Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Chloro-*N*-[3-cyano-1-(3,4-dichlorophenyl)-1*H*-pyrazol-5-yl]acetamide

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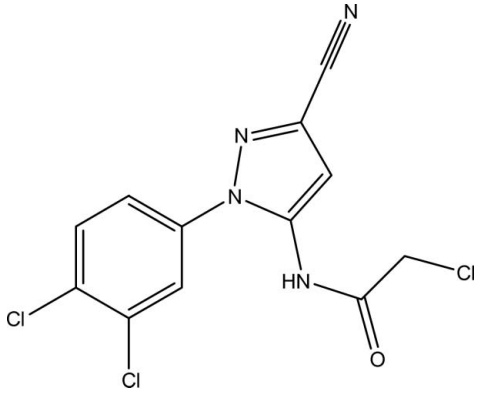
Received 22 February 2012; accepted 23 February 2012

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.042; wR factor = 0.139; data-to-parameter ratio = 14.1.

In the title compound, $C_{12}H_7Cl_3N_4O$, the dihedral angle between the pyrazole and benzene rings is 35.6 (3)°. In the crystal, molecules are linked by N—H···O hydrogen bonds generating $C(4)$ chains propagating in [100].

Related literature

For background to the properties of *N*-pyrazoles, see: Liu *et al.* (2010); Zhao *et al.* (2010).



Experimental

Crystal data

$C_{12}H_7Cl_3N_4O$
M_r = 329.57
Monoclinic, $P2_1/n$
a = 4.6280 (9) Å
b = 17.245 (3) Å
c = 17.468 (4) Å
β = 94.04 (3)°
V = 1390.7 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.66 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.827, T_{max} = 0.937
5687 measured reflections
2564 independent reflections
1880 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.139
S = 1.01
2564 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···O^{i}	0.86	1.95	2.743 (3)	153

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6649).

References

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supplementary materials

Acta Cryst. (2012). E**68**, o1150 [doi:10.1107/S1600536812008094]

2-Chloro-*N*-[3-cyano-1-(3,4-dichlorophenyl)-1*H*-pyrazol-5-yl]acetamide

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Experimental

To a stirred solution of 5-amino-1-(3,4-dichlorophenyl)-1H-pyrazole-3-carbonitrile (5 mmol) in THF (20 ml) was added 2-chloroacetyl chloride (5 mmol) dropwise at 0-5°C. After the addition, the reaction mixture was allowed to raise to room temperature and stirred for 2 h. The crude product (I) precipitated and was filterd. Pure compound (I) was obtained by crystallization from ethanol. Colourless blocks of (I) were obtained by slow evaporation of an acetone solution.

Refinement

All H atoms bonded to the C atoms were placed geometrically at the distances of 0.93-0.97 Å and included in the refinement in riding motion approximation with $U_{iso}(H) = 1.2$ or $1.5U_{eq}$ of the carrier atom.

Computing details

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software* (Enraf–Nonius, 1989); data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *SHELXL97* (Sheldrick, 2008).

