Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1,5-Bis(2-hydroxy-3-methoxybenzylidene)carbonohydrazide methanol 0.47-solvate

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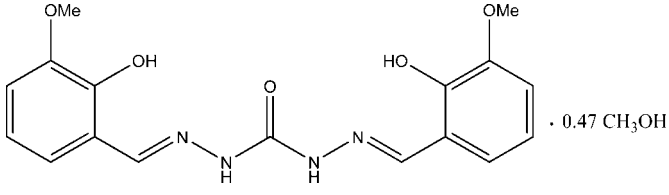
Received 11 February 2014; accepted 2 March 2014

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; disorder in main residue; R factor = 0.044; wR factor = 0.111; data-to-parameter ratio = 5.9.

In the title compound, $C_{17}H_{18}N_4O_5\cdot0.47CH_3OH$, the virtually planar (r.m.s. deviation = 0.128 Å) carbonohydrazide molecule is located on a twofold axis and conformation of its C=N bonds is *E*. There are short intramolecular O—H···N hydrogen bonds between the hydroxy groups and hydrazide N atoms. In the crystal, bifurcated N—H···(O,O) hydrogen bonds assemble the carbonohydrazide molecules into a three-dimensional network. There are C_2 symmetric voids in this network, 47% of which are occupied by disordered methanol molecules.

Related literature

For related structures, see: Du & Zhang (2010); He *et al.* (2010); Kong *et al.* (2010). For the biological activity of carbonohydrazides, see: Bacchi *et al.* (1999); El-Gammal *et al.* (2012).



Experimental

Crystal data

$C_{17}H_{18}N_4O_5\cdot0.47CH_4O$
M_r = 373.40
Orthorhombic, *Fdd*2
a = 9.4470 (7) Å
b = 17.5850 (9) Å
c = 22.8714 (12) Å
V = 3799.5 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.1 × 0.08 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
9573 measured reflections
862 independent reflections
658 reflections with $I > 2\sigma(I)$
R_{int} = 0.105
2 standard reflections every 120 min
intensity decay: 2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.111
S = 1.25
862 reflections
146 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*O*···N1	0.91 (5)	1.86 (5)	2.703 (5)	152 (5)
N2—H2*N*···O3^{i}	0.94 (6)	2.38 (5)	3.044 (6)	128 (4)
N2—H2*N*···O1^{i}	0.94 (6)	2.33 (6)	3.204 (6)	155 (4)

Symmetry code: (i) $x+\frac{1}{4}, -y+\frac{1}{4}, z+\frac{1}{4}$.

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick,2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97*.

Supporting information for this paper is available from the IUCr electronic archives (Reference: GK2603).

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supporting information

Acta Cryst. (2014). E**70**, o423 [doi:10.1107/S1600536814004802]

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S1. Comment

Carbonohydrazide derivatives give rise to a large spectrum of biological properties such as antioxidant (El-Gammal *et al.*, 2012) and anticancer activities (Bacchi *et al.*, 1999). We report here the crystal structure of the title compound synthesized according to literature (He *et al.*, 2010; Du *et al.*, 2010). All parameters are within normal ranges and comparable with the related structures (Kong *et al.*, 2010).

The molecular structure of the title compound is shown in Fig. 1. The complete carbonohydrazide molecule is generated by a twofold crystallographic axis passing throuth the atoms C8 and O2. A three-center O···(N)H···O intermolecular hydrogen bond involving the amido H atoms and the phenoxo and methoxy O atoms is observed (Fig. 2). There are voids in a three dimensional network containing solvent methanol molecules. Only one methanol molecule can be accommodated in a small void that has C_2 symmetry. This leads to disorder of methanol molecules. In addition refinement of occupancy factors of methanol O and C atoms converged at 0.234 (1), indicating that 47% of voids are occupied by the solvent.

S2. Experimental

In a round bottomed flask, carbonohydrazide (1.0 g, 11.11 mmol) was introduced with methanol (10 ml). *o*-Vanillin (3.3 g, 22.22 mmol) dissolved in 10 ml of the same solvent was added. Two drops of glacial acetic acid were added while stirring. After one hour under reflux, the precipitate formed that after cooling to room temperature was filtered off and washed with cold methanol. The resulting solid was dried in air. The filtrate was left at room temperature. Slow evaporation of the solvent gave colorless crystals after one day. Yield: 95%; m.p. 378 K. Anal. Calc. for [$C_{17}H_{18}N_4O_5$] (%): C, 56.98; H, 5.06, N, 15.63. Found: C, 56.96; H, 5.04; N, 15.60. Selected IR data (cm^{-1}, KBr pellet): 3291, 2942, 1696, 1553, 1200, 1167. ^{1}H-NMR (DMSO-d6) δ: 3.8 (s, 6H, O—CH3); 6.7 – 7.1 (m, 6H, $H_{Aromatic}$); 8.5 (s, 2H, H—C═N); 7.3 (s, 1H, H—N); 11 (s, 2H, H—O) p.p.m. ^{13}C-NMR (DMSO-d_6) d: 151.8 (C═O); 147.8, 146.1, 119.5, 119.4, 118.8, 112.8 ($C_{Aromatic}$); 58,7 (–O—CH_3).

