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3-(4-Chlorophenyl)-1-(4,6-dimethylpyrimidin-2-yl)-1*H*-pyrazole-4-carbaldehyde

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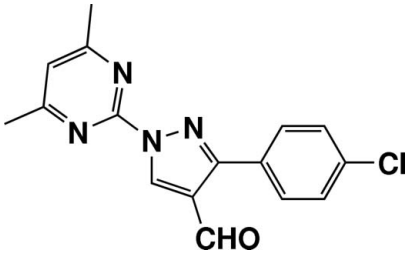
Key indicators: single-crystal X-ray study; T = 160 K; mean σ(C–C) = 0.004 Å; R factor = 0.058; wR factor = 0.165; data-to-parameter ratio = 16.3.

The asymmetric unit of the title compound, $C_{16}H_{13}ClN_4O$, contains two crystallographically independent molecules, A and B. The pyrazole ring of A makes dihedral angles of 5.8 (1) and 7.2 (1)° with the pyrimidine ring and the benzene ring, respectively; the corresponding values in molecule B are 7.1 (1) and 13.4 (1)°. The independent molecules display intramolecular C—H···N and C—H···O hydrogen bonds.

Related literature

For related literature, see: Wichmann *et al.* (1999); El-Bendary *et al.* (1998); Kirpal (1999); Tsuji & Ishikawa (1994); Mohan *et al.* (1989); Baraldi *et al.* (1996); Baraldi *et al.* (2003); Sanjay *et al.*, (2006).



Experimental

Crystal data

$C_{16}H_{13}ClN_4O$
M_r = 312.75
Triclinic, $P\overline{1}$
a = 7.2458 (2) Å
b = 13.6704 (4) Å
c = 14.4757 (3) Å
α = 90.718 (2)°
β = 92.618 (2)°
γ = 93.623 (1)°
V = 1429.33 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.28 mm^{-1}
T = 160 (1) K
0.38 × 0.23 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (Blessing, 1995)
T_{min} = 0.830, T_{max} = 0.948
34357 measured reflections
6537 independent reflections
5563 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.165
S = 1.17
6537 reflections
401 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.42 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C32A—H32A···N2A	0.95	2.43	2.770 (3)	101
C32B—H32B···N2B	0.95	2.47	2.800 (3)	100
C36A—H36A···O41A	0.95	2.21	3.076 (3)	152
C36B—H36B···O41B	0.95	2.23	3.054 (3)	144

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *PLATON* (Spek, 2003).

The data collection was carried out by Dr A. Linden of the Institute of Organic Chemistry at the University of Zürich. This help is gratefully acknowledged by AT.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2033).

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supplementary materials

Acta Cryst. (2007). E**63**, o2911 [**doi:10.1107/S1600536807024440**]

3-(4-Chlorophenyl)-1-(4,6-dimethylpyrimidin-2-yl)-1*H*-pyrazole-4-carbaldehyde

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Comment

Literature surveys shows that large numbers of simple, N–bridged, nitrogen and sulfur containing heterocyclic compounds carrying pyrimidine moiety are found to be associated with diverse biological activities, (Wichmann *et al.*, 1999; El-Bendary *et al.*, 1998; Kirpal, 1999; Tsuji & Ishikawa, 1994). Pyrazoles constitute an important class of heterocycles, which display interesting biological properties such as fungicidal, anti-inflamatory, anti-arthritic, anti-depressant and antiviral activity. Many pyrazolines find industrial use as polymer intermediates (Mohan *et al.*, 1989; Baraldi *et al.*, 1996; Baraldi *et al.*, 2003; Sanjay *et al.*, 2006).

The asymmetric unit of (I), Fig. 1, contains two crystallographically independent molecules, A and B. The pyrazole ring of A makes dihedral angles of 5.8 (1)° and 7.2 (1)° with the pyrimidine ring and the phenyl ring respectively; the corresponding values in molecule B are 7.1 (1)° and 13.4 (1)°. The independent molecules contain intramolecular C32A—H32A···N2A, C32B—H32B···N2B, C36A—H36A···O41A, and C36B—H36B···O41B, hydrogen bonds. Further, the molecules A and B are linked by a number of relatively short C—H···N and C—H···O intermolecular contacts.

Experimental

To the Vilsmeier–Haack complex, prepared from DMF (30 ml) and $POCl_3$ (4.6 g, 0.03 mol) at 273–278 K was added 1-(4-chloro-phenylethanone-(4,6-dimethylpyrimidin-2-yl)hydrazone (2.74 g, 0.01 mol). The reaction mixture was stirred at 333–343 K for 4 h, and monitored by TLC. The contents were cooled, poured into ice cold water and neutralized using sodium carbonate. The product that separated was filtered and recrystallized from ethyl acetate to give yellow crystals (2.1 g, 67%).

Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.95–0.98 Å and U_{iso}=1.2 or 1.5 times U_{eq}(C).

