Supporting Information

The AIE-Active Flavonoids in Orange Peel for Photocatalytic Oxidation Reactions

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Table of Contents

I. General Information

All chromatographic analyses for both the extracts and standards were acquired using an Agilent 1260 Infinity II HPLC system equipped with a Sunfire™ C18 column maintained at 25 °C. The selected analytical method was as follows: the mobile phase consisted of acetonitrile (A) and water (B) employing a gradient elution program: 35% A (0-15 min), 55% A (15-20 min), 100% A (20-35 min). The injection volume was 20 μL, the flow rate was 1.0 mL·min $^{-1}$, and the detection wavelength was set at 280 nm. Each standard compound (tangeretin, nobiletin, sinensetin;1.0 mg each) was dissolved in 1.0 mL of methanol to form stock solutions, which were filtered through a 0.22 μm micropore filter prior to analysis.

Magnetic resonance spectra (1H NMR and 13C NMR) were recorded on Bruker ACF 400 MHz or 600 MHz with CDCl3 as the solvent, and chemical shifts were internally referenced to TMS and residual portion solvent signals (note: TMS referenced at 0.00 ppm; CDCl3 referenced at 7.26 and 77.0 ppm, respectively). Data are shown as follows: chemical shifts (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sext = sextet, h = heptet, m = multiplet, dd = doublet of doublets, dt = doublet of triplets, br = broad), coupling constant (J Hz). Commercially available reagents and solvents were purchased from Adamas and Aladdin, and used as received unless otherwise specified. High-resolution mass spectra (HRMS) were obtained using a Q Exactive Plus (Thermo Fisher Scientific), which utilizes advanced quadrupole technology (AQT) and MassWorks software for matching. The resulting data is in terms of mass-to-charge ratio (m/z) units.

II. HPLC Chromatogram of Three Flavonoid Standards

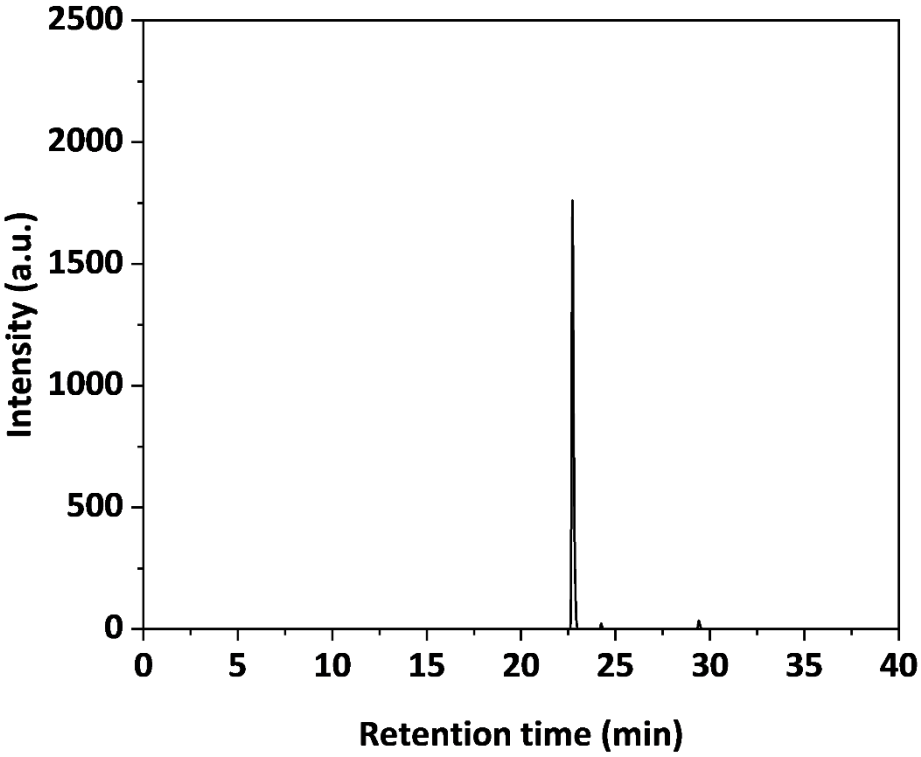


Fig.S1 HPLC chromatogram of Tangeretin.

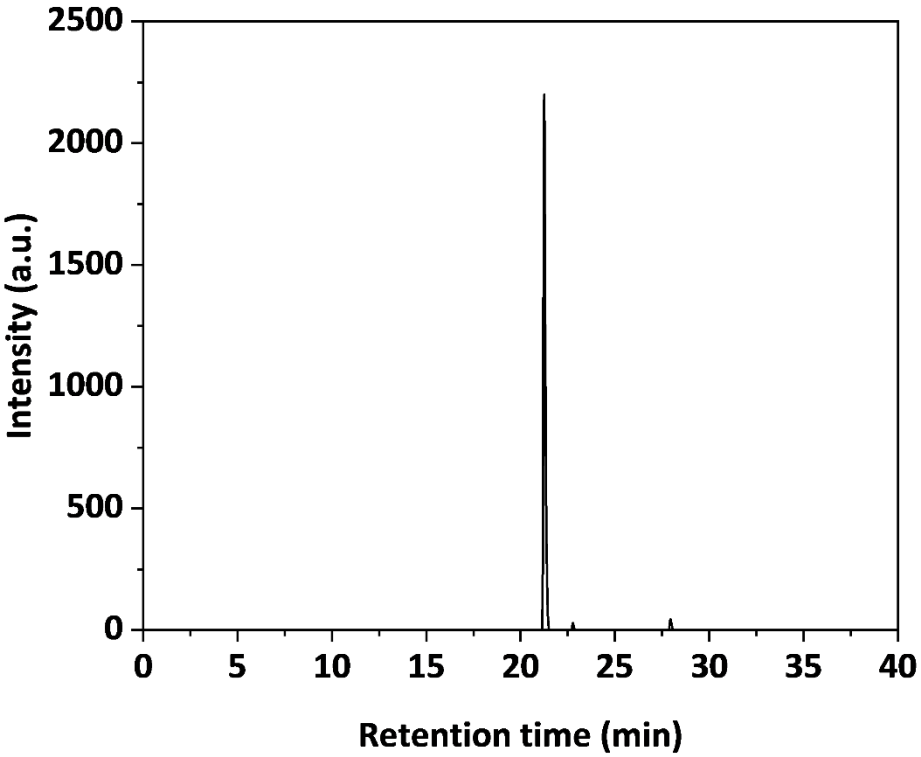


Fig.S2 HPLC chromatogram of Nobiletin.

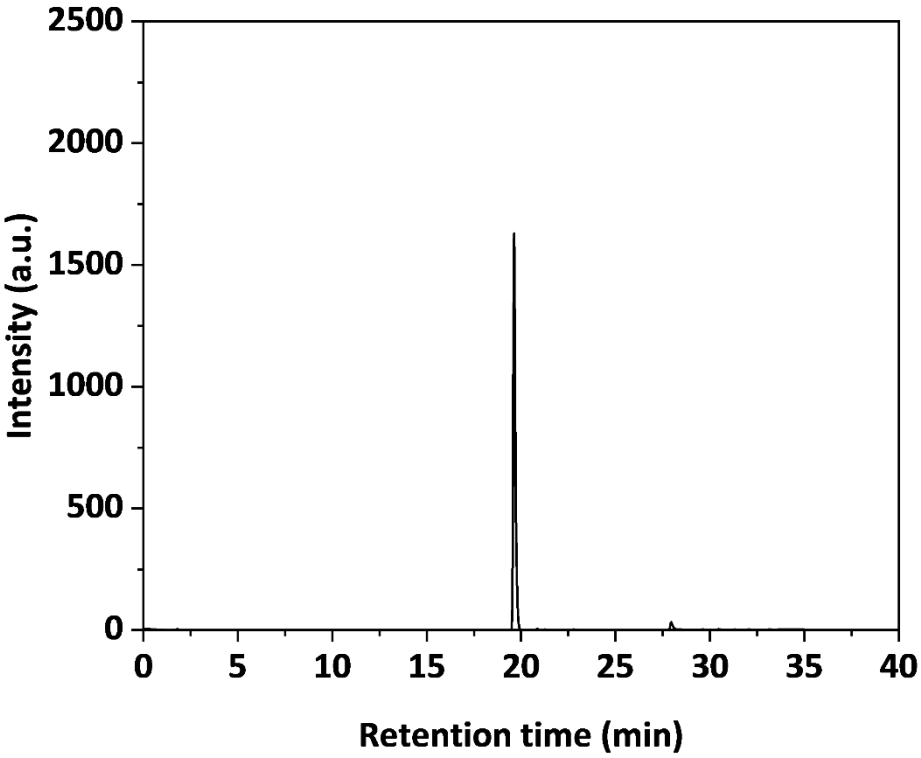


Fig.S3 HPLC chromatogram of Sinensetin.

Table S1 Comparison of the retention time of cellulite extract and three reference materials.

Extracts	RT (s)	RM	RT (s)
1	19.79	Sinensetin	19.62
2	21.39	Nobiletin	21.24
3	22.94	Tangeretin	22.71

III. High-Resolution Liquid Chromatography Mass Spectrometry

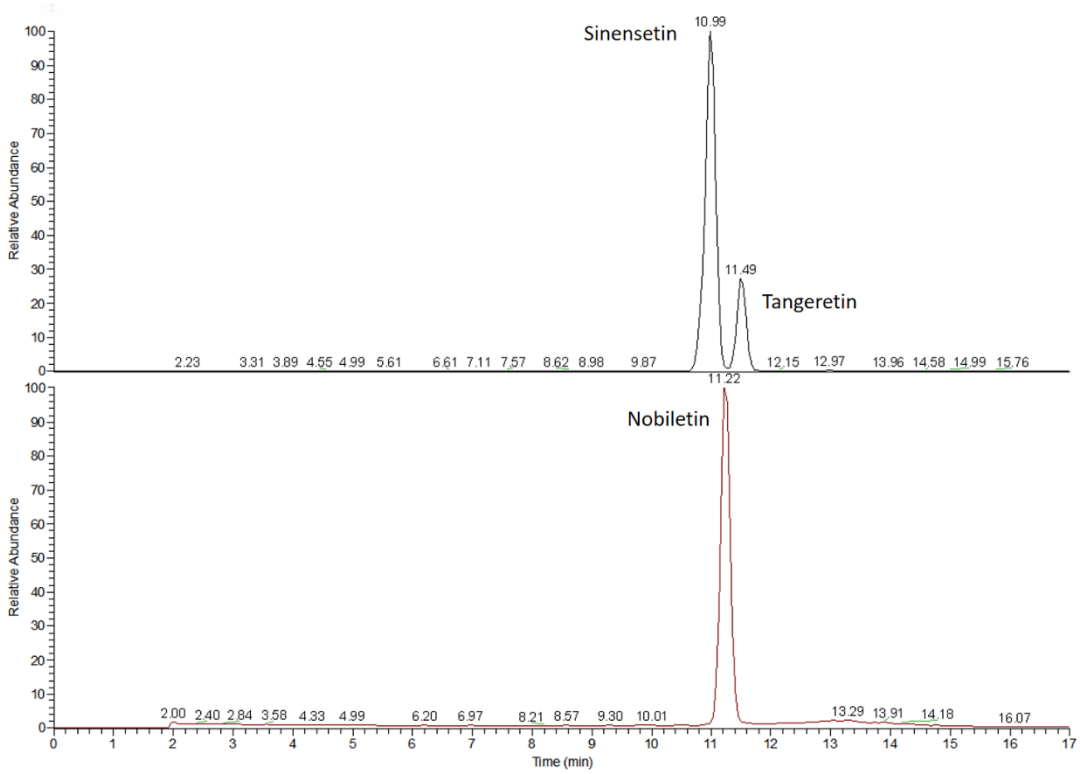


Fig.S4 HPLC chromatogram of orange peel extract.

