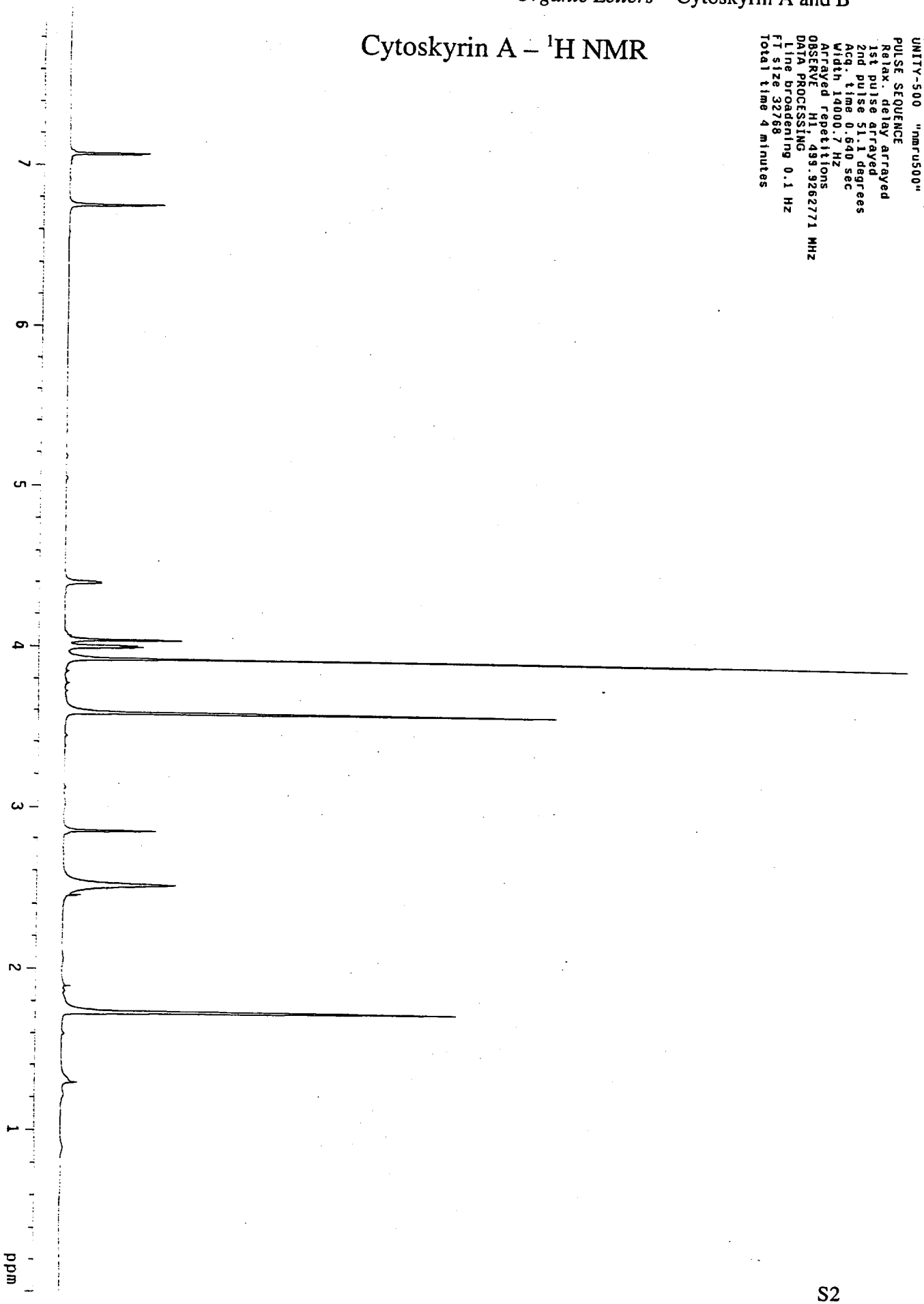
Supplementary Material for

Cytoskyrin A and B, New BIA Active Bisanthraquinones Isolated from an Endophytic Fungus

