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3-[(2*E*)-2-(Butan-2-ylidene)hydrazinyl]-6-chloropyridazine

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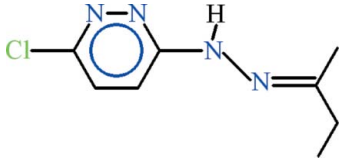
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.099; data-to-parameter ratio = 15.0.

The asymmetric unit of the title compound, $C_8H_{11}ClN_4$, contains two independent molecules (*A* and *B*) with slightly different conformations: the dihedral angles between the 3-chloro-6-hydrazinylpyridazine units and butyl side chains are 4.5 (2) and 11.98 (16)°. In the crystal, the *A* and *B* molecules are linked by a pair of N—H···N hydrogen bonds, generating an $R_2^2(8)$ loop.

Related literature

For related structures, see: Ather *et al.* (2009, 2010). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_{11}ClN_4$
M_r = 198.66
Triclinic, $P\overline{1}$
a = 8.0623 (4) Å
b = 11.6768 (5) Å
c = 12.1314 (5) Å
α = 113.858 (1)°
β = 91.370 (2)°
γ = 104.880 (2)°
V = 998.85 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.34 mm^{-1}
T = 296 K
0.25 × 0.15 × 0.14 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.982, T_{max} = 0.988
14983 measured reflections
3585 independent reflections
2652 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.099
S = 1.05
3585 reflections
239 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3*A*···N6^{i}	0.86	2.30	3.0674 (15)	148
N7—H7···N2^{i}	0.86	2.24	3.0689 (15)	161

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5623).

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supplementary materials

Acta Cryst. (2010). E**66**, o2499 [**doi:10.1107/S160053681003504X**]

3-[(2*E*)-2-(Butan-2-ylidene)hydrazinyl]-6-chloropyridazine

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Comment

In continuation of our studies of pyrazolylpyridazine derivatives (Ather *et al.*, 2009, 2010), the title compound (I, Fig. 1) is being reported here.

The title compound (I), consists of two independent molecules. In one molecule, the 3-chloro-6-hydrazinylpyridazine moiety A (C1—C4/N1—N4/CL1) and the butane group B (C5—C8) is planar with r. m. s. deviation of 0.0217 and 0.0130 Å. The dihedral angle between A/B is 4.53 (24)°. In second molecule, the 3-chloro-6-hydrazinylpyridazine moiety C (C9—C12/N5—N8/CL2) and the butane group D (C13—C16) is planar with r. m. s. deviation of 0.0453 and 0.0446 Å. The dihedral angle between C/D is 11.98 (16)°. The title compound consists of dimers due to N—H···N type of H-bonding (Table 1, Fig. 2) with $R_2^2(8)$ ring motif (Bernstein *et al.*, 1995).

Experimental

3-Chloro-6-hydrazinylpyridazine (0.5 g, 3.46 mmol), dissolved in ethyl-methylketone was refluxed for 30 min. The unreacted ethyl-methylketone was distilled off yielding the crude material. The product was re-crystallized in alcohol to affoard colorless needles of (I).

Refinement

The H-atoms were positioned geometrically (N–H = 0.86, C–H = 0.93–0.97 Å) and were included in the refinement in the riding model approximation, with U_{iso}(H) = xU_{eq}(C, N), where x = 1.5 for methyl and x = 1.2 for all other H-atoms.

Figures

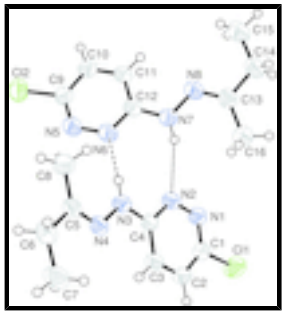


Fig. 1. Two independent molecules of (I) with 50% probability displacement ellipsoids. Dashed lines denote intermolecular hydrogen bonds, forming a dimer: the N6 moleucle shown is generated by the symmetry operation (1–x, 1–y, –z) from the asymmetric atoms.

