

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

**(3*R*,4*S*)-3,4-Isopropylidenedioxy-3,4-dihydro-2*H*-pyrrole 1-oxide**Mari Fe Flores,<sup>a</sup> Pilar Garcia,<sup>a</sup> Narciso M. Garrido,<sup>a</sup> Francisca Sanz<sup>b</sup> and David Diez<sup>a\*</sup><sup>a</sup>Departamento de Química Orgánica, Universidad de Salamanca, Plaza de los Caidos, 37008-Salamanca, Spain, and <sup>b</sup>Servicio General de Rayos X, Universidad de Salamanca, Plaza de los Caidos, 37008-Salamanca, Spain

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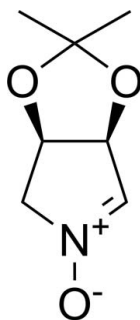
Received 31 March 2011; accepted 7 April 2011

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.033;  $wR$  factor = 0.091; data-to-parameter ratio = 11.6.

The title compound  $\text{C}_7\text{H}_{11}\text{NO}_3$  was prepared by intramolecular nucleophilic displacement of 2,3-*O*-isopropylidene-*D*-erythronolactol. There are two molecules in the asymmetric unit, which are related by a pseudo-inversion centre. The crystal structure determination confirms unequivocally the configuration of the chiral centres as 3*S*,4*R*. In the crystal structure, intermolecular  $\text{C}-\text{H}\cdots\text{O}$  interactions link the molecules (into infinite zigzag chains along the  $a$  axis).

## Related literature

Nitrones play a useful role in the synthesis of complex molecular frameworks, undergoing several synthetically useful reactions such as 1,3-dipolar cycloadditions (Tufariello, 1984) and nucleophilic addition (Merino *et al.*, 2000; Lombardo & Trombini, 2002). They also allow direct access to nitrones by simple reactions, see: Döpp & Döpp (1990); Hamer & Macaluso (1964). For the use of the title compound as a starting material in the synthesis of potential therapeutic (antibiotic, antiviral, antitumoral) agents, see: Hall *et al.* (1997); Closa & Wightman (1998); McCaig *et al.* (1998); Cicchi *et al.* (2002); Revuelta *et al.* (2007). For a related structure, see: Keleşoğlu *et al.* (2010). For the preparation of the title compound, see: Flores *et al.* (2010); Cicchi *et al.* (2006).



## Experimental

## Crystal data

|                                      |                                   |
|--------------------------------------|-----------------------------------|
| $\text{C}_7\text{H}_{11}\text{NO}_3$ | $V = 1604.2$ (6) Å <sup>3</sup>   |
| $M_r = 157.17$                       | $Z = 8$                           |
| Monoclinic, $C_2$                    | Cu $K\alpha$ radiation            |
| $a = 11.335$ (2) Å                   | $\mu = 0.86$ mm <sup>-1</sup>     |
| $b = 5.4467$ (11) Å                  | $T = 298$ K                       |
| $c = 26.508$ (5) Å                   | $0.15 \times 0.10 \times 0.08$ mm |
| $\beta = 101.40$ (3)°                |                                   |

## Data collection

|  |  |
|--|--|
| Bruker APEXII CCD area-detector diffractometer           | 4369 measured reflections              |
| Absorption correction: multi-scan (SADABS; Bruker, 2006) | 2365 independent reflections           |
| $T_{\min} = 0.902$ , $T_{\max} = 0.934$                  | 2261 reflections with $I > 2\sigma(I)$ |
|  | $R_{\text{int}} = 0.022$               |

## Refinement

|                                 |   |
|---------------------------------|---|
| $R[F^2 > 2\sigma(F^2)] = 0.033$ | H-atom parameters constrained                       |
| $wR(F^2) = 0.091$               | $\Delta\rho_{\text{max}} = 0.16$ e Å <sup>-3</sup>  |
| $S = 1.05$                      | $\Delta\rho_{\text{min}} = -0.11$ e Å <sup>-3</sup> |
| 2365 reflections                | Absolute structure: Flack (1983),                   |
| 204 parameters                  | 761 Friedel pairs                                   |
| 1 restraint                     | Flack parameter: 0.0 (2)                            |

