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# 2,4-Dimethylpyrido[1,2-*a*]pyrimidin-5-ium perchlorate

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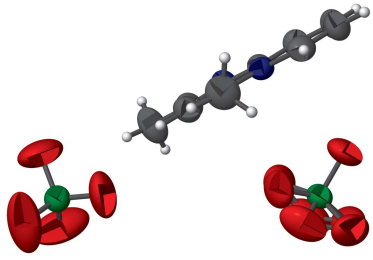
**Keywords:** crystal structure; pyrido[1,2-a]pyri-]pyrimidin-5-ium; pyrimidinium; perchlorate; hydrogen bonding; framework.

**CCDC reference**: 1507737

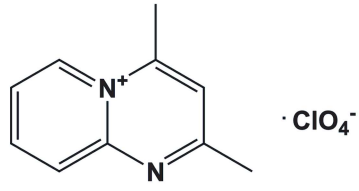
**Structural data**: full structural data are available from iucrdata.iucr.org

In the title molecular salt, $C_{10}H_{11}N_2^+\cdot ClO_4^-$, the pyrido[1,2-*a*]pyrimidin-5-ium cation is planar, with an r.m.s. deviation of 0.027 Å for all 12 non-H atoms. The perchlorate anions are distributed over two twofold rotation axes; for one, three of the O atoms are disordered over two sites (occupancies of 1/2). In the crystal, the cations are linked *via* C—H···N hydrogen bonds, forming chains propagating along [001]. The chains are linked by a C—H···O hydrogen bond involving the non-disordered perchlorate anion, forming double layers parallel to the *bc* plane. These layers are linked by a number of weak C—H···O hydrogen bonds involving the disordered perchlorate anion, forming a three-dimensional framework.

**3D view**



**Chemical scheme**




## Structure description

Reports on the crystal structures of pyrido[1,2-*a*]pyrimidin-5-ium cations are rare (Koval'chukova *et al.*, 2000, 2003). The title compound was obtained when studying the reaction of the ligand 5,7-bis(2-aminopyridine)-5*H*,-6,7-dihydropyrrolo[3.4-*b*]pyrazine (L1) with $Mn(ClO_4)_2\cdot 6H_2O$ in methanol in the presence of triethylamine (Posel, 1998). The solid obtained from this reaction was recrystallized from a mixture of solvents (methanol/acetonitrile/water) also containing acetylacetone, and dark-brown crystals of the title molecular salt were obtained.

The molecular structure of the title molecular salt is illustrated in Fig. 1. The perchlorate anions are distributed over two twofold rotation axes and for one, involving atom Cl1, three O atoms are disordered over two sites (occupancies of 1/2). The pyrido[1,2-*a*]pyrimidin-5-ium cation is planar (r.m.s. deviation of 0.027 Å for all twelve non-H atoms). The bond distances and angles in the cation are very similar to those

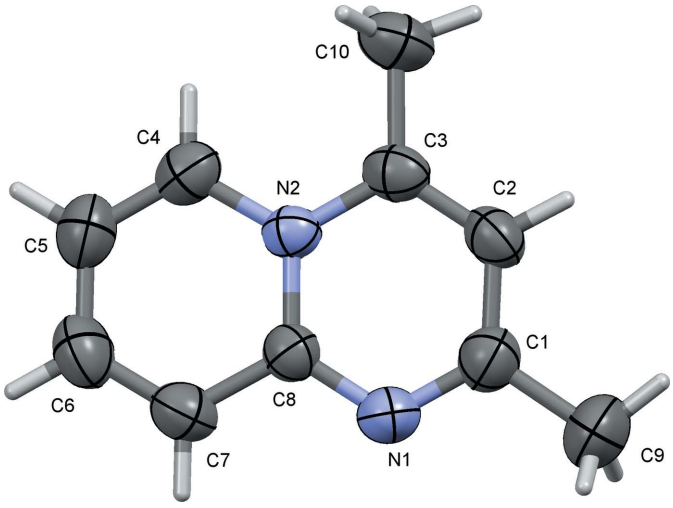



**Figure 1**
A view of the molecular structure of the title molecular salt, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level. For clarity, the perchlorate anions have been omitted.

