A New Unusual Natural Pigment from *Selaginella sinensis* and Its Noticeable Physicochemical Properties

*Li-Ping Zhang, Yong-Min Liang, Xiu-Cheng Wei and Dong-Liang Cheng**

State Key Laboratory of Applied Organic Chemistry (SKLAOC), Lanzhou University,
Lanzhou 730000, P. R. China

*E-mail: chengdl@lzu.edu.cn

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Isolation

The air-dried whole plant of *Selaginella sinensis* (5.5 kg), collected in Guangxi Autonomous Region of China in August 1999, was powdered and extracted with acetone three times at room temperature, each extraction lasting five days. The acetone solution was filtered and evaporated under reduced pressure to give an acetone extract (28 g) which was subjected to column chromatography on silica gel eluting with a gradient of petroleum/acetone (from 50:1 to 1:1) to give eight fractions (monitored by TLC analysis) from which the seventh fraction, petroleum/acetone (2:1), yielded pure Selaginellin (**1**) (108 mg).

General experimental procedures: Infrared spectra were recorded on a AVATAR 360 FT-IR spectrophotometer. The UV spectra were recorded on a UV-260 spectrophotometer, and the circular dichroism on DSM-1000. ¹H, ¹³C NMR (DEPT) spectral data and H-H COSY, NOESY, HMQC, HMBC experiments were measured on spectrometers (300 and 400-MHz). The fluorescence spectra were recorded on a LS55 Luminescence spectrometer.

The structure of compound 1a

The packing diagram of compound **1a**

The packing diagram of compound **1a**, *(R)*-**1a** and *(S)*-**1a** are indicated by solid and dash arrows, respectively.

Table S1. Crystal data and structure refinement for y2038.

Identification code	y2038
Empirical formula	C38 H32 O5
Formula weight	568.64
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	a = 9.882(2) Å alpha = 103.72(1) deg. b = 11.409(2) Å beta = 95.62(2) deg. c = 14.987(3) Å gamma = 108.65(1) deg.
Volume, Z	1527.46(59) Å ³ , 2
Density (calculated)	1.236 Mg/m ³
Absorption coefficient	0.081 mm ⁻¹
F(000)	600
Crystal size	0.56 x 0.48 x 0.46 mm
Theta range for data collection	1.42 to 25.50 deg.
Limiting indices	0 ≤ h ≤ 11, -13 ≤ k ≤ 12, -18 ≤ l ≤ 18
Reflections collected	6180
Independent reflections	5629 [R(int) = 0.0111]
Absorption correction	None
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	5629 / 0 / 393

Goodness-of-fit on F^2	0.921
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0369$, $wR_2 = 0.0885$
R indices (all data)	$R_1 = 0.0649$, $wR_2 = 0.0951$
Extinction coefficient	0.0216(15)
Largest diff. peak and hole	0.138 and -0.142 e. $\text{\AA}^{-3}$

