Supporting Informations

Oxidant controlled regioselective mono- and di-functionalizations of coumarins

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Contents:

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 ^{13}C 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]

1. M. Ghandi, A.-T. Ghomi, and M. Kubicki, *J. Org. Chem.*, 2013, **78**, 2611.

Experimental procedure:

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

To an oven dried 25 mL round bottom flask fitted with reflux condenser, 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) 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 ^oC. 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 Na_2SO_4, 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-2*H*-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 ^{o}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 (Na_2SO_4), 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

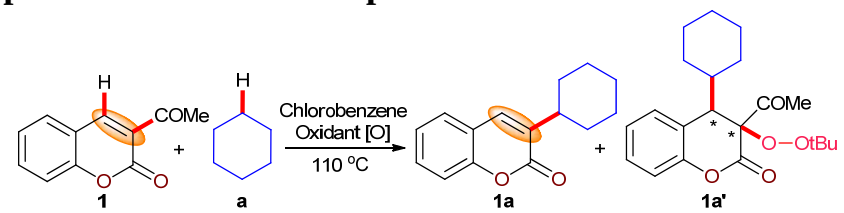
Crystal data were collected with Bruker Smart Apex-II CCD diffractometer using graphite monochromated MoKα radiation (λ = 0.71073 Å) at 298 K. Cell parameters were retrieved using SMART[a] software and refined with SAINT[a] on all observed reflections. Data reduction was performed with the SAINT software 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.

a. SMART V 4.043 Software for the CCD Detector System; Siemens Analytical Instruments Division: Madison, WI, 1995.
b. SAINT V 4.035 Software for the CCD Detector System; Siemens Analytical Instruments Division: Madison, WI, 1995.
c. Sheldrick, G. M. SHELXL-97, Program for the Refinement of Crystal Structures; University of Göttingen: Göttingen (Germany), 1997.

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.

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) Å^3 ; Z = 2; ρ_{cal}= 1.246g/cm^3; μ (mm^{-1}) = 0.080; *F (000)* = 276.0; Reflection collected / unique = 2348 / 1909; Refinement method = Full-matrix least-squares on F^2; Final R indices [I>2σ_I] 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



Entry	Catalyst (mol %)	Additive (equiv.)	Oxidant[d] (equiv.)	Yield (%)[a,b] **1a**	**1a'**
1	——	——	DTBP (3.0)	20	00
2	——	——	DTBP (4.0)	40	00
3	$Cu(OAc)_2$(10.0)	——	DTBP (4.0)	34	00
4	$CuCl_2$(10.0)	——	DTBP (4.0)	38	00
5	$FeCl_2$(10.0)	——	DTBP (4.0)	26	00
6	Fe_3O_4(10.0)	——	DTBP (4.0)	Trace	00
7	$Fe(acac)_3$ (10.0)	——	DTBP (4.0)	53	00
8	**$Fe(acac)_3$ (5.0)**	**——**	**DTBP (4.0)**	**52**	**00**
9	$Fe(acac)_3$ (5.0)	AcOH (1.0)	DTBP (4.0)	47	00
10	——	AcOH (1.0)	DTBP (4.0)	00	00
11	——	PTSA (1.0)	DTBP (4.0)	00	00
12[c]	——	AcOH (1.0)	TBHP (4.0)	00	54
13[c]	——	PTSA (1.0)	TBHP (4.0)	00	00
14[c]	——	TfOH (1.0)	TBHP (4.0)	00	00
15[c]	——	PhCOOH (1.0)	TBHP (4.0)	00	30
16[c]	——	PivOH (1.0)	TBHP (4.0)	00	27
17[c]	**——**	**AcOH (2.0)**	**TBHP (4.0)**	**00**	**65**
18[c]	——	——	TBHP (4.0)	00	08

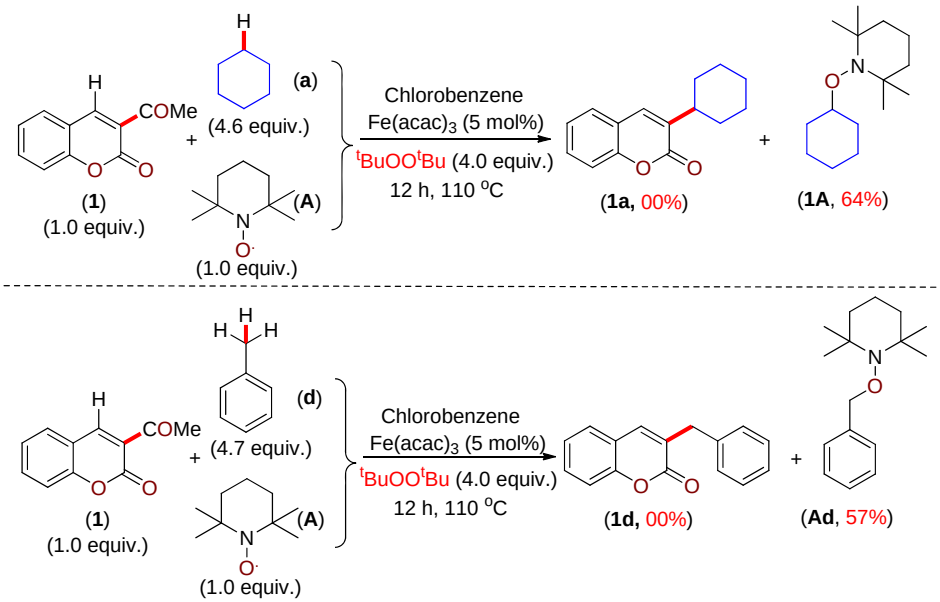
[a]Reaction conditions: **1** (0.25 mmol), cyclohexane (**a**) (0.5 mL, 4.6 mmol) in chlorobenzene (1.0 mL) at 110 °C for 12 h. [b]Isolated yield. [c]Reaction performed for 3 h. [d]Oxidants were used portion wise (four times for both DTBP and TBHP).

Mechanistic investigation:

Trapping of cyclohexyl and benzyl intermediates with TEMPO during mono-functionalization:

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 ^{o}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**).

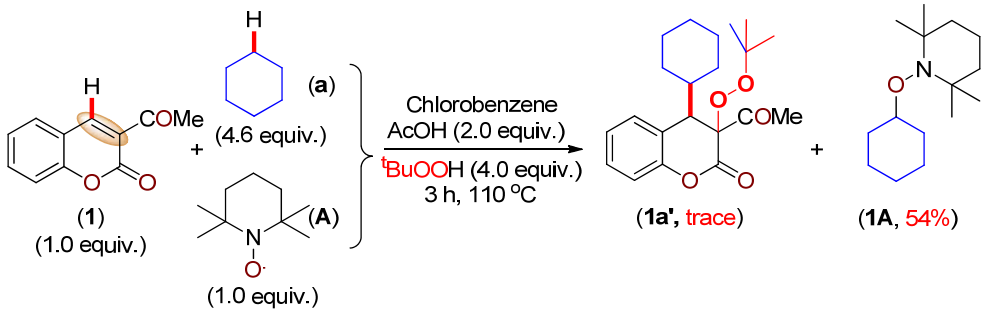
Similar reaction between 3-acetylcoumarin (**1**) (0.047g, 0.25 mmol) and toluene (**a**) (0.5 mL, 4.7 mmol) in the presence of radical scavenger TEMPO (**A**) (0.039 g, 0.25 mmol) under otherwise identical reaction conditions to that of above provided benzyl-TEMPO adduct 1-(benzyloxy)-2,2,6,6-tetramethylpiperidine (**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:

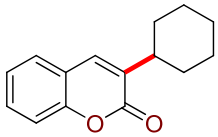
An oven-dried 25 mL round bottom flask was charged with 3-acetylcoumarin (**1**) (0.047g, 0.25 mmol), decane solution of TBHP (5−6 M) (200 µL, 1.0 mmol), AcOH (30 µL, 0.50 mmol), TEMPO (**A**) (0.039 g, 0.25 mmol) and cyclohexane (**a**) (0.5 mL, 4.6 mmol) in chlorobenzene (1.0 mL). The flask was fitted to a reflux condenser and the reaction mixture was stirred in a preheated oil bath at 110 ^{o}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

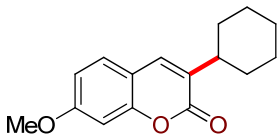
Spectral Data:

3-Cyclohexyl-2*H*-chromen-2-one (1a):



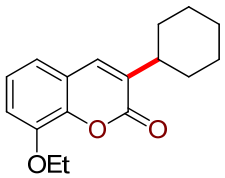
Semi-solid; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 7.47–7.44 (m, 3H), 7.31 (d, 1H, J = 8.4 Hz), 7.24 (t, 1H, J = 7.2 Hz), 2.81–2.76 (m, 1H), 1.98 (dd, 2H, J = 15.6, 4.2 Hz), 1.87–1.84 (m, 2H), 1.80–1.77 (m, 1H), 1.49–1.41 (m, 2H), 1.34–1.24 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 161.8, 152.9, 136.5, 135.2, 130.2, 127.5, 124.4, 119.9, 116.5, 38.4, 32.3, 26.7, 26.4; IR (KBr, cm^{-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 $C_{15}H_{16}O_2$ (M + H^+) 229.1229, found 229.1224.

3-Cyclohexyl-7-methoxy-2*H*-chromen-2-one (2a):



Semi-solid; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 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); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 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^{-1}): 2959, 2924, 2850, 1701, 1652, 1628, 1593, 1572, 1464, 1433, 1403, 1297, 1265, 1158, 1138, 1034, 964, 937, 774; HRMS (ESI) calcd for $C_{16}H_{18}O_3$ (M + H^+) 259.1335, found 259.1327.

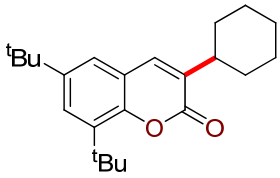
3-Cyclohexyl-8-ethoxy-2*H*-chromen-2-one (3a):



Semi-solid; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 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,

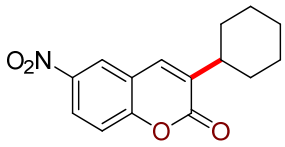
2H), 1.32–1.23 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 161.3, 146.5, 142.8, 136.6, 135.3, 124.2, 120.7, 118.9, 113.9, 65.1, 38.3, 32.3, 26.7, 26.4, 14.9; IR (KBr, cm^{-1}): 2977, 2926, 2852, 1719, 1608, 1579, 1471, 1449, 1392, 1355, 1276, 1181, 1114, 1098, 1064, 1044, 982, 770, 731; HRMS (ESI) calcd for $C_{17}H_{20}O_3$ (M + H^+) 273.1492, found 273.1485.

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



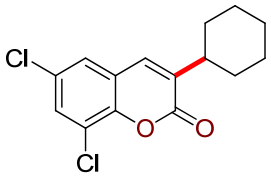
Semi-solid; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 7.49 (d, 1H, J = 2.4 Hz), 7.43 (s, 1H), 7.26 (s, 1H), 2.79–2.76 (m, 1H), 1.97 (d, 2H, J = 12.0 Hz), 1.86–1.83 (m, 2H), 1.78–1.76 (m, 1H), 1.51 (s, 9H), 1.46–1.42 (m, 3H), 1.35 (s, 9H), 1.33–1.29 (m, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 161.6, 149.6, 146.5, 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 $C_{23}H_{32}O_2$ (M + H^+) 341.2482, found 341.2470.

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



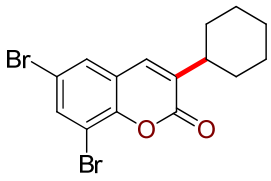
Semi-solid; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 8.39 (d, 1H, J = 2.4 Hz), 8.33 (d, 1H, J = 9.6 Hz), 7.51 (s, 1H), 7.42 (d, 1H, J = 9.0 Hz), 2.82–2.77 (m, 1H), 1.99 (d, 2H, J = 12.0, 4.2 Hz), 1.88 (dt, 2H, J = 13.2, 3.0 Hz), 1.80 (d, 1H, J = 13.2 Hz), 1.49–1.42 (m, 2H), 1.35–1.27 (m, 3H); ^{13}C NMR ($CDCl_3$, 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 $C_{15}H_{15}NO_4$ (M + H^+) 274.1081, found 274.1074.

6,8-Dichloro-3-cyclohexyl-2*H*-chromen-2-one (6a):



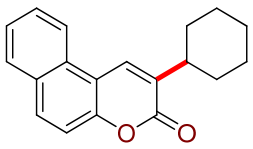
Semi-solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50 (d, 1H, J = 2.4 Hz), 7.35–7.34 (m, 2H), 2.81–2.75 (m, 1H), 1.98–1.95 (m, 2H), 1.88–1.77 (m, 3H), 1.50–1.39 (m, 2H), 1.33–1.19 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 159.9, 147.3, 137.4, 134.9, 130.6, 129.4, 125.4. 122.3, 121.7, 38.6, 32.2, 26.6, 26.3; IR (KBr, cm^{-1}): 2927, 2853, 1733, 1660, 1635, 1564, 1452, 1352, 1252, 1172, 1135, 1063, 992, 888, 859, 830, 778, 736; HRMS (ESI) calcd for $C_{15}H_{14}Cl_2O_2$ (M + H^+) 297.0450, found 297.0442.