S3. Refinement

H atoms of the NH and OH groups were located in the Fourier difference maps and refined without restraints. Otherg H atoms were geometrically optimized and refined as riding on their carriers with U_{iso}(H) = 1.2U_{eq}(C)(1.5 for CH_3 groups). Considerable disorder was detected for the solvent methanol molecule. The occupancy factor of the C and O atoms of methanol refined at 0.234 (1). Thus, there are 0.46 methanol molecules per one carbonohydrazide molecule in the crystal. Owing to a negligible anomalous dispersion effect the Friedel pairs were merged and the absolute structure was not determined.

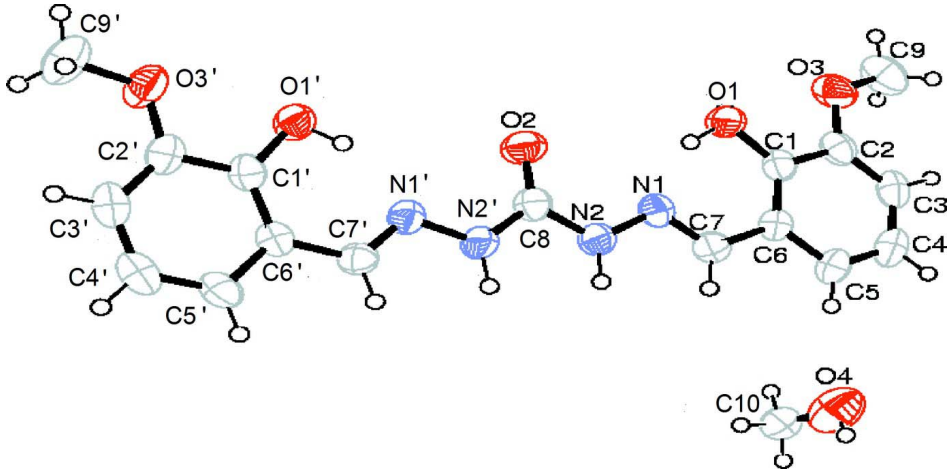

Figure 1

An *ORTEP* view of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are plotted at the 50% probability level. Only one position of the disordered solvent methanol molecule is shown for clarity. The symmetry code for generating primed atoms is 2-x,-y,z

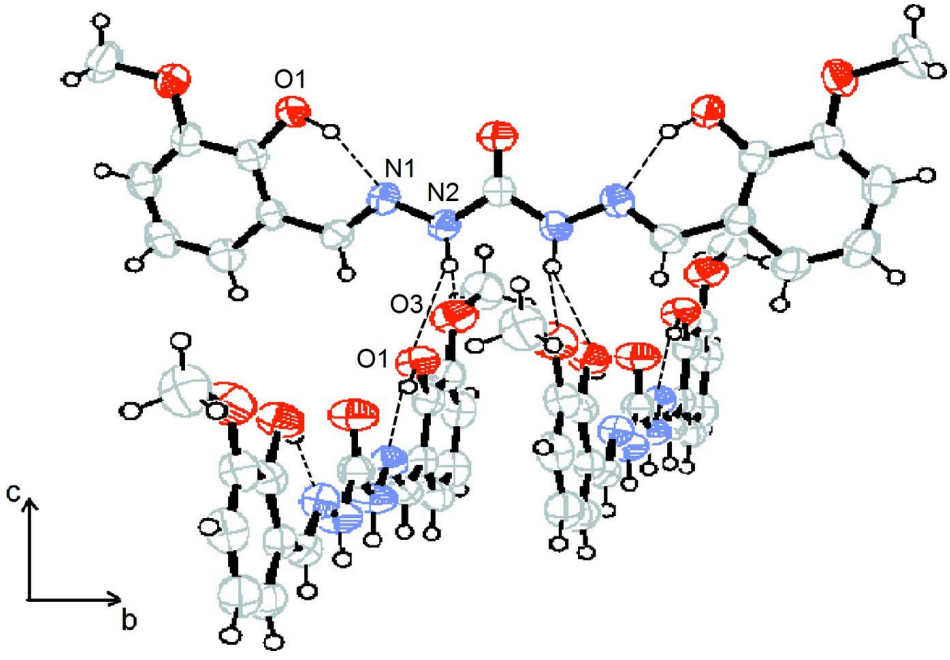

Figure 2

Intramoleculr and intermolecular hydrogen bonds. Solvent methanol molecules are omitted as they do not form hydrogen bonds.

