Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 4-Aminopyridinium *cis*-2-carboxycyclohexane-1-carboxylate

**Graham Smith* and Urs D. Wermuth**

Faculty of Science and Technology, Queensland University of Technology, GPO Box 2434, Brisbane, Queensland 4001, Australia
Correspondence e-mail: g.smith@qut.edu.au

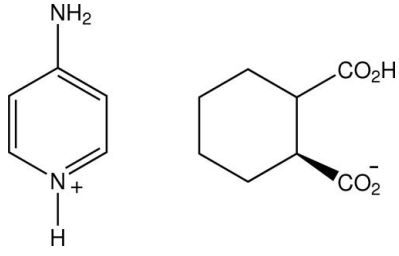
Received 19 September 2011; accepted 26 September 2011

Key indicators: single-crystal X-ray study; $T$ = 200 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.028; $wR$ factor = 0.060; data-to-parameter ratio = 9.1.

In the structure of the title molecular salt, $C_5H_7N_2^+\cdot C_8H_{11}O_4^-$, the *cis* monoanions associate through short O—H···O hydrogen bonds in the carboxylic acid groups [graph set $C(7)$], forming zigzag chains which extend along the $c$ axis. These are interlinked through pyridinium and amine N—H···O hydrogen bonds, giving a three-dimensional network structure.

## Related literature

For the structure of racemic *cis*-cyclohexane-1,2-dicarboxylic acid, see: Benedetti *et al.* (1970). For the structure of the racemic ammonium and 2-aminopyridinium salts of *cis*-2-carboxycyclohexane-1-carboxylate, see: Smith & Wermuth (2011*a*,*b*). For graph-set analysis, see Etter *et al.* (1990).




## Experimental

### Crystal data

$C_5H_7N_2^+\cdot C_8H_{11}O_4^-$
$M_r$ = 266.29
Orthorhombic, $Pna2_1$
$a$ = 12.1359 (3) Å
$b$ = 9.8351 (3) Å
$c$ = 11.1850 (3) Å
$V$ = 1335.02 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 200 K
0.30 × 0.25 × 0.20 mm

### Data collection

Oxford Diffraction Gemini-S CCD-detector diffractometer
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min}$ = 0.948, $T_{max}$ = 0.990
9670 measured reflections
1709 independent reflections
1448 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.060
$S$ = 0.99
1709 reflections
188 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.16 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1$A$—H1$A$···O12$^{i}$ | 0.88 (2) | 1.91 (2) | 2.795 (2) | 180 (3) |
| N41$A$—H41$A$···O12$^{ii}$ | 0.86 (2) | 2.14 (2) | 2.989 (2) | 168 (2) |
| N41$A$—H42$A$···O22 | 0.91 (2) | 2.13 (2) | 2.974 (2) | 152.6 (18) |
| O21—H21···O11$^{iii}$ | 0.95 (3) | 1.59 (3) | 2.5302 (17) | 170 (3) |

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

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008) within *WinGX* (Farrugia, 1999); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *PLATON*.

The authors acknowledge financial support from the Australian Research Council, the Faculty of Science and Technology and the University Library, Queensland University of Technology.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2453).

## References

Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
Benedetti, E., Pedone, C. & Allegra, G. (1970). *J. Phys. Chem.* **74**, 512–516.
Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* B**46**, 256–262.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Smith, G. & Wermuth, U. D. (2011*a*). *Acta Cryst.* E**67**, o174.
Smith, G. & Wermuth, U. D. (2011*b*). *Acta Cryst.* E**67**, o1900.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

# supporting information

*Acta Cryst.* (2011). E**67**, o2794 [https://doi.org/10.1107/S1600536811039547]

## 4-Aminopyridinium *cis*-2-carboxycyclohexane-1-carboxylate

**Graham Smith and Urs D. Wermuth**

### S1. Comment

The structures of Lewis base salts of *cis*-cyclohexane-1,2-dicarboxylic acid (*cis*-CHDC) are rare in the crystallographic literature and like the parent *cis*-acid (Benedetti *et al.*, 1970), exist only in the unresolved racemic form. We have reported the structures of the 1:1 ammonium salt (Smith & Wermuth, 2011*a*) and the 1:1 2-aminopyridinium salt (Smith & Wermuth, 2011*b*) and in our parallel 1:1 stoichiometric reaction of *cis*-CHDC anhydride with 4-aminopyridine in 50% ethanol–water solution we also obtained minor crystals of the title compound, *cis*-$C_5H_7N_2^+$ $C_8H_{11}O_4^-$ (Fig. 1) and the structure is reported here.

