



Crystal structure of (1*R*,4*R*)-*tert*-butyl 3-oxo-2-oxa-5-azabicyclo[2.2.2]octane-5-carboxylate

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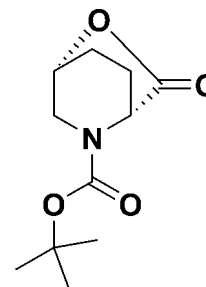
In the title compound, C₁₁H₁₇NO₄, commonly known as *N*-*tert*-butoxycarbonyl-5-hydroxy-D-pipecolic acid lactone, the absolute configuration is (1*R*,4*R*) due to the enantiomeric purity of the starting material which remains unchanged during the course of the reaction. In the crystal there no intermolecular hydrogen bonds.

Keywords: crystal structure; (1*R*,4*R*)- aza-oxa bicyclic chiral lactone.

CCDC reference: 1062075

1. Related literature

For background information on 5-hydroxypipicolinic acid and related compounds, see: Witkop & Foltz (1957); Hoarau *et al.* (1996); Sun *et al.* (2008). For the synthesis of a related compound, see: Krishnamurthy *et al.* (2014). For crystal structures of related lactones, see: (1*S*,4*S*) conformer, racemic mixture, Moriguchi, Krishnamurthy, Arai & Tsuge (2014); Moriguchi, Krishnamurthy, Arai, Matsumoto *et al.* (2014).



2. Experimental

2.1. Crystal data

C₁₁H₁₇NO₄
M_r = 227.26
Orthorhombic, P2₁2₁2₁
a = 9.6472 (4) Å
b = 9.7084 (4) Å
c = 12.2323 (5) Å

V = 1145.66 (8) Å³
Z = 4
Mo Kα radiation
μ = 0.10 mm⁻¹
T = 90 K
0.45 × 0.40 × 0.40 mm

2.2. Data collection

Bruker APEX2 KY CCD
diffractometer
Absorption correction: multi-scan
SADABS (Bruker, 2009)
T_{min} = 0.870, T_{max} = 0.961

13518 measured reflections
2791 independent reflections
2728 reflections with I > 2σ(I)
R_{int} = 0.021

2.3. Refinement

R[F² > 2σ(F²)] = 0.030
wR(F²) = 0.081
S = 1.03
2791 reflections
148 parameters
H-atom parameters constrained

Δρ_{max} = 0.24 e Å⁻³
Δρ_{min} = -0.27 e Å⁻³
Absolute structure: Flack (1983),
2933 Friedel pairs
Absolute structure parameter:
0.1 (7)

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: ZS2333).

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supporting information

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Crystal structure of (1*R*,4*R*)-*tert*-butyl 3-oxo-2-oxa-5-azabicyclo[2.2.2]octane-5-carboxylate

Suvratha Krishnamurthy, Venkataprasad Jalli, Tarun Chand Vagvala, Tetsuji Moriguchi and Akihiko Tsuge

S1. Comment

5-Hydroxypipicolinic acid is a higher homologue of 4-hydroxyproline, which is found in dates (Witkop & Foltz, 1957). 4-Hydroxyproline is formed by post-translational modification of proline in collagen and is responsible for enhancing its stability. Literature reports the synthesis of 5-hydroxypipicolinic acid derivatives (Hoarau *et al.*, 1996; Sun *et al.*, 2008), generally forming diastereomeric mixtures of *cis*- and *trans*-5-hydroxypipicolinic acids. Therefore such syntheses suffer from disadvantages of separation of the diastereomers making the procedure very tedious. A facile procedure to isolate this amino acid was desirable.

