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Bis(triethylammonium) tetrachloridocobaltate(II)

Reza Azadbakht,[a]* Hassan Hadadzadeh[b] and Hadi Amiri Rudbari[c]

[a]Department of Chemistry, Payame Noor University, Tehran, Iran, [b]Department of Chemistry, Isfahan University of Technology, Isfahan 84156-83111, Iran, and [c]Dipartimento di Chimica Inorganica, Vill. S. Agata, Salita Sperone 31, Universita di Messina 98166 Messina, Italy
Correspondence e-mail: r_azadbakht@yahoo.com

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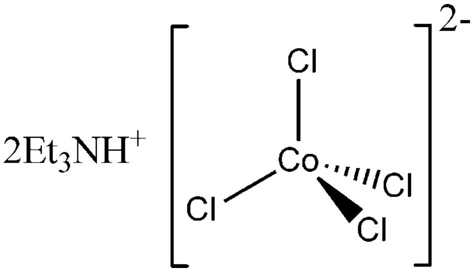
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.009 Å; R factor = 0.052; wR factor = 0.119; data-to-parameter ratio = 22.7.

The crystal structure of the title compound, $(C_6H_{16}N)_2[CoCl_4]$, is comprised of a tetrahedral $[CoCl_4]^{2-}$ anion and two independent triethylammonium cations. The latter are featureless while the $[CoCl_4]^{2-}$ anion exhibits typical Co—Cl bond lengths [2.2428 (15)–2.2847 (16) Å] and a Cl—Co—Cl angular range of 107.58 (6)–112.73 (7)°. In the crystal, N—H···Cl hydrogen bonds between the two crystallographically independent cations and the $[CoCl_4]^{2-}$ anion generate discrete ion triplets. The two Co—Cl bonds involved in these interactions are slightly longer than the remaining two.

Related literature

For the crystal structure of a related complex, see: Clegg & Martin (2007).



Experimental

Crystal data

$(C_6H_{16}N)_2[CoCl_4]$
M_r = 405.13
Orthorhombic, $Pbca$
a = 11.981 (5) Å
b = 13.226 (5) Å
c = 25.946 (10) Å
V = 4112 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 1.35 mm^{-1}
T = 296 K
0.18 × 0.14 × 0.13 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
T_{min} = 0.661, T_{max} = 0.745
42005 measured reflections
4090 independent reflections
2078 reflections with $I > 2\sigma(I)$
R_{int} = 0.117

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.119
S = 1.00
4090 reflections
180 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···Cl4	0.84 (4)	2.39 (5)	3.216 (6)	170 (4)
N2—H2···Cl1	0.90 (4)	2.34 (4)	3.214 (5)	163 (4)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

We are grateful to Payame Noor University (PNU) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2461).

References

Bruker (2007). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Clegg, W. & Martin, N. C. (2007). *Acta Cryst.* E**63**, m1151.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supplementary materials

Acta Cryst. (2012). E**68**, m859 [doi:10.1107/S1600536812022441]

Bis(triethylammonium) tetrachloridocobaltate(II)

Reza Azadbakht, Hassan Hadadzadeh and Hadi Amiri Rudbari

Comment

The crystal structure of the title compound, Bis (triethylammonium) tetrachloridocobalt (II), is comprised of a $[CoCl_4]^{2-}$ anion and two independent triethylammonium cations. The crystal structure of triethylammonium is similar to that of its analogue salts, Clegg and Martin (2007). The $[CoCl_4]^{2-}$ anion possesses typical Co—Cl bonds (range: 2.2428 (15) to 2.2847 (16) Å), while the Cl—Co—Cl angles range from 107.58 (6) to 112.73 (7)°. An *ORTEP* diagram of the asymmetric unit is presented in Fig. 1. In the crystal structure intermolecular N—H—Cl hydrogen bonds are observed between each of the two crystallographically independent cations and the tetrahedral $[CoCl_4]^{2-}$ anion that generate discrete ion triplets (Table 1). The Co—Cl bonds involving the hydrogen-bonded Cl atoms are slightly longer than the remaining two. The hydrogen bonds between the organic cations and the $[CoCl_4]^{2-}$ anions contribute to the stability of crystal packing (Fig. 2).