3-[(2*E*)-2-(Butan-2-ylidene)hydrazinyl]-6-chloropyridazine

Crystal data

$C_8H_{11}ClN_4$	$Z = 4$
$M_r = 198.66$	$F(000) = 416$
Triclinic, $P\overline{1}$	$D_x = 1.321$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.0623$ (4) Å	Cell parameters from 2652 reflections
$b = 11.6768$ (5) Å	$\theta = 2.1$–$25.3°$
$c = 12.1314$ (5) Å	$\mu = 0.34$ mm^{-1}
$\alpha = 113.858$ (1)°	$T = 296$ K
$\beta = 91.370$ (2)°	Needle, colorless
$\gamma = 104.880$ (2)°	0.25 × 0.15 × 0.14 mm
$V = 998.85$ (8) Å^3	

Data collection

Bruker Kappa APEXII CCD diffractometer	3585 independent reflections
Radiation source: fine-focus sealed tube	2652 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.031$
Detector resolution: 8.10 pixels mm^{-1}	$\theta_{max} = 25.3°$, $\theta_{min} = 2.1°$
ω scans	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	$k = -13 \rightarrow 13$
$T_{min} = 0.982$, $T_{max} = 0.988$	$l = -14 \rightarrow 11$
14983 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.099$	H-atom parameters constrained
$S = 1.05$	$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2 + 0.1867P]$ where $P = (F_o^2 + 2F_c^2)/3$
3585 reflections	$(\Delta/\sigma)_{max} = 0.001$
239 parameters	$\Delta\rho_{max} = 0.15$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.20$ e Å^{-3}

Special details

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 $F^2 > \sigma(F^2)$ 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	0.56878 (7)	1.01144 (5)	0.14461 (4)	0.0711 (2)
N1	0.65078 (7)	0.81818 (4)	−0.01826 (4)	0.0494 (5)
N2	0.69138 (9)	0.70411 (6)	−0.06328 (4)	0.0477 (5)
N3	0.73568 (11)	0.52631 (8)	−0.04789 (4)	0.0485 (5)
N4	0.75811 (18)	0.46292 (14)	0.02371 (12)	0.0446 (5)
C1	0.6218 (2)	0.86549 (17)	0.09480 (15)	0.0457 (6)
C2	0.6309 (2)	0.80724 (18)	0.17388 (15)	0.0482 (6)
C3	0.6692 (2)	0.69257 (17)	0.12939 (14)	0.0456 (6)
C4	0.6972 (2)	0.64155 (16)	0.00695 (14)	0.0403 (6)
C5	0.8018 (2)	0.35764 (18)	−0.02463 (16)	0.0446 (6)
C6	0.8294 (3)	0.29374 (19)	0.05683 (17)	0.0546 (7)
C7	0.8037 (3)	0.3631 (2)	0.18693 (18)	0.0706 (9)
C8	0.8324 (3)	0.29472 (19)	−0.15401 (16)	0.0571 (7)
Cl2	0.61890 (8)	0.92216 (5)	0.55956 (5)	0.0741 (2)
N5	0.4684 (2)	0.70148 (16)	0.37821 (14)	0.0577 (6)
N6	0.3743 (2)	0.57428 (16)	0.32230 (13)	0.0577 (6)
N7	0.2290 (2)	0.38112 (15)	0.32596 (13)	0.0557 (6)
N8	0.1391 (2)	0.31693 (16)	0.38987 (13)	0.0527 (6)
C9	0.4960 (2)	0.75931 (18)	0.49629 (16)	0.0499 (6)
C10	0.4326 (3)	0.69965 (19)	0.57130 (16)	0.0572 (7)
C11	0.3386 (3)	0.57264 (19)	0.51703 (16)	0.0560 (7)
C12	0.3135 (2)	0.51037 (18)	0.38917 (15)	0.0461 (6)
C13	0.0646 (3)	0.19453 (19)	0.33475 (16)	0.0514 (7)
C14	−0.0380 (3)	0.1305 (2)	0.40720 (18)	0.0656 (8)
C15	−0.0180 (3)	0.2152 (2)	0.5419 (2)	0.0842 (10)
C16	0.06718 (9)	0.10973 (7)	0.20380 (5)	0.0690 (8)
H2	0.61145	0.84592	0.25415	0.0578*
H3	0.67676	0.64881	0.17770	0.0547*
H3A	0.74569	0.49409	−0.12405	0.0582*
H6A	0.75051	0.20566	0.02287	0.0655*
H6B	0.94655	0.28638	0.05598	0.0655*
H7A	0.88583	0.44865	0.22347	0.1059*
H7B	0.68809	0.37098	0.18942	0.1059*
H7C	0.82074	0.31382	0.23104	0.1059*