Figures

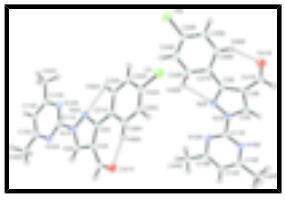


Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

3-(4-Chlorophenyl)-1-(4,6-dimethylpyrimidin-2-yl)-1*H*-pyrazole-4-carbaldehyde

Crystal data

$C_{16}H_{13}ClN_4O$	$Z = 4$
$M_r = 312.75$	$F_{000} = 648$
Triclinic, $P\overline{1}$	$D_x = 1.453$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 442(1) K
$a = 7.2458$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 13.6704$ (4) Å	Cell parameters from 53782 reflections
$c = 14.4757$ (3) Å	$\theta = 2.0$–27.5º
$\alpha = 90.718$ (2)º	$\mu = 0.28$ mm^{-1}
$\beta = 92.618$ (2)º	$T = 160$ (1) K
$\gamma = 93.623$ (1)º	Block, light brown
$V = 1429.33$ (6) Å^3	0.38 × 0.23 × 0.20 mm

Data collection

Nonius KappaCCD area-detector diffractometer	6537 independent reflections
Radiation source: Nonius FR590 sealed tube generator	5563 reflections with $I > 2\sigma(I)$
Monochromator: horizontally mounted graphite crystal	$R_{int} = 0.060$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 27.5$º
$T = 160(1)$ K	$\theta_{min} = 2.0$º
φ and ω scans with κ offsets	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (Blessing, 1995)	$k = -17 \rightarrow 17$
$T_{min} = 0.830$, $T_{max} = 0.948$	$l = -18 \rightarrow 18$
34357 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.058$	H-atom parameters constrained
$wR(F^2) = 0.165$	$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2 + 1.7016P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.17$	$(\Delta/\sigma)_{max} = <0.001$
6537 reflections	$\Delta\rho_{max} = 0.42$ e Å^{-3}
401 parameters	$\Delta\rho_{min} = -0.46$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. Solvent used: Ethyl acetate Cooling Device: Oxford Cryosystems Cryostream 700 Crystal mount: glued on a glass fibre Mosaicity (°.): 1.077 (2) Frames collected: 374 Seconds exposure per frame: 72 Degrees rotation per frame: 1.8 Crystal-Detector distance (mm): 30.0

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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}
Cl1	0.51295 (10)	0.68301 (5)	0.01191 (4)	0.0365 (2)
O41A	0.5611 (3)	0.74111 (14)	0.48792 (13)	0.0342 (6)
N1A	0.7911 (3)	0.43476 (14)	0.48997 (13)	0.0218 (5)
N2A	0.7564 (3)	0.45038 (14)	0.39789 (13)	0.0221 (5)
N12A	0.9017 (3)	0.28056 (15)	0.46061 (14)	0.0241 (6)
N16A	0.8668 (3)	0.34137 (15)	0.61465 (14)	0.0224 (6)
C3A	0.6944 (3)	0.54000 (16)	0.39297 (16)	0.0206 (6)
C4A	0.6906 (3)	0.58290 (17)	0.48430 (16)	0.0219 (6)
C5A	0.7543 (3)	0.51247 (17)	0.54265 (17)	0.0229 (7)
C11A	0.8575 (3)	0.34531 (17)	0.52289 (16)	0.0216 (6)
C13A	0.9668 (3)	0.19776 (17)	0.49692 (17)	0.0252 (7)
C14A	0.9818 (3)	0.18455 (18)	0.59144 (18)	0.0267 (7)
C15A	0.9311 (3)	0.25858 (18)	0.64966 (16)	0.0236 (6)
C31A	0.6484 (3)	0.57771 (17)	0.30050 (16)	0.0213 (6)
C32A	0.6891 (3)	0.52279 (17)	0.22255 (16)	0.0237 (7)
C33A	0.6465 (4)	0.55425 (18)	0.13403 (17)	0.0271 (7)
C34A	0.5645 (3)	0.64273 (18)	0.12273 (17)	0.0256 (7)
C35A	0.5241 (3)	0.69938 (18)	0.19835 (18)	0.0259 (7)
C36A	0.5643 (3)	0.66604 (18)	0.28682 (17)	0.0258 (7)
C41A	0.6371 (4)	0.67460 (18)	0.52475 (17)	0.0267 (7)
C44A	1.0221 (4)	0.1229 (2)	0.42840 (19)	0.0342 (8)
C46A	0.9439 (4)	0.25196 (19)	0.75273 (17)	0.0278 (7)
Cl2	−0.16204 (10)	0.51727 (5)	−0.18594 (5)	0.0392 (2)
O41B	0.0848 (3)	1.00278 (14)	−0.23689 (13)	0.0395 (7)
N1B	0.2953 (3)	1.00178 (14)	0.06769 (13)	0.0213 (6)
N2B	0.2305 (3)	0.90661 (14)	0.05160 (14)	0.0221 (6)
N12B	0.3945 (3)	0.96995 (15)	0.22009 (14)	0.0243 (6)
N16B	0.4076 (3)	1.13192 (15)	0.15907 (14)	0.0237 (6)
C3B	0.1713 (3)	0.90258 (17)	−0.03703 (15)	0.0194 (6)
C4B	0.2013 (3)	0.99737 (17)	−0.07844 (16)	0.0217 (6)