Table S2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for y2038.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	8253(1)	10173(1)	7276(1)	72(1)
O(2)	3885(1)	2044(1)	1465(1)	70(1)
O(3)	7172(2)	10960(1)	1504(1)	83(1)
O(4)	513(1)	6754(1)	5263(1)	74(1)
O(5)	-81(2)	6229(1)	-1725(1)	86(1)
C(1)	7843(2)	8436(2)	4813(1)	50(1)
C(2)	8278(2)	9458(2)	5618(1)	53(1)
C(3)	7890(2)	9242(2)	6439(1)	51(1)
C(4)	7074(2)	8009(2)	6446(1)	57(1)
C(5)	6659(2)	6998(2)	5641(1)	52(1)
C(6)	7020(2)	7191(1)	4799(1)	45(1)
C(7)	6537(2)	6107(1)	3920(1)	44(1)
C(8)	6976(2)	5052(2)	3839(1)	57(1)
C(9)	6542(2)	4058(2)	3021(1)	58(1)
C(10)	5664(2)	4068(1)	2251(1)	48(1)
C(11)	5214(2)	5127(1)	2312(1)	42(1)
C(12)	5640(2)	6138(1)	3150(1)	41(1)
C(13)	4299(2)	5199(1)	1534(1)	47(1)
C(14)	3551(2)	5335(1)	920(1)	50(1)
C(15)	2635(2)	5576(2)	239(1)	49(1)
C(16)	2313(2)	6688(2)	448(1)	66(1)
C(17)	1414(2)	6941(2)	-185(1)	69(1)
C(18)	827(2)	6077(2)	-1049(1)	60(1)
C(19)	1166(2)	4983(2)	-1279(1)	71(1)
C(20)	2056(2)	4724(2)	-648(1)	65(1)
C(21)	5069(2)	7216(1)	3228(1)	39(1)
C(22)	5585(2)	8163(1)	2805(1)	40(1)
C(23)	4949(2)	9138(1)	2786(1)	46(1)
C(24)	5450(2)	10046(2)	2356(1)	53(1)
C(25)	6686(2)	10117(2)	1894(1)	56(1)
C(26)	7322(2)	9144(2)	1912(1)	55(1)
C(27)	6798(2)	8225(1)	2330(1)	48(1)
C(28)	3885(2)	7148(1)	3776(1)	40(1)
C(29)	4020(2)	8103(1)	4576(1)	50(1)
C(30)	2926(2)	8008(2)	5097(1)	53(1)

C(31)	1663(2)	6949(2)	4809(1)	50(1)
C(32)	1500(2)	5991(2)	4002(1)	59(1)
C(33)	2605(2)	6077(2)	3501(1)	52(1)
C(34)	9083(2)	11446(2)	7310(2)	83(1)
C(35)	5174(2)	2962(2)	1379(1)	59(1)
C(36)	3295(3)	981(2)	653(2)	109(1)
C(37)	649(2)	7661(2)	6120(1)	75(1)
C(38)	-603(2)	7256(2)	-1485(2)	96(1)

Table S3. Bond lengths [Å] and angles [deg] for y2038.

O(1)-C(3)	1.3657(19)
O(1)-C(34)	1.406(2)
O(2)-C(35)	1.4074(19)
O(2)-C(36)	1.415(2)
O(3)-C(25)	1.2342(18)
O(4)-C(31)	1.3656(18)
O(4)-C(37)	1.409(2)
O(5)-C(18)	1.366(2)
O(5)-C(38)	1.412(2)
C(1)-C(2)	1.382(2)
C(1)-C(6)	1.388(2)
C(1)-H(1)	0.9300
C(2)-C(3)	1.377(2)
C(2)-H(2)	0.9300
C(3)-C(4)	1.383(2)
C(4)-C(5)	1.377(2)
C(4)-H(4)	0.9300
C(5)-C(6)	1.392(2)
C(5)-H(5)	0.9300
C(6)-C(7)	1.490(2)
C(7)-C(8)	1.389(2)
C(7)-C(12)	1.400(2)
C(8)-C(9)	1.378(2)
C(8)-H(8)	0.9300
C(9)-C(10)	1.378(2)
C(9)-H(9)	0.9300
C(10)-C(11)	1.4018(19)
C(10)-C(35)	1.496(2)
C(11)-C(12)	1.408(2)
C(11)-C(13)	1.439(2)
C(12)-C(21)	1.4947(19)
C(13)-C(14)	1.195(2)
C(14)-C(15)	1.431(2)
C(15)-C(16)	1.377(2)
C(15)-C(20)	1.386(2)
C(16)-C(17)	1.380(2)
C(16)-H(16)	0.9300
C(17)-C(18)	1.367(2)
C(17)-H(17)	0.9300
C(18)-C(19)	1.369(2)
C(19)-C(20)	1.376(2)

