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# 2,6-Anhydro-1-deoxy-3,4-*O*-isopropylidene-3-*C*-methyl-*β*-D-*ribo*-hex-2-ulofuranose

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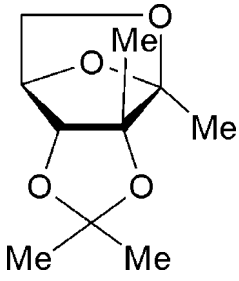
Key indicators: single-crystal X-ray study; $T$ = 150 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.031; $wR$ factor = 0.074; data-to-parameter ratio = 10.8.

The relative configuration of the title compound, $C_{10}H_{16}O_4$, was firmly established by X-ray crystallographic analysis. The absolute configuration was determined by the use of 2-*C*-methyl-D-ribono-1,4-lactone as the starting material.

## Related literature

For related literature see: Curran *et al.* (2007); Jones *et al.* (2007); Hotchkiss *et al.* (2006).




## Experimental

### *Crystal data*

$C_{10}H_{16}O_4$
$M_r$ = 200.23
Orthorhombic, $P2_12_12_1$
$a$ = 6.6307 (2) Å
$b$ = 11.1029 (4) Å
$c$ = 14.1409 (5) Å
$V$ = 1041.05 (6) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 150 K
0.60 × 0.50 × 0.10 mm

### *Data collection*

Nonius KappaCCD diffractometer
Absorption correction: multi-scan *DENZO/SCALEPACK* (Otwinowski & Minor, 1997)
$T_{min}$ = 0.95, $T_{max}$ = 1.00
6557 measured reflections
1370 independent reflections
1160 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.045

### *Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.074
$S$ = 0.93
1370 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.24 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2449).

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# supplementary materials

*Acta Cryst.* (2007). E**63**, o3388 [ **doi:10.1107/S1600536807031546** ]

# 2,6-Anhydro-1-deoxy-3,4-*O*-isopropylidene-3-*C*-methyl-*β*-D-*ribo*-hex-2-ulofuranose

**L. A. Curran, S. F. Jenkinson, N. A. Jones, D. J. Watkin and G. W. J. Fleet**

## Comment

For background information see the previous paper (Curran *et al.*, 2007). The crystal structure of the title compound (Fig. 1) exists as alternating layers of molecules running parallel to the *ab*-face (Fig.2). There is no hydrogen bonding.

## Experimental

Treatment of 1-deoxy-3-*C*-methyl-D-psicose **2** (Jones *et al.* in preparation) derived from 2-*C*-methyl-D-ribono-1,4-lactone **1** (Hotchkiss *et al.*, 2006), with sodium cyanide, gave a mixture of 2,6-anhydro derivative **3** and lactone **4** (Curran *et al.* 2007) (Fig. 3). X-ray analysis firmly established the structure of the title compound as the 2,6-anhydro furanose **3**. m.p. 321–326 K; $[\alpha]_D^{22}$ −47.0 (*c*, 1.0 in acetone).

## Refinement

In the absence of significant anomalous scattering, Friedel pairs were merged and the absolute configuration was assigned from the starting material.

The H atoms were all located in a difference map, but those attached to carbon atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry (C—H in the range 0.93–0.98, O—H = 0.82 Å) and $U_{iso}$(H) (in the range 1.2–1.5 times $U_{eq}$ of the parent atom), after which the positions were refined with riding constraints.

## Figures

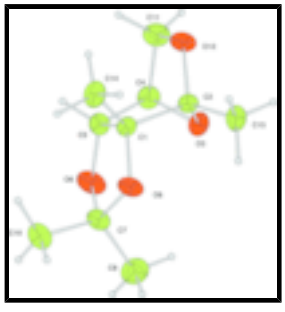


Fig. 1. The title compound with displacement ellipsoids drawn at the 50% probability level. H atoms are shown as spheres of arbitary radius.

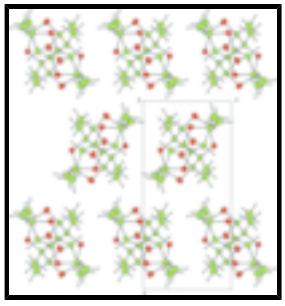


Fig. 2. Packing of the title compound projected along the *b*-axis.

Fig. 3. The reaction scheme.