C5B	0.2803 (3)	1.05697 (17)	−0.00814 (16)	0.0226 (6)
C11B	0.3705 (3)	1.03589 (17)	0.15578 (16)	0.0216 (6)
C13B	0.4647 (4)	1.00728 (19)	0.30196 (17)	0.0266 (7)
C14B	0.5048 (3)	1.10726 (19)	0.31526 (17)	0.0269 (7)
C15B	0.4774 (3)	1.16840 (18)	0.24049 (17)	0.0243 (7)
C31B	0.0901 (3)	0.80849 (17)	−0.07541 (16)	0.0214 (6)
C32B	0.0442 (4)	0.73270 (19)	−0.01571 (18)	0.0310 (8)
C33B	−0.0313 (4)	0.6433 (2)	−0.0493 (2)	0.0349 (8)
C34B	−0.0604 (3)	0.62868 (18)	−0.14341 (19)	0.0269 (7)
C35B	−0.0130 (4)	0.7013 (2)	−0.20448 (18)	0.0321 (8)
C36B	0.0617 (4)	0.79150 (19)	−0.17014 (18)	0.0294 (7)
C41B	0.1626 (4)	1.04061 (19)	−0.16822 (17)	0.0285 (7)
C44B	0.5025 (5)	0.9345 (2)	0.37569 (19)	0.0394 (9)
C46B	0.5244 (4)	1.27659 (19)	0.24509 (19)	0.0323 (8)
H5A	0.76945	0.51769	0.60810	0.0274*
H14A	1.02609	0.12587	0.61604	0.0320*
H32A	0.74699	0.46284	0.23056	0.0285*
H33A	0.67301	0.51584	0.08170	0.0325*
H35A	0.46969	0.76023	0.18981	0.0311*
H36A	0.53419	0.70385	0.33889	0.0309*
H41A	0.66616	0.68331	0.58911	0.0320*
H44A	0.91088	0.08764	0.40056	0.0513*
H44B	1.09989	0.07644	0.45998	0.0513*
H44C	1.09147	0.15590	0.37990	0.0513*
H46A	1.07426	0.25825	0.77451	0.0417*
H46B	0.88815	0.18848	0.77132	0.0417*
H46C	0.87780	0.30481	0.77992	0.0417*
H4A	0.38606	0.89951	0.39133	0.0591*
H4B	0.55884	0.96872	0.43085	0.0591*
H4C	0.58734	0.88742	0.35318	0.0591*
H5B	0.31737	1.12461	−0.01242	0.0271*
H6A	0.59384	1.29624	0.19111	0.0484*
H6B	0.59991	1.29309	0.30168	0.0484*
H6C	0.41009	1.31127	0.24549	0.0484*
H14B	0.54977	1.13317	0.37380	0.0323*
H32B	0.06511	0.74263	0.04909	0.0372*
H33B	−0.06302	0.59224	−0.00784	0.0418*
H35B	−0.03094	0.68996	−0.26925	0.0385*
H36B	0.09374	0.84217	−0.21193	0.0353*
H41B	0.20395	1.10753	−0.17374	0.0342*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0482 (4)	0.0357 (3)	0.0253 (3)	0.0061 (3)	−0.0074 (3)	0.0069 (3)
O41A	0.0463 (12)	0.0249 (9)	0.0321 (10)	0.0079 (8)	0.0013 (8)	−0.0020 (8)
N1A	0.0259 (10)	0.0197 (9)	0.0192 (9)	−0.0006 (8)	−0.0014 (8)	0.0011 (7)
N2A	0.0263 (10)	0.0207 (9)	0.0191 (9)	0.0013 (8)	−0.0015 (8)	0.0032 (7)

