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4-Methylanilinium trichloroacetate

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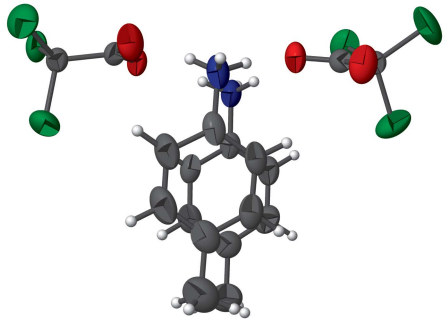
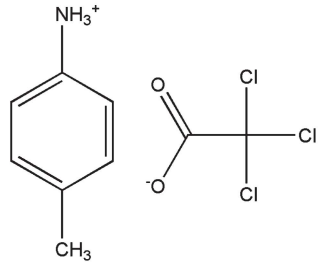
The asymmetric unit of the title molecular salt, $C_7H_{10}N^+\cdot C_2Cl_3O_2^-$, consists of two cations and two anions. In the crystal, N—H···O hydrogen bonds link the components into [100] chains incorporating $R_2^3(10)$ loops and weak π–π stacking [centroid-to-centroid distance = 3.865 (2) Å] is also observed.

3D view



Chemical scheme



Structure description

We herewith report the synthesis and the crystal structure of the title molecular salt. Its geometric parameters agree well with those for reported similar structures (Babu *et al.*, 2014; Benali-Cherif *et al.*, 2009; Kalaiyarasi *et al.*, 2017).

The asymmetric unit of the title compound (Fig. 1) comprises a pair of 4-methylanilinium cations and trichloroacetate anions. The dihedral angle between the benzene rings of the cations is 6.32 (1)°. Within the chosen asymmetric unit, N1—H1*A*···O4, N1—H1*C*···O2, N2—H2*B*···O3 and N2—H2*C*···O2 hydrogen bonds link the components, thereby generating an $R_2^3(10)$ loop and when symmetry-generated N2—H2*A*···O4^{ii}, N1—H1*B*···O1^{i} and N2—H2*C*···O2 hydrogen bonds are considered, another $R_2^3(10)$ loop is generated (Fig. 2, Table 1). The overall result is a supramolecular chain propagating along the *a*-axis direction (Fig. 3). The packing is further consolidated by weak π–π interactions [*Cg*1···*Cg*2 = 3.865 (2) Å; *Cg*1 and *Cg*2 are the centroids of the C1–C6 and C8–C13 rings, respectively].

Synthesis and crystallization

p-Toluidine (1.33 g) and trichloroacetic acid (1.48 g) were taken in a 1:1 ratio and dissolved in water at room temperature and the solution was stirred for 6 h. It was filtered and kept for slow evaporation and colourless blocks were obtained after four weeks.

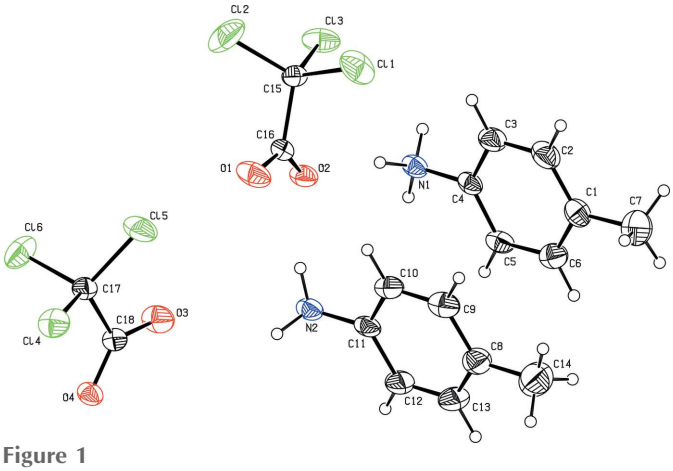


Figure 1
The molecular structure with 30% probability displacement ellipsoids.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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References