H8A	0.72987	0.27583	−0.20780	0.0858*
H8B	0.92751	0.35314	−0.16869	0.0858*
H8C	0.85936	0.21485	−0.16845	0.0858*
H7	0.23145	0.34080	0.24923	0.0668*
H10	0.45400	0.74553	0.65567	0.0687*
H11	0.29214	0.52778	0.56265	0.0673*
H14A	−0.00369	0.05316	0.39609	0.0787*
H14B	−0.15974	0.10162	0.37404	0.0787*
H15A	0.10043	0.23887	0.57731	0.1264*
H15B	−0.09207	0.16776	0.57958	0.1264*
H15C	−0.04957	0.29300	0.55442	0.1264*
H16A	−0.00022	0.13112	0.15265	0.1035*
H16B	0.01880	0.01944	0.18790	0.1035*
H16C	0.18459	0.12428	0.18693	0.1035*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1018 (5)	0.0565 (3)	0.0605 (3)	0.0378 (3)	0.0197 (3)	0.0210 (3)
N1	0.0617 (10)	0.0490 (9)	0.0437 (8)	0.0192 (8)	0.0131 (7)	0.0235 (7)
N2	0.0627 (10)	0.0477 (9)	0.0393 (8)	0.0201 (8)	0.0144 (7)	0.0222 (7)
N3	0.0670 (10)	0.0479 (9)	0.0362 (7)	0.0190 (8)	0.0112 (7)	0.0219 (7)
N4	0.0485 (9)	0.0478 (9)	0.0427 (8)	0.0109 (7)	0.0063 (7)	0.0261 (7)
C1	0.0480 (11)	0.0428 (10)	0.0437 (10)	0.0106 (8)	0.0075 (8)	0.0173 (8)
C2	0.0534 (11)	0.0505 (11)	0.0360 (9)	0.0100 (9)	0.0095 (8)	0.0167 (8)
C3	0.0521 (11)	0.0494 (11)	0.0367 (9)	0.0091 (9)	0.0078 (8)	0.0230 (8)
C4	0.0404 (10)	0.0420 (10)	0.0373 (9)	0.0066 (8)	0.0055 (7)	0.0188 (8)
C5	0.0414 (10)	0.0454 (11)	0.0467 (10)	0.0070 (8)	0.0060 (8)	0.0228 (9)
C6	0.0529 (12)	0.0582 (12)	0.0621 (12)	0.0144 (10)	0.0074 (9)	0.0358 (10)
C7	0.0867 (16)	0.0831 (16)	0.0590 (12)	0.0271 (13)	0.0127 (11)	0.0454 (12)
C8	0.0663 (13)	0.0581 (12)	0.0506 (11)	0.0227 (10)	0.0131 (9)	0.0236 (10)
Cl2	0.0911 (4)	0.0514 (3)	0.0707 (4)	0.0066 (3)	0.0057 (3)	0.0259 (3)
N5	0.0740 (12)	0.0536 (10)	0.0461 (9)	0.0099 (9)	0.0093 (8)	0.0269 (8)
N6	0.0790 (12)	0.0537 (10)	0.0401 (8)	0.0104 (9)	0.0088 (8)	0.0249 (8)
N7	0.0760 (11)	0.0497 (10)	0.0379 (8)	0.0079 (8)	0.0112 (8)	0.0213 (7)
N8	0.0610 (10)	0.0546 (10)	0.0466 (9)	0.0123 (8)	0.0115 (7)	0.0282 (8)
C9	0.0580 (12)	0.0459 (11)	0.0482 (10)	0.0162 (9)	0.0093 (9)	0.0215 (9)
C10	0.0795 (14)	0.0535 (13)	0.0373 (10)	0.0183 (11)	0.0123 (9)	0.0185 (9)
C11	0.0786 (14)	0.0534 (12)	0.0409 (10)	0.0169 (11)	0.0182 (9)	0.0256 (9)
C12	0.0540 (11)	0.0488 (11)	0.0394 (9)	0.0147 (9)	0.0086 (8)	0.0225 (9)
C13	0.0534 (12)	0.0540 (12)	0.0493 (10)	0.0128 (10)	0.0069 (9)	0.0261 (10)
C14	0.0692 (14)	0.0642 (14)	0.0642 (13)	0.0077 (11)	0.0132 (10)	0.0349 (11)
C15	0.1004 (19)	0.0848 (18)	0.0671 (15)	0.0104 (14)	0.0291 (13)	0.0410 (13)
C16	0.0818 (16)	0.0572 (13)	0.0572 (12)	0.0078 (11)	0.0125 (11)	0.0212 (10)