Our previous communication reported the synthesis of a 4-hydroxyproline derivative from an amino acid bearing epoxide (Krishnamurthy *et al.* 2014). It is reported in this study that the *cis*-isomer undergoes intramolecular lactonization to *tert*-butyl- 3-oxo-2-oxa-5-azabicyclo[2.2.1]heptane-5-carboxylate, making the isolation from the *trans* ester highly feasible. Based on this observation it can be expected that *cis*-5-hydroxypipicolinic acids would also undergo *in situ* intramolecular lactonization. In fact, when a mixture of a *cis*- and *trans*-5-hydroxypipicolinic acid derivatives was reacted under acidic conditions, the *cis*-isomer successfully converted to the lactone (I), subsequently readily separated from the remaining *trans*-isomer. We had previously reported the crystal structure of racemic *tert*-butyl- 3-oxo-2-oxa-5-azabicyclo[2.2.2]octane-5-carboxylate (Moriguchi, Krishnamurthy, Arai, Tsuge *et al.*, 2014) and (1*S*,4*S*)-*tert*-butyl 3-oxo-2-oxa-5-azabicyclo[2.2.2]octane-5-carboxylate (Moriguchi, Krishnamurthy, Arai, & Tsuge, 2014). Herein we would like to report the crystal structure of enantiomerically pure (1*R*,4*R*)-*tert*-butyl- 3-oxo-2-oxa-5-azabicyclo[2.2.2]octane-5-carboxylate, C₁₁H₁₇N O₄, (I).

The title compound (I), commonly known as *N-tert*-butoxycarbonyl-5-hydroxy-D-pipicolinic acid lactone, was derived from a starting product having a *cis* configuration for both hydroxyl and carboxyl groups, leading to lactone formation (Fig. 1). The nitrogen atom N1 appears next to the bridge-head atom within the bicyclic ring system. The absolute configuration of the compound was found to be (1*R*,4*R*) due to the configuration of the starting material (Fig. 3). The Flack structure parameter (Flack, 1983) determined for (I) [0.1 (7)], although not definitive because of the uncertainty factor, is considered to provide adequate supporting evidence for this configuration. The desired hydrophobic conformer (1*R*,4*R*), (I) was easily isolated from hydrophilic (1*R*,4*S*)-(4) (Fig. 3). The intramolecular lactonization is possible only in (1*R*,4*R*)-(3) isomer due to its configuration. The hydroxyl and the carboxyl groups are in close proximity due to the *cis*- configuration of (1*R*,4*R*)-(3), which leads to the intramolecular lactonization with loss of EtOH. With the (1*R*,4*S*)-(4) isomer the hydroxyl and the carboxyl groups are far apart due to the *trans*- configuration, thus preventing the lactonization. In the crystal there no formal intramolecular hydrogen bonds (Fig. 2).

This work represents the first structural characterization of this (1*R*,4*R*)- aza and oxa bicyclic chiral lactone characterized by X-ray analysis.

S2. Experimental

The basic reaction scheme for preparation of the title compound (I) is shown in Fig. 3. To a ice cooled solution of 4 mol/L HCl in 1,4-dioxane (16 mL), a solution of diastereomeric (1) (0.97 g, 3.56 mmol) in 0.5 mL of 1,4-dioxane was added. This reaction mixture was then warmed to room temperature and stirred. After 3 h most of the volatile materials were removed under vacuum resulting in a crude oily mixture. Trituration with diethyl ether followed by decantation resulted in (2) as a foam (0.71 g, 95 %). DIEA (0.89 mL, 5.07 mmol) was added to a solution of (2) (0.71 g, 3.38 mmol) in DMF (13 mL) and stirred at 50 °C. After 6 h the solution was warmed to room temperature, followed by addition of Boc₂O (3.96 g, 18.1 mmol), additional DIEA (0.3 mL, 1.69 mmol) and stirred at room temperature for 18 h. The DMF was evaporated and the crude mixture was subsequently washed with 10% aqueous citric acid, 4% aqueous NaHCO₃, brine, dried (MgSO₄), filtered and evaporated to obtain an oil. The crude product was purified by silica gel column chromatography with (CHCl₃/MeOH, 100:0 to 98:2, v/v) to yield (1*R*,4*R*), (I) (0.18 g, 23%) as a white solid. Single crystals were obtained by vapour diffusion method at room temperature, i.e., hexane vapour was allowed to diffuse into an EtOAc (0.5 ml) solution of (1*R*,4*R*), (I) at room temperature. Single crystals suitable for analysis were obtained after a week.