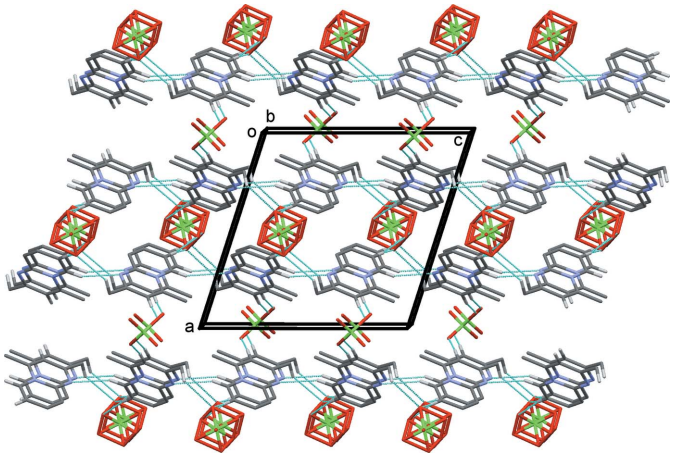



**Figure 2**
A view along the *b* axis of the crystal packing of the title molecular salt. The hydrogen bonds are shown as dashed lines (see Table 1) and only the H atoms involved in these interactions have been included.

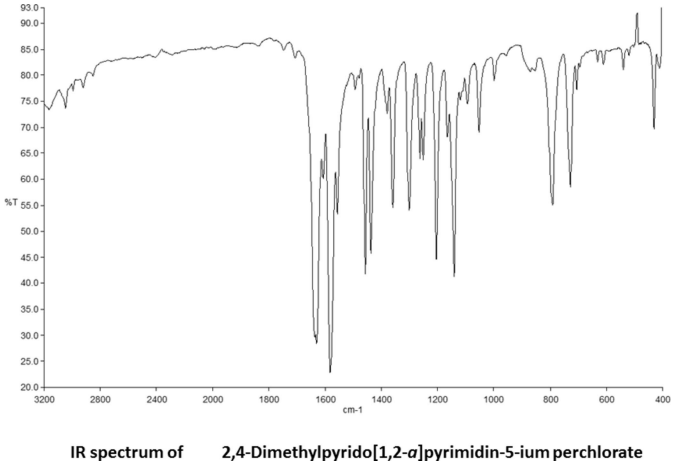



**Figure 3**
The IR spectrum of the title molecular salt.

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C4—H4···N1$^{i}$ | 0.93 | 2.49 | 3.349 (6) | 154 |
| C2—H2···O21 | 0.93 | 2.47 | 3.345 (7) | 158 |
| C5—H5···O12$^{ii}$ | 0.93 | 2.62 | 3.541 (14) | 170 |
| C5—H5···O14$^{iii}$ | 0.93 | 2.61 | 3.449 (12) | 150 |
| C9—H9$B$···O14$^{iv}$ | 0.96 | 2.64 | 3.493 (13) | 149 |

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

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_{10}H_{11}N_2^+\cdot ClO_4^-$ |
| $M_r$ | 258.66 |
| Crystal system, space group | Monoclinic, $P2/c$ |
| Temperature (K) | 293 |
| $a, b, c$ (Å) | 12.5178 (11), 7.8321 (9), 12.6354 (15) |
| $\beta$ (°) | 107.763 (8) |
| $V$ (Å$^3$) | 1179.7 (2) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.33 |
| Crystal size (mm) | 0.30 × 0.27 × 0.27 |
| Data collection | |
| Diffractometer | Stoe AED2 four-circle |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 2198, 2198, 1447 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.606 |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.072, 0.168, 1.13 |
| No. of reflections | 2198 |
| No. of parameters | 172 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.43, −0.24 |

Computer programs: *STADI4* and *X-RED* (Stoe & Cie, 1997), *SHELXS97* (Sheldrick, 2008), *Mercury* (Macrae *et al.*, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

observed for 2,4-dimethyl-9-hydroxypyrido[1,2-*a*]pyrimidi-]pyrimidinium perchlorate (Koval'chukova *et al.*, 2000).

