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1,3-Bis(2,4,6-trimethylphenyl)imidazolium perchlorate

Ruiyao Wang[a] and Manuel A.S. Aquino[b]*

[a]Department of Chemistry, Xi'an Jiaotong-Liverpool University, 111 Renai Road, Suzhou, Jiangsu, 215123, People's Republic of China, and [b]Department of Chemistry, St. Francis Xavier University, P.O. Box 5000, Antigonish, Nova Scotia, B2G 2W5, Canada. *Correspondence e-mail: maquino@stfx.ca

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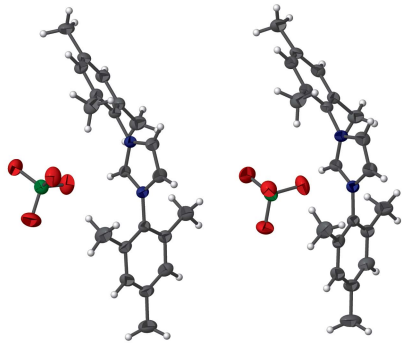
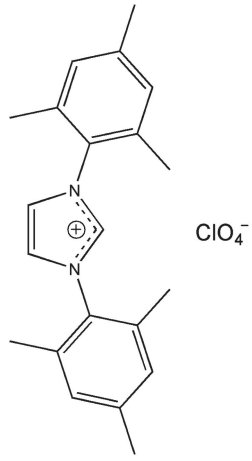
The title compound, $C_{21}H_{25}N_2^+\cdot ClO_4^-$, arose as an unexpected oxidation product of the carbene 1,3-bis(2,4,6-trimethylphenyl)-1,3-dihydro-2*H*-imidazol-2-ylidine in methanol. It crystallizes with two unique cations and anions in the *P*-type monoclinic unit cell. The five-membered cationic imidazolium rings are essentially planar and in each imidazolium cation the phenyl rings of the 2,4,6-trimethylphenyl groups are staggered with respect to the imidazolium ring [dihedral angles ranging from 60.9 (3) to 86.3 (3)°]. In the crystal, a hydrogen-bonding network is created *via* C—H···O interactions between the imidazolium ring H atoms and the perchlorate-anion oxygen atoms. The crystal studied was refined as an inversion twin.

3D view



Chemical scheme



Structure description

In past years, our research into the chemistry of diruthenium (II,III) tetracarboxylates has led us to attempt axial coordination of N-heterocyclic carbenes to the diruthenium (II,III) core. This has only been accomplished on analogous dirhodium(II,II) systems in the past (André *et al.*, 2008). Thus far our attempts have been unsuccessful, but we were able to isolate crystals of an oxidized imidizolium species as a perchlorate salt in the course of a number of these reactions.

The asymmetric unit of the title compound consists of two unique imidazolium cations and perchlorate anions. The salt consists of an imidazolium core with 2,4,6-trimethylphenyl substituents attached to each heterocyclic nitrogen atom (Fig. 1). The bond lengths in the heterocycles of the two unique imidazolium molecules are: C2—C3 = 1.347 (5), C23—C24 = 1.335 (5), N1—C1 = 1.327 (4), N2—C1 = 1.333 (4), N3—C22 = 1.326 (4) and N4—C22 = 1.328 (4) Å, and are consistent with a double bond between the C2 and C3, and the C23 and C24 carbon atoms and bond delocalization over the N—C—N portion of the ring in both cases. This is very similar to other 2,4,6-trimethylphenyl

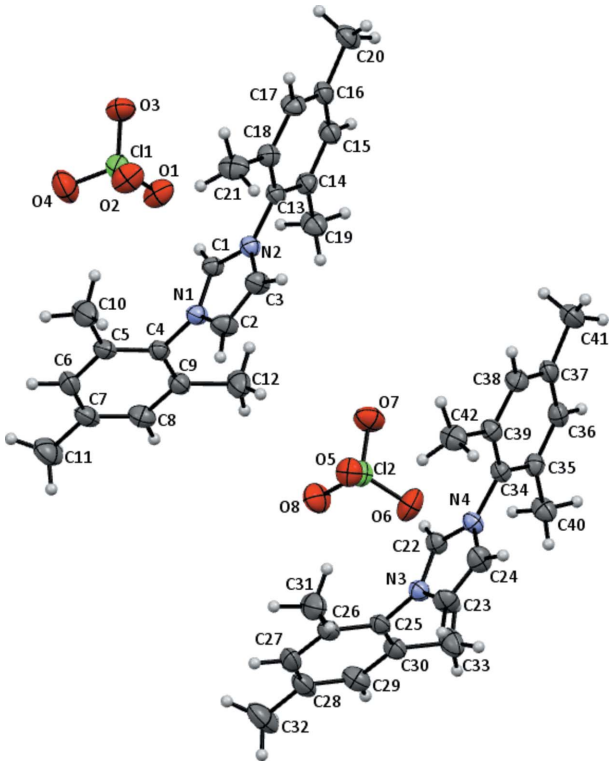


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

derivatives (*e.g.* Arduengo *et al.*, 1995; Cole *et al.* 2002; Kennedy *et al.* 2014). In the crystal, C—H···O hydrogen bonding is seen between the two alternating, unique, imidazolium molecules, and the interspersed perchlorate anions (Table 1) when viewed along the [100] axis (Fig. 2).

