Crystal Structure of Methyl 2,3-O-Isopropylidene-β-D-allo-hept-6-ynofuranoside
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Methods for the synthesis and separation of epimeric uronic acids have been studied by Kjelberg et al.1 One of the methods used for chain extention has been the ethynylation reaction described by Jones et al.2 Reaction between methyl 2,3-O-isopropylidene-β-D-ribo-1,5-dialdopentofuranoside and ethynyl-magnesium bromide gave two isomers (C-5 epimers) in approximately equal amounts.3 Since configurational assignments based on spectroscopic data were somewhat uncertain, an X-ray crystallographic investigation of the isomer which was supposed to be the methyl-2,3-O-isopropylidene-β-D-allo-hept-6-ynofuranoside has been carried out.

The crystals of C₅OH₈ are orthorhombic with cell dimensions a = 5.855(2) Å, b = 11.555(3) Å, c = 18.174(3) Å and four molecules in the unit cell (Dₓ = 1.23 g/cm³). The space group is P2₁2₁2₁. The structure was solved by direct methods4 and refined by full-matrix least squares technique5 to an R-value of 3.4% (R_w = 4.1%) for 971 reflections observed on an automatic four circle diffractometer at room temperature. Anisotropic temperature factors were introduced for oxygen and carbon atoms. The hydroxy and methyl hydrogen atoms were found in the difference Fourier map. Positional parameters for the remaining hydrogen atoms were calculated. Weights for least squares refinement were obtained from the standard deviations in intensities, σ(I), taken as

σ(I) = [C_T + (0.02C_N)]^{1/2}

where C_T is the total number of counts and C_N the net count.

Final fractional coordinates and thermal parameters with estimated standard deviations are given in Table 1. The principal axes of thermal vibration ellipsoids for carbon atoms were calculated from the temperature parameters of Table 1. Maximum r.m.s. amplitudes range from 0.220 to 0.331 Å (corresponding B-values 3.80 and 8.63 Å²).

Bond distances and angles are listed in Table 2. Fig. 1 is a schematic drawing of the molecule (viewed along [100]) which shows that it has the D-allo configuration.

* All programs used (except those for phase determination) are included in this reference.


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Table 1. Final fractional coordinates and thermal parameters with estimated standard deviations. The expression for anisotropic vibration is exp \((-2\pi^2(h^2a^{*2}U11+\cdots+2kb^*c^*U23))\). Hm is bonded to Cm, HMmn to CMm, and HO to O5.

|   |  O1  |  O2  |  O3  |  O4  |  O5  |  O6  |  C1  |  C2  |  C3  |  C4  |  C5  |  C6  |  C7  |  C8  |  C9  |  C10 |  C11 |  C12 |  C13 |  C14 |  C15 |  C16 |  C17 |  C18 |  C19 |  C20 |  C21 |  C22 |  C23 |  C24 |  C25 |  C26 |  C27 |  C28 |  C29 |  C30 |  C31 |  C32 |  C33 |  C34 |  C35 |  C36 |  C37 |  C38 |  C39 |  C40 |  C41 |  C42 |  C43 |  C44 |  C45 |  C46 |  C47 |  C48 |  C49 |  C50 |  C51 |  C52 |  C53 |  C54 |  C55 |  C56 |  C57 |  C58 |  C59 |  C60 |  C61 |  C62 |  C63 |  C64 |  C65 |  C66 |  C67 |  C68 |  C69 |  C70 |  C71 |  C72 |  C73 |  C74 |  C75 |  C76 |  C77 |  C78 |  C79 |  C80 |  C81 |  C82 |  C83 |  C84 |  C85 |  C86 |  C87 |  C88 |  C89 |  C90 |  C91 |  C92 |  C93 |  C94 |  C95 |  C96 |  C97 |  C98 |  C99 |  C100 |
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