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3-[5-(Pyridin-4-yl)-1,3,4-oxadiazol-2-yl]-4*H*-chromen-4-one

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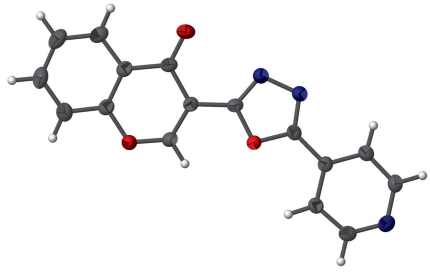
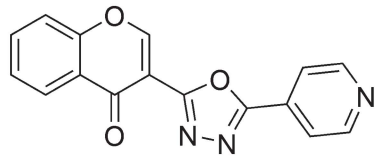
Structural data: full structural data are available from iucrdata.iucr.org

In the title molecule, $C_{16}H_9N_3O_3$, the plane of oxadiazole ring is almost coplanar with attached pyridine ring and chromenyl ring system, forming dihedral angles of 2.37 (3) and 6.71 (2)°, respectively. In the crystal, a pair of C—H···O and C—H···N hydrogen-bond interactions lead to the formation of dimers, which are linked together into [100] chains.

3D view



Chemical scheme



Structure description

Heterocycles possessing five-membered rings have attracted lots of interest due to their versatile pharmacological and biological activities. Oxadiazole is a furan-type five-membered ring heterocycle in which two nitrogen atoms substitute two carbons. Depending on the positions of the replacing nitrogen atoms, several types of oxadiazole isomers are formed. Examples of oxadiazole structures have been published (dos Santos *et al.*, 2014; Sharma *et al.*, 2014). In particular, 1,3,4-oxadiazoles have been extensively investigated in medicinal chemistry. They have been found to show anti-fungal (Wani *et al.* 2015), anti-inflammatory (Banerjee *et al.* 2015), anti-microbial (Li *et al.* 2015), and anticancer activities (Mochona *et al.* 2016).

We have focused our research on chalcones (Shin *et al.* 2014, Lee *et al.* 2016), which have a central conjugated enone system connecting two aromatic rings. In a continuation of this research program, the central enone of chalcone was modified to an oxadiazole ring. Herein, the crystal structure of title oxadiazole compound is reported.

The molecular structure is shown in Fig. 1. The plane of the oxadiazole ring is almost co-planar with attached pyridine ring and chromenyl ring system, making dihedral angles of 2.37 (3) and 6.71 (2)°, respectively. In the crystal, a pair of C—H···O and C—H···N hydrogen bonds (Table 1) lead to the formation of dimers, which are linked together into [100] chains (Fig. 2).

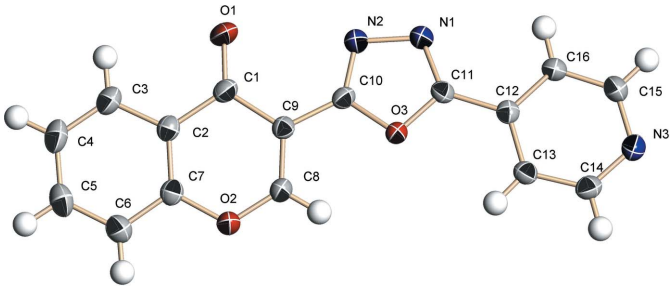


Figure 1
The molecular structure of the title compound, showing displacement ellipsoids drawn at the 50% probability level.

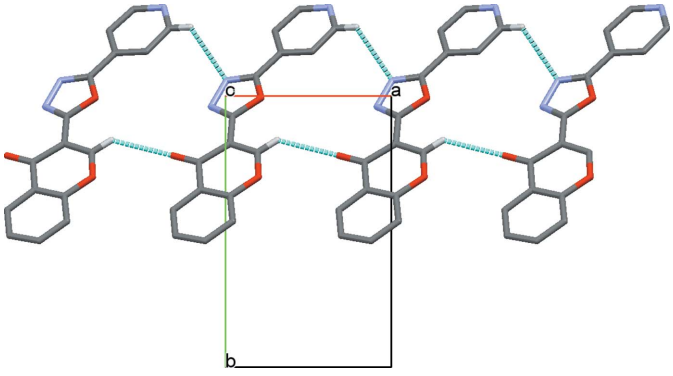


Figure 2
Part of the crystal structure of the title compound, showing the C—H···O and C—H···N hydrogen bonds as dashed lines. H atoms not involved in these interactions have been omitted for clarity.

