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1-(2-Hydroxy-5-methoxyphenyl)ethan-1-one *N*-[(*E*)-1-(2-hydroxy-5-methoxyphenyl)ethylidene]hydrazone

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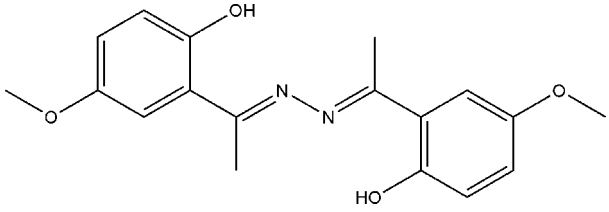
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.047; wR factor = 0.178; data-to-parameter ratio = 16.4.

In the title molecule, $C_{18}H_{20}N_2O_4$, which resides on a crystallographic centre of inversion (at the centre of the N—N bond), all non-H atoms apart from the methoxy substituent are approximately coplanar. The structure displays intramolecular O—H···N hydrogen bonding.

Related literature

For related literature, see: Saroja *et al.* (1995); Sreerama *et al.* (2007); Sreerama & Pal (2005); Tian *et al.* (2007).



Experimental

Crystal data

$C_{18}H_{20}N_2O_4$
M_r = 328.36
Monoclinic, $P2_1/c$
a = 8.5545 (7) Å
b = 6.4614 (4) Å
c = 14.3548 (10) Å
β = 91.243 (5)°
V = 793.26 (10) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 (2) K
0.39 × 0.23 × 0.06 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
T_{min} = 0.963, T_{max} = 0.995
7584 measured reflections
1837 independent reflections
1306 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.178
S = 1.05
1837 reflections
112 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 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	2.5523 (18)	146

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GG2057).

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supporting information

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1-(2-Hydroxy-5-methoxyphenyl)ethan-1-one *N*-[(*E*)-1-(2-hydroxy-5-methoxyphenyl)ethylidene]hydrazone

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S1. Comment

In bis Schiff base systems derived from hydrazine, the two chelating sites are connected directly by a single bond, (Saroja *et al.* 1995, Sreerama *et al.* 2005, 2007, Tian *et al.* 2007). However, To date, there has been no crystal structure report of the compound 2,2′-(1*E*,1′*E*)-1,1′-(hydrazine-1,2-diylidene)bis(ethan-1-yl-1-ylidene)bis(4-methoxyphenol). We report here the crystal structure of the title compound (Fig. 1).

In the title compound (Fig. 1), all bond lengths and angles are normal. Apart from the methoxy substituent, all non-H atoms of the molecule are coplanar to within 0.029 Å. In the crystal structure, intramolecular O—H···N hydrogen bonds are observed.

S2. Experimental

A mixture of 1-(2-hydroxy-5-methoxyphenyl)ethanone (166 mg, 1 mmol), hydrazine sulfate (67 mg, 0.5 mmol) and triethylamine (153 mg, 1.5 mmol) in alcohol (10 ml) was heated to reflux for 32 h. After cooling, the precipitate was filtrated and washed with water to afford the product in 60% yield. Crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a solution of the solid in ethyl acetate at room temperature for 10 d.

S3. Refinement

All H atoms were placed in geometrically calculated positions and refined using a riding model with C—H = 0.97 Å (for CH_2 groups) and 0.96 Å (for CH_3 groups), their isotropic displacement parameters were set to 1.2 times (1.5 times for CH_3 groups) the equivalent displacement parameter of their parent atoms.