Experimental

The compound was obtained unexpectedly in an unsuccessful attempt to prepare a cobalt(II) macrocyclic Schiff-base complex. To 2,6-diformyl-4-chlorophenol (1 mmol) and $CoCl_2.6H_2O$ (2 mmol) in 25ml of MeOH, a methanolic solution (25 ml) of 1,2-bis (2′-aminophenoxy)benzene (1 mmol) and NEt_3 (1 mmol) was added dropwise. The resulting solution was stirred under reflux for 3 h. The precipitate obtained by partial evaporation of the solution was allowed it to stand overnight in refrigerator and was collected by filtration. Suitable crystals for the X-ray crystal structure determination were grown by recrystallization by diffusion of diethylether in the solution of the complex in acetonitrile

Refinement

N-bound H atoms were located in a difference map and refined isotropically. Other H atoms were positioned geometrically and refined with a riding model (including free rotation about C—C bonds), with $U_{iso}(H)$ = 1.2 (1.5 for methyl groups) times $U_{eq}(C)$. Atoms C5 and C6 from one of the ethyl branches of the N1 cationic groups show large displacement factors, much larger than neighbouring atoms and (due to libration effects) giving rise to a short, non realistic C-C $sp^3\cdots sp^3$ distance of 1.250 (9)Å.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

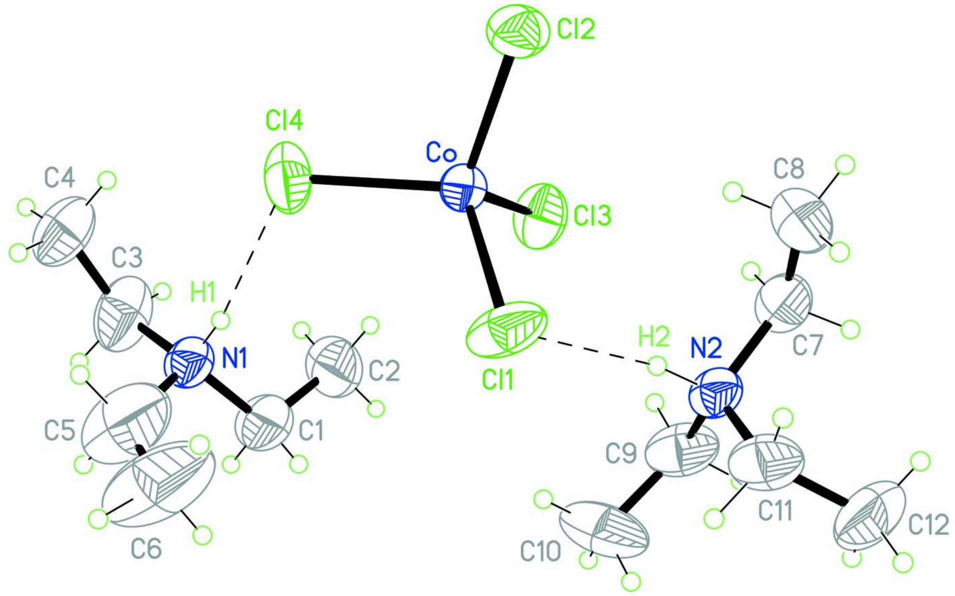


Figure 1

The asymmetric unit with atom labels and 50% probability ellipsoids for non-H atoms. Hydrogen bonds are shown as dashed lines.

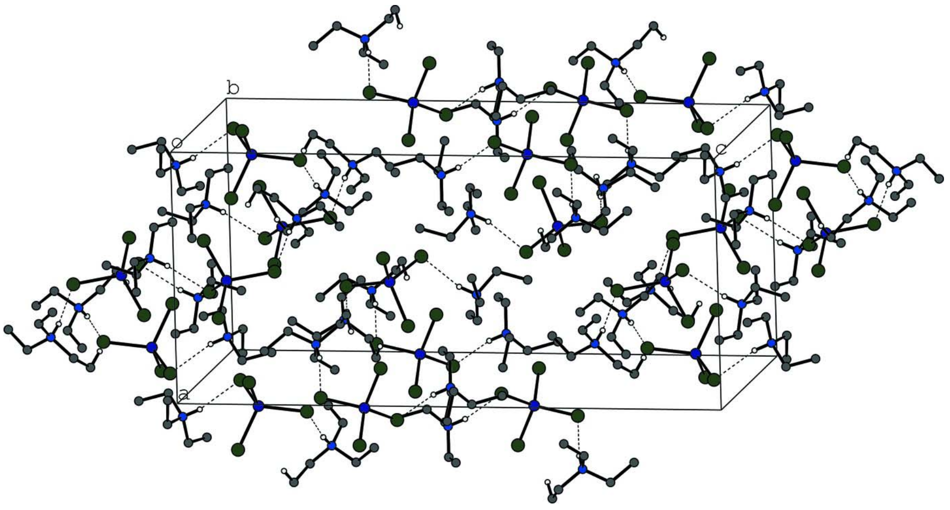


Figure 2

The packing, viewed along the *c* axis. Hydrogen bonds are shown as dashed lines, and H atoms not involved in hydrogen bonding have been omitted.

