

2-Methyl-3-(3-methylisoxazol-5-yl)-4-oxo-4H-pyrido[1,2-a]pyrimidin-1-ium chloride

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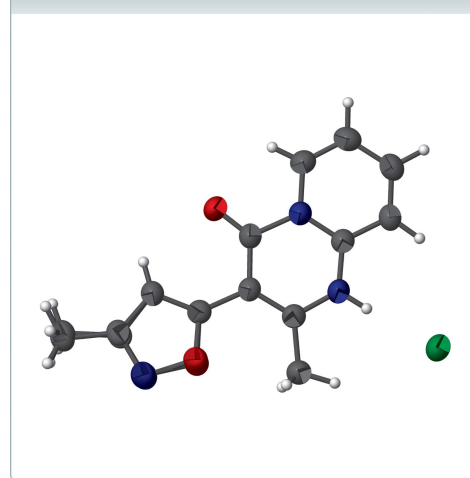
Keywords: crystal structure; hydrogen bond; pyridopyrimidinium chloride.

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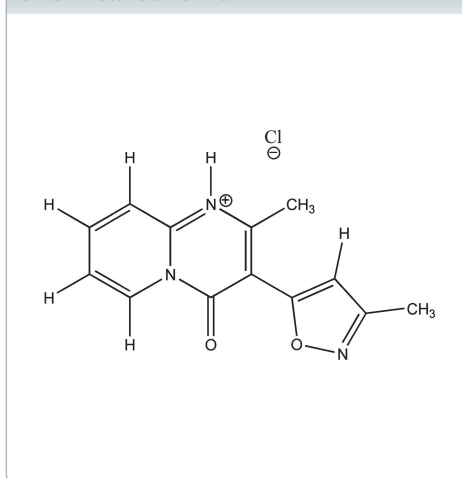
Structural data: full structural data are available from iucrdata.iucr.org

In the title molecular salt, $C_{13}H_{12}N_3O_2^+ \cdot Cl^-$, the oxazolyl ring is disordered over two orientations in a 0.536 (15):0.464 (15) ratio, both of which approximate to envelopes with the N atom as the flap in each case. The cation and anion are linked by a charge-assisted N—H \cdots Cl hydrogen bond. In the extended structure, C—H \cdots N, C—H \cdots O and C—H \cdots Cl interactions link the components into a three-dimensional network.

3D view



Chemical scheme



Structure description

Pyridopyrimidine compounds have found use as antimalarial agents (Mane *et al.*, 2014), anti-allergic agents (Awouters *et al.*, 1986) and urease inhibitors (Rauf *et al.*, 2012). The isoxazole nucleus is known to exhibit anticancer (Han *et al.*, 2002), anti-HIV (Deng *et al.*, 2006) and fungicide (Raffa *et al.*, 1999) activities. The present work reporting the synthesis and structure of the title molecular salt (Fig. 1) is a continuation of our work on pyridopyrimidine derivatives (Djerrari *et al.*, 2002).

The bicyclic core of the cation is slightly folded along the C5—N1 axis by 1.07 (14)°. In the oxazolyl substituent, the oxygen and nitrogen atoms and the C—CH₃ grouping are disordered over two sets of sites, which represent the two possible envelope conformations of the five-membered ring (flap atom = N). The cation and anion are strongly associated through the N2—H2A \cdots Cl1 hydrogen bond (Table 1 and Fig. 1). In the extended structure, the ion pairs are arranged in rows running along the *a*-axis direction with weak, bifurcated C13A—H13A \cdots O2Aⁱ and C13A—H13A \cdots N3Aⁱ [symmetry code: (i) $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$] hydrogen bonds (Table 1 and Fig. 2) as well as weak C—H \cdots Cl interactions.

