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6-Chloro-N^4,N^4-dimethylpyrimidine-2,4-diamine

Yuan-Yuan Pang, Kai Yu, Bin Sun and Dian-Shun Guo*

Department of Chemistry, Shandong Normal University, Jinan 250014, People's Republic of China
Correspondence e-mail: chdsguo@sdnu.edu.cn

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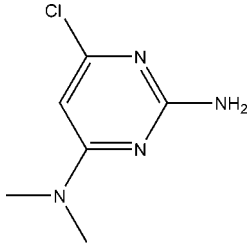
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.004 Å; R factor = 0.058; wR factor = 0.136; data-to-parameter ratio = 13.9.

The asymmetric unit of the title compound, $C_6H_9ClN_4$, contains four independent molecules (*A*, *B*, *C* and *D*). Their main difference is the torsion angles, ranging from 1.6 (5) to 5.9 (5)°, between the methyl group and the pyrimidine plane. A pair of intermolecular N—H···N hydrogen bonds link molecules *A* and *C* into a twisted dimer with a dihedral angle of 32.9 (1)° between the two pyrimidine rings, creating an $R_2^2(8)$ motif. In the packing, each two molecules of *B*, *C* and *D* form centrosymmetric dimers through two intermolecular N—H···N hydrogen bonds, locally creating $R_2^2(8)$ motifs. The dimers of *C* and *D* are alternately bridged by *A* into an infinite zigzag strip, locally creating two different $R_2^2(8)$ motifs with dihedral angles of 32.9 (1) and 63.4 (1)° between the pyrimidine rings. Finally, these strips together with the dimers of *B* associate into a complicated three-dimensional framework.

Related literature

For background to pyrimidine derivatives, see: Ligthart *et al.* (2005); Rabie *et al.* (2007); Goswami *et al.*(2008); Sherrington & Taskinen (2001). For similar structures, see: Cetina *et al.* (2005); Fun *et al.* (2006); Li *et al.* (2008); Ebenezer & Muthiah (2010); Cheng *et al.* (2011). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_6H_9ClN_4$
M_r = 172.62
Triclinic, $P\overline{1}$
a = 7.9438 (19) Å
b = 14.225 (3) Å
c = 14.895 (3) Å
α = 100.114 (3)°
β = 94.421 (4)°
γ = 100.524 (4)°
V = 1618.7 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.41 mm^{-1}
T = 298 K
0.21 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.919, T_{max} = 0.937
8239 measured reflections
5619 independent reflections
3769 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.136
S = 0.97
5619 reflections
405 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N11—H11*B*···N1	0.87	2.48	3.310 (4)	161
N3—H3*A*···N9	0.86	2.41	3.264 (4)	168
N16—H16*B*···N15^{i}	0.87	2.17	3.038 (4)	174
N11—H11*A*···N10^{ii}	0.87	2.21	3.086 (4)	174
N7—H7*B*···N5^{iii}	0.88	2.19	3.064 (3)	178
N3—H3*B*···N6^{iv}	0.89	2.35	3.230 (4)	169
N7—H7*A*···N2^{iv}	0.87	2.33	3.136 (3)	155

Symmetry codes: (i) $-x+2, -y+2, -z+2$; (ii) $-x, -y, -z+1$; (iii) $-x+1, -y+1, -z$; (iv) $-x+1, -y+1, -z+1$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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: ZQ2159).

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supplementary materials

Acta Cryst. (2012). E**68**, o1294–o1295 [doi:10.1107/S1600536812013517]

6-Chloro-N^4,N^4-dimethylpyrimidine-2,4-diamine

Yuan-Yuan Pang, Kai Yu, Bin Sun and Dian-Shun Guo

Comment

Functionalized pyrimidines play a major role in molecular recognition and supramolecular chemistry because of their potential ability to form stable hydrogen-bonded chains *via* their stereochemically associated amino groups and annular N atoms (Sherrington *et al.*, 2001; Ligthart *et al.*, 2005; Rabie *et al.*, 2007; Goswami *et al.*, 2008; Cheng *et al.*, 2011). Lots of substituted 2-aminopyrimidine compounds have been elucidated (Fun *et al.*, 2006; Li *et al.*, 2008; Ebenezer *et al.*, 2010; Cheng *et al.*, 2011). We report here the crystal structure of a related organic compound, 6-chloro-N^4,N^4-dimethylpyrimidine-2,4-diamine.

The title compound, $C_6H_9ClN_4$, contains four crystallographically independent molecules (Fig. 1), A, B, C and D, in the asymmetric unit, among which A and C are connected into a twisted dimer by two intermolecular N3—H3*A*···N9 and N11—H11*B*···N1 hydrogen bonds (Table 1), with a dihedral angle of 32.9 (1)° between the two pyrimidine rings. The bond lengths and angles of all molecules are similar except for the torsion angles between the methyl groups and the pyrimidine rings, ranging from 1.6 (5) to 5.9 (5)°.

