# organic compounds

## 4-Butyl-1-(2,3,4-tri-*O*-acetyl-*β*-L-fucopyranosyl)-1*H*-1,2,3-triazole

### Abdul-Basit Alhassan, Peter Norris and Matthias Zeller*

Department of Chemistry, Youngstown State University, 1 University Plaza, Youngstown, OH 44555-3663, USA
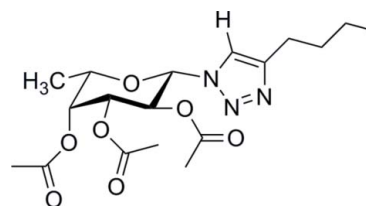Correspondence e-mail: mzeller@ysu.edu

The title compound, $C_{18}H_{27}N_3O_7$, was synthesized by $Cu^I$-catalysed coupling of an azide with an alkyne as part of a study into the synthesis of *N*-glycosyl-1,2,3-triazoles. The crystal structure confirms the selective formation of the *β*-conformer of the pyranose *N*-glycoside, thus confirming the retention of stereochemistry during heterocycle formation with the *N*-glycosyl triazole group occupying the equatorial position at the anomeric C atom. The structure exhibits two crystallographically independent molecules (*A* and *B*) with essentially identical conformations with a weighted r.m.s. deviation of only 0.09 Å. The molecules are arranged in layers with hydrophobic and more polar sections built from the butyl triazole units on the one hand and the more polar moieties dominated by the carbohydrate units on the other. Within the polar layers, intermolecular interactions are dominated by a three-dimensional network of weak C—H···O hydrogen bonds with the acetyl keto O atoms as the hydrogen-bond acceptors. The triazole units interact with each other *via* C—H···N hydrogen bonds which connect the molecules into two infinite chains of molecules made up of either *A* molecules or *B* molecules that stretch parallel to each other along [100]. Between the butyl groups no directional interactions are observed.

## Related literature

For background information on *N*-glycosidic mimics of naturally occurring carbohydrates, see: Norris (2008); Temelkoff *et al.* (2006). For details of the synthesis of the carbohydrate starting material used, see: Zhang *et al.* (2007).

## Experimental

### Crystal data

| | |
|---|---|
| $C_{18}H_{27}N_3O_7$ | $\gamma$ = 91.227 (1)° |
| $M_r$ = 397.43 | $V$ = 1020.22 (9) Å³ |
| Triclinic, $P1$ | $Z$ = 2 |
| $a$ = 5.5173 (3) Å | Mo $K\alpha$ radiation |
| $b$ = 7.7442 (4) Å | $\mu$ = 0.10 mm⁻¹ |
| $c$ = 24.1013 (13) Å | $T$ = 100 K |
| $\alpha$ = 94.507 (1)° | 0.36 × 0.35 × 0.09 mm |
| $\beta$ = 96.151 (1)° | |

### Data collection

| | |
|---|---|
| Bruker SMART APEX CCD diffractometer | 10512 measured reflections |
| Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003) | 5041 independent reflections |
| | 4839 reflections with $I > 2\sigma(I)$ |
| $T_{min}$ = 0.867, $T_{max}$ = 0.991 | $R_{int}$ = 0.020 |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)]$ = 0.046 | 3 restraints |
| $wR(F^2)$ = 0.120 | H-atom parameters constrained |
| $S$ = 1.11 | $\Delta\rho_{max}$ = 0.42 e Å⁻³ |
| 5041 reflections | $\Delta\rho_{min}$ = −0.21 e Å⁻³ |
| 515 parameters | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C1$B$—H1$B$···O3$B^i$ | 1.00 | 2.53 | 3.362 (3) | 141 |
| C2$A$—H2$A$···O3$A$ | 1.00 | 2.26 | 2.701 (3) | 105 |
| C2$B$—H2$B$···O3$B$ | 1.00 | 2.26 | 2.698 (3) | 105 |
| C3$A$—H3$A$···O3$A^{ii}$ | 1.00 | 2.32 | 3.214 (3) | 149 |
| C3$B$—H3$B$···O3$B^i$ | 1.00 | 2.27 | 3.165 (3) | 148 |
| C4$A$—H4$A$···O5$A$ | 1.00 | 2.56 | 3.046 (3) | 110 |
| C4$A$—H4$A$···O7$A$ | 1.00 | 2.23 | 2.682 (3) | 106 |
| C4$B$—H4$B$···O5$B$ | 1.00 | 2.57 | 3.067 (3) | 110 |
| C4$B$—H4$B$···O7$B$ | 1.00 | 2.21 | 2.670 (3) | 106 |
| C7$A$—H7$A$···N3$A^{ii}$ | 0.95 | 2.39 | 3.308 (4) | 161 |
| C7$B$—H7$B$···N3$B^i$ | 0.95 | 2.40 | 3.313 (4) | 161 |
| C14$A$—H14$A$···O1$A^{iii}$ | 0.98 | 2.47 | 3.377 (3) | 154 |
| C14$B$—H14$D$···O1$B^{iii}$ | 0.98 | 2.35 | 3.261 (3) | 155 |
| C16$A$—H16$A$···O7$B^{iii}$ | 0.98 | 2.41 | 3.295 (4) | 150 |
| C16$B$—H16$F$···O7$A$ | 0.98 | 2.51 | 3.401 (4) | 150 |

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

Data collection: *SMART* for WNT/2000 (Bruker, 2002); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *SHELXTL* and *publCIF* (Westrip, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FL2257).

## References

Bruker (2002). *SMART* for WNT/2000. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (2003). *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.

Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.

Norris, P. (2008). *Curr. Top. Med. Chem.* **8**, 101–113.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Temelkoff, D. P., Zeller, M. & Norris, P. (2006). *Carbohydr. Res.* **341**, 1081–1090.

Westrip, S. P. (2009). *publCIF*. In preparation.

Zhang, J., Chen, H.-N., Chiang, F.-I., Takemoto, J. Y., Bensaci, M. & Chang, C.-W. T. (2007). *J. Comb. Chem.* **9**, 17–19.

**supplementary materials**

# 4-Butyl-1-(2,3,4-tri-*O*-acetyl-*β*-L-fucopyranosyl)-1*H*-1,2,3-triazole

## A.-B. Alhassan, P. Norris and M. Zeller

## Comment

*N*-Glycosidic analogs of naturally occurring carbohydrates are receiving a growing amount of attention due to their potential in medicinal chemistry (Norris, 2008; Temelkoff *et al.*, 2006). As part of a study into the synthesis of *N*-glycosyl-1,2,3-triazoles, the title compound was found to be the only 1,2,3-triazole product formed from the reaction of 2,3,4-tri-*O*-acetyl-β-L-fucopyranosyl azide (Zhang *et al.*, 2007) with 1-hexyne and catalytic $CuSO_4$/ascorbic acid (Fig. 1).