In the structure of the title compound, the monoanions associate through strong carboxylic acid–carboxyl O—H···O hydrogen bonds (Table 1) giving zigzag chains [graph set *C*(7) (Etter *et al.*, 1990)] which extend along *c* (Fig. 2). The cations provide links between these chains through both pyridinium and amine *N*—*H*···$O_{carboxyl}$ hydrogen bonds, resulting in a three-dimensional structure (Figs. 2,3).

### S2. Experimental

The title compound was synthesized by heating a solution of 1 mmol of cyclohexane-1,2-dicarboxylic anhydride and 1 mmol of 4-aminopyridine in 50 ml of 1:1 ethanol–water under reflux for 10 min. After concentration to 30 ml the solution was allowed to evaporate at room temperature, giving finally a residual viscous oil in which minor well formed colourless crystals of the title compound were found.

### S3. Refinement

Hydrogen atoms involved in hydrogen-bonding interactions were located by difference methods and their positional and isotropic displacement parameters were refined. Other H-atoms were included in the refinement at calculated positions [C–H = 0.93–0.98 Å] and with $U_{iso}$(H) = 1.2$U_{eq}$(C), using a riding-model approximation. In the absence of a suitable heavy atom in the structure, the Friedel pairs (1332) were merged for the final cycles of the refinement. In the structure reported here, the *cis*-CHDC anion has the (1*S*,2*R*) configuration.

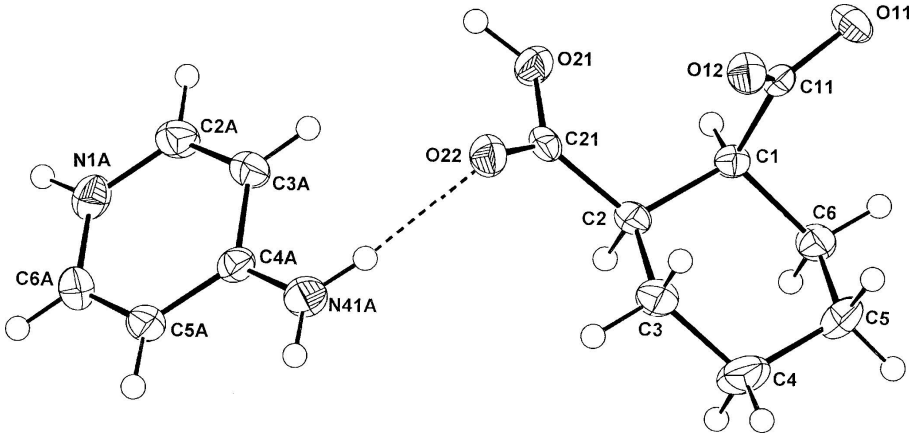



**Figure 1**

Molecular configuration and atom naming scheme for the cation the anion species in the title salt. Inter-species hydrogen bonds are shown as dashed lines and displacement ellipsoids are drawn at the 50% probability level.