N12A	0.0282 (10)	0.0204 (10)	0.0232 (10)	−0.0007 (8)	−0.0019 (8)	0.0006 (8)
N16A	0.0226 (10)	0.0228 (10)	0.0215 (10)	−0.0001 (8)	−0.0008 (8)	0.0023 (8)
C3A	0.0206 (11)	0.0184 (10)	0.0223 (11)	−0.0019 (8)	0.0006 (9)	−0.0001 (8)
C4A	0.0220 (11)	0.0213 (11)	0.0220 (11)	−0.0008 (9)	0.0013 (9)	0.0005 (9)
C5A	0.0248 (12)	0.0218 (11)	0.0216 (11)	−0.0013 (9)	0.0005 (9)	−0.0007 (9)
C11A	0.0205 (11)	0.0193 (10)	0.0244 (11)	−0.0013 (8)	−0.0021 (9)	0.0024 (9)
C13A	0.0275 (12)	0.0208 (11)	0.0267 (12)	−0.0005 (9)	−0.0005 (9)	−0.0002 (9)
C14A	0.0285 (12)	0.0231 (12)	0.0284 (12)	0.0037 (10)	−0.0023 (10)	0.0042 (9)
C15A	0.0233 (11)	0.0245 (11)	0.0224 (11)	−0.0020 (9)	−0.0008 (9)	0.0037 (9)
C31A	0.0210 (11)	0.0189 (10)	0.0234 (11)	−0.0020 (8)	0.0001 (9)	0.0013 (9)
C32A	0.0293 (12)	0.0184 (11)	0.0236 (12)	0.0019 (9)	0.0012 (9)	0.0016 (9)
C33A	0.0345 (13)	0.0235 (12)	0.0233 (12)	0.0001 (10)	0.0039 (10)	0.0012 (9)
C34A	0.0274 (12)	0.0262 (12)	0.0227 (11)	−0.0003 (10)	−0.0020 (9)	0.0045 (9)
C35A	0.0253 (12)	0.0231 (11)	0.0296 (12)	0.0040 (9)	0.0004 (10)	0.0038 (9)
C36A	0.0284 (12)	0.0236 (12)	0.0259 (12)	0.0049 (9)	0.0031 (10)	0.0005 (9)
C41A	0.0317 (13)	0.0250 (12)	0.0232 (12)	0.0001 (10)	0.0029 (10)	−0.0022 (9)
C44A	0.0467 (16)	0.0254 (13)	0.0307 (14)	0.0073 (11)	−0.0014 (12)	−0.0035 (10)
C46A	0.0334 (13)	0.0268 (12)	0.0234 (12)	0.0036 (10)	−0.0013 (10)	0.0050 (9)
Cl2	0.0381 (4)	0.0259 (3)	0.0514 (4)	−0.0078 (3)	−0.0047 (3)	−0.0083 (3)
O41B	0.0637 (14)	0.0291 (10)	0.0249 (10)	0.0072 (9)	−0.0126 (9)	−0.0010 (8)
N1B	0.0245 (10)	0.0181 (9)	0.0210 (10)	0.0015 (7)	−0.0024 (8)	−0.0026 (7)
N2B	0.0241 (10)	0.0188 (9)	0.0228 (10)	−0.0009 (8)	−0.0014 (8)	−0.0032 (7)
N12B	0.0276 (10)	0.0237 (10)	0.0211 (10)	0.0007 (8)	−0.0016 (8)	−0.0009 (8)
N16B	0.0269 (10)	0.0208 (10)	0.0230 (10)	0.0002 (8)	−0.0015 (8)	−0.0024 (8)
C3B	0.0181 (10)	0.0216 (11)	0.0188 (10)	0.0029 (8)	0.0016 (8)	−0.0024 (8)
C4B	0.0231 (11)	0.0199 (11)	0.0224 (11)	0.0047 (9)	0.0000 (9)	−0.0019 (9)
C5B	0.0242 (11)	0.0194 (11)	0.0242 (11)	0.0033 (9)	−0.0020 (9)	0.0002 (9)
C11B	0.0204 (11)	0.0236 (11)	0.0207 (11)	0.0027 (9)	−0.0006 (9)	−0.0037 (9)
C13B	0.0296 (13)	0.0288 (12)	0.0215 (11)	0.0030 (10)	0.0001 (9)	−0.0011 (9)
C14B	0.0287 (12)	0.0283 (12)	0.0232 (12)	0.0023 (10)	−0.0039 (9)	−0.0049 (9)
C15B	0.0236 (11)	0.0232 (12)	0.0258 (12)	0.0010 (9)	−0.0004 (9)	−0.0058 (9)
C31B	0.0193 (10)	0.0218 (11)	0.0230 (11)	0.0022 (9)	−0.0003 (9)	−0.0029 (9)
C32B	0.0406 (15)	0.0284 (13)	0.0230 (12)	−0.0044 (11)	0.0005 (10)	−0.0012 (10)
C33B	0.0429 (16)	0.0262 (13)	0.0347 (14)	−0.0068 (11)	0.0046 (12)	0.0047 (11)
C34B	0.0206 (11)	0.0224 (11)	0.0368 (14)	−0.0017 (9)	−0.0010 (10)	−0.0055 (10)
C35B	0.0408 (15)	0.0299 (13)	0.0240 (12)	−0.0041 (11)	−0.0058 (11)	−0.0039 (10)
C36B	0.0389 (14)	0.0249 (12)	0.0236 (12)	−0.0027 (10)	−0.0012 (10)	0.0007 (10)
C41B	0.0385 (14)	0.0219 (12)	0.0252 (12)	0.0057 (10)	−0.0028 (10)	0.0015 (9)
C44B	0.0601 (19)	0.0315 (14)	0.0253 (13)	−0.0003 (13)	−0.0086 (12)	0.0035 (11)
C46B	0.0404 (15)	0.0224 (12)	0.0328 (14)	−0.0013 (10)	−0.0046 (11)	−0.0068 (10)

Geometric parameters (Å, °)

Cl1—C34A	1.735 (3)	C33A—H33A	0.9500
Cl2—C34B	1.742 (3)	C35A—H35A	0.9500
O41A—C41A	1.210 (3)	C36A—H36A	0.9500
O41B—C41B	1.213 (3)	C41A—H41A	0.9500
N1A—N2A	1.367 (3)	C44A—H44C	0.9800
N1A—C5A	1.347 (3)	C44A—H44B	0.9800

N1A—C11A	1.422 (3)	C44A—H44A	0.9800
N2A—C3A	1.333 (3)	C46A—H46A	0.9800
N12A—C13A	1.356 (3)	C46A—H46B	0.9800
N12A—C11A	1.320 (3)	C46A—H46C	0.9800
N16A—C11A	1.329 (3)	C3B—C4B	1.443 (3)
N16A—C15A	1.347 (3)	C3B—C31B	1.472 (3)
N1B—C5B	1.344 (3)	C4B—C5B	1.376 (3)
N1B—C11B	1.425 (3)	C4B—C41B	1.457 (3)
N1B—N2B	1.368 (3)	C13B—C14B	1.388 (4)
N2B—C3B	1.333 (3)	C13B—C44B	1.496 (4)
N12B—C13B	1.349 (3)	C14B—C15B	1.391 (4)
N12B—C11B	1.317 (3)	C15B—C46B	1.497 (4)
N16B—C11B	1.323 (3)	C31B—C32B	1.391 (3)
N16B—C15B	1.339 (3)	C31B—C36B	1.392 (3)
C3A—C31A	1.472 (3)	C32B—C33B	1.380 (4)
C3A—C4A	1.442 (3)	C33B—C34B	1.379 (4)
C4A—C5A	1.376 (3)	C34B—C35B	1.375 (4)
C4A—C41A	1.459 (3)	C35B—C36B	1.392 (4)
C13A—C14A	1.383 (4)	C5B—H5B	0.9500
C13A—C44A	1.501 (4)	C14B—H14B	0.9500
C14A—C15A	1.387 (3)	C32B—H32B	0.9500
C15A—C46A	1.495 (3)	C33B—H33B	0.9500
C31A—C32A	1.400 (3)	C35B—H35B	0.9500
C31A—C36A	1.399 (3)	C36B—H36B	0.9500
C32A—C33A	1.384 (3)	C41B—H41B	0.9500
C33A—C34A	1.389 (4)	C44B—H4A	0.9800
C34A—C35A	1.386 (4)	C44B—H4B	0.9800
C35A—C36A	1.389 (4)	C44B—H4C	0.9800
C5A—H5A	0.9500	C46B—H6A	0.9800
C14A—H14A	0.9500	C46B—H6B	0.9800
C32A—H32A	0.9500	C46B—H6C	0.9800
Cl1···C32B	3.512 (3)	C4A···H36A	2.9200
Cl1···C33B^{i}	3.531 (3)	C4B···H36B	2.9000
Cl2···C32A^{ii}	3.567 (2)	C14A···H4A^{vi}	2.8600
Cl1···H6A^{iii}	3.0300	C15A···H4A^{vi}	3.0900
Cl1···H5B^{iii}	2.8300	C33A···H33B^{i}	3.0300
Cl2···H32A^{ii}	3.1000	C34B···H6C^{v}	3.0400
Cl2···H46C^{iv}	2.9700	C34B···H32A^{ii}	2.9800
Cl2···H5A^{iv}	3.0000	C35A···H46A^{ix}	2.9400
Cl2···H6C^{v}	3.1500	C35A···H46C^{vi}	2.9400
O41A···C44B	3.162 (3)	C35B···H6C^{v}	2.9100
O41A···C36A	3.076 (3)	C35B···H32A^{ii}	3.0800
O41A···C11A^{vi}	3.181 (3)	C35B···H6A^{iii}	3.0300
O41B···C36B	3.054 (3)	C36A···H46A^{ix}	2.9300
O41B···C44A^{ii}	3.279 (3)	C36B···H6A^{iii}	2.8600
O41A···H36A	2.2100	C41A···H36A	2.8000

