Crystal structure of ethyl (3a*R*,4*R*,5*R*,6*R*,7a*R*)-4,5-diacetoxy-6-acetoxymethyl-2-oxy-3a,5,6,7a-tetrahydro-4*H*-pyrano[3,2-*d*]isoxazole-3-carboxylate, $C_{16}H_{21}NO_{11}$

K. Peters*[I], E.-M. Peters[I], B. G. Kim[II] and T. Linker[II]

[I] Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, 70506 Stuttgart, Germany
[II] Universität Potsdam, Institut für Organische Chemie, Karl-Liebknecht-Straße 24-25, 14476 Golm, Germany

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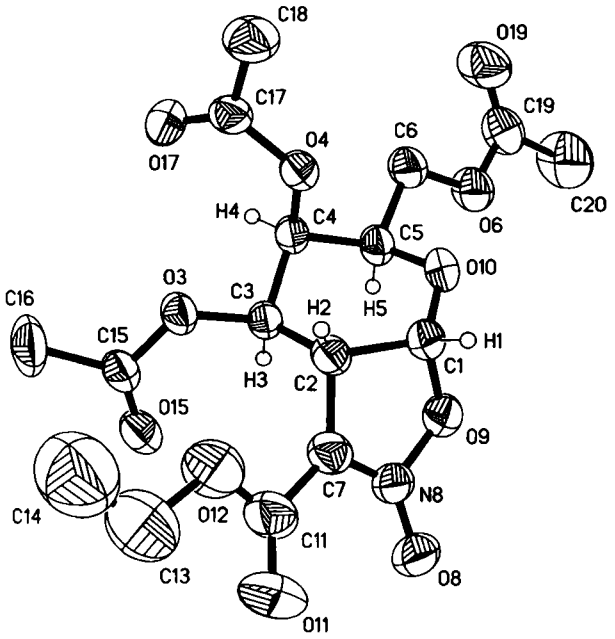

Abstract

$C_{16}H_{21}NO_{11}$, monoclinic, $P12_11$ (no. 4), $a = 8.181(1)$ Å, $b = 11.604(2)$ Å, $c = 10.193(1)$ Å, $\beta = 101.59(1)°$, $V = 947.9$ Å^3, $Z = 2$, $R_{gt}(F) = 0.068$, $wR_{ref}(F^2) = 0.177$, $T = 293$ K.

Source of material

The title compound was prepared by radical addition of ethyl nitroacetate to tri-*O*-acetyl-D-galactal [1].

Discussion

The compound crystallizes non-centrosymmetrically. The absolute configuration of the molecule could not be determined, but the presented chirality of this natural compound is well known.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.15 × 0.7 × 1.65 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.21 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$:	54.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	5218, 4331
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3982
$N(param)_{refined}$:	257
Programs:	SHELXS-97 [2], SHELXL-97 [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	0.4972	0.1458	0.6262	0.08
H(2)	2*a*	0.3143	0.2185	0.7438	0.08
H(3)	2*a*	0.0361	0.2006	0.5252	0.08
H(4)	2*a*	0.0665	0.3943	0.4660	0.08
H(5)	2*a*	0.1848	0.2479	0.3514	0.08
H(6A)	2*a*	0.2297	0.4489	0.2963	0.08
H(6B)	2*a*	0.4120	0.4347	0.3811	0.08
H(13A)	2*a*	−0.0456	0.0260	0.9602	0.08
H(13B)	2*a*	0.1335	0.0038	1.0454	0.08
H(14A)	2*a*	0.0101	0.1184	1.1521	0.08
H(14B)	2*a*	−0.0424	0.1986	1.0270	0.08
H(14C)	2*a*	0.1458	0.1891	1.0978	0.08
H(16A)	2*a*	−0.3122	0.2682	0.7929	0.08
H(16B)	2*a*	−0.2896	0.3762	0.7059	0.08
H(16C)	2*a*	−0.1592	0.3505	0.8378	0.08
H(18A)	2*a*	0.2994	0.6438	0.7819	0.08
H(18B)	2*a*	0.3838	0.6499	0.6569	0.08
H(18C)	2*a*	0.4340	0.5523	0.7637	0.08
H(20A)	2*a*	0.4707	0.3889	−0.0595	0.08
H(20B)	2*a*	0.3897	0.2782	−0.0115	0.08
H(20C)	2*a*	0.5781	0.3061	0.0452	0.08

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*a*	0.3877(3)	0.1601(3)	0.5691(3)	0.035(1)	0.043(1)	0.057(1)	−0.0006(9)	0.012(1)	−0.003(1)
C(2)	2*a*	0.2606(3)	0.1808(3)	0.6602(2)	0.037(1)	0.043(1)	0.044(1)	−0.0031(9)	0.0105(9)	−0.0004(9)
C(3)	2*a*	0.1091(3)	0.2510(3)	0.5885(2)	0.033(1)	0.037(1)	0.041(1)	−0.0026(8)	0.0125(8)	−0.0060(9)
O(3)	2*a*	0.0167(2)	0.2954(2)	0.6834(2)	0.0445(9)	0.0477(9)	0.0453(9)	−0.0065(7)	0.0217(7)	−0.0097(7)
C(4)	2*a*	0.1642(3)	0.3510(3)	0.5121(2)	0.033(1)	0.040(1)	0.040(1)	0.0000(8)	0.0113(8)	−0.0027(9)