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                                  | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|--------------|--------------------|-------------|----------------------|
| $\text{C3}-\text{H3}\cdots\text{O1}^{\text{i}}$       | 0.93         | 2.44               | 3.366 (3)   | 171                  |
| $\text{C1}-\text{H1}\cdots\text{O3}^{\text{ii}}$      | 0.98         | 2.38               | 3.355 (3)   | 171                  |
| $\text{C2}-\text{H2A}\cdots\text{O3}^{\text{iii}}$    | 0.97         | 2.70               | 3.441 (3)   | 134                  |
| $\text{C2}-\text{H2B}\cdots\text{O3}^{\text{iv}}$     | 0.97         | 2.41               | 3.120 (3)   | 130                  |
| $\text{C2}'-\text{H2}'1\cdots\text{O2}'^{\text{v}}$   | 0.97         | 2.49               | 3.247 (2)   | 135                  |
| $\text{C4}'-\text{H4}'\cdots\text{O3}'^{\text{vi}}$   | 0.98         | 2.48               | 3.375 (3)   | 152                  |
| $\text{C2}'-\text{H2}'2\cdots\text{O3}'^{\text{vii}}$ | 0.97         | 2.61               | 3.254 (2)   | 124                  |
| $\text{C3}'-\text{H3}'\cdots\text{O3}'^{\text{viii}}$ | 0.93         | 2.48               | 3.345 (3)   | 156                  |

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

Data collection: APEX2 (Bruker, 2006); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: Mercury (Macrae *et al.*, 2006); software used to prepare material for publication: SHELXL97.

The authors are grateful to the MICINN (CTQ2009-11172), Junta de Castilla y Leon, for financial support (GR178 and SA001A09) and for the doctoral fellowships awarded to MFF.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5507).

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**supplementary materials**

*Acta Cryst.* (2011). E67, o1116-o1117 [ doi:10.1107/S1600536811013055 ]

### (3*R*,4*S*)-3,4-Isopropylidenedioxy-3,4-dihydro-2*H*-pyrrole 1-oxide

M. F. Flores, P. Garcia, N. M. Garrido, F. Sanz and D. Diez

#### Comment

Nitrones have been the subject of intense research efforts, because of the wide role played in the synthesis of complex molecular frameworks. They undergo several synthetically useful reactions such as 1,3-dipolar cycloadditions, (Tufariello *et al.*, 1984) nucleophilic additions, (Merino *et al.*, 2000; Lombardo *et al.*, 2002). Both the reactions give rise to the formation of new carbon-carbon bonds, often with a high degree of stereocontrol. These features, together with the direct access to nitrones by simple reactions (Hamer *et al.*, 1964; Döpp *et al.*, 1990), and their stability which permits isolation and long storage, make nitrones ideal tools for application in organic syntheses, particularly in the field of alkaloids, nitrogen containing natural products or bioactive analogues. The construction of highly functionalized nitrogen heterocycles in a stereoselective manner is an important focus of medicinal and natural product chemistry. Although, in the last few years, the title compound has been reported more and more in the literature as a starting material due to its biological relevance in the synthesis of polyhydroxypyrrolidines or polyhydroxypyrrolizidines, both interesting compounds as potential glycosidase inhibitors and consequently as potential therapeutic (antibiotic, antiviral, antitumoral) agents (Hall *et al.*, 1997; Closa *et al.*, 1998; McCaig *et al.*, 1998; Cicchi *et al.*, 2002; Revuelta *et al.*, 2007) there was not any crystallographic data.

Following our special interest in nitrogen compounds such as isoxazolidines, we prepared the title N-oxide, and its crystal structure is reported here.

The asymmetric unit contains two symmetrically independent molecules. The title molecule consists of a pyrroline-N-oxide ring with an isopropylidenedioxy as substituent. All the bond lengths and angles are within the normal ranges. The carbonyl group at N1 is coplanar with the pyrroline ring being the O3—N1=C3—C4 and O3'-N1'=C3'-C4' torsion angles of 179.1 (4)° and 179.2 (7)°, respectively. These results are in good agreement with the literature (Keleşoğlu *et al.*, 2010).