**2,6-Anhydro-1-deoxy-3,4-*O*-isopropylidene-3-*C*-methyl-β-D– *ribo*-hex-2-ulofuranose**

*Crystal data*

| | |
|---|---|
| $C_{10}H_{16}O_4$ | $F_{000}$ = 432 |
| $M_r$ = 200.23 | $D_x$ = 1.277 Mg m$^{-3}$ |
| Orthorhombic, $P2_12_12_1$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: P 2ac 2ab | Cell parameters from 1321 reflections |
| $a$ = 6.6307 (2) Å | θ = 5–27º |
| $b$ = 11.1029 (4) Å | μ = 0.10 mm$^{-1}$ |
| $c$ = 14.1409 (5) Å | $T$ = 150 K |
| $V$ = 1041.05 (6) Å$^3$ | Plate, colourless |
| $Z$ = 4 | 0.60 × 0.50 × 0.10 mm |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 1160 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.045 |
| $T$ = 150 K | $\theta_{max}$ = 27.5º |
| ω scans | $\theta_{min}$ = 5.3º |
| Absorption correction: multi-scan DENZO/SCALEPACK (Otwinowski & Minor, 1997) | $h$ = −8→8 |
| $T_{min}$ = 0.95, $T_{max}$ = 1.00 | $k$ = −14→14 |
| 6557 measured reflections | $l$ = −18→18 |
| 1370 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)]$ = 0.031 | $w = 1/[\sigma^2(F^2) + (0.03P)^2 + 0.18P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$ |
| $wR(F^2)$ = 0.074 | $(\Delta/\sigma)_{max}$ = 0.0002 |

| | |
|---|---|
| $S$ = 0.93 | $\Delta\rho_{max}$ = 0.24 e Å$^{-3}$ |
| 1370 reflections | $\Delta\rho_{min}$ = −0.19 e Å$^{-3}$ |
| 127 parameters | Extinction correction: None |
| Primary atom site location: structure-invariant direct methods | |

