Crystal structure of (3a*R**,6a*R**,9a*R**,9b*R**)-spiro[(1′,3′-dioxolane)-2′,6-(decahydro-9a-methoxycarbonyl-9-oxoazuleno[4,5-*c*]furan-1(3*H*)-one], $C_2H_4O_2[C_{12}H_{13}O_3]COOCH_3$

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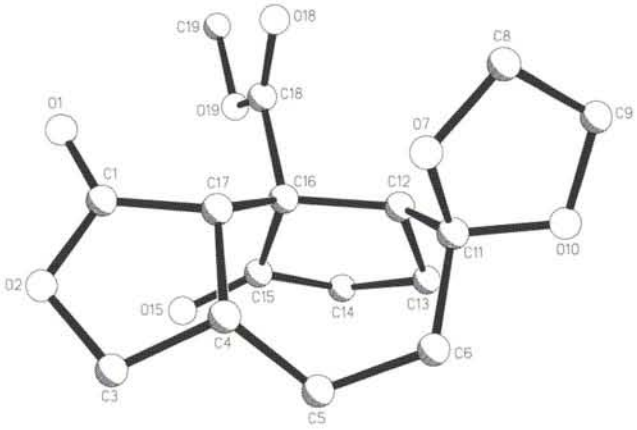

Abstract

$C_{16}H_{20}O_7$, orthorhombic, $P2_12_12_1$ (No. 19), a = 10.673(1) Å, b = 10.783(1) Å, c = 13.504(1) Å, V = 1554.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.048, $R_w(F)$ = 0.048, T = 293 K.

Source of material

The title compound was prepared as described in [1].

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.5 x 0.55 x 0.65 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.10 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2684, 2485
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 2310
$N(param)_{refined}$:	209
Program:	SHELXTL-plus [2]

