molecule, $C_{15}H_{18}BrNO_3$, contains a cyclohexyl ring in a chair conformation linked to an aromatic ring through an HC═N—CH$_2$ group. The molecular conformation may be partially controlled by an intramolecular O—H···N hydrogen bond, while the crystal structure is stabilized by intermolecular O—H···O hydrogen bonds.

Comment

Schiff bases are synthesized from an aromatic or aliphatic amine and carbonyl compound in nucleophilic addition to a hemiaminal followed by elimination of water to the imine. Schiff bases are an important class of ligands having many applications. In this paper, the synthesis and crystal structure of the title Schiff base, (I), which was obtained by condensation of 5-bromosalicyldehyde and tranexamic acid, are reported.



(I)

The molecular structure of (I) (Fig. 1) is composed of a cyclohexyl ring which adopts a classical chair conformation with puckering parameters (Cremer & Pople, 1975) *Q* = 0.563 (2) Å, $\theta = 0.0$ (2)° and $\varphi = 341$ (15)° and an almost planar (5-bromo-2-hydroxyphenyl)methylideneaminomethyl unit in which atoms O1 and C8 deviate from the Br1/N1/O1/C1–C8 least-squares plane by 0.041 (2) and 0.053 (2) Å, respectively. Selected bond lengths and angles are listed in Table 1. The hydroxyl H atom is involved in an intramolecular interaction with an amino N atom, resulting in a six-membered ring that may be expressed in graph-set terms as an *S*(6) motif (Bernstein *et al.*, 1995). In the crystal structure, molecules form dimeric pairs through hydrogen bonds involving carboxylic acid groups related by inversion centers (Fig. 2), representing a motif with graph set $R_2^2(8)$; details of the hydrogen-bonding geometry are provided in Table 2.

Experimental

Tranexamic acid (2.2 g, 14 mmol) and 5-bromosalicyldehyde (2.8 g, 14 mmol) were suspended in ethanol (50 ml) in a round-bottomed two-necked flask. The mixture was stirred continuously and refluxed for 3 h. The solvent was evaporated under reduced pressure and a yellow product was obtained. The product was redissolved in ethanol and after two weeks yellow crystals suitable for X-ray analysis were obtained (yield 65%, m.p. 453–455 K).

Crystal data

$C_{15}H_{18}BrNO_3$
M_r = 340.21
Monoclinic, $P2_1/n$
a = 9.786 (3) Å
b = 12.589 (5) Å
c = 12.475 (4) Å
β = 104.437 (19)°
V = 1488.3 (9) Å^3
Z = 4
D_x = 1.518 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 2.77 mm^{-1}
T = 173 (2) K
Prism, yellow
0.18 × 0.12 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
$T_{\min}$ = 0.623, $T_{\max}$ = 0.717
6399 measured reflections
3364 independent reflections
2581 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.026
$\theta_{\max}$ = 27.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.084
S = 1.01
3364 reflections
183 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.042P)^2 + 0.55P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.27 e Å^{-3}
$\Delta\rho_{\min}$ = −0.44 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C5	1.901 (2)	O3—C15	1.221 (3)
O1—C2	1.346 (3)	N1—C7	1.271 (3)
O2—C15	1.311 (3)	N1—C8	1.459 (3)
C7—N1—C8	120.1 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.84	1.82	2.566 (3)	148
O2—H2···O3$^{\rm i}$	0.84	1.83	2.674 (2)	178

Symmetry code: (i) $-x-1, -y, -z$.

H-atoms were located in difference Fourier maps and were subsequently included in the refinement in geometrically idealized positions with O—H = 0.84 Å and C—H = 0.96 Å, and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O) and 1.2$U_{\rm eq}$(C).

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

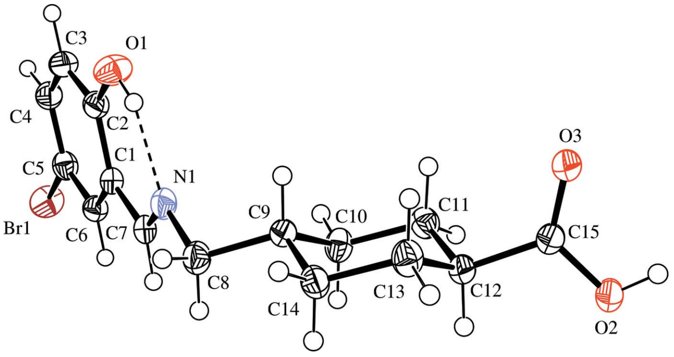


Figure 1
The molecular structure of (I), with displacement ellipsoids plotted at the 50% probability level. The dashed line indicates a hydrogen bond.

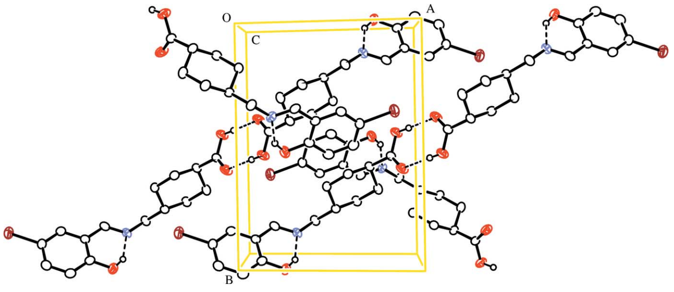


Figure 2
Part of the crystal structure of (I) showing intra- and intermolecular hydrogen bonds as dashed lines. H atoms not involved in these interactions have been omitted.

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