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(3,6-Dichloropyridin-2-yl)(3,5-dimethyl-1*H*-pyrazol-1-yl)methanone

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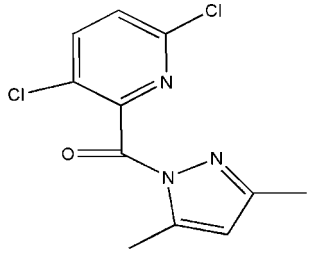
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.127; data-to-parameter ratio = 13.6.

In the crystal structure of the title compound, $C_{11}H_9Cl_2N_3O$, molecules are held together by short intermolecular Cl···Cl contacts [3.319 (1) Å] and C—H···N hydrogen bonds, forming two-dimensional networks parallel to (01$\bar{1}$).

Related literature

For related literature, see: Mann *et al.* (1992); Perevalov *et al.* (2001).



Experimental

Crystal data

$C_{11}H_9Cl_2N_3O$
M_r = 270.11
Triclinic, $P\bar{1}$
a = 7.3440 (10) Å
b = 8.7981 (12) Å
c = 9.6490 (14) Å
α = 75.554 (2)°
β = 89.627 (3)°
γ = 86.819 (2)°
V = 602.79 (15) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 298 (2) K
0.50 × 0.49 × 0.48 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.780, T_{max} = 0.787
3145 measured reflections
2102 independent reflections
1543 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.126
S = 1.04
2101 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C11—H11*C*···N1^{i}	0.96	2.56	3.514 (4)	174

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); 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: BG2182).

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supporting information

Acta Cryst. (2008). E**64**, o1224 [doi:10.1107/S1600536808015006]

(3,6-Dichloropyridin-2-yl)(3,5-dimethyl-1*H*-pyrazol-1-yl)methanone

Yue Zhuang, Shan-Shan Zhang, Xian-Hong Yin, Kai Zhao and Cui-Wu Lin

S1. Comment

The chemical and pharmacological properties of pyrazoles have been investigated extensively, owing to their chelating ability with metal ions and their potentially beneficial chemical and biological activities (Mann *et al.*, 1992, Perevalov *et al.*, 2001). As part of our studies on the synthesis and characterization of these compounds, we report here the synthesis and crystal structure of (3,6-dichloropyridin-2-yl)(3,5-dimethyl-1*H*-pyrazol-1-yl)methanone.

The crystal structure of the monomeric title compound is built up by $C_{11}H_9Cl_2N_3O$ molecules (Fig.1), linked by intermolecular C—H···N hydrogen bonds along [100] (Table 1) and by Cl···Cl short contacts along the [011] direction (Cl1···Cl1^{ii}: 3.319 (1)Å, (ii): 2-x, 1-y, 1-z) , forming a two-dimensional network parallel to (01$\bar{1}$).

The short C=O bond length (1.199 (3)Å) indicates that the molecule is in a keto form (Fig.1). The two rings are nearly perpendicular to each other (dihedral angle: 82.319 (84) °), and this fact helps in minimizing steric effects between them. Finally, there is an intermolecular π—π contact between pyridine groups with and intercentroid distance of 3.40 (1) Å, which contributes to the stability of the crystal packing.

S2. Experimental

A solution of 3,6-dichloropicolinoyl chloride (10 mmol) in 50 ml toluene was added to a solution of 3,5-dimethyl-1*H*-pyrazole (10 mmol) in 10 ml toluene. The reaction mixture was refluxed for 1 h with stirring then the resulting white precipitate was obtained by filtration, washed several times with ethanol and dried *in vacuo* (yield 90%). Elemental analysis calculate: Elemental analysis: found: C, 48.89; H, 3.33; N, 15.56; calc. for $C_{11}H_9Cl_2N_3O$: C, 48.99; H, 3.23; N, 15.46.

S3. Refinement

Data collection: 2102 independent reflections but 2101 in Refinement. H atoms on C atoms were positoned geometrically and refined using a riding model with C—H = 0.96Å and U_{iso}(H) = 1.2U_{eq}(C).

