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5-[5-(4-Chlorophenyl)isoxazol-3-yl]-*N*-phenyl-1,3,4-oxadiazol-2-amine

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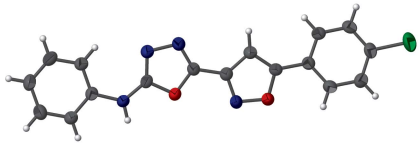
CCDC reference: 1893181

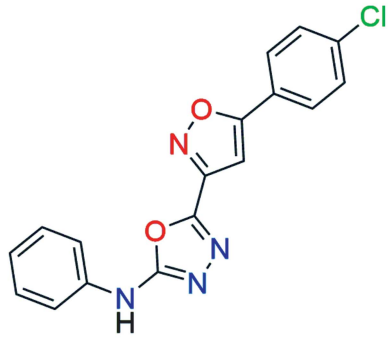
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{17}H_{11}ClN_4O_2$, consists of chlorophenyl (*A*), isoxazolyl (*B*), oxadiazolyl (C) and phenyl (D) rings with twist angles between the planes through neighbouring rings *A*/*B*, *B*/*C* and *C*/*D* of 14.9 (1), 1.8 (1) and 8.9 (1)° respectively. In the crystal, adjacent molecules related by inversion symmetry form columns parallel to the [010] direction through π–π stacking [shortest centroid–centroid separation = 3.6203 (10) Å]; these are cross-linked by N—H···N interactions in the [001] direction.

3D view



Chemical scheme



Structure description

Substituted 1,3,4-oxadiazol-2-amines can act as anticancer and antiproliferative agents (Ahsan *et al.*, 2014; Gonda *et al.*, 2017; Kumar *et al.*, 2009, 2011). As part of our studies in this area, we now describe the synthesis and structure of the title compound.

The molecule consists of chlorophenyl (*A*), isoxazolyl (*B*), oxadiazolyl (*C*) and phenyl (*D*) rings (Fig. 1). The molecule is close to planar, as indicated by the twist angles between the planes through neighbouring rings *A*/*B*, *B*/*C* and *C*/*D*, namely 14.9 (1), 1.8 (1) and 8.9 (1)° respectively. A short intramolecular C—H···N contact is observed (Table 1). In the crystal, adjacent molecules related by inversion symmetry form columns parallel to the [010] direction through π-π stacking [shortest centroid–centroid separation = 3.6203 (10) Å]. The columns of molecules are linked by N—H···N interactions in the [001] direction (Fig. 2).

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

D—H···A	D—H	H···A	D···A	D—H···A
C17—H17···N3	0.93	2.33	2.955 (2)	125
N4—H4···N2^{i}	0.86	2.42	3.176 (2)	146

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

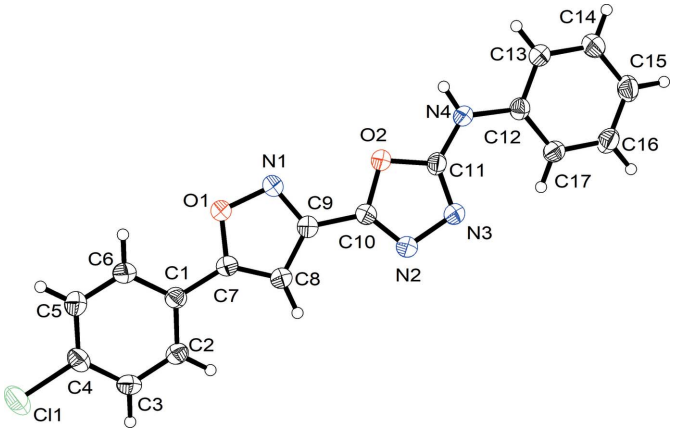


Figure 1
The molecular structure of the title compound showing 50% probability ellipsoids.

Synthesis and crystallization

2-(3-(4-Chlorophenyl)isoxazole-5-carbonyl)-*N*-phenylhydrazinecarbothioamide was treated with anhydrous sodium acetate in ethanol under reflux for 5 h. The resulting solid was collected by filtration, washed with ethanol, dried and recrystallized from dimethylformamide solution to give colourless plates, m.p. 277–278°C.