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



Semi-solid; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 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): δ 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 $C_{15}H_{14}Br_2O_2$ (M + H^+) 384.9433, found 384.9424.

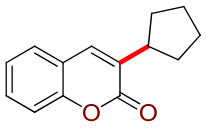
2-Cyclohexyl-3*H*-benzo[*f*]chromen-3-one (8a):



Semi-solid; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 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): δ 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,

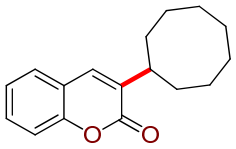
782, 738; HRMS (ESI) calcd for $C_{19}H_{18}O_2$ (M + H^+) 279.1386, found 279.1389

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



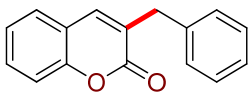
Semi-solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50 (s, 1H), 7.45 (m, 2H), 7.31 (m, 1H), 7.26–7.23 (m, 1H), 3.20–3.11 (m, 1H), 2.14–2.06 (m, 2H), 1.85–1.76 (m, 4H), 1.73–1.53 (m, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 161.9, 153.1, 136.2, 133.7, 130.6, 127.4, 124.4, 119.8, 116.5, 40.9, 31.9, 25.3; IR (KBr, cm^{-1}): 2955, 2872, 1722, 1630, 1609, 1454, 1276, 1224, 1173, 1121, 1057, 1022, 948, 922, 755; HRMS (ESI) calcd for $C_{14}H_{14}O_2$ (M + H^+) 215.1073, found 215.1064.

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



Crystalline needles; m.p. 85-86 ^{o}C; ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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 $C_{17}H_{20}O_2$ (M + H^+) 257.1543, found 257.1539.

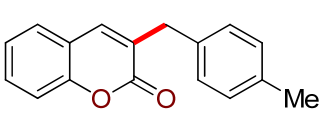
3-Benzyl-2*H*-chromen-2-one (1d):



Semi-solid; ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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,

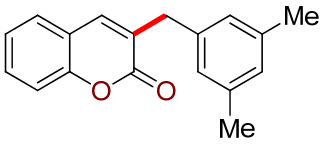
119.6, 116.6, 36.8; IR (KBr, cm^{-1}): 3022, 2988, 2917, 2841, 1712, 1631, 1608, 1489, 1455, 1434, 1387, 1224, 147, 1123, 1075, 1051, 1028, 956, 930, 825, 753, 731, 726, 700; HRMS (ESI) calcd for $C_{16}H_{12}O_2$ ($M + H^+$) 237.0917, found 237.0920.

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



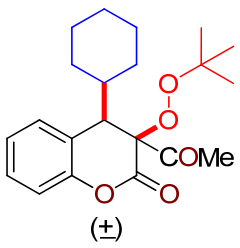
Semi-solid; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.44 (t, 1H, J = 7.4 Hz), 7.35–7.29 (m, 2H), 7.25–7.23 (m, 1H), 7.21–7.14 (m, 5H), 3.82 (s, 2H), 2.34 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 161.2, 153.2, 139.3, 136.6, 134.7, 130.9, 129.9, 129.7, 129.5, 127.6, 124.4, 119.7, 116.6, 36.3, 21.3; IR (KBr, cm^{-1}): 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; ^1H NMR ($CDCl_3$, 400 MHz): δ 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): δ 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.

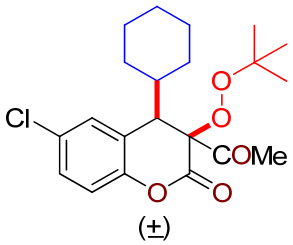
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (1a'):



Solid; m.p. 180-181 ^oC; ^1H NMR ($CDCl_3$, 400 MHz): δ 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

($CDCl_3$, 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, 1380, 1365, 1352, 1301, 1260, 1247, 1198, 1174, 1124, 1068, 1015, 918, 876, 776, 768; HRMS (ESI) calcd for $C_{21}H_{28}O_5$ ($M + Na^+$) 383.1834, found 383.1842.

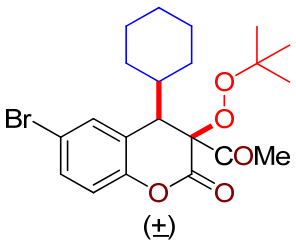
3-Acetyl-3-(*tert*-butylperoxy)-6-chloro-4-cyclohexylchroman-2-one (13a'):



(±)

Solid; m.p. 168-170 °C; ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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, 1120, 1094, 1083, 1066, 1041, 1034, 930, 911, 896, 891, 882, 870, 848, 812, 787, 759, 745, 707; HRMS (ESI) calcd for $C_{21}H_{27}ClO_5$ ($M + Na^+$) 417.1445, found 417.1436.

3-Acetyl-6-bromo-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (14a'):

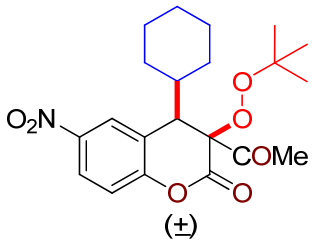


(±)

Solid; m.p. 155-158 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40 (dd, 1H, J_1 = 8.0 Hz, J_2 = 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); ^{13}C NMR ($CDCl_3$, 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,

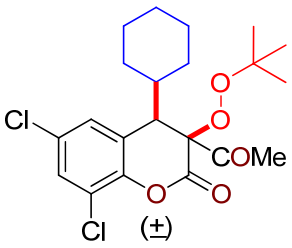
1478, 1450, 1412, 1389, 1364, 1260, 1245, 1224, 1175, 1153, 1092, 1026, 912, 891, 873, 858, 810, 775; HRMS (ESI) calcd for $C_{21}H_{27}BrO_5$ (M + Na^+) 461.0940, found 461.0932.

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



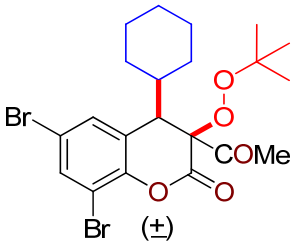
Semi-solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.19 (dd, 1H, J_1 = 8.8 Hz, J_2 = 2.4 Hz), 7.99 (d, 1H, J = 2.8 Hz), 7.15 (d, 1H, J = 8.8 Hz), 3.07 (d, 1H, J = 2.8 Hz), 2.47 (s, 3H), 1.71–1.51 (m, 6H), 1.27–1.05 (m, 3H), 0.99 (s, 9H), 0.88–0.79 (m, 1H), 0.54–0.44 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 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, 26.2, 25.8, 25.6; IR (KBr, cm^{-1}): 3092, 2984, 2924, 2850, 1798, 1720, 1627, 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'):



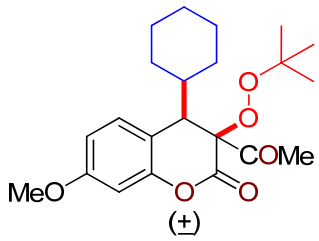
Semi-solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.38 (s, 1H), 6.98 (s, 1H), 2.95 (d, 1H, J = 2.4 Hz), 2.47 (s, 3H), 1.73–1.70 (m, 2H), 1.63–1.55 (m, 4H), 1.25–1.12 (m, 3H), 1.04 (s, 9H), 0.95–0.88 (m, 1H), 0.62–0.54 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 202.5, 159.4, 145.7, 129.0, 128.8, 128.2, 124.3, 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 $C_{21}H_{26}Cl_2O_5$ (M + Na^+) 451.1055, found 451.1049.

3-Acetyl-6,8-dibromo-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (7a'):



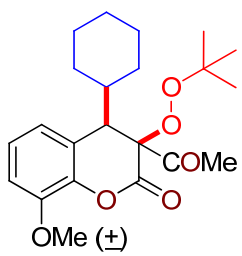
Semi-solid; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.64 (d, 1H, J = 2.4 Hz), 7.13 (d, 1H, J = 2.0 Hz), 2.90 (d, 1H, J = 3.2 Hz), 2.44 (s, 3H), 1.70–1.48 (m, 6H), 1.24–1.09 (m, 3H), 1.01 (s, 9H), 0.92–0.86 (m, 1H), 0.55–0.51 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 202.6, 159.5, 147.4, 134.7, 131.7, 124.7, 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 $C_{21}H_{26}Br_2O_5$ (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): δ 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): δ 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, 26.6, 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.

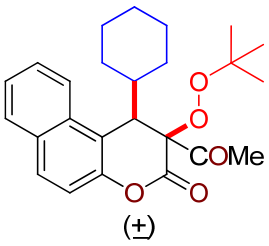
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexyl-8-methoxychroman-2-one (15a'):



Semi-solid; ^{1}H NMR ($CDCl_3$, 600 MHz): δ 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,

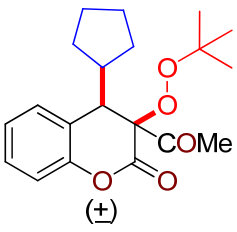
1H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 203.5, 160.6, 147.1, 140.4, 123.7, 122.2, 121.7, 111.4, 85.8, 82.2, 56.2, 48.9, 38.2, 32.4, 27.2, 27.0, 26.6, 26.0, 25.9, 25.8; IR (KBr, cm^{-1}): 2982, 2930, 2856, 1780, 1724, 1620, 1590, 1554, 1504, 1485, 1460, 1366, 1320, 1304, 1273, 1250. 1182, 1160, 1107, 1062, 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 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 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): δ 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, 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.

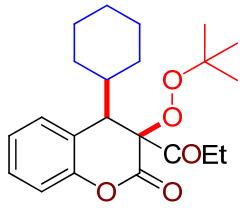
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclopentylchroman-2-one (1b'):



Solid; m.p. 160-162 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 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): δ 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.

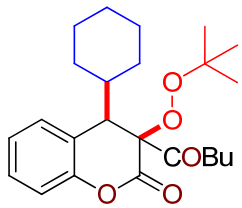
3-(*tert*-Butylperoxy)-4-cyclohexyl-3-propionylchroman-2-one (9a'):



(±)

Solid; m.p. 162-164 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 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); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 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, 26.2, 25.8, 25.7, 7.5; IR (KBr, cm^{-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.

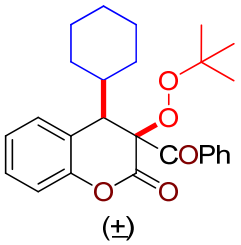
3-(*tert*-Butylperoxy)-4-cyclohexyl-3-pentanoylchroman-2-one (10a'):



(±)

Solid; m.p. 132-136 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 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); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 205.3, 161.3, 151.1, 130.1, 128.5, 123.8, 121.1, 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^{-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.

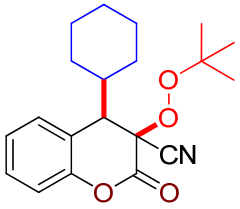
3-Benzoyl-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (11a'):



(±)

Solid; m.p. 158-160 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 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 ($CDCl_3$, 100 MHz): δ 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^{-1}): 2970, 2928, 2854, 1777, 1734, 1692, 1612, 1600, 1490, 1458, 1448, 1375, 1276, 1245, 1226, 1198, 1170, 1133, 1116, 1066, 1019, 952, 923, 880, 812, 770, 752; HRMS (ESI) calcd for $C_{26}H_{30}O_5$ (M + Na$^+$) 445.1991, found 445.1984.

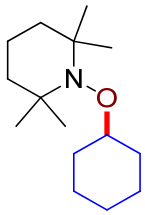
3-(*tert*-Butylperoxy)-4-cyclohexyl-2-oxochroman-3-carbonitrile (12a'):



(±)

Solid; m.p. 154-156 °C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 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 ($CDCl_3$, 100 MHz): δ 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^{-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 $C_{20}H_{25}NO_4$ (M + Na$^+$) 366.1681, found 366.1678.