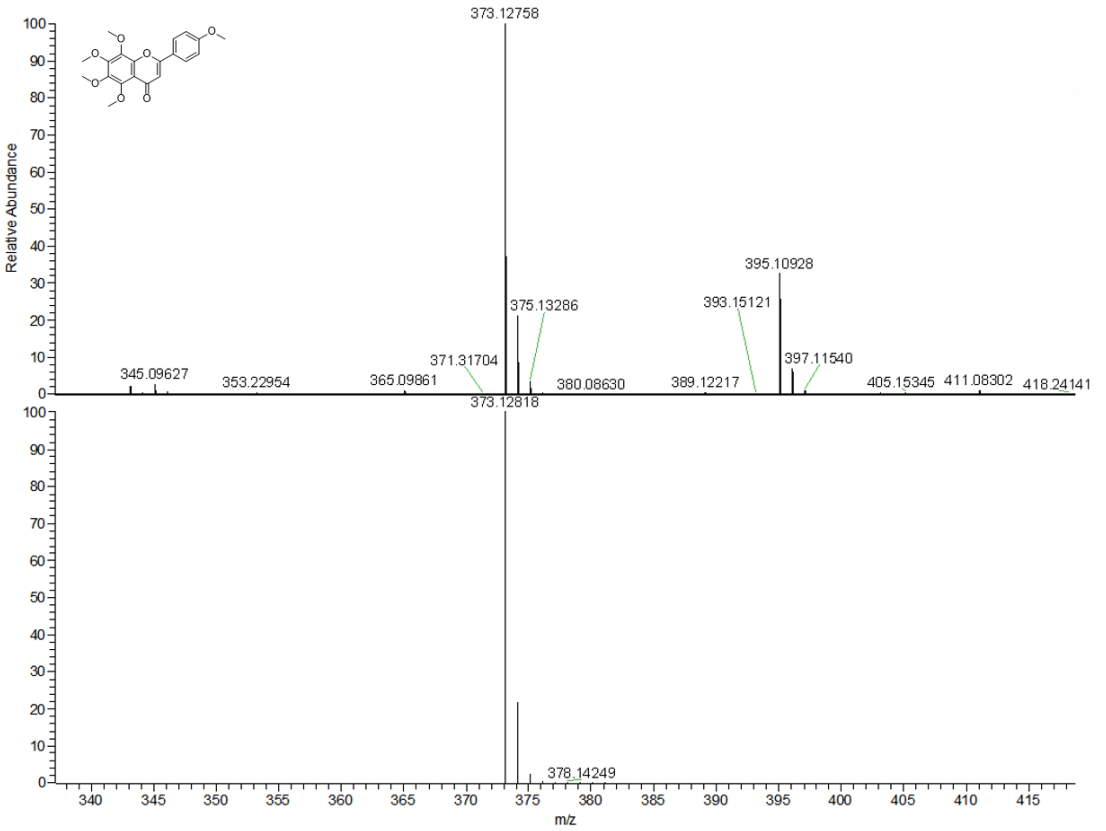


Fig.S5 Mass spectrum of Tangeretin.

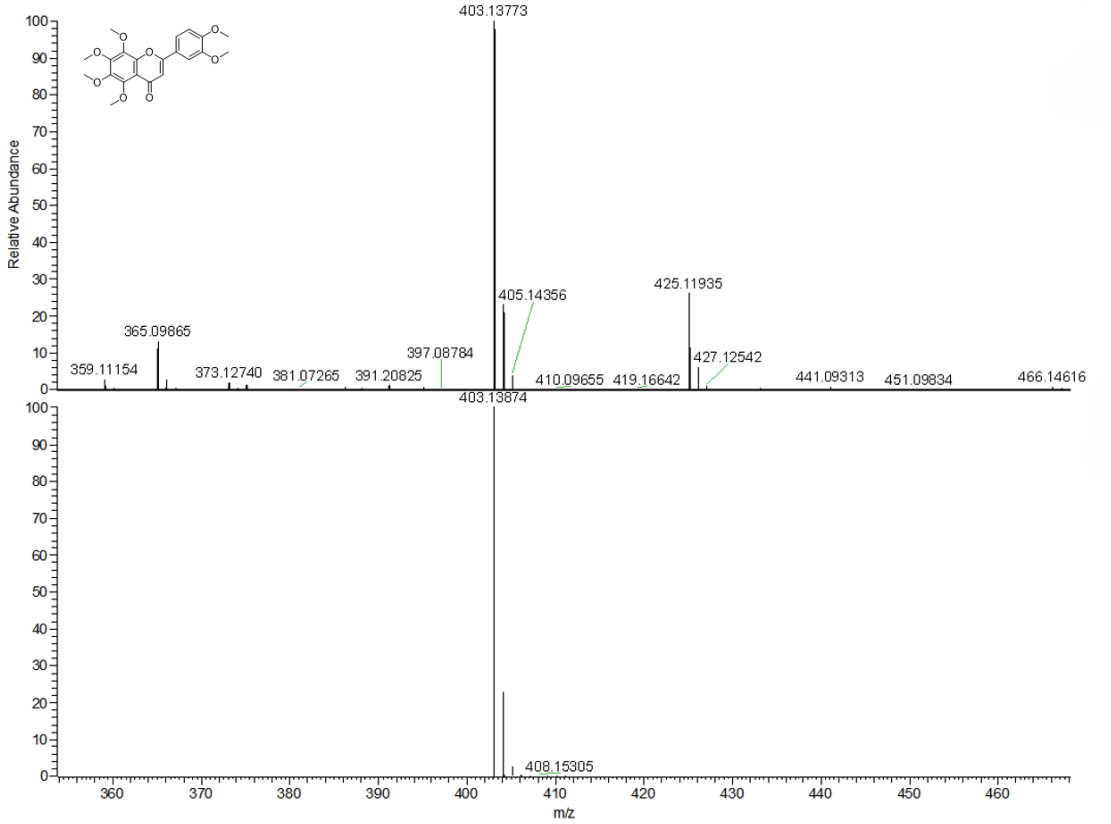


Fig.S6 Mass spectrum of Nobiletin.

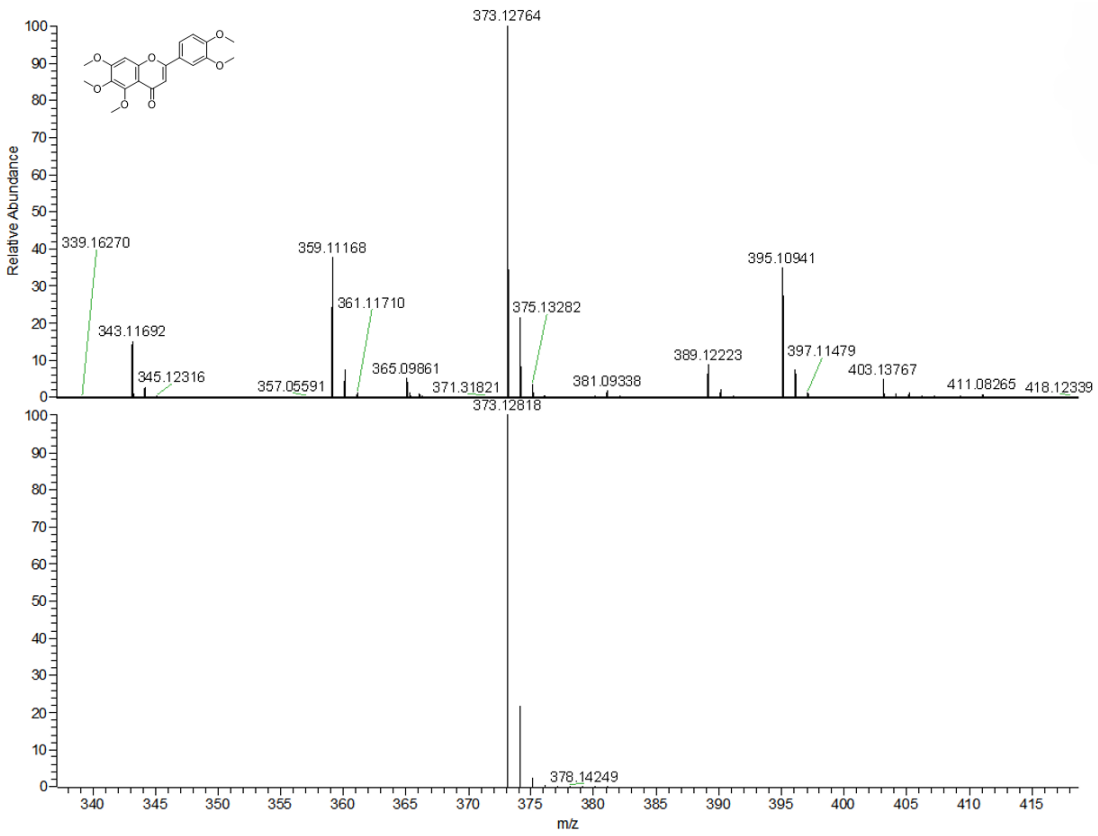


Fig.S7 Mass spectrum of Sinensetin.

