Supporting Information

Heliespirones B and C, two New Plant Heliespiranes with a Novel Spiro Heterocyclic Sesquiterpene Skeleton

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Figure S1. ^1H NMR of Heliespirone B (**2**, CDCl_3 , 600 MHz)

Figure S2. ^{13}C NMR of Heliespirone B (**2**, CDCl_3 , 100 MHz)

Figure S3. gHSQC of Heliespirone B (**2**, CDCl₃, 600 MHz)

Figure S4. ^1H NMR of Heliespirone C (**3**, CDCl_3 , 400 MHz)

Figure S5. ^{13}C NMR of Heliespirone C (**3**, CDCl_3 , 100 MHz)

Figure S6. COSY of Heliespirone C (**3**, CDCl₃, 400 MHz)

Table S1. ^{13}C NMR calculated and experimental data of heliespirones A and C (**3**). Calculation of the chemical shifts using NMRPredict program ccss v. 3.0.25 by Modgraph Consultants Ltd v. 2.0.

carbon #	calcd ^{13}C shifts for 1	exptl ^{13}C shifts for heliespirone A	exptl ^{13}C shifts for heliespirone C	calcd ^{13}C shifts for 3
1	83.6	87.6	86.9	85.1
2	194.8	201.5	196.5	190.2
3	136.9	137.0	136.9	136.9
4	152.7	153.4	151.7	152.7
5	192.1	197.5	196.3	191.3
6	44.0	51.8	48.5	41.5
7	136.7	135.3	134.6	134.7
8	41.9	57.1	47.0	49.5
9	29.2	31.9	32.4	27.9
10	74.0	86.7	86.7	83.8
11	79.0	70.1	70.3	71.2
12	27.9	28.3	27.5	25.9
13	27.9	25.3	24.5	25.9
14	116.9	118.4	119.6	117.9
15	16.5	15.9	16.1	16.5

Table S2. Crystal Data and Structure Refinement for heliespirone B (**2**)*Crystal Data*

Empirical formula	C ₁₅ H ₂₄ O ₄
Formula Weight	268.34
Temperature	105 K
Mo K α radiation	
Wavelength	0.71073 Å
Cell parameters from 5178 reflections	
Crystal system, space group	Monoclinic, P21
Unit cell dimensions	
	a = 10.3007 (14) Å
	b = 11.932 (2) Å
	c = 12.210 (2) Å
	$\alpha = 0^\circ$
	$\beta = 106.984 (9)^\circ$
	$\gamma = 0^\circ$
Volume	1435.3 (4) Å ³
Z	4
Calculated density	D _x = 1.242 Mg/m ³
	D _m not measured
Absorption coefficient	$\mu = 0.088 \text{ mm}^{-1}$
Crystal size	0.38 × 0.35 × 0.32 mm, colorless
Theta range for data collection	$\theta = 2.5\text{--}32.6^\circ$

Data Collection

KappaCCD (with Oxford Cryostream) diffractometer

 ω scans with κ offsets

Absorption correction: none

32482 measured reflections

5406 independent reflections

4753 reflections with $I > 2\sigma(I)$ R_{int} = 0.025

$h = -15 \rightarrow 15$

$k = -18 \rightarrow 14$

$l = -18 \rightarrow 18$

Intensity decay: <2%

Refinement

Refinement on F^2

$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 0.2847P]$ where $P = (F_o^2 + 2Fc^2)/3$

$R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.091$

$S = 1.029$

$(\Delta/\sigma)_{\max} = 0.000$

$\Delta\rho_{\max} = 0.32 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

5406 reflections

364 parameters

Extinction correction: SHELXL

Extinction coefficient: 0.012 (2)

H atoms treated by a mixture of independent and constrain refinement

Scattering factors from *International Tables for Crystallography* (Vol. C)

Figure S6. X-Ray Model of Heliespirone B (**2**)

Figure S7. X-Ray Model of Heliespirone B (**2**). Showing Intermolecular Interactions in the Crystal.