Crystal structure of the antibiotic SY-1 (20-deoxy-salinomycin): sodium 2-(6-[2-(5-ethyl-5-hydroxy-6-methyl-tetrahydro-pyran-2-yl)-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 — methanol solvate (1:0.69), $C_{42}H_{69}NaO_{10} \cdot 0.69CH_3OH$

E. F. Paulus^{*I} and L. Vértessy^{II}

^I Institut für Mineralogie/Kristallographie der Johann Wolfgang Goethe-Universität, Senckenberganlage 30, D-60325 Frankfurt/Main, Germany

^{II} Natural Product Research, Aventis Pharma Deutschland GmbH, D-65926 Frankfurt/Main, Germany

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Abstract

$C_{42.693}H_{71.772}NaO_{10.693}$, orthorhombic, $P2_12_12_1$ (No. 19), $a = 11.295(1) \text{ \AA}$, $b = 18.628(1) \text{ \AA}$, $c = 21.823(1) \text{ \AA}$, $V = 4591.6 \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.037$, $wR_{\text{ref}}(F^2) = 0.099$, $T = 293 \text{ K}$.

Source of material

The antibiotic SY-1 is a microbial metabolite of *Streptomyces albus* ATCC 21838. It is generated by fermentation during the production of the anticoccidial agent Salinomycin. The chemical structure has been deduced from that of SY-2 [1] and the constitution was confirmed by mass spectrometry [2]. It is the 20-deoxy derivative of Salinomycin and has weak anticoccidial and more-over activity against gram-positive bacteria and is used for treatment of coccidial infection in chickens [2]. Crystals were made by crystallization from *n*-hexane, nevertheless it is a salt.

Experimental details

The average $I/\sigma(I)$ of the measured X-ray intensities is 33.8. Except the hydrogens of one ethyl group, all the other hydrogens could be determined experimentally. The largest difference peak and hole were $0.205 \text{ e} \cdot \text{\AA}^{-3}$ and $-0.173 \text{ e} \cdot \text{\AA}^{-3}$ respectively. The Flack parameter is $-0.1(3)$ [3]. According to the Hamilton test [4], the hypothesis that the given absolute configuration might be wrong can be rejected at the 0.5 % level [$(R_1/R_2)_{\text{exp}} = 1.001$; $(R_1/R_2)_{(1,6036, 0.005)} = 1.00068$].

Discussion

The absolute configuration is (2R,3R,6S,7R,8S,9S,10S,12R,13S,14S,16R,17R,21S, 24S,25R,28R,29S), as it was analogously determined for Salinomycin [2,5–7]. The solvent molecule methanol is a remainder of the chromatographical separation from the ingredients of the original natural extract and was not removed during the recrystallization from *n*-hexane. Its hydrogen bond to O02 is relatively strong ($2.779 \text{ \AA}$). Alike in Salinomycin itself sodium has again the coordination number 5. Because there are no hydrophilic solvent molecules available for the coordination of Na^+ , another oxygen of the carboxylic group is used for that purpose, what takes away the possibility for a second isomer like in salinomycin [6]. The rest of the coordination is the same as that for salinomycin. The double spiro system and the sodium coordination sphere do not allow any changes in the centre of the molecule. Conformational differences occur only at the periphery. The conformations of SY-1 are the “averages” between the two different

* Correspondence author (e-mail: e.paulus@kristall.uni-frankfurt.de)

molecules of Salinomycin [6], which are in a certain way extreme cases of conformations. The figure (bottom) shows the least squares fit to molecule 1 of salinomycin, fitting only sodium and its configuration. The two intramolecular hydrogen bonds ($d(\text{O}04\text{H}\cdots\text{O}02) = 2.787 \text{ \AA}$; $d(\text{O}10\text{H}\cdots\text{O}01) = 2.856 \text{ \AA}$, "head to tail") fix the carboxyl group in its position. We have an ion pair, in which the sodium ion is configured in the centre of the complex by oxygens of negative polarity. The ion pair has lost its hydrophilicity. The average esd for a C—C and a C—O bond length is $0.003 \text{ \AA}$, for a C—H and O—H bond length $0.04 \text{ \AA}$, for a O—Na coordination distance $0.002 \text{ \AA}$, for bond angles (without hydrogens) $0.2 \text{ \AA}$ and for torsion angles 0.3° .

Table 1. Data collection and handling.

