Supporting Informations

Oxidant controlled regioselective mono- and di-functionalizations of coumarins

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**Instrumentation and Chemicals:**

All the reagents were commercial grade and purified according to the established procedures. Organic extracts were dried over anhydrous sodium sulphate. Solvents were removed in a rotary evaporator under reduced pressure. Silica gel (60-120 mesh size) was used for the column chromatography. Reactions were monitored by TLC on silica gel 60 F 254 (0.25 mm). NMR spectra were recorded in CDCl 3 with tetramethylsilane as the internal standard for 1H NMR (400 and 600 MHz), CDCl 3 solvent as the internal standard for 13C NMR (100 and 150 MHz). MS spectra were recorded using ESI mode. IR spectra were recorded in KBr or neat.

Starting materials (3-substituted coumarins) (1-15) are prepared by reacting salicylaldehydes with active methylene compounds (diethyl malonate / ethylcyanoacetate / ethyl acetoacetate etc.) in presence of catalytic amount of piperidine using Knoevenagel condensation.1


**Experimental procedure:**

(A) Synthesis of 3-cyclohexyl-2H-chromen-2-one (1a) from 3-acetyl-2H-chromen-2-one (I) and cyclohexane (a)

To an oven dried 25 mL round bottom flask fitted with reflux condenser, 3-acetylcoumarin (I) (0.047g, 0.25 mmol), di-tert-butylperoxide DTBP (0.146g, 1.0 mmol), Fe(acac)3 (0.004g, 0.013 mmol) and cyclohexane (a) (0.5 mL, 4.6 mmol) were added together in chlorobenzene (1.0 mL) solvent. The reaction mixture was then heated in an oil bath at 110 °C. The progress of the reaction was monitored by TLC and after completion of reaction (12 h) solvents were evaporated under reduced pressure. The reaction mixture was then cooled to room
temperature, admixed with water (2 mL). It was then extracted with ethyl acetate (3 x 10 mL), dried over anhydrous sodium sulphate Na₂SO₄, and evaporated under reduced pressure. The crude product obtained here was further purified over a short column of silica gel (hexane / ethyl acetate, 10:0.1) to give pure 3-cyclohexyl-2H-chromen-2-one (1a) (0.030g, yield 52%). The identity and purity of the product was confirmed by spectroscopic analysis.

(B) Synthesis of 3-Acetyl-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (1a') from 3-acetyl-2H-chromen-2-one (1), tert-butyl hydroperoxide and cyclohexane (a)

To an oven-dried 25 mL round bottom flask fitted with a reflux condenser was added 3-acetylcoumarin (1) (0.047g, 0.25 mmol), decane solution of TBHP (5–6 M) (200 µL, 1.0 mmol), AcOH (30 µL, 0.50 mmol), and cyclohexane (a) (0.5 mL, 4.6 mmol) in chlorobenzene (1.0 mL) solvent. The reaction mixture was heated in an oil bath at 110 °C. After completion (3 h) of the reaction, solvents were evaporated under reduced pressure. The reaction mixture was then cooled to room temperature and admixed with water (2 mL). The product was extracted with ethyl acetate (2 x 10 mL), dried over anhydrous sodium sulphate (Na₂SO₄), and evaporated under reduced pressure. The crude product so obtained was purified over a column of silica gel (hexane / ethyl acetate, 10:0.1) to give pure 3-acetyl-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (1a') (0.058g, yield 65%). The identity and purity of the product was confirmed by spectroscopic analysis.
Crystallographic Description

Crystal data were collected with Bruker Smart Apex-II CCD diffractometer using graphite monochromated MoKα radiation (λ = 0.71073 Å) at 298 K. Cell parameters were retrieved using SMART\(^a\) software and refined with SAINT\(^a\) on all observed reflections. Data reduction was performed with the SAINT software and corrected for Lorentz and polarization effects. Absorption corrections were applied with the program SADABS\(^b\). The structure was solved by direct methods implemented in SHELX-97\(^c\) program and refined by full-matrix least-squares methods on F2. All non-hydrogen atomic positions were located in difference Fourier maps and refined anisotropically. The hydrogen atoms were placed in their geometrically generated positions. colourless crystals were isolated in rectangular shape from acetonitrile at room temperature.


**CCDC number for compound 1c:** CCDC 1412809. This data can be obtained free of charge from the Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/datarequest/cif](http://www.ccdc.cam.ac.uk/datarequest/cif).
Crystallographic description of 1c: Crystal dimension (mm): 0.38 x 0.20 x 0.16. C_{17}H_{20}O_{2}, Mr = 256.33. triclinic, space group p -1; a = 6.7321 (3) Å, b = 9.3720 (4) Å, c = 11.4758 (5) Å; α = 95.498 (3)°, β = 99.490 (3)°, γ = 104.725 (3)°, V = 683.45 (5) Å³ ; Z = 2; ρ_{cal}= 1.246g/cm³; μ (mm⁻¹) = 0.080; F (000) = 276.0; Reflection collected / unique = 2348 / 1909; Refinement method = Full-matrix least-squares on F²; Final R indices [I>2σ₁] R1 = 0.0389, wR2 = 0.0985, R indices (all data) R1 = 0.0480, wR2 = 0.1132; goodness of fit = 1.079.

Table S1. Optimization of reaction parameters

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<th>Entry</th>
<th>Catalyst (mol %)</th>
<th>Additive (equiv.)</th>
<th>Oxidant⁴ (equiv.)</th>
<th>Yield (%)ᵃᵇ</th>
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ᵃReaction conditions: 1 (0.25 mmol), cyclohexane (a) (0.5 mL, 4.6 mmol) in chlorobenzene (1.0 mL) at 110 °C for 12 h.ᵇIsolated yield.ᶜReaction performed for 3 h.⁴Oxidants were used portion wise (four times for both DTBP and TBHP).
Mechanistic investigation:
Trapping of cyclohexyl and benzyl intermediates with TEMPO during monofunctionalization:

An oven-dried 25 mL round bottom flask was charged with 3-acetylcoumarin (1) (0.047g, 0.25 mmol), di-tert-butylperoxide DTBP (0.146g, 1.0 mmol), Fe(acac)₃ (0.004g, 0.013 mmol), TEMPO (A) (0.039 g, 0.25 mmol) and cyclohexane (a) (0.5 mL, 4.6 mmol) in chlorobenzene (1.0 mL). The flask was fitted with a reflux condenser and the reaction mixture was stirred in a preheated oil bath at 110 °C for 12 h. After 12 h of the reaction and usual work up the cyclohexyl-TEMPO adduct 1-(cyclohexyloxy)-2,2,6,6-tetramethylpiperidine (1A) was obtained in 64% isolated yield with no traces of the desired product (1a).

