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$N^2,N^{2'}$-Bis(2-hydroxy-3-methoxybenzylidene)terephthalohydrazide dihydrate

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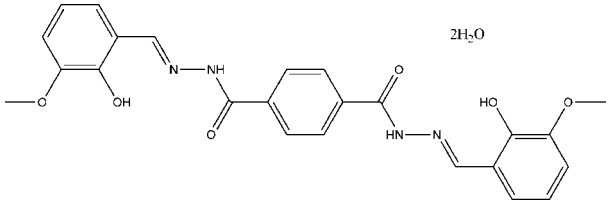
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.055; wR factor = 0.133; data-to-parameter ratio = 12.6.

The organic molecule of the title compound, $C_{24}H_{22}N_4O_6\cdot 2H_2O$, is centrosymmetric. The dihedral angle between the two benzene rings is 3.25 (7)°. The structure is stabilized by intramolecular O—H···O, and intermolecular N—H···O and O—H···O hydrogen bonds, forming an intricate three-dimensional network.

Related literature

For the structures of some organotin(IV) complexes with the Schiff base *o*-vanillin-2-thiophenoylhydrazone, see Yin & Chen (2006).



Experimental

Crystal data

$C_{24}H_{22}N_4O_6\cdot 2H_2O$
M_r = 498.49
Monoclinic, $P2_1/c$
a = 7.2854 (12) Å
b = 25.711 (2) Å
c = 6.4780 (11) Å
β = 102.613 (2)°
V = 1184.1 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.28 × 0.17 × 0.14 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.971, T_{max} = 0.985
5724 measured reflections
2052 independent reflections
1003 reflections with $I > 2\sigma(I)$
R_{int} = 0.067

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.133
S = 1.00
2052 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O4^{i}	0.86	2.00	2.835 (3)	163
O4—H14···O1^{ii}	0.85	1.92	2.751 (3)	167
O2—H2···N2	0.82	1.89	2.599 (4)	145
O4—H13···O3	0.85	2.29	3.021 (3)	144
O4—H13···O2	0.85	2.41	3.099 (3)	139

Symmetry codes: (i) $x-1, y, z-1$; (ii) $x, y, z+1$.

Data collection: *SMART* (Siemens, 1996); 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: AT2490).

References

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supplementary materials

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N^2,$N^{2'}$-Bis(2-hydroxy-3-methoxybenzylidene)terephthalohydrazide dihydrate

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Comment

Recently, we have reported some organotin(IV) complexes with Schiff base of *o*-vanillin-2-thiophenoylhydrazone (Yin & Chen, 2006). As an extension of our work on the structural characterization of Schiff base compounds, the title compound, (I), is reported here (Fig. 1).

In Fig. 1, it can be viewed as a centrosymmetric configuration, where one half of the molecule comprises the crystallographic asymmetric unit and the other half is generated by an inversion centre. In the compound, the N2═C5 bond length of 1.283 (4) Å (Table 1) conforms to the value for a double bond, while the N1—C1 bond [1.360 (4) Å] and N1—N2 bond [1.376 (3) Å] are intermediate between a double bond and a single bond because of conjugation effects in the molecule. The two benzene rings make a dihedral angle of 3.2 °.

The occurrence O—H···O and N—H···–O hydrogen bonds (Table 2) results in the formation of an intricated three dimensionnal network (Fig. 2).

Experimental

Compound (I) was synthesized by the reaction of terephthalohydrazide (5 mmol) with 2-hydroxy-3-methoxybenzaldehyde (10 mmol). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of an ethanol solution.

Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic), C—H = 0.96 Å (methylaromatic) and N—H = 0.86 Å with U_{iso}(H) = 1.2 or 1.5U_{eq} (C or N). H atoms of water molecule were located in difference Fourier maps and included in the subsequent refinement using restraints [O—H = 0.85 (1)Å and H···H = 1.39 (2) Å] with U_{iso}(H) = 1.5U_{eq}(O).

Figures

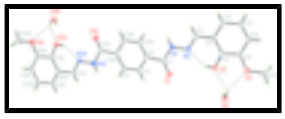

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme·The intramolecular hydrogen bonds are shown as dashed lines. [Symmetry code: (A) −*x*, −*y* + 1, −*z* − 1]

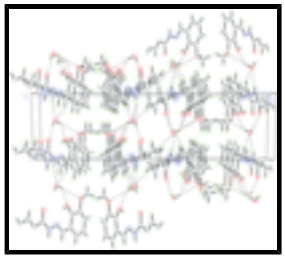

Fig. 2. The crystal packing of the title compound.