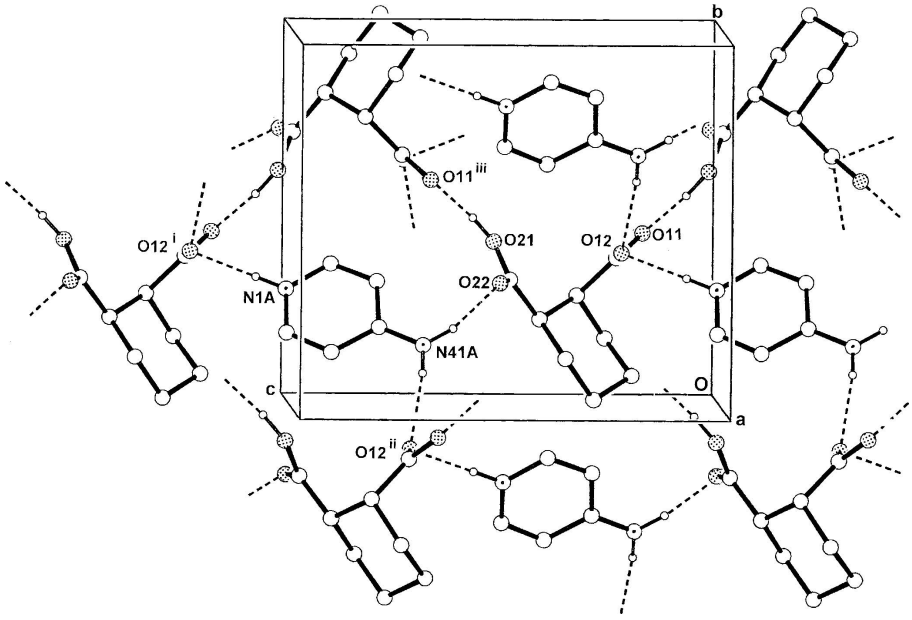



**Figure 2**

A perspective view of the unit cell showing the hydrogen-bonded zigzag *C*(7) *cis*-CHDC monoanion chains and their inter-linking cations, with hydrogen bonds shown as dashed lines. Non-associative H atoms are omitted. For symmetry codes, see Table 1.

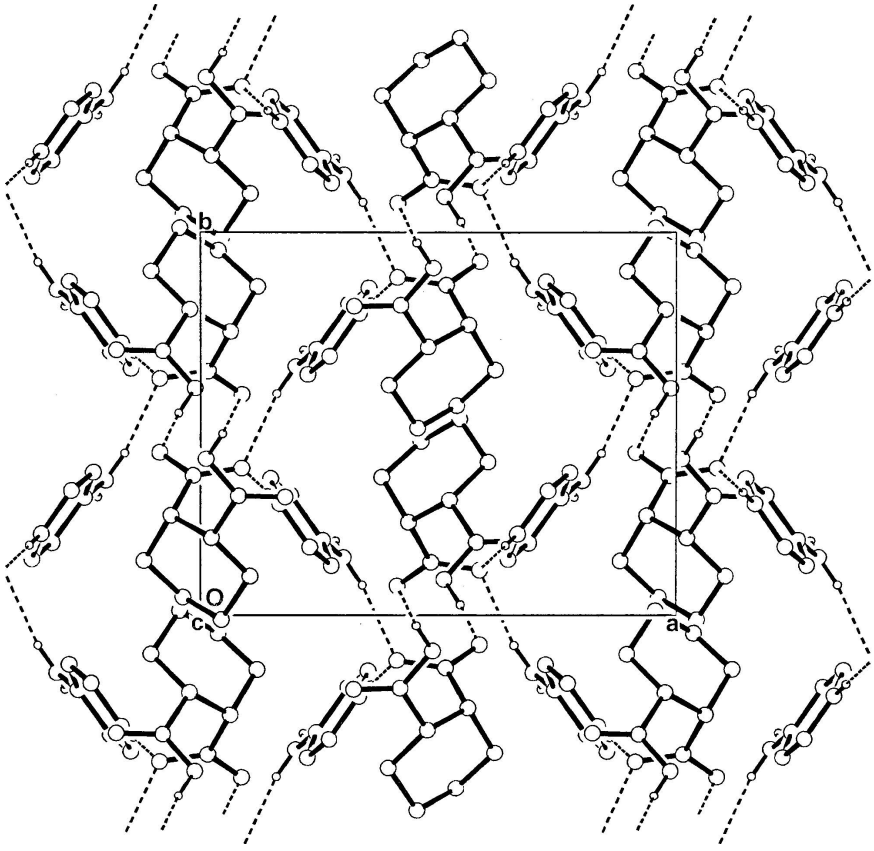



**Figure 3**

A view of the hydrogen-bonded structure looking down the *c* axis.

