# research communications

![CrossMark]

# Crystal structure of 3-deoxy-3-nitromethyl-1,2;5,6-di-O-isopropylidene-α-D-allofuranose
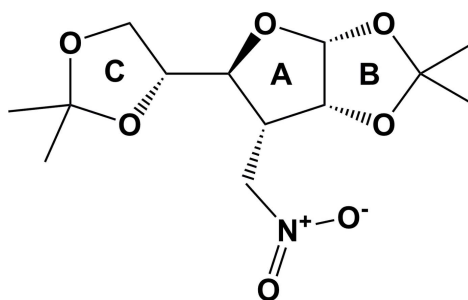
Jevgeņija Lugiņina,[a] Vitālijs Rjabovs[a]* and Dmitrijs Stepanovs[b]*

[a]Institute of Technology of Organic Chemistry, Faculty of Materials Science and, Applied Chemistry, Riga Technical University, P. Valdena 3/7, Riga, LV-1048, Latvia, and [b]Latvian Institute of Organic Synthesis, Str. Aizkraukles 21, Riga, LV 1006, Latvia. *Correspondence e-mail: vitalijs.rjabovs@rtu.lv, d_stepanovs@osi.lv

The title compound, $C_{13}H_{21}NO_7$ {systematic name: (3a$R$,5$S$,6$R$,6a$R$)-5-[($R$)-2,2-dimethyl-1,3-dioxolan-4-yl]-2,2-dimethyl-6-(nitromethyl)tetrahydrofuro[2,3-$d$]-[1,3]dioxole}, consists of a substituted 2,2-dimethyltetrahydrofuro[2,3-$d$][1,3]-dioxolane skeleton. The furanose ring $A$ adopts a $^oT_4$ conformation. The fused dioxolane ring $B$ and the substituent dioxolane ring $C$ also have twisted conformations. There are no strong hydrogen bonds in the crystal structure: only weak C—H···O contacts are present, which link the molecules to form a three-dimensional structure.
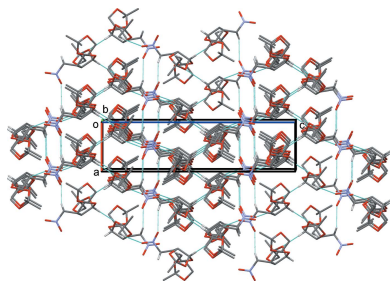
## 1. Chemical context

The title compound **1**, has been used for the syntheses of isoxazoles (Lugiņina *et al.*, 2013) and carbohydrate-based amines and amino acids (Rjabovs *et al.*, 2015*a*). Carbohydrates with amino groups are valuable synthetic precursors and are easily converted to spirocyclic carbohydrate derivatives (Turks *et al.*, 2013), imino sugars (Filichev & Pedersen, 2001), nucleic acid mimetics (Rozners *et al.*, 2003), and azido sugars (Mackeviča *et al.*, 2014; Rjabova *et al.*, 2012). The latter are widely used for the syntheses of triazoles (Uzuleņa *et al.*, 2015; Grigorjeva *et al.*, 2015) and THF-amino acids (sugar amino acids) (Rjabovs & Turks, 2013).



## 2. Structural commentary

The title compound **1**, consists of a tetrahydrofuran core (ring $A$) fused with a dioxolane ring ($B$) and substituted with a dioxolane (ring $C$) and a nitromethyl group (Fig. 1). The conformational analysis of the furanose ring ($A$) based on the internal dihedral angles of the ring shows that its pseudo-rotational phase angle $P = 70°$ (Altona & Sundaralingam, 1972; Taha *et al.*, 2013). Thus, this ring adopts a conformation close to $^oT_4$, where O1 and C4 deviate by 0.214 (2) and −0.340 (3) Å, respectively, from the plane through atoms
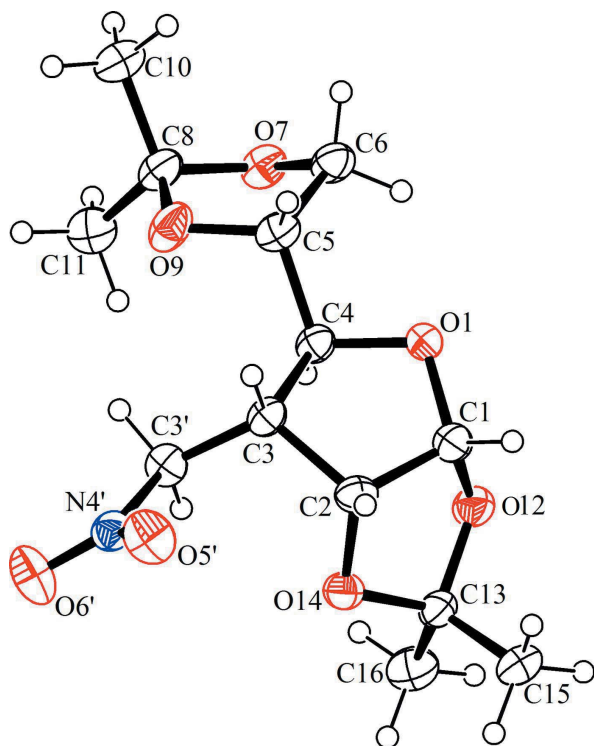
**Figure 1**
The molecular structure of the title compound **1**, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level.