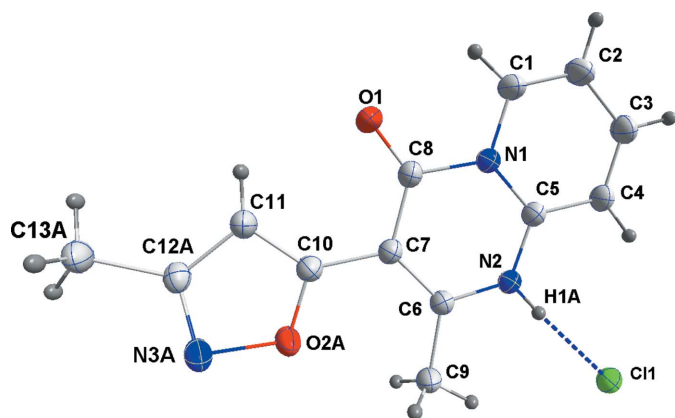


Figure 1
The title molecule with the atom-labelling scheme and 25% probability ellipsoids. The N—H···Cl hydrogen bond is shown as a dotted line. Only the major component of the disorder in the oxazolyl substituent is shown.

Synthesis and crystallization

A mixture of 1-(2-methyl-4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-3-yl)butane-1,3-dione (0.7 g, 2.86 mmol) and of hydroxylamine hydrochloride (0.4 g, 5.75 mmol) in methanol (30 ml) was heated at reflux for 4 h. The completion of the reaction was confirmed by TLC. The solid obtained upon cooling the mixture was recrystallized from ethanol solution to afford orange blocks (yield: 76%. m.p.: 465–467 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. In the pendant oxazolyl substituent, all atoms except C10 and C11 are disordered over two sets of sites in a 0.536 (15):0.464 (15) ratio. The two components of the disorder were refined with restraints that their geometries be comparable.

Acknowledgements

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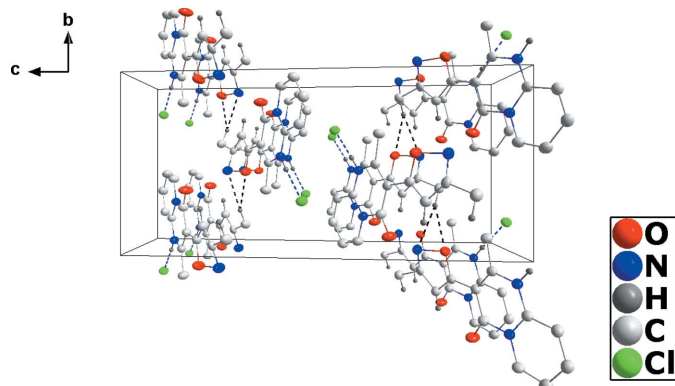


Figure 2
Packing viewed along the *a* axis. N—H···Cl and C—H···O hydrogen bonds are shown, respectively, as blue and black dotted lines.

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

| <i>D</i> —H··· <i>A</i> | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|--------------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H1A···Cl1 | 0.88 (4) | 2.15 (4) | 3.024 (3) | 177 (3) |
| C2—H2···Cl1 ⁱ | 0.98 (4) | 2.78 (4) | 3.745 (4) | 173 (2) |
| C3—H3···Cl1 ⁱⁱ | 0.98 (4) | 2.71 (4) | 3.445 (3) | 132 (3) |
| C11—H11···N3A ⁱⁱⁱ | 0.90 (4) | 2.67 (4) | 3.390 (16) | 138 (3) |
| C13A—H13A···O2A ⁱⁱⁱ | 0.96 | 2.34 | 3.21 (4) | 151 |
| C13A—H13A···N3A ⁱⁱⁱ | 0.96 | 2.50 | 3.37 (4) | 150 |

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

Table 2
Experimental details.

| | |
|--|---|
| Crystal data | |
| Chemical formula | C ₁₃ H ₁₂ N ₃ O ₂ ⁺ ·Cl [−] |
| <i>M_r</i> | 277.70 |
| Crystal system, space group | Monoclinic, <i>P</i> ₂ ₁ / <i>n</i> |
| Temperature (K) | 298 |
| <i>a</i> , <i>b</i> , <i>c</i> (Å) | 6.2796 (12), 9.6912 (18), 21.152 (4) |
| β (°) | 95.294 (3) |
| <i>V</i> (Å ³) | 1281.8 (4) |
| <i>Z</i> | 4 |
| Radiation type | Mo Kα |
| μ (mm ^{−1}) | 0.30 |
| Crystal size (mm) | 0.32 × 0.20 × 0.19 |
| Data collection | |
| Diffractometer | Bruker SMART APEX CCD |
| Absorption correction | Multi-scan (<i>TWINABS</i> ; Sheldrick, 2009) |
| <i>T_{min}</i> , <i>T_{max}</i> | 0.75, 0.94 |
| No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections | 28190, 6596, 4582 |
| <i>R_{int}</i> | 0.068 |
| (sin θ/λ) _{max} (Å ^{−1}) | 0.678 |
| Refinement | |
| <i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i> | 0.064, 0.192, 1.04 |
| No. of reflections | 6596 |
| No. of parameters | 222 |
| No. of restraints | 53 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| Δρ _{max} , Δρ _{min} (e Å ^{−3}) | 0.36, −0.33 |

