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N′-[(1*E*)-1-(2-Hydroxy-5-methylphenyl)-ethylidene]-4-methoxybenzohydrazide

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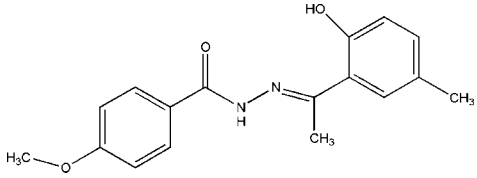
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.004 Å; R factor = 0.051; wR factor = 0.178; data-to-parameter ratio = 12.9.

The title compound, $C_{17}H_{18}N_2O_3$, displays a *trans* conformation with respect to the C=N double bond. The crystal structure is stabilized by intramolecular O—H···N and intermolecular N—H···O hydrogen bonds.

Related literature

For related literature, see: Carcelli *et al.* (1995); Salem (1998); Singh *et al.* (1982).



Experimental

Crystal data

$C_{17}H_{18}N_2O_3$
M_r = 298.33
Monoclinic, $P2_1/c$
a = 15.2171 (13) Å
b = 4.9253 (4) Å
c = 20.0906 (16) Å
β = 93.021 (3)°
V = 1503.7 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 273 (2) K
0.35 × 0.24 × 0.13 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.971, T_{max} = 0.992
16192 measured reflections
2641 independent reflections
1598 reflections with $I > 2\check{s}I$)
R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.178
S = 1.00
2641 reflections
204 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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	1.81	2.528 (3)	145
N2—H2···O2^{i}	0.86	2.17	2.903 (1)	142

Symmetry code: (i) $x, y+1, z$.

Data collection: *APEXII* (Bruker, 2005); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2040).

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supplementary materials

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N'-[(1*E*)-1-(2-Hydroxy-5-methylphenyl)ethylidene]-4-methoxybenzohydrazide

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Comment

The chemistry of aroylhydrazones continues to attract much attention due to their coordination ability to metal ions (Singh *et al.*, 1982; Salem, 1998) and their biological activity (Singh *et al.*, 1982; Carcelli *et al.*, 1995). As an extension of work on the structural characterization of aroylhydrazone derivatives, the title compound (I) was synthesized and its crystal structure is reported here.

The title molecule displays a *trans* conformation with respect to the C8=N1 double bond (Fig. 1). The dihedral angle between the two benzene rings is 4.66 (16)°. The crystal structure is stabilized by intramolecular O—H···N and intermolecular N—H···O hydrogen bonds (Table 1. and Fig. 2).

Experimental

4-methoxybenzohydrazide (0.01 mol,1.66 g) was dissolved in anhydrous ethanol (50 ml), and 1-(2-hydroxy-5-methylphenyl)ethanone (0.01 mol, 1.50 g) was added. The reaction mixture was refluxed for 4 h with stirring, then the resulting precipitate was collected by filtration, washed several times with ethanol and dried *in vacuo* (yield 81%). The compound (1.0 mmol,0.29 g) was dissolved in dimethylformamide (15 ml) and kept at room temperature for 20 d to obtain colourless single crystals suitable for X-ray diffraction.

Refinement

All H atoms were positioned geometrically and treated as riding on their parent atoms,with *C*—*H*(methyl) = 0.96 Å, C—H(aromatic) = 0.93 Å, O—H = 0.82 Å, and N—H = 0.86 Å and with U_{iso}(H) = 1.5U_{eq}(C_{methyl},*O*) and 1.2U_{eq}($C_{aromatic}$,*N*).

Figures

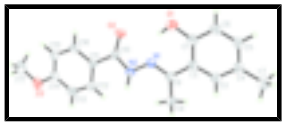

Fig. 1. The molecular structure of compound (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

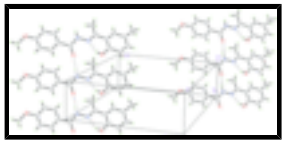

Fig. 2. The crystal packing of (I), viewed along the *c* axis. Dashed lines show intra-and intermolecular hydrogen bonds.