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Crystal data

$C_{17}H_{18}N_4O_5 \cdot 0.47CH_4O$
$M_r = 373.40$
Orthorhombic, *Fdd*2
Hall symbol: F 2 -2d

$a = 9.4470\ (7)$ Å
$b = 17.5850\ (9)$ Å
$c = 22.8714\ (12)$ Å
$V = 3799.5\ (4)$ Å^3

$Z = 8$
$F(000) = 1571.4$
$D_x = 1.306$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections

$\theta = 11–15°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Prismatic, colorless
0.1 × 0.08 × 0.05 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
non–profiled $\omega/2\theta$ scans
9573 measured reflections
862 independent reflections
658 reflections with $I > 2\sigma(I)$

$R_{int} = 0.105$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.6°$
$h = -11 \rightarrow 11$
$k = -1 \rightarrow 20$
$l = -27 \rightarrow 27$
2 standard reflections every 120 min
intensity decay: 2%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.111$
$S = 1.25$
862 reflections
146 parameters
1 restraint
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0306P)^2 + 5.2614P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ 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}	Occ. (<1)
O1	0.6805 (4)	0.1635 (2)	0.02775 (15)	0.0559 (10)	
O2	1.0000	0.0000	0.0525 (2)	0.0641 (14)	
O3	0.4920 (4)	0.2637 (2)	−0.00424 (17)	0.0680 (12)	
N1	0.8123 (4)	0.0896 (2)	0.11591 (18)	0.0471 (10)	
N2	0.9127 (5)	0.0436 (2)	0.1418 (2)	0.0533 (11)	
C1	0.6048 (5)	0.1974 (3)	0.0728 (2)	0.0459 (12)	
C2	0.5016 (5)	0.2512 (3)	0.0560 (2)	0.0512 (13)	
C3	0.4200 (5)	0.2864 (3)	0.0996 (3)	0.0567 (14)	
H3	0.3505	0.3211	0.0888	0.068*	
C4	0.4409 (5)	0.2704 (3)	0.1594 (3)	0.0595 (15)	

H4	0.3856	0.2946	0.1874	0.071*	
C5	0.5434 (5)	0.2189 (3)	0.1765 (2)	0.0528 (13)	
H5	0.5580	0.2087	0.2159	0.063*	
C6	0.6269 (5)	0.1813 (3)	0.1329 (2)	0.0423 (11)	
C7	0.7364 (5)	0.1284 (3)	0.1531 (2)	0.0459 (12)	
H7	0.7519	0.1223	0.1930	0.055*	
C8	1.0000	0.0000	0.1068 (3)	0.0467 (17)	
C9	0.4052 (7)	0.3261 (4)	−0.0244 (3)	0.0779 (19)	
H9A	0.4081	0.3283	−0.0663	0.117*	
H9B	0.4405	0.3729	−0.0086	0.117*	
H9C	0.3093	0.3185	−0.0118	0.117*	
O4	0.579 (2)	0.1913 (12)	0.3182 (9)	0.098 (9)	0.234 (11)
H1M	0.5601	0.1599	0.3453	0.148*	0.234 (11)
C10	0.703 (3)	0.2337 (18)	0.3207 (10)	0.075 (10)	0.234 (11)
H10A	0.7221	0.2638	0.3554	0.113*	0.234 (11)
H10B	0.7221	0.2638	0.2861	0.113*	0.234 (11)
H10C	0.7632	0.1890	0.3207	0.113*	0.234 (11)
H1O	0.743 (5)	0.134 (3)	0.048 (2)	0.057 (15)*	
H2N	0.916 (5)	0.040 (3)	0.183 (3)	0.052 (15)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.062 (2)	0.055 (2)	0.051 (2)	0.0187 (18)	−0.0048 (18)	−0.0067 (17)
O2	0.077 (4)	0.069 (3)	0.047 (3)	0.019 (3)	0.000	0.000
O3	0.076 (3)	0.063 (2)	0.066 (3)	0.026 (2)	−0.0196 (19)	−0.001 (2)
N1	0.043 (2)	0.045 (2)	0.053 (2)	0.004 (2)	−0.0026 (19)	0.0038 (19)
N2	0.054 (3)	0.059 (2)	0.047 (3)	0.020 (2)	−0.001 (2)	0.004 (2)
C1	0.038 (3)	0.044 (2)	0.056 (3)	0.000 (2)	−0.002 (2)	−0.005 (2)
C2	0.046 (3)	0.042 (2)	0.065 (3)	0.004 (2)	−0.010 (3)	0.001 (3)
C3	0.039 (3)	0.049 (3)	0.082 (4)	0.006 (2)	−0.001 (3)	−0.005 (3)
C4	0.047 (3)	0.052 (3)	0.080 (4)	0.002 (3)	0.017 (3)	−0.008 (3)
C5	0.046 (3)	0.053 (3)	0.059 (3)	−0.002 (2)	0.014 (2)	0.002 (3)
C6	0.039 (3)	0.035 (2)	0.052 (3)	−0.001 (2)	0.004 (2)	−0.002 (2)
C7	0.047 (3)	0.044 (3)	0.046 (3)	−0.001 (2)	0.000 (2)	0.006 (2)
C8	0.047 (4)	0.040 (4)	0.053 (5)	0.003 (3)	0.000	0.000
C9	0.082 (4)	0.058 (3)	0.094 (5)	0.015 (3)	−0.025 (4)	0.014 (3)
O4	0.117 (19)	0.087 (15)	0.091 (17)	0.004 (12)	0.030 (13)	0.010 (12)
C10	0.07 (2)	0.10 (3)	0.057 (16)	0.021 (19)	−0.001 (11)	−0.018 (15)

