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N′-(2-Hydroxy-3-methoxybenzylidene)pyrazine-2-carbohydrazide monohydrate

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Keywords: crystal structure; Schiff base; acylhydrazone ligand; hydrogen bonding.

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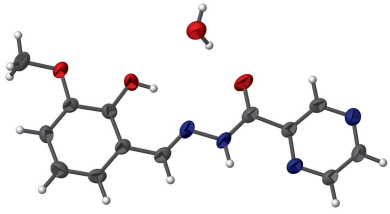
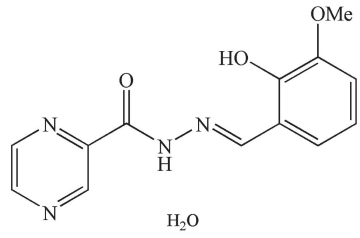
Structural data: full structural data are available from iucrdata.iucr.org

In the title hydrated Schiff base, $C_{13}H_{12}N_4O_3\cdot H_2O$, the dihedral angle between the aromatic rings is 5.06 (11)° and an intramolecular O—H···N hydrogen bond closes an S(6) ring. In the crystal, O_w—H···O and O_w—H···N (w = water) hydrogen bonds link the components into centrosymmetric tetramers (two Schiff bases and two water molecules). Longer N—H···O hydrogen bonds link the tetramers into [010] chains. A weak C—H···O hydrogen bond and aromatic π–π stacking between the pyrazine and phenyl rings [centroid–centroid separations = 3.604 (2) and 3.715 (2) Å] are also observed.

3D view



Chemical scheme



Structure description

Hydrazone-type Schiff base ligands have attracted attention from inorganic chemists because of their simple synthesis and variety arising from changing the aldyhyde or ketone and acylhydrazide precursors. Their applications include molecular switches (Coskun *et al.*, 2012), sensors (Albelda *et al.*, 2012) and single molecular magnets (SMMs) (Anwar *et al.*, 2018). As part of our studies in this area, we now describe the synthesis and structure of the title pyrazine-containing hydrazone, which crystallized as a monohydrate (Fig. 1).

The dihedral angle between the aromatic rings is 5.06 (11)° and an intramolecular O2—H2···N2 hydrogen bond closes an *S*(6) ring. The C7—N2 bond length [1.278 (3) Å] is consistent with a normal carbon–nitrogen double bond. In the crystal, O_w—H···O and O_w—H···N (w = water) hydrogen bonds link the components into centrosymmetric tetramers (two Schiff base and two water molecules). Longer N—H···O hydrogen bonds link the tetramers into [010] chains (Table 1, Fig. 2). The packing is consolidated by a weak C—H···O hydrogen bond and aromatic π–π stacking between the pyrazine and phenyl rings [centroid–centroid separations = 3.604 (2) and 3.715 (2) Å].

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···N3	0.88	2.34	2.708 (3)	105
N1—H1···O4^{i}	0.88	2.49	3.119 (3)	129
O2—H2···N2	0.84	1.94	2.668 (3)	145
O4—H4A···O3	0.87	1.99	2.846 (3)	167
O4—H4B···N4^{ii}	0.87	2.17	2.998 (3)	160
C13—H13A···O2^{iii}	0.98	2.56	3.335 (4)	135

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

Synthesis and crystallization

Pyrazine-2-carbohydrazide (2.76 g, 20 mmol) was reacted with 2-hydroxy-3-methoxybenzaldehyde (3.04 g, 20 mmol) under reflux in 25 ml methanol for 8 h. After cooling and solvent removal by rotary evaporation, a light yellow solid was obtained, which was recrystallized from methanol solution at room temperature to obtain colourless crystals of the title compound.

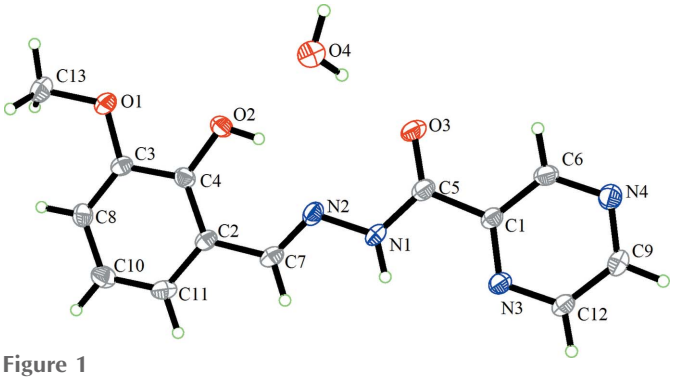


Figure 1
The molecular structure of the title compound showing displacement ellipsoids drawn at the 30% probability level.