In the packing of the title compound, each inversion-related two molecules of B, C and D form a centrosymmetric dimer through two intermolecular N—H···N hydrogen bonds (Table 1), locally creating an $R_2^2(8)$ motif (Bernstein *et al.*, 1995). For example, in the dimer of D (Fig. 2), hydrogen bonds arise from atoms N7—H7*B* at (*x*, *y*, *z*) and (-*x* + 1, -*y* + 1, -*z*), which act as hydrogen-bond donors, respectively, to atoms N5 at (-*x* + 1, -*y* + 1, -*z*) and (*x*, *y*, *z*). The dimers of C and D are alternately arranged and bridged by the molecule A, creating an infinite zigzag strip (Cetina *et al.*, 2005) (Fig. 3). In such a strip, the amino group of each molecule acts as a dual donor in N—H···N hydrogen bonds, while the pyrimidine ring serves as a dual acceptor. Interestingly, two different $R_2^2(8)$ motifs are formed with dihedral angles of 32.9 (1) and 63.4 (1)° between two adjacent pyrimidine rings. Finally, these strips together with the dimers of B are packed into a complicated three dimensional framework.

Experimental

2-Amino-4,6-dichloropyrimidine (0.082 g, 0.5 mmol) and K_2CO_3 (0.276 g, 2 mmol) were dissolved in DMF (2 ml) and H_2O (2 ml), the mixture was heated at 343 K for 0.5 h and then cooled to room temperature. The resulting mixture was extracted with ethyl acetate. The organic layer was separated and washed with brine, then dried over anhydrous $MgSO_4$. Removal of the solvent under reduced pressure gave the title compound as a yellow solid (yield 85%). Single crystals suitable for X-ray diffraction analysis were obtained by liquid–liquid diffusion from hexane and CH_2Cl_2 at 298 K.

Refinement

All non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atoms attached to anisotropically refined atoms were placed in geometrically idealized positions and included as riding atoms with C—H = 0.93 Å and U_{iso}(H) = 1.2 U_{eq}(C) (aromatic); C—H = 0.96 Å and U_{iso}(H) = 1.5 U_{eq}(C) (methyl); N—H = 0.87–0.88 Å and

U_{iso}(H) = 1.2U_{eq}(N).

Computing details

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT* (Bruker, 1999); 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* (Sheldrick, 2008).

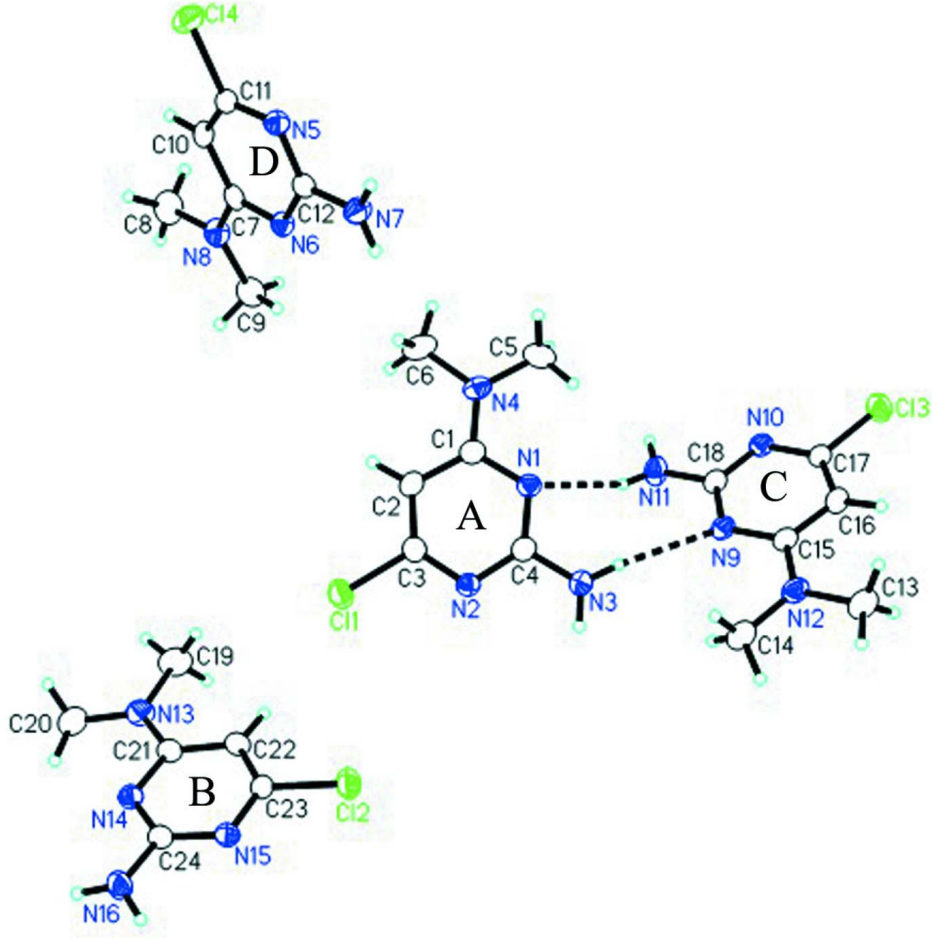

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. Dashed lines indicate hydrogen bonds.

