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Methyl 5-(4-hydroxyphenyl)-6-oxo-1,6-dihydropyrazine-2-carboxylate monohydrate

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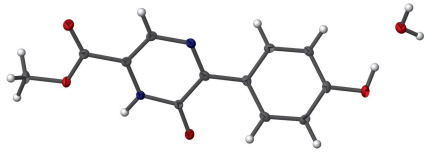
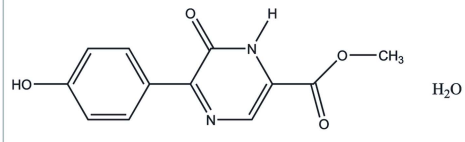
Structural data: full structural data are available from iucrdata.iucr.org

In the title hydrate, $C_{12}H_{10}N_2O_4\cdot H_2O$, the dihedral angle between the benzene and pyrazine rings is 37.70 (10)°. In the crystal, molecules are connected by hydrogen bonds (N—H···O, O—H···O and O—H···O) to generate ($1\bar{1}0$) sheets. Aromatic π–π stacking [centroid–centroid separation = 3.7070 (13) Å] links the sheets into a three-dimensional network.

3D view



Chemical scheme



Structure description

As part of our ongoing studies of amoxicillin degradation (Eltayeb, 2016), we report herein the synthesis and crystal structure of the title compound (Fig. 1). The dihedral angle between the benzene and pyrazine rings is 37.70 (10)°. In the crystal, the molecules are connected by various hydrogen bonds (N1—H1*N*···O2, O1—H1*O*···O1*W*, O1*W*—H1*W*···O4 and O1*W*—H2*W*···N2 (Fig. 2, Table 1), which generate ($1\bar{1}0$) sheets.

Aromatic π–π stacking is also observed: *Cg1*···*Cg1*^{iv} [centroid–centroid separation = 3.7074 (13) Å] and *Cg2*···*Cg2*^{v} [centroid–centroid separation = 3.7071 (13)] [symmetry codes: (iv) $-1+x, y, x$; (v) $1+x, y, z$; *Cg1* is the centroid of the C7/C8/N1/C9/C10/N2 ring and *Cg2* is the centroid of the C1–C6 ring]. The end result is a three-dimensional network.

Synthesis and crystallization

Amoxicillin trihydrate (0.5 mmol, 0.21 g) and 2,4-dihydroxybenzaldehyde (0.5 mmol, 0.07 g) were dissolved in methanol in a round-bottomed flask, then 0.1 g copper sulfate solution in 5 ml water was added. The mixture was refluxed for about 2 h. The product was filtered. Golden blocks of the title compound were formed on slow evaporation of the solution in a few days.

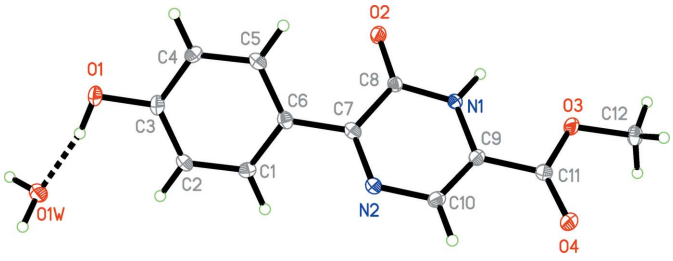


Figure 1
The molecular structure of the title compound, shown with 50% probability displacement ellipsoids. The O—H···O hydrogen bond is indicated by a dashed line.

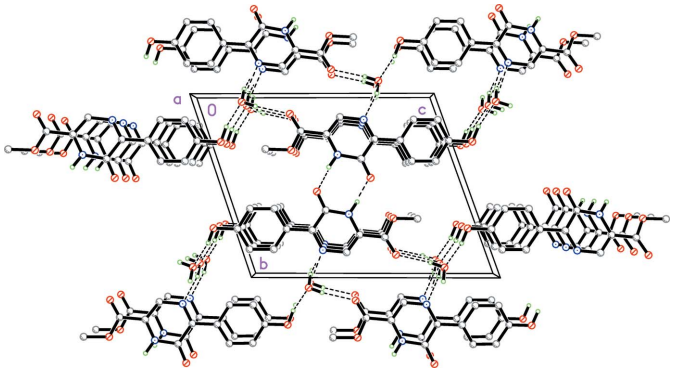


Figure 2
The crystal packing of the title compound, viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The highest residual electron density peak is located at 1.78 Å from C10 and the deepest hole is located at 0.81 Å from N1.

