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2-[((*E*)-2-{2-[(*E*)-2-Hydroxybenzylidene]-hydrazinecarbonyl}hydrazinylidene)-methyl]phenol

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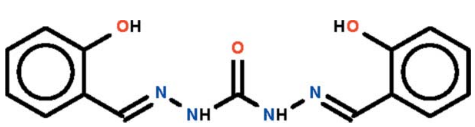
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.027; wR factor = 0.075; data-to-parameter ratio = 6.9.

The molecule of the title compound, $C_{15}H_{14}N_4O_3$, is completed by the application of crystallographic twofold symmetry, with the carbonyl group lying on the rotation axis. The molecule is close to planar: the greatest deviation of a torsion angle from 0° is 7.3 (2)° about the bond linking the phenol ring to the rest of the molecule. An intramolecular O—H···N(imine) hydrogen bond is formed in each half of the molecule. The carbonyl O atom is *anti* with respect to the amine H atoms and this allows for the formation of N—H···O(hydroxyl) hydrogen bonds in the crystal, which results in supramolecular layers lying parallel to (100).

Related literature

For the structures of related carbohydrazides, see: Bikas *et al.* (2010*a*,*b*).



Experimental

Crystal data

$C_{15}H_{14}N_4O_3$
M_r = 298.30
Orthorhombic, $Aba2$
a = 14.3101 (4) Å
b = 9.3620 (2) Å
c = 10.2697 (2) Å
V = 1375.84 (6) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.86 mm^{-1}
T = 100 K
0.25 × 0.25 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.342, T_{max} = 1.000
2323 measured reflections
757 independent reflections
750 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.075
S = 1.10
757 reflections
110 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.16 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
O2—H2···N2	0.86 (3)	1.79 (4)	2.5710 (17)	150 (3)
N1—H1···O2^i	0.89 (3)	2.12 (3)	2.983 (2)	161 (2)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001) and *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The authors are grateful to the Islamic Azad University (Tabriz Branch), the University of Zanjan and the University of Malaya for support of this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6561).

References

Agilent (2010). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bikas, R., Hosseini Monfared, H., Bijanzad, K., Koroglu, A. & Kazak, C. (2010*a*). *Acta Cryst.* E**66**, o2073.
Bikas, R., Hosseini Monfared, H., Kazak, C., Arslan, N. B. & Bijanzad, K. (2010*b*). *Acta Cryst.* E**66**, o2015.
Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

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supplementary materials

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2-[((*E*)-2-{2-[(*E*)-2-Hydroxybenzylidene]hydrazinecarbonyl}hydrazinylidene)methyl]phenol

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Comment

In connection with previous structural studies of carbohydrazide derivatives (Bikas *et al.*, 2010*a*,*b*), the title compound, (I), was investigated. The molecule, Fig. 1, has crystallographic twofold symmetry. The molecule is essentially planar with a r.m.s. deviation for all 22 atoms comprising the full molecule being 0.074 Å. The maximum deviation from 0° for a torsion angle in the molecule is 7.3 (2)° for N2—C2—C3—C8. The carbonyl-O atom is *anti* with respect to the amine-H atoms, and the conformation about the C2═N2 imine bond [1.2857 (19) Å] is *E*.The hydroxyl-H atom forms an intramolecular hydrogen bond to the imine-H atom, Table 1.

In the crystal, the amine-H atoms form hydrogen bonds to the hydroxyl-O atoms to form supramolecular layers parallel to (100), Fig. 2 and Table 1.

Experimental

All reagents were commercially available and used as received. A methanol (10 ml) solution of 2-hydroxybenzaldehyde (3 mmol) was added drop-wise to a methanol solution (10 ml) of carbohydrazide (1.5 mmol), and the mixture was refluxed for 3 h. Then the solution was evaporated on a steam bath to 5 ml and cooled to room temperature. White precipitates of the title compound were separated and filtered off, washed with cooled methanol (3 ml) and then dried in air. Crystals of the title compound were obtained from its methanol solution by slow solvent evaporation. Yield: 94%. *M*.pt. 496–497 K. Selected IR data (cm^{-1}): 3272 (v. broad, N—H), 1721 (C═O); 1625 (s, C═N(azomethine)); 959 (m, N—N); 1353, 1273 (s, C—O).

Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H = 0.95 Å, U_{iso}(H) = 1.2 U_{eq}(C)] and were included in the refinement in the riding model approximation. The hydroxyl and amino H-atoms were refined freely. In the absence of significant anomalous scattering effects, 242 Friedel pairs were averaged in the final refinement.

Figures

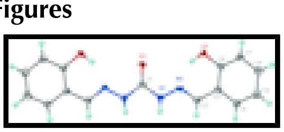

Fig. 1. Molecular structure of (I) showing displacement ellipsoids at the 70% probability level. The molecule has twofold symmetry and the unlabelled atoms are related by the symmetry operation 1 - *x*, 1 - *y*, *z*.