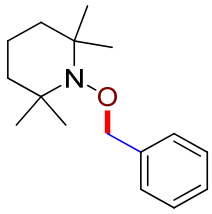
1-(Cyclohexyloxy)-2,2,6,6-tetramethylpiperidine (1A):



Gummy; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 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,

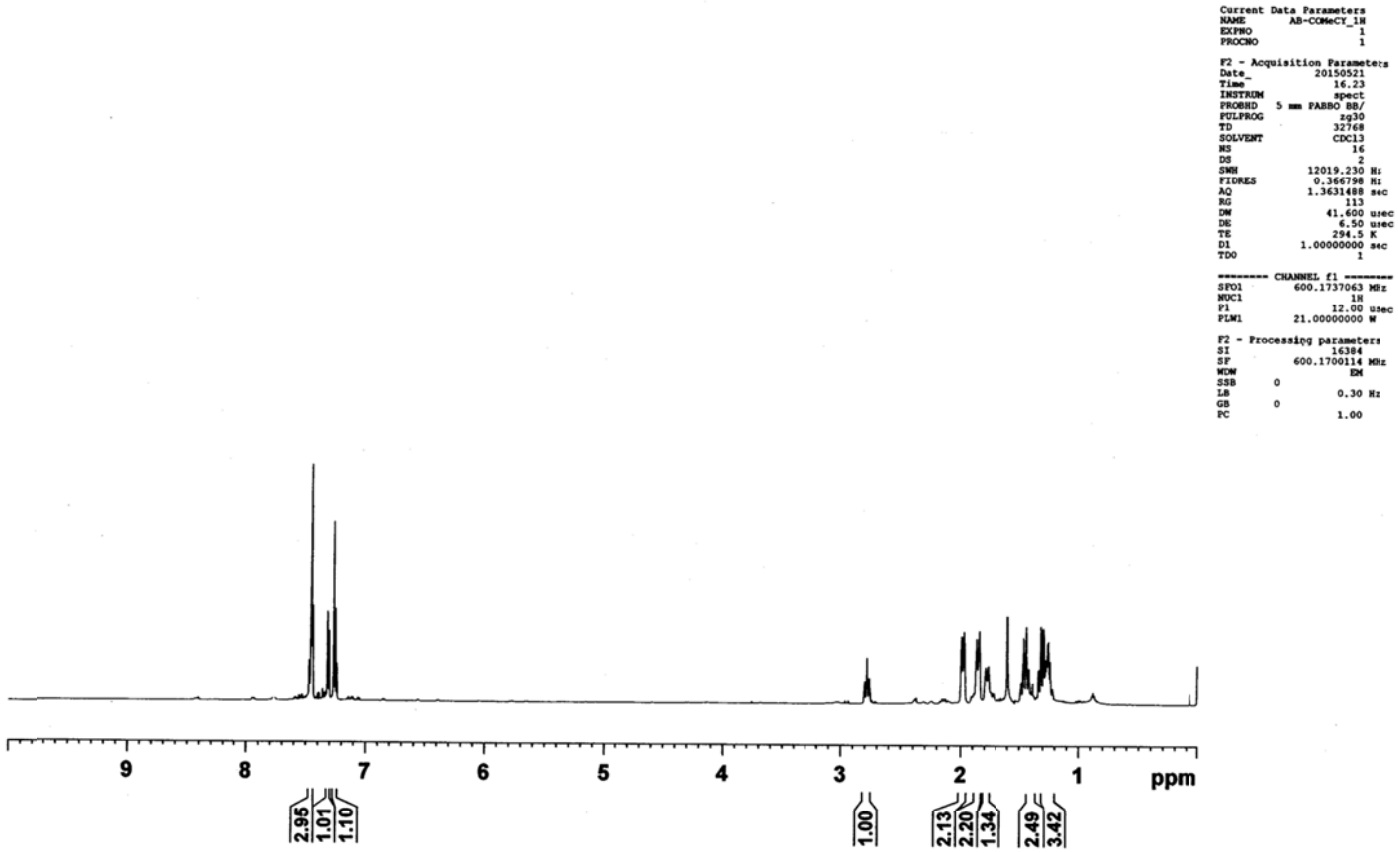
6H), 1.11 (s, 6H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 81.9, 59.8, 40.5, 34.7, 33.1, 26.2, 25.3, 17.6; IR (KBr, cm^{-1}): 2972, 2931, 2855, 1467, 1452, 1374, 1359, 1257, 1242, 1208, 1132, 1058, 1044, 1021, 966, 913, 785, 710; HRMS (ESI) calcd for $C_{15}H_{29}NO$ (M + H^+) 240.2329, found 240.2335.

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

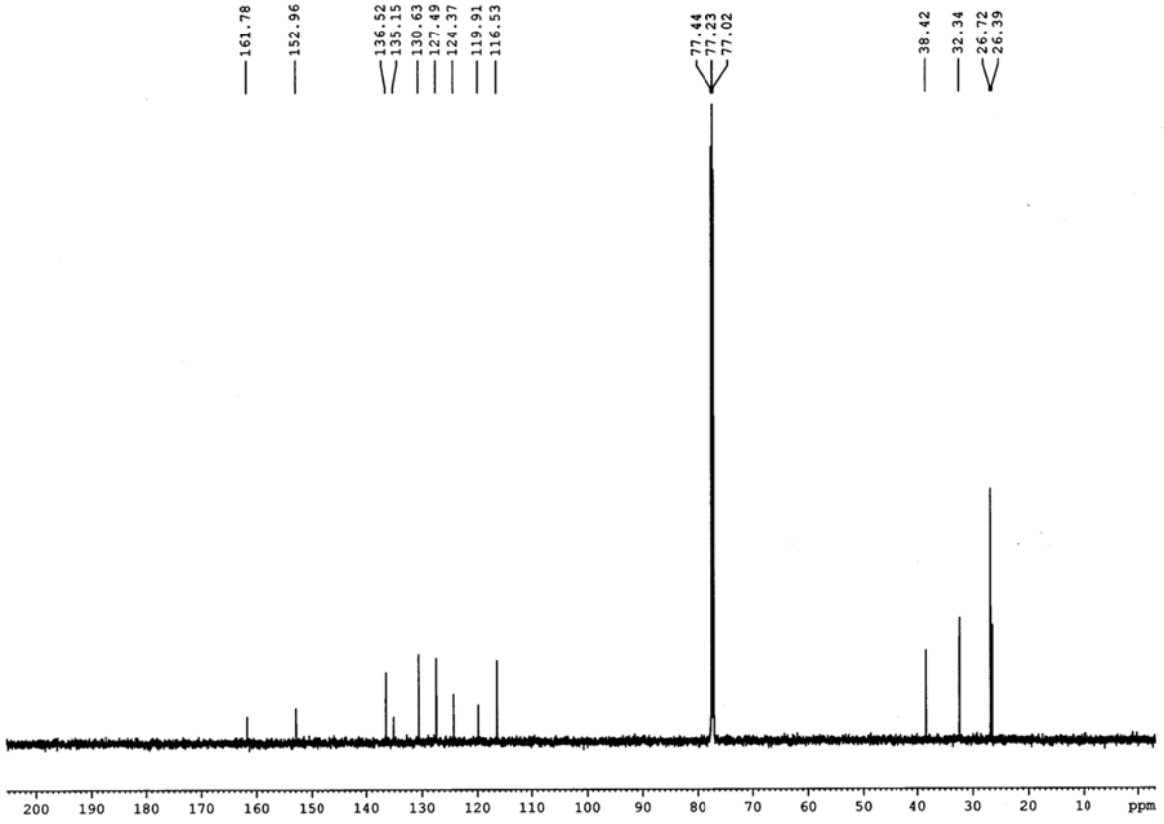


Gummy; ^1H NMR ($CDCl_3$, 400 MHz): δ 7.38-7.25 (m, 5H), 4.82 (s, 2H), 1.58–1.48 (m, 5H), 1.37–1.33 (m, 1H), 1.26 (s, 6H), 1.15 (s, 6H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 138.5, 128.4, 127.7, 127.5, 78.9, 60.2, 39.9, 33.3, 20.5, 17.3; IR (KBr, cm^{-1}): 2973, 2929, 2871, 1496, 1469, 1452, 1373, 1359, 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-2*H*-chromen-2-one (1a): ^{1}H NMR ($CDCl_3$, 600 MHz)



3-Cyclohexyl-2*H*-chromen-2-one (1a): ^{13}C NMR ($CDCl_3$, 150 MHz)

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EXPNO                 1
PROCNO                1

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Date_          20150302
Time              10.28
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD              32768
SOLVENT           CDCl3
NS                  201
DS                    2
SWH           36057.691 Hz
FIDRES         1.100393 Hz
AQ            0.4543829 sec
RG                65.24
DW               13.867 usec
DE                6.50 usec
TE              298.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9279571 MHz
NUC1                13C
P1                10.50 usec
PLW1        95.00000000 W

======== CHANNEL f2 ========
SFO2        600.1724007 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             70.00 usec
PLW2        21.00000000 W
PLW12        0.61714000 W
PLW13        0.30239999 W

F2 - Processing parameters
SI                16384
SF          150.9128355 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
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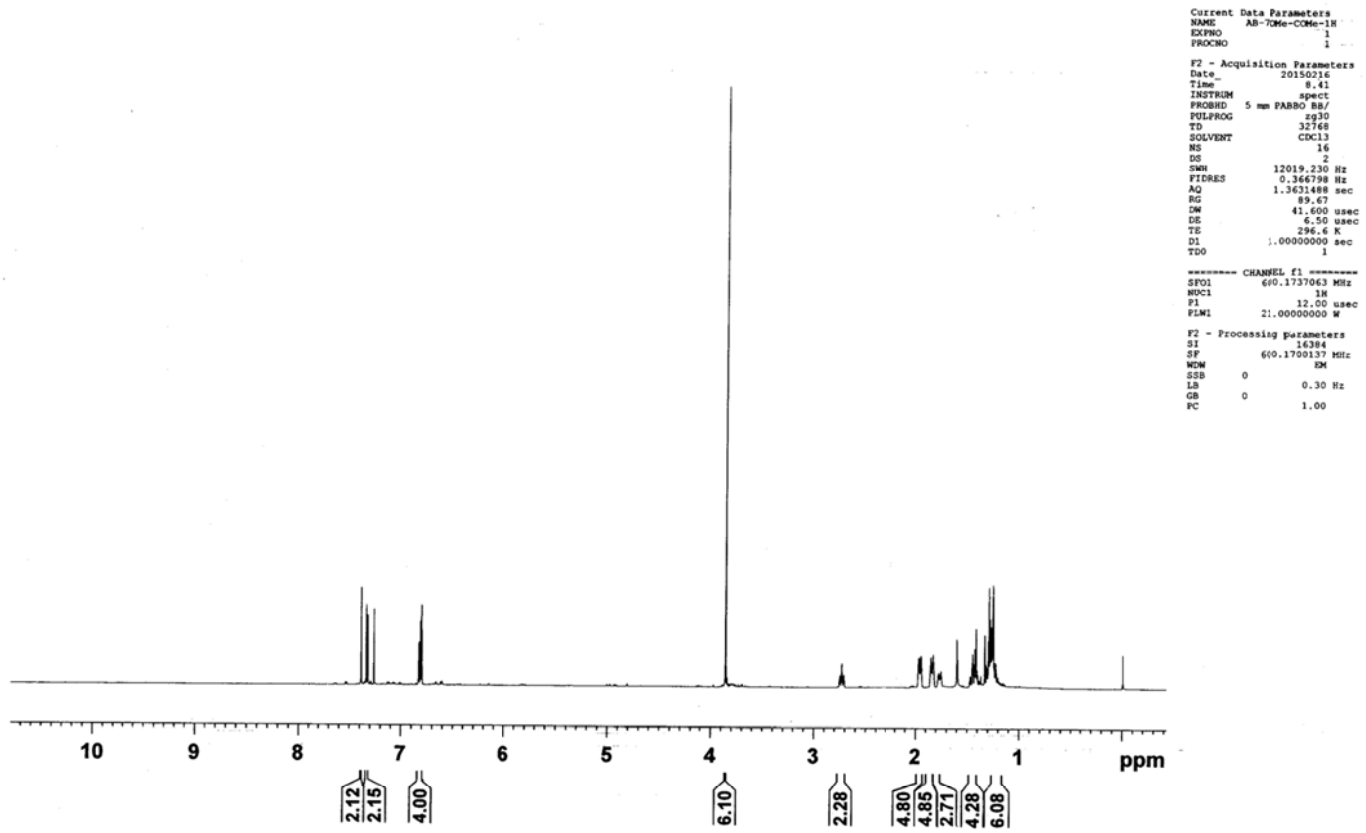
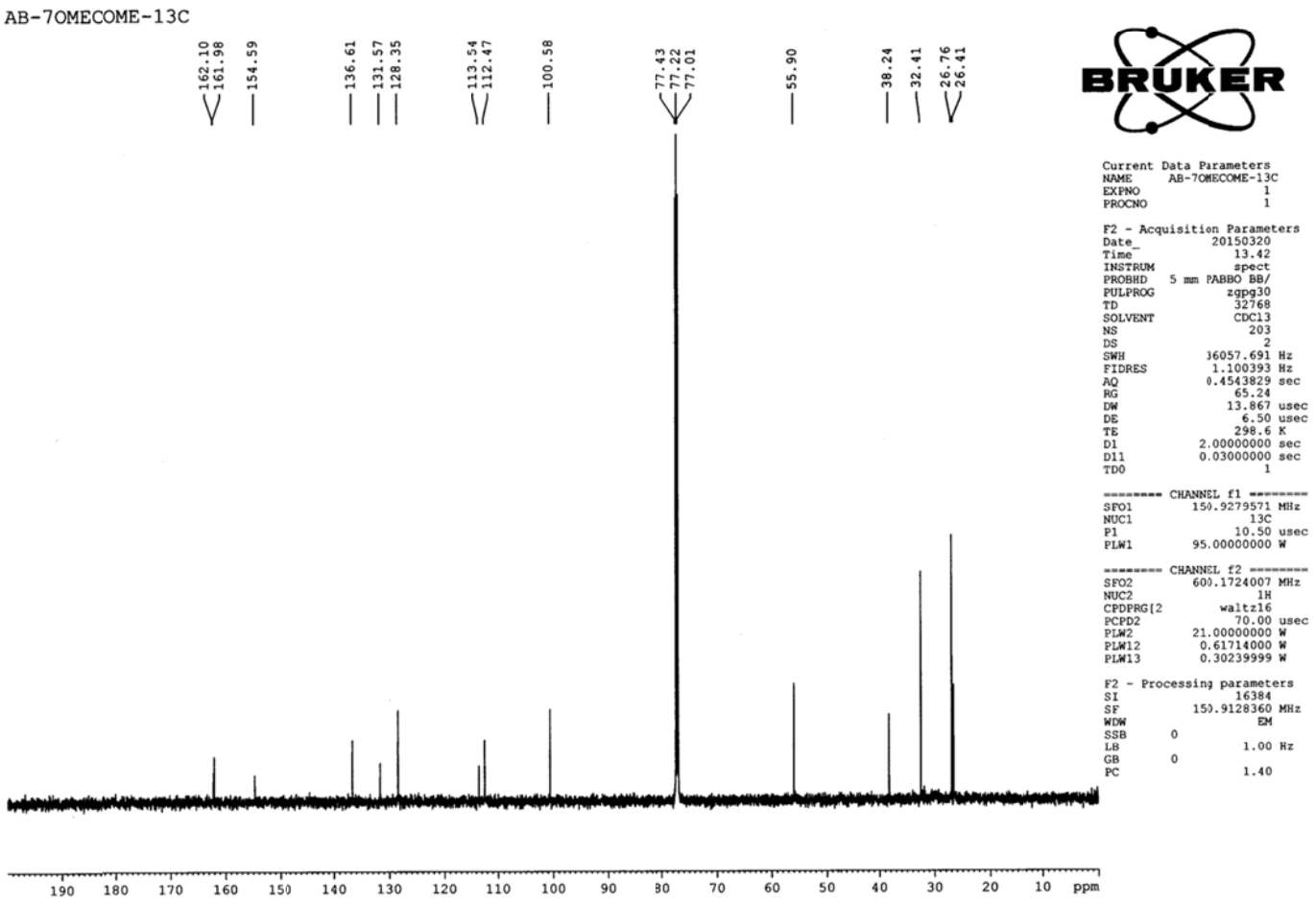
3-Cyclohexyl-7-methoxy-2*H*-chromen-2-one (2a): ^{1}H NMR ($CDCl_3$, 600 MHz)