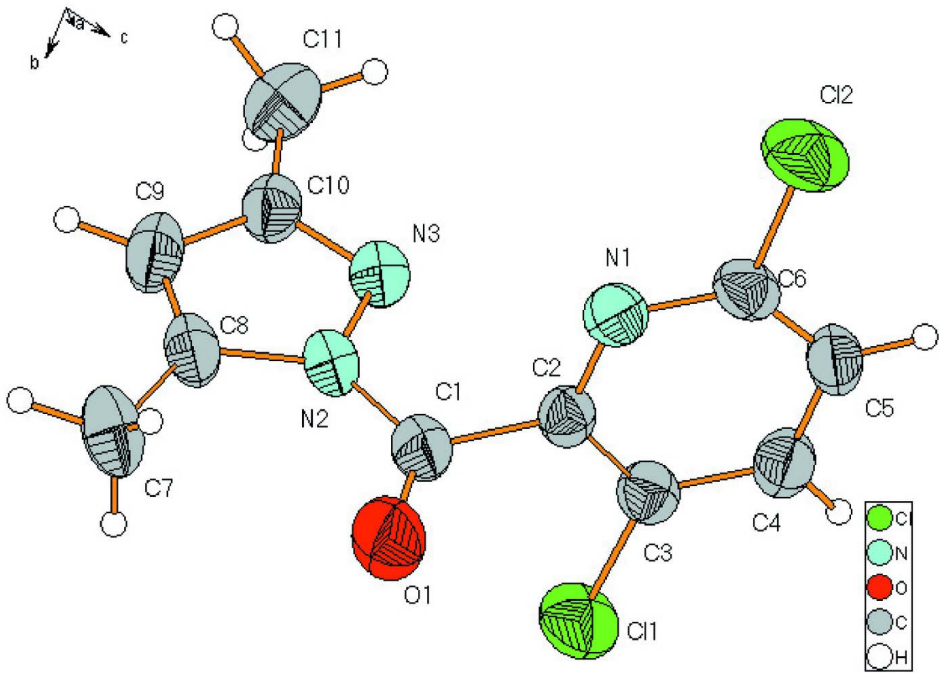

Figure 1

The structure of the title compound (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

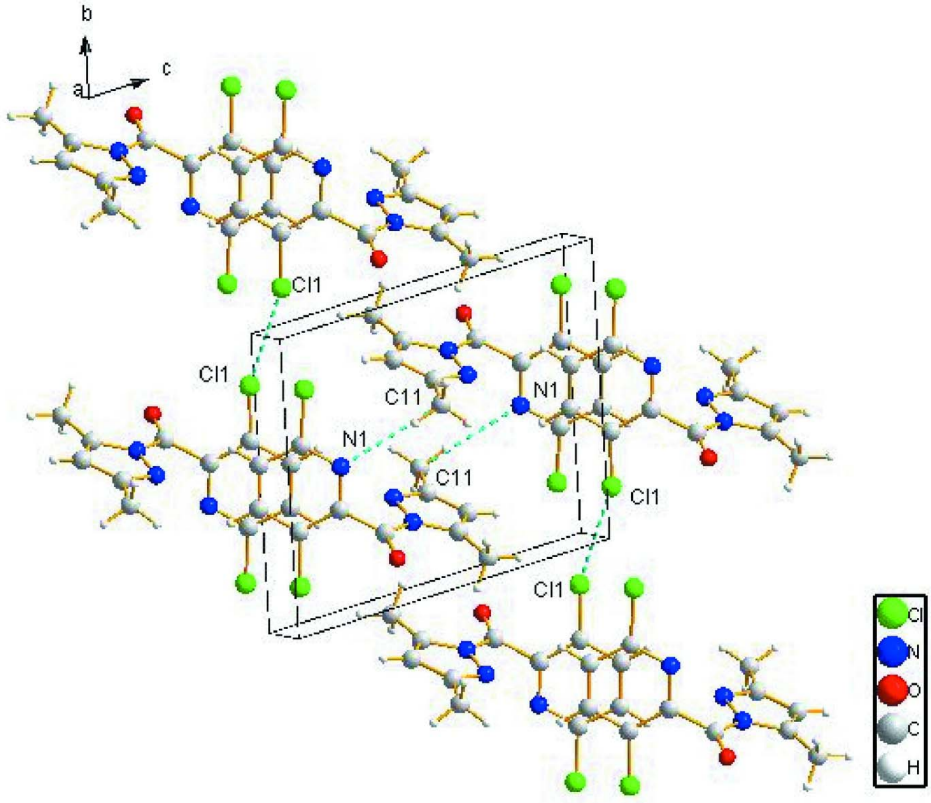

Figure 2

Crystal packing of (I) showing the short contacts interactions as dashed lines.

