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Crystal structure of (*E*)-4-hydroxy-3-{1-[(4-hydroxyphenyl)imino]ethyl}-6-methyl-2*H*-pyran-2-one

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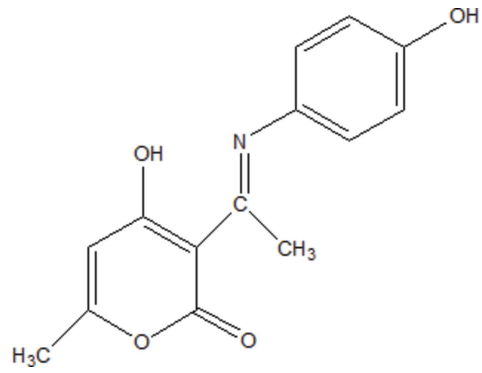
In the title Schiff base, $C_{14}H_{13}NO_4$, which adopts the phenol–imine tautomeric form, the dihedral angle between the planes of the benzene and heterocyclic (r.m.s. deviation = 0.037 Å) rings is 53.31 (11)°. An intramolecular O—H···N hydrogen bond closes an $S(6)$ ring. In the crystal, molecules are linked by O—H···O hydrogen bonds to generate $C(11)$ chains propagating in the [010] direction. A weak C—H···O link is also observed, leading to the formation of $R_5^5(32)$ rings extending parallel to the (101) plane.

Keywords: crystal structure; hydroxy Schiff base; pyran-2-one; phenol–imine tautomer; hydrogen bonding; proton-transfer processes.

CCDC reference: 1410367

1. Related literature

For photochromic and thermochromic properties of hydroxy Schiff bases, see: Garnovskii *et al.* (1993); Hadjoudis *et al.* (2004). For potential materials for optical memory and switch devices, see: Zhao *et al.* (2007). For proton-transfer processes, see: Lussier *et al.* (1987). For Schiff base structures, see: Djedouani *et al.* (2007, 2008). For Schiff base bond lengths and angles, see: Girija & Begum (2004); Girija *et al.* (2004); Bai & Jing (2007).



2. Experimental

2.1. Crystal data

$C_{14}H_{13}NO_4$
M_r = 259.26
Monoclinic, $P2_1/c$
a = 7.8730 (5) Å
b = 11.7930 (8) Å
c = 13.5330 (8) Å
β = 99.896 (2)°
V = 1237.79 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.10 × 0.06 × 0.03 mm

2.2. Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.875, T_{max} = 0.947
17976 measured reflections
2582 independent reflections
2061 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.148
S = 1.07
2582 reflections
173 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N1	0.82	1.83	2.560 (2)	147
O2—H2···O1^{i}	0.82	1.90	2.710 (2)	169
C12—H12B···O3^{ii}	0.96	2.55	3.137 (3)	120

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

Data collection: *COLLECT* (Nonius, 2002); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *OLEX2.refine* (Dolomanov *et al.*, 2009); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *PARST* (Nardelli, 1995).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7460).

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supporting information

Acta Cryst. (2015). E**71**, o564–o565 [https://doi.org/10.1107/S2056989015012840]

Crystal structure of (*E*)-4-hydroxy-3-{1-[(4-hydroxyphenyl)imino]ethyl}-6-methyl-2*H*-pyran-2-one

Amel Djedouani, Sihem Boufas, Franck Cleymand, Michel François and Solenne Fleutot

S1. Comment

Hydroxy Schiff bases have been extensively studied due to their biological, photochromic and thermochromic properties (Garnovskii *et al.*, 1993; Hadjoudis *et al.*, 2004). They are potential materials for optical memory and switch devices (Zhao *et al.*, 2007). Proton transfer in these compounds forms the basis for an explanation of the mechanisms of various biological processes where proton transfer is the rate-determining step (Lussier *et al.*, 1987). In general, *O*-hydroxy Schiff bases exhibit two possible tautomeric forms, the phenol-imine (or benzenoid) and ketoamine (or quinoid) forms. Depending on the tautomers, two types of intra-molecular hydrogen bonds are possible: O—H···N in benzenoid and N—H···O in quinoid tautomers.