N'-[(1E)-1-(2-Hydroxy-5-methylphenyl)ethylidene]-4-methoxybenzohydrazide

Crystal data

$C_{17}H_{18}N_2O_3$	F_{000} = 632
M_r = 298.33	D_x = 1.318 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1229 reflections
a = 15.2171 (13) Å	θ = 2.4–21.3º
b = 4.9253 (4) Å	μ = 0.09 mm^{-1}
c = 20.0906 (16) Å	T = 273 (2) K
β = 93.021 (3)º	Plate, colourless
V = 1503.7 (2) Å^3	0.35 × 0.24 × 0.13 mm
Z = 4	

Data collection

Bruker APEXII CCD area-detector diffractometer	2641 independent reflections
Radiation source: fine-focus sealed tube	1598 reflections with $I > 2s^{-}I$)
Monochromator: graphite	R_{int} = 0.072
T = 273(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.3º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −17→18
T_{min} = 0.971, T_{max} = 0.992	k = −5→5
16192 measured reflections	l = −23→23

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.051	$w = 1/[\sigma^2(F_o^2) + (0.105P)^2 + 0.0006P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.178	$(\Delta/\sigma)_{max}$ = 0.003
S = 1.00	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
2641 reflections	$\Delta\rho_{min}$ = −0.20 e Å^{-3}
204 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.007 (2)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.24549 (12)	−0.3586 (4)	0.14131 (10)	0.0686 (6)
H1	0.1961	−0.2985	0.1321	0.103*
O2	0.01369 (11)	−0.4028 (3)	0.09357 (9)	0.0591 (6)
O3	−0.35729 (13)	0.0552 (4)	−0.00730 (10)	0.0760 (6)
N1	0.11590 (13)	−0.0443 (4)	0.15312 (10)	0.0479 (6)
N2	0.03244 (13)	0.0274 (4)	0.12841 (9)	0.0473 (6)
H2	0.0115	0.1877	0.1338	0.057*
C1	0.28167 (17)	−0.2245 (5)	0.19477 (12)	0.0503 (7)
C2	0.23917 (16)	−0.0114 (5)	0.22697 (12)	0.0450 (6)
C3	0.28441 (17)	0.1048 (5)	0.28221 (12)	0.0528 (7)
H3	0.2571	0.2445	0.3045	0.063*
C4	0.36719 (17)	0.0243 (6)	0.30568 (13)	0.0564 (7)
C5	0.40601 (18)	−0.1847 (6)	0.27243 (15)	0.0626 (8)
H5	0.4617	−0.2442	0.2872	0.075*
C6	0.36474 (18)	−0.3072 (6)	0.21818 (15)	0.0631 (8)
H6	0.3928	−0.4477	0.1968	0.076*
C7	0.4129 (2)	0.1559 (7)	0.36580 (15)	0.0768 (9)
H7A	0.3776	0.3031	0.3807	0.115*
H7B	0.4691	0.2239	0.3541	0.115*
H7C	0.4210	0.0243	0.4008	0.115*
C8	0.15058 (16)	0.0812 (5)	0.20433 (12)	0.0444 (6)
C9	0.10478 (17)	0.3000 (6)	0.24057 (13)	0.0591 (8)
H9A	0.1290	0.4732	0.2295	0.089*
H9B	0.1128	0.2704	0.2877	0.089*
H9C	0.0431	0.2972	0.2278	0.089*
C10	−0.01451 (16)	−0.1688 (5)	0.09532 (11)	0.0437 (6)
C11	−0.10208 (16)	−0.0933 (5)	0.06576 (11)	0.0432 (6)
C12	−0.15304 (18)	0.1148 (5)	0.09037 (13)	0.0536 (7)
H12	−0.1298	0.2249	0.1246	0.064*
C13	−0.23702 (19)	0.1592 (6)	0.06474 (14)	0.0611 (8)
H13	−0.2708	0.2969	0.0821	0.073*
C14	−0.27177 (18)	−0.0003 (5)	0.01309 (13)	0.0549 (7)
C15	−0.22169 (19)	−0.2015 (6)	−0.01320 (13)	0.0588 (7)