In the crystal structure, intermolecular C—H···O interactions (Table 1) link the molecules (Fig. 2) into infinite zigzag chains along the *a* axis.

#### Experimental

The title N-oxide was obtained by intramolecular nucleophilic displacement, which is based on a simple one-pot procedure employing NH<sub>2</sub>OSiMe<sub>2</sub>*t*-Bu, methanesulfonyl chloride, and 2,3-*O*-iso-propylidene-*D*-erythronolactol, according to the methodology described by Cicchi *et al.* (2006) and by us (Flores *et al.*, 2010). Well shaped colourless single crystals were obtained by crystallization from CH<sub>2</sub>Cl<sub>2</sub>/MeOH.

#### Refinement

Hydrogen atoms were positioned geometrically, with C—H distances constrained to 0.93 Å (aromatic CH), 0.96 Å (methyl), 0.97 Å (methylene) and 0.98 Å (methine) and refined in riding mode with  $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C})$ , where  $x = 1.5$  for methyl H atoms and  $x = 1.2$  for all other atoms.

Figures

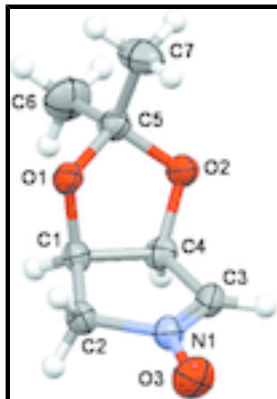


Fig. 1. Molecular structure of  $C_7H_{11}NO_3$ .

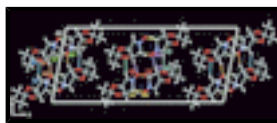


Fig. 2. Crystal packing of  $C_7H_{11}NO_3$  view along  $b$  axis, showing intermolecular hydrogen bonding.

**(3*R*,4*S*)-3,4-Isopropylidenedioxy-3,4-dihydro-2*H*-pyrrole 1-oxide**

*Crystal data*

$C_7H_{11}NO_3$

$M_r = 157.17$

Monoclinic,  $C_2$

Hall symbol:  $C\ 2y$

$a = 11.335\ (2)\ \text{\AA}$

$b = 5.4467\ (11)\ \text{\AA}$

$c = 26.508\ (5)\ \text{\AA}$

$\beta = 101.40\ (3)^\circ$

$V = 1604.2\ (6)\ \text{\AA}^3$

$Z = 8$

$F(000) = 672$

$D_x = 1.302\ \text{Mg m}^{-3}$

Cu  $K\alpha$  radiation,  $\lambda = 1.54178\ \text{\AA}$

Cell parameters from 2365 reflections

$\theta = 1.7\text{--}66.9^\circ$

$\mu = 0.86\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Prismatic, colourless

$0.15 \times 0.10 \times 0.08\ \text{mm}$

*Data collection*

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

phi and  $\omega$  scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2006)

$T_{\min} = 0.902$ ,  $T_{\max} = 0.934$

4369 measured reflections

2365 independent reflections

2261 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$

$\theta_{\max} = 66.9^\circ$ ,  $\theta_{\min} = 1.7^\circ$

$h = -13 \rightarrow 11$

$k = -6 \rightarrow 5$

$l = -28 \rightarrow 31$

Refinement

|  |   |
|--|---|
| Refinement on $F^2$  | Hydrogen site location: inferred from neighbouring sites  |
| Least-squares matrix: full                                     | H-atom parameters constrained   |
| $R[F^2 > 2\sigma(F^2)] = 0.033$                                | $w = 1/[\sigma^2(F_o^2) + (0.0479P)^2 + 0.4187P]$<br>where $P = (F_o^2 + 2F_c^2)/3$                   |
| $wR(F^2) = 0.091$  | $(\Delta/\sigma)_{\max} = 0.001$  |
| $S = 1.05$   | $\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$   |
| 2365 reflections   | $\Delta\rho_{\min} = -0.11 \text{ e } \text{\AA}^{-3}$  |
| 204 parameters   | Extinction correction: <i>SHELXL</i> ,<br>$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| 1 restraint  | Extinction coefficient: 0.00105 (18)  |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 761 Friedel pairs   |
| Secondary atom site location: difference Fourier map           | Flack parameter: 0.0 (2)  |