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

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|---|---|---|---|---|
| C3′—H3′2···O5′[i] | 0.97 | 2.53 | 3.355 (4) | 143 |
| C1—H1···O12[ii] | 0.98 | 2.41 | 3.386 (5) | 178 |
| C15—H15A···O6′[iii] | 0.96 | 2.48 | 3.433 (5) | 174 |

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

dimethyl-1,3-dioxolan-4-yl group, ring *C*, also adopts a twisted conformation, on bond C6—O7; these atoms deviate by 0.143 (4) and −0.381 (2) Å, respectively from plane C5/O9/C8.

## 3. Supramolecular features

In the crystal, molecules are linked *via* C—H···O hydrogen bonds, forming chains along [100]. The chains are linked *via* further C—H···O hydrogen bonds, forming a three-dimensional structure (Table 1 and Fig. 2).

## 4. Database survey

A search of the Cambridge Structural Database (Version 5.37; Groom & Allen, 2014) for substructure S1 (Fig. 3) gave 137 hits, while a search for substructure S2 (Fig. 3) gave only five hits. Amongst the latter compounds, four concern the structures with a hydroxyl and a nitromethyl group attached to atom C3 (BOGFOU: Turks *et al.*, 2014; BOGFUA: Turks *et al.*, 2014; CIDVO: Turks *et al.*, 2013; USODEM: Zhang *et al.*, 2011). They are diastereomers crystallizing in space groups $P2_12_12_1$, $P3_2$, $C2$ and $P6_1$, respectively. In the fifth compound (KATWIN; Lugiņina *et al.*, 2012), the extra substituent at atom C3 is a methylthio group; it crystallizes in space group $C2$.

C1/C2/C3. Such a conformation of the furanose ring is rather unusual for 3-*C*-monosubstituted 3-deoxy-1,2-*O*-isopropylidene-α-D-allofuranoses. For example, previously reported structures (Rjabovs *et al.*, 2014, 2015*a,b*) had conformations between $^3E$ and $^3T_4$. The fused dioxolane ring *B* also adopts a twisted conformation on bond C13—O12; these atoms deviate by −0.324 (4) and 0.224 (3) Å, respectively, from plane C1/C2/O14. The dihedral angle subtended by the mean planes of rings *A* and *B* is 63.7 (2)°. The five-membered ring of the 2,2-
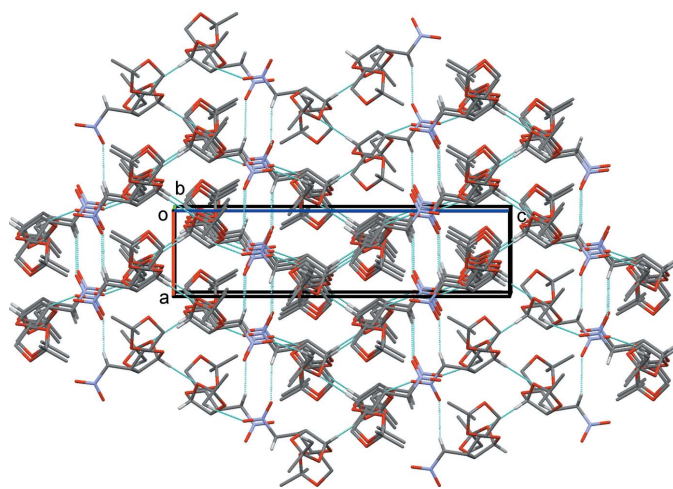