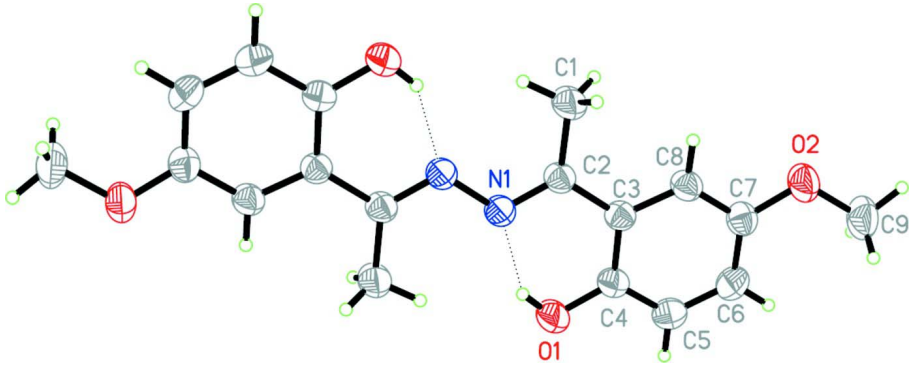

Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

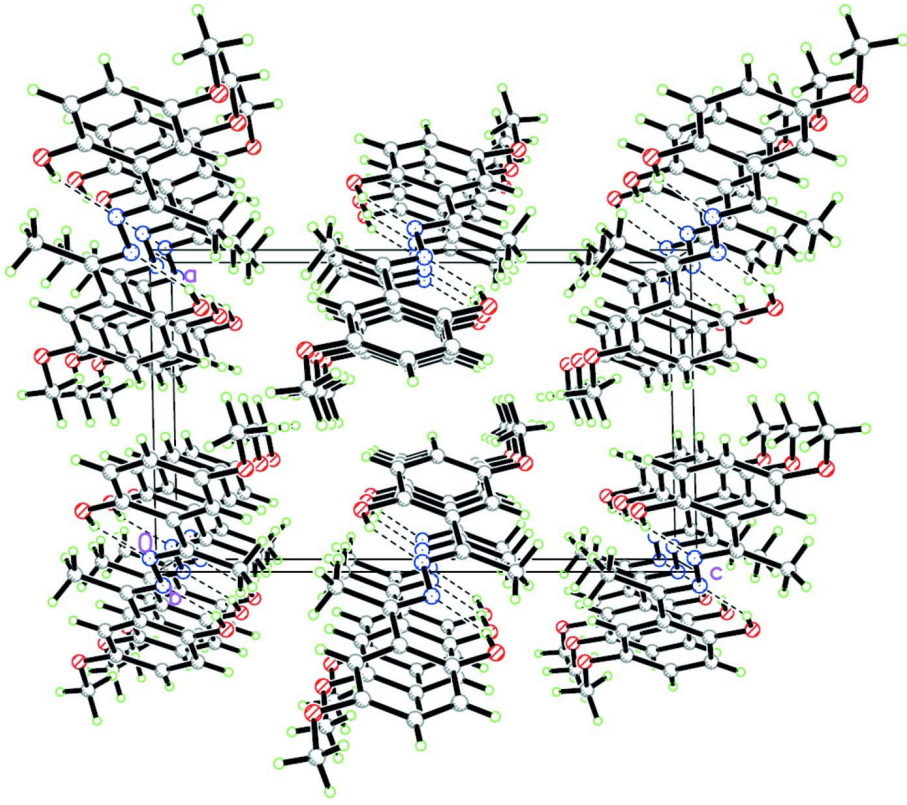

Figure 2

Packing view of (I), shown along the *b* axis direction.

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Crystal data

$C_{18}H_{20}N_2O_4$
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$\beta = 91.243\ (5)°$
$V = 793.26\ (10)$ Å^3
$Z = 2$
$F(000) = 348$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1667 reflections
$\theta = 2.4–27.6°$
$\mu = 0.10$ mm^{-1}
$T = 296$ K
Plate, orange-yellow
0.39 × 0.23 × 0.06 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
$T_{min} = 0.963$, $T_{max} = 0.995$
7584 measured reflections
1837 independent reflections
1306 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.4°$
$h = -11\rightarrow9$
$k = -8\rightarrow8$
$l = -18\rightarrow18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.178$
$S = 1.05$
1837 reflections
112 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
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1017P)^2 + 0.1301P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.
Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0145 (3)	0.1588 (3)	0.15985 (12)	0.0546 (5)
H1A	−0.0618	0.0500	0.1561	0.082*
H1B	−0.0328	0.2815	0.1842	0.082*
H1C	0.0996	0.1168	0.2003	0.082*
C2	0.0749 (2)	0.2025 (2)	0.06448 (11)	0.0371 (4)
C3	0.17276 (19)	0.3862 (2)	0.04965 (11)	0.0356 (4)
C4	0.2295 (2)	0.4376 (3)	−0.03962 (11)	0.0413 (4)
C5	0.3199 (2)	0.6135 (3)	−0.05005 (13)	0.0528 (5)
H5	0.3562	0.6473	−0.1087	0.063*
C6	0.3574 (2)	0.7398 (3)	0.02449 (13)	0.0500 (5)
H6	0.4185	0.8570	0.0159	0.060*
C7	0.3037 (2)	0.6916 (3)	0.11227 (12)	0.0417 (4)
C8	0.2134 (2)	0.5179 (3)	0.12370 (11)	0.0398 (4)
H8	0.1779	0.4868	0.1829	0.048*
C9	0.4389 (3)	0.9765 (3)	0.18430 (16)	0.0620 (6)
H9A	0.5389	0.9266	0.1650	0.093*
H9B	0.4503	1.0430	0.2439	0.093*
H9C	0.3989	1.0740	0.1393	0.093*
N1	0.04606 (16)	0.0865 (2)	−0.00768 (9)	0.0394 (4)
O1	0.19804 (19)	0.3216 (2)	−0.11624 (9)	0.0595 (5)
H1	0.1487	0.2184	−0.1014	0.089*
O2	0.33331 (18)	0.8075 (2)	0.19129 (9)	0.0572 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0789 (14)	0.0512 (10)	0.0339 (9)	−0.0200 (9)	0.0095 (9)	0.0003 (8)
C2	0.0445 (10)	0.0346 (8)	0.0322 (8)	−0.0006 (6)	0.0023 (7)	0.0020 (6)
C3	0.0387 (9)	0.0346 (8)	0.0334 (8)	0.0006 (6)	0.0017 (6)	0.0019 (6)
C4	0.0461 (10)	0.0440 (9)	0.0340 (9)	−0.0039 (7)	0.0047 (7)	−0.0005 (7)
C5	0.0597 (12)	0.0576 (12)	0.0415 (10)	−0.0159 (9)	0.0113 (8)	0.0027 (8)
C6	0.0524 (12)	0.0466 (10)	0.0514 (11)	−0.0145 (8)	0.0065 (9)	0.0038 (8)
C7	0.0463 (10)	0.0363 (8)	0.0423 (9)	−0.0012 (7)	−0.0017 (7)	−0.0014 (7)
C8	0.0487 (10)	0.0379 (8)	0.0330 (8)	−0.0020 (7)	0.0031 (7)	0.0005 (7)
C9	0.0744 (14)	0.0471 (11)	0.0641 (13)	−0.0195 (10)	−0.0072 (11)	−0.0069 (9)
N1	0.0482 (9)	0.0351 (7)	0.0351 (7)	−0.0052 (6)	0.0049 (6)	−0.0007 (6)
O1	0.0799 (11)	0.0644 (9)	0.0348 (7)	−0.0262 (7)	0.0129 (6)	−0.0073 (6)
O2	0.0758 (10)	0.0464 (7)	0.0494 (8)	−0.0202 (6)	0.0028 (7)	−0.0088 (6)