C(19)-H(19)	0.9300
C(20)-H(20)	0.9300
C(21)-C(22)	1.3659(19)
C(21)-C(28)	1.485(2)
C(22)-C(27)	1.445(2)
C(22)-C(23)	1.4461(19)
C(23)-C(24)	1.335(2)
C(23)-H(23)	0.9300
C(24)-C(25)	1.452(2)
C(24)-H(24)	0.9300
C(25)-C(26)	1.443(2)
C(26)-C(27)	1.337(2)
C(26)-H(26)	0.9300
C(27)-H(27)	0.9300
C(28)-C(29)	1.378(2)
C(28)-C(33)	1.391(2)
C(29)-C(30)	1.386(2)
C(29)-H(29)	0.9300
C(30)-C(31)	1.374(2)
C(30)-H(30)	0.9300
C(31)-C(32)	1.380(2)
C(32)-C(33)	1.375(2)
C(32)-H(32)	0.9300
C(33)-H(33)	0.9300
C(34)-H(34A)	0.9600
C(34)-H(34B)	0.9600
C(34)-H(34C)	0.9600
C(35)-H(35A)	0.9700
C(35)-H(35B)	0.9700
C(36)-H(36A)	0.9600
C(36)-H(36B)	0.9600
C(36)-H(36C)	0.9600
C(37)-H(37A)	0.9600
C(37)-H(37B)	0.9600
C(37)-H(37C)	0.9600
C(38)-H(38A)	0.9600
C(38)-H(38B)	0.9600
C(38)-H(38C)	0.9600
C(3)-O(1)-C(34)	118.24(15)
C(35)-O(2)-C(36)	112.31(15)
C(31)-O(4)-C(37)	118.25(14)
C(18)-O(5)-C(38)	118.34(16)
C(2)-C(1)-C(6)	122.19(15)

C(2)-C(1)-H(1)	118.9
C(6)-C(1)-H(1)	118.9
C(3)-C(2)-C(1)	119.39(15)
C(3)-C(2)-H(2)	120.3
C(1)-C(2)-H(2)	120.3
O(1)-C(3)-C(2)	124.60(16)
O(1)-C(3)-C(4)	115.80(15)
C(2)-C(3)-C(4)	119.60(15)
C(5)-C(4)-C(3)	120.46(15)
C(5)-C(4)-H(4)	119.8
C(3)-C(4)-H(4)	119.8
C(4)-C(5)-C(6)	121.14(15)
C(4)-C(5)-H(5)	119.4
C(6)-C(5)-H(5)	119.4
C(1)-C(6)-C(5)	117.21(15)
C(1)-C(6)-C(7)	121.40(14)
C(5)-C(6)-C(7)	121.39(14)
C(8)-C(7)-C(12)	118.37(14)
C(8)-C(7)-C(6)	121.05(14)
C(12)-C(7)-C(6)	120.57(13)
C(9)-C(8)-C(7)	120.91(15)
C(9)-C(8)-H(8)	119.5
C(7)-C(8)-H(8)	119.5
C(10)-C(9)-C(8)	121.86(14)
C(10)-C(9)-H(9)	119.1
C(8)-C(9)-H(9)	119.1
C(9)-C(10)-C(11)	118.37(14)
C(9)-C(10)-C(35)	120.91(14)
C(11)-C(10)-C(35)	120.71(14)
C(10)-C(11)-C(12)	120.08(14)
C(10)-C(11)-C(13)	121.23(14)
C(12)-C(11)-C(13)	118.69(13)
C(7)-C(12)-C(11)	120.41(13)
C(7)-C(12)-C(21)	119.96(13)
C(11)-C(12)-C(21)	119.56(13)
C(14)-C(13)-C(11)	175.94(16)
C(13)-C(14)-C(15)	175.40(17)
C(16)-C(15)-C(20)	117.65(16)
C(16)-C(15)-C(14)	120.32(14)
C(20)-C(15)-C(14)	122.03(16)
C(15)-C(16)-C(17)	121.89(16)
C(15)-C(16)-H(16)	119.1
C(17)-C(16)-H(16)	119.1
C(18)-C(17)-C(16)	119.63(18)

