

# **A new Prenylated Flavonoid induces G0/G1 arrest and apoptosis through p38/JNK MAPK pathways in Human Hepatocellular Carcinoma cells**

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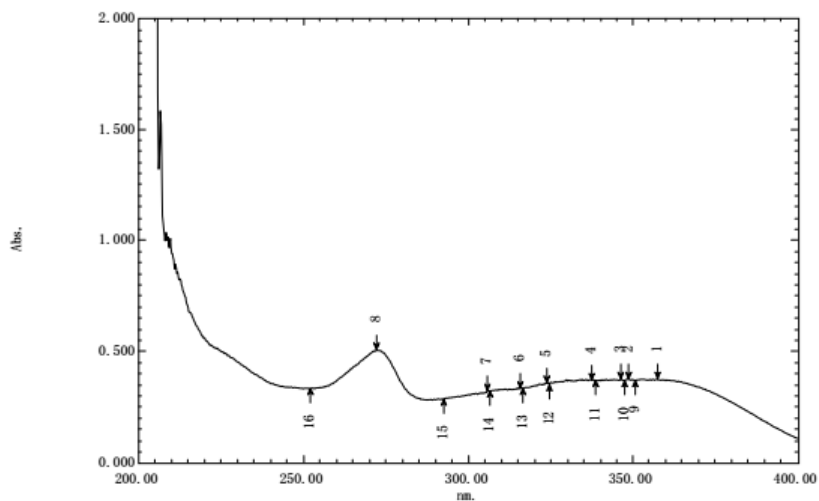
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<sup>4</sup>*School of Pharmaceutical Engineering, Shenyang Pharmaceutical University, Shenyang, 110016, People's Republic of China*

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<sup>+</sup>these authors contributed equally to this work



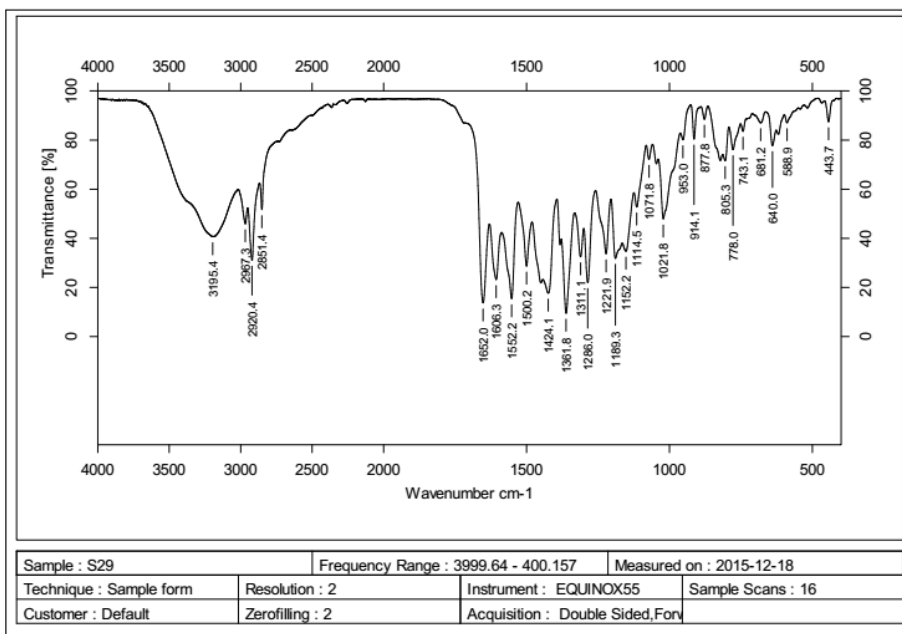
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 扫描模式: 单一的

试样准备属性  
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 稀释: 10  
 光程长: 407  
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仪器属性  
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 测定方式: 吸收值  
 狭缝宽: 1.0 nm  
 光源改变波长: 340.8 nm  
 S/R 转换: 标准

No.	P/V	Wavelength	Abs.	描述
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2	●	348.80	.375	
3	●	346.40	.374	
4	●	337.40	.373	
5	●	323.80	.357	
6	●	316.00	.337	
7	●	306.00	.325	
8	●	272.20	.506	
9	●	350.60	.370	
10	●	347.40	.370	
11	●	338.80	.368	
12	●	324.60	.355	
13	●	316.80	.333	
14	●	306.80	.322	
15	●	292.40	.284	
16	●	252.20	.332	

Supplementary Figure S1.1 UV spectrum of daphnegiravone D



Sample : S29		Frequency Range : 3999.64 - 400.157	Measured on : 2015-12-18
Technique : Sample form	Resolution : 2	Instrument : EQUINOX55	Sample Scans : 16
Customer : Default	Zerofilling : 2	Acquisition : Double Sided,For	

