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

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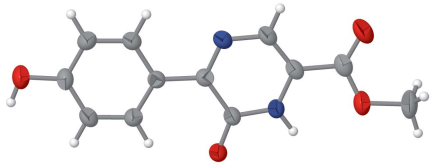
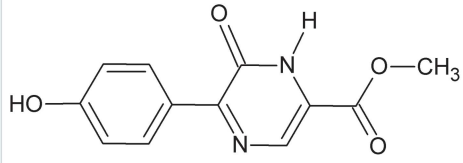
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{12}H_{10}N_2O_4$, is approximately planar, with dihedral angles of 5.53 (9) and 2.48 (13)°, respectively, between the benzene and pyrazine rings, and between the pyrazine ring and the methyl carboxylate plane. An intramolecular C—H···O hydrogen bond with an *S*(6) ring motif is observed. In the crystal, intermolecular O—H···O and N—H···O hydrogen bonds link molecules into a layer parallel to the *ab* plane. Adjacent layers interpenetrate each other through a π–π interaction [centroid–centroid distance of 3.4746 (11) Å].

3D view



Chemical scheme



Structure description

The title compound was obtained unexpectedly by the reaction of amoxicillin trihydrate and copper sulfate in a mixture of methanol and water. The molecule is approximately planer, as indicated by dihedral angles 5.53 (9) and 2.48 (13)°, respectively, between the C1–C6 and C7/C8/N1/C9/C10/N2 rings, and between the C7/C8/N1/C9/C10/N2 ring and the O3/C11/O4/C12 plane. An intramolecular C5—H5···O3 hydrogen bond generates an *S*(6) ring motif (Table 1 and Fig. 1).

In the crystal, molecules are linked through intermolecular O—H···O and N—H···O hydrogen bonds (Table 1) into a layer parallel to the *ab* plane (Figs. 2 and 3). Adjacent layers interpenetrate each other and a π–π stacking interaction is present between the layers. The $Cg\cdots Cg^{iii}$ separation is 3.4746 (11) Å [symmetry code: (iii) $1-x, -y, 1-z$]; *Cg* is the centroid of the C7/C8/N1/C9/C10/N2 ring.

Synthesis and crystallization

Amoxicillin trihydrate (0.5 mmol, 0.21 g) was dissolved in methanol in a round-bottomed flask, then 0.1 g copper sulfate in 5 ml water were added. The mixture was refluxed for

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1O1···O3^{i}	0.88 (3)	1.92 (3)	2.770 (2)	164 (3)
N1—H1N1···O2^{ii}	0.93 (2)	1.89 (2)	2.792 (2)	166 (2)
C5—H5···O2	0.93	2.15	2.816 (2)	127

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

about 2 h. The product was filtered off. Single crystals suitable for X-ray diffraction were formed on slow evaporation of the solution in a few days.

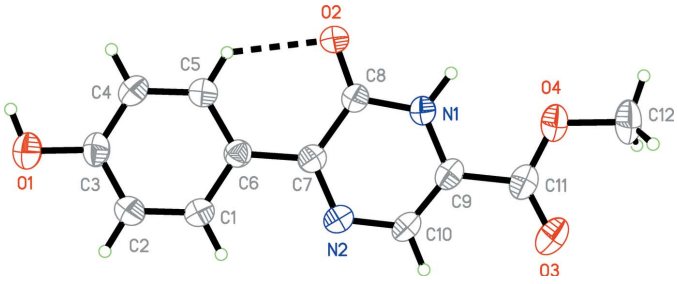


Figure 1
The molecular structure of the title compound, shown with 50% probability displacement ellipsoids and the atom-numbering scheme. The intramolecular hydrogen bond is shown as a dashed line.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{10}N_2O_4$
M_r	246.22
Crystal system, space group	Monoclinic, $C2/c$
Temperature (K)	273
a, b, c (Å)	23.9226 (19), 7.1372 (6), 13.0562 (11)
β (°)	99.158 (5)
V (Å^3)	2200.8 (3)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.30 × 0.20 × 0.03
Data collection	
Diffractometer	Bruker D8 Quest
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.656, 0.745
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	20352, 2011, 1450
R_{int}	0.075
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.602
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.115, 1.07
No. of reflections	2011
No. of parameters	171
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.14, −0.21

Computer programs: *APEX3* (Bruker, 2016), *SAINT* (Bruker, 2016), *SHELXS2014* (Sheldrick, 2008), *SHELXTL* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *PLATON* (Spek, 2015).

