

Supplementary Material

Methodologies used for Synthesis/Chemical Characterization

Materials and Methods

Chemical reagents used were commercially available. All reactions were conducted with magnetic stirring under the atmosphere of argon in oven dried flasks. Reactions were monitored by TLC until deemed complete using silica-gel-coated glass plates (Merck Kieselgel 60 F254). Plates were visualized under UV light (254 nm). Plates were dyed with 10% phosphomolybdic acid (PMA) in ethanol.

HPLC analysis

Agilent 1100 HPLC system equipped with a quaternary pump, an Agilent diode array detector 1200 Series and normal phase silica gel column (Phenomenex[®] Spherclone, 250 × 10 mm, 5 μm) with gradient system *n*-hexane to ethyl acetate in 20 min at 2 mL/min.

Characterization

¹H, ¹³C NMR spectra were recorded at 500 (¹H), 125 MHz (¹³C) on Agilent Inova 500, and Jeol Eclipse 400 MHz 400 (¹H), 100 MHz (¹³C) spectrometers. Chemical shifts (δ) are reported in parts per million (ppm) from the residual solvent peak and coupling constant (*J*) in Hz. Proton multiplicity is reported in: singlet (s), doublet (d), triplet (t), quartet (quart.), quintet (quint.), septet (sept), multiplet (m), broad (br). Infrared measurements were carried out on a Bruker Platinum ATR Alpha instrument. The HRMS analyses were carried out on a Bruker Daltonics microTOF-QIII spectrometer.

General procedure 1 (GP1) for the synthesis of Alkyl succinate monoesters (S1-S3)

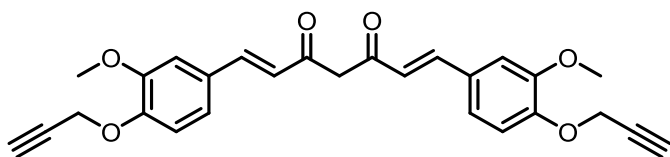
An oven dried round bottom flask was charged with alcohol (854 mg, 14.7 mmol), solvent (dichloromethane) (5 mL), and *N,N*-diisopropylethylamine (1.3 mL, 7.35 mmol, 0.5 equiv.) at room temperature (RT). After 2 h, succinic anhydride (735 mg, 7.35 mmol, 0.5 equiv.), and 4-dimethylaminopyridine (448 mg, 3.67 mmol, 0.25 equiv.) were added, and the reaction stirred at RT. After 48 h, the reaction mixture was diluted with brine/1M HCl (3:1, 10 mL). The aqueous layer was extracted with dichloromethane (3 × 10 mL), and the combined organic phases dried over anhydrous sodium sulfate (Na₂SO₄) and concentrated under reduced pressure. The crude product was washed with *n*-hexanes (3 × 20 mL) to give the desired product.

General procedure 2 (GP2) for the synthesis of dialkylcurcumin and monoalkylcurcumin derivatives (5-10)

An oven dried round bottom flask, equipped with magnetic stirrer and 3 Å molecular sieves, was flushed with argon and charged with succinate (207 mg, 1.3 mmol, 10 equiv.), pyridine (3 mL), 4-dimethylaminopyridine (366 mg, 0.39 mmol, 3 equiv.), and 1-ethyl-3-(3-dimethylaminopropyl) carbodiimide hydrochloride (75 mg, 0.39 mmol, 3 equiv.). The reaction was stirred for 4 h at RT. Concurrently a solution of curcumin (**1**) (50 mg, 0.13 mmol) in pyridine (3 mL) was stirred for 4 h at RT. Then the curcumin solution was added to 4-oxobutanoic acid reaction, and allowed to stir for 48 h at RT. The reaction mixture was then diluted with a 0.5 M aqueous solution of Na₂CO₃/brine (1:1, 10 mL), and the aqueous layer was extracted with ethyl acetate (EtOAc) (3 × 10 mL). The combined organic phases were dried over Na₂SO₄, concentrated under reduced pressure, and purified by HPLC using normal

phase silica gel column (Phenomenex® Sphereclone, 250 × 10 mm, 5 μm) with the gradient system of *n*-hexane to ethyl acetate in 20 min at 2 mL/min to give the desired product.

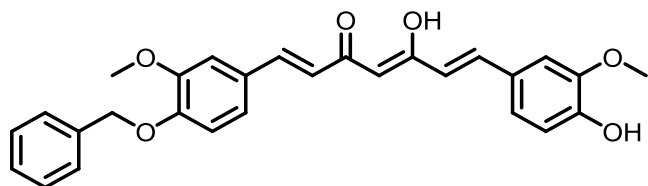
(1*E*,6*E*)-1,7-Bis(3-methoxy-4-(prop-2-yn-1-yloxy)phenyl)hepta-1,6-diene-3,5-dioneⁱ (2)



Curcumin (200 mg, 0.54 mmol), *N,N*-dimethylformamide (6 mL), K₂CO₃ (150 mg, 1.08 mmol, 2 equiv.), and propargyl

bromide (300 μL, 2.7 mmol, 5 equiv.) were stirred for 48 h at RT. The reaction mixture was diluted with water (10 mL), and extracted with EtOAc (3 × 10 mL). The combined organic phases were dried over Na₂SO₄, and concentrated under reduced pressure. The crude product was washed with hexane (3 × 20 mL) to yield diether **2** (168 mg, 70 %). – ¹H NMR (400 MHz): 3.17 (2 H, d, *J* = 2 Hz), 3.94 (6 H, s), 4.79–4.81 (6 H, m), 6.67 (2 H, d, *J* = 16 Hz), 6.99–7.03 (4 H, m), 7.15 (2 H, dd, *J* = 2, 8 Hz), 7.73 (2 H, d, *J* = 16 Hz) ppm. – ¹³C NMR (100 MHz): 56.0, 56.1, 56.6, 72.1, 76.3, 110.9, 113.7, 118.6, 123.5, 128.1, 145.6, 149.5, 149.8, 193.5 ppm. – HRMS (*m/z*) calcd for C₂₇H₂₄NaO₆: 467.1465; found: 467.1474 [M+Na⁺].

(1*E*, 4*Z*, 6*E*)-1-(4-(Benzyloxy)-3-methoxyphenyl)-5-hydroxy-7-(4-hydroxy-3-methoxyphenyl)hepta-1,4,6-trien-3-one (3)



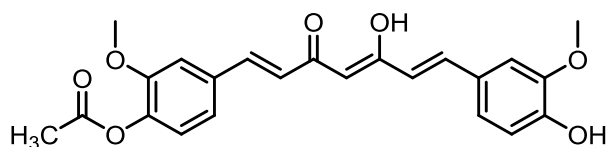
Curcumin (100 mg, 0.27 mmol), acetone (5 mL), Cs₂CO₃ (88 mg, 0.27 mmol, 1 equiv.), and benzyl bromide (1 mL, 8.1 mmol, 30 equiv.) were 56 °C

for 24 h. The reaction mixture was diluted with water (20 mL), and extracted with EtOAc (3 × 10 mL).

The combined organic phases were dried over Na₂SO₄, and concentrated under reduced pressure. The

crude product was purified by column chromatography [hexane/EtOAc, silica gel] to give curcumin ether **3** (98 mg, 80 %). – ¹H NMR (400 MHz): 3.94 (6 H, s), 5.20 (2 H, s), 5.81 (1 H, s), 6.48 (2 H, d, *J* = 16 Hz), 6.87–6.94 (2 H, m), 7.05–7.09 (4 H, m), 7.31–7.44 (5 H, m), 7.58 (2 H, d, *J* = 16 Hz) ppm. – ¹³C NMR (100 MHz): 55.9, 56.0, 70.9, 101.2, 109.7, 110.4, 113.5, 114.8, 115.9, 121.8, 122.1, 122.3, 122.9, 127.2, 127.7, 128.0, 128.6, 130.0, 136.6, 140.3, 140.6, 146.8, 147.9, 149.8, 150.2, 183.0, 183.4 ppm. – IR: 1136, 1270, 1430, 1464, 1512, 1585, 1625, 2850, 2917, 2955, 3370 cm⁻¹. – HRMS (*m/z*) calcd for C₂₈H₂₆NaO₆: 481.1622; found: 481.1619 [M+Na⁺].