Sean F. Brady,[†] Maya P. Singh,[‡] Jeff E. Janso[‡] and Jon Clardy[†*]

Department of Chemistry and Chemical Biology, Cornell University, Ithaca, NY 14853
Natural Products Microbiology, Wyeth-Ayerst Research, Pearl River, NY 10956

Cytoskyrin A – ^{1}H NMR



Cytoskyrin A – ^{13}C NMR

Cytoskyrin B – ^{1}H NMR

Cytoskyrin B – ^{13}C NMR

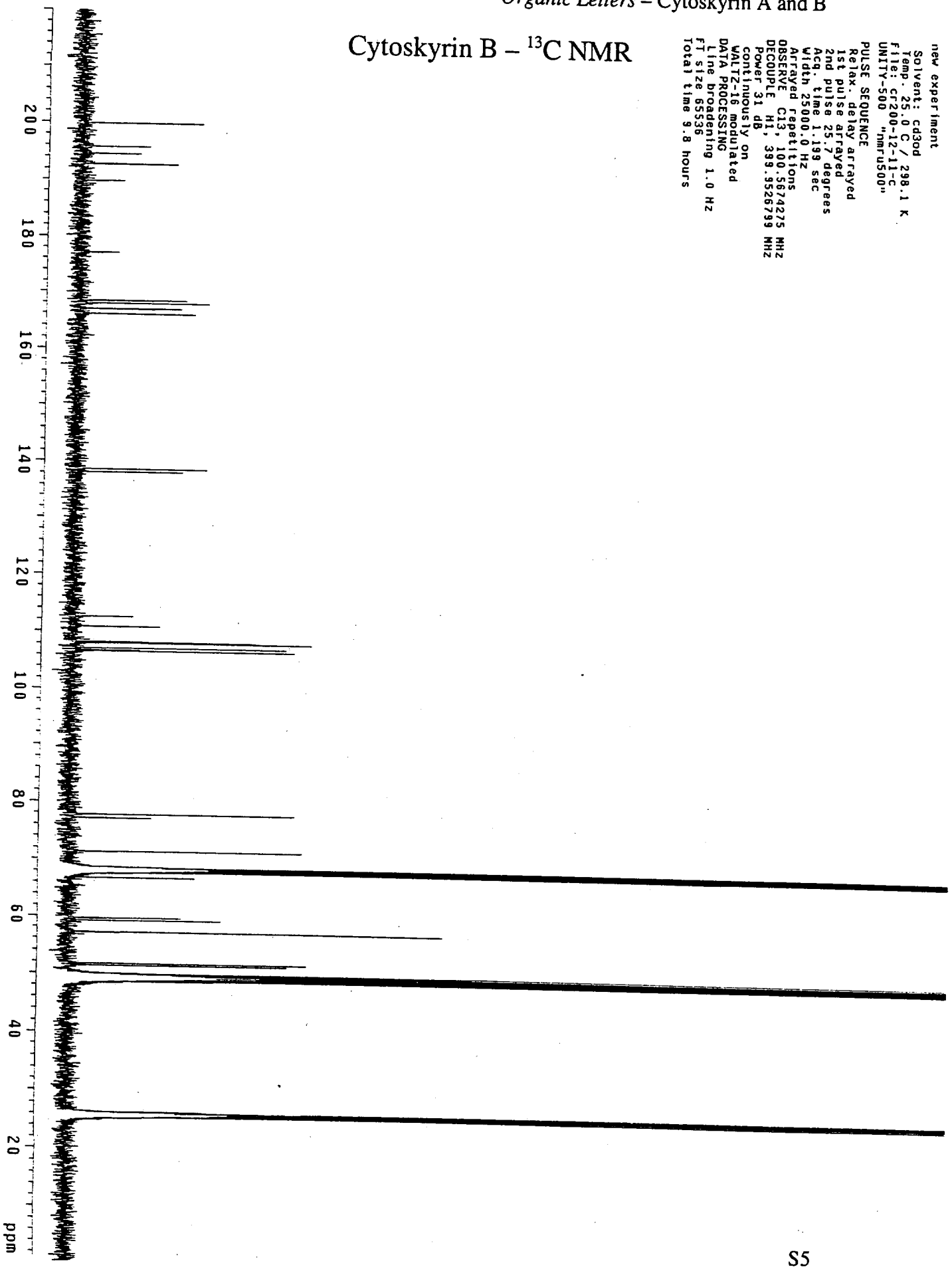


Table 1. Crystal data and structure refinement for Cytoskyrin A.

Identification code	Cytoskyrin A	
Empirical formula	C36 H36 N O12	
Formula weight	674.66	
Temperature	173(2) K	
Wavelength	1.54180 Å	
Crystal system	Triclinic	
Space group	P1	
Unit cell dimensions	a = 7.3742(2) Å	α= 72.976(2)°.
	b = 10.2464(4) Å	β= 78.717(2)°.
	c = 11.3595(4) Å	γ = 74.046(3)°.
Volume	782.87(5) $Å^3$	
Z	1	
Density (calculated)	1.431 Mg/m^3	
Absorption coefficient	0.904 mm^{-1}	
F(000)	355	
Crystal size	0.30 x 0.15 x 0.05 mm^3	
Theta range for data collection	4.64 to 62.27°.	
Index ranges	-7<=h<=8, -11<=k<=10, -12<=l<=8	
Reflections collected	3656	
Independent reflections	2381 [R(int) = 0.0407]	
Completeness to theta = 62.27°	85.4 %	
Absorption correction	SADABS	
Max. and min. transmission	1 and 0.670126	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2381 / 3 / 462	
Goodness-of-fit on F^2	0.984	
Final R indices [I>2sigma(I)]	R1 = 0.0471, wR2 = 0.1102	
R indices (all data)	R1 = 0.0565, wR2 = 0.1141	
Absolute structure parameter	0.7(3)	
Largest diff. peak and hole	0.224 and -0.333 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for Cytoskyrin A. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	5178(6)	7832(4)	10283(3)	24(1)
O(2)	6218(6)	6774(4)	6520(3)	25(1)
O(3)	7905(6)	8663(4)	4716(3)	30(1)