The structure exhibits two crystallographically independent molecules A and B (Fig. 2) with essentially identical conformations as can be seen in the overlay shown in Fig. 3. The weighted r.m.s. deviation of the two molecules is only 0.09 Å. Both molecules exhibit unexceptional chair conformations for the pyranose ring and straight all-*trans* chains for the butyl chains. The crystal structure reveals the β-configuration of the pyranose *N*-glycoside (Fig. 2). This confirms the retention of stereochemistry during heterocycle formation with the *N*-glycosyl triazole group occupying the equatorial position at the anomeric carbon atom. Also, the complete regioselectivity of the cycloaddition process is supported with only the 1,4–1*H*-1,2,3-triazole being formed as the ^{1}H NMR spectrum of the crude reaction mixture did not show any additional signals that may indicate the formation of the corresponding 1,5-isomer.

The molecules arrange in the solid state in layers with mainly hydrophobic sections built from the butyl triazole units on the one hand and the more polar moieties dominated by the carbohydrate units on the other. Within the polar layers intermolecular interactions are dominated by a three-dimensional network of weak C—H···O hydrogen bonds with the acetyl keto oxygen atoms as the H bond acceptors (Fig. 4, Table 1). The triazole units interact with each other *via* C—H···N hydrogen bonds that connect the molecules into two infinite chains made up of either A molecules or B molecules that stretch parallel to each other along the [1 0 0] direction (Figure 4, Table 1). In the hydrophobic layer dominated by the butyl groups no directional interactions are observed.

## Experimental

The triazole was prepared from 2,3,4-tri-*O*-acetyl-β-*L*-fucosyl azide (0.4 g, 1.27 mmol), 1-hexyne (0.16 ml, 1.38 mmol), 1*M* $CuSO_4$ (0.3 ml, 0.3 mmol), 1*M* ascorbic acid (0.4 ml, 0.4 mmol) and 10 ml of 1:1 ethanol/$H_2O$ as solvent. The mixture was heated to 345.5 K (70 °C) and allowed to stir vigorously until TLC showed the completion of the reaction. The reaction was monitored by TLC (1:1, hexane-ethyl acetate, $R_f$ = 0.41). After cooling to room temperature, ice water was added to the mixture which led to the precipitation of the triazole product which was then isolated by filtration through a glass frit. Purification by flash column chromatography (1:1, hexane-ethyl acetate) and recrystallization with isopropanol gave the title compound as a white solid (0.42 g, 83.3%). Crystals suitable for data collection were grown by slow evaporation from isopropanol. ^{1}H NMR ($CDCl_3$): δ 0.91 (t, 3H, *J* = 7.32 Hz), 1.23 (d, 3H, *J* = 6.22 Hz), 1.35 (m, 2H, *J* = 7.32 Hz), 1.64 (m, 2H, *J* = 7.32 Hz), 1.85 (s, 3H, $COCH_3$), 1.98 (s, 3H, $COCH_3$), 2.22 (s, 3H, $COCH_3$), 2.70 (t, 2H, *J* = 7.32 Hz), 4.09 (q, 1H, *J* = 6.59 Hz), 5.21 (dd, 1H, *J* = 2.93, 10.25 Hz), 5.37 (d, 1H, *J* = 3.30 Hz), 5.50 (t, 1H, H-2, *J* = 9.89 Hz), 5.76 (d, 1H, H-1, *J* =

# supplementary materials

9.89 Hz), 7.54 (s, 1H, H-triazole); $^{13}$C NMR (CDCl$_3$): δ 15.08, 17.33, 21.55, 21.83, 21.96, 23.45, 26.56, 32.53, 69.01, 71.07, 72.45, 73.75, 87.32, 119.86, 150.02, 170.20, 170.90, 171.39; MS: m/z calculated: 397.18, m/z found (ESI): 420.2 (+Na).

**Refinement**

Treatment of hydrogen atoms: All hydrogen atoms were added in calculated positions with a C—H bond distance of 0.95 Å (triazole H), 0.98 Å (methyl) or 1.00 Å (others). They were refined with isotropic displacement parameters of 1.5 times (methyl) or 1.2 times (others) that of the equivalent isotropic displacement parameter of the adjacent carbon atom. Methyl hydrogen atoms were allowed to rotate to best fit the experimental electron density.

Friedel pairs were merged prior to refinement. The absolute structure was assigned based on the known stereochemistry of carbon atoms not being changed during the synthesis of the compound.

**Figures**



Fig. 1. Synthesis of the title compound.
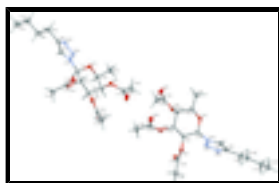


Fig. 2. Thermal ellipsoid representation of both crystallographically independent molecules. Displacement ellipsoids are at the 50% level, hydrogen atoms are shown as spheres of arbitrary radii.
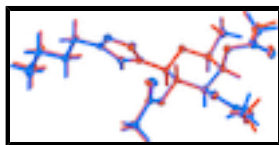


Fig. 3. Overlay of the A and B molecules.
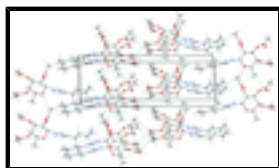


Fig. 4. View of the packing arrangement. Blue dotted lines represent C—H···O and C—H···N interactions.

**4-Butyl-1-(2,3,4-tri-*O*-acetyl-β-*L*-fucopyranosyl)-1*H*-1,2,3-triazole**

*Crystal data*

| | |
|---|---|
| C$_{18}$H$_{27}$N$_3$O$_7$ | $Z = 2$ |
| $M_r = 397.43$ | $F_{000} = 424$ |
| Triclinic, $P1$ | $D_x = 1.294$ Mg m$^{-3}$ |
| Hall symbol: P 1 | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 5.5173\ (3)$ Å | Cell parameters from 9354 reflections |
| $b = 7.7442\ (4)$ Å | $\theta = 2.6$–30.5º |
| $c = 24.1013\ (13)$ Å | $\mu = 0.10$ mm$^{-1}$ |
| $\alpha = 94.5070\ (10)$º | $T = 100$ K |
| $\beta = 96.1510\ (10)$º | Plate, colourless |