Acknowledgements

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References

Bruker (2016). *APEX3*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···O2^{i}	0.94 (3)	1.88 (3)	2.805 (2)	172 (3)
O1—H1*O*···O1*W*	0.93 (3)	1.77 (3)	2.682 (3)	168 (3)
O1*W*—H1*W*···O4^{ii}	0.86 (4)	2.02 (4)	2.864 (2)	167 (3)
O1*W*—H2*W*···N2^{iii}	0.90 (3)	1.97 (3)	2.838 (3)	163 (3)

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{10}N_2O_4{\cdot}H_2O$
M_r	264.24
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	100
a, b, c (Å)	3.7072 (3), 11.4251 (9), 14.3713 (13)
α, β, γ (°)	70.748 (4), 88.425 (5), 83.284 (5)
V (Å^3)	570.68 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.12
Crystal size (mm)	0.16 × 0.12 × 0.07
Data collection	
Diffractometer	Bruker D8 Quest CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.689, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	10223, 2091, 1556
R_{int}	0.079
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.049, 0.119, 1.02
No. of reflections	2091
No. of parameters	189
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.27, −0.34

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2015).

Eltayeb, N. E. (2016). *IUCrData*, **1**, x161689.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
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full crystallographic data

IUCrData (2016). **1**, x161741 [https://doi.org/10.1107/S2414314616017417]

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Crystal data

$C_{12}H_{10}N_2O_4·H_2O$
M_r = 264.24
Triclinic, $P\overline{1}$
a = 3.7072 (3) Å
b = 11.4251 (9) Å
c = 14.3713 (13) Å
α = 70.748 (4)°
β = 88.425 (5)°
γ = 83.284 (5)°
V = 570.68 (8) Å^3
Z = 2
$F(000)$ = 276
D_x = 1.538 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3588 reflections
θ = 2.8–25.3°
μ = 0.12 mm^{-1}
T = 100 K
Block, gold
0.16 × 0.12 × 0.07 mm