H15	−0.2441	−0.3049	−0.0489	0.071*
C16	−0.13797 (17)	−0.2490 (5)	0.01381 (12)	0.0521 (7)
H16	−0.1048	−0.3888	−0.0033	0.062*
C17	−0.3986 (2)	−0.1216 (8)	−0.05566 (16)	0.0916 (11)
H17A	−0.3719	−0.0983	−0.0976	0.137*
H17B	−0.4601	−0.0793	−0.0608	0.137*
H17C	−0.3914	−0.3064	−0.0411	0.137*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0628 (13)	0.0653 (13)	0.0770 (13)	0.0135 (10)	−0.0026 (10)	−0.0234 (11)
O2	0.0629 (12)	0.0356 (10)	0.0775 (12)	0.0072 (9)	−0.0083 (9)	−0.0033 (9)
O3	0.0581 (13)	0.0836 (15)	0.0846 (14)	0.0056 (11)	−0.0118 (11)	0.0048 (12)
N1	0.0463 (13)	0.0395 (12)	0.0572 (12)	0.0029 (10)	−0.0036 (10)	−0.0015 (10)
N2	0.0490 (13)	0.0350 (11)	0.0575 (12)	0.0053 (10)	−0.0007 (10)	−0.0055 (10)
C1	0.0458 (15)	0.0461 (15)	0.0592 (16)	−0.0028 (13)	0.0057 (12)	−0.0021 (13)
C2	0.0452 (15)	0.0385 (14)	0.0516 (14)	−0.0014 (12)	0.0060 (12)	0.0026 (11)
C3	0.0514 (17)	0.0497 (16)	0.0578 (16)	0.0001 (13)	0.0082 (13)	−0.0013 (13)
C4	0.0458 (16)	0.0582 (18)	0.0650 (17)	−0.0068 (14)	0.0009 (13)	0.0050 (14)
C5	0.0453 (16)	0.0640 (19)	0.0783 (19)	0.0015 (14)	0.0023 (15)	0.0045 (16)
C6	0.0505 (17)	0.0578 (18)	0.0814 (19)	0.0076 (14)	0.0082 (15)	−0.0054 (16)
C7	0.063 (2)	0.092 (2)	0.0745 (19)	−0.0073 (17)	−0.0084 (16)	−0.0066 (18)
C8	0.0471 (15)	0.0366 (13)	0.0499 (14)	−0.0008 (11)	0.0063 (12)	0.0033 (11)
C9	0.0572 (17)	0.0591 (17)	0.0610 (16)	0.0086 (13)	0.0022 (13)	−0.0113 (14)
C10	0.0509 (16)	0.0337 (14)	0.0466 (13)	0.0015 (12)	0.0041 (12)	−0.0002 (11)
C11	0.0507 (15)	0.0354 (13)	0.0434 (13)	0.0020 (11)	0.0035 (12)	0.0024 (11)
C12	0.0575 (18)	0.0449 (16)	0.0580 (15)	0.0025 (13)	−0.0003 (13)	−0.0065 (13)
C13	0.0576 (18)	0.0522 (17)	0.0734 (18)	0.0142 (14)	0.0034 (15)	−0.0049 (15)
C14	0.0517 (17)	0.0560 (17)	0.0566 (16)	0.0039 (14)	−0.0011 (13)	0.0107 (14)
C15	0.068 (2)	0.0585 (17)	0.0493 (15)	0.0000 (15)	−0.0052 (14)	−0.0038 (13)
C16	0.0580 (17)	0.0470 (16)	0.0512 (15)	0.0085 (13)	0.0028 (13)	−0.0035 (12)
C17	0.069 (2)	0.121 (3)	0.081 (2)	0.000 (2)	−0.0258 (18)	0.008 (2)

