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N-Methyl-4-nitroanilinium chloride

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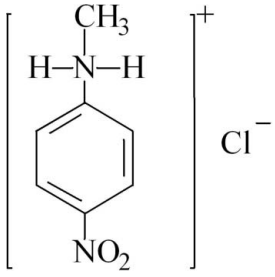
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.124; data-to-parameter ratio = 18.1.

The asymmetric unit of the title salt, $C_7H_9N_2O_2^+\cdot Cl^-$, contains two independent cations and anions. In the crystal, each *N*-methyl-4-nitroanilinium cation is linked to two Cl^- anions *via* N—H···Cl hydrogen bonds. π–π stacking is observed between the benzene rings of adjacent cations [centroid-to-centroid distances = 3.7684 (14) and 3.7917 (7) Å].

Related literature

For applications of *N*-methyl-4-nitrobenzenamine, see: Bellamy & Sammour (1993); Sammour (1994); Williams & Friedlander (2000); Davies & Provatas (2006).



Experimental

Crystal data

$C_7H_9N_2O_2^+\cdot Cl^-$
M_r = 188.61
Monoclinic, $P2_1/c$
a = 7.0509 (14) Å
b = 19.120 (4) Å
c = 13.443 (3) Å
β = 95.20 (3)°
V = 1804.8 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.39 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.12 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.927, T_{max} = 0.955
17869 measured reflections
4282 independent reflections
3022 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.124
S = 1.03
4282 reflections
236 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl2^{i}	0.90 (1)	2.18 (1)	3.0702 (17)	170 (2)
N2—H2B···Cl1^{i}	0.89 (1)	2.16 (1)	3.0482 (19)	173 (2)
N4—H4A···Cl2^{ii}	0.90 (1)	2.15 (1)	3.0361 (18)	168 (2)
N4—H4B···Cl1	0.89 (1)	2.26 (1)	3.1157 (18)	163 (2)

Symmetry codes: (i) $-x+1, y-\frac{1}{2}, -z+\frac{3}{2}$; (ii) $-x, -y+1, -z+1$.

Data collection: *RAPID-AUTO* (Rigaku, 2000); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: XU5532).

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supplementary materials

Acta Cryst. (2012). E**68**, o1973 [doi:10.1107/S1600536812024117]

N-Methyl-4-nitroanilinium chloride

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Comment

As a stablizer, *N*-methyl-4-nitrobenzenamine is used in order to lengthen the useful service life of double-base and minimum smoke propellants (Bellamy & Sammour, 1993; Sammour, 1994; Williams & Friedlander, 2000). As a important ingredient, *N*-methyl-4-nitrobenzenamine can improve melt-cast explosive systems mechanical properties (Davies & Provatas, 2006). In order to research reaction properties, here we report the synthesis and the crystal structure of the title compound (Fig. 1).

The title compound consists of a *N*-(4-nitrophenyl)-methylammonium cation and a chloride anion. The hydrochloric acid deprotonated and the *N*-methyl-4-nitrobenzenamine accepts the proton to produce the protonated organic cation, namely *N*-(4-nitrophenyl)-methylammonium chloride. In the crystal structure, contains two cations and two anions. They are linked by N—H···Cl hydrogen bonds to form a three-dimensional complex network.

Experimental

The title compound was synthesized by *N*-methyl-4-nitrobenzenamine and concentrated hydrochloric acid in acetone at room temperature. Single crystals suitable for X-ray diffraction were obtained by evaporation of a solution of the title compound in acetone at room temperature.

Refinement

H atoms bonded to N atoms were located in a difference Fourier map and refined isotropically with bond restraint of N—H = 0.89 (2) Å. Other H atoms were positioned geometrically and treated as riding with C—H = 0.93–0.96 Å, and refined as riding with U_{iso}(H) = 1.2–1.5U_{eq}(C).