As part of our ongoing studies of Schiff bases (Djedouani *et al.*, 2007, 2008), we now describe the synthesis and the structure of the title compound, which takes the form phenol-imine and complete a six-membered pseudocycle *via* an intramolecular O—H···O hydrogen bond.

The dehydroacetic acid ring and phenyl ring are almost planer with r.m.s deviation for the mean plane are 0.0260 and 0.0027 respectively. The dihedral angle between the two rings is 53.30 (0.05) °. The two torsional angles τ1 (N1—C5—C14—C4) and τ2 (C5—N1—C1—C6) defining the confirmation of the molecule.

The N1—C5 distance of 1.324 (2) Å agree with similar bond in related compounds (Girija & Begum, 2004; Girija *et al.* 2004), slightly longer than a typical C=N bond (1.283 (4) Å) (Bai & Jing, 2007); but much shorter than the single carbon-nitrogen bond (Table. 1), N1—C1=1.432 (3) Å because of the resonance. The carbon-carbon bond connecting the phenol and imine groups exhibits intermediate distances between a single and a double bond and agree well with those observed in other azomethines. The C5—N1—C14 and C14—C5—N1 bond angle of 117.70 (2)° and 117.47 () ° respectively in the Schiff base ligand are smaller than typical hexagonal of 120°. This is due to the effect of substitution on O of pyron & OH of the DHA ring.

In the crystal, molecules are aligned head to foot along *b* axis, in columns along to [0 0 1] axis and the structure is stabilized by an O—H···O hydrogen bond and another weak C—H···O interaction, leading to the formation of $R_5^5(32)$ rings extending parallel to the (101) plane (Fig. 2, Table.1).

S2. Experimental

Compound (I) was prepared by refluxing a mixture of a solution containing dehydroacetic acid (0.01 mmol) and *para*-4-aminophenol (0.01 mmol) in ethanol (40 ml). The reaction mixture was stirred for 2 h under reflux and left to cool. Yellow crystals grew after a few days.

S3. Refinement

C—H and O—H hydrogen atoms were placed in calculated positions and refined as riding atoms with C—H distances of 0.93 Å with U_{iso}(H) = 1.2Ueq(C) and O—H distances of 0.82 Å, with U_{iso}(H) = 1.2Ueq(N).

The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å) with U_{iso}(H) = 1.2Ueq(C), but were allowed to rotate freely about the C—C bonds.

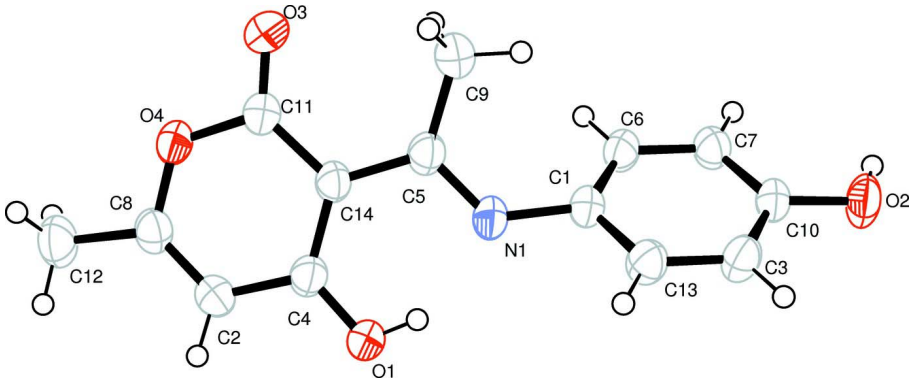

Figure 1

The structure of the title compound in 50% probability ellipsoids.