C(18)-C(17)-H(17)	120.2
C(16)-C(17)-H(17)	120.2
O(5)-C(18)-C(17)	124.41(18)
O(5)-C(18)-C(19)	116.25(17)
C(17)-C(18)-C(19)	119.34(17)
C(18)-C(19)-C(20)	121.14(17)
C(18)-C(19)-H(19)	119.4
C(20)-C(19)-H(19)	119.4
C(19)-C(20)-C(15)	120.31(17)
C(19)-C(20)-H(20)	119.8
C(15)-C(20)-H(20)	119.8
C(22)-C(21)-C(28)	123.49(12)
C(22)-C(21)-C(12)	121.78(13)
C(28)-C(21)-C(12)	114.72(12)
C(21)-C(22)-C(27)	121.03(13)
C(21)-C(22)-C(23)	122.91(13)
C(27)-C(22)-C(23)	116.05(13)
C(24)-C(23)-C(22)	122.25(15)
C(24)-C(23)-H(23)	118.9
C(22)-C(23)-H(23)	118.9
C(23)-C(24)-C(25)	121.43(15)
C(23)-C(24)-H(24)	119.3
C(25)-C(24)-H(24)	119.3
O(3)-C(25)-C(26)	121.47(17)
O(3)-C(25)-C(24)	122.11(16)
C(26)-C(25)-C(24)	116.42(14)
C(27)-C(26)-C(25)	121.77(16)
C(27)-C(26)-H(26)	119.1
C(25)-C(26)-H(26)	119.1
C(26)-C(27)-C(22)	122.05(14)
C(26)-C(27)-H(27)	119.0
C(22)-C(27)-H(27)	119.0
C(29)-C(28)-C(33)	117.73(14)
C(29)-C(28)-C(21)	122.19(13)
C(33)-C(28)-C(21)	120.05(13)
C(28)-C(29)-C(30)	121.64(15)
C(28)-C(29)-H(29)	119.2
C(30)-C(29)-H(29)	119.2
C(31)-C(30)-C(29)	119.73(15)
C(31)-C(30)-H(30)	120.1
C(29)-C(30)-H(30)	120.1
O(4)-C(31)-C(30)	124.88(14)
O(4)-C(31)-C(32)	115.67(15)
C(30)-C(31)-C(32)	119.45(15)

C(33)-C(32)-C(31)	120.46(15)
C(33)-C(32)-H(32)	119.8
C(31)-C(32)-H(32)	119.8
C(32)-C(33)-C(28)	120.95(14)
C(32)-C(33)-H(33)	119.5
C(28)-C(33)-H(33)	119.5
O(1)-C(34)-H(34A)	109.5
O(1)-C(34)-H(34B)	109.5
H(34A)-C(34)-H(34B)	109.5
O(1)-C(34)-H(34C)	109.5
H(34A)-C(34)-H(34C)	109.5
H(34B)-C(34)-H(34C)	109.5
O(2)-C(35)-C(10)	107.97(13)
O(2)-C(35)-H(35A)	110.1
C(10)-C(35)-H(35A)	110.1
O(2)-C(35)-H(35B)	110.1
C(10)-C(35)-H(35B)	110.1
H(35A)-C(35)-H(35B)	108.4
O(2)-C(36)-H(36A)	109.5
O(2)-C(36)-H(36B)	109.5
H(36A)-C(36)-H(36B)	109.5
O(2)-C(36)-H(36C)	109.5
H(36A)-C(36)-H(36C)	109.5
H(36B)-C(36)-H(36C)	109.5
O(4)-C(37)-H(37A)	109.5
O(4)-C(37)-H(37B)	109.5
H(37A)-C(37)-H(37B)	109.5
O(4)-C(37)-H(37C)	109.5
H(37A)-C(37)-H(37C)	109.5
H(37B)-C(37)-H(37C)	109.5
O(5)-C(38)-H(38A)	109.5
O(5)-C(38)-H(38B)	109.5
H(38A)-C(38)-H(38B)	109.5
O(5)-C(38)-H(38C)	109.5
H(38A)-C(38)-H(38C)	109.5
H(38B)-C(38)-H(38C)	109.5

Table S4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for y2038.

