SUPPLEMENTARY MATERIAL

Chiroptical Study and Absolute Configuration of Securinine Oxidation Products

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Abstract

TDDFT ECD spectra prediction was carried out to study the absolute configuration of phyllanthidine-type derivatives **5** and **6**, derived from securinine (**1**) and its enantiomer virosecurinine (**2**), respectively. This method demonstrated to be very reliable in this alkaloid series. Thus, **5** and **6** shared the same stereochemistry as their parent precursors, confirming the retentive nature of the oxidation sequence. In addition, this study highlighted the key role of the methylene bridge (BC ring) in the chiroptical activity of these compounds. These results fully clarified the stereochemical relationships between the phyllanthidine and the securinine subgroups.

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1. NMR and Mass spectra

¹H NMR spectra were recorded in CDCl₃ on a BRUKER AVANCE spectrometer at 400 MHz. Chemical shifts were corrected to δ_{H} 7.26 (¹H) and δ_{C} 77.00 (¹³C) for CDCl₃. FID signals were processed with ACD/NMR software. Mass spectra were measured with a ZQ mass spectrometer (ESI) and high resolution mass spectra were recorded at Institut de Chimie Organique d'Orléans (ICOA).

Securinine

AW-AT2viet-17-F2.001.esp VerticalScaleFactor = 1

Virosecurinine

15-Nov-2012
17:01:52
Scan ES+
1.29e8

ExT.1(fr.3-7) CH₂Cl₂/CH₃OH/HCOOH Vc 40V
Z5183 739 (25.877) Cm (736.771)

Analysis Info

Sample Name **FHP-AT2**
Analysis Name X016896CYC.d

Acquisition Date 26/11/2014 13:39:45
Instrument / Ser# maXis 255552.00086
Method Positif.m

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan End	3000 m/z	Set Collision Cell RF	500.0 Vpp	Set Dry Gas	7.0 l/min

Meas. m/z	z	#	Ion Formula	m/z	err [ppm]	mSigma	rdb	e ⁻	Conf
218.117517	1+	1	C ₁₃ H ₁₆ NO ₂	218.117555	0.2	0.8	6.5	even	

Allosecurinine

FHP-Securinine Lot China.002
VerticalScaleFactor = 1

Derivative 5'

fhp-v80-213c400.002.esp VerticalScaleFactor = 1

Derivative 6

06-Dec-2013
14:29:43
Scan ES+
2.23e7

K9282 Vc 20V
Z6365 4 (0.140) Cm (1:19)

Analysis Info

Sample Name **K9282**
Analysis Name X016902CYC.d

Acquisition Date 26/11/2014 13:48:52
Instrument / Ser# maXis 255552.00086
Method Positif.m

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.6 Bar
Scan Begin	50 m/z	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan End	3000 m/z	Set Collision Cell RF	500.0 Vpp	Set Dry Gas	7.0 l/min

Meas. m/z	z	#	Ion Formula	m/z	err [ppm]	mSigma	rdb	e ⁻	Conf
234.112485	1+	1	C ₁₃ H ₁₆ NO ₃	234.112470	0.1	0.9	6.5	even	

Phyllanthidine

Phyllanthidine

fhp-v83-214a.002.esp VerticalScaleFactor = 1

2. Optical Rotation Values

Rotary values were recorded on a Perkin Elmer Model 341 polarimeter at 20°C.

Compound	$[\alpha]_D$ in CHCl_3	$[\alpha]_D$ in ethanol
securinine (1)		-1100, (c 0.19), lit -1042 (c 0.43)
virosecurinine (2)		+1028 (c 1.2), lit +1035 (c 0.43)
Phyllanthidine (7)	- 420 (c 0.03), lit - 450 (c 0.33)	- 300 (c 0.03)
<i>ent</i> -Phyllanthidine (8)	lit + 333 (concentration not given)	no data
derivative 5 from 1	- 268 (c 0.04)	- 324 (c 0.14)
derivative 6 from 2	+ 254 (c 0.05), lit + 370 (c 0.73)	+ 312 (c 0.7)

3. X-Ray Analysis

3.1 Crystal Data and Details of the Structure Determination of Virosecurinine 2

Crystal Data for Virosecurinine

Formula	C ₁₃ H ₁₅ N O ₂
Formula Weight	217.26
Crystal System	orthorhombic
Space group	P2 ₁ 2 ₁ 2 ₁ (No. 19)
a, b, c [Angstrom]	7.0429(1) 9.4757(1) 16.4775(11)
V [Ang**3]	1099.65(8)
Z	4

D(calc) [g/cm ³]	1.312
Mu(CuKα) [/mm]	0.713
F(000)	464
Crystal Size [mm]	0.55 x 0.30 x 0.25

Data Collection

Diffractometer	Rigaku RAXIS-UNKNOWN
Radiation	CuKα (λ = 1.54187 Å)
Voltage, Current	50kV, 40mA
Temperature	20.0°C
Detector Aperture	460 x 256 mm
Data Images	69 exposures
ω oscillation Range	20.0 - 200.0°
Exposure Rate	30.0 sec. /°
ω oscillation Range	20.0 - 185.0°
Exposure Rate	30.0 sec. /°
ω oscillation Range	20.0 - 200.0°
Exposure Rate	30.0 sec. /°
ω oscillation Range	20.0 - 185.0°
Exposure Rate	30.0 sec. /°
Detector Position	127.40 mm
Pixel Size	0.100 mm
2θ _{max}	136.5°
No. of Reflections Measured	Total: 4989 Unique: 1963 (R _{int} = 0.0274) Friedel pairs: 788
Corrections	Lorentz-polarization Absorption
(trans. factors: 0.646 - 0.875)	

Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F ²
Function Minimized	Σ w (Fo ² - Fc ²) ²
Least Squares Weights	w = 1 / [σ ² (Fo ²) + (0.0373 · P) ² + 0.1445 · P] where P = (Max(Fo ² , 0) + 2Fc ²)/3
2θ _{max} cutoff	136.5°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	1960
No. Variables	146
Reflection/Parameter Ratio	13.42
Residuals: R1 (I > 2.00σ(I))	0.0295
Residuals: R (All reflections)	0.0351

Residuals: wR2 (All reflections)	0.0789
Goodness of Fit Indicator	1.017
Flack Parameter (Friedel pairs = 788)	-0.1(3)
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.14 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.13 e ⁻ /Å ³

Table S2 - Final Coordinates and Equivalent Isotropic Displacement
Parameters of the non-Hydrogen atoms

Atom	x	y	z	U(eq) [Ang ²]
----	---	---	---	-----
O10	0.52543(16)	0.48742(12)	0.35169(7)	0.0486(4)
O11	0.74421(19)	0.45018(17)	0.44802(8)	0.0714(5)
N1	0.16370(19)	0.41013(15)	0.20833(7)	0.0448(4)
C2	0.3555(2)	0.37323(16)	0.23509(8)	0.0389(5)
C3	0.4951(2)	0.4353(2)	0.17566(10)	0.0518(6)
C4	0.4519(3)	0.3724(2)	0.09202(10)	0.0612(7)
C5	0.2436(3)	0.3891(3)	0.06846(10)	0.0636(7)
C6	0.1102(3)	0.3366(2)	0.13452(10)	0.0557(6)
C7	0.0452(2)	0.4140(2)	0.28186(9)	0.0505(5)
C8	0.1729(3)	0.50896(18)	0.33381(10)	0.0491(6)
C9	0.3541(2)	0.42087(16)	0.32515(9)	0.0380(4)
C11	0.6008(2)	0.4114(2)	0.41526(10)	0.0500(5)
C12	0.4783(2)	0.29065(18)	0.43064(9)	0.0498(6)
C13	0.3320(2)	0.29604(16)	0.37969(8)	0.0412(5)
C14	0.1585(3)	0.21598(19)	0.36868(9)	0.0498(5)
C15	0.0212(2)	0.2713(2)	0.32273(10)	0.0552(6)

U(eq) = 1/3 of the trace of the orthogonalized U Tensor

Table S3 - Hydrogen Atom Positions and Isotropic Displacement
Parameters

Atom	x	y	z	U(iso) [Ang ²]
----	---	---	---	-----
H2	0.36808	0.27026	0.23364	0.0467
H3A	0.62396	0.41233	0.19177	0.0622
H3B	0.48238	0.53717	0.17411	0.0622
H4A	0.53055	0.41855	0.05165	0.0735
H4B	0.48425	0.27288	0.09225	0.0735

H5A	0.21752	0.48789	0.05778	0.0764
H5B	0.21965	0.33684	0.01888	0.0764
H6A	-0.02058	0.35735	0.12046	0.0669
H6B	0.12342	0.23545	0.14155	0.0669
H7	-0.07787	0.45860	0.27124	0.0606
H8A	0.18661	0.60286	0.31111	0.0589
H8B	0.12994	0.51512	0.38963	0.0589
H12	0.49887	0.22129	0.46959	0.0597
H14	0.14319	0.12819	0.39305	0.0597
H15	-0.09132	0.22143	0.31558	0.0662

The Temperature Factor has the Form of $\text{Exp}(-T)$ Where
 $T = 8 \cdot (\pi^2) \cdot U \cdot (\sin(\theta)/\lambda)^2$ for Isotropic Atoms

Table S4 - (An)isotropic Displacement Parameters

Atom	U(1,1) or U	U(2,2)	U(3,3)	U(2,3)	U(1,3)	U(1,2)
----	-----	-----	-----	-----	-----	-----
O10	0.0479(7)	0.0494(7)	0.0486(6)	0.0055(5)	-0.0120(5)	-0.0095(5)
O11	0.0584(8)	0.0942(12)	0.0615(8)	0.0030(8)	-0.0228(7)	-0.0091(7)
N1	0.0394(7)	0.0560(8)	0.0389(7)	-0.0010(7)	-0.0048(6)	-0.0025(7)
C2	0.0394(8)	0.0382(8)	0.0392(8)	0.0025(6)	0.0005(7)	-0.0013(7)
C3	0.0453(9)	0.0611(11)	0.0491(9)	0.0090(8)	0.0034(7)	-0.0090(9)
C4	0.0703(12)	0.0694(13)	0.0439(10)	0.0063(9)	0.0169(9)	-0.0096(10)
C5	0.0801(13)	0.0748(14)	0.0360(8)	0.0000(10)	-0.0016(9)	-0.0170(11)
C6	0.0557(11)	0.0688(12)	0.0427(9)	-0.0036(9)	-0.0063(8)	-0.0118(9)
C7	0.0369(8)	0.0702(11)	0.0444(8)	-0.0069(9)	-0.0011(7)	0.0040(9)
C8	0.0518(10)	0.0510(10)	0.0444(9)	-0.0059(8)	-0.0037(8)	0.0118(9)
C9	0.0368(8)	0.0379(8)	0.0393(7)	-0.0003(7)	-0.0028(6)	-0.0019(7)
C11	0.0474(9)	0.0609(11)	0.0416(8)	-0.0035(8)	-0.0070(7)	0.0053(8)
C12	0.0632(11)	0.0488(10)	0.0373(8)	0.0020(7)	-0.0035(8)	0.0062(9)
C13	0.0497(9)	0.0413(8)	0.0327(7)	-0.0031(7)	0.0062(7)	0.0024(8)
C14	0.0613(10)	0.0516(10)	0.0364(8)	-0.0018(8)	0.0119(8)	-0.0145(9)
C15	0.0441(9)	0.0750(12)	0.0464(9)	-0.0103(9)	0.0101(8)	-0.0189(9)

The Temperature Factor has the Form of $\text{Exp}(-T)$ Where
 $T = 8 \cdot (\pi^2) \cdot U \cdot (\sin(\theta)/\lambda)^2$ for Isotropic Atoms
 $T = 2 \cdot (\pi^2) \cdot \sum_{ij} h(i) \cdot h(j) \cdot U(i,j) \cdot A^*(i) \cdot A^*(j)$, for
Anisotropic Atoms. $A^*(i)$ are Reciprocal Axial Lengths and
 $h(i)$ are the Reflection Indices.