Computing details

Data collection: *RAPID-AUTO* (Rigaku, 2000); cell refinement: *RAPID-AUTO* (Rigaku, 2000); data reduction: *CrystalStructure* (Rigaku/MSC, 2000); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

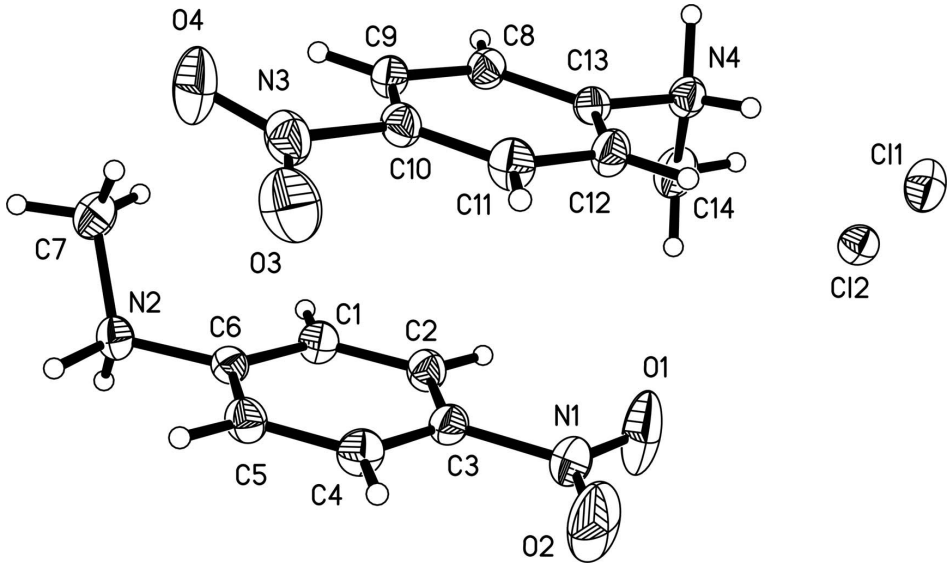

Figure 1

The molecular structure of the title compound.

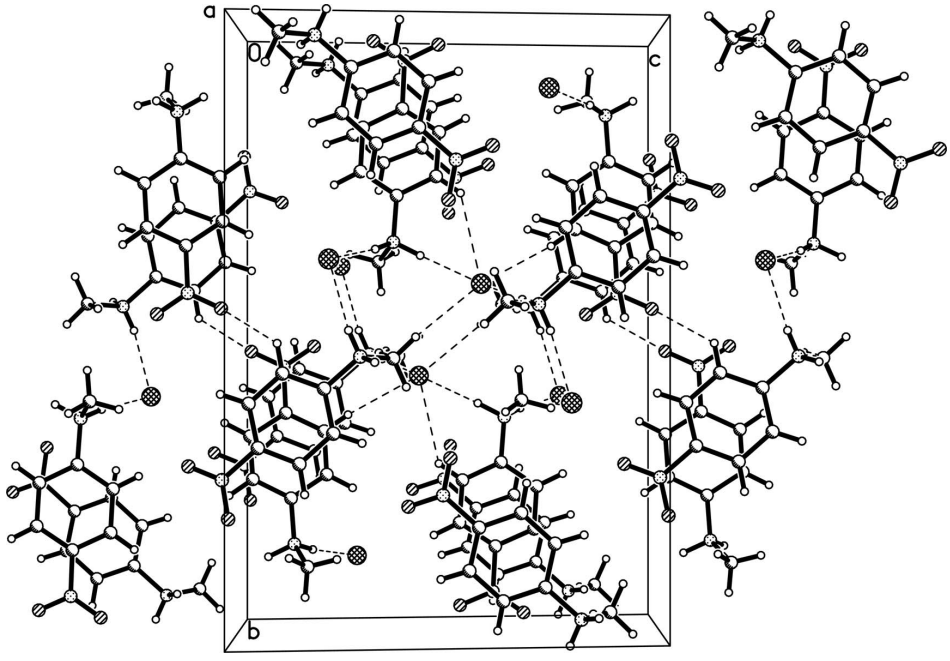

Figure 2

The packing of the title compound.

N-Methyl-4-nitroanilinium chloride

Crystal data

$C_7H_9N_2O_2^+\cdot Cl^-$
M_r = 188.61
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 7.0509 (14) Å
b = 19.120 (4) Å
c = 13.443 (3) Å
β = 95.20 (3)°
V = 1804.8 (6) Å^3
Z = 8

$F(000) = 784$
$D_x = 1.388$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4042 reflections
$\theta = 2.6–27.9°$
$\mu = 0.39$ mm^{-1}
$T = 293$ K
Block, yellow
0.20 × 0.20 × 0.12 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.927$, $T_{max} = 0.955$
17869 measured reflections
4282 independent reflections
3022 reflections with $I > 2\sigma(I)$
$R_{int} = 0.051$
$\theta_{max} = 27.9°$, $\theta_{min} = 2.6°$
$h = -9\rightarrow9$
$k = -25\rightarrow25$
$l = -17\rightarrow12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.124$
$S = 1.03$
4282 reflections
236 parameters
4 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0651P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.24$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.073 (4)