Similar reaction between 3-acetylcoumarin (1) (0.047g, 0.25 mmol) and toluene (a) (0.5 mL, 4.7 mmol) in the presence of radical scavenger TEMPO (A) (0.039 g, 0.25 mmol) under otherwise identical reaction conditions to that of above provided benzyl-TEMPO adduct 1-(benzyloxy)-2,2,6,6-tetramethylpiperidine (1B) in 57% isolated yield with no trace of desired product (1a).

Scheme S 2: Control experiments with radical scavenger TEMPO
Trapping of cyclohexyl intermediate with TEMPO during di-functionalization:

An oven-dried 25 mL round bottom flask was charged with 3-acetylcoumarin (1) (0.047g, 0.25 mmol), decane solution of TBHP (5–6 M) (200 µL, 1.0 mmol), AcOH (30 µL, 0.50 mmol), TEMPO (A) (0.039 g, 0.25 mmol) and cyclohexane (a) (0.5 mL, 4.6 mmol) in chlorobenzene (1.0 mL). The flask was fitted to a reflux condenser and the reaction mixture was stirred in a preheated oil bath at 110 °C. After 3 h of the reaction and usual work up, the cyclohexyl-TEMPO adduct 1-(cyclohexyloxy)-2,2,6,6-tetramethylpiperidine (1A) was obtained in 54% isolated yield along with a trace of the desired product (1a').

Scheme S3: control experiment with radical scavenger TEMPO
Spectral Data:

3-Cyclohexyl-2\textit{H}-chromen-2-one (1a):

Semi-solid; \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 600 MHz): \(\delta\) 7.47–7.44 (m, 3H), 7.31 (d, 1H, \(J = 8.4\) Hz), 7.24 (t, 1H, \(J = 7.2\) Hz), 2.81–2.76 (m, 1H), 1.98 (dd, 2H, \(J = 15.6, 4.2\) Hz), 1.87–1.84 (m, 2H), 1.80–1.77 (m, 1H), 1.49–1.41 (m, 2H), 1.34–1.24 (m, 3H); \textsuperscript{13}C NMR (CDCl\textsubscript{3}, 150 MHz): \(\delta\) 161.8, 152.9, 136.5, 135.2, 130.2, 127.5, 124.4, 119.9, 116.5, 38.4, 32.3, 26.7, 26.4; IR (KBr, cm\textsuperscript{-1}): 3058, 2928, 2850, 1710, 1652, 1632, 1609, 1488, 1455, 1387, 1277, 1255, 1234, 1184, 1173, 1136, 1064, 1042, 986, 956, 924, 890, 781, 754; HRMS (ESI) calcd for C\textsubscript{15}H\textsubscript{16}O\textsubscript{2} (M + H\textsuperscript{+}) 229.1229, found 229.1224.

3-Cyclohexyl-7-methoxy-2\textit{H}-chromen-2-one (2a):

Semi-solid; \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 600 MHz): \(\delta\) 7.38 (s, 1H), 7.33 (d, 1H, \(J = 8.4\) Hz), 6.83–6.79 (m, 2H), 3.85 (s, 3H), 2.75–2.71 (m, 1H), 1.96 (d, 2H, \(J = 12.0\) Hz), 1.86–1.82 (m, 2H), 1.78–1.75 (m, 1H), 1.45–1.40 (m, 2H), 1.30–1.26 (m, 3H); \textsuperscript{13}C NMR (CDCl\textsubscript{3}, 150 MHz): \(\delta\) 162.1, 161.9, 154.6, 136.6, 131.2, 128.4, 113.5, 112.5, 100.6, 55.9, 38.2, 32.4, 26.8, 26.4; IR (KBr, cm\textsuperscript{-1}): 2959, 2924, 2850, 1701, 1652, 1628, 1593, 1572, 1464, 1433, 1403, 1297, 1265, 1158, 1138, 1034, 964, 937, 774; HRMS (ESI) calcd for C\textsubscript{16}H\textsubscript{18}O\textsubscript{3} (M + H\textsuperscript{+}) 259.1335, found 259.1327.

3-Cyclohexyl-8-ethoxy-2\textit{H}-chromen-2-one (3a):

Semi-solid; \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 600 MHz): \(\delta\) 7.41 (s, 1H), 7.14 (t, 1H, \(J = 8.4\) Hz), 6.99 (d, 2H, \(J = 7.2\) Hz), 4.19–4.16 (q, 2H, \(J = 7.2\) Hz), 2.81–2.76 (m, 1H), 1.97 (d, 2H, \(J = 12.6\) Hz), 1.86–1.82 (m, 2H), 1.78–1.76 (m, 1H), 1.48 (t, 3H, \(J = 10.0\) Hz), 1.46–1.41 (m,
2H), 1.32–1.23 (m, 3H); ¹³C NMR (CDCl₃, 150 MHz): δ 161.3, 146.5, 142.8, 136.6, 135.3, 124.2, 120.7, 118.9, 113.9, 65.1, 38.3, 32.3, 26.7, 26.4, 14.9; IR (KBr, cm⁻¹): 2977, 2926, 2852, 1719, 1608, 1579, 1471, 1449, 1392, 1355, 1276, 1181, 1114, 1098, 1064, 1044, 982, 770, 731; HRMS (ESI) calcd for C₁₇H₂₀O₃ (M + H⁺) 273.1492, found 273.1485.

6,8-Di-tert-butyl-3-cyclohexyl-2H-chromen-2-one (4a):

Semi-solid; ¹H NMR (CDCl₃, 600 MHz): δ 7.49 (d, 1H, J = 2.4 Hz), 7.43 (s, 1H), 7.26 (s, 1H), 2.79–2.76 (m, 2H), 1.97 (d, 2H, J = 12.0 Hz), 1.86–1.83 (m, 2H), 1.78–1.76 (m, 1H), 1.51 (s, 9H), 1.46–1.42 (m, 3H), 1.35 (s, 9H), 1.33–1.29 (m, 2H); ¹³C NMR (CDCl₃, 150 MHz): δ 161.6, 149.6, 146.5, 137.8, 137.0, 133.8, 125.8, 122.2, 119.7, 38.1, 35.2, 34.8, 32.4, 31.6, 30.1, 26.7, 26.4; IR (KBr, cm⁻¹): 2961, 2929, 2853, 1711, 1586, 1477, 1445, 1393, 1363, 1243, 1217, 1170, 1135, 1068, 1032, 1004, 983, 939, 888, 783; HRMS (ESI) calcd for C₂₃H₃₂O₂ (M + H⁺) 341.2482, found 341.2470.