**Figure 2**
A view along the *b* axis of the crystal packing of the title compound **1**. Hydrogen bonds are shown as dashed lines (see Table 1) and H atoms not involved in these interactions have been omitted for clarity.
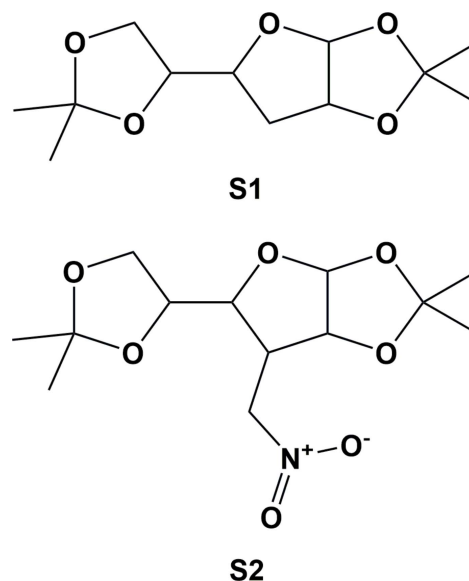


**Figure 3**
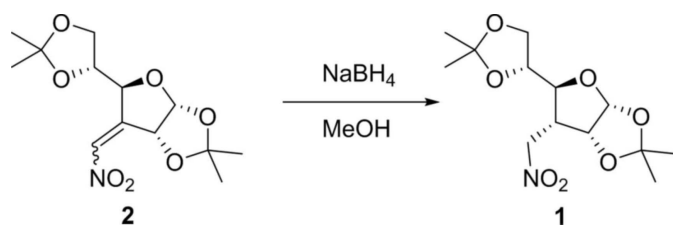Substructures used for the database survey.

**Figure 4**
Synthesis of the title compound.

## 5. Synthesis and crystallization

The title compound **1**, was synthesized by reduction of the nitro olefin **2** (Albrecht & Moffatt, 1970; Filichev *et al.*, 2001; Lugiņina *et al.*, 2012) with sodium borohydride in methanol solution, as illustrated in Fig. 4. NaBH$_4$ (6.2 g, 163.9 mmol, 5.5 eq.) was added portion wise to a solution of **2** (9.1 g, 30.0 mmol, 1.0 eq.) in MeOH (200 ml) over 30 min at 273 K. After completion (monitored by TLC) the reaction mixture was acidified using 10% aqueous solution of AcOH to pH 6–7 and then evaporated to dryness. The residue was dissolved in EtOAc (90 ml), washed with brine (3 × 10 ml), dried over NaSO$_4$, and evaporated. The product **1** was purified by column chromatography on silica gel (Hexanes/EtOAc 3:1 → 2:1) giving a white crystalline solid (yield: 6.5 g, 72%; m.p. 355–356 K). $R_f$ = 0.9 (hexanes/EtOAc 1:1). $^1$H NMR (CDCl$_3$, 300 MHz): δ 5.84 (d, J = 3.7 Hz, 1H), 4.88–4.82 (m, 21H), 4.68 (dd, AB syst., J = 14.9 Hz, J = 10.4 Hz, 1H), 4.14 (dd, J = 8.0 Hz, J = 5.5 Hz, 1H), 4.02–3.92 (m, 2H), 3.65 (dd, J = 9.9 Hz, J = 8.4 Hz, 1H), 2.74 (tt, J = 10.1 Hz, J = 4.4 Hz, 1H), 1.52 (s, 3H), 1.40, (s, 3H), 1.33 (s, 3H), 1.32 (s, 3H). $^{13}$C-NMR (75 MHz, CDCl$_3$): δ 112.6, 110.1, 105.4, 80.5, 79.0, 77.8, 70.8, 68.2, 46.6, 26.8, 26.7, 26.4, 25.1. X-ray quality single crystals were obtained by slow evaporation of a dichloromethane solution at ambient temperature.

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The H atoms were included in calculated positions and refined as riding atoms: C—H = 0.96–0.98 Å with $U_{iso}$(H) = 1.5$U_{eq}$(C-methyl) and 1.2$U_{eq}$(C) for other H atoms. The absolute configuration is based on that of the starting material.

## Acknowledgements

## References

Albrecht, H. P. & Moffatt, J. G. (1970). *Tetrahedron Lett.* **11**, 1063–1066.
Altona, C. & Sundaralingam, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.

