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2-[2-(2-Pyridyl)ethyl]isoindolinium perchlorate

Ray J. Butcher,* Yohannes T. Tesema, Teshome B. Yisgedu and Yilma Gultneh

Department of Chemistry, Howard University, 525 College Street NW, Washington, DC 20059, USA

Correspondence e-mail: rbutcher99@yahoo.com

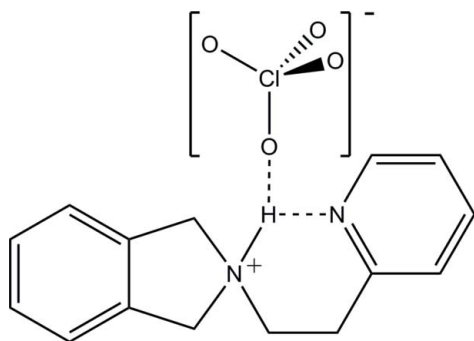
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 Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in solvent or counterion; R factor = 0.044; wR factor = 0.120; data-to-parameter ratio = 6.1.

In the title salt, $\text{C}_{15}\text{H}_{17}\text{N}_2^+\cdot\text{ClO}_4^-$, the isoindoline N atom is protonated and an intramolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bond occurs. In the crystal, $\text{N}-\text{H}\cdots\text{O}$ and numerous weak $\text{C}-\text{H}\cdots\text{O}$ interactions occur between the cation and anion. The O atoms of the perchlorate anion are disordered over four sets of sites with occupancies of 0.438 (4), 0.270 (9), 0.155 (8) and 0.138 (5).

Related literature

For further information on the synthesis, see: Bonnett *et al.* (1983); Meyers & Santiago (1995).



Experimental

Crystal data

 $\text{C}_{15}\text{H}_{17}\text{N}_2^+\cdot\text{ClO}_4^-$
 $M_r = 324.76$

 Orthorhombic, $P2_12_12_1$
 $a = 7.7941$ (9) Å

 $b = 11.7032$ (19) Å

 $c = 16.850$ (3) Å

 $V = 1537.0$ (4) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.27$ mm⁻¹
 $T = 293$ K

 $0.5 \times 0.2 \times 0.08$ mm

Data collection

Bruker P4 diffractometer

 Absorption correction: ψ scan

 (North *et al.*, 1968)

 $T_{\min} = 0.241$, $T_{\max} = 0.265$

(expected range = 0.890–0.979)

