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# 4-Amino-3,5-dichloropyridinium 3-hydroxypicolinate monohydrate

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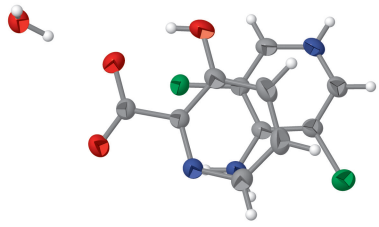
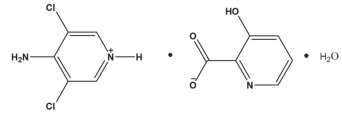
**Structural data:** full structural data are available from iucrdata.iucr.org

In the title hydrated salt, $C_5H_5Cl_2N_2^+\cdot C_6H_4NO_3^-\cdot H_2O$, the pyridine N atom of the cation is protonated and an intramolecular O—H···O hydrogen bond is observed in the anion, which generates an $S(6)$ ring. The crystal packing features N—H···N, O—H···O, N—H···O, C—H···Cl and C—H···O hydrogen bonds, which generate a three-dimensional network.

3D view



Chemical scheme



## Structure description

4-Aminopyridine and its derivatives are used clinically to treat Lambert–Eaton myasthenic syndrome and multiple sclerosis because they block potassium channels, which prolongs action potentials and increases transmitter release at the neuromuscular junction (Judge & Bever, 2006). Picolinic acid, which contains N and O donors, has attracted much attention for the design and synthesis of self-assembling systems (*e.g.*, Steiner, 2002). In this regard, 3-hydroxypicolinic acid is of interest because it can be used as a neutral ligand or, depending on the pH value, as an anionic or cationic ligand. In addition, due to the arrangement of its functional groups, it can act as a monodentate or bidentate ligand, which allows it to form five- or six-membered chelate rings. As part of our work in this area, we now report the synthesis and structure of the title hydrated molecular salt.

The asymmetric unit (Fig. 1) of the title salt contains a 4-amino-3,5-dichloropyridinium cation, a 3-hydroxy picolinate anion and a water molecule. The pyridinium cation is essentially planar, with a maximum deviation of 0.010 (2) Å for atom C2. A wider than normal angle [C5—N1—C1 = 120.41 (12)°] is subtended at the protonated N1 atom. In the anion, a typical intramolecular O—H···O hydrogen bond, which generates an $S(6)$ ring, is seen. In the extended structure, the cations, anions and water molecules are

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O3—H3···O2 | 0.82 | 1.79 | 2.5155 (17) | 147 |
| N1—H1···N3$^{i}$ | 0.86 | 1.90 | 2.7546 (17) | 171 |
| N2—H2$A$···O1$W^{ii}$ | 0.86 | 2.13 | 2.9414 (17) | 157 |
| N2—H2$B$···O1$W^{iii}$ | 0.86 | 2.05 | 2.8269 (17) | 149 |
| O1$W$—H1$W$···O2 | 0.85 | 1.90 | 2.7442 (17) | 170 |
| O1$W$—H2$W$···O1$^{iv}$ | 0.85 | 1.98 | 2.8181 (18) | 170 |
| C5—H5···O1$^{i}$ | 0.93 | 2.31 | 2.9864 (18) | 129 |
| C5—H5···Cl2$^{v}$ | 0.93 | 2.97 | 3.7363 (16) | 141 |
| C7—H7···O3$^{vi}$ | 0.93 | 2.52 | 3.399 (2) | 157 |

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

connected by N—H···N, O—H···O, C—H···Cl, N—H···O and C—H···O hydrogen bonds (Table 1), forming a three-dimensional network (Figs. 2 and 3).