### 4-Aminopyridinium *cis*-2-carboxycyclohexane-1-carboxylate

*Crystal data*

$C_5H_7N_2^+·C_8H_{11}O_4^-$
$M_r$ = 266.29
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
$a$ = 12.1359 (3) Å
$b$ = 9.8351 (3) Å
$c$ = 11.1850 (3) Å
$V$ = 1335.02 (6) Å$^3$
$Z$ = 4
$F(000)$ = 568
$D_x$ = 1.325 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 4840 reflections
$\theta$ = 3.2–28.7°
$\mu$ = 0.10 mm$^{-1}$
$T$ = 200 K
Block, colourless
0.30 × 0.25 × 0.20 mm

*Data collection*

Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm$^{-1}$
$\omega$ scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
$T_{min}$ = 0.948, $T_{max}$ = 0.990
9670 measured reflections
1709 independent reflections
1448 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.029
$\theta_{max}$ = 28.8°, $\theta_{min}$ = 3.2°
$h$ = −16→15
$k$ = −12→13
$l$ = −13→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.028$
$wR(F^2) = 0.060$
$S = 0.99$
1709 reflections
188 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.15$ e Å$^{-3}$
$\Delta\rho_{min} = -0.16$ e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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 (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O11 | −0.08623 (9) | 0.42384 (12) | 0.16082 (11) | 0.0293 (4) |
| O12 | 0.08627 (8) | 0.38250 (12) | 0.21757 (10) | 0.0262 (3) |
| O21 | 0.01171 (10) | 0.40723 (12) | 0.51058 (12) | 0.0285 (3) |
| O22 | 0.17703 (9) | 0.30714 (12) | 0.50187 (12) | 0.0296 (3) |
| C1 | −0.06239 (12) | 0.25883 (16) | 0.31330 (14) | 0.0202 (4) |
| C2 | 0.02258 (13) | 0.20096 (16) | 0.40246 (15) | 0.0223 (5) |
| C3 | 0.10393 (14) | 0.10098 (18) | 0.34520 (18) | 0.0311 (5) |
| C4 | 0.04378 (15) | −0.01169 (19) | 0.2767 (2) | 0.0410 (6) |
| C5 | −0.03704 (15) | 0.04676 (19) | 0.18597 (18) | 0.0340 (6) |
| C6 | −0.11879 (14) | 0.14099 (17) | 0.24603 (17) | 0.0272 (5) |
| C11 | −0.01574 (12) | 0.36284 (16) | 0.22481 (14) | 0.0200 (5) |
| C21 | 0.07930 (13) | 0.30994 (17) | 0.47466 (14) | 0.0222 (5) |
| N1A | 0.16603 (12) | 0.28917 (15) | 0.99803 (15) | 0.0301 (4) |
| N41A | 0.30073 (13) | 0.14997 (18) | 0.68510 (15) | 0.0320 (5) |
| C2A | 0.14635 (14) | 0.35553 (19) | 0.89470 (16) | 0.0306 (5) |
| C3A | 0.18908 (13) | 0.31222 (17) | 0.78984 (16) | 0.0285 (5) |
| C4A | 0.25659 (12) | 0.19462 (16) | 0.78670 (15) | 0.0236 (5) |
| C5A | 0.27516 (14) | 0.12848 (18) | 0.89698 (16) | 0.0289 (5) |
| C6A | 0.22940 (14) | 0.17671 (18) | 0.99826 (17) | 0.0309 (5) |
| H1 | −0.11940 | 0.30510 | 0.36020 | 0.0240* |
| H2 | −0.02010 | 0.14740 | 0.46010 | 0.0270* |
| H21 | 0.046 (2) | 0.473 (3) | 0.561 (3) | 0.069 (8)* |
| H31B | 0.15200 | 0.14990 | 0.29080 | 0.0370* |
| H32B | 0.14940 | 0.06050 | 0.40700 | 0.0370* |
| H41B | 0.09730 | −0.06800 | 0.23550 | 0.0490* |