Babu, K. S. S., Peramaiyan, G., NizamMohideen, M. & Mohan, R. (2014). *Acta Cryst.* E**70**, o391–o392.
Benali-Cherif, N., Boussekine, H., Boutobba, Z. & Dadda, N. (2009). *Acta Cryst.* E**65**, o2744.
Bruker (2004). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Kalaiyarasi, S., Suresh, S., Akilan, R., Kumar, R. M. & Chakkaravarthi, G. (2017). *IUCrData*, **2**, x170254.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O4	0.89	1.92	2.801 (3)	169
N1—H1B···O1^{i}	0.89	1.91	2.796 (3)	172
N1—H1C···O2	0.89	1.96	2.834 (3)	167
N2—H2A···O4^{ii}	0.89	1.98	2.851 (3)	165
N2—H2B···O3	0.89	1.86	2.745 (3)	172
N2—H2C···O2	0.89	1.92	2.789 (3)	164

Symmetry codes: (i) $x-1, y, z$; (ii) $x+1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_7H_{10}N^+\cdot C_2Cl_3O_2^-$
M_r	270.53
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	295
a, b, c (Å)	6.7395 (2), 11.3491 (3), 16.1078 (5)
α, β, γ (°)	75.681 (2), 88.031 (2), 86.856 (2)
V (Å^3)	1191.69 (6)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	6.82
Crystal size (mm)	0.26 × 0.22 × 0.18
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.386, 0.754
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	37675, 4685, 3713
R_{int}	0.066
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.619
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.051, 0.128, 1.02
No. of reflections	4685
No. of parameters	275
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.48

Computer programs: *APEX2* (Bruker, 2004), *SAINT* (Bruker, 2004), *SHELXT2016/6* (Sheldrick, 2015*a*), *SHELXL2016/6* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2009).

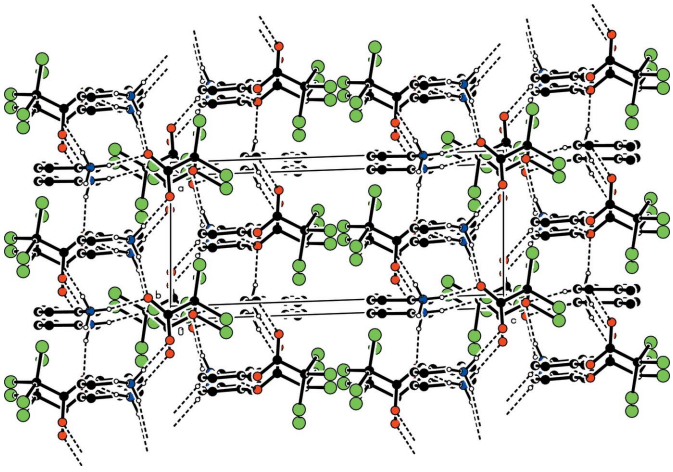

Figure 2
The crystal packing viewed down [010]. Hydrogen bonds are shown as dashed lines. H atoms not involving in hydrogen bonding have been omitted for clarity.

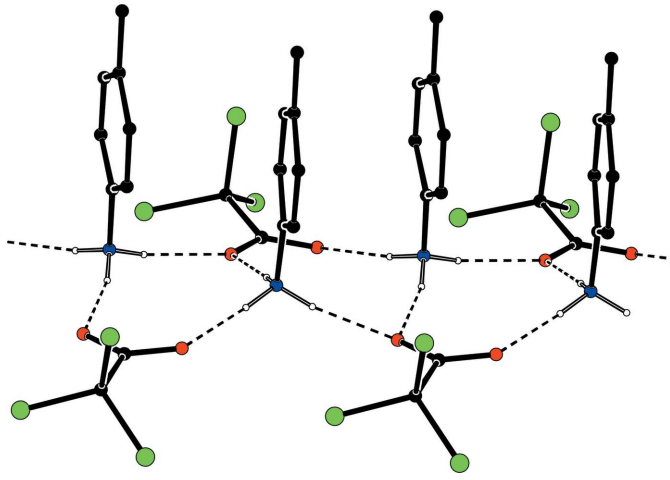

Figure 3
A partial view of the crystal packing, showing part of a [100] chain.

full crystallographic data

IUCrData (2017). **2**, x171767 [https://doi.org/10.1107/S2414314617017679]