A search of the Cambridge Structural Database (Version 5.43, update November 2022; Groom *et al.*, 2016) for the 3,5-dichloro-4-amino pyridine fragment with additional substituents yielded hexaaquamagnesium(II) bis(4-amino-3,5,6-trichloro-picolinate) tetrahydrate (CSD refcode BAWGOV; Smith *et al.*, 1981), [(4-amino-3,5-dichloro-6-fluoropyridin-2-yl)oxy]acetic acid (EZONOY; Park *et al.*, 2016), sodium picloramate hexahydrate (CURLIM; Smith *et al.*, 2015), guanidinium 4-amino-3,5,6-trichloropicolinate (GUPICL10; Parthasarathi *et al.*, 1982), and 6-chloro-3-(trifluoromethoxy) pyridine-2-carboxylic acid (MAFTEU; Manteau *et al.*, 2010).

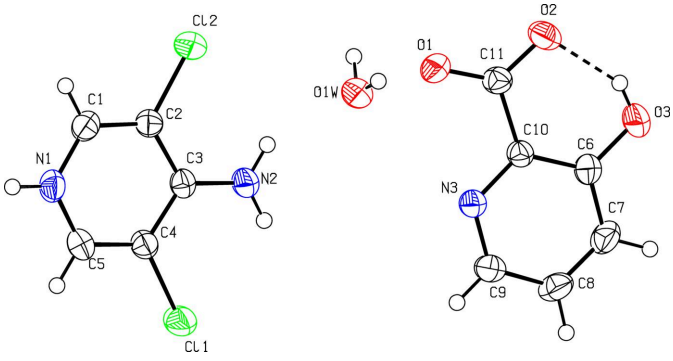



**Figure 1**
The molecular structure of the title compound showing 50% displacement ellipsoids. The intramolecular hydrogen bond is shown with dashed lines.

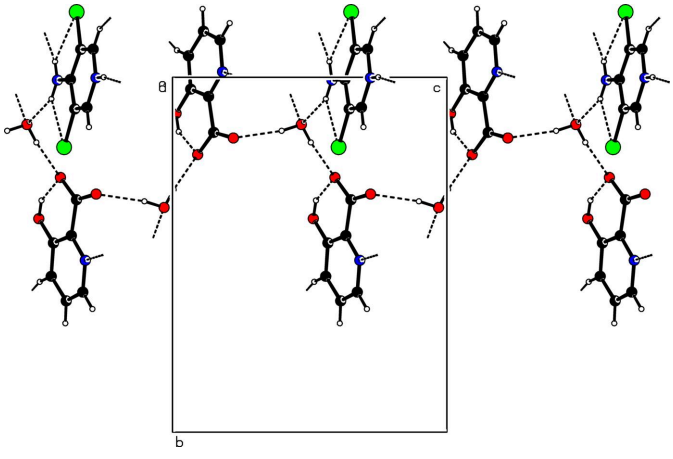



**Figure 2**
One-dimensional supramolecular hydrogen-bonded chain mediated by water molecules in the title compound.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | $C_5H_5Cl_2N_2^+ \cdot C_6H_4NO_3^- \cdot H_2O$ |
| $M_r$ | 320.13 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 296 |
| $a, b, c$ (Å) | 8.4267 (19), 14.084 (3), 10.900 (2) |
| $\beta$ (°) | 91.953 (8) |
| $V$ (Å$^3$) | 1292.9 (5) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.52 |
| Crystal size (mm) | 0.46 × 0.32 × 0.13 |
| | |
| Data collection | |
| Diffractometer | Agilent Xcalibur, Atlas, Gemini |
| Absorption correction | Multi-scan |
| $T_{min}$, $T_{max}$ | 0.819, 0.937 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 45801, 3296, 2853 |
| $R_{int}$ | 0.038 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.675 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.033, 0.091, 1.03 |
| No. of reflections | 3296 |
| No. of parameters | 185 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.24, −0.35 |

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

## Synthesis and crystallization

A hot methanol solution of 3-hydroxy picolinic acid (40 mg) was mixed with a hot aqueous solution of 4-amino 3,5-dichloro pyridine (34 mg). The mixture was cooled slowly and kept at