Ⅳ. Verification of AIE Properties

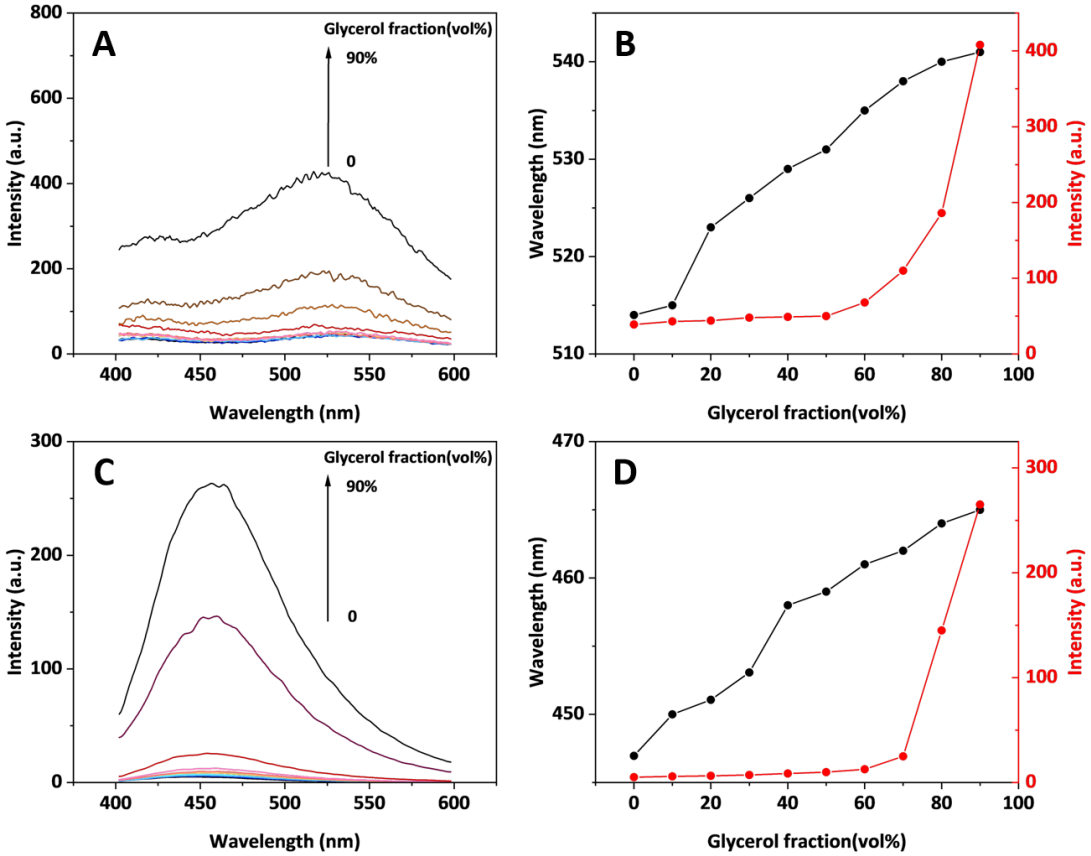


Fig.S8 Experiment on viscosity changes of Tangeretin(A,B), Nobiletin(C,D).

V.Cyclic Voltammetry Diagram

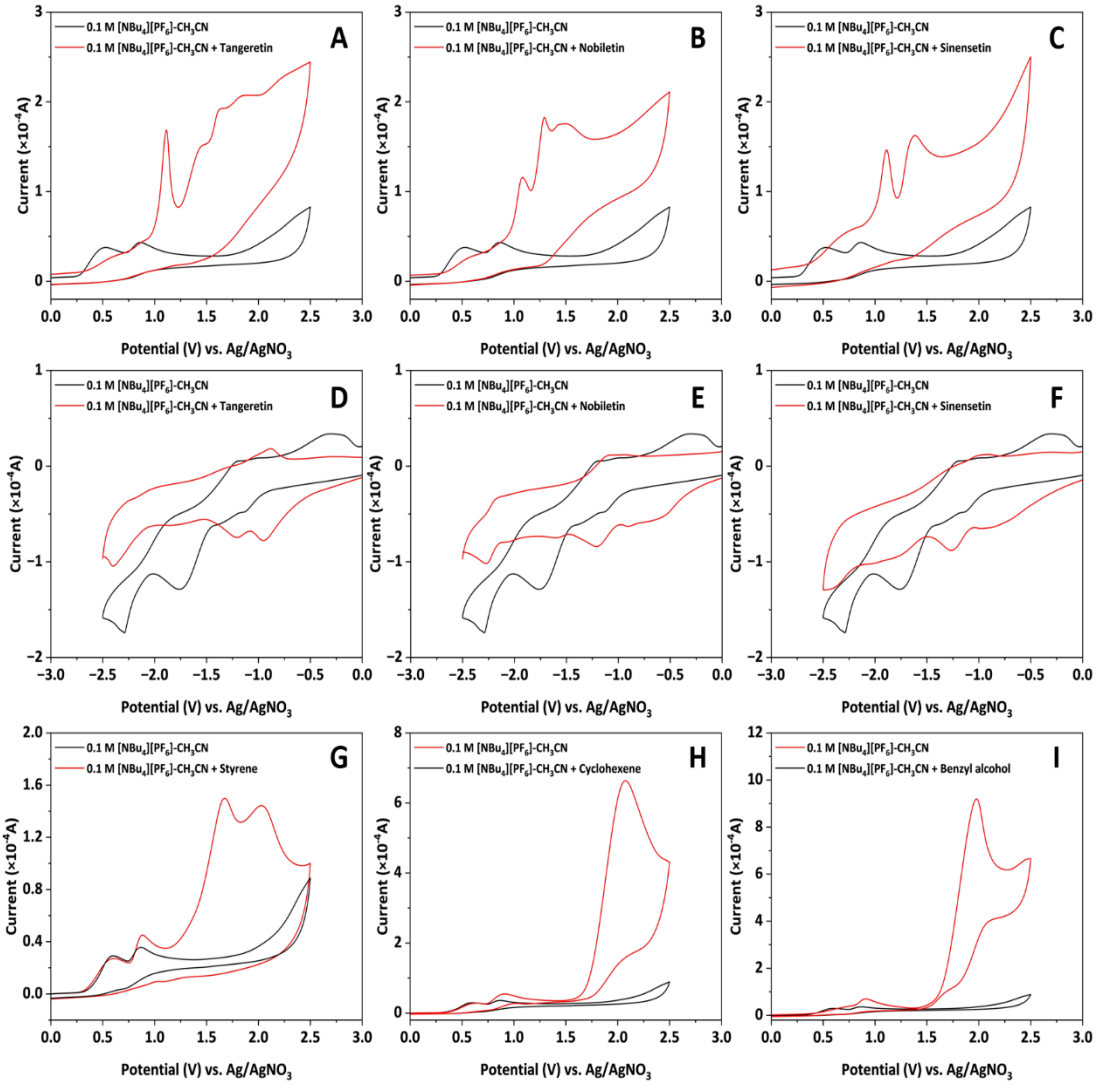


Fig.S9 Cyclic voltammograms of the three flavonoid compounds and all reaction substrates. A standard three-electrode configuration was employed for all experiments in an acetonitrile-based system: a glassy carbon working electrode, a platinum wire counter electrode, and an $Ag/AgNO_3$ reference electrode. The scan rate was set at 0.2 V/s over a potential range of -2.5 V to +2.5 V. To ensure sufficient conductivity, 0.1 M tetrabutylammonium hexafluorophosphate ($[NBu_4][PF_6]$) in acetonitrile was added as the supporting electrolyte.

Table S2 The half-wave oxidation potentials of all reaction substrates.

	Styrene	Cyclohexene	Benzyl alcohol
$E_{p/2}^{ox}$/V	1.45	1.86	1.79

VI.Physical and Fluorescent Properties of Nanoparticles

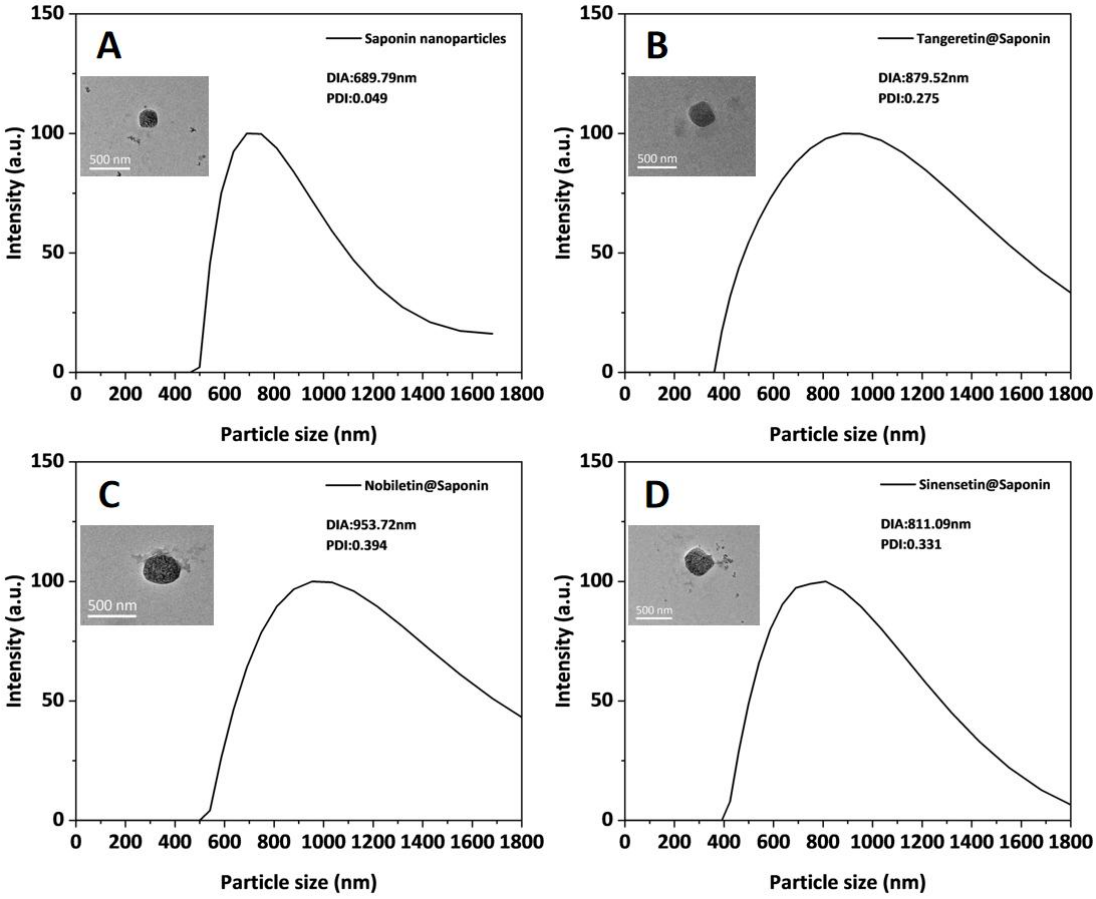


Fig.S10 Particle size distribution diagram and particle morphology. Pure saponin Nanoparticles (A). Photocatalyst @ Saponin Nanoparticles(B,C,D).Inset: TEM images for both nanoparticles. Scale bar=500nm.

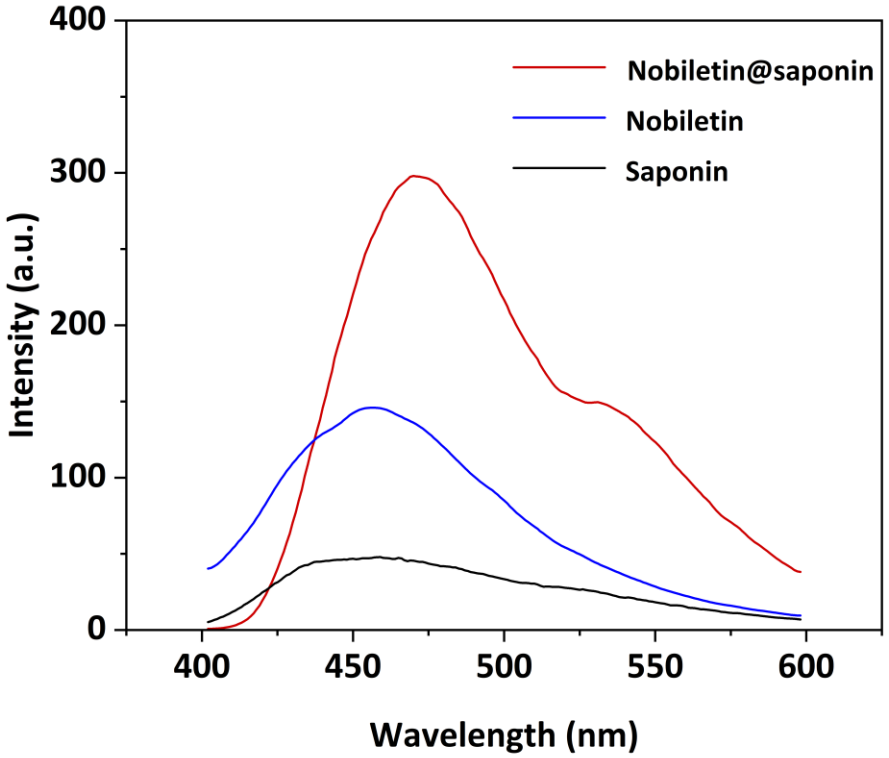


Fig.S11 Fluorescence emission spectra of Nobiletin, Saponin, and Nobiletin@saponin nanoparticles.