N^2,$N^{2'}$-Bis(2-hydroxy-3- methoxybenzylidene)terephthalohydrazide dihydrate

Crystal data

$C_{24}H_{22}N_4O_6 \cdot 2H_2O$	$F_{000} = 524$
$M_r = 498.49$	$D_x = 1.398$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1112 reflections
$a = 7.2854$ (12) Å	$\theta = 2.9$–27.6º
$b = 25.711$ (2) Å	$\mu = 0.11$ mm^{-1}
$c = 6.4780$ (11) Å	$T = 298$ (2) K
$\beta = 102.613$ (2)º	Block, colourless
$V = 1184.1$ (3) Å^3	0.28 × 0.17 × 0.14 mm
$Z = 2$	

Data collection

Siemens SMART CCD area-detector diffractometer	2052 independent reflections
Radiation source: fine-focus sealed tube	1003 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.067$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 1.6$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 8$
$T_{min} = 0.971$, $T_{max} = 0.985$	$k = -30 \rightarrow 28$
5724 measured reflections	$l = -7 \rightarrow 7$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.055$	H-atom parameters constrained
$wR(F^2) = 0.133$	$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} < 0.001$
2052 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}

163 parameters	$\Delta\rho_{min} = -0.22$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	−0.0134 (4)	0.57438 (9)	−0.0098 (4)	0.0298 (7)
H1	−0.1305	0.5725	−0.0711	0.036*
N2	0.0440 (4)	0.60098 (9)	0.1775 (4)	0.0299 (7)
O1	0.2850 (3)	0.55158 (9)	−0.0035 (4)	0.0475 (7)
O2	0.3017 (3)	0.63702 (8)	0.4869 (4)	0.0415 (7)
H2	0.2621	0.6214	0.3757	0.062*
O3	0.3871 (3)	0.68755 (8)	0.8377 (4)	0.0427 (7)
O4	0.5917 (3)	0.58503 (9)	0.8496 (4)	0.0513 (7)
H13	0.5422	0.6126	0.7893	0.077*
H14	0.5073	0.5706	0.9022	0.077*
C1	0.1207 (5)	0.55120 (12)	−0.0950 (5)	0.0306 (9)
C2	0.0522 (5)	0.52507 (11)	−0.3037 (5)	0.0286 (8)
C3	0.1867 (5)	0.50515 (12)	−0.4035 (6)	0.0367 (9)
H3	0.3134	0.5085	−0.3394	0.044*
C4	0.1356 (5)	0.48038 (12)	−0.5968 (5)	0.0343 (9)
H4	0.2283	0.4672	−0.6610	0.041*
C5	−0.0843 (5)	0.62087 (12)	0.2609 (5)	0.0354 (9)
H5	−0.2104	0.6168	0.1955	0.042*
C6	−0.0321 (5)	0.64951 (12)	0.4562 (5)	0.0282 (8)
C7	0.1552 (5)	0.65613 (11)	0.5602 (5)	0.0284 (8)
C8	0.1981 (5)	0.68380 (11)	0.7509 (5)	0.0302 (8)
C9	0.0568 (5)	0.70477 (12)	0.8354 (6)	0.0378 (9)
H9	0.0861	0.7234	0.9614	0.045*
C10	−0.1276 (5)	0.69803 (13)	0.7330 (6)	0.0402 (9)
H10	−0.2226	0.7122	0.7908	0.048*
C11	−0.1739 (5)	0.67075 (12)	0.5469 (6)	0.0369 (9)
H11	−0.2996	0.6663	0.4804	0.044*
C12	0.4402 (6)	0.71070 (15)	1.0399 (6)	0.0602 (12)
H12A	0.3977	0.7461	1.0325	0.090*
H12B	0.5747	0.7098	1.0860	0.090*
H12C	0.3844	0.6918	1.1385	0.090*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0271 (16)	0.0405 (17)	0.0199 (17)	−0.0041 (13)	0.0008 (13)	−0.0077 (14)
N2	0.0338 (18)	0.0308 (16)	0.0243 (18)	−0.0018 (13)	0.0046 (15)	−0.0073 (13)
O1	0.0263 (16)	0.0760 (19)	0.0380 (17)	−0.0021 (13)	0.0022 (14)	−0.0161 (14)
O2	0.0286 (15)	0.0549 (15)	0.0391 (16)	0.0065 (12)	0.0029 (13)	−0.0126 (12)
O3	0.0401 (16)	0.0468 (15)	0.0379 (16)	−0.0051 (12)	0.0013 (13)	−0.0162 (13)
O4	0.0289 (15)	0.0623 (16)	0.0606 (19)	0.0045 (12)	0.0054 (14)	0.0005 (15)