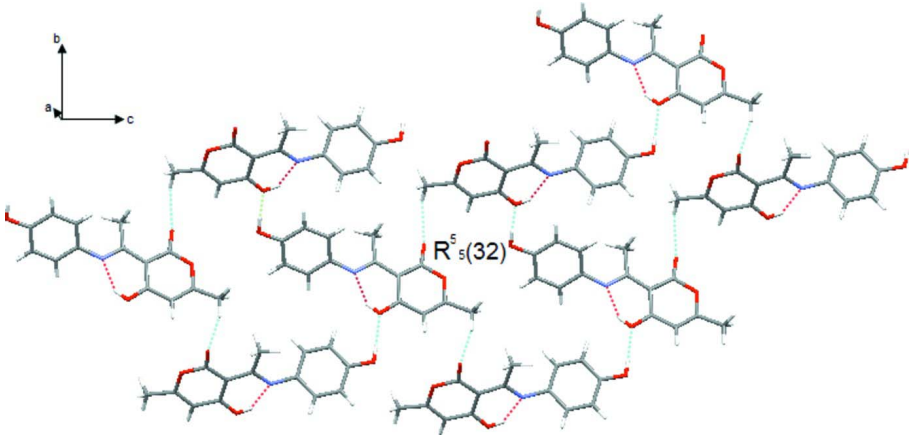

Figure 2

Part of the crystal structure of (I), showing the formation of *S*(6) rings with dashed red lines. C—H···O and O—H···O hydrogen bonds are shown as blue dashed lines.

(*E*)-4-hydroxy-3-{1-[(4-hydroxyphenyl)imino]ethyl}-6-methyl-2*H*-pyran-2-one

Crystal data

$C_{14}H_{13}NO_4$
M_r = 259.26
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Hall symbol: -P 2ybc
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b = 11.7930 (8) Å
c = 13.5330 (8) Å
β = 99.896 (2)°
V = 1237.79 (14) Å^3
Z = 4

F(000) = 544.3271
D_x = 1.391 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 0 reflections
θ = 2.9–27.5°
μ = 0.10 mm^{-1}
T = 293 K
Block, yellow
0.10 × 0.06 × 0.03 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: Enraf–Nonius FR590
Graphite monochromator
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thin slices scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
T_{min} = 0.875, T_{max} = 0.947

17976 measured reflections
2582 independent reflections
2061 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.7°, θ_{min} = 2.3°
h = −9→9
k = −14→14
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.148
S = 1.07
2582 reflections
173 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 0.6848P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
O1	0.6637 (2)	−0.08723 (12)	0.50633 (10)	0.0520 (4)
H1	0.6696 (2)	−0.05535 (12)	0.45317 (10)	0.0779 (6)*
O2	0.4993 (2)	0.21003 (13)	−0.00201 (10)	0.0543 (4)
H2	0.4624 (2)	0.27519 (13)	−0.00450 (10)	0.0815 (7)*
O3	1.0343 (3)	0.20593 (17)	0.65333 (13)	0.0814 (7)
O4	0.92760 (19)	0.08216 (12)	0.74439 (9)	0.0443 (4)
C1	0.6821 (3)	0.11371 (17)	0.29043 (13)	0.0390 (4)
C2	0.7568 (3)	−0.07161 (18)	0.67874 (15)	0.0444 (5)
H2a	0.7031 (3)	−0.13959 (18)	0.68968 (15)	0.0533 (6)*
C3	0.6279 (3)	0.07382 (18)	0.11415 (14)	0.0433 (5)
H3	0.6333 (3)	0.02421 (18)	0.06136 (14)	0.0520 (6)*
C4	0.7488 (3)	−0.03156 (16)	0.57810 (14)	0.0391 (4)
C5	0.8305 (3)	0.12191 (16)	0.46720 (14)	0.0382 (4)
C6	0.6138 (3)	0.22135 (17)	0.27136 (14)	0.0416 (5)
H6	0.6089 (3)	0.27092 (17)	0.32425 (14)	0.0499 (6)*
C7	0.5531 (3)	0.25514 (17)	0.17428 (14)	0.0416 (5)
H7	0.5074 (3)	0.32751 (17)	0.16191 (14)	0.0499 (6)*
C8	0.8390 (3)	−0.01396 (17)	0.75665 (14)	0.0407 (5)
C9	0.9259 (3)	0.22691 (19)	0.44950 (16)	0.0489 (5)
H9a	0.9215 (18)	0.2372 (8)	0.37874 (17)	0.0733 (8)*
H9b	0.8738 (13)	0.2909 (3)	0.4764 (11)	0.0733 (8)*
H9c	1.0438 (6)	0.2203 (6)	0.4820 (10)	0.0733 (8)*
C10	0.5598 (3)	0.18172 (17)	0.09476 (13)	0.0389 (4)
C11	0.9398 (3)	0.12553 (18)	0.65006 (15)	0.0454 (5)
C12	0.8492 (3)	−0.0416 (2)	0.86465 (15)	0.0554 (6)