¹H NMR 4.61-4.82 (2H, m), 3.63 (1H, m), 3.45 (1H, m), 2.22 (1H, br s), 2.11 (1H, m), 2.00 (1H, m), 1.80 (1H, m), 1.47 (9H, s); MS (FAB *m/z*): 228 (74), 190 (44), 172 (100), 137 (50), 128 (68), 55 (47). HRMS(FAB) calcd for C₁₁H₁₈N₁O₄ [M + H]⁺ 228.12358, found 228.1243

S3. Refinement

All hydrogen atoms were placed in calculated positions (C—H = 0.98–1.00 Å) and allowed to ride, with $U_{\text{iso}}\text{H} = 1.5U_{\text{eq}}\text{C}(\text{methyl})$ or $1.2U_{\text{eq}}\text{C}(\text{methine and methylene})$. The absolute structure parameter (Flack, 1983) for (I) [0.01 (7) for 2933 Friedel pairs], although not definitive is sufficient to confirm the (1*R*,4*R*) identity, as distinct from that of the known (1*S*,4*S*) conformer (Moriguchi, Krishnamurthy, Arai & Tsuge, 2014).

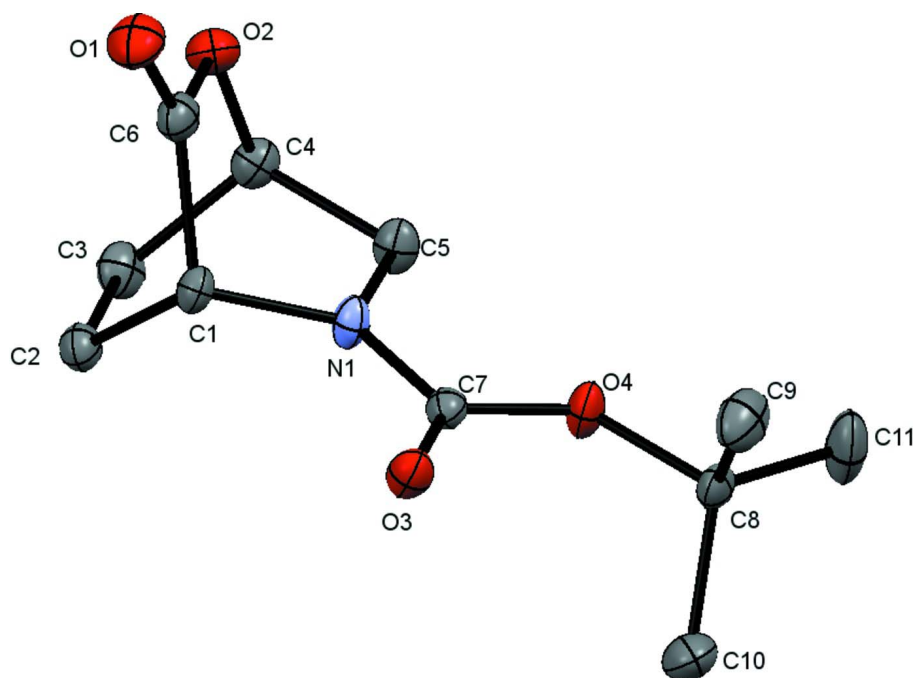


Figure 1

Molecular configuration and atom numbering scheme for the title compound with displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms are omitted for clarity.

