

Supporting Information

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Chemical Components and Their α -Glucosidase Inhibitory Activity from the Leaves of *Ficus carica* Linn.

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1. Experimental and Chemistry

1.1. General Experimental Procedures

¹H and ¹³C, 1D and 2D Nuclear Magnetic Resonance (NMR) spectra were obtained by Bruker Avance Neo-600 MHz NMR spectrometer (Bruker, Germany); a VG-Autospec-3000 mass spectrometer (Beckman Coulter, Inc. America) was adopted to acquire HR-ESIMS spectra. Thin Layer Chromatograph (TLC) plates of Silica gel GF₂₅₄ were purchased from Yantai Jiangyou Silicon Development Company (Yantai, China), and spots were observed by being exposed under UV light or heated after being sprayed with H₂SO₄ dissolved in ethanol (5% v/v). Purification by HPLC was carried out with LC-20AR pumps and an SPD-M20A UV detector (Shimadzu, Kyoto, Japan) An ELX800 microplate reader (BioTek, USA) was used to measure absorbance.

Chemicals: CHCl₃ and acetone were purchased from Chongqing Chuandong Chemical Group Co., Ltd.; MeOH, 95% ethanol, petroleum ether and ethyl acetate, CH₂Cl₂ were from Sinopharm Chemical Reagent Co., Ltd.; methanol for HPLC purification was acquired from Energy Chemical. Sodium dihydrogen phosphate dihydrate and Disodium hydrogen phosphate dodecahydrate were from Tianjin Kemiou Chemical Reagent Co., Ltd; α -Glucosidase from *Saccharomyces cerevisiae* was acquired from Macklin (Shanghai, China); the substrate (*p*-Nitrophenyl α -D-glucopyranoside, pNPG) and acarbose (positive drug) were acquired from Aladdin (Shanghai, China) and Shanghai Yuanye Bio-Technology Co., Ltd.

1.2. Extraction and Isolation

The air-dried and powdered leaves of *F. carica* (10 kg) were extracted under reflux with 95% ethanol 3 times, 2 h for each time, and the combined ethanol was concentrated to obtain crude extract. After being suspended with water, the crude extract was partitioned with EtOAc to obtain EtOAc part (310 g).

The EtOAc (310 g) partition was subjected to column chromatography on silica gel (gradient solvents: petroleum ether-EtOAc 100 : 0-0 : 100) to give fourteen fractions of Fr.E1~Fr.E14. Fr.E7 was then fractionated on an MCI column chromatography (MeOH-H₂O, 50-100% MeOH) to yield subfractions Fr.E7-1~Fr.E7-13. Fr.E7-6 was loaded to columns of silica gel (petroleum ether and EtOAc 98 : 2) to obtain Fr. E7-6-1~Fr.E7-6-8. Subfractions (Fr.E7-6-5-1 to Fr.E7-6-5-10) of Fr.E7-6-5 were obtained by a Sephadex LH-20 (MeOH), in which Fr.E7-6-5-4 was further recrystallized in MeOH to provide compounds **3** (15.2 mg) and **14** (4.3 mg). Compound **19** (5.3 mg) was obtained from the separation of Fr.E7-7 through columns of silica gel (petroleum ether-EtOAc 98 : 2), Sephadex LH-20 (MeOH-CH₂Cl₂ 70:30), semi-preparative HPLC (60% MeOH) and preparative TLC (petroleum ether-EtOAc 80 : 20).

Fr.E8 was isolated by an MCI column chromatography (MeOH-H₂O, 40-100% MeOH) to obtain Fr.E8-1~Fr.E8-12. Compounds **1** (26.1 mg) and **2** (35.6 mg) were obtained by recrystallization in MeOH from Fr.E8-4 and Fr.E8-10. Fr.E8-3 underwent silica gel column chromatography (petroleum ether-EtOAc 95:5), silica gel column chromatography (petroleum ether-acetone 9:1), and a semi-preparative HPLC (50% MeOH) to give compound **4** (4.7 mg). Fr. E8-4 was subjected to silica gel column (petroleum ether-acetone 19:1) to obtain Fr. E8-4-1~Fr. E8-4-7, then Fr. E8-4-7 was recrystallized in petroleum ether to obtain compound **10**. Compound **5** (7.2 mg) was isolated from Fr. E8-8 after two Sephadex LH-20 column chromatography (MeOH). Fr. E8-9 was chromatographed over a Sephadex LH-20 column (MeOH) to obtain Fr. E8-9-1~Fr. E8-9-7, then Fr. E8-9-3 was subjected to silica gel column (CHCl₃-MeOH 300:1) to obtain compound **17** (10.3 mg). Compound **18** (12.2 mg) was afforded from subfraction Fr. E8-9-7 by column chromatography on silica gel eluted with CHCl₃-MeOH (200:1). Fr. E8-11 was separated using Sephadex LH-20 column chromatography eluted with MeOH-CH₂Cl₂ (70:30) to obtain compound **15** (22.3 mg). Fr. E9 was eluted by MeOH-H₂O (40:60→100:0) on an MCI column to afford Fr. E9-1 ~ Fr. E9-7. Then, compound **6** (7.2 mg) was

directly crystallized from Fr. E9-4. Fr. E9-3 was separated by silica gel column chromatography (petroleum ether-acetone 50: 1) and a semi-preparative HPLC (50% MeOH) to afford compounds **16** (12.7 mg) and **11** (15.4 mg). Fr.E10 was subjected to column chromatography of MCI (MeOH-H₂O, 40-100% MeOH) to obtain Fr.E10-1~Fr.E10-6. Fr.E10-3 underwent column chromatography of Sephadex LH-20 (MeOH) and silica gel (petroleum ether and acetone 15:1), and then further purified by a semi-preparative HPLC (40% MeOH) to obtain compounds **12** (15.5mg) and **13** (13.2mg). Fr.E10-5 was separated by chromatography on a Sephadex LH-20 column (MeOH) and a silica gel column using a mixture of petroleum ether and acetone (10:1) to furnish Fr.E10-5-3-1~Fr.E10-5-3-3. Fr.E10-5-3-2 was recrystallized in petroleum ether to obtain compound **7** (15.7 mg). Fr. E10-5-3-3 was subjected to purification over a semi-preparative HPLC (43% MeOH) to yield compound **8** (17.3 mg). Fr.E11 was chromatographed over an MCI column (50% MeOH) and was recrystallized in petroleum ether to provide compound **9** (23.5 mg).

1.3 Identified compounds

The isolated compounds were identified from their spectroscopic data and by comparison with the data reported in the literature for umbelliferone (**1**) [1], psoralen (**2**) [2], furopinnarin (**3**) [3], 6,7-furano-hydrocoumaric acid (**4**) [4], (*E*)-3-[5-(6-hydroxy) benzofuranyl] propenoic acid (**5**) [5], (*E*)-3-(6-hydroxy-4-methoxy-5-benzofuranyl) propenoic acid (**6**) [6], nodakenetin (**7**) [7], oxypeucedanin hydrate (**8**) [8], dihydrofurocoumarin (**9**) [9], (*E*)-4-hydroxy-3,3,5-trimethyl-4-(3-oxobut-1-en-1-yl)-cyclohexan-1-one (**10**) [10], dehydrovomifoliol (**11**) [11], 4,5-dihydroblumenol A (**12**) [12], blumenol A (**13**) [12], flavonoids 5-hydroxy-4',7-dimethoxyisoflavone (**14**) [13], cajanin (**15**) [14], loliolide (**16**) [15], indole-3-carboxaldehyde (**17**) [16], 1H-indole-3-carboxylic acid (**18**) [17] and vitamin E quinone (**19**) [18], among which compounds **1-9** were coumarins, **10-13** were sesquiterpenoids, and **14-15** were flavonoids.

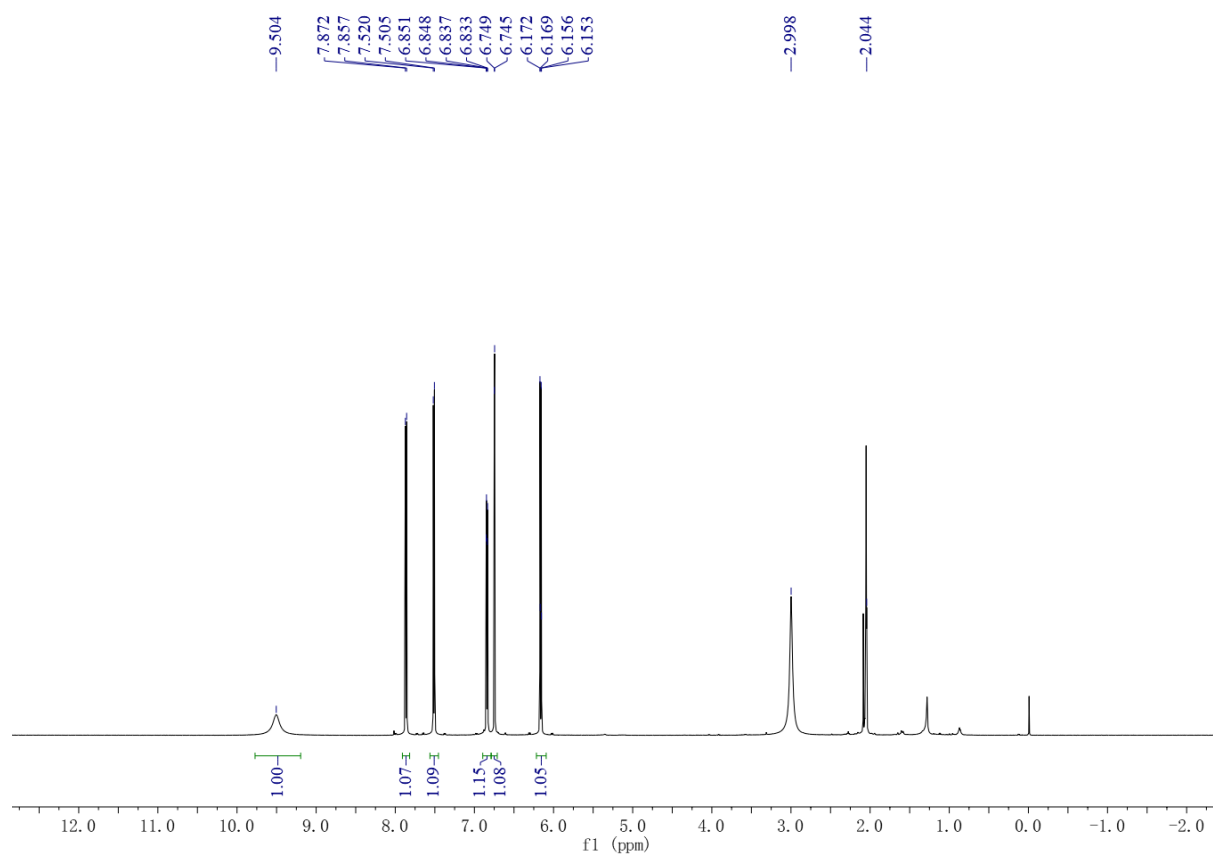


Figure S1: ¹H-NMR (600 MHz, CD₃COCD₃) spectrum of **1** (umbelliferone)

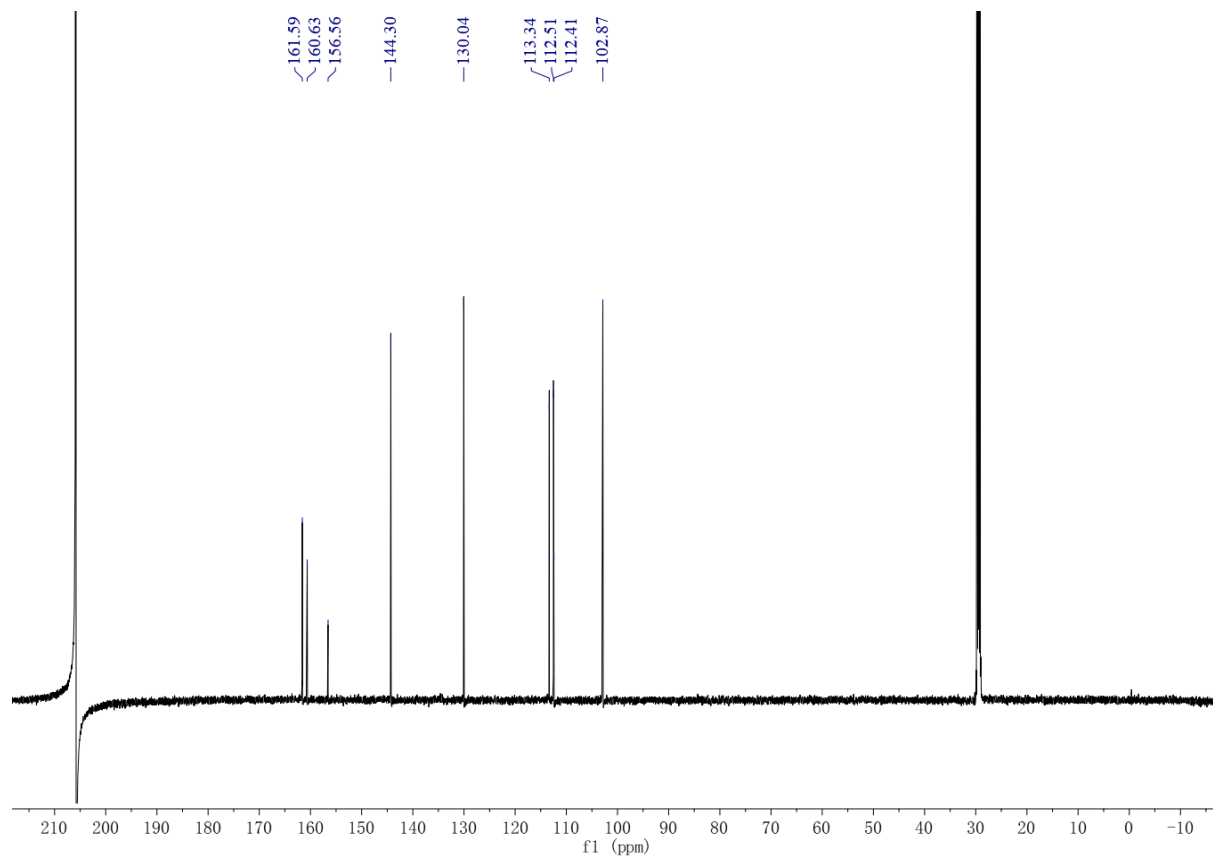


Figure S2: ^{13}C -NMR (150 MHz, CD_3COCD_3) spectrum of **1** (umbelliferone)

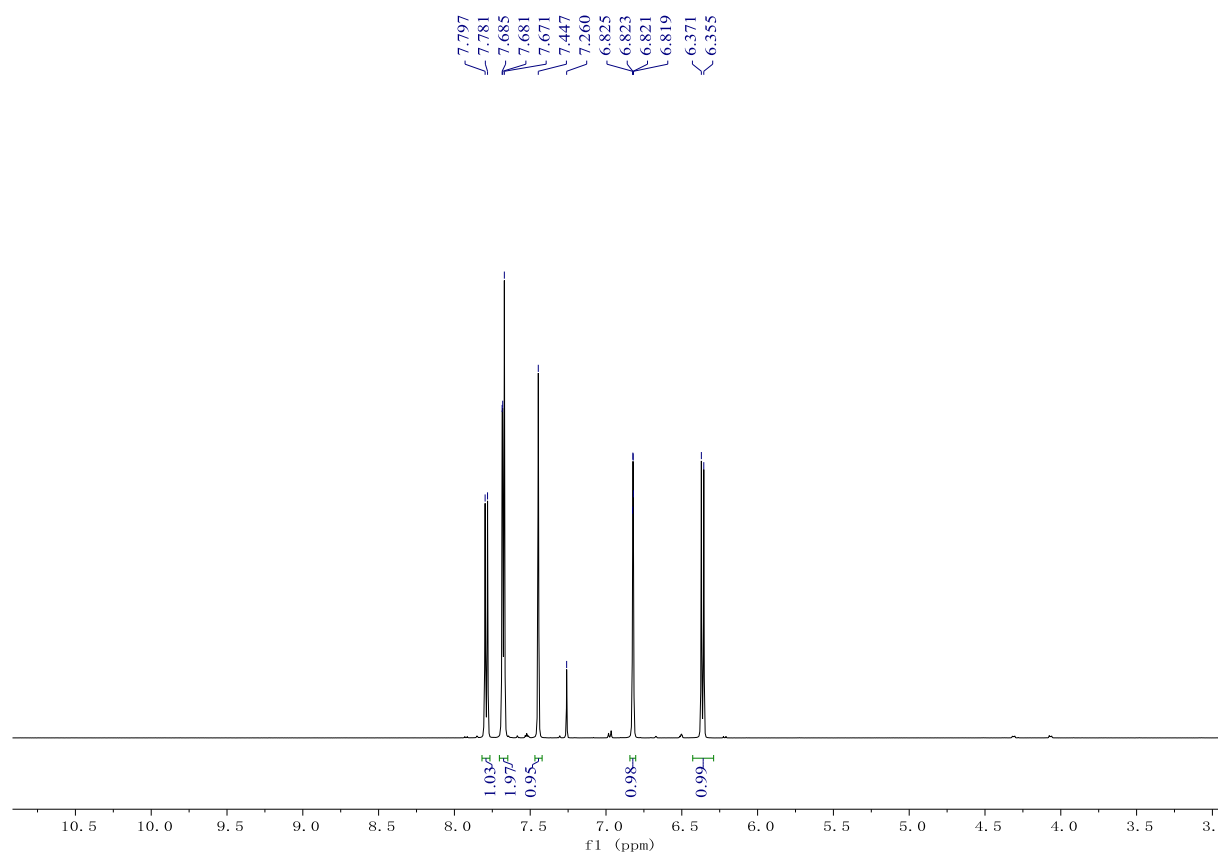


Figure S3: ^1H -NMR (600 MHz, CDCl_3) spectrum of **2** (psoralen)