H12a	0.9678 (4)	−0.0434 (15)	0.8967 (3)	0.0831 (9)*
H12b	0.789 (2)	0.0151 (9)	0.8959 (3)	0.0831 (9)*
H12c	0.797 (2)	−0.1144 (7)	0.87109 (15)	0.0831 (9)*
C13	0.6877 (3)	0.03998 (17)	0.21150 (15)	0.0419 (5)
H13	0.7319 (3)	−0.03271 (17)	0.22414 (15)	0.0502 (6)*
C14	0.8397 (2)	0.07122 (16)	0.56378 (13)	0.0368 (4)
N1	0.7321 (2)	0.07168 (14)	0.39048 (12)	0.0434 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0748 (11)	0.0408 (8)	0.0347 (7)	−0.0120 (7)	−0.0066 (7)	0.0022 (6)
O2	0.0822 (11)	0.0507 (9)	0.0265 (7)	0.0122 (8)	−0.0009 (7)	0.0018 (6)
O3	0.1131 (16)	0.0775 (13)	0.0444 (9)	−0.0536 (12)	−0.0125 (9)	0.0064 (9)
O4	0.0563 (9)	0.0434 (8)	0.0295 (7)	−0.0021 (6)	−0.0029 (6)	−0.0003 (6)
C1	0.0454 (11)	0.0404 (10)	0.0286 (9)	−0.0016 (8)	−0.0009 (7)	0.0027 (8)
C2	0.0565 (13)	0.0373 (10)	0.0383 (10)	−0.0024 (9)	0.0051 (9)	0.0042 (8)
C3	0.0508 (12)	0.0445 (11)	0.0329 (10)	0.0055 (9)	0.0021 (8)	−0.0065 (8)
C4	0.0466 (11)	0.0347 (10)	0.0332 (9)	0.0032 (8)	−0.0009 (8)	−0.0006 (8)
C5	0.0431 (10)	0.0367 (10)	0.0329 (9)	0.0046 (8)	0.0012 (8)	0.0004 (8)
C6	0.0557 (12)	0.0394 (10)	0.0290 (9)	0.0000 (9)	0.0052 (8)	−0.0029 (8)
C7	0.0532 (12)	0.0359 (10)	0.0343 (10)	0.0040 (9)	0.0036 (8)	0.0028 (8)
C8	0.0475 (11)	0.0389 (10)	0.0346 (10)	0.0077 (8)	0.0046 (8)	0.0027 (8)
C9	0.0536 (13)	0.0506 (12)	0.0397 (11)	−0.0060 (10)	0.0007 (9)	0.0079 (9)
C10	0.0451 (11)	0.0425 (11)	0.0275 (9)	0.0004 (8)	0.0018 (7)	0.0032 (8)
C11	0.0555 (13)	0.0423 (11)	0.0348 (10)	−0.0052 (10)	−0.0028 (9)	0.0032 (8)
C12	0.0773 (16)	0.0557 (14)	0.0329 (11)	0.0044 (12)	0.0088 (10)	0.0034 (9)
C13	0.0462 (11)	0.0379 (10)	0.0385 (10)	0.0066 (8)	−0.0008 (8)	0.0005 (8)
C14	0.0439 (11)	0.0336 (9)	0.0306 (9)	0.0029 (8)	−0.0002 (8)	0.0021 (7)
N1	0.0560 (10)	0.0414 (9)	0.0290 (8)	−0.0014 (8)	−0.0033 (7)	0.0036 (7)