O41A···H4A	2.9200	C41B···H46B^{viii}	3.0300
O41A···H4C	2.8200	C41B···H36B	2.7900
O41A···H14B^{vii}	2.8000	C46A···H41B^{xii}	2.9900
O41B···H36B	2.2300	C46A···H32B^{vi}	2.8700
O41B···H14A^{viii}	2.7600	C46B···H41A^{vii}	2.8900
O41B···H44A^{ii}	2.6600	C46B···H32A^{xiii}	2.9400
N1A···C5A^{ix}	3.383 (3)	H4A···O41A	2.9200
N1A···C41A^{vi}	3.354 (4)	H4A···C14A^{vi}	2.8600
N1B···C5B^{iii}	3.374 (3)	H4A···C15A^{vi}	3.0900
N2A···N12A	2.756 (3)	H4B···H14B	2.4100
N2B···N12B	2.768 (3)	H4B···H4B^{vii}	2.3800
N12A···C41A^{ix}	3.356 (4)	H4C···H36A	2.5200
N12A···N2A	2.756 (3)	H4C···O41A	2.8200
N12A···C4A^{ix}	3.449 (3)	H5A···N16A	2.5600
N12B···N2B	2.768 (3)	H5A···Cl2^{xiv}	3.0000
N12B···C41B^{iii}	3.341 (4)	H5A···H41A	2.4400
N2A···H32A	2.4300	H5B···Cl1^{iii}	2.8300
N2A···H6B^{x}	2.7100	H5B···N16B	2.5400
N2B···H32B	2.4700	H5B···H41B	2.4400
N12B···H46B^{vi}	2.8900	H6A···H32A^{xiii}	2.5200
N16A···H5A	2.5600	H6A···C36B^{iii}	2.8600
N16B···H5B	2.5400	H6A···Cl1^{iii}	3.0300
C3A···C5A^{vi}	3.457 (3)	H6A···C35B^{iii}	3.0300
C3B···C4B^{v}	3.574 (3)	H6B···H41A^{vii}	2.5800
C3B···C5B^{v}	3.443 (3)	H6B···H14B	2.4500
C4A···C11A^{ix}	3.365 (3)	H6B···N2A^{xiii}	2.7100
C4A···C5A^{vi}	3.404 (3)	H6C···C34B^{v}	3.0400
C4A···C4A^{vi}	3.510 (3)	H6C···C35B^{v}	2.9100
C4A···N12A^{ix}	3.449 (3)	H6C···Cl2^{v}	3.1500
C4B···C3B^{v}	3.574 (3)	H6C···H41A^{vii}	2.4800
C4B···C11B^{iii}	3.401 (3)	H14A···H44B	2.4400
C5A···C11A^{ix}	3.492 (3)	H14A···O41B^{xii}	2.7600
C5A···C3A^{vi}	3.457 (3)	H14B···H6B	2.4500
C5A···C4A^{vi}	3.404 (3)	H14B···H4B	2.4100
C5A···N1A^{ix}	3.383 (3)	H14B···O41A^{vii}	2.8000
C5B···N1B^{iii}	3.374 (3)	H32A···N2A	2.4300
C5B···C3B^{v}	3.443 (3)	H32A···C35B^{ii}	3.0800
C5B···C31B^{v}	3.594 (3)	H32A···H6A^{x}	2.5200
C11A···C5A^{ix}	3.492 (3)	H32A···Cl2^{ii}	3.1000
C11A···C4A^{ix}	3.365 (3)	H32A···C46B^{x}	2.9400
C11A···O41A^{vi}	3.181 (3)	H32A···C34B^{ii}	2.9800