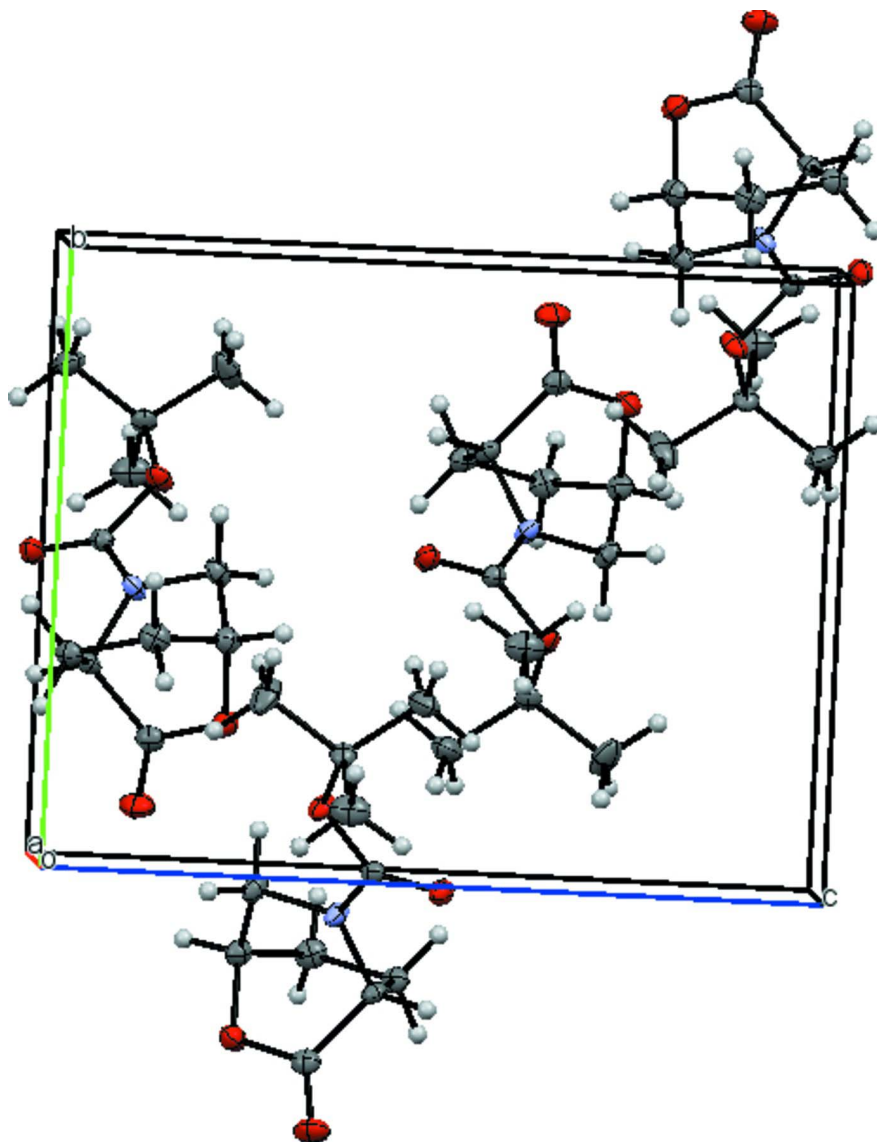
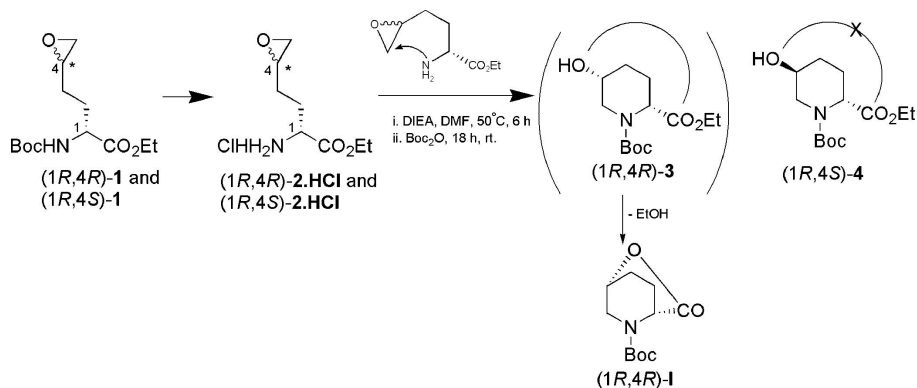


Figure 2

Crystal packing diagram of the title compound.


