

2-(3-Methoxysalicylideneamino)-1H-benzimidazole

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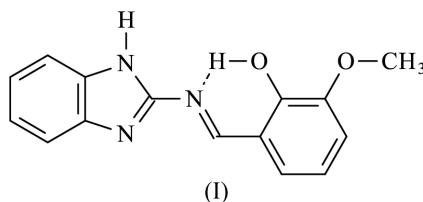
Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.039
wR factor = 0.087
Data-to-parameter ratio = 13.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The molecule of the title compound, $\text{C}_{15}\text{H}_{13}\text{O}_2\text{N}_3$, deviates slightly from planarity. There is a strong intramolecular $\text{N} \cdots \text{O}$ hydrogen bond of $2.5831(19) \text{ \AA}$. The molecule exists in the phenol–imine form and the dihedral angle between the two aromatic ring systems is $14.61(5)^\circ$.

Comment

The chemical background of (I) was described by Albayrak *et al.* (2005). The molecular structure with the atom-labelling scheme is shown in Fig.1. Selected bond lengths and angles are listed in Table 1.



In (I), the dihedral angle between the salicylidene and benzimidazole ring systems is $14.61(5)^\circ$ and the molecule is less nearly planar than that in the structure of its monohydrate (Albayrak, *et al.*, 2005), in which the corresponding dihedral angle is $1.04(5)^\circ$. In (I), the phenol–imine tautomer is favoured over the keto–amine form in the solid state. This fact is evident from the $\text{O1}-\text{C2}$ bond distance of $1.3571(19) \text{ \AA}$, which is consistent with an $\text{O}-\text{C}$ single bond; additionally, the $\text{C7}-\text{N1}$ distance of $1.285(2) \text{ \AA}$ is consistent with a $\text{C}=\text{N}$ double bond, as in *N*-(2-fluoro-3-methoxy)salicylaldimine [$\text{O}-\text{C} = 1.347(3) \text{ \AA}$ and $\text{C}=\text{N} 1.280(3) \text{ \AA}$; Ünver *et al.*, 2002] and 3-methoxysalicylidene-2-aminobenzimidazole monohydrate [$\text{O}-\text{C} = 1.357(2) \text{ \AA}$ and $\text{C}=\text{N} 1.287(2) \text{ \AA}$; Albayrak, *et al.*, 2005].

Compound (I) exhibits a strong intramolecular $\text{O}-\text{H} \cdots \text{N}$ hydrogen bond and a weak $\text{C}-\text{H} \cdots \text{N}$ interaction, in addition to intermolecular $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds (Table 2).

Experimental

The title compound was prepared by refluxing a mixture of a solution containing *o*-vanillin (0.5 g, 3.2 mmol) in ethanol (20 ml) and a solution containing 2-aminobenzimidazole (0.43 g, 3.2 mmol) in ethanol (20 ml). The reaction mixture was stirred for 1 h under reflux. The resulting orange precipitate was filtered off and recrystallized from ethanol by slow evaporation. Crystals of (I) suitable for X-ray analysis were obtained from the same solution as the crystals of the monohydrate compound (Albayrak, *et al.*, 2005) (yield 95%; m.p. 492–494 K).

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Crystal data

C₁₅H₁₃N₃O₂
M_r = 267.28
 Monoclinic, *P*2₁/*c*
a = 12.5450 (9) Å
b = 8.4858 (8) Å
c = 13.2955 (10) Å
 β = 111.755 (6)°
V = 1314.56 (18) Å³
Z = 4

D_x = 1.351 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 17 957 reflections
 θ = 1.8–26.0°
 μ = 0.09 mm⁻¹
T = 293 K
 Prism, red
 0.28 × 0.27 × 0.26 mm

Data collection

Stoe IPDS-II diffractometer
 ω scans
 23 491 measured reflections
 2590 independent reflections
 1519 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.072
 θ_{max} = 26.0°
h = -15 → 15
k = -10 → 10
l = -16 → 16

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.039
wR (*F*²) = 0.087
S = 0.88
 2590 reflections
 189 parameters

H atoms treated by a mixture of independent and constrained refinement
w = 1/[σ²(*F*_o²) + (0.0435*P*)²]
 where *P* = (*F*_o² + 2*F*_c²)/3
 (Δ/σ)_{max} < 0.001
 Δρ_{max} = 0.10 e Å⁻³
 Δρ_{min} = -0.18 e Å⁻³

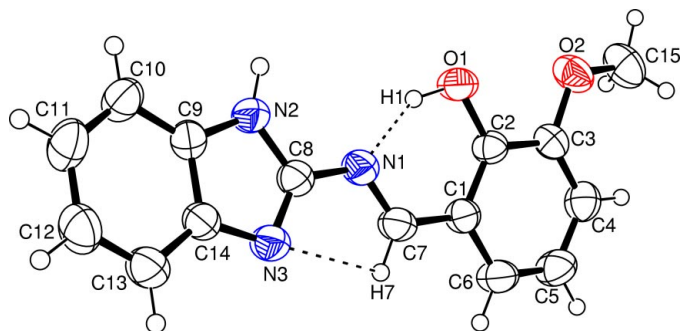


Figure 1
 A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

Table 1

Selected geometric parameters (Å, °).

C2–O1	1.3571 (19)	C8–N1	1.389 (2)
C3–O2	1.370 (2)	C9–N2	1.375 (2)
C7–N1	1.285 (2)	C14–N3	1.391 (2)
C8–N3	1.310 (2)	C15–O2	1.423 (2)
C8–N2	1.359 (2)		
N1–C7–C1	121.73 (15)	C7–N1–C8	119.33 (14)
N3–C8–N2	114.09 (15)	C8–N2–C9	106.57 (14)
N3–C8–N1	128.03 (15)	C8–N3–C14	103.84 (14)
N2–C8–N1	117.86 (15)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
O1–H1...N1	0.96 (3)	1.71 (3)	2.5831 (19)	150 (2)
C7–H7...N3	0.93	2.51	2.832 (2)	100
N2–H2...O1 ¹	0.92 (2)	2.06 (2)	2.9317 (19)	156.5 (18)
N2–H2...O2 ¹	0.92 (2)	2.55 (2)	3.211 (2)	129.1 (16)

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

H atoms bonded to N and O were located in a difference map and refined isotropically. Other H atoms were placed in calculated positions (C–H = 0.93–0.96 Å), with *U*_{iso}(H) values constrained to be 1.5*U*_{eq} of the carrier atom for the methyl-group H atoms and 1.2*U*_{eq} for the remaining H atoms.

Data collection: *X*-AREA (Stoe & Cie, 2002); cell refinement: *X*-AREA; data reduction: *X*-RED (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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