Crystal:	colourless plate, size $0.25 \times 0.5 \times 0.6 \text{ mm}$
Wavelength:	Mo K_α radiation ($0.71073 \text{ \AA}$)
μ :	0.88 cm^{-1}
Diffractionmeter, scan mode:	AXS CCD, $\Delta\omega = 0.3^\circ$
$2\theta_{\text{max}}$:	50.46°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	25297, 7600
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 6540
$N(\text{param})_{\text{refined}}$:	757
Programs:	SHELXS-97 [8], SHELXL-97 [9], SHELXTL-plus [10]

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(40A)	4a		−0.2839	0.5878	0.1842	0.164
H(40B)	4a		−0.2222	0.5922	0.2483	0.164
H(41A)	4a		−0.2543	0.7053	0.2202	0.196
H(41B)	4a		−0.2034	0.692	0.1544	0.196
H(41C)	4a		−0.1181	0.6934	0.2110	0.196
H(02)	4a		0.344(2)	0.738(1)	0.219(1)	0.052(6)
H(03)	4a		0.194(2)	0.813(1)	0.1367(9)	0.045(5)
H(041)	4a		0.304(2)	0.883(1)	0.216(1)	0.065(7)
H(042)	4a		0.311(2)	0.917(2)	0.152(1)	0.075(8)
H(051)	4a		0.498(2)	0.917(1)	0.192(1)	0.064(6)
H(052)	4a		0.494(2)	0.834(1)	0.213(1)	0.069(7)
H(06)	4a		0.601(3)	0.839(2)	0.120(1)	0.091(9)
H(07)	4a		0.483(2)	0.743(1)	0.1299(9)	0.033(4)
H(08)	4a		0.466(2)	0.777(1)	0.008(1)	0.044(5)
H(09)	4a		0.362(2)	0.678(1)	0.0361(8)	0.036(5)
H(10)	4a		0.545(2)	0.621(1)	−0.034(1)	0.050(6)
H(12)	4a		0.502(2)	0.521(1)	−0.101(1)	0.062(6)
H(13)	4a		0.542(2)	0.491(1)	0.005(1)	0.058(6)
H(14)	4a		0.539(2)	0.368(1)	−0.074(1)	0.057(6)
H(151)	4a		0.687(2)	0.393(2)	0.028(1)	0.074(8)
H(152)	4a		0.689(3)	0.324(2)	−0.013(1)	0.092(9)

Table 2. Continued.