In the crystal, the cations are linked *via* C—H···N hydrogen bonds, forming chains propagating along the *c*-axis direction (Table 1 and Fig. 2). The chains are linked by a C—H···O interaction involving the Cl2 perchlorate anion, forming double layers parallel to the *bc* plane. The layers are linked by weak C—H···O hydrogen bonds (H···$A$ > 2.6 Å), involving the Cl1 disordered perchlorate anion, forming a three-dimensional framework (Table 1 and Fig. 2).

## Synthesis and crystallization

To a mixture of 5,7-bis(2-aminopyridine)-5*H*,-6,7-dihydro-pyrrolo[3.4 − *b*]pyrazine [L1] (0.0646 g, 0.0001 mol) in 7 ml of dry methanol and 0.1 ml of triethylamine was added $Mn(ClO_4)_2\cdot 6H_2O$ (0.0362 g, 0.0001 mol) in 3 ml of dry methanol, and the mixture stirred at room temperature under nitrogen for four days. The mixture was then filtered and the filtrate left to evaporate, but no crystals were obtained. The

solid left after evaporation of the solvent was recrystallized several times from different solvents (containing acetylacetone). Finally a mixture of solvents, methanol/acetonitrile/water (1/4/3) was used and it gave a small amount of brown block-like crystals that were examined by X-ray diffraction analysis, and shown to be the title molecular salt (yield 0.0036 g, m.p. > 623 K). The IR spectrum (KBr pellet, $cm^{-1}$) is shown in Fig. 3. Compound L1 was synthesized by reacting 2,3-dicyanopyrazine with 2-aminopyridine (Posel, 1998). It is possible that during the reaction of L1 with $Mn(ClO_4)_2$, it decomposed reforming 2-aminopyridine which then reacted with the acetylacetone to form the title compound.

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The perchlorate anions are distributed over two twofold rotation axes and for one, involving atom Cl1, the O atoms (O12–O14) are disordered with occupancies of 0.5. Only one equivalent of data were measured, hence $R_{int} = 0$.

## Acknowledgements

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# full crystallographic data

*IUCrData* (2016). **1**, x161543 [https://doi.org/10.1107/S2414314616015431]

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### 2,4-Dimethylpyrido[1,2-*a*]pyrimidin-5-ium perchlorate

*Crystal data*

$C_{10}H_{11}N_2^+\cdot ClO_4^-$
$M_r$ = 258.66
Monoclinic, $P2/c$
$a$ = 12.5178 (11) Å
$b$ = 7.8321 (9) Å
$c$ = 12.6354 (15) Å
$\beta$ = 107.763 (8)°
$V$ = 1179.7 (2) Å$^3$
$Z$ = 4
$F(000)$ = 536
$D_x$ = 1.456 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 25 reflections
$\theta$ = 14.1–17.4°
$\mu$ = 0.33 mm$^{-1}$
$T$ = 293 K
Block, brown
0.30 × 0.27 × 0.27 mm

*Data collection*

Stoe AED2 four-circle diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
2198 measured reflections
2198 independent reflections
1447 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.0
$\theta_{max}$ = 25.5°, $\theta_{min}$ = 2.6°
$h$ = −15→14
$k$ = 0→9
$l$ = 0→15
2 standard reflections every 120 min
intensity decay: 2%

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.072
$wR(F^2)$ = 0.168
$S$ = 1.13
2198 reflections
172 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0364P)^2 + 1.9648P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.43 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0139 (17)

