Crystal structure of 4-*O*-acetyl-2,3-*O*-isopropylidene-D-*lyxo*-5-hexenose-*N*-(*tert*-butoxycarbonyl) hydrazone, $C_{16}H_{26}N_2O_6$

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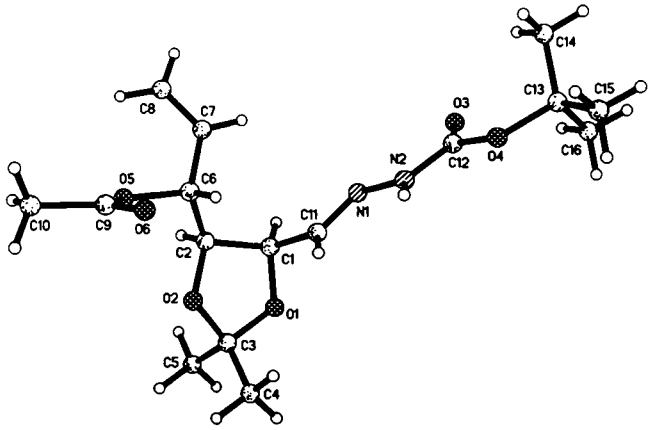

Abstract

$C_{16}H_{26}N_2O_6$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 11.2063(6)$ Å, $b = 12.3253(8)$ Å, $c = 13.973(2)$ Å, $V = 1930.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.048$, $wR_{ref}(F^2) = 0.142$, $T = 293$ K.

Source of material

The title compound has been obtained by heating 2,3-*O*-isopropylidene-D-*lyxo*-5,6-dideoxy-5-hexenose [1] and *N*-(*tert*-butoxycarbonyl) hydrazine in methanol [2-4]. Purification by column chromatography (ethyl acetate/petroleum ether) gave the title compound with a E:Z ratio > 99:1. Crystallization from *i*-PrOH/ethyl acetate gave colourless crystals (mp 402 K – 403 K, $[\alpha]_{20}^{D} = -27$, c = 0.50, MeOH).

Discussion

The atoms of the terminal moieties of the molecule show large displacement parameters which indicate a strong thermal vibration. We observe a nearly linear inter-molecular hydrogen bond interaction between N2-H2 as donor and O2 of the dioxolan moiety as acceptor. The H2···O2 distance is 2.17 Å and the N2–H2···O2 angle is 175.5°. In the *ab*-view the orientation of the molecules shows a fishbone pattern. We also observe nonpolar channels along the *a*-axis made up by the *tert*-butyl groups.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.20 × 0.40 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	7.51 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	134.82°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2412, 2238
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1787
$N(param)_{refined}$:	218
Programs:	SHELXS-97 [5], SHELXL-97 [6]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*a*	1.1019	0.5343	0.4238	0.070
H(2)	4*a*	0.7346	0.5754	0.4976	0.068
H(2A)	4*a*	1.2318	0.6496	0.4922	0.068
H(4A)	4*a*	1.0777	0.9240	0.3294	0.133
H(4B)	4*a*	0.9712	0.8457	0.3523	0.133
H(4C)	4*a*	1.0324	0.8423	0.2515	0.133
H(5A)	4*a*	1.2853	0.8487	0.3185	0.127
H(5B)	4*a*	1.2470	0.7629	0.2415	0.127
H(5C)	4*a*	1.3057	0.7248	0.3377	0.127
H(6)	4*a*	1.0273	0.7010	0.6038	0.075
H(7)	4*a*	1.0683	0.5142	0.6224	0.107
H(8A)	4*a*	1.2448	0.5902	0.7371	0.143
H(8B)	4*a*	1.1907	0.4692	0.7354	0.143
H(10A)	4*a*	1.2965	0.8731	0.7440	0.168
H(10B)	4*a*	1.1971	0.8999	0.8194	0.168
H(10C)	4*a*	1.2154	0.9755	0.7303	0.168
H(11)	4*a*	0.8982	0.6696	0.4606	0.069
H(14A)	4*a*	0.5278	0.3147	0.6381	0.194
H(14B)	4*a*	0.6035	0.2426	0.5684	0.194
H(14C)	4*a*	0.4660	0.2225	0.5788	0.194
H(15A)	4*a*	0.3790	0.4669	0.4658	0.224
H(15B)	4*a*	0.3973	0.4548	0.5765	0.224
H(15C)	4*a*	0.3254	0.3666	0.5198	0.224
H(16A)	4*a*	0.4851	0.3541	0.3561	0.170
H(16B)	4*a*	0.4383	0.2473	0.4041	0.170
H(16C)	4*a*	0.5758	0.2664	0.3921	0.170

