Crystal structure of 2-(6-[2-(5-ethyl-5-hydroxy-6-methyl-tetrahydro-pyran-2-yl)-15-oxo-2,10,12-trimethyl-1,6,8-trioxa-dispiro[4.1.5.3]pentadec-13-en-9-yl]-2-hydroxy-1,3-dimethyl-4-oxo-heptyl-5-methyl-tetrahydro-pyran-2-yl)-butyrate sodium, $\text{Na}(\text{C}_{42}\text{H}_{67}\text{O}_{11})$, SY-9 – antibiotic 20-oxo-salinomycin

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Abstract

$\text{C}_{42}\text{H}_{67}\text{NaO}_{11}$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 10.5617(5)$ Å, $b = 19.1580(9)$ Å, $c = 21.5366(9)$ Å, $V = 4357.7$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.035$, $wR_{\text{ref}}(F^2) = 0.082$, $T = 293$ K.

Source of material

The title compound, the antibiotic SY-9 [1] is produced by fermentation with *Streptomyces albus* ATCC 21838 as a minor component, while salinomycin is the main product. Although it is a salt, crystals were grown from *n*-hexane.

Experimental details

The average I/σ of the measured X-ray intensities is 37.9. All hydrogens could be determined experimentally. The largest difference peak and hole were 0.098 and -0.117 e/Å³, respectively. The Flack parameter is $-0.2(4)$ [2]. According to Hamilton [3] the hypothesis, that the given absolute configuration might be wrong, can be rejected at the 0.5 % level [$(R_1/R_2)_{\text{exp}} = 0.0818/0.0817 = 1.001$; $(R_1/R_2)_{1,6036,0.005;\text{theor}} = 1.00068$].

Discussion

SY-9 [1] is a member of the polyether family of antibiotics and is related to salinomycin [4]. The absolute configuration is (2*R*,3*R*,6*S*,7*R*,8*S*,9*S*,10*S*,12*R*,13*S*,14*S*,16*R*,17*R*,21*S*,24*S*,25*R*,28*R*,29*S*), as it was analogously determined for salinomycin [5–8] and SY-1 [9]. In contrast to SY-1 there is no solvent molecule methanol as a remainder of the chromatographical separation from the ingredients of the original natural extract. Like in SY-1 and in salinomycin itself sodium has coordination number 5 (figure, top). Because there are no solvent water molecules like in salinomycin, O10 is taken for completing the coordination of sodium instead of O02 as in SY-1. Figure, bottom shows the least squares fit to molecule 2 (M2) of salinomycin, a molecule with a relatively large head-to-tail distance $d(\text{O10H}\cdots\text{O}_{\text{exo}} \text{ positioned}) = 3.255$ Å, fitting only sodium and three oxygens of its configuration. The fit to molecule 1 (M1) is much worse, because only in M2 O_{endo} positioned is coordinated to Na like in SY-9. O_{endo} positioned and O_{exo} positioned are the two oxygens at C01. The average e.s.d. for a C—C bond length is 0.004 Å, for a C—O bond length 0.003 Å, for a C—H and O—H bond length 0.03 Å, for a O—Na coordination distance 0.002 Å, for bond angles (without hydrogens) 0.2° and for torsion angles 0.3°.

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Table 1. Data collection and handling.

Crystal:	colorless thin plate, size 0.1 × 0.4 × 0.5 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.92 cm ⁻¹
Diffractionmeter, scan mode:	Three-circle AXS/CCD, ω with $\Delta\omega = 0.3^\circ$
$2\theta_{\max}$:	46.52°
$N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}$:	22386, 6211
Criterion for $I_{\text{obs}}, N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5231
$N(\text{param})_{\text{refined}}$:	755
Programs:	SHELXS-97 [10], SHELXL-97 [11] SHELXTL-PLUS [12]

