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N,*N*′-(2-Hydroxypropane-1,3-diyl)bis(2-hydroxybenzamide) monohydrate

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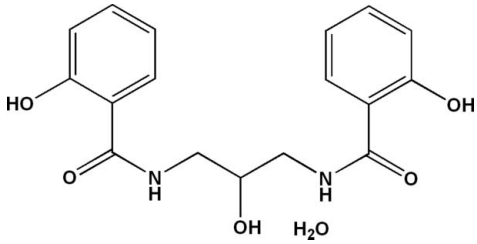
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.048; wR factor = 0.123; data-to-parameter ratio = 15.4.

In the title hydrate, $C_{17}H_{18}N_2O_5\cdot H_2O$, the complete organic molecule is generated by a crystallographic mirror plane with one C and one O atom lying on the mirror plane. The O atom of the water molecule has *m* site symmetry. Two symmetry-related intramolecular O—H···O hydrogen bonds complete $S(6)$ rings in the organic molecule. In the crystal, the components are linked into (010) sheets by O—H···O and N—H···O hydrogen bonds.

Related literature

For the synthesis of similar compounds and their complexes see: Kumar & Debashis (2006); Azam *et al.* (2012); Sarkar (1999); Louhibi *et al.* (2007); Kui *et al.* (2009).



Experimental

Crystal data

$C_{17}H_{18}N_2O_5\cdot H_2O$
M_r = 348.35
Orthorhombic, *Pnma*
a = 12.8969 (10) Å
b = 28.001 (2) Å
c = 4.5330 (4) Å
V = 1637.0 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 150 K
0.38 × 0.12 × 0.04 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2011)
T_{min} = 0.877, T_{max} = 0.996
7880 measured reflections
1904 independent reflections
1195 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.123
S = 1.04
1904 reflections
124 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 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
O1—H1···O1W^{i}	0.96 (3)	1.82 (3)	2.778 (3)	179 (3)
O1W—H1W···O6^{ii}	0.92 (2)	1.92 (2)	2.827 (2)	174 (2)
O13—H13···O6	0.84	1.75	2.500 (2)	147
N4—H4···O13^{iii}	0.88	2.07	2.915 (2)	160

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x, y, z+1$; (iii) $x+\frac{1}{2}, y, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2011); cell refinement: *SAINT* (Bruker, 2011); data reduction: *SAINT*; program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012) and *DIAMOND* (Brandenburg & Berndt, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 2012) and *CRYSCAL* (T. Roisnel, local program).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7142).

References

Azam, M., Zahid, H., Warad, I., Al-Resayes, S. I., Shahnawaz Khan, M., Shakir, M., Trzesowska-Kruszynska, A. & Kruszynski, R. (2012). *Dalton Trans.* **41**, 10854–10864.
Brandenburg, K. & Berndt, M. (2001). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
Bruker (2011). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Kui, C., Zheng, Q. Z., Qian, Y., Shi, L., Jing Zhao, J. & Zhu, H. (2009). *Bioorg. Med. Chem.* **7**, 7861–7871.
Kumar, P. N. & Debashis, R. (2006). *J. Chem. Res.* pp. 632–635.
Louhibi, S., Yacouta, A., Vendier, L., Costes, J. P. & Tuchagues, J. P. (2007). *Polyhedron*, **26**, 3448–3454.
Sarkar, B. (1999). *Chem. Rev.* **99**, 2535–2544.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

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Sihem Yebedri, Samira Louhibi, Sofiane Bouacida, Ali Ourari and Thierry Roisnel

S1. Comment

The chelating agents containing in their molecular structures 2-hydroxy-1,3-diaminopropane appear in a variey of ligands such as salicylamides (Kumar & Debashis, 2006) and Schiff bases (Azam *et al.* 2012) or as mixture of both previous functions in the same molecular structures (Kui *et al.* 2009). This kind of compounds are very attractive and interesting, especially for their coordinating properties with transition metal ions as those involved in metalloenzymes where, for example, the copper complexes are participating in the process of copper transport in humans (Sarkar, 1999). So, the title compound and its analogs are currently used in the synthesis of mono- and polynuclear manganese complexes to investigate their magnetic properties (Louhibi *et al.*, 2007). However, the resulting complexes are, in this case, often bi- or polynuclear owing to their corresponding polydentate nature (NNOOO).

Herein we report the synthesis and crystal structure of *N*,*N*-Bis-Salicylamide(2-hydroxy-1,3-diaminopropane), (I). The molecule structure of (I), and the atomic numbering used, is illustrated in Fig. 1. The asymmetric unit of (I) consists of one-half of the molecule, with the other half generated by a crystallographic mirror plane.