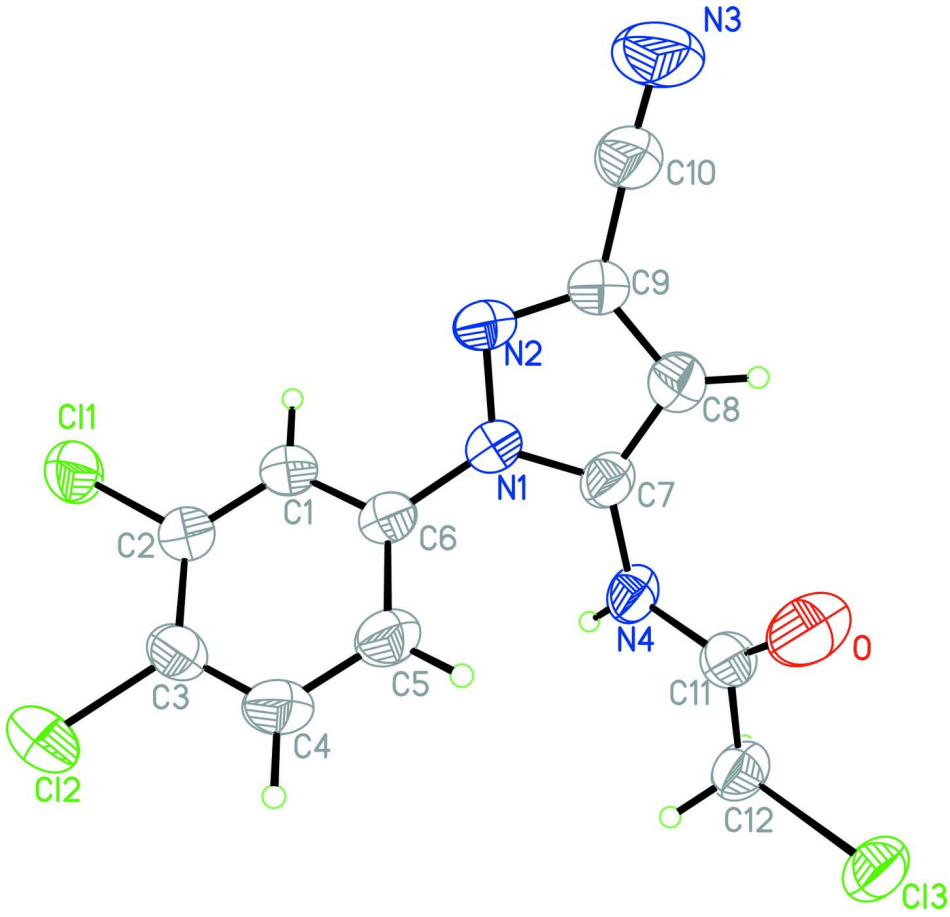

Figure 1

A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level.