O(4)	9364(6)	10447(4)	3129(4)	40(1)
O(5)	13803(6)	12324(4)	4115(4)	41(1)
O(6)	11121(6)	9500(4)	8255(4)	36(1)
C(1)	8259(8)	7922(6)	9120(5)	24(1)
C(2)	6200(8)	7956(6)	9056(5)	22(1)
C(3)	6466(8)	6660(5)	8598(5)	20(1)
C(4A)	8423(8)	7937(5)	6823(5)	20(1)
C(4)	7058(8)	7089(5)	7207(5)	19(1)
C(5)	10282(9)	10510(6)	4027(5)	29(2)
C(6)	11536(10)	11388(6)	3696(6)	33(2)
C(7)	12515(9)	11468(6)	4574(6)	30(2)
C(8)	12215(8)	10747(5)	5806(5)	26(1)
C(8A)	10928(8)	9889(6)	6139(5)	23(1)
C(9)	10531(8)	9186(6)	7451(5)	22(1)
C(9A)	9514(8)	7988(5)	7819(5)	21(1)
C(10A)	9988(9)	9708(6)	5257(5)	22(1)
C(10)	8712(8)	8712(5)	5603(5)	21(1)
C(11)	15126(10)	12241(7)	4916(7)	45(2)
O(1')	11890(6)	7052(4)	10025(4)	32(1)
O(2')	12081(6)	4995(4)	6803(4)	29(1)
O(3')	9992(6)	3263(4)	7147(4)	31(1)
O(4')	7424(7)	1935(5)	7214(4)	37(1)
O(5')	2488(6)	1915(4)	10631(4)	32(1)
O(6')	6932(6)	4435(4)	11431(4)	36(1)
C(1')	9017(8)	6425(5)	9867(5)	22(1)
C(2')	11140(9)	6188(7)	9523(5)	23(1)
C(3')	11263(8)	6613(6)	8105(5)	22(1)
C(4')	10914(9)	5417(6)	7750(5)	23(1)
C(5')	6598(9)	2342(6)	8271(6)	28(1)
C(6')	4964(9)	1891(6)	8880(6)	29(2)
C(7')	4114(8)	2261(6)	9976(5)	25(1)
C(8')	4898(9)	3034(6)	10486(5)	26(1)
C(9')	7176(9)	4484(6)	10331(6)	25(1)
C(4A')	9476(8)	4798(6)	8417(5)	21(1)
C(8A')	6456(8)	3532(6)	9846(5)	22(1)
C(9A')	8095(8)	5552(5)	9326(5)	19(1)
C(10')	9014(8)	3732(6)	8050(5)	23(1)
C(10B)	7353(9)	3192(6)	8709(6)	25(1)
C(11')	1672(10)	1016(6)	10216(6)	36(2)
N(1)	16102(7)	7066(4)	4020(4)	21(1)
C(1S)	16920(9)	5498(6)	4271(6)	33(2)
C(6S)	12969(10)	6990(8)	3472(7)	46(2)
C(5S)	13992(8)	7403(7)	4308(6)	31(2)
C(4S)	15868(10)	9354(6)	2333(6)	34(2)
C(3S)	16784(9)	7800(6)	2707(5)	27(1)
C(2S)	19047(11)	5111(7)	4225(7)	44(2)

