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6-Chloro-N^2,N^4-di-*p*-tolyl-1,3,5-triazine-2,4-diamine acetone solvate

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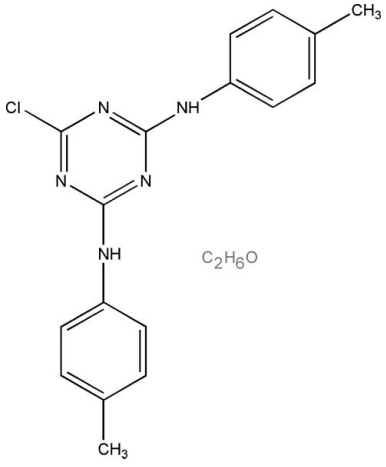
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.009 Å; R factor = 0.077; wR factor = 0.329; data-to-parameter ratio = 15.7.

The title compound, $C_{18}H_{16}ClN_5\cdot C_2H_6O$, was prepared by the reaction of *p*-toluidine with 2,4,6-trichloro-1,3,5-triazine at room temperature. The three rings are not coplanar; the dihedral angles between the triazine ring and the phenyl rings are 41.32 and 6.58°, and that between the two phenyl rings is 35.58°. The molecular structure and packing are stabilized by N—H···N, C—H···O and C—H···N hydrogen-bond interactions and C—H···π interactions.

Related literature

For related literature, see: Manasek & Hrdlovik (1990); Mathias & Simanek (1994); Zeng, Dong & Shu (2005); Zeng, Dong, Shu *et al.* (2005).



Experimental

Crystal data

$C_{18}H_{16}ClN_5\cdot C_2H_6O$
M_r = 383.88
Monoclinic, $P2_1/c$
a = 7.8610 (16) Å
b = 22.579 (5) Å
c = 12.012 (2) Å
β = 104.22 (3)°
V = 2066.7 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.20 mm^{-1}
T = 295 (2) K
0.30 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: none
4122 measured reflections
3833 independent reflections
1855 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
3 standard reflections every 100 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.329
S = 1.06
3833 reflections
244 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

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

*Cg*1 and *Cg*2 are the centroids of the C2–C7 and C11–C16 phenyl rings.

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*A*···N3^{i}	0.86	2.16	3.018 (6)	176
N5—H5*A*···N2^{ii}	0.86	2.45	3.300 (6)	171
C4—H4*B*···O1^{iii}	0.93	2.36	3.264 (9)	165
C6—H6*A*···N4	0.93	2.59	2.999 (6)	107
C12—H12*A*···N4	0.93	2.30	2.912 (7)	123
C1—H1*C*···*Cg*1^{iv}	0.96	3.01	3.806 (2)	141
C17—H17*C*···*Cg*2^{v}	0.96	2.92	3.745 (2)	145

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2472).

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supplementary materials

Acta Cryst. (2007). E**63**, o4937 [**doi:10.1107/S1600536807060151**]

6-Chloro-N^2,N^4-di-*p*-tolyl-1,3,5-triazine-2,4-diamine acetone solvate

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Comment

The amine derivatives of 2,4,6-trichloro[1,3,5]triazine possess valuable properties, as they are widely used as starting materials for many products, including drugs and light stabilizers (Mathias & Simanek, 1994; Manasek & Hrdlovik, 1990). The title compound has been synthesized and we report here its crystal structure.

In the crystal structure (Fig. 1), it contains a solution molecule of acetone, and there is an interaction between the major molecule and the solution molecule. The bond lengths and angles of the major molecule (Table 1) is agreement with those of the similar compound 2,4-dichloro-6-aniline-1,3,5-triazine (Zeng, Dong & Shu, 2005; Zeng, Dong, Shu, Li & Huang, 2005). The atom N1 lies in the plan of phenyl C1—C7(p1). The atom N5 lies in the plan of phenyl C11—C17(p2). The dihedral angles formed by the triazine ring with p1 and p2 are 40.9 (8) and 6.3 (1)°, respectively. The dihedral angles between the plane p1 and p2 is 35.8 (1)°.

It exists two kind of C—H···Π interaction [C1···C_g1 = 3.806 (2), C17···C_g2 = 3.745 (2)Å and C1—H1···C_g1 = 141.1 (1), C17—H17C···C_g2 = 145.2 (2)°] (C_g1 = phenyl ring C2–C7, C_g2 = phenyl ring C11—C16). In addtion, there exist N—H···N and C—H···O intermolecular interactions (Table 1). All above interactions stabilize the title structure.