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

Atom	Site	x	y	z	U_{iso}
H(3A)	4*a*	0.3987(3)	0.6656(3)	0.8816(3)	0.08
H(3B)	4*a*	0.3175(3)	0.6878(3)	0.7865(3)	0.08
H(4)	4*a*	0.3915(3)	0.8774(2)	0.9183(2)	0.08
H(5A)	4*a*	0.4648(3)	0.8694(3)	0.7586(3)	0.08
H(5B)	4*a*	0.3272(3)	0.8973(3)	0.7247(3)	0.08
H(6A)	4*a*	0.4307(3)	1.0814(3)	0.7215(3)	0.08
H(6B)	4*a*	0.4837(3)	1.0597(3)	0.8284(3)	0.08
H(8A)	4*a*	0.1814(5)	1.2411(3)	0.9808(3)	0.08
H(8B)	4*a*	0.3163(5)	1.2714(3)	1.0198(3)	0.08
H(9A)	4*a*	0.3514(5)	1.3862(3)	0.8916(3)	0.08
H(9B)	4*a*	0.2114(5)	1.3687(3)	0.8593(3)	0.08
H(12)	4*a*	0.1209(2)	1.1532(2)	0.8194(2)	0.08
H(13A)	4*a*	0.2445(3)	1.0771(3)	0.6477(2)	0.08
H(13B)	4*a*	0.1300(3)	1.1680(3)	0.6523(2)	0.08
H(14A)	4*a*	0.0980(3)	0.9494(3)	0.5846(2)	0.08
H(14B)	4*a*	−0.0101(3)	1.0130(3)	0.6446(2)	0.08
H(17)	4*a*	0.2051(2)	0.9437(2)	0.9517(2)	0.08
H(19A)	4*a*	−0.2873(2)	0.9375(3)	0.8272(3)	0.08
H(19B)	4*a*	−0.2366(2)	1.0568(3)	0.8799(3)	0.08
H(19C)	4*a*	−0.2292(2)	0.9281(3)	0.9335(3)	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}
O(1)	4*a*	0.0412(2)	0.7467(2)	0.9644(2)	0.077(1)	0.067(1)	0.079(1)	−0.013(1)	0.014(1)	0.016(1)
C(1)	4*a*	0.1414(3)	0.7620(3)	0.9261(2)	0.066(2)	0.048(1)	0.052(1)	−0.003(1)	−0.007(1)	0.006(1)
O(2)	4*a*	0.2173(2)	0.6661(2)	0.9081(2)	0.078(1)	0.0434(9)	0.088(2)	0.005(1)	−0.003(1)	0.004(1)
C(3)	4*a*	0.3258(3)	0.7065(3)	0.8557(3)	0.069(2)	0.055(2)	0.085(2)	0.019(2)	−0.008(2)	−0.008(2)
C(4)	4*a*	0.3350(3)	0.8479(2)	0.8683(2)	0.044(1)	0.051(1)	0.068(2)	0.011(1)	−0.008(1)	−0.005(1)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(5)	4*a*	0.3879(3)	0.9100(3)	0.7761(3)	0.039(1)	0.068(2)	0.086(2)	0.010(1)	0.012(1)	−0.003(2)
C(6)	4*a*	0.4122(3)	1.0490(3)	0.7861(3)	0.037(1)	0.074(2)	0.103(2)	−0.006(1)	0.002(2)	0.009(2)
O(7)	4*a*	0.3068(2)	1.1255(2)	0.9316(2)	0.081(1)	0.051(1)	0.069(1)	−0.005(1)	−0.030(1)	−0.001(1)
C(8)	4*a*	0.2687(5)	1.2435(3)	0.9638(3)	0.152(4)	0.057(2)	0.087(2)	−0.005(2)	−0.026(3)	−0.011(2)
C(9)	4*a*	0.2873(5)	1.3264(3)	0.8769(3)	0.128(3)	0.048(1)	0.096(3)	−0.004(2)	−0.035(3)	0.005(2)
O(10)	4*a*	0.3264(2)	1.2512(2)	0.7986(2)	0.079(1)	0.049(1)	0.087(2)	−0.019(1)	−0.012(1)	0.010(1)
C(11)	4*a*	0.3035(3)	1.1254(3)	0.8266(2)	0.046(1)	0.050(1)	0.065(2)	−0.009(1)	−0.010(1)	0.005(1)
C(12)	4*a*	0.1719(2)	1.0894(2)	0.7906(2)	0.043(1)	0.040(1)	0.047(1)	0.002(1)	−0.003(1)	0.001(1)
C(13)	4*a*	0.1630(3)	1.0906(3)	0.6758(2)	0.060(2)	0.070(2)	0.047(1)	−0.007(2)	−0.003(1)	0.011(1)
C(14)	4*a*	0.0751(3)	0.9845(3)	0.6474(2)	0.063(2)	0.070(2)	0.040(1)	0.000(2)	−0.006(1)	−0.004(1)
C(15)	4*a*	0.0922(2)	0.8908(2)	0.7282(2)	0.036(1)	0.052(1)	0.043(1)	−0.001(1)	0.001(1)	−0.009(1)
O(15)	4*a*	0.0906(2)	0.7787(2)	0.7212(1)	0.065(1)	0.052(1)	0.062(1)	−0.004(1)	−0.001(1)	−0.016(1)
C(16)	4*a*	0.1203(2)	0.9603(2)	0.8259(2)	0.0319(9)	0.040(1)	0.039(1)	0.0013(9)	0.0003(8)	−0.005(1)
C(17)	4*a*	0.2014(2)	0.8841(2)	0.8989(2)	0.042(1)	0.040(1)	0.042(1)	0.000(1)	−0.004(1)	−0.004(1)
C(18)	4*a*	−0.0046(2)	0.9864(2)	0.8803(2)	0.041(1)	0.044(1)	0.045(1)	0.004(1)	0.005(1)	−0.002(1)
O(18)	4*a*	−0.0098(2)	1.0386(2)	0.9585(2)	0.053(1)	0.094(2)	0.056(1)	0.004(1)	0.009(1)	−0.026(1)
O(19)	4*a*	−0.1020(2)	0.9487(2)	0.8282(1)	0.0345(8)	0.065(1)	0.056(1)	0.0025(8)	−0.0002(8)	−0.0075(9)
C(19)	4*a*	−0.2240(2)	0.9694(3)	0.8707(3)	0.034(1)	0.088(2)	0.081(2)	0.006(1)	0.007(1)	−0.007(2)

References

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2. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.