3-Cyclohexyl-7-methoxy-2*H*-chromen-2-one (2a): ^{13}C NMR ($CDCl_3$, 150 MHz)



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Current Data Parameters
NAME AB-7OMECOME-13C
EXPNO 1
PROCNO 1

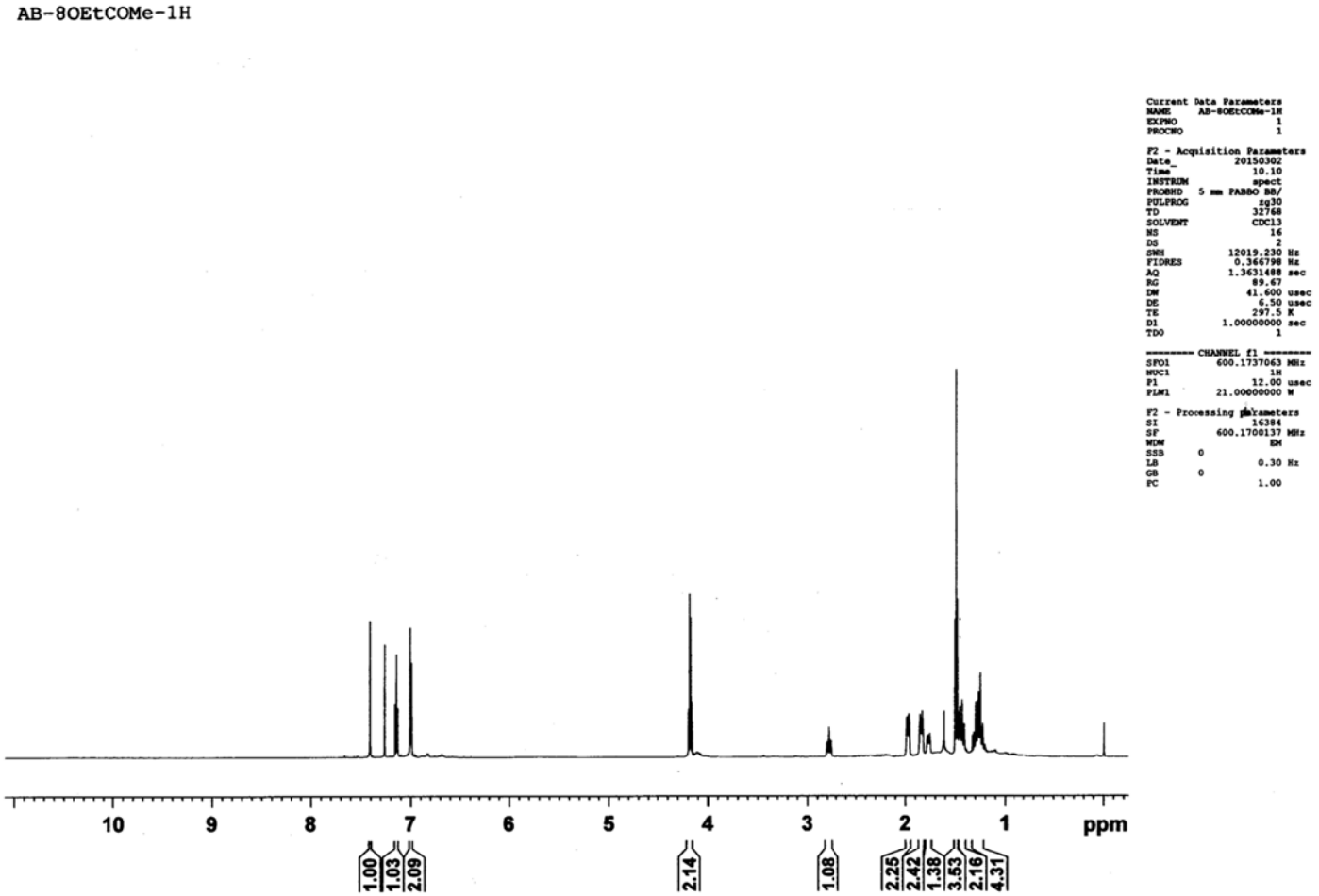
F2 - Acquisition Parameters
Date_ 20150320
Time 13.42
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 203
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

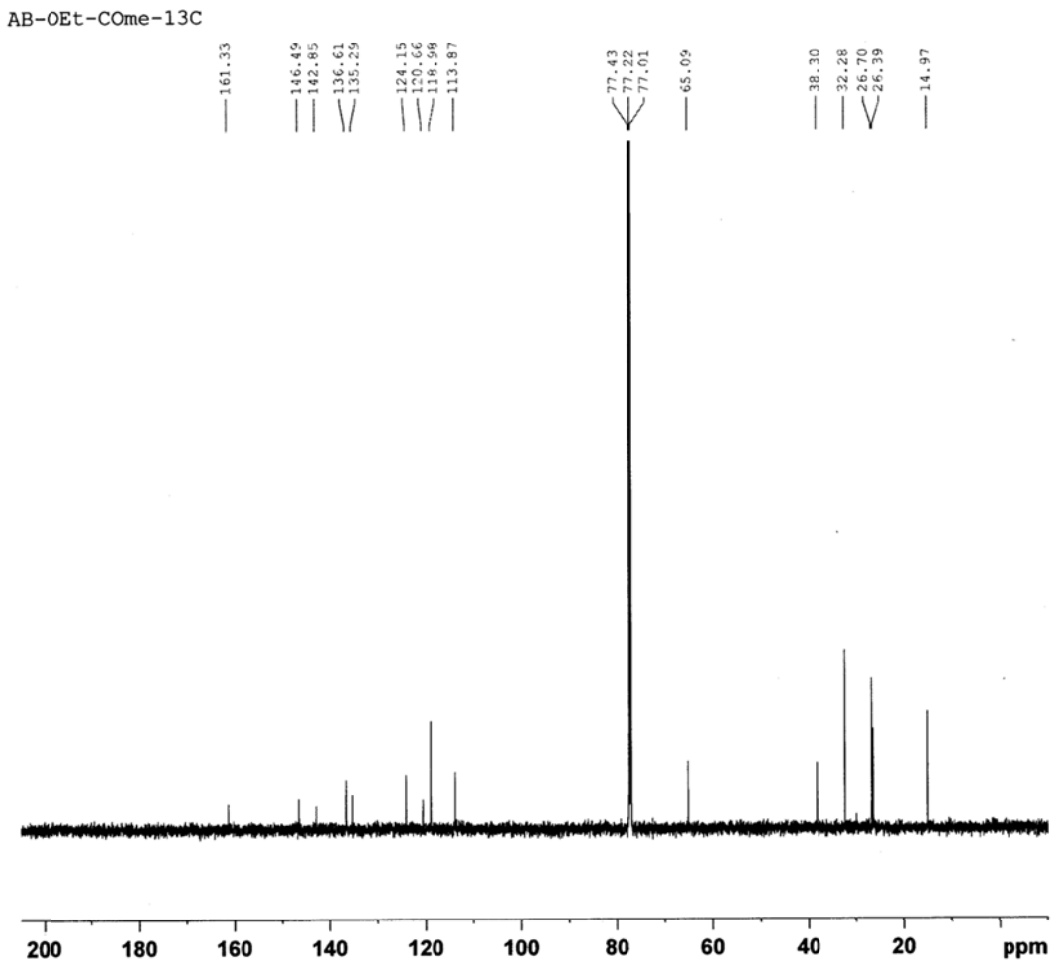
======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.9128360 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

3-Cyclohexyl-8-ethoxy-2*H*-chromen-2-one (3a): ^{1}H NMR ($CDCl_3$, 600 MHz)



3-Cyclohexyl-8-ethoxy-2*H*-chromen-2-one (3a): ^{13}C NMR ($CDCl_3$, 150 MHz)



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Current Data Parameters
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EXPNO                  1
PROCNO                 1

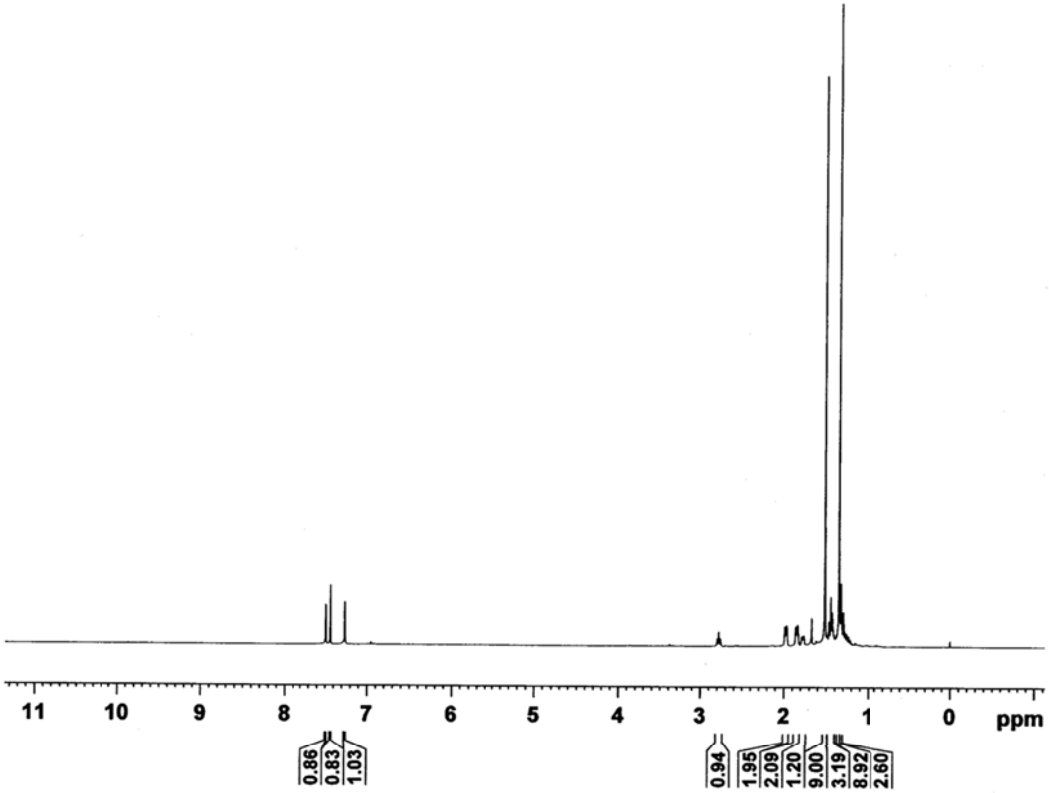
F2 - Acquisition Parameters
Date_           20150305
Time              12.26
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                32768
SOLVENT           CDCl3
NS                  201
DS                    2
SWH           36057.691 Hz
FIDRES         1.100393 Hz
AQ            0.4543829 sec
RG               200.18
DW               13.867 usec
DE                6.50 usec
TE               298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9279571 MHz
NUC1               13C
P1               10.50 usec
PLW1        95.00000000 W

======== CHANNEL f2 ========
SFO2        600.1724007 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            70.00 usec
PLW2        21.00000000 W
PLW12        0.61714000 W
PLW13        0.30239999 W

F2 - Processing parameters
SI                16384
SF          150.9128372 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```