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(02)	4a	0.755(2)	0.793(1)	0.902(1)	0.045(6)
H(03)	4a	0.498(3)	0.739(1)	0.883(1)	0.072(8)
H(041)	4a	0.643(3)	0.638(2)	0.885(1)	0.09(1)
H(042)	4a	0.529(3)	0.639(2)	0.938(1)	0.09(1)
H(051)	4a	0.791(3)	0.676(1)	0.956(1)	0.068(8)
H(052)	4a	0.718(3)	0.603(2)	0.985(1)	0.09(1)
H(06)	4a	0.728(3)	0.695(2)	1.063(1)	0.068(9)
H(07)	4a	0.592(2)	0.789(1)	0.995(1)	0.044(6)
H(08)	4a	0.517(2)	0.804(1)	1.083(1)	0.055(7)
H(09)	4a	0.509(2)	0.890(1)	1.006(1)	0.049(7)
H(10)	4a	0.628(3)	0.957(1)	1.110(1)	0.065(8)
H(12)	4a	0.592(2)	1.078(1)	1.118(1)	0.053(7)
H(16)	4a	0.912(3)	1.193(2)	0.996(1)	0.068(8)
H(14)	4a	0.793(3)	1.171(1)	1.094(1)	0.061(7)
H(151)	4a	1.016(3)	1.145(2)	1.084(1)	0.076(9)
H(152)	4a	0.982(3)	1.066(2)	1.063(1)	0.09(1)
H(18)	4a	0.903(3)	1.003(2)	0.976(1)	0.075(9)
H(19)	4a	0.894(2)	0.956(1)	0.875(1)	0.061(8)
H(221)	4a	0.858(4)	1.170(2)	0.770(2)	0.11(1)
H(222)	4a	0.792(3)	1.218(2)	0.819(2)	0.10(1)
H(231)	4a	0.623(3)	1.219(2)	0.750(2)	0.09(1)
H(232)	4a	0.662(3)	1.137(2)	0.732(1)	0.074(9)
H(25)	4a	0.404(2)	1.137(1)	0.766(1)	0.037(6)
H(261)	4a	0.558(3)	1.014(1)	0.778(1)	0.064(8)
H(262)	4a	0.525(2)	1.058(1)	0.717(1)	0.058(7)

Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(271)	4a	0.407(3)	0.948(2)	0.724(2)	0.09(1)
H(272)	4a	0.326(2)	1.016(1)	0.711(1)	0.053(7)
H(29)	4a	0.236(3)	1.041(1)	0.873(1)	0.068(8)
H(301)	4a	0.659(3)	0.733(2)	0.790(2)	0.08(1)
H(302)	4a	0.764(3)	0.696(2)	0.828(2)	0.11(1)
H(311)	4a	0.851(5)	0.766(3)	0.741(3)	0.18(2)
H(312)	4a	0.806(4)	0.834(2)	0.772(2)	0.11(2)
H(313)	4a	0.905(8)	0.798(4)	0.811(3)	0.27(4)
H(321)	4a	0.539(3)	0.680(2)	1.106(2)	0.10(1)
H(322)	4a	0.569(4)	0.611(2)	1.074(2)	0.12(1)
H(323)	4a	0.473(4)	0.668(2)	1.039(2)	0.11(1)
H(331)	4a	0.675(3)	0.855(2)	1.148(2)	0.10(1)
H(332)	4a	0.712(4)	0.774(2)	1.133(2)	0.12(1)
H(333)	4a	0.776(4)	0.841(2)	1.100(2)	0.11(1)
H(341)	4a	0.355(4)	0.936(2)	1.098(2)	0.12(1)
H(342)	4a	0.418(3)	0.975(2)	1.150(2)	0.10(1)
H(343)	4a	0.461(3)	0.892(2)	1.152(2)	0.11(1)
H(351)	4a	0.506(3)	1.150(2)	1.015(2)	0.09(1)
H(352)	4a	0.587(3)	1.189(2)	1.068(1)	0.09(1)
H(361)	4a	0.346(5)	1.110(3)	1.086(2)	0.15(2)
H(362)	4a	0.423(5)	1.156(3)	1.138(3)	0.19(2)
H(363)	4a	0.360(4)	1.202(3)	1.085(2)	0.14(2)
H(371)	4a	0.760(5)	1.091(3)	1.177(2)	0.16(3)
H(372)	4a	0.893(4)	1.114(2)	1.181(2)	0.11(1)
H(373)	4a	0.861(4)	1.035(2)	1.152(2)	0.15(2)
H(381)	4a	1.129(4)	1.173(2)	0.980(2)	0.12(2)
H(382)	4a	1.070(4)	1.158(2)	0.914(2)	0.14(2)
H(383)	4a	1.102(4)	1.091(2)	0.965(2)	0.12(2)
H(391)	4a	0.587(3)	1.234(2)	0.873(2)	0.09(1)
H(392)	4a	0.463(3)	1.239(2)	0.836(2)	0.09(1)
H(393)	4a	0.476(4)	1.187(2)	0.897(2)	0.12(1)
H(401)	4a	0.118(3)	0.939(2)	0.826(2)	0.11(1)
H(402)	4a	0.129(3)	0.962(2)	0.752(1)	0.09(1)
H(411)	4a	0.249(4)	0.839(2)	0.798(2)	0.13(2)
H(412)	4a	0.259(4)	0.865(2)	0.724(2)	0.11(1)
H(413)	4a	0.124(4)	0.833(2)	0.759(2)	0.11(1)
H(421)	4a	0.087(4)	1.072(2)	0.793(2)	0.11(1)
H(422)	4a	0.196(3)	1.104(2)	0.750(2)	0.09(1)
H(423)	4a	0.171(3)	1.140(2)	0.817(1)	0.07(1)
H(13)	4a	0.760(2)	1.031(1)	1.052(1)	0.048(6)
H(4)	4a	0.688(3)	0.910(2)	0.966(2)	0.09(1)
H(10)	4a	0.423(3)	0.912(2)	0.830(2)	0.10(1)