Table S5 - Bond Distances (Angstrom)

O10	-C9	1.4300(18)	C13	-C14	1.450(2)
O10	-C11	1.378(2)	C14	-C15	1.335(2)
O11	-C11	1.203(2)	C2	-H2	0.9800
N1	-C2	1.4634(19)	C3	-H3A	0.9700
N1	-C6	1.451(2)	C3	-H3B	0.9700
N1	-C7	1.4717(19)	C4	-H4A	0.9700
C2	-C3	1.507(2)	C4	-H4B	0.9700
C2	-C9	1.551(2)	C5	-H5A	0.9700
C3	-C4	1.532(2)	C5	-H5B	0.9700
C4	-C5	1.526(3)	C6	-H6A	0.9700
C5	-C6	1.522(3)	C6	-H6B	0.9700
C7	-C8	1.533(2)	C7	-H7	0.9800
C7	-C15	1.520(3)	C8	-H8A	0.9700
C8	-C9	1.532(2)	C8	-H8B	0.9700
C9	-C13	1.494(2)	C12	-H12	0.9300
C11	-C12	1.455(2)	C14	-H14	0.9300
C12	-C13	1.330(2)	C15	-H15	0.9300

Table S6 - Bond Angles (Degrees)

C9	-O10	-C11	109.09(12)	C13	-C14	-C15	118.42(16)
C2	-N1	-C6	112.18(13)	C7	-C15	-C14	121.33(15)
C2	-N1	-C7	106.34(11)	N1	-C2	-H2	108.00
C6	-N1	-C7	123.68(14)	C3	-C2	-H2	108.00
N1	-C2	-C3	108.25(12)	C9	-C2	-H2	108.00
N1	-C2	-C9	102.29(11)	C2	-C3	-H3A	110.00
C3	-C2	-C9	120.81(13)	C2	-C3	-H3B	110.00
C2	-C3	-C4	107.65(14)	C4	-C3	-H3A	110.00
C3	-C4	-C5	112.30(15)	C4	-C3	-H3B	110.00
C4	-C5	-C6	112.20(16)	H3A	-C3	-H3B	108.00
N1	-C6	-C5	106.39(16)	C3	-C4	-H4A	109.00
N1	-C7	-C8	98.14(12)	C3	-C4	-H4B	109.00
N1	-C7	-C15	113.93(14)	C5	-C4	-H4A	109.00
C8	-C7	-C15	109.87(13)	C5	-C4	-H4B	109.00
C7	-C8	-C9	96.71(13)	H4A	-C4	-H4B	108.00
O10	-C9	-C2	114.55(12)	C4	-C5	-H5A	109.00
O10	-C9	-C8	115.74(13)	C4	-C5	-H5B	109.00
O10	-C9	-C13	104.66(11)	C6	-C5	-H5A	109.00
C2	-C9	-C8	104.66(12)	C6	-C5	-H5B	109.00
C2	-C9	-C13	110.23(12)	H5A	-C5	-H5B	108.00
C8	-C9	-C13	106.78(12)	N1	-C6	-H6A	110.00
O10	-C11	-O11	120.34(16)	N1	-C6	-H6B	110.00
O10	-C11	-C12	108.37(12)	C5	-C6	-H6A	110.00

O11	-C11	-C12	131.29(17)	C5	-C6	-H6B	110.00
C11	-C12	-C13	108.61(14)	H6A	-C6	-H6B	109.00
C9	-C13	-C12	109.24(13)	N1	-C7	-H7	111.00
C9	-C13	-C14	115.28(12)	C8	-C7	-H7	111.00
C12	-C13	-C14	135.39(14)	C15	-C7	-H7	111.00
C7	-C8	-H8A	112.00	C13	-C12	-H12	126.00
C7	-C8	-H8B	112.00	C13	-C14	-H14	121.00
C9	-C8	-H8A	112.00	C15	-C14	-H14	121.00
C9	-C8	-H8B	112.00	C7	-C15	-H15	119.00
H8A	-C8	-H8B	110.00	C14	-C15	-H15	119.00
C11	-C12	-H12	126.00				

Table S7 - Torsion Angles (Degrees)

C11	-O10	-C9	-C13	0.28(15)
C9	-O10	-C11	-C12	0.75(17)
C11	-O10	-C9	-C8	117.51(14)
C9	-O10	-C11	-O11	-178.70(15)
C11	-O10	-C9	-C2	-120.55(14)
C7	-N1	-C2	-C3	-152.32(14)
C7	-N1	-C2	-C9	-23.72(15)
C6	-N1	-C2	-C9	-161.77(13)
C7	-N1	-C6	-C5	165.97(16)
C6	-N1	-C7	-C15	65.98(19)
C2	-N1	-C6	-C5	-64.47(18)
C6	-N1	-C7	-C8	-177.97(15)
C6	-N1	-C2	-C3	69.64(17)
C2	-N1	-C7	-C15	-65.94(15)
C2	-N1	-C7	-C8	50.11(15)
C9	-C2	-C3	-C4	-176.95(14)
N1	-C2	-C9	-O10	-139.64(12)
C3	-C2	-C9	-C13	-137.15(14)
N1	-C2	-C9	-C8	-11.84(15)
N1	-C2	-C3	-C4	-59.72(17)
C3	-C2	-C9	-C8	108.37(15)
C3	-C2	-C9	-O10	-19.44(19)
N1	-C2	-C9	-C13	102.65(13)
C2	-C3	-C4	-C5	52.5(2)
C3	-C4	-C5	-C6	-51.2(2)
C4	-C5	-C6	-N1	54.2(2)
N1	-C7	-C8	-C9	-53.89(14)
C8	-C7	-C15	-C14	-31.1(2)
N1	-C7	-C15	-C14	77.8(2)