$\gamma = 91.2270\ (10)°$

$0.36 \times 0.35 \times 0.09$ mm

$V = 1020.22\ (9)$ Å$^3$

*Data collection*

| | |
|---|---|
| Bruker SMART APEX CCD diffractometer | 5041 independent reflections |
| Radiation source: fine-focus sealed tube | 4839 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.020$ |
| $T = 100$ K | $\theta_{max} = 28.3°$ |
| $\omega$ scans | $\theta_{min} = 0.9°$ |
| Absorption correction: multi-scan (SADABS in SAINT-Plus; Bruker, 2003) | $h = -7\rightarrow7$ |
| $T_{min} = 0.867$, $T_{max} = 0.991$ | $k = -10\rightarrow10$ |
| 10512 measured reflections | $l = -31\rightarrow32$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | H-atom parameters constrained |
| $wR(F^2) = 0.120$ | $w = 1/[\sigma^2(F_o^2) + (0.0771P)^2 + 0.17P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.11$ | $(\Delta/\sigma)_{max} < 0.001$ |
| 5041 reflections | $\Delta\rho_{max} = 0.42$ e Å$^{-3}$ |
| 515 parameters | $\Delta\rho_{min} = -0.21$ e Å$^{-3}$ |
| 3 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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 (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| C1A | 0.1284 (4) | 0.5904 (3) | 0.25737 (10) | 0.0160 (4) |
| H1A | 0.2899 | 0.5447 | 0.2490 | 0.019* |

| | | | |
|---|---|---|---|
| C2A | 0.0093 (4) | 0.4715 (3) | 0.29447 (10) | 0.0160 (4) |
| H2A | −0.1530 | 0.5159 | 0.3027 | 0.019* |
| C3A | 0.1762 (4) | 0.4617 (3) | 0.34855 (10) | 0.0172 (4) |
| H3A | 0.3249 | 0.3967 | 0.3405 | 0.021* |
| C4A | 0.2522 (4) | 0.6418 (3) | 0.37653 (10) | 0.0178 (5) |
| H4A | 0.3898 | 0.6301 | 0.4064 | 0.021* |
| C5A | 0.3353 (5) | 0.7604 (3) | 0.33445 (11) | 0.0201 (5) |
| H5A | 0.4950 | 0.7206 | 0.3229 | 0.024* |
| C6A | 0.3639 (6) | 0.9487 (4) | 0.35723 (13) | 0.0282 (6) |
| H6A1 | 0.4299 | 1.0173 | 0.3295 | 0.042* |
| H6A2 | 0.4757 | 0.9583 | 0.3919 | 0.042* |
| H6A3 | 0.2045 | 0.9923 | 0.3650 | 0.042* |
| C7A | 0.0505 (5) | 0.6379 (3) | 0.15536 (11) | 0.0186 (5) |
| H7A | 0.2127 | 0.6396 | 0.1455 | 0.022* |
| C8A | −0.1595 (4) | 0.6638 (3) | 0.12178 (11) | 0.0189 (5) |
| C9A | −0.1947 (5) | 0.7003 (4) | 0.06140 (12) | 0.0238 (5) |
| H9A1 | −0.2156 | 0.8262 | 0.0590 | 0.029* |
| H9A2 | −0.3463 | 0.6399 | 0.0434 | 0.029* |
| C10A | 0.0155 (5) | 0.6439 (4) | 0.02915 (11) | 0.0226 (5) |
| H10A | 0.0396 | 0.5186 | 0.0323 | 0.027* |
| H10B | 0.1665 | 0.7070 | 0.0463 | 0.027* |
| C11A | −0.0269 (5) | 0.6775 (5) | −0.03262 (12) | 0.0307 (6) |
| H11A | −0.1812 | 0.6180 | −0.0494 | 0.037* |
| H11B | −0.0451 | 0.8034 | −0.0357 | 0.037* |
| C12A | 0.1782 (6) | 0.6156 (5) | −0.06548 (13) | 0.0351 (7) |
| H12A | 0.3314 | 0.6749 | −0.0494 | 0.053* |
| H12B | 0.1425 | 0.6416 | −0.1046 | 0.053* |
| H12C | 0.1936 | 0.4903 | −0.0637 | 0.053* |
| C13A | −0.2440 (4) | 0.2243 (3) | 0.26034 (11) | 0.0192 (5) |
| C14A | −0.2384 (5) | 0.0438 (3) | 0.23312 (12) | 0.0248 (5) |
| H14A | −0.1490 | −0.0299 | 0.2590 | 0.037* |
| H14B | −0.1569 | 0.0458 | 0.1990 | 0.037* |
| H14C | −0.4056 | −0.0026 | 0.2235 | 0.037* |
| C15A | 0.1526 (5) | 0.3080 (3) | 0.43018 (12) | 0.0234 (5) |
| C16A | −0.0247 (6) | 0.2183 (5) | 0.46177 (14) | 0.0367 (7) |
| H16A | 0.0645 | 0.1544 | 0.4906 | 0.055* |
| H16B | −0.1292 | 0.1373 | 0.4359 | 0.055* |
| H16C | −0.1257 | 0.3043 | 0.4795 | 0.055* |
| C17A | 0.0802 (5) | 0.7476 (3) | 0.45847 (11) | 0.0207 (5) |
| C18A | −0.1564 (5) | 0.7616 (4) | 0.48340 (13) | 0.0286 (6) |
| H18A | −0.1241 | 0.7955 | 0.5236 | 0.043* |
| H18B | −0.2447 | 0.6494 | 0.4777 | 0.043* |
| H18C | −0.2553 | 0.8492 | 0.4653 | 0.043* |
| N1A | −0.0240 (4) | 0.6093 (3) | 0.20568 (9) | 0.0166 (4) |
| N2A | −0.2688 (4) | 0.6186 (3) | 0.20427 (10) | 0.0204 (4) |
| N3A | −0.3509 (4) | 0.6499 (3) | 0.15312 (10) | 0.0203 (4) |
| O1A | 0.1606 (3) | 0.7576 (2) | 0.28548 (8) | 0.0195 (4) |
| O2A | −0.0188 (3) | 0.3011 (2) | 0.26588 (8) | 0.0181 (3) |
| O3A | −0.4211 (3) | 0.2926 (3) | 0.27577 (9) | 0.0262 (4) |