*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* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| N1 | 0.2789 (3) | 0.1607 (5) | 0.4607 (3) | 0.0569 (10) | |
| N2 | 0.2637 (3) | 0.0349 (4) | 0.2851 (3) | 0.0448 (9) | |
| C1 | 0.2140 (4) | 0.2878 (6) | 0.4095 (4) | 0.0611 (13) | |
| C2 | 0.1683 (4) | 0.2900 (6) | 0.2934 (4) | 0.0579 (12) | |
| H2 | 0.1214 | 0.3791 | 0.2591 | 0.069* | |
| C3 | 0.1918 (4) | 0.1641 (6) | 0.2309 (4) | 0.0540 (11) | |
| C4 | 0.2936 (4) | −0.0962 (6) | 0.2268 (4) | 0.0571 (12) | |
| H4 | 0.2671 | −0.0963 | 0.1496 | 0.069* | |
| C5 | 0.3607 (4) | −0.2242 (6) | 0.2802 (5) | 0.0681 (14) | |
| H5 | 0.3798 | −0.3117 | 0.2396 | 0.082* | |
| C6 | 0.4009 (4) | −0.2262 (7) | 0.3946 (5) | 0.0697 (14) | |
| H6 | 0.4465 | −0.3153 | 0.4311 | 0.084* | |
| C7 | 0.3741 (4) | −0.0984 (6) | 0.4536 (4) | 0.0607 (13) | |
| H7 | 0.4024 | −0.0992 | 0.5307 | 0.073* | |
| C8 | 0.3036 (3) | 0.0364 (5) | 0.3996 (3) | 0.0481 (10) | |
| C9 | 0.1890 (5) | 0.4263 (8) | 0.4800 (5) | 0.0931 (19) | |
| H9C | 0.1338 | 0.5022 | 0.4343 | 0.140* | |
| H9B | 0.2564 | 0.4888 | 0.5154 | 0.140* | |
| H9A | 0.1607 | 0.3766 | 0.5356 | 0.140* | |
| C10 | 0.1435 (5) | 0.1578 (7) | 0.1067 (4) | 0.0770 (16) | |
| H10C | 0.0931 | 0.2521 | 0.0817 | 0.115* | |
| H10B | 0.1033 | 0.0526 | 0.0852 | 0.115* | |
| H10A | 0.2030 | 0.1646 | 0.0737 | 0.115* | |
| Cl1 | 0.5000 | 0.3239 (2) | 0.2500 | 0.0559 (5) | |
| O11 | 0.5000 | 0.1448 (6) | 0.2500 | 0.101 (2) | |
| O12 | 0.5458 (15) | 0.4207 (12) | 0.3408 (8) | 0.113 (4) | 0.5 |
| O13 | 0.5533 (12) | 0.3630 (13) | 0.1648 (11) | 0.116 (4) | 0.5 |
| O14 | 0.3885 (7) | 0.3640 (14) | 0.1956 (14) | 0.125 (5) | 0.5 |
| Cl2 | 0.0000 | 0.7762 (2) | 0.2500 | 0.0607 (5) | |
| O21 | 0.0652 (5) | 0.6759 (7) | 0.2040 (5) | 0.151 (2) | |
| O22 | −0.0649 (5) | 0.8791 (8) | 0.1674 (4) | 0.163 (3) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.068 (2) | 0.057 (2) | 0.044 (2) | 0.005 (2) | 0.0147 (19) | −0.0025 (19) |
| N2 | 0.049 (2) | 0.047 (2) | 0.0399 (19) | −0.0041 (17) | 0.0152 (16) | 0.0012 (17) |
| C1 | 0.072 (3) | 0.053 (3) | 0.061 (3) | 0.001 (3) | 0.023 (2) | −0.003 (2) |
| C2 | 0.062 (3) | 0.051 (3) | 0.056 (3) | 0.004 (2) | 0.011 (2) | 0.011 (2) |
| C3 | 0.057 (3) | 0.057 (3) | 0.047 (2) | −0.009 (2) | 0.015 (2) | 0.006 (2) |
| C4 | 0.065 (3) | 0.056 (3) | 0.053 (3) | −0.008 (2) | 0.023 (2) | −0.008 (2) |
| C5 | 0.073 (3) | 0.054 (3) | 0.080 (4) | 0.007 (3) | 0.028 (3) | −0.006 (3) |
| C6 | 0.063 (3) | 0.063 (3) | 0.078 (4) | 0.013 (3) | 0.014 (3) | 0.007 (3) |
| C7 | 0.060 (3) | 0.066 (3) | 0.051 (3) | 0.006 (3) | 0.011 (2) | 0.007 (2) |