Table 3. Bond lengths [Å] and angles [°] for Cytoskyrin A.

O(1)-C(2)	1.437(7)
O(2)-C(4)	1.242(6)
O(3)-C(10)	1.285(6)
O(4)-C(5)	1.355(7)
O(5)-C(7)	1.392(7)
O(5)-C(11)	1.429(8)
O(6)-C(9)	1.237(6)
C(1)-C(2)	1.524(8)
C(1)-C(1')	1.530(8)
C(1)-C(9A)	1.577(8)
C(2)-C(3)	1.515(7)
C(3)-C(4)	1.520(8)
C(3)-C(9A')	1.583(7)
C(4A)-C(10)	1.389(7)
C(4A)-C(4)	1.431(7)
C(4A)-C(9A)	1.530(7)
C(5)-C(6)	1.389(8)
C(5)-C(10A)	1.408(8)
C(6)-C(7)	1.373(9)
C(7)-C(8)	1.384(8)
C(8)-C(8A)	1.393(7)
C(8A)-C(10A)	1.403(7)
C(8A)-C(9)	1.464(8)
C(9)-C(9A)	1.525(7)
C(9A)-C(3')	1.628(8)
C(10A)-C(10)	1.493(7)
O(1')-C(2')	1.450(7)
O(2')-C(4')	1.338(7)
O(3')-C(10')	1.270(7)
O(4')-C(5')	1.361(7)
O(5')-C(7')	1.359(7)
O(5')-C(11')	1.450(6)
O(6')-C(9')	1.215(7)
C(1')-C(2')	1.506(8)
C(1')-C(9A')	1.569(7)
C(2')-C(3')	1.532(8)
C(3')-C(4')	1.496(8)
C(4')-C(4A')	1.369(8)
C(5')-C(6')	1.391(8)
C(5')-C(10B)	1.395(8)
C(6')-C(7')	1.388(9)
C(7')-C(8')	1.396(8)
C(8')-C(8A')	1.374(8)
C(9')-C(8A')	1.501(7)
C(9')-C(9A')	1.527(7)
C(4A')-C(10')	1.417(7)
C(4A')-C(9A')	1.524(8)
C(8A')-C(10B)	1.424(9)
C(10')-C(10B)	1.463(8)
N(1)-C(5S)	1.490(7)
N(1)-C(1S)	1.514(7)
N(1)-C(3S)	1.517(7)
C(1S)-C(2S)	1.503(10)
C(6S)-C(5S)	1.530(8)
C(4S)-C(3S)	1.515(8)
C(7)-O(5)-C(11)	117.1(5)
C(2)-C(1)-C(1')	103.3(5)
C(2)-C(1)-C(9A)	112.1(4)