Supplementary Figure S1.2 IR spectrum of daphnegiravone D

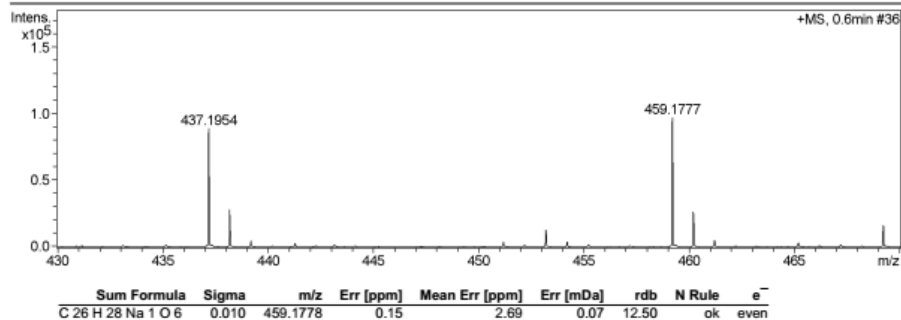
## Mass Spectrum Molecular Formula Report

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**Sample Name** SQ-29 **Instrument / Ser#** Bruker Customer 125  
**Comment**

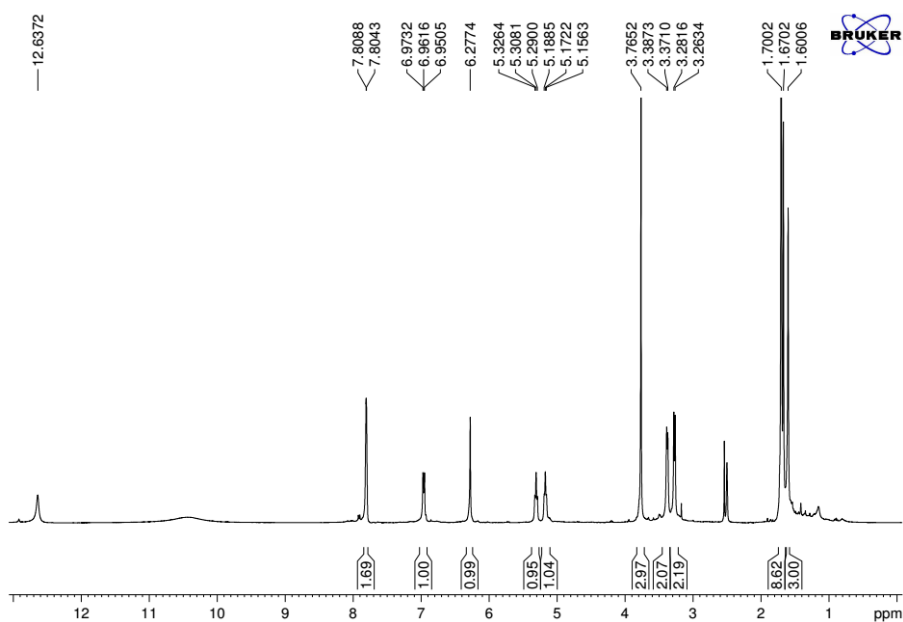
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 Scan End 3000 m/z Set Collision Cell RF 600.0 Vpp Set Divert Valve Source

**Generate Molecular Formula Parameter**

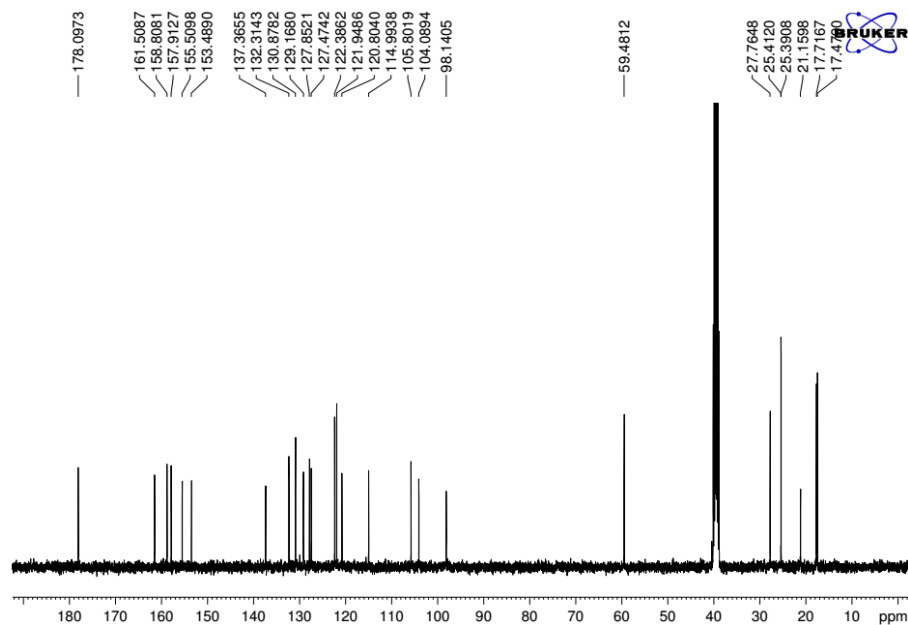
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Filter H/C Ratio	no	Minimum	0						
Estimate Carbon	yes								