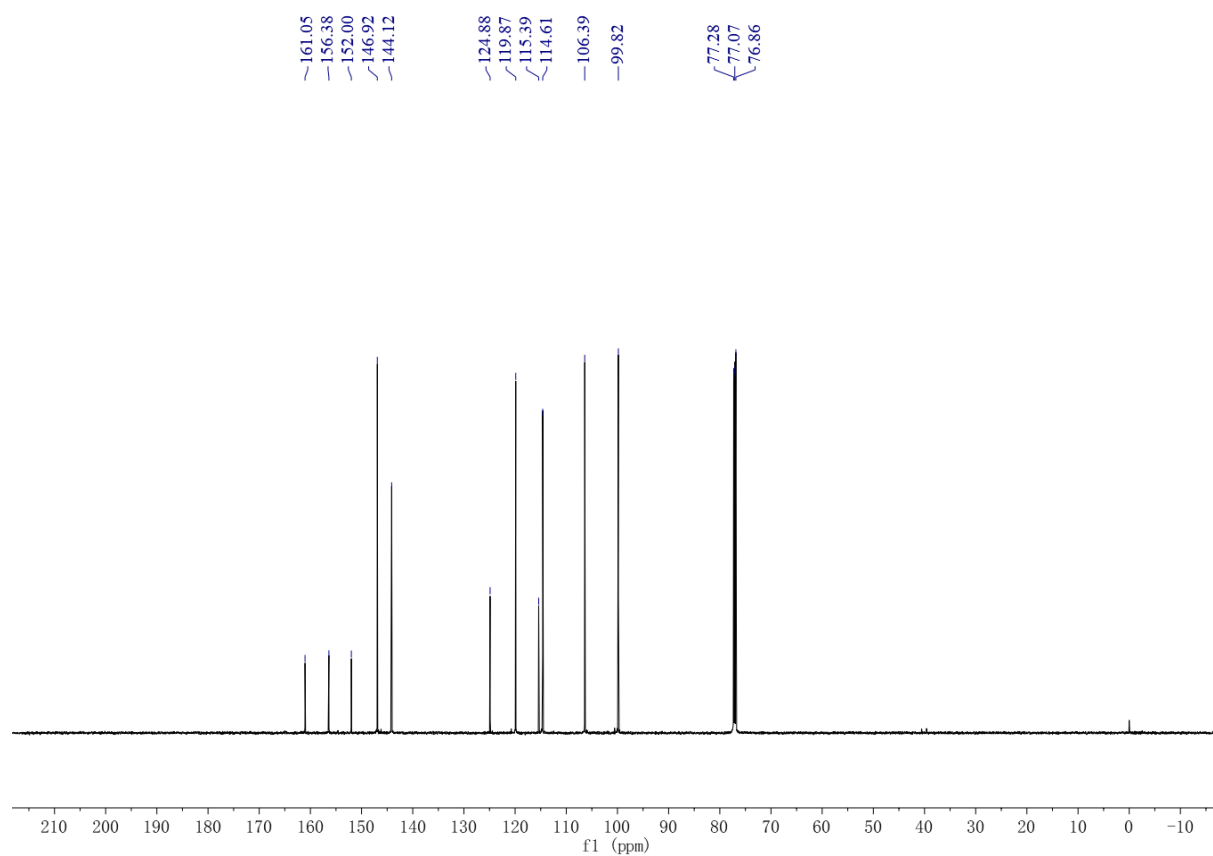


Figure S4: ^{13}C -NMR (150 MHz, CDCl_3) spectrum of **2** (psoralen)

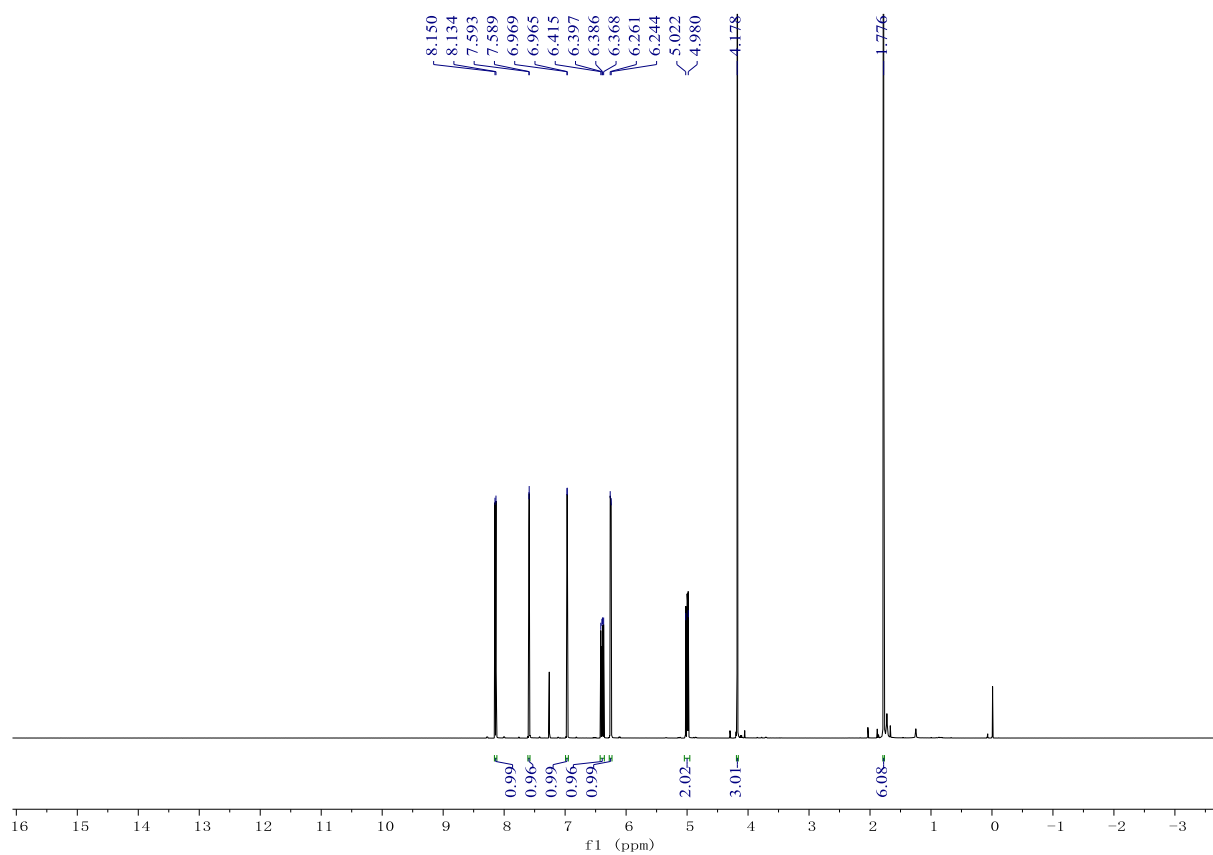


Figure S5: ¹H-NMR (600 MHz, CDCl₃) spectrum of **3** (furopinnarin)

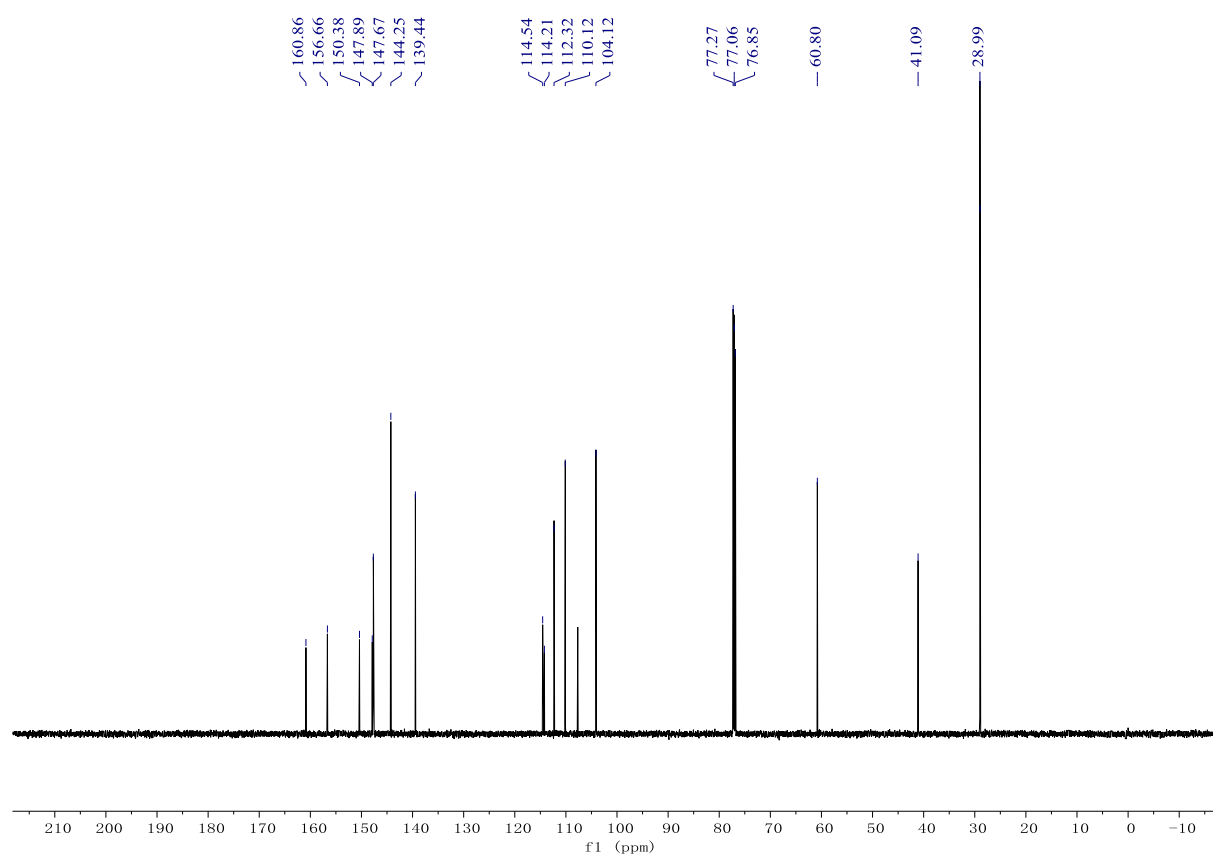


Figure S6: ^{13}C -NMR (150 MHz, CDCl_3) spectrum of **3** (fuopinnarin)

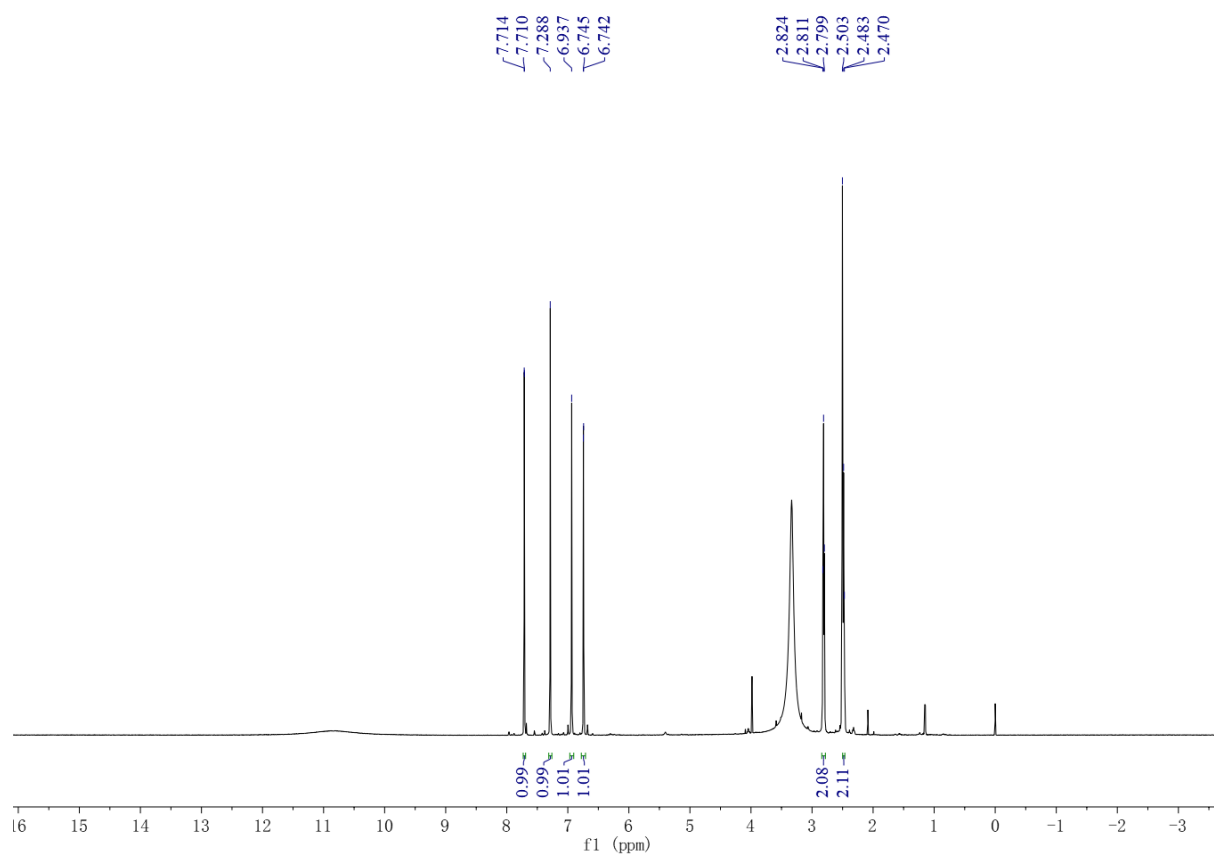


Figure S7: ^1H -NMR (600 MHz, DMSO-d_6) spectrum of **4** (6,7-Furano-hydrocoumaric acid)

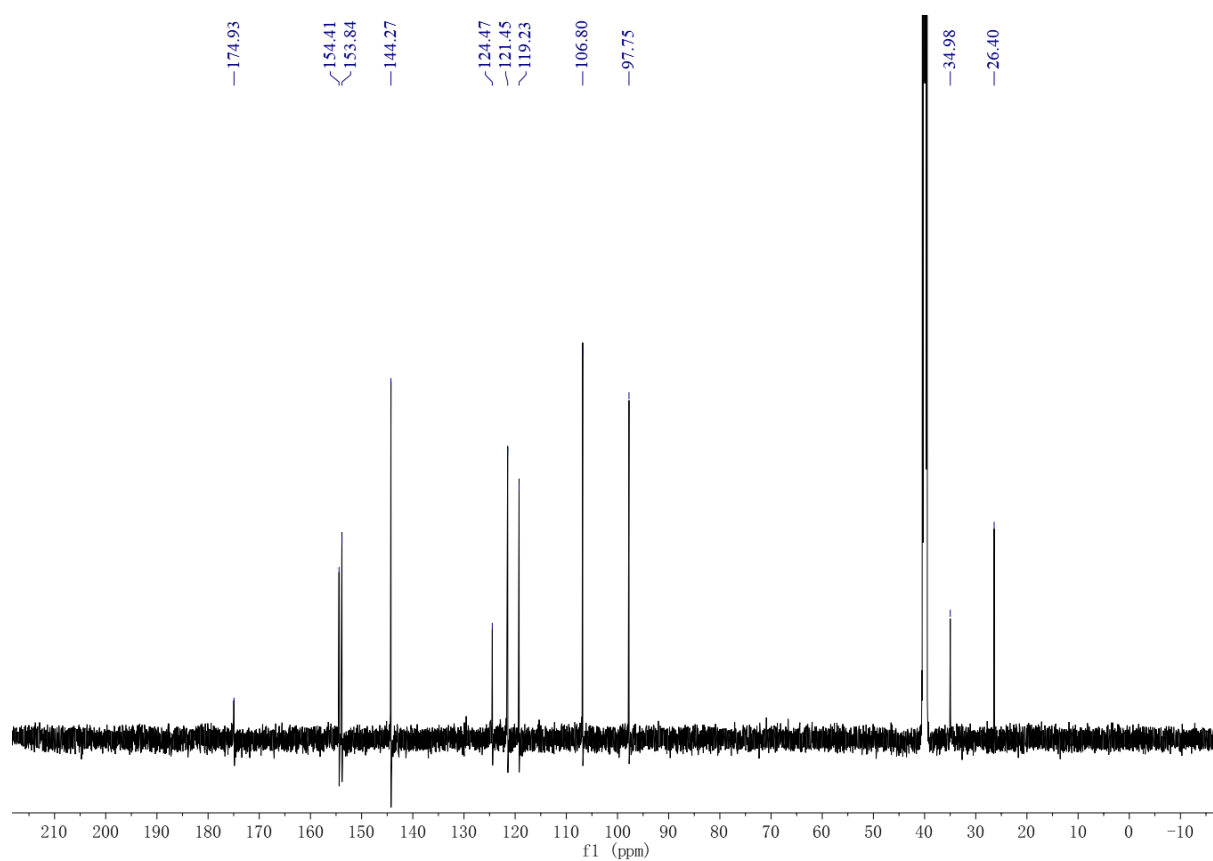


Figure S8: ^{13}C -NMR (150 MHz, DMSO-d_6) spectrum of **4** (6,7-Furano-hydrocoumaric acid)