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *CELL_NOW* (Sheldrick, 2008a) and *SHELXT* (Sheldrick, 2015a), *SHELXL2014* (Sheldrick, 2015b), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008b).

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full crystallographic data

IUCrData (2016). **1**, x161978 [<https://doi.org/10.1107/S2414314616019787>]

2-Methyl-3-(3-methylisoxazol-5-yl)-4-oxo-4*H*-pyrido[1,2-*a*]pyrimidin-1-ium chloride

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Crystal data

$C_{13}H_{12}N_3O_2^+ \cdot Cl^-$

$M_r = 277.70$

Monoclinic, $P2_1/n$

$a = 6.2796$ (12) Å

$b = 9.6912$ (18) Å

$c = 21.152$ (4) Å

$\beta = 95.294$ (3)°

$V = 1281.8$ (4) Å³

$Z = 4$

$F(000) = 576$

$D_x = 1.439$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5128 reflections

$\theta = 2.3$ – 25.9 °

$\mu = 0.30$ mm⁻¹

$T = 298$ K

Block, orange

$0.32 \times 0.20 \times 0.19$ mm

Data collection

Bruker SMART APEX CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.3333 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan
(*TWINABS*; Sheldrick, 2009)

$T_{\min} = 0.75$, $T_{\max} = 0.94$

28190 measured reflections

6596 independent reflections

4582 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.068$

$\theta_{\max} = 28.8$ °, $\theta_{\min} = 1.9$ °

$h = -8$ → 8

$k = -13$ → 13

$l = -28$ → 28

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.064$

$wR(F^2) = 0.192$

$S = 1.04$

6596 reflections

222 parameters

53 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0802P)^2 + 0.5444P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 0.36$ e Å⁻³

$\Delta\rho_{\min} = -0.33$ e Å⁻³

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω , collected at $\varphi = 0.00, 90.00$ and 180.00° and 2 sets of 800 frames, each of width 0.45° in φ , collected at $\omega = -30.00$ and 210.00° . The scan time was 20 sec/frame. Analysis of 1600 reflections having $I/\sigma(I) > 12$ and chosen from the full data set with *CELL_NOW* (Sheldrick, 2008a) showed the crystal to belong to the monoclinic system and to be twinned by a 180° rotation about the c^* axis. The raw data were processed using the multi-component version of *SAINTE* under control of the two-component orientation file generated by *CELL_NOW*.

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. 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 $F^2 > 2\sigma(F^2)$ 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. The methyl oxazole moiety is disordered over two partially resolved sites with the disorder mainly involving alternate conformations of the heteroatom prtion of the ring.