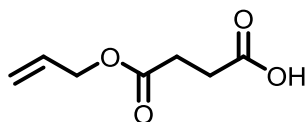
4-((1*E*,4*Z*,6*E*)-5-Hydroxy-7-(4-hydroxy-3-methoxyphenyl)-3-oxohepta-1,4,6-trien-1-yl)-2-methoxyphenyl acetateⁱⁱ (4**)**



Curcumin (100 mg, 0.27 mmol) and K₂CO₃ (38 mg, 0.27 mmol, 1 equiv.) in CH₂Cl₂ (10 mL) were stirred at RT. After 8 h, a solution of acetyl

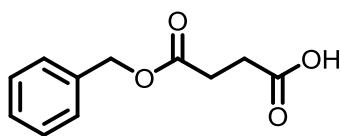
chloride (96 μL, 1.35 mmol, 5 equiv.) in CH₂Cl₂ (5 mL) was sonicated for 5 min and added to the curcumin solution. The reaction was stirred for 24 h at RT. Column chromatography [hexane/EtOAc, silica gel] afforded acetate **4** (79 mg) in 71 % yield. – ¹H NMR (400 MHz): 2.32 (3 H, s), 3.88 (3 H, s), 3.94 (3 H, s), 5.81–5.85 (2 H, m), 6.52–6.58 (2 H, m), 7.05–7.17 (6 H, m), 7.58–7.63 (2 H, m), 15.8 (1 H, br s) ppm. – ¹³C NMR (100 MHz): 20.6, 55.9, 56.0, 101.5, 109.7, 111.5, 114.8, 121.0, 121.8, 123.0, 123.3, 124.3, 127.6, 134.1, 139.4, 141.1, 146.8, 148.0, 151.4, 168.8, 181.8, 184.4 ppm. – HRMS (*m/z*) calcd for C₂₃H₂₂NaO₇: 433.1258; found: 433.1263 [M+Na⁺].

4-(Allyloxy)-4-oxobutanoic acidⁱⁱⁱ (S1**)**



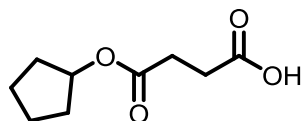
According to GP1, allyl alcohol (854 mg, 14.7 mmol), and *N,N*-diisopropylethylamine (640 μ L, 3.67 mmol, 0.25 equiv.) were stirred in CH_2Cl_2 (5 mL) at RT. After 2 h, succinic anhydride (735 mg, 7.35 mmol, 0.5 equiv.) and 4-dimethylaminopyridine (448 mg, 3.67 mmol, 0.25 equiv.) were added, and the reaction mixture stirred for 48 h at RT to yield monoester **S1** (790 mg) in 40% yield. ^1H NMR (500 MHz): 2.56 (2 H, m), 2.65 (2 H, m), 4.61 (2 H, d, $J = 6$ Hz), 5.29 (1 H, dd, $J = 1, 10$ Hz), 5.35 (1 H, dd, $J = 2, 17$ Hz) 5.86 (1 H, m), 9.70 (1 H, br s) ppm. ^{13}C NMR (125 MHz): 28.9, 29.1, 65.5, 118.4, 134.1, 171.6, 178.2 ppm.

4-(Benzyloxy)-4-oxobutanoic acid^{iv} (**S2**)



According to GP1, benzyl alcohol (3 g, 27.7 mmol, 2.88 mL), pyridine (20 mL), 4-dimethylaminopyridine (508 mg, 4.15 mmol, 0.15 equiv.), and succinic anhydride (1.3 g, 13.8 mmol, 0.5 equiv.) were combined and the reaction stirred for 6 h at 100 $^\circ\text{C}$ to yield monoester **S2** (3.74 g) in 65% yield. ^1H NMR (500 MHz): 2.70 (4 H, m), 5.15 (2 H, s), 7.36 (5 H, br s), 9.42 (1 H, br s) ppm. ^{13}C NMR (125 MHz): 28.9, 29.1, 66.7, 128.2, 128.3, 128.6, 135.7, 172.0, 178.3 ppm.

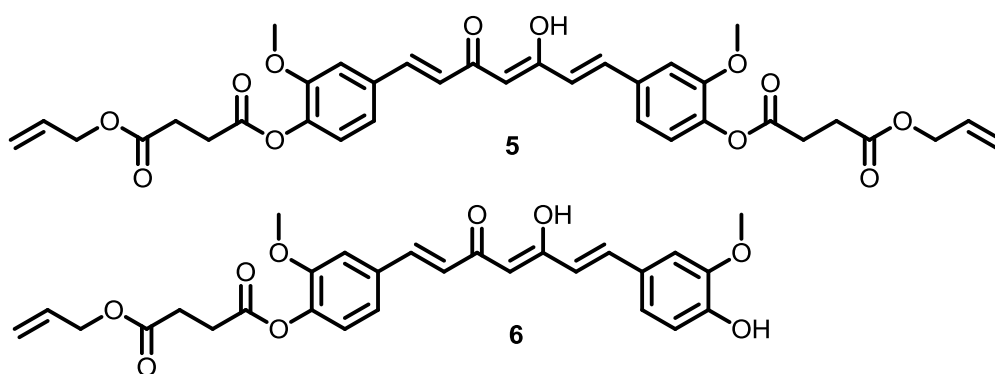
4-(Cyclopentyloxy)-4-oxobutanoic acid^v (**S3**)



According to GP1, cyclopentanol (949 mg, 11 mmol, 1 mL) and *N,N*-diisopropylethylamine (958 μ L, 5.5 mmol, 0.5 equiv.) were stirred in CH_2Cl_2 (5 mL) at RT. After 2 h, succinic anhydride (550 mg, 5.5 mmol, 0.5 equiv.) and 4-dimethylaminopyridine (672 mg, 5.5 mmol, 0.5 equiv.) were added and the reaction mixture was stirred for 48 h at RT to yield monoester **S3** (1 g) in 50% yield. ^1H NMR (500 MHz): 1.54–1.71 (8 H, m),

2.55 (2 H, m), 2.63 (2 H, m), 5.12 (1 H, s), 9.70 (1 H, br s) ppm. – ^{13}C NMR (125 MHz): 23.5, 28.9, 29.1, 32.4, 77.5, 171.9, 177.8 ppm.