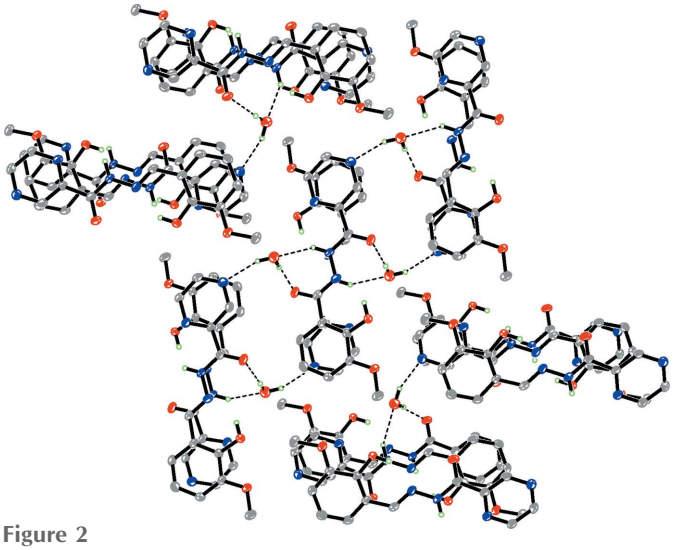


Figure 2
The crystal packing viewed along the -axis direction.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{13}H_{12}N_4O_3·H_2O$
M_r	290.28
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	189
a, b, c (Å)	7.018 (3), 9.041 (4), 20.828 (8)
β (°)	91.481 (7)
V (Å^3)	1321.1 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.25 × 0.15 × 0.12
Data collection	
Diffractometer	Bruker D8 Venture
Absorption correction	Multi-scan (*SADABS*; Bruker, 2014)
T_{min}, T_{max}	0.626, 0.746
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	7707, 2996, 1745
R_{int}	0.053
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.653
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.063, 0.161, 1.00
No. of reflections	2996
No. of parameters	195
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.31, −0.28

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Funding information

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full crystallographic data

IUCrData (2020). **5**, x191731 [https://doi.org/10.1107/S2414314619017310]

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Crystal data

$C_{13}H_{12}N_4O_3·H_2O$
M_r = 290.28
Monoclinic, $P2_1/c$
a = 7.018 (3) Å
b = 9.041 (4) Å
c = 20.828 (8) Å
β = 91.481 (7)°
V = 1321.1 (9) Å^3
Z = 4
$F(000)$ = 608
D_x = 1.459 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1318 reflections
θ = 2.5–24.4°
μ = 0.11 mm^{-1}
T = 189 K
Block, colourless
0.25 × 0.15 × 0.12 mm

Data collection

Bruker D8 Venture diffractometer
Multi–scan
Absorption correction: multi-scan (SADABS; Bruker, 2014)
T_{min} = 0.626, T_{max} = 0.746
7707 measured reflections
2996 independent reflections
1745 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 27.7°, θ_{min} = 2.5°
h = −9→8
k = −11→11
l = −18→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.161
S = 1.00
2996 reflections
195 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.076P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.8752 (3)	0.71932 (19)	0.73561 (7)	0.0466 (5)