Refinement

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

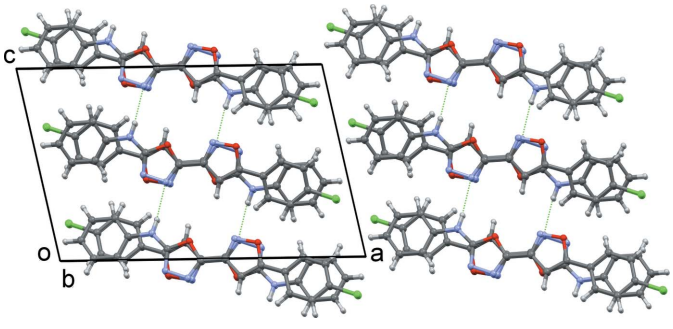


Figure 2
A segment of the crystal structure showing N—H···N hydrogen bonds as dotted lines.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{17}H_{11}ClN_4O_2$
M_r	338.75
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	296
a, b, c (Å)	17.8925 (15), 7.4168 (6), 11.5006 (9)
β (°)	102.061 (8)
V (Å^3)	1492.5 (2)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.27
Crystal size (mm)	0.32 × 0.17 × 0.04
Data collection	
Diffractometer	Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Atlas
Absorption correction	Gaussian (*CrysAlis PRO*; Rigaku OD, 2015)
T_{min}, T_{max}	0.768, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13114, 3720, 2622
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.699
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.112, 1.06
No. of reflections	3720
No. of parameters	218
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.23

Computer programs: *CrysAlis PRO* (Rigaku OD, 2015), *SHELXS* (Sheldrick, 2008), *SHELXL2018* (Sheldrick, 2015), *ORTEP-3 for Windows* and *WinGX* (Farrugia, 2012) and *CHEMDRAW Ultra* (Cambridge Soft, 2001).

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full crystallographic data

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Crystal data

$C_{17}H_{11}ClN_4O_2$
M_r = 338.75
Monoclinic, $P2_1/c$
a = 17.8925 (15) Å
b = 7.4168 (6) Å
c = 11.5006 (9) Å
β = 102.061 (8)°
V = 1492.5 (2) Å^3
Z = 4
$F(000)$ = 696
D_x = 1.508 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3445 reflections
θ = 3.3–26.7°
μ = 0.27 mm^{-1}
T = 296 K
Plate, colourless
0.32 × 0.17 × 0.04 mm

Data collection

Rigaku Oxford Diffraction SuperNova, Dual, Cu at zero, Atlas diffractometer
ω scans
Absorption correction: gaussian (CrysAlisPro; Rigaku OD, 2015)
T_{min} = 0.768, T_{max} = 1.000
13114 measured reflections
3720 independent reflections
2622 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 29.8°, θ_{min} = 3.0°
h = −24→23
k = −10→10
l = −13→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.112
S = 1.06
3720 reflections
218 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.039P)^2 + 0.4804P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: SHELXL-2018/1 (Sheldrick 2018), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0044 (7)

