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5-(4-Methoxyphenyl)-1,3,4-oxadiazol-2-amine

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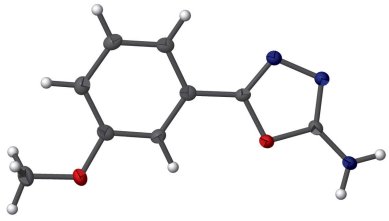
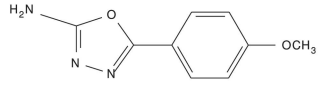
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_9H_9N_3O_2$, the dihedral angle between the aromatic rings is 8.64 (10)°. The crystal structure features inversion-related dimers linked by pairs of N—H···N hydrogen bonds, generating $R_2^2(8)$ loops. A further N—H···N hydrogen bond links the dimers into (100) sheets.

3D view



Chemical scheme



Structure description

Derivatives of 1,3,4-oxadiazole exhibit a broad spectrum of pharmaceutical applications such as antibacterial, anticonvulsant (Taha *et al.*, 2016), anti-inflammatory, anticancer, analgesic and fungicidal. As a part of our ongoing research on such molecules (Yasser *et al.*, 2016) we report herein on the synthesis and crystal structure of the title compound (Fig. 1).

The molecule is approximately planar as indicated by the dihedral angle value of 8.64 (10)° between the aromatic rings. The methoxy group lies almost in the plane of the phenyl ring as indicated by the torsion angle value of −5.1 (3)° for C9—O2—C5—C6. The crystal structure features inversion-related dimers linked by pairs of N—H···N hydrogen bonds generating $R_2^2(8)$ loops (Table 1 and Fig. 2). A further N—H···N hydrogen bond links the dimers into (100) sheets.

Synthesis and crystallization

To a solution of 1-(4-methoxybenzylidene)semicarbazide in ethanol, chloramine-T was added and refluxed. The reaction was monitored by TLC and the after completion of the reaction, the sodium chloride formed in the reaction was filtered and the filtrate was

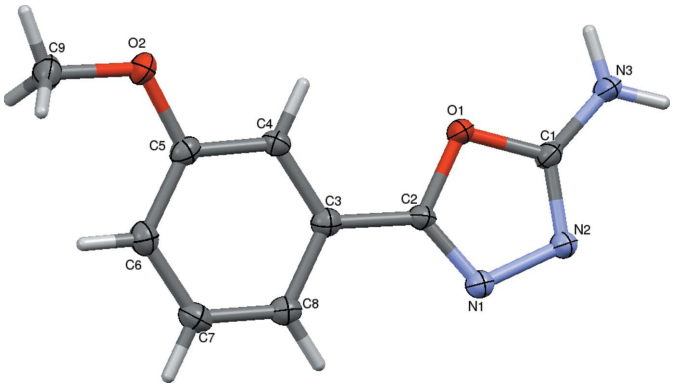


Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

concentrated and extracted to dichloromethane. The organic layer was washed with 10% hydrochloric acid, the aqueous layer was neutralized with 10% sodium hydroxide and the white solid obtained was further purified. Colourless crystals formed after 3 days due to the slow evaporation of the solvent. Yield 84%, m.p. 246–248°C.

IR (KBr, γ/cm^{-1}): 3409–3490 (COOH), 3050 (CH), 1730 (CO of COOH), 1675 (CO of $OCOCH_3$). ^{1}H NMR (400 MHz, DMSO-d^6): δ 3.9 (*s*, 3H,CH_3) 5.1 (*s*, 2H,NH_2), 7.1–08.1 (*m*, 4H, ArH), ^{13}C NMR: 155.32, 150.06, 129.21, 125.43, 124.31, 54.32. LCMS (M^+): (191). Analysis calculated for $C_9H_9N_3O_2$: C, 56.54; H, 4.74; N, 21.98; found: C, 56.01; H, 4.56; N, 21.68%.