VII. Comparison of Catalytic Performance

Table S3 Comparison of Catalytic Performance.

Entry	Photocatalysts	Time (h)	Conv. (%)
1	Tangeretin	16	62.9
2	Nobiletin	16	86.0
3	Sinensetin	16	65.5
4	Mo-SIM[38]	7	93.0
5	Mo-ZrO_2[38]	7	97.0
6	Ti_{12}PTA[39]	8	100.0
7	AuNP[40]	3	28.3

38 H. Noh, Y. Cui, A. W. Peters, *J. Am. Chem. Soc.*, 2016, **138**, 14720-14726.
39 H. Yu, X. Wang and T. Kong, *Chem. Commun.*, 2024, **60**, 15051-15054.
40 V. P. Chauke, Y. Arslanoglu, T. Nyokong, *J. Photochem. Photobiol.*, A, 2011, **221**, 38-46.

Ⅷ. Catalyst Cycling Experiment

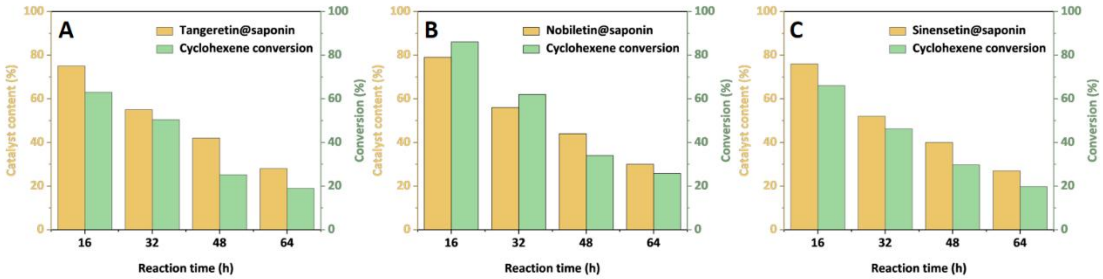


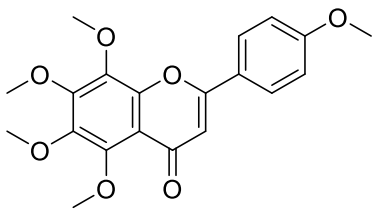
Fig.S12 Catalyst Cycling Experiment. Tangeretin(A). Nobiletin(B). Sinensetin(C).

IX. Green Metrics

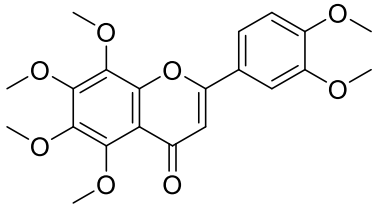
Table S4 Key Green Metrics for the Photo-oxidation of Benzyl Alcohol.

Substrate	Solvent	Photocatalysts	Yield (%)	AE (%)	E factor	Solvent contribution (%)
Benzyl alcohol	H_2O	Tangeretin	43.5	85	2.36	0
Benzyl alcohol	H_2O	Nobiletin	52.4	85	1.79	0
Benzyl alcohol	H_2O	Sinensetin	42.5	85	2.45	0

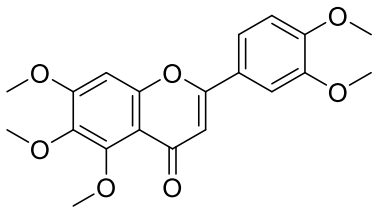
X .Characterization Data of Compounds



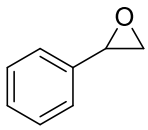
Tangeretin. Following the general procedure to afford the product. EtOAc/PE (1:1) as the eluent, R_f=0.4. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.88 (d, *J* = 7.9 Hz, 1H), 7.02 (d, *J* = 8.7 Hz, 1H), 6.61 (s, 1H), 4.11 – 4.09 (m, 1H), 4.02 (d, *J* = 1.0 Hz, 1H), 3.94 (d, *J* = 0.9 Hz, 3H), 3.88 (d, *J* = 1.0 Hz, 1H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.44, 162.31, 161.26, 151.41, 148.40, 147.75, 144.10, 144.09, 138.08, 127.76, 127.74, 123.80, 114.53, 106.65, 106.60, 62.27, 62.05, 61.84, 61.67, 55.51. HRMS m/z: (ESI) calculated for [M+H]+ $C_{20}H_{20}O_7$: 373.1281, found: 373.1276.



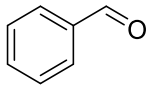
Nobiletin. Following the general procedure to afford the product. EtOAc/PE (1:1) as the eluent, R_f=0.3. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.56 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.41 (d, *J* = 2.1 Hz, 1H), 6.99 (d, *J* = 8.5 Hz, 1H), 6.61 (s, 1H), 4.10 (s, 3H), 4.02 (s, 3H), 3.99 – 3.93 (m, 12H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.36, 161.04, 151.94, 151.43, 149.30, 148.43, 147.73, 144.10, 138.02, 124.02, 119.63, 114.87, 111.23, 108.56, 106.89, 62.27, 61.97, 61.84, 61.69, 56.10, 55.98. HRMS m/z: (ESI) calculated for [M+H]+ $C_{21}H_{22}O_8$: 403.1387, found: 403.1377.



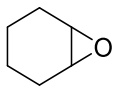
Sinensetin. Following the general procedure to afford the product. EtOAc/PE (1:1) as the eluent, R_f=0.2. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.51 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.33 (d, *J* = 2.2 Hz, 1H), 6.97 (d, *J* = 8.4 Hz, 1H), 6.80 (s, 1H), 6.59 (s, 1H), 3.99 (s, 5H), 3.98 (s, 3H), 3.96 (s, 3H), 3.92 (s, 3H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 177.21, 161.14, 157.66, 154.54, 154.51, 152.60, 151.86, 151.81, 149.28, 140.36, 124.13, 119.64, 119.59, 112.89, 111.13, 108.67, 108.65, 107.42, 107.28, 96.26, 62.21, 61.56, 56.34, 56.32, 56.14, 56.09. HRMS m/z: (ESI) calculated for [M+H]+ $C_{20}H_{20}O_7$: 373.1281, found: 373.1275.



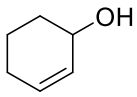
Styrene oxide(1). Following the general procedure to afford product **1** (12.6% yield). EtOAc/PE (1:20) as the eluent, R_f=0.7. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.37 – 7.27 (m, 5H), 3.89 – 3.85 (m, 1H), 3.18 – 3.13 (m, 1H), 2.81 (dd, *J* = 5.5, 2.6 Hz, 1H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 137.59, 128.51, 128.20, 125.50, 52.38, 51.23. HRMS m/z: (ESI) calculated for [M+H]+ C_8H_9O: 121.0648, found: 121.0645.



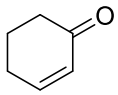
Benzaldehyde(2). Following the general procedure to afford product **2** (41.1% yield). EtOAc/PE (1:20) as the eluent, R_f=0.5. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 10.03 (s, 1H), 7.89 (dd, *J* = 8.1, 1.4 Hz, 2H), 7.63 (dtd, *J* = 12.5, 7.3, 1.3 Hz, 1H), 7.54 (t, *J* = 7.7 Hz, 2H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 192.45, 136.42, 134.49, 130.21, 129.77, 129.02, 128.50. HRMS m/z: (ESI) calculated for [M+H]+ C_7H_7O: 107.0491, found: 107.0489.



Cyclohexene oxide(3). Following the general procedure to afford product **3** (8.6% yield). EtOAc/PE (1:20) as the eluent. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 3.12 (d, *J* = 2.5 Hz, 1H), 1.95 (dt, *J* = 15.2, 6.3 Hz, 1H), 1.86 – 1.75 (m, 1H), 1.42 (tt, *J* = 9.2, 3.7 Hz, 1H), 1.29 – 1.15 (m, 1H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 52.16, 24.44, 19.44. HRMS m/z: (ESI) calculated for [M+H]+ $C_6H_{11}O$: 99.0804, found: 99.0802.

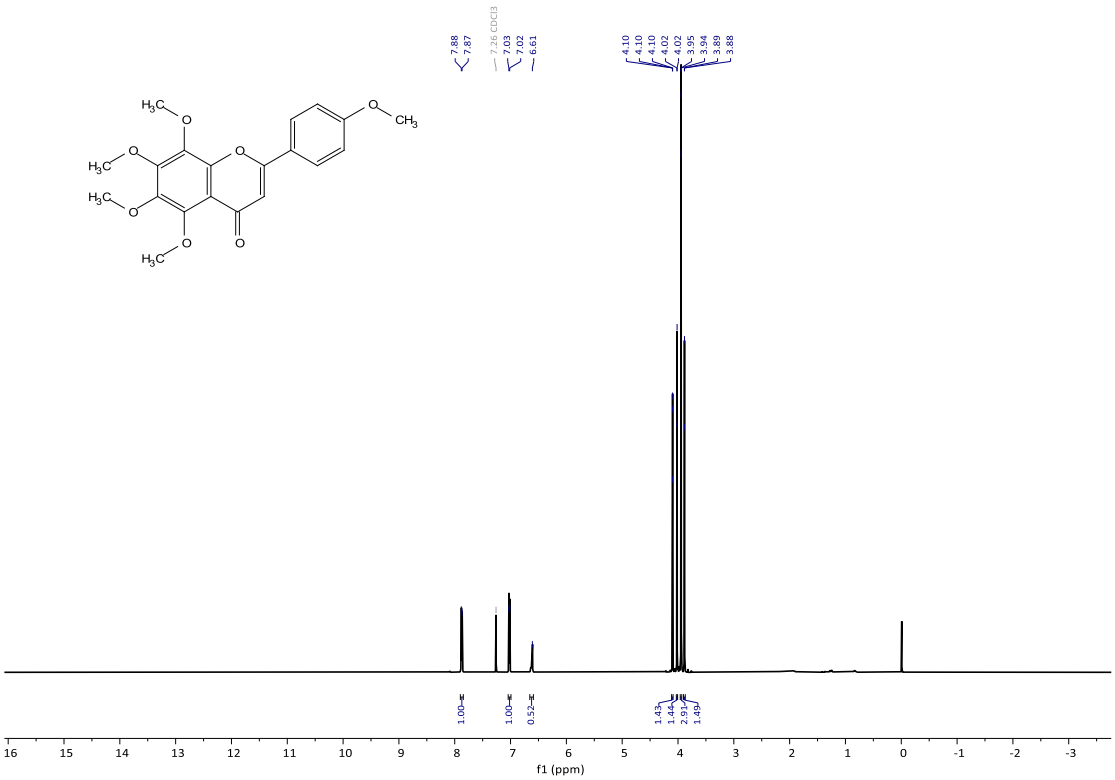


2-cyclohexen-1-ol(4). Following the general procedure to afford product **4** (28.9% yield). EtOAc/PE (1:20) as the eluent, R_f=0.3. ^{1}H NMR (600 MHz, CDCl3) δ 5.85 – 5.80 (m, 1H), 5.75 (m, 1H), 4.19 (s, 1H), 2.08 – 2.01 (m, 1H), 1.99 – 1.93 (m, 1H), 1.91 – 1.84 (m, 1H), 1.78 – 1.69 (m, 1H), 1.60 (m, 3H).13C NMR (151 MHz, Chloroform-*d*) δ 130.64, 129.82, 65.52, 32.00, 25.05, 18.92. HRMS m/z: (ESI) calculated for [M+H]+ $C_6H_{11}O$: 99.0804, found: 99.0803.