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Crystal data

$(C_6H_{16}N)_2[CoCl_4]$
$M_r = 405.13$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 11.981$ (5) Å
$b = 13.226$ (5) Å
$c = 25.946$ (10) Å
$V = 4112$ (3) Å^3
$Z = 8$
$F(000) = 1704$
$D_x = 1.309$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3697 reflections
$\theta = 2.3–19.2°$
$\mu = 1.35\ \text{mm}^{-1}$
$T = 296$ K
Irregular, green
$0.18 \times 0.14 \times 0.13$ mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2007)
$T_{\text{min}} = 0.661$, $T_{\text{max}} = 0.745$
42005 measured reflections
4090 independent reflections
2078 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.117$
$\theta_{\text{max}} = 26.1°$, $\theta_{\text{min}} = 1.6°$
$h = -14 \rightarrow 12$
$k = -16 \rightarrow 16$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.119$
$S = 1.00$
4090 reflections
180 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0298P)^2 + 6.2054P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\text{max}} < 0.001$
$\Delta\rho_{\text{max}} = 0.45$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.51$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
Co	0.05270 (5)	0.21711 (5)	0.12639 (2)	0.0501 (2)
Cl1	−0.02174 (14)	0.34750 (11)	0.08048 (6)	0.0963 (6)
Cl2	−0.06848 (12)	0.08799 (10)	0.12358 (5)	0.0758 (4)
Cl4	0.08198 (12)	0.25981 (12)	0.21063 (5)	0.0840 (5)
Cl3	0.21929 (11)	0.17851 (12)	0.09060 (5)	0.0786 (4)
N1	0.2550 (4)	0.4391 (4)	0.23571 (17)	0.0634 (12)
N2	0.1187 (4)	0.3092 (3)	−0.02326 (17)	0.0593 (12)
C2	0.3882 (5)	0.3716 (5)	0.1714 (2)	0.106 (2)
H2A	0.4369	0.3915	0.1438	0.159*
H2B	0.3332	0.3252	0.1585	0.159*
H2C	0.4310	0.3393	0.1980	0.159*
C1	0.3327 (6)	0.4609 (5)	0.1925 (2)	0.098 (2)
H1A	0.2913	0.4938	0.1651	0.118*

H1B	0.3891	0.5080	0.2044	0.118*
C3	0.3132 (5)	0.4070 (6)	0.2830 (2)	0.106 (2)
H3A	0.3725	0.3609	0.2734	0.127*
H3B	0.3478	0.4660	0.2985	0.127*
C4	0.2438 (5)	0.3579 (5)	0.3221 (2)	0.099 (2)
H4A	0.2892	0.3396	0.3511	0.149*
H4B	0.2104	0.2982	0.3078	0.149*
H4C	0.1862	0.4036	0.3331	0.149*
C6	0.1254 (8)	0.5774 (8)	0.2169 (4)	0.207 (6)
H6A	0.0886	0.6314	0.2349	0.311*
H6B	0.0705	0.5342	0.2013	0.311*
H6C	0.1727	0.6051	0.1906	0.311*
C5	0.1830 (8)	0.5274 (7)	0.2479 (4)	0.188 (5)
H5A	0.1314	0.5041	0.2742	0.225*
H5B	0.2315	0.5760	0.2647	0.225*
C10	0.2315 (6)	0.4566 (4)	0.0047 (3)	0.121 (3)
H10A	0.3066	0.4816	0.0066	0.182*
H10B	0.2003	0.4532	0.0387	0.182*
H10C	0.1874	0.5013	−0.0161	0.182*
C9	0.2319 (5)	0.3537 (5)	−0.0188 (2)	0.0868 (18)
H9A	0.2651	0.3575	−0.0528	0.104*
H9B	0.2780	0.3093	0.0020	0.104*
C7	0.1263 (5)	0.2003 (4)	−0.0402 (2)	0.0769 (17)
H7A	0.1468	0.1980	−0.0764	0.092*
H7B	0.1847	0.1669	−0.0207	0.092*
C8	0.0190 (5)	0.1446 (4)	−0.0327 (2)	0.095 (2)
H8A	0.0279	0.0758	−0.0437	0.143*
H8B	−0.0386	0.1763	−0.0527	0.143*
H8C	−0.0011	0.1460	0.0031	0.143*
C12	0.0734 (7)	0.3786 (5)	−0.1106 (2)	0.137 (3)
H12A	0.0194	0.4182	−0.1291	0.206*
H12B	0.0776	0.3122	−0.1254	0.206*
H12C	0.1451	0.4107	−0.1128	0.206*
C11	0.0390 (5)	0.3705 (4)	−0.0550 (2)	0.0858 (18)
H11A	0.0336	0.4379	−0.0405	0.103*
H11B	−0.0344	0.3398	−0.0532	0.103*
H2	0.089 (3)	0.310 (3)	0.0084 (17)	0.052 (14)*
H1	0.212 (4)	0.394 (4)	0.2253 (18)	0.055 (17)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co	0.0547 (4)	0.0482 (4)	0.0476 (3)	−0.0002 (3)	0.0041 (3)	−0.0008 (3)
Cl1	0.1149 (13)	0.0708 (10)	0.1032 (12)	0.0411 (9)	0.0525 (10)	0.0382 (9)
Cl2	0.0832 (10)	0.0699 (9)	0.0741 (9)	−0.0231 (8)	−0.0188 (8)	0.0107 (7)
Cl4	0.0758 (9)	0.1138 (12)	0.0623 (8)	−0.0229 (9)	0.0088 (7)	−0.0328 (8)
Cl3	0.0671 (9)	0.0994 (11)	0.0693 (9)	0.0168 (8)	0.0126 (7)	−0.0066 (8)
N1	0.064 (3)	0.067 (3)	0.059 (3)	0.001 (3)	0.015 (3)	0.003 (2)
N2	0.067 (3)	0.057 (3)	0.053 (3)	0.008 (2)	0.020 (2)	0.005 (2)
C2	0.116 (5)	0.098 (5)	0.104 (5)	−0.016 (4)	0.050 (4)	−0.020 (4)

