Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-Hydroxy-6-[(2-methoxyphenyl)aminomethylene]cyclohexa-2,4-dienone

Onur Şahin,[a] Orhan Büyükgüngör,[a] Çiğdem Albayrak[b] and Mustafa Odabaşoğlu[b]*

[a]Department of Physics, Ondokuz Mayıs University, TR-55139, Samsun, Turkey, and [b]Department of Chemistry, Ondokuz Mayıs University, TR-55139, Samsun, Turkey

Correspondence e-mail: onurs@omu.edu.tr

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.096
Data-to-parameter ratio = 16.4

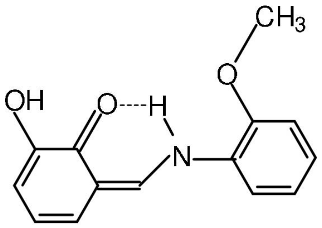
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 15 April 2005
Accepted 26 April 2005
Online 7 May 2005

The molecule of the title compound, $C_{14}H_{13}NO_3$, adopts the keto–amine tautomeric form. An intramolecular N—H···O hydrogen bond dictates the essentially planar molecular geometry; the two benzene rings make a dihedral angle of 7.93 (7)°. The crystal packing is stabilized by intermolecular O—H···O and C—H···O hydrogen bonds.

Comment

It is known that the thermo- and photochromism observed in N-substituted *ortho*-hydroxyimines in the solid state are possibly due to H-atom transfer from the hydroxy O atom to the N atom (Hadjoudis *et al.*, 1987; Xu *et al.*, 1994). It is also known that the chemical conductivity of Schiff bases containing *ortho*-hydroxyphenyl fragments is strongly influenced by an H-atom transfer reaction between the phenol–imine and keto–amine tautomers (Yüce *et al.*, 2004). The phenol–imine form is dominant in salicylaldimine, while the keto–amine form is preferred in *ortho*-hydroxynaphthaldimine Schiff bases. It has also been observed that, in the solid state, the keto–amine form is present in hydroxynaphthaldimine, while the phenol–imine form exists in salicylaldimine Schiff bases (Kaitner & Pavlovic *et al.*, 1996; Yıldız *et al.*, 1998). The title compound, (I) (Fig. 1), is in the keto–amine tautomeric form.



(I)

In the molecule of (I), the C10=O2 and N1—C8 bond lengths of 1.2931 (17) and 1.3043 (19) Å, respectively (Table 1), are in good agreement with those observed in 2-[(2-hydroxy-4-nitrophenyl)aminomethylene]cyclohexa-3,5-dien-1(2*H*)-one [1.298 (2) and 1.308 (2) Å; Ersanlı *et al.*, 2003] and 1-[(4-nitrophenylamino)methylene]naphthalen-2(1*H*)-one [1.289 (3) and 1.306 (3) Å; Özek *et al.*, 2004]. The C1—N1 bond length is 1.4135 (18) Å, corresponding to 1.4155 (18) Å in 3-(4-acetylphenyliminomethyl)-1,2-dihydroxybenzene (Yüce *et al.*, 2004). The C8=C9 bond length and C8—N1—C1 bond angle are 1.404 (2) Å and 127.29 (13)°, respectively, compared with 1.396 (3) Å and 126.35 (19)°, respectively, in 1-[(4-nitrophenylamino)methylene]naphthalen-2(1*H*)-one (Özek *et al.*, 2004).

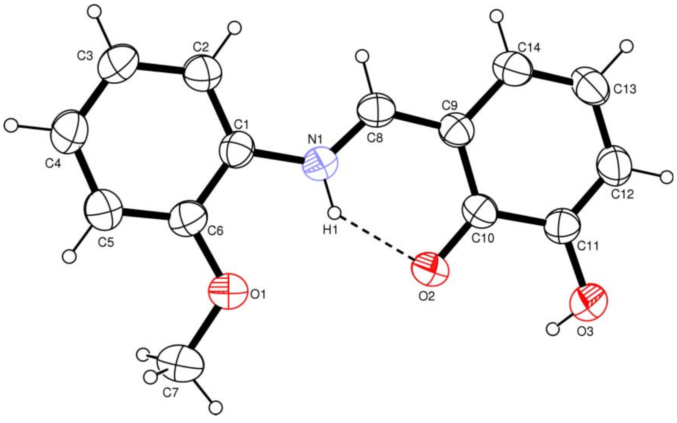


Figure 1
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. The dashed line indicates a hydrogen bond.

There is a strong intramolecular N1—H1···O2 hydrogen bond (Table 2), with the H atom transferred from the O to the N atom. The N1···O2 distance [2.5870 (16) Å] is shorter than the sum of the van der Waals radii of O and N [3.07 Å; Bondi *et al.*, 1964], and comparable with those observed in 2-{[tris(hydroxymethyl)methyl]aminomethylene}cyclohexa-3,5-dien-1(2*H*)-one and its 6-hydroxy and 6-methoxy derivatives [2.5851 (16) Å; Odabaşoğlu *et al.*, 2003]. This intramolecular N—H···O hydrogen bond dictates the essentially planar molecular geometry; the two benzene rings make a dihedral angle of 7.93 (7)°.

In the crystal structure of (I), there is an intermolecular O—H···O hydrogen bond with an O···O distance of 2.7160 Å (Table 2); this may be compared with the sum of the van der Waals radii of two O atoms [3.04 Å; Pizzala *et al.*, 2000]. The arrangement of O—H···O hydrogen bonds can be described by the graph-set notation $R_2^2(10)$, and this generates a centrosymmetric dimer with an *S*(6) motif (Bernstein *et al.*, 1995). The crystal packing (Fig. 2) is further stabilized by weak intermolecular C—H···O hydrogen bonds (Table 2).