The crystal packing can be described by alterning layers in zigzag parallel to (100) planes (Fig. 2) and the water molecule is sandwished betwen these layers. It features O—H···O and N—H···O hydrogen bonds (Fig. 2, Table 1). A O—H···O intramolecular interaction is also observed. These interactions link the molecules within the layers and also link the layers together and reinforcing the cohesion of the structure.

S2. Experimental

90 mg (1.0 mmol) of 2-hydroxy-1,3-diaminopropane was dissolved in 10 ml of absolute ethanol and placed in three necked flask of 50 ml. 396 mg (2.0 mmol) of phenylsalicylate were also dissolved in the same solvent (10 ml absolute ethanol). This solution was added in one portion to the previous solution. The resulting mixture, under nitrogen atmosphere and stirring, was heated to reflux for three hours, after which the reaction mixture was filtered as hot solution. Yellow prisms were formed after some days by slow evaporation (yield 70%).

S3. Refinement

H1W and H1 protons were located in a difference Fourier map and refined isotropically with U_{iso}(H) = 1.5Ueq(O). The remaining H atoms were localized on Fourier maps but introduced in calculated positions and treated as riding on their parent atom (C,*O* and N) with C—H = 0.99 Å (Methylene)or 0.95 Å (aromatic) or 1.00 Å (methine), O—H = 0.84 Å and N—H = 0.88 Å; with U_{iso}(H) = 1.2U_{eq} and U_{iso}(H) = 1.5U_{eq}(hydroxy).

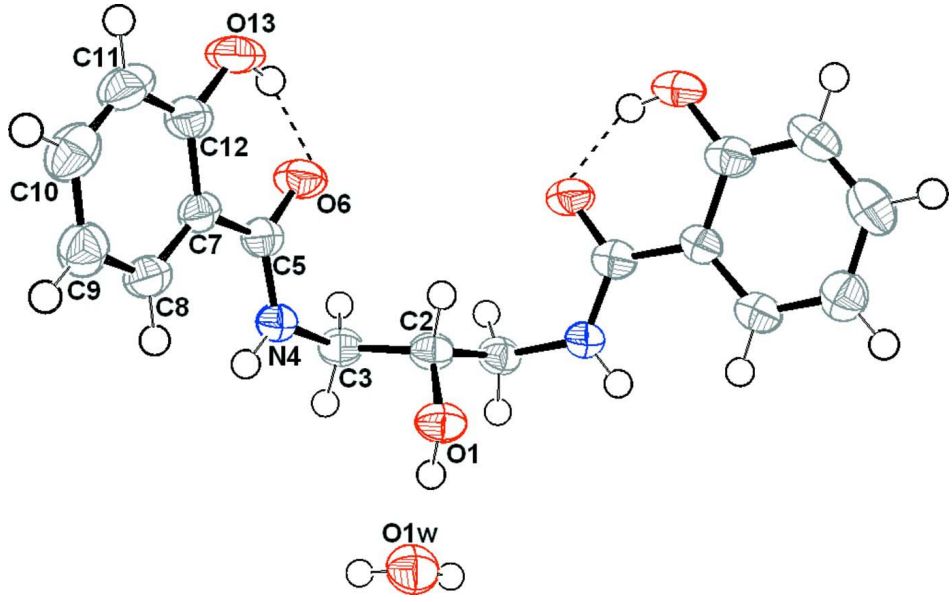

Figure 1

The molecular structure of the title compound with displacement drawn at the 50% probability level. Only the contents of the asymmetric unit are numbered.

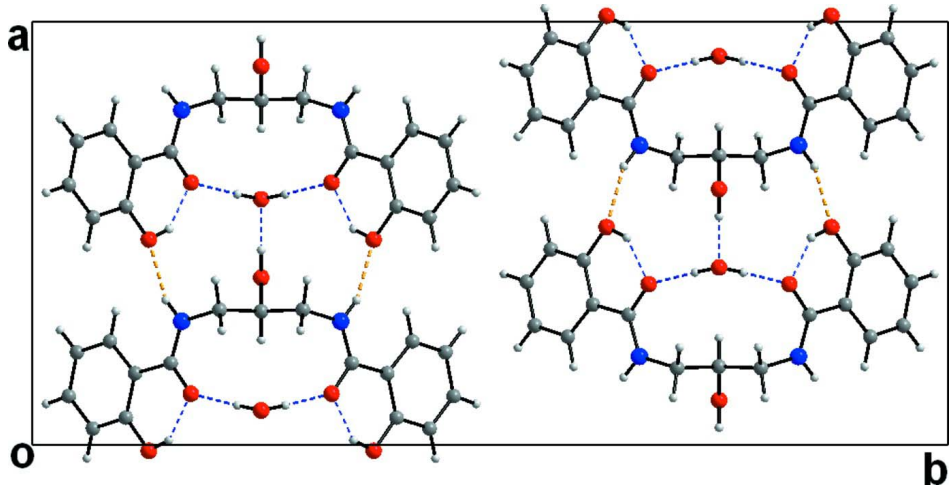

Figure 2

Alternating layers in zigzag parallel to (100) plane of (I) viewed *via c* axis showing hydrogen bond as dashed line [O—H···O and N—H···O interactions].