Data collection

Bruker D8 Quest CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.689, T_{max} = 0.745
10223 measured reflections
2091 independent reflections
1556 reflections with $I > 2\sigma(I)$
R_{int} = 0.079
θ_{max} = 25.4°, θ_{min} = 2.8°
h = −4→4
k = −13→13
l = −17→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.119
S = 1.02
2091 reflections
189 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.2621P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 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	0.5384 (5)	0.72898 (15)	−0.07116 (12)	0.0208 (4)
O2	0.1695 (4)	0.53640 (13)	0.38440 (11)	0.0185 (4)
O1W	0.2759 (5)	0.93868 (16)	−0.21323 (13)	0.0215 (4)
O3	0.0237 (4)	0.68091 (13)	0.65526 (11)	0.0166 (4)
O4	0.2049 (5)	0.87280 (14)	0.61292 (11)	0.0198 (4)
N1	0.1665 (5)	0.65746 (16)	0.48160 (14)	0.0125 (4)
N2	0.4133 (5)	0.84534 (16)	0.32977 (13)	0.0137 (4)
C1	0.3041 (6)	0.84329 (19)	0.13426 (16)	0.0133 (5)
H1	0.2106	0.9183	0.1458	0.016*
C2	0.3441 (6)	0.84230 (19)	0.03887 (16)	0.0143 (5)
H2	0.2730	0.9157	−0.0149	0.017*
C3	0.4887 (6)	0.7337 (2)	0.02149 (16)	0.0145 (5)
C4	0.5883 (6)	0.6262 (2)	0.09990 (16)	0.0154 (5)
H4	0.6897	0.5523	0.0879	0.018*
C5	0.5405 (6)	0.62616 (19)	0.19544 (16)	0.0147 (5)
H5	0.6038	0.5516	0.2489	0.018*
C6	0.3997 (6)	0.73496 (19)	0.21435 (16)	0.0123 (5)
C7	0.3548 (6)	0.74126 (19)	0.31435 (16)	0.0128 (5)
C8	0.2279 (6)	0.63618 (19)	0.39424 (16)	0.0115 (5)
C9	0.2278 (6)	0.76628 (19)	0.49603 (16)	0.0121 (5)
C10	0.3518 (6)	0.85802 (19)	0.42030 (16)	0.0137 (5)
H10	0.3972	0.9332	0.4303	0.016*
C11	0.1537 (6)	0.78086 (19)	0.59361 (16)	0.0137 (5)
C12	−0.0733 (6)	0.6846 (2)	0.75253 (17)	0.0179 (5)
H12A	−0.1903	0.6103	0.7886	0.027*
H12B	−0.2415	0.7596	0.7466	0.027*
H12C	0.1462	0.6863	0.7883	0.027*
H1N	0.070 (8)	0.593 (3)	0.531 (2)	0.043 (9)*
H1O	0.451 (9)	0.807 (3)	−0.114 (2)	0.056 (10)*
H1W	0.224 (9)	0.914 (3)	−0.261 (3)	0.049 (10)*
H2W	0.388 (9)	1.008 (3)	−0.238 (2)	0.048 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0321 (10)	0.0195 (9)	0.0097 (9)	0.0020 (7)	0.0026 (7)	−0.0052 (7)
O2	0.0303 (10)	0.0138 (8)	0.0131 (9)	−0.0090 (7)	0.0062 (7)	−0.0054 (7)
O1W	0.0334 (11)	0.0164 (9)	0.0145 (10)	−0.0058 (7)	0.0007 (8)	−0.0038 (7)
O3	0.0252 (9)	0.0146 (8)	0.0116 (9)	−0.0058 (7)	0.0059 (7)	−0.0056 (6)
O4	0.0313 (10)	0.0151 (9)	0.0152 (9)	−0.0061 (7)	0.0014 (7)	−0.0069 (7)
N1	0.0165 (10)	0.0102 (9)	0.0103 (10)	−0.0025 (7)	0.0026 (8)	−0.0026 (8)
N2	0.0169 (10)	0.0117 (9)	0.0113 (10)	−0.0004 (7)	−0.0010 (8)	−0.0026 (8)
C1	0.0131 (12)	0.0117 (11)	0.0152 (12)	−0.0004 (8)	−0.0007 (9)	−0.0047 (9)
C2	0.0162 (12)	0.0130 (11)	0.0113 (12)	−0.0011 (9)	−0.0009 (9)	−0.0008 (9)
C3	0.0150 (12)	0.0187 (12)	0.0105 (12)	−0.0034 (9)	0.0011 (9)	−0.0054 (9)

C4	0.0156 (12)	0.0140 (11)	0.0167 (13)	0.0018 (9)	0.0027 (9)	−0.0066 (10)
C5	0.0157 (12)	0.0127 (11)	0.0132 (12)	−0.0012 (9)	−0.0006 (9)	−0.0008 (9)
C6	0.0109 (11)	0.0142 (11)	0.0116 (12)	−0.0045 (8)	0.0017 (9)	−0.0029 (9)
C7	0.0108 (11)	0.0127 (11)	0.0131 (12)	−0.0012 (8)	0.0004 (9)	−0.0019 (9)
C8	0.0117 (11)	0.0116 (11)	0.0104 (12)	−0.0007 (9)	0.0009 (9)	−0.0029 (9)
C9	0.0112 (11)	0.0124 (11)	0.0129 (12)	0.0002 (8)	−0.0014 (9)	−0.0047 (9)
C10	0.0151 (12)	0.0120 (11)	0.0152 (13)	−0.0014 (9)	−0.0006 (9)	−0.0060 (9)
C11	0.0145 (12)	0.0126 (12)	0.0136 (12)	−0.0003 (9)	−0.0004 (9)	−0.0042 (9)
C12	0.0212 (13)	0.0217 (13)	0.0110 (12)	−0.0027 (10)	0.0053 (10)	−0.0061 (10)

Geometric parameters (Å, º)