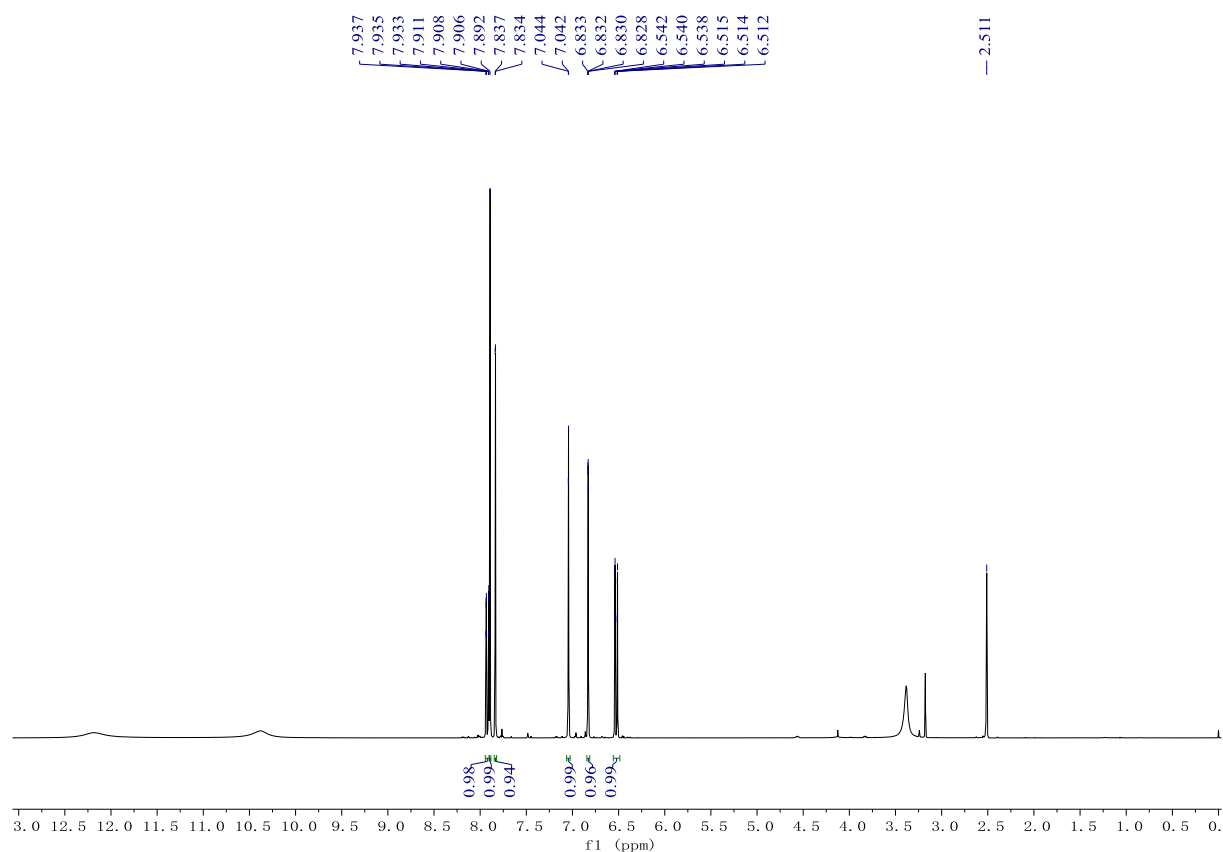


Figure S9: ^1H -NMR (600 MHz, DMSO-d_6) spectrum of **5** ((*E*)-3-[5-(6-hydroxy) benzofuranyl] propenoic acid)

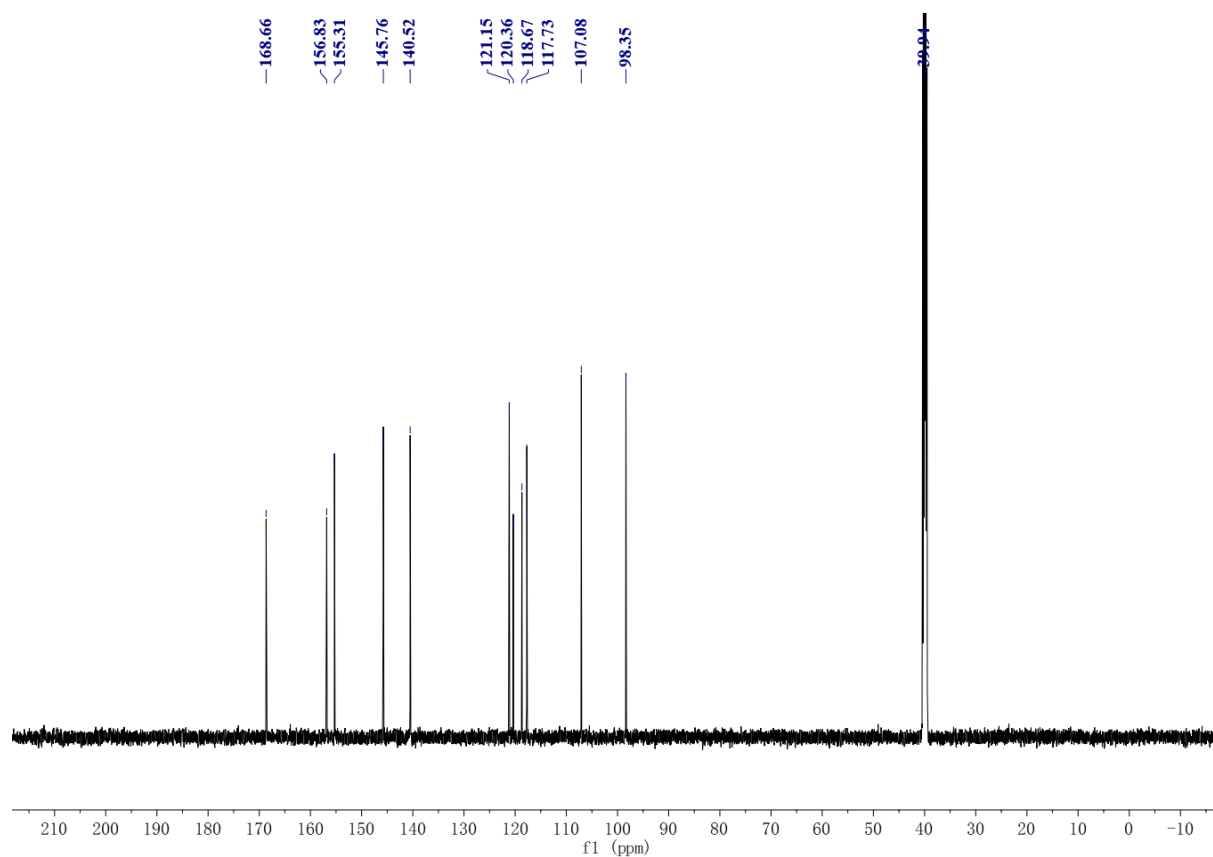


Figure S10: ^{13}C -NMR (150 MHz, DMSO-d_6) spectrum of **5** ((*E*)-3-[5-(6-hydroxy) benzofuranyl] propenoic acid)

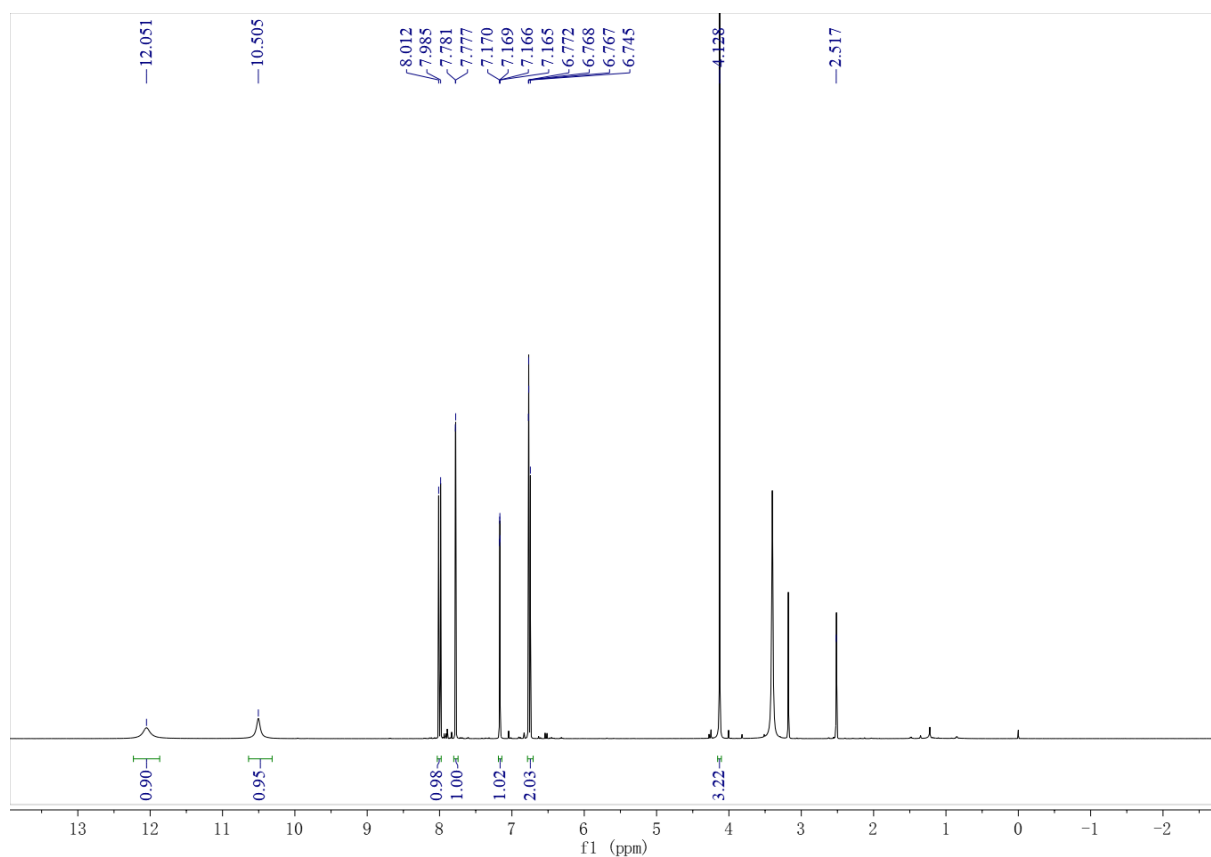


Figure S11: ^1H -NMR (600 MHz, DMSO-d_6) spectrum of **6** (*(E)*-3-(6-hydroxy-4-methoxy-5-benzofuranyl) propenoic acid)

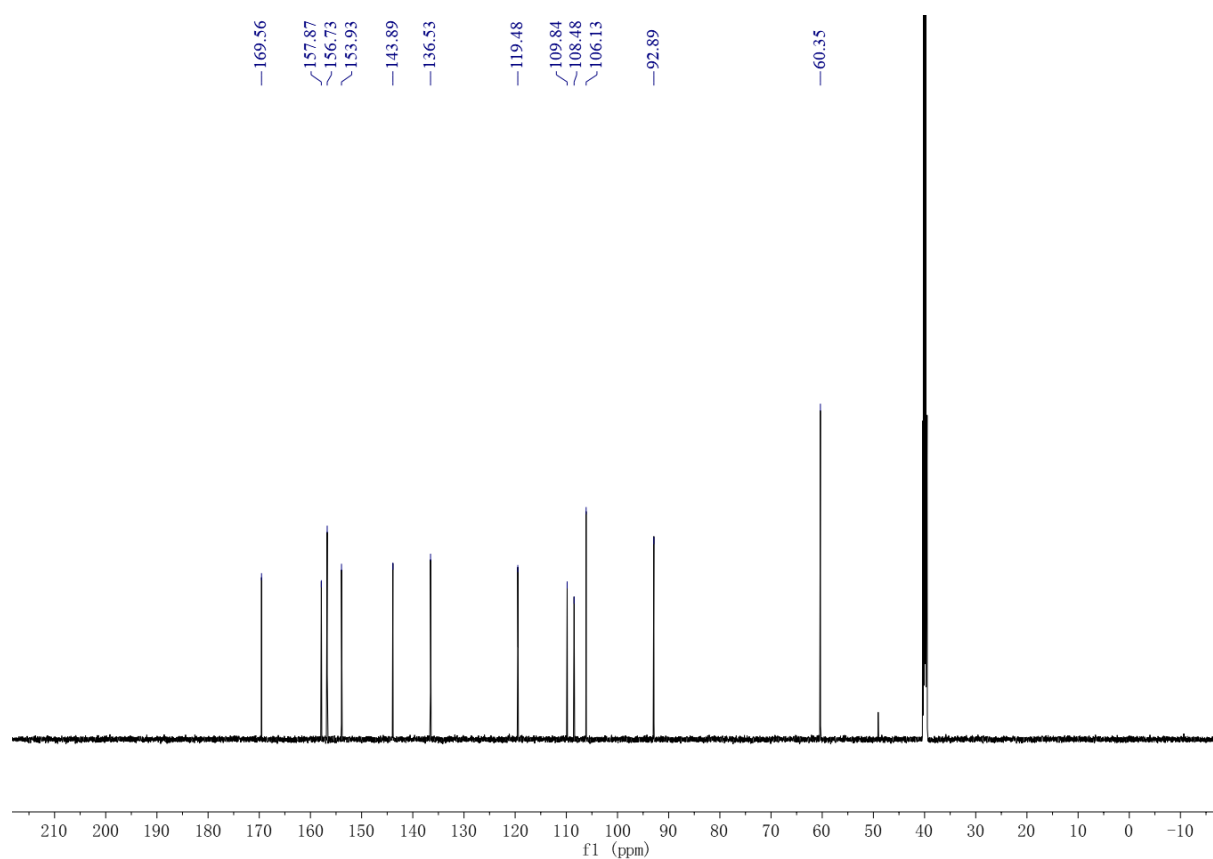


Figure S12: ^{13}C -NMR (150 MHz, DMSO- d_6) spectrum of **6** ((*E*)-3-(6-hydroxy-4-methoxy-5-benzofuranyl) propenoic acid)

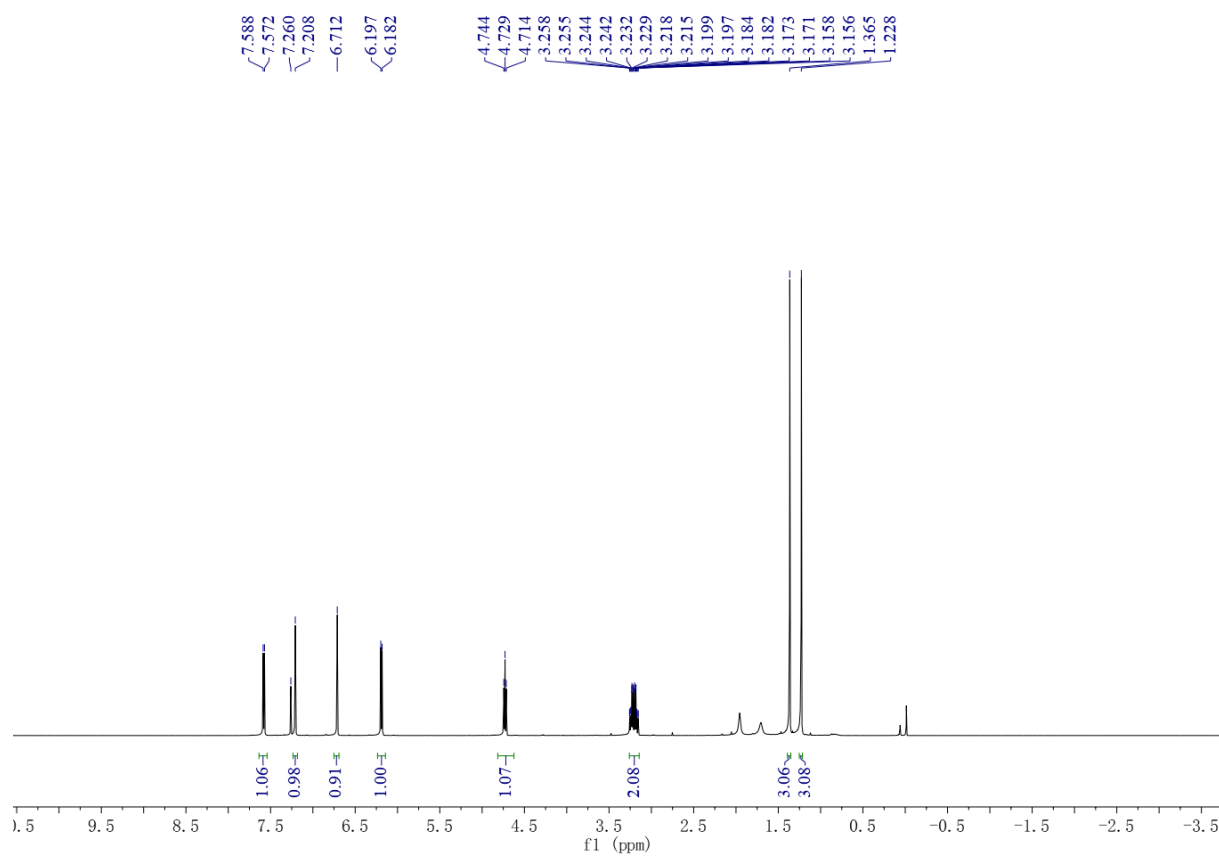


Figure S13: ¹H-NMR (600 MHz, CDCl₃) spectrum of **7** (nodakenetin)