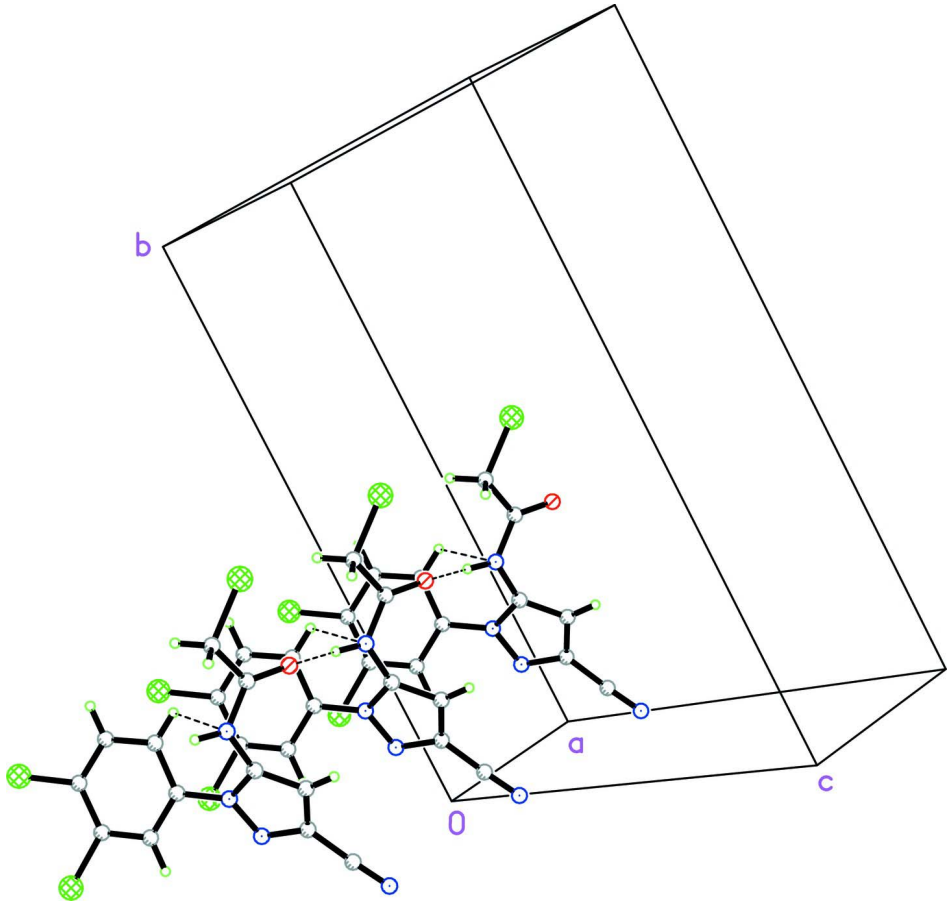

Figure 2

Packing diagram for (I).

2-Chloro-N-[3-cyano-1-(3,4-dichlorophenyl)-1*H*-pyrazol-5-yl]acetamide

Crystal data

$C_{12}H_7Cl_3N_4O$
$M_r = 329.57$
Monoclinic, $P2_1/n$
$a = 4.6280$ (9) Å
$b = 17.245$ (3) Å
$c = 17.468$ (4) Å
$\beta = 94.04$ (3)°
$V = 1390.7$ (5) Å^3
$Z = 4$
$F(000) = 664$
$D_x = 1.574$ Mg m^{-3}
Melting point: 473 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.66$ mm^{-1}
$T = 293$ K
Block, colorless
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.827$, $T_{max} = 0.937$
5687 measured reflections
2564 independent reflections
1880 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.7°$
$h = 0 \rightarrow 5$

$k = -20 \rightarrow 20$
$l = -21 \rightarrow 21$

3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.139$
$S = 1.01$
2564 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.090P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.31$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.019 (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}
O	0.8240 (5)	0.38138 (13)	0.35313 (18)	0.0813 (8)
Cl1	−0.40136 (18)	0.21696 (4)	−0.00295 (5)	0.0641 (3)
C1	−0.0120 (6)	0.23735 (15)	0.11596 (15)	0.0438 (6)
H1A	−0.0419	0.1852	0.1266	0.053*
Cl2	−0.3173 (2)	0.39579 (5)	−0.03563 (5)	0.0738 (3)
N1	0.3510 (5)	0.24173 (12)	0.22197 (12)	0.0426 (5)
N2	0.4349 (5)	0.16733 (12)	0.21185 (13)	0.0488 (6)
C2	−0.1647 (6)	0.27227 (15)	0.05502 (15)	0.0447 (6)
Cl3	0.69321 (17)	0.53185 (4)	0.41501 (5)	0.0602 (3)
C3	−0.1258 (7)	0.35054 (16)	0.04024 (15)	0.0496 (7)
N3	0.8375 (9)	0.01856 (18)	0.2981 (2)	0.0950 (11)
N4	0.3729 (4)	0.34308 (13)	0.31669 (13)	0.0456 (6)
H4A	0.1933	0.3562	0.3119	0.055*
C4	0.0724 (8)	0.39217 (16)	0.08606 (17)	0.0582 (8)
H4B	0.0990	0.4446	0.0762	0.070*
C5	0.2322 (7)	0.35752 (15)	0.14625 (16)	0.0521 (7)
H5A	0.3688	0.3857	0.1763	0.062*
C6	0.1850 (6)	0.27986 (14)	0.16112 (14)	0.0412 (6)
C7	0.4522 (5)	0.26933 (16)	0.29174 (15)	0.0425 (6)
C8	0.6121 (7)	0.21299 (17)	0.32747 (17)	0.0541 (7)
H8A	0.7116	0.2150	0.3756	0.065*
C9	0.5948 (7)	0.15113 (15)	0.27610 (16)	0.0489 (7)