Experimental

The title compound was prepared by refluxing a solution containing 3-hydroxysalicylaldehyde (3.5 mmol) in ethanol (10 ml) and a solution containing 2-methoxyaniline (3.5 mmol) in ethanol (10 ml). The reaction mixture was stirred for 1 h under reflux and then allowed to cool. The powder product, (I), was recrystallized from ethanol. Suitable single crystals were obtained from methanol (yield 83%; m.p. 478–479 K).

Crystal data

$C_{14}H_{13}NO_3$
$M_r = 243.25$
Monoclinic, $P2_1/n$
$a = 7.5517$ (7) Å
$b = 11.3022$ (12) Å
$c = 13.4646$ (13) Å
$\beta = 95.186$ (8)°
$V = 1144.5$ (2) Å^3
$Z = 4$
$D_x = 1.412$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 5462 reflections
$\theta = 2.4$–27.9°
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Prism, red
0.30 × 0.21 × 0.15 mm

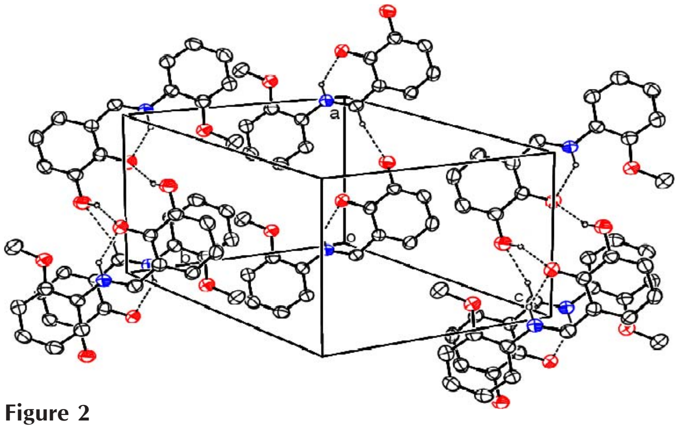


Figure 2
A packing diagram for (I). Dashed lines indicate hydrogen bonds. Other H atoms have been omitted for clarity.

Data collection

Stoe IPDS-II diffractometer
Rotation *ω* scans
Absorption correction: integration (*X-RED*; Stoe & Cie, 2002)
$T_{\rm min} = 0.973$, $T_{\rm max} = 0.993$
13 408 measured reflections
2724 independent reflections
1551 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.070$
$\theta_{\rm max} = 27.9°$
$h = -9 \rightarrow 9$
$k = -14 \rightarrow 14$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.096$
$S = 0.85$
2724 reflections
166 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.15$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.017 (2)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.4135 (18)	C9—C10	1.425 (2)
C8—N1	1.3043 (19)	C10—O2	1.2931 (17)
C8—C9	1.404 (2)	C11—O3	1.3673 (18)
C2—C1—N1	123.54 (14)	O2—C10—C9	122.59 (13)
N1—C8—C9	123.20 (13)	O3—C11—C10	119.47 (14)
C8—C9—C10	120.11 (13)	C8—N1—C1	127.29 (13)
N1—C8—C9—C10	0.7 (2)	C2—C1—N1—C8	6.0 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O2	0.86	1.89	2.5870 (16)	137
O3—H3*A*···O2$^{\rm i}$	0.82	1.95	2.7160 (16)	154
C8—H8···O3$^{\rm ii}$	0.93	2.59	3.3597 (19)	141

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

After their location in a difference Fourier map, all H atoms were positioned geometrically and refined using a riding model, with C—H distances in the range 0.93–0.96 Å, O—H = 0.82 Å and N—H = 0.86 Å, and with $U_{\rm iso}$(H) = 1.2–1.5$U_{\rm eq}$ of the parent atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s)

o1580 Şahin *et al.* • $C_{14}H_{13}NO_3$ *Acta Cryst.* (2005). E**61**, o1579–o1581

used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDS-II diffractometer (purchased under grant No. F279 of the University Research Fund).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

Ersanlı, C. C., Albayrak, Ç., Odabaşoğlu, M. & Erdönmez, A. (2003). *Acta Cryst.* C**59**, o601–o602.

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

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

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

Kaitner, B. & Pavlovic, G. (1996). *Acta Cryst.* C**52**, 2573–2575.

Odabaşoğlu, M., Albayrak, Ç., Büyükgüngör, O. & Lönnecke, P. (2003). *Acta Cryst.* C**59**, o616–o619.

Özek, A., Yüce, S., Albayrak, Ç., Odabaşoğlu, M. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o826–o827.

Pizzala, H., Carles, M., Stone, W. E. E. & Thevand, A. (2000). *J. Chem. Soc. Perkin Trans. 2*, pp. 935–939.

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

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

Xu, X., You, X., Sun, Z., Wang, X. & Liu, H. (1994). *Acta Cryst.* C**50**, 1169–1171.

Yıldız, M., Kılı ç, Z. & Hökelek, T. (1998). *J. Mol. Struct.* **441**, 1–10.

Yüce, S., Özek, A., Albayrak, Ç., Odabaşoğlu, M. & Büyükgüngör, O. (2004). *Acta Cryst.* E**60**, o810–o812.

Acta Cryst. (2005). E**61**, o1579–o1581 Şahin *et al.* • $C_{14}H_{13}NO_3$ **o1581**