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Crystal data

$C_{11}H_9Cl_2N_3O$
$M_r = 270.11$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.344$ (1) Å
$b = 8.7981$ (12) Å
$c = 9.6490$ (14) Å
$\alpha = 75.554$ (2)°
$\beta = 89.627$ (3)°
$\gamma = 86.819$ (2)°
$V = 602.79$ (15) Å^3
$Z = 2$
$F(000) = 276$
$D_x = 1.488$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1363 reflections
$\theta = 2.2$–$27.4°$
$\mu = 0.52$ mm^{-1}
$T = 298$ K
Block, colourless
0.50 × 0.49 × 0.48 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.780$, $T_{max} = 0.787$
3145 measured reflections
2102 independent reflections
1543 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$
$h = -8 \rightarrow 8$
$k = -9 \rightarrow 10$
$l = -9 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.126$
$S = 1.04$
2101 reflections
154 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
$w = 1/[\sigma^2(F_o^2) + (0.0468P)^2 + 0.2168P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
Cl1	0.60627 (11)	0.85128 (9)	0.94981 (8)	0.0634 (3)
Cl2	0.90422 (12)	0.19393 (9)	0.92556 (10)	0.0712 (3)
N1	0.8160 (3)	0.4899 (3)	0.8173 (2)	0.0434 (5)
N2	0.6305 (3)	0.7559 (2)	0.5846 (2)	0.0415 (5)
N3	0.4844 (3)	0.6627 (2)	0.6266 (2)	0.0421 (5)
O1	0.8838 (3)	0.8463 (3)	0.6636 (2)	0.0712 (7)
C1	0.7630 (4)	0.7572 (3)	0.6853 (3)	0.0440 (6)
C2	0.7517 (3)	0.6330 (3)	0.8231 (2)	0.0368 (6)
C3	0.6925 (3)	0.6653 (3)	0.9493 (3)	0.0407 (6)
C4	0.6996 (4)	0.5468 (3)	1.0739 (3)	0.0469 (7)
H4	0.6596	0.5662	1.1598	0.056*
C5	0.7666 (4)	0.4004 (3)	1.0686 (3)	0.0488 (7)
H5	0.7746	0.3181	1.1508	0.059*
C6	0.8220 (4)	0.3782 (3)	0.9384 (3)	0.0445 (7)
C7	0.7566 (5)	0.9440 (4)	0.3653 (3)	0.0670 (9)
H7A	0.7593	1.0332	0.4056	0.101*
H7B	0.7259	0.9789	0.2654	0.101*
H7C	0.8743	0.8890	0.3764	0.101*
C8	0.6167 (4)	0.8362 (3)	0.4409 (3)	0.0483 (7)
C9	0.4615 (4)	0.7924 (3)	0.3939 (3)	0.0548 (8)
H9	0.4145	0.8254	0.3013	0.066*
C10	0.3819 (4)	0.6865 (3)	0.5110 (3)	0.0444 (6)
C11	0.2080 (4)	0.6066 (4)	0.5140 (3)	0.0622 (8)
H11A	0.1074	0.6782	0.5223	0.093*
H11B	0.2091	0.5166	0.5944	0.093*

H11C	0.1953	0.5733	0.4272	0.093*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0743 (6)	0.0459 (4)	0.0697 (5)	0.0120 (4)	0.0039 (4)	−0.0172 (4)
Cl2	0.0775 (6)	0.0408 (4)	0.0948 (7)	0.0118 (4)	−0.0144 (5)	−0.0188 (4)
N1	0.0444 (13)	0.0417 (13)	0.0441 (13)	0.0003 (10)	−0.0022 (10)	−0.0110 (10)
N2	0.0448 (13)	0.0369 (12)	0.0379 (12)	−0.0010 (10)	0.0017 (10)	−0.0006 (9)
N3	0.0440 (13)	0.0414 (12)	0.0388 (12)	−0.0010 (10)	0.0006 (10)	−0.0062 (9)
O1	0.0706 (15)	0.0756 (16)	0.0611 (14)	−0.0326 (13)	−0.0013 (11)	0.0012 (11)
C1	0.0466 (16)	0.0413 (15)	0.0422 (15)	−0.0033 (13)	0.0020 (12)	−0.0066 (12)
C2	0.0338 (13)	0.0387 (14)	0.0360 (13)	−0.0027 (11)	−0.0032 (10)	−0.0058 (11)
C3	0.0382 (14)	0.0390 (14)	0.0438 (15)	0.0000 (12)	−0.0031 (11)	−0.0084 (12)
C4	0.0507 (17)	0.0510 (17)	0.0380 (15)	−0.0034 (13)	−0.0011 (12)	−0.0088 (12)
C5	0.0527 (18)	0.0462 (17)	0.0406 (15)	−0.0082 (13)	−0.0079 (13)	0.0036 (12)
C6	0.0415 (15)	0.0343 (14)	0.0559 (17)	0.0001 (11)	−0.0102 (12)	−0.0083 (12)
C7	0.079 (2)	0.062 (2)	0.0488 (18)	−0.0075 (18)	0.0099 (16)	0.0091 (15)
C8	0.0604 (19)	0.0408 (15)	0.0373 (15)	0.0049 (13)	0.0037 (13)	0.0009 (12)
C9	0.064 (2)	0.0595 (19)	0.0359 (15)	0.0079 (16)	−0.0081 (14)	−0.0047 (13)
C10	0.0488 (16)	0.0445 (15)	0.0382 (14)	0.0057 (12)	−0.0024 (12)	−0.0090 (12)
C11	0.0570 (19)	0.076 (2)	0.0561 (19)	−0.0005 (17)	−0.0078 (15)	−0.0219 (16)