C10	0.7278 (8)	0.07622 (19)	0.28682 (18)	0.0644 (9)
C11	0.5659 (6)	0.39367 (15)	0.34757 (15)	0.0426 (6)
C12	0.4304 (6)	0.46675 (15)	0.37652 (18)	0.0503 (7)
H12A	0.3004	0.4534	0.4157	0.060*
H12B	0.3171	0.4916	0.3346	0.060*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O	0.0337 (12)	0.0558 (13)	0.154 (3)	0.0049 (10)	0.0002 (13)	−0.0304 (15)
Cl1	0.0735 (6)	0.0549 (5)	0.0603 (5)	−0.0049 (4)	−0.0212 (4)	0.0010 (3)
C1	0.0494 (16)	0.0346 (13)	0.0471 (15)	−0.0013 (12)	0.0018 (12)	0.0003 (11)
Cl2	0.0949 (7)	0.0570 (5)	0.0673 (5)	0.0099 (4)	−0.0091 (5)	0.0194 (4)
N1	0.0480 (13)	0.0351 (11)	0.0440 (12)	−0.0008 (10)	−0.0004 (10)	−0.0051 (9)
N2	0.0626 (15)	0.0326 (11)	0.0507 (13)	0.0051 (10)	0.0013 (11)	−0.0040 (10)
C2	0.0482 (15)	0.0410 (14)	0.0448 (14)	0.0018 (12)	0.0021 (12)	−0.0013 (11)
Cl3	0.0585 (5)	0.0442 (4)	0.0767 (5)	−0.0064 (3)	−0.0043 (4)	−0.0129 (3)
C3	0.0626 (19)	0.0423 (15)	0.0440 (15)	0.0078 (13)	0.0044 (13)	0.0068 (11)
N3	0.138 (3)	0.0591 (19)	0.087 (2)	0.036 (2)	−0.001 (2)	0.0074 (16)
N4	0.0297 (11)	0.0483 (13)	0.0576 (13)	0.0067 (9)	−0.0046 (10)	−0.0182 (11)
C4	0.080 (2)	0.0360 (14)	0.0583 (18)	−0.0048 (14)	0.0049 (17)	0.0047 (12)
C5	0.067 (2)	0.0373 (14)	0.0515 (16)	−0.0100 (13)	0.0020 (14)	−0.0023 (12)
C6	0.0457 (14)	0.0339 (13)	0.0441 (14)	−0.0012 (11)	0.0040 (12)	−0.0037 (10)
C7	0.0361 (14)	0.0427 (14)	0.0485 (15)	0.0016 (11)	0.0005 (12)	−0.0111 (12)
C8	0.0557 (18)	0.0526 (17)	0.0526 (16)	0.0086 (14)	−0.0061 (14)	−0.0082 (13)
C9	0.0557 (17)	0.0393 (14)	0.0508 (16)	0.0054 (13)	−0.0012 (13)	−0.0021 (12)
C10	0.086 (2)	0.0515 (18)	0.0538 (17)	0.0120 (17)	−0.0060 (17)	−0.0009 (14)
C11	0.0333 (14)	0.0406 (14)	0.0535 (16)	0.0032 (11)	−0.0002 (12)	−0.0024 (11)
C12	0.0385 (15)	0.0445 (15)	0.0673 (18)	0.0007 (12)	−0.0006 (13)	−0.0132 (13)

Geometric parameters (Å, º)

O—C11	1.210 (3)	N4—C11	1.335 (3)
Cl1—C2	1.726 (3)	N4—C7	1.402 (3)
C1—C2	1.374 (4)	N4—H4A	0.8600
C1—C6	1.375 (3)	C4—C5	1.378 (4)
C1—H1A	0.9300	C4—H4B	0.9300
Cl2—C3	1.728 (3)	C5—C6	1.384 (4)
N1—N2	1.356 (3)	C5—H5A	0.9300
N1—C7	1.360 (3)	C7—C8	1.348 (4)
N1—C6	1.427 (3)	C8—C9	1.393 (4)
N2—C9	1.330 (3)	C8—H8A	0.9300
C2—C3	1.388 (4)	C9—C10	1.438 (4)
Cl3—C12	1.754 (3)	C11—C12	1.511 (4)
C3—C4	1.376 (4)	C12—H12A	0.9700
N3—C10	1.127 (4)	C12—H12B	0.9700
C2—C1—C6	119.7 (2)	C1—C6—C5	121.0 (3)
C2—C1—H1A	120.2	C1—C6—N1	118.8 (2)
C6—C1—H1A	120.2	C5—C6—N1	120.1 (2)