| | | | |
|---|---|---|---|
| O4A | 0.0358 (3) | 0.3647 (2) | 0.38316 (8) | 0.0216 (4) |
| O5A | 0.3664 (4) | 0.3301 (3) | 0.44336 (9) | 0.0329 (5) |
| O6A | 0.0444 (3) | 0.7086 (2) | 0.40218 (8) | 0.0205 (4) |
| O7A | 0.2785 (4) | 0.7661 (4) | 0.48414 (9) | 0.0385 (6) |
| C1B | 0.2703 (4) | 0.9784 (3) | 0.80834 (10) | 0.0174 (5) |
| H1B | 0.1076 | 0.9344 | 0.8168 | 0.021* |
| C2B | 0.3807 (4) | 0.8444 (3) | 0.76952 (11) | 0.0166 (4) |
| H2B | 0.5439 | 0.8862 | 0.7608 | 0.020* |
| C3B | 0.2060 (4) | 0.8121 (3) | 0.71631 (10) | 0.0186 (5) |
| H3B | 0.0553 | 0.7500 | 0.7248 | 0.022* |
| C4B | 0.1364 (4) | 0.9804 (3) | 0.69039 (11) | 0.0193 (5) |
| H4B | −0.0028 | 0.9557 | 0.6605 | 0.023* |
| C5B | 0.0619 (5) | 1.1164 (4) | 0.73393 (11) | 0.0212 (5) |
| H5B | −0.0975 | 1.0789 | 0.7460 | 0.025* |
| C6B | 0.0375 (6) | 1.2960 (4) | 0.71358 (12) | 0.0275 (6) |
| H6B1 | −0.0147 | 1.3756 | 0.7434 | 0.041* |
| H6B2 | −0.0838 | 1.2920 | 0.6806 | 0.041* |
| H6B3 | 0.1953 | 1.3367 | 0.7036 | 0.041* |
| C7B | 0.3525 (5) | 1.0728 (3) | 0.91047 (11) | 0.0195 (5) |
| H7B | 0.1904 | 1.0772 | 0.9203 | 0.023* |
| C8B | 0.5642 (4) | 1.1177 (3) | 0.94397 (11) | 0.0187 (5) |
| C9B | 0.6021 (5) | 1.1841 (4) | 1.00422 (12) | 0.0244 (5) |
| H9B1 | 0.6318 | 1.3111 | 1.0067 | 0.029* |
| H9B2 | 0.7496 | 1.1320 | 1.0222 | 0.029* |
| C10B | 0.3876 (5) | 1.1448 (4) | 1.03638 (11) | 0.0232 (5) |
| H10C | 0.2409 | 1.1995 | 1.0190 | 0.028* |
| H10D | 0.3552 | 1.0180 | 1.0332 | 0.028* |
| C11B | 0.4303 (6) | 1.2089 (5) | 1.09785 (12) | 0.0309 (6) |
| H11C | 0.4583 | 1.3361 | 1.1010 | 0.037* |
| H11D | 0.5800 | 1.1568 | 1.1149 | 0.037* |
| C12B | 0.2200 (6) | 1.1658 (5) | 1.13071 (13) | 0.0351 (7) |
| H12D | 0.0720 | 1.2198 | 1.1149 | 0.053* |
| H12E | 0.2593 | 1.2100 | 1.1699 | 0.053* |
| H12F | 0.1931 | 1.0399 | 1.1285 | 0.053* |
| C13B | 0.6276 (5) | 0.6187 (3) | 0.80445 (11) | 0.0192 (5) |
| C14B | 0.6181 (5) | 0.4532 (4) | 0.83252 (13) | 0.0264 (6) |
| H14D | 0.5291 | 0.3635 | 0.8068 | 0.040* |
| H14E | 0.5345 | 0.4719 | 0.8663 | 0.040* |
| H14F | 0.7844 | 0.4160 | 0.8428 | 0.040* |
| C15B | 0.1945 (6) | 0.6211 (4) | 0.63345 (12) | 0.0272 (6) |
| C16B | 0.3516 (7) | 0.5229 (5) | 0.59592 (14) | 0.0402 (8) |
| H16D | 0.2618 | 0.4193 | 0.5779 | 0.060* |
| H16E | 0.4996 | 0.4886 | 0.6181 | 0.060* |
| H16F | 0.3965 | 0.5967 | 0.5672 | 0.060* |
| C17B | 0.3086 (5) | 1.0620 (4) | 0.60971 (11) | 0.0218 (5) |
| C18B | 0.5441 (5) | 1.0822 (5) | 0.58499 (13) | 0.0306 (6) |
| H18D | 0.5115 | 1.1205 | 0.5472 | 0.046* |
| H18E | 0.6251 | 0.9709 | 0.5832 | 0.046* |
| H18F | 0.6498 | 1.1686 | 0.6084 | 0.046* |