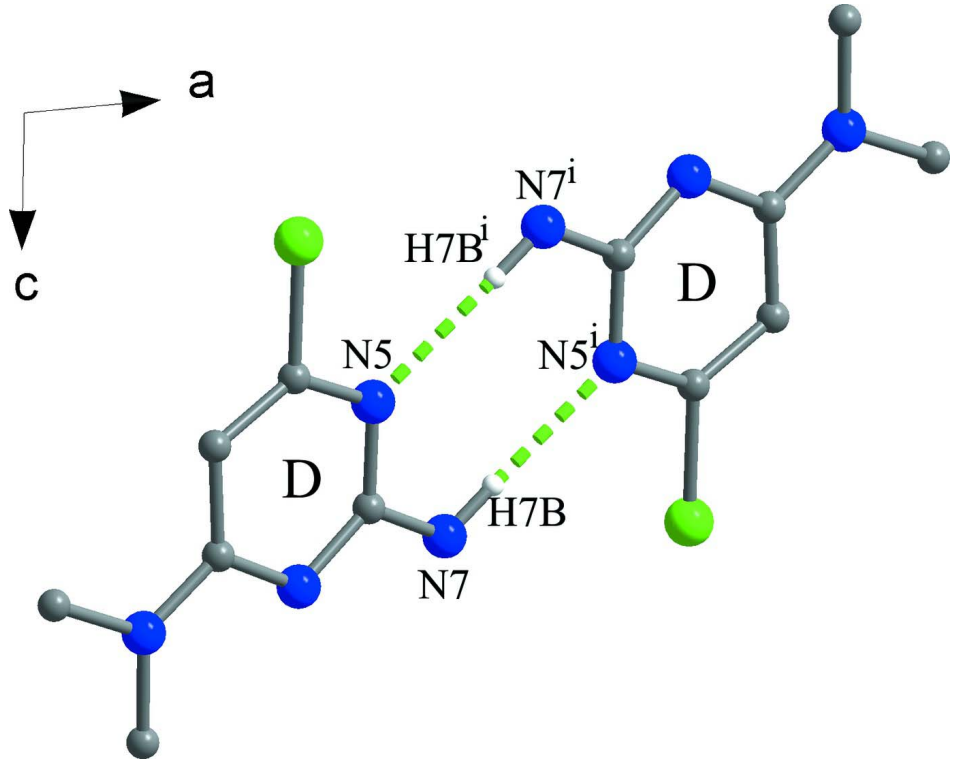

Figure 2

A centrosymmetric dimer of the D molecule, viewed along the crystallographic *b* axis, showing the $R_2^2(8)$ motif. For the sake of clarity, H atoms not involved in the motif have been omitted [symmetry code: (i) -*x* + 1, -*y* + 1, -*z*].

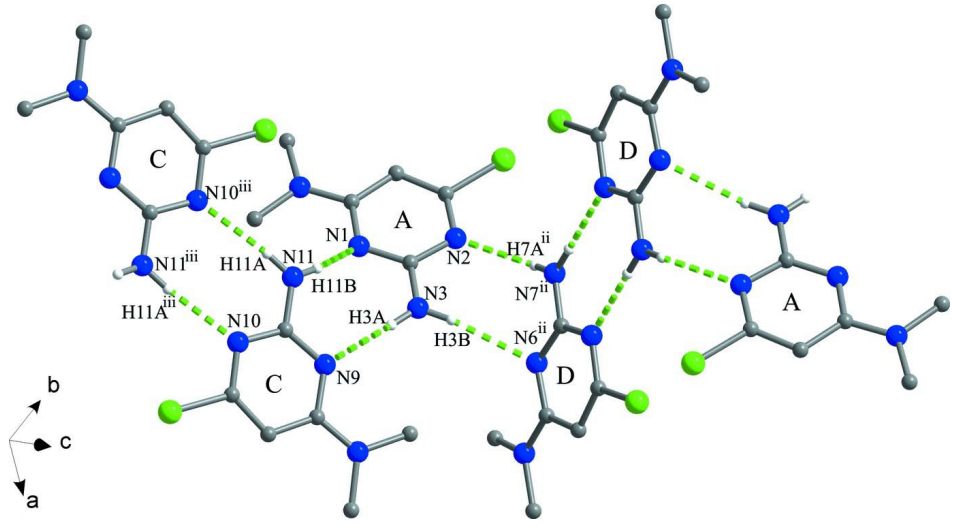

Figure 3

An infinite zigzag strip formed by a combination of A, C and D through hydrogen bonds, showing different $R_2^2(8)$ motifs. For the sake of clarity, H atoms not involved in the motifs have been omitted [symmetry codes: (ii) -*x* + 1, -*y* + 1, -*z* + 1; (iii) -*x*, -*y*, -*z* + 1].