Geometric parameters (Å, °)

O1—C1	1.353 (3)	C7—H7B	0.9600
O1—H1	0.8200	C7—H7C	0.9600
O2—C10	1.231 (3)	C8—C9	1.494 (3)
O3—C14	1.371 (3)	C9—H9A	0.9600
O3—C17	1.426 (4)	C9—H9B	0.9600
N1—C8	1.289 (3)	C9—H9C	0.9600
N1—N2	1.385 (3)	C10—C11	1.478 (3)
N2—C10	1.355 (3)	C11—C16	1.384 (3)
N2—H2	0.8600	C11—C12	1.392 (3)
C1—C6	1.386 (4)	C12—C13	1.370 (4)
C1—C2	1.408 (3)	C12—H12	0.9300
C2—C3	1.397 (3)	C13—C14	1.384 (4)
C2—C8	1.472 (3)	C13—H13	0.9300

C3—C4	1.380 (4)	C14—C15	1.373 (4)
C3—H3	0.9300	C15—C16	1.378 (3)
C4—C5	1.377 (4)	C15—H15	0.9300
C4—C7	1.507 (4)	C16—H16	0.9300
C5—C6	1.369 (4)	C17—H17A	0.9600
C5—H5	0.9300	C17—H17B	0.9600
C6—H6	0.9300	C17—H17C	0.9600
C7—H7A	0.9600		
C1—O1—H1	109.5	C8—C9—H9A	109.5
C14—O3—C17	117.3 (2)	C8—C9—H9B	109.5
C8—N1—N2	119.5 (2)	H9A—C9—H9B	109.5
C10—N2—N1	116.34 (19)	C8—C9—H9C	109.5
C10—N2—H2	121.8	H9A—C9—H9C	109.5
N1—N2—H2	121.8	H9B—C9—H9C	109.5
O1—C1—C6	116.8 (2)	O2—C10—N2	120.4 (2)
O1—C1—C2	123.4 (2)	O2—C10—C11	122.2 (2)
C6—C1—C2	119.7 (2)	N2—C10—C11	117.3 (2)
C3—C2—C1	116.8 (2)	C16—C11—C12	118.1 (2)
C3—C2—C8	121.6 (2)	C16—C11—C10	118.4 (2)
C1—C2—C8	121.6 (2)	C12—C11—C10	123.4 (2)
C4—C3—C2	123.8 (3)	C13—C12—C11	120.7 (2)
C4—C3—H3	118.1	C13—C12—H12	119.7
C2—C3—H3	118.1	C11—C12—H12	119.7
C5—C4—C3	117.1 (3)	C12—C13—C14	120.2 (2)
C5—C4—C7	121.2 (3)	C12—C13—H13	119.9
C3—C4—C7	121.6 (3)	C14—C13—H13	119.9
C6—C5—C4	121.7 (3)	O3—C14—C15	124.5 (3)
C6—C5—H5	119.2	O3—C14—C13	115.5 (2)
C4—C5—H5	119.2	C15—C14—C13	120.0 (3)
C5—C6—C1	120.8 (3)	C14—C15—C16	119.4 (3)
C5—C6—H6	119.6	C14—C15—H15	120.3
C1—C6—H6	119.6	C16—C15—H15	120.3
C4—C7—H7A	109.5	C15—C16—C11	121.5 (2)
C4—C7—H7B	109.5	C15—C16—H16	119.2
H7A—C7—H7B	109.5	C11—C16—H16	119.2
C4—C7—H7C	109.5	O3—C17—H17A	109.5
H7A—C7—H7C	109.5	O3—C17—H17B	109.5
H7B—C7—H7C	109.5	H17A—C17—H17B	109.5
N1—C8—C2	115.3 (2)	O3—C17—H17C	109.5
N1—C8—C9	123.6 (2)	H17A—C17—H17C	109.5
C2—C8—C9	121.1 (2)	H17B—C17—H17C	109.5
C8—N1—N2—C10	−153.5 (2)	C1—C2—C8—C9	177.5 (2)
O1—C1—C2—C3	179.1 (2)	N1—N2—C10—O2	6.6 (3)
C6—C1—C2—C3	−0.5 (3)	N1—N2—C10—C11	−177.19 (18)
O1—C1—C2—C8	0.2 (4)	O2—C10—C11—C16	−26.0 (3)
C6—C1—C2—C8	−179.3 (2)	N2—C10—C11—C16	157.8 (2)
C1—C2—C3—C4	0.7 (4)	O2—C10—C11—C12	149.4 (2)
C8—C2—C3—C4	179.6 (2)	N2—C10—C11—C12	−26.7 (3)