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}
C1	0.27509 (9)	0.6433 (2)	0.57408 (14)	0.0354 (4)
C2	0.27554 (10)	0.5711 (2)	0.68585 (14)	0.0380 (4)
H2	0.321696	0.540461	0.735750	0.046*
C3	0.20791 (11)	0.5447 (2)	0.72307 (15)	0.0406 (4)
H3	0.208175	0.496146	0.797718	0.049*
C4	0.13988 (10)	0.5911 (2)	0.64829 (16)	0.0419 (4)
C5	0.13775 (10)	0.6597 (3)	0.53621 (16)	0.0443 (4)
H5	0.091345	0.687890	0.486141	0.053*
C6	0.20539 (10)	0.6856 (2)	0.49975 (15)	0.0410 (4)
H6	0.204587	0.732140	0.424398	0.049*
C7	0.34717 (10)	0.6763 (2)	0.53732 (14)	0.0368 (4)
C8	0.42112 (10)	0.6850 (2)	0.59279 (15)	0.0411 (4)
H8	0.441861	0.666766	0.673050	0.049*
C9	0.45996 (10)	0.7280 (2)	0.50222 (15)	0.0395 (4)
C10	0.54121 (10)	0.7559 (2)	0.50955 (14)	0.0386 (4)
C11	0.64134 (9)	0.8153 (2)	0.44570 (14)	0.0363 (4)
C12	0.76067 (9)	0.8721 (2)	0.37782 (14)	0.0348 (4)
C13	0.78904 (10)	0.9338 (2)	0.28169 (15)	0.0416 (4)
H13	0.755572	0.967507	0.211870	0.050*
C14	0.86646 (11)	0.9450 (3)	0.28962 (17)	0.0480 (5)
H14	0.885249	0.984768	0.224560	0.058*
C15	0.91682 (11)	0.8977 (3)	0.39347 (17)	0.0494 (5)
H15	0.969268	0.906781	0.398840	0.059*
C16	0.88857 (10)	0.8372 (3)	0.48854 (17)	0.0473 (5)
H16	0.922266	0.805371	0.558565	0.057*
C17	0.81049 (10)	0.8229 (2)	0.48169 (15)	0.0392 (4)
H17	0.791856	0.780592	0.546287	0.047*
N1	0.41386 (9)	0.7429 (3)	0.39846 (14)	0.0606 (5)
N2	0.59647 (9)	0.7436 (2)	0.59928 (13)	0.0490 (4)
N3	0.66384 (8)	0.7833 (2)	0.55885 (13)	0.0487 (4)
N4	0.68044 (8)	0.8585 (2)	0.36244 (12)	0.0405 (4)
H4	0.654043	0.880249	0.292363	0.049*
O1	0.34130 (7)	0.7092 (2)	0.42024 (11)	0.0600 (4)
O2	0.56429 (6)	0.80070 (17)	0.40728 (10)	0.0409 (3)
Cl1	0.05523 (3)	0.56575 (9)	0.69766 (5)	0.0682 (2)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0365 (9)	0.0360 (9)	0.0339 (8)	−0.0021 (7)	0.0078 (7)	−0.0019 (7)
C2	0.0399 (9)	0.0403 (9)	0.0324 (8)	−0.0005 (7)	0.0046 (7)	−0.0025 (7)
C3	0.0490 (11)	0.0427 (10)	0.0317 (8)	−0.0024 (8)	0.0119 (7)	−0.0009 (8)
C4	0.0389 (10)	0.0444 (10)	0.0465 (10)	−0.0004 (8)	0.0178 (8)	−0.0048 (8)
C5	0.0372 (10)	0.0494 (11)	0.0455 (10)	0.0037 (8)	0.0069 (8)	0.0012 (9)
C6	0.0422 (10)	0.0467 (10)	0.0344 (9)	0.0018 (8)	0.0088 (7)	0.0054 (8)
C7	0.0375 (9)	0.0409 (9)	0.0315 (8)	−0.0010 (7)	0.0061 (7)	0.0015 (7)
C8	0.0373 (9)	0.0538 (11)	0.0316 (8)	−0.0017 (8)	0.0062 (7)	−0.0005 (8)
C9	0.0346 (9)	0.0462 (10)	0.0373 (9)	−0.0011 (8)	0.0068 (7)	0.0013 (8)
C10	0.0348 (9)	0.0480 (10)	0.0338 (8)	−0.0012 (8)	0.0087 (7)	0.0001 (8)
C11	0.0288 (8)	0.0430 (9)	0.0362 (9)	0.0006 (7)	0.0046 (7)	−0.0043 (7)
C12	0.0326 (9)	0.0356 (9)	0.0357 (8)	−0.0002 (7)	0.0063 (7)	−0.0050 (7)
C13	0.0367 (9)	0.0515 (11)	0.0355 (9)	−0.0013 (8)	0.0049 (7)	0.0017 (8)
C14	0.0435 (11)	0.0560 (12)	0.0471 (11)	−0.0051 (9)	0.0154 (8)	0.0002 (9)
C15	0.0327 (9)	0.0614 (12)	0.0544 (11)	−0.0017 (9)	0.0101 (8)	−0.0043 (10)
C16	0.0372 (10)	0.0564 (12)	0.0443 (10)	0.0034 (9)	−0.0003 (8)	−0.0018 (9)
C17	0.0380 (9)	0.0444 (10)	0.0344 (9)	0.0005 (8)	0.0059 (7)	−0.0010 (8)
N1	0.0337 (9)	0.1058 (15)	0.0422 (9)	−0.0077 (9)	0.0076 (7)	0.0204 (9)
N2	0.0364 (8)	0.0768 (12)	0.0341 (8)	−0.0046 (8)	0.0084 (6)	0.0010 (8)
N3	0.0349 (8)	0.0784 (12)	0.0323 (8)	−0.0047 (8)	0.0059 (6)	−0.0001 (8)
N4	0.0304 (7)	0.0583 (9)	0.0316 (7)	0.0004 (7)	0.0035 (6)	0.0050 (7)
O1	0.0337 (7)	0.1065 (12)	0.0386 (7)	−0.0082 (7)	0.0048 (5)	0.0204 (7)
O2	0.0308 (6)	0.0564 (8)	0.0345 (6)	−0.0015 (5)	0.0050 (5)	0.0041 (6)
Cl1	0.0494 (3)	0.0880 (4)	0.0766 (4)	0.0034 (3)	0.0345 (3)	0.0077 (3)

