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

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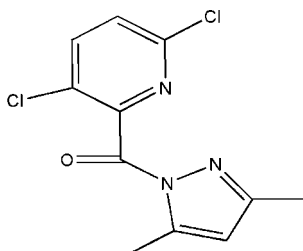
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.045; wR factor = 0.127; data-to-parameter ratio = 13.6.

In the crystal structure of the title compound, $\text{C}_{11}\text{H}_9\text{Cl}_2\text{N}_3\text{O}$, molecules are held together by short intermolecular $\text{Cl}\cdots\text{Cl}$ contacts [3.319 (1) Å] and $\text{C}-\text{H}\cdots\text{N}$ 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

$\text{C}_{11}\text{H}_9\text{Cl}_2\text{N}_3\text{O}$

$M_r = 270.11$

Triclinic, $P\bar{1}$

$a = 7.3440$ (10) 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) Å³

$Z = 2$

Mo $K\alpha$ radiation

$\mu = 0.52$ 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)

$T_{\text{min}} = 0.780$, $T_{\text{max}} = 0.787$

3145 measured reflections

2102 independent reflections

1543 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

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

$\Delta\rho_{\text{max}} = 0.22$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| $\text{C11}-\text{H11C}\cdots\text{N1}^i$ | 0.96 | 2.56 | 3.514 (4) | 174 |

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINTE* (Siemens, 1996); data reduction: *SAINTE*; 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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- Mann, F., Chiment, F., Balasco, A., Cenicola, M. L., Amico, M. D., Parrilo, C., Rossi, F. & Marmo, E. (1992). *Eur. J. Med. Chem.* **27**, 633–639.
Perevalov, S. G., Burgart, Y. V., Saloutin, V. I. & Chupakhin, O. N. (2001). *Russ. Chem. Rev.* **70**, 921–925.
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supplementary materials

Acta Cryst. (2008). E64, o1224 [doi:10.1107/S1600536808015006]

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

Y. Zhuang, S.-S. Zhang, X.-H. Yin, K. Zhao and C.-W. Lin

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-1H-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 \cdots N hydrogen bonds along [100] (Table 1) and by Cl \cdots Cl short contacts along the [011] direction (Cl1 \cdots Cl1ⁱⁱ: 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 an intercentroid distance of 3.40 (1) Å, which contributes to the stability of the crystal packing.

Experimental

A solution of 3,6-dichloropicolinoyl chloride (10 mmol) in 50 ml toluene was added to a solution of 3,5-dimethyl-1H-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.

Refinement

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

Figures

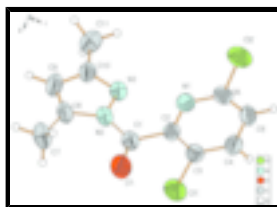


Fig. 1. The structure of the title compound (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

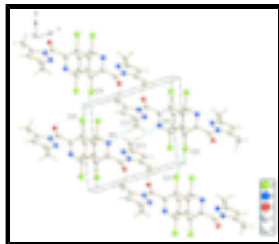


Fig. 2. Crystal packing of (I) showing the short contacts interactions as dashed lines.

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

Crystal data

$C_{11}H_9Cl_2N_3O$

$M_r = 270.11$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.3440$ (10) 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) Å³

$Z = 2$

$F_{000} = 276$

$D_x = 1.488$ 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

$T = 298$ (2) K

Block, colourless

$0.50 \times 0.49 \times 0.48$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298$ (2) K

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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.038$

$\theta_{\max} = 25.0$ °

$\theta_{\min} = 2.2$ °

$h = -8 \rightarrow 8$

$k = -9 \rightarrow 10$

$l = -9 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.044$

$wR(F^2) = 0.126$

$S = 1.04$

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 $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

2101 reflections $\Delta\rho_{\max} = 0.22 \text{ e } \text{\AA}^{-3}$
 154 parameters $\Delta\rho_{\min} = -0.27 \text{ e } \text{\AA}^{-3}$
 Primary atom site location: structure-invariant direct methods Extinction correction: none

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|-------------|--------------|----------------------------------|
| 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* |

supplementary materials

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C11 | 0.0743 (6) | 0.0459 (4) | 0.0697 (5) | 0.0120 (4) | 0.0039 (4) | -0.0172 (4) |
| C12 | 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 (\AA , $^\circ$)

| | | | |
|-----------|-----------|------------|-----------|
| C11—C3 | 1.722 (3) | C5—C6 | 1.375 (4) |
| C12—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—C12 | 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—C11 | 120.5 (2) | C10—C9—H9 | 126.2 |
| C2—C3—C11 | 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—C12 | 115.9 (2) | H11B—C11—H11C | 109.5 |
| C1—N2—N3—C10 | 179.3 (2) | C11—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—C12 | 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—C12 | -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—C11 | -178.62 (19) | C8—C9—C10—N3 | -0.9 (3) |
| C1—C2—C3—C11 | 6.4 (3) | C8—C9—C10—C11 | 179.3 (3) |
| C2—C3—C4—C5 | 0.4 (4) | | |

Hydrogen-bond geometry (\AA , $^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| C11—H11C \cdots N1 ⁱ | 0.96 | 2.56 | 3.514 (4) | 174 |

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

Fig. 1

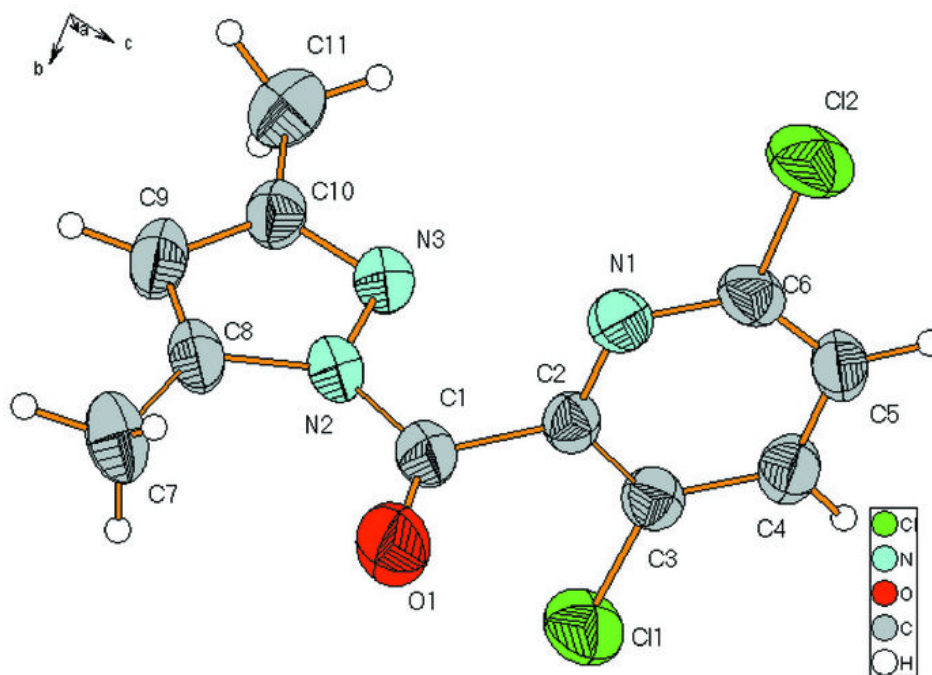


Fig. 2