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 ( $\text{\AA}^2$ )

|      | $x$          | $y$        | $z$         | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|--------------|------------|-------------|----------------------------------|
| O1'  | 0.30975 (12) | 0.5460 (3) | 0.84350 (5) | 0.0571 (4)                       |
| O2'  | 0.44334 (11) | 0.8279 (3) | 0.88116 (5) | 0.0566 (4)                       |
| O3'  | 0.14788 (12) | 0.7976 (4) | 0.96810 (6) | 0.0693 (5)                       |
| N1'  | 0.23631 (12) | 0.7223 (3) | 0.94785 (5) | 0.0481 (4)                       |
| C1'  | 0.33483 (16) | 0.4762 (4) | 0.89604 (7) | 0.0476 (4)                       |
| H1'  | 0.3729       | 0.3142     | 0.9013      | 0.057*                           |
| C3'  | 0.34095 (15) | 0.8196 (4) | 0.95186 (6) | 0.0464 (4)                       |
| H3'  | 0.3657       | 0.9601     | 0.9709      | 0.056*                           |
| C4'  | 0.41764 (14) | 0.6811 (4) | 0.92243 (6) | 0.0463 (4)                       |
| H4'  | 0.4907       | 0.6168     | 0.9445      | 0.056*                           |
| C5'  | 0.40764 (17) | 0.6940 (4) | 0.83486 (7) | 0.0525 (5)                       |
| C6'  | 0.3627 (3)   | 0.8714 (6) | 0.79227 (9) | 0.0866 (8)                       |
| H6'1 | 0.2985       | 0.9674     | 0.8009      | 0.130*                           |
| H6'2 | 0.3337       | 0.7826     | 0.7610      | 0.130*                           |
| H6'3 | 0.4271       | 0.9780     | 0.7876      | 0.130*                           |

## supplementary materials

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|      |              |            |              |             |
|------|--------------|------------|--------------|-------------|
| C7'  | 0.5109 (3)   | 0.5377 (6) | 0.82529 (12) | 0.0928 (9)  |
| H7'1 | 0.4860       | 0.4458     | 0.7941       | 0.139*      |
| H7'2 | 0.5349       | 0.4266     | 0.8536       | 0.139*      |
| H7'3 | 0.5776       | 0.6413     | 0.8221       | 0.139*      |
| C2'  | 0.22244 (16) | 0.4919 (4) | 0.91839 (8)  | 0.0553 (5)  |
| H2'1 | 0.1510       | 0.4965     | 0.8913       | 0.066*      |
| H2'2 | 0.2168       | 0.3528     | 0.9406       | 0.066*      |
| O1   | 0.09613 (11) | 0.3663 (3) | 0.62016 (5)  | 0.0557 (4)  |
| O2   | 0.21761 (13) | 0.6589 (3) | 0.66182 (5)  | 0.0666 (5)  |
| O3   | 0.36792 (14) | 0.3733 (4) | 0.52993 (7)  | 0.0861 (6)  |
| N1   | 0.29356 (13) | 0.4731 (4) | 0.55436 (6)  | 0.0544 (4)  |
| C1   | 0.10961 (15) | 0.5348 (4) | 0.58059 (7)  | 0.0514 (5)  |
| H1   | 0.0341       | 0.6186     | 0.5659       | 0.062*      |
| C2   | 0.16439 (16) | 0.4088 (4) | 0.54033 (7)  | 0.0556 (5)  |
| H2A  | 0.1530       | 0.2325     | 0.5413       | 0.067*      |
| H2B  | 0.1293       | 0.4687     | 0.5062       | 0.067*      |
| C3   | 0.31507 (17) | 0.6385 (4) | 0.58935 (8)  | 0.0586 (5)  |
| H3   | 0.3910       | 0.7049     | 0.6014       | 0.070*      |
| C4   | 0.20620 (17) | 0.7127 (4) | 0.60855 (7)  | 0.0522 (5)  |
| H4   | 0.1847       | 0.8848     | 0.6007       | 0.063*      |
| C5   | 0.12362 (18) | 0.4967 (4) | 0.66756 (7)  | 0.0558 (5)  |
| C6   | 0.0151 (3)   | 0.6365 (7) | 0.67681 (11) | 0.0971 (10) |
| H6A  | -0.0464      | 0.5228     | 0.6816       | 0.146*      |
| H6B  | -0.0148      | 0.7394     | 0.6477       | 0.146*      |
| H6C  | 0.0374       | 0.7365     | 0.7070       | 0.146*      |
| C7   | 0.1714 (3)   | 0.3142 (6) | 0.70956 (9)  | 0.0882 (8)  |
| H7A  | 0.2404       | 0.2320     | 0.7016       | 0.132*      |
| H7B  | 0.1101       | 0.1958     | 0.7120       | 0.132*      |
| H7C  | 0.1942       | 0.3990     | 0.7418       | 0.132*      |