2027 measured reflections

2001 independent reflections

 1660 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

3 standard reflections

every 97 reflections

intensity decay: <2%

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.120$
 $S = 1.03$

2001 reflections

329 parameters

245 restraints

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N2}-\text{H2B}\cdots\text{N1}$ | 0.91 | 2.11 | 2.769 (4) | 129 |
| $\text{N2}-\text{H2B}\cdots\text{O4}$ | 0.91 | 2.39 | 3.197 (9) | 148 |
| $\text{N2}-\text{H2B}\cdots\text{O2A}$ | 0.91 | 2.53 | 3.222 (19) | 133 |
| $\text{N2}-\text{H2B}\cdots\text{O1C}$ | 0.91 | 2.59 | 3.390 (16) | 147 |
| $\text{C2}-\text{H2A}\cdots\text{O1B}^{\text{i}}$ | 0.93 | 2.30 | 3.062 (11) | 139 |
| $\text{C3}-\text{H3A}\cdots\text{O1A}^{\text{ii}}$ | 0.93 | 2.62 | 3.193 (10) | 121 |
| $\text{C7}-\text{H7B}\cdots\text{O2A}^{\text{iii}}$ | 0.97 | 2.50 | 3.232 (16) | 132 |
| $\text{C8}-\text{H8A}\cdots\text{O3}$ | 0.97 | 2.55 | 3.383 (10) | 144 |
| $\text{C8}-\text{H8A}\cdots\text{O2B}$ | 0.97 | 2.39 | 3.168 (19) | 136 |
| $\text{C8}-\text{H8A}\cdots\text{O3C}$ | 0.97 | 2.29 | 3.189 (17) | 154 |
| $\text{C8}-\text{H8B}\cdots\text{O4B}^{\text{iv}}$ | 0.97 | 2.48 | 3.167 (17) | 128 |
| $\text{C11}-\text{H11A}\cdots\text{O3A}^{\text{v}}$ | 0.93 | 2.55 | 3.27 (2) | 135 |
| $\text{C11}-\text{H11A}\cdots\text{O3B}^{\text{v}}$ | 0.93 | 2.57 | 3.47 (2) | 162 |
| $\text{C13}-\text{H13A}\cdots\text{O3}^{\text{vi}}$ | 0.93 | 2.48 | 3.299 (9) | 148 |
| $\text{C13}-\text{H13A}\cdots\text{O4A}^{\text{vi}}$ | 0.93 | 2.20 | 2.976 (8) | 140 |
| $\text{C13}-\text{H13A}\cdots\text{O1B}^{\text{vi}}$ | 0.93 | 2.56 | 3.44 (2) | 158 |
| $\text{C13}-\text{H13A}\cdots\text{O3C}^{\text{vi}}$ | 0.93 | 2.70 | 3.444 (17) | 138 |
| $\text{C15}-\text{H15A}\cdots\text{O1}^{\text{iii}}$ | 0.97 | 2.55 | 3.423 (9) | 150 |
| $\text{C15}-\text{H15B}\cdots\text{O2}^{\text{vi}}$ | 0.97 | 2.48 | 3.425 (10) | 163 |
| $\text{C15}-\text{H15B}\cdots\text{O4A}^{\text{vi}}$ | 0.97 | 2.55 | 3.194 (19) | 124 |

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

Data collection: *XSCANS* (Bruker, 1997); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

RJB wishes to acknowledge the Laboratory for the Structure of Matter at the Naval Research Laboratory for access to their diffractometers.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5043).

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supplementary materials

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2-[2-(2-Pyridyl)ethyl]isoindolinium perchlorate

R. J. Butcher, Y. T. Tesema, T. B. Yisgedu and Y. Gultneh

Comment

The structure of the title compound, (I), is shown below. Dimensions are available in the archived CIF.

The crystal structure shows that the N atom of 2,3-dihydro-1*H*-isoindoline is protonated and involved in hydrogen bonding to the nearest pyridyl N atom (Table 1). The perchlorate ion O atoms are also involved in the hydrogen bonding with the isoindoline H atom. The amine H atom is also involved in hydrogen bonding to the perchlorate O atoms and there are extensive but weak interionic C—H···O interactions between the cation and anion (Table 1).

Experimental

The neutral ligand 2-(2-pyridylethyl)-2,3-dihydro-1*H*-isoindole, was synthesized by reacting 2-(2-aminoethyl)pyridine and α,α' -dibromo-*o*-xylene in THF in the presence of triethylamine as a base and purified by column chromatography using modified literature methods of Bonnett *et al.* (1983) & Meyers *et al.* (1995). The perchlorate salt of the protonated ligand (1) was formed by addition of one equivalent of an aqueous solution of HClO₄ to a solution of the neutral ligand in methanol and allowing the resulting solution to stand yielded pale pink needles of (I).

Refinement

The perchlorate anion is disordered over four conformations with occupancy factors of 0.438 (4), 0.270 (9), 0.155 (8), and 0.138 (5). These were constrained to adopt a tetrahedral geometry. This disorder and its modelling resulted in 245 restraints. The H atoms were idealized with an N—H distance of 0.91 Å and C—H distances were idealized at 0.93 (aromatic C—H), 0.96 (CH₃), and 0.97 (CH₂) Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ ($1.5U_{\text{eq}}(\text{C})$ for the CH₃ protons). Since a unique data set was collected there were no Friedel pairs and no attempt was made to establish the absolute structure.

Figures

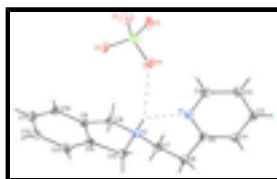


Fig. 1. The title compound showing the N—H···N bond (dashed bonds). Ellipsoids are drawn at the 20% probability level.

