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4-Chloro-6-(piperidin-1-yl)-N^2-(2,4,4-trimethylpentan-2-yl)-1,3,5-triazin-2-amine

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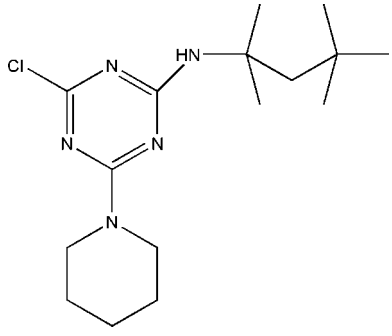
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.052; wR factor = 0.116; data-to-parameter ratio = 21.0.

In the title compound, $C_{16}H_{28}ClN_5$, the piperidine ring has a classical chair conformation. In the crystal structure, weak intermolecular N—H···N hydrogen bonds link two molecules, related by a twofold axis of symmetry, into dimers.

Related literature

For a related crystal structure, see: Deng *et al.* (2006). For related literature, see: Borzatta & Carrozza (1991); Kaiser & Thurston (1951).



Experimental

Crystal data

$C_{16}H_{28}ClN_5$
M_r = 325.88
Monoclinic, $C2/c$
a = 23.603 (3) Å
b = 7.9486 (12) Å
c = 19.659 (3) Å
β = 95.818 (13)°
V = 3669.3 (9) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.21 mm^{-1}
T = 113 (2) K
0.34 × 0.24 × 0.20 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.920, T_{max} = 0.959
16244 measured reflections
4366 independent reflections
3747 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.116
S = 1.11
4366 reflections
208 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5···N3^{i}	0.886 (19)	2.20 (2)	3.0860 (19)	173.5 (17)

Symmetry code: (i) $-x, y, -z+\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 1997); cell refinement: *CrystalStructure* (Rigaku, 1997); data reduction: *CrystalStructure*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2257).

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supplementary materials

Acta Cryst. (2007). E**63**, o3288 [**doi:10.1107/S1600536807027341**]

4-Chloro-6-(piperidin-1-yl)-N^2-(2,4,4-trimethylpentan-2-yl)-1,3,5-triazin-2-amine

X.-Y. Tang, F. Wen, L. Qin, L. Xu and Y.-G. Yin

Comment

The title compound (I), has attracted much attention as an important intermediate of hindered light stabilizers (Borzatta & Carrozza, 1991). These compounds containing triazine ring are widely used (Deng *et al.*, 2006). Herein we reported the crystal structure of the title compound, (I) (Fig. 1).

The triazine ring in (I) is essentially planar with an r.m.s. deviation from the mean plane of 0.0133 Å. In the crystal, the molecules are linked into dimers by intermolecular N—H···N hydrogen bonds (Table 1).

Experimental

The title compound was prepared according to the method of Kaiser & Thurston (1951). 2,4,6-Trichloro-1,3,5-triazine (40.0 g, 0.217 mol) was dissolved in toluene (120 ml) and then cooled to 278 K. With stirring, a solution of 2,4,4-trimethylpentan-2-amine (27.5 g, 0.213 mol) in toluene (50 ml) was then added dropwise to the mixture over a period of 0.5 h. Then a solution of Na_2CO_3 (23.02 g, 0.217 mol) in water (50 ml) was added dropwise for 0.5 h. The mixture was stirred at 273–278 K for a further 3 h. Piperidine (18.46 g, 0.217 mol) and solid Na_2CO_3 (23.02 g, 0.217 mol) were added to the mixture, maintaining the temperature at 338 k for 5 h. The organic layer was washed with water and then concentrated *in vacuo*. The title compound (54.50 g) was obtained in powder form in a yield of 82.1%. Crystals of (I) were obtained by slow evaporation of a solution of methanol (m.p. 427–429 K).

Refinement

The atom H5 (attached to N5) was located from difference map and refined isotropically. The rest H atoms were positioned geometrically (C—H = 0.98–0.99 Å), and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(C).