Figure 3

Synthetic scheme for the title compound (I).

(1*R*,4*R*)-tert-butyl 3-oxo-2-oxa-5-azabicyclo[2.2.2]octane-5-carboxylate
Crystal data
 $C_{11}H_{17}NO_4$
 $M_r = 227.26$

 Orthorhombic, $P2_12_12_1$
 $a = 9.6472$ (4) Å

 $b = 9.7084$ (4) Å

 $c = 12.2323$ (5) Å

 $V = 1145.66$ (8) Å³
 $Z = 4$
 $F(000) = 488$
 $D_x = 1.318$ Mg m⁻³

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

Cell parameters from 9630 reflections

 $\theta = 2.7$ – 28.7°
 $\mu = 0.10$ mm⁻¹
 $T = 90$ K

Prism, colorless

 $0.45 \times 0.40 \times 0.40$ mm

Data collection

 Bruker APEX2 KY CCD
 diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 Detector resolution: 16.6666 pixels mm⁻¹
 φ and ω -scans

Absorption correction: multi-scan

SADABS (Bruker, 2009)

 $T_{\min} = 0.870$, $T_{\max} = 0.961$

13518 measured reflections

2791 independent reflections

 2728 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\max} = 28.7^\circ$, $\theta_{\min} = 2.7^\circ$
 $h = -12 \rightarrow 12$
 $k = -12 \rightarrow 12$
 $l = -16 \rightarrow 16$
Refinement

 Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.030$
 $wR(F^2) = 0.081$
 $S = 1.02$

2791 reflections

148 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

 $w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.1302P]$

 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.27$ e Å⁻³

 Extinction correction: *SHELXL97*

Extinction coefficient: 0.0015

 Absolute structure: Flack (1983), 2933 Friedel
 pairs

Absolute structure parameter: 0.1 (7)