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(01)	4a	0.6114(3)	0.8498(2)	0.8562(1)	0.069(2)	0.061(2)	0.051(2)	0.001(2)	−0.008(2)	−0.006(1)
C(02)	4a	0.6769(3)	0.7812(1)	0.8739(1)	0.061(2)	0.053(2)	0.056(2)	0.008(1)	−0.012(1)	−0.012(1)
C(03)	4a	0.5782(3)	0.7382(1)	0.9098(1)	0.061(2)	0.050(2)	0.069(2)	0.001(1)	−0.011(2)	−0.011(1)
C(04)	4a	0.6108(4)	0.6617(2)	0.9239(2)	0.086(2)	0.053(2)	0.086(2)	0.001(2)	−0.002(2)	−0.013(2)
C(05)	4a	0.7063(4)	0.6549(2)	0.9762(2)	0.089(3)	0.056(2)	0.085(2)	0.014(2)	0.002(2)	0.006(2)
C(06)	4a	0.6640(3)	0.6943(2)	1.0337(2)	0.077(2)	0.063(2)	0.074(2)	0.011(2)	−0.007(2)	0.012(2)
C(07)	4a	0.6420(3)	0.7704(1)	1.0140(1)	0.055(2)	0.056(2)	0.056(2)	0.003(1)	−0.007(1)	0.009(1)
C(08)	4a	0.6019(3)	0.8212(2)	1.0652(1)	0.063(2)	0.060(2)	0.050(2)	−0.000(1)	−0.001(1)	0.005(1)
C(09)	4a	0.5819(3)	0.8939(1)	1.0372(1)	0.058(2)	0.058(2)	0.048(1)	−0.006(1)	0.003(1)	−0.003(1)
C(10)	4a	0.5515(3)	0.9514(1)	1.0840(1)	0.072(2)	0.057(2)	0.043(1)	−0.007(2)	0.002(1)	−0.002(1)
C(11)	4a	0.5277(3)	1.0195(1)	1.0499(1)	0.072(2)	0.068(2)	0.039(1)	0.001(2)	0.009(1)	−0.011(1)
C(12)	4a	0.5926(3)	1.0843(2)	1.0748(1)	0.096(2)	0.056(2)	0.040(2)	0.005(2)	0.004(2)	−0.006(1)
C(13)	4a	0.7310(3)	1.0810(2)	1.0529(1)	0.088(2)	0.048(2)	0.043(1)	0.000(2)	−0.006(1)	−0.006(1)
C(14)	4a	0.8254(4)	1.1213(2)	1.0922(1)	0.102(3)	0.062(2)	0.054(2)	−0.001(2)	−0.019(2)	−0.009(2)
C(15)	4a	0.9522(4)	1.1184(2)	1.0604(1)	0.093(3)	0.069(2)	0.068(2)	−0.009(2)	−0.030(2)	−0.010(2)
C(16)	4a	0.9486(3)	1.1447(2)	0.9935(1)	0.078(2)	0.063(2)	0.067(2)	−0.011(2)	−0.017(2)	−0.010(2)
C(17)	4a	0.8458(3)	1.1059(1)	0.9566(1)	0.064(2)	0.056(2)	0.054(2)	−0.005(1)	−0.006(1)	−0.006(1)
C(18)	4a	0.8791(3)	1.0321(2)	0.9401(1)	0.062(2)	0.058(2)	0.068(2)	−0.000(1)	−0.003(2)	−0.005(2)
C(19)	4a	0.8758(3)	1.0071(2)	0.8826(1)	0.058(2)	0.064(2)	0.078(2)	−0.001(2)	0.002(2)	−0.019(2)
C(20)	4a	0.8359(2)	1.0507(2)	0.8313(1)	0.047(2)	0.080(2)	0.064(2)	−0.008(2)	0.000(1)	−0.020(2)
C(21)	4a	0.7735(3)	1.1198(1)	0.8487(1)	0.061(2)	0.064(2)	0.047(1)	−0.014(1)	−0.000(1)	−0.005(1)
C(22)	4a	0.7828(4)	1.1761(2)	0.7989(2)	0.091(3)	0.084(3)	0.054(2)	−0.029(2)	−0.003(2)	0.007(2)