O1—C3	1.357 (3)	C2—C3	1.390 (3)
O1—H1O	0.93 (3)	C2—H2	0.9500
O2—C8	1.238 (2)	C3—C4	1.385 (3)
O1W—H1W	0.86 (4)	C4—C5	1.380 (3)
O1W—H2W	0.90 (3)	C4—H4	0.9500
O3—C11	1.326 (3)	C5—C6	1.398 (3)
O3—C12	1.447 (3)	C5—H5	0.9500
O4—C11	1.208 (3)	C6—C7	1.466 (3)
N1—C8	1.364 (3)	C7—C8	1.475 (3)
N1—C9	1.371 (3)	C9—C10	1.352 (3)
N1—H1N	0.94 (3)	C9—C11	1.480 (3)
N2—C7	1.322 (3)	C10—H10	0.9500
N2—C10	1.366 (3)	C12—H12A	0.9800
C1—C2	1.378 (3)	C12—H12B	0.9800
C1—C6	1.401 (3)	C12—H12C	0.9800
C1—H1	0.9500		
C3—O1—H1O	106 (2)	C5—C6—C7	122.84 (19)
H1W—O1W—H2W	108 (3)	C1—C6—C7	118.6 (2)
C11—O3—C12	116.91 (16)	N2—C7—C6	118.44 (19)
C8—N1—C9	123.15 (19)	N2—C7—C8	121.0 (2)
C8—N1—H1N	115.4 (18)	C6—C7—C8	120.44 (18)
C9—N1—H1N	121.4 (18)	O2—C8—N1	121.07 (19)
C7—N2—C10	120.27 (19)	O2—C8—C7	124.0 (2)
C2—C1—C6	120.7 (2)	N1—C8—C7	114.90 (18)
C2—C1—H1	119.6	C10—C9—N1	119.0 (2)
C6—C1—H1	119.6	C10—C9—C11	121.71 (19)
C1—C2—C3	119.9 (2)	N1—C9—C11	119.30 (19)
C1—C2—H2	120.1	C9—C10—N2	121.63 (19)
C3—C2—H2	120.1	C9—C10—H10	119.2
O1—C3—C4	118.0 (2)	N2—C10—H10	119.2
O1—C3—C2	121.90 (19)	O4—C11—O3	125.3 (2)
C4—C3—C2	120.1 (2)	O4—C11—C9	123.6 (2)
C5—C4—C3	120.1 (2)	O3—C11—C9	111.11 (18)
C5—C4—H4	119.9	O3—C12—H12A	109.5
C3—C4—H4	119.9	O3—C12—H12B	109.5

C4—C5—C6	120.6 (2)	H12A—C12—H12B	109.5
C4—C5—H5	119.7	O3—C12—H12C	109.5
C6—C5—H5	119.7	H12A—C12—H12C	109.5
C5—C6—C1	118.6 (2)	H12B—C12—H12C	109.5
C6—C1—C2—C3	−1.5 (3)	C9—N1—C8—C7	−2.0 (3)
C1—C2—C3—O1	−178.9 (2)	N2—C7—C8—O2	−179.8 (2)
C1—C2—C3—C4	0.8 (3)	C6—C7—C8—O2	3.6 (3)
O1—C3—C4—C5	−179.5 (2)	N2—C7—C8—N1	2.1 (3)
C2—C3—C4—C5	0.7 (3)	C6—C7—C8—N1	−174.48 (19)
C3—C4—C5—C6	−1.5 (3)	C8—N1—C9—C10	0.7 (3)
C4—C5—C6—C1	0.8 (3)	C8—N1—C9—C11	−179.89 (19)
C4—C5—C6—C7	−177.9 (2)	N1—C9—C10—N2	0.7 (3)
C2—C1—C6—C5	0.7 (3)	C11—C9—C10—N2	−178.7 (2)
C2—C1—C6—C7	179.5 (2)	C7—N2—C10—C9	−0.5 (3)
C10—N2—C7—C6	175.74 (19)	C12—O3—C11—O4	1.8 (3)
C10—N2—C7—C8	−0.9 (3)	C12—O3—C11—C9	−177.80 (18)
C5—C6—C7—N2	143.9 (2)	C10—C9—C11—O4	−1.6 (3)
C1—C6—C7—N2	−34.8 (3)	N1—C9—C11—O4	179.0 (2)
C5—C6—C7—C8	−39.4 (3)	C10—C9—C11—O3	178.00 (19)
C1—C6—C7—C8	141.9 (2)	N1—C9—C11—O3	−1.4 (3)
C9—N1—C8—O2	179.85 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O2^{i}	0.94 (3)	1.88 (3)	2.805 (2)	172 (3)
O1—H1*O*···O1*W*	0.93 (3)	1.77 (3)	2.682 (3)	168 (3)
O1*W*—H1*W*···O4^{ii}	0.86 (4)	2.02 (4)	2.864 (2)	167 (3)
O1*W*—H2*W*···N2^{iii}	0.90 (3)	1.97 (3)	2.838 (3)	163 (3)

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