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

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | 0.3568 (2) | 0.21011 (14) | 0.20100 (10) | 0.0212 |
| C2 | 0.4422 (2) | 0.31928 (14) | 0.14619 (10) | 0.0217 |
| O3 | 0.56240 (17) | 0.37828 (10) | 0.21534 (8) | 0.0263 |
| C4 | 0.6883 (2) | 0.27622 (15) | 0.23612 (11) | 0.0272 |
| C5 | 0.5351 (2) | 0.18346 (15) | 0.26887 (10) | 0.0245 |
| O6 | 0.46475 (18) | 0.21350 (13) | 0.36068 (7) | 0.0352 |
| C7 | 0.2509 (3) | 0.22921 (16) | 0.35888 (11) | 0.0270 |
| O8 | 0.19903 (16) | 0.25108 (11) | 0.26189 (7) | 0.0283 |
| C9 | 0.1985 (3) | 0.34073 (17) | 0.41531 (13) | 0.0403 |
| C10 | 0.1483 (3) | 0.11679 (18) | 0.39421 (13) | 0.0396 |
| C11 | 0.7600 (3) | 0.24199 (19) | 0.13709 (12) | 0.0353 |
| O12 | 0.58629 (17) | 0.27261 (11) | 0.07985 (8) | 0.0317 |
| C13 | 0.2974 (3) | 0.40276 (16) | 0.09881 (12) | 0.0305 |
| C14 | 0.2857 (3) | 0.10662 (15) | 0.13904 (12) | 0.0308 |
| H41 | 0.8009 | 0.2937 | 0.2816 | 0.0355* |
| H51 | 0.5810 | 0.0978 | 0.2636 | 0.0310* |
| H91 | 0.0522 | 0.3542 | 0.4084 | 0.0621* |
| H92 | 0.2310 | 0.3255 | 0.4825 | 0.0614* |
| H93 | 0.2804 | 0.4099 | 0.3910 | 0.0617* |
| H101 | 0.0017 | 0.1299 | 0.3883 | 0.0594* |
| H102 | 0.1804 | 0.1039 | 0.4613 | 0.0595* |
| H103 | 0.1903 | 0.0467 | 0.3550 | 0.0594* |
| H111 | 0.7881 | 0.1541 | 0.1327 | 0.0480* |
| H112 | 0.8841 | 0.2875 | 0.1191 | 0.0473* |
| H131 | 0.3743 | 0.4706 | 0.0698 | 0.0479* |
| H132 | 0.1972 | 0.4328 | 0.1459 | 0.0487* |
| H133 | 0.2281 | 0.3599 | 0.0465 | 0.0489* |
| H141 | 0.2242 | 0.0438 | 0.1796 | 0.0485* |
| H142 | 0.4037 | 0.0743 | 0.1055 | 0.0487* |
| H143 | 0.1825 | 0.1383 | 0.0923 | 0.0481* |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0180 (7) | 0.0224 (8) | 0.0231 (7) | 0.0020 (7) | −0.0018 (6) | −0.0006 (7) |
| C2 | 0.0217 (7) | 0.0228 (8) | 0.0207 (7) | 0.0003 (7) | −0.0004 (7) | −0.0020 (6) |
| O3 | 0.0255 (6) | 0.0219 (5) | 0.0316 (6) | −0.0021 (5) | −0.0053 (5) | −0.0018 (5) |
| C4 | 0.0185 (7) | 0.0304 (9) | 0.0326 (8) | 0.0012 (8) | −0.0031 (7) | 0.0014 (8) |
| C5 | 0.0219 (8) | 0.0267 (8) | 0.0248 (7) | 0.0014 (8) | −0.0033 (7) | 0.0028 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O6 | 0.0274 (6) | 0.0560 (8) | 0.0221 (5) | 0.0010 (7) | −0.0034 (5) | 0.0044 (6) |
| C7 | 0.0257 (8) | 0.0348 (10) | 0.0204 (7) | −0.0004 (8) | −0.0008 (6) | 0.0021 (7) |