Synthesis and crystallization

The synthetic procedure is shown in Fig. 3. To a solution of 4-oxo-4*H*-chromene-3-carbaldehyde (348 mg, 2 mmol) in 20 ml of anhydrous ethanol was added isonicotinohydrazide (274 mg, 2 mmol) and a catalytic amount of glacial acetic acid. The temperature of the mixture was adjusted to around 358 K in an oil-bath and the mixture was refluxed for 5 h. Then the reaction mixture was cooled down to the room temperature to give a precipitate. Filtration and washing with ethanol afforded a solid of the intermediate compound (**I**, 88%) which was used in the next step without further purification. The intermediate compound (**I**, 196 mg, 0.5 mmol) was dissolved in

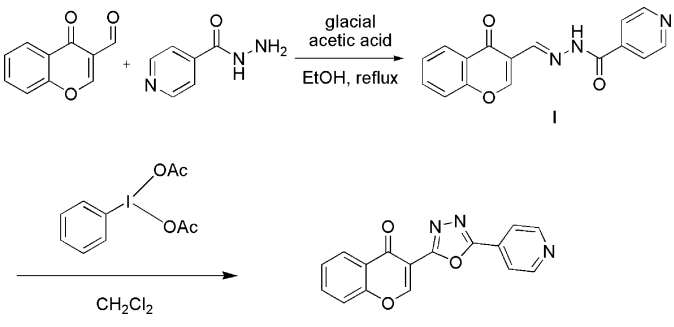


Figure 3
The synthetic procedure for the title compound.

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1^{i}	0.94	2.55	3.2517 (19)	132
C14—H14···N1^{i}	0.94	2.63	3.248 (2)	123

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_9N_3O_3$
M_r	291.26
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	223
a, b, c (Å)	6.7439 (2), 10.8441 (3), 17.7415 (6)
β (°)	100.7317 (13)
V (Å^3)	1274.77 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.11
Crystal size (mm)	0.13 × 0.12 × 0.09
Data collection	
Diffractometer	Bruker SMART CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2000)
T_{min}, T_{max}	0.986, 0.990
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	38727, 3188, 2438
R_{int}	0.050
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.670
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.112, 1.07
No. of reflections	3188
No. of parameters	199
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.19

Computer programs: *APEX2* and *SAINT* (Bruker, 2000) and *SHELXTL* (Sheldrick, 2008).

10 ml of dichloromethane and iodobenzenediacetate (276 mg, 0.8 mmol) was added. The reaction mixture was stirred at room temperature for 12 h and the solvent was evaporated under vacuum to produce a solid. Recrystallization of the solid from methanol solution gave yellow block-shaped crystals for this study (m.p. 513–514 K).

Refinement

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

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full crystallographic data

IUCrData (2016). **1**, x161625 [https://doi.org/10.1107/S2414314616016254]

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Crystal data

$C_{16}H_9N_3O_3$
M_r = 291.26
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 6.7439 (2) Å
b = 10.8441 (3) Å
c = 17.7415 (6) Å
β = 100.7317 (13)°
V = 1274.77 (7) Å^3
Z = 4
$F(000)$ = 600
D_x = 1.518 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9683 reflections
θ = 2.2–28.3°
μ = 0.11 mm^{-1}
T = 223 K
Block, yellow
0.13 × 0.12 × 0.09 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2000)
T_{min} = 0.986, T_{max} = 0.990
38727 measured reflections
3188 independent reflections
2438 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 28.4°, θ_{min} = 2.2°
h = −9→9
k = −14→14
l = −23→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.112
S = 1.07
3188 reflections
199 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 0.5495P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 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.