Figures

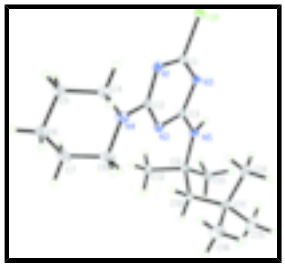


Fig. 1. A view of the molecule of (I). Displacement ellopsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

4-Chloro-6-(piperidin-1-yl)-N^2-(2,4,4-trimethylpentan-2-yl)- 1,3,5-triazin-2-amine

Crystal data

$C_{16}H_{28}ClN_5$	$D_x = 1.180$ Mg m^{-3}
$M_r = 325.88$	Melting point: 103-106 K
Monoclinic, $C2/c$	Mo $K\alpha$ radiation $\lambda = 0.71070$ Å
$a = 23.603$ (3) Å	Cell parameters from 3587 reflections
$b = 7.9486$ (12) Å	$\theta = 2.7–25.0°$
$c = 19.659$ (3) Å	$\mu = 0.21$ mm^{-1}
$\beta = 95.818$ (13)°	$T = 113$ (2) K
$V = 3669.3$ (9) Å^3	Prism, colourless
$Z = 8$	0.34 × 0.24 × 0.20 mm
$F_{000} = 1408$	

Data collection

Rigaku Saturn diffractometer	3747 reflections with $I > 2\sigma(I)$
Radiation source: rotating anode	$R_{int} = 0.045$
Monochromator: confocal	$\theta_{max} = 27.9°$
$T = 113(2)$ K	$\theta_{min} = 1.7°$
ω scans	$h = -30\rightarrow30$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -10\rightarrow10$
$T_{min} = 0.920$, $T_{max} = 0.959$	$l = -25\rightarrow25$
16244 measured reflections	Standard reflections: ?
4366 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.052$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.116$	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 1.7404P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} < 0.001$
4366 reflections	$\Delta\rho_{max} = 0.20$ e Å^{-3}
208 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-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 $F^2 > 2\text{sigma}(F^2)$ 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	0.100756 (18)	0.56099 (6)	0.13014 (2)	0.02726 (13)
N1	0.16998 (6)	0.47978 (17)	0.23565 (6)	0.0196 (3)
N2	0.13973 (5)	0.33320 (17)	0.33276 (6)	0.0187 (3)
N3	0.07186 (6)	0.40830 (17)	0.23796 (6)	0.0185 (3)
N4	0.23465 (6)	0.3886 (2)	0.32432 (7)	0.0286 (4)
N5	0.04278 (6)	0.27903 (17)	0.33219 (6)	0.0182 (3)
C1	0.11603 (7)	0.4726 (2)	0.21150 (8)	0.0180 (3)
C2	0.18014 (7)	0.4003 (2)	0.29723 (8)	0.0191 (3)
C3	0.08661 (6)	0.34137 (19)	0.30201 (8)	0.0171 (3)
C4	0.28234 (7)	0.4602 (2)	0.29193 (9)	0.0273 (4)
H4A	0.2681	0.5116	0.2476	0.033*
H4B	0.3095	0.3700	0.2830	0.033*
C5	0.31245 (8)	0.5928 (2)	0.33808 (9)	0.0281 (4)
H5A	0.2865	0.6894	0.3423	0.034*
H5B	0.3463	0.6341	0.3173	0.034*
C6	0.33093 (8)	0.5218 (3)	0.40907 (10)	0.0369 (5)
H6A	0.3611	0.4366	0.4058	0.044*
H6B	0.3468	0.6134	0.4394	0.044*
C7	0.28043 (9)	0.4416 (3)	0.43953 (10)	0.0452 (6)
H7A	0.2528	0.5301	0.4489	0.054*
H7B	0.2939	0.3867	0.4834	0.054*
C8	0.25106 (8)	0.3113 (3)	0.39086 (10)	0.0389 (5)
H8A	0.2772	0.2160	0.3855	0.047*
H8B	0.2168	0.2672	0.4101	0.047*
C9	0.04368 (7)	0.1958 (2)	0.39985 (7)	0.0185 (3)
C10	0.08715 (7)	0.0497 (2)	0.41039 (8)	0.0202 (3)
H10A	0.0860	0.0106	0.4580	0.024*
H10B	0.1252	0.1006	0.4082	0.024*
C11	0.08562 (8)	−0.1109 (2)	0.36542 (9)	0.0258 (4)
C12	0.05982 (8)	0.3252 (2)	0.45668 (8)	0.0260 (4)
H12A	0.0990	0.3635	0.4539	0.039*
H12B	0.0569	0.2730	0.5014	0.039*
H12C	0.0338	0.4215	0.4510	0.039*