O2	0.8069 (3)	0.55020 (18)	0.63673 (8)	0.0485 (5)
H2	0.784669	0.504827	0.602149	0.073*
O3	0.6841 (3)	0.2318 (2)	0.50428 (8)	0.0551 (6)
N1	0.7156 (3)	0.4515 (2)	0.45331 (9)	0.0390 (5)
H1	0.713885	0.499088	0.416450	0.047*
N2	0.7502 (3)	0.5261 (2)	0.51003 (9)	0.0380 (5)
N3	0.6642 (3)	0.3166 (2)	0.33806 (9)	0.0366 (5)
N4	0.5903 (3)	0.0122 (2)	0.33186 (10)	0.0411 (5)
C1	0.6523 (3)	0.2336 (3)	0.39063 (10)	0.0324 (6)
C2	0.8247 (3)	0.7542 (3)	0.56268 (10)	0.0329 (6)
C3	0.8709 (3)	0.7883 (3)	0.67721 (11)	0.0325 (6)
C4	0.8336 (3)	0.6947 (3)	0.62434 (11)	0.0336 (6)
C5	0.6843 (3)	0.3044 (3)	0.45503 (11)	0.0370 (6)
C6	0.6124 (3)	0.0832 (3)	0.38743 (11)	0.0378 (6)
H6	0.600418	0.029255	0.426269	0.045*
C7	0.7873 (3)	0.6640 (3)	0.50590 (11)	0.0368 (6)
H7	0.790496	0.708891	0.464695	0.044*
C8	0.8975 (3)	0.9374 (3)	0.66768 (12)	0.0388 (6)
H8	0.924031	1.000252	0.703370	0.047*
C9	0.6062 (4)	0.0945 (3)	0.27911 (12)	0.0400 (6)
H9	0.594317	0.048417	0.238199	0.048*
C10	0.8859 (4)	0.9966 (3)	0.60621 (13)	0.0431 (7)
H10	0.902978	1.099855	0.600130	0.052*
C11	0.8502 (3)	0.9074 (3)	0.55447 (12)	0.0395 (6)
H11	0.842440	0.948922	0.512593	0.047*
C12	0.6397 (3)	0.2452 (3)	0.28243 (11)	0.0387 (6)
H12	0.645434	0.299940	0.243586	0.046*
C13	0.8953 (4)	0.8115 (3)	0.79116 (11)	0.0505 (7)
H13A	1.018576	0.862348	0.790623	0.076*
H13B	0.888839	0.750474	0.829937	0.076*
H13C	0.792400	0.884721	0.790985	0.076*
O4	0.4937 (3)	0.2931 (2)	0.62032 (9)	0.0573 (6)
H4A	0.567749	0.271798	0.588742	0.086*
H4B	0.487099	0.211456	0.642269	0.086*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0713 (13)	0.0426 (11)	0.0256 (9)	−0.0046 (9)	−0.0038 (8)	−0.0012 (8)
O2	0.0798 (14)	0.0288 (10)	0.0360 (10)	−0.0038 (9)	−0.0165 (9)	0.0014 (8)
O3	0.0919 (15)	0.0470 (12)	0.0263 (10)	0.0178 (10)	−0.0010 (9)	0.0063 (8)
N1	0.0536 (14)	0.0429 (13)	0.0202 (10)	0.0006 (10)	−0.0043 (9)	−0.0019 (9)
N2	0.0425 (12)	0.0447 (14)	0.0265 (11)	0.0051 (10)	−0.0051 (9)	−0.0052 (9)
N3	0.0413 (12)	0.0401 (12)	0.0283 (11)	0.0018 (9)	−0.0009 (9)	0.0017 (9)
N4	0.0436 (13)	0.0414 (13)	0.0383 (13)	0.0000 (10)	−0.0011 (10)	−0.0032 (10)
C1	0.0333 (13)	0.0379 (14)	0.0259 (12)	0.0062 (10)	−0.0013 (9)	0.0001 (11)
C2	0.0319 (13)	0.0409 (15)	0.0258 (12)	0.0006 (11)	−0.0034 (10)	0.0012 (11)
C3	0.0318 (13)	0.0385 (15)	0.0270 (12)	0.0007 (10)	−0.0024 (10)	0.0033 (10)

C4	0.0363 (14)	0.0288 (14)	0.0353 (14)	0.0023 (10)	−0.0039 (10)	0.0025 (10)
C5	0.0418 (15)	0.0433 (16)	0.0258 (13)	0.0101 (11)	0.0002 (11)	0.0014 (11)
C6	0.0452 (15)	0.0388 (15)	0.0294 (13)	0.0062 (11)	0.0013 (11)	0.0063 (11)
C7	0.0371 (14)	0.0472 (17)	0.0257 (13)	0.0019 (11)	−0.0039 (10)	0.0036 (11)
C8	0.0439 (15)	0.0346 (15)	0.0376 (14)	−0.0042 (11)	−0.0034 (11)	−0.0059 (11)
C9	0.0428 (14)	0.0477 (17)	0.0294 (13)	−0.0024 (12)	−0.0011 (11)	−0.0078 (12)
C10	0.0449 (15)	0.0361 (15)	0.0482 (16)	−0.0040 (12)	−0.0038 (12)	0.0050 (12)
C11	0.0418 (14)	0.0422 (16)	0.0341 (14)	−0.0046 (12)	−0.0044 (11)	0.0115 (11)
C12	0.0462 (15)	0.0438 (16)	0.0258 (13)	−0.0019 (12)	−0.0037 (11)	0.0014 (11)
C13	0.0620 (19)	0.0581 (19)	0.0314 (14)	0.0005 (14)	−0.0002 (13)	−0.0079 (13)
O4	0.0784 (15)	0.0496 (12)	0.0441 (12)	0.0017 (10)	0.0057 (10)	0.0024 (9)

Geometric parameters (Å, º)