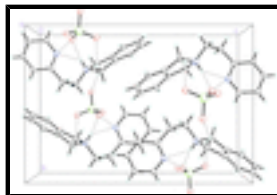


Fig. 2. The packing arrangement viewed down the *a* axis showing the N—H···O and intermolecular C—H···O interactions (dashed bonds).

2-[2-(2-Pyridyl)ethyl]isoindolinium perchlorate

Crystal data

| | |
|-----------------------------------|---|
| $C_{15}H_{17}N_2^+ \cdot ClO_4^-$ | $F_{000} = 680$ |
| $M_r = 324.76$ | $D_x = 1.403 \text{ Mg m}^{-3}$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$ |
| Hall symbol: P 2ac 2ab | Cell parameters from 39 reflections |
| $a = 7.7941 (9) \text{ \AA}$ | $\theta = 5.1\text{--}12.5^\circ$ |
| $b = 11.7032 (19) \text{ \AA}$ | $\mu = 0.27 \text{ mm}^{-1}$ |
| $c = 16.850 (3) \text{ \AA}$ | $T = 293 \text{ K}$ |
| $V = 1537.0 (4) \text{ \AA}^3$ | Needle, pale pink |
| $Z = 4$ | $0.5 \times 0.2 \times 0.08 \text{ mm}$ |