**Table 2**
Experimental details.

| Crystal data | |
|---|---|
| Chemical formula | C$_{13}$H$_{21}$NO$_7$ |
| $M_r$ | 303.31 |
| Crystal system, space group | Orthorhombic, $P2_12_12_1$ |
| Temperature (K) | 173 |
| $a, b, c$ (Å) | 5.5044 (2), 12.6144 (4), 21.6348 (9) |
| $V$ (Å$^3$) | 1502.21 (10) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.11 |
| Crystal size (mm) | 0.26 × 0.08 × 0.06 |
| | |
| Data collection | |
| Diffractometer | Nonius KappaCCD |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 4225, 4225, 2316 |
| $(\sin \theta/\lambda)_{max}$ (Å$^{-1}$) | 0.705 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.065, 0.127, 1.01 |
| No. of reflections | 4225 |
| No. of parameters | 194 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.20, −0.26 |

Computer programs: *KappaCCD Server Software* (Nonius, 1997), *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997), *SIR2011* (Burla *et al.*, 2012), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

Burla, M. C., Caliandro, R., Camalli, M., Carrozzini, B., Cascarano, G. L., Giacovazzo, C., Mallamo, M., Mazzone, A., Polidori, G. & Spagna, R. (2012). *J. Appl. Cryst.* **45**, 357–361.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Filichev, V. V., Brandt, M. & Pedersen, E. B. (2001). *Carbohydr. Res.* **333**, 115–122.
Filichev, V. V. & Pedersen, E. B. (2001). *Tetrahedron*, **57**, 9163–9168.
Grigorjeva, J., Uzuleņa, J., Rjabovs, V. & Turks, M. (2015). *Chem. Heterocycl. Compd.* **51**, 883–890.
Groom, C. R. & Allen, F. H. (2014). *Angew. Chem. Int. Ed.* **53**, 662–671.
Lugiņina, J., Rjabovs, V., Belyakov, S. & Turks, M. (2012). *Carbohydr. Res.* **350**, 86–89.
Luginina, J., Rjabovs, V., Belyakov, S. & Turks, M. (2013). *Tetrahedron Lett.* **54**, 5328–5331.
Mackeviča, J., Ostrovskis, P., Leffler, H., Nilsson, U. J., Rudovica, V., Viksna, A., Belyakov, S. & Turks, M. (2014). *ARKIVOC*, (**iii**), 90–112.
Nonius (1997). *KappaCCD Server Software*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Rjabova, J., Rjabovs, V., Moreno Vargas, A. J., Clavijo, E. M. & Turks, M. (2012). *Cent. Eur. J. Chem.* **10**, 386–394.
Rjabovs, V., Mishnev, A., Kiselovs, G. & Turks, M. (2014). *Acta Cryst.* E**70**, o524–o525.
Rjabovs, V., Ostrovskis, P., Posevins, D., Kiselovs, G., Kumpiņš, V., Mishnev, A. & Turks, M. (2015a). *Eur. J. Org. Chem.* pp. 5572–5584.
Rjabovs, V., Stepanovs, D. & Turks, M. (2015b). *Acta Cryst.* E**71**, 1212–1215.
Rjabovs, V. & Turks, M. (2013). *Tetrahedron*, **69**, 10693–10710.
Rozners, E., Katkevica, D., Bizdena, E. & Strömberg, R. (2003). *J. Am. Chem. Soc.* **125**, 12125–12136.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Taha, H. A., Richards, M. R. & Lowary, T. L. (2013). *Chem. Rev.* **113**, 1851–1876.

Turks, M., Rodins, V., Rolava, E., Ostrovskis, P. & Belyakov, S. (2013). *Carbohydr. Res.* **375**, 5–15.

Turks, M., Vēze, K., Kiselovs, G., Mackeviča, J., Lugiņina, J., Mishnev, A. & Marković, D. (2014). *Carbohydr. Res.* **391**, 82–88.

Uzuleņa, J., Rjabovs, V., Moreno-Vargas, A. J. & Turks, M. (2015). *Chem. Heterocycl. Compd*, **51**, 664–671.

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

Zhang, Q., Ke, Y., Cheng, W., Li, P. & Liu, H. (2011). *Acta Cryst.* E**67**, o1402.