C15	-C7	-C8	-C9	65.28(15)
C7	-C8	-C9	-C2	40.49(14)
C7	-C8	-C9	-C13	-76.39(14)
C7	-C8	-C9	-O10	167.57(12)
O10	-C9	-C13	-C14	175.57(13)
C2	-C9	-C13	-C12	122.33(13)
C8	-C9	-C13	-C12	-124.54(14)
C8	-C9	-C13	-C14	52.36(17)
C2	-C9	-C13	-C14	-60.77(16)
O10	-C9	-C13	-C12	-1.32(16)
O10	-C11	-C12	-C13	-1.63(18)
O11	-C11	-C12	-C13	177.75(18)
C11	-C12	-C13	-C9	1.79(17)
C11	-C12	-C13	-C14	-174.21(17)
C9	-C13	-C14	-C15	-11.3(2)
C12	-C13	-C14	-C15	164.48(18)
C13	-C14	-C15	-C7	0.6(2)

Table S8 - Contact Distances(Angstrom)

O10	.C3	2.950(2)	C5	.C2	2.861(2)
O10	.C12	2.298(2)	C6	.C3	2.947(3)
O11	.C15_b	3.308(2)	C6	.C15	3.224(2)
O10	.H3A	2.8200	C7	.C9	2.290(2)
O10	.H8B	2.8700	C7	.C13	2.816(2)
O10	.H4B_a	2.8600	C8	.C3	3.525(2)
O10	.H8A	2.7100	C8	.C2	2.440(2)
O11	.H12	2.8000	C8	.C12	3.384(2)
O11	.H4A_c	2.6400	C8	.C14	2.837(2)
O11	.H12_d	2.7700	C8	.C11	3.426(3)
O11	.H14_d	2.8100	C9	.C12	2.304(2)
N1	.C4	2.814(2)	C9	.C15	2.740(2)
N1	.C8	2.271(2)	C9	.C7	2.290(2)
N1	.C13	3.2475(18)	C11	.C8	3.426(3)
N1	.C14	3.220(2)	C11	.C13	2.263(2)
N1	.H3B	2.6100	C11	.C15_b	3.585(2)
N1	.H5A	2.6200	C11	.C2	3.454(2)
N1	.H8A	2.5000	C12	.C9	2.304(2)
C2	.C5	2.861(2)	C12	.C14_d	3.542(2)
C2	.C8	2.440(2)	C12	.C8	3.384(2)
C2	.C11	3.454(2)	C12	.C2	3.427(2)
C2	.C12	3.427(2)	C13	.C11	2.263(2)
C2	.C14	2.998(2)	C13	.C7	2.816(2)

C2	.C15	2.926(2)	C13	.N1	3.2475(18)
C3	.O10	2.950(2)	C14	.C8	2.837(2)
C3	.C6	2.947(3)	C14	.N1	3.220(2)
C3	.C8	3.525(2)	C14	.C2	2.998(2)
C4	.N1	2.814(2)	C14	.C12_g	3.542(2)
C15	.C9	2.740(2)	C14	.H2	2.7200
C15	.C6	3.224(2)	C14	.H8B	2.8600
C15	.C11_h	3.585(2)	C14	.H12	2.9200
C15	.C2	2.926(2)	C15	.H2	2.8500
C15	.O11_h	3.308(2)	C15	.H6B	3.0900
C2	.H6B	2.6000	C15	.H8B	2.6700
C2	.H4B	2.7000	C15	.H8A_i	3.0900
C2	.H8A	2.7800	H2	.C4	2.5900
C3	.H5A	2.8000	H2	.C5	3.0700
C4	.H6B	2.7800	H2	.C6	2.5200
C4	.H2	2.5900	H2	.C7	2.7700
C5	.H3B	2.8000	H2	.C13	2.4300
C5	.H2	3.0700	H2	.C14	2.7200
C6	.H2	2.5200	H2	.C15	2.8500
C6	.H7	2.8600	H2	.H3A	2.3500
C6	.H4B	2.7900	H2	.H4B	2.4700
C7	.H2	2.7700	H2	.H6B	2.3200
C7	.H6A	2.7500	H3A	.O10	2.8200
C7	.H6B	2.9200	H3A	.C9	2.9100
C8	.H6B_e	3.0200	H3A	.H2	2.3500
C9	.H3A	2.9100	H3A	.H4A	2.4000
C9	.H3B	2.8700	H3A	.H4B	2.3200
C12	.H14	2.8900	H3A	.H7_b	2.5100
C12	.H3B_f	2.9700	H3B	.N1	2.6100
C13	.H8B	2.5200	H3B	.C5	2.8000
C13	.H2	2.4300	H3B	.C9	2.8700
C13	.H3B_f	2.9200	H3B	.H4A	2.3300
C14	.H12_g	2.9500	H3B	.C12_a	2.9700
H3B	.C13_a	2.9200	H6B	.H5B	2.3400
H4A	.H3A	2.4000	H6B	.C8_i	3.0200
H4A	.H3B	2.3300	H7	.C6	2.8600
H4A	.H5A	2.3000	H7	.H3A_h	2.5100
H4A	.H5B	2.3800	H7	.H8A	2.4000
H4A	.O11_j	2.6400	H7	.H8B	2.5000
H4B	.C2	2.7000	H7	.H15	2.3700
H4B	.C6	2.7900	H8A	.O10	2.7100
H4B	.H2	2.4700	H8A	.N1	2.5000
H4B	.H3A	2.3200	H8A	.C2	2.7800
H4B	.H5B	2.3000	H8A	.H7	2.4000