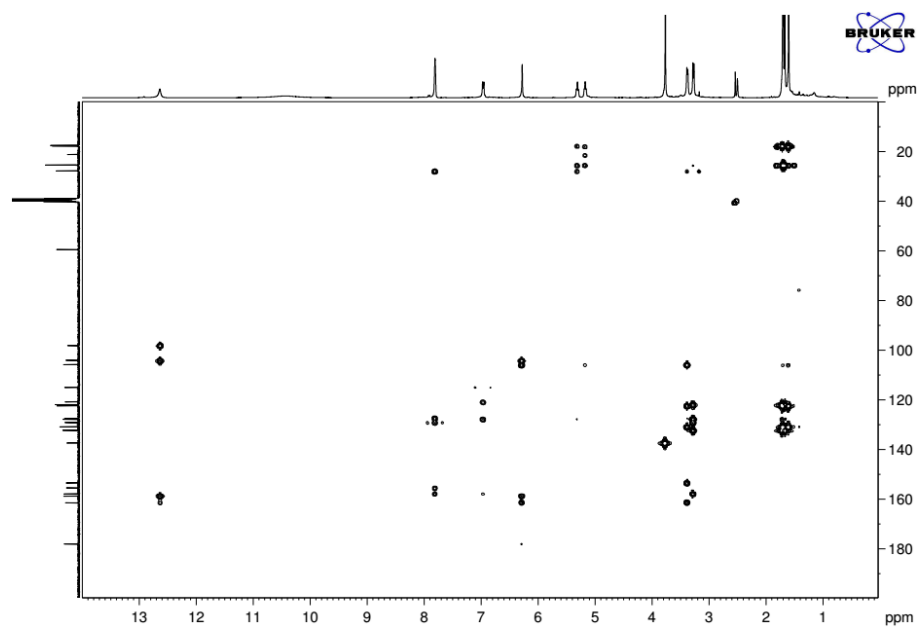
**Supplementary Figure S1.3** HRESIMS spectrum of daphnegiravone D



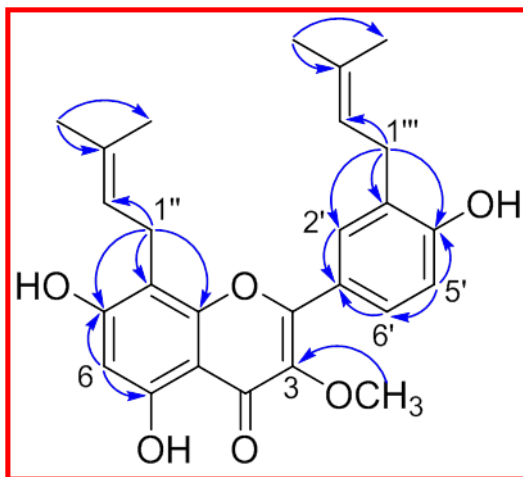
**Supplementary Figure S1.4** <sup>1</sup>H NMR spectrum (400 MHz, DMSO-*d*<sub>6</sub>) of daphnegiravone D



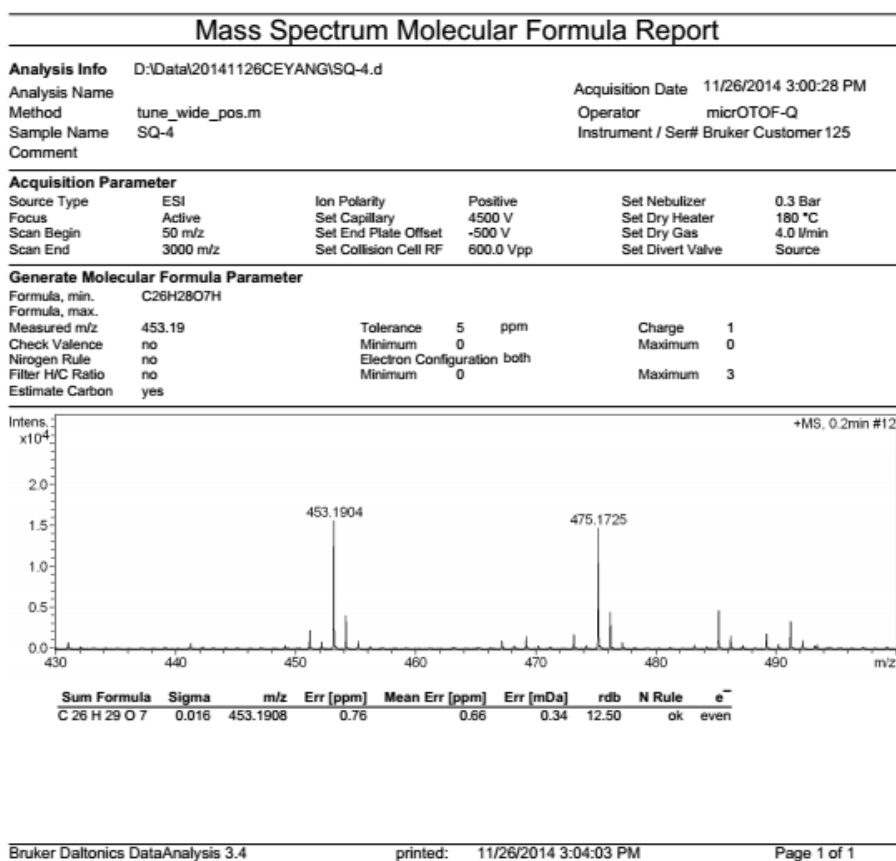
**Supplementary Figure S1.5**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{DMSO-}d_6$ ) of daphnegiravone D