3-Cyclohexyl-6-nitro-2H-chromen-2-one (5a):

Semi-solid; ¹H NMR (CDCl₃, 600 MHz): δ 8.39 (d, 1H, J = 2.4 Hz), 8.33 (d, 1H, J = 9.6 Hz), 7.51 (s, 1H), 7.42 (d, 1H, J = 9.0 Hz), 2.82–2.77 (m, 1H), 1.99 (d, 2H, J = 12.0, 4.2 Hz), 1.88 (dt, 2H, J = 13.2, 3.0 Hz), 1.80 (d, 1H, J = 13.2 Hz), 1.49–1.42 (m, 2H), 1.35–1.27 (m, 3H); ¹³C NMR (CDCl₃, 150 MHz): δ 160.1, 156.5, 144.2, 137.7, 135.2, 125.5, 123.4, 119.9, 117.6, 38.8, 32.2, 26.6, 26.2; IR (KBr, cm⁻¹): 2927, 2855, 1728, 1652, 1632, 1617, 1529, 1486, 1342, 1270, 1169, 1090, 1060, 1041, 983, 935, 844, 836, 749, 668; HRMS (ESI) calcd for C₁₃H₁₅NO₄ (M + H⁺) 274.1081, found 274.1074.
6,8-Dichloro-3-cyclohexyl-2H-chromen-2-one (6a):

Semi-solid; $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 7.50 (d, 1H, $J$ = 2.4 Hz), 7.35–7.34 (m, 2H), 2.81–2.75 (m, 1H), 1.98–1.95 (m, 2H), 1.88–1.77 (m, 3H), 1.50–1.39 (m, 2H), 1.33–1.19 (m, 3H); $^{13}$C NMR (CDCl$_3$, 150 MHz): $\delta$ 159.9, 147.3, 137.4, 134.9, 130.6, 129.4, 125.4. 122.3, 121.7, 38.6, 32.2, 26.6, 26.3; IR (KBr, cm$^{-1}$): 2927, 2853, 1733, 1660, 1635, 1564, 1452, 1352, 1172, 1135, 1063, 992, 888, 859, 830, 778, 736; HRMS (ESI) calcd for C$_{15}$H$_{14}$Cl$_2$O$_2$ (M + H$^+$) 297.0450, found 297.0442.

3-Cyclohexyl-6-nitro-2H-chromen-2-one (7a):

Semi-solid; $^1$H NMR (CDCl$_3$, 600 MHz): $\delta$ 7.79 (s, 1H), 7.53 (s, 1H), 7.31 (s, 1H), 2.80–2.76 (m, 1H), 1.96 (d, 2H, $J$ = 12.0 Hz), 1.86–1.84 (m, 2H), 1.79–1.76 (m, 1H), 1.45–1.41 (m, 2H), 1.35–1.27 (m, 3H); $^{13}$C NMR (CDCl$_3$, 150 MHz): $\delta$ 160.0, 148.9, 137.3, 136.1, 134.8, 129.1, 122.2, 116.8, 111.0, 38.6, 32.2, 26.6, 26.3; IR (KBr, cm$^{-1}$): 2964, 2922, 2846, 1735, 1658, 1641, 1635, 1448, 1260, 1020, 796; HRMS (ESI) calcd for C$_{15}$H$_{14}$Br$_2$O$_2$ (M + H$^+$) 384.9433, found 384.9424.

2-Cyclohexyl-3H-benzof[chromen-3-one (8a):

Semi-solid; $^1$H NMR (CDCl$_3$, 600 MHz): $\delta$ 8.28 (d, 1H, $J$ = 8.4 Hz), 8.24 (s, 1H), 7.91 (t, 2H, $J$ = 8.4 Hz), 7.67 (t, 1H, $J$ = 7.8 Hz), 7.55 (t, 1H, $J$ = 7.8 Hz), 7.45 (d, 1H, $J$ = 9.0 Hz), 2.88–2.86 (m, 1H), 2.06 (d, 2H, $J$ = 12.0 Hz), 1.91–1.89 (m, 2H), 1.81 (d, 1H, $J$ = 13.2 Hz), 1.51–1.42 (m, 4H), 1.34–1.27 (m, 1H); $^{13}$C NMR (CDCl$_3$, 150 MHz): $\delta$ 161.8, 152.2, 134.3, 132.2, 131.8, 130.5, 129.2, 128.0, 125.9, 121.7, 116.9, 113.8, 38.8, 32.5, 26.8, 26.4; IR (KBr, cm$^{-1}$): 3066, 2921, 2849, 1705, 1635, 1591, 1517, 1448, 1438, 1236, 1213, 1171, 1135, 1088, 1075, 1044, 986, 882, 813,
782, 738; HRMS (ESI) calcd for C₁₉H₁₈O₂ (M + H⁺) 279.1386, found 279.1389

3-Cyclopentyl-2H-chromen-2-one (1b):

Semi-solid; ¹H NMR (CDCl₃, 400 MHz): δ 7.50 (s, 1H), 7.45 (m, 2H), 7.31 (m, 1H), 7.26–7.23 (m, 1H), 3.20–3.11 (m, 1H), 2.14–2.06 (m, 2H), 1.85–1.76 (m, 4H), 1.73–1.53 (m, 2H); ¹³C NMR (CDCl₃, 150 MHz): δ 161.9, 153.1, 136.2, 133.7, 130.6, 127.4, 124.4, 119.8, 116.5, 40.9, 31.9, 25.3; IR (KBr, cm⁻¹): 2955, 2872, 1722, 1630, 1454, 1276, 1224, 1173, 1121, 1057, 1022, 948, 922, 755; HRMS (ESI) calcd for C₁₄H₁₄O₂ (M + H⁺) 215.1073, found 215.1064.

3-Cyclooctyl-2H-chromen-2-one (1c):

Crystalline needles; m.p. 85-86 °C; ¹H NMR (CDCl₃, 600 MHz): δ 7.46 (s, 1H), 7.44 (d, 2H, J = 6.0 Hz), 7.29 (d, 1H, J = 8.4 Hz), 7.24 (t, 1H, J = 7.5 Hz), 3.10–3.03 (m, 1H), 1.84–1.82 (m, 2H), 1.80–1.73 (m, 4H), 1.72–1.64 (m, 8H); ¹³C NMR (CDCl₃, 150 MHz): δ 161.7, 152.9, 139.8, 136.5, 130.5, 127.4, 124.3, 119.8, 116.4, 38.2, 31.8, 26.9, 26.4, 25.8; IR (KBr, cm⁻¹): 3057, 2963, 2922, 2856, 1717, 1627, 1608, 1489, 1473, 1462, 1454, 1388, 1360, 1281, 1274, 1258, 1230, 1182, 1274, 1258, 1230, 1181, 1174, 1146, 1124, 1058, 1011, 1027, 1000, 971, 956, 932, 923, 872, 853, 830, 777, 754, 733; HRMS (ESI) calcd for C₁₇H₂₀O₂ (M + H⁺) 257.1543, found 257.1539.