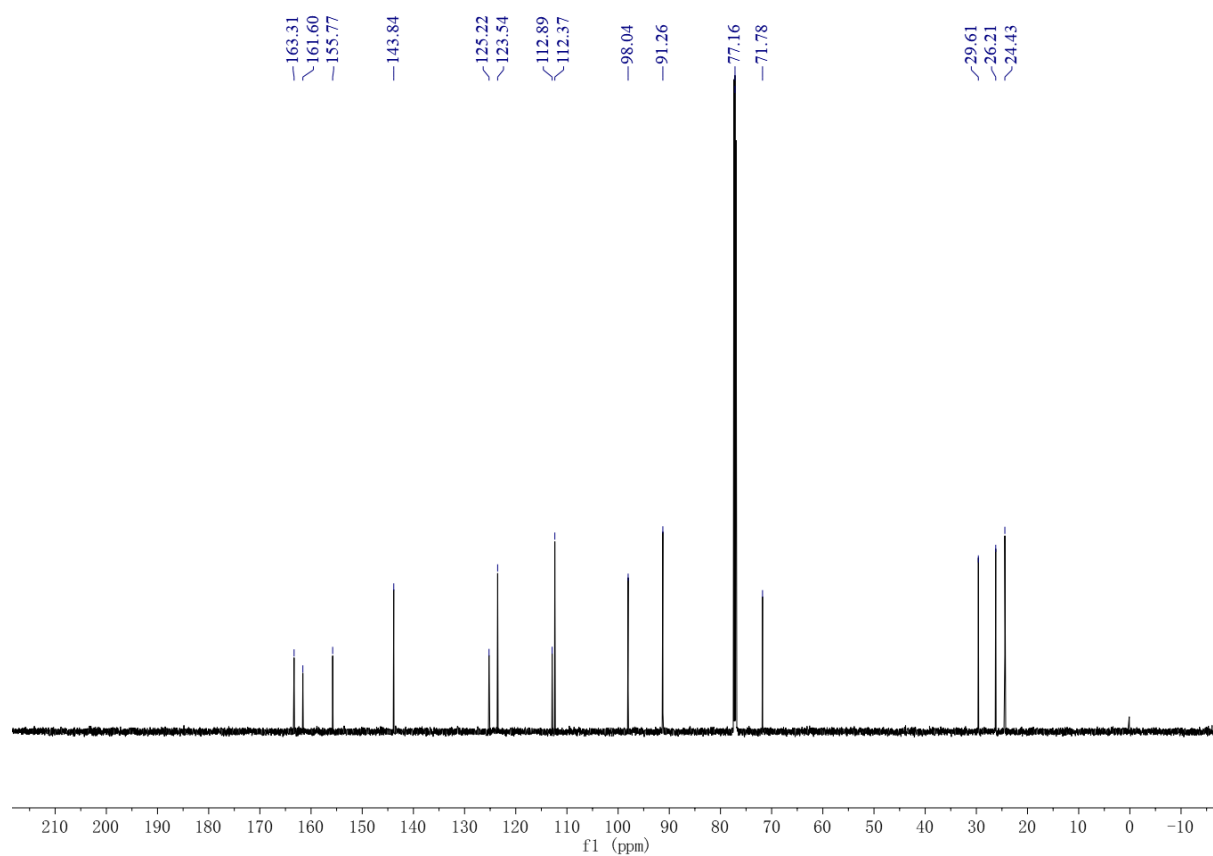


Figure S14: ¹³C-NMR (150 MHz, CDCl₃) spectrum of **7** (nodakenetin)

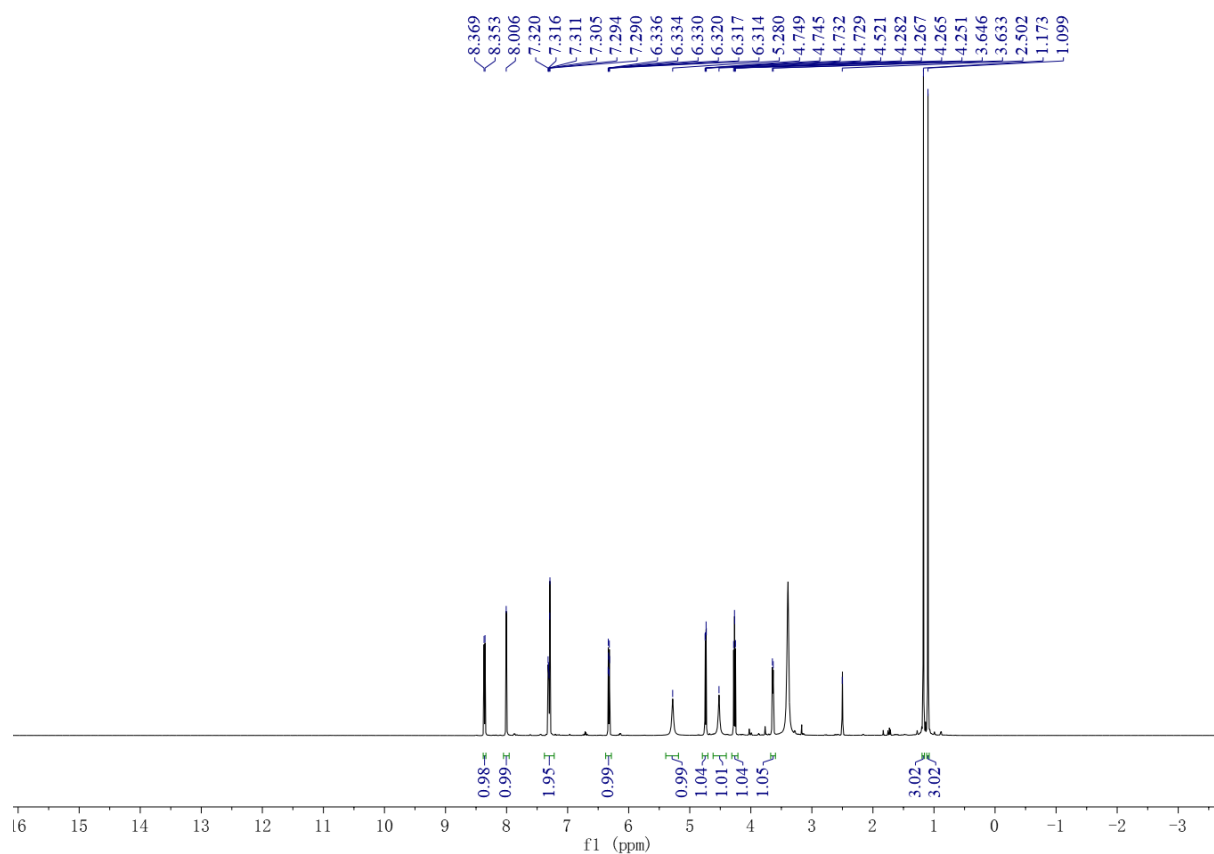


Figure S15: ^1H -NMR (600 MHz, DMSO-d_6) spectrum of **8** (oxypeucedanin hydrate)

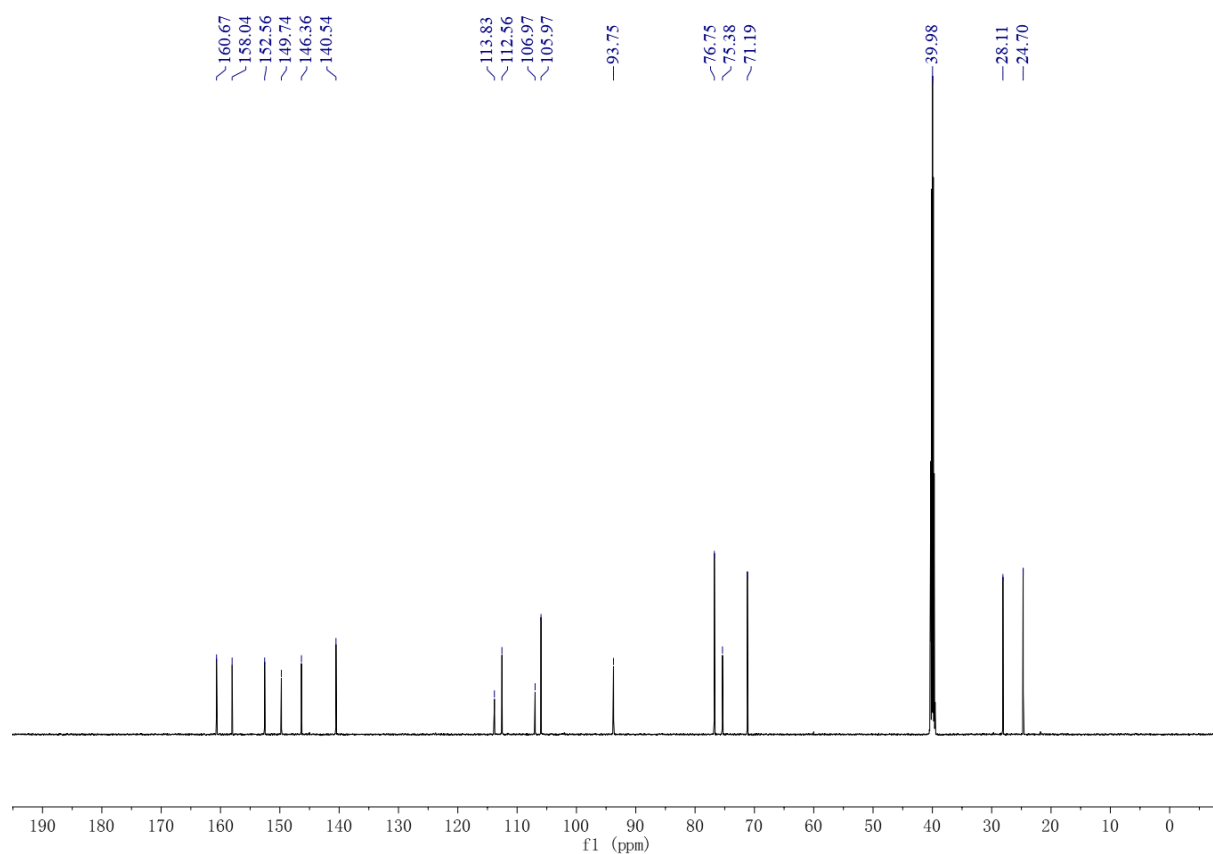


Figure S16: ^{13}C -NMR (150 MHz, DMSO-d_6) spectrum of **8** (oxypeucedanin hydrate)

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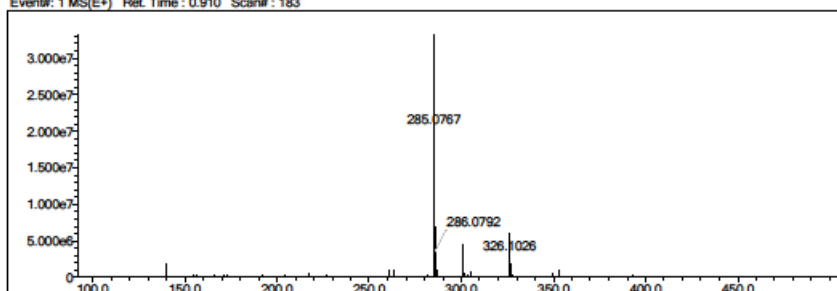
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C	4	0	50	F	1	0	0	Cl	1	0	0	Ru	2	0	0	
N	3	0	0	P	3	0	0	K	1	0	0					

Error Margin (ppm): 20
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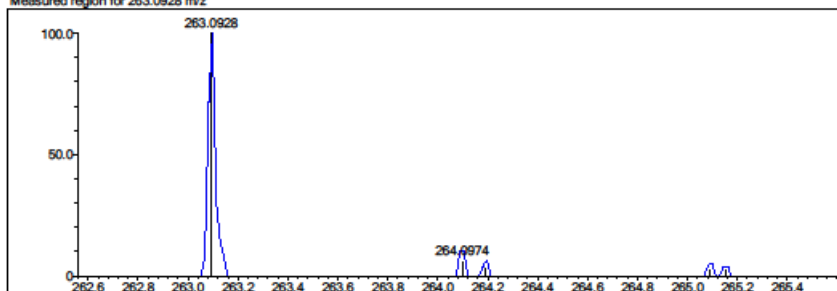
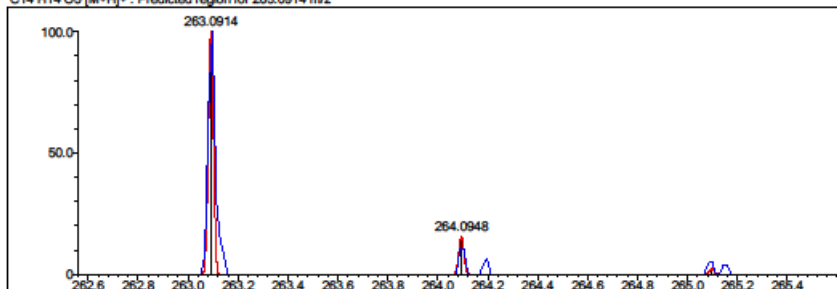
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 MSn Logic Mode: AND

Electron Ions: both
 Use MSn Info: no
 Isotope Res: 10000
 Max Results: 50

Event#: 1 MS(E+) Ret. Time : 0.910 Scan#: 183



Measured region for 263.0928 m/z

C14 H14 O5 [M+H]⁺ : Predicted region for 263.0914 m/z

Rank	Score	Formula (M)	Ion	Meas. m/z	Pred. m/z	Df. (mDa)	Df. (ppm)	Iso	DBE
1	41.60	C14 H14 O5	[M+H] ⁺	263.0928	263.0914	1.4	5.32	47.93	8.0

Figure S17: HR-ESI-MS spectrum of **9** (dihydrofurocoumarin)

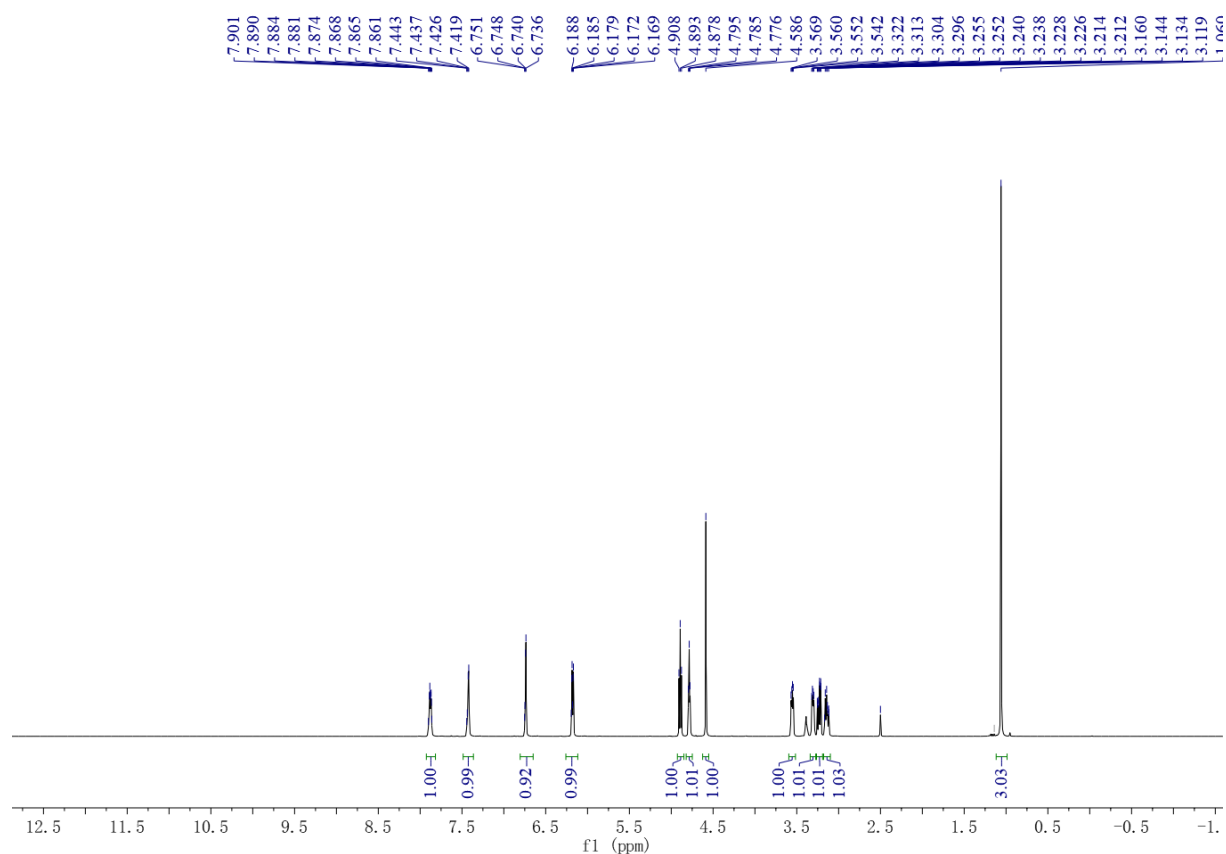


Figure S18: ^1H -NMR (600 MHz, DMSO-d_6) spectrum of **9** (dihydrofurocoumarin)