C1	0.116 (5)	0.089 (5)	0.091 (4)	−0.009 (4)	0.053 (4)	0.002 (4)
C3	0.079 (4)	0.166 (7)	0.072 (4)	−0.027 (5)	−0.013 (4)	−0.005 (4)
C4	0.088 (4)	0.143 (6)	0.066 (4)	−0.001 (4)	−0.009 (4)	0.022 (4)
C6	0.188 (10)	0.214 (11)	0.220 (11)	0.125 (9)	0.093 (8)	0.125 (9)
C5	0.222 (11)	0.164 (8)	0.178 (9)	0.113 (8)	0.118 (8)	0.064 (7)
C10	0.103 (5)	0.063 (4)	0.199 (8)	−0.020 (4)	0.031 (5)	−0.003 (5)
C9	0.067 (4)	0.084 (5)	0.109 (5)	0.001 (3)	0.027 (4)	0.026 (4)
C7	0.099 (5)	0.067 (4)	0.065 (3)	0.029 (3)	0.012 (3)	0.001 (3)
C8	0.118 (5)	0.077 (4)	0.091 (4)	−0.010 (4)	−0.013 (4)	−0.013 (4)
C12	0.220 (9)	0.112 (6)	0.080 (5)	0.032 (6)	−0.019 (5)	0.036 (4)
C11	0.088 (4)	0.062 (4)	0.107 (5)	0.023 (3)	0.003 (4)	0.017 (3)

Geometric parameters (Å, º)

Co—Cl2	2.2428 (15)	C6—C5	1.250 (9)
Co—Cl3	2.2597 (16)	C6—H6A	0.9600
Co—Cl1	2.2778 (16)	C6—H6B	0.9600
Co—Cl4	2.2847 (16)	C6—H6C	0.9600
N1—C3	1.474 (7)	C5—H5A	0.9700
N1—C1	1.486 (6)	C5—H5B	0.9700
N1—C5	1.486 (8)	C10—C9	1.492 (8)
N1—H1	0.84 (4)	C10—H10A	0.9600
N2—C9	1.483 (7)	C10—H10B	0.9600
N2—C11	1.498 (6)	C10—H10C	0.9600
N2—C7	1.508 (6)	C9—H9A	0.9700
N2—H2	0.90 (4)	C9—H9B	0.9700
C2—C1	1.462 (7)	C7—C8	1.494 (7)
C2—H2A	0.9600	C7—H7A	0.9700
C2—H2B	0.9600	C7—H7B	0.9700
C2—H2C	0.9600	C8—H8A	0.9600
C1—H1A	0.9700	C8—H8B	0.9600
C1—H1B	0.9700	C8—H8C	0.9600
C3—C4	1.464 (7)	C12—C11	1.503 (8)
C3—H3A	0.9700	C12—H12A	0.9600
C3—H3B	0.9700	C12—H12B	0.9600
C4—H4A	0.9600	C12—H12C	0.9600
C4—H4B	0.9600	C11—H11A	0.9700
C4—H4C	0.9600	C11—H11B	0.9700
Cl2—Co—Cl3	112.73 (7)	C5—C6—H6C	109.5
Cl2—Co—Cl1	107.82 (7)	H6A—C6—H6C	109.5
Cl3—Co—Cl1	107.58 (6)	H6B—C6—H6C	109.5
Cl2—Co—Cl4	108.58 (6)	C6—C5—N1	126.8 (9)
Cl3—Co—Cl4	108.26 (6)	C6—C5—H5A	105.6
Cl1—Co—Cl4	111.92 (7)	N1—C5—H5A	105.6
C3—N1—C1	112.8 (5)	C6—C5—H5B	105.6
C3—N1—C5	108.9 (6)	N1—C5—H5B	105.6
C1—N1—C5	111.8 (5)	H5A—C5—H5B	106.1
C3—N1—H1	111 (3)	C9—C10—H10A	109.5
C1—N1—H1	106 (3)	C9—C10—H10B	109.5