| C8 | 0.051 (3) | 0.047 (2) | 0.047 (3) | −0.001 (2) | 0.015 (2) | 0.002 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.120 (5) | 0.076 (4) | 0.079 (4) | 0.025 (4) | 0.023 (4) | −0.016 (3) |
| C10 | 0.091 (4) | 0.085 (4) | 0.050 (3) | 0.007 (3) | 0.013 (3) | 0.014 (3) |
| Cl1 | 0.0524 (10) | 0.0447 (9) | 0.0658 (11) | 0.000 | 0.0110 (8) | 0.000 |
| O11 | 0.124 (5) | 0.044 (3) | 0.141 (6) | 0.000 | 0.048 (4) | 0.000 |
| O12 | 0.172 (12) | 0.080 (7) | 0.066 (6) | −0.005 (8) | 0.005 (7) | −0.020 (5) |
| O13 | 0.143 (9) | 0.099 (7) | 0.141 (11) | −0.010 (7) | 0.098 (9) | 0.011 (7) |
| O14 | 0.051 (5) | 0.095 (8) | 0.204 (15) | 0.026 (5) | 0.003 (7) | 0.019 (8) |
| Cl2 | 0.0508 (9) | 0.0570 (10) | 0.0725 (12) | 0.000 | 0.0161 (8) | 0.000 |
| O21 | 0.148 (5) | 0.124 (4) | 0.214 (6) | 0.063 (4) | 0.103 (5) | 0.008 (4) |
| O22 | 0.174 (5) | 0.210 (6) | 0.104 (4) | 0.122 (5) | 0.041 (3) | 0.056 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C1 | 1.322 (6) | C10—H10C | 0.9600 |
| N1—C8 | 1.337 (5) | C10—H10B | 0.9600 |
| N2—C8 | 1.378 (5) | C10—H10A | 0.9600 |
| N2—C4 | 1.381 (5) | Cl1—O12$^{i}$ | 1.349 (9) |
| N2—C3 | 1.388 (5) | Cl1—O12 | 1.349 (9) |
| C1—C2 | 1.402 (6) | Cl1—O14 | 1.390 (8) |
| C1—C9 | 1.497 (7) | Cl1—O14$^{i}$ | 1.391 (8) |
| C2—C3 | 1.351 (6) | Cl1—O11 | 1.402 (5) |
| C2—H2 | 0.9300 | Cl1—O13 | 1.461 (8) |
| C3—C10 | 1.501 (6) | Cl1—O13$^{i}$ | 1.461 (8) |
| C4—C5 | 1.349 (7) | O12—O14$^{i}$ | 1.147 (13) |
| C4—H4 | 0.9300 | O12—O13$^{i}$ | 1.301 (14) |
| C5—C6 | 1.378 (7) | O13—O12$^{i}$ | 1.301 (14) |
| C5—H5 | 0.9300 | O13—O14$^{i}$ | 1.690 (15) |
| C6—C7 | 1.350 (7) | O14—O12$^{i}$ | 1.147 (13) |
| C6—H6 | 0.9300 | O14—O13$^{i}$ | 1.690 (15) |
| C7—C8 | 1.411 (6) | Cl2—O22$^{ii}$ | 1.371 (5) |
| C7—H7 | 0.9300 | Cl2—O22 | 1.371 (5) |
| C9—H9C | 0.9600 | Cl2—O21$^{ii}$ | 1.382 (5) |
| C9—H9B | 0.9600 | Cl2—O21 | 1.382 (5) |
| C9—H9A | 0.9600 | | |
| | | | |
| C1—N1—C8 | 118.8 (4) | H10C—C10—H10A | 109.5 |
| C8—N2—C4 | 119.7 (4) | H10B—C10—H10A | 109.5 |
| C8—N2—C3 | 119.0 (4) | O12$^{i}$—Cl1—O12 | 111.5 (9) |
| C4—N2—C3 | 121.2 (4) | O12$^{i}$—Cl1—O14 | 49.5 (6) |
| N1—C1—C2 | 121.0 (4) | O12—Cl1—O14 | 113.3 (7) |
| N1—C1—C9 | 117.6 (4) | O12$^{i}$—Cl1—O14$^{i}$ | 113.3 (7) |
| C2—C1—C9 | 121.4 (5) | O12—Cl1—O14$^{i}$ | 49.5 (6) |
| C3—C2—C1 | 120.7 (4) | O14—Cl1—O14$^{i}$ | 153.9 (9) |
| C3—C2—H2 | 119.6 | O12$^{i}$—Cl1—O11 | 124.2 (4) |
| C1—C2—H2 | 119.6 | O12—Cl1—O11 | 124.2 (4) |
| C2—C3—N2 | 117.8 (4) | O14—Cl1—O11 | 103.1 (5) |
| C2—C3—C10 | 123.1 (5) | O14$^{i}$—Cl1—O11 | 103.1 (5) |
| N2—C3—C10 | 119.1 (4) | O12$^{i}$—Cl1—O13 | 55.0 (6) |

