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2,3:5,6-Di-*O*-isopropylidene-2-*C*-hydroxymethyl-D-talono-1,4-lactone

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.073
Data-to-parameter ratio = 10.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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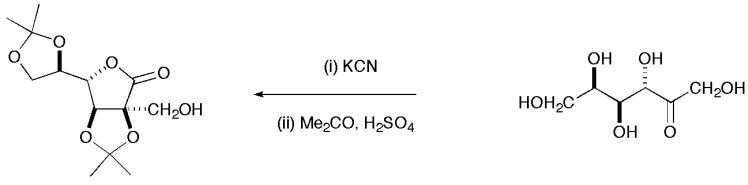
The title diacetonide, $C_{13}H_{20}O_7$, readily available in quantity from D-tagatose, is likely to be a useful carbohydrate starting material. The current structure analysis resolves any ambiguities arising from the synthetic route over the configuration at the new chiral centre and the size of the lactone ring, but otherwise shows no unusual features.

Comment

Sugars provide the largest group of readily available chiral building blocks and bio-active scaffolds (Lichtenthaler & Peters, 2004; Bols, 1996). Although little studied since initial investigations by Kiliani (Kiliani, 1885, 1928; Gorin & Perlin, 1958), the reaction of ketoses with aqueous potassium cyanide easily produces a mixture of branched sugar lactones under aqueous conditions. The reaction of the lactones produced from D-fructose and L-sorbose with acetone in the presence of acid gives rise to readily crystallized diacetonides likely to furnish a new family of carbohydrate-derived chiral building blocks with branched carbon chains (Hotchkiss *et al.*, 2004). The full exploitation of this technology requires access to a wide range of ketoses; in the past, only D-fructose and L-sorbose have been readily available. However, the impetus for the development of low calorie sweeteners has led to an extensive biotechnology which provides almost any hexose by combinations of microbial oxidations and enzyme-catalysed epimerizations (Granstrom *et al.*, 2004). Thus D-tagatose (2) (see scheme), previously considered a rare sugar, is prepared on an industrial scale for use in soft drinks and ready-to-eat cereals (Skytte, 2002).



(1) (2)

The Kiliani reaction of cyanide with D-tagatose (2) gave an excellent yield of different amounts of two lactones. Extraction of this mixture with acetone in the presence of sulfuric acid gave a mixture of diacetonides; the major product (1) was easily isolated as a crystalline material. The current structure analysis of (1) resolves any ambiguities arising from the synthetic route over the configuration at the new chiral centre and the size of the lactone ring. The diacetonide (1) is likely to be a useful starting material for the preparation of a number of branched sugar mimics.

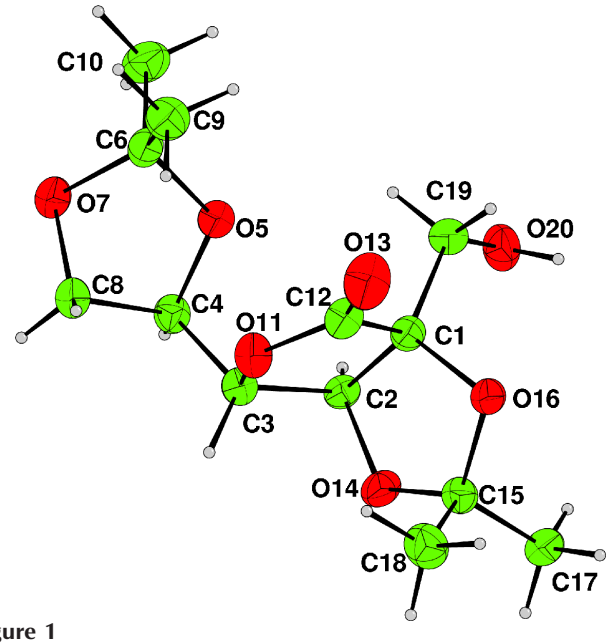


Figure 1
The title compound, with displacement ellipsoids drawn at the 50% probability level. H atoms are of arbitrary radii.

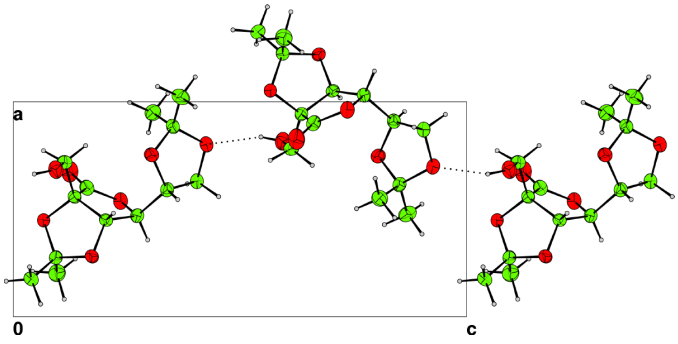


Figure 2
Packing diagram viewed along the *b* axis. Molecules are linked into ribbons by hydrogen bonds (dashed lines).

The crystal and molecular structures of (1) show no unusual features. As expected for sugar derivatives, hydrogen bonding occurs between molecules, in this case, linking molecules into ribbons parallel to the *c* axis.

Experimental

The title compound was crystallized from diethyl ether by inward diffusion of *n*-hexane to yield plate-like colourless crystals. These did not cleave well, leading to the use of a large crystal. The multi-scan technique was used to correct for changes in illuminated volume.

Crystal data

$C_{13}H_{20}O_7$
M_r = 288.30
Orthorhombic, $P2_12_12_1$
a = 7.8609 (3) Å
b = 10.7470 (4) Å
c = 16.5516 (6) Å
V = 1398.30 (9) Å^3
Z = 4
D_x = 1.369 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1577 reflections
θ = 5–27°
μ = 0.11 mm^{-1}
T = 190 K
Block, colourless
0.65 × 0.25 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.97, T_{max} = 0.98
2981 measured reflections
1804 independent reflections
1544 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 27.5°
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -21 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.074
S = 0.92
1803 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F) + 0.028 + 0.385P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

All H atoms were observed in a difference electron-density map. The hydroxyl H atom was placed as found and the others were placed geometrically with isotropic displacement parameters related to the U_{eq} values of the adjacent atoms. The H-atom positions and U_{iso} values were regularized by refinement with slack restraints and the refinement completed with H-atom riding constraints [C—H = 0.98±0.02 Å; U_{iso}(H) = U_{eq}(C) ± 0.002 Å^2; O—H no restraints]. In the absence of significant anomalous scattering effects, Friedel pairs were merged.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO*/*SCALEPACK*; program(s) used to solve structure: *SIR*92 (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*.

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