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}
O1	0.5920 (3)	0.47358 (8)	0.82213 (13)	0.0978 (7)
O2	0.6814 (3)	0.46456 (9)	0.97433 (15)	0.1113 (9)
O3	0.2318 (3)	0.28345 (10)	1.09445 (12)	0.0814 (6)
O4	0.1062 (3)	0.20296 (9)	0.99778 (13)	0.0869 (6)
N1	0.6303 (3)	0.43952 (9)	0.89526 (14)	0.0532 (5)
N2	0.5910 (2)	0.14490 (8)	0.86838 (12)	0.0408 (4)
N3	0.1557 (2)	0.26321 (10)	1.01448 (14)	0.0542 (5)
N4	0.0461 (2)	0.45901 (8)	0.70054 (11)	0.0400 (4)
C1	0.5619 (3)	0.26091 (10)	0.79113 (13)	0.0405 (4)
H1	0.5343	0.2393	0.7295	0.049*

C2	0.5707 (3)	0.33269 (10)	0.79740 (13)	0.0414 (4)
H2	0.5485	0.3603	0.7406	0.050*
C3	0.6131 (2)	0.36260 (9)	0.88971 (14)	0.0394 (4)
C4	0.6468 (3)	0.32360 (10)	0.97592 (14)	0.0442 (5)
H4	0.6757	0.3453	1.0374	0.053*
C5	0.6365 (3)	0.25178 (10)	0.96866 (13)	0.0416 (4)
H5	0.6580	0.2242	1.0256	0.050*
C6	0.5942 (2)	0.22099 (9)	0.87655 (12)	0.0351 (4)
C7	0.4023 (3)	0.11454 (11)	0.83074 (18)	0.0602 (6)
H7A	0.3053	0.1324	0.8696	0.090*
H7B	0.4074	0.0645	0.8365	0.090*
H7C	0.3732	0.1272	0.7620	0.090*
C8	0.0468 (3)	0.33800 (9)	0.76075 (13)	0.0388 (4)
H8	0.0125	0.3233	0.6956	0.047*
C9	0.0732 (3)	0.28977 (9)	0.83732 (14)	0.0411 (4)
H9	0.0568	0.2422	0.8251	0.049*
C10	0.1243 (3)	0.31429 (10)	0.93243 (13)	0.0402 (4)
C11	0.1482 (3)	0.38398 (10)	0.95434 (14)	0.0474 (5)
H11	0.1817	0.3987	1.0195	0.057*
C12	0.1211 (3)	0.43201 (10)	0.87722 (13)	0.0423 (4)
H12	0.1360	0.4796	0.8898	0.051*
C13	0.0720 (2)	0.40829 (9)	0.78202 (12)	0.0347 (4)
C14	0.1958 (3)	0.45515 (11)	0.62862 (15)	0.0557 (6)
H14A	0.3184	0.4640	0.6634	0.084*
H14B	0.1701	0.4896	0.5772	0.084*
H14C	0.1948	0.4094	0.5991	0.084*
Cl1	0.10531 (8)	0.61298 (3)	0.77271 (4)	0.05256 (19)
Cl2	0.31979 (7)	0.57619 (3)	0.42629 (3)	0.04808 (18)
H2A	0.632 (3)	0.1249 (11)	0.9271 (10)	0.062 (7)*
H2B	0.684 (2)	0.1328 (12)	0.8311 (15)	0.075 (8)*
H4A	−0.0708 (18)	0.4519 (12)	0.6697 (15)	0.068 (7)*
H4B	0.041 (3)	0.5011 (7)	0.7277 (15)	0.064 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.187 (2)	0.0365 (10)	0.0681 (12)	−0.0036 (11)	0.0040 (12)	0.0119 (8)
O2	0.181 (2)	0.0497 (11)	0.0926 (13)	−0.0070 (12)	−0.0467 (15)	−0.0217 (10)
O3	0.1046 (14)	0.0890 (14)	0.0489 (10)	0.0101 (11)	−0.0028 (9)	0.0215 (9)
O4	0.1261 (17)	0.0515 (11)	0.0827 (12)	0.0016 (10)	0.0070 (11)	0.0284 (9)
N1	0.0590 (11)	0.0368 (10)	0.0633 (12)	−0.0019 (8)	0.0029 (9)	−0.0075 (9)
N2	0.0475 (10)	0.0353 (9)	0.0395 (9)	0.0044 (7)	0.0038 (7)	0.0004 (7)
N3	0.0558 (11)	0.0538 (12)	0.0544 (11)	0.0097 (9)	0.0121 (9)	0.0178 (9)
N4	0.0442 (9)	0.0350 (9)	0.0396 (9)	−0.0033 (7)	−0.0021 (7)	0.0040 (7)
C1	0.0491 (11)	0.0400 (11)	0.0317 (9)	−0.0016 (8)	0.0002 (8)	−0.0022 (7)
C2	0.0455 (11)	0.0402 (11)	0.0380 (10)	0.0005 (8)	0.0019 (8)	0.0049 (8)
C3	0.0381 (10)	0.0339 (10)	0.0463 (11)	−0.0009 (7)	0.0047 (8)	−0.0031 (8)
C4	0.0525 (12)	0.0453 (11)	0.0341 (10)	−0.0021 (9)	−0.0003 (8)	−0.0069 (8)
C5	0.0477 (11)	0.0428 (11)	0.0337 (9)	0.0026 (8)	0.0001 (8)	0.0035 (8)
C6	0.0331 (9)	0.0339 (9)	0.0381 (10)	0.0019 (7)	0.0026 (7)	−0.0008 (7)