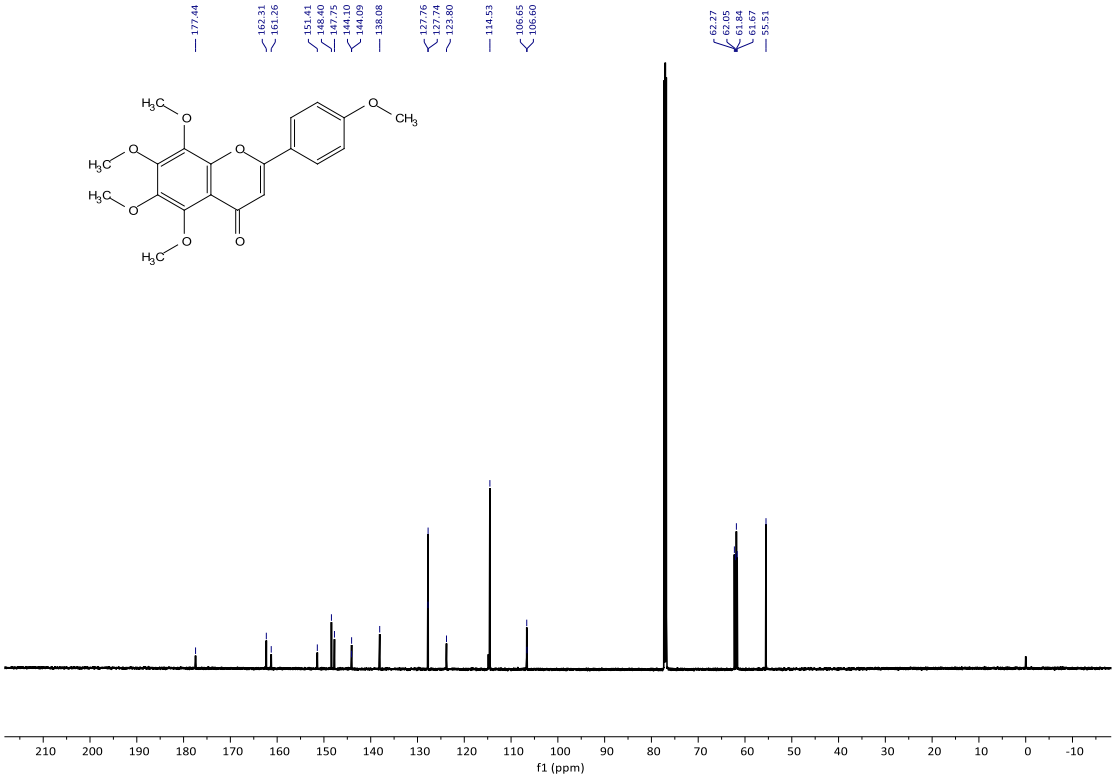


2-Cyclohexen-1-one(5). Following the general procedure to afford product **5** (28.6% yield). EtOAc/PE (1:20) as the eluent, R_f=0.5. ^{1}H NMR (600 MHz, Chloroform-*d*) δ 6.99 (dt, *J* = 9.8, 4.1 Hz, 1H), 6.02 (dt, *J* = 10.1, 2.1 Hz, 1H), 2.46 – 2.40 (m, 2H), 2.35 (tdd, *J* = 6.1, 4.1, 2.1 Hz, 2H), 2.06 – 1.98 (m, 2H). ^{13}C NMR (151 MHz, Chloroform-*d*) δ 199.80, 150.69, 129.96, 38.12, 25.69, 22.74. HRMS m/z: (ESI) calculated for [M+H]+ C_6H_9O: 97.0648, found: 97.0645.

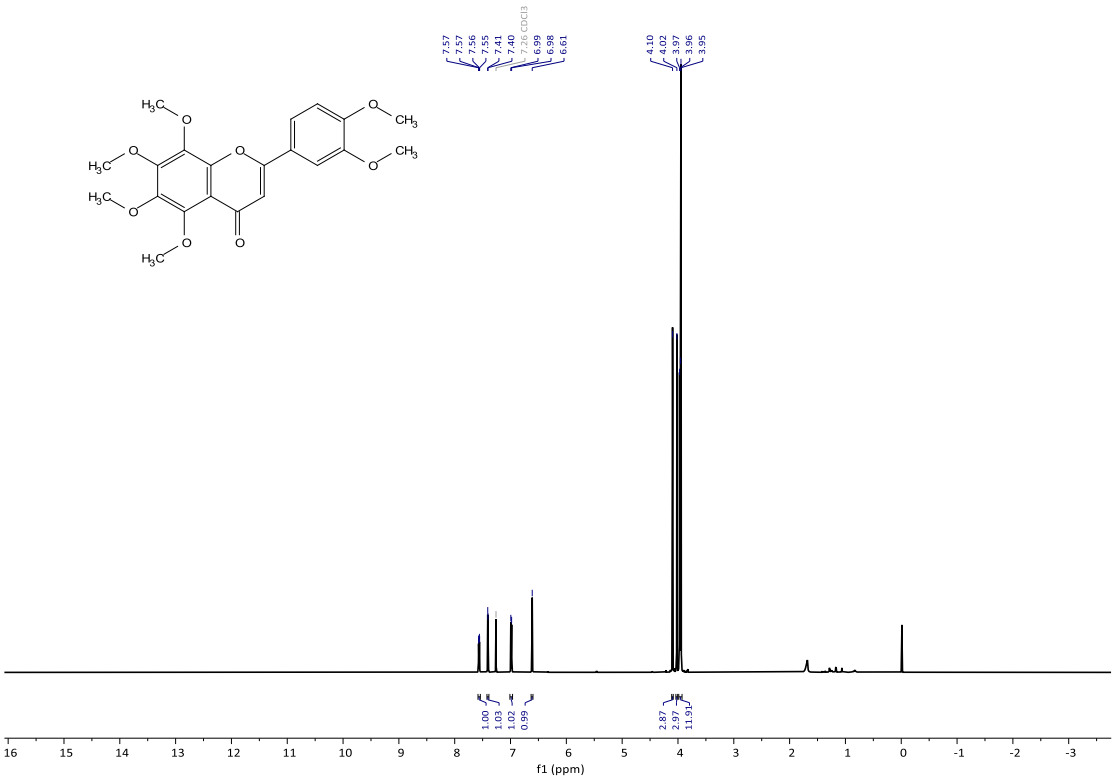
XI.The NMR Spectra of Compounds



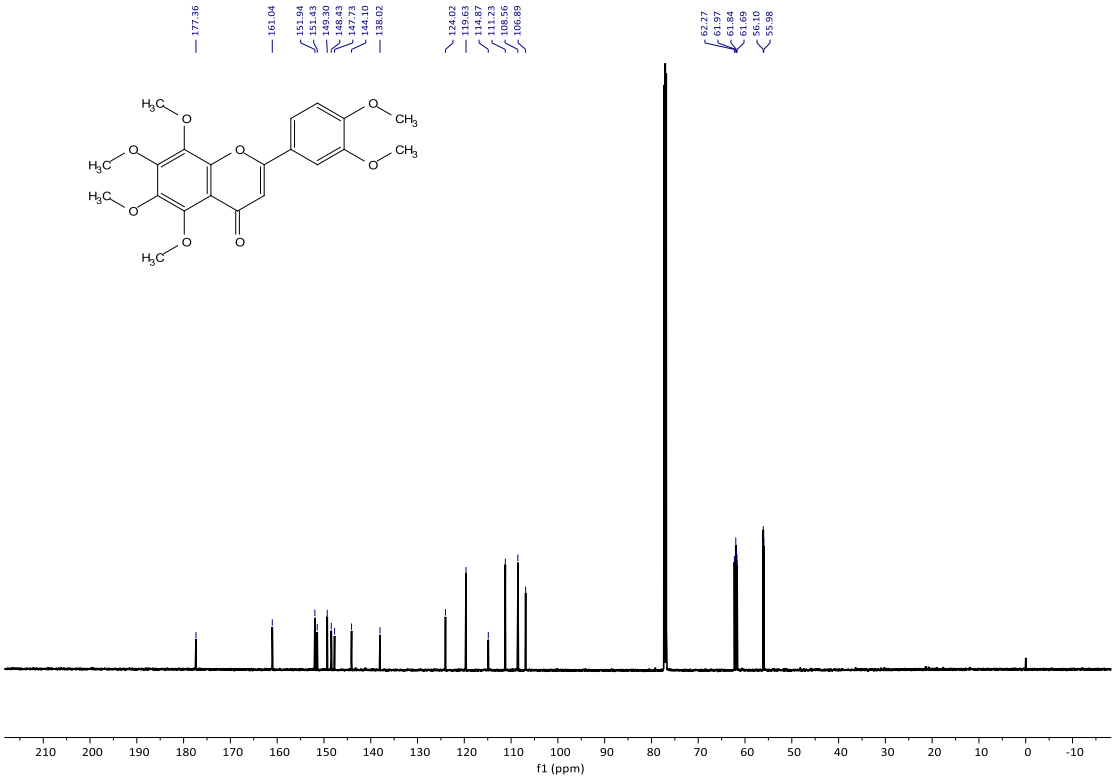
^{1}H NMR spectrum of compound Tangeretin (600 MHz, $CDCl_3$).