Geometric parameters (Å, º)

O1—C1	1.389 (6)	C4—H4	0.9300
O1—H1O	0.91 (5)	C5—C6	1.433 (7)
O2—C8	1.243 (8)	C5—H5	0.9300
O3—C2	1.397 (6)	C6—C7	1.466 (6)
O3—C9	1.445 (6)	C7—H7	0.9300
N1—C7	1.305 (6)	C8—N2^{i}	1.381 (6)

N1—N2	1.381 (5)	C9—H9A	0.9600
N2—C8	1.381 (6)	C9—H9B	0.9600
N2—H2N	0.94 (6)	C9—H9C	0.9600
C1—C2	1.413 (6)	O4—C10	1.39 (3)
C1—C6	1.418 (7)	O4—H1M	0.8500
C2—C3	1.405 (8)	C10—C10^{ii}	1.06 (5)
C3—C4	1.411 (8)	C10—H10A	0.9700
C3—H3	0.9300	C10—H10B	0.9700
C4—C5	1.382 (7)	C10—H10C	0.9700
C1—O1—H1O	102 (3)	N1—C7—C6	121.0 (4)
C2—O3—C9	118.1 (4)	N1—C7—H7	119.5
C7—N1—N2	113.9 (4)	C6—C7—H7	119.5
N1—N2—C8	119.1 (5)	O2—C8—N2	125.4 (3)
N1—N2—H2N	120 (3)	O2—C8—N2^{i}	125.4 (3)
C8—N2—H2N	121 (3)	N2—C8—N2^{i}	109.2 (7)
O1—C1—C2	116.1 (5)	O3—C9—H9A	109.5
O1—C1—C6	123.9 (4)	O3—C9—H9B	109.5
C2—C1—C6	120.0 (5)	H9A—C9—H9B	109.5
O3—C2—C3	126.5 (5)	O3—C9—H9C	109.5
O3—C2—C1	114.8 (5)	H9A—C9—H9C	109.5
C3—C2—C1	118.7 (5)	H9B—C9—H9C	109.5
C2—C3—C4	121.7 (5)	C10—O4—H1M	119.9
C2—C3—H3	119.2	C10^{ii}—C10—O4	177.6 (17)
C4—C3—H3	119.2	C10^{ii}—C10—H10A	63.1
C5—C4—C3	120.2 (5)	O4—C10—H10A	118.9
C5—C4—H4	119.9	C10^{ii}—C10—H10B	63.1
C3—C4—H4	119.9	O4—C10—H10B	114.5
C4—C5—C6	119.4 (5)	H10A—C10—H10B	109.6
C4—C5—H5	120.3	C10^{ii}—C10—H10C	87.1
C6—C5—H5	120.3	O4—C10—H10C	93.3
C1—C6—C5	120.1 (4)	H10A—C10—H10C	109.6
C1—C6—C7	122.3 (4)	H10B—C10—H10C	109.6
C5—C6—C7	117.5 (4)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···N1	0.91 (5)	1.86 (5)	2.703 (5)	152 (5)
N2—H2*N*···O3^{iii}	0.94 (6)	2.38 (5)	3.044 (6)	128 (4)
N2—H2*N*···O1^{iii}	0.94 (6)	2.33 (6)	3.204 (6)	155 (4)

Symmetry code: (iii) *x*+1/4, −*y*+1/4, *z*+1/4.