C7	0.0611 (14)	0.0381 (12)	0.0789 (16)	−0.0080 (10)	−0.0066 (12)	−0.0052 (10)
C8	0.0414 (10)	0.0345 (10)	0.0400 (10)	−0.0010 (8)	0.0009 (8)	−0.0033 (8)
C9	0.0399 (10)	0.0312 (10)	0.0526 (12)	0.0011 (7)	0.0065 (8)	0.0024 (8)
C10	0.0398 (10)	0.0397 (11)	0.0416 (10)	0.0034 (8)	0.0067 (8)	0.0108 (8)
C11	0.0588 (13)	0.0462 (12)	0.0364 (10)	0.0007 (9)	−0.0010 (9)	0.0004 (8)
C12	0.0534 (12)	0.0314 (10)	0.0408 (10)	−0.0031 (8)	−0.0028 (8)	−0.0024 (8)
C13	0.0332 (9)	0.0340 (9)	0.0366 (9)	−0.0004 (7)	0.0015 (7)	0.0048 (7)
C14	0.0711 (15)	0.0500 (13)	0.0483 (12)	−0.0070 (10)	0.0177 (10)	0.0066 (10)
Cl1	0.0642 (4)	0.0381 (3)	0.0568 (3)	−0.0017 (2)	0.0129 (2)	0.0025 (2)
Cl2	0.0535 (3)	0.0514 (3)	0.0381 (3)	−0.0033 (2)	−0.0030 (2)	−0.0018 (2)

Geometric parameters (Å, º)

O1—N1	1.190 (2)	C4—C5	1.378 (3)
O2—N1	1.191 (2)	C4—H4	0.9300
O3—N3	1.220 (2)	C5—C6	1.379 (2)
O4—N3	1.218 (2)	C5—H5	0.9300
N1—C3	1.477 (2)	C7—H7A	0.9600
N2—C6	1.459 (2)	C7—H7B	0.9600
N2—C7	1.497 (3)	C7—H7C	0.9600
N2—H2A	0.901 (9)	C8—C9	1.382 (2)
N2—H2B	0.891 (10)	C8—C13	1.382 (2)
N3—C10	1.475 (2)	C8—H8	0.9300
N4—C13	1.462 (2)	C9—C10	1.379 (3)
N4—C14	1.496 (3)	C9—H9	0.9300
N4—H4A	0.898 (10)	C10—C11	1.372 (3)
N4—H4B	0.885 (10)	C11—C12	1.385 (3)
C1—C2	1.376 (3)	C11—H11	0.9300
C1—C6	1.380 (2)	C12—C13	1.372 (2)
C1—H1	0.9300	C12—H12	0.9300
C2—C3	1.374 (2)	C14—H14A	0.9600
C2—H2	0.9300	C14—H14B	0.9600
C3—C4	1.380 (3)	C14—H14C	0.9600
O1—N1—O2	123.02 (19)	C5—C6—C1	121.13 (16)
O1—N1—C3	119.47 (17)	C5—C6—N2	119.60 (15)
O2—N1—C3	117.52 (18)	C1—C6—N2	119.23 (15)
C6—N2—C7	114.80 (14)	N2—C7—H7A	109.5
C6—N2—H2A	110.9 (14)	N2—C7—H7B	109.5
C7—N2—H2A	109.6 (14)	H7A—C7—H7B	109.5
C6—N2—H2B	107.1 (16)	N2—C7—H7C	109.5
C7—N2—H2B	112.7 (16)	H7A—C7—H7C	109.5
H2A—N2—H2B	101 (2)	H7B—C7—H7C	109.5
O4—N3—O3	123.90 (19)	C9—C8—C13	119.26 (16)
O4—N3—C10	117.86 (18)	C9—C8—H8	120.4
O3—N3—C10	118.23 (19)	C13—C8—H8	120.4
C13—N4—C14	113.82 (15)	C10—C9—C8	118.03 (17)
C13—N4—H4A	107.0 (14)	C10—C9—H9	121.0
C14—N4—H4A	111.5 (14)	C8—C9—H9	121.0
C13—N4—H4B	107.5 (14)	C11—C10—C9	123.10 (17)

