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(Z)-3-Hydroxy-6-[(2-methoxyphenylamino)-methylene]cyclohexa-2,4-dienone

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The title compound, $C_{14}H_{13}N_1O_3$, adopts the keto–amine tautomeric form, with an intramolecular N—H···O resonance-assisted hydrogen bond. The two benzene rings are nearly coplanar [dihedral angle = 14.3 (2)°]. The molecules are linked by intermolecular O—H···O hydrogen bonds and C—H···π interactions between methyl groups and benzene rings.

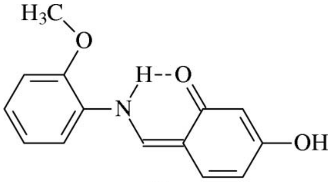
Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
R factor = 0.059
wR factor = 0.192
Data-to-parameter ratio = 11.8

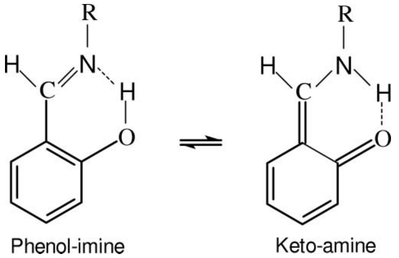
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

There is an important interest in Schiff base ligands and their complexes with regards to their significant antitumour activities (Zhou *et al.*, 2000). Schiff base compounds can be classified by their photochromic and thermochromic characteristics (Cohen *et al.*, 1964; Moustakali *et al.*, 1978; Hadjoudis *et al.*, 1987). Based on studies of some thermochromic and photochromic Schiff base compounds, it has been proposed that molecules exhibiting monochromism are planar, while those exhibiting photochromism are non-planar (Moustakali *et al.*, 1978). *o*-Hydroxy Schiff bases exist in an enol form (Yıldız *et al.*, 1998; Elmalı & Elerman *et al.*, 1998; Dey *et al.*, 2001; Ünver, Yıldız *et al.*, 2002; Yang, Vittal *et al.*, 2003), a keto form (Ünver, Kabak *et al.*, 2002; Hökelek *et al.*, 2000) or as enol–keto mixtures (Nazır *et al.*, 2000; Szady-Chelmienicecka *et al.*, 2001) as a result of H-atom transfer from the hydroxy O atom to the N atom. Such H-atom tautomerism plays a significant role in many fields of chemistry, in particular, in biochemistry (Hem *et al.*, 2002).



(I)



An *ORTEP-3* (Farrugia, 1997) wiew of the molecule of (I) and a packing diagram are shown in Figs. 1 and 2, respectively. Compound (I) exists primarily as the keto–amine tautomer (see scheme), as indicated by the C2═O1, C7—N1, C8—N1

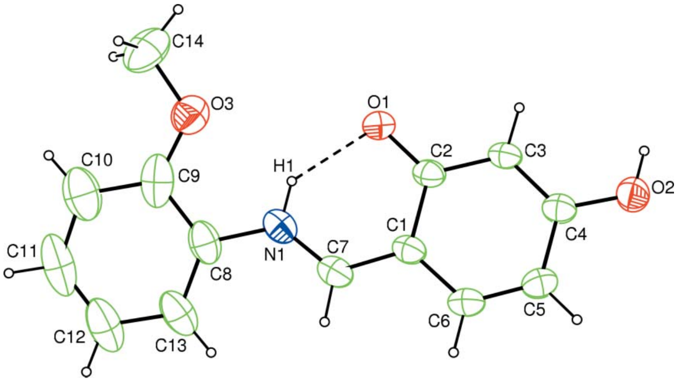


Figure 1
A view of (I), with the atom-numbering scheme and 40% probability displacement ellipsoids. The intramolecular hydrogen bond is shown as a dashed line.

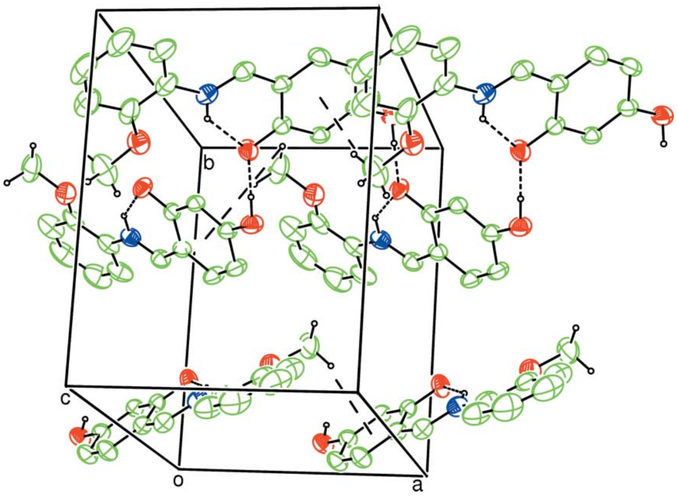


Figure 2
A packing diagram for (I) showing hydrogen bonds (short dashes) and C—H$\cdots\pi$ interactions (long dashes). H atoms not involved in these interactions have been omitted for clarity.

and C1═C7 bond lengths (Table 1). These bonds are comparable with those of 2-hydroxy-6-[(2-methoxyphenyl)aminomethylene]cyclohexa-2,4-dienone [1.293 (2), 1.304 (2), 1.414 (2) and 1.404 (2)Å, respectively; Şahin *et al.*, 2005]. In this keto–amine tautomer an intramolecular N—H$\cdots$O hydrogen bond, with a short N$\cdots$O distance of 2.582 (4) Å (Table 2), is an example of a hydrogen bond assisted by resonance due to conjugation with a π system that results in its pronounced covalent character (Bertolasi *et al.*, 1997; Jeffrey, 1997).