C11B···C4B^{iii}	3.401 (3)	H32B···C46A^{vi}	2.8700
C13A···C41A^{ix}	3.293 (4)	H32B···H46C^{vi}	2.5900
C13B···C41B^{iii}	3.484 (4)	H32B···N2B	2.4700
C15B···C36B^{iii}	3.547 (4)	H33A···H33B^{i}	2.5400
C31B···C5B^{v}	3.594 (3)	H33B···H33A^{xi}	2.5400
C32A···C46B^{x}	3.523 (3)	H33B···C33A^{xi}	3.0300
C32A···Cl2^{ii}	3.567 (2)	H36A···C4A	2.9200
C32B···Cl1	3.512 (3)	H36A···O41A	2.2100
C33B···Cl1^{xi}	3.531 (3)	H36A···H4C	2.5200
C35A···C46A^{vi}	3.597 (4)	H36A···C41A	2.8000
C36A···C41A	3.460 (3)	H36B···C4B	2.9000
C36A···O41A	3.076 (3)	H36B···C41B	2.7900
C36B···C41B	3.437 (4)	H36B···O41B	2.2300
C36B···O41B	3.054 (3)	H41A···H5A	2.4400
C36B···C46B^{iii}	3.415 (4)	H41A···H6C^{vii}	2.4800
C36B···C15B^{iii}	3.547 (4)	H41A···H6B^{vii}	2.5800
C41A···C13A^{ix}	3.293 (4)	H41A···C46B^{vii}	2.8900
C41A···N12A^{ix}	3.356 (4)	H41B···H46A^{viii}	2.4300
C41A···C36A	3.460 (3)	H41B···H5B	2.4400
C41A···N1A^{vi}	3.354 (4)	H41B···C46A^{viii}	2.9900
C41B···N12B^{iii}	3.341 (4)	H44A···O41B^{ii}	2.6600
C41B···C46A^{viii}	3.559 (4)	H44B···H14A	2.4400
C41B···C13B^{iii}	3.484 (4)	H46A···C36A^{ix}	2.9300
C41B···C36B	3.437 (4)	H46A···H41B^{xii}	2.4300
C44A···O41B^{ii}	3.279 (3)	H46A···C35A^{ix}	2.9400
C44B···O41A	3.162 (3)	H46B···N12B^{vi}	2.8900
C46A···C35A^{vi}	3.597 (4)	H46B···C41B^{xii}	3.0300
C46A···C41B^{xii}	3.559 (4)	H46C···H32B^{vi}	2.5900
C46B···C32A^{xiii}	3.523 (3)	H46C···Cl2^{xiv}	2.9700
C46B···C36B^{iii}	3.415 (4)	H46C···C35A^{vi}	2.9400
N2A—N1A—C5A	112.13 (19)	H46B—C46A—H46C	109.00
N2A—N1A—C11A	122.00 (19)	C15A—C46A—H46A	109.00
C5A—N1A—C11A	125.9 (2)	C15A—C46A—H46B	109.00
N1A—N2A—C3A	105.52 (18)	C15A—C46A—H46C	109.00
C11A—N12A—C13A	114.2 (2)	H46A—C46A—H46B	109.00
C11A—N16A—C15A	115.2 (2)	H46A—C46A—H46C	109.00
C5B—N1B—C11B	125.1 (2)	N2B—C3B—C4B	110.1 (2)
N2B—N1B—C5B	112.43 (19)	N2B—C3B—C31B	118.4 (2)
N2B—N1B—C11B	122.43 (19)	C4B—C3B—C31B	131.5 (2)
N1B—N2B—C3B	105.31 (18)	C3B—C4B—C5B	104.8 (2)
C11B—N12B—C13B	114.3 (2)	C3B—C4B—C41B	136.7 (2)
C11B—N16B—C15B	115.4 (2)	C5B—C4B—C41B	118.5 (2)
N2A—C3A—C4A	110.2 (2)	N1B—C5B—C4B	107.3 (2)
N2A—C3A—C31A	117.6 (2)	N1B—C11B—N12B	117.4 (2)

