Crystal structure of (3a*R**,6a*R**,9a*R**,9b*R**)-spiro[(1',3'-dioxolane)-2',6-(decahydro-8,8-dimethyl-9-oxoazuleno[4,5-*c*]furan-1(3*H*)-one)], (CH₃)₂C₁₄H₁₆O₅

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Abstract

C₁₆H₂₂O₅, monoclinic, *P*12₁/*n*1 (No. 14), *a* = 10.715(1) Å, *b* = 9.426(1) Å, *c* = 29.529(1) Å, β = 93.82(1)°, *V* = 2975.8 Å³, *Z* = 8, *R*_{gt}(*F*) = 0.060, *R*_w(*F*) = 0.061, *T* = 293 K.

Source of material

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

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Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.5 x 0.55 x 0.65 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	1.00 cm ⁻¹
Diffractometer, scan mode:	Siemens P4, ω
2θ _{max} :	55°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	7642, 6840
Criterion for <i>F</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>F</i> _{obs} > 3 σ(<i>F</i> _{obs}), 5644
<i>N</i> (<i>param</i>) _{refined} :	380
Program:	SHELXTL-plus [2]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(13A)	4e	-0.7216(3)	-0.0517(3)	-0.03645(8)	0.08
H(13B)	4e	-0.6057(3)	-0.1252(3)	-0.01112(8)	0.08
H(14A)	4e	-0.8287(2)	-0.2487(2)	-0.10385(7)	0.08
H(14B)	4e	-0.7782(2)	-0.0949(2)	-0.11121(7)	0.08
H(15A)	4e	-0.6407(2)	-0.1974(2)	-0.16262(7)	0.08
H(15B)	4e	-0.7820(2)	-0.1933(2)	-0.18011(7)	0.08
H(17A)	4e	-0.5471(2)	-0.6268(2)	-0.17829(6)	0.08
H(17B)	4e	-0.6771(2)	-0.6721(2)	-0.16158(6)	0.08
H(110)	4e	-0.7499(2)	-0.5082(2)	-0.08970(5)	0.08
H(111)	4e	-0.5498(2)	-0.4133(2)	-0.13806(5)	0.08
H(112)	4e	-0.5654(2)	-0.3920(2)	-0.04239(6)	0.08
H(113)	4e	-0.5802(2)	-0.1902(2)	-0.08793(7)	0.08
H(15A)	4e	-0.9638(2)	-0.5198(3)	-0.21112(8)	0.08
H(15B)	4e	-0.9491(2)	-0.3544(3)	-0.20874(8)	0.08
H(16A)	4e	-0.8223(3)	-0.3812(4)	-0.26320(9)	0.08
H(16B)	4e	-0.8001(3)	-0.5416(4)	-0.25111(9)	0.08
H(18A)	4e	-0.3540(2)	-0.7180(3)	-0.13329(8)	0.08
H(18B)	4e	-0.3548(2)	-0.7048(3)	-0.08040(8)	0.08
H(18C)	4e	-0.3796(2)	-0.5707(3)	-0.11107(8)	0.08
H(19A)	4e	-0.5230(2)	-0.9059(2)	-0.13584(9)	0.08
H(19B)	4e	-0.6464(2)	-0.8760(2)	-0.11180(9)	0.08
H(19C)	4e	-0.5202(2)	-0.8989(2)	-0.08275(9)	0.08
H(23A)	4e	0.1113(2)	0.8984(3)	0.29680(7)	0.08
H(23B)	4e	0.1664(2)	1.0516(3)	0.29262(7)	0.08
H(24A)	4e	0.1048(2)	0.7734(2)	0.20685(7)	0.08
H(24B)	4e	-0.0064(2)	0.7934(2)	0.23818(7)	0.08
H(25A)	4e	-0.1125(2)	0.9160(2)	0.17657(7)	0.08
H(25B)	4e	-0.0969(2)	0.7529(2)	0.16817(7)	0.08
H(27A)	4e	0.1426(2)	0.9846(2)	0.06386(6)	0.08
H(27B)	4e	0.0351(2)	1.0976(2)	0.06228(6)	0.08
H(210)	4e	0.2391(2)	0.9228(2)	0.15061(6)	0.08
H(211)	4e	0.0161(2)	1.0779(2)	0.14004(6)	0.08
H(212)	4e	0.2180(2)	1.1224(2)	0.20827(6)	0.08
H(213)	4e	0.0225(2)	1.0365(2)	0.22684(6)	0.08
H(15C)	4e	0.0817(2)	0.6262(2)	0.06503(8)	0.08
H(15D)	4e	-0.0206(2)	0.6003(2)	0.09968(8)	0.08

Table 2. Continued.