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.1618 (2)	0.24444 (14)	0.01388 (8)	0.0301 (3)
O1	−0.31711 (18)	0.22005 (13)	0.03664 (8)	0.0540 (4)
C2	−0.1492 (2)	0.34564 (14)	−0.04011 (8)	0.0282 (3)
C3	−0.3161 (2)	0.42116 (15)	−0.06691 (9)	0.0361 (4)
H3	−0.4382	0.4078	−0.0498	0.043*
C4	−0.3024 (3)	0.51438 (16)	−0.11791 (10)	0.0421 (4)
H4	−0.4145	0.5652	−0.1353	0.050*
C5	−0.1229 (3)	0.53389 (16)	−0.14399 (10)	0.0417 (4)
H5	−0.1156	0.5972	−0.1796	0.050*
C6	0.0439 (3)	0.46201 (15)	−0.11854 (9)	0.0373 (4)
H6	0.1654	0.4756	−0.1360	0.045*
C7	0.0282 (2)	0.36883 (13)	−0.06637 (8)	0.0287 (3)
O2	0.19977 (16)	0.29944 (10)	−0.04199 (6)	0.0342 (3)
C8	0.1928 (2)	0.20816 (14)	0.00834 (9)	0.0299 (3)
H8	0.3118	0.1630	0.0251	0.036*
C9	0.0281 (2)	0.17627 (13)	0.03673 (8)	0.0254 (3)
C10	0.0427 (2)	0.07610 (13)	0.09176 (8)	0.0266 (3)
O3	0.21616 (14)	0.00778 (9)	0.10556 (5)	0.0247 (2)
C11	0.1816 (2)	−0.07098 (13)	0.16119 (8)	0.0265 (3)
N1	0.0061 (2)	−0.05671 (13)	0.17827 (9)	0.0408 (3)
N2	−0.0862 (2)	0.04037 (13)	0.13246 (9)	0.0411 (4)
C12	0.3365 (2)	−0.15833 (13)	0.19546 (8)	0.0257 (3)
C13	0.5241 (2)	−0.16371 (13)	0.17404 (8)	0.0285 (3)
H13	0.5565	−0.1107	0.1362	0.034*
C14	0.6622 (2)	−0.24943 (14)	0.21005 (9)	0.0319 (3)
H14	0.7887	−0.2532	0.1951	0.038*
N3	0.6283 (2)	−0.32645 (12)	0.26410 (8)	0.0350 (3)
C15	0.4466 (2)	−0.31961 (15)	0.28450 (9)	0.0363 (4)
H15	0.4194	−0.3732	0.3230	0.044*
C16	0.2980 (2)	−0.23835 (14)	0.25208 (9)	0.0320 (3)
H16	0.1727	−0.2371	0.2680	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0233 (7)	0.0345 (8)	0.0325 (7)	0.0026 (6)	0.0051 (6)	0.0007 (6)
O1	0.0276 (6)	0.0684 (9)	0.0695 (9)	0.0109 (6)	0.0183 (6)	0.0293 (7)
C2	0.0285 (7)	0.0289 (7)	0.0260 (7)	0.0024 (6)	0.0014 (5)	−0.0017 (6)
C3	0.0293 (8)	0.0385 (9)	0.0381 (8)	0.0055 (6)	0.0002 (6)	−0.0002 (7)
C4	0.0415 (9)	0.0385 (9)	0.0411 (9)	0.0092 (7)	−0.0056 (7)	0.0044 (7)