Experimental

A mixture of 2,4,6-trichloro-1,3,5-triazine (0.02 mol) and *p*-toluidine (0.04 mol) was stirred with acetone (50 ml) at 293 K for 5 h, affording the title compound (4.8 g, yield 90%). Single crystals suitable for X-ray measurements were obtained by recrystallization from acetone at room temperature.

Refinement

H atoms were positioned geometrically and allowed to ride on their parent atoms, with N—H and C—H distances of 0.86 and 0.93–0.96 Å, respectively, and with U_{iso}(H) = 1.2 or 1.5U_{eq} of the parent atoms.

Figures

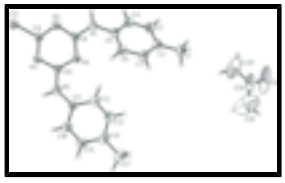


Fig. 1. The molecular structure of the title compound with the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level.

6-Chloro-N^2,N^4-di-*p*-tolyl-1,3,5-triazine-2,4-diamine acetone solvate

Crystal data

$C_{18}H_{16}ClN_5 \cdot C_2H_6O_1$	F_{000} = 808
M_r = 383.88	D_x = 1.234 Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 221.3 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 7.8610 (16) Å	Cell parameters from 25 reflections
b = 22.579 (5) Å	θ = 4–14º
c = 12.012 (2) Å	μ = 0.20 mm^{-1}
β = 104.22 (3)º	T = 295 (2) K
V = 2066.7 (7) Å^3	Block, white
Z = 4	0.30 × 0.20 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.052
Radiation source: fine-focus sealed tube	θ_{max} = 25.5º
Monochromator: graphite	θ_{min} = 1.8º
T = 295(2) K	h = 0→9
ω scans	k = 0→26
Absorption correction: none	l = −14→14
4122 measured reflections	3 standard reflections
3833 independent reflections	every 100 reflections
1855 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.077	H-atom parameters constrained
$wR(F^2)$ = 0.329	$w = 1/[\sigma^2(F_o^2) + (0.1873P)^2 + 0.955P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max} < 0.001$
3833 reflections	$\Delta\rho_{max}$ = 0.39 e Å^{-3}
244 parameters	$\Delta\rho_{min}$ = −0.48 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	0.1703 (2)	0.34124 (6)	0.34531 (14)	0.0828 (6)
N1	−0.0587 (6)	0.17534 (18)	0.0908 (4)	0.0636 (12)
H1A	−0.0266	0.1972	0.0411	0.076*
N2	0.0415 (6)	0.25289 (17)	0.2107 (4)	0.0618 (11)
N3	0.0340 (6)	0.24672 (17)	0.4102 (3)	0.0555 (11)
N4	−0.0697 (5)	0.16526 (16)	0.2826 (3)	0.0539 (10)
N5	−0.0776 (6)	0.16432 (17)	0.4782 (4)	0.0596 (11)
H5A	−0.0589	0.1850	0.5401	0.071*
C1	−0.3658 (9)	−0.0448 (3)	−0.0980 (6)	0.0835 (18)
H1B	−0.4447	−0.0375	−0.1714	0.125*
H1C	−0.4286	−0.0630	−0.0479	0.125*
H1D	−0.2735	−0.0707	−0.1074	0.125*
C2	−0.2877 (7)	0.0130 (2)	−0.0466 (5)	0.0627 (13)
C3	−0.3263 (8)	0.0658 (3)	−0.1043 (5)	0.0755 (17)
H3B	−0.4030	0.0658	−0.1769	0.091*
C4	−0.2545 (8)	0.1189 (2)	−0.0577 (5)	0.0718 (16)
H4B	−0.2851	0.1540	−0.0983	0.086*
C5	−0.1370 (7)	0.1200 (2)	0.0493 (5)	0.0589 (13)
C6	−0.0954 (7)	0.0678 (2)	0.1096 (5)	0.0611 (14)
H6A	−0.0180	0.0679	0.1819	0.073*
C7	−0.1702 (7)	0.0153 (2)	0.0614 (5)	0.0669 (15)
H7A	−0.1410	−0.0197	0.1025	0.080*
C8	−0.0285 (6)	0.1978 (2)	0.1980 (4)	0.0519 (12)
C9	0.0696 (7)	0.2725 (2)	0.3187 (5)	0.0573 (13)
C10	−0.0386 (6)	0.19227 (19)	0.3868 (4)	0.0495 (11)
C11	−0.1436 (6)	0.1068 (2)	0.4875 (4)	0.0546 (12)
C12	−0.1980 (8)	0.0673 (2)	0.3971 (5)	0.0697 (15)
H12A	−0.1983	0.0788	0.3227	0.084*
C13	−0.2515 (8)	0.0108 (2)	0.4176 (5)	0.0751 (16)
H13A	−0.2861	−0.0151	0.3561	0.090*
C14	−0.2555 (7)	−0.0086 (2)	0.5272 (6)	0.0678 (15)
C15	−0.2045 (8)	0.0315 (3)	0.6166 (6)	0.0783 (17)
H15A	−0.2064	0.0201	0.6907	0.094*