6-Chloro-N^4,N^4-dimethylpyrimidine-2,4-diamine

Crystal data

$C_6H_9ClN_4$
M_r = 172.62
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 7.9438 (19) Å
b = 14.225 (3) Å
c = 14.895 (3) Å
α = 100.114 (3)°
β = 94.421 (4)°
γ = 100.524 (4)°
V = 1618.7 (7) Å^3
Z = 8
$F(000)$ = 720
D_x = 1.417 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2311 reflections
θ = 2.6–27.7°
μ = 0.41 mm^{-1}
T = 298 K
Block, colourless
0.21 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.919, T_{max} = 0.937
8239 measured reflections
5619 independent reflections
3769 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 25.0°, θ_{min} = 1.5°
h = −9→8
k = −16→16
l = −17→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.136
S = 0.97
5619 reflections
405 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
$w = 1/[\sigma^2(F_o^2) + (0.0681P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.3407 (3)	0.34115 (18)	0.54662 (16)	0.0383 (6)
N2	0.4648 (3)	0.47960 (18)	0.66542 (16)	0.0369 (6)
N3	0.5150 (4)	0.32771 (19)	0.67110 (18)	0.0534 (8)
N4	0.1803 (4)	0.3573 (2)	0.41724 (17)	0.0498 (8)

N5	0.3131 (3)	0.56029 (17)	0.00737 (15)	0.0377 (6)
N6	0.2031 (3)	0.57494 (17)	0.15383 (16)	0.0356 (6)
N7	0.4281 (4)	0.49791 (19)	0.12364 (16)	0.0469 (8)
N8	−0.0361 (4)	0.64285 (19)	0.18002 (18)	0.0451 (7)
N9	0.4193 (3)	0.08990 (18)	0.62696 (16)	0.0379 (6)
N10	0.1931 (3)	−0.03793 (18)	0.54295 (16)	0.0390 (7)
N11	0.1596 (4)	0.12009 (19)	0.5723 (2)	0.0542 (8)
N12	0.6743 (4)	0.0572 (2)	0.68555 (18)	0.0478 (7)
N13	0.3235 (4)	0.9764 (2)	0.83692 (19)	0.0511 (8)
N14	0.5994 (4)	1.03959 (18)	0.90517 (17)	0.0405 (7)
N15	0.8008 (3)	0.93718 (19)	0.93227 (17)	0.0412 (7)
N16	0.8765 (4)	1.10315 (19)	0.9711 (2)	0.0567 (8)
C1	0.2689 (4)	0.3996 (2)	0.4992 (2)	0.0387 (8)
C2	0.2883 (4)	0.4998 (2)	0.5353 (2)	0.0405 (8)
H2	0.2382	0.5412	0.5047	0.049*
C3	0.3852 (4)	0.5320 (2)	0.6179 (2)	0.0370 (8)
C4	0.4379 (4)	0.3849 (2)	0.6250 (2)	0.0399 (8)
C5	0.1705 (5)	0.2558 (3)	0.3791 (2)	0.0619 (11)
H5A	0.2531	0.2309	0.4139	0.093*
H5B	0.1951	0.2490	0.3164	0.093*
H5C	0.0568	0.2198	0.3815	0.093*
C6	0.1034 (5)	0.4146 (3)	0.3586 (2)	0.0667 (11)
H6A	0.0397	0.4559	0.3942	0.100*
H6B	0.0272	0.3715	0.3092	0.100*
H6C	0.1929	0.4541	0.3340	0.100*
C7	0.0773 (4)	0.6171 (2)	0.1222 (2)	0.0362 (8)
C8	−0.1760 (5)	0.6879 (3)	0.1505 (3)	0.0643 (11)
H8A	−0.1297	0.7441	0.1262	0.096*
H8B	−0.2361	0.7072	0.2020	0.096*
H8C	−0.2544	0.6420	0.1039	0.096*
C9	−0.0343 (5)	0.6179 (3)	0.2706 (2)	0.0570 (10)
H9A	−0.1284	0.5649	0.2705	0.086*
H9B	−0.0458	0.6735	0.3150	0.086*
H9C	0.0725	0.5990	0.2861	0.086*
C10	0.0687 (4)	0.6347 (2)	0.0317 (2)	0.0376 (8)
H10	−0.0150	0.6648	0.0084	0.045*
C11	0.1901 (4)	0.6050 (2)	−0.01859 (19)	0.0363 (8)
C12	0.3096 (4)	0.5455 (2)	0.09442 (19)	0.0348 (7)
C13	0.7969 (5)	−0.0067 (3)	0.6925 (2)	0.0623 (11)
H13A	0.7474	−0.0597	0.7204	0.093*
H13B	0.9006	0.0295	0.7293	0.093*
H13C	0.8232	−0.0321	0.6322	0.093*
C14	0.7279 (5)	0.1570 (2)	0.7337 (3)	0.0610 (11)
H14A	0.7736	0.1970	0.6921	0.092*
H14B	0.8149	0.1607	0.7833	0.092*
H14C	0.6306	0.1797	0.7577	0.092*
C15	0.5186 (4)	0.0239 (2)	0.63617 (19)	0.0375 (8)
C16	0.4614 (4)	−0.0744 (2)	0.5956 (2)	0.0418 (8)
H16	0.5303	−0.1203	0.5987	0.050*