3-Benzyl-2H-chromen-2-one (1d):

Semi-solid; ¹H NMR (CDCl₃, 600 MHz): δ 7.44 (t, 1H, J = 7.2 Hz), 7.34–7.31 (m, 2H), 7.29–7.24 (m, 5H), 7.19 (t, 2H, J = 7.2 Hz), 3.86 (s, 2H); ¹³C NMR (CDCl₃, 150 MHz): δ 161.9, 153.3, 139.5, 137.8, 130.9, 130.0, 129.6, 128.9, 127.6, 127.0, 124.5,
119.6, 116.6, 36.8; IR (KBr, cm\(^{-1}\)): 3022, 2988, 2917, 2841, 1712, 1631, 1608, 1489, 1455, 1434, 1387, 1224, 147, 1123, 1075, 1051, 1028, 956, 930, 825, 753, 731, 726, 700; HRMS (ESI) calcd for C\(_{16}H_{12}O_2\) (M + H\(^+\)) 237.0917, found 237.0920.

3-(4-Methylbenzyl)-2\(H\)-chromen-2-one (1e):

Semi-solid; \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.44 (t, 1H, \(J = 7.4\) Hz), 7.35–7.29 (m, 2H), 7.25–7.23 (m, 1H), 7.21–7.14 (m, 5H), 3.82 (s, 2H), 2.34 (s, 3H); \(^{13}\)C NMR (CDCl\(_3\), 100 MHz): \(\delta\) 161.2, 153.2, 139.3, 136.6, 134.7, 130.9, 129.9, 129.7, 129.5, 127.6, 124.4, 119.7, 116.6, 36.3, 21.3; IR (KBr, cm\(^{-1}\)): 2956, 2923, 2846, 1709, 1631, 1608, 1533, 1513, 1487, 1455, 1388, 1287, 1230, 1125, 1050, 956, 786, 752, 722; HRMS (ESI) calcd for C\(_{17}H_{14}O_2\) (M + H\(^+\)) 251.1073, found 251.1086.

3-(3,5-Dimethylbenzyl)-2\(H\)-chromen-2-one (1f):

Semi-solid; \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.39 (t, 1H, \(J = 7.4\) Hz), 7.30 (d, 1H, \(J = 7.6\) Hz), 7.25–7.19 (m, 2H), 7.15 (t, 1H, \(J = 7.2\) Hz), 6.83 (s, 3H), 3.75 (s, 2H), 2.24 (s, 6H); \(^{13}\)C NMR (CDCl\(_3\), 150 MHz): \(\delta\) 161.9, 153.3, 139.4, 138.5, 137.7, 130.9, 129.8, 128.7, 127.6, 127.4, 124.4, 119.7, 116.6, 36.5, 21.5; IR (KBr, cm\(^{-1}\)): 3043, 3014, 2995, 2919, 2849, 1714, 1632, 1606, 487, 1456, 1429, 1386, 1257, 1232, 1196, 1163, 1124, 1055, 958, 923, 854, 837, 753, 704; HRMS (ESI) calcd for C\(_{18}H_{16}O_2\) (M + H\(^+\)) 265.1229, found 265.1220.

3-Acetyl-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (1a'):

Solid; m.p. 180–181 °C; \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta\) 7.24–7.19 (m, 1H), 7.06–6.99 (m, 2H), 6.96 (d, 1H, \(J = 8.0\) Hz), 2.90 (d, 1H, \(J = 4.2\) Hz), 2.40 (s, 3H), 1.69–1.44 (m, 6H), 1.24–1.04 (m, 3H), 0.94 (s, 9H), 0.84–0.73 (m, 1H), 0.56–0.46 (m, 1H); \(^{13}\)C NMR
(CDCl₃, 150 MHz): δ 203.4, 161.2, 151.1, 130.2, 128.6, 123.9, 121.1, 116.3, 86.0, 82.3, 48.9, 38.4, 32.3, 27.1, 26.9, 26.7, 26.2, 25.9, 25.8; IR (KBr, cm⁻¹): 2990, 2972, 2935, 2952, 1770, 1714, 1611, 1587, 1499, 1462, 1442, 1420, 1390, 1365, 1352, 1301, 1260, 1247, 1198, 1174, 1124, 1068, 1015, 918, 876, 776, 768; HRMS (ESI) calcd for C₂₁H₂₈O₅ (M + Na⁺) 383.1834, found 383.1842.

3-Acetyl-3-(tert-butyldperoxy)-6-chloro-4-cyclohexylchroman-2-one (13a):

Solid; m.p. 168-170 °C; ¹H NMR (CDCl₃, 400 MHz): δ 7.22–7.18 (m, 1H), 7.00 (d, 1H, J = 2.4 Hz), 6.91 (d, 1H, J = 8.8 Hz), 2.87 (d, 1H, J = 2.8 Hz), 2.40 (s, 3H), 1.66–1.63 (m, 2H), 1.54–1.47 (m, 4H), 1.22–1.04 (m, 3H), 0.96 (s, 9H), 0.92–0.78 (m, 1H), 0.55–0.45 (m, 1H); ¹³C NMR (CDCl₃, 100 MHz): δ 203.0, 160.5, 149.7, 129.8, 128.9, 128.6, 122.8, 117.5, 85.5, 82.5, 48.7, 38.2, 32.2, 27.0, 26.8, 26.5, 26.2, 25.8, 25.7; IR (KBr, cm⁻¹): 2984, 2942, 2921, 2853, 1790, 1725, 1482, 1455, 1415, 1367, 1351, 1273, 1260, 1226, 1217, 1194, 1180, 1148, 1200, 1104, 1083, 1066, 1041, 1034, 930, 911, 896, 891, 882, 870, 848, 812, 787, 759, 745, 707; HRMS (ESI) calcd for C₂₁H₂₇ClO₅ (M + Na⁺) 417.1445, found 417.1436.

3-Acetyl-6-bromo-3-(tert-butyldperoxy)-4-cyclohexylchroman-2-one (14a):

Solid; m.p. 155-158 °C; ¹H NMR (CDCl₃, 400 MHz): δ 7.40 (dd, 1H, J₁ = 8.0 Hz, J₂ = 2.4 Hz), 7.21 (d, 1H, J = 2.4 Hz), 6.92 (d, 1H, J = Hz), 2.93 (d, 1H, J = 2.8 Hz), 2.46 (s, 3H), 1.73–1.69 (m, 2H), 1.61–1.53 (m, 4H), 1.25–1.11 (m, 3H), 1.02 (s, 9H), 0.91–0.86 (m, 1H), 0.58–0.55 (m, 1H); ¹³C NMR (CDCl₃, 150 MHz): δ 202.2, 160.6, 150.3, 132.7, 131.7, 131.2, 123.4, 118.9, 117.9, 85.6, 82.6, 48.7, 38.3, 32.3, 27.1, 26.9, 26.6, 26.3, 25.9, 25.7; IR (KBr, cm⁻¹): 2940, 2926, 2850, 1791, 1700, 1697, 1681, 1650, 1633, 1560,
1478, 1450, 1412, 1389, 1364, 1260, 1245, 1224, 1175, 1153, 1092, 1026, 912, 891, 873, 858, 810, 775; HRMS (ESI) calcd for C_{21}H_{27}BrO_5 (M + Na^+) 461.0940, found 461.0932.