Geometric parameters (Å, °)

Cl1—C1	1.734 (2)	C6—H6B	0.9700
Cl2—C9	1.733 (2)	C6—H6A	0.9700

N1—N2	1.3504 (9)	C7—H7A	0.9600
N1—C1	1.3075 (17)	C7—H7C	0.9600
N2—C4	1.3345 (19)	C7—H7B	0.9600
N3—N4	1.3843 (18)	C8—H8A	0.9600
N3—C4	1.360 (2)	C8—H8C	0.9600
N4—C5	1.276 (3)	C8—H8B	0.9600
N3—H3A	0.8600	C9—C10	1.388 (3)
N5—C9	1.297 (2)	C10—C11	1.348 (3)
N5—N6	1.350 (3)	C11—C12	1.405 (2)
N6—C12	1.333 (3)	C13—C16	1.4981 (19)
N7—N8	1.381 (2)	C13—C14	1.505 (3)
N7—C12	1.357 (3)	C14—C15	1.508 (3)
N8—C13	1.272 (3)	C10—H10	0.9300
N7—H7	0.8600	C11—H11	0.9300
C1—C2	1.392 (3)	C14—H14A	0.9700
C2—C3	1.347 (3)	C14—H14B	0.9700
C3—C4	1.408 (2)	C15—H15A	0.9600
C5—C8	1.498 (3)	C15—H15B	0.9600
C5—C6	1.501 (3)	C15—H15C	0.9600
C6—C7	1.503 (3)	C16—H16A	0.9600
C2—H2	0.9300	C16—H16B	0.9600
C3—H3	0.9300	C16—H16C	0.9600
N2—N1—C1	118.43 (10)	H8A—C8—H8C	109.00
N1—N2—C4	119.67 (8)	C5—C8—H8A	109.00
N4—N3—C4	117.38 (10)	H8B—C8—H8C	109.00
N3—N4—C5	118.39 (13)	H8A—C8—H8B	109.00
C4—N3—H3A	121.00	C5—C8—H8C	109.00
N4—N3—H3A	121.00	C5—C8—H8B	109.00
N6—N5—C9	119.09 (18)	Cl2—C9—C10	119.98 (14)
N5—N6—C12	119.53 (15)	Cl2—C9—N5	115.69 (16)
N8—N7—C12	117.28 (14)	N5—C9—C10	124.3 (2)
N7—N8—C13	119.01 (15)	C9—C10—C11	117.37 (17)
N8—N7—H7	121.00	C10—C11—C12	117.67 (19)
C12—N7—H7	121.00	N7—C12—C11	122.28 (19)
N1—C1—C2	124.75 (16)	N6—C12—N7	115.75 (15)
Cl1—C1—C2	119.92 (13)	N6—C12—C11	121.96 (19)
Cl1—C1—N1	115.33 (13)	N8—C13—C14	116.84 (17)
C1—C2—C3	117.28 (16)	N8—C13—C16	125.68 (18)
C2—C3—C4	117.42 (17)	C14—C13—C16	117.47 (17)
N2—C4—N3	114.99 (12)	C13—C14—C15	115.48 (19)
N3—C4—C3	122.59 (16)	C9—C10—H10	121.00
N2—C4—C3	122.41 (16)	C11—C10—H10	121.00
C6—C5—C8	117.44 (18)	C10—C11—H11	121.00
N4—C5—C8	125.76 (19)	C12—C11—H11	121.00
N4—C5—C6	116.78 (16)	C13—C14—H14A	108.00
C5—C6—C7	115.54 (19)	C13—C14—H14B	108.00
C3—C2—H2	121.00	C15—C14—H14A	108.00
C1—C2—H2	121.00	C15—C14—H14B	108.00
C4—C3—H3	121.00	H14A—C14—H14B	107.00