| O8 | 0.0205 (5) | 0.0431 (7) | 0.0214 (5) | 0.0029 (6) | 0.0011 (5) | 0.0025 (5) |
| C9 | 0.0467 (12) | 0.0394 (10) | 0.0347 (9) | 0.0016 (10) | −0.0041 (9) | −0.0070 (8) |
| C10 | 0.0441 (11) | 0.0375 (10) | 0.0372 (9) | −0.0035 (10) | 0.0063 (9) | 0.0071 (9) |
| C11 | 0.0221 (8) | 0.0443 (11) | 0.0394 (10) | 0.0043 (9) | 0.0037 (8) | 0.0026 (9) |
| O12 | 0.0273 (6) | 0.0415 (7) | 0.0262 (5) | 0.0031 (6) | 0.0057 (5) | 0.0001 (6) |
| C13 | 0.0350 (10) | 0.0261 (8) | 0.0304 (8) | 0.0022 (9) | −0.0060 (8) | 0.0045 (7) |
| C14 | 0.0323 (9) | 0.0254 (8) | 0.0348 (9) | −0.0029 (8) | −0.0085 (8) | −0.0010 (8) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C2 | 1.546 (2) | C7—C10 | 1.507 (3) |
| C1—C5 | 1.551 (2) | C9—H91 | 0.986 |
| C1—O8 | 1.4293 (18) | C9—H92 | 0.988 |
| C1—C14 | 1.520 (2) | C9—H93 | 1.001 |
| C2—O3 | 1.4214 (18) | C10—H101 | 0.986 |
| C2—O12 | 1.4357 (18) | C10—H102 | 0.983 |
| C2—C13 | 1.493 (2) | C10—H103 | 0.995 |
| O3—C4 | 1.438 (2) | C11—O12 | 1.448 (2) |
| C4—C5 | 1.519 (2) | C11—H111 | 0.995 |
| C4—C11 | 1.527 (2) | C11—H112 | 0.998 |
| C4—H41 | 1.004 | C13—H131 | 0.998 |
| C5—O6 | 1.4193 (19) | C13—H132 | 0.999 |
| C5—H51 | 1.001 | C13—H133 | 0.993 |
| O6—C7 | 1.429 (2) | C14—H141 | 0.991 |
| C7—O8 | 1.4348 (18) | C14—H142 | 0.983 |
| C7—C9 | 1.514 (2) | C14—H143 | 1.014 |
| C2—C1—C5 | 100.40 (12) | C7—O8—C1 | 110.27 (12) |
| C2—C1—O8 | 108.70 (12) | C7—C9—H91 | 107.3 |
| C5—C1—O8 | 104.24 (11) | C7—C9—H92 | 108.4 |
| C2—C1—C14 | 114.66 (13) | H91—C9—H92 | 109.6 |
| C5—C1—C14 | 116.67 (13) | C7—C9—H93 | 108.8 |
| O8—C1—C14 | 111.16 (13) | H91—C9—H93 | 112.5 |
| C1—C2—O3 | 102.81 (11) | H92—C9—H93 | 110.0 |
| C1—C2—O12 | 106.76 (12) | C7—C10—H101 | 107.1 |
| O3—C2—O12 | 104.05 (12) | C7—C10—H102 | 110.0 |
| C1—C2—C13 | 118.43 (14) | H101—C10—H102 | 108.4 |
| O3—C2—C13 | 112.53 (13) | C7—C10—H103 | 109.6 |
| O12—C2—C13 | 111.02 (13) | H101—C10—H103 | 110.2 |
| C2—O3—C4 | 95.90 (11) | H102—C10—H103 | 111.4 |
| O3—C4—C5 | 102.05 (13) | C4—C11—O12 | 101.92 (12) |
| O3—C4—C11 | 100.93 (13) | C4—C11—H111 | 111.1 |
| C5—C4—C11 | 108.61 (15) | O12—C11—H111 | 110.1 |
| O3—C4—H41 | 114.2 | C4—C11—H112 | 111.4 |
| C5—C4—H41 | 115.7 | O12—C11—H112 | 113.2 |
| C11—C4—H41 | 113.8 | H111—C11—H112 | 109.0 |
| C1—C5—C4 | 101.05 (12) | C11—O12—C2 | 104.40 (11) |
| C1—C5—O6 | 105.70 (12) | C2—C13—H131 | 108.9 |