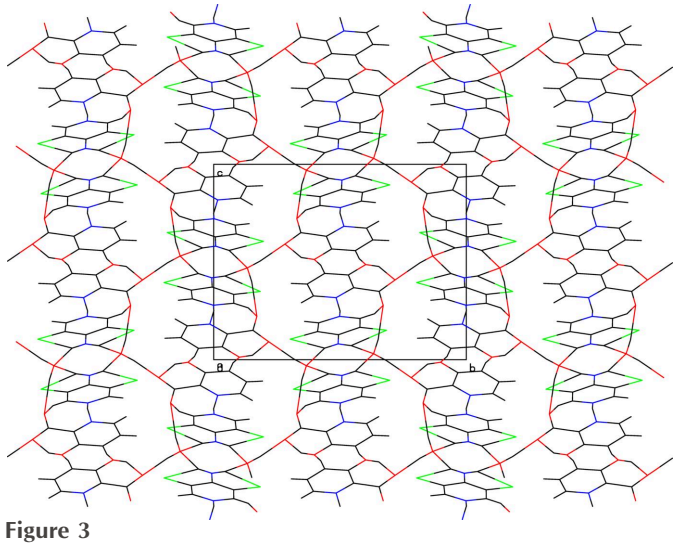



**Figure 3**
Crystal packing viewed down [100] in the title compound.

room temperature. After a few days, colourless block shaped crystals were obtained.

## Refinement

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

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# full crystallographic data

*IUCrData* (2023). **8**, x230821 [https://doi.org/10.1107/S2414314623008210]

## 4-Amino-3,5-dichloropyridinium 3-hydroxypicolinate monohydrate

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**4-Amino-3,5-dichloropyridinium 3-hydroxypyridine-2-carboxylate monohydrate**

*Crystal data*

$C_5H_5Cl_2N_2^+·C_6H_4NO_3^-·H_2O$
$M_r$ = 320.13
Monoclinic, $P2_1/c$
$a$ = 8.4267 (19) Å
$b$ = 14.084 (3) Å
$c$ = 10.900 (2) Å
$\beta$ = 91.953 (8)°
$V$ = 1292.9 (5) Å$^3$
$Z$ = 4
$F(000)$ = 656
$D_x$ = 1.645 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 3676 reflections
$\theta$ = 2.5–28.8°
$\mu$ = 0.52 mm$^{-1}$
$T$ = 296 K
Plate, colourless
0.46 × 0.32 × 0.13 mm

*Data collection*

Agilent Xcalibur, Atlas, Gemini diffractometer
Radiation source: fine-focus sealed tube
$\omega$ scans
Absorption correction: multi-scan
$T_{min}$ = 0.819, $T_{max}$ = 0.937
45801 measured reflections
3296 independent reflections
2853 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.038
$\theta_{max}$ = 28.7°, $\theta_{min}$ = 2.4°
$h$ = −11→11
$k$ = −18→18
$l$ = −14→14

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
$S$ = 1.03
3296 reflections
185 parameters
0 restraints
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.3959P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.35 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.

**Refinement**. The water H atoms were located in a difference Fourier map and allowed to refine freely. The remaining H atoms were positioned geometrically (C—H = 0.93 and N—H = 0.86 Å) and were refined using a riding model, with $U_{iso}$(H) = 1.2 $U_{eq}$(C).