C17	0.2997 (4)	−0.0987 (2)	0.55161 (19)	0.0367 (8)
C18	0.2619 (4)	0.0556 (2)	0.5810 (2)	0.0380 (8)
C19	0.1863 (5)	0.8969 (3)	0.7904 (3)	0.0694 (12)
H19A	0.2290	0.8584	0.7410	0.104*
H19B	0.0928	0.9228	0.7663	0.104*
H19C	0.1462	0.8569	0.8330	0.104*
C20	0.2873 (5)	1.0738 (3)	0.8461 (2)	0.0594 (11)
H20A	0.3797	1.1194	0.8848	0.089*
H20B	0.1816	1.0755	0.8730	0.089*
H20C	0.2765	1.0909	0.7867	0.089*
C21	0.4788 (4)	0.9608 (2)	0.8677 (2)	0.0394 (8)
C22	0.5135 (4)	0.8663 (2)	0.8614 (2)	0.0447 (9)
H22	0.4315	0.8106	0.8359	0.054*
C23	0.6750 (5)	0.8624 (2)	0.8952 (2)	0.0419 (8)
C24	0.7528 (4)	1.0241 (2)	0.9355 (2)	0.0396 (8)
Cl1	0.41068 (13)	0.65460 (6)	0.66924 (6)	0.0559 (3)
Cl2	0.72990 (15)	0.74846 (7)	0.89197 (7)	0.0725 (3)
Cl3	0.21621 (13)	−0.21962 (6)	0.49962 (6)	0.0594 (3)
Cl4	0.19291 (13)	0.62621 (7)	−0.13080 (5)	0.0581 (3)
H7A	0.4312	0.4882	0.1794	0.070*
H11B	0.2061	0.1818	0.5803	0.070*
H16A	0.8516	1.1614	0.9822	0.070*
H3A	0.4991	0.2653	0.6522	0.070*
H7B	0.5017	0.4822	0.0852	0.070*
H16B	0.9661	1.0935	1.0027	0.070*
H3B	0.5805	0.3546	0.7235	0.070*
H11A	0.0579	0.1009	0.5408	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0343 (17)	0.0405 (16)	0.0382 (14)	0.0061 (13)	−0.0005 (12)	0.0055 (12)
N2	0.0337 (17)	0.0400 (16)	0.0369 (14)	0.0079 (13)	0.0031 (12)	0.0067 (12)
N3	0.066 (2)	0.0450 (17)	0.0496 (17)	0.0224 (16)	−0.0095 (15)	0.0045 (13)
N4	0.047 (2)	0.060 (2)	0.0405 (16)	0.0082 (15)	−0.0063 (14)	0.0101 (14)
N5	0.0362 (17)	0.0406 (16)	0.0368 (15)	0.0086 (13)	0.0070 (12)	0.0070 (11)
N6	0.0336 (17)	0.0365 (15)	0.0388 (14)	0.0103 (13)	0.0078 (12)	0.0078 (11)
N7	0.050 (2)	0.0593 (19)	0.0398 (15)	0.0284 (16)	0.0106 (13)	0.0122 (13)
N8	0.0385 (18)	0.0476 (17)	0.0540 (17)	0.0158 (14)	0.0164 (14)	0.0101 (13)
N9	0.0298 (16)	0.0432 (16)	0.0411 (15)	0.0069 (13)	−0.0009 (12)	0.0115 (12)
N10	0.0314 (17)	0.0422 (17)	0.0415 (15)	0.0040 (14)	−0.0016 (12)	0.0093 (12)
N11	0.0368 (19)	0.0418 (17)	0.081 (2)	0.0086 (14)	−0.0111 (16)	0.0110 (14)
N12	0.0370 (18)	0.0517 (19)	0.0523 (17)	0.0066 (15)	−0.0087 (14)	0.0122 (14)
N13	0.0315 (18)	0.059 (2)	0.0599 (18)	0.0097 (15)	−0.0050 (14)	0.0063 (14)
N14	0.0359 (18)	0.0409 (16)	0.0439 (15)	0.0071 (14)	0.0011 (13)	0.0086 (12)
N15	0.0354 (17)	0.0395 (17)	0.0452 (15)	0.0040 (14)	−0.0049 (13)	0.0062 (12)
N16	0.048 (2)	0.0376 (17)	0.077 (2)	0.0010 (15)	−0.0156 (16)	0.0083 (14)
C1	0.0256 (19)	0.049 (2)	0.0412 (18)	0.0038 (16)	0.0070 (15)	0.0107 (15)
C2	0.033 (2)	0.050 (2)	0.0437 (19)	0.0134 (16)	0.0066 (15)	0.0154 (15)
C3	0.032 (2)	0.0403 (19)	0.0410 (18)	0.0072 (16)	0.0159 (15)	0.0094 (14)

