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1-[5-(2-Chloropyridin-3-yl)-2-methyl-2-phenyl-1,3,4-oxadiazol-3(2*H*)-yl]-ethanone

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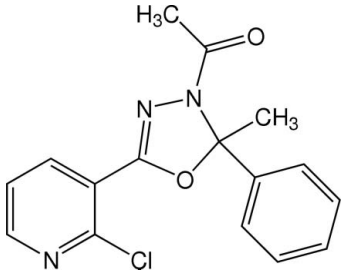
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.105; data-to-parameter ratio = 18.6.

In the title compound, $C_{16}H_{14}ClN_3O_2$, all bond lengths and angles are normal. The mean plane of the oxadiazoline ring makes dihedral angles of 1.94 (3) and 82.05 (3)° with the substituted pyridine and benzene rings, respectively. No classical hydrogen bonds are evident in the crystal packing.

Related literature

For the crystal structures of isomers of the title compound, see: Song, Zhang, Dong *et al.* (2006); Song, Zhang & Tiekink (2006). For the pharmacological properties of 2,5-disubstituted 1,3,4-oxadiazolines, see: Hassan *et al.* (1983); Khalil *et al.* (1993). For the normal values of bond lengths in organic compounds, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{16}H_{14}ClN_3O_2$
M_r = 315.75
Monoclinic, $P2_1/c$
a = 7.7788 (7) Å
b = 24.625 (2) Å
c = 7.9948 (8) Å
β = 93.030 (1)°
V = 1529.3 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 273 (2) K
0.12 × 0.10 × 0.08 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.970, T_{max} = 0.980
13995 measured reflections
3751 independent reflections
2953 reflections with $I > 2\sigma(I)$
R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.105
S = 1.00
3751 reflections
202 parameters
H-atom parameters not refined
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2288).

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supplementary materials

Acta Cryst. (2007). E**63**, o3988 [**doi:10.1107/S1600536807042729**]

1-[5-(2-Chloropyridin-3-yl)-2-methyl-2-phenyl-1,3,4-oxadiazol-3(2*H*)-yl]ethanone

L.-F. Pan, Y.-Q. Lu, Q. Qin, C.-Z. Qi and Q.-B. Song

Comment

In continuation of our study of 1,3,4-oxadiazolines (Song, Zhang, Dong *et al.*, 2006; Song, Zhang & Tiekink, 2006), which possess a wide range of pharmaceutical activities (Hassan *et al.*, 1983; Khalil *et al.*, 1993), we present here the crystal structure of the title compound, (I).

In (I) (Fig. 1), all bond lengths and angles are normal (Allen *et al.*, 1987). The molecule is twisted around the C7—C11 bond. Within the five-membered oxadiazoline ring, there is a formal C6=N2 double bond (1.2755 (17) Å), the bond C6—O2 [1.3648 (16) Å] is shortened as compared with C7—O2 [1.4615 (16) Å], showing some delocalization of electron density over the O2—C6—N2 chromophore.

Experimental

A solution of 2-chloropyridine-3-carboxylic acid phenylethylidene hydrazide (0.5 g, 1.91 mmol) in 10 ml of acetic anhydride was refluxed until the reaction was finished. The acetic anhydride was distilled in vacuum. The residue was recrystallized from ethanol (10 ml). Colourless crystals (0.38 g) of the title compound were obtained by slow evaporation of the solvent after 2 days at room temperature.

Refinement

H atoms were placed in calculated positions and allowed to ride on their parent atoms, with C—H = 0.93–0.96 Å and U_{iso}(H) = 1.2Ueq(C) or 1.5Ueq(C_{methyl}).

Figures

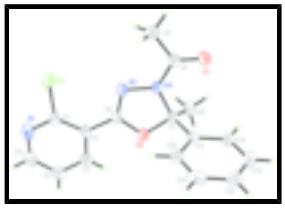

Fig. 1. The molecular structure of (I) showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 40% probability level.

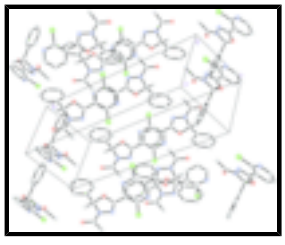

Fig. 2. The molecular packing viewed along the *a* axis. H atoms omitted for clarity.