| | | | |
|---|---|---|---|
| N1B | 0.4259 (4) | 1.0209 (3) | 0.86032 (9) | 0.0166 (4) |
| N2B | 0.6712 (4) | 1.0341 (3) | 0.86152 (10) | 0.0202 (4) |
| N3B | 0.7542 (4) | 1.0919 (3) | 0.91256 (9) | 0.0201 (4) |
| O1B | 0.2423 (3) | 1.1344 (2) | 0.78221 (8) | 0.0195 (4) |
| O2B | 0.4016 (3) | 0.6857 (2) | 0.79666 (8) | 0.0187 (3) |
| O3B | 0.8064 (3) | 0.6837 (3) | 0.78989 (10) | 0.0285 (4) |
| O4B | 0.3322 (4) | 0.7019 (2) | 0.67849 (8) | 0.0228 (4) |
| O5B | −0.0221 (4) | 0.6326 (3) | 0.62559 (10) | 0.0363 (5) |
| O6B | 0.3468 (3) | 1.0408 (2) | 0.66523 (8) | 0.0209 (4) |
| O7B | 0.1098 (4) | 1.0641 (4) | 0.58456 (9) | 0.0352 (5) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1A | 0.0121 (10) | 0.0187 (11) | 0.0174 (11) | 0.0013 (8) | 0.0022 (8) | 0.0014 (9) |
| C2A | 0.0137 (10) | 0.0157 (10) | 0.0185 (11) | 0.0005 (8) | 0.0032 (8) | −0.0015 (8) |
| C3A | 0.0154 (11) | 0.0187 (11) | 0.0183 (11) | 0.0008 (8) | 0.0032 (8) | 0.0040 (9) |
| C4A | 0.0139 (11) | 0.0202 (11) | 0.0191 (11) | 0.0011 (9) | 0.0026 (8) | −0.0017 (9) |
| C5A | 0.0167 (11) | 0.0218 (12) | 0.0215 (12) | −0.0022 (9) | 0.0013 (9) | 0.0007 (9) |
| C6A | 0.0322 (15) | 0.0227 (13) | 0.0281 (14) | −0.0071 (11) | −0.0006 (11) | −0.0002 (10) |
| C7A | 0.0155 (11) | 0.0213 (11) | 0.0195 (12) | 0.0015 (9) | 0.0042 (9) | 0.0003 (9) |
| C8A | 0.0134 (11) | 0.0216 (11) | 0.0213 (12) | 0.0008 (9) | 0.0035 (9) | −0.0022 (9) |
| C9A | 0.0177 (12) | 0.0311 (14) | 0.0230 (13) | 0.0057 (10) | 0.0015 (9) | 0.0051 (10) |
| C10A | 0.0212 (12) | 0.0263 (13) | 0.0209 (12) | 0.0020 (10) | 0.0039 (9) | 0.0031 (10) |
| C11A | 0.0249 (14) | 0.0474 (18) | 0.0208 (13) | 0.0034 (12) | 0.0035 (10) | 0.0067 (12) |
| C12A | 0.0313 (16) | 0.0507 (19) | 0.0253 (15) | 0.0038 (13) | 0.0087 (12) | 0.0065 (13) |
| C13A | 0.0171 (11) | 0.0213 (12) | 0.0192 (12) | 0.0014 (9) | 0.0006 (9) | 0.0023 (9) |
| C14A | 0.0240 (13) | 0.0202 (12) | 0.0288 (14) | 0.0010 (10) | 0.0005 (10) | −0.0029 (10) |
| C15A | 0.0291 (14) | 0.0182 (11) | 0.0231 (13) | 0.0025 (10) | 0.0035 (10) | 0.0021 (9) |
| C16A | 0.0382 (18) | 0.0403 (17) | 0.0343 (16) | −0.0012 (14) | 0.0052 (13) | 0.0192 (13) |
| C17A | 0.0157 (11) | 0.0221 (12) | 0.0243 (12) | 0.0011 (9) | 0.0034 (9) | −0.0006 (9) |
| C18A | 0.0159 (12) | 0.0402 (16) | 0.0297 (14) | 0.0012 (11) | 0.0062 (10) | −0.0018 (12) |
| N1A | 0.0123 (9) | 0.0177 (9) | 0.0199 (10) | 0.0007 (7) | 0.0017 (7) | 0.0010 (7) |
| N2A | 0.0121 (9) | 0.0221 (10) | 0.0270 (11) | −0.0006 (8) | 0.0027 (8) | 0.0018 (8) |
| N3A | 0.0148 (10) | 0.0221 (10) | 0.0243 (11) | −0.0001 (8) | 0.0037 (8) | 0.0019 (8) |
| O1A | 0.0183 (8) | 0.0172 (8) | 0.0222 (9) | −0.0004 (6) | −0.0001 (7) | 0.0001 (7) |
| O2A | 0.0133 (8) | 0.0174 (8) | 0.0233 (9) | 0.0017 (6) | 0.0033 (6) | −0.0017 (7) |
| O3A | 0.0172 (9) | 0.0231 (9) | 0.0385 (11) | −0.0029 (7) | 0.0079 (8) | −0.0019 (8) |
| O4A | 0.0183 (8) | 0.0242 (9) | 0.0230 (9) | −0.0012 (7) | 0.0035 (7) | 0.0049 (7) |
| O5A | 0.0300 (11) | 0.0354 (12) | 0.0319 (11) | 0.0004 (9) | −0.0069 (9) | 0.0089 (9) |
| O6A | 0.0144 (8) | 0.0229 (9) | 0.0238 (9) | 0.0038 (7) | 0.0023 (7) | −0.0020 (7) |
| O7A | 0.0173 (10) | 0.0686 (17) | 0.0267 (11) | 0.0044 (10) | 0.0018 (8) | −0.0144 (11) |
| C1B | 0.0139 (10) | 0.0202 (11) | 0.0173 (11) | −0.0023 (9) | −0.0002 (8) | 0.0009 (9) |
| C2B | 0.0140 (10) | 0.0161 (10) | 0.0195 (11) | −0.0026 (8) | 0.0013 (8) | 0.0023 (8) |
| C3B | 0.0180 (11) | 0.0201 (11) | 0.0171 (11) | −0.0015 (9) | 0.0013 (8) | 0.0005 (9) |
| C4B | 0.0134 (11) | 0.0241 (12) | 0.0202 (12) | −0.0022 (9) | 0.0008 (9) | 0.0037 (9) |
| C5B | 0.0164 (11) | 0.0252 (12) | 0.0219 (12) | 0.0006 (9) | 0.0003 (9) | 0.0043 (9) |
| C6B | 0.0311 (14) | 0.0244 (13) | 0.0266 (14) | 0.0054 (11) | −0.0020 (11) | 0.0046 (11) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C7B | 0.0155 (11) | 0.0209 (11) | 0.0220 (12) | −0.0009 (9) | 0.0032 (9) | 0.0007 (9) |
| C8B | 0.0144 (11) | 0.0185 (11) | 0.0232 (12) | −0.0022 (9) | 0.0017 (9) | 0.0033 (9) |
| C9B | 0.0185 (12) | 0.0321 (14) | 0.0216 (13) | −0.0063 (10) | 0.0013 (9) | −0.0007 (10) |
| C10B | 0.0199 (12) | 0.0279 (13) | 0.0213 (12) | −0.0035 (10) | 0.0029 (9) | −0.0011 (10) |
| C11B | 0.0248 (14) | 0.0444 (17) | 0.0222 (14) | −0.0059 (12) | 0.0040 (10) | −0.0046 (12) |
| C12B | 0.0304 (16) | 0.0494 (19) | 0.0253 (14) | −0.0049 (14) | 0.0091 (12) | −0.0043 (13) |
| C13B | 0.0184 (12) | 0.0193 (11) | 0.0190 (12) | −0.0015 (9) | 0.0016 (9) | −0.0029 (9) |
| C14B | 0.0224 (13) | 0.0248 (13) | 0.0324 (15) | 0.0004 (10) | 0.0006 (10) | 0.0071 (11) |
| C15B | 0.0409 (17) | 0.0186 (12) | 0.0204 (13) | −0.0067 (11) | −0.0024 (11) | 0.0012 (10) |
| C16B | 0.058 (2) | 0.0306 (15) | 0.0298 (16) | −0.0025 (15) | 0.0037 (14) | −0.0102 (12) |
| C17B | 0.0167 (12) | 0.0260 (13) | 0.0228 (12) | 0.0006 (9) | 0.0043 (9) | 0.0000 (10) |
| C18B | 0.0187 (13) | 0.0454 (17) | 0.0281 (14) | −0.0006 (11) | 0.0073 (11) | −0.0006 (12) |
| N1B | 0.0121 (9) | 0.0196 (10) | 0.0179 (10) | −0.0014 (7) | 0.0018 (7) | 0.0005 (8) |
| N2B | 0.0117 (9) | 0.0252 (11) | 0.0233 (11) | −0.0001 (8) | 0.0014 (7) | 0.0003 (8) |
| N3B | 0.0146 (9) | 0.0244 (11) | 0.0212 (10) | 0.0009 (8) | 0.0023 (8) | 0.0008 (8) |
| O1B | 0.0187 (8) | 0.0184 (8) | 0.0207 (9) | −0.0015 (6) | −0.0008 (7) | 0.0015 (7) |
| O2B | 0.0153 (8) | 0.0194 (8) | 0.0217 (9) | −0.0017 (6) | 0.0017 (6) | 0.0049 (7) |
| O3B | 0.0175 (9) | 0.0261 (10) | 0.0432 (12) | 0.0005 (7) | 0.0081 (8) | 0.0059 (9) |
| O4B | 0.0266 (10) | 0.0210 (9) | 0.0200 (9) | −0.0018 (7) | 0.0025 (7) | −0.0028 (7) |
| O5B | 0.0365 (13) | 0.0351 (12) | 0.0329 (12) | −0.0066 (9) | −0.0092 (9) | −0.0049 (9) |
| O6B | 0.0149 (8) | 0.0252 (9) | 0.0225 (9) | −0.0050 (7) | 0.0006 (7) | 0.0054 (7) |
| O7B | 0.0190 (10) | 0.0659 (16) | 0.0212 (10) | −0.0011 (10) | 0.0010 (7) | 0.0101 (10) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1A—O1A | 1.412 (3) | C1B—O1B | 1.409 (3) |
| C1A—N1A | 1.446 (3) | C1B—N1B | 1.452 (3) |
| C1A—C2A | 1.519 (3) | C1B—C2B | 1.523 (3) |
| C1A—H1A | 1.0000 | C1B—H1B | 1.0000 |
| C2A—O2A | 1.437 (3) | C2B—O2B | 1.438 (3) |
| C2A—C3A | 1.521 (3) | C2B—C3B | 1.520 (3) |
| C2A—H2A | 1.0000 | C2B—H2B | 1.0000 |
| C3A—O4A | 1.440 (3) | C3B—O4B | 1.443 (3) |
| C3A—C4A | 1.530 (3) | C3B—C4B | 1.528 (3) |
| C3A—H3A | 1.0000 | C3B—H3B | 1.0000 |
| C4A—O6A | 1.446 (3) | C4B—O6B | 1.451 (3) |
| C4A—C5A | 1.520 (4) | C4B—C5B | 1.522 (4) |
| C4A—H4A | 1.0000 | C4B—H4B | 1.0000 |
| C5A—O1A | 1.440 (3) | C5B—O1B | 1.444 (3) |
| C5A—C6A | 1.516 (4) | C5B—C6B | 1.515 (4) |
| C5A—H5A | 1.0000 | C5B—H5B | 1.0000 |
| C6A—H6A1 | 0.9800 | C6B—H6B1 | 0.9800 |
| C6A—H6A2 | 0.9800 | C6B—H6B2 | 0.9800 |
| C6A—H6A3 | 0.9800 | C6B—H6B3 | 0.9800 |
| C7A—N1A | 1.354 (3) | C7B—N1B | 1.350 (3) |
| C7A—C8A | 1.369 (3) | C7B—C8B | 1.369 (3) |
| C7A—H7A | 0.9500 | C7B—H7B | 0.9500 |
| C8A—N3A | 1.369 (3) | C8B—N3B | 1.367 (3) |
| C8A—C9A | 1.497 (4) | C8B—C9B | 1.493 (4) |