C13	−0.01778 (7)	0.1390 (2)	0.40468 (9)	0.0256 (4)
H13A	−0.0422	0.2379	0.4072	0.038*
H13B	−0.0195	0.0701	0.4458	0.038*
H13C	−0.0310	0.0725	0.3642	0.038*
C14	0.08364 (9)	−0.0723 (2)	0.28890 (9)	0.0329 (4)
H14A	0.0866	−0.1775	0.2635	0.049*
H14B	0.1155	0.0017	0.2807	0.049*
H14C	0.0476	−0.0164	0.2735	0.049*
C15	0.03606 (10)	−0.2286 (3)	0.37689 (12)	0.0483 (6)
H15A	0.0000	−0.1753	0.3595	0.072*
H15B	0.0360	−0.2511	0.4259	0.072*
H15C	0.0405	−0.3347	0.3526	0.072*
C16	0.14161 (10)	−0.2037 (3)	0.38666 (12)	0.0498 (6)
H16A	0.1429	−0.3077	0.3601	0.075*
H16B	0.1439	−0.2311	0.4355	0.075*
H16C	0.1738	−0.1317	0.3780	0.075*
H5	0.0088 (8)	0.308 (2)	0.3122 (10)	0.030 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0267 (2)	0.0337 (3)	0.0210 (2)	−0.00372 (18)	0.00062 (16)	0.00908 (16)
N1	0.0193 (7)	0.0206 (7)	0.0192 (6)	−0.0013 (6)	0.0037 (5)	0.0017 (5)
N2	0.0149 (7)	0.0208 (7)	0.0203 (6)	−0.0010 (5)	0.0015 (5)	0.0028 (5)
N3	0.0177 (7)	0.0202 (7)	0.0177 (6)	−0.0004 (5)	0.0021 (5)	0.0023 (5)
N4	0.0162 (7)	0.0402 (9)	0.0293 (8)	−0.0037 (6)	0.0012 (6)	0.0138 (7)
N5	0.0151 (7)	0.0219 (7)	0.0175 (6)	0.0005 (5)	0.0017 (5)	0.0033 (5)
C1	0.0200 (8)	0.0166 (8)	0.0178 (7)	0.0000 (6)	0.0031 (6)	0.0014 (6)
C2	0.0186 (8)	0.0182 (8)	0.0207 (8)	−0.0010 (6)	0.0023 (6)	0.0011 (6)
C3	0.0167 (8)	0.0154 (7)	0.0195 (7)	0.0009 (6)	0.0030 (6)	−0.0006 (6)
C4	0.0172 (9)	0.0372 (10)	0.0281 (9)	−0.0030 (7)	0.0056 (7)	0.0056 (7)
C5	0.0242 (9)	0.0298 (10)	0.0311 (9)	−0.0006 (7)	0.0059 (7)	0.0054 (7)
C6	0.0308 (11)	0.0475 (12)	0.0311 (10)	−0.0103 (9)	−0.0030 (8)	0.0081 (9)
C7	0.0320 (11)	0.0757 (16)	0.0270 (10)	−0.0091 (11)	−0.0019 (8)	0.0178 (10)
C8	0.0199 (9)	0.0534 (13)	0.0419 (11)	−0.0070 (9)	−0.0042 (8)	0.0288 (10)
C9	0.0193 (8)	0.0200 (8)	0.0166 (7)	0.0001 (6)	0.0042 (6)	0.0028 (6)
C10	0.0212 (8)	0.0202 (8)	0.0190 (7)	0.0004 (6)	0.0011 (6)	0.0021 (6)
C11	0.0321 (10)	0.0182 (8)	0.0285 (9)	0.0032 (7)	0.0089 (7)	−0.0005 (7)
C12	0.0335 (10)	0.0245 (9)	0.0207 (8)	0.0012 (7)	0.0054 (7)	−0.0033 (7)
C13	0.0221 (9)	0.0298 (9)	0.0257 (8)	0.0009 (7)	0.0069 (7)	0.0075 (7)
C14	0.0436 (12)	0.0287 (10)	0.0277 (9)	−0.0005 (8)	0.0094 (8)	−0.0086 (7)
C15	0.0667 (16)	0.0258 (11)	0.0573 (14)	−0.0143 (10)	0.0303 (12)	−0.0109 (10)
C16	0.0594 (15)	0.0381 (12)	0.0510 (13)	0.0258 (11)	0.0006 (11)	−0.0073 (10)