Refinement

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

Acknowledgements

The authors thank the Institution of Excellence, Vijnana Bhavana, University of Mysore, India, for providing the single-crystal X-ray diffractometer facility. NP gratefully acknowledges the financial support of UGC MRP(S)-0551–13-14/KAMY013/UGC-SWRO. Zabiulla gratefully acknowledges the financial support provided by the Department of Science and Technology, New Delhi, under the INSPIRE–Fellowship scheme. SAK thankfully acknowledges the financial support provided by VGST, Bangalore, under the CISEE Programme.

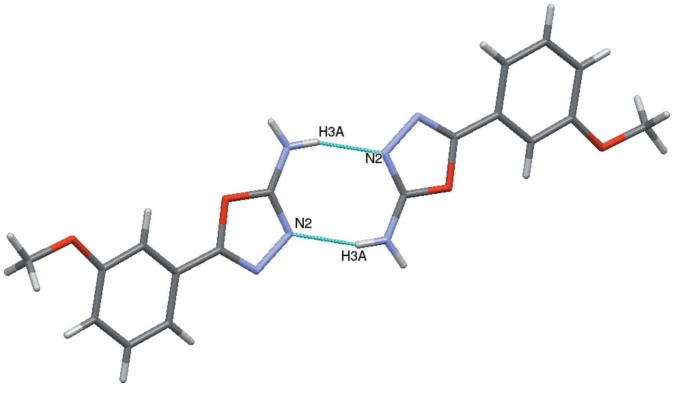


Figure 2
Inversion-related dimers linked by pairs of N—H···O hydrogen bonds, generating $R_2^2(8)$ loops. The right-hand molecule is generated by the symmetry operation $2-x, 3-y, 2-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···N2^{i}	0.86	2.09	2.924 (2)	163
N3—H3B···N1^{ii}	0.86	2.13	2.969 (2)	164

Symmetry codes: (i) $-x+2, -y+3, -z+2$; (ii) $x, -y+\frac{5}{2}, z-\frac{1}{2}$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_9H_9N_3O_2$
M_r	191.19
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	16.2029 (9), 5.0730 (3), 11.1133 (6)
β (°)	108.096 (3)
V (Å^3)	868.30 (9)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.90
Crystal size (mm)	0.28 × 0.26 × 0.23
Data collection	
Diffractometer	Bruker X8 Proteum
Absorption correction	Multi-scan (*SADABS*; Bruker, 2013)
T_{min}, T_{max}	0.788, 0.821
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5230, 1430, 1308
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.587
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.142, 1.15
No. of reflections	1430
No. of parameters	129
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.32, −0.31

Computer programs: *APEX2* and *SAINT* (Bruker, 2013), *SHELXS97* and *SHELXL97* (Sheldrick, 2008) and *Mercury* (Macrae *et al.*, 2008).

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full crystallographic data

IUCrData (2016). **1**, x161896 [https://doi.org/10.1107/S2414314616018964]

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5-(4-Methoxyphenyl)-1,3,4-oxadiazol-2-amine

Crystal data

$C_9H_9N_3O_2$
M_r = 191.19
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 16.2029 (9) Å
b = 5.0730 (3) Å
c = 11.1133 (6) Å
β = 108.096 (3)°
V = 868.30 (9) Å^3
Z = 4
$F(000)$ = 400
D_x = 1.463 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 1308 reflections
θ = 8.0–64.8°
μ = 0.90 mm^{-1}
T = 296 K
Block, colourless
0.28 × 0.26 × 0.23 mm

Data collection

Bruker X8 Proteum diffractometer
Radiation source: Bruker MicroStar microfocus rotating anode
Helios multilayer optics monochromator
Detector resolution: 18.4 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2013)
T_{min} = 0.788, T_{max} = 0.821
5230 measured reflections
1430 independent reflections
1308 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 64.8°, θ_{min} = 8.0°
h = −18→18
k = −5→5
l = −12→12

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.142
S = 1.15
1430 reflections
129 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
$w = 1/[\sigma^2(F_o^2) + (0.0816P)^2 + 0.4652P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Extinction correction: SHELXL97 (Sheldrick, 2008), FC*=KFC[1+0.001XFC$^2\Lambda^3$/SIN(2Θ)]$^{-1/4}$
Extinction coefficient: 0.0050 (13)