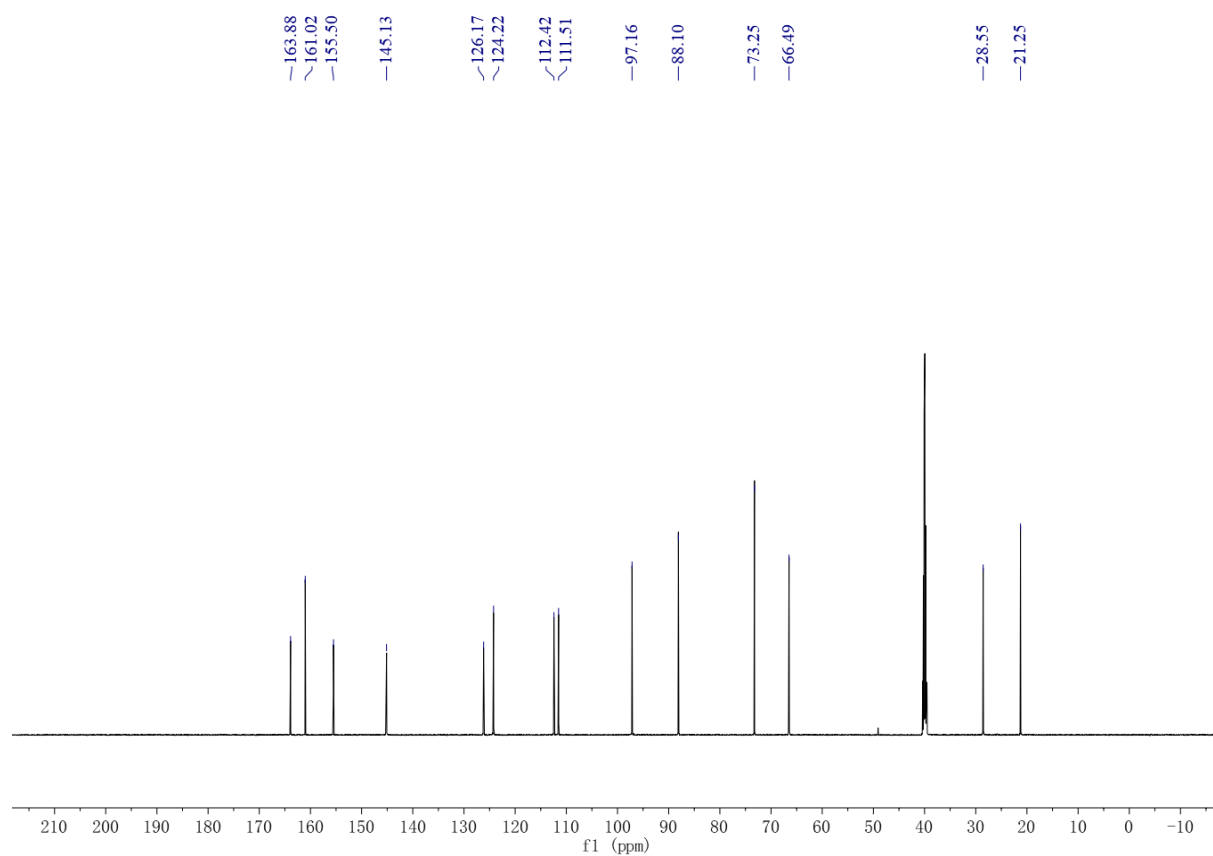


Figure S19: ^{13}C -NMR (150 MHz, DMSO-d_6) spectrum of **9** (dihydrofurocoumarin)

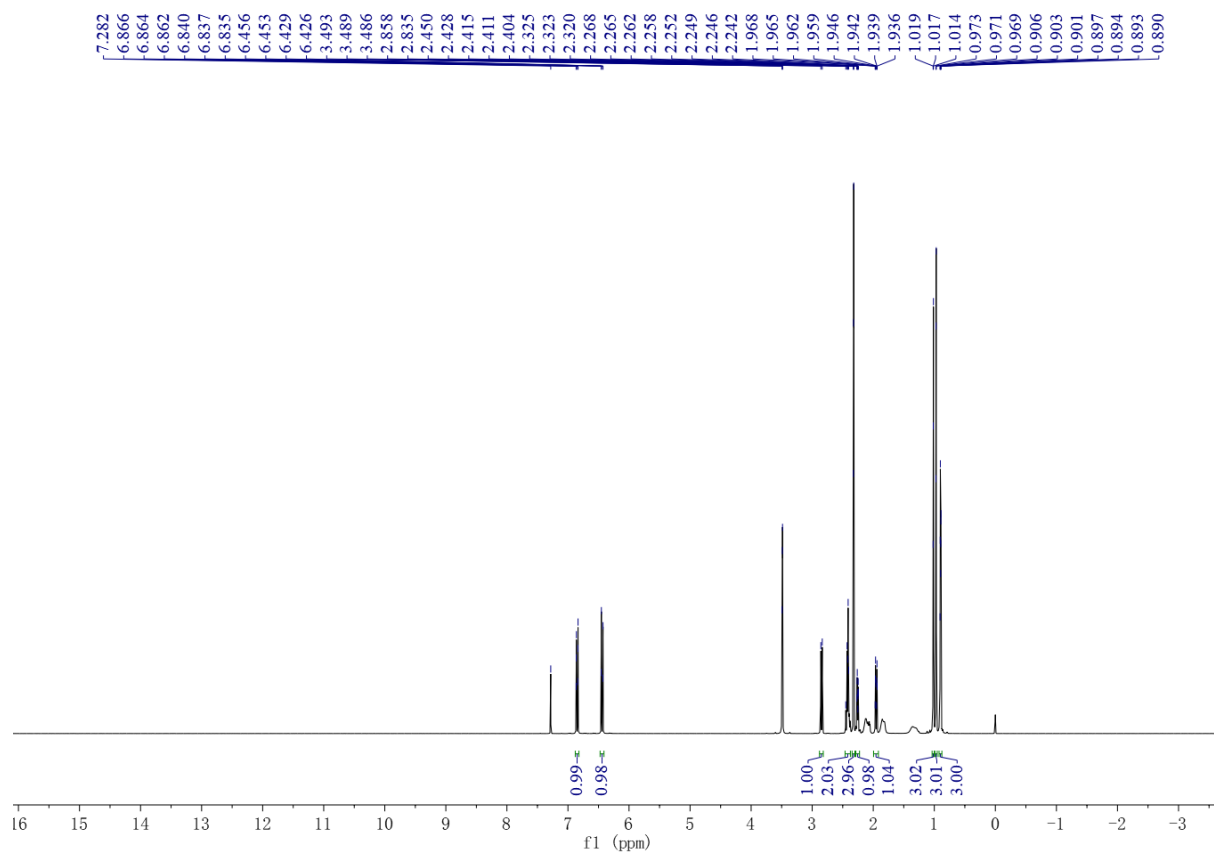


Figure S20: ^1H -NMR (600 MHz, CDCl_3) spectrum of **10** ((*E*)-4-hydroxy3,3,5-trimethyl-4-(3-oxobut-1-en-1-yl)-cyclohexan-1-one)

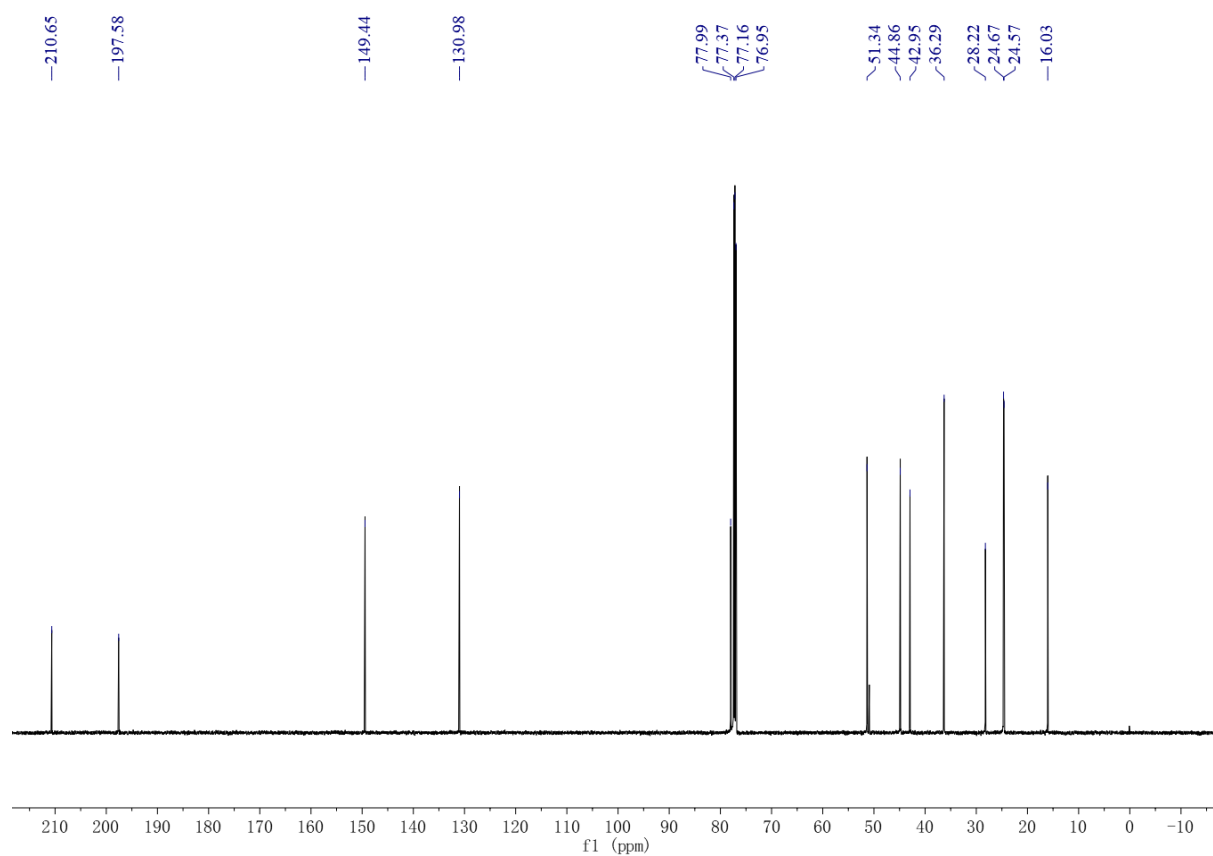


Figure S21: ¹³C-NMR (150 MHz, CDCl₃) spectrum of **10** ((*E*)-4-hydroxy3,3,5-trimethyl-4-(3-oxobut-1-en-1-yl)-cyclohexan-1-one)

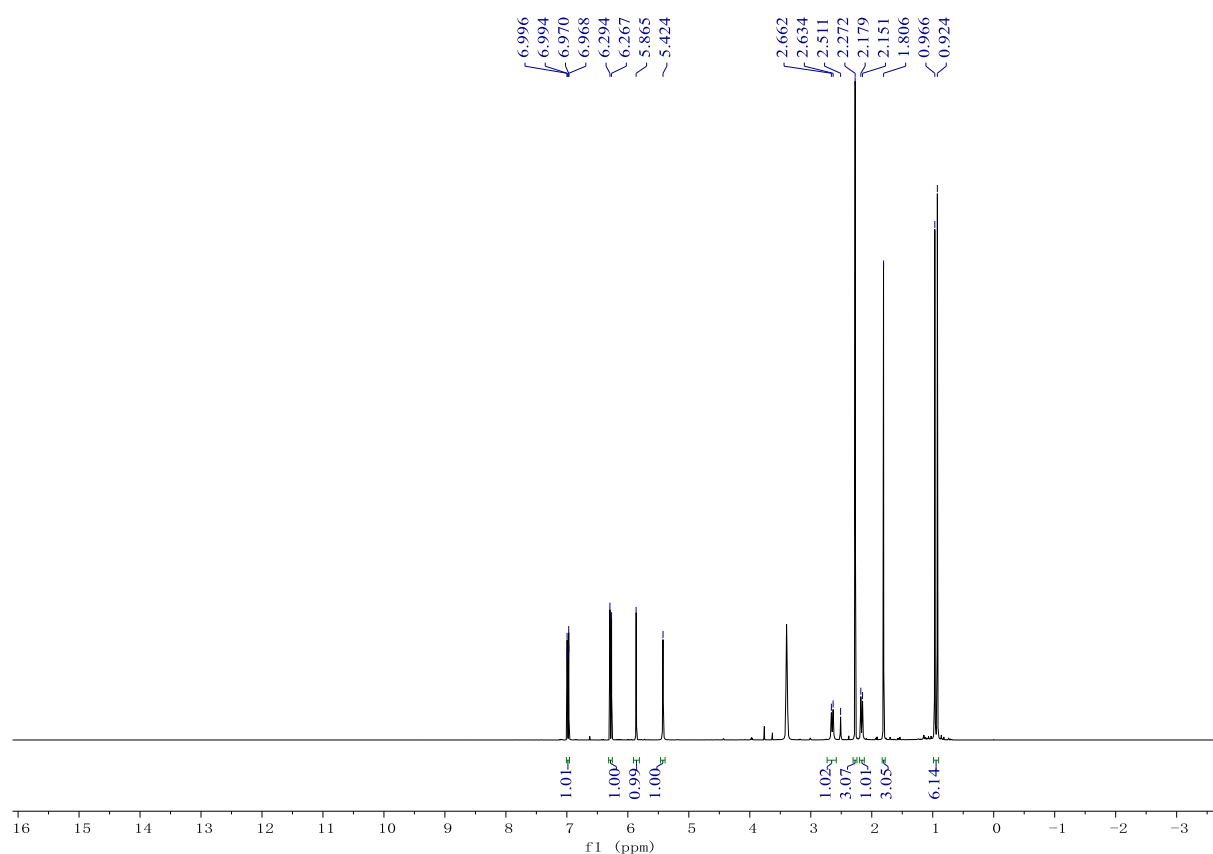


Figure S22: ¹H-NMR (600 MHz, DMSO-d₆) spectrum of **11** (dehydromifoliol)

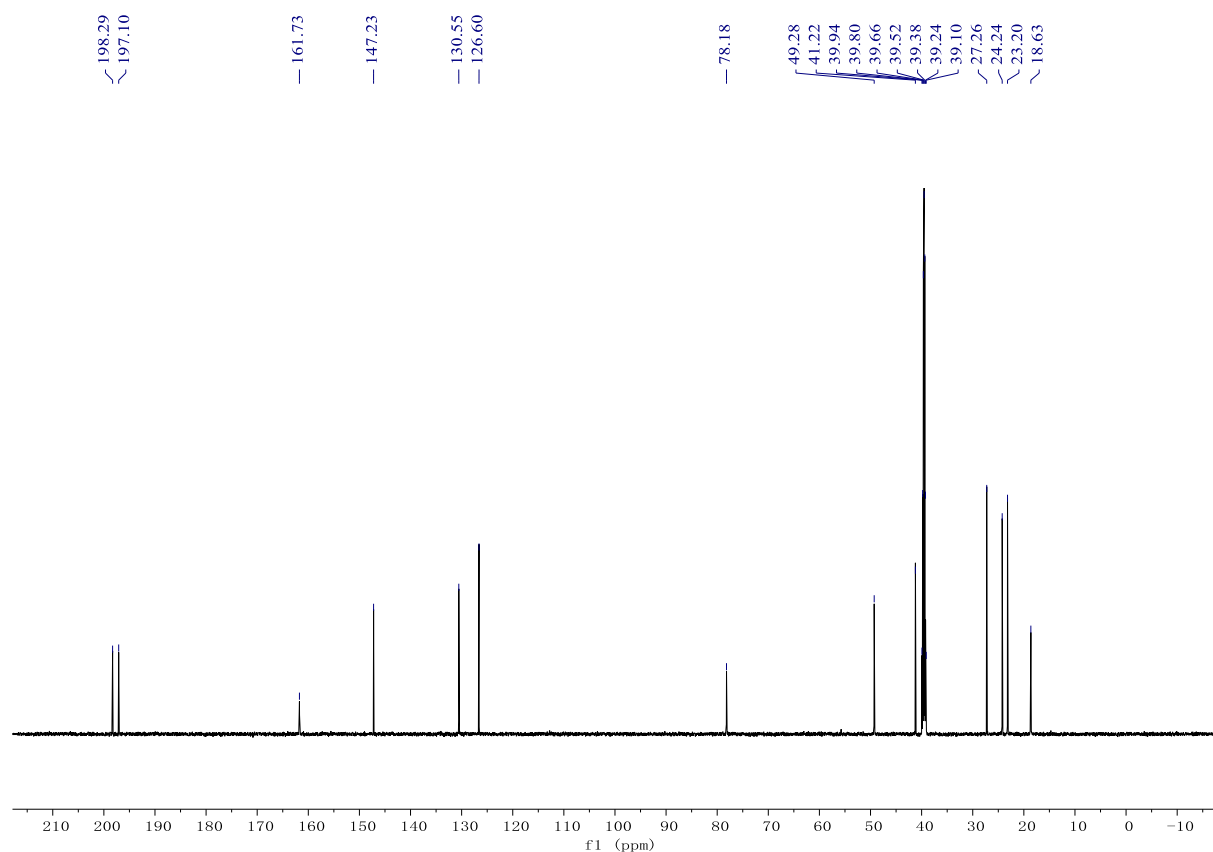


Figure S23: ^{13}C -NMR (150 MHz, DMSO-d_6) spectrum of **11** (dehydrovomifoliol)

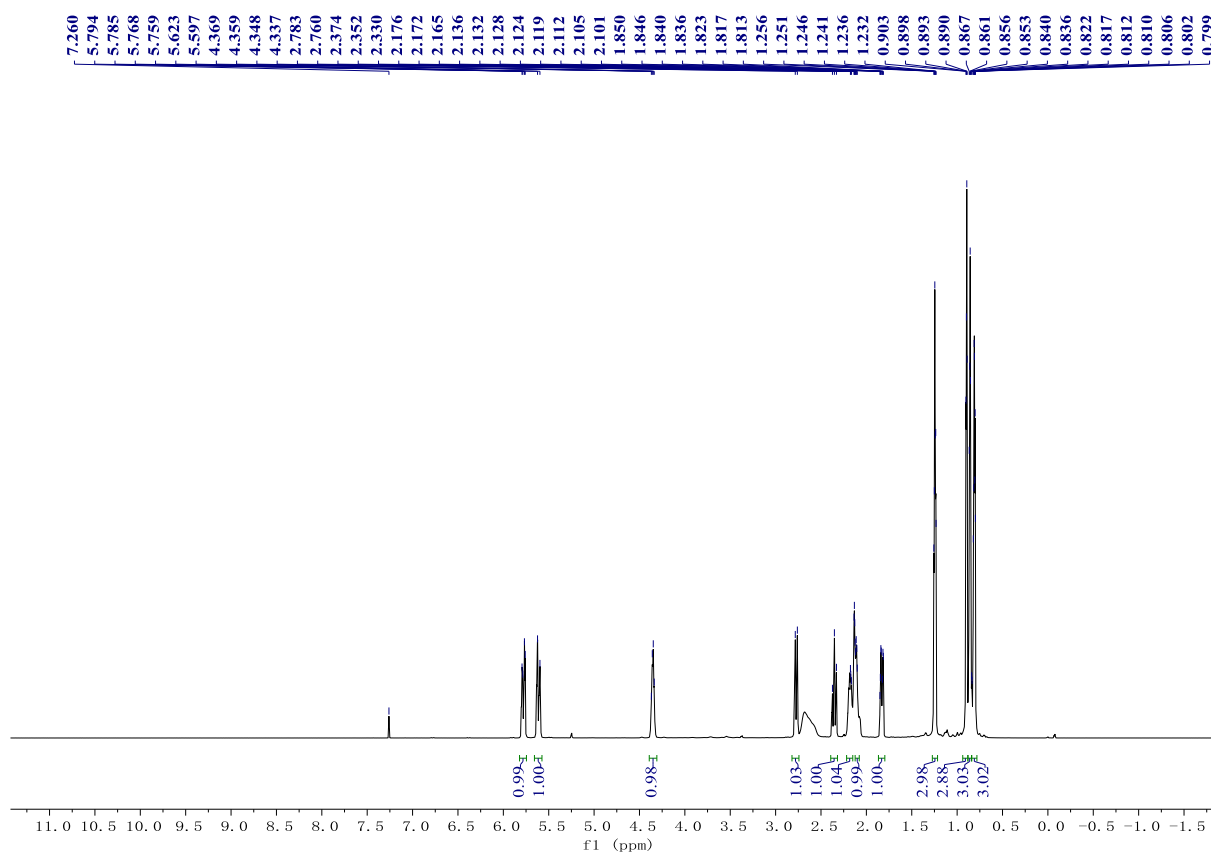


Figure S24: ^1H -NMR (600 MHz, CDCl_3) spectrum of **12** (4,5-dihydroblumenol A)