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) |
|------|------------|-------------|--------------|----------------------------------|------------|
| O1 | 0.6819 (4) | 0.8510 (2) | 0.65445 (14) | 0.0747 (8) | |
| N1 | 0.9648 (4) | 0.7726 (2) | 0.60427 (12) | 0.0472 (6) | |
| H1A | 1.092 (5) | 0.470 (4) | 0.5794 (15) | 0.048 (8)* | |
| N2 | 1.0175 (4) | 0.5368 (3) | 0.59451 (11) | 0.0460 (6) | |
| O2A | 0.497 (2) | 0.4524 (5) | 0.7017 (5) | 0.067 (2) | 0.536 (15) |
| N3A | 0.374 (2) | 0.4582 (16) | 0.7600 (5) | 0.068 (3) | 0.536 (15) |
| O2B | 0.549 (2) | 0.4604 (7) | 0.7216 (6) | 0.067 (2) | 0.464 (15) |
| N3B | 0.320 (2) | 0.4573 (19) | 0.7389 (7) | 0.068 (3) | 0.464 (15) |
| C1 | 1.0220 (6) | 0.9076 (3) | 0.59290 (16) | 0.0571 (8) | |
| H1 | 0.933 (6) | 0.971 (4) | 0.6091 (17) | 0.063 (10)* | |
| C2 | 1.1882 (6) | 0.9357 (4) | 0.55943 (17) | 0.0614 (8) | |
| H2 | 1.227 (5) | 1.032 (4) | 0.5534 (15) | 0.058 (9)* | |
| C3 | 1.3048 (5) | 0.8268 (4) | 0.53573 (16) | 0.0578 (8) | |
| H3 | 1.424 (6) | 0.848 (4) | 0.5106 (16) | 0.060 (9)* | |
| C4 | 1.2516 (5) | 0.6939 (3) | 0.54654 (14) | 0.0505 (7) | |
| H4 | 1.327 (5) | 0.616 (3) | 0.5311 (14) | 0.046 (8)* | |
| C5 | 1.0770 (5) | 0.6671 (3) | 0.58186 (13) | 0.0448 (6) | |
| C7 | 0.7321 (5) | 0.6085 (3) | 0.65240 (13) | 0.0450 (6) | |
| C6 | 0.8520 (5) | 0.5045 (3) | 0.62902 (13) | 0.0445 (6) | |
| C9 | 0.8178 (6) | 0.3526 (3) | 0.6367 (2) | 0.0566 (8) | |
| H9A | 0.841 (7) | 0.328 (4) | 0.678 (2) | 0.090 (14)* | |
| H9B | 0.685 (7) | 0.327 (4) | 0.6228 (19) | 0.075 (12)* | |
| H9C | 0.921 (7) | 0.298 (5) | 0.614 (2) | 0.089 (13)* | |
| C8 | 0.7780 (5) | 0.7504 (3) | 0.63969 (15) | 0.0510 (7) | |
| C10 | 0.5567 (5) | 0.5859 (3) | 0.69230 (13) | 0.0476 (7) | |
| C11 | 0.4164 (5) | 0.6703 (3) | 0.71767 (15) | 0.0510 (7) | |
| H11 | 0.400 (6) | 0.762 (4) | 0.7120 (16) | 0.065 (11)* | |
| C12A | 0.279 (3) | 0.5827 (14) | 0.7485 (9) | 0.046 (3) | 0.536 (15) |