3-Acetyl-3-(tert-butylperoxy)-4-cyclohexyl-6-nitrochroman-2-one (5a'):

Semi-solid; \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta \) 8.19 (dd, 1H, \(J_1 = 8.8\) Hz, \(J_2 = 2.4\) Hz), 7.99 (d, 1H, \(J = 2.8\) Hz), 7.15 (d, 1H, \(J = 8.8\) Hz), 3.07 (d, 1H, \(J = 2.8\) Hz), 2.47 (s, 3H), 1.71–1.51 (m, 6H), 1.27–1.05 (m, 3H), 0.99 (s, 9H), 0.88–0.79 (m, 1H), 0.54–0.44 (m, 1H); \(^13\)C NMR (CDCl\(_3\), 100 MHz): \(\delta \) 202.4, 159.6, 155.5, 143.8, 125.7, 124.7, 122.6, 117.1, 85.2, 82.8, 48.8, 38.2, 32.1, 27.0, 26.9, 26.4, 25.8, 25.6; IR (KBr, cm\(^{-1}\)): 3092, 2984, 2924, 2850, 1798, 1720, 1586, 1559, 1525, 1483, 1450, 1433, 1392, 1367, 1341, 1308, 1260, 1233, 1219, 1184, 1150, 1112, 1087, 1066, 1044, 1029, 1010, 967, 939, 915, 902, 868, 841, 812, 777, 753, 752, 740; HRMS (ESI) calcd for C\(_{21}\)H\(_{27}\)NO\(_7\) (M + Na\(^+\)) 428.1685, found 428.1676.

3-Acetyl-3-(tert-butylperoxy)-6,8-dichloro-4-cyclohexylchroman-2-one (6a'):

Semi-solid; \(^1\)H NMR (CDCl\(_3\), 400 MHz): \(\delta \) 7.38 (s, 1H), 6.98 (s, 1H), 2.95 (d, 1H, \(J = 2.4\) Hz), 2.47 (s, 3H), 1.73–1.70 (m, 2H), 1.63–1.55 (m, 4H), 1.25–1.12 (m, 3H), 1.04 (s, 9H), 0.95–0.88 (m, 1H), 0.62–0.54 (m, 1H); \(^13\)C NMR (CDCl\(_3\), 100 MHz): \(\delta \) 202.5, 159.4, 145.7, 129.0, 128.8, 128.2, 124.3, 122.1, 85.2, 82.6, 49.0, 38.0, 32.2, 27.0, 26.9, 26.4, 26.0, 25.7, 25.6; IR (KBr, cm\(^{-1}\)): 3084, 2979, 2928, 2852, 1795, 1721, 1460, 1420, 1363, 1262, 1249, 1212, 1182, 1154, 1123, 1100, 1070, 1044, 1014, 973, 937, 917, 900, 881, 870, 855, 825, 786, 754, 746; HRMS (ESI) calcd for C\(_{21}\)H\(_{26}\)Cl\(_2\)O\(_5\) (M + Na\(^+\)) 451.1055, found 451.1049.
3-Acetyl-6,8-dibromo-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (7a’):

Semi-solid; $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 7.64 (d, 1H, $J = 2.4$ Hz), 7.13 (d, 1H, $J = 2.0$ Hz), 2.90 (d, 1H, $J = 3.2$ Hz), 2.44 (s, 3H), 1.70–1.48 (m, 6H), 1.24–1.09 (m, 3H), 1.01 (s, 9H), 0.92–0.86 (m, 1H), 0.55–0.51 (m, 1H); $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta$ 202.6, 159.5, 147.4, 134.7, 131.7, 124.7, 116.4, 111.0, 85.3, 82.7, 49.2, 38.1, 32.3, 27.2, 27.1, 26.8, 26.5, 26.2, 25.8, 25.6; IR (KBr, cm$^{-1}$): 2960, 2926, 2856, 1800, 1725, 1683, 1643, 1565, 1560, 1449, 1413, 1365, 1254, 1190, 1148, 1095, 1026, 931, 865, 800; HRMS (ESI) calcd for C$_{21}$H$_{26}$Br$_2$O$_5$ (M + Na$^+$) 539.0039, found 539.0027.

3-Acetyl-3-(tert-butylperoxy)-4-cyclohexyl-7-methoxychroman-2-one (2a’):

Semi-solid; $^1$H NMR (CDCl$_3$, 600 MHz): $\delta$ 6.94 (d, 1H, $J = 8.4$ Hz), 6.64 (dd, 1H, $J_1 = 9.0$ Hz, $J_2 = 3.0$ Hz), 6.56 (d, 1H, $J = 2.4$ Hz), 3.78 (s, 3H), 2.89 (d, 1H, $J = 3.0$ Hz), 2.42 (s, 3H), 1.70–1.64 (m, 2H), 1.58–1.47 (m, 4H), 1.24–1.10 (m, 3H), 1.00 (s, 9H), 0.86–0.84 (m, 1H), 0.57–0.56 (m, 1H); $^{13}$C NMR (CDCl$_3$, 150 MHz): $\delta$ 203.4, 161.2, 159.9, 151.9, 130.7, 112.9, 109.9, 101.9, 86.1, 82.3, 55.7, 48.3, 38.5, 32.3, 27.0, 26.8, 26.5, 26.3, 25.9, 25.8; IR (KBr, cm$^{-1}$): 2990, 2970, 2928, 2850, 1776, 1722, 1624, 1585, 1559, 1507, 1453, 1432, 1364, 1321, 1285, 1277, 1238, 1210, 1189, 1156, 1124, 1090, 1045, 1030, 922, 902, 886, 870, 859, 838, 806, 792, 763, 747, 712; HRMS (ESI) calcd for C$_{22}$H$_{30}$O$_6$ (M + Na$^+$) 413.1940, found 413.1944.

3-Acetyl-3-(tert-butylperoxy)-4-cyclohexyl-8-methoxychroman-2-one (15a’):

Semi-solid; $^1$H NMR (CDCl$_3$, 600 MHz): $\delta$ 7.00 (d, 1H, $J = 7.8$ Hz), 6.85 (d, 1H, $J = 7.8$ Hz), 6.61 (d, 1H, $J = 7.2$ Hz), 3.83 (s, 3H), 2.92 (d, 1H, $J = 3.0$ Hz), 2.42 (s, 3H), 1.71–1.47 (m, 6H), 1.23–1.08 (m, 3H), 0.96 (s, 9H), 0.86–0.80 (m, 1H), 0.63–0.58 (m,
1H); $^{13}$C NMR (CDCl$_3$, 150 MHz): $\delta$ 203.5, 160.6, 147.1, 140.4, 123.7, 122.2, 121.7, 111.4, 85.8, 82.2, 56.2, 48.9, 38.2, 32.4, 27.2, 27.0, 26.6, 26.0, 25.9, 25.8; IR (KBr, cm$^{-1}$): 2982, 2930, 2856, 1780, 1724, 1620, 1590, 1554, 1504, 1485, 1460, 1366, 1320, 1304, 1273, 1250. 1182, 1160, 1107, 1062, 1016, 971, 913, 866, 804, 785, 732; HRMS (ESI) calcd for C$_{22}$H$_{30}$O$_6$ (M + Na$^+$) 413.1940, found 413.1931.