### Atomic displacement parameters ( $\text{\AA}^2$ )

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| O1' | 0.0642 (8)  | 0.0626 (10) | 0.0440 (7)  | -0.0248 (7)  | 0.0098 (6)  | -0.0060 (6)  |
| O2' | 0.0573 (7)  | 0.0637 (9)  | 0.0517 (7)  | -0.0225 (7)  | 0.0178 (6)  | -0.0098 (7)  |
| O3' | 0.0556 (7)  | 0.0876 (12) | 0.0713 (9)  | 0.0236 (8)   | 0.0283 (7)  | 0.0071 (9)   |
| N1' | 0.0431 (7)  | 0.0554 (10) | 0.0467 (8)  | 0.0091 (7)   | 0.0108 (6)  | 0.0067 (7)   |
| C1' | 0.0541 (9)  | 0.0433 (10) | 0.0471 (9)  | 0.0010 (9)   | 0.0142 (8)  | 0.0032 (8)   |
| C3' | 0.0484 (9)  | 0.0476 (10) | 0.0420 (9)  | -0.0002 (8)  | 0.0061 (7)  | 0.0000 (8)   |
| C4' | 0.0374 (8)  | 0.0565 (12) | 0.0434 (9)  | 0.0022 (8)   | 0.0042 (7)  | 0.0000 (9)   |
| C5' | 0.0612 (11) | 0.0527 (12) | 0.0467 (10) | -0.0157 (9)  | 0.0176 (8)  | -0.0056 (9)  |
| C6' | 0.115 (2)   | 0.0827 (19) | 0.0616 (13) | -0.0206 (17) | 0.0163 (13) | 0.0152 (14)  |
| C7' | 0.1036 (19) | 0.084 (2)   | 0.108 (2)   | 0.0040 (17)  | 0.0634 (17) | -0.0118 (18) |
| C2' | 0.0509 (10) | 0.0578 (12) | 0.0581 (11) | -0.0114 (10) | 0.0127 (8)  | -0.0032 (10) |
| O1  | 0.0564 (7)  | 0.0588 (9)  | 0.0530 (7)  | -0.0180 (7)  | 0.0137 (6)  | -0.0052 (7)  |
| O2  | 0.0758 (9)  | 0.0742 (12) | 0.0499 (8)  | -0.0306 (9)  | 0.0126 (6)  | -0.0074 (8)  |
| O3  | 0.0679 (9)  | 0.1030 (15) | 0.0940 (11) | 0.0252 (10)  | 0.0316 (8)  | -0.0034 (12) |
| N1  | 0.0472 (8)  | 0.0598 (10) | 0.0560 (9)  | 0.0068 (8)   | 0.0098 (7)  | 0.0045 (9)   |