The anisotropic displacement factor exponent takes the form:

$$-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$$

	U11	U22	U33	U23	U13	U12
O(1)	79(1)	66(1)	65(1)	4(1)	11(1)	28(1)
O(2)	82(1)	58(1)	55(1)	5(1)	13(1)	13(1)
O(3)	97(1)	71(1)	103(1)	57(1)	34(1)	31(1)
O(4)	58(1)	88(1)	70(1)	11(1)	28(1)	22(1)
O(5)	75(1)	95(1)	78(1)	29(1)	-21(1)	23(1)
C(1)	47(1)	57(1)	48(1)	22(1)	6(1)	17(1)
C(2)	48(1)	49(1)	61(1)	18(1)	3(1)	14(1)
C(3)	46(1)	57(1)	52(1)	10(1)	4(1)	26(1)
C(4)	56(1)	69(1)	52(1)	24(1)	16(1)	26(1)
C(5)	50(1)	52(1)	58(1)	23(1)	12(1)	18(1)
C(6)	39(1)	50(1)	50(1)	19(1)	5(1)	19(1)
C(7)	42(1)	44(1)	51(1)	21(1)	10(1)	17(1)
C(8)	59(1)	57(1)	65(1)	28(1)	6(1)	30(1)
C(9)	67(1)	50(1)	73(1)	26(1)	19(1)	36(1)
C(10)	56(1)	44(1)	54(1)	20(1)	22(1)	24(1)
C(11)	44(1)	41(1)	47(1)	18(1)	14(1)	17(1)
C(12)	41(1)	39(1)	49(1)	18(1)	13(1)	17(1)
C(13)	54(1)	42(1)	48(1)	13(1)	14(1)	21(1)
C(14)	54(1)	48(1)	48(1)	14(1)	12(1)	17(1)
C(15)	48(1)	53(1)	42(1)	15(1)	7(1)	13(1)
C(16)	75(1)	74(1)	46(1)	4(1)	-3(1)	35(1)
C(17)	74(1)	72(1)	62(1)	14(1)	2(1)	36(1)
C(18)	50(1)	66(1)	55(1)	22(1)	-4(1)	8(1)
C(19)	80(1)	63(1)	51(1)	8(1)	-10(1)	12(1)
C(20)	79(1)	54(1)	55(1)	11(1)	3(1)	19(1)
C(21)	37(1)	39(1)	40(1)	9(1)	2(1)	15(1)
C(22)	39(1)	38(1)	44(1)	12(1)	4(1)	15(1)
C(23)	45(1)	43(1)	52(1)	14(1)	6(1)	19(1)
C(24)	60(1)	43(1)	61(1)	21(1)	5(1)	24(1)
C(25)	64(1)	48(1)	58(1)	24(1)	7(1)	16(1)
C(26)	56(1)	58(1)	61(1)	28(1)	20(1)	25(1)
C(27)	50(1)	49(1)	54(1)	20(1)	12(1)	25(1)
C(28)	42(1)	39(1)	45(1)	14(1)	6(1)	18(1)
C(29)	43(1)	43(1)	58(1)	5(1)	7(1)	14(1)
C(30)	53(1)	55(1)	48(1)	3(1)	9(1)	23(1)

C(31)	47(1)	58(1)	50(1)	17(1)	13(1)	22(1)
C(32)	48(1)	53(1)	62(1)	7(1)	14(1)	5(1)
C(33)	51(1)	45(1)	51(1)	4(1)	11(1)	12(1)
C(34)	71(1)	72(1)	88(2)	-2(1)	2(1)	23(1)
C(35)	80(1)	52(1)	59(1)	21(1)	29(1)	34(1)
C(36)	131(2)	87(2)	71(1)	-10(1)	-9(1)	20(2)
C(37)	68(1)	108(2)	62(1)	21(1)	24(1)	48(1)
C(38)	70(1)	116(2)	115(2)	61(2)	0(1)	35(1)

Table S5. Hydrogen coordinates (x 10⁴) and isotropic displacement parameters (Å² x 10³) for y2038.