Data collection

| | |
|---|------------------------------------|
| Bruker P4 diffractometer | $R_{\text{int}} = 0.025$ |
| Radiation source: fine-focus sealed tube | $\theta_{\text{max}} = 27.5^\circ$ |
| Monochromator: graphite | $\theta_{\text{min}} = 2.4^\circ$ |
| $T = 293 \text{ K}$ | $h = 0 \rightarrow 10$ |
| ω scans | $k = 0 \rightarrow 15$ |
| Absorption correction: ψ scan (North <i>et al.</i> , 1968) | $l = 0 \rightarrow 21$ |
| $T_{\text{min}} = 0.241$, $T_{\text{max}} = 0.265$ | 3 standard reflections |
| 2027 measured reflections | every 97 reflections |
| 2001 independent reflections | intensity decay: $<2\%$ |
| 1660 reflections with $I > 2\sigma(I)$ | |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.044$ | H-atom parameters constrained |
| $wR(F^2) = 0.120$ | $w = 1/[\sigma^2(F_o^2) + (0.0626P)^2 + 0.2989P]$ |
| $S = 1.03$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 2001 reflections | $(\Delta/\sigma)_{\text{max}} = 0.005$ |
| 329 parameters | $\Delta\rho_{\text{max}} = 0.21 \text{ e \AA}^{-3}$ |
| 245 restraints | $\Delta\rho_{\text{min}} = -0.15 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|---------------|-------------|--------------|----------------------------------|-----------|
| Cl | -0.45920 (11) | 0.96522 (8) | 0.73473 (5) | 0.0643 (3) | |
| O1 | -0.4598 (13) | 1.0879 (4) | 0.7432 (7) | 0.093 (3) | 0.438 (4) |
| O2 | -0.5971 (10) | 0.9347 (9) | 0.6824 (5) | 0.101 (3) | 0.438 (4) |
| O3 | -0.4861 (13) | 0.9137 (8) | 0.8101 (3) | 0.074 (2) | 0.438 (4) |
| O4 | -0.3007 (9) | 0.9305 (9) | 0.7017 (6) | 0.105 (3) | 0.438 (4) |
| O1A | -0.497 (3) | 0.8798 (16) | 0.6772 (10) | 0.099 (5) | 0.155 (8) |
| O2A | -0.2811 (10) | 0.9589 (19) | 0.7554 (14) | 0.100 (6) | 0.155 (8) |
| O3A | -0.494 (3) | 1.0759 (10) | 0.7025 (15) | 0.103 (5) | 0.155 (8) |
| O4A | -0.561 (3) | 0.948 (2) | 0.8038 (8) | 0.104 (5) | 0.155 (8) |
| O1B | -0.466 (3) | 0.9618 (18) | 0.8191 (3) | 0.100 (6) | 0.138 (5) |
| O2B | -0.404 (3) | 0.8561 (9) | 0.7054 (11) | 0.099 (5) | 0.138 (5) |
| O3B | -0.341 (2) | 1.0506 (14) | 0.7094 (12) | 0.100 (4) | 0.138 (5) |
| O4B | -0.6256 (14) | 0.9896 (18) | 0.7036 (13) | 0.104 (5) | 0.138 (5) |
| O1C | -0.2804 (8) | 0.9697 (14) | 0.7176 (9) | 0.088 (4) | 0.270 (9) |
| O2C | -0.5525 (18) | 0.9411 (15) | 0.6637 (6) | 0.111 (5) | 0.270 (9) |
| O3C | -0.492 (2) | 0.8776 (12) | 0.7918 (8) | 0.096 (4) | 0.270 (9) |
| O4C | -0.5146 (19) | 1.0728 (8) | 0.7659 (10) | 0.111 (4) | 0.270 (9) |
| N1 | -0.0054 (4) | 0.8416 (2) | 0.61178 (17) | 0.0633 (7) | |
| N2 | -0.0414 (3) | 0.7346 (2) | 0.75736 (15) | 0.0546 (6) | |
| H2B | -0.0805 | 0.7936 | 0.7272 | 0.065 (10)* | |
| C1 | -0.0438 (5) | 0.9290 (3) | 0.5639 (2) | 0.0716 (9) | |
| H1A | -0.0860 | 0.9961 | 0.5861 | 0.084 (13)* | |
| C2 | -0.0232 (5) | 0.9233 (4) | 0.4823 (2) | 0.0762 (10) | |
| H2A | -0.0468 | 0.9865 | 0.4507 | 0.114 (16)* | |
| C3 | 0.0328 (5) | 0.8230 (4) | 0.4489 (2) | 0.0798 (11) | |
| H3A | 0.0460 | 0.8167 | 0.3943 | 0.077 (11)* | |
| C4 | 0.0691 (5) | 0.7319 (4) | 0.4976 (2) | 0.0702 (9) | |
| H4A | 0.1059 | 0.6629 | 0.4762 | 0.086 (13)* | |
| C5 | 0.0502 (5) | 0.7442 (3) | 0.57909 (19) | 0.0609 (8) | |
| C6 | 0.0970 (5) | 0.6493 (3) | 0.6367 (2) | 0.0677 (9) | |
| H6A | 0.2089 | 0.6658 | 0.6591 | 0.083 (13)* | |
| H6B | 0.1062 | 0.5782 | 0.6074 | 0.073 (11)* | |
| C7 | -0.0297 (5) | 0.6324 (3) | 0.7044 (2) | 0.0652 (8) | |