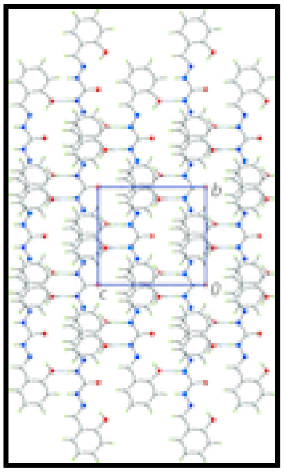

Fig. 2. A view of the supramolecular layer parallel to (100) in (I). The N—H···O hydrogen bonds are shown as blue dashed lines.

2-[((*E*)-2-{2-[(*E*)-2- Hydroxybenzylidene]hydrazinecarbonyl}hydrazinylidene)methyl]phenol

Crystal data

$C_{15}H_{14}N_4O_3$	$F(000) = 624$
$M_r = 298.30$	$D_x = 1.440$ Mg m^{-3}
Orthorhombic, *Aba*2	Cu *K*α radiation, λ = 1.54178 Å
Hall symbol: A 2 -2ac	Cell parameters from 1858 reflections
$a = 14.3101$ (4) Å	θ = 3.1–76.5°
$b = 9.3620$ (2) Å	μ = 0.86 mm^{-1}
$c = 10.2697$ (2) Å	$T = 100$ K
$V = 1375.84$ (6) Å^3	Prism, colourless
$Z = 4$	0.25 × 0.25 × 0.10 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector	757 independent reflections
Radiation source: SuperNova (Cu) X-ray Source	750 reflections with $I > 2\sigma(I)$
Mirror	$R_{int} = 0.014$
Detector resolution: 10.4041 pixels mm^{-1}	$\theta_{max} = 76.7°$, $\theta_{min} = 6.2°$
ω scan	$h = -17\rightarrow17$
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)	$k = -11\rightarrow10$
$T_{min} = 0.342$, $T_{max} = 1.000$	$l = -10\rightarrow12$
2323 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.027$	H atoms treated by a mixture of independent and constrained refinement

$wR(F^2) = 0.075$	$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.2173P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
757 reflections	$\Delta\rho_{max} = 0.16$ e Å^{-3}
110 parameters	$\Delta\rho_{min} = -0.21$ e Å^{-3}
1 restraint	Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0051 (7)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.5000	0.5000	0.50067 (16)	0.0218 (4)
O2	0.59665 (8)	0.87891 (12)	0.43307 (12)	0.0197 (3)
N1	0.52439 (9)	0.61383 (13)	0.69670 (15)	0.0186 (3)
N2	0.55917 (9)	0.73346 (13)	0.63891 (13)	0.0170 (3)
C1	0.5000	0.5000	0.6191 (2)	0.0174 (4)
C2	0.57681 (11)	0.84255 (16)	0.71089 (15)	0.0172 (3)
H2A	0.5646	0.8395	0.8018	0.021*
C3	0.61574 (10)	0.97076 (15)	0.65176 (15)	0.0159 (4)
C4	0.64432 (11)	1.08454 (17)	0.73122 (16)	0.0191 (4)
H4	0.6380	1.0769	0.8230	0.023*
C5	0.68171 (11)	1.20828 (18)	0.67764 (18)	0.0211 (4)
H5	0.7009	1.2847	0.7324	0.025*
C6	0.69086 (11)	1.21928 (18)	0.54321 (19)	0.0219 (4)
H6	0.7165	1.3036	0.5062	0.026*
C7	0.66290 (11)	1.10842 (16)	0.46270 (15)	0.0195 (4)
H7	0.6698	1.1169	0.3710	0.023*
C8	0.62482 (9)	0.98474 (16)	0.51579 (14)	0.0159 (4)
H1	0.5006 (17)	0.614 (2)	0.777 (3)	0.023 (5)*
H2	0.579 (2)	0.809 (3)	0.481 (3)	0.051 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