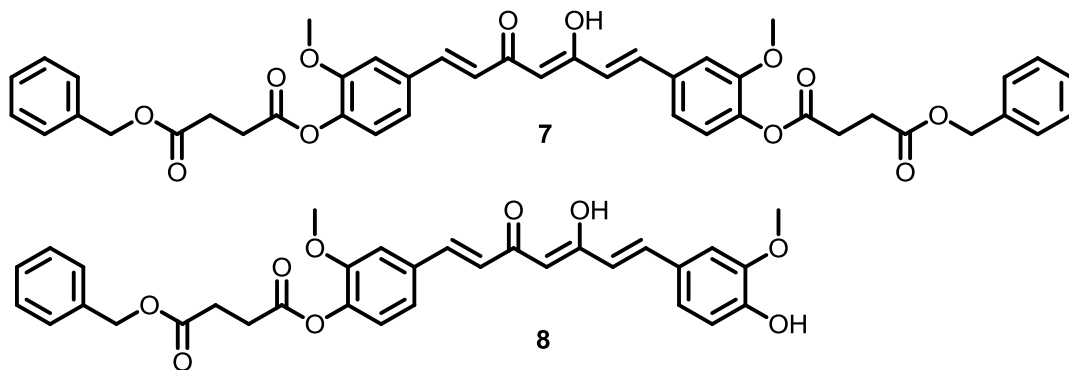
Diallyl *O,O'*-(((1*E*,3*Z*,6*E*)-3-hydroxy-5-oxohepta-1,3,6-triene-1,7-diyl)bis(2-methoxy-4,1-phenylene)) disuccinate (**5**), and allyl (4-(((1*E*,4*Z*,6*E*)-5-hydroxy-7-(4-hydroxy-3-methoxyphenyl)-3-oxohepta-1,4,6-trien-1-yl)-2-methoxyphenyl) succinate (**6**)



According to GP2, monosuccinate **S1** (207 mg, 0.67 mmol, 10 equiv), pyridine (3 mL), 4-dimethylaminopyridine (366 mg, 0.39 mmol, 3 equiv.), and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (75 mg, 0.39 mmol, 3 equiv.) were combined and stirred at RT. Concurrently, a solution of curcumin (50 mg, 0.13 mmol) in pyridine (3 mL) was prepared at RT. After 4 h, the reactions were combined and allowed to stir for 48 h at RT. The crude product was purified by HPLC to obtain diester **5** (19 mg, 22%) and monoester **6** (27 mg, 39%). **Compound 5**: ^1H NMR (400 MHz): 2.80 (4 H, t, $J = 14$ Hz), 2.95 (4 H, t, $J = 13$ Hz), 3.87 (6 H, s), 4.63 (4 H, d, $J = 5$ Hz), 5.25 (2H, dd, $J = 1, 10$ Hz), 5.34 (2 H, dd, $J = 2, 17$ Hz), 5.85 (1 H, s), 5.87-5.97 (2 H, m), 6.56 (2 H, d, $J = 16$ Hz), 7.06-7.17 (6 H, m), 7.61 (2 H, d, $J = 16$ Hz), 15.8 (1 H, br s) ppm. – ^{13}C NMR (100 MHz): 28.9, 29.1, 55.9, 65.5, 101.8, 111.5, 118.4, 121.0, 123.3, 124.3, 132.0, 134.0, 140.0, 141.2, 151.4, 170.2, 171.6, 183.1 ppm. – IR: 1031, 1132, 1202, 1259, 1509, 1599, 1736, 1762, 2851, 2922 cm^{-1} . HRMS (m/z) calcd for $\text{C}_{35}\text{H}_{36}\text{NaO}_{12}$:

671.2099; found: 671.2068 [M+Na⁺]. **Compound 6**: ¹H NMR (400 MHz): 2.79 (2 H, t, *J* = 14 Hz), 2.95 (2 H, t, *J* = 14 Hz), 3.86 (3 H, s), 3.94 (3 H, s), 4.63 (2 H, d, *J* = 6 Hz), 5.24 (1 H, dd, *J* = 1, 10 Hz), 5.33 (1 H, dd, *J* = 2, 17 Hz), 5.83 (1 H, s), 5.87-5.97 (2 H, m), 6.51 (2 H, dd, *J* = 16, 22 Hz), 6.93 (1 H, d, *J* = 8 Hz), 7.05-7.16 (5 H, m), 7.60 (2 H, dd, *J* = 5, 16 Hz), 15.9 (1 H, br s) ppm. – ¹³C NMR (100 MHz): 28.9, 29.1, 55.9, 56.0, 65.5, 101.5, 109.7, 111.5, 114.9, 118.4, 120.9, 121.8, 123.0, 123.2, 124.3, 127.6, 131.9, 134.1, 139.4, 141.1, 146.8, 148.0, 151.3, 170.3, 171.7, 181.8, 184.6 ppm. – IR: 1131, 1203, 1269, 1510, 1588, 1627, 1736, 2850, 2918, 2956, 3401 cm⁻¹. HRMS (*m/z*) calcd for C₂₈H₂₈NaO₉: 531.1626; found: 531.1606 [M+Na⁺].

Dibenzyl *O,O'*-(((1*E*,3*Z*,6*E*)-3-hydroxy-5-oxohepta-1,3,6-triene-1,7-diyl)bis(2-methoxy-4,1-phenylene)) disuccinate (7), and benzyl (4-(((1*E*,4*Z*,6*E*)-5-hydroxy-7-(4-hydroxy-3-methoxyphenyl)-3-oxohepta-1,4,6-trien-1-yl)-2-methoxyphenyl) succinate (8)

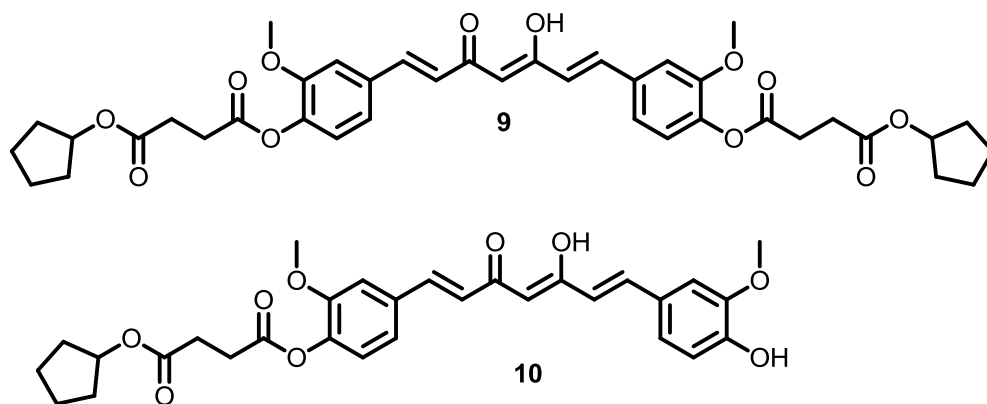


According to GP2, monosuccinate **S2** (64 mg, 1.3 mmol, 10 equiv.), pyridine (3 mL), 4-dimethylaminopyridine (366 mg, 0.39 mmol, 3 equiv.), and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (75 mg, 0.39 mmol, 3 equiv.) were stirred at RT. Concurrently, a solution of curcumin (50 mg, 0.13 mmol) in pyridine (3 mL) was stirred at RT. After 4 h, were combined, and allowed to stir for 48 h at RT. The crude product was purified by HPLC-DAD to obtain diester **7** (21 mg, 21%) and monoester **8** (28 mg, 37%). **Compound 7**: ¹H NMR (400 MHz): 2.81 (4

