

2-[(4-Bromophenyl)iminomethyl]-6-methoxyphenol

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Received 8 September 2005
Accepted 30 September 2005
Online 12 October 2005

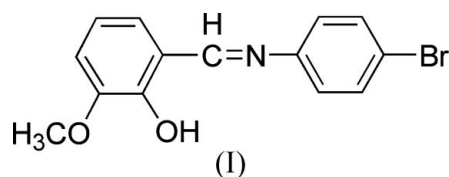
Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.028
 wR factor = 0.070
Data-to-parameter ratio = 13.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{14}\text{H}_{12}\text{BrNO}_2$, was prepared from 2-hydroxy-3-methoxybenzaldehyde and 4-bromophenylamine. The 4-hydroxy-3-methoxyphenyl and bromobenzene groups are *trans* with respect to the imino bond. The planarity of the molecule is supported by the conjugation of the imino group and the aromatic system, together with a resonance-assisted intramolecular hydrogen bond [$\text{O}-\text{H}\cdots\text{N} = 2.610$ (3) Å].

Comment

The synthesis and structure determination of the title compound, (I) (Fig. 1), have been carried out in the context of the design of novel crystalline solids based on crystal engineering (Tynan *et al.*, 2005; Parashar *et al.*, 1988). The bond lengths of $\text{C7}-\text{C8}$ [1.429 (4) Å] and $\text{C7}=\text{N1}$ [1.277 (4) Å] are in good agreement with the values observed in a similar system (Jing *et al.*, 2005). The 4-bromophenylimino moiety ($\text{C1}-\text{C6}/\text{N1}/\text{Br1}$) is planar, with an r.m.s. deviation of fitted atoms of 0.0046 Å. The *o*-vanillin moiety ($\text{C7}-\text{C13}/\text{O1}/\text{O2}$) is planar, with an r.m.s. deviation of fitted atoms of 0.0055 Å. The dihedral angle between these two planes is 12.74 (8)°. The values of the $\text{N1}-\text{C7}-\text{C8}-\text{C13}$ and $\text{N1}-\text{C6}-\text{C5}-\text{C4}$ torsion angles of -178.9 (3) and 179.9 (3)°, respectively, illustrate the planarity of the molecule.



An intramolecular resonance-assisted hydrogen bond (Gigli *et al.*, 1989) (Table 2) contributes to the planarity of the molecule. Crystal packing is *via* $\text{C}-\text{H}(\text{ar})\cdots\text{Br}$ interactions [3.704 (4) Å] connecting molecules along the *c* axis, and $\text{C}-\text{H}\cdots\text{O}$ interactions [3.346 (3) Å] involving an $\text{O}sp^3$ atom (Lommerse *et al.*, 1997; Molčanov *et al.*, 2004) (Table 2).

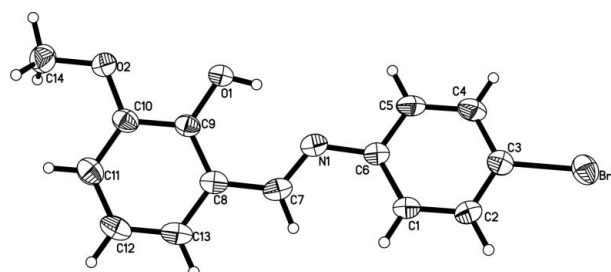


Figure 1
A view of (I), with 30% probability displacement ellipsoids.

Experimental

An anhydrous ethanol solution of 2-hydroxy-3-methoxybenzaldehyde (1.52 g, 10 mmol) was added to an anhydrous ethanol solution of 4-bromophenylamine (1.72 g, 10 mmol), and the mixture was stirred at 350 K for 5 h under nitrogen. A yellow precipitate was obtained. The product was isolated, recrystallized from ethanol and dried *in vacuo* to give pure compound (I) in 83% yield. Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution of (I).

Crystal data

C₁₄H₁₂BrNO₂ Mo K α radiation
M_r = 306.16 Cell parameters from 3003 reflections
Orthorhombic, P2₁2₁2₁ θ = 2.6–24.6°
a = 4.9175 (9) Å μ = 3.24 mm⁻¹
b = 12.524 (2) Å T = 293 (2) K
c = 20.551 (4) Å Prism, colourless
V = 1265.6 (4) Å³ 0.26 × 0.24 × 0.12 mm
Z = 4
D_x = 1.607 Mg m⁻³

Data collection

Bruker SMART CCD area-detector 2214 independent reflections
diffractometer 1993 reflections with I > 2σ(I)
φ and ω scans R_{int} = 0.019
Absorption correction: multi-scan θ_{max} = 25.0°
(SADABS; Bruker, 1999) h = -5 → 5
T_{min} = 0.420, T_{max} = 0.678 k = -14 → 12
6772 measured reflections l = -23 → 24

Refinement

Refinement on F² w = 1/[σ²(F_o²) + (0.0257P)²
R[F² > 2σ(F²)] = 0.028 + 0.2106P]
wR(F²) = 0.070 where P = (F_o² + 2F_c²)/3
S = 1.07 (Δ/σ)_{max} < 0.001
2214 reflections Δρ_{max} = 0.38 e Å⁻³
165 parameters Δρ_{min} = -0.41 e Å⁻³
H-atom parameters constrained Absolute structure: Flack (1983),
with 883 Friedel pairs
Flack parameter: 0.004 (12)

Table 1

Selected geometric parameters (Å, °).

Br1—C3	1.899 (3)	N1—C7	1.277 (4)
O2—C10	1.357 (3)	N1—C6	1.406 (4)
O2—C14	1.413 (4)		
C10—O2—C14	117.5 (2)	C1—C6—N1	125.1 (3)
C7—N1—C6	121.7 (2)	N1—C7—C8	123.3 (3)
C5—C6—N1	117.5 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
O1—H1...N1	0.82	1.88	2.610 (3)	147
C2—H2...O2 ⁱ	0.93	2.50	3.346 (3)	152
C11—H11...Br1 ⁱⁱ	0.93	2.88	3.704 (4)	149

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

The H atom of the hydroxyl group was found in a difference; it and the other H atoms were included and refined with free coordinates and an isotropic U parameter. The other H atoms were included in calculated positions and refined using a riding-model approximation, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic CH, C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl CH₃, and O—H = 0.82 Å and U_{iso}(H) = 1.2U_{eq}(O) for the hydroxyl group.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

This work was supported by the Science Fund of Tianjin University of Science and Technology (grant No. 118181), which is gratefully acknowledged.

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