4-Methylanilinium trichloroacetate

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bis-(4-Methylanilinium trichloroacetate)

Crystal data

$C_7H_{10}N^+\cdot C_2Cl_3O_2^-$
M_r = 270.53
Triclinic, $P\overline{1}$
a = 6.7395 (2) Å
b = 11.3491 (3) Å
c = 16.1078 (5) Å
α = 75.681 (2)°
β = 88.031 (2)°
γ = 86.856 (2)°
V = 1191.69 (6) Å^3
Z = 4
$F(000)$ = 552
D_x = 1.508 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9965 reflections
θ = 2.8–70.5°
μ = 6.82 mm^{-1}
T = 295 K
Block, colourless
0.26 × 0.22 × 0.18 mm

Data collection

Bruker APEXII CCD diffractometer
ω and φ scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.386, T_{max} = 0.754
37675 measured reflections
4685 independent reflections
3713 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 72.5°, θ_{min} = 2.8°
h = −8→8
k = −14→13
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.128
S = 1.02
4685 reflections
275 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 1.3481P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.0236 (4)	0.8176 (3)	0.3452 (2)	0.0550 (8)
C2	0.0345 (5)	0.7010 (4)	0.3974 (2)	0.0626 (9)
H2	0.032750	0.690668	0.456533	0.075*
C3	0.0480 (5)	0.5996 (3)	0.36460 (19)	0.0560 (8)
H3	0.054310	0.521988	0.401045	0.067*
C4	0.0519 (4)	0.6150 (3)	0.27675 (17)	0.0412 (6)
C5	0.0444 (4)	0.7297 (3)	0.22276 (18)	0.0464 (7)
H5	0.049155	0.739887	0.163650	0.056*
C6	0.0297 (4)	0.8296 (3)	0.2575 (2)	0.0538 (8)
H6	0.023774	0.907162	0.220901	0.065*
C7	0.0015 (7)	0.9265 (4)	0.3818 (3)	0.0829 (12)
H7A	−0.045476	0.995783	0.338455	0.124*
H7B	0.127876	0.942503	0.401343	0.124*
H7C	−0.092142	0.911241	0.429039	0.124*
C8	0.5084 (5)	0.9077 (3)	0.2121 (2)	0.0620 (9)
C9	0.5367 (4)	0.7938 (3)	0.2677 (2)	0.0579 (9)
H9	0.542346	0.787788	0.326206	0.069*
C10	0.5568 (4)	0.6891 (3)	0.2385 (2)	0.0506 (7)
H10	0.577655	0.613701	0.276898	0.061*
C11	0.5456 (4)	0.6972 (3)	0.15239 (19)	0.0482 (7)
C12	0.5148 (6)	0.8089 (3)	0.0954 (2)	0.0672 (10)
H12	0.506275	0.814448	0.037024	0.081*
C13	0.4969 (6)	0.9119 (4)	0.1260 (3)	0.0755 (11)
H13	0.476311	0.987076	0.087357	0.091*
C14	0.4872 (7)	1.0201 (4)	0.2444 (3)	0.0934 (14)
H14A	0.584074	1.076280	0.216207	0.140*
H14B	0.507760	0.999367	0.305112	0.140*
H14C	0.356226	1.056932	0.232833	0.140*
C15	0.4706 (4)	0.2780 (3)	0.40294 (18)	0.0430 (6)
C16	0.5665 (4)	0.3595 (3)	0.32056 (17)	0.0395 (6)
C17	−0.0049 (4)	0.7259 (3)	−0.07151 (18)	0.0441 (6)
C18	0.0910 (4)	0.6342 (3)	0.00763 (17)	0.0399 (6)
N1	0.0600 (3)	0.5075 (2)	0.24171 (15)	0.0443 (6)
H1A	0.048514	0.530754	0.185045	0.066*
H1B	−0.039197	0.460317	0.264266	0.066*
H1C	0.175484	0.466167	0.254491	0.066*
N2	0.5668 (3)	0.5879 (2)	0.12006 (16)	0.0503 (6)
H2A	0.686813	0.583562	0.095871	0.076*
H2B	0.474359	0.590779	0.081347	0.076*
H2C	0.552053	0.522479	0.163241	0.076*
O1	0.7457 (3)	0.3714 (3)	0.32515 (15)	0.0670 (7)
O2	0.4537 (3)	0.40509 (19)	0.26125 (12)	0.0499 (5)
O3	0.2719 (3)	0.6182 (2)	0.00216 (16)	0.0661 (7)
O4	−0.0249 (3)	0.59187 (19)	0.06740 (12)	0.0466 (5)
Cl1	0.45338 (17)	0.36565 (11)	0.47998 (6)	0.0839 (3)