1-[5-(2-Chloropyridin-3-yl)-2-methyl-2-phenyl-1,3,4-oxadiazol-3(2*H*)-yl]ethanone

Crystal data

$C_{16}H_{14}ClN_3O_2$	F_{000} = 656
M_r = 315.75	D_x = 1.371 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4932 reflections
a = 7.7788 (7) Å	θ = 2.6–27.6º
b = 24.625 (2) Å	μ = 0.26 mm^{-1}
c = 7.9948 (8) Å	T = 273 (2) K
β = 93.030 (1)º	Block, colourless
V = 1529.3 (2) Å^3	0.12 × 0.10 × 0.08 mm
Z = 4	

Data collection

Bruker APEXII CCD area-detector diffractometer	3751 independent reflections
Radiation source: fine-focus sealed tube	2953 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.019
T = 273(2) K	θ_{max} = 28.2º
φ and ω scans	θ_{min} = 2.6º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −10→10
T_{min} = 0.970, T_{max} = 0.980	k = −32→32
13995 measured reflections	l = −10→10

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters not refined
$R[F^2 > 2\sigma(F^2)]$ = 0.036	$w = 1/[\sigma^2(F_o^2) + (0.0526P)^2 + 0.2948P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.105	$(\Delta/\sigma)_{max} < 0.001$
S = 1.00	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
3751 reflections	$\Delta\rho_{min}$ = −0.25 e Å^{-3}
202 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0039 (12)
Secondary atom site location: difference Fourier map	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.28346 (18)	0.52714 (5)	−0.02816 (18)	0.0444 (3)
C2	0.27771 (17)	0.50607 (5)	0.13417 (17)	0.0412 (3)
C3	0.3456 (2)	0.53918 (6)	0.2626 (2)	0.0546 (4)
H3	0.3440	0.5276	0.3732	0.066*
C4	0.4152 (2)	0.58909 (7)	0.2260 (2)	0.0653 (4)
H4	0.4613	0.6114	0.3107	0.078*
C5	0.4145 (2)	0.60475 (7)	0.0619 (2)	0.0668 (5)
H5	0.4621	0.6383	0.0375	0.080*
C6	0.20863 (17)	0.45270 (5)	0.17689 (16)	0.0415 (3)
C7	0.14495 (19)	0.38600 (5)	0.36193 (16)	0.0448 (4)
C8	0.01531 (18)	0.32860 (5)	0.12329 (17)	0.0443 (3)
C9	−0.0074 (2)	0.32372 (7)	−0.06330 (19)	0.0611 (4)
H9A	0.1034	0.3215	−0.1103	0.092*
H9B	−0.0680	0.3550	−0.1076	0.092*
H9C	−0.0723	0.2915	−0.0915	0.092*
C10	−0.0073 (2)	0.39235 (7)	0.47005 (19)	0.0563 (4)
H10A	0.0315	0.4056	0.5785	0.084*
H10B	−0.0627	0.3578	0.4818	0.084*
H10C	−0.0876	0.4177	0.4185	0.084*
C11	0.28433 (17)	0.34702 (5)	0.42702 (16)	0.0419 (3)
C12	0.2521 (2)	0.30728 (6)	0.54395 (18)	0.0528 (4)
H12	0.1425	0.3042	0.5844	0.063*
C13	0.3805 (2)	0.27214 (7)	0.6013 (2)	0.0667 (5)
H13	0.3576	0.2463	0.6821	0.080*
C14	0.5419 (2)	0.27503 (7)	0.5398 (2)	0.0674 (5)
H14	0.6281	0.2513	0.5784	0.081*
C15	0.5750 (2)	0.31361 (8)	0.4199 (2)	0.0640 (5)
H15	0.6832	0.3153	0.3759	0.077*
C16	0.4476 (2)	0.34980 (7)	0.36515 (18)	0.0530 (4)
H16	0.4717	0.3761	0.2864	0.064*
Cl1	0.20295 (6)	0.491105 (16)	−0.20175 (5)	0.06009 (14)
N1	0.34943 (18)	0.57466 (5)	−0.06525 (18)	0.0590 (3)
N2	0.13810 (15)	0.41731 (4)	0.07904 (14)	0.0432 (3)