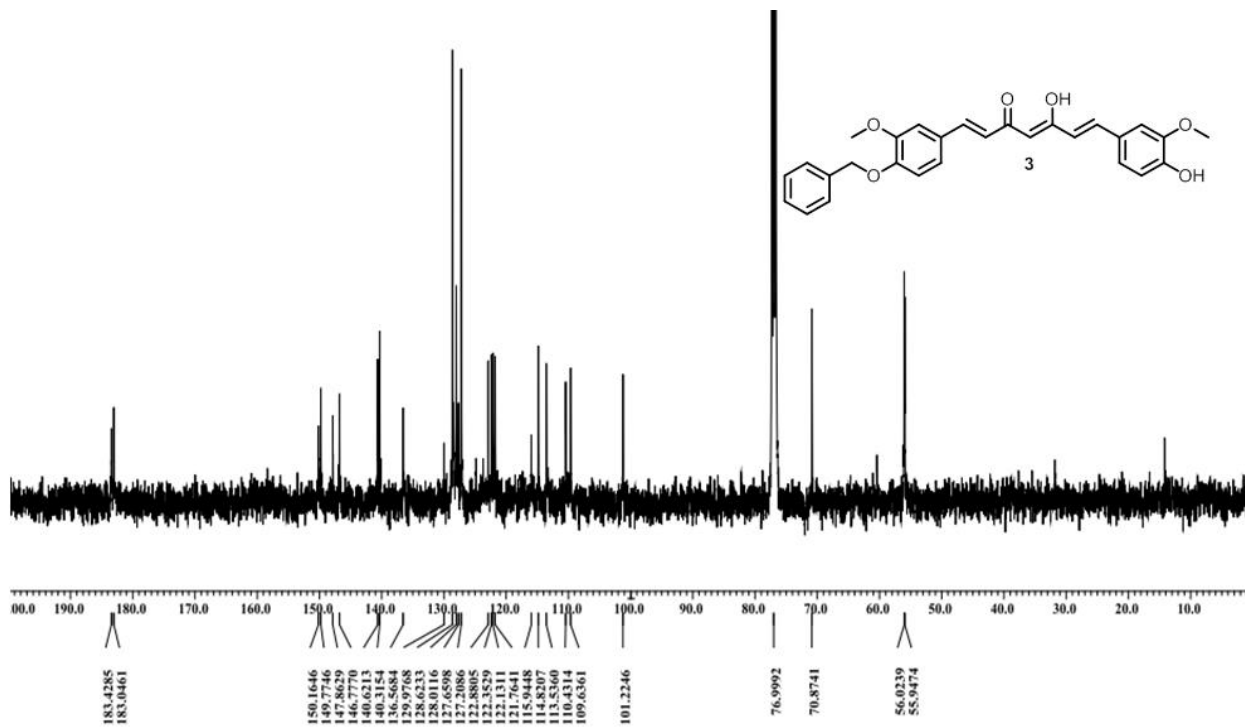
H, t, $J = 13$ Hz), 2.96 (4 H, t, $J = 14$ Hz), 3.85 (6 H, s), 5.17 (4 H, s), 5.86 (1 H, s), 6.56 (2 H, d, $J = 16$ Hz), 7.02 (2 H, d, $J = 8$ Hz), 7.14 (4 H, m), 7.36 (10 H, s), 7.62 (2 H, d, $J = 16$ Hz), 15.8 (1 H, br s) ppm. – ^{13}C NMR (100 MHz): 29.0, 29.2, 55.9, 66.7, 101.8, 111.5, 121.0, 123.3, 124.3, 128.2, 128.3, 128.6, 134.0, 135.7, 140.0, 141.2, 151.4, 170.2, 171.8, 183.1 ppm. – IR: 1131, 1203, 1260, 1465, 1509, 1599, 1736, 1764, 2850, 2910, 2955 cm^{-1} . – HRMS (m/z) calcd for $\text{C}_{43}\text{H}_{40}\text{NaO}_{12}$: 771.2412; found: 771.2391 [$\text{M}+\text{Na}^+$].

Compound 8: ^1H NMR (400 MHz): 2.82 (2 H, t, $J = 14$ Hz), 2.94 (2 H, t, $J = 14$ Hz), 3.84 (3 H, s), 3.94 (3 H, s), 5.20 (2 H, s), 5.83 (1 H, s), 5.93 (1 H, br s), 6.52 (2H, dd, $J = 16, 21$ Hz), 6.93 (1H, d, $J = 8$ Hz), 7.00-7.14 (5 H, m), 7.61 (2 H, dd, $J = 6, 16$ Hz), 15.9 (1H, br s) ppm. – ^{13}C NMR (100 MHz): 29.0, 29.2, 55.9, 56.0, 66.7, 101.5, 109.7, 111.5, 114.9, 120.9, 121.8, 123.0, 123.2, 124.3, 127.6, 128.2, 128.3, 128.6, 134.1, 135.7, 139.4, 141.1, 141.2, 146.8, 148.0, 151.4, 170.2, 171.8, 181.8, 184.5 ppm. – IR: 1031, 1101, 1268, 1510, 1588, 1627, 1736, 2851, 2920, 2955, 3360 cm^{-1} . – HRMS (m/z) calcd for $\text{C}_{32}\text{H}_{30}\text{NaO}_9$: 581.1782; found: 581.1776 [$\text{M}+\text{Na}^+$].

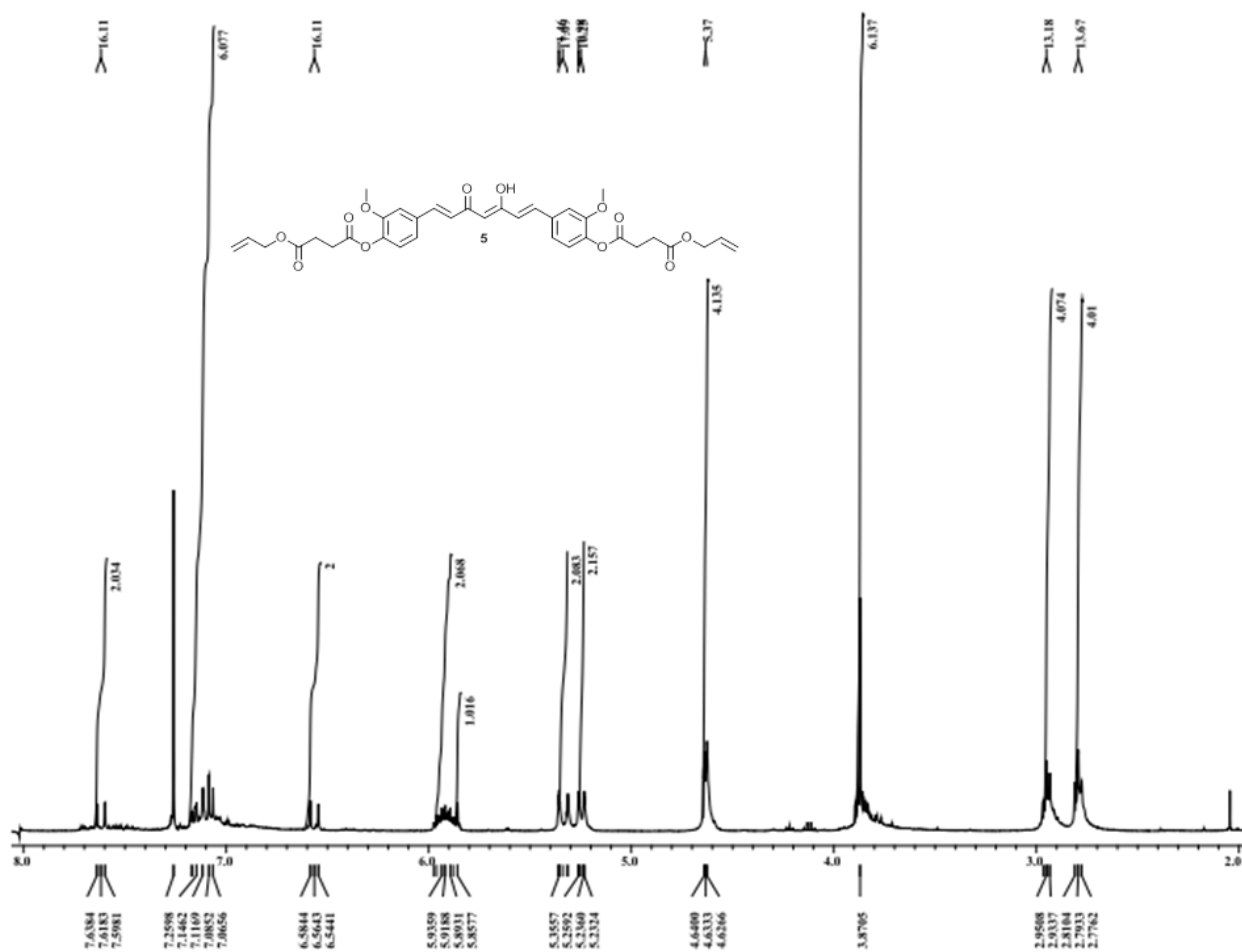
Dicyclopentyl *O,O'*-(((1*E*,3*Z*,6*E*)-3-hydroxy-5-oxohepta-1,3,6-triene-1,7-diyl)bis(2-methoxy-4,1-phenylene)) disuccinate (**9**), and cyclopentyl 4-(((1*E*,4*Z*,6*E*)-5-hydroxy-7-(4-hydroxy-3-methoxyphenyl)-3-oxohepta-1,4,6-trien-1-yl)-2-methoxyphenyl) succinate (**10**).