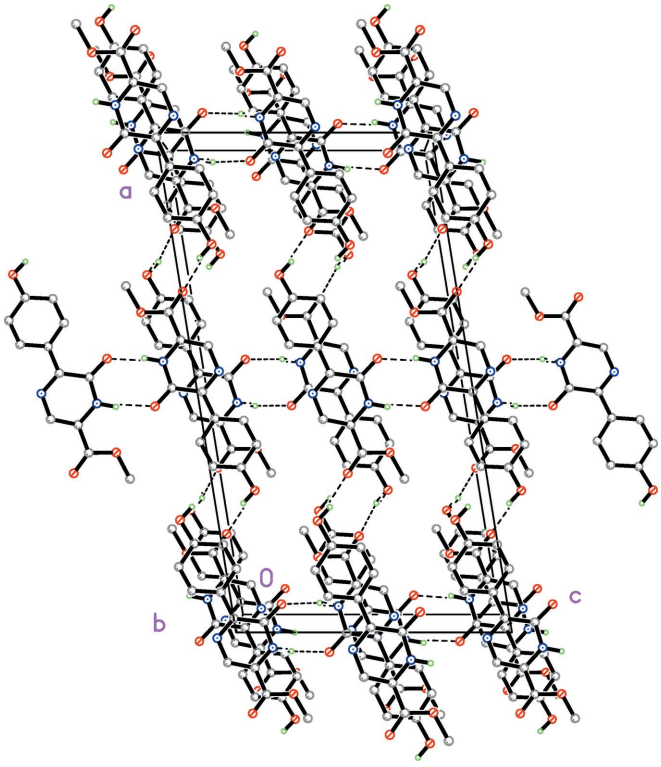


Figure 2
The crystal packing of the title compound viewed down the b axis. Hydrogen bonds are shown as dashed lines and H atoms not involved in the hydrogen bonds have been omitted.

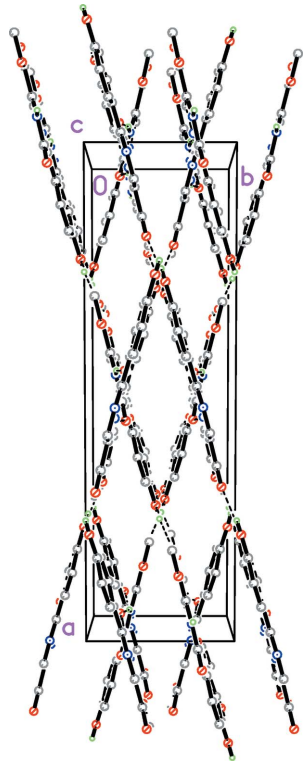


Figure 3
The crystal packing of the title compound viewed down the c axis. Hydrogen bonds are shown as dashed lines and H atoms not involved in the hydrogen bonds have been omitted.

Refinement

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

Acknowledgements

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full crystallographic data

IUCrData (2016). **1**, x161689 [https://doi.org/10.1107/S2414314616016898]

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Crystal data

$C_{12}H_{10}N_2O_4$
M_r = 246.22
Monoclinic, $C2/c$
a = 23.9226 (19) Å
b = 7.1372 (6) Å
c = 13.0562 (11) Å
β = 99.158 (5)°
V = 2200.8 (3) Å^3
Z = 8
$F(000)$ = 1024
D_x = 1.486 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 184 reflections
θ = 3.2–18.6°
μ = 0.11 mm^{-1}
T = 273 K
Plate, colourless
0.30 × 0.20 × 0.03 mm

Data collection

Bruker D8 Quest diffractometer
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2016)
T_{min} = 0.656, T_{max} = 0.745
20352 measured reflections
2011 independent reflections
1450 reflections with $I > 2\sigma(I)$
R_{int} = 0.075
θ_{max} = 25.4°, θ_{min} = 3.0°
h = −28→28
k = −8→8
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.115
S = 1.07
2011 reflections
171 parameters
0 restraints
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 1.083P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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	0.27612 (6)	0.4678 (2)	0.63884 (13)	0.0586 (5)