# supporting information

## Crystal structure of 3-deoxy-3-nitromethyl-1,2;5,6-di-*O*-isopropylidene-*α*-D-allofuranose

**Jevgeņija Lugiņina, Vitālijs Rjabovs and Dmitrijs Stepanovs**

**Computing details**

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR2011* (Burla *et al.*, 2012); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

**(3a*R*,5*S*,6*R*,6a*R*)-5-[(*R*)-2,2-Dimethyl-1,3-\ dioxolan-4-yl]-2,2-dimethyl-6-(nitromethyl)tetrahydrofuro[2,3-*d*] [1,3]\ dioxole**

*Crystal data*

| | |
|---|---|
| $C_{13}H_{21}NO_7$ | $D_x = 1.341$ Mg m$^{-3}$ |
| $M_r = 303.31$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Orthorhombic, $P2_12_12_1$ | Cell parameters from 26214 reflections |
| $a = 5.5044$ (2) Å | $\theta = 1.0$–30.0° |
| $b = 12.6144$ (4) Å | $\mu = 0.11$ mm$^{-1}$ |
| $c = 21.6348$ (9) Å | $T = 173$ K |
| $V = 1502.21$ (10) Å$^3$ | Needle, colourless |
| $Z = 4$ | $0.26 \times 0.08 \times 0.06$ mm |
| $F(000) = 648$ | |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 2316 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $\theta_{max} = 30.1°$, $\theta_{min} = 3.2°$ |
| $\varphi$ and $\omega$ scan | $h = -7 \rightarrow 7$ |
| 4225 measured reflections | $k = -17 \rightarrow 17$ |
| 4225 independent reflections | $l = -30 \rightarrow 30$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.065$ | H-atom parameters constrained |
| $wR(F^2) = 0.127$ | $w = 1/[\sigma^2(F_o^2) + (0.0367P)^2 + 0.3523P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.01$ | |
| 4225 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 194 parameters | $\Delta\rho_{max} = 0.20$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.25$ e Å$^{-3}$ |

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.1756 (4) | 0.94348 (18) | 0.03395 (11) | 0.0372 (6) |
| C1 | 0.2580 (7) | 0.8418 (3) | 0.04912 (17) | 0.0368 (9) |
| H1 | 0.3478 | 0.8095 | 0.0148 | 0.044* |
| C2 | 0.4157 (6) | 0.8508 (3) | 0.10747 (17) | 0.0339 (8) |
| H2 | 0.5873 | 0.8361 | 0.0990 | 0.041* |
| C3 | 0.3744 (6) | 0.9665 (2) | 0.12881 (17) | 0.0306 (8) |
| H3 | 0.5155 | 1.0090 | 0.1166 | 0.037* |
| C4 | 0.1536 (6) | 1.0022 (2) | 0.09017 (16) | 0.0309 (8) |
| H4 | 0.0025 | 0.9832 | 0.1114 | 0.037* |
| C5 | 0.1502 (6) | 1.1182 (3) | 0.07239 (18) | 0.0360 (9) |
| H5 | 0.3020 | 1.1371 | 0.0514 | 0.043* |
| C6 | −0.0661 (6) | 1.1468 (3) | 0.03179 (19) | 0.0392 (9) |
| H6A | −0.0244 | 1.2023 | 0.0026 | 0.047* |
| H6B | −0.1248 | 1.0854 | 0.0093 | 0.047* |
| O7 | −0.2414 (4) | 1.18276 (17) | 0.07540 (12) | 0.0391 (6) |
| C8 | −0.1090 (6) | 1.2369 (3) | 0.12245 (19) | 0.0393 (9) |
| O9 | 0.1197 (4) | 1.18174 (19) | 0.12615 (13) | 0.0470 (7) |
| C10 | −0.0637 (8) | 1.3514 (3) | 0.1055 (2) | 0.0489 (11) |
| H10A | −0.2159 | 1.3882 | 0.1027 | 0.073* |
| H10B | 0.0184 | 1.3548 | 0.0664 | 0.073* |
| H10C | 0.0354 | 1.3840 | 0.1367 | 0.073* |
| C11 | −0.2428 (8) | 1.2243 (3) | 0.18262 (19) | 0.0554 (11) |
| H11A | −0.2530 | 1.1505 | 0.1930 | 0.083* |
| H11B | −0.4035 | 1.2530 | 0.1786 | 0.083* |
| H11C | −0.1572 | 1.2614 | 0.2147 | 0.083* |
| O12 | 0.0607 (5) | 0.77711 (19) | 0.06819 (12) | 0.0397 (6) |
| C13 | 0.1517 (6) | 0.7096 (3) | 0.11560 (17) | 0.0346 (9) |
| O14 | 0.3131 (4) | 0.77660 (18) | 0.14980 (11) | 0.0362 (6) |
| C15 | 0.2887 (8) | 0.6163 (3) | 0.08712 (19) | 0.0489 (11) |
| H15A | 0.3527 | 0.5723 | 0.1194 | 0.073* |
| H15B | 0.4198 | 0.6425 | 0.0621 | 0.073* |
| H15C | 0.1798 | 0.5756 | 0.0619 | 0.073* |
| C16 | −0.0501 (7) | 0.6743 (3) | 0.1567 (2) | 0.0484 (11) |
| H16A | −0.1272 | 0.7352 | 0.1748 | 0.073* |
| H16B | 0.0138 | 0.6299 | 0.1889 | 0.073* |
| H16C | −0.1670 | 0.6352 | 0.1330 | 0.073* |
| C3′ | 0.3314 (6) | 0.9814 (3) | 0.19700 (17) | 0.0339 (8) |
| H3′1 | 0.2859 | 1.0546 | 0.2044 | 0.041* |
| H3′2 | 0.1959 | 0.9372 | 0.2095 | 0.041* |