Geometric parameters (Å, °)

Cl1—C1	1.7506 (15)	C8—H8B	0.9900
N1—C1	1.314 (2)	C9—C13	1.531 (2)
N1—C2	1.3650 (19)	C9—C12	1.538 (2)

N2—C3	1.3371 (19)	C9—C10	1.549 (2)
N2—C2	1.348 (2)	C10—C11	1.551 (2)
N3—C1	1.315 (2)	C10—H10A	0.9900
N3—C3	1.3787 (19)	C10—H10B	0.9900
N4—C2	1.345 (2)	C11—C14	1.531 (2)
N4—C8	1.461 (2)	C11—C15	1.532 (3)
N4—C4	1.464 (2)	C11—C16	1.534 (3)
N5—C3	1.339 (2)	C12—H12A	0.9800
N5—C9	1.4839 (19)	C12—H12B	0.9800
N5—H5	0.886 (19)	C12—H12C	0.9800
C4—C5	1.519 (2)	C13—H13A	0.9800
C4—H4A	0.9900	C13—H13B	0.9800
C4—H4B	0.9900	C13—H13C	0.9800
C5—C6	1.527 (2)	C14—H14A	0.9800
C5—H5A	0.9900	C14—H14B	0.9800
C5—H5B	0.9900	C14—H14C	0.9800
C6—C7	1.527 (3)	C15—H15A	0.9800
C6—H6A	0.9900	C15—H15B	0.9800
C6—H6B	0.9900	C15—H15C	0.9800
C7—C8	1.528 (3)	C16—H16A	0.9800
C7—H7A	0.9900	C16—H16B	0.9800
C7—H7B	0.9900	C16—H16C	0.9800
C8—H8A	0.9900		
C1—N1—C2	112.15 (13)	N5—C9—C13	105.04 (12)
C3—N2—C2	115.26 (13)	N5—C9—C12	109.45 (13)
C1—N3—C3	111.98 (13)	C13—C9—C12	108.58 (13)
C2—N4—C8	122.67 (14)	N5—C9—C10	113.77 (13)
C2—N4—C4	123.21 (14)	C13—C9—C10	112.92 (13)
C8—N4—C4	114.03 (13)	C12—C9—C10	106.99 (12)
C3—N5—C9	128.38 (13)	C9—C10—C11	124.32 (13)
C3—N5—H5	114.4 (12)	C9—C10—H10A	106.2
C9—N5—H5	116.2 (13)	C11—C10—H10A	106.2
N1—C1—N3	130.69 (14)	C9—C10—H10B	106.2
N1—C1—Cl1	114.51 (12)	C11—C10—H10B	106.2
N3—C1—Cl1	114.79 (11)	H10A—C10—H10B	106.4
N4—C2—N2	117.78 (14)	C14—C11—C15	108.62 (16)
N4—C2—N1	117.28 (14)	C14—C11—C16	107.66 (16)
N2—C2—N1	124.94 (14)	C15—C11—C16	108.39 (17)
N2—C3—N5	120.86 (14)	C14—C11—C10	113.06 (14)
N2—C3—N3	124.76 (14)	C15—C11—C10	113.11 (15)
N5—C3—N3	114.37 (13)	C16—C11—C10	105.74 (15)
N4—C4—C5	110.12 (15)	C9—C12—H12A	109.5
N4—C4—H4A	109.6	C9—C12—H12B	109.5
C5—C4—H4A	109.6	H12A—C12—H12B	109.5
N4—C4—H4B	109.6	C9—C12—H12C	109.5
C5—C4—H4B	109.6	H12A—C12—H12C	109.5
H4A—C4—H4B	108.2	H12B—C12—H12C	109.5
C4—C5—C6	111.24 (15)	C9—C13—H13A	109.5
C4—C5—H5A	109.4	C9—C13—H13B	109.5