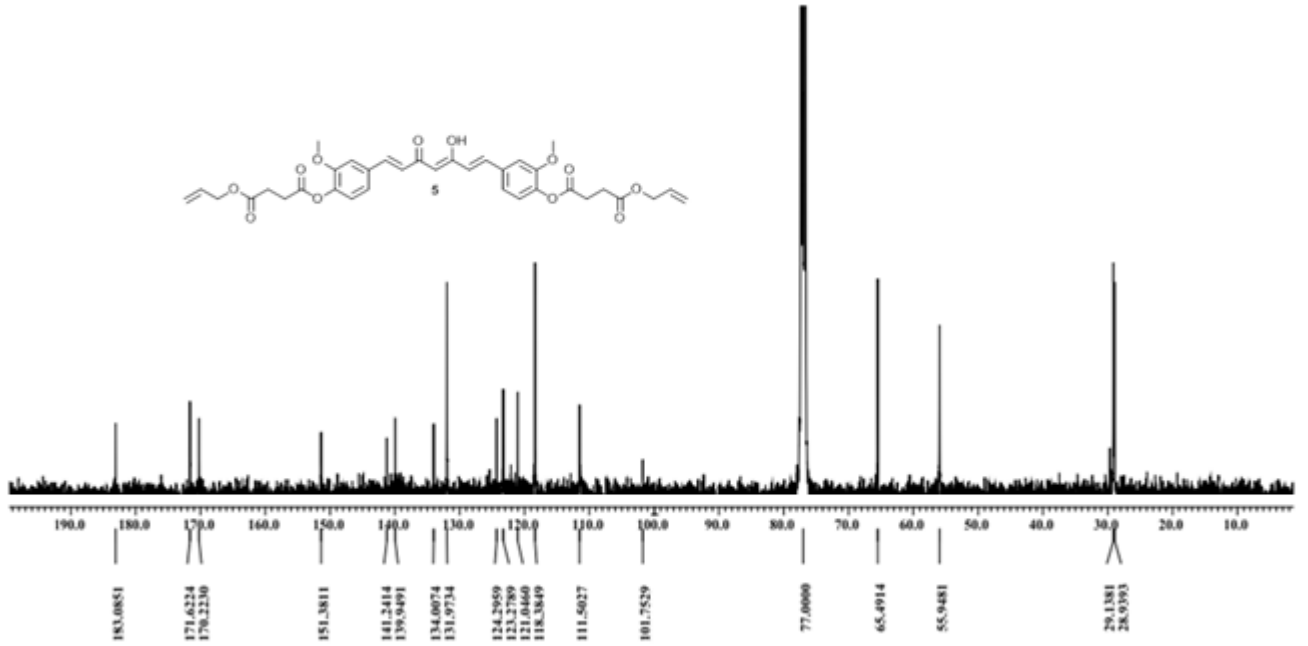
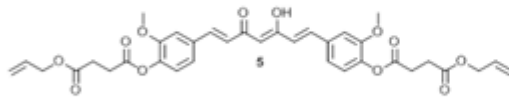


According to GP2, monoester **S3** (245 mg, 1.3 mmol, 10 equiv.), pyridine (3 mL), 4-dimethylaminopyridine (366 mg, 0.39 mmol, 3 equiv.), and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide hydrochloride (75 mg, 0.39 mmol, 3 equiv.) was stirred at RT. Concurrently, a solution of curcumin (50 mg, 0.13 mmol) in pyridine (3 mL) was stirred at RT. After 4 h, the reaction mixtures were combined and allowed to stir for 48 h at RT. The crude product was purified by HPLC to obtain diester **9** (17 mg, 18%) and monoester **10** (25 mg, 34%). **Compound 9**: $^1\text{H NMR}$ (400 MHz): 1.57-1.61 (4 H, m), 1.70-1.71 (8 H, m), 1.84-1.87 (4 H, m), 2.70 (4 H, t, $J = 14$ Hz), 2.92 (4 H, t, $J = 14$ Hz), 3.87 (6 H, s), 5.17-5.22 (2 H, m), 5.85 (1 H, s), 6.56 (2 H, d, $J = 16$ Hz), 7.06-7.17 (6 H, m), 7.60 (2 H, d, $J = 16$ Hz), 15.8 (1 H, br s) ppm. – $^{13}\text{C NMR}$ (100 MHz): 23.7, 29.0, 29.5, 32.6, 55.9, 77.6, 101.8, 111.5, 121.0, 122.1, 123.3, 124.3, 129.2, 134.0, 140.0, 141.3, 151.4, 170.3, 171.7, 183.1 ppm. – IR: 1132, 1157, 1417, 1465, 1509, 1600, 1731, 1765, 2852, 2923, 2956 cm^{-1} . – HRMS (m/z) calcd for $\text{C}_{39}\text{H}_{44}\text{NaO}_{12}$: 727.2725; found: 727.2709 [$\text{M}+\text{Na}^+$]. **Compound 10**: $^1\text{H NMR}$ (400 MHz): 1.57-1.59 (2 H, m), 1.70-1.72 (4 H, m), 1.84-1.87 (2 H, m), 2.71 (2 H, t, $J = 14$ Hz), 2.92 (2 H, t, $J = 14$ Hz), 3.87 (3 H, s), 3.95 (3 H, s), 5.20 (1 H, m), 5.83 (1 H, s), 6.52 (2 H, dd, $J = 16, 22$ Hz), 6.94 (1 H, d, $J = 8$ Hz), 7.06-7.16 (5 H, m), 7.60 (2 H, dd, J