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 esds are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating -R-factor-obs 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.
H atoms were fixed geometrically (C—H = 0.93–0.96 Å) and allowed to ride on their parent atoms with U_{iso}(H) = 1.5U_{eq}(C) for the methyl H atoms and = 1.2U_{eq}(C) for the others.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.84349 (8)	1.0817 (3)	0.83232 (11)	0.0197 (4)
O2	0.58911 (9)	0.4726 (3)	0.65817 (13)	0.0230 (4)
N1	0.88643 (10)	1.0232 (3)	1.03973 (15)	0.0186 (5)
N2	0.93522 (10)	1.2299 (3)	1.01268 (14)	0.0190 (5)
N3	0.93358 (12)	1.4284 (4)	0.81916 (15)	0.0264 (6)
C1	0.90787 (12)	1.2572 (4)	0.88958 (17)	0.0183 (6)
C2	0.83441 (12)	0.9410 (4)	0.93398 (17)	0.0164 (6)
C3	0.76937 (12)	0.7335 (4)	0.91245 (17)	0.0186 (6)
C4	0.70870 (12)	0.6920 (4)	0.79427 (17)	0.0178 (6)
C5	0.64685 (12)	0.4939 (4)	0.77827 (18)	0.0190 (6)
C6	0.64528 (13)	0.3346 (4)	0.87986 (19)	0.0217 (6)
C7	0.70707 (13)	0.3786 (4)	0.99784 (18)	0.0223 (6)
C8	0.76843 (12)	0.5742 (4)	1.01542 (18)	0.0196 (6)
C9	0.52835 (14)	0.2595 (4)	0.6353 (2)	0.0267 (7)
H3A	0.97340	1.54120	0.85420	0.0320*
H3B	0.91050	1.42720	0.73820	0.0320*
H4	0.70950	0.79660	0.72590	0.0210*
H6	0.60400	0.20200	0.86940	0.0260*
H7	0.70660	0.27310	1.06610	0.0270*
H8	0.80890	0.60070	1.09470	0.0230*
H9A	0.55920	0.09510	0.65060	0.0400*
H9B	0.49150	0.26560	0.54900	0.0400*
H9C	0.49360	0.27480	0.69090	0.0400*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0222 (7)	0.0219 (8)	0.0132 (7)	−0.0060 (6)	0.0029 (5)	−0.0003 (5)
O2	0.0206 (8)	0.0251 (8)	0.0201 (7)	−0.0072 (6)	0.0016 (6)	−0.0015 (6)
N1	0.0196 (9)	0.0198 (9)	0.0157 (8)	−0.0016 (7)	0.0045 (7)	0.0003 (6)
N2	0.0197 (9)	0.0217 (9)	0.0149 (8)	−0.0040 (7)	0.0042 (6)	0.0002 (6)
N3	0.0309 (10)	0.0317 (11)	0.0131 (8)	−0.0154 (8)	0.0017 (7)	0.0001 (7)
C1	0.0177 (10)	0.0206 (11)	0.0151 (9)	−0.0029 (8)	0.0030 (7)	−0.0026 (7)
C2	0.0186 (10)	0.0176 (10)	0.0132 (9)	0.0007 (8)	0.0053 (7)	0.0016 (7)

C3	0.0193 (10)	0.0191 (11)	0.0178 (10)	0.0023 (8)	0.0065 (8)	−0.0003 (7)
C4	0.0199 (10)	0.0183 (11)	0.0161 (9)	−0.0009 (8)	0.0069 (8)	0.0016 (8)
C5	0.0192 (10)	0.0197 (11)	0.0176 (10)	0.0028 (8)	0.0050 (8)	−0.0025 (8)
C6	0.0208 (10)	0.0202 (11)	0.0254 (11)	−0.0012 (8)	0.0089 (8)	−0.0004 (8)
C7	0.0261 (11)	0.0212 (11)	0.0215 (10)	0.0033 (8)	0.0103 (8)	0.0047 (8)
C8	0.0199 (10)	0.0216 (11)	0.0172 (10)	0.0031 (8)	0.0058 (8)	0.0007 (8)
C9	0.0256 (11)	0.0225 (12)	0.0288 (11)	−0.0055 (9)	0.0039 (9)	−0.0034 (8)

