Crystal structure of (3*S*,8*R*,9*R*)-3,9-dihydroxy-6′-methoxy-3-phenyl-10,11-dinorcinchonane hemihydrate, $(C_{24}H_{26}N_2O_3) \cdot 0.5H_2O$

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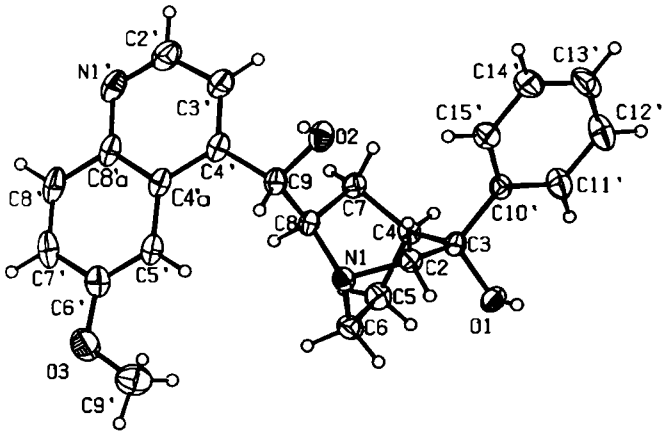

Source of material: The preparation is described in ref. 1. The compound is called *endo*-2d in ref.1. The Flack x parameter was not refinable. An intermolecular hydrogen bond exists between O1 and N1′. The solvent water molecule is probably disordered and its position on the twofold axis is an average position. *Endo*-2d exhibits an *anti*-open conformation which is characteristic for C-9 unprotected cinchona alkaloids (see ref. 2).

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless plate paralle (100), size 0.15 x 0.4 x 0.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.90 cm^{-1}
Diffractometer:	Stoe IPDS
Scan mode:	150 exposures, $\Delta\varphi = 1.5°$
$T_{measurement}$:	300 K
$2\theta_{max}$:	56.4°
$N(hkl)_{unique}$:	4946
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	281
Programs:	SHELXS-86, SHELXL-93, PLATON

$C_{24}H_{27}N_2O_{3.5}$, monoclinic, *I*121 (No. 5), $a = 11.108(2)$ Å, $b = 10.057(2)$ Å, $c = 19.187(3)$ Å, $\beta = 104.29(2)°$, $V = 2077.1$ Å^3, $Z = 4$, $R(F) = 0.036$, $R_w(F^2) = 0.038$.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	4*c*	1.0674(2)	0.5728(2)	0.91293(9)	0.039(2)
H(2)	4*c*	0.9773(2)	0.6792(2)	0.93193(9)	0.039(2)
H(3)	4*c*	1.137(3)	0.648(3)	0.813(1)	0.109(4)
H(4)	4*c*	0.8475(2)	0.7293(2)	0.7324(1)	0.030(2)
H(5)	4*c*	0.9055(2)	0.5083(2)	0.7183(1)	0.039(2)
H(6)	4*c*	0.7666(2)	0.5031(2)	0.7240(1)	0.039(2)
H(7)	4*c*	0.8357(2)	0.3631(2)	0.8146(1)	0.039(2)
H(8)	4*c*	0.9743(2)	0.4060(2)	0.8228(1)	0.039(2)
H(9)	4*c*	0.7730(2)	0.7796(2)	0.84020(9)	0.039(2)
H(10)	4*c*	0.6772(2)	0.6981(2)	0.78159(9)	0.039(2)
H(11)	4*c*	0.7044(2)	0.5149(2)	0.84407(9)	0.030(2)
H(12)	4*c*	0.7566(2)	0.5508(2)	0.9737(1)	0.030(2)
H(13)	4*c*	0.766(3)	0.742(3)	0.996(1)	0.109(4)
H(14)	4*c*	0.3464(2)	0.8131(2)	0.8848(1)	0.055(2)
H(15)	4*c*	0.5613(2)	0.8182(2)	0.9156(1)	0.055(2)
H(16)	4*c*	0.6629(2)	0.3671(2)	0.9357(1)	0.055(2)
H(17)	4*c*	0.3308(2)	0.1889(3)	0.8811(1)	0.055(2)
H(18)	4*c*	0.2332(2)	0.3901(3)	0.8706(1)	0.055(2)
H(19)	4*c*	0.7090(2)	0.0285(2)	0.9456(1)	0.109(4)
H(20)	4*c*	0.7185(2)	0.1648(2)	0.9863(1)	0.109(4)
H(21)	4*c*	0.7224(2)	0.1610(2)	0.9052(1)	0.109(4)
H(22)	4*c*	1.0869(2)	0.8610(2)	0.7502(1)	0.055(2)
H(23)	4*c*	1.1303(3)	1.0843(2)	0.7618(1)	0.055(2)
H(24)	4*c*	1.0831(2)	1.2027(2)	0.8545(1)	0.055(2)
H(25)	4*c*	0.9928(2)	1.0989(2)	0.9351(1)	0.055(2)
H(26)	4*c*	0.9539(2)	0.8739(2)	0.9255(1)	0.055(2)
H(27)	4*c*	–0.059(3)	0.379(2)	–0.010(2)	0.18(2)