| | | | |
|---|---|---|---|
| C4—C5—O6 | 109.83 (14) | C2—C13—H132 | 109.6 |
| C1—C5—H51 | 111.5 | H131—C13—H132 | 111.3 |
| C4—C5—H51 | 114.7 | C2—C13—H133 | 109.5 |
| O6—C5—H51 | 113.0 | H131—C13—H133 | 107.0 |
| C5—O6—C7 | 109.79 (12) | H132—C13—H133 | 110.5 |
| O6—C7—O8 | 106.00 (12) | C1—C14—H141 | 109.1 |
| O6—C7—C9 | 108.58 (16) | C1—C14—H142 | 107.9 |
| O8—C7—C9 | 108.08 (14) | H141—C14—H142 | 110.5 |
| O6—C7—C10 | 109.95 (16) | C1—C14—H143 | 108.8 |
| O8—C7—C10 | 110.41 (14) | H141—C14—H143 | 110.0 |
| C9—C7—C10 | 113.52 (15) | H142—C14—H143 | 110.5 |

**Fig. 1**

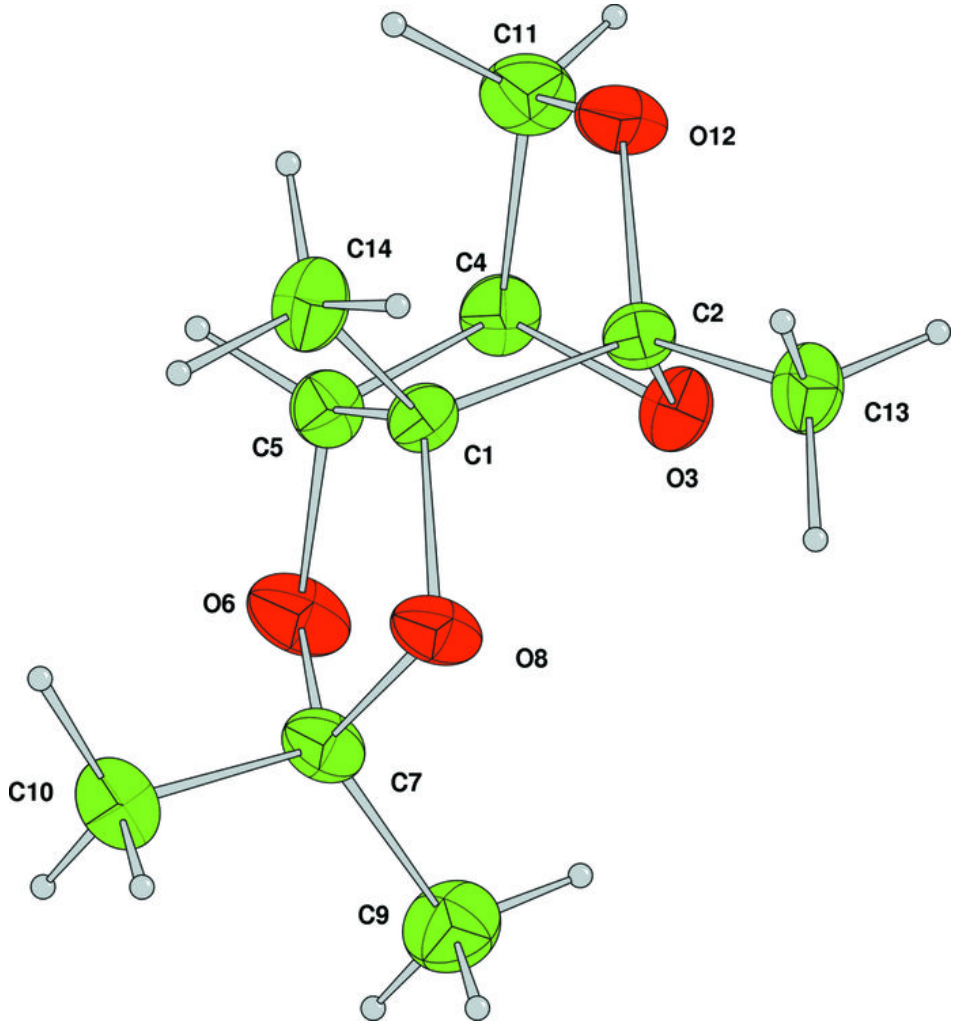


**Fig. 2**

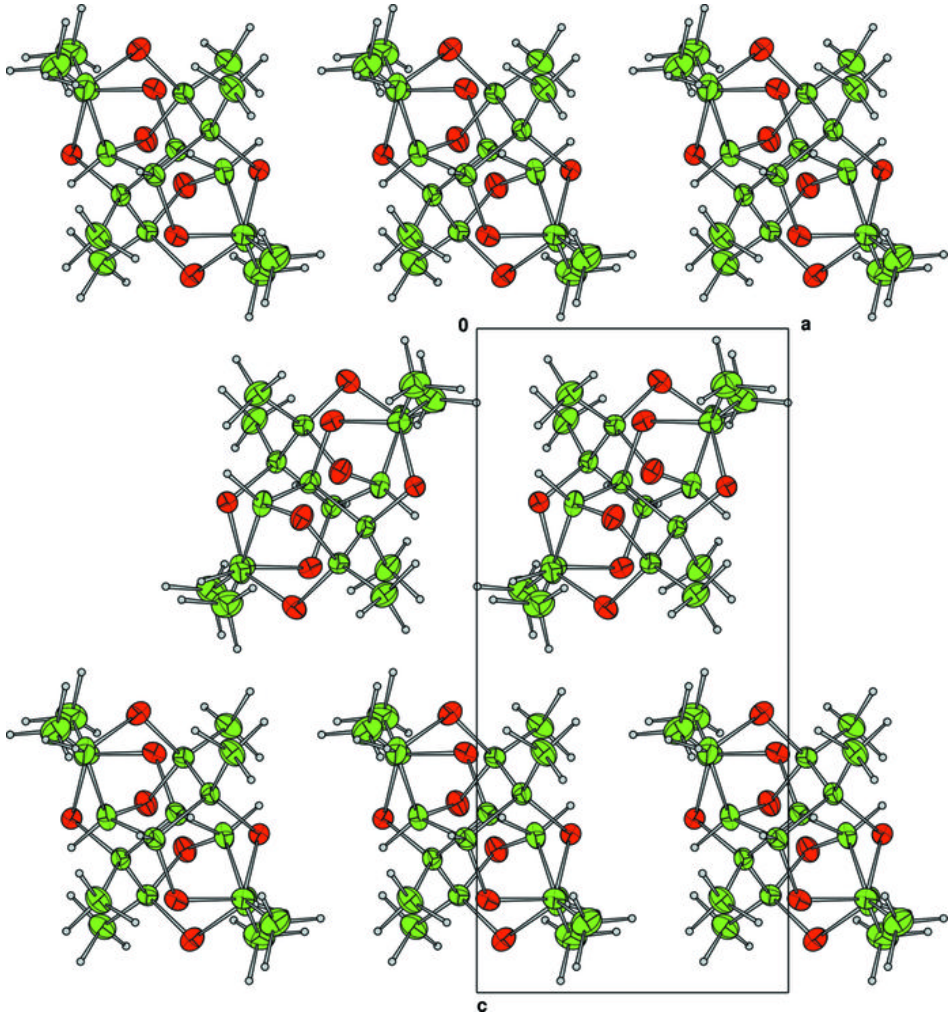
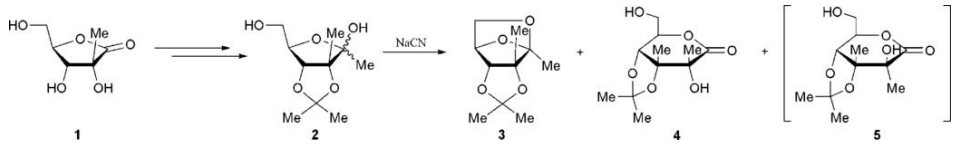


**Fig. 3**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