Crystal structures of perchlorate salts of imidazolium derivatives are rare (Minaker *et al.*, 2018; Crees *et al.*, 2010; Fürstner *et al.*, 2006; Pesch *et al.*, 2004) and none have been reported for the 2,4,6-trimethylphenyl derivative.

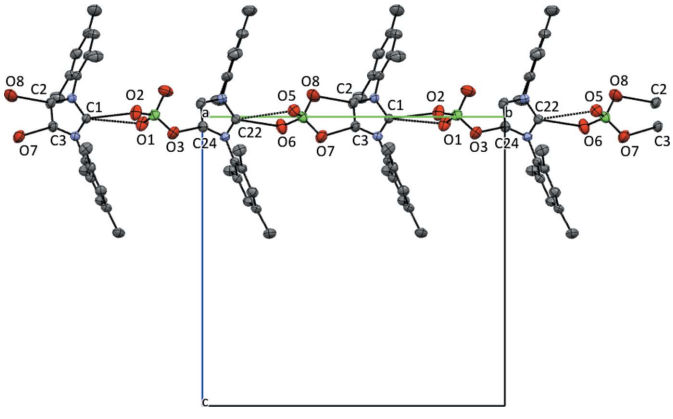


Figure 2
Packing diagram showing hydrogen-bonding interactions as dashed lines, viewed along the [100] axis.

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···O1	0.95	2.16	3.078 (4)	162
C2—H2A···O8^{i}	0.95	2.61	3.191 (5)	120
C3—H3A···O7^{i}	0.95	2.38	3.225 (5)	148
C22—H22A···O5	0.95	2.37	3.119 (4)	136
C22—H22A···O6	0.95	2.34	3.259 (4)	163
C24—H24A···O3^{ii}	0.95	2.50	3.295 (5)	141

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{21}H_{25}N_2^+\cdot ClO_4^-$
M_r	404.88
Crystal system, space group	Monoclinic, *Pc*
Temperature (K)	180
a, b, c (Å)	8.4771 (2), 16.0726 (4), 15.7922 (3)
β (°)	103.958 (1)
V (Å^3)	2088.14 (8)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.21
Crystal size (mm)	0.25 × 0.20 × 0.08
Data collection	
Diffractometer	Bruker APEXII CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2010)
T_{min}, T_{max}	0.949, 0.983
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9700, 5877, 5049
R_{int}	0.025
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.099, 1.04
No. of reflections	5877
No. of parameters	518
No. of restraints	2
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.29
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.11 (7)

Computer programs: *APEX2* and *SAINT* (Bruker, 2010), *SHELXS97* and *SHELXTL* (Sheldrick, 2008) and *SHELXL2018* (Sheldrick, 2015).

Synthesis and crystallization

Crystals of the title compound were isolated as a byproduct of the reaction of $[Ru_2(\mu\text{-}O_2CCH_3)_4(MeOH)_2](ClO_4)$ (0.100 g, 0.166 mmol) in 15 ml of methanol with a twofold excess of the carbene 1,3-bis(2,4,6-trimethylphenyl)-1,3-dihydro-2*H*-imidazol-2-ylidene (0.101 g, 0.333 mmol) in 5 ml of methanol. Crystals were obtained by slow evaporation of the reaction mixture.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The crystal studied was refined as an inversion twin.

Funding information

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full crystallographic data

IUCrData (2019). **4**, x190494 [https://doi.org/10.1107/S2414314619004942]

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Crystal data

$C_{21}H_{25}N_2^+ \cdot ClO_4^-$
M_r = 404.88
Monoclinic, *Pc*
a = 8.4771 (2) Å
b = 16.0726 (4) Å
c = 15.7922 (3) Å
β = 103.958 (1)°
V = 2088.14 (8) Å^3
Z = 4
F(000) = 856
D_x = 1.288 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 3592 reflections
θ = 2.7–26.2°
μ = 0.21 mm^{-1}
T = 180 K
Plate, colourless
0.25 × 0.20 × 0.08 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, 2010)
T_{min} = 0.949, T_{max} = 0.983
9700 measured reflections
5877 independent reflections
5049 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.0°, θ_{min} = 1.8°
h = −10→9
k = −19→18
l = −17→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.099
S = 1.04
5877 reflections
518 parameters
2 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0521P)^2 + 0.1933P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.29 e Å^{-3}
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.11 (7)

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.