C16	−0.1509 (8)	0.0881 (3)	0.5978 (5)	0.0733 (16)
H16A	−0.1191	0.1142	0.6592	0.088*
C17	−0.3135 (8)	−0.0711 (2)	0.5475 (6)	0.087 (2)
H17A	−0.3052	−0.0767	0.6279	0.130*
H17B	−0.2392	−0.0992	0.5224	0.130*
H17C	−0.4328	−0.0769	0.5050	0.130*
C20	−0.4657 (14)	−0.1911 (5)	−0.1900 (12)	0.190 (6)
H20A	−0.4986	−0.1768	−0.2676	0.285*
H20B	−0.3402	−0.1934	−0.1649	0.285*
H20C	−0.5090	−0.1644	−0.1410	0.285*
O1	−0.6395 (12)	−0.2727 (4)	−0.2586 (10)	0.270 (7)
C19	−0.5396 (11)	−0.2489 (4)	−0.1845 (10)	0.128 (4)
C18	−0.4935 (19)	−0.2761 (7)	−0.0704 (18)	0.282 (11)
H18A	−0.5479	−0.3143	−0.0738	0.423*
H18B	−0.5339	−0.2514	−0.0172	0.423*
H18C	−0.3684	−0.2804	−0.0456	0.423*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.1211 (13)	0.0545 (8)	0.0670 (11)	−0.0223 (7)	0.0123 (8)	−0.0058 (6)
N1	0.103 (3)	0.049 (2)	0.041 (3)	−0.010 (2)	0.022 (2)	−0.0016 (18)
N2	0.089 (3)	0.046 (2)	0.050 (3)	−0.007 (2)	0.017 (2)	−0.0037 (18)
N3	0.075 (3)	0.045 (2)	0.045 (2)	0.0009 (19)	0.0128 (19)	0.0006 (18)
N4	0.077 (3)	0.044 (2)	0.041 (2)	−0.0004 (18)	0.0162 (19)	−0.0022 (17)
N5	0.085 (3)	0.050 (2)	0.046 (3)	−0.002 (2)	0.020 (2)	−0.0019 (18)
C1	0.103 (5)	0.068 (4)	0.082 (5)	−0.015 (3)	0.027 (4)	−0.022 (3)
C2	0.073 (3)	0.058 (3)	0.061 (3)	−0.005 (3)	0.023 (3)	−0.005 (3)
C3	0.089 (4)	0.070 (4)	0.059 (4)	−0.001 (3)	0.001 (3)	−0.010 (3)
C4	0.098 (4)	0.053 (3)	0.060 (4)	0.006 (3)	0.010 (3)	0.004 (3)
C5	0.074 (3)	0.047 (3)	0.058 (3)	0.001 (2)	0.020 (3)	−0.002 (2)
C6	0.073 (3)	0.052 (3)	0.058 (3)	0.007 (2)	0.015 (3)	−0.003 (2)
C7	0.089 (4)	0.046 (3)	0.070 (4)	0.004 (3)	0.027 (3)	−0.001 (2)
C8	0.069 (3)	0.047 (3)	0.040 (3)	0.003 (2)	0.014 (2)	−0.004 (2)
C9	0.072 (3)	0.041 (2)	0.055 (3)	−0.002 (2)	0.007 (2)	−0.002 (2)
C10	0.065 (3)	0.045 (2)	0.039 (3)	0.005 (2)	0.015 (2)	0.003 (2)
C11	0.059 (3)	0.053 (3)	0.052 (3)	0.002 (2)	0.012 (2)	0.008 (2)
C12	0.087 (4)	0.062 (3)	0.061 (4)	−0.011 (3)	0.021 (3)	−0.003 (3)
C13	0.092 (4)	0.063 (3)	0.074 (4)	−0.013 (3)	0.028 (3)	−0.006 (3)
C14	0.062 (3)	0.054 (3)	0.088 (4)	0.006 (2)	0.019 (3)	0.012 (3)
C15	0.100 (4)	0.064 (4)	0.072 (4)	0.003 (3)	0.022 (3)	0.020 (3)
C16	0.095 (4)	0.065 (3)	0.058 (4)	0.000 (3)	0.014 (3)	0.005 (3)
C17	0.083 (4)	0.061 (4)	0.119 (6)	−0.002 (3)	0.029 (4)	0.023 (3)
C20	0.115 (7)	0.120 (8)	0.317 (18)	−0.006 (6)	0.021 (9)	−0.032 (9)
O1	0.188 (7)	0.160 (7)	0.364 (14)	0.037 (6)	−0.122 (8)	−0.155 (8)
C19	0.088 (5)	0.097 (6)	0.171 (9)	0.018 (4)	−0.024 (5)	−0.060 (6)
C18	0.156 (11)	0.27 (2)	0.36 (3)	−0.053 (13)	−0.040 (15)	0.123 (18)