C4	0.033 (2)	0.046 (2)	0.0418 (18)	0.0081 (16)	0.0063 (15)	0.0116 (16)
C5	0.061 (3)	0.060 (3)	0.050 (2)	−0.008 (2)	−0.0051 (19)	−0.0023 (17)
C6	0.054 (3)	0.095 (3)	0.051 (2)	0.019 (2)	−0.0105 (19)	0.017 (2)
C7	0.034 (2)	0.0230 (17)	0.0479 (18)	0.0003 (15)	0.0042 (15)	0.0028 (13)
C8	0.047 (3)	0.069 (3)	0.086 (3)	0.029 (2)	0.020 (2)	0.015 (2)
C9	0.057 (3)	0.059 (2)	0.058 (2)	0.010 (2)	0.0266 (19)	0.0089 (17)
C10	0.035 (2)	0.0297 (18)	0.0488 (19)	0.0062 (15)	0.0008 (16)	0.0107 (14)
C11	0.039 (2)	0.0290 (17)	0.0374 (17)	−0.0008 (15)	0.0005 (15)	0.0074 (13)
C12	0.033 (2)	0.0321 (18)	0.0383 (17)	0.0067 (15)	0.0053 (15)	0.0040 (13)
C13	0.041 (2)	0.076 (3)	0.071 (3)	0.018 (2)	−0.009 (2)	0.017 (2)
C14	0.050 (3)	0.053 (2)	0.073 (3)	−0.0062 (19)	−0.019 (2)	0.0236 (19)
C15	0.031 (2)	0.047 (2)	0.0357 (17)	0.0060 (16)	0.0044 (15)	0.0143 (14)
C16	0.033 (2)	0.048 (2)	0.0485 (19)	0.0157 (17)	0.0056 (16)	0.0128 (16)
C17	0.034 (2)	0.0422 (19)	0.0354 (17)	0.0076 (16)	0.0033 (15)	0.0108 (14)
C18	0.030 (2)	0.046 (2)	0.0405 (17)	0.0097 (17)	0.0055 (15)	0.0134 (15)
C19	0.038 (2)	0.088 (3)	0.076 (3)	0.006 (2)	−0.010 (2)	0.014 (2)
C20	0.052 (3)	0.074 (3)	0.061 (2)	0.032 (2)	0.0055 (19)	0.0148 (19)
C21	0.035 (2)	0.043 (2)	0.0380 (17)	0.0033 (17)	0.0050 (15)	0.0064 (14)
C22	0.039 (2)	0.042 (2)	0.0471 (19)	−0.0002 (17)	−0.0058 (16)	0.0035 (15)
C23	0.046 (2)	0.037 (2)	0.0430 (18)	0.0104 (17)	−0.0022 (17)	0.0085 (14)
C24	0.038 (2)	0.039 (2)	0.0386 (17)	0.0002 (17)	0.0019 (15)	0.0080 (14)
Cl1	0.0686 (7)	0.0406 (5)	0.0582 (5)	0.0122 (5)	0.0097 (5)	0.0062 (4)
Cl2	0.0785 (8)	0.0406 (6)	0.0913 (7)	0.0163 (5)	−0.0270 (6)	0.0051 (5)
Cl3	0.0610 (7)	0.0407 (5)	0.0700 (6)	0.0063 (5)	−0.0074 (5)	0.0037 (4)
Cl4	0.0674 (7)	0.0670 (6)	0.0436 (5)	0.0108 (5)	0.0059 (4)	0.0233 (4)

Geometric parameters (Å, º)

N1—C4	1.333 (4)	C1—C2	1.409 (4)
N1—C1	1.353 (4)	C2—C3	1.357 (4)
N2—C3	1.321 (4)	C2—H2	0.9300
N2—C4	1.347 (4)	C3—Cl1	1.746 (3)
N3—C4	1.350 (4)	C5—H5A	0.9600
N3—H3A	0.8649	C5—H5B	0.9600
N3—H3B	0.8870	C5—H5C	0.9600
N4—C1	1.343 (4)	C6—H6A	0.9600
N4—C5	1.441 (4)	C6—H6B	0.9600
N4—C6	1.467 (4)	C6—H6C	0.9600
N5—C11	1.329 (4)	C7—C10	1.411 (4)
N5—C12	1.351 (3)	C8—H8A	0.9600
N6—C12	1.338 (4)	C8—H8B	0.9600
N6—C7	1.351 (4)	C8—H8C	0.9600
N7—C12	1.344 (4)	C9—H9A	0.9600
N7—H7A	0.8654	C9—H9B	0.9600
N7—H7B	0.8789	C9—H9C	0.9600
N8—C7	1.348 (4)	C10—C11	1.349 (4)
N8—C9	1.453 (4)	C10—H10	0.9300
N8—C8	1.456 (4)	C11—Cl4	1.751 (3)
N9—C18	1.341 (4)	C13—H13A	0.9600
N9—C15	1.350 (4)	C13—H13B	0.9600