| | | | | | |
|------|--------------|-------------|-------------|------------|------------|
| C13A | 0.117 (4) | 0.630 (4) | 0.7921 (13) | 0.059 (2) | 0.536 (15) |
| H13A | 0.0628 | 0.7190 | 0.7787 | 0.089* | 0.536 (15) |
| H13B | 0.0014 | 0.5650 | 0.7908 | 0.089* | 0.536 (15) |
| H13C | 0.1839 | 0.6364 | 0.8347 | 0.089* | 0.536 (15) |
| C12B | 0.306 (4) | 0.5894 (16) | 0.7596 (11) | 0.046 (3) | 0.464 (15) |
| C13B | 0.110 (5) | 0.617 (5) | 0.7933 (15) | 0.059 (2) | 0.464 (15) |
| H13D | 0.1091 | 0.7114 | 0.8067 | 0.089* | 0.464 (15) |
| H13E | -0.0157 | 0.5989 | 0.7649 | 0.089* | 0.464 (15) |
| H13F | 0.1097 | 0.5576 | 0.8297 | 0.089* | 0.464 (15) |
| Cl1 | 1.28278 (13) | 0.31411 (8) | 0.53955 (4) | 0.0574 (3) | |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|------|-------------|-------------|-------------|--------------|-------------|--------------|
| O1 | 0.0733 (16) | 0.0429 (12) | 0.115 (2) | 0.0045 (11) | 0.0483 (15) | -0.0048 (13) |
| N1 | 0.0485 (13) | 0.0420 (12) | 0.0527 (13) | -0.0001 (10) | 0.0139 (11) | -0.0023 (10) |
| N2 | 0.0463 (13) | 0.0418 (12) | 0.0514 (13) | 0.0045 (10) | 0.0130 (11) | -0.0006 (10) |
| O2A | 0.088 (5) | 0.0476 (14) | 0.073 (5) | -0.0073 (16) | 0.045 (4) | -0.008 (2) |
| N3A | 0.090 (6) | 0.0550 (18) | 0.065 (6) | -0.004 (4) | 0.042 (5) | -0.002 (5) |
| O2B | 0.088 (5) | 0.0476 (14) | 0.073 (5) | -0.0073 (16) | 0.045 (4) | -0.008 (2) |
| N3B | 0.090 (6) | 0.0550 (18) | 0.065 (6) | -0.004 (4) | 0.042 (5) | -0.002 (5) |
| C1 | 0.063 (2) | 0.0418 (16) | 0.069 (2) | -0.0008 (14) | 0.0182 (16) | -0.0025 (14) |
| C2 | 0.067 (2) | 0.0487 (18) | 0.071 (2) | -0.0085 (15) | 0.0187 (17) | 0.0023 (15) |
| C3 | 0.0541 (18) | 0.0594 (19) | 0.0626 (19) | -0.0049 (15) | 0.0190 (15) | 0.0000 (15) |
| C4 | 0.0468 (16) | 0.0529 (17) | 0.0538 (16) | 0.0014 (13) | 0.0149 (13) | -0.0018 (13) |
| C5 | 0.0451 (14) | 0.0425 (14) | 0.0475 (15) | 0.0017 (11) | 0.0076 (12) | -0.0004 (11) |
| C7 | 0.0470 (15) | 0.0427 (14) | 0.0465 (14) | 0.0010 (12) | 0.0111 (12) | -0.0025 (11) |
| C6 | 0.0449 (14) | 0.0424 (14) | 0.0466 (14) | 0.0020 (11) | 0.0065 (12) | 0.0016 (11) |
| C9 | 0.058 (2) | 0.0432 (16) | 0.071 (2) | 0.0025 (15) | 0.0213 (18) | 0.0033 (15) |
| C8 | 0.0477 (16) | 0.0471 (16) | 0.0603 (17) | 0.0009 (13) | 0.0168 (14) | -0.0031 (13) |
| C10 | 0.0522 (16) | 0.0436 (15) | 0.0480 (15) | -0.0021 (12) | 0.0106 (12) | -0.0031 (12) |
| C11 | 0.0510 (17) | 0.0476 (17) | 0.0565 (17) | 0.0006 (13) | 0.0172 (13) | -0.0001 (13) |
| C12A | 0.049 (4) | 0.0526 (19) | 0.037 (6) | -0.008 (2) | 0.006 (5) | -0.010 (3) |
| C13A | 0.064 (2) | 0.055 (6) | 0.063 (2) | -0.007 (3) | 0.0261 (17) | -0.004 (2) |
| C12B | 0.049 (4) | 0.0526 (19) | 0.037 (6) | -0.008 (2) | 0.006 (5) | -0.010 (3) |
| C13B | 0.064 (2) | 0.055 (6) | 0.063 (2) | -0.007 (3) | 0.0261 (17) | -0.004 (2) |
| Cl1 | 0.0549 (4) | 0.0508 (4) | 0.0684 (5) | 0.0076 (3) | 0.0152 (4) | -0.0032 (3) |

Geometric parameters (Å, °)

| | | | |
|---------|-----------|--------|-----------|
| O1—C8 | 1.203 (4) | C4—H4 | 0.96 (3) |
| N1—C5 | 1.352 (4) | C7—C6 | 1.377 (4) |
| N1—C1 | 1.383 (4) | C7—C8 | 1.436 (4) |
| N1—C8 | 1.465 (4) | C7—C10 | 1.465 (4) |
| N2—C5 | 1.351 (4) | C6—C9 | 1.498 (4) |
| N2—C6 | 1.360 (4) | C9—H9C | 0.99 (4) |
| N2—H1A | 0.88 (4) | C9—H9B | 0.89 (4) |
| O2A—C10 | 1.368 (5) | C9—H9A | 0.90 (5) |