C1	0.036 (2)	0.032 (2)	0.025 (2)	−0.0032 (17)	0.0081 (19)	−0.0007 (17)
C2	0.031 (2)	0.0276 (18)	0.027 (2)	−0.0023 (15)	0.0053 (18)	−0.0008 (15)
C3	0.026 (2)	0.048 (2)	0.036 (2)	−0.0066 (17)	0.0047 (18)	−0.0127 (19)
C4	0.032 (2)	0.042 (2)	0.030 (2)	−0.0017 (16)	0.0083 (19)	−0.0098 (18)
C5	0.032 (2)	0.040 (2)	0.033 (2)	0.0029 (17)	0.0032 (18)	−0.0064 (18)
C6	0.030 (2)	0.0297 (19)	0.024 (2)	0.0040 (15)	0.0028 (18)	−0.0010 (16)
C7	0.035 (2)	0.0237 (18)	0.027 (2)	−0.0002 (16)	0.0092 (19)	0.0016 (16)
C8	0.033 (2)	0.0237 (18)	0.031 (2)	−0.0014 (16)	0.0009 (18)	0.0011 (16)
C9	0.047 (3)	0.039 (2)	0.027 (2)	−0.0007 (18)	0.006 (2)	−0.0081 (17)
C10	0.046 (3)	0.047 (2)	0.030 (2)	0.0123 (18)	0.013 (2)	−0.0019 (19)
C11	0.031 (2)	0.043 (2)	0.038 (2)	0.0076 (17)	0.0081 (19)	−0.0022 (18)
C12	0.059 (3)	0.083 (3)	0.032 (3)	−0.012 (2)	−0.004 (2)	−0.022 (2)

Geometric parameters (Å, °)

N1—C1	1.360 (4)	C4—C2^{i}	1.387 (4)
N1—N2	1.376 (3)	C4—H4	0.9300
N1—H1	0.8600	C5—C6	1.441 (4)
N2—C5	1.283 (4)	C5—H5	0.9300
O1—C1	1.214 (4)	C6—C7	1.394 (4)
O2—C7	1.352 (3)	C6—C11	1.406 (4)
O2—H2	0.8200	C7—C8	1.400 (4)
O3—C8	1.372 (4)	C8—C9	1.377 (4)
O3—C12	1.414 (4)	C9—C10	1.373 (5)
O4—H13	0.8500	C9—H9	0.9300
O4—H14	0.8500	C10—C11	1.372 (5)
C1—C2	1.495 (5)	C10—H10	0.9300
C2—C3	1.384 (4)	C11—H11	0.9300
C2—C4^{i}	1.387 (4)	C12—H12A	0.9600
C3—C4	1.381 (4)	C12—H12B	0.9600
C3—H3	0.9300	C12—H12C	0.9600
C1—N1—N2	118.0 (3)	C7—C6—C5	122.0 (3)
C1—N1—H1	121.0	C11—C6—C5	119.2 (3)
N2—N1—H1	121.0	O2—C7—C6	123.4 (3)
C5—N2—N1	117.4 (3)	O2—C7—C8	117.0 (3)
C7—O2—H2	109.5	C6—C7—C8	119.7 (3)
C8—O3—C12	117.2 (3)	O3—C8—C9	125.4 (3)
H13—O4—H14	106.0	O3—C8—C7	114.1 (3)
O1—C1—N1	121.2 (3)	C9—C8—C7	120.5 (3)
O1—C1—C2	122.9 (3)	C10—C9—C8	119.7 (3)
N1—C1—C2	115.9 (3)	C10—C9—H9	120.1
C3—C2—C4^{i}	118.2 (3)	C8—C9—H9	120.1
C3—C2—C1	117.3 (3)	C11—C10—C9	121.1 (3)
C4^{i}—C2—C1	124.5 (3)	C11—C10—H10	119.5
C4—C3—C2	121.1 (3)	C9—C10—H10	119.5
C4—C3—H3	119.5	C10—C11—C6	120.3 (3)
C2—C3—H3	119.5	C10—C11—H11	119.9
C3—C4—C2^{i}	120.7 (3)	C6—C11—H11	119.9