Geometric parameters (Å, º)

C1—C2	1.391 (2)	C10—O2	1.3665 (19)
C1—C6	1.393 (2)	C11—N3	1.301 (2)
C1—C7	1.459 (2)	C11—N4	1.338 (2)
C2—C3	1.379 (2)	C11—O2	1.3611 (19)
C2—H2	0.9300	C12—C17	1.382 (2)
C3—C4	1.379 (3)	C12—C13	1.387 (2)
C3—H3	0.9300	C12—N4	1.413 (2)
C4—C5	1.379 (3)	C13—C14	1.372 (3)
C4—Cl1	1.7351 (18)	C13—H13	0.9300
C5—C6	1.375 (2)	C14—C15	1.383 (3)
C5—H5	0.9300	C14—H14	0.9300
C6—H6	0.9300	C15—C16	1.372 (3)
C7—C8	1.345 (2)	C15—H15	0.9300
C7—O1	1.351 (2)	C16—C17	1.387 (2)
C8—C9	1.404 (2)	C16—H16	0.9300
C8—H8	0.9300	C17—H17	0.9300
C9—N1	1.306 (2)	N1—O1	1.3952 (19)
C9—C10	1.453 (2)	N2—N3	1.411 (2)

C10—N2	1.275 (2)	N4—H4	0.8600
C2—C1—C6	119.05 (16)	N3—C11—N4	131.36 (16)
C2—C1—C7	119.83 (15)	N3—C11—O2	113.02 (15)
C6—C1—C7	121.11 (15)	N4—C11—O2	115.62 (14)
C3—C2—C1	120.36 (16)	C17—C12—C13	119.92 (16)
C3—C2—H2	119.8	C17—C12—N4	122.98 (15)
C1—C2—H2	119.8	C13—C12—N4	117.08 (15)
C4—C3—C2	119.23 (16)	C14—C13—C12	119.99 (17)
C4—C3—H3	120.4	C14—C13—H13	120.0
C2—C3—H3	120.4	C12—C13—H13	120.0
C5—C4—C3	121.56 (16)	C13—C14—C15	120.58 (18)
C5—C4—Cl1	119.36 (14)	C13—C14—H14	119.7
C3—C4—Cl1	119.07 (14)	C15—C14—H14	119.7
C6—C5—C4	118.86 (17)	C16—C15—C14	119.29 (17)
C6—C5—H5	120.6	C16—C15—H15	120.4
C4—C5—H5	120.6	C14—C15—H15	120.4
C5—C6—C1	120.92 (16)	C15—C16—C17	120.94 (17)
C5—C6—H6	119.5	C15—C16—H16	119.5
C1—C6—H6	119.5	C17—C16—H16	119.5
C8—C7—O1	109.14 (15)	C12—C17—C16	119.28 (16)
C8—C7—C1	135.40 (16)	C12—C17—H17	120.4
O1—C7—C1	115.45 (14)	C16—C17—H17	120.4
C7—C8—C9	104.44 (15)	C9—N1—O1	104.75 (14)
C7—C8—H8	127.8	C10—N2—N3	106.85 (14)
C9—C8—H8	127.8	C11—N3—N2	105.10 (13)
N1—C9—C8	112.40 (16)	C11—N4—C12	126.68 (14)
N1—C9—C10	118.31 (15)	C11—N4—H4	116.7
C8—C9—C10	129.29 (16)	C12—N4—H4	116.7
N2—C10—O2	113.04 (15)	C7—O1—N1	109.26 (13)
N2—C10—C9	129.36 (16)	C11—O2—C10	101.99 (13)
O2—C10—C9	117.60 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C17—H17···N3	0.93	2.33	2.955 (2)	125
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