Refinement. Refined as a 2-component inversion twin. 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.2216 (4)	0.56851 (17)	−0.06414 (17)	0.0294 (6)
N2	0.1903 (3)	0.57959 (17)	0.06729 (17)	0.0284 (6)
N3	0.6168 (4)	0.06465 (17)	−0.06288 (17)	0.0296 (7)
N4	0.5868 (3)	0.07431 (17)	0.06929 (17)	0.0279 (6)
C1	0.2506 (4)	0.6165 (2)	0.0063 (2)	0.0274 (7)
H1A	0.305234	0.668566	0.012313	0.033*
C2	0.1378 (5)	0.4991 (2)	−0.0484 (2)	0.0383 (9)
H2A	0.100289	0.454850	−0.087960	0.046*
C3	0.1191 (5)	0.5058 (2)	0.0337 (2)	0.0361 (9)
H3A	0.066664	0.466832	0.062939	0.043*
C4	0.2781 (4)	0.5859 (2)	−0.1422 (2)	0.0281 (8)
C5	0.1696 (5)	0.6240 (2)	−0.2123 (2)	0.0336 (8)
C6	0.2302 (5)	0.6416 (2)	−0.2856 (2)	0.0404 (10)
H6A	0.160705	0.667849	−0.334511	0.048*
C7	0.3861 (5)	0.6223 (2)	−0.2892 (2)	0.0385 (9)
C8	0.4882 (5)	0.5830 (2)	−0.2179 (2)	0.0390 (9)
H8A	0.596135	0.569337	−0.220130	0.047*
C9	0.4347 (5)	0.5634 (2)	−0.1430 (2)	0.0333 (8)
C10	−0.0005 (5)	0.6463 (3)	−0.2089 (3)	0.0541 (12)
H10A	0.002536	0.681936	−0.158141	0.081*
H10B	−0.061391	0.595436	−0.203965	0.081*
H10C	−0.053771	0.676104	−0.262248	0.081*
C11	0.4493 (6)	0.6432 (3)	−0.3678 (3)	0.0563 (12)
H11A	0.379572	0.685500	−0.402777	0.084*
H11B	0.449207	0.592976	−0.403026	0.084*
H11C	0.560481	0.664637	−0.348748	0.084*
C12	0.5465 (5)	0.5209 (3)	−0.0664 (2)	0.0483 (11)
H12A	0.563672	0.557230	−0.015094	0.073*
H12B	0.651022	0.509474	−0.080179	0.073*
H12C	0.497272	0.468529	−0.054071	0.073*
C13	0.1951 (4)	0.6141 (2)	0.1526 (2)	0.0291 (8)
C14	0.3458 (4)	0.6305 (2)	0.2093 (2)	0.0317 (8)
C15	0.3442 (5)	0.6652 (2)	0.2898 (2)	0.0357 (9)
H15A	0.445012	0.677350	0.329421	0.043*
C16	0.2030 (5)	0.6825 (2)	0.3145 (2)	0.0330 (8)
C17	0.0552 (5)	0.6646 (2)	0.2568 (2)	0.0353 (8)
H17A	−0.043016	0.675867	0.273555	0.042*
C18	0.0480 (4)	0.6303 (2)	0.1747 (2)	0.0321 (8)
C19	0.5043 (5)	0.6155 (3)	0.1858 (3)	0.0432 (10)

H19A	0.592296	0.614705	0.239025	0.065*
H19B	0.523974	0.660089	0.147300	0.065*
H19C	0.500343	0.561912	0.155792	0.065*
C20	0.2073 (5)	0.7232 (2)	0.4015 (2)	0.0435 (10)
H20A	0.317745	0.719905	0.438848	0.065*
H20B	0.131934	0.694377	0.429776	0.065*
H20C	0.175135	0.781692	0.392175	0.065*
C21	−0.1132 (5)	0.6144 (3)	0.1114 (3)	0.0450 (10)
H21A	−0.198908	0.644818	0.130297	0.067*
H21B	−0.137225	0.554651	0.109730	0.067*
H21C	−0.108348	0.633255	0.053112	0.067*
C22	0.6483 (4)	0.1107 (2)	0.0089 (2)	0.0290 (8)
H22A	0.705933	0.161930	0.015982	0.035*
C23	0.5310 (5)	−0.0048 (2)	−0.0470 (2)	0.0381 (9)
H23A	0.492428	−0.048828	−0.086827	0.046*
C24	0.5125 (5)	0.0015 (2)	0.0343 (2)	0.0382 (9)
H24A	0.458141	−0.037154	0.062926	0.046*
C25	0.6691 (4)	0.0848 (2)	−0.1410 (2)	0.0299 (8)
C26	0.5517 (5)	0.1085 (2)	−0.2152 (2)	0.0353 (9)
C27	0.6090 (5)	0.1336 (2)	−0.2872 (2)	0.0417 (10)
H27A	0.533204	0.153149	−0.337817	0.050*
C28	0.7706 (6)	0.1309 (2)	−0.2873 (2)	0.0439 (10)
C29	0.8819 (5)	0.1048 (3)	−0.2122 (2)	0.0413 (10)
H29A	0.994130	0.102928	−0.211641	0.050*
C30	0.8325 (5)	0.0815 (2)	−0.1379 (2)	0.0344 (9)
C31	0.3741 (5)	0.1083 (3)	−0.2191 (3)	0.0530 (12)
H31A	0.316293	0.140696	−0.269655	0.079*
H31B	0.355442	0.132997	−0.165633	0.079*
H31C	0.333752	0.050876	−0.224619	0.079*
C32	0.8276 (7)	0.1557 (3)	−0.3665 (3)	0.0636 (14)
H32A	0.761409	0.202142	−0.395873	0.095*
H32B	0.816930	0.108347	−0.406574	0.095*
H32C	0.941792	0.172928	−0.348958	0.095*
C33	0.9582 (5)	0.0541 (3)	−0.0571 (2)	0.0501 (11)
H33A	0.918355	0.004857	−0.032095	0.075*
H33B	0.977447	0.099179	−0.014142	0.075*
H33C	1.060010	0.040410	−0.073036	0.075*
C34	0.5987 (4)	0.1071 (2)	0.1559 (2)	0.0281 (8)
C35	0.7530 (4)	0.1161 (2)	0.2124 (2)	0.0292 (8)
C36	0.7620 (5)	0.1519 (2)	0.2932 (2)	0.0339 (9)
H36A	0.865316	0.158801	0.332486	0.041*
C37	0.6224 (5)	0.1781 (2)	0.3183 (2)	0.0338 (8)
C38	0.4722 (5)	0.1654 (2)	0.2613 (2)	0.0358 (9)
H38A	0.376804	0.180914	0.278999	0.043*
C39	0.4567 (4)	0.1307 (2)	0.1789 (2)	0.0311 (8)
C40	0.9067 (4)	0.0915 (2)	0.1869 (2)	0.0384 (9)
H40A	0.995085	0.085272	0.239428	0.058*
H40B	0.935438	0.134556	0.149317	0.058*