C5	0.0524 (10)	0.0362 (9)	0.0337 (8)	0.0030 (8)	0.0007 (7)	0.0089 (7)
C6	0.0413 (9)	0.0365 (8)	0.0343 (8)	−0.0002 (7)	0.0079 (7)	0.0042 (7)
C7	0.0295 (7)	0.0287 (7)	0.0268 (7)	0.0024 (6)	0.0027 (6)	−0.0012 (6)
O2	0.0285 (5)	0.0367 (6)	0.0390 (6)	0.0048 (4)	0.0109 (4)	0.0109 (5)
C8	0.0260 (7)	0.0306 (7)	0.0332 (7)	0.0042 (6)	0.0060 (6)	0.0041 (6)
C9	0.0243 (7)	0.0245 (7)	0.0269 (7)	0.0002 (5)	0.0036 (5)	−0.0011 (5)
C10	0.0219 (7)	0.0262 (7)	0.0318 (7)	0.0015 (5)	0.0057 (6)	−0.0016 (6)
O3	0.0234 (5)	0.0253 (5)	0.0257 (5)	0.0014 (4)	0.0050 (4)	0.0013 (4)
C11	0.0275 (7)	0.0241 (7)	0.0285 (7)	−0.0029 (5)	0.0066 (6)	0.0007 (6)
N1	0.0312 (7)	0.0402 (8)	0.0542 (9)	0.0060 (6)	0.0163 (6)	0.0192 (7)
N2	0.0323 (7)	0.0403 (8)	0.0543 (9)	0.0080 (6)	0.0171 (6)	0.0183 (7)
C12	0.0268 (7)	0.0235 (7)	0.0261 (7)	−0.0020 (5)	0.0027 (5)	−0.0020 (5)
C13	0.0290 (7)	0.0269 (7)	0.0305 (7)	−0.0017 (6)	0.0073 (6)	0.0013 (6)
C14	0.0261 (7)	0.0327 (8)	0.0367 (8)	−0.0013 (6)	0.0055 (6)	−0.0023 (6)
N3	0.0305 (7)	0.0331 (7)	0.0402 (7)	0.0027 (5)	0.0037 (6)	0.0049 (6)
C15	0.0360 (8)	0.0352 (8)	0.0377 (8)	0.0000 (7)	0.0071 (7)	0.0105 (7)
C16	0.0287 (8)	0.0327 (8)	0.0357 (8)	−0.0013 (6)	0.0088 (6)	0.0050 (6)

Geometric parameters (Å, º)

C1—O1	1.2198 (19)	C9—C10	1.452 (2)
C1—C9	1.4687 (19)	C10—N2	1.2886 (19)
C1—C2	1.470 (2)	C10—O3	1.3677 (16)
C2—C7	1.385 (2)	O3—C11	1.3579 (16)
C2—C3	1.402 (2)	C11—N1	1.2844 (19)
C3—C4	1.371 (2)	C11—C12	1.4566 (19)
C3—H3	0.9400	N1—N2	1.4026 (19)
C4—C5	1.390 (3)	C12—C16	1.388 (2)
C4—H4	0.9400	C12—C13	1.389 (2)
C5—C6	1.374 (2)	C13—C14	1.385 (2)
C5—H5	0.9400	C13—H13	0.9400
C6—C7	1.388 (2)	C14—N3	1.323 (2)
C6—H6	0.9400	C14—H14	0.9400
C7—O2	1.3804 (17)	N3—C15	1.343 (2)
O2—C8	1.3398 (17)	C15—C16	1.377 (2)
C8—C9	1.347 (2)	C15—H15	0.9400
C8—H8	0.9400	C16—H16	0.9400
O1—C1—C9	123.91 (14)	C10—C9—C1	120.58 (12)
O1—C1—C2	122.36 (14)	N2—C10—O3	112.35 (13)
C9—C1—C2	113.73 (12)	N2—C10—C9	129.32 (13)
C7—C2—C3	117.99 (14)	O3—C10—C9	118.32 (12)
C7—C2—C1	120.88 (13)	C11—O3—C10	102.35 (11)
C3—C2—C1	121.13 (14)	N1—C11—O3	112.72 (13)
C4—C3—C2	120.38 (16)	N1—C11—C12	126.91 (13)
C4—C3—H3	119.8	O3—C11—C12	120.36 (12)
C2—C3—H3	119.8	C11—N1—N2	106.41 (12)
C3—C4—C5	120.12 (15)	C10—N2—N1	106.13 (12)