C(1')-C(1)-C(9A)	103.1(5)
O(1)-C(2)-C(3)	112.4(4)
O(1)-C(2)-C(1)	108.6(4)
C(3)-C(2)-C(1)	100.6(5)
C(2)-C(3)-C(4)	105.9(4)
C(2)-C(3)-C(9A')	103.8(4)
C(4)-C(3)-C(9A')	113.0(5)
C(10)-C(4A)-C(4)	121.4(4)
C(10)-C(4A)-C(9A)	121.3(5)
C(4)-C(4A)-C(9A)	117.3(5)
O(2)-C(4)-C(4A)	126.6(5)
O(2)-C(4)-C(3)	117.8(5)
C(4A)-C(4)-C(3)	115.3(4)
O(4)-C(5)-C(6)	117.9(5)
O(4)-C(5)-C(10A)	121.3(5)
C(6)-C(5)-C(10A)	120.8(5)
C(7)-C(6)-C(5)	119.8(6)
C(6)-C(7)-C(8)	121.6(5)
C(6)-C(7)-O(5)	114.3(5)
C(8)-C(7)-O(5)	124.1(5)
C(7)-C(8)-C(8A)	118.4(5)
C(8)-C(8A)-C(10A)	121.8(5)
C(8)-C(8A)-C(9)	118.5(5)
C(10A)-C(8A)-C(9)	119.7(5)
O(6)-C(9)-C(8A)	120.2(5)
O(6)-C(9)-C(9A)	119.9(5)
C(8A)-C(9)-C(9A)	119.7(4)
C(9)-C(9A)-C(4A)	114.7(4)
C(9)-C(9A)-C(1)	111.6(4)
C(4A)-C(9A)-C(1)	112.5(5)
C(9)-C(9A)-C(3')	102.1(4)
C(4A)-C(9A)-C(3')	111.5(4)
C(1)-C(9A)-C(3')	103.2(4)
C(8A)-C(10A)-C(5)	117.5(5)
C(8A)-C(10A)-C(10)	121.6(5)
C(5)-C(10A)-C(10)	120.9(5)
O(3)-C(10)-C(4A)	124.1(4)
O(3)-C(10)-C(10A)	115.7(5)
C(4A)-C(10)-C(10A)	120.2(4)
C(7')-O(5')-C(11')	118.2(5)
C(2')-C(1')-C(1)	103.8(5)
C(2')-C(1')-C(9A')	113.4(5)
C(1)-C(1')-C(9A')	101.9(4)
O(1')-C(2')-C(1')	110.4(5)
O(1')-C(2')-C(3')	114.0(4)
C(1')-C(2')-C(3')	100.3(4)
C(4')-C(3')-C(2')	106.0(4)
C(4')-C(3')-C(9A)	110.7(4)
C(2')-C(3')-C(9A)	103.8(5)
O(2')-C(4')-C(4A')	124.0(5)
O(2')-C(4')-C(3')	116.9(5)
C(4A')-C(4')-C(3')	119.1(5)
O(4')-C(5')-C(6')	117.3(5)
O(4')-C(5')-C(10B)	121.0(6)
C(6')-C(5')-C(10B)	121.7(6)
C(5')-C(6')-C(7')	118.6(5)
O(5')-C(7')-C(6')	123.8(5)
O(5')-C(7')-C(8')	114.9(5)
C(6')-C(7')-C(8')	121.3(6)
C(8A')-C(8')-C(7')	119.5(6)
O(6')-C(9')-C(8A')	121.3(5)