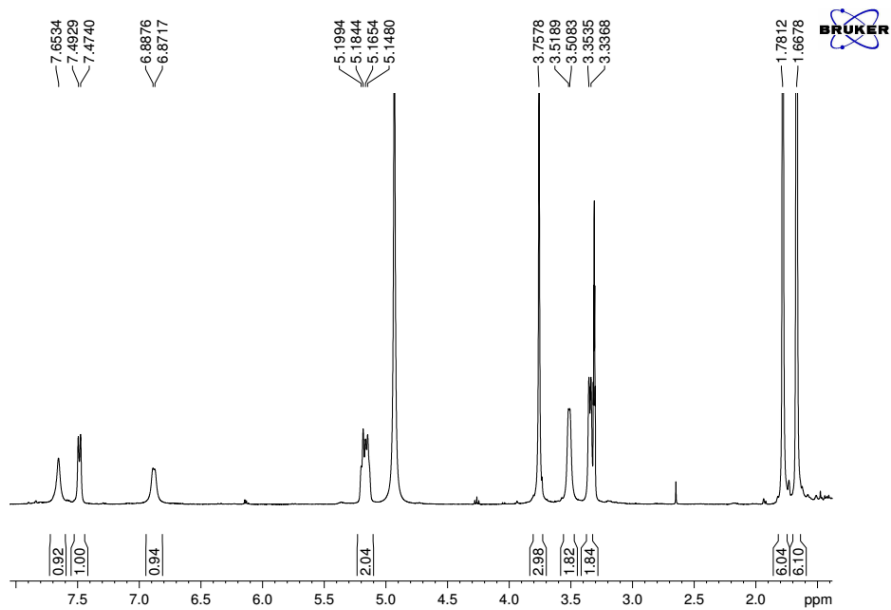
**Supplementary Figure S1.6** HMBC spectrum (600 MHz,  $\text{DMSO-}d_6$ ) of daphnegiravone D



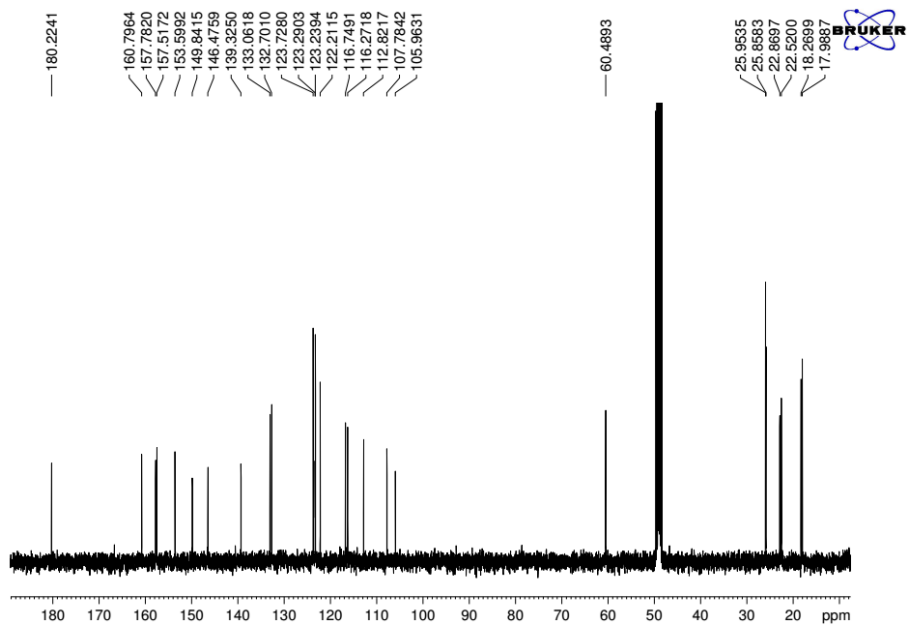
Supplementary Figure S1.7 Key HMBC correlations of daphnegiravone D.



Supplementary Figure S2.1 HRESIMS spectrum of brousoflavonol B



**Supplementary Figure S2.2**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{DMSO-}d_6$ ) of brousoflavonol B



**Supplementary Figure S2.3**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{DMSO-}d_6$ ) of brousoflavonol B

## Mass Spectrum Molecular Formula Report

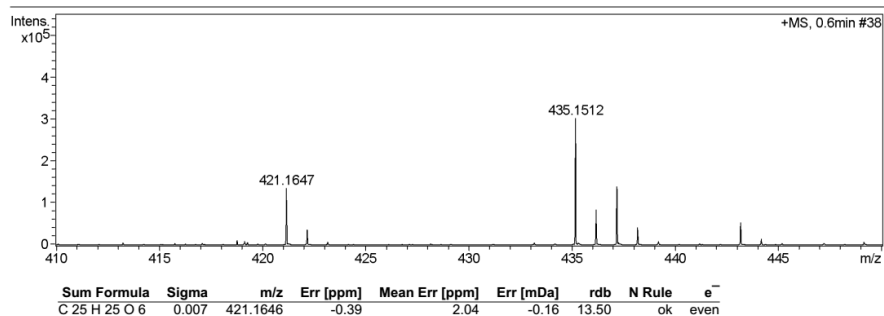
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Acquisition Date 12/11/2014 5:17:21 PM  
Operator micrOTOF-Q  
Instrument / Ser# Bruker Customer 125