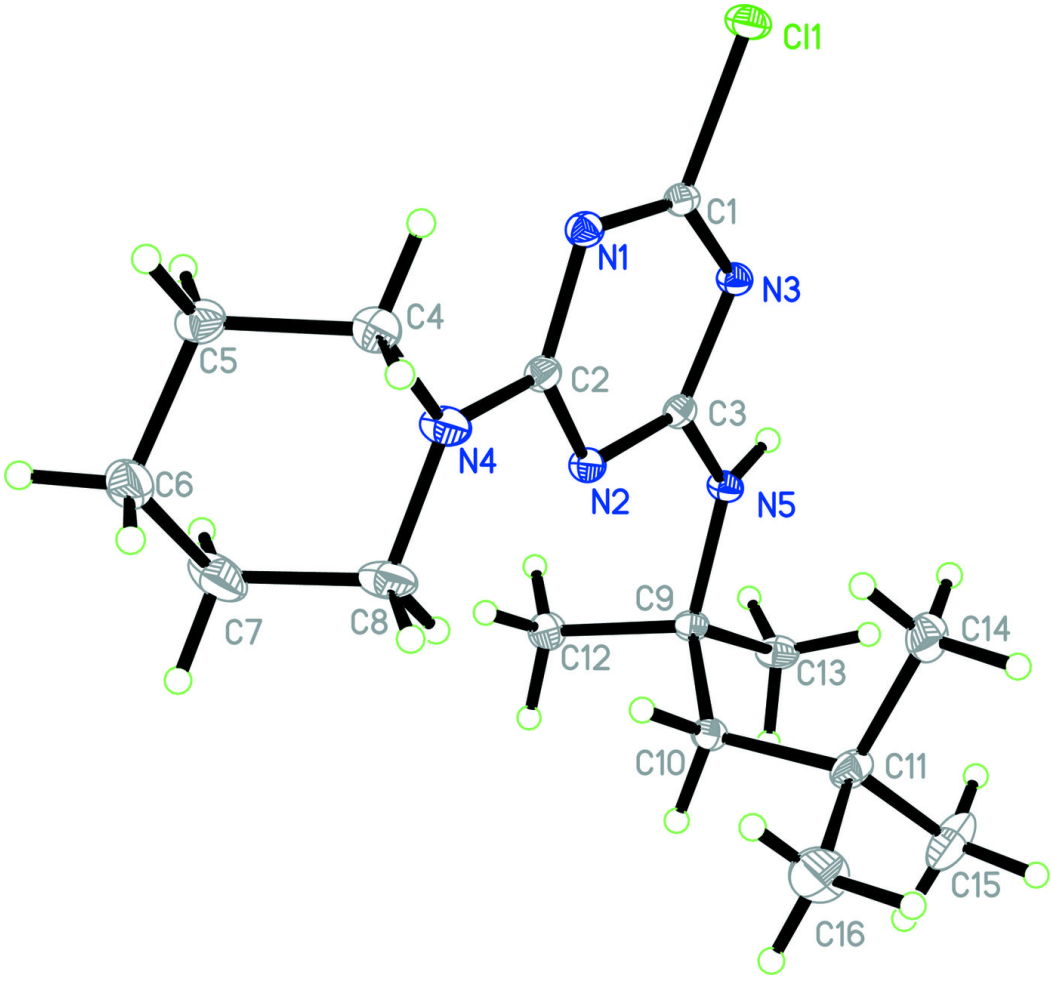
C6—C5—H5A	109.4	H13A—C13—H13B	109.5
C4—C5—H5B	109.4	C9—C13—H13C	109.5
C6—C5—H5B	109.4	H13A—C13—H13C	109.5
H5A—C5—H5B	108.0	H13B—C13—H13C	109.5
C7—C6—C5	110.53 (15)	C11—C14—H14A	109.5
C7—C6—H6A	109.5	C11—C14—H14B	109.5
C5—C6—H6A	109.5	H14A—C14—H14B	109.5
C7—C6—H6B	109.5	C11—C14—H14C	109.5
C5—C6—H6B	109.5	H14A—C14—H14C	109.5
H6A—C6—H6B	108.1	H14B—C14—H14C	109.5
C6—C7—C8	111.12 (18)	C11—C15—H15A	109.5
C6—C7—H7A	109.4	C11—C15—H15B	109.5
C8—C7—H7A	109.4	H15A—C15—H15B	109.5
C6—C7—H7B	109.4	C11—C15—H15C	109.5
C8—C7—H7B	109.4	H15A—C15—H15C	109.5
H7A—C7—H7B	108.0	H15B—C15—H15C	109.5
N4—C8—C7	109.77 (16)	C11—C16—H16A	109.5
N4—C8—H8A	109.7	C11—C16—H16B	109.5
C7—C8—H8A	109.7	H16A—C16—H16B	109.5
N4—C8—H8B	109.7	C11—C16—H16C	109.5
C7—C8—H8B	109.7	H16A—C16—H16C	109.5
H8A—C8—H8B	108.2	H16B—C16—H16C	109.5
C2—N1—C1—N3	2.7 (2)	C2—N4—C4—C5	118.14 (18)
C2—N1—C1—Cl1	−176.28 (11)	C8—N4—C4—C5	−58.5 (2)