Atom	Site	Occ.	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(16)	4a		0.494(2)	0.292(2)	0.021(1)	0.073(7)
H(18)	4a		0.544(2)	0.470(1)	0.097(1)	0.069(8)
H(19)	4a		0.468(2)	0.475(1)	0.193(1)	0.065(7)
H(201)	4a		0.287(2)	0.423(2)	0.208(1)	0.072(8)
H(202)	4a		0.372(3)	0.349(2)	0.208(1)	0.100(9)
H(221)	4a		0.151(3)	0.318(2)	0.182(2)	0.081(9)
H(222)	4a		0.219(3)	0.268(2)	0.143(2)	0.10(1)
H(231)	4a		0.006(3)	0.304(2)	0.102(1)	0.085(8)
H(232)	4a		0.121(3)	0.286(2)	0.053(2)	0.094(9)
H(25)	4a		−0.089(2)	0.425(1)	0.085(1)	0.070(7)
H(261)	4a		0.057(3)	0.441(2)	0.193(1)	0.097(9)
H(262)	4a		−0.037(3)	0.380(2)	0.185(2)	0.12(1)
H(271)	4a		−0.200(3)	0.460(2)	0.186(2)	0.10(1)
H(272)	4a		−0.134(3)	0.476(2)	0.242(2)	0.10(1)
H(29)	4a		−0.055(2)	0.611(1)	0.099(1)	0.058(6)
H(301)	4a		0.193(3)	0.803(2)	0.261(1)	0.088(9)
H(302)	4a		0.089(3)	0.767(2)	0.221(2)	0.11(1)
H(311)	4a		0.105(3)	0.715(2)	0.325(2)	0.11(1)
H(312)	4a		0.143(3)	0.651(2)	0.273(2)	0.095(9)
H(313)	4a		0.240(5)	0.694(2)	0.318(2)	0.15(2)
H(321)	4a		0.543(3)	0.899(2)	0.028(2)	0.10(1)
H(322)	4a		0.537(3)	0.958(2)	0.085(2)	0.11(1)
H(323)	4a		0.429(3)	0.918(2)	0.056(2)	0.10(1)
H(331)	4a		0.676(3)	0.720(2)	0.059(2)	0.10(1)
H(332)	4a		0.673(3)	0.796(2)	0.033(1)	0.094(9)
H(333)	4a		0.662(3)	0.720(2)	−0.007(2)	0.10(1)
H(341)	4a		0.494(3)	0.725(2)	−0.094(1)	0.084(8)
H(342)	4a		0.355(3)	0.702(1)	−0.086(1)	0.078(8)
H(343)	4a		0.442(2)	0.654(2)	−0.129(1)	0.074(7)
H(351)	4a		0.273(3)	0.454(2)	−0.083(2)	0.10(1)
H(352)	4a		0.373(3)	0.421(2)	−0.125(1)	0.090(9)
H(361)	4a		0.247(3)	0.484(2)	−0.186(1)	0.077(9)
H(362)	4a		0.263(3)	0.552(2)	−0.155(2)	0.12(1)
H(363)	4a		0.380(4)	0.518(2)	−0.192(2)	0.15(2)
H(371)	4a		0.747(3)	0.397(2)	−0.103(2)	0.10(1)
H(372)	4a		0.650(5)	0.459(3)	−0.126(3)	0.17(2)
H(373)	4a		0.734(4)	0.477(3)	−0.067(2)	0.16(2)
H(381)	4a		0.530(3)	0.266(2)	0.122(1)	0.08(1)
H(382)	4a		0.652(4)	0.248(2)	0.084(2)	0.13(1)
H(383)	4a		0.622(3)	0.318(2)	0.127(2)	0.10(1)
H(391)	4a		0.164(3)	0.385(2)	−0.007(1)	0.077(8)
H(392)	4a		0.012(2)	0.385(1)	−0.022(1)	0.056(6)
H(393)	4a		0.106(2)	0.461(2)	−0.005(1)	0.076(8)
H(421)	4a		−0.243(4)	0.497(2)	0.083(2)	0.13(1)
H(422)	4a		−0.186(3)	0.550(2)	0.029(2)	0.11(1)
H(423)	4a		−0.267(4)	0.588(2)	0.078(2)	0.16(2)
H(010)	4a		0.004(4)	0.610(2)	0.190(2)	0.10(1)
H(4)	4a		0.458(2)	0.622(2)	0.104(1)	0.066(8)
H(1A)	4a	0.693(8)	0.5768	0.6015	0.3186	0.335
H(1B)	4a	0.693	0.4418	0.6168	0.3291	0.335
H(1C)	4a	0.693	0.5189	0.6710	0.2912	0.335
H(1)	4a	0.693	0.4421	0.6156	0.2348	0.373

Table 3. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	Occ.	<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		0.2447(2)	0.6734(1)	0.16388(9)	0.051(1)	0.046(1)	0.045(1)	−0.005(1)	0.0104(9)	0.0003(9)
C(02)	4a		0.2715(2)	0.7443(1)	0.1976(1)	0.055(1)	0.046(1)	0.050(1)	−0.003(1)	0.003(1)	−0.0052(9)
C(03)	4a		0.2777(2)	0.8063(1)	0.15088(9)	0.048(1)	0.046(1)	0.050(1)	0.0036(9)	0.0030(9)	−0.0048(9)
C(04)	4a		0.3326(3)	0.8759(1)	0.1764(1)	0.087(2)	0.045(1)	0.065(2)	−0.005(1)	0.014(1)	−0.010(1)
C(05)	4a		0.4680(2)	0.8719(1)	0.1788(1)	0.081(2)	0.054(1)	0.069(2)	−0.023(1)	−0.007(1)	−0.013(1)
C(06)	4a		0.5203(2)	0.8502(1)	0.1170(1)	0.053(1)	0.054(1)	0.071(2)	−0.014(1)	−0.006(1)	−0.004(1)
C(07)	4a		0.4641(2)	0.7777(1)	0.0986(1)	0.037(1)	0.046(1)	0.051(1)	−0.0037(8)	−0.0061(8)	0.003(1)
C(08)	4a		0.5019(2)	0.7459(1)	0.0366(1)	0.040(1)	0.050(1)	0.051(1)	−0.0057(9)	0.0023(9)	0.004(1)
C(09)	4a		0.4454(2)	0.6707(1)	0.02914(9)	0.041(1)	0.046(1)	0.046(1)	0.0030(9)	0.0040(9)	0.0028(9)
C(10)	4a		0.4637(2)	0.6346(1)	−0.03351(9)	0.042(1)	0.050(1)	0.048(1)	0.0070(9)	0.0094(9)	0.0003(9)
C(11)	4a		0.3853(2)	0.5680(1)	−0.03791(9)	0.055(1)	0.050(1)	0.040(1)	0.007(1)	−0.0012(9)	0.0005(9)

Table 3. Continued.