Table 3. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(23)	4a	0.6550(3)	1.1738(2)	0.7665(1)	0.089(2)	0.070(2)	0.055(2)	−0.017(2)	−0.005(2)	0.012(2)
C(24)	4a	0.5640(3)	1.1492(1)	0.8171(1)	0.072(2)	0.052(2)	0.050(2)	0.008(2)	−0.003(1)	0.005(1)
C(25)	4a	0.4547(3)	1.1057(1)	0.7925(1)	0.056(2)	0.061(2)	0.043(1)	0.007(2)	0.002(1)	0.012(1)
C(26)	4a	0.4911(3)	1.0411(2)	0.7560(1)	0.060(2)	0.075(2)	0.045(2)	−0.002(2)	0.004(1)	−0.006(1)
C(27)	4a	0.3767(3)	0.9948(2)	0.7422(1)	0.058(2)	0.080(2)	0.048(2)	−0.005(2)	−0.005(1)	0.001(2)
C(28)	4a	0.2964(3)	0.9808(2)	0.7997(1)	0.058(2)	0.067(2)	0.049(1)	0.004(1)	−0.004(1)	0.012(1)
C(29)	4a	0.2637(3)	1.0498(2)	0.8302(1)	0.058(2)	0.074(2)	0.060(2)	0.005(2)	0.012(1)	0.016(2)
C(30)	4a	0.7267(4)	0.7450(2)	0.8154(2)	0.089(3)	0.076(2)	0.069(2)	0.015(2)	−0.006(2)	−0.022(2)
C(31)	4a	0.8265(6)	0.7866(3)	0.7819(2)	0.131(4)	0.123(4)	0.074(3)	−0.007(3)	0.026(3)	−0.009(3)
C(32)	4a	0.5510(5)	0.6614(2)	1.0651(2)	0.115(4)	0.074(3)	0.092(3)	−0.006(2)	0.017(3)	0.023(2)
C(33)	4a	0.7003(5)	0.8218(2)	1.1172(2)	0.103(3)	0.085(3)	0.061(2)	0.010(2)	−0.019(2)	0.000(2)
C(34)	4a	0.4368(4)	0.9370(2)	1.1253(2)	0.103(3)	0.076(2)	0.063(2)	−0.009(2)	0.025(2)	−0.011(2)
C(35)	4a	0.5237(4)	1.1522(2)	1.0595(2)	0.109(3)	0.062(2)	0.076(2)	0.021(2)	0.016(2)	−0.005(2)
C(36)	4a	0.3946(6)	1.1579(3)	1.0909(3)	0.145(4)	0.106(4)	0.119(4)	0.055(4)	0.052(3)	0.007(3)
C(37)	4a	0.8333(7)	1.0902(3)	1.1583(2)	0.149(5)	0.130(4)	0.058(2)	−0.015(4)	−0.036(3)	0.002(2)
C(38)	4a	1.0777(4)	1.1417(3)	0.9623(2)	0.079(3)	0.117(4)	0.101(3)	−0.029(3)	−0.009(2)	−0.017(3)