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

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Cl1 | 0.85851 (4) | 0.68420 (3) | 0.15096 (4) | 0.04683 (12) |
| Cl2 | 0.89478 (5) | 0.30311 (2) | 0.10521 (4) | 0.04737 (12) |
| O1 | 0.42463 (12) | 0.67099 (7) | 0.27823 (11) | 0.0439 (3) |
| O2 | 0.61861 (13) | 0.71788 (7) | 0.40818 (11) | 0.0459 (3) |
| O3 | 0.82471 (14) | 0.60173 (8) | 0.48694 (13) | 0.0535 (3) |
| H3 | 0.780642 | 0.653220 | 0.477230 | 0.080* |
| N3 | 0.50360 (13) | 0.48436 (8) | 0.31752 (10) | 0.0321 (2) |
| N1 | 1.19792 (14) | 0.49866 (8) | 0.22147 (11) | 0.0371 (3) |
| H1 | 1.294531 | 0.500301 | 0.249479 | 0.045* |
| N2 | 0.73770 (13) | 0.49223 (8) | 0.08094 (11) | 0.0355 (3) |
| H2A | 0.685153 | 0.544264 | 0.072057 | 0.043* |
| H2B | 0.694906 | 0.439155 | 0.059109 | 0.043* |
| C10 | 0.59379 (14) | 0.55395 (9) | 0.36845 (11) | 0.0288 (3) |
| C11 | 0.53872 (15) | 0.65512 (9) | 0.34965 (13) | 0.0326 (3) |
| C3 | 0.88438 (14) | 0.49383 (9) | 0.12820 (11) | 0.0291 (3) |
| C4 | 0.96128 (15) | 0.57881 (9) | 0.16585 (12) | 0.0321 (3) |
| C6 | 0.73510 (16) | 0.53325 (10) | 0.43364 (13) | 0.0353 (3) |
| C2 | 0.97853 (16) | 0.41096 (9) | 0.14404 (12) | 0.0325 (3) |
| C5 | 1.11412 (16) | 0.57934 (10) | 0.21103 (13) | 0.0362 (3) |
| H5 | 1.160941 | 0.636508 | 0.234961 | 0.043* |
| C1 | 1.13192 (17) | 0.41529 (10) | 0.18845 (13) | 0.0373 (3) |
| H1A | 1.191375 | 0.359836 | 0.195919 | 0.045* |
| C9 | 0.55198 (17) | 0.39478 (10) | 0.32671 (14) | 0.0388 (3) |
| H9 | 0.488901 | 0.347163 | 0.291583 | 0.047* |
| C8 | 0.69351 (18) | 0.37002 (10) | 0.38699 (15) | 0.0428 (3) |
| H8 | 0.725723 | 0.306886 | 0.390273 | 0.051* |
| C7 | 0.78549 (17) | 0.43927 (11) | 0.44166 (15) | 0.0431 (3) |
| H7 | 0.880028 | 0.423758 | 0.483433 | 0.052* |
| O1W | 0.48614 (13) | 0.86692 (8) | 0.53048 (11) | 0.0441 (3) |
| H1W | 0.518086 | 0.816660 | 0.495934 | 0.066* |
| H2W | 0.462944 | 0.849278 | 0.602250 | 0.066* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Cl1 | 0.0388 (2) | 0.03527 (19) | 0.0658 (3) | 0.00513 (13) | −0.00686 (17) | −0.01076 (16) |
| Cl2 | 0.0497 (2) | 0.03070 (18) | 0.0608 (3) | −0.00776 (14) | −0.01108 (18) | 0.00405 (15) |
| O1 | 0.0385 (5) | 0.0339 (5) | 0.0585 (7) | 0.0032 (4) | −0.0120 (5) | 0.0042 (5) |
| O2 | 0.0429 (6) | 0.0320 (5) | 0.0621 (7) | −0.0008 (4) | −0.0073 (5) | −0.0116 (5) |
| O3 | 0.0416 (6) | 0.0449 (6) | 0.0723 (8) | −0.0023 (5) | −0.0249 (6) | −0.0072 (6) |
| N3 | 0.0294 (5) | 0.0297 (5) | 0.0371 (6) | 0.0000 (4) | −0.0023 (4) | −0.0008 (4) |
| N1 | 0.0295 (5) | 0.0419 (6) | 0.0393 (6) | −0.0021 (5) | −0.0075 (5) | 0.0026 (5) |
| N2 | 0.0278 (5) | 0.0345 (6) | 0.0438 (6) | −0.0027 (4) | −0.0051 (5) | −0.0019 (5) |