Atom	Site	Occ.	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(12)	4a		0.4346(2)	0.5038(1)	−0.0740(1)	0.071(2)	0.054(1)	0.045(1)	0.006(1)	0.004(1)	−0.008(1)
C(13)	4a		0.4988(2)	0.4561(1)	−0.0260(1)	0.058(1)	0.051(1)	0.056(1)	0.005(1)	0.003(1)	−0.012(1)
C(14)	4a		0.5861(3)	0.4003(1)	−0.0520(1)	0.071(2)	0.064(2)	0.078(2)	0.012(1)	0.002(1)	−0.021(1)
C(15)	4a		0.6367(3)	0.3593(2)	0.0030(2)	0.063(2)	0.065(2)	0.102(2)	0.020(1)	−0.004(2)	−0.016(2)
C(16)	4a		0.5415(3)	0.3265(1)	0.0439(1)	0.078(2)	0.056(1)	0.091(2)	0.020(1)	−0.018(2)	−0.006(1)
C(17)	4a		0.4485(2)	0.3832(1)	0.0636(1)	0.067(1)	0.048(1)	0.063(1)	0.009(1)	−0.014(1)	0.002(1)
C(18)	4a		0.4891(3)	0.4361(1)	0.1107(1)	0.074(2)	0.062(1)	0.058(1)	0.002(1)	−0.016(1)	0.002(1)
C(19)	4a		0.4397(3)	0.4424(2)	0.1656(1)	0.094(2)	0.075(2)	0.057(2)	0.006(2)	−0.026(2)	−0.003(1)
C(20)	4a		0.3354(3)	0.3974(2)	0.1839(1)	0.098(2)	0.107(2)	0.054(2)	0.012(2)	−0.009(2)	0.007(2)
C(21)	4a		0.2698(3)	0.3689(1)	0.1286(1)	0.086(2)	0.060(1)	0.059(1)	0.003(1)	−0.008(1)	0.016(1)
C(22)	4a		0.1799(4)	0.3086(2)	0.1417(2)	0.113(3)	0.057(2)	0.098(2)	0.000(2)	0.012(2)	0.025(2)
C(23)	4a		0.0881(3)	0.3163(1)	0.0917(2)	0.092(2)	0.054(1)	0.104(2)	−0.016(1)	0.006(2)	0.005(2)
C(24)	4a		0.0889(2)	0.3975(1)	0.0760(1)	0.061(1)	0.051(1)	0.073(2)	−0.013(1)	−0.004(1)	0.001(1)
C(25)	4a		−0.0155(2)	0.4387(1)	0.1052(1)	0.062(1)	0.059(1)	0.077(2)	−0.022(1)	−0.002(1)	0.004(1)
C(26)	4a		−0.0288(3)	0.4299(2)	0.1747(2)	0.089(2)	0.074(2)	0.088(2)	−0.022(2)	0.026(2)	0.020(2)
C(27)	4a		−0.1269(3)	0.4774(2)	0.1988(2)	0.082(2)	0.094(2)	0.092(2)	−0.031(2)	0.033(2)	0.005(2)
C(28)	4a		−0.1087(2)	0.5561(2)	0.1808(1)	0.066(2)	0.084(2)	0.083(2)	−0.021(1)	0.021(1)	−0.009(2)
C(29)	4a		−0.0917(2)	0.5616(1)	0.1104(1)	0.049(1)	0.062(2)	0.088(2)	−0.008(1)	0.009(1)	−0.004(1)
C(30)	4a		0.1748(3)	0.7591(2)	0.2458(1)	0.108(2)	0.060(2)	0.070(2)	−0.002(2)	0.037(2)	−0.010(1)
C(31)	4a		0.1654(4)	0.7018(2)	0.2951(1)	0.124(3)	0.085(2)	0.054(2)	−0.023(2)	0.024(2)	−0.006(2)
C(32)	4a		0.5049(4)	0.9089(2)	0.0689(2)	0.093(2)	0.059(2)	0.090(2)	−0.021(2)	0.014(2)	0.007(1)
C(33)	4a		0.6369(2)	0.7447(2)	0.0285(2)	0.045(1)	0.077(2)	0.087(2)	−0.007(1)	0.011(1)	−0.005(2)