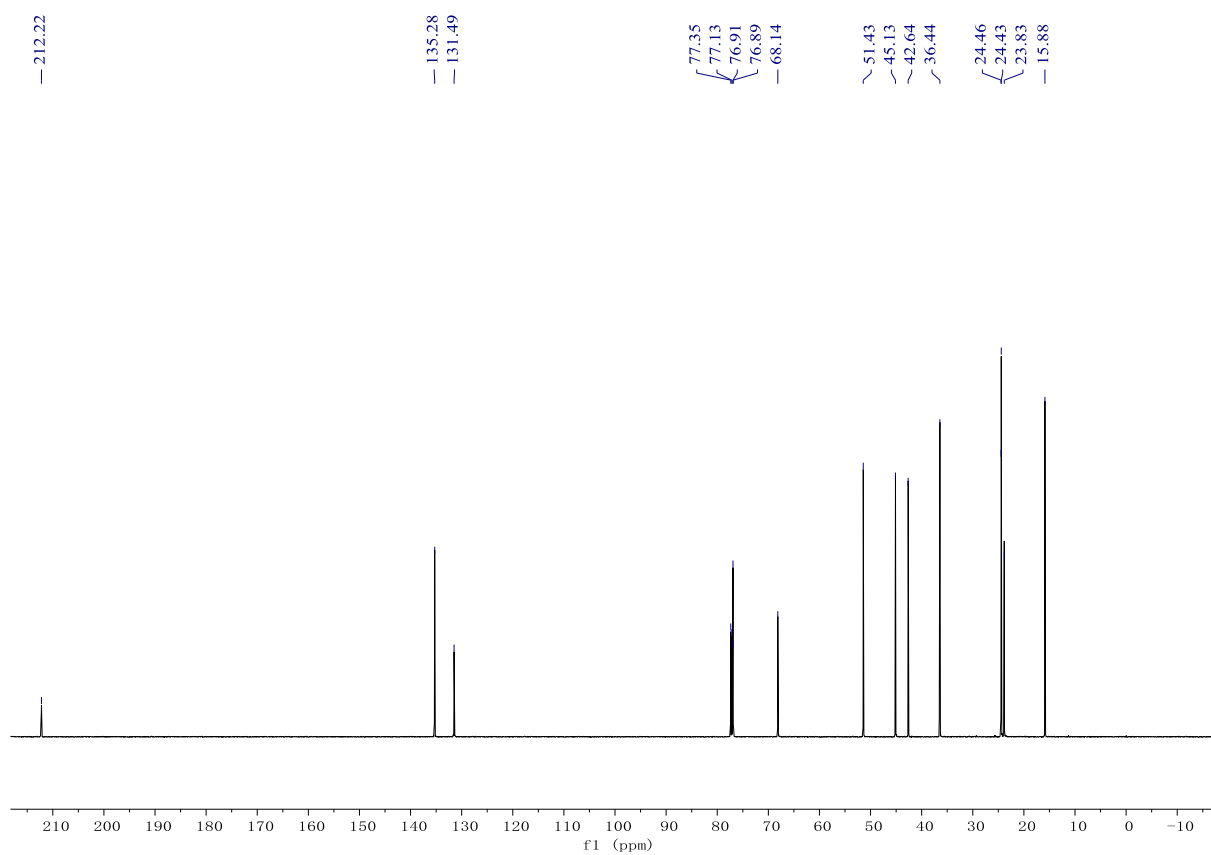


Figure S25: ^{13}C -NMR (150 MHz, CDCl_3) spectrum of **12** (4,5-dihydroblumenol A)

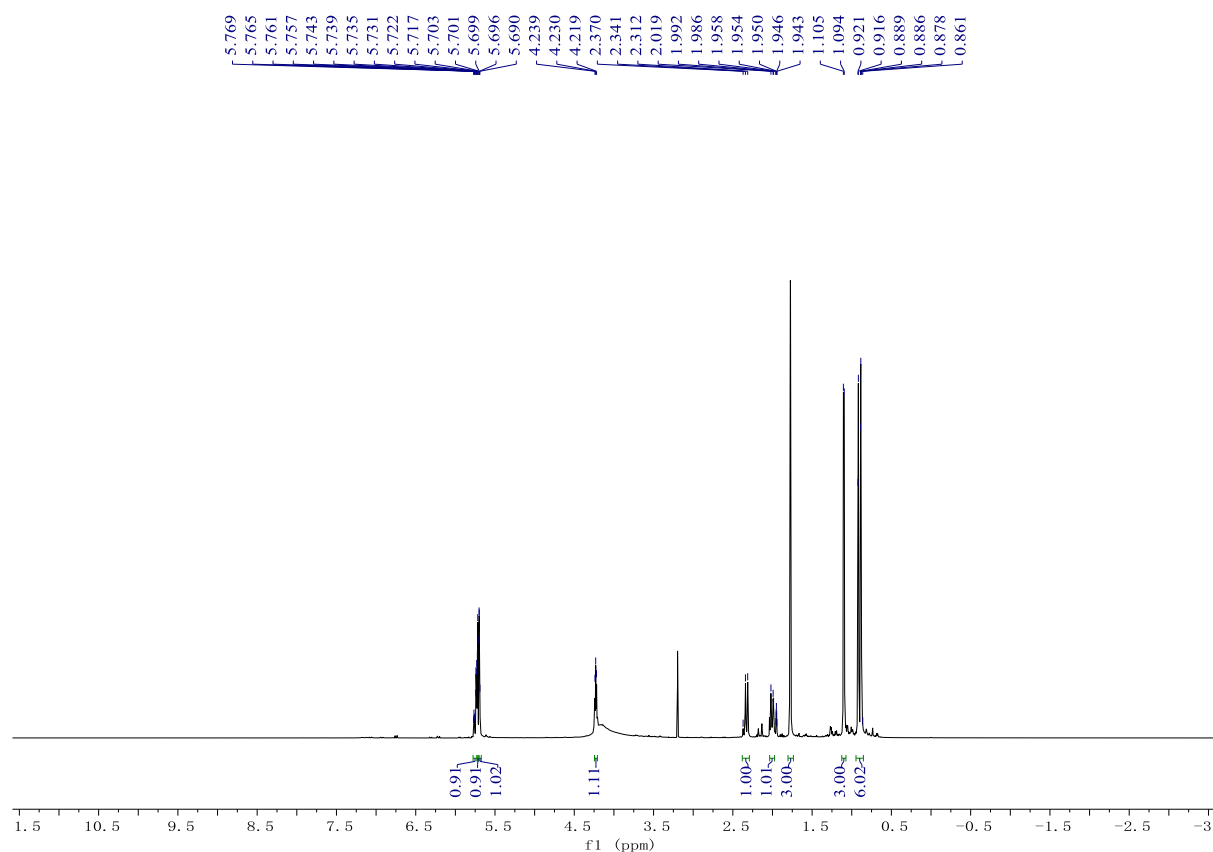


Figure S26: $^1\text{H-NMR}$ (600 MHz, CD_3COCD_3) spectrum of **13** (blumenol A)

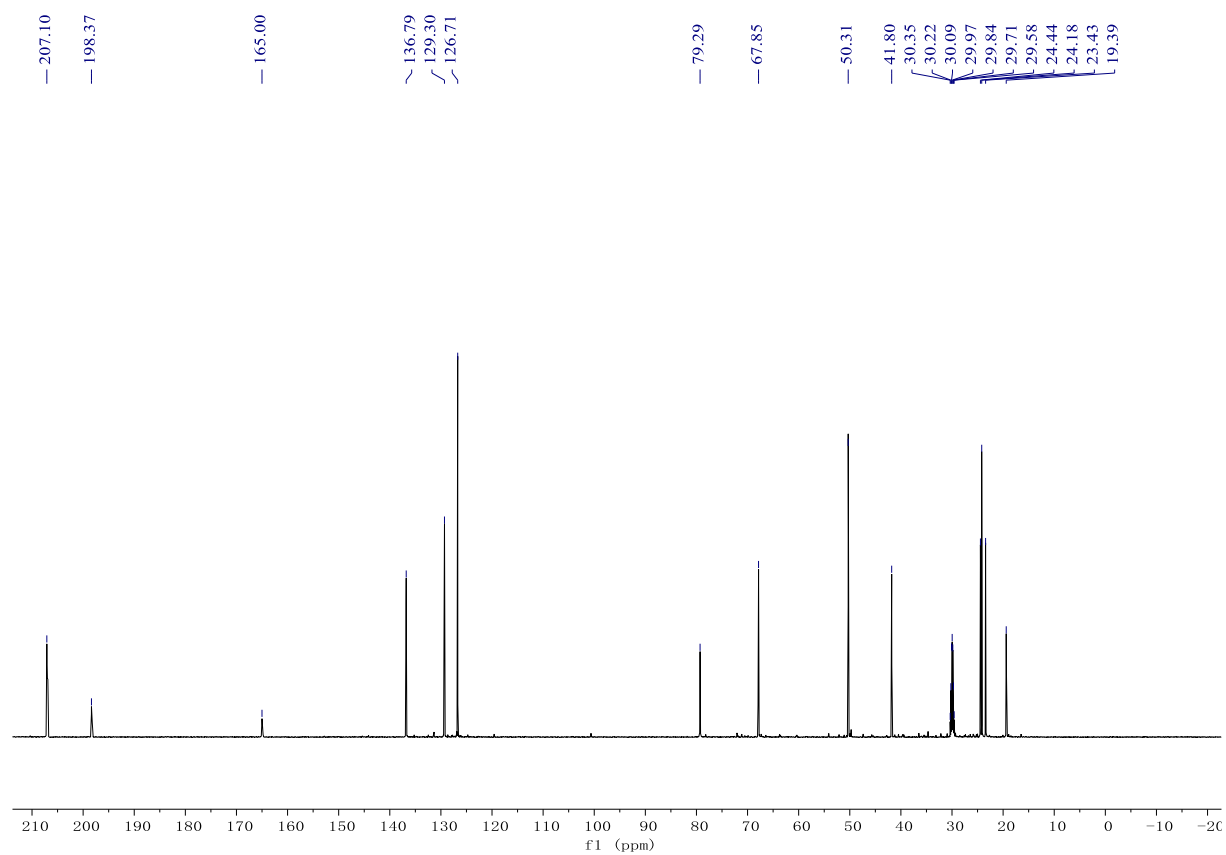


Figure S27: ^{13}C -NMR (150 MHz, CD_3COCD_3) spectrum of **13** (blumenol A)

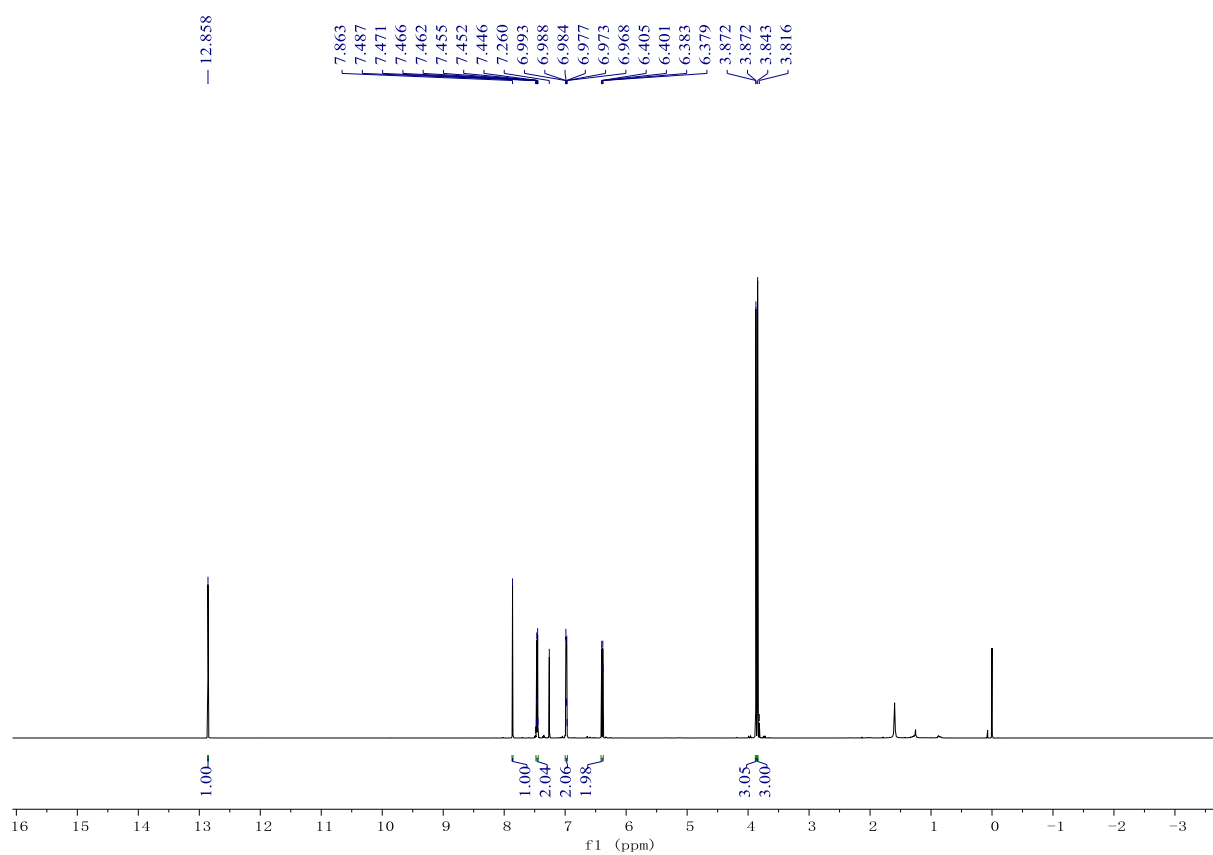


Figure S28: ^1H -NMR (600 MHz, CDCl_3) spectrum of **14** (5-hydroxy-4',7-dimethoxyisoflavone)

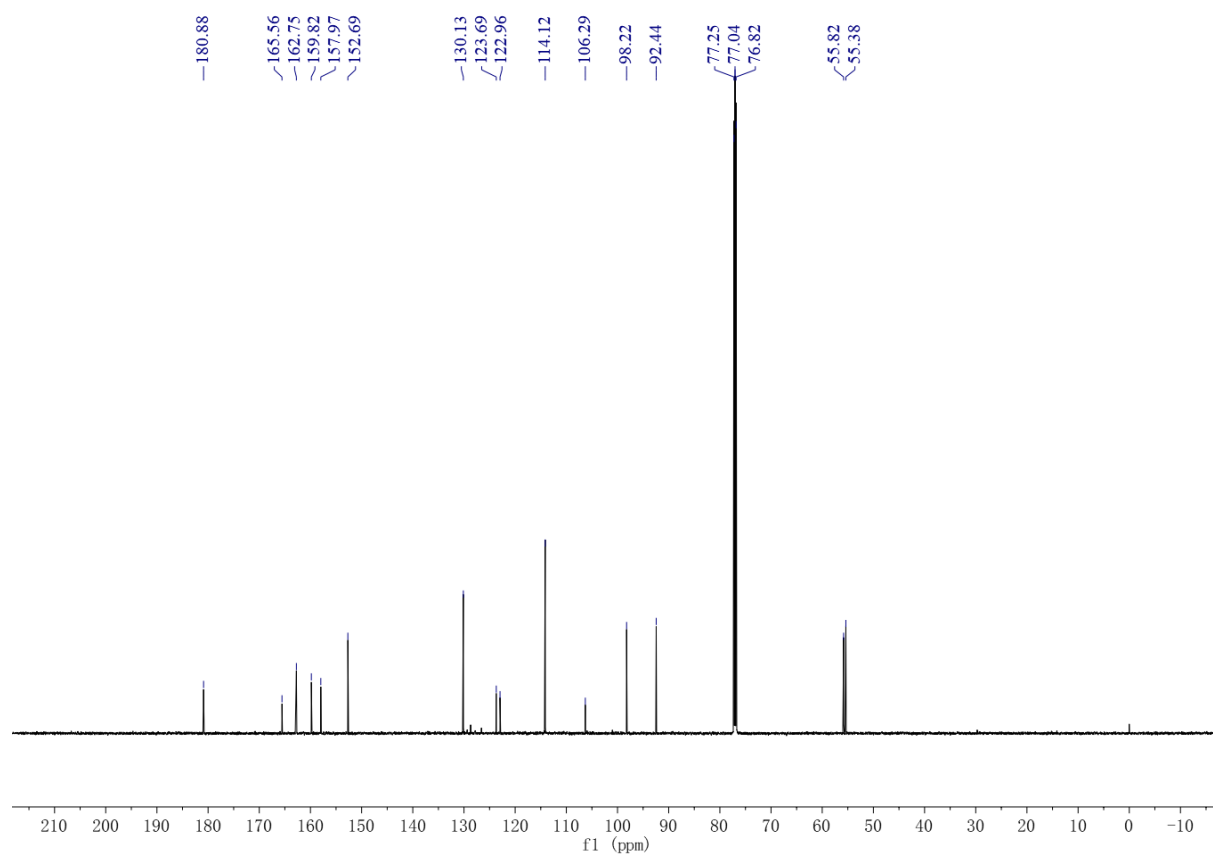


Figure S29: ^{13}C -NMR (150 MHz, CDCl_3) spectrum of **14** (5-hydroxy-4',7-dimethoxyisoflavone)