H40C	0.889510	0.038503	0.155199	0.058*
C41	0.6383 (6)	0.2226 (3)	0.4039 (2)	0.0499 (11)
H41A	0.552190	0.203848	0.431176	0.075*
H41B	0.628567	0.282686	0.393653	0.075*
H41C	0.744623	0.210108	0.442704	0.075*
C42	0.2906 (5)	0.1227 (3)	0.1162 (3)	0.0433 (10)
H42A	0.299361	0.135682	0.056868	0.065*
H42B	0.214734	0.161535	0.133280	0.065*
H42C	0.250677	0.065660	0.117998	0.065*
Cl1	0.19602 (10)	0.84117 (6)	−0.00581 (6)	0.0367 (2)
Cl2	0.80684 (10)	0.33141 (6)	0.00185 (6)	0.0359 (2)
O1	0.3521 (3)	0.80080 (18)	0.02427 (18)	0.0481 (7)
O2	0.0698 (3)	0.78046 (16)	−0.01380 (19)	0.0495 (7)
O3	0.1824 (4)	0.90408 (17)	0.05591 (18)	0.0492 (7)
O4	0.1895 (4)	0.8787 (2)	−0.08888 (19)	0.0627 (9)
O5	0.6417 (3)	0.30266 (17)	−0.02108 (17)	0.0425 (7)
O6	0.9118 (3)	0.26184 (17)	0.03387 (19)	0.0513 (8)
O7	0.8257 (4)	0.39378 (17)	0.06874 (18)	0.0498 (8)
O8	0.8495 (4)	0.36686 (18)	−0.07326 (19)	0.0536 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0359 (17)	0.0268 (15)	0.0277 (14)	−0.0026 (13)	0.0117 (12)	−0.0018 (12)
N2	0.0315 (16)	0.0282 (15)	0.0269 (15)	−0.0005 (13)	0.0099 (12)	0.0008 (12)
N3	0.0331 (16)	0.0297 (15)	0.0252 (14)	−0.0013 (13)	0.0055 (12)	−0.0010 (12)
N4	0.0289 (16)	0.0308 (15)	0.0236 (14)	−0.0056 (12)	0.0057 (11)	−0.0015 (12)
C1	0.0299 (18)	0.0252 (17)	0.0284 (18)	−0.0020 (14)	0.0096 (14)	−0.0023 (14)
C2	0.045 (2)	0.032 (2)	0.040 (2)	−0.0103 (18)	0.0151 (17)	−0.0081 (16)
C3	0.047 (2)	0.0279 (18)	0.037 (2)	−0.0091 (17)	0.0167 (17)	−0.0023 (15)
C4	0.034 (2)	0.0252 (17)	0.0257 (17)	−0.0025 (15)	0.0083 (14)	−0.0022 (14)
C5	0.038 (2)	0.036 (2)	0.0270 (18)	0.0032 (17)	0.0090 (16)	−0.0062 (15)
C6	0.053 (3)	0.041 (2)	0.0250 (19)	0.003 (2)	0.0041 (17)	0.0024 (16)
C7	0.054 (3)	0.036 (2)	0.0287 (19)	−0.0071 (19)	0.0168 (17)	−0.0058 (16)
C8	0.038 (2)	0.045 (2)	0.037 (2)	−0.0036 (18)	0.0140 (17)	−0.0064 (17)
C9	0.036 (2)	0.035 (2)	0.0289 (18)	−0.0003 (17)	0.0078 (15)	−0.0011 (16)
C10	0.042 (3)	0.077 (3)	0.040 (2)	0.020 (2)	0.0049 (18)	−0.002 (2)
C11	0.073 (3)	0.062 (3)	0.042 (2)	−0.005 (3)	0.029 (2)	0.002 (2)
C12	0.041 (2)	0.064 (3)	0.040 (2)	0.016 (2)	0.0085 (18)	0.008 (2)
C13	0.037 (2)	0.0279 (18)	0.0246 (17)	−0.0002 (15)	0.0118 (14)	0.0019 (14)
C14	0.0295 (19)	0.034 (2)	0.0312 (19)	0.0025 (16)	0.0075 (14)	0.0013 (15)
C15	0.035 (2)	0.042 (2)	0.0289 (19)	0.0013 (17)	0.0062 (15)	0.0016 (16)
C16	0.042 (2)	0.0332 (19)	0.0251 (18)	−0.0001 (17)	0.0103 (15)	0.0044 (15)
C17	0.036 (2)	0.038 (2)	0.037 (2)	0.0011 (17)	0.0187 (16)	0.0000 (16)
C18	0.032 (2)	0.0318 (19)	0.0335 (19)	−0.0020 (16)	0.0103 (15)	0.0002 (15)
C19	0.033 (2)	0.060 (3)	0.037 (2)	0.0034 (19)	0.0098 (17)	−0.0009 (19)
C20	0.060 (3)	0.046 (2)	0.0287 (19)	−0.005 (2)	0.0183 (18)	0.0007 (17)
C21	0.029 (2)	0.055 (3)	0.051 (2)	−0.0012 (19)	0.0109 (17)	−0.011 (2)