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}
N(1)	4*a*	0.8900(2)	0.5141(2)	0.4620(2)	0.042(1)	0.045(1)	0.082(2)	–0.004(1)	0.005(1)	–0.009(2)
O(1)	4*a*	1.0865(3)	0.6624(2)	0.3391(2)	0.078(2)	0.066(2)	0.069(2)	–0.027(2)	0.011(1)	–0.019(2)
C(1)	4*a*	1.0710(3)	0.6085(3)	0.4284(3)	0.053(2)	0.040(2)	0.083(2)	–0.006(2)	0.009(2)	–0.014(2)
N(2)	4*a*	0.7701(2)	0.5157(2)	0.4834(2)	0.041(1)	0.039(1)	0.090(2)	–0.002(1)	0.008(1)	–0.004(2)
O(2)	4*a*	1.1391(2)	0.7807(2)	0.4572(2)	0.062(1)	0.047(1)	0.069(1)	–0.010(1)	–0.002(1)	–0.008(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(2)	4*a*	1.1489(3)	0.6744(3)	0.4966(3)	0.046(2)	0.044(2)	0.079(2)	–0.003(1)	0.004(2)	–0.008(2)
O(3)	4*a*	0.7522(2)	0.3337(2)	0.4657(3)	0.054(1)	0.047(1)	0.181(3)	–0.001(1)	0.014(2)	–0.013(2)
C(3)	4*a*	1.1316(3)	0.7696(3)	0.3545(3)	0.065(2)	0.053(2)	0.066(2)	–0.010(2)	0.003(2)	–0.013(2)
O(4)	4*a*	0.5957(2)	0.4407(2)	0.5004(2)	0.044(1)	0.054(1)	0.114(2)	–0.007(1)	0.016(1)	–0.015(2)
C(4)	4*a*	1.0455(5)	0.8528(4)	0.3187(3)	0.099(3)	0.083(3)	0.083(3)	0.009(3)	–0.019(2)	–0.010(3)
O(5)	4*a*	1.1850(2)	0.7536(2)	0.6495(2)	0.064(1)	0.064(2)	0.078(2)	–0.006(1)	–0.009(1)	–0.009(2)
C(5)	4*a*	1.2535(4)	0.7772(4)	0.3089(4)	0.076(2)	0.082(3)	0.095(3)	–0.023(2)	0.025(2)	–0.005(3)
C(6)	4*a*	1.1104(3)	0.6765(3)	0.6003(3)	0.056(2)	0.054(2)	0.079(2)	–0.006(2)	0.003(2)	–0.010(2)
O(6)	4*a*	1.0268(3)	0.8492(3)	0.6939(3)	0.092(2)	0.096(2)	0.158(3)	0.013(2)	0.008(2)	–0.047(3)
C(7)	4*a*	1.1198(5)	0.5675(4)	0.6452(4)	0.112(4)	0.070(3)	0.084(3)	–0.010(3)	0.010(3)	0.007(3)
C(8)	4*a*	1.1917(7)	0.5398(5)	0.7120(5)	0.149(5)	0.092(4)	0.117(4)	0.020(4)	0.034(4)	0.031(4)
C(9)	4*a*	1.1303(5)	0.8351(4)	0.6971(3)	0.093(3)	0.068(2)	0.069(2)	–0.012(3)	0.006(2)	–0.008(2)
C(10)	4*a*	1.2177(6)	0.9019(5)	0.7527(4)	0.144(5)	0.111(4)	0.081(3)	–0.042(4)	–0.003(3)	–0.031(3)
C(11)	4*a*	0.9410(3)	0.6055(3)	0.4526(3)	0.044(2)	0.047(2)	0.082(2)	–0.004(1)	0.005(2)	–0.010(2)
C(12)	4*a*	0.7093(3)	0.4196(3)	0.4815(3)	0.039(1)	0.045(2)	0.079(2)	–0.000(1)	0.004(2)	–0.000(2)
C(13)	4*a*	0.5045(3)	0.3543(3)	0.4997(3)	0.046(2)	0.062(2)	0.084(2)	–0.018(2)	0.007(2)	–0.013(2)
C(14)	4*a*	0.5275(5)	0.2767(5)	0.5781(5)	0.114(4)	0.157(5)	0.117(4)	–0.079(4)	–0.025(3)	0.049(4)
C(15)	4*a*	0.3914(4)	0.4162(5)	0.5170(6)	0.053(2)	0.112(4)	0.283(9)	–0.015(3)	0.043(4)	–0.067(5)
C(16)	4*a*	0.5006(4)	0.3008(5)	0.4046(4)	0.089(3)	0.159(5)	0.093(3)	–0.056(4)	0.000(3)	–0.021(4)

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