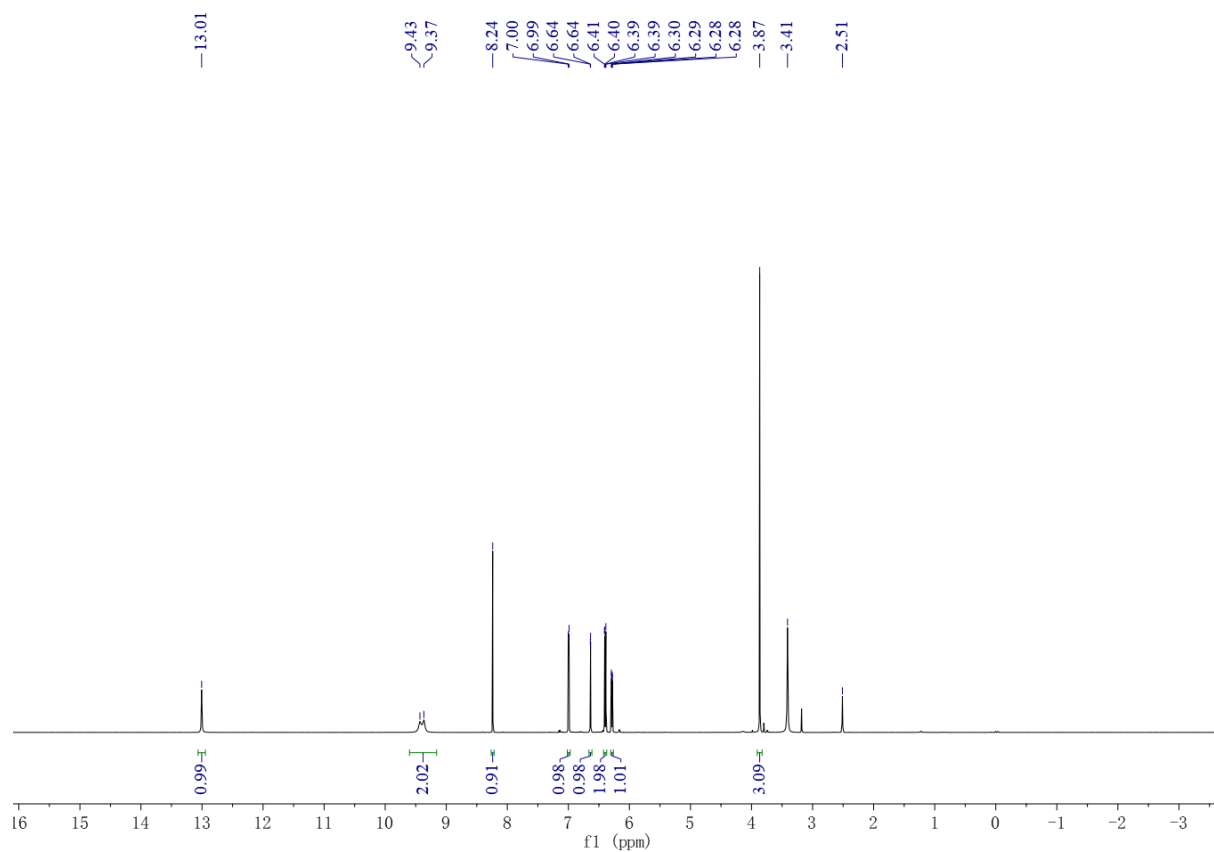


Figure S30: ^1H -NMR (600 MHz, DMSO-d_6) spectrum of **15** (cajanin)

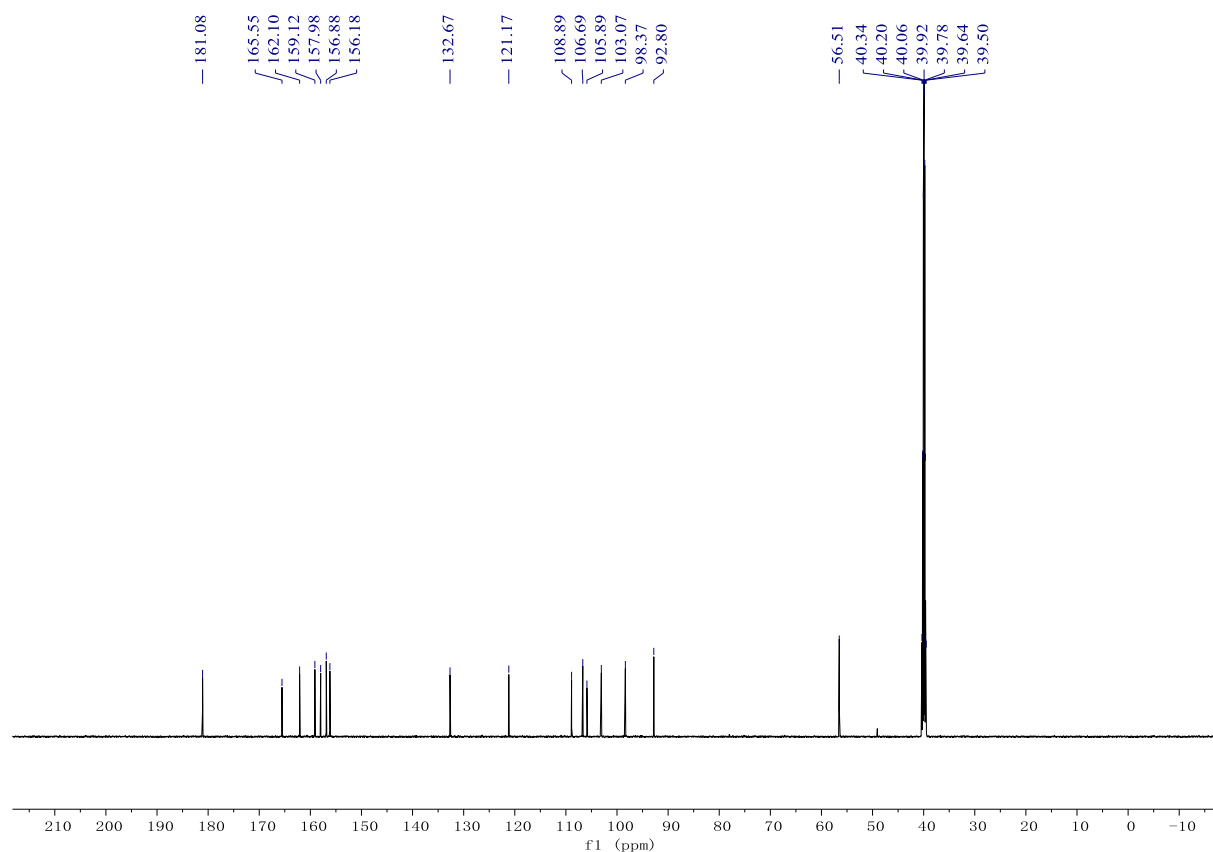


Figure S31: ^{13}C -NMR (150 MHz, DMSO-d_6) spectrum of **15** (cajanin)

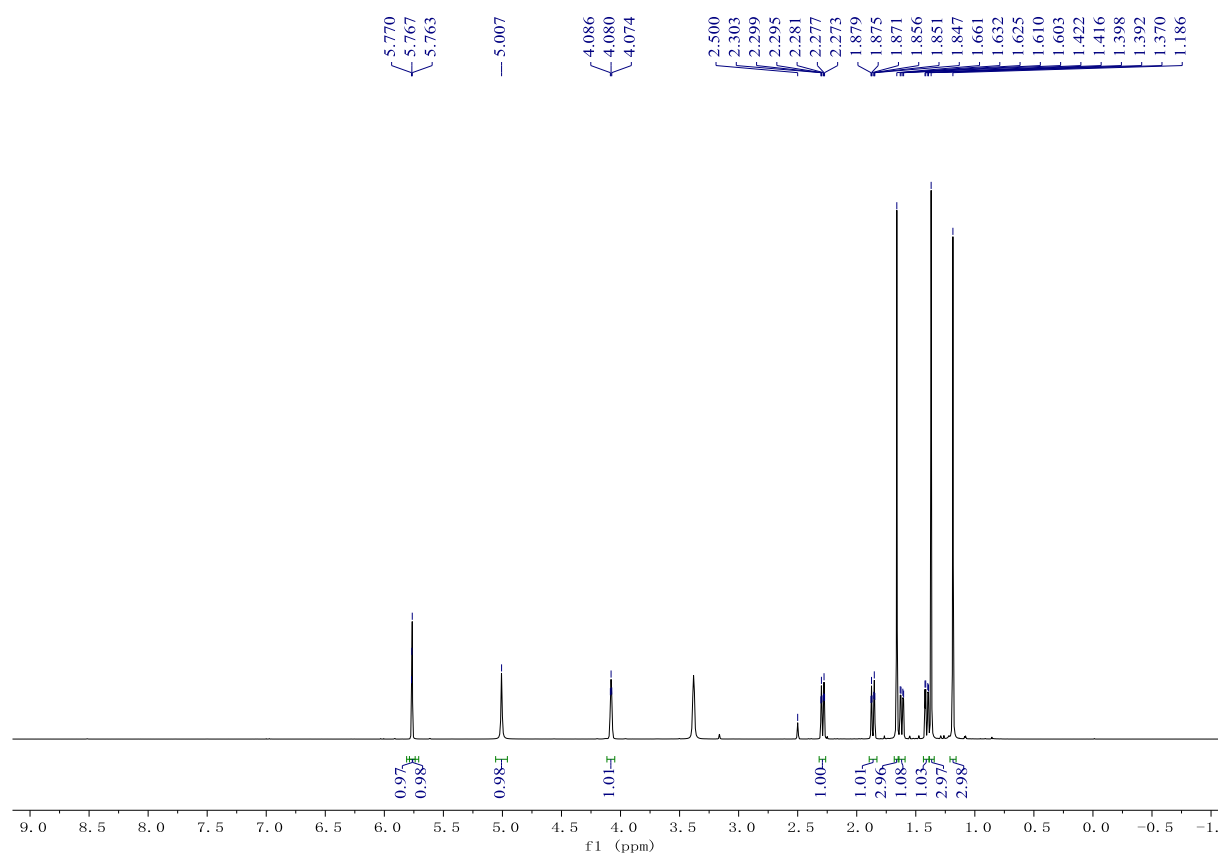


Figure S32: ^1H -NMR (600 MHz, DMSO-d_6) spectrum of **16** (loliolide)

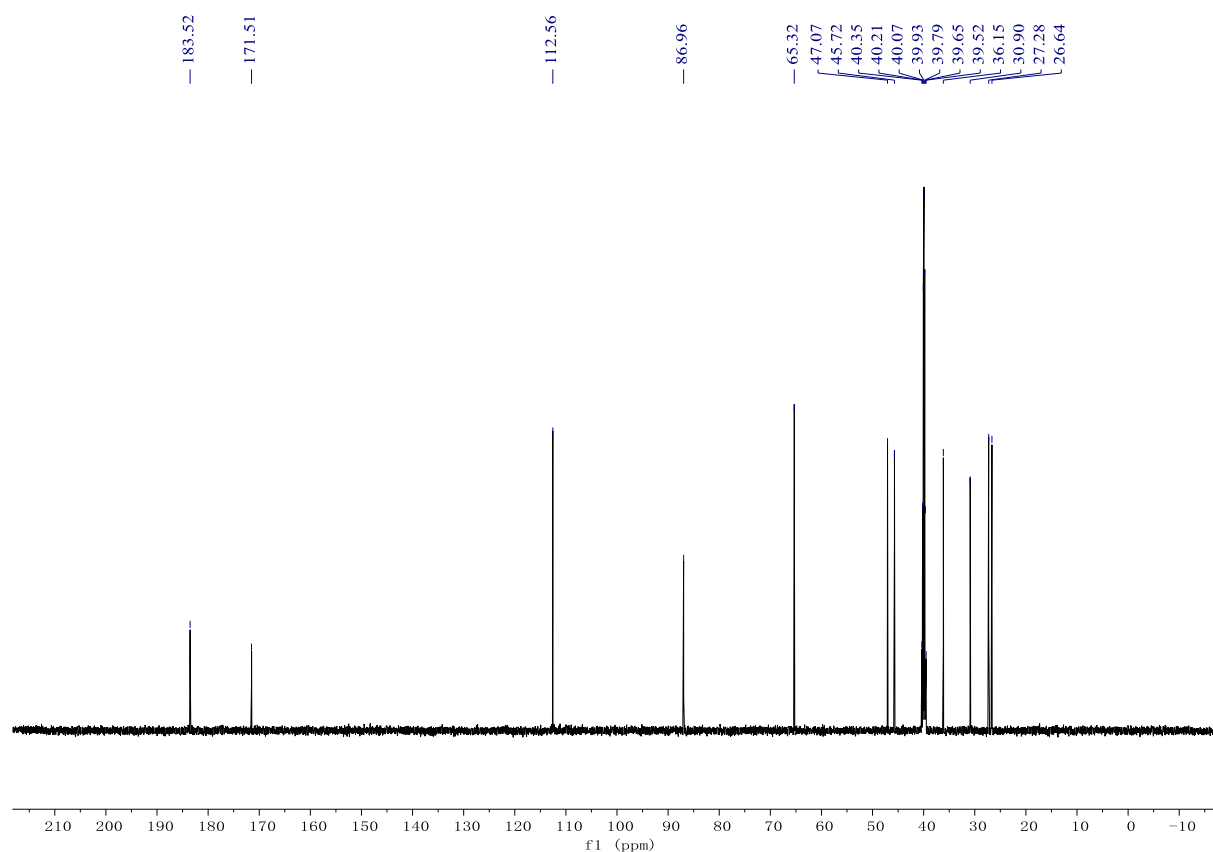


Figure S33: ^{13}C -NMR (150 MHz, DMSO-d_6) spectrum of **16** (loliolide)

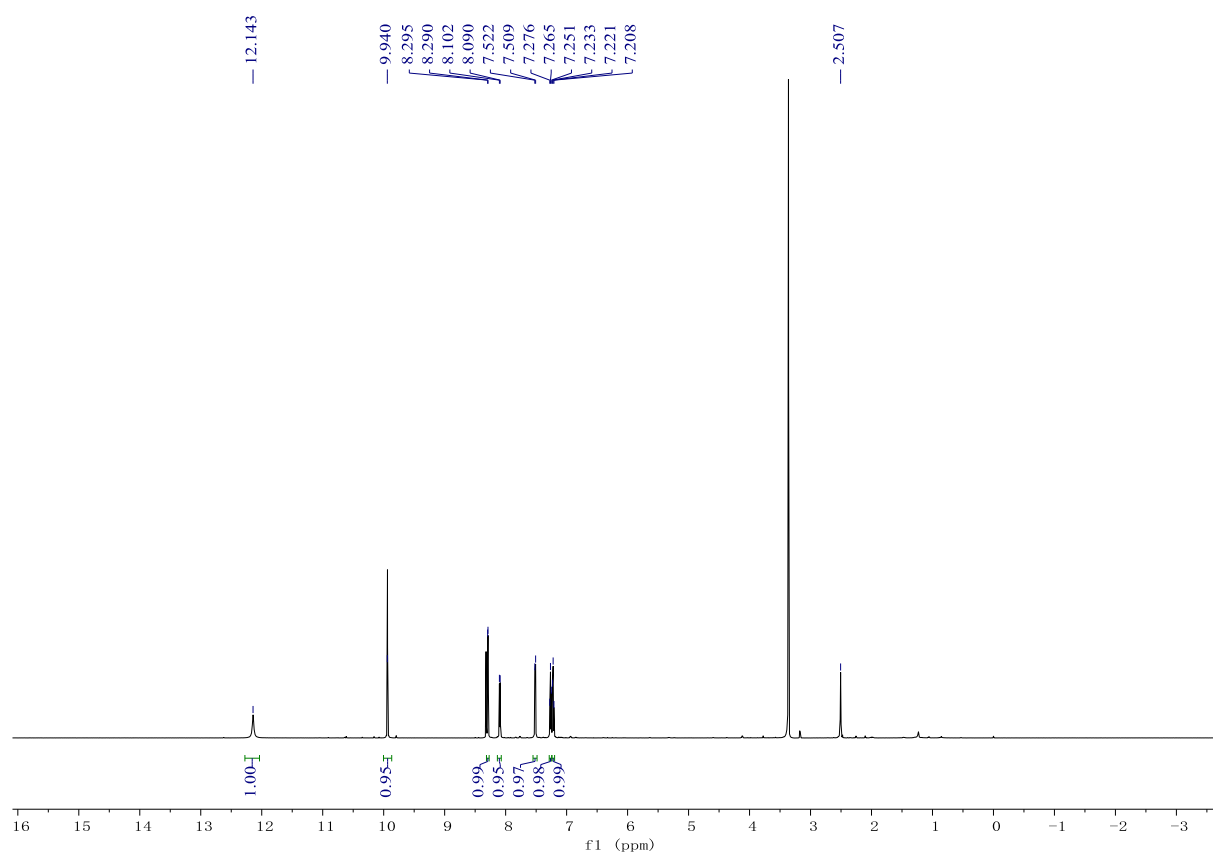


Figure S34: ¹H-NMR (600 MHz, DMSO-d₆) spectrum of **17** (indole-3-carboxaldehyde)