C4A—C3A—C31A	132.2 (2)	N1B—C11B—N16B	112.7 (2)
C3A—C4A—C5A	104.7 (2)	N12B—C11B—N16B	129.9 (2)
C3A—C4A—C41A	136.9 (2)	N12B—C13B—C14B	121.5 (2)
C5A—C4A—C41A	118.4 (2)	N12B—C13B—C44B	116.1 (2)
N1A—C5A—C4A	107.5 (2)	C14B—C13B—C44B	122.4 (2)
N1A—C11A—N12A	117.4 (2)	C13B—C14B—C15B	118.2 (2)
N12A—C11A—N16A	130.0 (2)	N16B—C15B—C14B	120.7 (2)
N1A—C11A—N16A	112.7 (2)	N16B—C15B—C46B	116.3 (2)
N12A—C13A—C14A	121.4 (2)	C14B—C15B—C46B	123.0 (2)
C14A—C13A—C44A	122.7 (2)	C3B—C31B—C32B	119.4 (2)
N12A—C13A—C44A	115.9 (2)	C3B—C31B—C36B	122.2 (2)
C13A—C14A—C15A	118.8 (2)	C32B—C31B—C36B	118.4 (2)
N16A—C15A—C46A	116.5 (2)	C31B—C32B—C33B	120.9 (2)
N16A—C15A—C14A	120.6 (2)	C32B—C33B—C34B	119.5 (2)
C14A—C15A—C46A	122.9 (2)	Cl2—C34B—C33B	119.7 (2)
C3A—C31A—C36A	122.9 (2)	Cl2—C34B—C35B	119.3 (2)
C3A—C31A—C32A	118.9 (2)	C33B—C34B—C35B	121.1 (2)
C32A—C31A—C36A	118.3 (2)	C34B—C35B—C36B	119.1 (2)
C31A—C32A—C33A	121.2 (2)	C31B—C36B—C35B	120.9 (2)
C32A—C33A—C34A	119.1 (2)	O41B—C41B—C4B	128.8 (2)
Cl1—C34A—C33A	119.28 (19)	N1B—C5B—H5B	126.00
Cl1—C34A—C35A	119.58 (19)	C4B—C5B—H5B	126.00
C33A—C34A—C35A	121.1 (2)	C13B—C14B—H14B	121.00
C34A—C35A—C36A	119.1 (2)	C15B—C14B—H14B	121.00
C31A—C36A—C35A	121.1 (2)	C31B—C32B—H32B	120.00
O41A—C41A—C4A	129.2 (2)	C33B—C32B—H32B	120.00
N1A—C5A—H5A	126.00	C32B—C33B—H33B	120.00
C4A—C5A—H5A	126.00	C34B—C33B—H33B	120.00
C15A—C14A—H14A	121.00	C34B—C35B—H35B	120.00
C13A—C14A—H14A	121.00	C36B—C35B—H35B	120.00
C31A—C32A—H32A	119.00	C31B—C36B—H36B	120.00
C33A—C32A—H32A	119.00	C35B—C36B—H36B	120.00
C32A—C33A—H33A	120.00	O41B—C41B—H41B	116.00
C34A—C33A—H33A	120.00	C4B—C41B—H41B	116.00
C36A—C35A—H35A	120.00	C13B—C44B—H4A	109.00
C34A—C35A—H35A	120.00	C13B—C44B—H4B	110.00
C31A—C36A—H36A	119.00	C13B—C44B—H4C	109.00
C35A—C36A—H36A	119.00	H4A—C44B—H4B	109.00
O41A—C41A—H41A	115.00	H4A—C44B—H4C	109.00
C4A—C41A—H41A	115.00	H4B—C44B—H4C	109.00
H44A—C44A—H44B	110.00	C15B—C46B—H6A	109.00
H44A—C44A—H44C	109.00	C15B—C46B—H6B	109.00
C13A—C44A—H44A	109.00	C15B—C46B—H6C	109.00
C13A—C44A—H44B	109.00	H6A—C46B—H6B	110.00
C13A—C44A—H44C	109.00	H6A—C46B—H6C	109.00
H44B—C44A—H44C	110.00	H6B—C46B—H6C	109.00
C5A—N1A—N2A—C3A	−0.6 (3)	C41A—C4A—C5A—N1A	178.9 (2)
C11A—N1A—N2A—C3A	179.1 (2)	C3A—C4A—C5A—N1A	−0.4 (2)
N2A—N1A—C5A—C4A	0.6 (3)	C5A—C4A—C41A—O41A	−171.7 (3)

C11A—N1A—C5A—C4A	−179.1 (2)	C3A—C4A—C41A—O41A	7.2 (5)
N2A—N1A—C11A—N12A	6.3 (3)	N12A—C13A—C14A—C15A	−0.9 (3)
N2A—N1A—C11A—N16A	−174.4 (2)	C44A—C13A—C14A—C15A	178.6 (2)
C5A—N1A—C11A—N12A	−174.0 (2)	C13A—C14A—C15A—C46A	−179.4 (2)
C5A—N1A—C11A—N16A	5.3 (3)	C13A—C14A—C15A—N16A	0.6 (3)
N1A—N2A—C3A—C4A	0.4 (3)	C3A—C31A—C32A—C33A	179.1 (2)
N1A—N2A—C3A—C31A	178.90 (19)	C36A—C31A—C32A—C33A	−0.4 (3)
C13A—N12A—C11A—N1A	179.0 (2)	C3A—C31A—C36A—C35A	179.6 (2)
C13A—N12A—C11A—N16A	−0.2 (4)	C32A—C31A—C36A—C35A	−0.8 (3)
C11A—N12A—C13A—C14A	0.6 (3)	C31A—C32A—C33A—C34A	1.0 (4)
C11A—N12A—C13A—C44A	−178.9 (2)	C32A—C33A—C34A—C35A	−0.3 (4)
C15A—N16A—C11A—N1A	−179.2 (2)	C32A—C33A—C34A—Cl1	179.38 (19)
C15A—N16A—C11A—N12A	0.0 (4)	Cl1—C34A—C35A—C36A	179.39 (17)
C11A—N16A—C15A—C14A	−0.2 (3)	C33A—C34A—C35A—C36A	−0.9 (3)
C11A—N16A—C15A—C46A	179.8 (2)	C34A—C35A—C36A—C31A	1.5 (3)
N2B—N1B—C11B—N16B	−173.6 (2)	N2B—C3B—C4B—C5B	−0.5 (3)
C5B—N1B—C11B—N12B	−172.5 (2)	N2B—C3B—C4B—C41B	−177.3 (3)
C5B—N1B—C11B—N16B	6.9 (3)	C31B—C3B—C4B—C5B	179.4 (2)
N2B—N1B—C5B—C4B	0.4 (3)	C31B—C3B—C4B—C41B	2.5 (5)
C5B—N1B—N2B—C3B	−0.7 (3)	N2B—C3B—C31B—C32B	12.4 (3)
C11B—N1B—N2B—C3B	179.7 (2)	N2B—C3B—C31B—C36B	−165.9 (2)
C11B—N1B—C5B—C4B	180.0 (2)	C4B—C3B—C31B—C32B	−167.4 (2)
N2B—N1B—C11B—N12B	7.1 (3)	C4B—C3B—C31B—C36B	14.3 (4)
N1B—N2B—C3B—C4B	0.7 (3)	C3B—C4B—C5B—N1B	0.1 (2)
N1B—N2B—C3B—C31B	−179.19 (19)	C41B—C4B—C5B—N1B	177.6 (2)
C13B—N12B—C11B—N1B	−179.5 (2)	C3B—C4B—C41B—O41B	1.2 (5)
C13B—N12B—C11B—N16B	1.3 (4)	C5B—C4B—C41B—O41B	−175.4 (3)
C11B—N12B—C13B—C44B	−177.3 (2)	N12B—C13B—C14B—C15B	−2.5 (4)
C11B—N12B—C13B—C14B	0.7 (4)	C44B—C13B—C14B—C15B	175.3 (3)
C15B—N16B—C11B—N12B	−1.3 (4)	C13B—C14B—C15B—N16B	2.5 (3)
C15B—N16B—C11B—N1B	179.5 (2)	C13B—C14B—C15B—C46B	−176.9 (2)
C11B—N16B—C15B—C14B	−0.8 (3)	C3B—C31B—C32B—C33B	−179.7 (2)
C11B—N16B—C15B—C46B	178.7 (2)	C36B—C31B—C32B—C33B	−1.3 (4)
C31A—C3A—C4A—C5A	−178.3 (2)	C3B—C31B—C36B—C35B	179.1 (2)
N2A—C3A—C4A—C5A	0.0 (3)	C32B—C31B—C36B—C35B	0.8 (4)
N2A—C3A—C4A—C41A	−179.0 (3)	C31B—C32B—C33B—C34B	0.5 (4)
N2A—C3A—C31A—C36A	173.2 (2)	C32B—C33B—C34B—Cl2	−178.1 (2)
C4A—C3A—C31A—C32A	171.7 (2)	C32B—C33B—C34B—C35B	0.9 (4)
C4A—C3A—C31A—C36A	−8.7 (4)	Cl2—C34B—C35B—C36B	177.6 (2)
N2A—C3A—C31A—C32A	−6.4 (3)	C33B—C34B—C35B—C36B	−1.4 (4)
C31A—C3A—C4A—C41A	2.8 (5)	C34B—C35B—C36B—C31B	0.5 (4)