### Acquisition Parameter

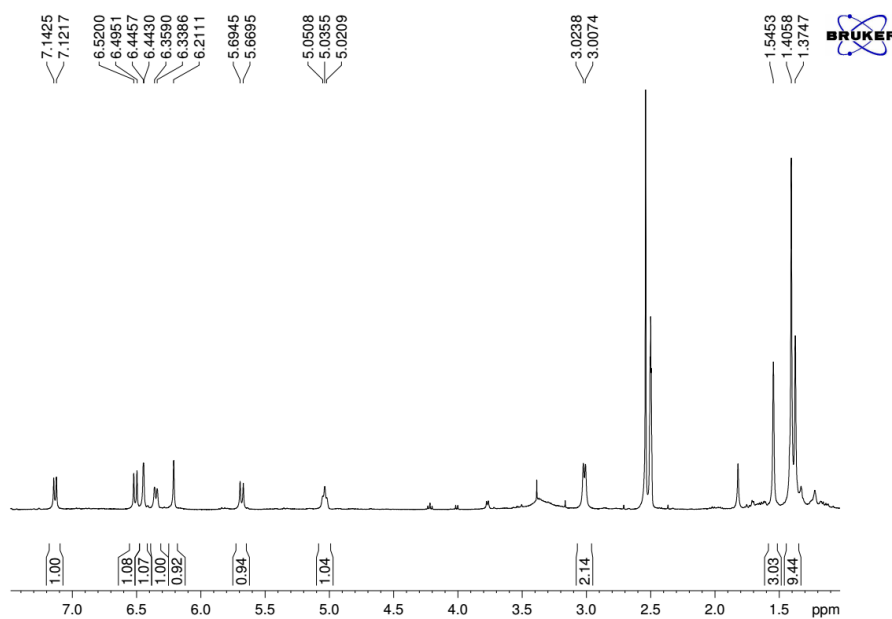
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### Generate Molecular Formula Parameter

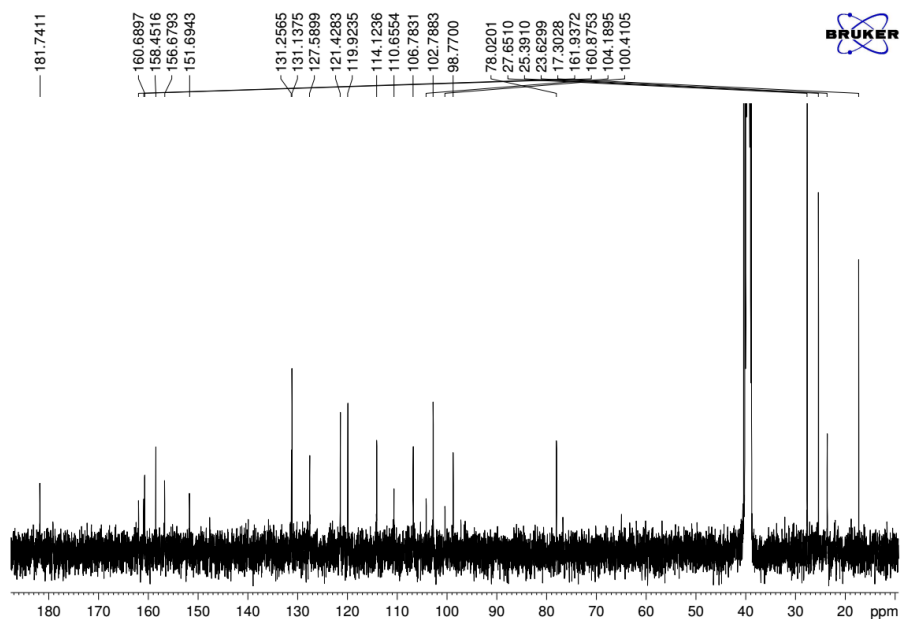
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Formula, max.					
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Check Valence	no	Minimum	0	Maximum	0
Nitrogen Rule	no	Electron Configuration	both		
Filter H/C Ratio	no	Minimum	0	Maximum	3
Estimate Carbon	yes				



## Supplementary Figure S3.1 HRESIMS spectrum of morusin



Supplementary Figure S3.2 <sup>1</sup>H NMR spectrum (400 MHz, DMSO-*d*<sub>6</sub>) of morusin



Supplementary Figure S3.3  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{DMSO-}d_6$ ) of morusin