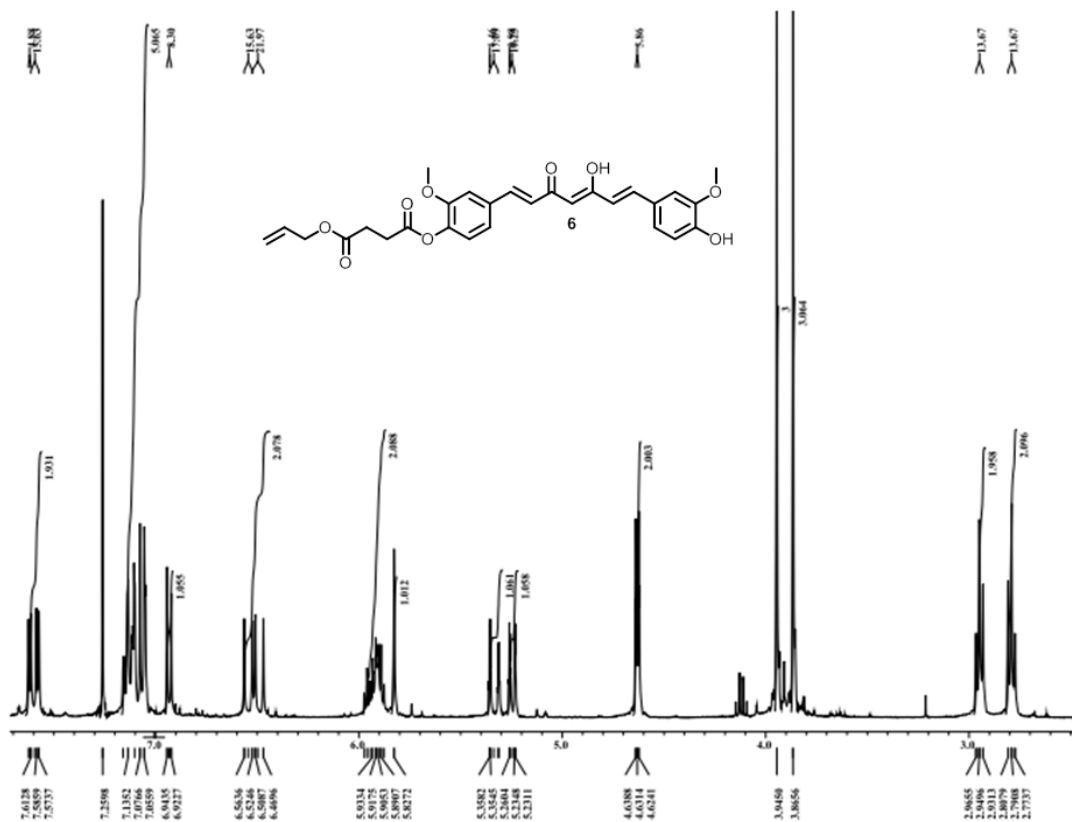


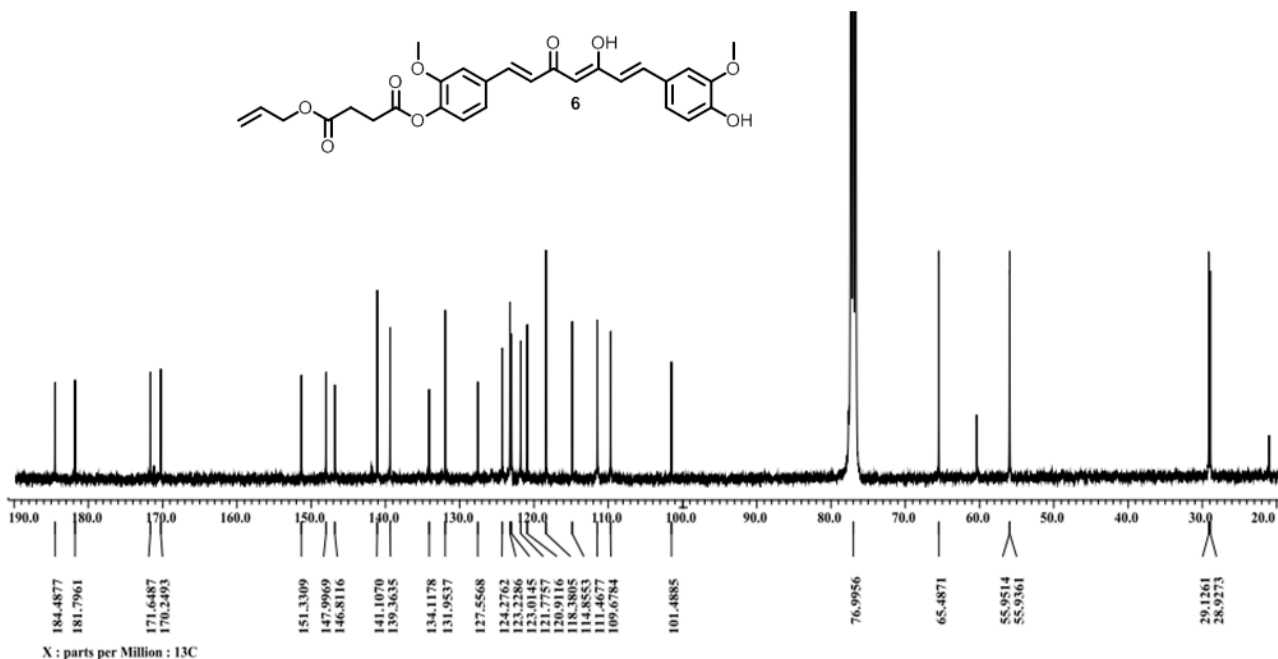
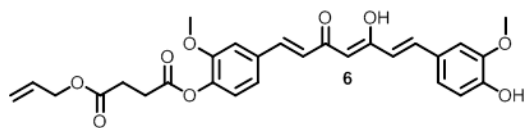
^1H and ^{13}C NMR Spectral Data of diallyl O,O'-(((1E,3Z,6E)-3-hydroxy-5-oxohepta-1,3,6-triene-1,7-diyl)bis(2-methoxy-4,1-phenylene)) disuccinate (5)





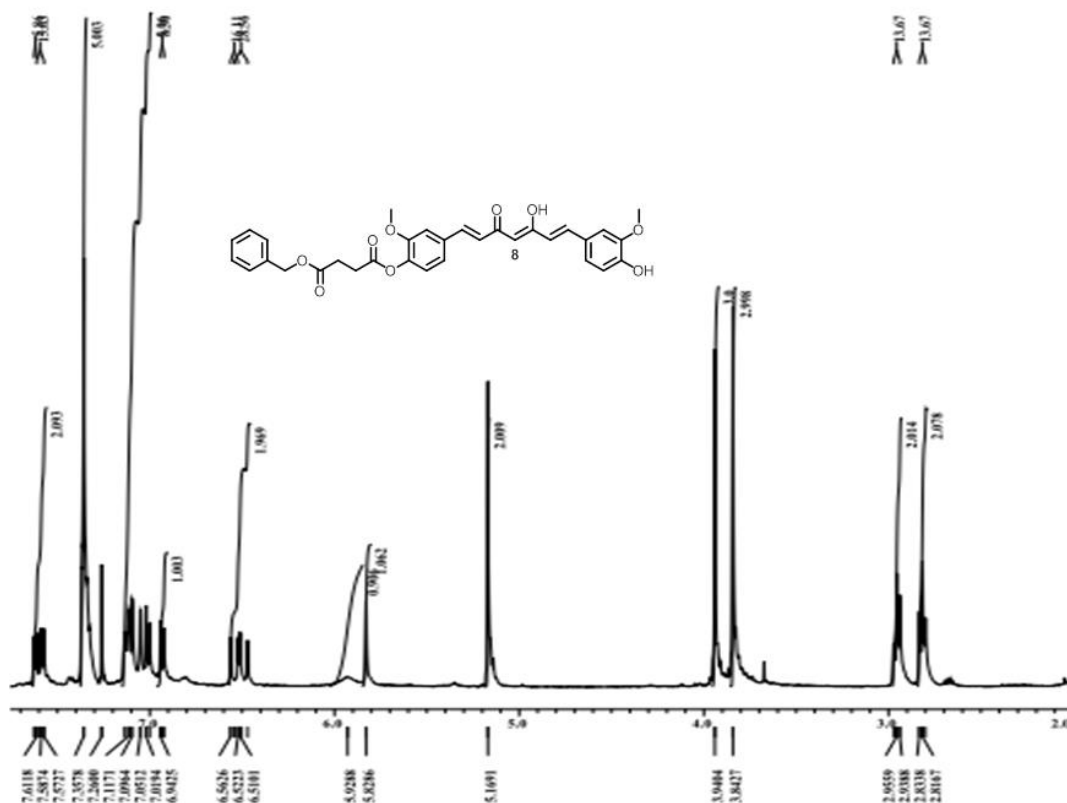
^1H and ^{13}C NMR Spectral Data of allyl (4-((1E,4Z,6E)-5-hydroxy-7-(4-hydroxy-3-methoxyphenyl)-3-oxohepta-1,4,6-trien-1-yl)-2-methoxyphenyl) succinate (6)