C(39)	4a	0.5175(5)	1.2091(2)	0.8576(2)	0.107(3)	0.063(2)	0.086(3)	0.017(2)	−0.008(3)	−0.014(2)
C(40)	4a	0.1770(3)	0.9374(2)	0.7847(2)	0.065(2)	0.099(3)	0.076(2)	−0.011(2)	−0.007(2)	0.027(2)
C(41)	4a	0.2048(5)	0.8629(2)	0.7658(2)	0.104(3)	0.093(3)	0.092(3)	−0.034(3)	−0.012(3)	0.002(2)
C(42)	4a	0.1721(4)	1.0965(2)	0.7951(2)	0.059(2)	0.090(3)	0.100(3)	0.017(2)	0.001(2)	0.025(2)
O(01)	4a	0.6414(2)	0.90506(9)	0.88525(8)	0.079(1)	0.055(1)	0.059(1)	0.000(1)	−0.014(1)	−0.0038(9)
O(02)	4a	0.5301(2)	0.8474(1)	0.81358(9)	0.097(2)	0.073(1)	0.073(1)	0.017(1)	−0.036(1)	−0.013(1)
O(03)	4a	0.5452(2)	0.77336(9)	0.96674(8)	0.056(1)	0.055(1)	0.061(1)	0.0029(9)	−0.0074(9)	−0.0047(8)
O(04)	4a	0.6941(2)	0.9170(1)	1.00573(9)	0.076(1)	0.065(1)	0.051(1)	−0.014(1)	0.009(1)	−0.0081(9)
O(05)	4a	0.4607(2)	1.0216(1)	1.00390(8)	0.085(1)	0.082(1)	0.048(1)	0.009(1)	−0.006(1)	−0.0075(9)
O(06)	4a	0.7281(2)	1.10668(9)	0.98957(7)	0.071(1)	0.058(1)	0.0427(9)	0.001(1)	−0.0051(9)	−0.0049(8)
O(07)	4a	0.8259(2)	1.14811(9)	0.90348(8)	0.076(1)	0.058(1)	0.048(1)	−0.013(1)	−0.0066(9)	−0.0024(9)
O(08)	4a	0.6423(2)	1.10306(9)	0.85554(7)	0.054(1)	0.055(1)	0.0478(9)	−0.0019(9)	−0.0013(8)	0.0036(8)
O(09)	4a	0.3796(2)	1.08681(9)	0.84559(7)	0.067(1)	0.064(1)	0.0441(9)	0.012(1)	0.0077(9)	0.0029(8)
O(10)	4a	0.3694(2)	0.9455(1)	0.84572(9)	0.074(1)	0.066(1)	0.055(1)	0.012(1)	−0.010(1)	0.0057(9)
O(11)	4a	0.8447(2)	1.0331(1)	0.7768(1)	0.084(2)	0.127(2)	0.067(1)	0.013(1)	0.004(1)	−0.035(1)
Na(1)	4a	0.5241(1)	1.00625(5)	0.90272(4)	0.0843(7)	0.0585(6)	0.0445(5)	0.0056(6)	−0.0072(5)	0.0011(5)

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