|    |             |             |             |              |             |              |
|----|-------------|-------------|-------------|--------------|-------------|--------------|
| C1 | 0.0395 (8)  | 0.0617 (14) | 0.0502 (10) | 0.0022 (9)   | 0.0022 (7)  | 0.0044 (9)   |
| C2 | 0.0511 (10) | 0.0646 (14) | 0.0488 (10) | -0.0048 (9)  | 0.0045 (8)  | -0.0025 (10) |
| C3 | 0.0465 (9)  | 0.0662 (14) | 0.0612 (11) | -0.0129 (9)  | 0.0061 (8)  | 0.0058 (11)  |
| C4 | 0.0600 (11) | 0.0397 (10) | 0.0572 (11) | -0.0048 (9)  | 0.0127 (9)  | 0.0008 (9)   |
| C5 | 0.0627 (11) | 0.0549 (12) | 0.0523 (10) | -0.0128 (10) | 0.0179 (8)  | -0.0059 (10) |
| C6 | 0.0920 (19) | 0.112 (3)   | 0.098 (2)   | 0.0096 (19)  | 0.0441 (16) | -0.018 (2)   |
| C7 | 0.127 (2)   | 0.0758 (18) | 0.0603 (13) | -0.0100 (18) | 0.0159 (13) | 0.0090 (14)  |

*Geometric parameters (Å, °)*

|             |             |           |             |
|-------------|-------------|-----------|-------------|
| O1'—C1'     | 1.417 (2)   | O1—C5     | 1.423 (2)   |
| O1'—C5'     | 1.426 (2)   | O1—C1     | 1.425 (2)   |
| O2'—C5'     | 1.416 (2)   | O2—C5     | 1.415 (2)   |
| O2'—C4'     | 1.431 (2)   | O2—C4     | 1.423 (2)   |
| O3'—N1'     | 1.2937 (19) | O3—N1     | 1.281 (2)   |
| N1'—C3'     | 1.284 (2)   | N1—C3     | 1.282 (3)   |
| N1'—C2'     | 1.470 (3)   | N1—C2     | 1.480 (2)   |
| C1'—C2'     | 1.510 (2)   | C1—C2     | 1.502 (3)   |
| C1'—C4'     | 1.535 (3)   | C1—C4     | 1.538 (3)   |
| C1'—H1'     | 0.9800      | C1—H1     | 0.9800      |
| C3'—C4'     | 1.484 (3)   | C2—H2A    | 0.9700      |
| C3'—H3'     | 0.9300      | C2—H2B    | 0.9700      |
| C4'—H4'     | 0.9800      | C3—C4     | 1.481 (3)   |
| C5'—C6'     | 1.497 (3)   | C3—H3     | 0.9300      |
| C5'—C7'     | 1.509 (4)   | C4—H4     | 0.9800      |
| C6'—H6'1    | 0.9600      | C5—C6     | 1.507 (3)   |
| C6'—H6'2    | 0.9600      | C5—C7     | 1.511 (4)   |
| C6'—H6'3    | 0.9600      | C6—H6A    | 0.9600      |
| C7'—H7'1    | 0.9600      | C6—H6B    | 0.9600      |
| C7'—H7'2    | 0.9600      | C6—H6C    | 0.9600      |
| C7'—H7'3    | 0.9600      | C7—H7A    | 0.9600      |
| C2'—H2'1    | 0.9700      | C7—H7B    | 0.9600      |
| C2'—H2'2    | 0.9700      | C7—H7C    | 0.9600      |
| C1'—O1'—C5' | 107.35 (14) | C5—O1—C1  | 106.97 (16) |
| C5'—O2'—C4' | 107.96 (15) | C5—O2—C4  | 108.22 (15) |
| C3'—N1'—O3' | 127.85 (19) | O3—N1—C3  | 127.89 (18) |
| C3'—N1'—C2' | 113.34 (15) | O3—N1—C2  | 119.33 (19) |
| O3'—N1'—C2' | 118.74 (16) | C3—N1—C2  | 112.67 (16) |
| O1'—C1'—C2' | 110.46 (15) | O1—C1—C2  | 110.37 (18) |
| O1'—C1'—C4' | 103.80 (15) | O1—C1—C4  | 102.75 (14) |
| C2'—C1'—C4' | 105.50 (15) | C2—C1—C4  | 105.98 (15) |
| O1'—C1'—H1' | 112.2       | O1—C1—H1  | 112.4       |
| C2'—C1'—H1' | 112.2       | C2—C1—H1  | 112.4       |
| C4'—C1'—H1' | 112.2       | C4—C1—H1  | 112.4       |
| N1'—C3'—C4' | 111.98 (18) | N1—C2—C1  | 103.94 (16) |
| N1'—C3'—H3' | 124.0       | N1—C2—H2A | 111.0       |
| C4'—C3'—H3' | 124.0       | C1—C2—H2A | 111.0       |
| O2'—C4'—C3' | 110.31 (17) | N1—C2—H2B | 111.0       |
| O2'—C4'—C1' | 104.86 (13) | C1—C2—H2B | 111.0       |