N2—N1—C7	111.4 (2)	C8—C7—N1	107.8 (2)
N2—N1—C6	118.88 (19)	C8—C7—N4	131.1 (2)
C7—N1—C6	129.7 (2)	N1—C7—N4	121.0 (2)
C9—N2—N1	103.7 (2)	C7—C8—C9	104.4 (3)
C1—C2—C3	120.3 (3)	C7—C8—H8A	127.8
C1—C2—Cl1	118.9 (2)	C9—C8—H8A	127.8
C3—C2—Cl1	120.9 (2)	N2—C9—C8	112.7 (2)
C4—C3—C2	119.3 (3)	N2—C9—C10	120.4 (2)
C4—C3—Cl2	119.7 (2)	C8—C9—C10	126.8 (3)
C2—C3—Cl2	121.0 (2)	N3—C10—C9	177.1 (4)
C11—N4—C7	122.4 (2)	O—C11—N4	123.1 (2)
C11—N4—H4A	118.8	O—C11—C12	123.3 (2)
C7—N4—H4A	118.8	N4—C11—C12	113.5 (2)
C3—C4—C5	121.2 (3)	C11—C12—Cl3	111.68 (19)
C3—C4—H4B	119.4	C11—C12—H12A	109.3
C5—C4—H4B	119.4	Cl3—C12—H12A	109.3
C4—C5—C6	118.6 (3)	C11—C12—H12B	109.3
C4—C5—H5A	120.7	Cl3—C12—H12B	109.3
C6—C5—H5A	120.7	H12A—C12—H12B	107.9

C7—N1—N2—C9	−1.5 (3)	N2—N1—C7—C8	1.7 (3)
C6—N1—N2—C9	176.8 (2)	C6—N1—C7—C8	−176.4 (3)
C6—C1—C2—C3	−1.6 (4)	N2—N1—C7—N4	−175.1 (2)
C6—C1—C2—Cl1	177.7 (2)	C6—N1—C7—N4	6.9 (4)
C1—C2—C3—C4	1.6 (4)	C11—N4—C7—C8	51.2 (5)
Cl1—C2—C3—C4	−177.7 (2)	C11—N4—C7—N1	−132.9 (3)
C1—C2—C3—Cl2	−179.5 (2)	N1—C7—C8—C9	−1.1 (3)
Cl1—C2—C3—Cl2	1.1 (4)	N4—C7—C8—C9	175.2 (3)
C2—C3—C4—C5	−0.1 (5)	N1—N2—C9—C8	0.8 (3)
Cl2—C3—C4—C5	−178.9 (2)	N1—N2—C9—C10	−179.3 (3)
C3—C4—C5—C6	−1.5 (5)	C7—C8—C9—N2	0.2 (4)
C2—C1—C6—C5	0.1 (4)	C7—C8—C9—C10	−179.7 (3)
C2—C1—C6—N1	−177.2 (2)	N2—C9—C10—N3	176 (8)
C4—C5—C6—C1	1.5 (4)	C8—C9—C10—N3	−4 (9)
C4—C5—C6—N1	178.7 (3)	C7—N4—C11—O	3.5 (5)
N2—N1—C6—C1	35.1 (3)	C7—N4—C11—C12	−175.1 (2)
C7—N1—C6—C1	−147.0 (3)	O—C11—C12—Cl3	2.4 (4)
N2—N1—C6—C5	−142.2 (3)	N4—C11—C12—Cl3	−179.0 (2)
C7—N1—C6—C5	35.7 (4)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···O^{i}	0.86	1.95	2.743 (3)	153

Symmetry code: (i) *x*−1, *y*, *z*.