| | | | | | |
|---|---|---|---|---|---|
| N4′ | 0.5468 (6) | 0.9550 (2) | 0.23638 (16) | 0.0373 (7) | |
| O5′ | 0.7368 (4) | 0.9282 (2) | 0.21135 (13) | 0.0518 (8) | |
| O6′ | 0.5199 (5) | 0.9616 (3) | 0.29175 (14) | 0.0612 (9) | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0535 (15) | 0.0301 (13) | 0.0281 (14) | 0.0051 (11) | −0.0038 (12) | −0.0004 (12) |
| C1 | 0.045 (2) | 0.0324 (19) | 0.033 (2) | 0.0065 (17) | 0.0013 (17) | −0.0011 (16) |
| C2 | 0.0328 (19) | 0.0309 (18) | 0.038 (2) | 0.0052 (15) | 0.0025 (17) | 0.0038 (17) |
| C3 | 0.0258 (17) | 0.0283 (19) | 0.038 (2) | −0.0020 (13) | −0.0002 (15) | −0.0018 (16) |
| C4 | 0.0273 (17) | 0.0285 (18) | 0.037 (2) | −0.0028 (14) | −0.0016 (16) | −0.0007 (17) |
| C5 | 0.0299 (18) | 0.0302 (19) | 0.048 (2) | 0.0000 (14) | −0.0025 (18) | 0.0015 (18) |
| C6 | 0.038 (2) | 0.0311 (19) | 0.048 (2) | 0.0058 (16) | −0.0062 (18) | 0.0003 (19) |
| O7 | 0.0308 (13) | 0.0349 (13) | 0.0517 (17) | −0.0016 (11) | −0.0035 (12) | −0.0014 (13) |
| C8 | 0.038 (2) | 0.0284 (19) | 0.052 (3) | 0.0024 (15) | −0.0039 (18) | −0.0024 (18) |
| O9 | 0.0418 (15) | 0.0345 (14) | 0.0646 (19) | 0.0089 (11) | −0.0173 (13) | −0.0132 (14) |
| C10 | 0.055 (2) | 0.030 (2) | 0.062 (3) | −0.0002 (18) | 0.003 (2) | 0.003 (2) |
| C11 | 0.068 (3) | 0.046 (2) | 0.052 (3) | −0.004 (2) | 0.002 (2) | 0.003 (2) |
| O12 | 0.0432 (14) | 0.0323 (13) | 0.0436 (15) | −0.0020 (11) | −0.0114 (12) | 0.0004 (13) |
| C13 | 0.040 (2) | 0.0257 (18) | 0.038 (2) | −0.0024 (15) | −0.0072 (17) | −0.0009 (16) |
| O14 | 0.0408 (14) | 0.0327 (13) | 0.0352 (15) | −0.0045 (11) | −0.0058 (12) | 0.0045 (12) |
| C15 | 0.065 (3) | 0.0281 (19) | 0.053 (3) | 0.0049 (18) | −0.004 (2) | −0.0008 (19) |
| C16 | 0.045 (2) | 0.042 (2) | 0.059 (3) | −0.0028 (18) | 0.000 (2) | 0.006 (2) |
| C3′ | 0.0245 (17) | 0.038 (2) | 0.040 (2) | −0.0017 (15) | −0.0016 (16) | −0.0010 (17) |
| N4′ | 0.0387 (18) | 0.0316 (17) | 0.042 (2) | −0.0026 (14) | −0.0078 (16) | 0.0001 (16) |
| O5′ | 0.0312 (14) | 0.0693 (19) | 0.0548 (19) | 0.0061 (14) | −0.0057 (14) | 0.0012 (15) |
| O6′ | 0.0627 (19) | 0.085 (2) | 0.0355 (18) | 0.0003 (16) | −0.0099 (16) | −0.0045 (17) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C1 | 1.399 (4) | C8—C10 | 1.511 (5) |
| O1—C4 | 1.429 (4) | C10—H10A | 0.9600 |
| C1—O12 | 1.420 (4) | C10—H10B | 0.9600 |
| C1—C2 | 1.537 (5) | C10—H10C | 0.9600 |
| C1—H1 | 0.9800 | C11—H11A | 0.9600 |
| C2—O14 | 1.427 (4) | C11—H11B | 0.9600 |
| C2—C3 | 1.547 (5) | C11—H11C | 0.9600 |
| C2—H2 | 0.9800 | O12—C13 | 1.424 (4) |
| C3—C3′ | 1.506 (5) | C13—O14 | 1.432 (4) |
| C3—C4 | 1.542 (5) | C13—C16 | 1.491 (5) |
| C3—H3 | 0.9800 | C13—C15 | 1.527 (5) |
| C4—C5 | 1.514 (4) | C15—H15A | 0.9600 |
| C4—H4 | 0.9800 | C15—H15B | 0.9600 |
| C5—O9 | 1.422 (4) | C15—H15C | 0.9600 |
| C5—C6 | 1.523 (5) | C16—H16A | 0.9600 |
| C5—H5 | 0.9800 | C16—H16B | 0.9600 |
| C6—O7 | 1.424 (4) | C16—H16C | 0.9600 |