C5—N1—H1	106 (3)	H10A—C10—H10B	109.5
C9—N2—C11	114.3 (4)	C9—C10—H10C	109.5
C9—N2—C7	110.2 (4)	H10A—C10—H10C	109.5
C11—N2—C7	113.2 (4)	H10B—C10—H10C	109.5
C9—N2—H2	107 (3)	N2—C9—C10	113.0 (5)
C11—N2—H2	104 (3)	N2—C9—H9A	109.0
C7—N2—H2	107 (3)	C10—C9—H9A	109.0
C1—C2—H2A	109.5	N2—C9—H9B	109.0
C1—C2—H2B	109.5	C10—C9—H9B	109.0
H2A—C2—H2B	109.5	H9A—C9—H9B	107.8
C1—C2—H2C	109.5	C8—C7—N2	112.4 (4)
H2A—C2—H2C	109.5	C8—C7—H7A	109.1
H2B—C2—H2C	109.5	N2—C7—H7A	109.1
C2—C1—N1	114.3 (5)	C8—C7—H7B	109.1
C2—C1—H1A	108.7	N2—C7—H7B	109.1
N1—C1—H1A	108.7	H7A—C7—H7B	107.9
C2—C1—H1B	108.7	C7—C8—H8A	109.5
N1—C1—H1B	108.7	C7—C8—H8B	109.5
H1A—C1—H1B	107.6	H8A—C8—H8B	109.5
C4—C3—N1	115.8 (5)	C7—C8—H8C	109.5
C4—C3—H3A	108.3	H8A—C8—H8C	109.5
N1—C3—H3A	108.3	H8B—C8—H8C	109.5
C4—C3—H3B	108.3	C11—C12—H12A	109.5
N1—C3—H3B	108.3	C11—C12—H12B	109.5
H3A—C3—H3B	107.4	H12A—C12—H12B	109.5
C3—C4—H4A	109.5	C11—C12—H12C	109.5
C3—C4—H4B	109.5	H12A—C12—H12C	109.5
H4A—C4—H4B	109.5	H12B—C12—H12C	109.5
C3—C4—H4C	109.5	N2—C11—C12	113.1 (5)
H4A—C4—H4C	109.5	N2—C11—H11A	109.0
H4B—C4—H4C	109.5	C12—C11—H11A	109.0
C5—C6—H6A	109.5	N2—C11—H11B	109.0
C5—C6—H6B	109.5	C12—C11—H11B	109.0
H6A—C6—H6B	109.5	H11A—C11—H11B	107.8

C3—N1—C1—C2	69.4 (8)	C11—N2—C9—C10	59.1 (7)
C5—N1—C1—C2	−167.5 (7)	C7—N2—C9—C10	−172.1 (5)
C1—N1—C3—C4	−163.6 (6)	C9—N2—C7—C8	165.6 (5)
C5—N1—C3—C4	71.6 (8)	C11—N2—C7—C8	−65.0 (6)
C3—N1—C5—C6	176.5 (11)	C9—N2—C11—C12	64.2 (7)
C1—N1—C5—C6	51.2 (14)	C7—N2—C11—C12	−63.1 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···Cl4	0.84 (4)	2.39 (5)	3.216 (6)	170 (4)
N2—H2···Cl1	0.90 (4)	2.34 (4)	3.214 (5)	163 (4)