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)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.65404 (10)	0.31574 (9)	0.06026 (7)	0.01427 (18)
H1	0.71	0.2937	-0.0063	0.017*
C2	0.49996 (10)	0.33409 (10)	0.03171 (8)	0.0176 (2)
H2A	0.4628	0.2485	-0.0011	0.021*
H2B	0.4883	0.41	-0.0215	0.021*
C3	0.42204 (10)	0.36786 (11)	0.13907 (9)	0.0206 (2)
H3B	0.378	0.4597	0.1335	0.025*
H3A	0.3485	0.2987	0.1521	0.025*
C4	0.52501 (11)	0.36654 (10)	0.23335 (8)	0.01785 (19)
H4	0.475	0.3836	0.3037	0.021*
C5	0.63878 (10)	0.47291 (11)	0.21909 (8)	0.0178 (2)
H5B	0.5993	0.567	0.2175	0.021*
H5A	0.7064	0.4671	0.2798	0.021*
C6	0.66134 (10)	0.19956 (10)	0.14366 (8)	0.01762 (19)
C7	0.81399 (10)	0.51222 (9)	0.07261 (8)	0.01444 (19)
C8	0.96933 (10)	0.70326 (10)	0.12123 (8)	0.01618 (19)
C9	1.10001 (11)	0.61760 (12)	0.11084 (11)	0.0276 (2)
H9A	1.0964	0.5638	0.0431	0.041*
H9B	1.1072	0.5551	0.1735	0.041*
H9C	1.181	0.6785	0.1092	0.041*
C10	0.94288 (14)	0.79172 (11)	0.02135 (10)	0.0272 (3)
H10A	0.8622	0.8507	0.0345	0.041*
H10B	0.925	0.7324	-0.0419	0.041*
H10C	1.0243	0.8492	0.0069	0.041*
C11	0.97332 (14)	0.79157 (13)	0.22359 (10)	0.0311 (3)
H11A	0.9866	0.7325	0.2877	0.047*
H11B	0.8858	0.842	0.2307	0.047*
H11C	1.0502	0.8572	0.2184	0.047*
N1	0.70560 (9)	0.44009 (8)	0.11448 (7)	0.01612 (17)
O1	0.71703 (8)	0.08995 (8)	0.13238 (7)	0.02526 (18)
O2	0.59161 (8)	0.23044 (8)	0.23684 (6)	0.01975 (16)
O3	0.86922 (8)	0.48744 (7)	-0.01469 (6)	0.01893 (16)
O4	0.84801 (7)	0.61426 (7)	0.14236 (6)	0.01846 (16)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0141 (4)	0.0119 (4)	0.0168 (4)	-0.0019 (3)	0.0008 (3)	-0.0028 (3)
C2	0.0155 (5)	0.0172 (4)	0.0201 (4)	-0.0010 (4)	-0.0030 (3)	-0.0007 (3)
C3	0.0129 (4)	0.0230 (5)	0.0259 (5)	0.0005 (4)	0.0000 (4)	-0.0014 (4)
C4	0.0160 (4)	0.0179 (4)	0.0196 (4)	-0.0001 (4)	0.0032 (4)	-0.0012 (4)
C5	0.0166 (4)	0.0198 (4)	0.0172 (4)	-0.0019 (4)	0.0050 (3)	-0.0052 (3)
C6	0.0143 (4)	0.0171 (4)	0.0215 (5)	-0.0016 (3)	-0.0017 (4)	-0.0002 (4)
C7	0.0145 (4)	0.0126 (4)	0.0162 (4)	0.0002 (3)	-0.0017 (3)	0.0000 (3)
C8	0.0151 (4)	0.0155 (4)	0.0179 (4)	-0.0058 (4)	-0.0001 (3)	-0.0001 (3)
C9	0.0166 (5)	0.0241 (5)	0.0421 (6)	-0.0010 (4)	-0.0024 (4)	0.0012 (5)
C10	0.0361 (6)	0.0179 (5)	0.0275 (5)	-0.0039 (4)	-0.0051 (4)	0.0051 (4)
C11	0.0330 (6)	0.0349 (6)	0.0255 (6)	-0.0171 (5)	0.0039 (5)	-0.0126 (5)
N1	0.0161 (4)	0.0161 (4)	0.0162 (4)	-0.0040 (3)	0.0032 (3)	-0.0056 (3)
O1	0.0252 (4)	0.0171 (3)	0.0334 (4)	0.0050 (3)	-0.0025 (4)	0.0002 (3)
O2	0.0211 (4)	0.0186 (3)	0.0195 (3)	0.0009 (3)	0.0015 (3)	0.0029 (3)
O3	0.0219 (4)	0.0184 (3)	0.0165 (3)	-0.0038 (3)	0.0041 (3)	-0.0022 (3)
O4	0.0175 (3)	0.0185 (3)	0.0194 (3)	-0.0073 (3)	0.0040 (3)	-0.0059 (3)

Geometric parameters (\AA , $^\circ$)

C1—N1	1.4645 (11)	C6—O2	1.3570 (12)
C1—C6	1.5225 (13)	C7—O3	1.2175 (12)
C1—C2	1.5373 (14)	C7—O4	1.3481 (11)
C1—H1	1.0	C7—N1	1.3587 (12)
C2—C3	1.5482 (14)	C8—O4	1.4776 (11)
C2—H2A	0.99	C8—C9	1.5156 (15)
C2—H2B	0.99	C8—C10	1.5150 (14)
C3—C4	1.5222 (15)	C8—C11	1.5180 (14)
C3—H3B	0.99	C9—H9A	0.98
C3—H3A	0.99	C9—H9B	0.98
C4—O2	1.4699 (13)	C9—H9C	0.98
C4—C5	1.5171 (13)	C10—H10A	0.98
C4—H4	1.0	C10—H10B	0.98
C5—N1	1.4677 (12)	C10—H10C	0.98
C5—H5B	0.99	C11—H11A	0.98
C5—H5A	0.99	C11—H11B	0.98
C6—O1	1.2000 (12)	C11—H11C	0.98
N1—C1—C6	106.95 (7)	O3—C7—O4	126.43 (9)
N1—C1—C2	109.62 (8)	O3—C7—N1	124.45 (9)
C6—C1—C2	106.43 (8)	O4—C7—N1	109.12 (8)
N1—C1—H1	111.2	O4—C8—C9	110.65 (8)
C6—C1—H1	111.2	O4—C8—C10	109.82 (8)
C2—C1—H1	111.2	C9—C8—C10	112.55 (9)
C1—C2—C3	107.54 (8)	O4—C8—C11	101.89 (8)
C1—C2—H2A	110.2	C9—C8—C11	110.98 (10)