### Mass Spectrum Molecular Formula Report

#### Analysis Info

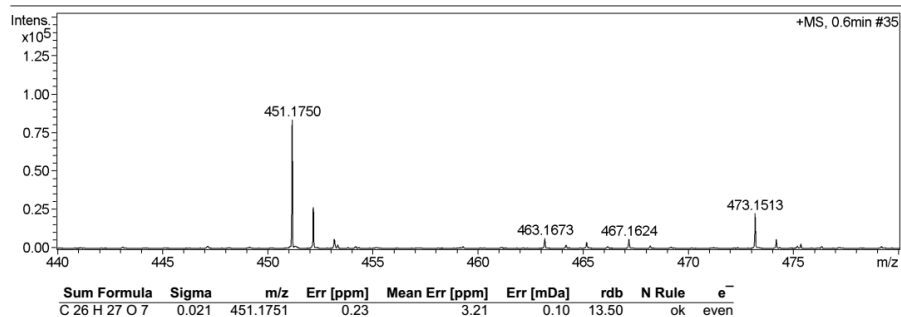
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Method	20150520yezhi.m	Operator	micrOTOF-Q
Sample Name	SQ-27	Instrument / Ser#	Bruker Customer 125
Comment			

#### Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 °C
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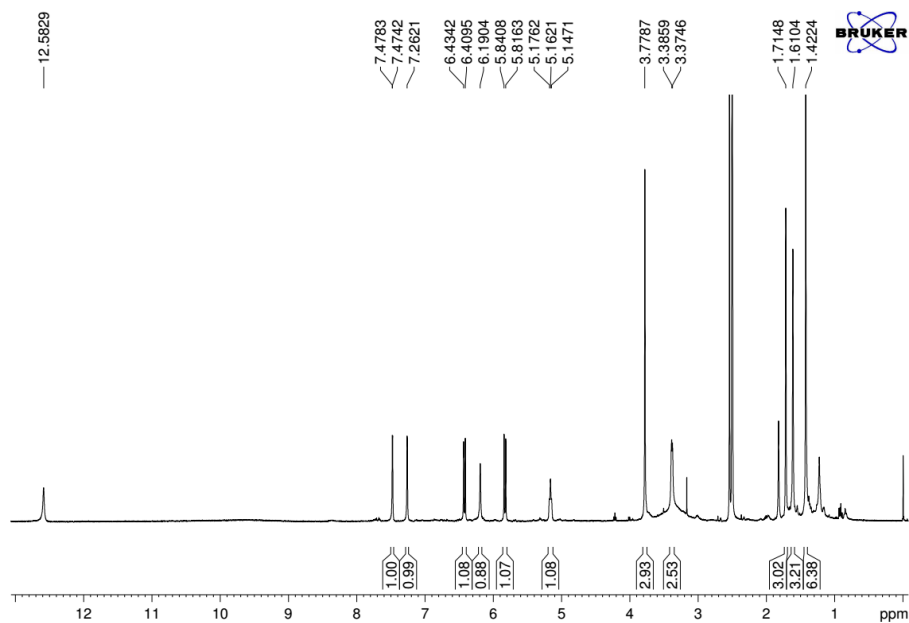
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Estimate Carbon	yes				