C22	0.0305 (19)	0.0287 (17)	0.0269 (18)	−0.0041 (15)	0.0050 (14)	−0.0009 (14)
C23	0.047 (2)	0.0303 (19)	0.035 (2)	−0.0129 (18)	0.0064 (17)	−0.0079 (16)
C24	0.043 (2)	0.0329 (19)	0.038 (2)	−0.0132 (17)	0.0088 (17)	0.0003 (16)
C25	0.040 (2)	0.0270 (18)	0.0216 (16)	−0.0024 (15)	0.0046 (14)	−0.0036 (13)
C26	0.042 (2)	0.035 (2)	0.0267 (18)	0.0068 (17)	0.0029 (16)	−0.0064 (15)
C27	0.057 (3)	0.038 (2)	0.0246 (19)	0.003 (2)	−0.0015 (17)	−0.0006 (16)
C28	0.066 (3)	0.041 (2)	0.0254 (19)	−0.008 (2)	0.0121 (18)	−0.0036 (17)
C29	0.040 (2)	0.051 (2)	0.035 (2)	−0.0072 (19)	0.0124 (17)	−0.0063 (17)
C30	0.039 (2)	0.036 (2)	0.0264 (18)	−0.0005 (17)	0.0048 (15)	−0.0029 (15)
C31	0.044 (3)	0.067 (3)	0.042 (2)	0.017 (2)	−0.0001 (19)	−0.003 (2)
C32	0.092 (4)	0.070 (3)	0.033 (2)	−0.023 (3)	0.024 (2)	−0.003 (2)
C33	0.038 (2)	0.077 (3)	0.034 (2)	0.009 (2)	0.0050 (17)	0.005 (2)
C34	0.0306 (19)	0.0299 (18)	0.0239 (17)	−0.0023 (15)	0.0065 (14)	0.0015 (14)
C35	0.0274 (18)	0.0314 (19)	0.0291 (18)	0.0005 (15)	0.0077 (14)	0.0044 (14)
C36	0.036 (2)	0.0358 (19)	0.0270 (18)	−0.0028 (17)	0.0023 (15)	0.0036 (15)
C37	0.047 (2)	0.0313 (19)	0.0231 (17)	−0.0007 (17)	0.0084 (15)	0.0036 (14)
C38	0.037 (2)	0.039 (2)	0.034 (2)	0.0031 (17)	0.0130 (16)	0.0022 (16)
C39	0.033 (2)	0.0328 (19)	0.0280 (18)	−0.0027 (16)	0.0089 (14)	0.0016 (14)
C40	0.031 (2)	0.052 (2)	0.032 (2)	−0.0015 (18)	0.0080 (16)	0.0002 (17)
C41	0.073 (3)	0.048 (2)	0.029 (2)	0.003 (2)	0.0127 (19)	−0.0027 (18)
C42	0.029 (2)	0.057 (3)	0.042 (2)	0.0013 (19)	0.0063 (16)	−0.0059 (19)
Cl1	0.0337 (5)	0.0368 (5)	0.0371 (5)	−0.0035 (4)	0.0038 (4)	−0.0019 (4)
Cl2	0.0359 (6)	0.0336 (5)	0.0388 (5)	−0.0037 (4)	0.0102 (4)	0.0033 (4)
O1	0.0275 (15)	0.0449 (16)	0.0651 (19)	−0.0001 (12)	−0.0016 (13)	−0.0105 (14)
O2	0.0327 (16)	0.0419 (15)	0.0703 (19)	−0.0129 (13)	0.0055 (13)	−0.0081 (14)
O3	0.0603 (19)	0.0398 (15)	0.0480 (17)	−0.0002 (14)	0.0139 (14)	−0.0100 (13)
O4	0.080 (2)	0.069 (2)	0.0383 (16)	−0.0050 (19)	0.0125 (15)	0.0101 (15)
O5	0.0319 (15)	0.0458 (16)	0.0477 (16)	−0.0068 (12)	0.0058 (12)	0.0009 (13)
O6	0.0397 (16)	0.0387 (15)	0.070 (2)	0.0044 (13)	0.0034 (14)	0.0113 (14)
O7	0.060 (2)	0.0413 (16)	0.0488 (17)	−0.0123 (14)	0.0152 (14)	−0.0116 (13)
O8	0.068 (2)	0.0515 (18)	0.0504 (17)	−0.0055 (16)	0.0313 (15)	0.0110 (14)

Geometric parameters (Å, º)

N1—C1	1.327 (4)	C21—H21B	0.9800
N1—C2	1.376 (4)	C21—H21C	0.9800
N1—C4	1.453 (4)	C22—H22A	0.9500
N2—C1	1.333 (4)	C23—C24	1.335 (5)
N2—C3	1.378 (4)	C23—H23A	0.9500
N2—C13	1.449 (4)	C24—H24A	0.9500
N3—C22	1.326 (4)	C25—C30	1.375 (5)
N3—C23	1.388 (4)	C25—C26	1.394 (5)
N3—C25	1.445 (4)	C26—C27	1.400 (5)
N4—C22	1.328 (4)	C26—C31	1.492 (6)
N4—C24	1.380 (4)	C27—C28	1.371 (6)
N4—C34	1.447 (4)	C27—H27A	0.9500
C1—H1A	0.9500	C28—C29	1.391 (6)
C2—C3	1.347 (5)	C28—C32	1.500 (6)