C3—C4—H4	119.6	O3—C12—H12A	109.5
C2^{i}—C4—H4	119.6	O3—C12—H12B	109.5
N2—C5—C6	119.8 (3)	H12A—C12—H12B	109.5
N2—C5—H5	120.1	O3—C12—H12C	109.5
C6—C5—H5	120.1	H12A—C12—H12C	109.5
C7—C6—C11	118.8 (3)	H12B—C12—H12C	109.5
C1—N1—N2—C5	−177.2 (3)	C11—C6—C7—C8	−0.3 (4)
N2—N1—C1—O1	3.8 (4)	C5—C6—C7—C8	−179.3 (3)
N2—N1—C1—C2	−177.0 (2)	C12—O3—C8—C9	6.1 (5)
O1—C1—C2—C3	−6.5 (5)	C12—O3—C8—C7	−174.2 (3)
N1—C1—C2—C3	174.4 (3)	O2—C7—C8—O3	−0.1 (4)
O1—C1—C2—C4^{i}	173.6 (3)	C6—C7—C8—O3	179.9 (3)
N1—C1—C2—C4^{i}	−5.5 (4)	O2—C7—C8—C9	179.6 (3)
C4^{i}—C2—C3—C4	−0.1 (5)	C6—C7—C8—C9	−0.4 (5)
C1—C2—C3—C4	180.0 (3)	O3—C8—C9—C10	−179.7 (3)
C2—C3—C4—C2^{i}	0.1 (5)	C7—C8—C9—C10	0.7 (5)
N1—N2—C5—C6	−179.5 (3)	C8—C9—C10—C11	−0.1 (5)
N2—C5—C6—C7	−0.5 (5)	C9—C10—C11—C6	−0.6 (5)
N2—C5—C6—C11	−179.5 (3)	C7—C6—C11—C10	0.8 (5)
C11—C6—C7—O2	179.7 (3)	C5—C6—C11—C10	179.8 (3)
C5—C6—C7—O2	0.7 (5)		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O4^{ii}	0.86	2.00	2.835 (3)	163
O4—H14···O1^{iii}	0.85	1.92	2.751 (3)	167
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O4—H13···O3	0.85	2.29	3.021 (3)	144
O4—H13···O2	0.85	2.41	3.099 (3)	139

Symmetry codes: (ii) *x*−1, *y*, *z*−1; (iii) *x*, *y*, *z*+1.

Fig. 1

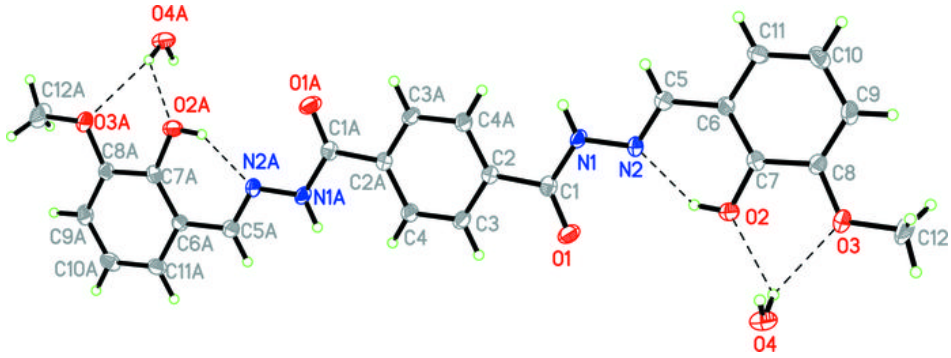
O4A
C12A
O3A
O2A
O1A
C1A
C3A
C4A
C8A
C7A
N2A
N1A
C2A
C2
N1
N2
C5
C11
C10
C9
C6
C1
C8
C7
O2
O3
C12
C9A
C6A
C5A
C4
C3
O1
C10A
C11A
O4

Fig. 2