supplementary materials

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|------|-------------|------------|--------------|-------------|
| H7A | -0.1422 | 0.6163 | 0.6825 | 0.069 (10)* |
| H7B | 0.0053 | 0.5668 | 0.7356 | 0.080 (11)* |
| C8 | -0.1673 (4) | 0.7187 (3) | 0.8247 (2) | 0.0626 (8) |
| H8A | -0.2813 | 0.7439 | 0.8098 | 0.105 (15)* |
| H8B | -0.1724 | 0.6395 | 0.8414 | 0.086 (12)* |
| C9 | -0.0935 (4) | 0.7931 (3) | 0.88894 (19) | 0.0552 (7) |
| C10 | -0.1673 (5) | 0.8278 (3) | 0.9602 (2) | 0.0690 (9) |
| H10A | -0.2794 | 0.8076 | 0.9729 | 0.095 (14)* |
| C11 | -0.0696 (6) | 0.8932 (3) | 1.0117 (2) | 0.0743 (11) |
| H11A | -0.1162 | 0.9156 | 1.0600 | 0.069 (10)* |
| C12 | 0.0928 (6) | 0.9252 (3) | 0.9930 (2) | 0.0721 (10) |
| H12A | 0.1547 | 0.9704 | 1.0282 | 0.112 (16)* |
| C13 | 0.1681 (5) | 0.8917 (3) | 0.9219 (2) | 0.0654 (8) |
| H13A | 0.2787 | 0.9146 | 0.9088 | 0.124 (19)* |
| C14 | 0.0738 (4) | 0.8232 (3) | 0.87123 (18) | 0.0546 (7) |
| C15 | 0.1273 (4) | 0.7717 (3) | 0.79305 (19) | 0.0612 (8) |
| H15A | 0.2032 | 0.7070 | 0.8009 | 0.064 (10)* |
| H15B | 0.1841 | 0.8277 | 0.7597 | 0.079 (12)* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Cl | 0.0620 (5) | 0.0772 (5) | 0.0537 (4) | -0.0053 (5) | 0.0037 (4) | -0.0008 (4) |
| O1 | 0.083 (6) | 0.077 (4) | 0.121 (7) | -0.012 (4) | -0.002 (6) | -0.003 (4) |
| O2 | 0.094 (6) | 0.146 (8) | 0.062 (4) | -0.043 (5) | -0.005 (4) | -0.008 (5) |
| O3 | 0.082 (5) | 0.093 (6) | 0.047 (3) | 0.015 (5) | 0.017 (3) | -0.004 (3) |
| O4 | 0.094 (5) | 0.110 (7) | 0.112 (6) | 0.028 (5) | 0.040 (5) | 0.003 (6) |
| O1A | 0.099 (10) | 0.117 (10) | 0.082 (8) | -0.002 (9) | -0.010 (8) | -0.028 (8) |
| O2A | 0.084 (9) | 0.092 (10) | 0.126 (12) | -0.007 (9) | 0.028 (10) | 0.011 (11) |
| O3A | 0.085 (9) | 0.111 (9) | 0.112 (11) | 0.031 (8) | -0.016 (9) | 0.023 (9) |
| O4A | 0.096 (10) | 0.133 (11) | 0.085 (9) | -0.003 (10) | 0.044 (8) | -0.022 (9) |
| O1B | 0.100 (10) | 0.121 (12) | 0.079 (9) | 0.001 (11) | 0.015 (9) | -0.017 (9) |
| O2B | 0.099 (10) | 0.102 (9) | 0.096 (9) | 0.012 (9) | 0.006 (9) | -0.013 (8) |
| O3B | 0.087 (8) | 0.095 (8) | 0.120 (9) | -0.014 (8) | 0.021 (8) | 0.002 (8) |
| O4B | 0.081 (9) | 0.128 (10) | 0.101 (10) | 0.022 (9) | -0.013 (9) | 0.000 (9) |
| O1C | 0.078 (6) | 0.079 (7) | 0.107 (9) | -0.010 (5) | 0.056 (6) | -0.025 (7) |
| O2C | 0.123 (10) | 0.150 (9) | 0.059 (6) | -0.021 (9) | -0.013 (7) | -0.010 (7) |
| O3C | 0.082 (7) | 0.107 (9) | 0.099 (8) | 0.007 (7) | 0.015 (7) | 0.016 (7) |
| O4C | 0.096 (8) | 0.117 (7) | 0.120 (8) | 0.039 (6) | 0.008 (7) | -0.025 (7) |
| N1 | 0.0600 (17) | 0.0669 (17) | 0.0630 (15) | 0.0055 (13) | -0.0045 (14) | -0.0003 (13) |
| N2 | 0.0552 (14) | 0.0559 (13) | 0.0526 (13) | 0.0009 (12) | -0.0042 (13) | 0.0014 (11) |
| C1 | 0.066 (2) | 0.069 (2) | 0.080 (2) | -0.0005 (19) | -0.004 (2) | 0.0048 (18) |
| C2 | 0.060 (2) | 0.091 (3) | 0.078 (2) | -0.004 (2) | 0.0013 (19) | 0.026 (2) |
| C3 | 0.0581 (19) | 0.122 (3) | 0.0588 (19) | 0.006 (2) | 0.0058 (18) | 0.010 (2) |
| C4 | 0.061 (2) | 0.090 (2) | 0.0599 (18) | 0.0087 (19) | -0.0008 (17) | -0.0076 (18) |
| C5 | 0.0543 (17) | 0.0722 (19) | 0.0561 (16) | 0.0054 (17) | -0.0032 (16) | -0.0035 (15) |
| C6 | 0.075 (2) | 0.070 (2) | 0.0584 (18) | 0.0152 (18) | -0.0067 (18) | -0.0095 (16) |
| C7 | 0.076 (2) | 0.0574 (17) | 0.0620 (17) | 0.0011 (17) | -0.0066 (19) | -0.0045 (14) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| C8 | 0.0541 (18) | 0.0631 (19) | 0.071 (2) | -0.0068 (16) | 0.0066 (16) | 0.0027 (16) |
| C9 | 0.0571 (18) | 0.0510 (15) | 0.0576 (17) | 0.0052 (14) | 0.0056 (14) | 0.0081 (14) |
| C10 | 0.070 (2) | 0.065 (2) | 0.072 (2) | 0.0034 (18) | 0.0226 (19) | 0.0092 (17) |
| C11 | 0.093 (3) | 0.075 (2) | 0.0546 (18) | 0.013 (2) | 0.015 (2) | -0.0035 (17) |
| C12 | 0.081 (3) | 0.078 (2) | 0.0577 (18) | 0.0099 (19) | -0.0069 (19) | -0.0030 (18) |
| C13 | 0.0591 (19) | 0.076 (2) | 0.0605 (18) | 0.0006 (18) | -0.0069 (16) | -0.0013 (17) |
| C14 | 0.0525 (17) | 0.0614 (17) | 0.0499 (15) | 0.0047 (14) | 0.0009 (13) | 0.0068 (13) |
| C15 | 0.0502 (16) | 0.078 (2) | 0.0550 (16) | -0.0031 (17) | 0.0003 (14) | -0.0035 (16) |