H4B	.O10_f	2.8600	H8A	.C15_e	3.0900
H5A	.N1	2.6200	H8A	.H15_e	2.4600
H5A	.C3	2.8000	H8B	.O10	2.8700
H5A	.H4A	2.3000	H8B	.C13	2.5200
H5A	.H6A	2.3300	H8B	.C14	2.8600
H5B	.H4A	2.3800	H8B	.C15	2.6700
H5B	.H4B	2.3000	H8B	.H7	2.5000
H5B	.H6A	2.3900	H12	.O11	2.8000
H5B	.H6B	2.3400	H12	.C14	2.9200
H6A	.C7	2.7500	H12	.O11_g	2.7700
H6A	.H5A	2.3300	H12	.C14_d	2.9500
H6A	.H5B	2.3900	H14	.C12	2.8900
H6B	.C2	2.6000	H14	.H15	2.2700
H6B	.C4	2.7800	H14	.O11_g	2.8100
H6B	.C7	2.9200	H15	.H7	2.3700
H6B	.C15	3.0900	H15	.H14	2.2700
H6B	.H2	2.3200	H15	.H8A_i	2.4600

3.2 Crystal Data and Details of the Structure Determination of Virosecurinine derivative 6

Formula	C ₁₃ H ₁₅ N O ₃		
Formula Weight	233.26		
Crystal System	monoclinic		
Space group	P21	(No. 4)	
a, b, c [Angstrom]	8.091(3)	7.089(3)	10.710(7)
alpha, beta, gamma [deg]	90	109.171(8)	90
V [Ang**3]	580.2(5)		
Z	2		
D(calc) [g/cm**3]	1.335		
Mu(CuKa) [/mm]	0.780		
F(000)	248		

Crystal Size [mm] 0.40 x 0.10 x 0.10

Data Collection

Temperature (K) 293(2)
 Radiation [Angstrom] CuK α 1.54187
 Theta Min-Max [Deg] 4.4, 68.2
 Dataset 9:-6 ; 8:-6 ; 8:-12
 Tot., Uniq. Data, R(int) 3999, 1808, 0.032
 Observed data [I > 0.2 sigma(I)] 1519

Refinement

Nref, Npar 1808, 158
 R, wR2, S 0.0326, 0.0784, 0.99
 $w = 1/[\sigma^2(F_o^2) + (0.0487P)^2 + 0.0360P]$ where $P = (F_o^2 + 2F_c^2)/3$
 Max. and Av. Shift/Error 0.00, 0.00
 Min. and Max. Resd. Dens. [e/Ang³] -0.23, 0.24

Table S2 - Final Coordinates and Equivalent Isotropic Displacement
 Parameters of the non-Hydrogen atoms

Atom	x	y	z	U(eq) [Ang ²]
----	---	---	---	-----
O7	0.17532(17)	0.0872(2)	0.62597(14)	0.0395(5)
O11	0.56298(16)	0.4628(2)	0.82598(13)	0.0370(5)
O12	0.8386(2)	0.5496(3)	0.93407(16)	0.0553(6)
N1	0.3234(2)	0.1126(3)	0.57830(15)	0.0315(5)
C2	0.4037(3)	0.2965(3)	0.62608(19)	0.0290(6)
C3	0.2870(2)	0.4651(3)	0.56953(19)	0.0338(7)
C4	0.2237(3)	0.4580(3)	0.4187(2)	0.0389(7)
C5	0.1275(3)	0.2738(3)	0.3719(2)	0.0405(7)
C6	0.2461(3)	0.1078(3)	0.43321(19)	0.0365(7)
C8	0.2441(3)	0.0638(3)	0.7681(2)	0.0427(7)
C9	0.3188(3)	0.2540(3)	0.8301(2)	0.0403(8)
C10	0.4702(2)	0.2882(3)	0.77917(18)	0.0304(6)
C12	0.7370(3)	0.4235(4)	0.8900(2)	0.0391(7)
C13	0.7603(3)	0.2195(3)	0.8947(2)	0.0405(7)
C14	0.6050(3)	0.1380(3)	0.83528(19)	0.0352(7)
C15	0.5477(3)	-0.0548(4)	0.83309(19)	0.0425(8)
C16	0.3770(3)	-0.0910(4)	0.8051(2)	0.0460(8)

U(eq) = 1/3 of the trace of the orthogonalized U Tensor

Table S3 - Hydrogen Atom Positions and Isotropic Displacement
 Parameters

Atom	x	y	z	U(iso) [Ang ²]
----	---	---	---	-----

H2	0.507(2)	0.297(3)	0.5958(19)	0.037(6)
H3A	0.35172	0.58085	0.59960	0.0406
H3B	0.18716	0.46412	0.60068	0.0406
H4A	0.14617	0.56348	0.38321	0.0466
H4B	0.32293	0.46752	0.38713	0.0466
H5A	0.09233	0.26620	0.27633	0.0486
H5B	0.02296	0.26914	0.39722	0.0486
H6A	0.17902	-0.00772	0.40813	0.0438
H6B	0.34019	0.10311	0.39586	0.0438
H8	0.14714	0.02959	0.79950	0.0513
H9A	0.23193	0.35322	0.80160	0.0483
H9B	0.35893	0.24695	0.92581	0.0483
H13	0.86571	0.15636	0.93274	0.0485
H15	0.62835	-0.15298	0.85095	0.0509
H16	0.34031	-0.21466	0.80860	0.0552

The Temperature Factor has the Form of $\text{Exp}(-T)$ Where
 $T = 8 \cdot (\pi^2) \cdot U \cdot (\sin(\Theta)/\lambda)^2$ for Isotropic Atoms