6,8-Di-*tert*-butyl-3-cyclohexyl-2*H*-chromen-2-one (4a): ^{1}H NMR ($CDCl_3$, 600 MHz)

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Current Data Parameters
NAME      SS-6,8-tBu-DTBP-1H
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20150318
Time                 10.33
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              12019.230 Hz
FIDRES           0.183399 Hz
AQ               2.7262976 sec
RG                   31.5
DW                 41.600 usec
DE                  6.50 usec
TE                 297.7 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          600.1737063 MHz
NUC1                   1H
P1                  12.00 usec
PLW1          21.00000000 W

F2 - Processing parameters
SI                  65536
SF            600.1700142 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```
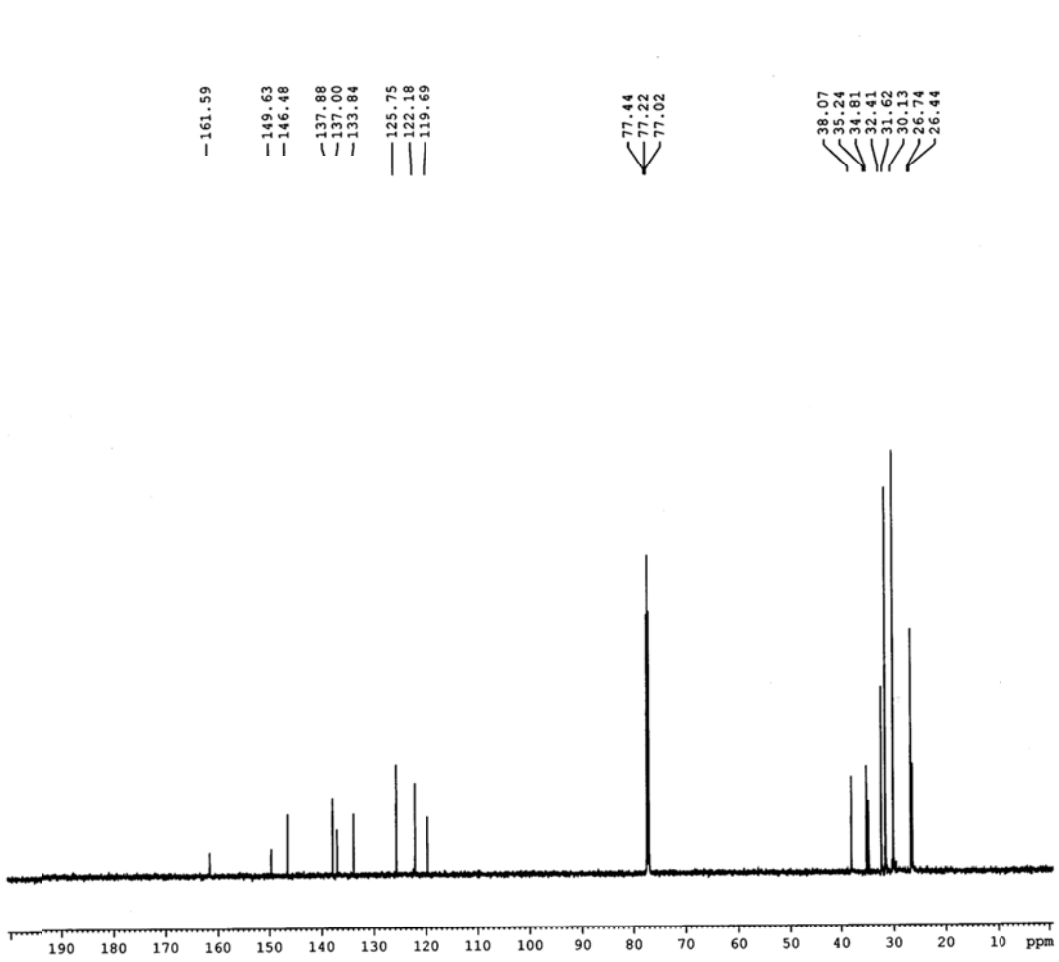

6,8-Di-*tert*-butyl-3-cyclohexyl-2*H*-chromen-2-one (4a): ^{13}C NMR ($CDCl_3$, 150 MHz)



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Current Data Parameters
NAME      SS-6,8-tBu-DTBP-13C
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150318
Time                 10.25
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   32768
SOLVENT              CDCl3
NS                     120
DS                       2
SWH              36057.691 Hz
FIDRES            1.100393 Hz
AQ               0.4543829 sec
RG                   65.24
DW                  13.867 usec
DE                    6.50 usec
TE                   298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           150.9279571 MHz
NUC1                   13C
P1                   10.50 usec
PLW1           95.00000000 W

======== CHANNEL f2 ========
SFO2           600.1724007 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                70.00 usec
PLW2           21.00000000 W
PLW12           0.61714000 W
PLW13           0.30239999 W

F2 - Processing parameters
SI                     16384
SF             150.9128381 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
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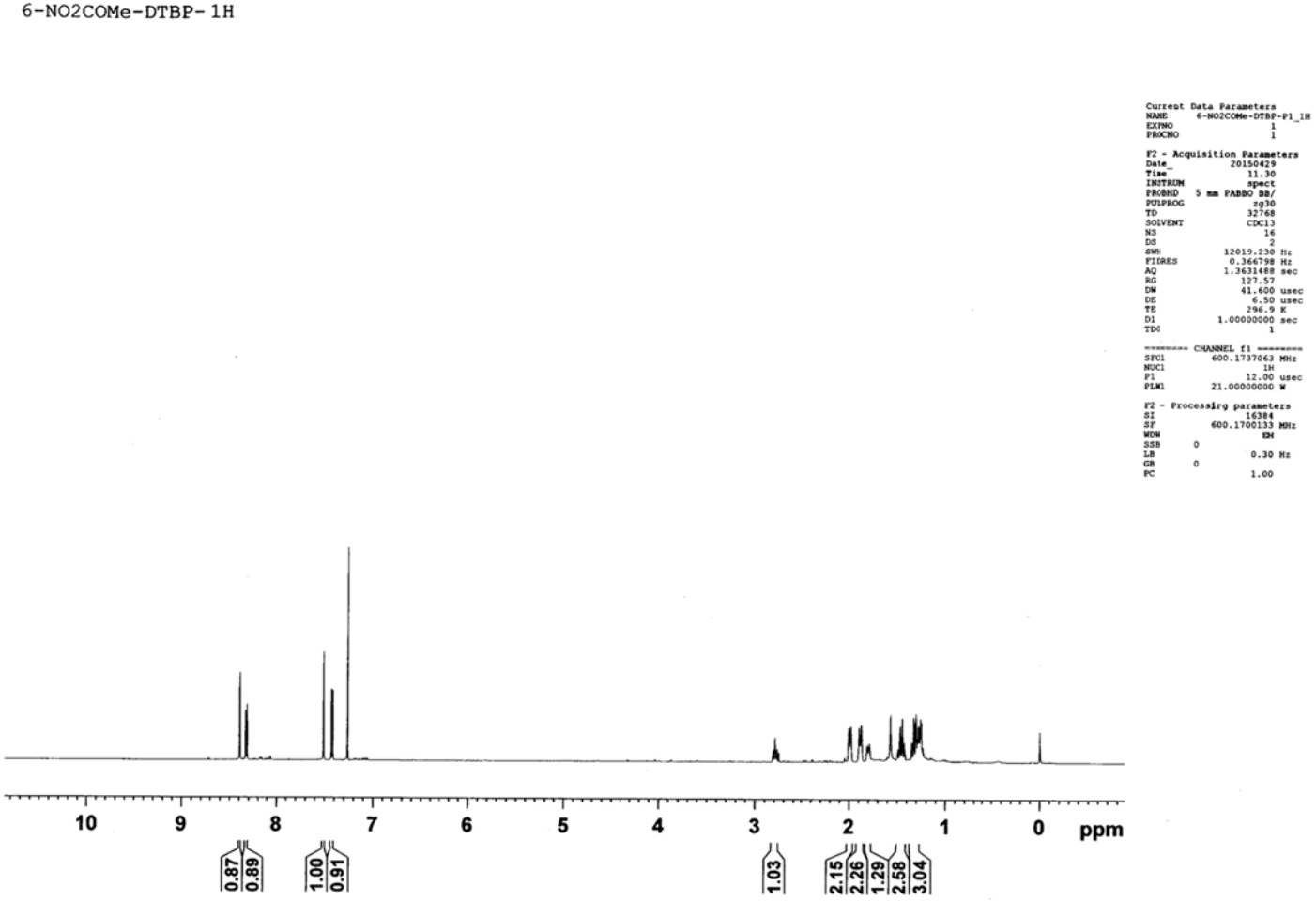
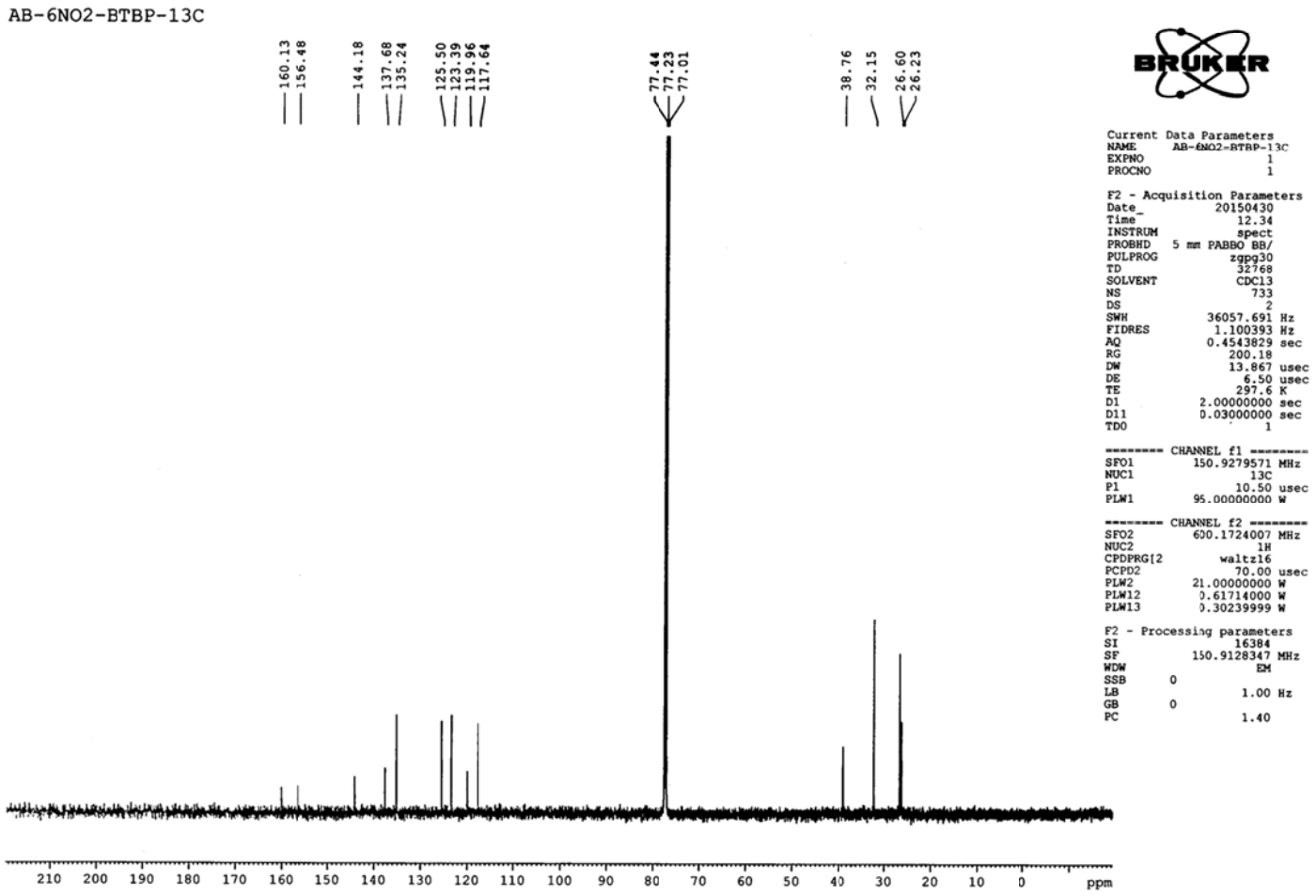
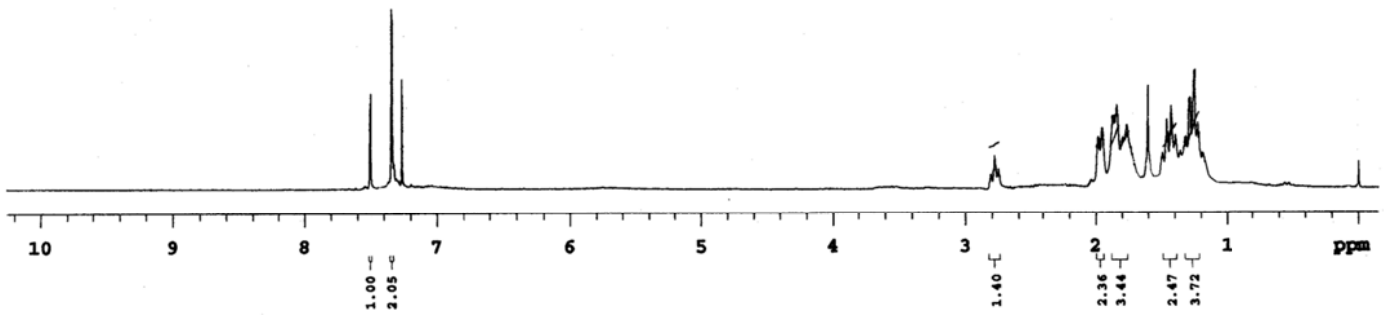
3-Cyclohexyl-6-nitro-2*H*-chromen-2-one (5a): ^{1}H NMR ($CDCl_3$, 600 MHz)