* Correspondence author (e-mail: karlpet@gmx.de)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(4)	2*a*	0.2760(2)	0.4264(2)	0.6001(2)	0.0378(8)	0.0434(9)	0.0477(9)	−0.0045(7)	0.0127(7)	−0.0042(7)
C(5)	2*a*	0.2575(3)	0.3015(3)	0.4106(2)	0.040(1)	0.046(1)	0.045(1)	−0.001(1)	0.0175(9)	−0.004(1)
C(6)	2*a*	0.3181(4)	0.3938(3)	0.3279(3)	0.055(1)	0.051(1)	0.050(1)	−0.004(1)	0.025(1)	−0.003(1)
O(6)	2*a*	0.3682(3)	0.3373(3)	0.2154(2)	0.071(1)	0.062(1)	0.051(1)	−0.006(1)	0.0324(9)	−0.0054(9)
C(7)	2*a*	0.2156(3)	0.0595(3)	0.6856(3)	0.040(1)	0.047(1)	0.058(2)	−0.002(1)	0.007(1)	0.008(1)
N(8)	2*a*	0.2523(3)	−0.0096(3)	0.5933(3)	0.035(1)	0.041(1)	0.075(2)	0.0012(8)	0.007(1)	0.003(1)
O(8)	2*a*	0.2377(3)	−0.1125(3)	0.5732(3)	0.051(1)	0.042(1)	0.115(2)	−0.0005(9)	0.015(1)	−0.002(1)
O(9)	2*a*	0.3286(2)	0.0523(2)	0.5002(2)	0.047(1)	0.046(1)	0.066(1)	−0.0011(8)	0.0190(9)	−0.0095(9)
O(10)	2*a*	0.4057(2)	0.2418(2)	0.4772(2)	0.0362(8)	0.051(1)	0.059(1)	0.0017(7)	0.0202(7)	0.0007(8)
C(11)	2*a*	0.1469(5)	0.0127(4)	0.7963(4)	0.067(2)	0.062(2)	0.077(2)	0.005(2)	0.022(2)	0.028(2)
O(11)	2*a*	0.1110(5)	−0.0854(3)	0.8091(4)	0.131(3)	0.070(2)	0.132(3)	−0.012(2)	0.061(2)	0.032(2)
O(12)	2*a*	0.1339(5)	0.0958(3)	0.8819(3)	0.113(2)	0.083(2)	0.068(2)	0.008(2)	0.039(2)	0.024(1)
C(13)	2*a*	0.062(1)	0.0614(7)	0.9937(7)	0.143(3)	0.124(3)	0.107(3)	−0.001(3)	0.055(2)	0.025(2)
C(14)	2*a*	0.043(1)	0.1477(9)	1.0730(8)	0.159(4)	0.157(4)	0.138(3)	−0.016(3)	0.047(3)	−0.002(3)
C(15)	2*a*	−0.1323(3)	0.2457(3)	0.6843(3)	0.047(1)	0.051(1)	0.052(1)	−0.006(1)	0.026(1)	−0.003(1)
O(15)	2*a*	−0.1769(3)	0.1574(3)	0.6305(3)	0.056(1)	0.058(1)	0.081(1)	−0.017(1)	0.034(1)	−0.011(1)
C(16)	2*a*	−0.2323(5)	0.3165(5)	0.7622(5)	0.073(2)	0.096(3)	0.102(3)	−0.012(2)	0.057(2)	−0.038(3)
C(17)	2*a*	0.2096(4)	0.5236(3)	0.6415(3)	0.054(1)	0.040(1)	0.043(1)	−0.006(1)	0.018(1)	0.0020(9)
O(17)	2*a*	0.0649(3)	0.5453(3)	0.6177(2)	0.056(1)	0.049(1)	0.063(1)	0.0090(9)	0.0209(9)	−0.0017(9)
C(18)	2*a*	0.3437(5)	0.5992(4)	0.7178(4)	0.078(2)	0.059(2)	0.067(2)	−0.020(2)	0.021(2)	−0.018(2)
C(19)	2*a*	0.4210(4)	0.4067(4)	0.1276(3)	0.059(2)	0.076(2)	0.045(1)	−0.003(1)	0.019(1)	0.005(1)
O(19)	2*a*	0.4327(4)	0.5070(3)	0.1398(3)	0.099(2)	0.076(2)	0.069(1)	−0.015(1)	0.036(1)	0.008(1)
C(20)	2*a*	0.4692(8)	0.3388(6)	0.0153(4)	0.131(4)	0.112(4)	0.061(2)	−0.012(3)	0.058(2)	−0.008(2)

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