Geometric parameters (Å, °)

Cl1—C9	1.736 (5)	C6—H6A	0.9300
N1—C8	1.350 (6)	C7—H7A	0.9300
N1—C5	1.428 (6)	C11—C12	1.389 (7)
N1—H1A	0.8600	C11—C16	1.404 (7)
N2—C9	1.337 (6)	C12—C13	1.384 (7)
N2—C8	1.353 (6)	C12—H12A	0.9300
N3—C9	1.333 (6)	C13—C14	1.396 (8)
N3—C10	1.356 (6)	C13—H13A	0.9300
N4—C8	1.356 (6)	C14—C15	1.386 (8)
N4—C10	1.359 (6)	C14—C17	1.521 (7)
N5—C10	1.364 (6)	C15—C16	1.381 (7)
N5—C11	1.414 (6)	C15—H15A	0.9300
N5—H5A	0.8600	C16—H16A	0.9300
C1—C2	1.509 (7)	C17—H17A	0.9600
C1—H1B	0.9600	C17—H17B	0.9600
C1—H1C	0.9600	C17—H17C	0.9600
C1—H1D	0.9600	C20—C19	1.436 (13)
C2—C3	1.375 (8)	C20—H20A	0.9600
C2—C7	1.397 (8)	C20—H20B	0.9600
C3—C4	1.384 (7)	C20—H20C	0.9600
C3—H3B	0.9300	O1—C19	1.164 (9)
C4—C5	1.386 (8)	C19—C18	1.463 (18)
C4—H4B	0.9300	C18—H18A	0.9600
C5—C6	1.380 (7)	C18—H18B	0.9600
C6—C7	1.385 (7)	C18—H18C	0.9600
C8—N1—C5	128.5 (4)	N4—C10—N5	120.5 (4)
C8—N1—H1A	115.8	C12—C11—C16	117.9 (5)
C5—N1—H1A	115.8	C12—C11—N5	125.2 (5)
C9—N2—C8	112.3 (4)	C16—C11—N5	116.9 (5)
C9—N3—C10	112.8 (4)	C13—C12—C11	120.1 (5)
C8—N4—C10	115.1 (4)	C13—C12—H12A	120.0
C10—N5—C11	130.4 (4)	C11—C12—H12A	120.0
C10—N5—H5A	114.8	C12—C13—C14	122.4 (5)
C11—N5—H5A	114.8	C12—C13—H13A	118.8
C2—C1—H1B	109.5	C14—C13—H13A	118.8
C2—C1—H1C	109.5	C15—C14—C13	117.2 (5)
H1B—C1—H1C	109.5	C15—C14—C17	121.5 (6)
C2—C1—H1D	109.5	C13—C14—C17	121.3 (6)
H1B—C1—H1D	109.5	C16—C15—C14	121.2 (6)
H1C—C1—H1D	109.5	C16—C15—H15A	119.4
C3—C2—C7	116.7 (5)	C14—C15—H15A	119.4
C3—C2—C1	121.8 (5)	C15—C16—C11	121.2 (6)
C7—C2—C1	121.5 (5)	C15—C16—H16A	119.4
C2—C3—C4	122.0 (5)	C11—C16—H16A	119.4
C2—C3—H3B	119.0	C14—C17—H17A	109.5
C4—C3—H3B	119.0	C14—C17—H17B	109.5