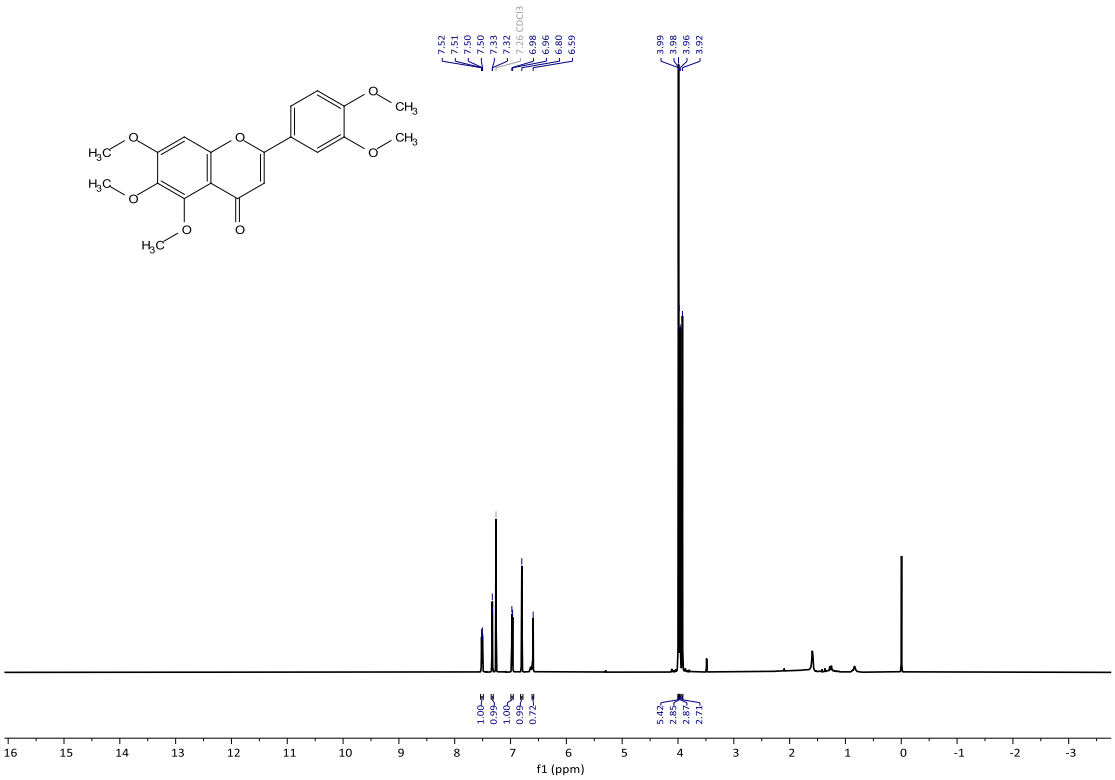
^{13}C NMR spectrum of compound Tangeretin (151 MHz, $CDCl_3$).



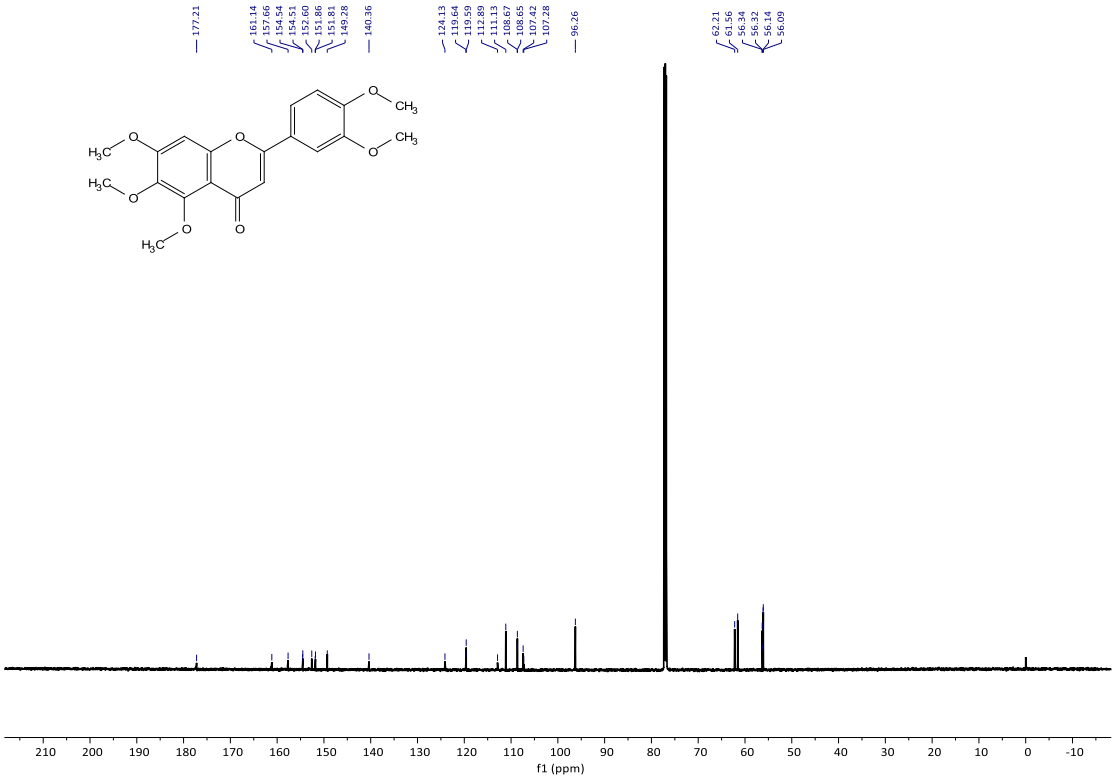
^{1}H NMR spectrum of compound Nobiletin (600 MHz, $CDCl_3$).



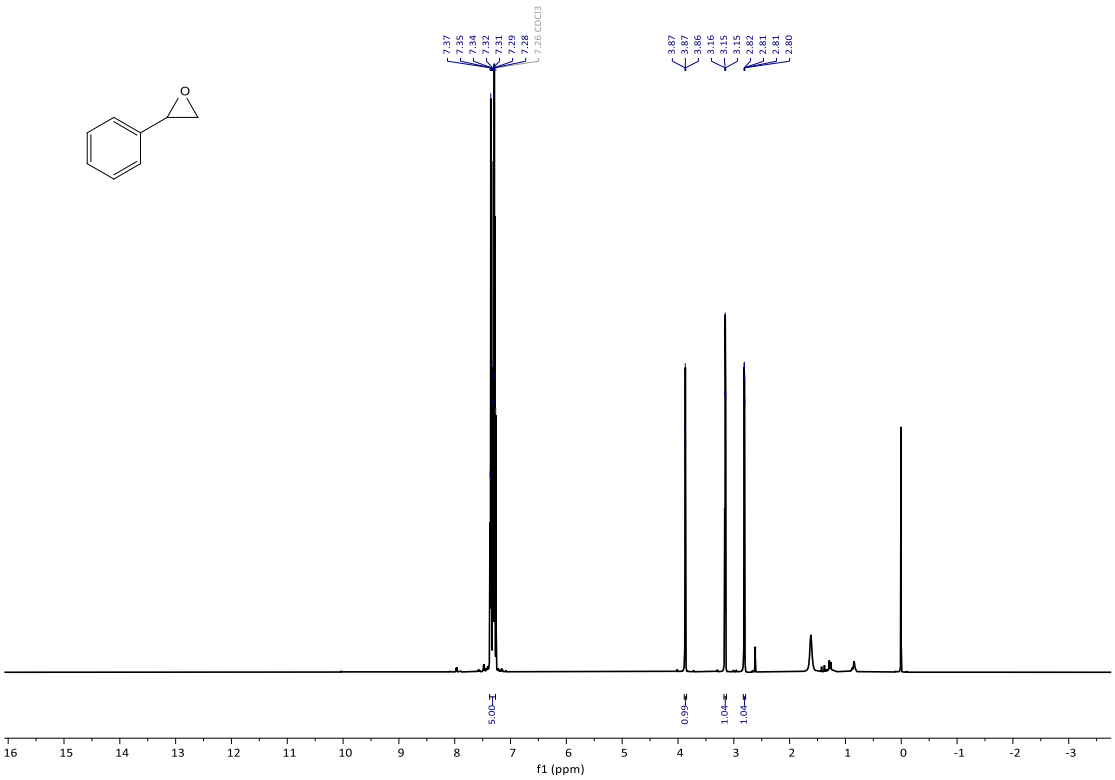
^{13}C NMR spectrum of compound Nobiletin (151 MHz, $CDCl_3$).



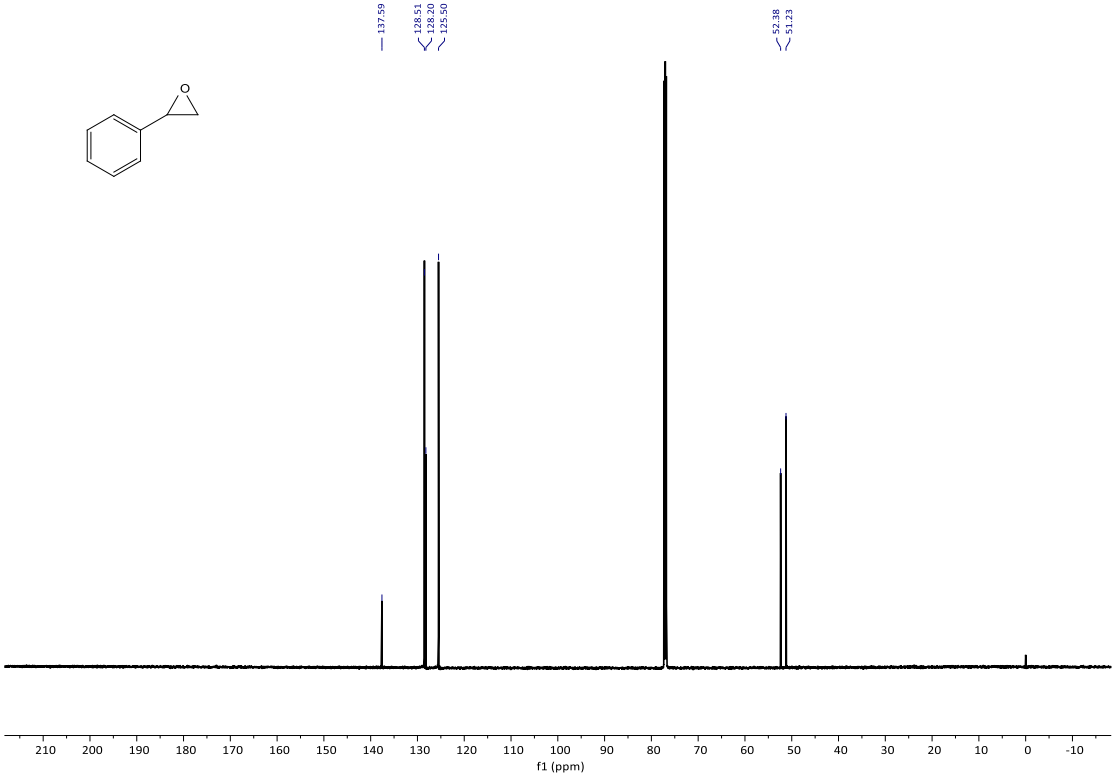
^{1}H NMR spectrum of compound Sinensetin (600 MHz, $CDCl_3$).