N3	0.09365 (16)	0.37466 (5)	0.18324 (14)	0.0463 (3)
O1	−0.02848 (15)	0.29362 (4)	0.22043 (13)	0.0579 (3)
O2	0.22132 (15)	0.43973 (4)	0.34306 (12)	0.0540 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0459 (7)	0.0390 (7)	0.0489 (7)	0.0045 (5)	0.0094 (6)	0.0056 (6)
C2	0.0427 (7)	0.0353 (6)	0.0460 (7)	0.0014 (5)	0.0052 (5)	0.0023 (5)
C3	0.0667 (10)	0.0438 (8)	0.0533 (8)	−0.0054 (7)	0.0025 (7)	−0.0007 (6)
C4	0.0774 (11)	0.0456 (8)	0.0727 (11)	−0.0125 (8)	0.0023 (9)	−0.0081 (8)
C5	0.0770 (11)	0.0414 (8)	0.0832 (12)	−0.0122 (8)	0.0152 (9)	0.0062 (8)
C6	0.0488 (7)	0.0384 (7)	0.0373 (6)	0.0001 (5)	0.0032 (5)	0.0039 (5)
C7	0.0613 (11)	0.0386 (9)	0.0344 (8)	−0.0103 (8)	0.0008 (7)	0.0020 (7)
C8	0.0501 (7)	0.0391 (7)	0.0435 (7)	−0.0024 (6)	0.0012 (6)	0.0001 (5)
C9	0.0839 (11)	0.0541 (9)	0.0447 (8)	−0.0056 (8)	−0.0037 (7)	−0.0077 (7)
C10	0.0645 (10)	0.0585 (9)	0.0464 (8)	0.0053 (7)	0.0071 (7)	0.0031 (7)
C11	0.0498 (7)	0.0417 (7)	0.0341 (6)	−0.0089 (6)	0.0006 (5)	−0.0002 (5)
C12	0.0547 (8)	0.0537 (9)	0.0503 (8)	−0.0033 (7)	0.0047 (6)	0.0130 (7)
C13	0.0710 (11)	0.0582 (10)	0.0700 (11)	0.0022 (8)	−0.0035 (9)	0.0183 (8)
C14	0.0628 (10)	0.0606 (10)	0.0771 (12)	0.0071 (8)	−0.0119 (9)	−0.0054 (9)
C15	0.0471 (8)	0.0777 (12)	0.0675 (10)	−0.0087 (8)	0.0041 (7)	−0.0211 (9)
C16	0.0563 (9)	0.0599 (9)	0.0431 (7)	−0.0147 (7)	0.0068 (6)	−0.0028 (6)
Cl1	0.0826 (3)	0.0537 (2)	0.0438 (2)	−0.00125 (18)	0.00217 (18)	0.00691 (15)
N1	0.0705 (9)	0.0428 (7)	0.0650 (8)	−0.0036 (6)	0.0162 (7)	0.0106 (6)
N2	0.0534 (7)	0.0389 (6)	0.0373 (6)	−0.0043 (5)	0.0033 (5)	0.0062 (4)
N3	0.0637 (7)	0.0406 (6)	0.0340 (5)	−0.0109 (5)	−0.0017 (5)	0.0051 (4)
O1	0.0746 (7)	0.0450 (6)	0.0541 (6)	−0.0142 (5)	0.0051 (5)	0.0044 (5)
O2	0.0836 (7)	0.0399 (5)	0.0376 (5)	−0.0148 (5)	−0.0035 (5)	0.0035 (4)

Geometric parameters (Å, °)

C1—N1	1.3179 (18)	C8—C9	1.498 (2)
C1—C2	1.4006 (19)	C9—H9A	0.9600
C1—Cl1	1.7360 (15)	C9—H9B	0.9600
C2—C3	1.393 (2)	C9—H9C	0.9600
C2—C6	1.4670 (18)	C10—H10A	0.9600
C3—C4	1.380 (2)	C10—H10B	0.9600
C3—H3	0.9300	C10—H10C	0.9600
C4—C5	1.367 (3)	C11—C12	1.3854 (19)
C4—H4	0.9300	C11—C16	1.389 (2)
C5—N1	1.336 (2)	C12—C13	1.382 (2)
C5—H5	0.9300	C12—H12	0.9300
C6—N2	1.2755 (17)	C13—C14	1.374 (3)
C6—O2	1.3648 (16)	C13—H13	0.9300
C7—O2	1.4615 (16)	C14—C15	1.383 (3)
C7—N3	1.4892 (16)	C14—H14	0.9300
C7—C10	1.511 (2)	C15—C16	1.387 (2)
C7—C11	1.519 (2)	C15—H15	0.9300