C2—C3—C4—C5	−0.7 (4)	C16—C11—C12—C13	1.5 (4)
C2—C3—C4—C7	−179.7 (2)	C10—C11—C12—C13	−174.0 (2)
C3—C4—C5—C6	0.3 (4)	C11—C12—C13—C14	−1.1 (4)
C7—C4—C5—C6	179.4 (3)	C17—O3—C14—C15	4.8 (4)
C4—C5—C6—C1	−0.1 (4)	C17—O3—C14—C13	−173.8 (2)
O1—C1—C6—C5	−179.4 (2)	C12—C13—C14—O3	177.9 (2)
C2—C1—C6—C5	0.2 (4)	C12—C13—C14—C15	−0.8 (4)
N2—N1—C8—C2	179.61 (17)	O3—C14—C15—C16	−176.3 (2)
N2—N1—C8—C9	1.4 (3)	C13—C14—C15—C16	2.3 (4)
C3—C2—C8—N1	−179.5 (2)	C14—C15—C16—C11	−1.8 (4)
C1—C2—C8—N1	−0.7 (3)	C12—C11—C16—C15	0.0 (4)
C3—C2—C8—C9	−1.2 (3)	C10—C11—C16—C15	175.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.81	2.528 (3)	145
N2—H2···O2^{i}	0.86	2.17	2.903 (1)	142

Symmetry codes: (i) *x*, *y*+1, *z*.

Fig. 1

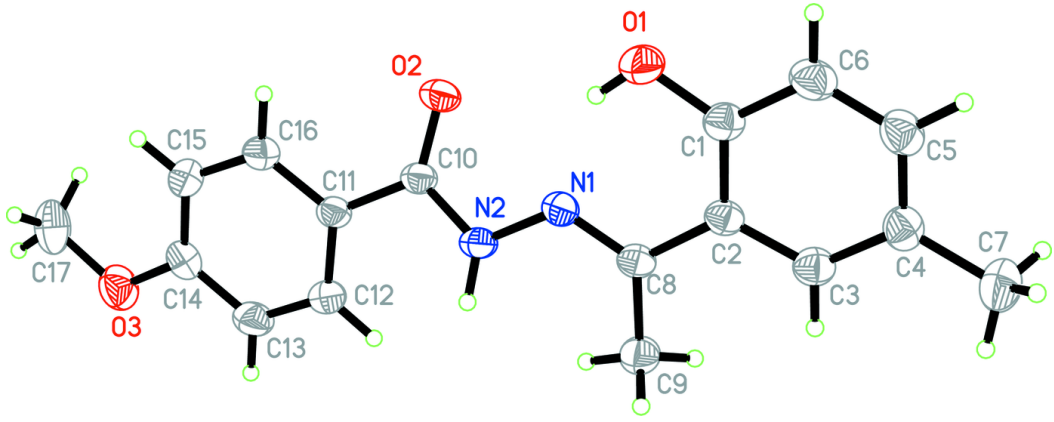
O1
O2
C6
C15
C16
C10
C1
C5
C11
N2
N1
C17
C2
C7
C14
C8
C4
C12
C3
O3
C13
C9

Fig. 2