2-Acetyl-2-(tert-butylperoxy)-1-cyclohexyl-1H-benzo[f]chromen-3(2H)-one (8a’):

Solid; m.p. 156-160 °C; $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 7.90 (d, 1H, $J = 8.8$ Hz), 7.86 (d, 1H, $J = 8.0$ Hz), 7.78 (d, 1H, $J = 8.8$ Hz), 7.56 (d, 1H, $J = 7.2$ Hz), 7.47 (t, 1H, $J = 7.0$ Hz), 7.22 (t, 1H, $J = 9.2$ Hz), 3.77 (d, 1H, $J = 3.6$ Hz), 2.56 (s, 3H), 1.86–1.43 (m, 6H), 1.20–1.06 (m, 3H), 0.90 (s, 9H), 0.85–0.81 (m, 1H), 0.77–0.68 (m, 1H); $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta$ 203.6, 161.3, 148.7, 132.2, 131.2, 129.4, 129.1, 127.1, 125.0, 123.8, 116.7, 116.1, 85.5, 82.3, 44.3, 39.1, 33.3, 28.5, 27.4, 26.8, 26.4, 26.1, 25.6; IR (KBr, cm$^{-1}$): 2942, 2927, 2850, 2800, 1780, 1745, 1720, 1628, 1602, 1558, 1516, 1507, 1463, 1438, 1395, 1367, 1264, 1220, 1186, 1162, 1115, 1080, 1063, 1045, 1028, 975, 864, 820, 789, 752; HRMS (ESI) calcd for C$_{25}$H$_{30}$O$_5$ (M + Na$^+$) 433.1991, found 433.1998.

3-Acetyl-3-(tert-butylperoxy)-4-cyclopentylchroman-2-one (1b’):

Solid; m.p. 156-162 °C; $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 7.24–7.19 (m, 1H), 7.07–7.01 (m, 2H), 6.96 (d, 1H, $J = 8.0$ Hz), 3.20 (d, 1H, $J = 4.0$ Hz), 2.41 (s, 3H), 1.87–1.79 (m, 1H), 1.63–1.58 (m, 2H), 1.38–1.32 (m, 4H), 1.26–1.15 (m, 1H), 0.94 (s, 9H), 0.73–0.63 (m, 1H); $^{13}$C NMR (CDCl$_3$, 100 MHz): $\delta$ 203.3, 161.2, 151.2, 130.3, 128.6, 123.9, 121.7, 116.2, 86.5, 82.3, 45.6, 39.7, 31.1, 27.1, 26.5, 26.2, 24.3, 23.5; IR (KBr, cm$^{-1}$): 2990, 2958, 2872, 1784, 1715, 1615, 1587, 1489, 1467, 1423, 1373, 1366, 1358, 1226, 1198,
1169, 1136, 1116, 1093, 1056, 1017, 986, 920, 904, 774, 755, 720; HRMS (ESI) calcd for C_{20}H_{26}O_{5} (M + Na^{+}) 369.1678, found 369.1672.

3-(tert-Butylperoxy)-4-cyclohexyl-3-propionylchroman-2-one (9a‘):

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Solid; m.p. 162-164 °C; \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 400 MHz): \(\delta\) 7.21 (t, 1H, \(J = 8.0\) Hz), 7.05–6.99 (m, 2H), 6.95 (d, 1H, \(J = 8.4\) Hz), 2.95–2.90 (m, 1H), 2.88 (d, 1H, \(J = 2.4\) Hz), 2.76–2.66 (m, 1H), 1.71–1.60 (m, 2H), 1.52–1.43 (m, 4H), 1.25–1.18 (m, 2H), 1.09 (t, 3H, \(J = 7.0\) Hz), 0.92 (s, 9H), 0.87–0.73 (m, 2H), 0.56–0.46 (m, 1H); \textsuperscript{13}C NMR (CDCl\textsubscript{3}, 100 MHz): \(\delta\) 205.9, 161.3, 151.1, 130.1, 128.5, 123.8, 121.2, 116.2, 86.2, 82.2, 49.2, 38.4, 32.2, 31.9, 26.9, 26.6, 25.8, 25.7, 7.5; IR (KBr, cm\textsuperscript{-1}): 2974, 2930, 2857, 1786, 1716, 1614, 1588, 1490, 1458, 1405, 1390, 1376, 1370, 1364, 1352, 1325, 1260, 1225, 1179, 1155, 1119, 1082, 1066, 1020, 965, 940, 907, 898, 875, 842, 788, 757, 749, 703; HRMS (ESI) calcd for C_{22}H_{30}O_{5} (M + Na^{+}) 397.1991, found 397.1980.

3-(tert-Butylperoxy)-4-cyclohexyl-3-pentanoylchroman-2-one (10a‘):

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Solid; m.p. 132-136 °C; \textsuperscript{1}H NMR (CDCl\textsubscript{3}, 400 MHz): \(\delta\) 7.21 (t, 1H, \(J = 7.4\) Hz), 7.05–6.99 (m, 2H), 6.95 (d, 1H, \(J = 8.4\) Hz), 2.88 (s, 1H), 2.84–2.81 (m, 1H), 2.71–2.62 (m, 1H), 1.71–1.60 (m, 4H), 1.51–1.40 (m, 4H), 1.16–1.04 (m, 4H), 0.92 (s, 9H), 0.91–0.86 (m, 3H), 0.82–0.73 (m, 2H), 0.54–0.45 (m, 1H); \textsuperscript{13}C NMR (CDCl\textsubscript{3}, 100 MHz): \(\delta\) 205.3, 161.3, 151.1, 130.1, 128.5, 123.8, 121.2, 116.2, 86.0, 82.1, 49.1, 40.4, 38.3, 32.2, 26.8, 26.6, 26.2, 25.8, 25.7, 16.7, 13.9; IR (KBr, cm\textsuperscript{-1}): 2980, 2927, 2850, 1787, 1721, 1615, 1586, 1490, 1464, 1366, 1260, 1227, 1182, 1158, 1148, 1112, 1084, 1021, 966, 948, 911, 898, 877, 790, 765, 704; HRMS (ESI) calcd for C_{24}H_{34}O_{5} (M + Na^{+}) 425.2304, found 425.2310.
3-Benzoyl-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (11a’):