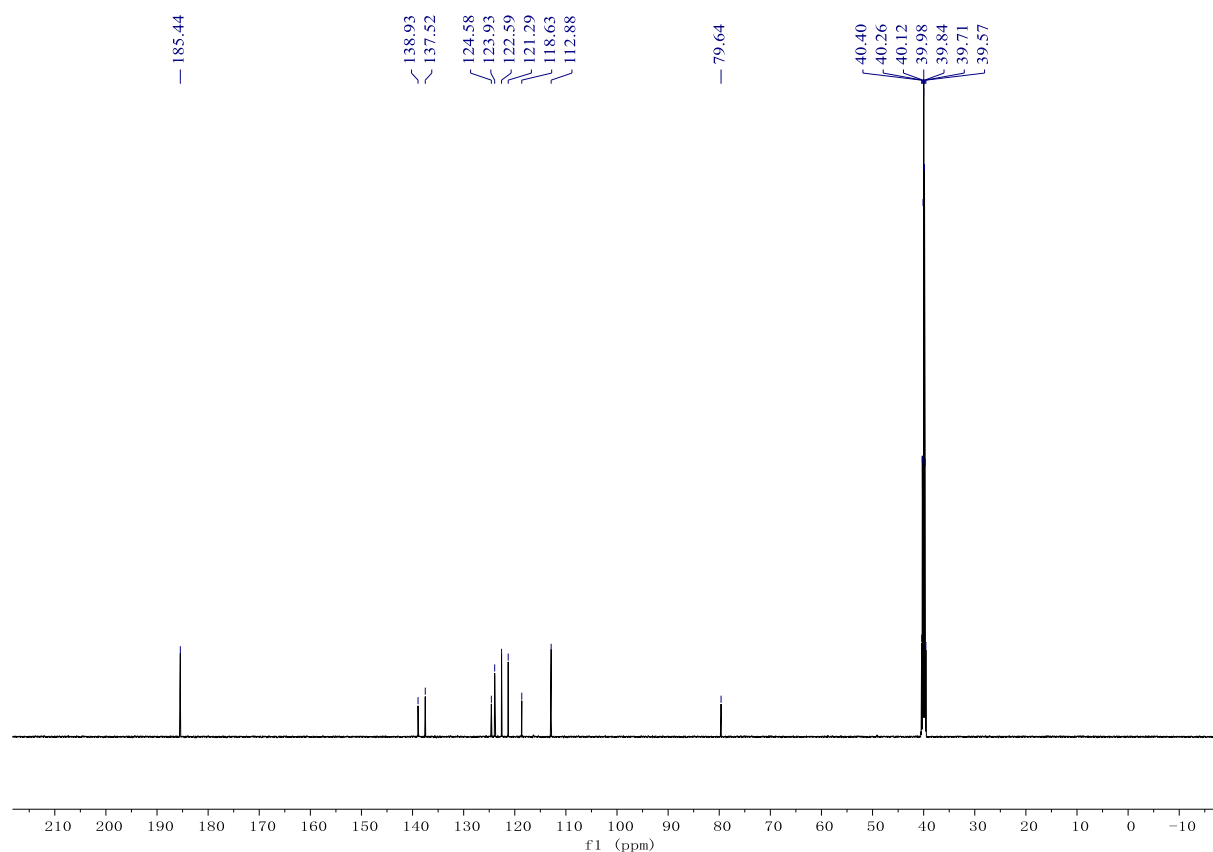


Figure S35: ¹³C-NMR (150 MHz, DMSO-d₆) spectrum of **17** (indole-3-carboxaldehyde)

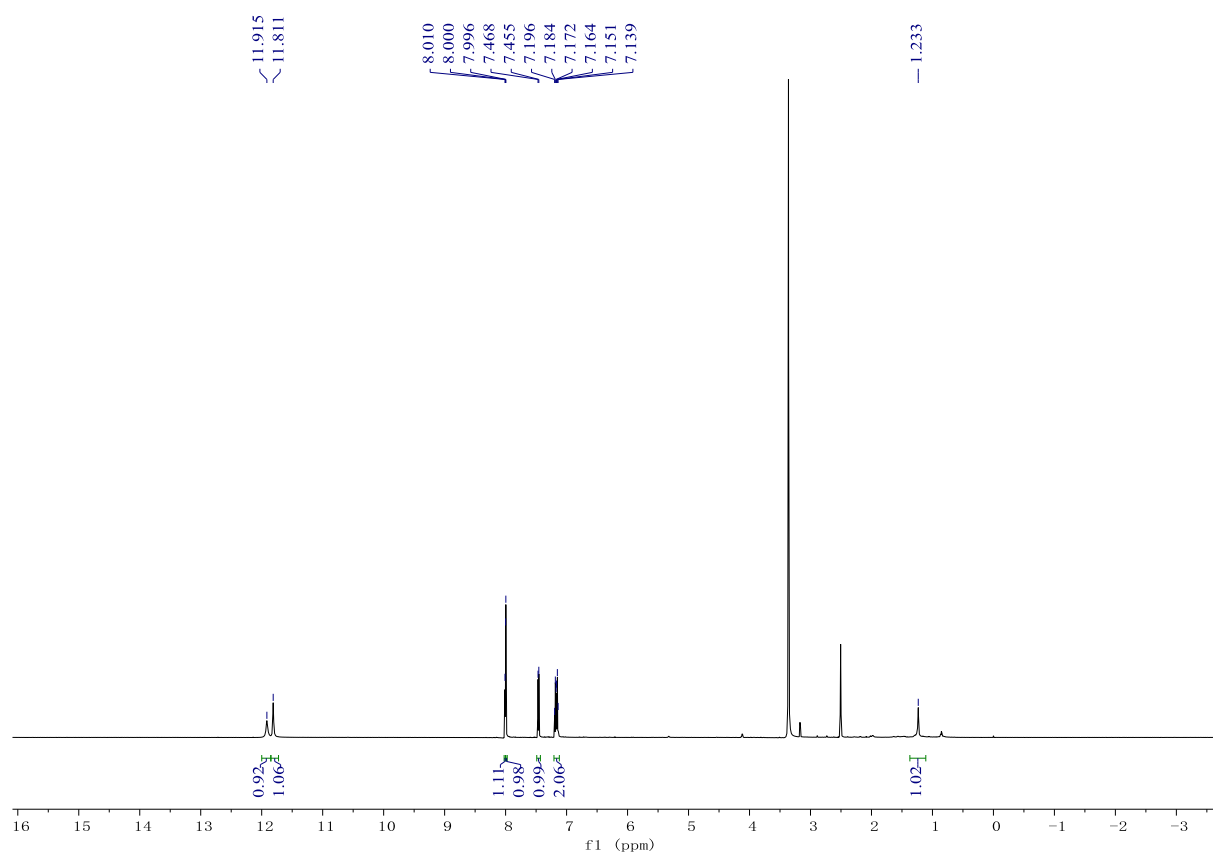


Figure S36: ^1H -NMR (600 MHz, DMSO-d_6) spectrum of **18** (1H-indole-3-carboxylic acid)

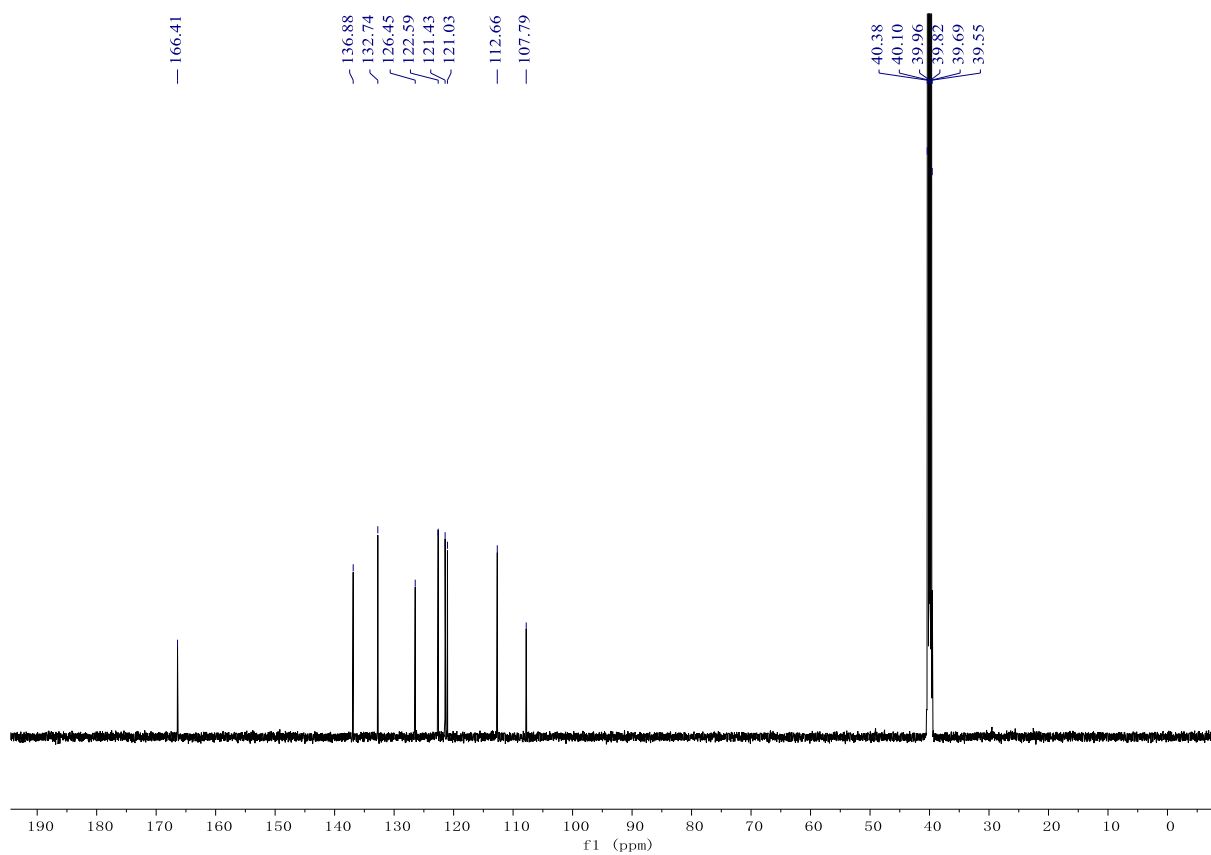


Figure S37: ^{13}C -NMR (150 MHz, DMSO-d_6) spectrum of **18** (1H-indole-3-carboxylic acid)

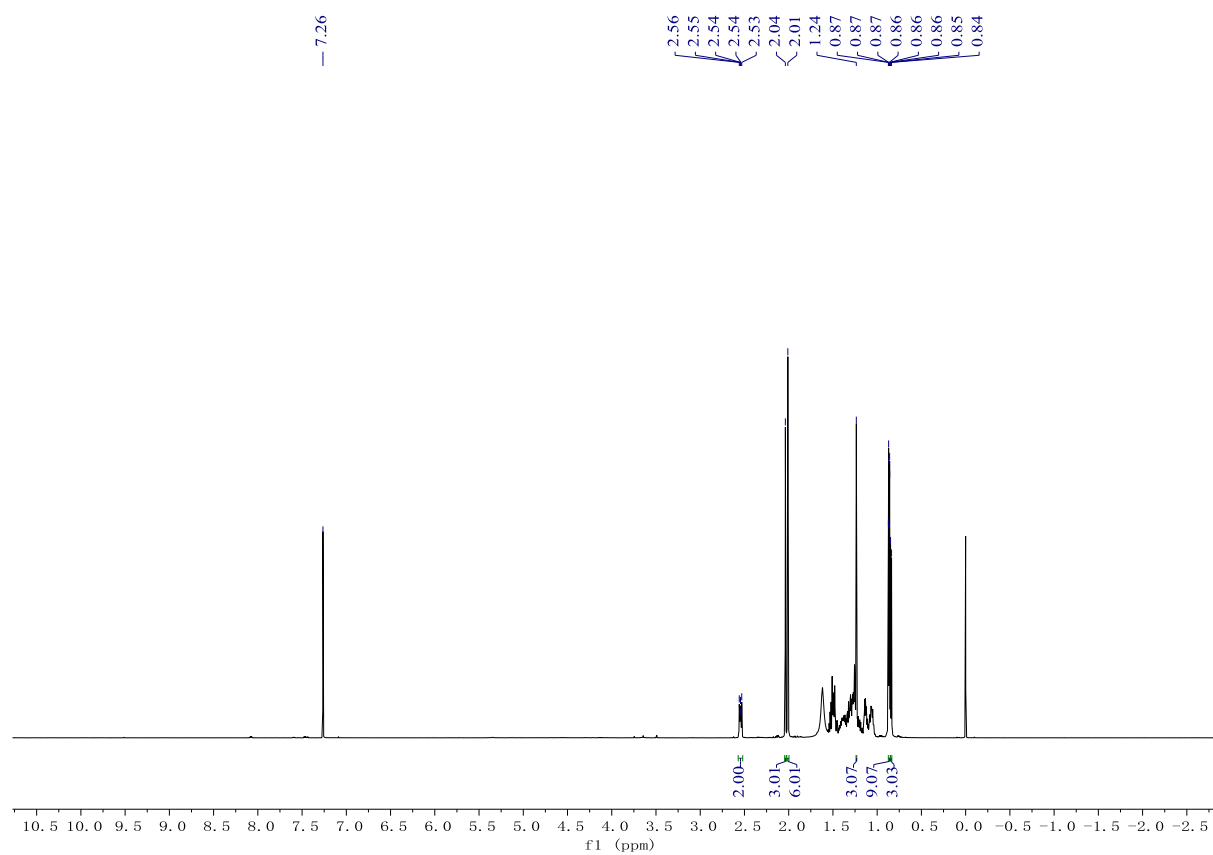


Figure S38: ¹H-NMR (600 MHz, CDCl₃) spectrum of **19** (vitamin E quinone)

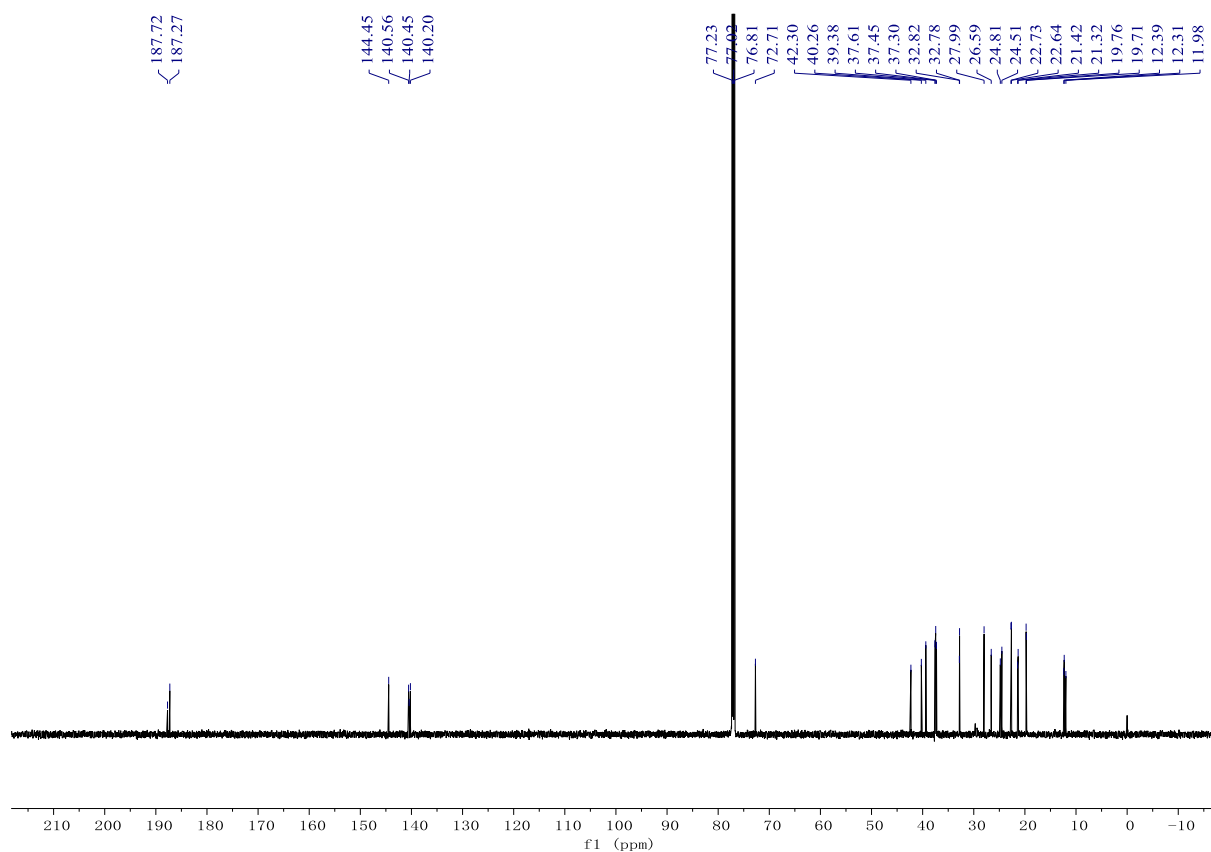


Figure S39: ^{13}C -NMR (150 MHz, CDCl_3) spectrum of **19** (vitamin E quinone)

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