| | | | | |
|---|---|---|---|---|
| H42B | 0.00420 | −0.06880 | 0.33280 | 0.0490* |
| H51B | 0.00320 | 0.09640 | 0.12510 | 0.0410* |
| H52B | −0.07640 | −0.02690 | 0.14720 | 0.0410* |
| H61B | −0.16300 | 0.08910 | 0.30200 | 0.0330* |
| H62B | −0.16790 | 0.17830 | 0.18600 | 0.0330* |
| H1A | 0.1408 (18) | 0.319 (2) | 1.0674 (19) | 0.038 (6)* |
| H2A | 0.10230 | 0.43290 | 0.89560 | 0.0370* |
| H3A | 0.17420 | 0.35960 | 0.71970 | 0.0340* |
| H5A | 0.31920 | 0.05120 | 0.89970 | 0.0350* |
| H6A | 0.24190 | 0.13130 | 1.06990 | 0.0370* |
| H41A | 0.3409 (18) | 0.078 (2) | 0.687 (2) | 0.047 (6)* |
| H42A | 0.2841 (16) | 0.193 (2) | 0.615 (2) | 0.038 (6)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O11 | 0.0297 (6) | 0.0270 (6) | 0.0312 (7) | 0.0010 (5) | −0.0051 (5) | 0.0106 (6) |
| O12 | 0.0244 (6) | 0.0291 (6) | 0.0251 (6) | −0.0047 (5) | 0.0004 (5) | 0.0028 (5) |
| O21 | 0.0321 (6) | 0.0263 (6) | 0.0272 (6) | 0.0042 (5) | −0.0021 (6) | −0.0066 (6) |
| O22 | 0.0304 (6) | 0.0316 (6) | 0.0269 (6) | 0.0036 (5) | −0.0062 (5) | −0.0019 (6) |
| C1 | 0.0205 (7) | 0.0188 (8) | 0.0212 (8) | 0.0009 (6) | 0.0009 (6) | −0.0003 (7) |
| C2 | 0.0267 (8) | 0.0193 (8) | 0.0209 (8) | −0.0009 (7) | 0.0007 (7) | 0.0046 (7) |
| C3 | 0.0325 (9) | 0.0253 (9) | 0.0354 (10) | 0.0084 (8) | −0.0064 (8) | −0.0016 (8) |
| C4 | 0.0439 (10) | 0.0247 (9) | 0.0543 (13) | 0.0080 (8) | −0.0046 (10) | −0.0118 (9) |
| C5 | 0.0387 (10) | 0.0270 (9) | 0.0363 (11) | −0.0064 (8) | −0.0040 (9) | −0.0106 (8) |
| C6 | 0.0269 (8) | 0.0226 (8) | 0.0322 (10) | −0.0068 (7) | −0.0053 (7) | 0.0039 (8) |
| C11 | 0.0254 (8) | 0.0166 (8) | 0.0179 (8) | −0.0006 (6) | −0.0006 (6) | −0.0031 (6) |
| C21 | 0.0287 (8) | 0.0222 (8) | 0.0157 (8) | 0.0017 (7) | −0.0004 (6) | 0.0051 (7) |
| N1A | 0.0318 (7) | 0.0344 (8) | 0.0240 (8) | 0.0016 (6) | 0.0021 (7) | −0.0067 (8) |
| N41A | 0.0366 (8) | 0.0293 (9) | 0.0302 (9) | 0.0098 (7) | 0.0024 (7) | −0.0044 (7) |
| C2A | 0.0324 (9) | 0.0258 (9) | 0.0335 (10) | 0.0077 (8) | 0.0012 (8) | −0.0007 (9) |
| C3A | 0.0334 (9) | 0.0241 (9) | 0.0281 (9) | 0.0057 (7) | −0.0019 (8) | 0.0023 (8) |
| C4A | 0.0225 (7) | 0.0209 (8) | 0.0275 (9) | −0.0010 (6) | −0.0021 (7) | −0.0032 (8) |
| C5A | 0.0305 (9) | 0.0233 (9) | 0.0329 (10) | 0.0054 (7) | −0.0085 (8) | −0.0012 (8) |
| C6A | 0.0334 (9) | 0.0328 (9) | 0.0264 (9) | −0.0001 (8) | −0.0080 (8) | 0.0013 (9) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O11—C11 | 1.2665 (19) | C1—H1 | 0.9800 |
| O12—C11 | 1.2556 (18) | C2—H2 | 0.9800 |
| O21—C21 | 1.323 (2) | C3—H32B | 0.9700 |
| O22—C21 | 1.2248 (19) | C3—H31B | 0.9700 |
| O21—H21 | 0.95 (3) | C4—H42B | 0.9700 |
| N1A—C6A | 1.347 (2) | C4—H41B | 0.9700 |
| N1A—C2A | 1.349 (2) | C5—H51B | 0.9700 |
| N41A—C4A | 1.331 (2) | C5—H52B | 0.9700 |
| N1A—H1A | 0.88 (2) | C6—H61B | 0.9700 |
| N41A—H41A | 0.86 (2) | C6—H62B | 0.9700 |