Table S4 - (An)isotropic Displacement Parameters

Atom	U(1,1) or U	U(2,2)	U(3,3)	U(2,3)	U(1,3)	U(1,2)
----	-----	-----	-----	-----	-----	-----
O7	0.0332(7)	0.0475(11)	0.0361(8)	-0.0016(7)	0.0091(6)	-0.0101(7)
O11	0.0316(7)	0.0324(10)	0.0397(7)	-0.0049(7)	0.0017(6)	-0.0035(7)
O12	0.0410(8)	0.0526(12)	0.0594(10)	-0.0106(9)	-0.0012(8)	-0.0161(8)
N1	0.0303(8)	0.0305(11)	0.0312(9)	-0.0022(7)	0.0066(7)	-0.0026(8)
C2	0.0251(9)	0.0281(12)	0.0327(9)	0.0018(10)	0.0082(8)	0.0009(9)
C3	0.0283(9)	0.0309(14)	0.0401(11)	-0.0004(10)	0.0082(9)	0.0022(9)
C4	0.0318(9)	0.0395(15)	0.0408(11)	0.0097(10)	0.0057(9)	0.0055(10)
C5	0.0346(10)	0.0480(17)	0.0326(10)	0.0017(10)	0.0025(9)	0.0075(10)
C6	0.0371(10)	0.0396(14)	0.0305(10)	-0.0029(9)	0.0078(8)	0.0049(10)
C8	0.0436(12)	0.0486(16)	0.0357(10)	-0.0033(11)	0.0126(9)	-0.0171(12)
C9	0.0388(11)	0.0481(18)	0.0326(11)	-0.0050(10)	0.0100(9)	-0.0081(11)
C10	0.0313(9)	0.0250(12)	0.0309(10)	-0.0039(9)	0.0047(8)	-0.0040(9)
C12	0.0319(10)	0.0468(16)	0.0336(11)	-0.0029(12)	0.0039(9)	-0.0045(11)
C13	0.0356(11)	0.0407(16)	0.0376(11)	-0.0003(10)	0.0019(9)	0.0100(10)
C14	0.0423(12)	0.0336(15)	0.0283(10)	-0.0004(9)	0.0098(9)	0.0028(11)
C15	0.0598(14)	0.0316(16)	0.0303(10)	0.0030(10)	0.0071(10)	0.0063(12)
C16	0.0652(15)	0.0336(15)	0.0325(11)	0.0032(10)	0.0071(11)	-0.0123(12)

The Temperature Factor has the Form of $\text{Exp}(-T)$ Where
 $T = 8 \cdot (\pi^2) \cdot U \cdot (\sin(\Theta)/\lambda)^2$ for Isotropic Atoms
 $T = 2 \cdot (\pi^2) \cdot \sum_{ij} h(i) \cdot h(j) \cdot U(i,j) \cdot A^*(i) \cdot A^*(j)$, for
Anisotropic Atoms. $A^*(i)$ are Reciprocal Axial Lengths and
 $h(i)$ are the Reflection Indices.

Table S5 - Bond Distances (Angstrom)

O7	-N1	1.460(2)	C14	-C15	1.441(4)
O7	-C8	1.448(3)	C15	-C16	1.338(4)
O11	-C10	1.449(3)	C2	-H2	0.991(18)
O11	-C12	1.377(3)	C3	-H3A	0.9700
O12	-C12	1.201(3)	C3	-H3B	0.9700
N1	-C2	1.472(3)	C4	-H4A	0.9700
N1	-C6	1.473(3)	C4	-H4B	0.9700
C2	-C3	1.520(3)	C5	-H5A	0.9700
C2	-C10	1.550(3)	C5	-H5B	0.9700
C3	-C4	1.527(3)	C6	-H6A	0.9700
C4	-C5	1.519(3)	C6	-H6B	0.9700
C5	-C6	1.524(3)	C8	-H8	0.9800
C8	-C9	1.536(3)	C9	-H9A	0.9700
C8	-C16	1.496(4)	C9	-H9B	0.9700
C9	-C10	1.515(3)	C13	-H13	0.9300
C10	-C14	1.501(3)	C15	-H15	0.9300
C12	-C13	1.457(4)	C16	-H16	0.9300
C13	-C14	1.339(3)			

Table S6 - Bond Angles (Degrees)

N1	-O7	-C8	107.79(15)	C13	-C14	-C15	131.6(2)
C10	-O11	-C12	109.17(17)	C14	-C15	-C16	119.2(2)
O7	-N1	-C2	107.88(16)	C8	-C16	-C15	121.2(2)
O7	-N1	-C6	104.67(15)	N1	-C2	-H2	102.3(12)
C2	-N1	-C6	111.93(17)	C3	-C2	-H2	110.3(12)
N1	-C2	-C3	114.36(18)	C10	-C2	-H2	108.0(11)
N1	-C2	-C10	107.13(16)	C2	-C3	-H3A	110.00
C3	-C2	-C10	113.94(17)	C2	-C3	-H3B	110.00
C2	-C3	-C4	110.14(17)	C4	-C3	-H3A	110.00
C3	-C4	-C5	109.58(17)	C4	-C3	-H3B	110.00
C4	-C5	-C6	109.86(19)	H3A	-C3	-H3B	108.00
N1	-C6	-C5	115.21(18)	C3	-C4	-H4A	110.00
O7	-C8	-C9	108.33(16)	C3	-C4	-H4B	110.00
O7	-C8	-C16	110.92(17)	C5	-C4	-H4A	110.00
C9	-C8	-C16	112.5(2)	C5	-C4	-H4B	110.00
C8	-C9	-C10	103.57(18)	H4A	-C4	-H4B	108.00
O11	-C10	-C2	107.10(16)	C4	-C5	-H5A	110.00
O11	-C10	-C9	113.60(16)	C4	-C5	-H5B	110.00
O11	-C10	-C14	103.98(15)	C6	-C5	-H5A	110.00
C2	-C10	-C9	110.25(16)	C6	-C5	-H5B	110.00
C2	-C10	-C14	113.87(17)	H5A	-C5	-H5B	108.00
C9	-C10	-C14	108.02(17)	N1	-C6	-H6A	108.00
O11	-C12	-O12	120.1(2)	N1	-C6	-H6B	108.00
O11	-C12	-C13	108.5(2)	C5	-C6	-H6A	108.00
O12	-C12	-C13	131.4(2)	C5	-C6	-H6B	108.00
C12	-C13	-C14	108.7(2)	H6A	-C6	-H6B	107.00
C10	-C14	-C13	109.22(18)	O7	-C8	-H8	108.00