| | | | |
|---|---|---|---|
| C6—H6A | 0.9700 | C3′—N4′ | 1.498 (4) |
| C6—H6B | 0.9700 | C3′—H3′1 | 0.9700 |
| O7—C8 | 1.426 (4) | C3′—H3′2 | 0.9700 |
| C8—O9 | 1.441 (4) | N4′—O6′ | 1.210 (4) |
| C8—C11 | 1.504 (6) | N4′—O5′ | 1.225 (4) |
| | | | |
| C1—O1—C4 | 107.6 (2) | C11—C8—C10 | 113.0 (3) |
| O1—C1—O12 | 110.3 (3) | C5—O9—C8 | 109.2 (3) |
| O1—C1—C2 | 107.9 (3) | C8—C10—H10A | 109.5 |
| O12—C1—C2 | 103.7 (3) | C8—C10—H10B | 109.5 |
| O1—C1—H1 | 111.5 | H10A—C10—H10B | 109.5 |
| O12—C1—H1 | 111.5 | C8—C10—H10C | 109.5 |
| C2—C1—H1 | 111.5 | H10A—C10—H10C | 109.5 |
| O14—C2—C1 | 104.8 (3) | H10B—C10—H10C | 109.5 |
| O14—C2—C3 | 111.7 (3) | C8—C11—H11A | 109.5 |
| C1—C2—C3 | 103.4 (3) | C8—C11—H11B | 109.5 |
| O14—C2—H2 | 112.1 | H11A—C11—H11B | 109.5 |
| C1—C2—H2 | 112.1 | C8—C11—H11C | 109.5 |
| C3—C2—H2 | 112.1 | H11A—C11—H11C | 109.5 |
| C3′—C3—C4 | 111.8 (3) | H11B—C11—H11C | 109.5 |
| C3′—C3—C2 | 115.7 (3) | C1—O12—C13 | 106.5 (3) |
| C4—C3—C2 | 103.3 (3) | O12—C13—O14 | 103.8 (2) |
| C3′—C3—H3 | 108.6 | O12—C13—C16 | 110.2 (3) |
| C4—C3—H3 | 108.6 | O14—C13—C16 | 109.3 (3) |
| C2—C3—H3 | 108.6 | O12—C13—C15 | 110.1 (3) |
| O1—C4—C5 | 106.6 (3) | O14—C13—C15 | 110.9 (3) |
| O1—C4—C3 | 104.1 (3) | C16—C13—C15 | 112.2 (3) |
| C5—C4—C3 | 115.5 (3) | C2—O14—C13 | 107.5 (2) |
| O1—C4—H4 | 110.1 | C13—C15—H15A | 109.5 |
| C5—C4—H4 | 110.1 | C13—C15—H15B | 109.5 |
| C3—C4—H4 | 110.1 | H15A—C15—H15B | 109.5 |
| O9—C5—C4 | 109.8 (3) | C13—C15—H15C | 109.5 |
| O9—C5—C6 | 104.2 (3) | H15A—C15—H15C | 109.5 |
| C4—C5—C6 | 112.7 (3) | H15B—C15—H15C | 109.5 |
| O9—C5—H5 | 110.0 | C13—C16—H16A | 109.5 |
| C4—C5—H5 | 110.0 | C13—C16—H16B | 109.5 |
| C6—C5—H5 | 110.0 | H16A—C16—H16B | 109.5 |
| O7—C6—C5 | 102.9 (3) | C13—C16—H16C | 109.5 |
| O7—C6—H6A | 111.2 | H16A—C16—H16C | 109.5 |
| C5—C6—H6A | 111.2 | H16B—C16—H16C | 109.5 |
| O7—C6—H6B | 111.2 | N4′—C3′—C3 | 113.9 (3) |
| C5—C6—H6B | 111.2 | N4′—C3′—H3′1 | 108.8 |
| H6A—C6—H6B | 109.1 | C3—C3′—H3′1 | 108.8 |
| C6—O7—C8 | 106.2 (2) | N4′—C3′—H3′2 | 108.8 |
| O7—C8—O9 | 104.8 (3) | C3—C3′—H3′2 | 108.8 |
| O7—C8—C11 | 108.5 (3) | H3′1—C3′—H3′2 | 107.7 |
| O9—C8—C11 | 109.2 (3) | O6′—N4′—O5′ | 124.1 (3) |
| O7—C8—C10 | 111.7 (3) | O6′—N4′—C3′ | 116.8 (3) |