C2—H2A	0.9500	C29—C30	1.389 (5)
C3—H3A	0.9500	C29—H29A	0.9500
C4—C9	1.379 (5)	C30—C33	1.516 (5)
C4—C5	1.399 (5)	C31—H31A	0.9800
C5—C6	1.404 (5)	C31—H31B	0.9800
C5—C10	1.499 (6)	C31—H31C	0.9800
C6—C7	1.372 (6)	C32—H32A	0.9800
C6—H6A	0.9500	C32—H32B	0.9800
C7—C8	1.395 (5)	C32—H32C	0.9800
C7—C11	1.503 (5)	C33—H33A	0.9800
C8—C9	1.400 (5)	C33—H33B	0.9800
C8—H8A	0.9500	C33—H33C	0.9800
C9—C12	1.509 (5)	C34—C39	1.391 (5)
C10—H10A	0.9800	C34—C35	1.402 (5)
C10—H10B	0.9800	C35—C36	1.384 (5)
C10—H10C	0.9800	C35—C40	1.507 (5)
C11—H11A	0.9800	C36—C37	1.400 (6)
C11—H11B	0.9800	C36—H36A	0.9500
C11—H11C	0.9800	C37—C38	1.386 (5)
C12—H12A	0.9800	C37—C41	1.507 (5)
C12—H12B	0.9800	C38—C39	1.393 (5)
C12—H12C	0.9800	C38—H38A	0.9500
C13—C14	1.396 (5)	C39—C42	1.518 (5)
C13—C18	1.398 (5)	C40—H40A	0.9800
C14—C15	1.392 (5)	C40—H40B	0.9800
C14—C19	1.497 (5)	C40—H40C	0.9800
C15—C16	1.374 (5)	C41—H41A	0.9800
C15—H15A	0.9500	C41—H41B	0.9800
C16—C17	1.390 (5)	C41—H41C	0.9800
C16—C20	1.514 (5)	C42—H42A	0.9800
C17—C18	1.396 (5)	C42—H42B	0.9800
C17—H17A	0.9500	C42—H42C	0.9800
C18—C21	1.508 (5)	Cl1—O3	1.428 (3)
C19—H19A	0.9800	Cl1—O2	1.431 (3)
C19—H19B	0.9800	Cl1—O4	1.433 (3)
C19—H19C	0.9800	Cl1—O1	1.447 (3)
C20—H20A	0.9800	Cl2—O5	1.435 (3)
C20—H20B	0.9800	Cl2—O7	1.437 (3)
C20—H20C	0.9800	Cl2—O8	1.439 (3)
C21—H21A	0.9800	Cl2—O6	1.443 (3)

C1—N1—C2	108.6 (3)	N3—C22—N4	109.6 (3)
C1—N1—C4	124.6 (3)	N3—C22—H22A	125.2
C2—N1—C4	126.8 (3)	N4—C22—H22A	125.2
C1—N2—C3	108.3 (3)	C24—C23—N3	107.5 (3)
C1—N2—C13	124.9 (3)	C24—C23—H23A	126.3
C3—N2—C13	126.8 (3)	N3—C23—H23A	126.3
C22—N3—C23	107.5 (3)	C23—C24—N4	107.5 (3)