Geometric parameters (Å, º)

O1—C1	1.369 (2)	C3—C4	1.391 (3)
O1—C2	1.382 (2)	C4—C5	1.391 (3)
O2—C5	1.376 (2)	C5—C6	1.395 (3)
O2—C9	1.431 (3)	C6—C7	1.399 (3)
N1—N2	1.401 (2)	C7—C8	1.375 (3)
N1—C2	1.285 (2)	C4—H4	0.9300
N2—C1	1.308 (2)	C6—H6	0.9300
N3—C1	1.320 (3)	C7—H7	0.9300
N3—H3A	0.8600	C8—H8	0.9300
N3—H3B	0.8600	C9—H9A	0.9600
C2—C3	1.456 (3)	C9—H9B	0.9600
C3—C8	1.405 (3)	C9—H9C	0.9600
C1—O1—C2	102.42 (14)	O2—C5—C6	124.18 (18)
C5—O2—C9	117.05 (16)	C5—C6—C7	118.59 (19)
N2—N1—C2	107.48 (15)	C6—C7—C8	121.59 (18)
N1—N2—C1	105.86 (15)	C3—C8—C7	119.32 (18)
H3A—N3—H3B	120.00	C3—C4—H4	120.00
C1—N3—H3A	120.00	C5—C4—H4	120.00
C1—N3—H3B	120.00	C5—C6—H6	121.00
O1—C1—N2	112.28 (17)	C7—C6—H6	121.00
N2—C1—N3	128.53 (19)	C6—C7—H7	119.00
O1—C1—N3	119.18 (16)	C8—C7—H7	119.00
N1—C2—C3	128.38 (17)	C3—C8—H8	120.00
O1—C2—C3	119.63 (16)	C7—C8—H8	120.00
O1—C2—N1	111.96 (17)	O2—C9—H9A	109.00
C2—C3—C4	121.87 (17)	O2—C9—H9B	109.00
C2—C3—C8	118.20 (17)	O2—C9—H9C	109.00
C4—C3—C8	119.93 (18)	H9A—C9—H9B	109.00
C3—C4—C5	120.00 (18)	H9A—C9—H9C	109.00
C4—C5—C6	120.57 (18)	H9B—C9—H9C	109.00
O2—C5—C4	115.25 (17)		
C2—O1—C1—N2	−0.4 (2)	N1—C2—C3—C4	170.2 (2)
C2—O1—C1—N3	−179.58 (19)	N1—C2—C3—C8	−9.3 (3)
C1—O1—C2—N1	0.5 (2)	C2—C3—C4—C5	−179.06 (19)
C1—O1—C2—C3	178.77 (18)	C8—C3—C4—C5	0.4 (3)
C9—O2—C5—C4	175.65 (18)	C2—C3—C8—C7	179.34 (19)

C9—O2—C5—C6	−5.1 (3)	C4—C3—C8—C7	−0.1 (3)
C2—N1—N2—C1	0.2 (2)	C3—C4—C5—O2	178.97 (18)
N2—N1—C2—O1	−0.5 (2)	C3—C4—C5—C6	−0.3 (3)
N2—N1—C2—C3	−178.51 (19)	O2—C5—C6—C7	−179.19 (19)
N1—N2—C1—O1	0.2 (2)	C4—C5—C6—C7	0.1 (3)
N1—N2—C1—N3	179.2 (2)	C5—C6—C7—C8	0.2 (3)
O1—C2—C3—C4	−7.8 (3)	C6—C7—C8—C3	−0.2 (3)
O1—C2—C3—C8	172.78 (18)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*A*···N2^{i}	0.86	2.09	2.924 (2)	163
N3—H3*B*···N1^{ii}	0.86	2.13	2.969 (2)	164

Symmetry codes: (i) −*x*+2, −*y*+3, −*z*+2; (ii) *x*, −*y*+5/2, *z*−1/2.