C3—C2—H2A	110.2	C10—C8—C11	110.44 (9)
C1—C2—H2B	110.2	C8—C9—H9A	109.5
C3—C2—H2B	110.2	C8—C9—H9B	109.5
H2A—C2—H2B	108.5	H9A—C9—H9B	109.5
C4—C3—C2	108.91 (8)	C8—C9—H9C	109.5
C4—C3—H3B	109.9	H9A—C9—H9C	109.5
C2—C3—H3B	109.9	H9B—C9—H9C	109.5
C4—C3—H3A	109.9	C8—C10—H10A	109.5
C2—C3—H3A	109.9	C8—C10—H10B	109.5
H3B—C3—H3A	108.3	H10A—C10—H10B	109.5
O2—C4—C5	107.40 (8)	C8—C10—H10C	109.5
O2—C4—C3	108.35 (8)	H10A—C10—H10C	109.5
C5—C4—C3	112.29 (9)	H10B—C10—H10C	109.5
O2—C4—H4	109.6	C8—C11—H11A	109.5
C5—C4—H4	109.6	C8—C11—H11B	109.5
C3—C4—H4	109.6	H11A—C11—H11B	109.5
N1—C5—C4	105.68 (8)	C8—C11—H11C	109.5
N1—C5—H5B	110.6	H11A—C11—H11C	109.5
C4—C5—H5B	110.6	H11B—C11—H11C	109.5
N1—C5—H5A	110.6	C7—N1—C1	121.03 (8)
C4—C5—H5A	110.6	C7—N1—C5	123.69 (8)
H5B—C5—H5A	108.7	C1—N1—C5	115.13 (8)
O1—C6—O2	120.96 (9)	C6—O2—C4	113.01 (7)
O1—C6—C1	126.91 (9)	C7—O4—C8	120.79 (7)
O2—C6—C1	112.10 (8)		
N1—C1—C2—C3	-56.55 (10)	C6—C1—N1—C7	123.18 (10)
C6—C1—C2—C3	58.77 (10)	C2—C1—N1—C7	-121.84 (9)
C1—C2—C3—C4	-1.58 (11)	C6—C1—N1—C5	-52.53 (11)
C2—C3—C4—O2	-57.69 (10)	C2—C1—N1—C5	62.46 (10)
C2—C3—C4—C5	60.76 (11)	C4—C5—N1—C7	-179.64 (9)
O2—C4—C5—N1	61.20 (10)	C4—C5—N1—C1	-4.06 (11)
C3—C4—C5—N1	-57.81 (10)	O1—C6—O2—C4	-177.27 (9)
N1—C1—C6—O1	-126.60 (11)	C1—C6—O2—C4	0.91 (11)
C2—C1—C6—O1	116.29 (11)	C5—C4—O2—C6	-61.30 (10)
N1—C1—C6—O2	55.36 (10)	C3—C4—O2—C6	60.21 (10)
C2—C1—C6—O2	-61.75 (10)	O3—C7—O4—C8	-4.98 (15)
O3—C7—N1—C1	5.74 (15)	N1—C7—O4—C8	175.60 (8)
O4—C7—N1—C1	-174.83 (8)	C10—C8—O4—C7	67.57 (11)
O3—C7—N1—C5	-178.93 (9)	C9—C8—O4—C7	-57.30 (12)
O4—C7—N1—C5	0.50 (13)	C11—C8—O4—C7	-175.35 (9)