C10	-C14	-C15	118.6(2)	C9	-C8	-H8	108.00
C16	-C8	-H8	108.00	C12	-C13	-H13	126.00
C8	-C9	-H9A	111.00	C14	-C13	-H13	126.00
C8	-C9	-H9B	111.00	C14	-C15	-H15	120.00
C10	-C9	-H9A	111.00	C16	-C15	-H15	120.00
C10	-C9	-H9B	111.00	C8	-C16	-H16	119.00
H9A	-C9	-H9B	109.00	C15	-C16	-H16	119.00

Table S7 - Torsion Angles (Degrees)

C8	-O7	-N1	-C2	-68.56(18)
C8	-O7	-N1	-C6	172.09(16)
N1	-O7	-C8	-C16	-53.7(2)
N1	-O7	-C8	-C9	70.3(2)
C10	-O11	-C12	-O12	178.29(19)
C12	-O11	-C10	-C14	5.90(19)
C10	-O11	-C12	-C13	-3.9(2)
C12	-O11	-C10	-C9	123.07(18)
C12	-O11	-C10	-C2	-114.96(18)
O7	-N1	-C2	-C10	62.1(2)
C6	-N1	-C2	-C3	49.5(2)
O7	-N1	-C2	-C3	-65.2(2)
C2	-N1	-C6	-C5	-49.1(3)
O7	-N1	-C6	-C5	67.5(2)
C6	-N1	-C2	-C10	176.74(17)
C10	-C2	-C3	-C4	-178.28(18)
N1	-C2	-C10	-O11	175.00(16)
N1	-C2	-C10	-C9	-61.0(2)
C3	-C2	-C10	-O11	-57.5(2)
N1	-C2	-C3	-C4	-54.6(2)
C3	-C2	-C10	-C14	-171.85(18)
N1	-C2	-C10	-C14	60.6(2)
C3	-C2	-C10	-C9	66.6(2)
C2	-C3	-C4	-C5	57.5(2)
C3	-C4	-C5	-C6	-56.5(2)
C4	-C5	-C6	-N1	53.4(3)
O7	-C8	-C9	-C10	-64.8(2)
C9	-C8	-C16	-C15	-28.6(3)
O7	-C8	-C16	-C15	92.9(2)
C16	-C8	-C9	-C10	58.2(2)
C8	-C9	-C10	-C2	60.0(2)
C8	-C9	-C10	-C14	-64.98(19)
C8	-C9	-C10	-O11	-179.77(15)
O11	-C10	-C14	-C15	166.24(17)
C2	-C10	-C14	-C13	110.3(2)
C9	-C10	-C14	-C13	-126.94(19)
C9	-C10	-C14	-C15	45.3(2)
C2	-C10	-C14	-C15	-77.6(2)
O11	-C10	-C14	-C13	-6.0(2)

O11	-C12	-C13	-C14	0.0(3)
O12	-C12	-C13	-C14	177.5(2)
C12	-C13	-C14	-C10	3.8(2)
C12	-C13	-C14	-C15	-167.1(2)
C10	-C14	-C15	-C16	-12.5(3)
C13	-C14	-C15	-C16	157.6(2)
C14	-C15	-C16	-C8	3.8(3)

Table S8 - Contact Distances(Angstrom)

O7	.C3	2.953(3)	N1	.C4	2.943(3)
O7	.C5	2.935(3)	N1	.C9	2.889(3)
O7	.C10	2.801(3)	N1	.C14	2.946(3)
O7	.C15	3.259(3)	N1	.C15	2.983(3)
O7	.C5_a	3.313(4)	N1	.C16	2.735(3)
O7	.C4_a	3.237(4)	N1	.H3B	2.7700
O11	.C13	2.301(3)	N1	.H5B	2.7900
O11	.C3	2.916(3)	N1	.H4B_f	2.9500
O12	.C13_d	3.382(4)	C2	.C5	2.904(4)
O7	.H5B	2.6800	C2	.C8	2.827(4)
O7	.H3B	2.6900	C2	.C12	3.327(4)
O7	.H16	2.9100	C2	.C13	3.383(4)
O7	.H5B_a	2.7300	C2	.C15	3.283(4)
O7	.H6A	2.4400	C2	.C16	3.397(4)
O7	.H4A_a	2.5800	C3	.C9	3.102(4)
O7	.H9A	2.6000	C3	.O7	2.953(3)
O11	.H2	2.63(2)	C3	.O11	2.916(3)
O11	.H3A	2.6000	C3	.C6	2.888(3)
O11	.H9B	2.7100	C4	.O7_g	3.237(4)
O11	.H15_b	2.7700	C4	.N1	2.943(3)
O11	.H16_b	2.8800	C5	.O7_g	3.313(4)
O11	.H6B_c	2.9100	C5	.C2	2.904(4)
O11	.H9A	2.7200	C5	.O7	2.935(3)
O12	.H13	2.8000	C5	.C8_g	3.543(4)
O12	.H15_b	2.6700	C6	.C3	2.888(3)
O12	.H8_e	2.8200	C8	.C2	2.827(4)
O12	.H9B_e	2.8900	C8	.C14	2.818(4)
O12	.H13_d	2.4700	C8	.C5_a	3.543(4)
C9	.C15	2.861(4)	C2	.H4B	2.7100
C9	.C13	3.417(4)	C3	.H5B	2.7000
C9	.C3	3.102(4)	C3	.H9A	2.7800
C9	.C12	3.447(4)	C3	.H6B_c	3.0800
C9	.N1	2.889(3)	C4	.H2	2.702(19)
C10	.O7	2.801(3)	C4	.H6B	2.7300
C10	.C13	2.317(3)	C5	.H3B	2.7000
C10	.C16	2.830(4)	C5	.H8_g	2.9900
C12	.C9	3.447(4)	C6	.H4B	2.7100
C12	.C14	2.274(4)	C6	.H2	2.62(2)
C12	.C2	3.327(4)	C6	.H2_f	3.06(2)
C13	.C10	2.317(3)	C8	.H4A_a	3.0400