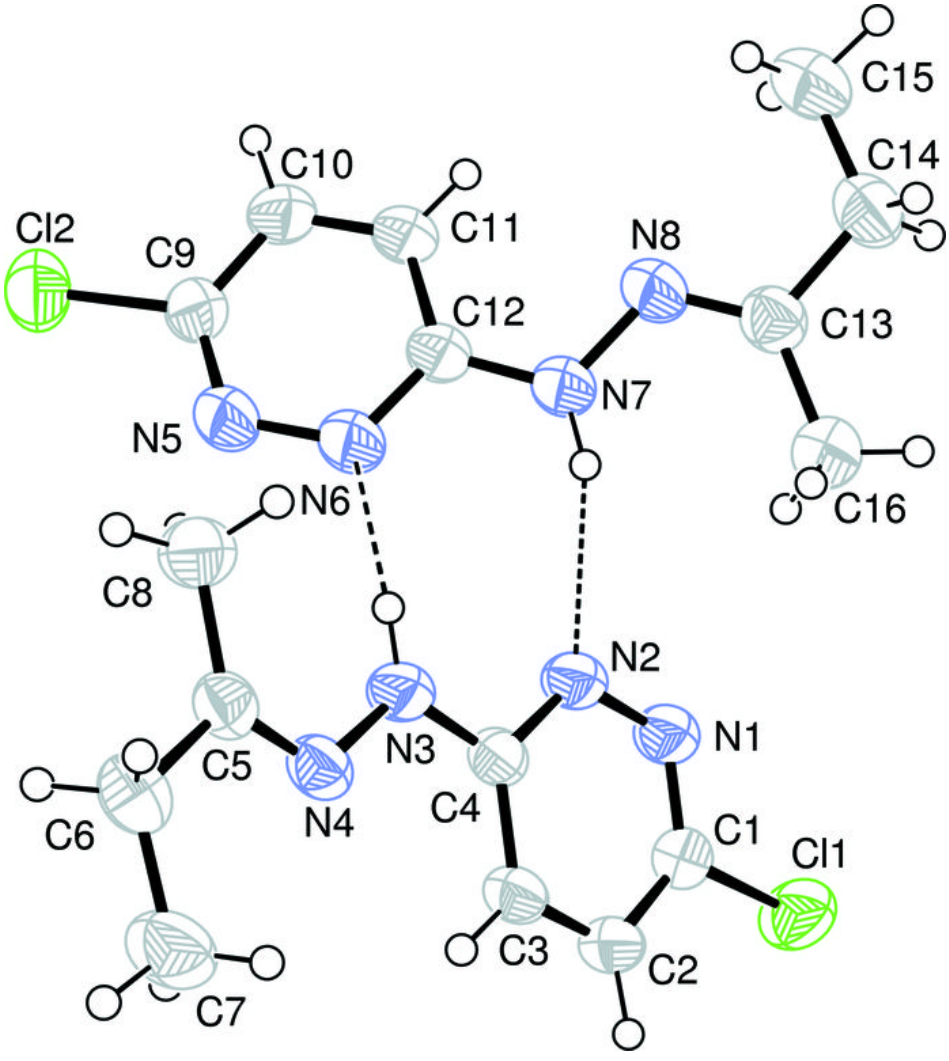
C2—C3—H3	121.00	C14—C15—H15A	109.00
C5—C6—H6A	108.00	C14—C15—H15B	109.00
C7—C6—H6B	108.00	C14—C15—H15C	109.00
C5—C6—H6B	108.00	H15A—C15—H15B	110.00
H6A—C6—H6B	107.00	H15A—C15—H15C	109.00
C7—C6—H6A	108.00	H15B—C15—H15C	110.00
H7A—C7—H7B	110.00	C13—C16—H16A	109.00
H7A—C7—H7C	109.00	C13—C16—H16B	109.00
C6—C7—H7C	109.00	C13—C16—H16C	109.00
H7B—C7—H7C	109.00	H16A—C16—H16B	109.00
C6—C7—H7B	109.00	H16A—C16—H16C	109.00
C6—C7—H7A	109.00	H16B—C16—H16C	109.00
C1—N1—N2—C4	1.45 (15)	N8—N7—C12—C11	12.0 (3)
N2—N1—C1—Cl1	−179.47 (8)	N7—N8—C13—C16	1.2 (3)
N2—N1—C1—C2	0.5 (2)	N7—N8—C13—C14	−177.43 (18)
N1—N2—C4—N3	178.67 (10)	N1—C1—C2—C3	−1.3 (3)
N1—N2—C4—C3	−2.6 (2)	Cl1—C1—C2—C3	178.68 (14)
C4—N3—N4—C5	−176.95 (15)	C1—C2—C3—C4	0.2 (3)