| | | | |
|---|---|---|---|
| N41A—H42A | 0.91 (2) | C2A—C3A | 1.351 (2) |
| C1—C2 | 1.543 (2) | C3A—C4A | 1.418 (2) |
| C1—C11 | 1.532 (2) | C4A—C5A | 1.413 (2) |
| C1—C6 | 1.542 (2) | C5A—C6A | 1.348 (3) |
| C2—C3 | 1.534 (2) | C2A—H2A | 0.9300 |
| C2—C21 | 1.508 (2) | C3A—H3A | 0.9300 |
| C3—C4 | 1.532 (3) | C5A—H5A | 0.9300 |
| C4—C5 | 1.524 (3) | C6A—H6A | 0.9300 |
| C5—C6 | 1.515 (3) | | |

| | | | |
|---|---|---|---|
| C21—O21—H21 | 113.6 (15) | C2—C3—H31B | 109.00 |
| C2A—N1A—C6A | 120.01 (16) | C2—C3—H32B | 109.00 |
| C6A—N1A—H1A | 117.9 (13) | C5—C4—H41B | 109.00 |
| C2A—N1A—H1A | 122.0 (13) | C5—C4—H42B | 109.00 |
| H41A—N41A—H42A | 122 (2) | C3—C4—H42B | 109.00 |
| C4A—N41A—H41A | 118.6 (15) | C3—C4—H41B | 109.00 |
| C4A—N41A—H42A | 119.4 (13) | H41B—C4—H42B | 108.00 |
| C2—C1—C6 | 109.55 (13) | C6—C5—H51B | 109.00 |
| C2—C1—C11 | 114.64 (12) | C4—C5—H52B | 109.00 |
| C6—C1—C11 | 110.53 (13) | C6—C5—H52B | 109.00 |
| C1—C2—C21 | 112.88 (13) | H51B—C5—H52B | 108.00 |
| C1—C2—C3 | 113.38 (14) | C4—C5—H51B | 109.00 |
| C3—C2—C21 | 112.67 (13) | C5—C6—H62B | 109.00 |
| C2—C3—C4 | 111.46 (14) | H61B—C6—H62B | 108.00 |
| C3—C4—C5 | 111.52 (15) | C1—C6—H61B | 109.00 |
| C4—C5—C6 | 110.92 (16) | C1—C6—H62B | 109.00 |
| C1—C6—C5 | 112.68 (14) | C5—C6—H61B | 109.00 |
| O11—C11—C1 | 115.58 (13) | N1A—C2A—C3A | 121.56 (17) |
| O11—C11—O12 | 123.82 (14) | C2A—C3A—C4A | 120.03 (16) |
| O12—C11—C1 | 120.59 (13) | N41A—C4A—C3A | 121.53 (16) |
| O21—C21—C2 | 113.18 (13) | C3A—C4A—C5A | 116.50 (15) |
| O22—C21—C2 | 123.99 (15) | N41A—C4A—C5A | 121.97 (15) |
| O21—C21—O22 | 122.77 (15) | C4A—C5A—C6A | 120.41 (16) |
| C6—C1—H1 | 107.00 | N1A—C6A—C5A | 121.50 (17) |
| C2—C1—H1 | 107.00 | N1A—C2A—H2A | 119.00 |
| C11—C1—H1 | 107.00 | C3A—C2A—H2A | 119.00 |
| C1—C2—H2 | 106.00 | C2A—C3A—H3A | 120.00 |
| C3—C2—H2 | 106.00 | C4A—C3A—H3A | 120.00 |
| C21—C2—H2 | 106.00 | C4A—C5A—H5A | 120.00 |
| C4—C3—H32B | 109.00 | C6A—C5A—H5A | 120.00 |
| H31B—C3—H32B | 108.00 | N1A—C6A—H6A | 119.00 |
| C4—C3—H31B | 109.00 | C5A—C6A—H6A | 119.00 |