C14—N4—H4B	111.6 (15)	C11—C10—N3	118.39 (17)
H4A—N4—H4B	104.9 (19)	C9—C10—N3	118.50 (17)
C2—C1—C6	119.82 (16)	C10—C11—C12	118.52 (17)
C2—C1—H1	120.1	C10—C11—H11	120.7
C6—C1—H1	120.1	C12—C11—H11	120.7
C3—C2—C1	118.37 (16)	C13—C12—C11	119.01 (17)
C3—C2—H2	120.8	C13—C12—H12	120.5
C1—C2—H2	120.8	C11—C12—H12	120.5
C2—C3—C4	122.68 (17)	C12—C13—C8	122.08 (16)
C2—C3—N1	118.04 (16)	C12—C13—N4	118.90 (16)
C4—C3—N1	119.23 (16)	C8—C13—N4	119.02 (15)
C5—C4—C3	118.36 (16)	N4—C14—H14A	109.5
C5—C4—H4	120.8	N4—C14—H14B	109.5
C3—C4—H4	120.8	H14A—C14—H14B	109.5
C4—C5—C6	119.64 (16)	N4—C14—H14C	109.5
C4—C5—H5	120.2	H14A—C14—H14C	109.5
C6—C5—H5	120.2	H14B—C14—H14C	109.5
C6—C1—C2—C3	0.4 (3)	C13—C8—C9—C10	−0.1 (3)
C1—C2—C3—C4	−0.1 (3)	C8—C9—C10—C11	0.7 (3)
C1—C2—C3—N1	177.47 (17)	C8—C9—C10—N3	−179.08 (16)
O1—N1—C3—C2	6.2 (3)	O4—N3—C10—C11	168.9 (2)
O2—N1—C3—C2	−174.3 (2)	O3—N3—C10—C11	−12.1 (3)
O1—N1—C3—C4	−176.2 (2)	O4—N3—C10—C9	−11.3 (3)
O2—N1—C3—C4	3.3 (3)	O3—N3—C10—C9	167.65 (18)
C2—C3—C4—C5	−0.3 (3)	C9—C10—C11—C12	−0.5 (3)
N1—C3—C4—C5	−177.77 (17)	N3—C10—C11—C12	179.22 (17)
C3—C4—C5—C6	0.3 (3)	C10—C11—C12—C13	−0.1 (3)
C4—C5—C6—C1	0.0 (3)	C11—C12—C13—C8	0.6 (3)
C4—C5—C6—N2	177.53 (17)	C11—C12—C13—N4	−179.17 (17)
C2—C1—C6—C5	−0.4 (3)	C9—C8—C13—C12	−0.5 (3)
C2—C1—C6—N2	−177.88 (17)	C9—C8—C13—N4	179.30 (16)
C7—N2—C6—C5	118.4 (2)	C14—N4—C13—C12	111.3 (2)
C7—N2—C6—C1	−64.1 (2)	C14—N4—C13—C8	−68.5 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···Cl2^{i}	0.90 (1)	2.18 (1)	3.0702 (17)	170 (2)
N2—H2*B*···Cl1^{i}	0.89 (1)	2.16 (1)	3.0482 (19)	173 (2)
N4—H4*A*···Cl2^{ii}	0.90 (1)	2.15 (1)	3.0361 (18)	168 (2)
N4—H4*B*···Cl1	0.89 (1)	2.26 (1)	3.1157 (18)	163 (2)

Symmetry codes: (i) −*x*+1, *y*−1/2, −*z*+3/2; (ii) −*x*, −*y*+1, −*z*+1.