N,N′-(2-Hydroxypropane-1,3-diyl)bis(2-hydroxybenzamide) monohydrate

Crystal data

$C_{17}H_{18}N_2O_5·H_2O$
M_r = 348.35
Orthorhombic, *Pnma*
Hall symbol: -P 2ac 2n
a = 12.8969 (10) Å
b = 28.001 (2) Å
c = 4.5330 (4) Å
V = 1637.0 (2) Å^3
Z = 4
F(000) = 736
D_x = 1.413 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 951 reflections
θ = 3.2–21.6°

μ = 0.11 mm^{-1}
T = 150 K

Prism, yellow
0.38 × 0.12 × 0.04 mm

Data collection

Bruker APEXII diffractometer
Graphite monochromator
CCD rotation images, thin slices scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2011)
T_{min} = 0.877, T_{max} = 0.996
7880 measured reflections

1904 independent reflections
1195 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 27.4°, θ_{min} = 3.2°
h = −16→15
k = −33→36
l = −3→5

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.123
S = 1.04
1904 reflections
124 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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	0.39517 (14)	0.25000	0.2499 (4)	0.0247 (6)
O6	0.12018 (10)	0.17317 (5)	−0.0499 (3)	0.0305 (5)
O13	−0.01478 (10)	0.12722 (5)	0.2259 (4)	0.0365 (5)
N4	0.29166 (11)	0.16318 (5)	0.0274 (3)	0.0222 (5)
C2	0.3155 (2)	0.25000	0.0315 (6)	0.0220 (8)
C3	0.31907 (14)	0.20435 (6)	−0.1551 (4)	0.0231 (6)
C5	0.19276 (14)	0.15119 (6)	0.0789 (4)	0.0221 (6)
C7	0.16854 (13)	0.11226 (6)	0.2905 (4)	0.0211 (6)
C8	0.24447 (14)	0.08491 (6)	0.4318 (4)	0.0246 (6)
C9	0.21836 (16)	0.04885 (7)	0.6277 (5)	0.0299 (7)
C10	0.11462 (16)	0.03987 (7)	0.6864 (5)	0.0324 (7)
C11	0.03789 (16)	0.06622 (7)	0.5520 (5)	0.0324 (7)
C12	0.06373 (14)	0.10227 (7)	0.3538 (5)	0.0266 (6)
O1W	0.08072 (15)	0.25000	0.5613 (5)	0.0345 (7)

H1	0.460 (2)	0.25000	0.145 (7)	0.0370*
H2	0.24766	0.25000	0.13838	0.0264*
H3A	0.26982	0.20707	−0.32177	0.0278*
H3B	0.38959	0.20000	−0.23708	0.0278*
H4	0.34148	0.14581	0.10545	0.0266*
H8	0.31554	0.09118	0.39263	0.0295*
H9	0.27095	0.03049	0.72084	0.0359*
H10	0.09631	0.01526	0.82091	0.0389*
H11	−0.03286	0.05978	0.59456	0.0389*
H13	0.01003	0.14928	0.12199	0.0547*
H1W	0.0926 (17)	0.2238 (8)	0.677 (6)	0.0517*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0203 (10)	0.0348 (11)	0.0189 (11)	0.0000	0.0017 (9)	0.0000
O6	0.0194 (7)	0.0361 (8)	0.0359 (9)	0.0012 (6)	−0.0039 (7)	0.0057 (7)
O13	0.0166 (7)	0.0423 (9)	0.0506 (11)	−0.0004 (6)	0.0023 (7)	0.0076 (8)
N4	0.0185 (8)	0.0223 (8)	0.0258 (10)	0.0002 (6)	0.0017 (7)	0.0005 (7)
C2	0.0183 (13)	0.0282 (14)	0.0196 (15)	0.0000	0.0013 (12)	0.0000
C3	0.0206 (10)	0.0277 (10)	0.0211 (10)	−0.0012 (8)	0.0026 (9)	0.0016 (9)
C5	0.0208 (9)	0.0252 (10)	0.0202 (10)	−0.0002 (8)	0.0012 (9)	−0.0063 (9)
C7	0.0196 (9)	0.0225 (9)	0.0211 (11)	−0.0024 (7)	0.0020 (9)	−0.0043 (8)
C8	0.0205 (10)	0.0272 (10)	0.0262 (11)	−0.0032 (7)	0.0014 (9)	−0.0036 (9)
C9	0.0323 (11)	0.0277 (11)	0.0298 (12)	−0.0008 (9)	−0.0029 (10)	−0.0020 (9)
C10	0.0375 (12)	0.0319 (12)	0.0279 (13)	−0.0092 (9)	0.0060 (10)	0.0005 (10)
C11	0.0263 (11)	0.0357 (12)	0.0353 (13)	−0.0105 (9)	0.0079 (10)	−0.0018 (10)
C12	0.0218 (10)	0.0290 (11)	0.0291 (12)	−0.0010 (8)	0.0009 (10)	−0.0072 (9)
O1W	0.0306 (11)	0.0388 (12)	0.0340 (14)	0.0000	−0.0103 (10)	0.0000