| | | | |
|--------------|------------|----------------|------------|
| O2A—N3A | 1.513 (6) | C10—C11 | 1.349 (4) |
| N3A—C12A | 1.359 (10) | C11—C12B | 1.412 (5) |
| O2B—C10 | 1.368 (5) | C11—C12A | 1.412 (5) |
| O2B—N3B | 1.515 (7) | C11—H11 | 0.90 (4) |
| N3B—C12B | 1.358 (10) | C12A—C13A | 1.506 (7) |
| C1—C2 | 1.342 (5) | C13A—H13A | 0.9600 |
| C1—H1 | 0.92 (4) | C13A—H13B | 0.9600 |
| C2—C3 | 1.403 (5) | C13A—H13C | 0.9600 |
| C2—H2 | 0.97 (4) | C12B—C13B | 1.506 (7) |
| C3—C4 | 1.355 (5) | C13B—H13D | 0.9600 |
| C3—H3 | 0.98 (4) | C13B—H13E | 0.9600 |
| C4—C5 | 1.407 (4) | C13B—H13F | 0.9600 |
| | | | |
| C5—N1—C1 | 120.1 (3) | C6—C9—H9A | 112 (3) |
| C5—N1—C8 | 122.4 (2) | H9C—C9—H9A | 105 (4) |
| C1—N1—C8 | 117.5 (2) | H9B—C9—H9A | 109 (3) |
| C5—N2—C6 | 124.1 (3) | O1—C8—C7 | 127.9 (3) |
| C5—N2—H1A | 117 (2) | O1—C8—N1 | 117.3 (3) |
| C6—N2—H1A | 119 (2) | C7—C8—N1 | 114.9 (2) |
| C10—O2A—N3A | 104.6 (9) | C11—C10—O2A | 108.5 (6) |
| C12A—N3A—O2A | 97.7 (12) | C11—C10—O2B | 107.8 (6) |
| C10—O2B—N3B | 101.7 (10) | C11—C10—C7 | 133.8 (3) |
| C12B—N3B—O2B | 98.5 (15) | O2A—C10—C7 | 117.2 (5) |
| C2—C1—N1 | 120.8 (3) | O2B—C10—C7 | 117.1 (6) |
| C2—C1—H1 | 126 (2) | C10—C11—C12B | 106.8 (9) |
| N1—C1—H1 | 113 (2) | C10—C11—C12A | 105.6 (7) |
| C1—C2—C3 | 119.5 (3) | C10—C11—H11 | 128 (2) |
| C1—C2—H2 | 119 (2) | C12B—C11—H11 | 125 (2) |
| C3—C2—H2 | 122 (2) | C12A—C11—H11 | 126 (2) |
| C4—C3—C2 | 120.7 (3) | N3A—C12A—C11 | 109.9 (13) |
| C4—C3—H3 | 120 (2) | N3A—C12A—C13A | 118.1 (16) |
| C2—C3—H3 | 119 (2) | C11—C12A—C13A | 125.1 (19) |
| C3—C4—C5 | 118.8 (3) | C12A—C13A—H13A | 109.5 |
| C3—C4—H4 | 123.6 (19) | C12A—C13A—H13B | 109.5 |
| C5—C4—H4 | 117.6 (19) | H13A—C13A—H13B | 109.5 |
| N2—C5—N1 | 118.3 (3) | C12A—C13A—H13C | 109.5 |
| N2—C5—C4 | 121.4 (3) | H13A—C13A—H13C | 109.5 |
| N1—C5—C4 | 120.2 (3) | H13B—C13A—H13C | 109.5 |
| C6—C7—C8 | 120.5 (3) | N3B—C12B—C11 | 105.8 (13) |
| C6—C7—C10 | 124.3 (3) | N3B—C12B—C13B | 113 (2) |
| C8—C7—C10 | 115.2 (2) | C11—C12B—C13B | 132 (2) |
| N2—C6—C7 | 119.6 (3) | C12B—C13B—H13D | 109.5 |
| N2—C6—C9 | 114.1 (3) | C12B—C13B—H13E | 109.5 |
| C7—C6—C9 | 126.3 (3) | H13D—C13B—H13E | 109.5 |
| C6—C9—H9C | 111 (3) | C12B—C13B—H13F | 109.5 |
| C6—C9—H9B | 112 (3) | H13D—C13B—H13F | 109.5 |
| H9C—C9—H9B | 108 (4) | H13E—C13B—H13F | 109.5 |