C3—N3—C1—N1	1.2 (2)	N4—C4—C5—C6	54.8 (2)
C3—N3—C1—Cl1	−179.80 (11)	C4—C5—C6—C7	−53.5 (2)
C8—N4—C2—N2	−1.9 (3)	C5—C6—C7—C8	53.7 (2)
C4—N4—C2—N2	−178.23 (15)	C2—N4—C8—C7	−118.13 (19)
C8—N4—C2—N1	178.03 (17)	C4—N4—C8—C7	58.5 (2)
C4—N4—C2—N1	1.7 (2)	C6—C7—C8—N4	−55.1 (2)
C3—N2—C2—N4	−176.96 (15)	C3—N5—C9—C13	−175.67 (15)
C3—N2—C2—N1	3.1 (2)	C3—N5—C9—C12	67.9 (2)
C1—N1—C2—N4	175.05 (15)	C3—N5—C9—C10	−51.7 (2)
C1—N1—C2—N2	−5.0 (2)	N5—C9—C10—C11	−60.8 (2)
C2—N2—C3—N5	−179.97 (14)	C13—C9—C10—C11	58.8 (2)
C2—N2—C3—N3	1.6 (2)	C12—C9—C10—C11	178.18 (15)
C9—N5—C3—N2	0.1 (2)	C9—C10—C11—C14	53.1 (2)
C9—N5—C3—N3	178.72 (14)	C9—C10—C11—C15	−70.9 (2)
C1—N3—C3—N2	−3.5 (2)	C9—C10—C11—C16	170.68 (16)
C1—N3—C3—N5	177.93 (13)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N5—H5···N3^{i}	0.886 (19)	2.20 (2)	3.0860 (19)	173.5 (17)

Symmetry codes: (i) −*x*, *y*, −*z*+1/2.

Fig. 1


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