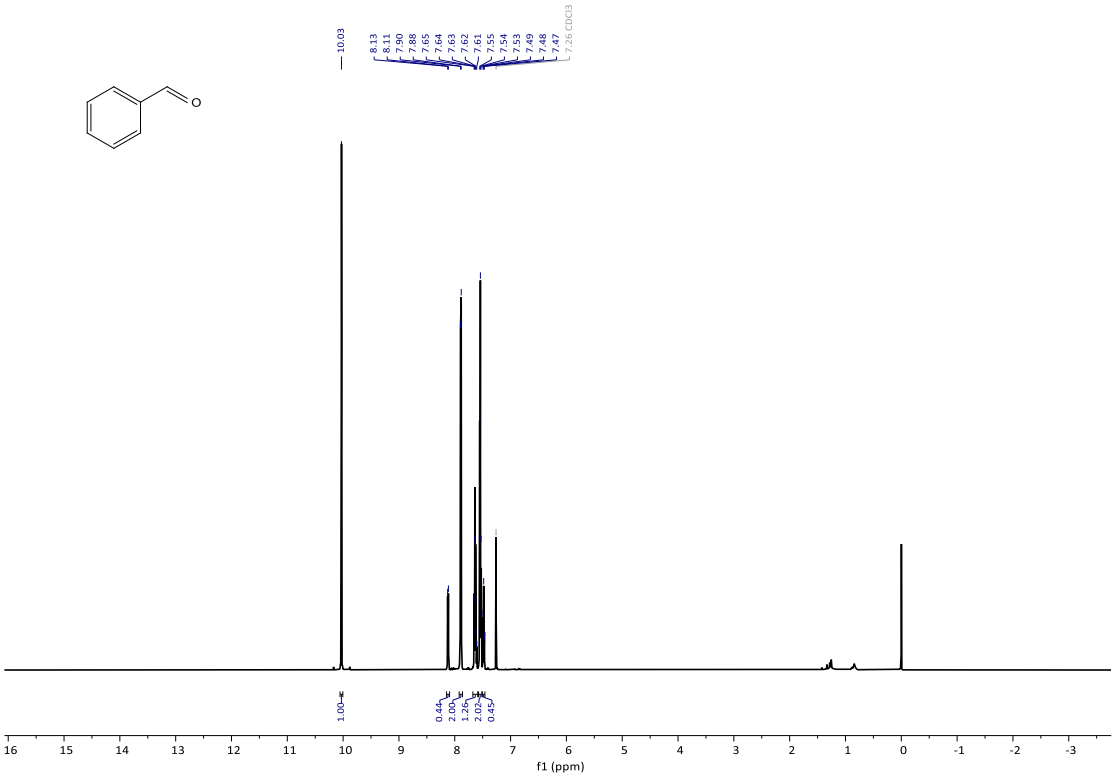
^{13}C NMR spectrum of compound Sinensetin (151 MHz, $CDCl_3$).



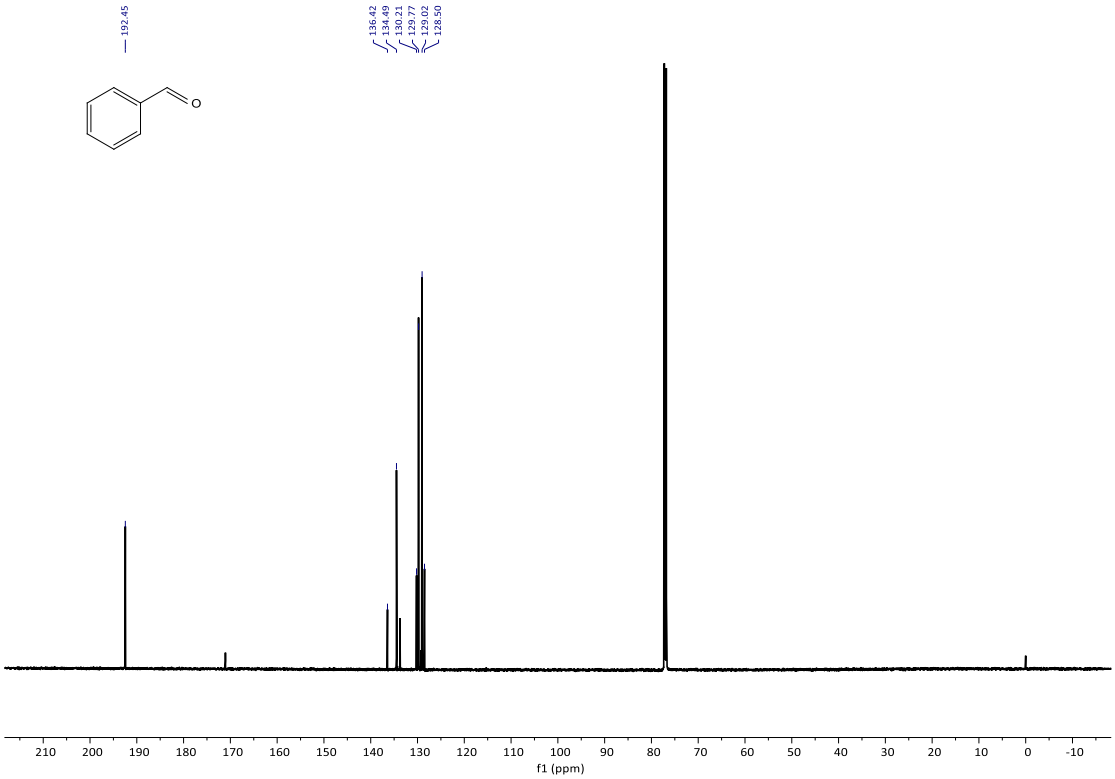
^{1}H NMR spectrum of compound 1 (600 MHz, $CDCl_3$).



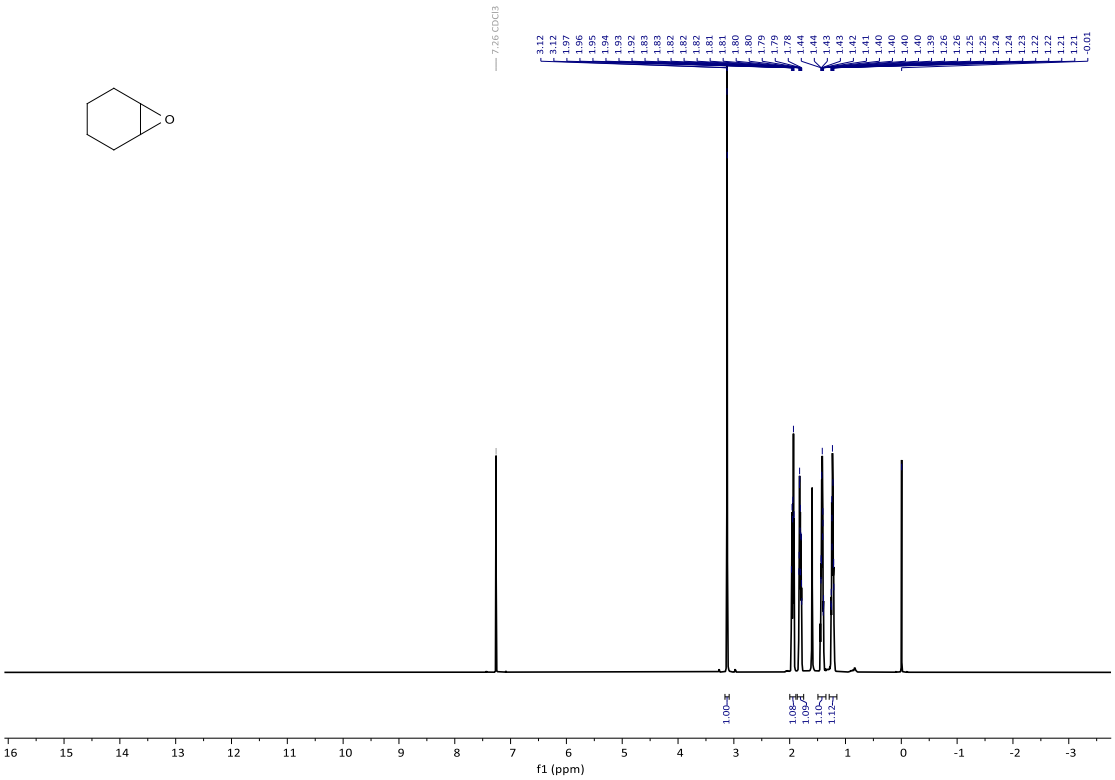
^{13}C NMR spectrum of compound 1 (151 MHz, $CDCl_3$).



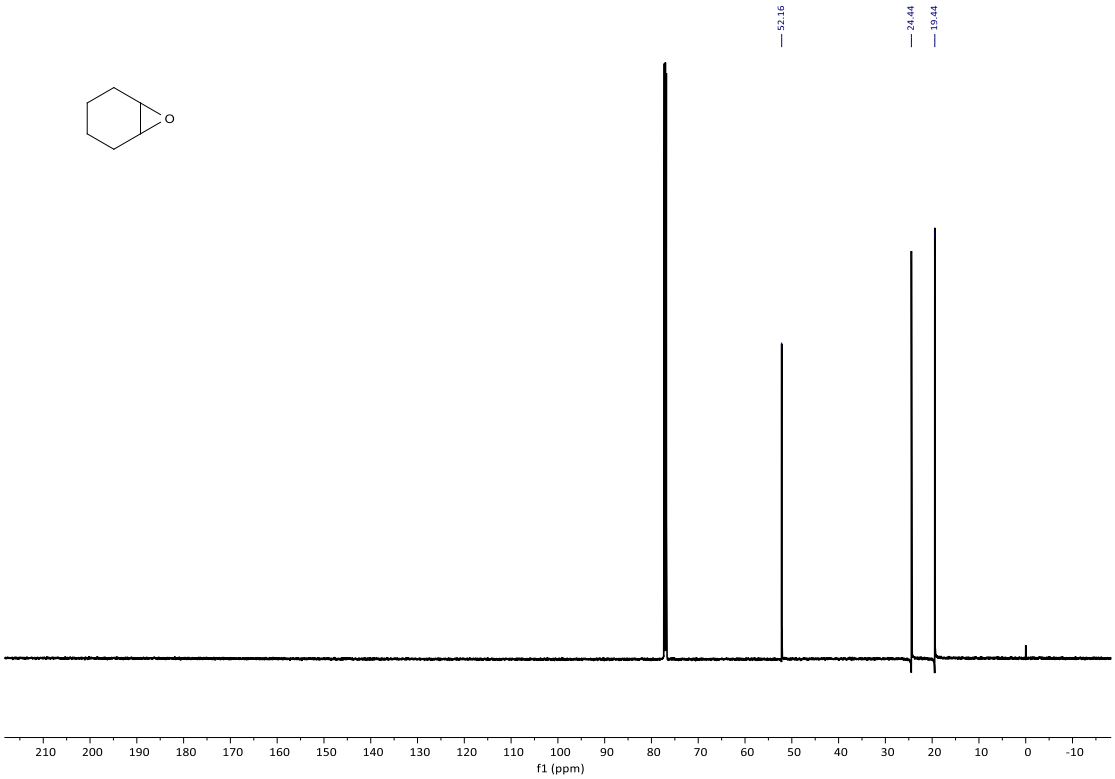
^{1}H NMR spectrum of compound 2 (600 MHz, $CDCl_3$).