Geometric parameters (Å, º)

Cl1—C3	1.722 (3)	C5—C6	1.375 (4)
Cl2—C6	1.732 (3)	C5—H5	0.9300
N1—C6	1.325 (3)	C7—C8	1.496 (4)
N1—C2	1.334 (3)	C7—H7A	0.9600
N2—C1	1.382 (3)	C7—H7B	0.9600
N2—N3	1.383 (3)	C7—H7C	0.9600
N2—C8	1.392 (3)	C8—C9	1.340 (4)
N3—C10	1.316 (3)	C9—C10	1.419 (4)
O1—C1	1.199 (3)	C9—H9	0.9300
C1—C2	1.502 (3)	C10—C11	1.488 (4)
C2—C3	1.380 (3)	C11—H11A	0.9600
C3—C4	1.380 (4)	C11—H11B	0.9600
C4—C5	1.366 (4)	C11—H11C	0.9600
C4—H4	0.9300		
C6—N1—C2	117.4 (2)	C5—C6—Cl2	119.8 (2)
C1—N2—N3	118.3 (2)	C8—C7—H7A	109.5
C1—N2—C8	130.2 (2)	C8—C7—H7B	109.5
N3—N2—C8	111.5 (2)	H7A—C7—H7B	109.5
C10—N3—N2	104.7 (2)	C8—C7—H7C	109.5
O1—C1—N2	123.1 (2)	H7A—C7—H7C	109.5
O1—C1—C2	121.5 (2)	H7B—C7—H7C	109.5
N2—C1—C2	115.3 (2)	C9—C8—N2	105.3 (2)

N1—C2—C3	122.1 (2)	C9—C8—C7	131.3 (3)
N1—C2—C1	114.8 (2)	N2—C8—C7	123.4 (3)
C3—C2—C1	123.0 (2)	C8—C9—C10	107.6 (2)
C4—C3—C2	119.4 (3)	C8—C9—H9	126.2
C4—C3—Cl1	120.5 (2)	C10—C9—H9	126.2
C2—C3—Cl1	120.03 (19)	N3—C10—C9	110.9 (2)
C5—C4—C3	118.7 (3)	N3—C10—C11	120.8 (2)
C5—C4—H4	120.6	C9—C10—C11	128.3 (2)
C3—C4—H4	120.6	C10—C11—H11A	109.5
C4—C5—C6	118.1 (2)	C10—C11—H11B	109.5
C4—C5—H5	121.0	H11A—C11—H11B	109.5
C6—C5—H5	121.0	C10—C11—H11C	109.5
N1—C6—C5	124.3 (3)	H11A—C11—H11C	109.5
N1—C6—Cl2	115.9 (2)	H11B—C11—H11C	109.5
C1—N2—N3—C10	179.3 (2)	Cl1—C3—C4—C5	179.1 (2)
C8—N2—N3—C10	−0.6 (3)	C3—C4—C5—C6	−0.7 (4)
N3—N2—C1—O1	−171.8 (3)	C2—N1—C6—C5	0.0 (4)
C8—N2—C1—O1	8.1 (5)	C2—N1—C6—Cl2	179.37 (18)
N3—N2—C1—C2	11.2 (3)	C4—C5—C6—N1	0.5 (4)
C8—N2—C1—C2	−169.0 (2)	C4—C5—C6—Cl2	−178.9 (2)
C6—N1—C2—C3	−0.2 (4)	C1—N2—C8—C9	−179.9 (3)
C6—N1—C2—C1	175.1 (2)	N3—N2—C8—C9	0.0 (3)
O1—C1—C2—N1	−98.9 (3)	C1—N2—C8—C7	1.1 (5)
N2—C1—C2—N1	78.2 (3)	N3—N2—C8—C7	−179.1 (3)
O1—C1—C2—C3	76.4 (4)	N2—C8—C9—C10	0.5 (3)
N2—C1—C2—C3	−106.5 (3)	C7—C8—C9—C10	179.5 (3)
N1—C2—C3—C4	0.0 (4)	N2—N3—C10—C9	0.9 (3)
C1—C2—C3—C4	−174.9 (2)	N2—N3—C10—C11	−179.3 (2)
N1—C2—C3—Cl1	−178.62 (19)	C8—C9—C10—N3	−0.9 (3)
C1—C2—C3—Cl1	6.4 (3)	C8—C9—C10—C11	179.3 (3)
C2—C3—C4—C5	0.4 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C11—H11*C*···N1^{i}	0.96	2.56	3.514 (4)	174

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