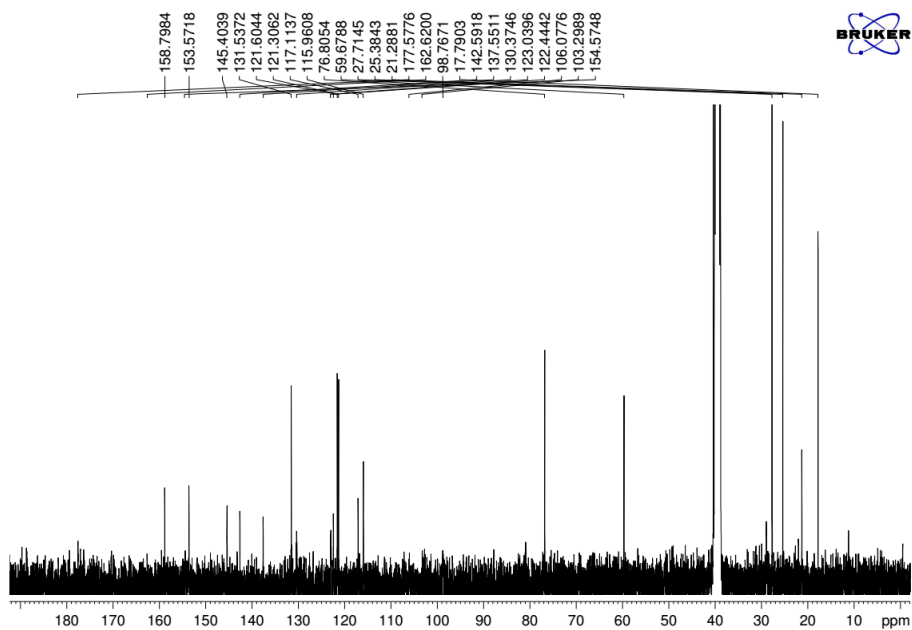


Supplementary Figure S4.1 HRESIMS spectrum of daphnegravone A





**Supplementary Figure S4.2**  $^1\text{H}$  NMR spectrum (400 MHz,  $\text{DMSO-}d_6$ ) of daphnegiravone A



**Supplementary Figure S4.3**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{DMSO-}d_6$ ) of daphnegiravone A

## Mass Spectrum Molecular Formula Report

### Analysis Info

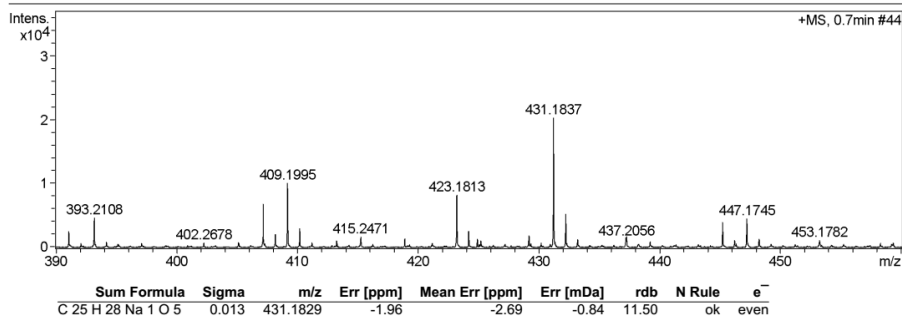
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Sample Name	HRX-30	Instrument / Ser#	Bruker Customer 125
Comment			

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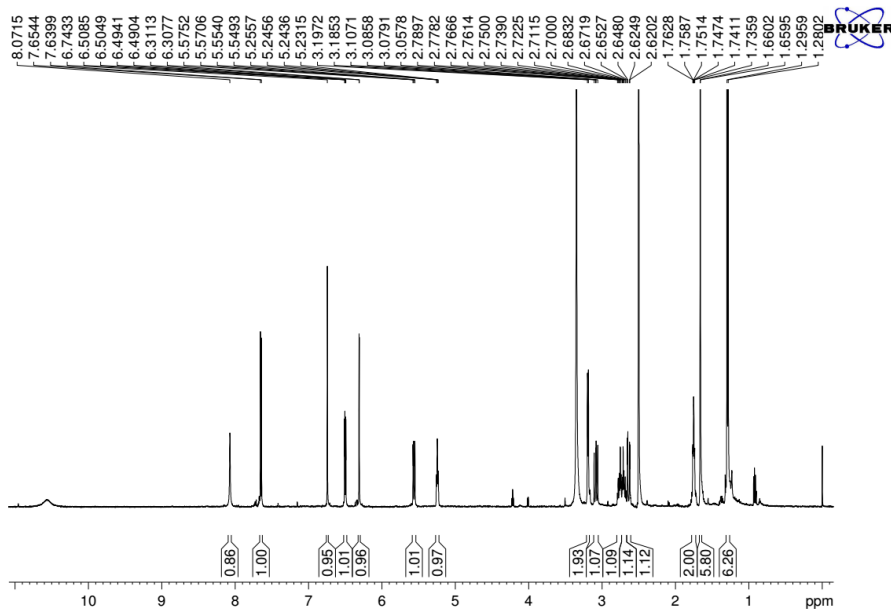
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### Generate Molecular Formula Parameter

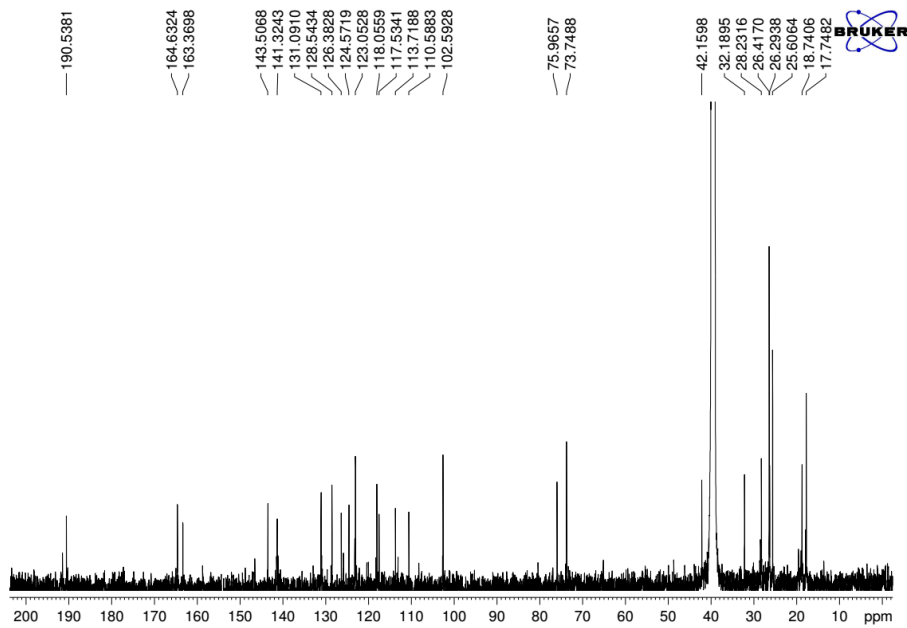
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Formula, max.			
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Filter H/C Ratio	yes	Minimum	0
Estimate Carbon	yes		
		Charge	1
		Maximum	0
		Maximum	3