| | | | |
|---|---|---|---|
| C9A—C10A | 1.517 (4) | C9B—C10B | 1.520 (4) |
| C9A—H9A1 | 0.9900 | C9B—H9B1 | 0.9900 |
| C9A—H9A2 | 0.9900 | C9B—H9B2 | 0.9900 |
| C10A—C11A | 1.526 (4) | C10B—C11B | 1.517 (4) |
| C10A—H10A | 0.9900 | C10B—H10C | 0.9900 |
| C10A—H10B | 0.9900 | C10B—H10D | 0.9900 |
| C11A—C12A | 1.514 (4) | C11B—C12B | 1.519 (4) |
| C11A—H11A | 0.9900 | C11B—H11C | 0.9900 |
| C11A—H11B | 0.9900 | C11B—H11D | 0.9900 |
| C12A—H12A | 0.9800 | C12B—H12D | 0.9800 |
| C12A—H12B | 0.9800 | C12B—H12E | 0.9800 |
| C12A—H12C | 0.9800 | C12B—H12F | 0.9800 |
| C13A—O3A | 1.200 (3) | C13B—O3B | 1.196 (3) |
| C13A—O2A | 1.355 (3) | C13B—O2B | 1.361 (3) |
| C13A—C14A | 1.500 (4) | C13B—C14B | 1.498 (4) |
| C14A—H14A | 0.9800 | C14B—H14D | 0.9800 |
| C14A—H14B | 0.9800 | C14B—H14E | 0.9800 |
| C14A—H14C | 0.9800 | C14B—H14F | 0.9800 |
| C15A—O5A | 1.193 (4) | C15B—O5B | 1.196 (4) |
| C15A—O4A | 1.351 (3) | C15B—O4B | 1.358 (3) |
| C15A—C16A | 1.495 (4) | C15B—C16B | 1.495 (5) |
| C16A—H16A | 0.9800 | C16B—H16D | 0.9800 |
| C16A—H16B | 0.9800 | C16B—H16E | 0.9800 |
| C16A—H16C | 0.9800 | C16B—H16F | 0.9800 |
| C17A—O7A | 1.198 (3) | C17B—O7B | 1.196 (3) |
| C17A—O6A | 1.359 (3) | C17B—O6B | 1.355 (3) |
| C17A—C18A | 1.497 (4) | C17B—C18B | 1.496 (4) |
| C18A—H18A | 0.9800 | C18B—H18D | 0.9800 |
| C18A—H18B | 0.9800 | C18B—H18E | 0.9800 |
| C18A—H18C | 0.9800 | C18B—H18F | 0.9800 |
| N1A—N2A | 1.351 (3) | N1B—N2B | 1.352 (3) |
| N2A—N3A | 1.310 (3) | N2B—N3B | 1.307 (3) |
| O1A—C1A—N1A | 106.41 (19) | O1B—C1B—N1B | 106.14 (19) |
| O1A—C1A—C2A | 108.84 (19) | O1B—C1B—C2B | 109.43 (19) |
| N1A—C1A—C2A | 111.89 (19) | N1B—C1B—C2B | 112.5 (2) |
| O1A—C1A—H1A | 109.9 | O1B—C1B—H1B | 109.6 |
| N1A—C1A—H1A | 109.9 | N1B—C1B—H1B | 109.6 |
| C2A—C1A—H1A | 109.9 | C2B—C1B—H1B | 109.6 |
| O2A—C2A—C1A | 108.36 (19) | O2B—C2B—C3B | 108.53 (19) |
| O2A—C2A—C3A | 108.51 (19) | O2B—C2B—C1B | 108.20 (19) |
| C1A—C2A—C3A | 108.96 (19) | C3B—C2B—C1B | 108.09 (19) |
| O2A—C2A—H2A | 110.3 | O2B—C2B—H2B | 110.6 |
| C1A—C2A—H2A | 110.3 | C3B—C2B—H2B | 110.6 |
| C3A—C2A—H2A | 110.3 | C1B—C2B—H2B | 110.6 |
| O4A—C3A—C2A | 104.62 (19) | O4B—C3B—C2B | 105.76 (19) |
| O4A—C3A—C4A | 111.7 (2) | O4B—C3B—C4B | 110.6 (2) |
| C2A—C3A—C4A | 111.91 (19) | C2B—C3B—C4B | 112.0 (2) |
| O4A—C3A—H3A | 109.5 | O4B—C3B—H3B | 109.5 |
| C2A—C3A—H3A | 109.5 | C2B—C3B—H3B | 109.5 |