Cl2	0.6160 (2)	0.14573 (9)	0.44281 (9)	0.1000 (4)
Cl3	0.22772 (14)	0.23875 (9)	0.38605 (6)	0.0722 (3)
Cl4	−0.25288 (12)	0.69394 (8)	−0.08705 (5)	0.0618 (2)
Cl5	0.12882 (16)	0.72723 (10)	−0.16713 (5)	0.0763 (3)
Cl6	−0.00309 (17)	0.87155 (8)	−0.04977 (7)	0.0793 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0387 (15)	0.067 (2)	0.060 (2)	−0.0023 (14)	0.0021 (14)	−0.0174 (17)
C2	0.065 (2)	0.079 (2)	0.0420 (17)	0.0003 (18)	0.0085 (15)	−0.0140 (17)
C3	0.0583 (19)	0.064 (2)	0.0375 (15)	0.0028 (16)	0.0036 (14)	0.0015 (14)
C4	0.0248 (12)	0.0574 (17)	0.0375 (14)	−0.0010 (11)	0.0005 (10)	−0.0046 (12)
C5	0.0349 (14)	0.0619 (19)	0.0368 (14)	−0.0077 (13)	−0.0016 (11)	−0.0001 (13)
C6	0.0408 (16)	0.0560 (19)	0.0592 (19)	−0.0052 (14)	−0.0050 (14)	−0.0028 (15)
C7	0.087 (3)	0.080 (3)	0.086 (3)	0.001 (2)	−0.002 (2)	−0.031 (2)
C8	0.0458 (18)	0.060 (2)	0.073 (2)	−0.0049 (15)	0.0028 (16)	−0.0038 (18)
C9	0.0386 (16)	0.075 (2)	0.0522 (18)	−0.0080 (15)	−0.0074 (13)	0.0011 (17)
C10	0.0324 (14)	0.0570 (19)	0.0515 (17)	−0.0041 (13)	−0.0056 (12)	0.0085 (14)
C11	0.0260 (13)	0.0595 (19)	0.0473 (16)	−0.0008 (12)	0.0054 (11)	0.0083 (14)
C12	0.074 (2)	0.064 (2)	0.0483 (19)	0.0094 (18)	0.0103 (17)	0.0124 (16)
C13	0.083 (3)	0.061 (2)	0.063 (2)	0.0072 (19)	0.0108 (19)	0.0175 (18)
C14	0.094 (3)	0.076 (3)	0.107 (4)	−0.003 (2)	−0.002 (3)	−0.017 (3)
C15	0.0493 (16)	0.0399 (15)	0.0378 (14)	−0.0077 (12)	0.0016 (12)	−0.0048 (12)
C16	0.0388 (14)	0.0420 (15)	0.0363 (14)	−0.0039 (11)	0.0059 (11)	−0.0072 (12)
C17	0.0481 (16)	0.0450 (16)	0.0368 (14)	−0.0029 (13)	0.0008 (12)	−0.0053 (12)
C18	0.0392 (14)	0.0414 (15)	0.0382 (14)	−0.0044 (11)	−0.0047 (11)	−0.0070 (12)
N1	0.0317 (11)	0.0589 (15)	0.0370 (12)	−0.0013 (10)	0.0004 (9)	−0.0024 (11)
N2	0.0333 (12)	0.0605 (16)	0.0456 (14)	0.0021 (11)	0.0035 (10)	0.0076 (12)
O1	0.0368 (11)	0.0967 (19)	0.0588 (14)	−0.0150 (12)	0.0041 (10)	−0.0004 (13)
O2	0.0431 (11)	0.0593 (13)	0.0380 (10)	−0.0017 (9)	0.0012 (9)	0.0056 (9)
O3	0.0335 (11)	0.0805 (17)	0.0731 (15)	−0.0048 (11)	−0.0063 (10)	0.0039 (13)
O4	0.0440 (11)	0.0584 (12)	0.0314 (10)	0.0035 (9)	0.0008 (8)	−0.0014 (9)
Cl1	0.0997 (8)	0.1091 (8)	0.0561 (5)	−0.0204 (6)	0.0153 (5)	−0.0441 (5)
Cl2	0.1093 (9)	0.0528 (5)	0.1176 (9)	0.0153 (5)	−0.0250 (7)	0.0163 (6)
Cl3	0.0624 (5)	0.0783 (6)	0.0699 (6)	−0.0369 (4)	0.0035 (4)	−0.0006 (4)
Cl4	0.0522 (4)	0.0709 (5)	0.0558 (5)	0.0019 (4)	−0.0188 (4)	−0.0020 (4)
Cl5	0.0892 (7)	0.0884 (7)	0.0452 (4)	−0.0131 (5)	0.0221 (4)	−0.0065 (4)
Cl6	0.1087 (8)	0.0431 (4)	0.0869 (7)	0.0003 (5)	−0.0135 (6)	−0.0166 (4)