	x	y	z	U(eq)
H(1)	8112	8589	4261	60
H(2)	8826	10283	5606	64
H(4)	6803	7862	6998	68
H(5)	6128	6171	5660	63
H(8)	7572	5015	4344	68
H(9)	6849	3362	2987	69
H(16)	2714	7284	1033	79
H(17)	1209	7697	-24	83
H(19)	789	4405	-1873	86
H(20)	2270	3974	-816	78
H(23)	4165	9133	3083	55
H(24)	4996	10645	2352	63
H(26)	8117	9157	1624	66
H(27)	7228	7607	2314	58
H(29)	4865	8829	4770	61
H(30)	3047	8659	5640	64
H(32)	637	5283	3795	71
H(33)	2494	5409	2972	62
H(34A)	9988	11467	7118	100
H(34B)	9273	11994	7937	100
H(34C)	8556	11745	6897	100
H(35A)	4997	3260	839	71
H(35B)	5918	2582	1297	71
H(36A)	3031	1273	134	131
H(36B)	2448	361	754	131
H(36C)	4006	588	518	131
H(37A)	1426	7677	6566	90
H(37B)	-244	7424	6349	90
H(37C)	859	8501	6027	90
H(38A)	-1081	7194	-962	116
H(38B)	-1280	7211	-2009	116
H(38C)	197	8062	-1323	116

The HR-ESIMS spectrum of compound **1a**

/u/data/TRAINING/zhangliping0917/1/pdata/1 xspec Fri Sep 17 15:53:18 2004

The HR-ESIMS spectrum of compound 1

/u/data/TRAINING/zhangliping1111/2/pdata/1 xspec Fri Nov 11 10:57:21 2005

¹H NMR spectrum of compound **1a** (400MHz, CDCl₃)

^{13}C NMR spectrum of compound **1a** (400MHz, CDCl_3)

DEPT spectrum of compound **1a** (400MHz, CDCl₃)

gCOSY spectrum of compound **1a** (400MHz, CDCl₃)

NOESY spectrum of compound **1a** (400MHz, CDCl₃)

STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory:
File: PROTON

Pulse Sequence: NOESY

Solvent: CDCl₃
Ambient temperature
Mercury-400SB "ULNOC400"

Relax. delay 1.000 sec
Mixing 0.400 sec
Acq. time 0.234 sec
Width 2163.4 Hz
40 us/pt 1133.4 Hz
2 x 656 increments
OBSERVE H1 400.1718148 MHz
DATA PROCESSING
Gauss apodization 0.108 sec
F1 DATA PROCESSING
Gauss apodization 0.084 sec
FT size 2048 x 2048
Total time 52 hr, 58 min, 15 sec

UV-Vis spectrum of Compound **1a**

UV-Vis spectrum of Compound **1a** in methanol (A versus λ),
 $c=9.859 \times 10^{-5}$ mol/L

Circular Dichroism spectrum of compound 1

Olis GlobalWorks Data Report

5/24/2006 10:54:11 AM

ZHANG LP-Subtract-Smooth-Smooth

Data name: ZHANG LP-Subtract-Smooth-Smooth
File name: C:\Program Files\OLIS GlobalWorks\Demo Data\Zhangliping-06.5.11\ZHANG LP-Subtract-Smooth.ols
Created: 5/24/2006 9:37:34 AM

Instrument: RSM
Collection mode: Scan
Number of points: 300
RSM Mono = 500 to 200 nm (2400 lines/mm)
Timing mode: Constant Time
Integration Time: 0.1 sec.
Reduction mode: Circular Dichroism
Scan mode: Fixed slitwidth
(manual Slit width = _____ mm)
Total Elapsed Time = 3:20.1 (min:sec)

Circular Dichroism spectrum of compound **1a**

Olis GlobalWorks Data Report

5/24/2006 11:31:24 AM

Sample-2-Subtract-Smooth-Smooth

Data name: Sample-2-Subtract-Smooth-Smooth
File name: C:\Program Files\OLIS GlobalWorks\Demo Data\Zhangliping-06.5.11\06.5.24r\CH3OH.ols
Created: 5/24/2006 9:37:34 AM

Instrument: RSM
Collection mode: Scan
Number of points: 300
RSM Mono = 500 to 200 nm (2400 lines/mm)
Timing mode: Constant Time
Integration Time: 0.1 sec.
Reduction mode: Circular Dichroism
Scan mode: Fixed slitwidth
(manual Slit width = _____ mm)
Total Elapsed Time = 3:20.1 (min:sec)