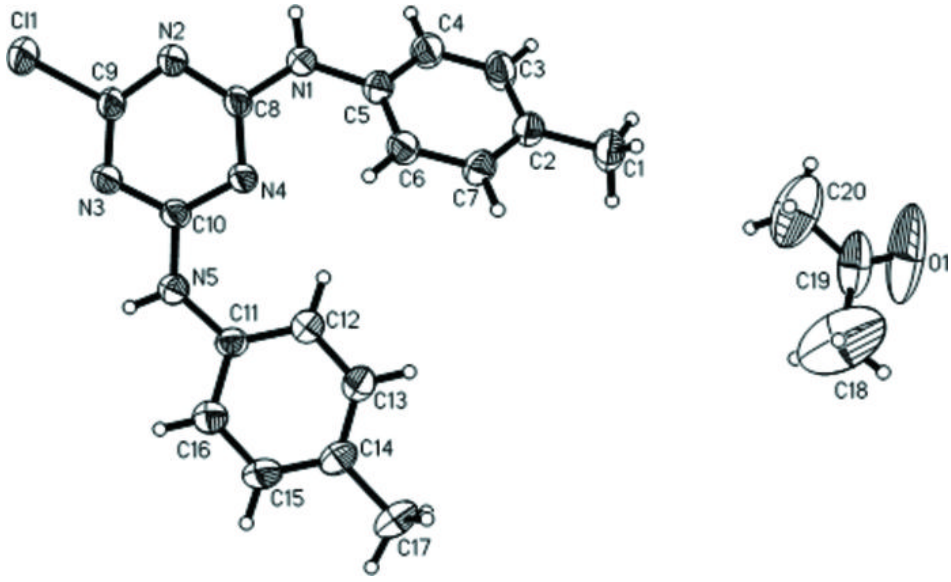
C3—C4—C5	120.2 (5)	H17A—C17—H17B	109.5
C3—C4—H4B	119.9	C14—C17—H17C	109.5
C5—C4—H4B	119.9	H17A—C17—H17C	109.5
C6—C5—C4	119.3 (5)	H17B—C17—H17C	109.5
C6—C5—N1	122.5 (5)	C19—C20—H20A	109.5
C4—C5—N1	118.1 (4)	C19—C20—H20B	109.5
C5—C6—C7	119.3 (5)	H20A—C20—H20B	109.5
C5—C6—H6A	120.3	C19—C20—H20C	109.5
C7—C6—H6A	120.3	H20A—C20—H20C	109.5
C6—C7—C2	122.4 (5)	H20B—C20—H20C	109.5
C6—C7—H7A	118.8	O1—C19—C20	126.1 (13)
C2—C7—H7A	118.8	O1—C19—C18	119.2 (13)
N1—C8—N2	115.1 (4)	C20—C19—C18	114.5 (10)
N1—C8—N4	119.5 (4)	C19—C18—H18A	109.5
N2—C8—N4	125.4 (4)	C19—C18—H18B	109.5
N3—C9—N2	129.6 (4)	H18A—C18—H18B	109.5
N3—C9—Cl1	114.7 (4)	C19—C18—H18C	109.5
N2—C9—Cl1	115.7 (4)	H18A—C18—H18C	109.5
N3—C10—N4	124.7 (4)	H18B—C18—H18C	109.5
N3—C10—N5	114.8 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···N3^{i}	0.86	2.16	3.018 (6)	176
N5—H5A···N2^{ii}	0.86	2.45	3.300 (6)	171
C4—H4B···O1^{iii}	0.93	2.36	3.264 (9)	165
C6—H6A···N4	0.93	2.59	2.999 (6)	107
C12—H12A···N4	0.93	2.30	2.912 (7)	123
C1—H1C···Cg1^{iv}	0.96	3.01	3.806 (2)	141
C17—H17C···Cg2^{v}	0.96	2.92	3.745 (2)	145

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

Fig. 1


Cl1
N2
C4
C9
N1
C3
C8
C5
C2
C1
C6
C7
N4
N3
C10
N5
C11
C12
C13
C16
C14
C15
C17
C20
C19
O1
C18