| | | | |
|---|---|---|---|
| C4A—C3A—H3A | 109.5 | C4B—C3B—H3B | 109.5 |
| O6A—C4A—C5A | 111.8 (2) | O6B—C4B—C5B | 110.9 (2) |
| O6A—C4A—C3A | 107.00 (19) | O6B—C4B—C3B | 107.1 (2) |
| C5A—C4A—C3A | 111.2 (2) | C5B—C4B—C3B | 111.6 (2) |
| O6A—C4A—H4A | 108.9 | O6B—C4B—H4B | 109.1 |
| C5A—C4A—H4A | 108.9 | C5B—C4B—H4B | 109.1 |
| C3A—C4A—H4A | 108.9 | C3B—C4B—H4B | 109.1 |
| O1A—C5A—C6A | 105.6 (2) | O1B—C5B—C6B | 105.7 (2) |
| O1A—C5A—C4A | 111.2 (2) | O1B—C5B—C4B | 110.5 (2) |
| C6A—C5A—C4A | 113.1 (2) | C6B—C5B—C4B | 114.1 (2) |
| O1A—C5A—H5A | 108.9 | O1B—C5B—H5B | 108.8 |
| C6A—C5A—H5A | 108.9 | C6B—C5B—H5B | 108.8 |
| C4A—C5A—H5A | 108.9 | C4B—C5B—H5B | 108.8 |
| C5A—C6A—H6A1 | 109.5 | C5B—C6B—H6B1 | 109.5 |
| C5A—C6A—H6A2 | 109.5 | C5B—C6B—H6B2 | 109.5 |
| H6A1—C6A—H6A2 | 109.5 | H6B1—C6B—H6B2 | 109.5 |
| C5A—C6A—H6A3 | 109.5 | C5B—C6B—H6B3 | 109.5 |
| H6A1—C6A—H6A3 | 109.5 | H6B1—C6B—H6B3 | 109.5 |
| H6A2—C6A—H6A3 | 109.5 | H6B2—C6B—H6B3 | 109.5 |
| N1A—C7A—C8A | 104.7 (2) | N1B—C7B—C8B | 104.4 (2) |
| N1A—C7A—H7A | 127.6 | N1B—C7B—H7B | 127.8 |
| C8A—C7A—H7A | 127.6 | C8B—C7B—H7B | 127.8 |
| C7A—C8A—N3A | 107.9 (2) | N3B—C8B—C7B | 108.0 (2) |
| C7A—C8A—C9A | 129.8 (2) | N3B—C8B—C9B | 122.2 (2) |
| N3A—C8A—C9A | 122.3 (2) | C7B—C8B—C9B | 129.8 (2) |
| C8A—C9A—C10A | 113.5 (2) | C8B—C9B—C10B | 113.5 (2) |
| C8A—C9A—H9A1 | 108.9 | C8B—C9B—H9B1 | 108.9 |
| C10A—C9A—H9A1 | 108.9 | C10B—C9B—H9B1 | 108.9 |
| C8A—C9A—H9A2 | 108.9 | C8B—C9B—H9B2 | 108.9 |
| C10A—C9A—H9A2 | 108.9 | C10B—C9B—H9B2 | 108.9 |
| H9A1—C9A—H9A2 | 107.7 | H9B1—C9B—H9B2 | 107.7 |
| C9A—C10A—C11A | 112.7 (2) | C11B—C10B—C9B | 113.1 (2) |
| C9A—C10A—H10A | 109.0 | C11B—C10B—H10C | 108.9 |
| C11A—C10A—H10A | 109.0 | C9B—C10B—H10C | 108.9 |
| C9A—C10A—H10B | 109.0 | C11B—C10B—H10D | 108.9 |
| C11A—C10A—H10B | 109.0 | C9B—C10B—H10D | 108.9 |
| H10A—C10A—H10B | 107.8 | H10C—C10B—H10D | 107.8 |
| C12A—C11A—C10A | 113.0 (2) | C10B—C11B—C12B | 113.6 (3) |
| C12A—C11A—H11A | 109.0 | C10B—C11B—H11C | 108.9 |
| C10A—C11A—H11A | 109.0 | C12B—C11B—H11C | 108.9 |
| C12A—C11A—H11B | 109.0 | C10B—C11B—H11D | 108.9 |
| C10A—C11A—H11B | 109.0 | C12B—C11B—H11D | 108.9 |
| H11A—C11A—H11B | 107.8 | H11C—C11B—H11D | 107.7 |
| C11A—C12A—H12A | 109.5 | C11B—C12B—H12D | 109.5 |
| C11A—C12A—H12B | 109.5 | C11B—C12B—H12E | 109.5 |
| H12A—C12A—H12B | 109.5 | H12D—C12B—H12E | 109.5 |
| C11A—C12A—H12C | 109.5 | C11B—C12B—H12F | 109.5 |
| H12A—C12A—H12C | 109.5 | H12D—C12B—H12F | 109.5 |
| H12B—C12A—H12C | 109.5 | H12E—C12B—H12F | 109.5 |

| | | | |
|---|---|---|---|
| O3A—C13A—O2A | 124.0 (2) | O3B—C13B—O2B | 123.8 (2) |
| O3A—C13A—C14A | 125.5 (2) | O3B—C13B—C14B | 125.6 (2) |
| O2A—C13A—C14A | 110.5 (2) | O2B—C13B—C14B | 110.6 (2) |
| C13A—C14A—H14A | 109.5 | C13B—C14B—H14D | 109.5 |
| C13A—C14A—H14B | 109.5 | C13B—C14B—H14E | 109.5 |
| H14A—C14A—H14B | 109.5 | H14D—C14B—H14E | 109.5 |
| C13A—C14A—H14C | 109.5 | C13B—C14B—H14F | 109.5 |
| H14A—C14A—H14C | 109.5 | H14D—C14B—H14F | 109.5 |
| H14B—C14A—H14C | 109.5 | H14E—C14B—H14F | 109.5 |
| O5A—C15A—O4A | 123.3 (3) | O5B—C15B—O4B | 123.1 (3) |
| O5A—C15A—C16A | 126.7 (3) | O5B—C15B—C16B | 126.2 (3) |
| O4A—C15A—C16A | 110.0 (2) | O4B—C15B—C16B | 110.7 (3) |
| C15A—C16A—H16A | 109.5 | C15B—C16B—H16D | 109.5 |
| C15A—C16A—H16B | 109.5 | C15B—C16B—H16E | 109.5 |
| H16A—C16A—H16B | 109.5 | H16D—C16B—H16E | 109.5 |
| C15A—C16A—H16C | 109.5 | C15B—C16B—H16F | 109.5 |
| H16A—C16A—H16C | 109.5 | H16D—C16B—H16F | 109.5 |
| H16B—C16A—H16C | 109.5 | H16E—C16B—H16F | 109.5 |
| O7A—C17A—O6A | 123.1 (2) | O7B—C17B—O6B | 123.2 (2) |
| O7A—C17A—C18A | 125.3 (3) | O7B—C17B—C18B | 125.3 (3) |
| O6A—C17A—C18A | 111.6 (2) | O6B—C17B—C18B | 111.5 (2) |
| C17A—C18A—H18A | 109.5 | C17B—C18B—H18D | 109.5 |
| C17A—C18A—H18B | 109.5 | C17B—C18B—H18E | 109.5 |
| H18A—C18A—H18B | 109.5 | H18D—C18B—H18E | 109.5 |
| C17A—C18A—H18C | 109.5 | C17B—C18B—H18F | 109.5 |
| H18A—C18A—H18C | 109.5 | H18D—C18B—H18F | 109.5 |
| H18B—C18A—H18C | 109.5 | H18E—C18B—H18F | 109.5 |
| N2A—N1A—C7A | 111.1 (2) | C7B—N1B—N2B | 111.4 (2) |
| N2A—N1A—C1A | 121.4 (2) | C7B—N1B—C1B | 126.5 (2) |
| C7A—N1A—C1A | 127.1 (2) | N2B—N1B—C1B | 121.5 (2) |
| N3A—N2A—N1A | 106.6 (2) | N3B—N2B—N1B | 106.3 (2) |
| N2A—N3A—C8A | 109.6 (2) | N2B—N3B—C8B | 109.8 (2) |
| C1A—O1A—C5A | 111.88 (18) | C1B—O1B—C5B | 111.96 (19) |
| C13A—O2A—C2A | 117.27 (19) | C13B—O2B—C2B | 117.18 (19) |
| C15A—O4A—C3A | 117.9 (2) | C15B—O4B—C3B | 116.7 (2) |
| C17A—O6A—C4A | 115.87 (19) | C17B—O6B—C4B | 115.64 (19) |