H1O1	0.2492 (12)	0.494 (4)	0.587 (2)	0.095 (10)*
O2	0.45124 (6)	0.2748 (3)	0.32435 (10)	0.0591 (5)
O3	0.68297 (6)	0.0924 (2)	0.50218 (12)	0.0608 (5)
O4	0.64150 (5)	0.1191 (2)	0.33671 (11)	0.0534 (4)
N1	0.54260 (6)	0.2092 (2)	0.38434 (12)	0.0364 (4)
H1N1	0.5495 (10)	0.216 (3)	0.3167 (18)	0.066 (7)*
N2	0.52255 (6)	0.2191 (2)	0.58370 (12)	0.0405 (4)
C1	0.42192 (8)	0.3329 (3)	0.64301 (14)	0.0428 (5)
H1	0.4536	0.3093	0.6926	0.051*
C2	0.37228 (8)	0.3858 (3)	0.67471 (15)	0.0467 (5)
H2	0.3709	0.3998	0.7451	0.056*
C3	0.32447 (8)	0.4184 (3)	0.60294 (15)	0.0400 (5)
C4	0.32729 (8)	0.3998 (3)	0.49911 (15)	0.0472 (5)
H4	0.2952	0.4215	0.4501	0.057*
C5	0.37741 (8)	0.3490 (3)	0.46728 (15)	0.0442 (5)
H5	0.3787	0.3381	0.3967	0.053*
C6	0.42609 (7)	0.3136 (2)	0.53830 (13)	0.0317 (4)
C7	0.48090 (7)	0.2593 (2)	0.50891 (13)	0.0326 (4)
C8	0.48888 (7)	0.2495 (3)	0.39935 (14)	0.0368 (5)
C9	0.58564 (7)	0.1695 (3)	0.46318 (14)	0.0357 (5)
C10	0.57426 (8)	0.1723 (3)	0.56099 (15)	0.0419 (5)
H10	0.6029	0.1410	0.6151	0.050*
C11	0.64207 (8)	0.1228 (3)	0.43776 (16)	0.0407 (5)
C12	0.69404 (9)	0.0771 (4)	0.2991 (2)	0.0649 (7)
H12A	0.6877	0.0789	0.2247	0.097*
H12B	0.7220	0.1693	0.3248	0.097*
H12C	0.7072	−0.0448	0.3231	0.097*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0377 (8)	0.0876 (13)	0.0530 (10)	0.0142 (8)	0.0146 (7)	0.0015 (9)
O2	0.0356 (8)	0.1114 (14)	0.0297 (7)	0.0175 (8)	0.0036 (6)	0.0044 (8)
O3	0.0334 (8)	0.0799 (12)	0.0665 (10)	0.0118 (8)	−0.0006 (7)	0.0050 (8)
O4	0.0345 (8)	0.0720 (11)	0.0562 (10)	0.0112 (7)	0.0151 (7)	0.0034 (8)
N1	0.0289 (8)	0.0481 (10)	0.0333 (9)	0.0019 (7)	0.0078 (7)	0.0035 (7)
N2	0.0326 (9)	0.0521 (11)	0.0349 (9)	0.0047 (7)	0.0002 (7)	0.0006 (7)
C1	0.0336 (10)	0.0590 (14)	0.0343 (10)	0.0054 (9)	0.0011 (8)	−0.0002 (9)
C2	0.0409 (11)	0.0674 (15)	0.0321 (10)	0.0045 (10)	0.0071 (9)	−0.0022 (10)
C3	0.0317 (10)	0.0442 (12)	0.0463 (12)	0.0018 (9)	0.0125 (9)	0.0003 (9)
C4	0.0310 (10)	0.0667 (15)	0.0421 (12)	0.0078 (10)	0.0006 (9)	0.0020 (10)
C5	0.0369 (11)	0.0627 (14)	0.0328 (10)	0.0082 (10)	0.0047 (8)	−0.0002 (10)
C6	0.0305 (9)	0.0327 (11)	0.0316 (9)	−0.0010 (7)	0.0041 (7)	−0.0001 (8)
C7	0.0313 (9)	0.0324 (10)	0.0334 (10)	−0.0004 (8)	0.0037 (8)	0.0015 (8)
C8	0.0289 (9)	0.0475 (12)	0.0341 (10)	0.0023 (8)	0.0051 (8)	0.0032 (9)
C9	0.0272 (9)	0.0343 (11)	0.0443 (11)	−0.0004 (8)	0.0020 (8)	0.0022 (9)
C10	0.0279 (9)	0.0544 (13)	0.0413 (11)	0.0070 (9)	−0.0013 (8)	0.0020 (9)
C11	0.0314 (10)	0.0378 (12)	0.0528 (13)	0.0014 (9)	0.0069 (9)	0.0033 (9)

C12	0.0405 (12)	0.0851 (18)	0.0751 (17)	0.0107 (12)	0.0273 (12)	−0.0004 (14)

Geometric parameters (Å, º)