C3—C4—H4	119.9	C16—C12—C13	118.32 (13)
C5—C4—H4	119.9	C16—C12—C11	119.51 (13)
C6—C5—C4	120.97 (16)	C13—C12—C11	122.17 (13)
C6—C5—H5	119.5	C14—C13—C12	118.09 (13)
C4—C5—H5	119.5	C14—C13—H13	121.0
C5—C6—C7	118.22 (16)	C12—C13—H13	121.0
C5—C6—H6	120.9	N3—C14—C13	124.45 (14)
C7—C6—H6	120.9	N3—C14—H14	117.8
O2—C7—C2	121.44 (13)	C13—C14—H14	117.8
O2—C7—C6	116.25 (14)	C14—N3—C15	116.74 (13)
C2—C7—C6	122.30 (14)	N3—C15—C16	123.53 (14)
C8—O2—C7	118.70 (12)	N3—C15—H15	118.2
O2—C8—C9	124.91 (13)	C16—C15—H15	118.2
O2—C8—H8	117.5	C15—C16—C12	118.86 (14)
C9—C8—H8	117.5	C15—C16—H16	120.6
C8—C9—C10	119.08 (13)	C12—C16—H16	120.6
C8—C9—C1	120.32 (13)		

O1—C1—C2—C7	−178.30 (15)	C1—C9—C10—N2	−7.4 (2)
C9—C1—C2—C7	1.2 (2)	C8—C9—C10—O3	−8.0 (2)
O1—C1—C2—C3	1.4 (2)	C1—C9—C10—O3	173.75 (12)
C9—C1—C2—C3	−179.09 (13)	N2—C10—O3—C11	−2.00 (16)
C7—C2—C3—C4	0.4 (2)	C9—C10—O3—C11	177.07 (12)
C1—C2—C3—C4	−179.31 (14)	C10—O3—C11—N1	1.97 (16)
C2—C3—C4—C5	0.6 (3)	C10—O3—C11—C12	−177.28 (12)
C3—C4—C5—C6	−1.0 (3)	O3—C11—N1—N2	−1.26 (18)
C4—C5—C6—C7	0.4 (3)	C12—C11—N1—N2	177.93 (14)
C3—C2—C7—O2	179.26 (13)	O3—C10—N2—N1	1.33 (18)
C1—C2—C7—O2	−1.1 (2)	C9—C10—N2—N1	−177.61 (14)
C3—C2—C7—C6	−1.0 (2)	C11—N1—N2—C10	−0.05 (18)
C1—C2—C7—C6	178.66 (14)	N1—C11—C12—C16	1.1 (2)
C5—C6—C7—O2	−179.60 (14)	O3—C11—C12—C16	−179.80 (13)
C5—C6—C7—C2	0.7 (2)	N1—C11—C12—C13	−178.16 (15)
C2—C7—O2—C8	−0.2 (2)	O3—C11—C12—C13	1.0 (2)
C6—C7—O2—C8	−179.93 (13)	C16—C12—C13—C14	0.5 (2)
C7—O2—C8—C9	1.3 (2)	C11—C12—C13—C14	179.71 (13)
O2—C8—C9—C10	−179.26 (13)	C12—C13—C14—N3	−0.5 (2)
O2—C8—C9—C1	−1.0 (2)	C13—C14—N3—C15	0.1 (2)
O1—C1—C9—C8	179.27 (16)	C14—N3—C15—C16	0.3 (2)
C2—C1—C9—C8	−0.3 (2)	N3—C15—C16—C12	−0.3 (2)
O1—C1—C9—C10	−2.5 (2)	C13—C12—C16—C15	−0.1 (2)
C2—C1—C9—C10	177.96 (12)	C11—C12—C16—C15	−179.37 (14)
C8—C9—C10—N2	170.89 (16)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1^{i}	0.94	2.55	3.2517 (19)	132

C14—H14···N1^{i}	0.94	2.63	3.248 (2)	123

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