3-Cyclohexyl-6-nitro-2*H*-chromen-2-one (5a): ^{13}C NMR ($CDCl_3$, 150 MHz)

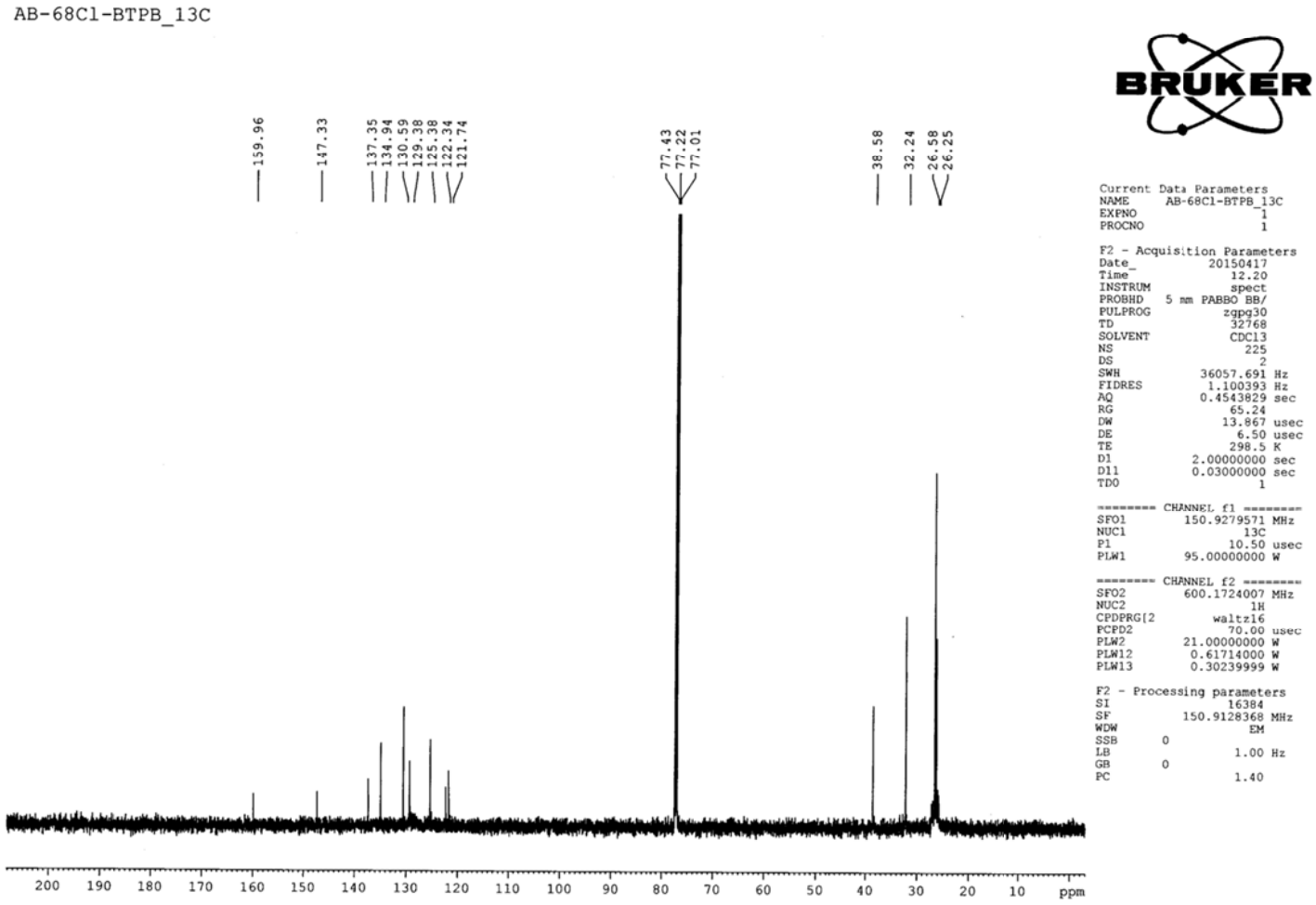


6,8-Dichloro-3-cyclohexyl-2*H*-chromen-2-one (6a): ^{1}H NMR ($CDCl_3$, 600 MHz)



PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509609	DATA PROCESSING FT size 32768 Total time 1 minutes		AB-68ClCOMe-DTSP-P1-1H Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem Mercury-400 "IITG-NMR"

6,8-Dichloro-3-cyclohexyl-2*H*-chromen-2-one (6a): ^{13}C NMR ($CDCl_3$, 150 MHz)



BRUKER

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Current Data Parameters
NAME      AB-68Cl-BTPB_13C
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
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Time                 12.20
INSTRUM              spect
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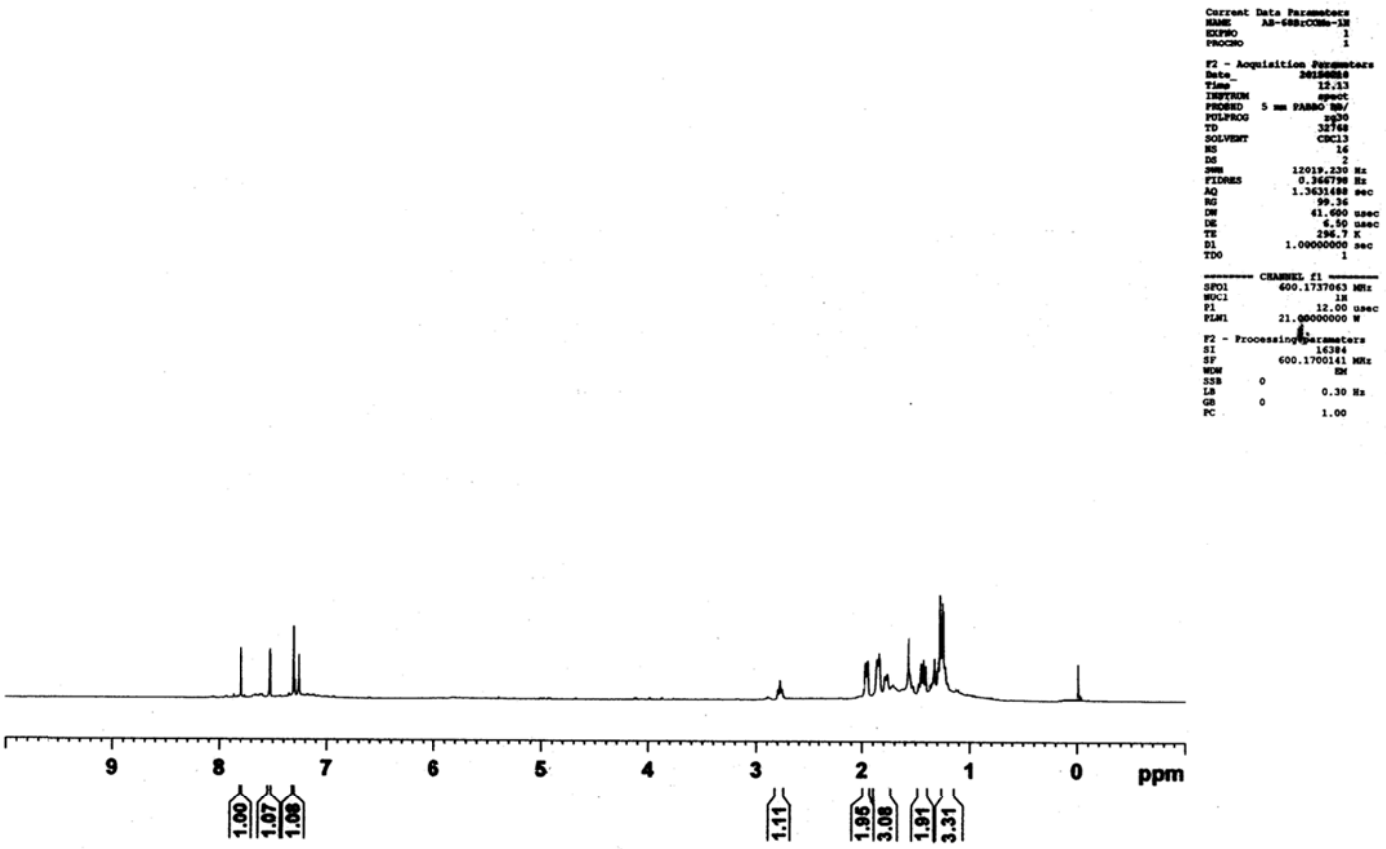
PULPROG           zgpg30
TD                  32768
SOLVENT             CDCl3
NS                    225
DS                      2
SWH              36057.691 Hz
FIDRES            1.100393 Hz
AQ               0.4543829 sec
RG                   65.24
DW                  13.867 usec
DE                    6.50 usec
TE                  298.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                  13C
P1                  10.50 usec
PLW1           95.00000000 W

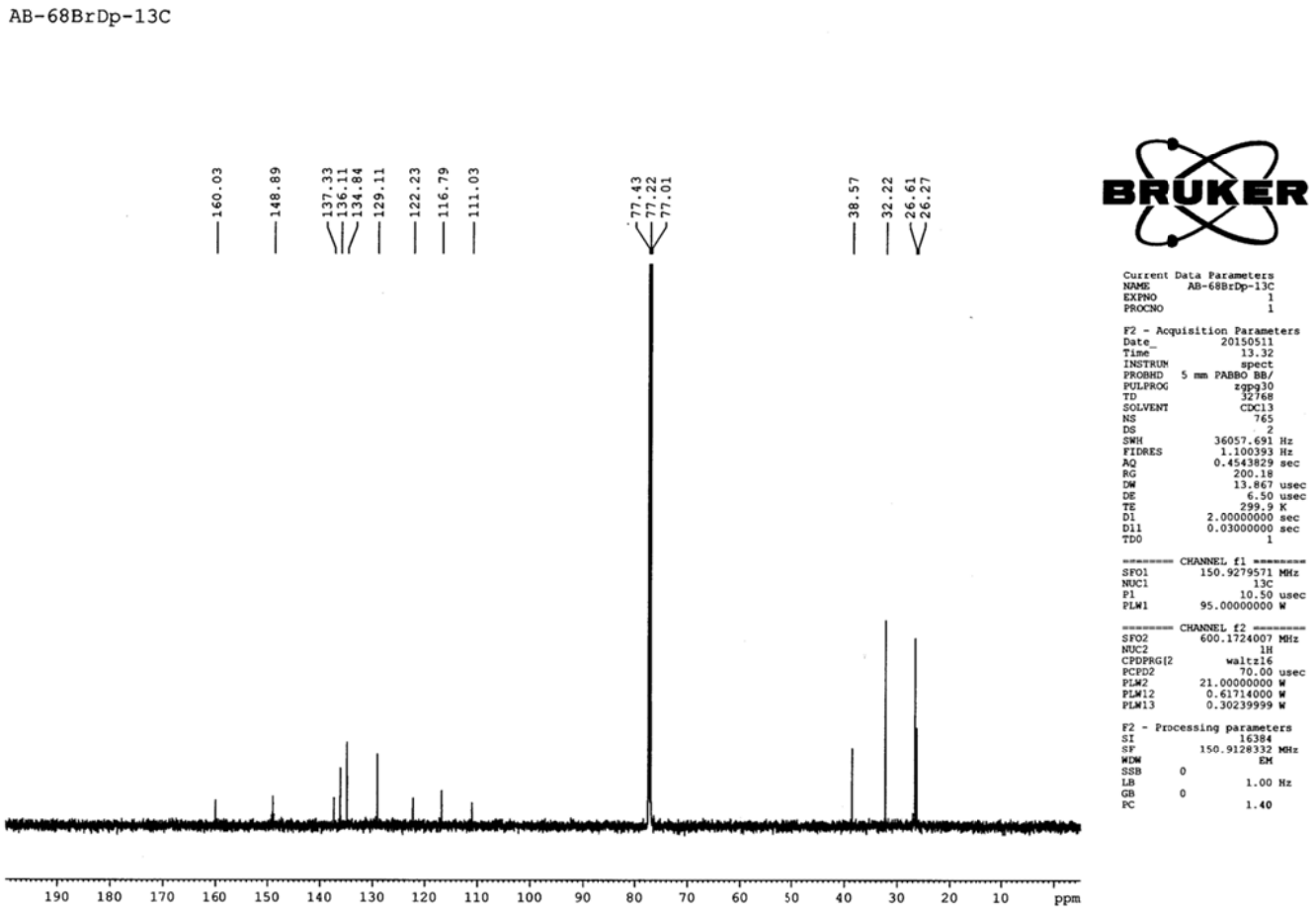
======== CHANNEL f2 ========
SFO2          600.1724007 MHz
NUC2                  1H
CPDPRG[2           waltz16
PCPD2               70.00 usec
PLW2           21.00000000 W
PLW12           0.61714000 W
PLW13           0.30239999 W