N10—C17	1.330 (4)	C13—H13C	0.9600
N10—C18	1.346 (4)	C14—H14A	0.9600
N11—C18	1.348 (4)	C14—H14B	0.9600
N11—H11B	0.8716	C14—H14C	0.9600
N11—H11A	0.8741	C15—C16	1.399 (4)
N12—C15	1.349 (4)	C16—C17	1.352 (4)
N12—C14	1.444 (4)	C16—H16	0.9300
N12—C13	1.458 (4)	C17—Cl3	1.742 (3)
N13—C21	1.353 (4)	C19—H19A	0.9600
N13—C20	1.450 (4)	C19—H19B	0.9600
N13—C19	1.452 (5)	C19—H19C	0.9600
N14—C24	1.337 (4)	C20—H20A	0.9600
N14—C21	1.338 (4)	C20—H20B	0.9600
N15—C23	1.325 (4)	C20—H20C	0.9600
N15—C24	1.353 (4)	C21—C22	1.409 (4)
N16—C24	1.348 (4)	C22—C23	1.356 (4)
N16—H16A	0.8773	C22—H22	0.9300
N16—H16B	0.8682	C23—Cl2	1.747 (3)

C4—N1—C1	116.3 (3)	H8B—C8—H8C	109.5
C3—N2—C4	113.1 (3)	N8—C9—H9A	109.5
C4—N3—H3A	122.0	N8—C9—H9B	109.5
C4—N3—H3B	118.9	H9A—C9—H9B	109.5
H3A—N3—H3B	119.1	N8—C9—H9C	109.5
C1—N4—C5	121.6 (3)	H9A—C9—H9C	109.5
C1—N4—C6	121.2 (3)	H9B—C9—H9C	109.5
C5—N4—C6	116.9 (3)	C11—C10—C7	115.4 (3)
C11—N5—C12	113.1 (3)	C11—C10—H10	122.3
C12—N6—C7	117.2 (2)	C7—C10—H10	122.3
C12—N7—H7A	118.4	N5—C11—C10	127.1 (3)
C12—N7—H7B	116.9	N5—C11—Cl4	114.1 (2)
H7A—N7—H7B	124.6	C10—C11—Cl4	118.8 (2)
C7—N8—C9	121.4 (3)	N6—C12—N7	117.7 (3)
C7—N8—C8	121.0 (3)	N6—C12—N5	126.5 (3)
C9—N8—C8	117.3 (3)	N7—C12—N5	115.8 (3)
C18—N9—C15	116.7 (3)	N12—C13—H13A	109.5
C17—N10—C18	113.7 (3)	N12—C13—H13B	109.5
C18—N11—H11B	119.1	H13A—C13—H13B	109.5
C18—N11—H11A	120.3	N12—C13—H13C	109.5
H11B—N11—H11A	117.1	H13A—C13—H13C	109.5
C15—N12—C14	122.1 (3)	H13B—C13—H13C	109.5
C15—N12—C13	121.0 (3)	N12—C14—H14A	109.5
C14—N12—C13	116.9 (3)	N12—C14—H14B	109.5
C21—N13—C20	121.8 (3)	H14A—C14—H14B	109.5
C21—N13—C19	121.7 (3)	N12—C14—H14C	109.5
C20—N13—C19	116.5 (3)	H14A—C14—H14C	109.5
C24—N14—C21	116.9 (3)	H14B—C14—H14C	109.5
C23—N15—C24	112.7 (3)	N12—C15—N9	117.0 (3)
C24—N16—H16A	120.6	N12—C15—C16	122.0 (3)

C24—N16—H16B	116.8	N9—C15—C16	120.9 (3)
H16A—N16—H16B	117.8	C17—C16—C15	115.9 (3)
N4—C1—N1	116.8 (3)	C17—C16—H16	122.1
N4—C1—C2	122.5 (3)	C15—C16—H16	122.1
N1—C1—C2	120.7 (3)	N10—C17—C16	126.1 (3)
C3—C2—C1	115.4 (3)	N10—C17—Cl3	114.6 (2)
C3—C2—H2	122.3	C16—C17—Cl3	119.3 (2)
C1—C2—H2	122.3	N9—C18—N10	126.6 (3)
N2—C3—C2	126.7 (3)	N9—C18—N11	117.6 (3)
N2—C3—Cl1	114.8 (2)	N10—C18—N11	115.8 (3)
C2—C3—Cl1	118.5 (2)	N13—C19—H19A	109.5
N1—C4—N2	127.6 (3)	N13—C19—H19B	109.5
N1—C4—N3	116.7 (3)	H19A—C19—H19B	109.5
N2—C4—N3	115.7 (3)	N13—C19—H19C	109.5
N4—C5—H5A	109.5	H19A—C19—H19C	109.5
N4—C5—H5B	109.5	H19B—C19—H19C	109.5
H5A—C5—H5B	109.5	N13—C20—H20A	109.5
N4—C5—H5C	109.5	N13—C20—H20B	109.5
H5A—C5—H5C	109.5	H20A—C20—H20B	109.5
H5B—C5—H5C	109.5	N13—C20—H20C	109.5
N4—C6—H6A	109.5	H20A—C20—H20C	109.5
N4—C6—H6B	109.5	H20B—C20—H20C	109.5
H6A—C6—H6B	109.5	N14—C21—N13	116.9 (3)
N4—C6—H6C	109.5	N14—C21—C22	121.0 (3)
H6A—C6—H6C	109.5	N13—C21—C22	122.1 (3)
H6B—C6—H6C	109.5	C23—C22—C21	115.3 (3)
N8—C7—N6	117.9 (3)	C23—C22—H22	122.4
N8—C7—C10	121.8 (3)	C21—C22—H22	122.4
N6—C7—C10	120.4 (3)	N15—C23—C22	126.9 (3)
N8—C8—H8A	109.5	N15—C23—Cl2	114.3 (2)
N8—C8—H8B	109.5	C22—C23—Cl2	118.8 (3)
H8A—C8—H8B	109.5	N14—C24—N16	117.3 (3)
N8—C8—H8C	109.5	N14—C24—N15	127.2 (3)
H8A—C8—H8C	109.5	N16—C24—N15	115.5 (3)