Solid; m.p. 158-160 °C; \( ^1H \) NMR (CDCl\textsubscript{3}, 400 MHz): \( \delta \) 8.13 (d, 2H, \( J = 7.2 \) Hz), 7.49 (t, 1H, \( J = 7.0 \) Hz), 7.37 (t, 2H, \( J = 7.4 \) Hz), 7.23–7.21 (m, 1H), 7.07–7.05 (m, 2H), 7.80 (d, 1H, \( J = 8.4 \) Hz), 3.27 (s, 1H), 1.82–1.78 (m, 1H), 1.63–1.50 (m, 4H), 1.45–1.41 (m, 2H), 1.18–1.10 (m, 3H), 0.81 (s, 9H), 0.60–0.53 (m, 1H); \( ^{13}C \) NMR (CDCl\textsubscript{3}, 100 MHz): \( \delta \) 194.2, 160.1, 151.2, 136.4, 132.9, 130.2, 129.5, 128.6, 128.2, 123.8, 120.9, 116.2, 87.8, 82.2, 50.6, 38.5, 32.3, 26.7, 26.5, 26.1, 25.9, 25.8; IR (KBr, cm\textsuperscript{-1}): 2970, 2928, 2854, 1777, 1734, 1692, 1612, 1548, 1448, 1375, 1276, 1245, 1226, 1198, 1170, 1133, 1116, 1066, 1019, 952, 923, 880, 812, 770, 752; HRMS (ESI) calcd for C\textsubscript{26}H\textsubscript{30}O\textsubscript{5} (M + Na\textsuperscript{+}) 445.1991, found 445.1984.

3-(tert-Butylperoxy)-4-cyclohexyl-2-oxochroman-3-carbonitrile (12a’):

Solid; m.p. 154-156 °C; \( ^1H \) NMR (CDCl\textsubscript{3}, 400 MHz): \( \delta \) 7.32 (t, 1H, \( J = 8.0 \) Hz), 7.16 (t, 1H, \( J = 7.4 \) Hz), 7.10–7.04 (m, 2H), 3.22 (d, 1H, \( J = 3.2 \) Hz), 2.15–2.08 (m, 1H), 1.77–1.73 (m, 2H), 1.63–1.53 (m, 3H), 1.35–1.18 (m, 3H), 1.06 (s, 9H), 0.95–0.85 (m, 1H), 0.70–0.60 (m, 1H); \( ^{13}C \) NMR (CDCl\textsubscript{3}, 100 MHz): \( \delta \) 159.5, 150.5, 130.2, 129.3, 124.8, 119.1, 116.5, 114.5, 83.3, 80.7, 50.1, 39.6, 32.1, 27.1, 26.4, 26.1, 25.8, 25.6; IR (KBr, cm\textsuperscript{-1}): 2980, 2927, 2855, 2212, 1768, 1620, 1558, 1486, 1459, 1367, 1361, 1260, 1239, 1150, 1135, 1064, 1050, 1030, 1008, 910, 896, 856, 798, 767, 751, 704; HRMS (ESI) calcd for C\textsubscript{26}H\textsubscript{30}NO\textsubscript{4} (M + Na\textsuperscript{+}) 366.1681, found 366.1678.

1-(Cyclohexyloxy)-2,2,6,6-tetramethylpiperidine (1A):

Gummy; \( ^1H \) NMR (CDCl\textsubscript{3}, 400 MHz): \( \delta \) 3.58 (bs, 1H), 2.04 (bs, 2H), 1.74 (bs, 2H), 1.54–1.45 (m, 7H), 1.31–1.19 (m, 5H), 1.14 (s,
6H), 1.11 (s, 6H); $^{13}$C NMR (CDCl$_3$, 150 MHz): $\delta$ 81.9, 59.8, 40.5, 34.7, 33.1, 26.2, 25.3, 17.6; IR (KBr, cm$^{-1}$): 2972, 2931, 2855, 1467, 1452, 1374, 1359, 1257, 1242, 1208, 1132, 1058, 1044, 1021, 966, 913, 785, 710; HRMS (ESI) calcd for C$_{15}$H$_{29}$NO (M + H$^+$) 240.2329, found 240.2335.

1-(Benzyloxy)-2,2,6,6-tetramethylpiperidine (1B):

Gummy; $^1$H NMR (CDCl$_3$, 400 MHz): $\delta$ 7.38-7.25 (m, 5H), 4.82 (s, 2H), 1.58–1.48 (m, 5H), 1.37–1.33 (m, 1H), 1.26 (s, 6H), 1.15 (s, 6H); $^{13}$C NMR (CDCl$_3$, 150 MHz): $\delta$ 138.5, 128.4, 127.7, 127.5, 78.9, 60.2, 39.9, 33.3, 20.5, 17.3; IR (KBr, cm$^{-1}$): 2973, 2929, 2871, 1496, 1469, 1452, 1373, 1359, 1262, 1243, 1207, 1183, 1133, 1081, 1045, 1028, 992, 955, 926, 732; HRMS (ESI) calcd for C$_{16}$H$_{25}$NO (M + H$^+$) 248.2016, found 248.2011.
3-Cyclohexyl-2H-chromen-2-one (1a): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Cyclohexyl-2H-chromen-2-one (1a): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-Cyclohexyl-7-methoxy-2H-chromen-2-one (2a): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Cyclohexyl-7-methoxy-2H-chromen-2-one (2a): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-Cyclohexyl-8-ethoxy-2H-chromen-2-one (3a): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Cyclohexyl-8-ethoxy-2H-chromen-2-one (3a): $^{13}$C NMR (CDCl$_3$, 150 MHz)
6,8-Di-tert-butyl-3-cyclohexyl-2H-chromen-2-one (4a): $^1$H NMR (CDCl$_3$, 600 MHz)
6,8-Di-tert-butyl-3-cyclohexyl-2H-chromen-2-one (4a): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-Cyclohexyl-6-nitro-2H-chromen-2-one (5a): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Cyclohexyl-6-nitro-2H-chromen-2-one (5a): $^{13}$C NMR (CDCl$_3$, 150 MHz)
6,8-Dichloro-3-cyclohexyl-2H-chromen-2-one (6a): $^1$H NMR (CDCl$_3$, 600 MHz)
6,8-Dichloro-3-cyclohexyl-2H-chromen-2-one (6a): $^{13}$C NMR (CDCl$_3$, 150 MHz)
6,8-Bromo-3-cyclohexyl-2H-chromen-2-one (7a): $^1$H NMR (CDCl$_3$, 600 MHz)
6,8-Bromo-3-cyclohexyl-2H-chromen-2-one (7a): $^{13}$C NMR (CDCl$_3$, 150 MHz)
2-Cyclohexyl-3H-benzo[f]chromen-3-one (8a): $^1$H NMR (CDCl$_3$, 600 MHz)
2-Cyclohexyl-3H-benzo[f]chromen-3-one (8a): $^{13}$C NMR (CDCl$_3$, 150 MHz)