N4—N3—C4—N2	174.58 (12)	C2—C3—C4—N2	1.8 (3)
N4—N3—C4—C3	−4.1 (2)	C2—C3—C4—N3	−179.62 (16)
N3—N4—C5—C6	178.44 (15)	N4—C5—C6—C7	−0.6 (3)
N3—N4—C5—C8	0.2 (3)	C8—C5—C6—C7	177.78 (19)
C9—N5—N6—C12	−0.8 (3)	Cl2—C9—C10—C11	−179.66 (18)
N6—N5—C9—Cl2	179.70 (14)	N5—C9—C10—C11	1.1 (3)
N6—N5—C9—C10	−1.0 (3)	C9—C10—C11—C12	0.6 (3)
N5—N6—C12—N7	−176.31 (16)	C10—C11—C12—N6	−2.3 (3)
N5—N6—C12—C11	2.5 (3)	C10—C11—C12—N7	176.4 (2)
C12—N7—N8—C13	−176.99 (19)	N8—C13—C14—C15	−8.9 (3)
N8—N7—C12—N6	−169.24 (16)	C16—C13—C14—C15	172.35 (18)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3A···N6^{i}	0.86	2.30	3.0674 (15)	148
N7—H7···N2^{i}	0.86	2.24	3.0689 (15)	161

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

Fig. 1


C15
C14
C10
Cl2
C9
C11
N8
C12
C13
N7
N5
N6
C16
C8
N2
N1
C5
N3
C4
C6
N4
C1
Cl1
C3
C2
C7