Atom	Site	x	y	z	<i>U</i> _{iso}
H(16C)	4e	−0.1388(2)	0.7211(2)	0.04670(7)	0.08
H(16D)	4e	−0.0246(2)	0.8144(2)	0.03453(7)	0.08
H(18D)	4e	0.0970(3)	1.3549(3)	0.0684(1)	0.08
H(18E)	4e	0.2039(3)	1.3919(3)	0.1053(1)	0.08

Table 2. Continued.

Atom	Site	x	y	z	<i>U</i> _{iso}
H(18F)	4e	0.0839(3)	1.3121(3)	0.1191(1)	0.08
H(19D)	4e	0.2529(2)	1.2225(3)	0.02271(8)	0.08
H(19E)	4e	0.3359(2)	1.1041(3)	0.04685(8)	0.08
H(19F)	4e	0.3596(2)	1.2641(3)	0.05920(8)	0.08

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(11)	4e	−0.8120(2)	−0.4650(2)	−0.00948(6)	0.107(2)	0.069(1)	0.074(1)	−0.012(1)	0.053(1)	−0.005(1)
C(11)	4e	−0.7499(2)	−0.3694(3)	−0.02204(7)	0.074(2)	0.055(1)	0.043(1)	0.005(1)	0.018(1)	−0.005(1)
O(12)	4e	−0.7643(2)	−0.2345(2)	−0.00668(6)	0.095(1)	0.059(1)	0.060(1)	0.011(1)	0.026(1)	−0.0153(8)
C(13)	4e	−0.6811(3)	−0.1409(3)	−0.02984(8)	0.082(2)	0.046(1)	0.060(1)	0.004(1)	0.003(1)	−0.013(1)
C(14)	4e	−0.7571(2)	−0.1939(2)	−0.11104(7)	0.042(1)	0.035(1)	0.058(1)	0.0064(9)	−0.0031(9)	0.0011(9)
C(15)	4e	−0.7223(2)	−0.2353(2)	−0.15845(7)	0.046(1)	0.043(1)	0.049(1)	0.0007(9)	−0.0040(9)	0.0141(9)
C(16)	4e	−0.7188(2)	−0.3938(2)	−0.16780(6)	0.037(1)	0.046(1)	0.0298(8)	−0.0008(8)	0.0011(7)	0.0071(8)
C(17)	4e	−0.5991(2)	−0.6263(2)	−0.15303(6)	0.0364(9)	0.042(1)	0.0369(9)	0.0009(8)	0.0018(8)	−0.0023(8)
C(18)	4e	−0.5329(2)	−0.7009(2)	−0.11192(6)	0.036(1)	0.0334(9)	0.043(1)	0.0030(8)	0.0035(8)	0.0024(8)
C(19)	4e	−0.5855(2)	−0.6246(2)	−0.07204(6)	0.0360(9)	0.037(1)	0.0372(9)	−0.0004(8)	0.0006(8)	0.0060(8)
O(19)	4e	−0.5667(2)	−0.6595(2)	−0.03279(5)	0.077(1)	0.060(1)	0.0389(8)	0.0178(9)	0.0020(8)	0.0135(7)
C(110)	4e	−0.6608(2)	−0.4959(2)	−0.08840(5)	0.0307(9)	0.0344(9)	0.0291(8)	0.0011(7)	0.0017(7)	0.0020(7)
C(111)	4e	−0.6220(2)	−0.4734(2)	−0.13722(5)	0.0303(9)	0.0374(9)	0.0294(8)	−0.0007(7)	0.0020(7)	0.0037(7)
C(112)	4e	−0.6482(2)	−0.3712(2)	−0.05509(6)	0.040(1)	0.039(1)	0.0340(9)	0.0025(8)	0.0002(8)	−0.0011(8)
C(113)	4e	−0.6535(2)	−0.2187(2)	−0.07329(7)	0.043(1)	0.035(1)	0.046(1)	0.0011(8)	−0.0009(9)	−0.0022(8)
O(114)	4e	−0.8383(1)	−0.4556(2)	−0.16325(5)	0.0357(7)	0.0527(8)	0.0423(7)	−0.0033(6)	−0.0074(6)	0.0062(6)
C(115)	4e	−0.9053(2)	−0.4432(3)	−0.20622(8)	0.057(1)	0.074(2)	0.054(1)	−0.009(1)	−0.026(1)	0.008(1)
C(116)	4e	−0.8053(3)	−0.4476(4)	−0.23889(9)	0.089(2)	0.150(3)	0.042(1)	−0.006(2)	−0.013(1)	−0.014(2)