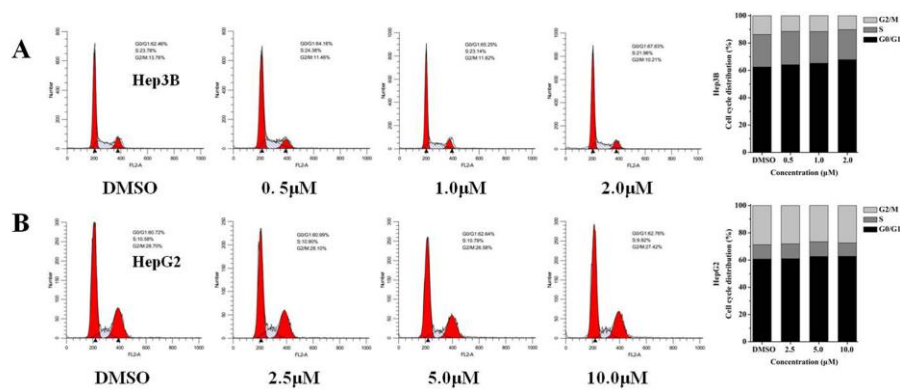
## Supplementary Figure S5.1 HRESIMS spectrum of daphnegiravone C



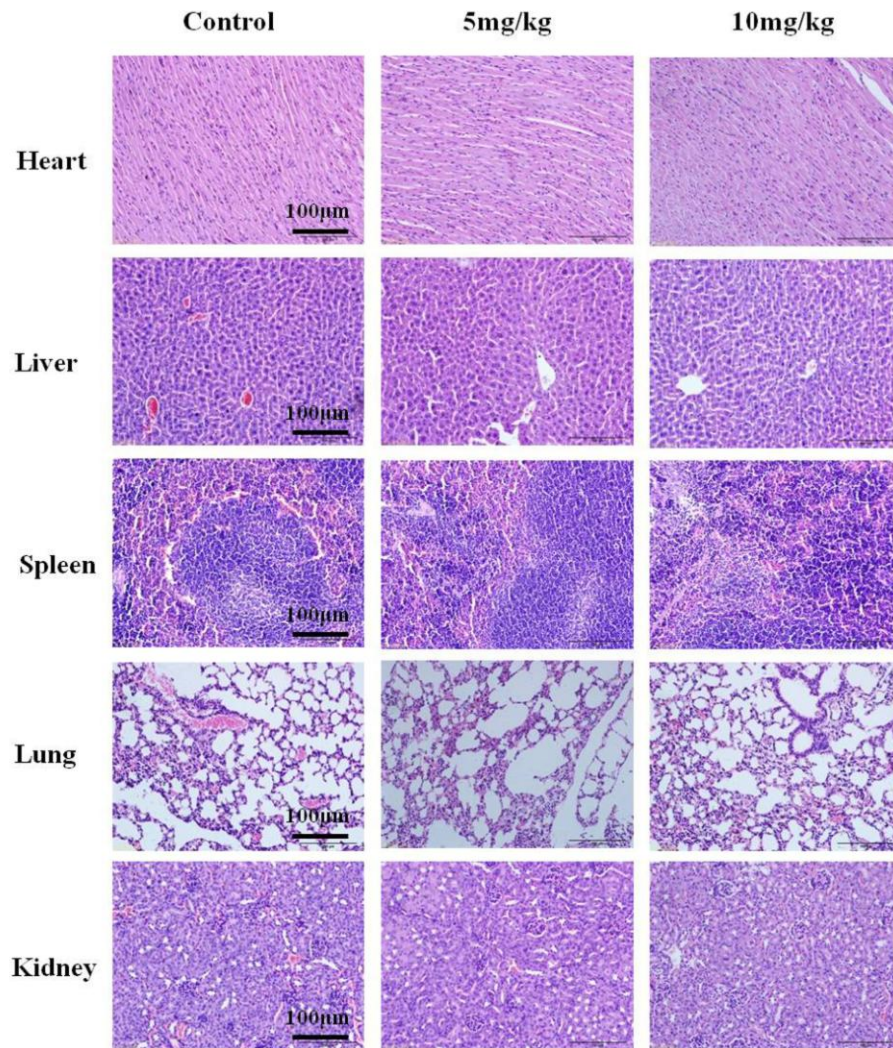
Supplementary Figure S5.2 <sup>1</sup>H NMR spectrum (400 MHz, DMSO-d<sub>6</sub>) of daphnegiravone C



**Supplementary Figure S5.3**  $^{13}\text{C}$  NMR spectrum (100 MHz,  $\text{DMSO-}d_6$ ) of daphnegiravone C



**Supplementary Figure S6** Cell cycle effects of daphnegiravone D on Hep3B and HepG2 cells. Cells were induced with a variety of concentrations (0.5–2.0  $\mu\text{M}$  for Hep3B and 2.5–10.0  $\mu\text{M}$  for HepG2 cells) for 24 h, and subsequently cells were harvested, stained and cell cycle were analyzed by flow cytometry. The histograms were presented to describe the effect of cell cycle.



**Supplementary Figure S7.** Histological analysis of heart, liver, spleen, lung and kidney obtained from nude mice after daphnegiravone D treatment. Hematoxylin-eosin staining; Scale bar, 100  $\mu\text{m}$ .