| C10 | 0.0268 (6) | 0.0282 (6) | 0.0313 (6) | −0.0006 (5) | 0.0004 (5) | −0.0009 (5) |
| C11 | 0.0294 (6) | 0.0290 (6) | 0.0397 (7) | 0.0010 (5) | 0.0023 (5) | −0.0010 (5) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C3 | 0.0273 (6) | 0.0342 (6) | 0.0258 (6) | −0.0031 (5) | 0.0007 (5) | 0.0009 (5) |
| C4 | 0.0299 (6) | 0.0327 (6) | 0.0335 (6) | −0.0009 (5) | −0.0010 (5) | −0.0027 (5) |
| C6 | 0.0290 (6) | 0.0371 (7) | 0.0396 (7) | −0.0004 (5) | −0.0039 (5) | 0.0003 (6) |
| C2 | 0.0340 (6) | 0.0304 (6) | 0.0329 (6) | −0.0047 (5) | −0.0028 (5) | 0.0039 (5) |
| C5 | 0.0323 (6) | 0.0387 (7) | 0.0374 (7) | −0.0054 (5) | −0.0035 (5) | −0.0036 (6) |
| C1 | 0.0357 (7) | 0.0363 (7) | 0.0396 (7) | 0.0008 (5) | −0.0048 (6) | 0.0065 (6) |
| C9 | 0.0387 (7) | 0.0292 (6) | 0.0483 (8) | −0.0009 (5) | −0.0021 (6) | −0.0018 (6) |
| C8 | 0.0413 (8) | 0.0320 (7) | 0.0549 (9) | 0.0084 (6) | 0.0016 (7) | 0.0068 (6) |
| C7 | 0.0327 (7) | 0.0436 (8) | 0.0526 (9) | 0.0074 (6) | −0.0066 (6) | 0.0075 (7) |
| O1W | 0.0418 (6) | 0.0358 (5) | 0.0542 (7) | 0.0069 (4) | −0.0077 (5) | −0.0016 (5) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Cl1—C4 | 1.7233 (14) | C10—C11 | 1.5103 (18) |
| Cl2—C2 | 1.7217 (14) | C3—C4 | 1.4151 (17) |
| O1—C11 | 1.2366 (17) | C3—C2 | 1.4184 (18) |
| O2—C11 | 1.2697 (17) | C4—C5 | 1.3631 (18) |
| O3—C6 | 1.3446 (17) | C6—C7 | 1.392 (2) |
| O3—H3 | 0.8200 | C2—C1 | 1.3661 (19) |
| N3—C9 | 1.3287 (18) | C5—H5 | 0.9300 |
| N3—C10 | 1.3481 (16) | C1—H1A | 0.9300 |
| N1—C5 | 1.3406 (18) | C9—C8 | 1.386 (2) |
| N1—C1 | 1.3430 (18) | C9—H9 | 0.9300 |
| N1—H1 | 0.8600 | C8—C7 | 1.370 (2) |
| N2—C3 | 1.3229 (16) | C8—H8 | 0.9300 |
| N2—H2A | 0.8600 | C7—H7 | 0.9300 |
| N2—H2B | 0.8600 | O1W—H1W | 0.8499 |
| C10—C6 | 1.3965 (18) | O1W—H2W | 0.8500 |
| | | | |
| C6—O3—H3 | 109.5 | O3—C6—C10 | 121.79 (13) |
| C9—N3—C10 | 119.47 (12) | C7—C6—C10 | 118.91 (13) |
| C5—N1—C1 | 120.41 (12) | C1—C2—C3 | 121.66 (12) |
| C5—N1—H1 | 119.8 | C1—C2—Cl2 | 120.10 (11) |
| C1—N1—H1 | 119.8 | C3—C2—Cl2 | 118.24 (10) |
| C3—N2—H2A | 120.0 | N1—C5—C4 | 120.96 (13) |
| C3—N2—H2B | 120.0 | N1—C5—H5 | 119.5 |
| H2A—N2—H2B | 120.0 | C4—C5—H5 | 119.5 |
| N3—C10—C6 | 121.12 (12) | N1—C1—C2 | 120.81 (13) |
| N3—C10—C11 | 117.62 (11) | N1—C1—H1A | 119.6 |
| C6—C10—C11 | 121.24 (11) | C2—C1—H1A | 119.6 |
| O1—C11—O2 | 125.31 (13) | N3—C9—C8 | 122.09 (13) |
| O1—C11—C10 | 118.99 (12) | N3—C9—H9 | 119.0 |
| O2—C11—C10 | 115.68 (12) | C8—C9—H9 | 119.0 |
| N2—C3—C4 | 122.67 (12) | C7—C8—C9 | 119.48 (13) |
| N2—C3—C2 | 123.00 (12) | C7—C8—H8 | 120.3 |
| C4—C3—C2 | 114.33 (11) | C9—C8—H8 | 120.3 |
| C5—C4—C3 | 121.81 (12) | C8—C7—C6 | 118.88 (13) |
| C5—C4—Cl1 | 119.66 (11) | C8—C7—H7 | 120.6 |