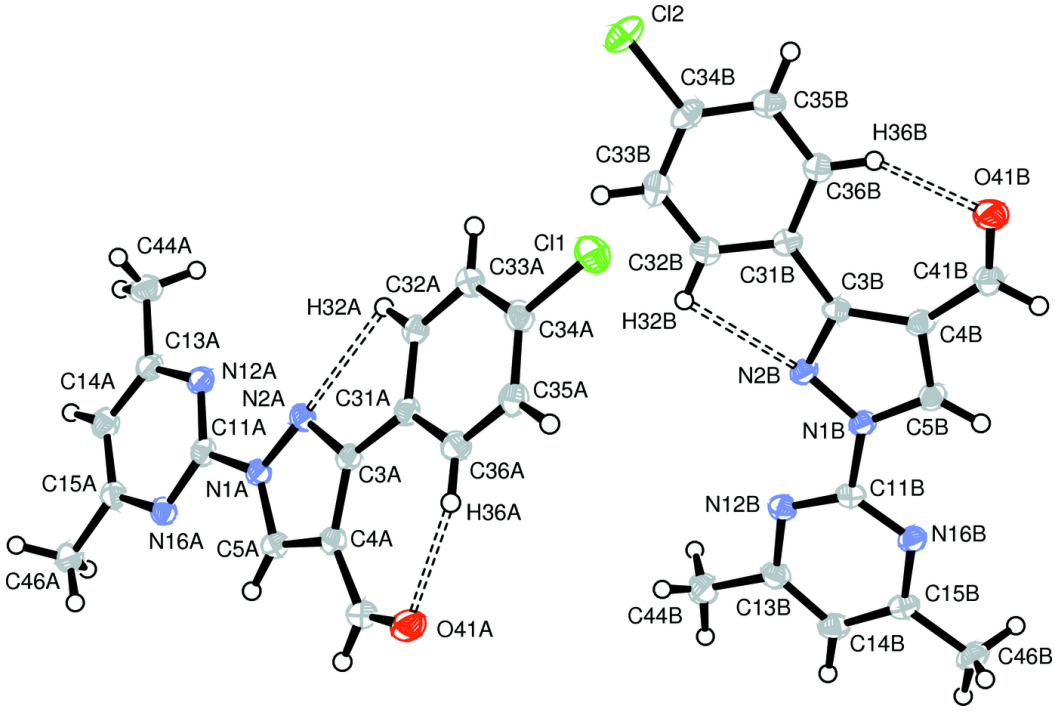
Symmetry codes: (i) *x*+1, *y*, *z*; (ii) −*x*+1, −*y*+1, −*z*; (iii) −*x*+1, −*y*+2, −*z*; (iv) *x*−1, *y*, *z*−1; (v) −*x*, −*y*+2, −*z*; (vi) −*x*+1, −*y*+1, −*z*+1; (vii) −*x*+1, −*y*+2, −*z*+1; (viii) *x*−1, *y*+1, *z*−1; (ix) −*x*+2, −*y*+1, −*z*+1; (x) *x*, *y*−1, *z*; (xi) *x*−1, *y*, *z*; (xii) *x*+1, *y*−1, *z*+1; (xiii) *x*, *y*+1, *z*; (xiv) *x*+1, *y*, *z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C32A—H32A···N2A	0.9500	2.4300	2.770 (3)	101.00

C32B—H32B···N2B	0.9500	2.4700	2.800 (3)	100.00
C36A—H36A···O41A	0.9500	2.2100	3.076 (3)	152.00
C36B—H36B···O41B	0.9500	2.2300	3.054 (3)	144.00

Fig. 1


Cl2
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C35B
H36B
C33B
C36B
O41B
C32B
C31B
C3B
C41B
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N2B
N1B
C5B
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C15A
N1A
H36A
N16A
C5A
C4A
C46A
O41A