O(117)	4e	−0.6928(1)	−0.4144(2)	−0.21423(4)	0.0592(9)	0.076(1)	0.0295(7)	0.0048(8)	0.0021(6)	0.0076(7)
C(118)	4e	−0.3921(2)	−0.6712(3)	−0.10885(8)	0.038(1)	0.058(1)	0.064(1)	0.008(1)	0.004(1)	0.008(1)
C(119)	4e	−0.5578(2)	−0.8603(2)	−0.11049(9)	0.072(2)	0.038(1)	0.070(2)	−0.001(1)	0.008(1)	0.000(1)
O(21)	4e	0.4018(2)	0.8841(2)	0.21336(5)	0.0487(9)	0.125(2)	0.0489(9)	0.024(1)	−0.0077(8)	−0.007(1)
C(21)	4e	0.3093(2)	0.9306(3)	0.22824(7)	0.045(1)	0.069(2)	0.036(1)	0.003(1)	−0.0070(9)	−0.006(1)
O(22)	4e	0.2802(2)	0.9002(2)	0.27113(5)	0.062(1)	0.083(1)	0.0364(8)	0.0095(9)	−0.0042(7)	0.0034(8)
C(23)	4e	0.1577(2)	0.9603(3)	0.27832(7)	0.069(2)	0.069(2)	0.038(1)	0.009(1)	0.011(1)	−0.001(1)
C(24)	4e	0.0370(2)	0.8343(2)	0.21393(7)	0.046(1)	0.051(1)	0.050(1)	−0.002(1)	0.010(1)	0.010(1)
C(25)	4e	−0.0535(2)	0.8419(2)	0.17157(7)	0.037(1)	0.047(1)	0.055(1)	−0.0048(9)	0.0073(9)	0.007(1)
C(26)	4e	0.0058(2)	0.8733(2)	0.12714(6)	0.0286(9)	0.035(1)	0.045(1)	0.0022(7)	−0.0015(7)	0.0011(8)
C(27)	4e	0.1076(2)	1.0626(2)	0.07961(6)	0.040(1)	0.045(1)	0.040(1)	−0.0024(9)	−0.0024(8)	0.0052(9)
C(28)	4e	0.2051(2)	1.1808(2)	0.08794(7)	0.038(1)	0.044(1)	0.049(1)	−0.0046(9)	0.0014(9)	0.0097(9)
C(29)	4e	0.2727(2)	1.1360(2)	0.13276(7)	0.039(1)	0.043(1)	0.040(1)	−0.0041(9)	0.0059(8)	−0.0050(8)
O(29)	4e	0.3667(1)	1.1902(2)	0.14959(5)	0.0524(9)	0.072(1)	0.0546(9)	−0.0266(8)	−0.0013(7)	−0.0035(8)
C(210)	4e	0.2023(2)	1.0150(2)	0.15350(6)	0.0326(9)	0.0341(9)	0.0344(9)	−0.0006(7)	0.0006(7)	−0.0014(7)
C(211)	4e	0.0742(2)	1.0151(2)	0.12688(6)	0.0314(9)	0.0334(9)	0.0389(9)	0.0018(7)	0.0019(7)	0.0005(7)
C(212)	4e	0.2071(2)	1.0217(2)	0.20541(6)	0.041(1)	0.043(1)	0.0346(9)	−0.0022(8)	0.0018(8)	−0.0050(8)
C(213)	4e	0.0937(2)	0.9748(2)	0.23071(6)	0.047(1)	0.050(1)	0.038(1)	0.0063(9)	0.0093(9)	0.0021(9)
O(214)	4e	0.0895(1)	0.7641(1)	0.11611(5)	0.0384(7)	0.0344(7)	0.0596(9)	0.0053(6)	−0.0017(6)	−0.0070(6)
C(215)	4e	0.0249(2)	0.6714(2)	0.08428(8)	0.055(1)	0.040(1)	0.059(1)	−0.004(1)	0.007(1)	−0.009(1)
C(216)	4e	−0.0652(2)	0.7696(2)	0.05879(7)	0.058(1)	0.048(1)	0.050(1)	−0.009(1)	−0.004(1)	−0.006(1)
O(217)	4e	−0.0943(1)	0.8723(2)	0.09232(5)	0.0363(7)	0.0463(9)	0.0579(9)	0.0016(6)	−0.0115(7)	−0.0054(7)
C(218)	4e	0.1414(3)	1.3232(3)	0.0958(1)	0.066(2)	0.041(1)	0.116(2)	−0.001(1)	−0.002(2)	0.016(1)
C(219)	4e	0.2968(2)	1.1946(3)	0.05070(8)	0.058(1)	0.103(2)	0.049(1)	−0.019(1)	0.003(1)	0.023(1)

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