| | | | |
|---|---|---|---|
| C5—C4—N2 | 121.0 (4) | O12—Cl1—O13 | 109.7 (7) |
| C5—C4—H4 | 119.5 | O14—Cl1—O13 | 101.7 (7) |
| N2—C4—H4 | 119.5 | O14$^{i}$—Cl1—O13 | 72.7 (7) |
| C4—C5—C6 | 120.4 (5) | O11—Cl1—O13 | 102.1 (4) |
| C4—C5—H5 | 119.8 | O12$^{i}$—Cl1—O13$^{i}$ | 109.7 (7) |
| C6—C5—H5 | 119.8 | O12—Cl1—O13$^{i}$ | 55.0 (6) |
| C7—C6—C5 | 119.9 (5) | O14—Cl1—O13$^{i}$ | 72.7 (6) |
| C7—C6—H6 | 120.1 | O14$^{i}$—Cl1—O13$^{i}$ | 101.7 (7) |
| C5—C6—H6 | 120.1 | O11—Cl1—O13$^{i}$ | 102.1 (4) |
| C6—C7—C8 | 120.8 (5) | O13—Cl1—O13$^{i}$ | 155.8 (9) |
| C6—C7—H7 | 119.6 | O14$^{i}$—O12—O13$^{i}$ | 129.2 (11) |
| C8—C7—H7 | 119.6 | O14$^{i}$—O12—Cl1 | 67.2 (7) |
| N1—C8—N2 | 122.6 (4) | O13$^{i}$—O12—Cl1 | 66.9 (7) |
| N1—C8—C7 | 119.1 (4) | O12$^{i}$—O13—Cl1 | 58.1 (5) |
| N2—C8—C7 | 118.3 (4) | O12$^{i}$—O13—O14$^{i}$ | 98.9 (8) |
| C1—C9—H9C | 109.5 | Cl1—O13—O14$^{i}$ | 51.7 (4) |
| C1—C9—H9B | 109.5 | O12$^{i}$—O14—Cl1 | 63.3 (6) |
| H9C—C9—H9B | 109.5 | O12$^{i}$—O14—O13$^{i}$ | 106.6 (10) |
| C1—C9—H9A | 109.5 | Cl1—O14—O13$^{i}$ | 55.6 (5) |
| H9C—C9—H9A | 109.5 | O22$^{ii}$—Cl2—O22 | 108.0 (6) |
| H9B—C9—H9A | 109.5 | O22$^{ii}$—Cl2—O21$^{ii}$ | 107.6 (3) |
| C3—C10—H10C | 109.5 | O22—Cl2—O21$^{ii}$ | 111.4 (4) |
| C3—C10—H10B | 109.5 | O22$^{ii}$—Cl2—O21 | 111.4 (4) |
| H10C—C10—H10B | 109.5 | O22—Cl2—O21 | 107.6 (3) |
| C3—C10—H10A | 109.5 | O21$^{ii}$—Cl2—O21 | 110.7 (5) |