## supplementary materials

|               |             |            |             |
|---------------|-------------|------------|-------------|
| C3'—C4'—C1'   | 103.90 (14) | H2A—C2—H2B | 109.0       |
| O2'—C4'—H4'   | 112.4       | N1—C3—C4   | 112.86 (17) |
| C3'—C4'—H4'   | 112.4       | N1—C3—H3   | 123.6       |
| C1'—C4'—H4'   | 112.4       | C4—C3—H3   | 123.6       |
| O2'—C5'—O1'   | 104.48 (13) | O2—C4—C3   | 111.49 (17) |
| O2'—C5'—C6'   | 108.5 (2)   | O2—C4—C1   | 105.37 (15) |
| O1'—C5'—C6'   | 109.05 (19) | C3—C4—C1   | 102.97 (17) |
| O2'—C5'—C7'   | 109.8 (2)   | O2—C4—H4   | 112.2       |
| O1'—C5'—C7'   | 111.2 (2)   | C3—C4—H4   | 112.2       |
| C6'—C5'—C7'   | 113.4 (2)   | C1—C4—H4   | 112.2       |
| C5'—C6'—H6'1  | 109.5       | O2—C5—O1   | 104.75 (13) |
| C5'—C6'—H6'2  | 109.5       | O2—C5—C6   | 111.0 (2)   |
| H6'1—C6'—H6'2 | 109.5       | O1—C5—C6   | 110.64 (19) |
| C5'—C6'—H6'3  | 109.5       | O2—C5—C7   | 108.8 (2)   |
| H6'1—C6'—H6'3 | 109.5       | O1—C5—C7   | 107.8 (2)   |
| H6'2—C6'—H6'3 | 109.5       | C6—C5—C7   | 113.4 (2)   |
| C5'—C7'—H7'1  | 109.5       | C5—C6—H6A  | 109.5       |
| C5'—C7'—H7'2  | 109.5       | C5—C6—H6B  | 109.5       |
| H7'1—C7'—H7'2 | 109.5       | H6A—C6—H6B | 109.5       |
| C5'—C7'—H7'3  | 109.5       | C5—C6—H6C  | 109.5       |
| H7'1—C7'—H7'3 | 109.5       | H6A—C6—H6C | 109.5       |
| H7'2—C7'—H7'3 | 109.5       | H6B—C6—H6C | 109.5       |
| N1'—C2'—C1'   | 104.30 (16) | C5—C7—H7A  | 109.5       |
| N1'—C2'—H2'1  | 110.9       | C5—C7—H7B  | 109.5       |
| C1'—C2'—H2'1  | 110.9       | H7A—C7—H7B | 109.5       |
| N1'—C2'—H2'2  | 110.9       | C5—C7—H7C  | 109.5       |
| C1'—C2'—H2'2  | 110.9       | H7A—C7—H7C | 109.5       |
| H2'1—C2'—H2'2 | 108.9       | H7B—C7—H7C | 109.5       |

### 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> |
|-------------------------------|-------------|---------------|-----------------------|-------------------------|
| C3—H3...O1 <sup>i</sup>       | 0.93        | 2.44          | 3.366 (3)             | 171                     |
| C1—H1...O3 <sup>ii</sup>      | 0.98        | 2.38          | 3.355 (3)             | 171                     |
| C2—H2A...O3 <sup>iii</sup>    | 0.97        | 2.70          | 3.441 (3)             | 134                     |
| C2—H2B...O3 <sup>iv</sup>     | 0.97        | 2.41          | 3.120 (3)             | 130                     |
| C2'—H2'1...O2' <sup>v</sup>   | 0.97        | 2.49          | 3.247 (2)             | 135                     |
| C4'—H4'...O3' <sup>vi</sup>   | 0.98        | 2.48          | 3.375 (3)             | 152                     |
| C2'—H2'2...O3' <sup>vii</sup> | 0.97        | 2.61          | 3.254 (2)             | 124                     |
| C3'—H3'...O3' <sup>viii</sup> | 0.93        | 2.48          | 3.345 (3)             | 156                     |

