

Prop-2-ynyl 1,2,3,4-tetra-*O*-acetyl- $\beta$ -D-glucopyranuronateDavid P. Temelkoff, Peter Norris  
and Matthias Zeller\*Department of Chemistry, Youngstown State  
University, 1 University Plaza, Youngstown,  
OH 44555-3663, USA

Correspondence e-mail: mzeller@cc.yzu.edu

## Key indicators

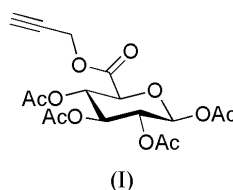
Single-crystal X-ray study  
 $T = 100$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.043  
 $wR$  factor = 0.106  
Data-to-parameter ratio = 9.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The structure of the title compound,  $\text{C}_{17}\text{H}_{20}\text{O}_{11}$ , has been determined by single-crystal X-ray diffraction at 100 K. The glucopyranose ring exhibits a chair conformation, with all non-H-atom substituents in equatorial positions, and the triple bond is sterically easily accessible, allowing for followup cycloaddition reactions. The glucopyranose ring exhibits chair conformation with all non-H substituents in equatorial positions, and the triple bond is sterically easily accessible, allowing for follow-up cycloaddition reactions.

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## Comment

Prop-2-ynyl 1,2,3,4-tetra-*O*-acetyl- $\beta$ -D-glucopyranuronate, (I), was prepared as part of a study on the synthesis of 1,2,3-triazole-linked sugars. In this study, various alkynes were coupled with a D-glucosyl azide in the presence of  $\text{Cu}^{\text{I}}$  to form 1,4-disubstituted 1,2,3-triazoles. This reaction was found to be regioselective, and proceeds with retention of the  $\beta$ -D-glucopyranosidic stereochemistry (Akula *et al.*, 2004). This rapid heterocycle-forming reaction fits well the criteria set out for processes useful in the so-called 'click chemistry' (Kolb *et al.*, 2001).



The molecular structure of (I) is shown in Fig. 1. The bond lengths and angles are within expected ranges for sugars. The six-membered ring exhibits the chair conformation expected for a glucopyranose, and all non-H substituents are found in equatorial positions. The acetate groups are all twisted to be roughly perpendicular to the equatorial plane and their  $\text{C}=\text{O}$  groups point alternately up and down (see Fig. 1).

The propynyl functional group is close to linear [ $\text{C}15-\text{C}16-\text{C}17 = 177.8(3)^\circ$ ] and the  $\text{C}16-\text{C}17-\text{H}17$  angle is  $176(2)^\circ$ . The  $\text{C}\equiv\text{C}$  triple bond distance is  $1.177(4)$  Å. Steric crowding around the triple bond is not very pronounced, and both the acidic H atom and the  $\pi$ -electron density of the alkyne are easily accessible from several sides of the molecule. Thus, the regioselectivity of the Cu-catalysed triazole-formation reaction is best explained by electronic effects induced by the copper catalyst itself, on either an intermediate or the transition state of the cycloaddition reaction.

## Experimental

Prop-2-ynyl 1,2,3,4-tetra-*O*-acetyl- $\beta$ -D-glucopyranuronate was prepared as described in the literature (Akula *et al.*, 2004). Crystals

suitable for single-crystal X-ray diffraction were obtained by slow cooling of a hot methanol solution.

*Crystal data*

$C_{17}H_{20}O_{11}$   
 $M_r = 400.33$   
 Orthorhombic,  $P2_12_12_1$   
 $a = 7.3827$  (9) Å  
 $b = 14.1462$  (17) Å  
 $c = 18.077$  (2) Å  
 $V = 1887.9$  (4) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.408$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
 Cell parameters from 6910 reflections  
 $\theta = 2.9\text{--}31.8^\circ$   
 $\mu = 0.12$  mm<sup>-1</sup>  
 $T = 100$  (2) K  
 Block, colourless  
 $0.37 \times 0.22 \times 0.22$  mm

*Data collection*

Bruker SMART APEX CCD diffractometer  
 $\varphi$  and  $\omega$  scans  
 Absorption correction: multi-scan (SADABS in S<sub>A</sub>I<sub>N</sub>T-Plus; Bruker, 2003)  
 $T_{\min} = 0.884$ ,  $T_{\max} = 0.97$   
 20 026 measured reflections

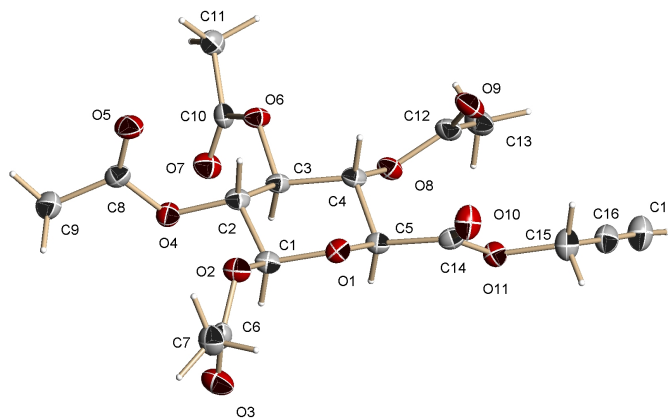
2663 independent reflections  
 2634 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.021$   
 $\theta_{\text{max}} = 28.3^\circ$   
 $h = -9 \rightarrow 9$   
 $k = -18 \rightarrow 18$   
 $l = -23 \rightarrow 23$

*Refinement*

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.043$   
 $wR(F^2) = 0.106$   
 $S = 1.19$   
 2663 reflections  
 282 parameters  
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.043P)^2 + 0.6857P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.38$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.22$  e Å<sup>-3</sup>

Methyl H atoms were placed in calculated positions, with a C–H bond length of 0.98 Å, and have been refined as riding, with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ . All other H atoms were located in a difference density Fourier map, and their atomic coordinates were refined. For the acetylenic H atom, the isotropic displacement parameter was also refined; for all other H atoms,  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ . In the absence of significant anomalous dispersion effects, Friedel pairs were merged before refinement. The absolute configuration assignment is based on the known configuration of C atoms retaining their configuration during the synthesis of the alkyne. The s.u. values of the cell parameters are taken from the software, recognizing that the values are unreasonably small (Herbstein, 2000).



**Figure 1**  
 The molecular structure of (I), showing 50% probability displacement ellipsoids.

Data collection: SMART (Bruker, 2002); cell refinement: S<sub>A</sub>I<sub>N</sub>T-Plus (Bruker, 2003); data reduction: S<sub>A</sub>I<sub>N</sub>T-Plus; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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