Table 3. Final 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*c*	0.8874(1)	0.5159(1)	0.88404(7)	0.034(1)	0.0369(8)	0.0403(9)	–0.0020(8)	0.0041(8)	0.0041(8)
C(2)	4*c*	0.9879(2)	0.6166(2)	0.89544(9)	0.029(1)	0.0374(9)	0.040(1)	–0.001(1)	–0.0018(9)	0.003(1)
C(3)	4*c*	0.9883(2)	0.6937(2)	0.8252(1)	0.027(1)	0.042(1)	0.037(1)	–0.0015(9)	0.005(1)	0.0003(9)
O(1)	4*c*	1.0757(1)	0.6349(1)	0.79072(8)	0.0352(9)	0.0555(9)	0.055(1)	0.0007(8)	0.0150(8)	–0.0095(7)

Table 3. (Continued)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(4)	4*c*	0.8597(2)	0.6701(2)	0.7741(1)	0.035(1)	0.038(1)	0.035(1)	–0.0034(9)	0.001(1)	0.0021(9)
C(5)	4*c*	0.8510(2)	0.5245(2)	0.7498(1)	0.041(1)	0.053(1)	0.046(1)	–0.008(1)	0.002(1)	–0.014(1)
C(6)	4*c*	0.8907(2)	0.4391(2)	0.8185(1)	0.045(2)	0.038(1)	0.057(1)	–0.002(1)	0.008(1)	–0.005(1)
C(7)	4*c*	0.7585(2)	0.6953(2)	0.81498(9)	0.030(1)	0.045(1)	0.038(1)	0.0007(9)	0.001(1)	0.0035(9)
C(8)	4*c*	0.7645(2)	0.5814(2)	0.86856(9)	0.026(1)	0.044(1)	0.037(1)	–0.0044(9)	0.005(1)	–0.0006(9)
C(9)	4*c*	0.7257(2)	0.6191(2)	0.9372(1)	0.033(1)	0.052(1)	0.044(1)	–0.005(1)	0.006(1)	0.002(1)
O(2)	4*c*	0.7783(1)	0.7441(2)	0.96422(7)	0.045(1)	0.0676(8)	0.054(1)	–0.0119(8)	0.0132(8)	–0.0092(9)
N(1')	4*c*	0.3235(2)	0.6238(2)	0.88312(9)	0.033(1)	0.087(1)	0.066(1)	0.004(1)	0.012(1)	0.006(1)
C(2')	4*c*	0.3890(2)	0.7326(2)	0.8916(1)	0.046(2)	0.077(2)	0.070(2)	0.012(1)	0.014(1)	0.008(1)
C(3')	4*c*	0.5200(2)	0.7370(2)	0.9105(1)	0.043(2)	0.066(2)	0.062(2)	0.004(1)	0.016(1)	0.003(1)
C(4')	4*c*	0.5853(2)	0.6211(2)	0.9210(1)	0.033(1)	0.064(1)	0.039(1)	–0.001(1)	0.010(1)	0.006(1)
C(4'A)	4*c*	0.5179(2)	0.4997(2)	0.91262(9)	0.032(1)	0.065(1)	0.039(1)	–0.001(1)	0.011(1)	0.009(1)
C(5')	4*c*	0.5767(2)	0.3725(2)	0.9219(1)	0.036(1)	0.066(1)	0.047(1)	–0.005(1)	0.010(1)	0.012(1)
C(6')	4*c*	0.5076(2)	0.2590(3)	0.9109(1)	0.050(2)	0.063(1)	0.051(1)	–0.008(1)	0.012(1)	0.007(1)
C(7')	4*c*	0.3776(2)	0.2665(3)	0.8900(1)	0.053(2)	0.078(2)	0.069(2)	–0.030(2)	0.012(1)	0.005(2)
C(8')	4*c*	0.3196(2)	0.3868(3)	0.8828(1)	0.033(2)	0.096(2)	0.072(2)	–0.012(2)	0.013(1)	0.010(1)
C(8'A)	4*c*	0.3877(2)	0.5058(2)	0.8936(1)	0.032(1)	0.076(2)	0.051(1)	–0.005(1)	0.013(1)	0.009(1)
O(3)	4*c*	0.5555(2)	0.1337(2)	0.92004(8)	0.069(1)	0.061(1)	0.086(1)	–0.008(1)	0.013(1)	0.0101(9)
C(9')	4*c*	0.6870(2)	0.1209(2)	0.9410(1)	0.079(2)	0.073(1)	0.077(2)	0.016(2)	0.004(2)	0.008(1)
C(10')	4*c*	1.0172(2)	0.8411(2)	0.8372(1)	0.029(1)	0.041(1)	0.040(1)	–0.0058(9)	0.001(1)	–0.002(1)
C(11')	4*c*	1.0690(2)	0.9079(2)	0.7882(1)	0.069(2)	0.054(1)	0.055(1)	–0.016(1)	0.023(1)	–0.000(1)
C(12')	4*c*	1.0943(3)	1.0417(2)	0.7946(1)	0.088(2)	0.064(2)	0.081(2)	–0.026(1)	0.029(2)	0.012(1)
C(13')	4*c*	1.0659(2)	1.1122(2)	0.8499(1)	0.060(2)	0.040(1)	0.100(2)	–0.014(1)	–0.004(1)	0.000(1)
C(14')	4*c*	1.0129(2)	1.0504(2)	0.8982(1)	0.064(2)	0.051(1)	0.080(2)	–0.008(1)	0.016(2)	–0.017(1)
C(15')	4*c*	0.9892(2)	0.9157(2)	0.8921(1)	0.054(2)	0.049(1)	0.057(2)	–0.004(1)	0.015(1)	–0.005(1)
O(4W)	2*a*	0	0.3353(3)	0	0.259(6)	0.040(1)	0.061(2)	0	–0.006(2)	0

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