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

Fig. 1

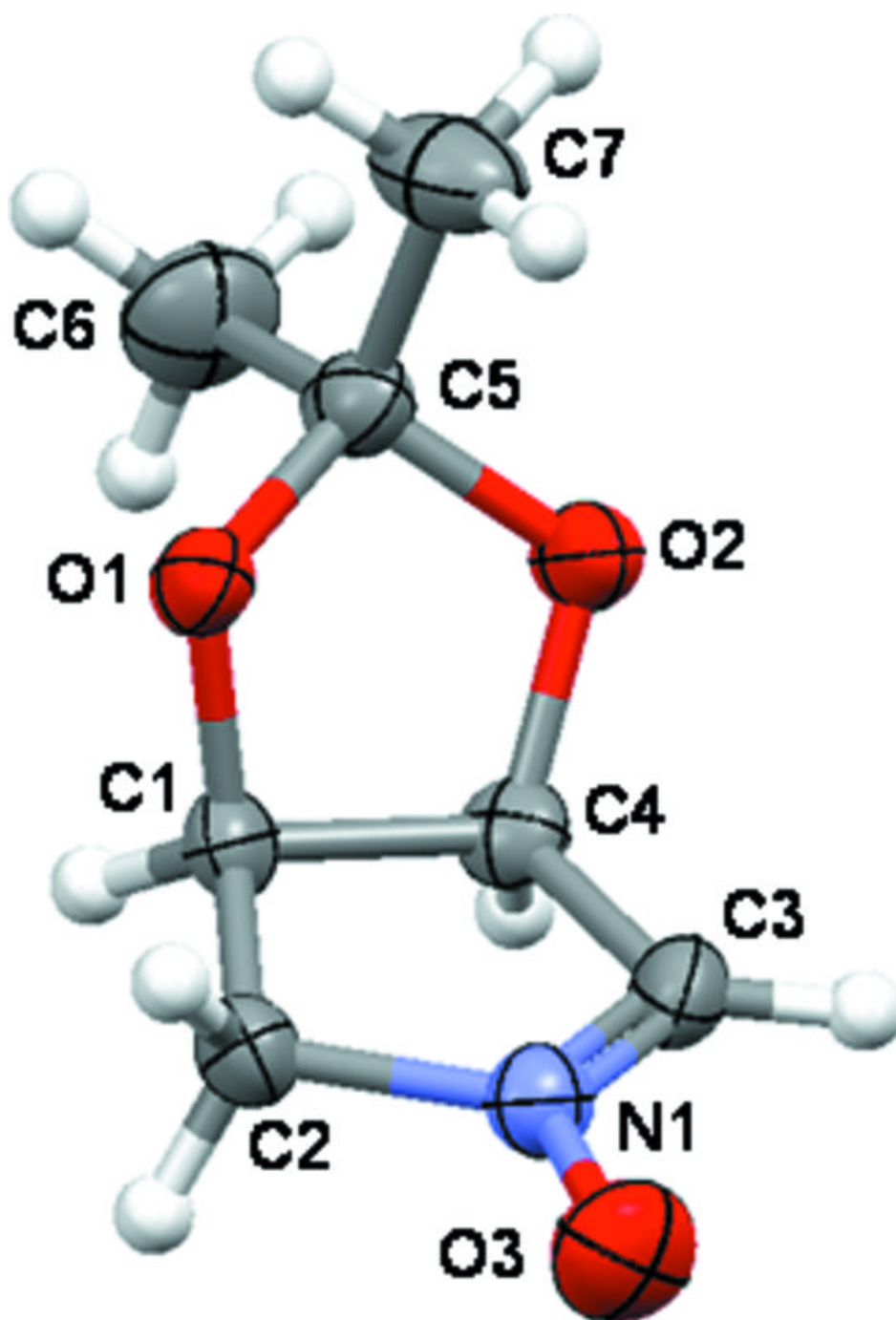


Fig. 2