C22—N3—C25	124.4 (3)	C23—C24—H24A	126.2
C23—N3—C25	128.0 (3)	N4—C24—H24A	126.2
C22—N4—C24	107.8 (3)	C30—C25—C26	123.3 (3)
C22—N4—C34	124.7 (3)	C30—C25—N3	118.3 (3)
C24—N4—C34	127.5 (3)	C26—C25—N3	118.3 (3)
N1—C1—N2	108.7 (3)	C25—C26—C27	116.4 (4)
N1—C1—H1A	125.6	C25—C26—C31	122.9 (4)
N2—C1—H1A	125.6	C27—C26—C31	120.8 (3)
C3—C2—N1	107.2 (3)	C28—C27—C26	122.3 (3)
C3—C2—H2A	126.4	C28—C27—H27A	118.8
N1—C2—H2A	126.4	C26—C27—H27A	118.8
C2—C3—N2	107.3 (3)	C27—C28—C29	118.7 (4)
C2—C3—H3A	126.4	C27—C28—C32	121.1 (4)
N2—C3—H3A	126.4	C29—C28—C32	120.2 (4)
C9—C4—C5	123.9 (3)	C30—C29—C28	121.4 (4)
C9—C4—N1	118.2 (3)	C30—C29—H29A	119.3
C5—C4—N1	117.8 (3)	C28—C29—H29A	119.3
C4—C5—C6	116.0 (4)	C25—C30—C29	117.7 (3)
C4—C5—C10	122.1 (3)	C25—C30—C33	122.6 (4)
C6—C5—C10	121.9 (4)	C29—C30—C33	119.7 (4)
C7—C6—C5	122.5 (4)	C26—C31—H31A	109.5
C7—C6—H6A	118.8	C26—C31—H31B	109.5
C5—C6—H6A	118.8	H31A—C31—H31B	109.5
C6—C7—C8	119.0 (4)	C26—C31—H31C	109.5
C6—C7—C11	121.5 (4)	H31A—C31—H31C	109.5
C8—C7—C11	119.5 (4)	H31B—C31—H31C	109.5
C7—C8—C9	121.3 (4)	C28—C32—H32A	109.5
C7—C8—H8A	119.4	C28—C32—H32B	109.5
C9—C8—H8A	119.4	H32A—C32—H32B	109.5
C4—C9—C8	117.2 (3)	C28—C32—H32C	109.5
C4—C9—C12	122.0 (3)	H32A—C32—H32C	109.5
C8—C9—C12	120.7 (4)	H32B—C32—H32C	109.5
C5—C10—H10A	109.5	C30—C33—H33A	109.5
C5—C10—H10B	109.5	C30—C33—H33B	109.5
H10A—C10—H10B	109.5	H33A—C33—H33B	109.5
C5—C10—H10C	109.5	C30—C33—H33C	109.5
H10A—C10—H10C	109.5	H33A—C33—H33C	109.5
H10B—C10—H10C	109.5	H33B—C33—H33C	109.5
C7—C11—H11A	109.5	C39—C34—C35	122.7 (3)
C7—C11—H11B	109.5	C39—C34—N4	118.6 (3)
H11A—C11—H11B	109.5	C35—C34—N4	118.8 (3)
C7—C11—H11C	109.5	C36—C35—C34	117.7 (3)
H11A—C11—H11C	109.5	C36—C35—C40	119.8 (3)
H11B—C11—H11C	109.5	C34—C35—C40	122.5 (3)
C9—C12—H12A	109.5	C35—C36—C37	121.6 (3)
C9—C12—H12B	109.5	C35—C36—H36A	119.2
H12A—C12—H12B	109.5	C37—C36—H36A	119.2
C9—C12—H12C	109.5	C38—C37—C36	118.6 (3)

H12A—C12—H12C	109.5	C38—C37—C41	121.4 (4)
H12B—C12—H12C	109.5	C36—C37—C41	119.9 (3)
C14—C13—C18	122.5 (3)	C37—C38—C39	122.0 (4)
C14—C13—N2	119.0 (3)	C37—C38—H38A	119.0
C18—C13—N2	118.5 (3)	C39—C38—H38A	119.0
C15—C14—C13	116.9 (3)	C34—C39—C38	117.4 (3)
C15—C14—C19	119.9 (3)	C34—C39—C42	122.1 (3)
C13—C14—C19	123.2 (3)	C38—C39—C42	120.5 (3)
C16—C15—C14	122.8 (3)	C35—C40—H40A	109.5
C16—C15—H15A	118.6	C35—C40—H40B	109.5
C14—C15—H15A	118.6	H40A—C40—H40B	109.5
C15—C16—C17	118.7 (3)	C35—C40—H40C	109.5
C15—C16—C20	120.9 (3)	H40A—C40—H40C	109.5
C17—C16—C20	120.4 (4)	H40B—C40—H40C	109.5
C16—C17—C18	121.4 (3)	C37—C41—H41A	109.5
C16—C17—H17A	119.3	C37—C41—H41B	109.5
C18—C17—H17A	119.3	H41A—C41—H41B	109.5
C17—C18—C13	117.6 (3)	C37—C41—H41C	109.5
C17—C18—C21	120.8 (3)	H41A—C41—H41C	109.5
C13—C18—C21	121.6 (3)	H41B—C41—H41C	109.5
C14—C19—H19A	109.5	C39—C42—H42A	109.5
C14—C19—H19B	109.5	C39—C42—H42B	109.5
H19A—C19—H19B	109.5	H42A—C42—H42B	109.5
C14—C19—H19C	109.5	C39—C42—H42C	109.5
H19A—C19—H19C	109.5	H42A—C42—H42C	109.5
H19B—C19—H19C	109.5	H42B—C42—H42C	109.5
C16—C20—H20A	109.5	O3—Cl1—O2	111.17 (19)
C16—C20—H20B	109.5	O3—Cl1—O4	109.58 (19)
H20A—C20—H20B	109.5	O2—Cl1—O4	110.09 (19)
C16—C20—H20C	109.5	O3—Cl1—O1	108.15 (17)
H20A—C20—H20C	109.5	O2—Cl1—O1	109.15 (17)
H20B—C20—H20C	109.5	O4—Cl1—O1	108.6 (2)
C18—C21—H21A	109.5	O5—Cl2—O7	109.80 (19)
C18—C21—H21B	109.5	O5—Cl2—O8	110.09 (18)
H21A—C21—H21B	109.5	O7—Cl2—O8	108.92 (18)
C18—C21—H21C	109.5	O5—Cl2—O6	108.70 (16)
H21A—C21—H21C	109.5	O7—Cl2—O6	109.47 (18)
H21B—C21—H21C	109.5	O8—Cl2—O6	109.85 (19)