C8—O1	1.2203 (17)	C16—H16	0.9300
C8—N3	1.3626 (17)	N2—N3	1.3953 (15)
N1—C1—C2	124.70 (14)	H9B—C9—H9C	109.5
N1—C1—Cl1	113.72 (11)	C7—C10—H10A	109.5
C2—C1—Cl1	121.58 (11)	C7—C10—H10B	109.5
C3—C2—C1	115.89 (13)	H10A—C10—H10B	109.5
C3—C2—C6	118.83 (12)	C7—C10—H10C	109.5
C1—C2—C6	125.28 (12)	H10A—C10—H10C	109.5
C4—C3—C2	120.19 (15)	H10B—C10—H10C	109.5
C4—C3—H3	119.9	C12—C11—C16	118.48 (14)
C2—C3—H3	119.9	C12—C11—C7	121.73 (13)
C5—C4—C3	118.21 (16)	C16—C11—C7	119.76 (12)
C5—C4—H4	120.9	C13—C12—C11	120.83 (15)
C3—C4—H4	120.9	C13—C12—H12	119.6
N1—C5—C4	123.77 (15)	C11—C12—H12	119.6
N1—C5—H5	118.1	C14—C13—C12	120.50 (16)
C4—C5—H5	118.1	C14—C13—H13	119.8
N2—C6—O2	116.35 (11)	C12—C13—H13	119.8
N2—C6—C2	128.33 (12)	C13—C14—C15	119.38 (16)
O2—C6—C2	115.32 (11)	C13—C14—H14	120.3
O2—C7—N3	99.23 (9)	C15—C14—H14	120.3
O2—C7—C10	107.49 (12)	C14—C15—C16	120.27 (16)
N3—C7—C10	112.94 (12)	C14—C15—H15	119.9
O2—C7—C11	108.71 (11)	C16—C15—H15	119.9
N3—C7—C11	111.13 (11)	C15—C16—C11	120.51 (15)
C10—C7—C11	115.87 (12)	C15—C16—H16	119.7
O1—C8—N3	119.90 (13)	C11—C16—H16	119.7
O1—C8—C9	123.70 (13)	C1—N1—C5	117.24 (14)
N3—C8—C9	116.38 (12)	C6—N2—N3	105.08 (10)
C8—C9—H9A	109.5	C8—N3—N2	122.46 (11)
C8—C9—H9B	109.5	C8—N3—C7	125.85 (11)
H9A—C9—H9B	109.5	N2—N3—C7	111.68 (10)
C8—C9—H9C	109.5	C6—O2—C7	107.66 (10)
H9A—C9—H9C	109.5		
N1—C1—C2—C3	0.9 (2)	C12—C11—C16—C15	−0.2 (2)
Cl1—C1—C2—C3	−179.60 (11)	C7—C11—C16—C15	−178.30 (13)
N1—C1—C2—C6	−178.39 (14)	C2—C1—N1—C5	−0.3 (2)
Cl1—C1—C2—C6	1.1 (2)	Cl1—C1—N1—C5	−179.82 (12)
C1—C2—C3—C4	−0.9 (2)	C4—C5—N1—C1	−0.4 (3)
C6—C2—C3—C4	178.49 (15)	O2—C6—N2—N3	0.54 (16)
C2—C3—C4—C5	0.3 (3)	C2—C6—N2—N3	−179.11 (13)
C3—C4—C5—N1	0.4 (3)	O1—C8—N3—N2	−176.44 (13)
C3—C2—C6—N2	178.23 (14)	C9—C8—N3—N2	5.2 (2)
C1—C2—C6—N2	−2.5 (2)	O1—C8—N3—C7	4.6 (2)
C3—C2—C6—O2	−1.43 (19)	C9—C8—N3—C7	−173.76 (14)
C1—C2—C6—O2	177.89 (13)	C6—N2—N3—C8	−179.60 (13)
O2—C7—C11—C12	141.40 (13)	C6—N2—N3—C7	−0.53 (15)
N3—C7—C11—C12	−110.40 (14)	O2—C7—N3—C8	179.35 (13)