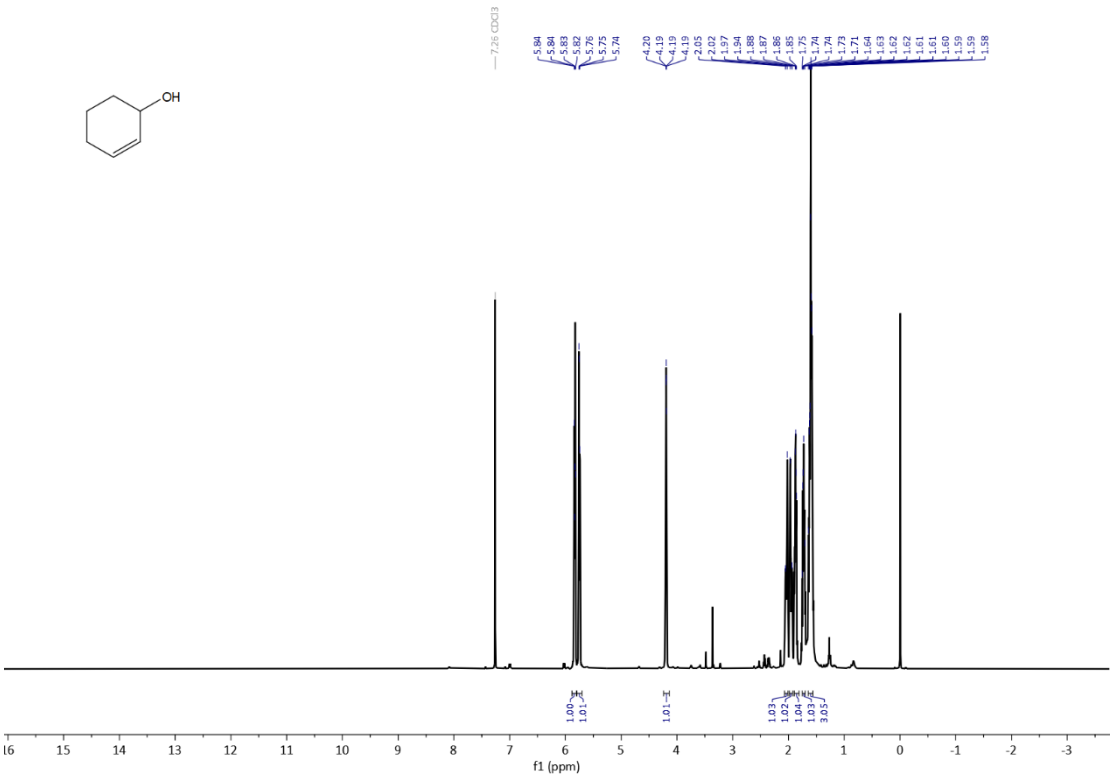
^{13}C NMR spectrum of compound 2 (151 MHz, $CDCl_3$).



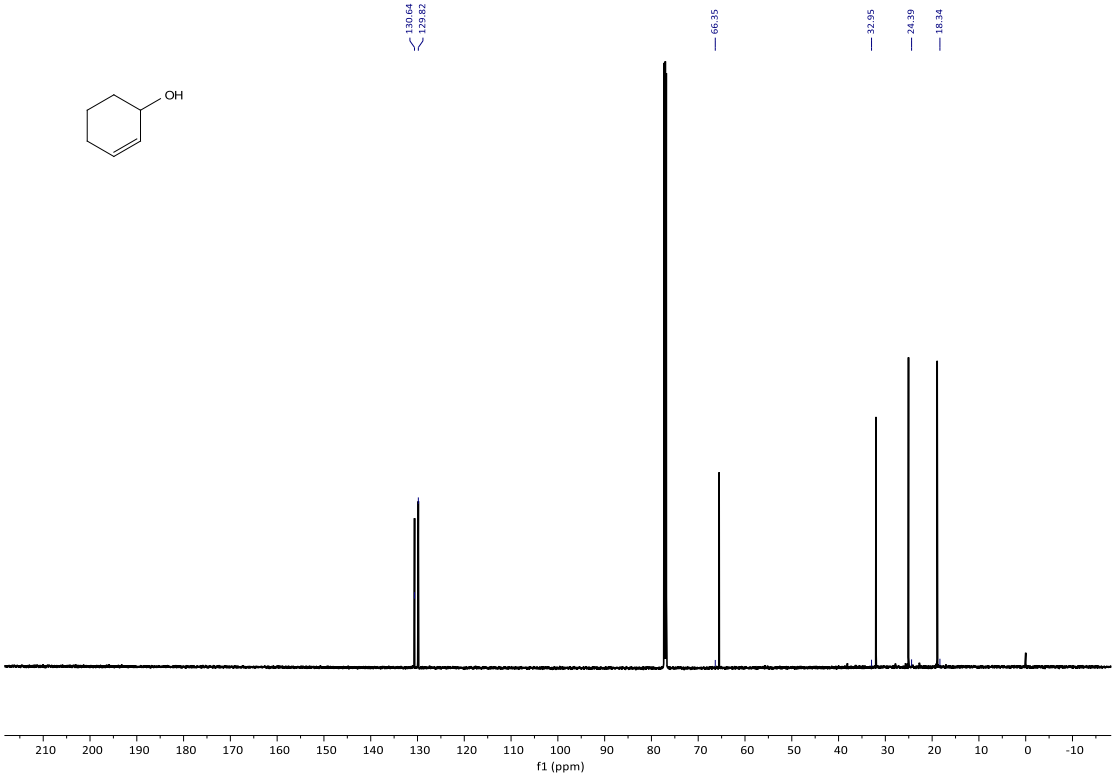
^{1}H NMR spectrum of compound 3 (600 MHz, $CDCl_3$).



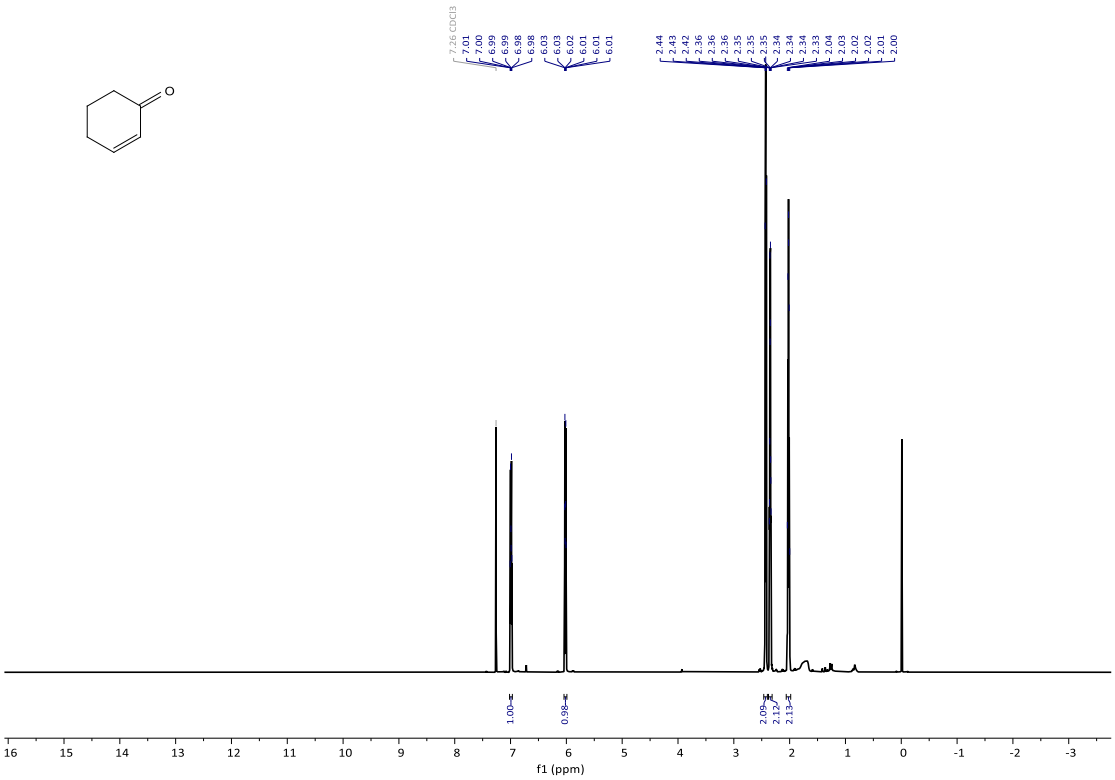
^{13}C NMR spectrum of compound 3 (151 MHz, $CDCl_3$).



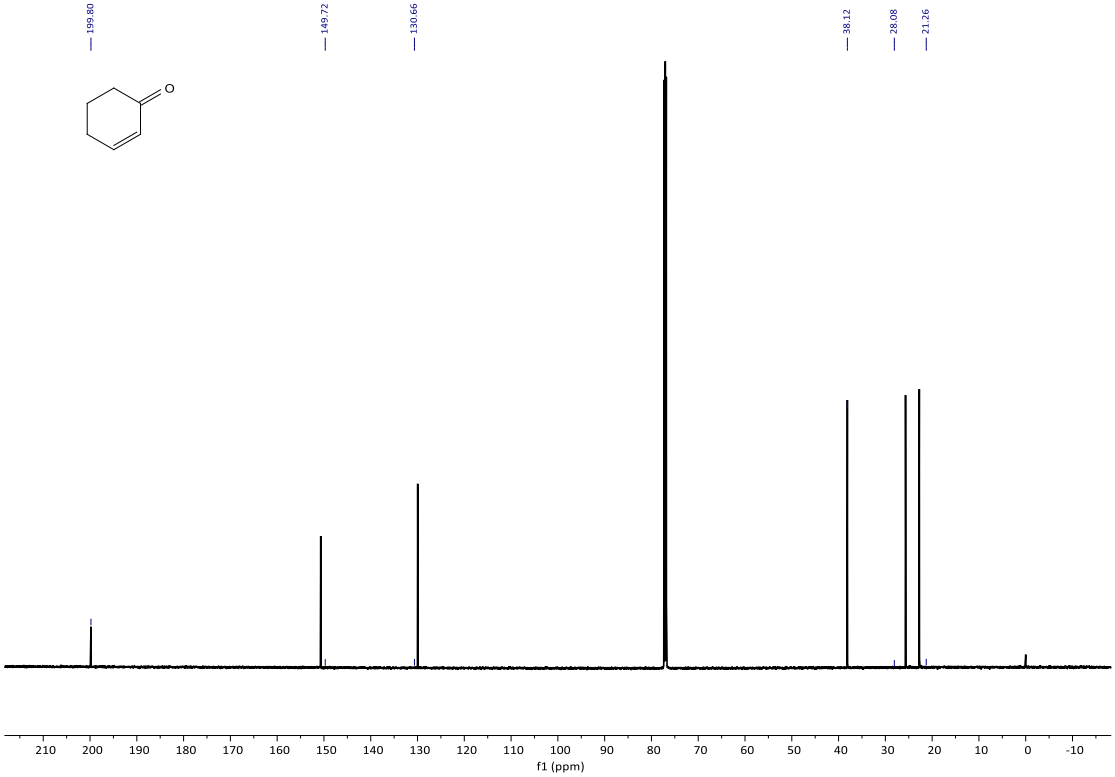
^{1}H NMR spectrum of compound 4 (600 MHz, $CDCl_3$).



^{13}C NMR spectrum of compound 4 (151 MHz, $CDCl_3$).



^{1}H NMR spectrum of compound 5 (600 MHz, $CDCl_3$).



^{13}C NMR spectrum of compound 5 (151 MHz, $CDCl_3$).