C(34)	4a		0.4365(2)	0.6828(1)	−0.0894(1)	0.063(2)	0.063(1)	0.049(1)	0.002(1)	0.012(1)	0.006(1)
C(35)	4a		0.3405(3)	0.4645(2)	−0.1122(1)	0.105(2)	0.068(2)	0.053(1)	0.009(2)	−0.018(2)	−0.015(1)
C(36)	4a		0.2997(4)	0.5086(2)	−0.1672(2)	0.106(3)	0.107(3)	0.060(2)	0.015(2)	−0.024(2)	−0.008(2)
C(37)	4a		0.6846(4)	0.4337(2)	−0.0907(2)	0.093(2)	0.106(3)	0.115(3)	0.023(2)	0.040(2)	−0.011(2)
C(38)	4a		0.5957(5)	0.2876(3)	0.0990(2)	0.119(3)	0.097(3)	0.122(3)	0.047(3)	−0.020(3)	0.020(3)
C(39)	4a		0.0941(3)	0.4087(2)	0.0066(1)	0.078(2)	0.065(2)	0.070(2)	−0.004(1)	−0.009(1)	−0.008(1)
C(40)	4a		−0.2118(3)	0.6022(2)	0.2050(2)	0.097(2)	0.105(3)	0.126(3)	−0.007(2)	0.039(2)	−0.021(2)
C(41)	4a		−0.1955(5)	0.6800(2)	0.1970(2)	0.143(4)	0.117(3)	0.132(3)	0.019(3)	0.030(3)	−0.001(3)
C(42)	4a		−0.2039(3)	0.5489(2)	0.0716(2)	0.061(2)	0.110(3)	0.114(3)	0.003(2)	−0.009(2)	−0.015(2)
Na(1)	4a		0.21217(8)	0.55275(4)	0.08997(4)	0.0681(5)	0.0475(4)	0.0654(5)	−0.0098(4)	0.0057(4)	−0.0077(4)
O(01)	4a		0.1462(1)	0.66719(9)	0.13736(8)	0.0558(9)	0.063(1)	0.073(1)	−0.0130(8)	−0.0012(8)	−0.0080(8)
O(02)	4a		0.3222(1)	0.62432(8)	0.16419(7)	0.0634(9)	0.0450(8)	0.0681(9)	0.0025(8)	0.0072(8)	0.0003(7)
O(03)	4a		0.3365(1)	0.78559(7)	0.09495(6)	0.0397(7)	0.0485(7)	0.0444(7)	−0.0008(6)	−0.0017(6)	−0.0029(6)
O(04)	4a		0.4948(2)	0.62045(8)	0.07294(8)	0.0628(9)	0.0554(8)	0.0526(9)	0.0121(8)	0.0042(8)	0.0071(7)
O(05)	4a		0.2877(1)	0.56534(8)	−0.01409(7)	0.0536(8)	0.0545(8)	0.0587(8)	−0.0019(7)	0.0075(7)	−0.0047(7)
O(06)	4a		0.4070(1)	0.42137(8)	0.00932(6)	0.0618(9)	0.0480(7)	0.0527(8)	0.0088(7)	−0.0086(7)	−0.0020(7)
O(07)	4a		0.3491(2)	0.34160(8)	0.08331(8)	0.082(1)	0.0474(8)	0.066(1)	0.0066(8)	−0.0063(9)	0.0042(7)
O(08)	4a		0.1972(1)	0.42573(8)	0.10414(7)	0.0636(9)	0.0457(7)	0.0616(9)	−0.0050(7)	−0.0060(7)	0.0040(7)
O(09)	4a		0.0038(1)	0.51364(8)	0.09070(7)	0.0508(8)	0.0528(8)	0.0695(9)	−0.0079(7)	0.0057(8)	0.0041(7)
O(10)	4a		−0.0009(2)	0.5773(2)	0.2111(1)	0.093(2)	0.102(2)	0.083(1)	−0.035(1)	−0.002(1)	0.002(1)
C(1)	4a	0.693(8)	0.5063(7)	0.6212(6)	0.3006(5)	0.114(5)	0.32(1)	0.24(1)	0.014(7)	−0.042(6)	−0.19(1)
O(1)	4a	0.693	0.4841(7)	0.5906(6)	0.2567(3)	0.192(6)	0.38(1)	0.170(5)	0.123(7)	−0.082(5)	−0.109(7)

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