O1—C3	1.362 (2)	C2—H2	0.9300
O1—H1O1	0.88 (3)	C3—C4	1.375 (3)
O2—C8	1.234 (2)	C4—C5	1.379 (3)
O3—C11	1.204 (2)	C4—H4	0.9300
O4—C11	1.317 (2)	C5—C6	1.391 (2)
O4—C12	1.451 (2)	C5—H5	0.9300
N1—C8	1.361 (2)	C6—C7	1.475 (2)
N1—C9	1.365 (2)	C7—C8	1.475 (3)
N1—H1N1	0.92 (2)	C9—C10	1.348 (3)
N2—C7	1.311 (2)	C9—C11	1.479 (3)
N2—C10	1.359 (2)	C10—H10	0.9300
C1—C2	1.371 (3)	C12—H12A	0.9600
C1—C6	1.393 (2)	C12—H12B	0.9600
C1—H1	0.9300	C12—H12C	0.9600
C2—C3	1.378 (3)		
C3—O1—H1O1	110.3 (19)	C1—C6—C7	119.11 (16)
C11—O4—C12	118.10 (16)	N2—C7—C8	120.78 (16)
C8—N1—C9	123.47 (16)	N2—C7—C6	117.73 (16)
C8—N1—H1N1	116.3 (14)	C8—C7—C6	121.49 (15)
C9—N1—H1N1	120.1 (14)	O2—C8—N1	120.22 (16)
C7—N2—C10	120.11 (16)	O2—C8—C7	124.90 (16)
C2—C1—C6	121.61 (17)	N1—C8—C7	114.88 (15)
C2—C1—H1	119.2	C10—C9—N1	118.02 (16)
C6—C1—H1	119.2	C10—C9—C11	123.04 (17)
C1—C2—C3	120.41 (18)	N1—C9—C11	118.93 (17)
C1—C2—H2	119.8	C9—C10—N2	122.58 (17)
C3—C2—H2	119.8	C9—C10—H10	118.7
O1—C3—C4	122.84 (17)	N2—C10—H10	118.7
O1—C3—C2	117.92 (18)	O3—C11—O4	125.00 (18)
C4—C3—C2	119.24 (17)	O3—C11—C9	123.59 (19)
C3—C4—C5	120.30 (18)	O4—C11—C9	111.40 (16)
C3—C4—H4	119.9	O4—C12—H12A	109.5
C5—C4—H4	119.9	O4—C12—H12B	109.5
C4—C5—C6	121.51 (18)	H12A—C12—H12B	109.5
C4—C5—H5	119.2	O4—C12—H12C	109.5
C6—C5—H5	119.2	H12A—C12—H12C	109.5
C5—C6—C1	116.93 (17)	H12B—C12—H12C	109.5
C5—C6—C7	123.96 (16)		
C6—C1—C2—C3	1.1 (3)	C9—N1—C8—C7	3.7 (3)
C1—C2—C3—O1	179.0 (2)	N2—C7—C8—O2	175.80 (19)
C1—C2—C3—C4	−0.9 (3)	C6—C7—C8—O2	−4.0 (3)
O1—C3—C4—C5	−179.8 (2)	N2—C7—C8—N1	−4.3 (3)

C2—C3—C4—C5	0.0 (3)	C6—C7—C8—N1	175.95 (16)
C3—C4—C5—C6	0.6 (3)	C8—N1—C9—C10	−0.7 (3)
C4—C5—C6—C1	−0.3 (3)	C8—N1—C9—C11	178.39 (18)
C4—C5—C6—C7	−179.86 (19)	N1—C9—C10—N2	−2.3 (3)
C2—C1—C6—C5	−0.5 (3)	C11—C9—C10—N2	178.66 (18)
C2—C1—C6—C7	179.01 (19)	C7—N2—C10—C9	1.7 (3)
C10—N2—C7—C8	1.7 (3)	C12—O4—C11—O3	−0.6 (3)
C10—N2—C7—C6	−178.52 (17)	C12—O4—C11—C9	179.36 (18)
C5—C6—C7—N2	−175.99 (19)	C10—C9—C11—O3	−3.7 (3)
C1—C6—C7—N2	4.5 (3)	N1—C9—C11—O3	177.29 (19)
C5—C6—C7—C8	3.8 (3)	C10—C9—C11—O4	176.33 (18)
C1—C6—C7—C8	−175.72 (18)	N1—C9—C11—O4	−2.7 (3)
C9—N1—C8—O2	−176.32 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*1···O3^{i}	0.88 (3)	1.92 (3)	2.770 (2)	164 (3)
N1—H1*N*1···O2^{ii}	0.93 (2)	1.89 (2)	2.792 (2)	166 (2)
C5—H5···O2	0.93	2.15	2.816 (2)	127

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