Geometric parameters (Å, º)

C1—C2	1.501 (2)	C6—C7	1.386 (3)
C1—H1A	0.9600	C6—H6	0.9300
C1—H1B	0.9600	C7—C8	1.374 (2)
C1—H1C	0.9600	C7—O2	1.378 (2)
C2—N1	1.298 (2)	C8—H8	0.9300
C2—C3	1.471 (2)	C9—O2	1.422 (2)
C3—C8	1.400 (2)	C9—H9A	0.9600
C3—C4	1.420 (2)	C9—H9B	0.9600
C4—O1	1.353 (2)	C9—H9C	0.9600
C4—C5	1.385 (2)	N1—N1^{i}	1.388 (3)
C5—C6	1.378 (3)	O1—H1	0.8200
C5—H5	0.9300		
C2—C1—H1A	109.5	C5—C6—C7	119.81 (16)
C2—C1—H1B	109.5	C5—C6—H6	120.1
H1A—C1—H1B	109.5	C7—C6—H6	120.1
C2—C1—H1C	109.5	C8—C7—O2	116.04 (15)
H1A—C1—H1C	109.5	C8—C7—C6	119.35 (16)
H1B—C1—H1C	109.5	O2—C7—C6	124.61 (16)
N1—C2—C3	116.73 (14)	C7—C8—C3	122.47 (16)
N1—C2—C1	123.78 (15)	C7—C8—H8	118.8
C3—C2—C1	119.50 (15)	C3—C8—H8	118.8
C8—C3—C4	117.32 (15)	O2—C9—H9A	109.5
C8—C3—C2	120.91 (15)	O2—C9—H9B	109.5
C4—C3—C2	121.77 (15)	H9A—C9—H9B	109.5
O1—C4—C5	117.95 (15)	O2—C9—H9C	109.5
O1—C4—C3	122.54 (15)	H9A—C9—H9C	109.5
C5—C4—C3	119.51 (16)	H9B—C9—H9C	109.5
C6—C5—C4	121.54 (17)	C2—N1—N1^{i}	115.92 (16)
C6—C5—H5	119.2	C4—O1—H1	109.5

C4—C5—H5	119.2	C7—O2—C9	117.68 (16)
N1—C2—C3—C8	−178.44 (15)	C5—C6—C7—C8	0.0 (3)
C1—C2—C3—C8	1.5 (3)	C5—C6—C7—O2	−179.58 (17)
N1—C2—C3—C4	1.7 (2)	O2—C7—C8—C3	179.54 (15)
C1—C2—C3—C4	−178.35 (17)	C6—C7—C8—C3	0.0 (3)
C8—C3—C4—O1	179.88 (15)	C4—C3—C8—C7	0.3 (3)
C2—C3—C4—O1	−0.2 (3)	C2—C3—C8—C7	−179.56 (15)
C8—C3—C4—C5	−0.6 (3)	C3—C2—N1—N1^{i}	179.83 (16)
C2—C3—C4—C5	179.33 (16)	C1—C2—N1—N1^{i}	−0.2 (3)
O1—C4—C5—C6	−179.92 (18)	C8—C7—O2—C9	173.87 (16)
C3—C4—C5—C6	0.5 (3)	C6—C7—O2—C9	−6.6 (3)
C4—C5—C6—C7	−0.2 (3)		

Symmetry code: (i) −*x*, −*y*, −*z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.83	2.5523 (18)	146