Current Data Parameters
- ESR: AB_NAPCOME-13C
- P3000
- F2: Acquisition Parameters
  - Resn: 2816.10628
  - Tune: 14.26
  - Dim D: 5 mm EPR03801
  - Millimeter operation
  - SQUID: GMF12
  - RF: 200 MHz
  - SW: 260.6265 Hz
  - FT: 5.48e-08 sec
  - DM: 65.28 Hz
  - EM: 13.667 sec
  - DS: 1.000000 sec
  - 100

--- CHANNEL 1 ---
- F1: 150.917571 MHz
- S1: 1.0000000 sec
- SIM: 0.0050000 W

--- CHANNEL 2 ---
- F2: 480.117120 MHz
- S2: 20.000000 sec
- SIM: 5.5110000 W
- SIM: 0.0050000 W

--- PROCESSING PARAMETERS ---
- DF: 150.917571 MHz
- SW: 100 MHz
- S1: 1.00 Hz
- S2: 1.00 Hz
- PC: 1.60
3-Cyclopentyl-2H-chromen-2-one (1b): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Cyclopentyl-2H-chromen-2-one (1b): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-Cyclooctyl-2H-chromen-2-one (1c): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Cyclooctyl-2H-chromen-2-one (1c): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-Benzyl-2H-chromen-2-one (1d): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Benzyl-2H-chromen-2-one (1d): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-(4-Methylbenzyl)-2H-chromen-2-one (1e): $^1$H NMR (CDCl$_3$, 400 MHz)
3-(4-Methylbenzyl)-2H-chromen-2-one (1e): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-(3,5-Dimethylbenzyl)-2H-chromen-2-one (1f): $^1$H NMR (CDCl$_3$, 400 MHz)
3-(3,5-Dimethylbenzyl)-2H-chromen-2-one (1f): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-Acetyl-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (1a'): $^1$H NMR (CDCl$_3$, 400 MHz)
3-Acetyl-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (1a'): $^{13}$C NMR (CDCl$_3$, 150 MHz)

AB-COME-CYCLO-13C
3-Acetyl-3-(tert-butylperoxy)-6-chloro-4-cyclohexylchroman-2-one (13a'): $^1$H NMR (CDCl$_3$, 400 MHz)
3-Acetyl-3-(tert-butylperoxy)-6-chloro-4-cyclohexylchroman-2-one (13a'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-Acetyl-6-bromo-3-(tert-butyldperoxy)-4-cyclohexylchroman-2-one (14a'): $^1$H NMR (CDCl$_3$, 400 MHz)
3-Acetyl-6-bromo-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (14a'): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-Acetyl-3-((tert-butyli peroxy)-4-cyclohexyl-6-nitrochroman-2-one (5a'): $^1$H NMR (CDCl$_3$, 400 MHz)
3-Acetyl-3-(tert-butyloperoxy)-4-cyclohexyl-6-nitrochroman-2-one (5a'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-Acetyl-3-(tert-buty]peroxy)-6,8-dichloro-4-cyclohexylchroman-2-one (6a''): $^1$H NMR (CDCl$_3$, 400 MHz)
3-Acetyl-3-(tert-butylperoxy)-6,8-dichloro-4-cyclohexylchroman-2-one (6a'): $^{13}$C NMR (CDCl₃, 100 MHz)
3-Acetyl-6,8-dibromo-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (7a'): $^1$H NMR (CDCl$_3$, 400 MHz)
3-Acetyl-6,8-dibromo-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (7a'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-Acetyl-3-((tert-butylperoxy)-4-cyclohexyl-7-methoxychroman-2-one (2a'): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Acetyl-3-(tert-butyldiroyloxy)-4-cyclohexyl-7-methoxychroman-2-one (2a'): $^{13}$C NMR (CDCl$_3$, 150 MHz)
3-Acetyl-3-( tert-butylperoxy)-4-cyclohexyl-8-methoxychroman-2-one (15a'): $^1$H NMR (CDCl$_3$, 600 MHz)
3-Acetyl-3-((tert-butylperoxy)-4-cyclohexyl-8-methoxychroman-2-one (15a'): $^{13}$C NMR (CDCl$_3$, 150 MHz)
2-Acetyl-2-(tert-butylperoxy)-1-cyclohexyl-1H-benzo[f]chromen-3(2H)-one (8a'): $^1$H NMR (CDCl$_3$, 400 MHz)
2-Acetyl-2-(tert-butylperoxy)-1-cyclohexyl-1H-benzo[f]chromen-3(2H)-one (8a'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-Acetyl-3-((tert-butylperoxy)-4-cyclopentylchroman-2-one (1b'): $^1$H NMR (CDCl$_3$, 400 MHz)
3-Acetyl-3-(tert-butyldiperoxyl)-4-cyclopentylchroman-2-one (1b\'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-((tert-Butylperoxy)-4-cyclohexyl-3-propionylchroman-2-one (9a'): $^1$H NMR (CDCl$_3$, 400 MHz)
3-(tert-Butylperoxy)-4-cyclohexyl-3-propionylchroman-2-one (9a'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-\((\text{tert-Buty卜peroxy})-4\text{-cyclohexyl-3-pentanoylchroman-2-one (10a')}: \text{\textsuperscript{1}H NMR (CDCl\textsubscript{3}, 400 MHz)}}}
3-((tert-Butylperoxy)-4-cyclohexyl-3-pentanoylchroman-2-one (10a'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-Benzoyl-3-(tert-butylperoxy)-4-cyclohexylchroman-2-one (11a'): \(^1\)H NMR (CDCl\(_3\), 400 MHz)
3-Benzoyl-3-(tert-butyldperoxy)-4-cyclohexylchroman-2-one (11a'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
3-\((\text{tert-Butylperoxy})-4\)-cyclohexyl-2-oxochroman-3-carbonitrile (12a'): $^1$H NMR (CDCl$_3$, 400 MHz)
3-(tert-Butylperoxy)-4-cyclohexyl-2-oxochroman-3-carbonitrile (12a'): $^{13}$C NMR (CDCl$_3$, 100 MHz)
1-(Cyclohexyloxy)-2,2,6,6-tetramethylpiperidine (1A): $^1$H NMR (CDCl$_3$, 400 MHz)
1-(Cyclohexyloxy)-2,2,6,6-tetramethylpiperidine (1A): $^{13}$C NMR (CDCl$_3$, 150 MHz)
1-(Benzyloxy)-2,2,6,6-tetramethylpiperidine (1B): $^1$H NMR (CDCl$_3$, 400 MHz)
1-(Benzyloxy)-2,2,6,6-tetramethylpiperidine (1B): $^{13}$C NMR (CDCl$_3$, 150 MHz)