Geometric parameters (Å, º)

O1—C2	1.427 (3)	C7—C8	1.399 (2)
O6—C5	1.263 (2)	C7—C12	1.410 (2)
O13—C12	1.360 (2)	C8—C9	1.386 (3)
O1—H1	0.96 (3)	C9—C10	1.387 (3)
O13—H13	0.8400	C10—C11	1.377 (3)
O1W—H1W^{i}	0.92 (2)	C11—C12	1.392 (3)
O1W—H1W	0.92 (2)	C2—H2	1.0000
N4—C5	1.340 (2)	C3—H3B	0.9900
N4—C3	1.462 (2)	C3—H3A	0.9900
N4—H4	0.8800	C8—H8	0.9500
C2—C3	1.534 (2)	C9—H9	0.9500
C2—C3^{i}	1.534 (2)	C10—H10	0.9500
C5—C7	1.485 (2)	C11—H11	0.9500
C2—O1—H1	106.4 (18)	C7—C12—C11	120.32 (18)
C12—O13—H13	109.00	O13—C12—C7	121.68 (18)

H1W—O1W—H1W^{i}	107 (2)	O13—C12—C11	118.00 (17)
C3—N4—C5	121.76 (14)	O1—C2—H2	107.00
C3—N4—H4	119.00	C3^{i}—C2—H2	107.00
C5—N4—H4	119.00	C3—C2—H2	107.00
O1—C2—C3^{i}	111.17 (13)	N4—C3—H3B	110.00
O1—C2—C3	111.17 (13)	C2—C3—H3A	110.00
C3—C2—C3^{i}	112.93 (19)	N4—C3—H3A	110.00
N4—C3—C2	109.75 (15)	H3A—C3—H3B	108.00
O6—C5—N4	120.19 (16)	C2—C3—H3B	110.00
N4—C5—C7	119.79 (15)	C7—C8—H8	119.00
O6—C5—C7	120.01 (16)	C9—C8—H8	119.00
C5—C7—C12	118.60 (16)	C10—C9—H9	120.00
C8—C7—C12	118.00 (16)	C8—C9—H9	120.00
C5—C7—C8	123.40 (15)	C9—C10—H10	120.00
C7—C8—C9	121.48 (17)	C11—C10—H10	120.00
C8—C9—C10	119.29 (19)	C10—C11—H11	120.00
C9—C10—C11	120.75 (19)	C12—C11—H11	120.00
C10—C11—C12	120.15 (19)		

C3—N4—C5—O6	5.8 (3)	C12—C7—C8—C9	0.3 (3)
C3—N4—C5—C7	−173.61 (15)	C5—C7—C12—O13	0.2 (3)
C5—N4—C3—C2	83.3 (2)	C5—C7—C12—C11	−179.68 (18)
O1—C2—C3—N4	67.2 (2)	C8—C7—C12—O13	179.97 (19)
C3^{i}—C2—C3—N4	−167.07 (16)	C8—C7—C12—C11	0.1 (3)
O6—C5—C7—C8	177.11 (17)	C7—C8—C9—C10	−0.4 (3)
O6—C5—C7—C12	−3.1 (3)	C8—C9—C10—C11	0.2 (3)
N4—C5—C7—C8	−3.5 (3)	C9—C10—C11—C12	0.2 (3)
N4—C5—C7—C12	176.29 (17)	C10—C11—C12—O13	179.8 (2)
C5—C7—C8—C9	−179.93 (17)	C10—C11—C12—C7	−0.4 (3)

Symmetry code: (i) x, $-y+1/2$, z.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O1*W*^{ii}	0.96 (3)	1.82 (3)	2.778 (3)	179 (3)
O1*W*—H1*W*···O6^{iii}	0.92 (2)	1.92 (2)	2.827 (2)	174 (2)
O13—H13···O6	0.84	1.75	2.500 (2)	147
N4—H4···O13^{iv}	0.88	2.07	2.915 (2)	160

Symmetry codes: (ii) $x+1/2$, $-y+1/2$, $-z+1/2$; (iii) x, y, $z+1$; (iv) $x+1/2$, y, $-z+1/2$.