C2—N1—C1—N2	0.9 (4)	C23—N3—C22—N4	−0.1 (4)
C4—N1—C1—N2	−176.1 (3)	C25—N3—C22—N4	−178.6 (3)
C3—N2—C1—N1	−0.6 (4)	C24—N4—C22—N3	0.0 (4)
C13—N2—C1—N1	−178.5 (3)	C34—N4—C22—N3	179.8 (3)
C1—N1—C2—C3	−0.9 (4)	C22—N3—C23—C24	0.2 (4)
C4—N1—C2—C3	176.0 (3)	C25—N3—C23—C24	178.6 (3)
N1—C2—C3—N2	0.5 (4)	N3—C23—C24—N4	−0.2 (4)
C1—N2—C3—C2	0.0 (4)	C22—N4—C24—C23	0.1 (4)
C13—N2—C3—C2	177.9 (3)	C34—N4—C24—C23	−179.6 (3)

C1—N1—C4—C9	82.7 (4)	C22—N3—C25—C30	68.2 (5)
C2—N1—C4—C9	−93.7 (4)	C23—N3—C25—C30	−110.0 (4)
C1—N1—C4—C5	−97.7 (4)	C22—N3—C25—C26	−110.3 (4)
C2—N1—C4—C5	85.9 (4)	C23—N3—C25—C26	71.5 (5)
C9—C4—C5—C6	−1.9 (5)	C30—C25—C26—C27	−3.1 (5)
N1—C4—C5—C6	178.5 (3)	N3—C25—C26—C27	175.3 (3)
C9—C4—C5—C10	179.2 (4)	C30—C25—C26—C31	177.2 (4)
N1—C4—C5—C10	−0.5 (5)	N3—C25—C26—C31	−4.4 (5)
C4—C5—C6—C7	0.4 (6)	C25—C26—C27—C28	3.4 (6)
C10—C5—C6—C7	179.3 (4)	C31—C26—C27—C28	−176.9 (4)
C5—C6—C7—C8	0.6 (6)	C26—C27—C28—C29	−2.0 (6)
C5—C6—C7—C11	−178.8 (4)	C26—C27—C28—C32	178.1 (4)
C6—C7—C8—C9	−0.2 (6)	C27—C28—C29—C30	0.1 (6)
C11—C7—C8—C9	179.2 (4)	C32—C28—C29—C30	−180.0 (4)
C5—C4—C9—C8	2.3 (5)	C26—C25—C30—C29	1.4 (5)
N1—C4—C9—C8	−178.1 (3)	N3—C25—C30—C29	−177.0 (3)
C5—C4—C9—C12	−178.9 (3)	C26—C25—C30—C33	−178.9 (4)
N1—C4—C9—C12	0.8 (5)	N3—C25—C30—C33	2.7 (5)
C7—C8—C9—C4	−1.1 (5)	C28—C29—C30—C25	0.2 (6)
C7—C8—C9—C12	180.0 (3)	C28—C29—C30—C33	−179.5 (4)
C1—N2—C13—C14	−60.9 (5)	C22—N4—C34—C39	115.3 (4)
C3—N2—C13—C14	121.6 (4)	C24—N4—C34—C39	−65.0 (5)
C1—N2—C13—C18	118.6 (4)	C22—N4—C34—C35	−62.8 (5)
C3—N2—C13—C18	−58.9 (5)	C24—N4—C34—C35	117.0 (4)
C18—C13—C14—C15	−0.7 (5)	C39—C34—C35—C36	−1.5 (5)
N2—C13—C14—C15	178.8 (3)	N4—C34—C35—C36	176.5 (3)
C18—C13—C14—C19	−178.4 (4)	C39—C34—C35—C40	−179.2 (3)
N2—C13—C14—C19	1.0 (5)	N4—C34—C35—C40	−1.2 (5)
C13—C14—C15—C16	0.5 (6)	C34—C35—C36—C37	0.2 (5)
C19—C14—C15—C16	178.3 (4)	C40—C35—C36—C37	177.9 (3)
C14—C15—C16—C17	0.1 (6)	C35—C36—C37—C38	1.8 (5)
C14—C15—C16—C20	−177.7 (3)	C35—C36—C37—C41	−175.4 (3)
C15—C16—C17—C18	−0.6 (6)	C36—C37—C38—C39	−2.7 (5)
C20—C16—C17—C18	177.2 (3)	C41—C37—C38—C39	174.5 (3)
C16—C17—C18—C13	0.4 (5)	C35—C34—C39—C38	0.7 (5)
C16—C17—C18—C21	−177.5 (3)	N4—C34—C39—C38	−177.3 (3)
C14—C13—C18—C17	0.3 (5)	C35—C34—C39—C42	178.4 (3)
N2—C13—C18—C17	−179.2 (3)	N4—C34—C39—C42	0.4 (5)
C14—C13—C18—C21	178.2 (4)	C37—C38—C39—C34	1.4 (5)
N2—C13—C18—C21	−1.3 (5)	C37—C38—C39—C42	−176.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···Cl1	0.95	2.92	3.639 (3)	134
C1—H1*A*···O1	0.95	2.16	3.078 (4)	162
C2—H2*A*···O8^{i}	0.95	2.61	3.191 (5)	120
C3—H3*A*···O7^{i}	0.95	2.38	3.225 (5)	148

C22—H22*A*···Cl2	0.95	2.88	3.804 (4)	165
C22—H22*A*···O5	0.95	2.37	3.119 (4)	136
C22—H22*A*···O6	0.95	2.34	3.259 (4)	163
C24—H24*A*···Cl1^{ii}	0.95	2.96	3.663 (4)	132
C24—H24*A*···O3^{ii}	0.95	2.50	3.295 (5)	141

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