C10—C7—C11—C12	20.27 (19)	C10—C7—N3—C8	−67.11 (18)
O2—C7—C11—C16	−40.53 (16)	C11—C7—N3—C8	65.07 (17)
N3—C7—C11—C16	67.68 (15)	O2—C7—N3—N2	0.32 (14)
C10—C7—C11—C16	−161.65 (13)	C10—C7—N3—N2	113.86 (13)
C16—C11—C12—C13	1.8 (2)	C11—C7—N3—N2	−113.96 (12)
C7—C11—C12—C13	179.85 (15)	N2—C6—O2—C7	−0.35 (17)
C11—C12—C13—C14	−1.8 (3)	C2—C6—O2—C7	179.35 (11)
C12—C13—C14—C15	0.2 (3)	N3—C7—O2—C6	−0.01 (14)
C13—C14—C15—C16	1.4 (3)	C10—C7—O2—C6	−117.73 (12)
C14—C15—C16—C11	−1.4 (2)	C11—C7—O2—C6	116.13 (12)

Fig. 1

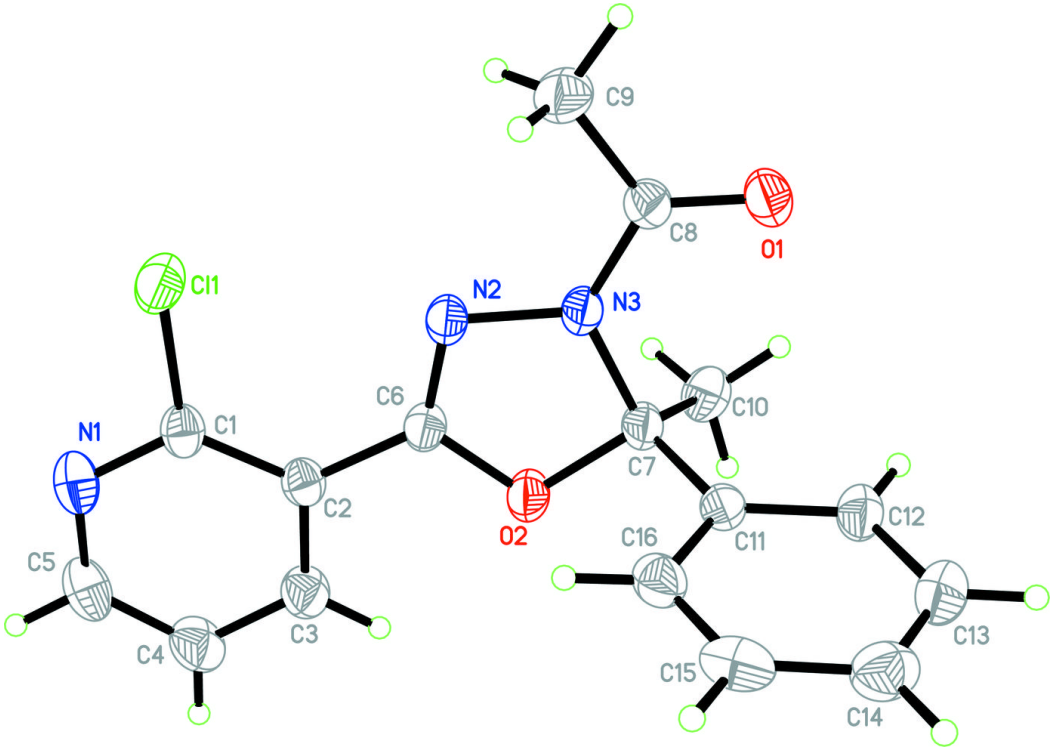
C9
C8
O1
Cl1
N2
N3
C6
C10
N1
C1
C7
C2
O2
C12
C16
C11
C5
C3
C13
C4
C15
C14

Fig. 2