O(6')-C(9')-C(9A')	124.2(5)
C(8A')-C(9')-C(9A')	114.3(5)
C(4')-C(4A')-C(10')	120.5(5)
C(4')-C(4A')-C(9A')	116.8(5)
C(10')-C(4A')-C(9A')	120.7(5)
C(8')-C(8A')-C(10B)	120.8(5)
C(8')-C(8A')-C(9')	119.4(5)
C(10B)-C(8A')-C(9')	119.8(5)
C(9')-C(9A')-C(4A')	108.8(4)
C(9')-C(9A')-C(1')	113.0(4)
C(4A')-C(9A')-C(1')	113.4(5)
C(9')-C(9A')-C(3)	107.8(5)
C(4A')-C(9A')-C(3)	108.2(4)
C(1')-C(9A')-C(3)	105.4(4)
O(3')-C(10')-C(4A')	121.2(5)
O(3')-C(10')-C(10B)	119.7(5)
C(4A')-C(10')-C(10B)	119.1(5)
C(5')-C(10B)-C(8A')	117.9(5)
C(5')-C(10B)-C(10')	122.5(5)
C(8A')-C(10B)-C(10')	119.6(5)
C(5S)-N(1)-C(1S)	111.6(4)
C(5S)-N(1)-C(3S)	112.8(4)
C(1S)-N(1)-C(3S)	110.7(4)
C(2S)-C(1S)-N(1)	112.8(5)
N(1)-C(5S)-C(6S)	114.1(5)
C(4S)-C(3S)-N(1)	114.1(5)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for Cytoskyrin A. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	20(3)	39(2)	20(2)	-15(2)	6(2)	-16(2)
O(2)	36(3)	36(2)	11(2)	-4(2)	2(2)	-24(2)
O(3)	40(3)	40(2)	16(2)	-3(2)	0(2)	-27(2)
O(4)	54(3)	50(3)	23(3)	-1(2)	-2(2)	-35(2)
O(5)	43(3)	44(3)	45(3)	-12(2)	11(2)	-35(2)
O(6)	44(3)	43(3)	30(3)	-11(2)	-2(2)	-27(2)
C(1)	22(4)	34(3)	21(3)	-11(2)	-2(3)	-11(3)
C(2)	22(4)	32(3)	17(3)	-11(2)	1(3)	-14(3)
C(3)	22(3)	23(3)	20(3)	-8(2)	-1(3)	-11(2)
C(4A)	24(4)	26(3)	15(3)	-8(2)	1(3)	-11(3)
C(4)	18(3)	24(3)	16(3)	-2(2)	0(3)	-9(2)
C(5)	39(4)	28(3)	23(4)	-7(3)	3(3)	-17(3)
C(6)	38(4)	33(4)	31(4)	-10(3)	9(3)	-22(3)
C(7)	28(4)	24(3)	43(4)	-15(3)	10(3)	-18(3)
C(8)	27(4)	25(3)	30(4)	-14(3)	3(3)	-12(3)
C(8A)	19(4)	27(3)	26(4)	-9(2)	5(3)	-13(3)
C(9)	17(3)	27(3)	27(3)	-10(2)	-5(3)	-8(3)
C(9A)	22(4)	24(3)	21(3)	-9(2)	-3(3)	-7(3)
C(10A)	26(4)	22(3)	21(3)	-9(2)	6(3)	-13(2)
C(10)	23(4)	20(3)	21(3)	-5(2)	-2(3)	-10(2)
C(11)	33(5)	43(4)	67(5)	-19(3)	4(4)	-23(3)
O(1')	26(3)	58(3)	24(2)	-15(2)	2(2)	-25(2)
O(2')	28(3)	35(3)	26(3)	-13(2)	12(2)	-14(2)
O(3')	37(3)	26(2)	30(3)	-12(2)	8(2)	-11(2)
O(4')	43(3)	43(3)	34(3)	-23(2)	10(2)	-20(2)
O(5')	35(3)	38(2)	29(3)	-10(2)	2(2)	-21(2)
O(6')	54(3)	42(2)	18(3)	-7(2)	5(2)	-29(2)
C(1')	22(4)	30(3)	19(3)	-5(2)	-7(3)	-10(3)
C(2')	18(4)	30(4)	21(3)	-3(3)	-5(3)	-8(3)
C(3')	12(3)	31(3)	27(3)	-8(2)	2(3)	-12(3)
C(4')	27(4)	21(3)	21(3)	-6(2)	1(3)	-6(3)
C(5')	30(4)	29(3)	26(4)	-11(3)	7(3)	-11(3)
C(6')	37(4)	25(3)	29(4)	-8(3)	-1(3)	-15(3)
C(7')	23(4)	26(3)	26(4)	-4(3)	0(3)	-9(3)
C(8')	33(4)	24(3)	20(3)	-3(2)	-1(3)	-13(3)
C(9')	33(4)	29(3)	14(3)	4(2)	0(3)	-18(3)
C(4A')	21(3)	26(3)	15(3)	-6(2)	1(3)	-6(3)
C(8A')	23(4)	23(3)	21(3)	-5(2)	0(3)	-10(3)
C(9A')	20(3)	21(3)	15(3)	-3(2)	1(3)	-9(2)
C(10')	24(4)	22(3)	19(3)	0(2)	2(3)	-9(3)
C(10B)	28(4)	27(3)	22(3)	-5(2)	-4(3)	-10(3)
C(11')	44(5)	37(4)	34(4)	-5(3)	-7(3)	-25(3)
N(1)	20(3)	27(3)	16(3)	-1(2)	0(2)	-12(2)
C(1S)	45(5)	22(3)	35(4)	-6(3)	-8(3)	-11(3)
C(6S)	34(5)	74(5)	39(4)	-18(4)	-1(4)	-24(4)
C(5S)	22(4)	46(4)	26(4)	-12(3)	-2(3)	-9(3)
C(4S)	41(5)	33(3)	25(4)	5(3)	-9(3)	-13(3)
C(3S)	33(4)	38(3)	9(3)	-3(2)	-4(3)	-12(3)
C(2S)	45(5)	33(4)	48(5)	-7(3)	-6(4)	-2(3)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for Cytoskyrin A.