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| C8—N1—C1—C2 | 2.2 (7) | O13$^{i}$—Cl1—O12—O14$^{i}$ | 157.6 (11) |
| C8—N1—C1—C9 | −179.3 (5) | O12$^{i}$—Cl1—O12—O13$^{i}$ | 99.6 (8) |
| N1—C1—C2—C3 | −1.6 (7) | O14$^{i}$—Cl1—O12—O13$^{i}$ | −157.6 (11) |
| C9—C1—C2—C3 | 179.9 (5) | O14—Cl1—O12—O13$^{i}$ | 45.8 (9) |
| C1—C2—C3—N2 | −1.0 (7) | O11—Cl1—O12—O13$^{i}$ | −80.4 (8) |
| C1—C2—C3—C10 | 178.6 (5) | O13—Cl1—O12—O13$^{i}$ | 158.7 (8) |
| C8—N2—C3—C2 | 2.8 (6) | O12—Cl1—O13—O12$^{i}$ | −103.0 (11) |
| C4—N2—C3—C2 | −178.6 (4) | O14$^{i}$—Cl1—O13—O12$^{i}$ | −136.4 (9) |
| C8—N2—C3—C10 | −176.8 (4) | O14—Cl1—O13—O12$^{i}$ | 17.2 (9) |
| C4—N2—C3—C10 | 1.8 (6) | O11—Cl1—O13—O12$^{i}$ | 123.5 (6) |
| C8—N2—C4—C5 | 0.6 (6) | O13$^{i}$—Cl1—O13—O12$^{i}$ | −56.5 (6) |
| C3—N2—C4—C5 | −177.9 (4) | O12$^{i}$—Cl1—O13—O14$^{i}$ | 136.4 (9) |
| N2—C4—C5—C6 | −0.1 (7) | O12—Cl1—O13—O14$^{i}$ | 33.4 (7) |
| C4—C5—C6—C7 | −0.7 (8) | O14—Cl1—O13—O14$^{i}$ | 153.6 (9) |
| C5—C6—C7—C8 | 1.0 (8) | O11—Cl1—O13—O14$^{i}$ | −100.1 (5) |
| C1—N1—C8—N2 | −0.3 (7) | O13$^{i}$—Cl1—O13—O14$^{i}$ | 79.9 (5) |
| C1—N1—C8—C7 | 179.1 (4) | O12—Cl1—O14—O12$^{i}$ | 99.1 (12) |
| C4—N2—C8—N1 | 179.1 (4) | O14$^{i}$—Cl1—O14—O12$^{i}$ | 55.9 (7) |
| C3—N2—C8—N1 | −2.3 (6) | O11—Cl1—O14—O12$^{i}$ | −124.1 (7) |
| C4—N2—C8—C7 | −0.3 (6) | O13—Cl1—O14—O12$^{i}$ | −18.6 (9) |
| C3—N2—C8—C7 | 178.3 (4) | O13$^{i}$—Cl1—O14—O12$^{i}$ | 137.1 (10) |
| C6—C7—C8—N1 | −179.9 (4) | O12$^{i}$—Cl1—O14—O13$^{i}$ | −137.1 (10) |

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| C6—C7—C8—N2 | −0.5 (7) | O12—Cl1—O14—O13$^{i}$ | −37.9 (7) |
| O12$^{i}$—Cl1—O12—O14$^{i}$ | −102.8 (10) | O14$^{i}$—Cl1—O14—O13$^{i}$ | −81.2 (5) |
| O14—Cl1—O12—O14$^{i}$ | −156.6 (9) | O11—Cl1—O14—O13$^{i}$ | 98.8 (5) |
| O11—Cl1—O12—O14$^{i}$ | 77.2 (10) | O13—Cl1—O14—O13$^{i}$ | −155.6 (9) |
| O13—Cl1—O12—O14$^{i}$ | −43.7 (10) | | |

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

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C4—H4···N1$^{iii}$ | 0.93 | 2.49 | 3.349 (6) | 154 |
| C2—H2···O21 | 0.93 | 2.47 | 3.345 (7) | 158 |
| C5—H5···O12$^{iv}$ | 0.93 | 2.62 | 3.541 (14) | 170 |
| C5—H5···O14$^{v}$ | 0.93 | 2.61 | 3.449 (12) | 150 |
| C9—H9*B*···O14$^{vi}$ | 0.96 | 2.64 | 3.493 (13) | 149 |

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