F2 - Processing parameters
SI                   16384
SF            150.9128368 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```

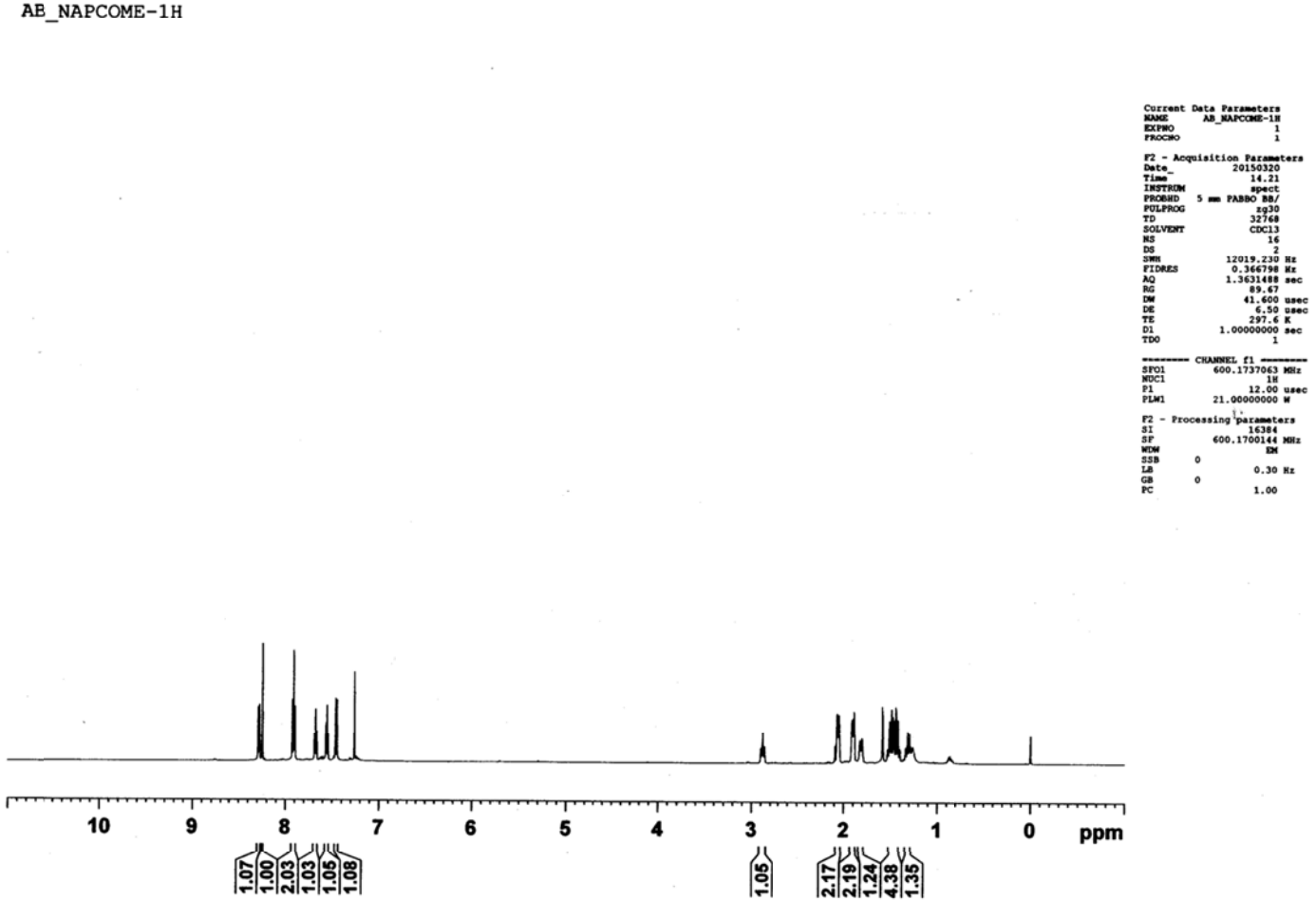
6,8-Bromo-3-cyclohexyl-2*H*-chromen-2-one (7a): ^{1}H NMR ($CDCl_3$, 600 MHz)



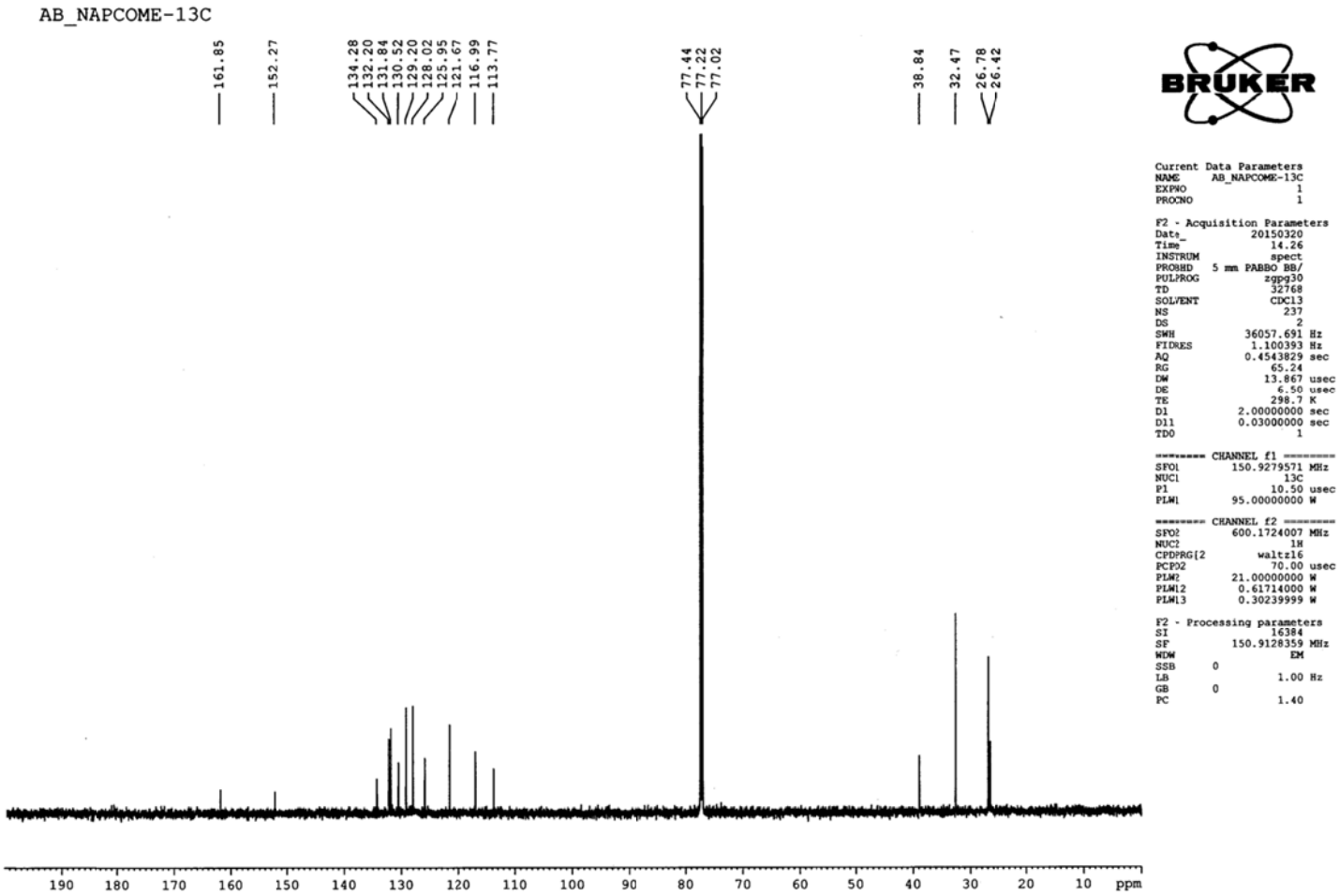
6,8-Bromo-3-cyclohexyl-2*H*-chromen-2-one (7a):^{13}C NMR ($CDCl_3$, 150 MHz)



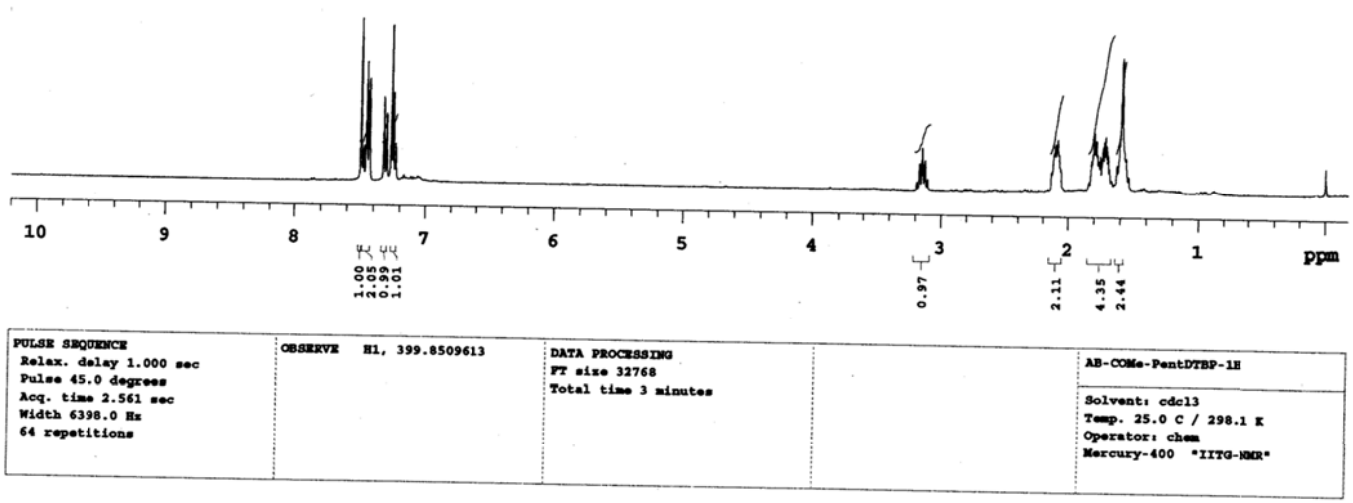
2-Cyclohexyl-3*H*-benzo[*f*]chromen-3-one (8a): ^{1}H NMR ($CDCl_3$, 600 MHz)



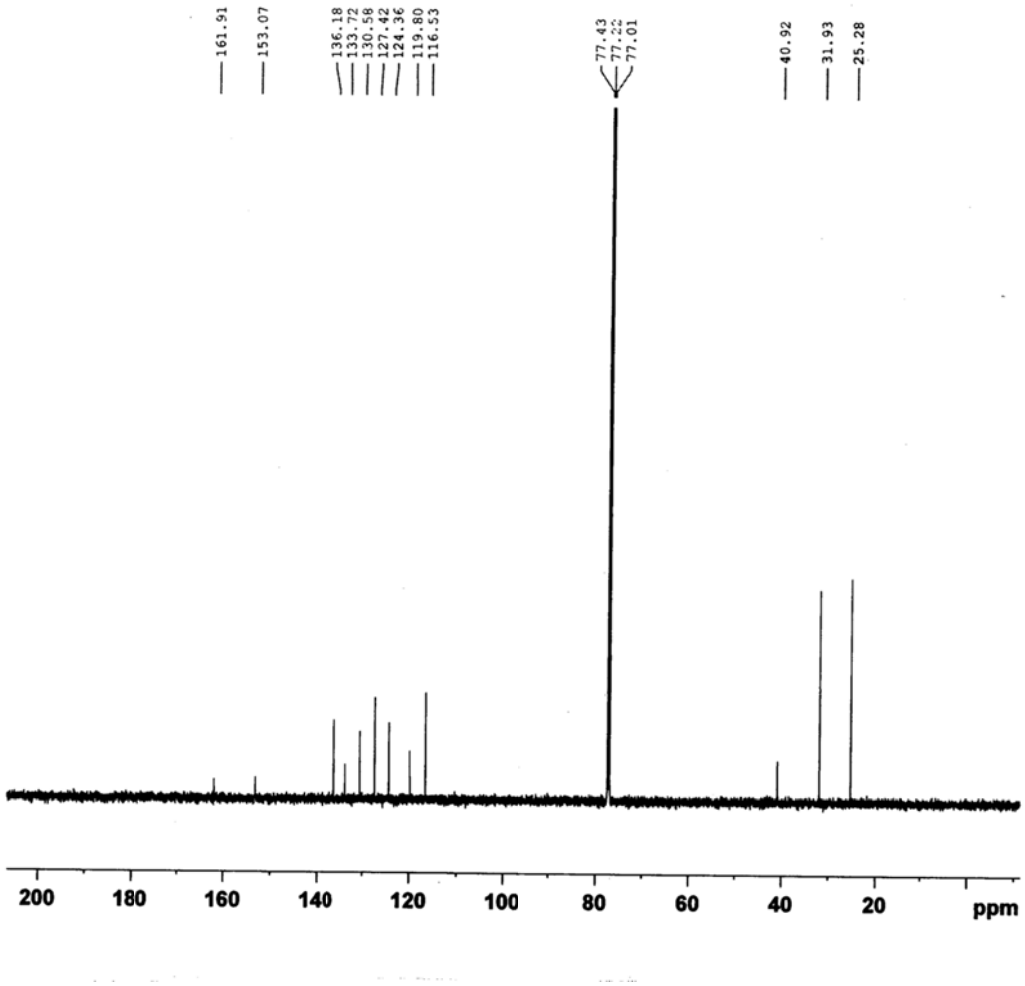
2-Cyclohexyl-3*H*-benzo[*f*]chromen-3-one (8a): ^{13}C NMR ($CDCl_3$, 150 MHz)