| | | | |
|---|---|---|---|
| O9—C8—C10 | 109.3 (3) | O5′—N4′—C3′ | 119.1 (3) |
| | | | |
| C4—O1—C1—O12 | 82.3 (3) | C6—O7—C8—O9 | −32.8 (3) |
| C4—O1—C1—C2 | −30.3 (3) | C6—O7—C8—C11 | −149.3 (3) |
| O1—C1—C2—O14 | 126.3 (3) | C6—O7—C8—C10 | 85.4 (3) |
| O12—C1—C2—O14 | 9.3 (3) | C4—C5—O9—C8 | −115.4 (3) |
| O1—C1—C2—C3 | 9.3 (4) | C6—C5—O9—C8 | 5.6 (4) |
| O12—C1—C2—C3 | −107.7 (3) | O7—C8—O9—C5 | 16.0 (4) |
| O14—C2—C3—C3′ | 23.3 (4) | C11—C8—O9—C5 | 132.1 (3) |
| C1—C2—C3—C3′ | 135.5 (3) | C10—C8—O9—C5 | −103.8 (3) |
| O14—C2—C3—C4 | −99.1 (3) | O1—C1—O12—C13 | −144.5 (3) |
| C1—C2—C3—C4 | 13.1 (3) | C2—C1—O12—C13 | −29.2 (3) |
| C1—O1—C4—C5 | 161.0 (3) | C1—O12—C13—O14 | 38.3 (3) |
| C1—O1—C4—C3 | 38.5 (3) | C1—O12—C13—C16 | 155.2 (3) |
| C3′—C3—C4—O1 | −155.8 (3) | C1—O12—C13—C15 | −80.4 (3) |
| C2—C3—C4—O1 | −30.8 (3) | C1—C2—O14—C13 | 13.7 (3) |
| C3′—C3—C4—C5 | 87.7 (4) | C3—C2—O14—C13 | 125.0 (3) |
| C2—C3—C4—C5 | −147.3 (3) | O12—C13—O14—C2 | −31.8 (3) |
| O1—C4—C5—O9 | 177.9 (3) | C16—C13—O14—C2 | −149.4 (3) |
| C3—C4—C5—O9 | −67.0 (4) | C15—C13—O14—C2 | 86.4 (3) |
| O1—C4—C5—C6 | 62.2 (4) | C4—C3—C3′—N4′ | −176.0 (3) |
| C3—C4—C5—C6 | 177.3 (3) | C2—C3—C3′—N4′ | 66.2 (4) |
| O9—C5—C6—O7 | −25.0 (3) | C3—C3′—N4′—O6′ | −177.3 (3) |
| C4—C5—C6—O7 | 94.0 (3) | C3—C3′—N4′—O5′ | 2.6 (4) |
| C5—C6—O7—C8 | 35.7 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C3′—H3′2···O5′i | 0.97 | 2.53 | 3.355 (4) | 143 |
| C1—H1···O12ii | 0.98 | 2.41 | 3.386 (5) | 178 |
| C15—H15A···O6′iii | 0.96 | 2.48 | 3.433 (5) | 174 |

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