C5—N4—C1—N1	−3.6 (5)	C14—N12—C15—N9	5.5 (5)
C6—N4—C1—N1	−177.5 (3)	C13—N12—C15—N9	−174.5 (3)
C5—N4—C1—C2	176.3 (3)	C14—N12—C15—C16	−174.8 (3)
C6—N4—C1—C2	2.4 (5)	C13—N12—C15—C16	5.3 (5)
C4—N1—C1—N4	175.7 (3)	C18—N9—C15—N12	−177.3 (3)
C4—N1—C1—C2	−4.2 (4)	C18—N9—C15—C16	2.9 (4)
N4—C1—C2—C3	−178.4 (3)	N12—C15—C16—C17	177.1 (3)
N1—C1—C2—C3	1.4 (5)	N9—C15—C16—C17	−3.2 (5)
C4—N2—C3—C2	−1.8 (5)	C18—N10—C17—C16	1.8 (5)
C4—N2—C3—Cl1	178.1 (2)	C18—N10—C17—Cl3	−178.0 (2)
C1—C2—C3—N2	1.8 (5)	C15—C16—C17—N10	0.8 (5)
C1—C2—C3—Cl1	−178.2 (2)	C15—C16—C17—Cl3	−179.5 (2)
C1—N1—C4—N2	4.4 (5)	C15—N9—C18—N10	−0.2 (5)
C1—N1—C4—N3	−177.7 (3)	C15—N9—C18—N11	179.0 (3)

C3—N2—C4—N1	−1.5 (5)	C17—N10—C18—N9	−2.1 (5)
C3—N2—C4—N3	−179.4 (3)	C17—N10—C18—N11	178.7 (3)
C9—N8—C7—N6	−5.8 (5)	C24—N14—C21—N13	−180.0 (3)
C8—N8—C7—N6	−179.5 (3)	C24—N14—C21—C22	0.4 (4)
C9—N8—C7—C10	175.3 (3)	C20—N13—C21—N14	−1.5 (5)
C8—N8—C7—C10	1.6 (5)	C19—N13—C21—N14	176.5 (3)
C12—N6—C7—N8	176.8 (3)	C20—N13—C21—C22	178.1 (3)
C12—N6—C7—C10	−4.3 (4)	C19—N13—C21—C22	−3.9 (5)
N8—C7—C10—C11	−179.9 (3)	N14—C21—C22—C23	−0.3 (4)
N6—C7—C10—C11	1.2 (4)	N13—C21—C22—C23	−179.9 (3)
C12—N5—C11—C10	−0.3 (5)	C24—N15—C23—C22	1.0 (5)
C12—N5—C11—Cl4	179.4 (2)	C24—N15—C23—Cl2	−178.6 (2)
C7—C10—C11—N5	1.2 (5)	C21—C22—C23—N15	−0.4 (5)
C7—C10—C11—Cl4	−178.6 (2)	C21—C22—C23—Cl2	179.2 (2)
C7—N6—C12—N7	−175.9 (3)	C21—N14—C24—N16	178.2 (3)
C7—N6—C12—N5	5.5 (5)	C21—N14—C24—N15	0.3 (5)
C11—N5—C12—N6	−3.2 (5)	C23—N15—C24—N14	−1.0 (5)
C11—N5—C12—N7	178.2 (3)	C23—N15—C24—N16	−178.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N11—H11*B*···N1	0.87	2.48	3.310 (4)	161
N3—H3*A*···N9	0.86	2.41	3.264 (4)	168
N16—H16*B*···N15^{i}	0.87	2.17	3.038 (4)	174
N11—H11*A*···N10^{ii}	0.87	2.21	3.086 (4)	174
N7—H7*B*···N5^{iii}	0.88	2.19	3.064 (3)	178
N3—H3*B*···N6^{iv}	0.89	2.35	3.230 (4)	169
N7—H7*A*···N2^{iv}	0.87	2.33	3.136 (3)	155

Symmetry codes: (i) −*x*+2, −*y*+2, −*z*+2; (ii) −*x*, −*y*, −*z*+1; (iii) −*x*+1, −*y*+1, −*z*; (iv) −*x*+1, −*y*+1, −*z*+1.