In the structure of (I), there is weak a C—H$\cdots\pi$ interaction between C14—H14*B* and the C1–C6 benzene ring (Table 2). The perpendicular distance between atom H14*B* and the plane of the benzene ring is 2.52Å. The crystal packing is supported by an intermolecular O—H$\cdots$O hydrogen bond (Table 2).

Experimental

The title compound was prepared as described by Şahin *et al.* (2005) using 2-methoxyaniline and 4-hydroxysalicylaldehyde as starting materials. Well-shaped crystals of (I) were obtained by slow evaporation of a solution in ethanol (yield 76%; m.p. 423-425 K).

Crystal data

$C_{14}H_{13}NO_3$
$M_r = 243.25$
Orthorhombic, $P2_12_12_1$
$a = 8.3391$ (7) Å
$b = 11.1748$ (15) Å
$c = 13.8056$ (19) Å
$V = 1286.5$ (3) Å^3
$Z = 4$
$D_x = 1.256$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 21062 reflections
$\theta = 2.3$–$26.0°$
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Prism, yellow
0.35 × 0.31 × 0.27 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{min} = 0.972$, $T_{max} = 0.981$
21062 measured reflections

1788 independent reflections
1007 reflections with $I > 2\sigma(I)$
$R_{int} = 0.077$
$\theta_{max} = 28.0°$
$h = -11 \rightarrow 10$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.192$
$S = 0.97$
1788 reflections
152 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1182P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C1—C7	1.391 (6)	C8—N1	1.401 (5)
C2—O1	1.286 (5)	C9—O3	1.342 (7)
C4—O2	1.333 (5)	C14—O3	1.419 (6)
C7—N1	1.309 (5)		
N1—C7—C1	123.8 (4)	C7—N1—C8	128.4 (4)
C13—C8—N1	122.6 (5)		
C2—C1—C7—N1	1.6 (6)	C1—C7—N1—C8	179.9 (4)

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

*Cg*1 is the centroid of the C1–C6 benzene ring.

D—H$\cdots$*A*	*D*—H	H$\cdots$*A*	*D*$\cdots$*A*	*D*—H$\cdots$*A*
N1—H1$\cdots$O1	0.86	1.89	2.582 (4)	137
O2—H2$\cdots$O1^{i}	0.91 (4)	1.60 (4)	2.506 (4)	175 (6)
C14—H14*B*$\cdots$*Cg*1^{ii}	0.96	2.60	3.529 (7)	162

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

In the absence of significant anomalous scattering, Friedel pairs were merged. H atoms attached to C atoms were refined using a riding model; C—H = 0.93Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for aromatic C atoms, and C—H = 0.96Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl C atoms. The H atom of the hydroxyl group was refined with O—H restrained to 0.88 (4)Å and $U_{iso}(H) = 1.5U_{eq}(O)$.

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)

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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).

References

Bertolasi, V., Gilli, P., Ferretti, V. & Gilli, G. (1997). *J. Chem. Soc. Perkin Trans. 2*, pp. 945–952.

Cohen, M. D., Schmidt, G. M. J. & Flavian, S. (1964). *J. Chem. Soc*. pp. 2041–2051.

Dey, D. K., Dey, S. P., Elmalı, A. & Elerman, Y. (2001). *J. Mol. Struct*. **562**, 177–184.

Elmalı, A. & Elerman, Y. (1998). *J. Mol. Struct*. **442**, 31–37.

Farrugia, L. J. (1997). *J. Appl. Cryst*. **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Hadjoudis, E., Vitterakis, M. Moustakali, I. & Mavridis, I. (1987). *Tetrahedron*, **43**, 1345–1360.

Hem, J., Fadhil, S. K., Cees, G., van der Gert, Z. & Liudmil, A. (2002). *J. Photochem. Photobiol. A*, **152**, 183–191.

Hökelek, T., Kılıç, Z., Işıklan, M. & Toy, M. (2000). *J. Mol. Struct*. **523**, 61–69.

Jeffrey, G. A. (1997). *An Introduction to Hydrogen Bonding*, pp 99–103. Oxford University Press.

Moustakali, I., Mavridis, I., Hadjoudis, E. & Mavridis, A. (1978). *Acta Cryst*. B**34**, 3709–3715.

Nazır, H., Yıldız, M., Yılmaz, H., Tahir, M. N. & Ülkü, D. (2000). *J. Mol. Struct*. **524**, 241–250.

Şahin, O., Büyükgüngör, O., Albayrak, Ç. & Odabaşoğlu, M. (2005). *Acta Cryst*. E**61**, o1579–o1581.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Stoe & Cie (2002). *X-AREA* and *X-RED*. Stoe & Cie, Darmstadt, German.

Szady-Chelmienicecka, A., Grech, E., Rozwadowski, Z., Dziembowska, T., Schilf, W. & Kamienski, B. (2001). *J. Mol. Struct*. **565–566**, 125–128.

Ünver, H., Kabak, M., Zengin, D. M. & Durlu, T. N. (2002). *J. Chem. Crystallogr*. **31**, 203–209.

Ünver, H., Yıldız, M., Zengin, D. M., Özbey, S. & Kendi, E. (2002). *J. Chem. Crystallogr*. 3**1**, 211–216.

Yang, C.-T. & Vittal, J. (2003). *Inorg. Chim. Acta*, **344**, 65–76.

Yıldız, M., Kılıç, Z. & Hökelek, T. (1998). *J. Mol. Struct*. **44**1, 1–10.

Zhou, Y.-S., Zhang, L.-J., Zeng, X.-R., Vittal, J. J. & You, X.-Z. (2000). *J. Mol. Struct.* **524**, 241–250.

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