Geometric parameters (Å, °)

| | | | |
|------------|-----------|------------|-----------|
| Cl—O4 | 1.415 (4) | C4—C5 | 1.389 (5) |
| Cl—O3 | 1.421 (4) | C4—H4A | 0.9300 |
| Cl—O4A | 1.423 (5) | C5—C6 | 1.520 (5) |
| Cl—O1B | 1.424 (5) | C6—C7 | 1.521 (5) |
| Cl—O1A | 1.424 (5) | C6—H6A | 0.9700 |
| Cl—O1C | 1.424 (5) | C6—H6B | 0.9700 |
| Cl—O3B | 1.424 (5) | C7—H7A | 0.9700 |
| Cl—O4B | 1.427 (5) | C7—H7B | 0.9700 |
| Cl—O3C | 1.428 (5) | C8—C9 | 1.503 (5) |
| Cl—O2C | 1.429 (5) | C8—H8A | 0.9700 |
| Cl—O3A | 1.429 (5) | C8—H8B | 0.9700 |
| Cl—O4C | 1.431 (5) | C9—C14 | 1.383 (5) |
| N1—C1 | 1.337 (4) | C9—C10 | 1.392 (4) |
| N1—C5 | 1.338 (4) | C10—C11 | 1.385 (5) |
| N2—C7 | 1.495 (4) | C10—H10A | 0.9300 |
| N2—C15 | 1.510 (4) | C11—C12 | 1.357 (6) |
| N2—C8 | 1.512 (4) | C11—H11A | 0.9300 |
| N2—H2B | 0.9100 | C12—C13 | 1.390 (5) |
| C1—C2 | 1.385 (6) | C12—H12A | 0.9300 |
| C1—H1A | 0.9300 | C13—C14 | 1.382 (5) |
| C2—C3 | 1.374 (6) | C13—H13A | 0.9300 |
| C2—H2A | 0.9300 | C14—C15 | 1.508 (4) |
| C3—C4 | 1.373 (6) | C15—H15A | 0.9700 |
| C3—H3A | 0.9300 | C15—H15B | 0.9700 |
| O4—Cl—O3 | 111.0 (4) | C7—C6—H6B | 108.6 |
| O4A—Cl—O1A | 110.1 (5) | H6A—C6—H6B | 107.6 |
| O1B—Cl—O3B | 110.0 (5) | N2—C7—C6 | 112.5 (3) |
| O1B—Cl—O4B | 109.8 (5) | N2—C7—H7A | 109.1 |
| O3B—Cl—O4B | 109.7 (5) | C6—C7—H7A | 109.1 |
| O1C—Cl—O3C | 109.7 (4) | N2—C7—H7B | 109.1 |
| O1C—Cl—O2C | 109.6 (4) | C6—C7—H7B | 109.1 |
| O3C—Cl—O2C | 109.3 (4) | H7A—C7—H7B | 107.8 |
| O4A—Cl—O3A | 109.5 (4) | C9—C8—N2 | 102.8 (2) |
| O1A—Cl—O3A | 109.8 (4) | C9—C8—H8A | 111.2 |
| O1C—Cl—O4C | 109.7 (4) | N2—C8—H8A | 111.2 |
| O3C—Cl—O4C | 109.4 (4) | C9—C8—H8B | 111.2 |
| O2C—Cl—O4C | 109.1 (4) | N2—C8—H8B | 111.2 |
| C1—N1—C5 | 118.4 (3) | H8A—C8—H8B | 109.1 |