Geometric parameters (Å, º)

O1—H1	0.82	C5—C9	1.489 (3)
O1—C4	1.265 (2)	C5—C14	1.428 (3)
O2—H2	0.82	C5—N1	1.324 (2)
O2—C10	1.356 (2)	C6—H6	0.93
O3—C11	1.201 (3)	C6—C7	1.377 (3)
O4—C8	1.356 (2)	C7—H7	0.93
O4—C11	1.394 (2)	C7—C10	1.389 (3)
C1—C6	1.385 (3)	C8—C12	1.486 (3)
C1—C13	1.384 (3)	C9—H9a	0.96
C1—N1	1.432 (2)	C9—H9b	0.96
C2—H2a	0.93	C9—H9c	0.96
C2—C4	1.433 (3)	C11—C14	1.442 (3)
C2—C8	1.326 (3)	C12—H12a	0.96
C3—H3	0.93	C12—H12b	0.96
C3—C10	1.388 (3)	C12—H12c	0.96

C3—C13	1.380 (3)	C13—H13	0.93
C4—C14	1.438 (3)		
C4—O1—H1	109.5	C12—C8—C2	127.3 (2)
C10—O2—H2	109.5	H9a—C9—C5	109.5
C11—O4—C8	122.46 (15)	H9b—C9—C5	109.5
C13—C1—C6	119.66 (17)	H9b—C9—H9a	109.5
N1—C1—C6	121.88 (17)	H9c—C9—C5	109.5
N1—C1—C13	118.18 (18)	H9c—C9—H9a	109.5
C4—C2—H2a	119.25 (12)	H9c—C9—H9b	109.5
C8—C2—H2a	119.25 (12)	C3—C10—O2	117.93 (17)
C8—C2—C4	121.5 (2)	C7—C10—O2	122.75 (18)
C10—C3—H3	119.90 (11)	C7—C10—C3	119.31 (17)
C13—C3—H3	119.90 (12)	O4—C11—O3	113.30 (18)
C13—C3—C10	120.19 (18)	C14—C11—O3	129.00 (19)
C2—C4—O1	119.33 (18)	C14—C11—O4	117.69 (18)
C14—C4—O1	122.99 (17)	H12a—C12—C8	109.5
C14—C4—C2	117.68 (17)	H12b—C12—C8	109.5
C14—C5—C9	123.23 (17)	H12b—C12—H12a	109.5
N1—C5—C9	119.29 (17)	H12c—C12—C8	109.5
N1—C5—C14	117.48 (18)	H12c—C12—H12a	109.5
H6—C6—C1	119.91 (11)	H12c—C12—H12b	109.5
C7—C6—C1	120.17 (18)	C3—C13—C1	120.30 (18)
C7—C6—H6	119.91 (12)	H13—C13—C1	119.85 (11)
H7—C7—C6	119.82 (12)	H13—C13—C3	119.85 (12)
C10—C7—C6	120.36 (18)	C5—C14—C4	121.89 (17)
C10—C7—H7	119.82 (11)	C11—C14—C4	118.74 (17)
C2—C8—O4	121.50 (18)	C11—C14—C5	119.35 (18)
C12—C8—O4	111.21 (18)	C5—N1—C1	127.99 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.83	2.560 (2)	147
O2—H2···O1^{i}	0.82	1.90	2.710 (2)	169
C12—H12B···O3^{ii}	0.96	2.55	3.137 (3)	120

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*+2, *y*−1/2, −*z*+3/2.