	x	y	z	U(eq)
H(2A)	6453	7228	5784	38
H(4B)	8685	9861	3429	59
H(1)	8368	8646	9516	29
H(3)	5268	6319	8789	24
H(6)	11715	11932	2865	39
H(8)	12870	10835	6410	31
H(11A)	14448	12643	5608	68
H(11B)	16079	12767	4449	68
H(11C)	15756	11257	5239	68
H(1')	8634	6305	10780	27
H(3')	12517	6826	7702	27
H(6')	4442	1341	8552	35
H(8')	4358	3214	11270	31
H(11D)	1388	1461	9362	54
H(11E)	497	871	10759	54
H(11F)	2578	109	10247	54
H(1SA)	16353	5036	5101	40
H(1SB)	16564	5140	3649	40
H(6SA)	11594	7230	3722	70
H(6SB)	13397	5977	3550	70
H(6SC)	13260	7497	2607	70
H(5SA)	13536	8424	4229	37
H(5SB)	13645	6915	5181	37
H(4SA)	16375	9754	1478	51
H(4SB)	16147	9825	2890	51
H(4SC)	14489	9486	2388	51
H(3SA)	18178	7679	2626	32
H(3SB)	16524	7340	2124	32
H(2SA)	19501	4089	4392	66
H(2SB)	19408	5444	4852	66
H(2SC)	19619	5549	3400	66
H(1O)	3920(120)	7690(80)	10140(70)	80(30)
H(2')	11770(100)	5430(70)	9750(60)	30(19)
H(2)	5510(80)	8880(60)	8390(50)	33(16)
H(2B)	11640(90)	4180(60)	6700(60)	38(18)
H(4A)	8360(150)	2320(100)	7070(90)	100(40)