supplementary materials

| | | | |
|--------------|------------|-----------------|------------|
| C7—N2—C15 | 114.5 (3) | C14—C9—C10 | 120.1 (3) |
| C7—N2—C8 | 112.9 (2) | C14—C9—C8 | 110.7 (3) |
| C15—N2—C8 | 107.5 (2) | C10—C9—C8 | 129.2 (3) |
| C7—N2—H2B | 107.2 | C11—C10—C9 | 118.3 (4) |
| C15—N2—H2B | 107.2 | C11—C10—H10A | 120.9 |
| C8—N2—H2B | 107.2 | C9—C10—H10A | 120.9 |
| N1—C1—C2 | 122.4 (4) | C12—C11—C10 | 121.3 (4) |
| N1—C1—H1A | 118.8 | C12—C11—H11A | 119.3 |
| C2—C1—H1A | 118.8 | C10—C11—H11A | 119.3 |
| C3—C2—C1 | 119.0 (4) | C11—C12—C13 | 121.0 (4) |
| C3—C2—H2A | 120.5 | C11—C12—H12A | 119.5 |
| C1—C2—H2A | 120.5 | C13—C12—H12A | 119.5 |
| C4—C3—C2 | 119.0 (4) | C14—C13—C12 | 118.1 (4) |
| C4—C3—H3A | 120.5 | C14—C13—H13A | 120.9 |
| C2—C3—H3A | 120.5 | C12—C13—H13A | 120.9 |
| C3—C4—C5 | 119.2 (4) | C13—C14—C9 | 121.0 (3) |
| C3—C4—H4A | 120.4 | C13—C14—C15 | 128.6 (3) |
| C5—C4—H4A | 120.4 | C9—C14—C15 | 110.3 (3) |
| N1—C5—C4 | 122.0 (3) | C14—C15—N2 | 102.8 (3) |
| N1—C5—C6 | 115.9 (3) | C14—C15—H15A | 111.2 |
| C4—C5—C6 | 122.1 (3) | N2—C15—H15A | 111.2 |
| C5—C6—C7 | 114.7 (3) | C14—C15—H15B | 111.2 |
| C5—C6—H6A | 108.6 | N2—C15—H15B | 111.2 |
| C7—C6—H6A | 108.6 | H15A—C15—H15B | 109.1 |
| C5—C6—H6B | 108.6 | | |
| C5—N1—C1—C2 | -2.1 (6) | N2—C8—C9—C10 | 168.3 (3) |
| N1—C1—C2—C3 | 2.5 (6) | C14—C9—C10—C11 | 0.5 (5) |
| C1—C2—C3—C4 | -1.0 (6) | C8—C9—C10—C11 | 177.3 (3) |
| C2—C3—C4—C5 | -0.8 (6) | C9—C10—C11—C12 | 1.4 (6) |
| C1—N1—C5—C4 | 0.2 (6) | C10—C11—C12—C13 | -1.2 (6) |
| C1—N1—C5—C6 | 178.2 (3) | C11—C12—C13—C14 | -0.9 (5) |
| C3—C4—C5—N1 | 1.2 (6) | C12—C13—C14—C9 | 2.8 (5) |
| C3—C4—C5—C6 | -176.7 (4) | C12—C13—C14—C15 | -177.3 (3) |
| N1—C5—C6—C7 | 43.7 (5) | C10—C9—C14—C13 | -2.6 (5) |
| C4—C5—C6—C7 | -138.3 (4) | C8—C9—C14—C13 | -180.0 (3) |
| C15—N2—C7—C6 | -56.6 (4) | C10—C9—C14—C15 | 177.5 (3) |
| C8—N2—C7—C6 | 179.9 (3) | C8—C9—C14—C15 | 0.1 (4) |
| C5—C6—C7—N2 | -63.2 (4) | C13—C14—C15—N2 | -165.4 (3) |
| C7—N2—C8—C9 | 150.7 (3) | C9—C14—C15—N2 | 14.4 (4) |
| C15—N2—C8—C9 | 23.4 (3) | C7—N2—C15—C14 | -149.7 (3) |
| N2—C8—C9—C14 | -14.6 (3) | C8—N2—C15—C14 | -23.4 (3) |