O1—C3	1.367 (3)	C3—C4	1.408 (3)
O1—C13	1.430 (3)	C3—C8	1.376 (3)
O2—H2	0.8400	C6—H6	0.9500
O2—C4	1.346 (3)	C7—H7	0.9500
O3—C5	1.218 (3)	C8—H8	0.9500
N1—H1	0.8800	C8—C10	1.388 (4)
N1—N2	1.376 (3)	C9—H9	0.9500
N1—C5	1.348 (3)	C9—C12	1.385 (4)
N2—C7	1.278 (3)	C10—H10	0.9500
N3—C1	1.332 (3)	C10—C11	1.364 (4)
N3—C12	1.333 (3)	C11—H11	0.9500
N4—C6	1.329 (3)	C12—H12	0.9500
N4—C9	1.334 (3)	C13—H13A	0.9800
C1—C5	1.498 (3)	C13—H13B	0.9800
C1—C6	1.390 (3)	C13—H13C	0.9800
C2—C4	1.393 (3)	O4—H4A	0.8702
C2—C7	1.455 (3)	O4—H4B	0.8697
C2—C11	1.407 (3)		
C3—O1—C13	117.0 (2)	N2—C7—C2	121.7 (2)
C4—O2—H2	109.5	N2—C7—H7	119.1
N2—N1—H1	120.5	C2—C7—H7	119.1
C5—N1—H1	120.5	C3—C8—H8	119.8
C5—N1—N2	119.1 (2)	C3—C8—C10	120.4 (2)
C7—N2—N1	116.9 (2)	C10—C8—H8	119.8
C1—N3—C12	115.6 (2)	N4—C9—H9	119.2
C6—N4—C9	116.0 (2)	N4—C9—C12	121.7 (2)
N3—C1—C5	119.0 (2)	C12—C9—H9	119.2
N3—C1—C6	121.9 (2)	C8—C10—H10	119.8
C6—C1—C5	119.1 (2)	C11—C10—C8	120.4 (2)
C4—C2—C7	122.4 (2)	C11—C10—H10	119.8
C4—C2—C11	119.3 (2)	C2—C11—H11	119.8
C11—C2—C7	118.3 (2)	C10—C11—C2	120.5 (2)
O1—C3—C4	114.9 (2)	C10—C11—H11	119.8

O1—C3—C8	125.2 (2)	N3—C12—C9	122.5 (2)
C8—C3—C4	120.0 (2)	N3—C12—H12	118.7
O2—C4—C2	123.3 (2)	C9—C12—H12	118.7
O2—C4—C3	117.2 (2)	O1—C13—H13A	109.5
C2—C4—C3	119.4 (2)	O1—C13—H13B	109.5
O3—C5—N1	123.9 (2)	O1—C13—H13C	109.5
O3—C5—C1	121.4 (2)	H13A—C13—H13B	109.5
N1—C5—C1	114.7 (2)	H13A—C13—H13C	109.5
N4—C6—C1	122.2 (2)	H13B—C13—H13C	109.5
N4—C6—H6	118.9	H4A—O4—H4B	104.5
C1—C6—H6	118.9		

O1—C3—C4—O2	−0.1 (3)	C6—N4—C9—C12	−1.3 (4)
O1—C3—C4—C2	179.5 (2)	C6—C1—C5—O3	3.2 (4)
O1—C3—C8—C10	−178.5 (2)	C6—C1—C5—N1	−177.8 (2)
N1—N2—C7—C2	179.5 (2)	C7—C2—C4—O2	−0.6 (4)
N2—N1—C5—O3	0.2 (4)	C7—C2—C4—C3	179.8 (2)
N2—N1—C5—C1	−178.74 (19)	C7—C2—C11—C10	−179.9 (2)
N3—C1—C5—O3	−176.4 (2)	C8—C3—C4—O2	−179.3 (2)
N3—C1—C5—N1	2.6 (3)	C8—C3—C4—C2	0.2 (3)
N3—C1—C6—N4	2.4 (4)	C8—C10—C11—C2	0.0 (4)
N4—C9—C12—N3	2.2 (4)	C9—N4—C6—C1	−0.9 (4)
C1—N3—C12—C9	−0.7 (3)	C11—C2—C4—O2	178.5 (2)
C3—C8—C10—C11	−0.8 (4)	C11—C2—C4—C3	−1.0 (3)
C4—C2—C7—N2	3.9 (4)	C11—C2—C7—N2	−175.3 (2)
C4—C2—C11—C10	0.9 (4)	C12—N3—C1—C5	178.1 (2)
C4—C3—C8—C10	0.7 (4)	C12—N3—C1—C6	−1.5 (3)
C5—N1—N2—C7	177.0 (2)	C13—O1—C3—C4	−174.5 (2)
C5—C1—C6—N4	−177.1 (2)	C13—O1—C3—C8	4.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···N3	0.88	2.34	2.708 (3)	105
N1—H1···O4^{i}	0.88	2.49	3.119 (3)	129
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C13—H13*A*···O2^{iii}	0.98	2.56	3.335 (4)	135

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