*Hydrogen-bond geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| C1B—H1B···O3B[i] | 1.00 | 2.53 | 3.362 (3) | 141 |
| C2A—H2A···O3A | 1.00 | 2.26 | 2.701 (3) | 105 |
| C2B—H2B···O3B | 1.00 | 2.26 | 2.698 (3) | 105 |
| C3A—H3A···O3A[ii] | 1.00 | 2.32 | 3.214 (3) | 149 |
| C3B—H3B···O3B[i] | 1.00 | 2.27 | 3.165 (3) | 148 |
| C4A—H4A···O5A | 1.00 | 2.56 | 3.046 (3) | 110 |
| C4A—H4A···O7A | 1.00 | 2.23 | 2.682 (3) | 106 |
| C4B—H4B···O5B | 1.00 | 2.57 | 3.067 (3) | 110 |

| | | | |
|---|---|---|---|
| C4B—H4B⋯O7B | 1.00 | 2.21 | 2.670 (3) | 106 |
| C7A—H7A⋯N3A[ii] | 0.95 | 2.39 | 3.308 (4) | 161 |
| C7B—H7B⋯N3B[i] | 0.95 | 2.40 | 3.313 (4) | 161 |
| C14A—H14A⋯O1A[iii] | 0.98 | 2.47 | 3.377 (3) | 154 |
| C14B—H14D⋯O1B[iii] | 0.98 | 2.35 | 3.261 (3) | 155 |
| C16A—H16A⋯O7B[iii] | 0.98 | 2.41 | 3.295 (4) | 150 |
| C16B—H16F⋯O7A | 0.98 | 2.51 | 3.401 (4) | 150 |

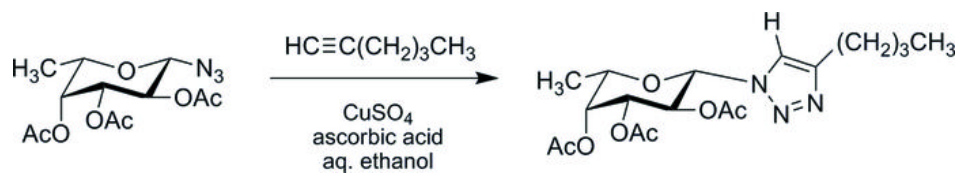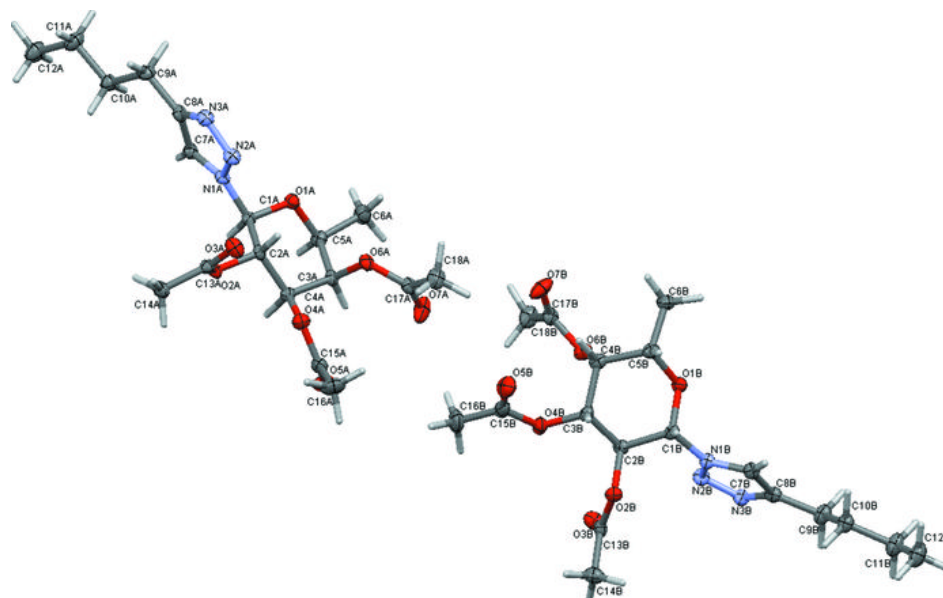Symmetry codes: (i) $x-1, y, z$; (ii) $x+1, y, z$; (iii) $x, y-1, z$.

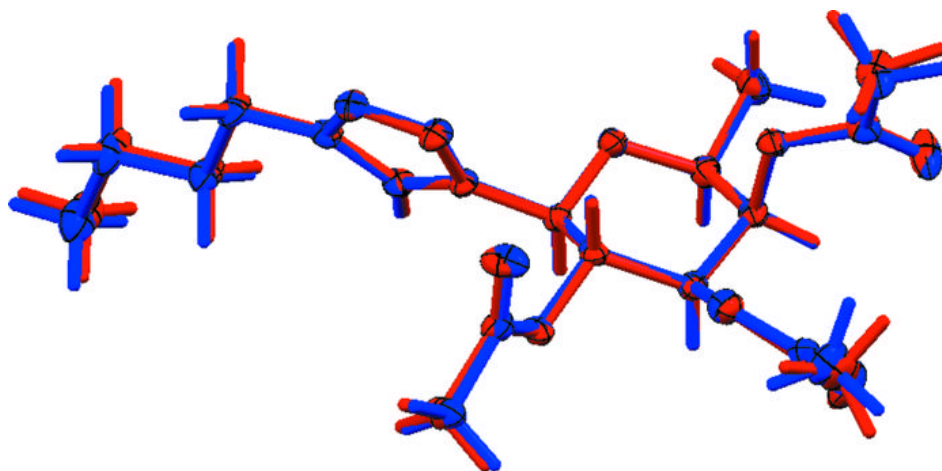# supplementary materials

Fig. 1

**Fig. 2**

**Fig. 3**

**Fig. 4**