Hydrogen-bond geometry (\AA , $^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| N2—H2B \cdots N1 | 0.91 | 2.11 | 2.769 (4) | 129 |
| N2—H2B \cdots O4 | 0.91 | 2.39 | 3.197 (9) | 148 |
| N2—H2B \cdots O2A | 0.91 | 2.53 | 3.222 (19) | 133 |
| N2—H2B \cdots O1C | 0.91 | 2.59 | 3.390 (16) | 147 |

| | | | | |
|----------------------------|------|------|------------|-----|
| C2—H2A…O1B ⁱ | 0.93 | 2.30 | 3.062 (11) | 139 |
| C3—H3A…O1A ⁱⁱ | 0.93 | 2.62 | 3.193 (10) | 121 |
| C7—H7B…O2A ⁱⁱⁱ | 0.97 | 2.50 | 3.232 (16) | 132 |
| C8—H8A…O3 | 0.97 | 2.55 | 3.383 (10) | 144 |
| C8—H8A…O2B | 0.97 | 2.39 | 3.168 (19) | 136 |
| C8—H8A…O3C | 0.97 | 2.29 | 3.189 (17) | 154 |
| C8—H8B…O4B ^{iv} | 0.97 | 2.48 | 3.167 (17) | 128 |
| C11—H11A…O3A ^v | 0.93 | 2.55 | 3.27 (2) | 135 |
| C11—H11A…O3B ^v | 0.93 | 2.57 | 3.47 (2) | 162 |
| C13—H13A…O3 ^{vi} | 0.93 | 2.48 | 3.299 (9) | 148 |
| C13—H13A…O4A ^{vi} | 0.93 | 2.20 | 2.976 (8) | 140 |
| C13—H13A…O1B ^{vi} | 0.93 | 2.56 | 3.44 (2) | 158 |
| C13—H13A…O3C ^{vi} | 0.93 | 2.70 | 3.444 (17) | 138 |
| C15—H15A…O1 ⁱⁱⁱ | 0.97 | 2.55 | 3.423 (9) | 150 |
| C15—H15B…O2 ^{vi} | 0.97 | 2.48 | 3.425 (10) | 163 |
| C15—H15B…O4A ^{vi} | 0.97 | 2.55 | 3.194 (19) | 124 |

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

Fig. 2