Geometric parameters (Å, º)

C1—C2	1.381 (5)	C12—C13	1.375 (6)
C1—C6	1.385 (5)	C12—H12	0.9300
C1—C7	1.494 (5)	C13—H13	0.9300
C2—C3	1.378 (5)	C14—H14A	0.9600
C2—H2	0.9300	C14—H14B	0.9600
C3—C4	1.382 (4)	C14—H14C	0.9600

C3—H3	0.9300	C15—C16	1.560 (4)
C4—C5	1.375 (4)	C15—Cl2	1.741 (3)
C4—N1	1.463 (4)	C15—Cl3	1.767 (3)
C5—C6	1.380 (5)	C15—Cl1	1.770 (3)
C5—H5	0.9300	C16—O1	1.230 (3)
C6—H6	0.9300	C16—O2	1.233 (3)
C7—H7A	0.9600	C17—C18	1.566 (4)
C7—H7B	0.9600	C17—Cl5	1.755 (3)
C7—H7C	0.9600	C17—Cl4	1.769 (3)
C8—C13	1.380 (6)	C17—Cl6	1.773 (3)
C8—C9	1.386 (5)	C18—O3	1.226 (3)
C8—C14	1.491 (6)	C18—O4	1.236 (3)
C9—C10	1.381 (5)	N1—H1A	0.8900
C9—H9	0.9300	N1—H1B	0.8900
C10—C11	1.372 (4)	N1—H1C	0.8900
C10—H10	0.9300	N2—H2A	0.8900
C11—C12	1.379 (4)	N2—H2B	0.8900
C11—N2	1.458 (4)	N2—H2C	0.8900