O1	0.0307 (8)	0.0208 (7)	0.0138 (8)	−0.0031 (6)	0.000	0.000
O2	0.0260 (6)	0.0186 (5)	0.0146 (5)	−0.0020 (4)	−0.0006 (5)	−0.0009 (4)
N1	0.0253 (7)	0.0169 (7)	0.0136 (6)	−0.0027 (5)	0.0024 (5)	0.0014 (5)
N2	0.0183 (6)	0.0166 (7)	0.0160 (7)	0.0001 (5)	−0.0004 (5)	0.0014 (5)
C1	0.0181 (10)	0.0175 (10)	0.0167 (10)	0.0017 (7)	0.000	0.000
C2	0.0176 (6)	0.0190 (7)	0.0149 (7)	0.0010 (5)	−0.0001 (5)	0.0002 (6)
C3	0.0147 (7)	0.0180 (7)	0.0150 (7)	0.0022 (6)	−0.0004 (6)	0.0005 (6)
C4	0.0181 (7)	0.0219 (8)	0.0174 (8)	0.0007 (6)	−0.0004 (6)	−0.0010 (7)
C5	0.0195 (7)	0.0197 (8)	0.0241 (8)	−0.0025 (5)	0.0002 (6)	−0.0040 (6)
C6	0.0186 (8)	0.0209 (8)	0.0261 (9)	−0.0031 (5)	0.0009 (7)	0.0035 (7)
C7	0.0190 (7)	0.0230 (7)	0.0166 (8)	−0.0003 (6)	0.0009 (6)	0.0027 (6)
C8	0.0136 (7)	0.0181 (7)	0.0161 (9)	0.0016 (5)	−0.0009 (6)	−0.0003 (6)

Geometric parameters (Å, °)

O1—C1	1.217 (3)	C3—C4	1.403 (2)
O2—C8	1.3660 (19)	C3—C8	1.408 (2)
O2—H2	0.86 (3)	C4—C5	1.390 (2)
N1—N2	1.3617 (16)	C4—H4	0.9500
N1—C1	1.3754 (19)	C5—C6	1.391 (3)
N1—H1	0.89 (3)	C5—H5	0.9500
N2—C2	1.2857 (19)	C6—C7	1.386 (2)
C1—N1^{i}	1.3754 (19)	C6—H6	0.9500
C2—C3	1.456 (2)	C7—C8	1.391 (2)
C2—H2A	0.9500	C7—H7	0.9500
C8—O2—H2	106 (2)	C5—C4—H4	119.5
N2—N1—C1	118.53 (14)	C3—C4—H4	119.5
N2—N1—H1	122.6 (13)	C4—C5—C6	119.41 (17)
C1—N1—H1	116.2 (14)	C4—C5—H5	120.3
C2—N2—N1	118.33 (13)	C6—C5—H5	120.3
O1—C1—N1	125.38 (10)	C7—C6—C5	120.64 (16)
O1—C1—N1^{i}	125.38 (10)	C7—C6—H6	119.7
N1—C1—N1^{i}	109.2 (2)	C5—C6—H6	119.7
N2—C2—C3	119.35 (14)	C6—C7—C8	120.17 (15)
N2—C2—H2A	120.3	C6—C7—H7	119.9
C3—C2—H2A	120.3	C8—C7—H7	119.9
C4—C3—C8	118.64 (14)	O2—C8—C7	118.40 (14)
C4—C3—C2	119.67 (14)	O2—C8—C3	121.46 (14)
C8—C3—C2	121.69 (14)	C7—C8—C3	120.14 (14)
C5—C4—C3	120.99 (16)		
C1—N1—N2—C2	176.05 (12)	C4—C5—C6—C7	0.1 (2)
N2—N1—C1—O1	−5.97 (14)	C5—C6—C7—C8	0.3 (2)
N2—N1—C1—N1^{i}	174.03 (14)	C6—C7—C8—O2	178.90 (13)
N1—N2—C2—C3	178.82 (12)	C6—C7—C8—C3	−0.9 (2)
N2—C2—C3—C4	−173.31 (13)	C4—C3—C8—O2	−178.76 (13)
N2—C2—C3—C8	7.3 (2)	C2—C3—C8—O2	0.6 (2)
C8—C3—C4—C5	−0.6 (2)	C4—C3—C8—C7	1.1 (2)
C2—C3—C4—C5	−179.99 (13)	C2—C3—C8—C7	−179.58 (13)

C3—C4—C5—C6	0.0 (2)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···N2	0.86 (3)	1.79 (4)	2.5710 (17)	150 (3)
N1—H1···O2^{ii}	0.89 (3)	2.12 (3)	2.983 (2)	161 (2)

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

Fig. 1

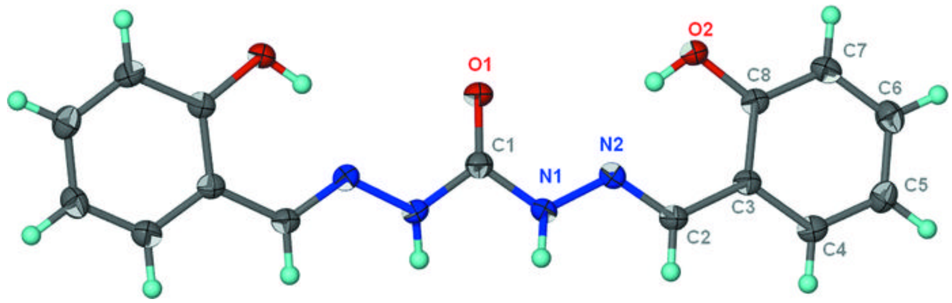

Fig. 2