| | | | |
|------------------|------------|-------------------|-------------|
| C10—O2A—N3A—C12A | 35.8 (19) | C1—N1—C8—O1 | 2.8 (5) |
| C10—O2B—N3B—C12B | -42.4 (18) | C5—N1—C8—C7 | 4.0 (4) |
| C5—N1—C1—C2 | 0.3 (5) | C1—N1—C8—C7 | -177.4 (3) |
| C8—N1—C1—C2 | -178.4 (3) | N3A—O2A—C10—C11 | -25.9 (13) |
| N1—C1—C2—C3 | 0.0 (6) | N3A—O2A—C10—C7 | 161.2 (9) |
| C1—C2—C3—C4 | -0.2 (6) | N3B—O2B—C10—C11 | 29.1 (12) |
| C2—C3—C4—C5 | 0.1 (5) | N3B—O2B—C10—C7 | -162.2 (8) |
| C6—N2—C5—N1 | -0.6 (4) | C6—C7—C10—C11 | -177.0 (3) |
| C6—N2—C5—C4 | 179.2 (3) | C8—C7—C10—C11 | 4.6 (5) |
| C1—N1—C5—N2 | 179.3 (3) | C6—C7—C10—O2A | -6.3 (8) |
| C8—N1—C5—N2 | -2.1 (4) | C8—C7—C10—O2A | 175.2 (7) |
| C1—N1—C5—C4 | -0.4 (4) | C6—C7—C10—O2B | 18.1 (8) |
| C8—N1—C5—C4 | 178.2 (3) | C8—C7—C10—O2B | -160.4 (7) |
| C3—C4—C5—N2 | -179.5 (3) | O2B—C10—C11—C12B | -5.3 (14) |
| C3—C4—C5—N1 | 0.2 (5) | C7—C10—C11—C12B | -171.3 (13) |
| C5—N2—C6—C7 | 1.1 (4) | O2A—C10—C11—C12A | 5.4 (12) |
| C5—N2—C6—C9 | -179.8 (3) | C7—C10—C11—C12A | 176.6 (11) |
| C8—C7—C6—N2 | 1.1 (4) | O2A—N3A—C12A—C11 | -33 (2) |
| C10—C7—C6—N2 | -177.3 (3) | O2A—N3A—C12A—C13A | 174 (2) |
| C8—C7—C6—C9 | -177.9 (3) | C10—C11—C12A—N3A | 20.2 (19) |
| C10—C7—C6—C9 | 3.8 (5) | C10—C11—C12A—C13A | 170 (2) |
| C6—C7—C8—O1 | 176.4 (4) | O2B—N3B—C12B—C11 | 40 (2) |
| C10—C7—C8—O1 | -5.1 (5) | O2B—N3B—C12B—C13B | -169 (2) |
| C6—C7—C8—N1 | -3.4 (4) | C10—C11—C12B—N3B | -24 (2) |
| C10—C7—C8—N1 | 175.1 (3) | C10—C11—C12B—C13B | -168 (3) |
| C5—N1—C8—O1 | -175.9 (3) | | |

Hydrogen-bond geometry (Å, °)

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|--------------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H1A...C11 | 0.88 (4) | 2.15 (4) | 3.024 (3) | 177 (3) |
| C2—H2...C11 ⁱ | 0.98 (4) | 2.78 (4) | 3.745 (4) | 173 (2) |
| C3—H3...C11 ⁱⁱ | 0.98 (4) | 2.71 (4) | 3.445 (3) | 132 (3) |
| C11—H11...N3A ⁱⁱⁱ | 0.90 (4) | 2.67 (4) | 3.390 (16) | 138 (3) |
| C13A—H13A...O2A ⁱⁱⁱ | 0.96 | 2.34 | 3.21 (4) | 151 |
| C13A—H13A...N3A ⁱⁱⁱ | 0.96 | 2.50 | 3.37 (4) | 150 |

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