C2—C1—C6	117.4 (3)	C12—C13—C8	122.3 (3)
C2—C1—C7	121.3 (3)	C12—C13—H13	118.8
C6—C1—C7	121.3 (3)	C8—C13—H13	118.8
C3—C2—C1	122.0 (3)	C8—C14—H14A	109.5
C3—C2—H2	119.0	C8—C14—H14B	109.5
C1—C2—H2	119.0	H14A—C14—H14B	109.5
C2—C3—C4	119.0 (3)	C8—C14—H14C	109.5
C2—C3—H3	120.5	H14A—C14—H14C	109.5
C4—C3—H3	120.5	H14B—C14—H14C	109.5
C5—C4—C3	120.6 (3)	C16—C15—Cl2	111.6 (2)
C5—C4—N1	120.2 (3)	C16—C15—Cl3	112.51 (19)
C3—C4—N1	119.2 (3)	Cl2—C15—Cl3	109.27 (16)
C4—C5—C6	119.1 (3)	C16—C15—Cl1	106.45 (19)
C4—C5—H5	120.4	Cl2—C15—Cl1	109.14 (16)
C6—C5—H5	120.4	Cl3—C15—Cl1	107.67 (16)
C5—C6—C1	121.9 (3)	O1—C16—O2	129.0 (3)
C5—C6—H6	119.1	O1—C16—C15	114.4 (2)
C1—C6—H6	119.1	O2—C16—C15	116.5 (2)
C1—C7—H7A	109.5	C18—C17—Cl5	112.3 (2)
C1—C7—H7B	109.5	C18—C17—Cl4	112.67 (19)
H7A—C7—H7B	109.5	Cl5—C17—Cl4	107.38 (16)
C1—C7—H7C	109.5	C18—C17—Cl6	106.09 (19)
H7A—C7—H7C	109.5	Cl5—C17—Cl6	109.53 (16)
H7B—C7—H7C	109.5	Cl4—C17—Cl6	108.85 (16)
C13—C8—C9	117.1 (4)	O3—C18—O4	129.5 (3)
C13—C8—C14	121.7 (4)	O3—C18—C17	114.8 (2)
C9—C8—C14	121.2 (4)	O4—C18—C17	115.6 (2)
C10—C9—C8	121.8 (3)	C4—N1—H1A	109.5
C10—C9—H9	119.1	C4—N1—H1B	109.5

C8—C9—H9	119.1	H1A—N1—H1B	109.5
C11—C10—C9	119.4 (3)	C4—N1—H1C	109.5
C11—C10—H10	120.3	H1A—N1—H1C	109.5
C9—C10—H10	120.3	H1B—N1—H1C	109.5
C10—C11—C12	120.4 (3)	C11—N2—H2A	109.5
C10—C11—N2	120.4 (3)	C11—N2—H2B	109.5
C12—C11—N2	119.2 (3)	H2A—N2—H2B	109.5
C13—C12—C11	119.1 (4)	C11—N2—H2C	109.5
C13—C12—H12	120.5	H2A—N2—H2C	109.5
C11—C12—H12	120.5	H2B—N2—H2C	109.5
C6—C1—C2—C3	−1.0 (5)	N2—C11—C12—C13	−179.4 (3)
C7—C1—C2—C3	177.7 (3)	C11—C12—C13—C8	−0.1 (6)
C1—C2—C3—C4	0.4 (5)	C9—C8—C13—C12	−0.8 (6)
C2—C3—C4—C5	0.6 (5)	C14—C8—C13—C12	−179.6 (4)
C2—C3—C4—N1	−178.1 (3)	Cl2—C15—C16—O1	−44.8 (3)
C3—C4—C5—C6	−1.0 (4)	Cl3—C15—C16—O1	−168.1 (2)
N1—C4—C5—C6	177.7 (2)	Cl1—C15—C16—O1	74.2 (3)
C4—C5—C6—C1	0.4 (4)	Cl2—C15—C16—O2	136.9 (2)
C2—C1—C6—C5	0.6 (5)	Cl3—C15—C16—O2	13.6 (3)
C7—C1—C6—C5	−178.1 (3)	Cl1—C15—C16—O2	−104.1 (3)
C13—C8—C9—C10	1.3 (5)	Cl5—C17—C18—O3	29.9 (3)
C14—C8—C9—C10	−179.9 (3)	Cl4—C17—C18—O3	151.3 (2)
C8—C9—C10—C11	−1.0 (4)	Cl6—C17—C18—O3	−89.7 (3)
C9—C10—C11—C12	0.0 (4)	Cl5—C17—C18—O4	−152.5 (2)
C9—C10—C11—N2	179.9 (2)	Cl4—C17—C18—O4	−31.1 (3)
C10—C11—C12—C13	0.5 (5)	Cl6—C17—C18—O4	87.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O4	0.89	1.92	2.801 (3)	169
N1—H1*B*···O1^{i}	0.89	1.91	2.796 (3)	172
N1—H1*C*···O2	0.89	1.96	2.834 (3)	167
N2—H2*A*···O4^{ii}	0.89	1.98	2.851 (3)	165
N2—H2*B*···O3	0.89	1.86	2.745 (3)	172
N2—H2*C*···O2	0.89	1.92	2.789 (3)	164

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