| | | | |
|---|---|---|---|
| C3—C4—Cl1 | 118.52 (10) | C6—C7—H7 | 120.6 |
| O3—C6—C7 | 119.29 (12) | H1W—O1W—H2W | 104.5 |
| | | | |
| C9—N3—C10—C6 | −1.8 (2) | C4—C3—C2—C1 | −1.97 (19) |
| C9—N3—C10—C11 | 176.65 (12) | N2—C3—C2—Cl2 | −2.54 (18) |
| N3—C10—C11—O1 | −7.84 (19) | C4—C3—C2—Cl2 | 178.22 (10) |
| C6—C10—C11—O1 | 170.60 (13) | C1—N1—C5—C4 | −0.6 (2) |
| N3—C10—C11—O2 | 173.80 (12) | C3—C4—C5—N1 | 0.0 (2) |
| C6—C10—C11—O2 | −7.76 (19) | Cl1—C4—C5—N1 | −178.74 (11) |
| N2—C3—C4—C5 | −178.05 (13) | C5—N1—C1—C2 | −0.2 (2) |
| C2—C3—C4—C5 | 1.20 (19) | C3—C2—C1—N1 | 1.5 (2) |
| N2—C3—C4—Cl1 | 0.74 (18) | Cl2—C2—C1—N1 | −178.65 (11) |
| C2—C3—C4—Cl1 | 179.99 (10) | C10—N3—C9—C8 | −0.2 (2) |
| N3—C10—C6—O3 | −178.70 (13) | N3—C9—C8—C7 | 1.6 (2) |
| C11—C10—C6—O3 | 2.9 (2) | C9—C8—C7—C6 | −0.9 (2) |
| N3—C10—C6—C7 | 2.4 (2) | O3—C6—C7—C8 | −179.94 (15) |
| C11—C10—C6—C7 | −175.97 (13) | C10—C6—C7—C8 | −1.0 (2) |
| N2—C3—C2—C1 | 177.27 (13) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O3—H3···O2 | 0.82 | 1.79 | 2.5155 (17) | 147 |
| N1—H1···N3$^{i}$ | 0.86 | 1.90 | 2.7546 (17) | 171 |
| N2—H2*A*···O1*W*$^{ii}$ | 0.86 | 2.13 | 2.9414 (17) | 157 |
| N2—H2*B*···O1*W*$^{iii}$ | 0.86 | 2.05 | 2.8269 (17) | 149 |
| O1*W*—H1*W*···O2 | 0.85 | 1.90 | 2.7442 (17) | 170 |
| O1*W*—H2*W*···O1$^{iv}$ | 0.85 | 1.98 | 2.8181 (18) | 170 |
| C5—H5···O1$^{i}$ | 0.93 | 2.31 | 2.9864 (18) | 129 |
| C5—H5···Cl2$^{v}$ | 0.93 | 2.97 | 3.7363 (16) | 141 |
| C7—H7···O3$^{vi}$ | 0.93 | 2.52 | 3.399 (2) | 157 |

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