| | | | |
|---|---|---|---|
| C6A—N1A—C2A—C3A | −0.2 (3) | C3—C2—C21—O21 | 171.15 (14) |
| C2A—N1A—C6A—C5A | 0.6 (3) | C3—C2—C21—O22 | −11.6 (2) |
| C6—C1—C2—C3 | 51.98 (17) | C1—C2—C21—O21 | 41.13 (19) |
| C11—C1—C2—C21 | 56.73 (18) | C1—C2—C21—O22 | −141.58 (16) |
| C6—C1—C2—C21 | −178.36 (13) | C2—C3—C4—C5 | 53.7 (2) |

| | | | |
|---|---|---|---|
| C11—C1—C2—C3 | −72.93 (17) | C3—C4—C5—C6 | −56.2 (2) |
| C2—C1—C11—O11 | −171.66 (14) | C4—C5—C6—C1 | 57.4 (2) |
| C2—C1—C11—O12 | 9.2 (2) | N1A—C2A—C3A—C4A | −0.2 (3) |
| C6—C1—C11—O11 | 63.95 (18) | C2A—C3A—C4A—N41A | −179.40 (16) |
| C6—C1—C11—O12 | −115.22 (16) | C2A—C3A—C4A—C5A | 0.0 (2) |
| C11—C1—C6—C5 | 72.82 (18) | N41A—C4A—C5A—C6A | 179.81 (17) |
| C2—C1—C6—C5 | −54.43 (19) | C3A—C4A—C5A—C6A | 0.4 (2) |
| C21—C2—C3—C4 | 177.72 (15) | C4A—C5A—C6A—N1A | −0.7 (3) |
| C1—C2—C3—C4 | −52.5 (2) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1*A*—H1*A*···O12$^{i}$ | 0.88 (2) | 1.91 (2) | 2.795 (2) | 180 (3) |
| N41*A*—H41*A*···O12$^{ii}$ | 0.86 (2) | 2.14 (2) | 2.989 (2) | 168 (2) |
| N41*A*—H42*A*···O22 | 0.91 (2) | 2.13 (2) | 2.974 (2) | 152.6 (18) |
| O21—H21···O11$^{iii}$ | 0.95 (3) | 1.59 (3) | 2.5302 (17) | 170 (3) |
| C2*A*—H2*A*···O21$^{iii}$ | 0.93 | 2.46 | 3.287 (2) | 149 |
| C3*A*—H3*A*···O22 | 0.93 | 2.49 | 3.225 (2) | 136 |
| C3*A*—H3*A*···O11$^{iii}$ | 0.93 | 2.47 | 3.222 (2) | 138 |
| C6*A*—H6*A*···O11$^{iv}$ | 0.93 | 2.38 | 3.048 (2) | 128 |
| C3—H31*B*···O12 | 0.97 | 2.56 | 3.123 (2) | 117 |

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