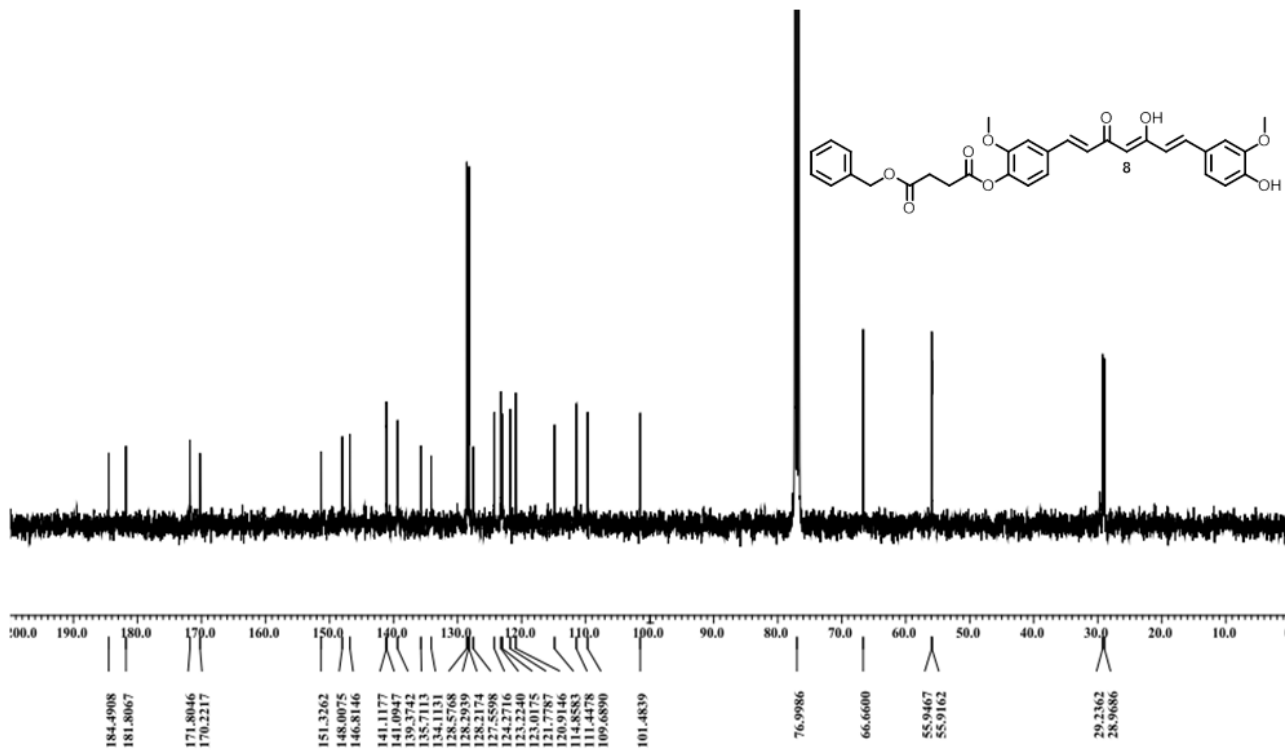




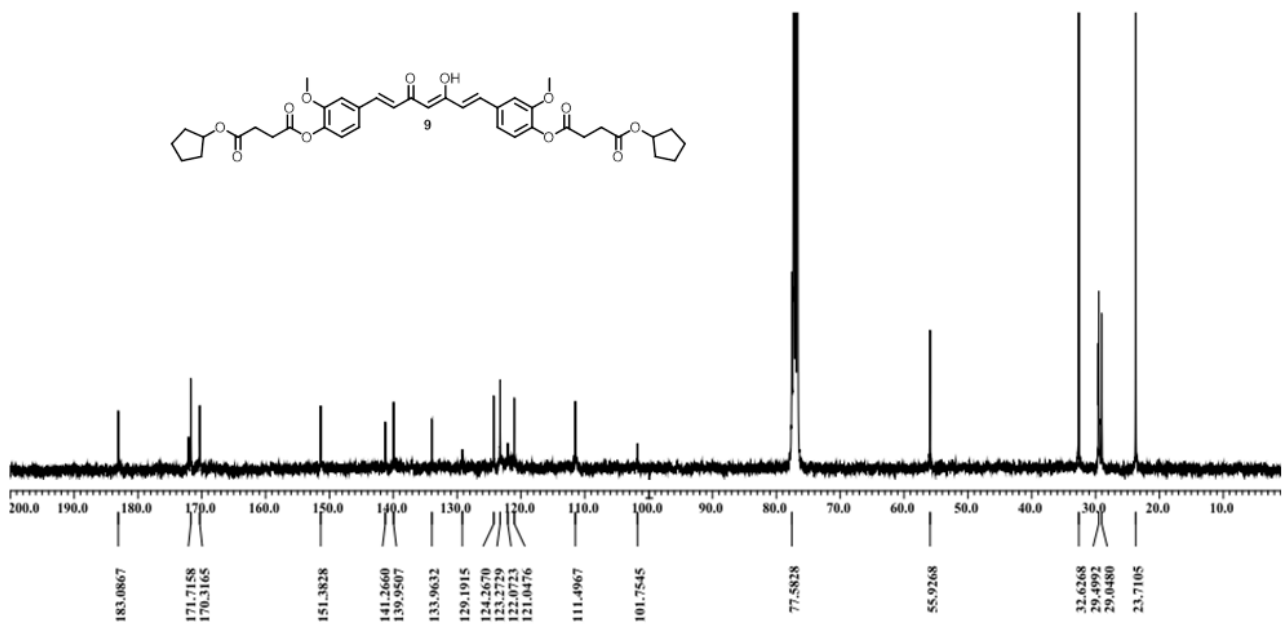
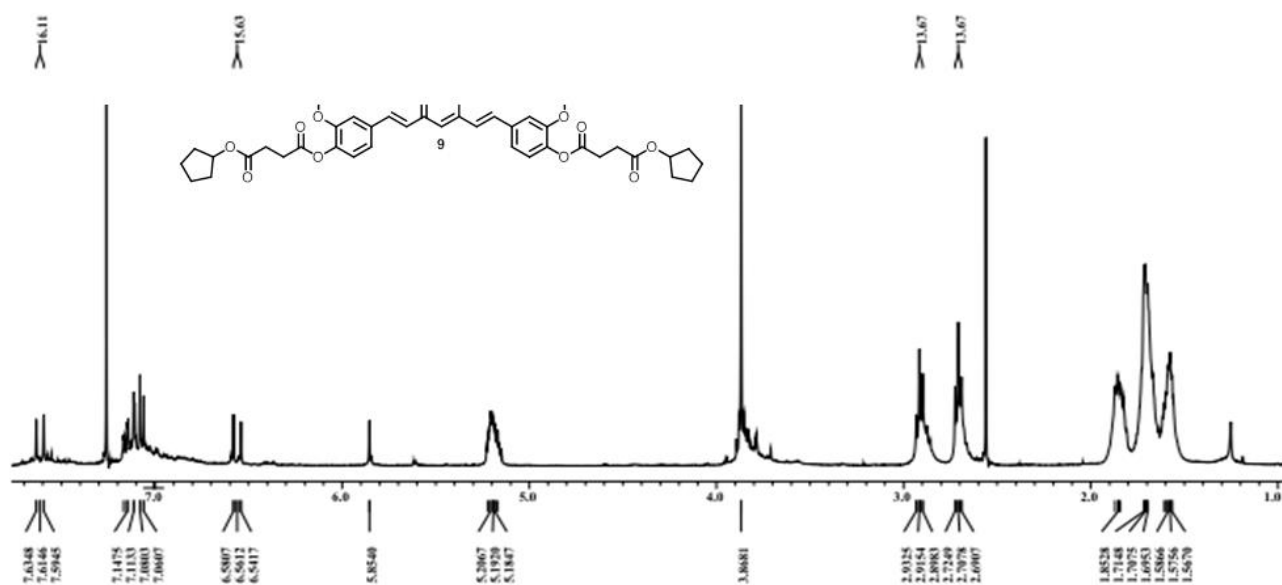
^1H and ^{13}C NMR Spectral Data of dibenzyl O,O'-(((1E,3Z,6E)-3-hydroxy-5-oxohepta-1,3,6-