C13	.C2	3.383(4)	C9	.H3B	2.7700
C13	.C9	3.417(4)	C10	.H3A	2.7800
C13	.O12_h	3.382(4)	C10	.H3B	2.7500
C14	.N1	2.946(3)	C13	.H15	2.8300
C14	.C8	2.818(4)	C14	.H9B	2.6000
C14	.C12	2.274(4)	C14	.H4B_f	2.9000
C15	.O7	3.259(3)	C14	.H2	2.67(2)
C15	.N1	2.983(3)	C15	.H9B	2.9800
C15	.C2	3.283(4)	C15	.H13	2.8600
C15	.C9	2.861(4)	C15	.H4B_f	2.8800
C16	.C10	2.830(4)	C15	.H9B_i	2.8200
C16	.N1	2.735(3)	C16	.H9B	2.7500
C16	.C2	3.397(4)	H2	.O11	2.63(2)
C2	.H6B	2.7200	H2	.C4	2.702(19)
C2	.H9A	2.7100	H2	.C6	2.62(2)
C2	.H6B_c	3.0600	H2	.C14	2.67(2)
H2	.H3A	2.3800	H4B	.H2	2.5500
H2	.H4B	2.5500	H4B	.H3A	2.3500
H2	.H6B	2.5300	H4B	.H5A	2.3400
H2	.C6_c	3.06(2)	H4B	.H6B	2.5900
H2	.H6B_c	2.4800	H4B	.N1_c	2.9500
H3A	.O11	2.6000	H4B	.C14_c	2.9000
H3A	.C10	2.7800	H4B	.C15_c	2.8800
H3A	.H2	2.3800	H5A	.H4A	2.3700
H3A	.H4A	2.3700	H5A	.H4B	2.3400
H3A	.H4B	2.3500	H5A	.H6A	2.3700
H3A	.H6B_c	2.4800	H5A	.H6B	2.3000
H3B	.O7	2.6900	H5B	.O7	2.6800
H3B	.N1	2.7700	H5B	.N1	2.7900
H3B	.C5	2.7000	H5B	.C3	2.7000
H3B	.C9	2.7700	H5B	.H3B	2.5500
H3B	.C10	2.7500	H5B	.H4A	2.3400
H3B	.H4A	2.3500	H5B	.H6A	2.3200
H3B	.H5B	2.5500	H5B	.O7_g	2.7300
H3B	.H9A	2.2100	H6A	.O7	2.4400
H4A	.H3A	2.3700	H6A	.H5A	2.3700
H4A	.H3B	2.3500	H6A	.H5B	2.3200
H4A	.H5A	2.3700	H6B	.C2	2.7200
H4A	.H5B	2.3400	H6B	.C4	2.7300
H4A	.O7_g	2.5800	H6B	.H2	2.5300
H4A	.C8_g	3.0400	H6B	.H4B	2.5900
H4A	.H8_g	2.5400	H6B	.H5A	2.3000
H4B	.C2	2.7100	H6B	.O11_f	2.9100
H4B	.C6	2.7100	H6B	.C2_f	3.0600
H6B	.C3_f	3.0800	H9B	.C16	2.7500
H6B	.H2_f	2.4800	H9B	.H8	2.3700
H6B	.H3A_f	2.4800	H9B	.O12_i	2.8900
H8	.H9A	2.3900	H9B	.C15_e	2.8200
H8	.H9B	2.3700	H9B	.H15_e	2.4600
H8	.H16	2.3100	H13	.O12	2.8000

H8	.O12_i	2.8200	H13	.C15	2.8600
H8	.C5_a	2.9900	H13	.O12_h	2.4700
H8	.H4A_a	2.5400	H15	.O11_j	2.7700
H9A	.O7	2.6000	H15	.O12_j	2.6700
H9A	.O11	2.7200	H15	.C13	2.8300
H9A	.C2	2.7100	H15	.H16	2.2700
H9A	.C3	2.7800	H15	.H9B_i	2.4600
H9A	.H3B	2.2100	H16	.O7	2.9100
H9A	.H8	2.3900	H16	.O11_j	2.8800
H9B	.O11	2.7100	H16	.H8	2.3100
H9B	.C14	2.6000	H16	.H15	2.2700
H9B	.C15	2.9800			

Table S9 - Hydrogen Bonds (Angstrom, Deg)
for:

C4 -- H4A .. O7	0.9700	2.5800	3.237(4)	125.00	2_556
C13 -- H13 .. O12	0.9300	2.4700	3.382(4)	167.00	2_747

Translation of Symmetry Code to Equiv.Pos

a=[2546.00] =	-x,-1/2+y,1-z
b=[1565.00] =	x,1+y,z
c=[2656.00] =	1-x,1/2+y,1-z
d=[2757.00] =	2-x,1/2+y,2-z
e=[2657.00] =	1-x,1/2+y,2-z
f=[2646.00] =	1-x,-1/2+y,1-z
g=[2556.00] =	-x,1/2+y,1-z
h=[2747.00] =	2-x,-1/2+y,2-z
i=[2647.00] =	1-x,-1/2+y,2-z
j=[1545.00] =	x,-1+y,z