^1H and ^{13}C NMR Spectral Data of benzyl (4-((1E,4Z,6E)-5-hydroxy-7-(4-hydroxy-3-methoxyphenyl)-3-oxohepta-1,4,6-trien-1-yl)-2-methoxyphenyl) succinate (8)

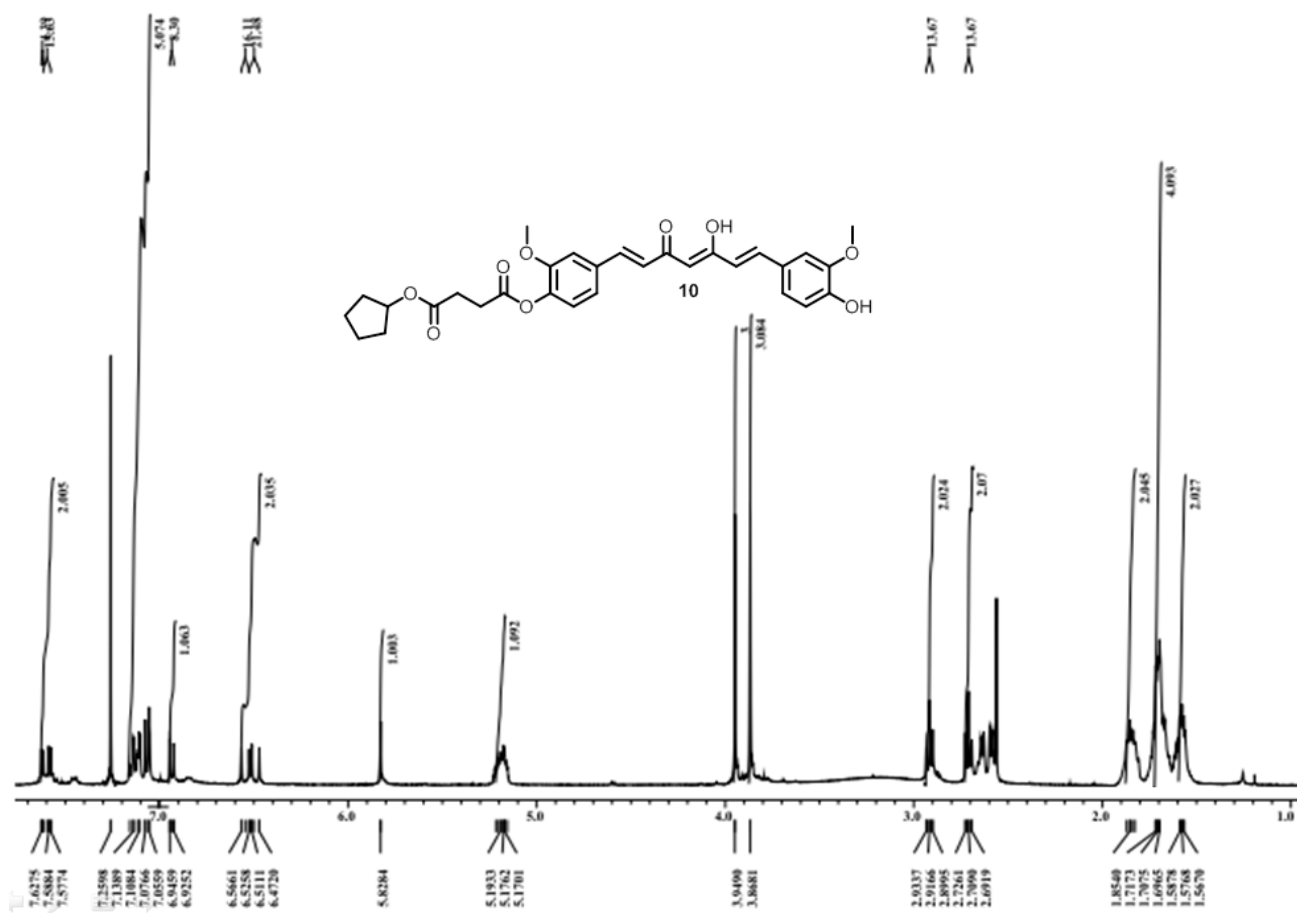


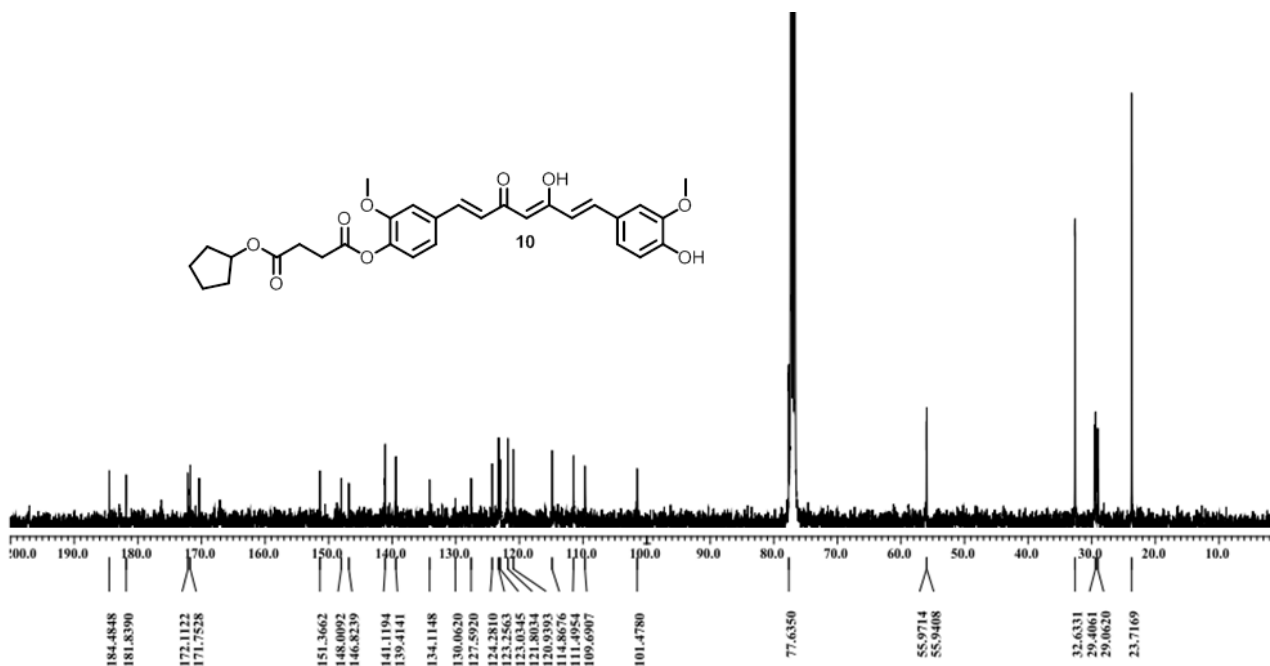
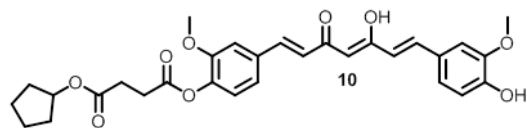


^1H and ^{13}C NMR Spectral Data of dicyclopentyl O,O'-(((1E,3Z,6E)-3-hydroxy-5-oxohepta-1,3,6-triene-1,7-diyl)bis(2-methoxy-4,1-phenylene)) disuccinate (9)



^1H and ^{13}C NMR Spectral Data of cyclopentyl (4-((1E,4Z,6E)-5-hydroxy-7-(4-hydroxy-3-methoxyphenyl)-3-oxohepta-1,4,6-trien-1-yl)-2-methoxyphenyl) succinate (10).





Supplementary References

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