3-Cyclopentyl-2*H*-chromen-2-one (1b): ^{1}H NMR ($CDCl_3$, 600 MHz)



3-Cyclopentyl-2*H*-chromen-2-one (1b): ^{13}C NMR ($CDCl_3$, 150 MHz)

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PROCNO                        1

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INSTRUM               spect
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PULPROG             zgpg30
TD                    32768
SOLVENT               CDCl3
NS                      274
DS                        2
SWH               36057.691 Hz
FIDRES             1.100393 Hz
AQ                 0.4543829 sec
RG                    200.1
DW                   13.867 usec
DE                     6.50 usec
TE                   298.8 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1          150.9279571 MHz
NUC1                    13C
P1                    10.50 usec
PLW1          95.00000000 W

======== CHANNEL f2 ========
SFO2          600.1724007 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 70.00 usec
PLW2          21.00000000 W
PLW12          0.61714000 W
PLW13          0.30239999 W

F2 - Processing parameters
SI                     16384
SF            150.9128358 MHz
WDW                       EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
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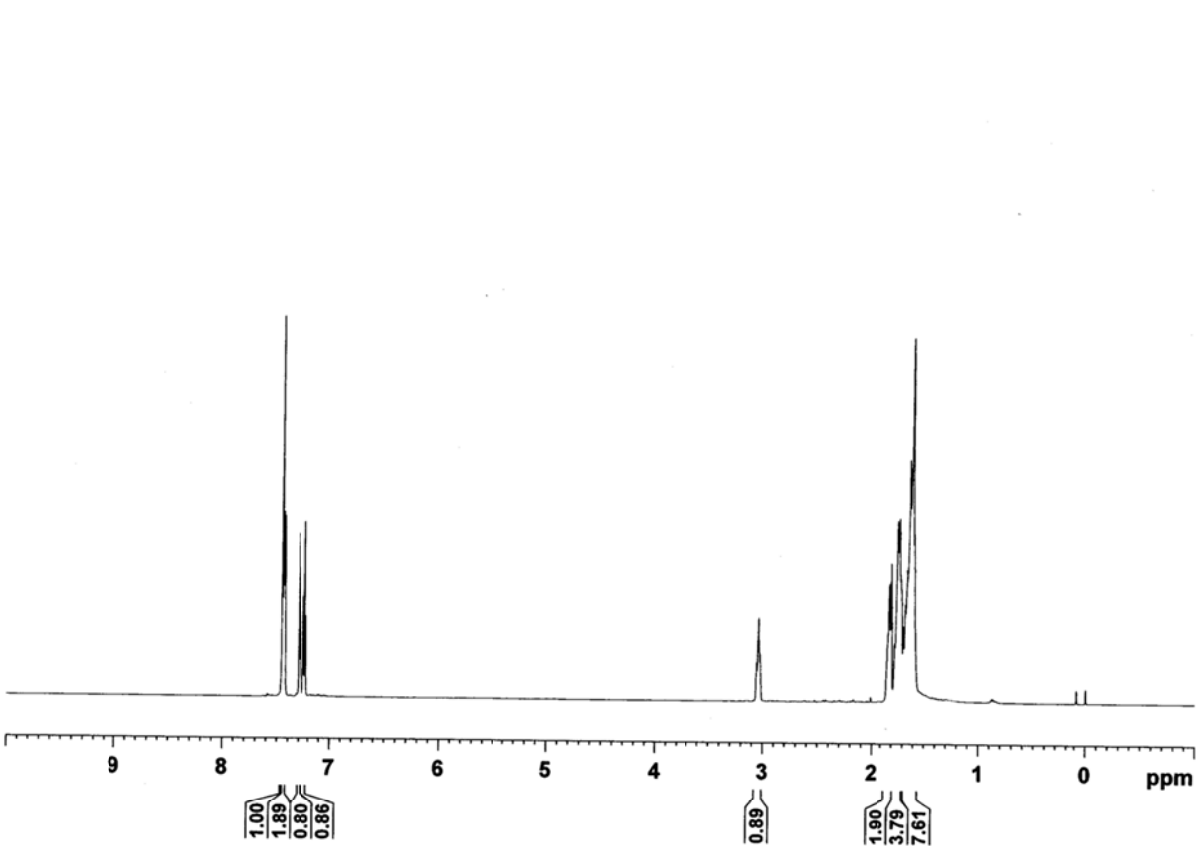
3-Cyclooctyl-2*H*-chromen-2-one (1c): ^{1}H NMR ($CDCl_3$, 600 MHz)

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EXPNO                    1
PROCNO                   1

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Time                 15.36
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PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
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DS                      2
SWH              12019.230 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                   18.03
DW                  41.600 usec
DE                   6.50 usec
TE                  297.1 K
D1               1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
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NUC1                   1H
P1                  12.00 usec
PLW1        21.00000000 W

F2 - Processing parameters
SI                  16384
SF           600.1700051 MHz
WDW                    EM
SSB      0
LB                   0.30 Hz
GB       0
PC                   1.00
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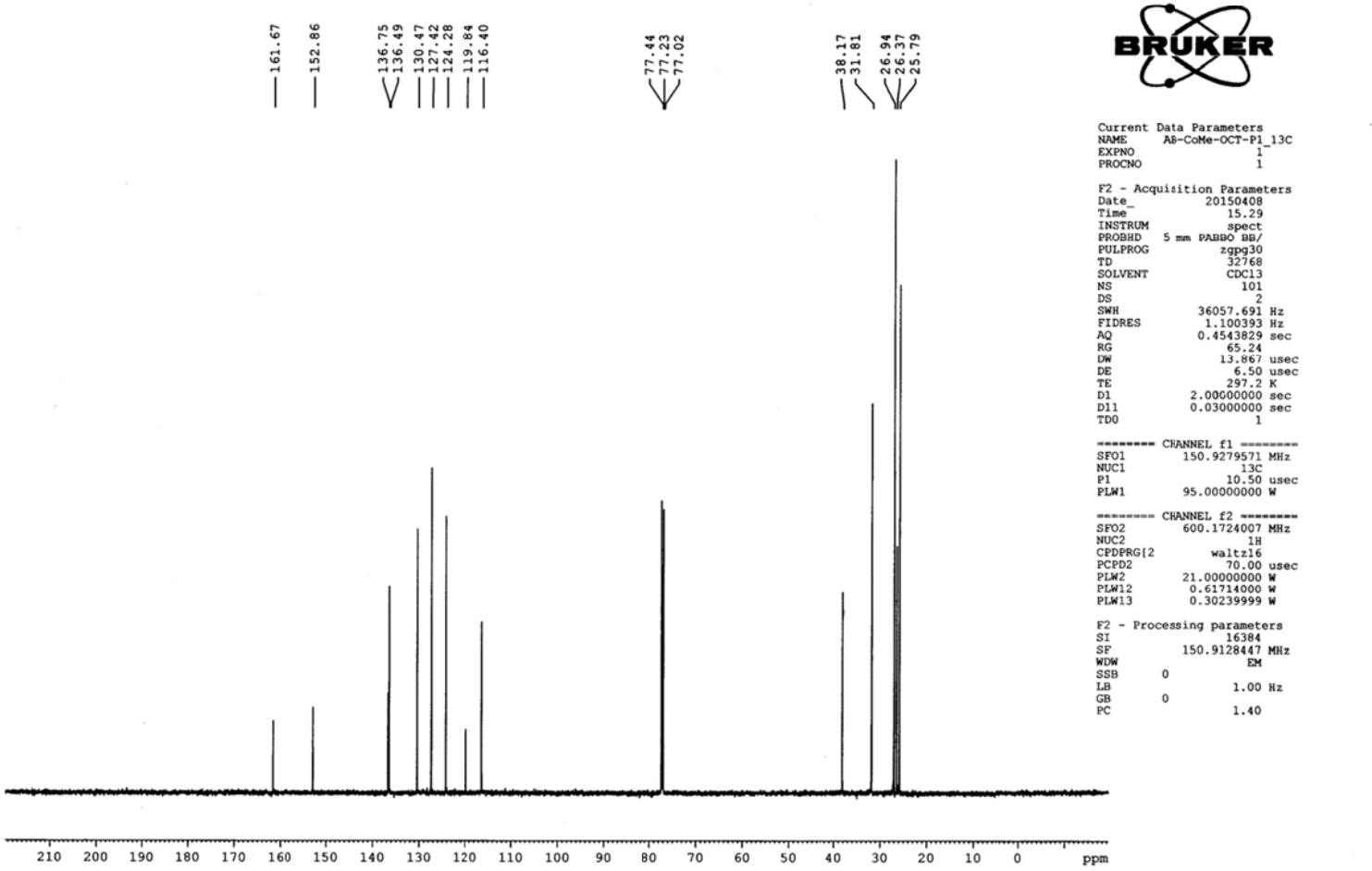
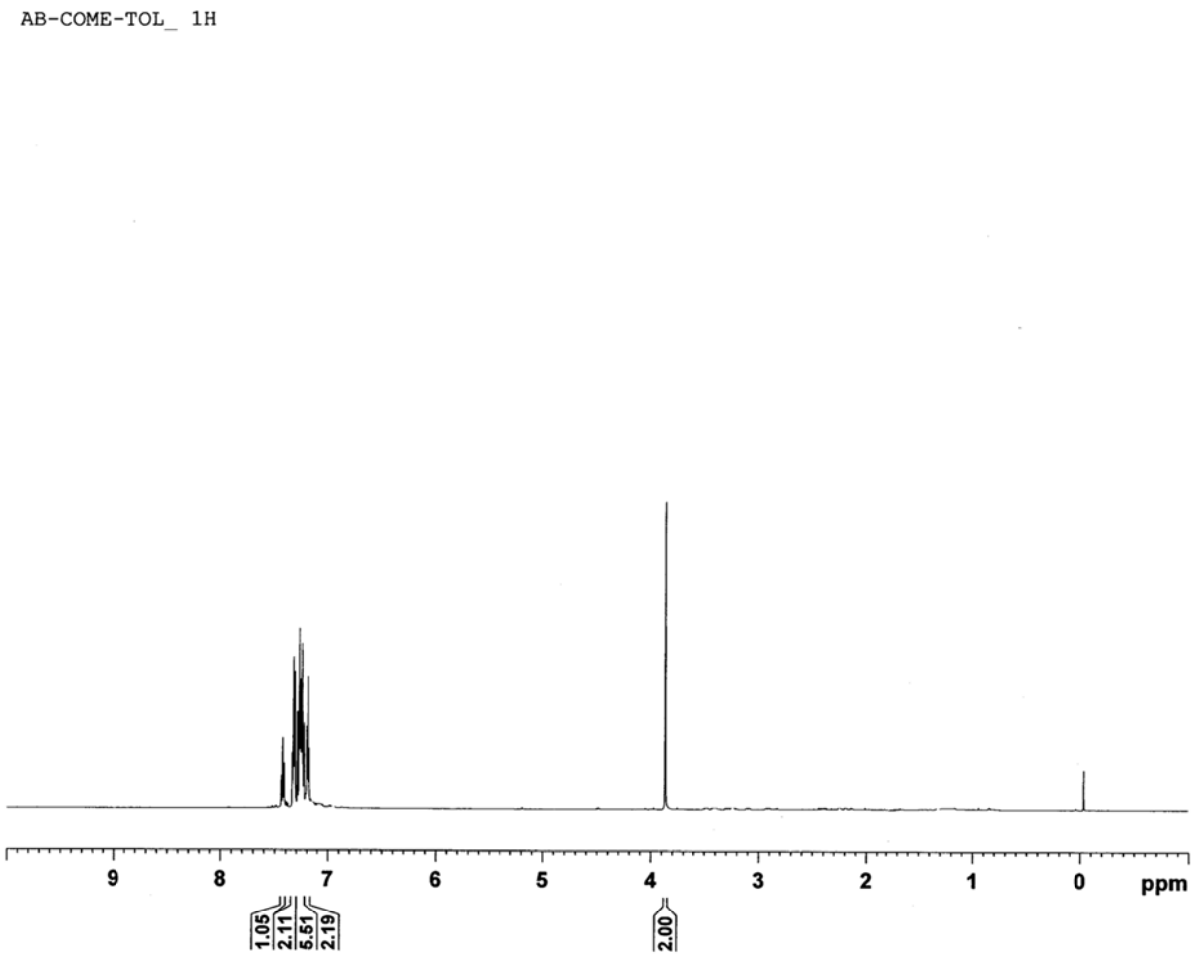
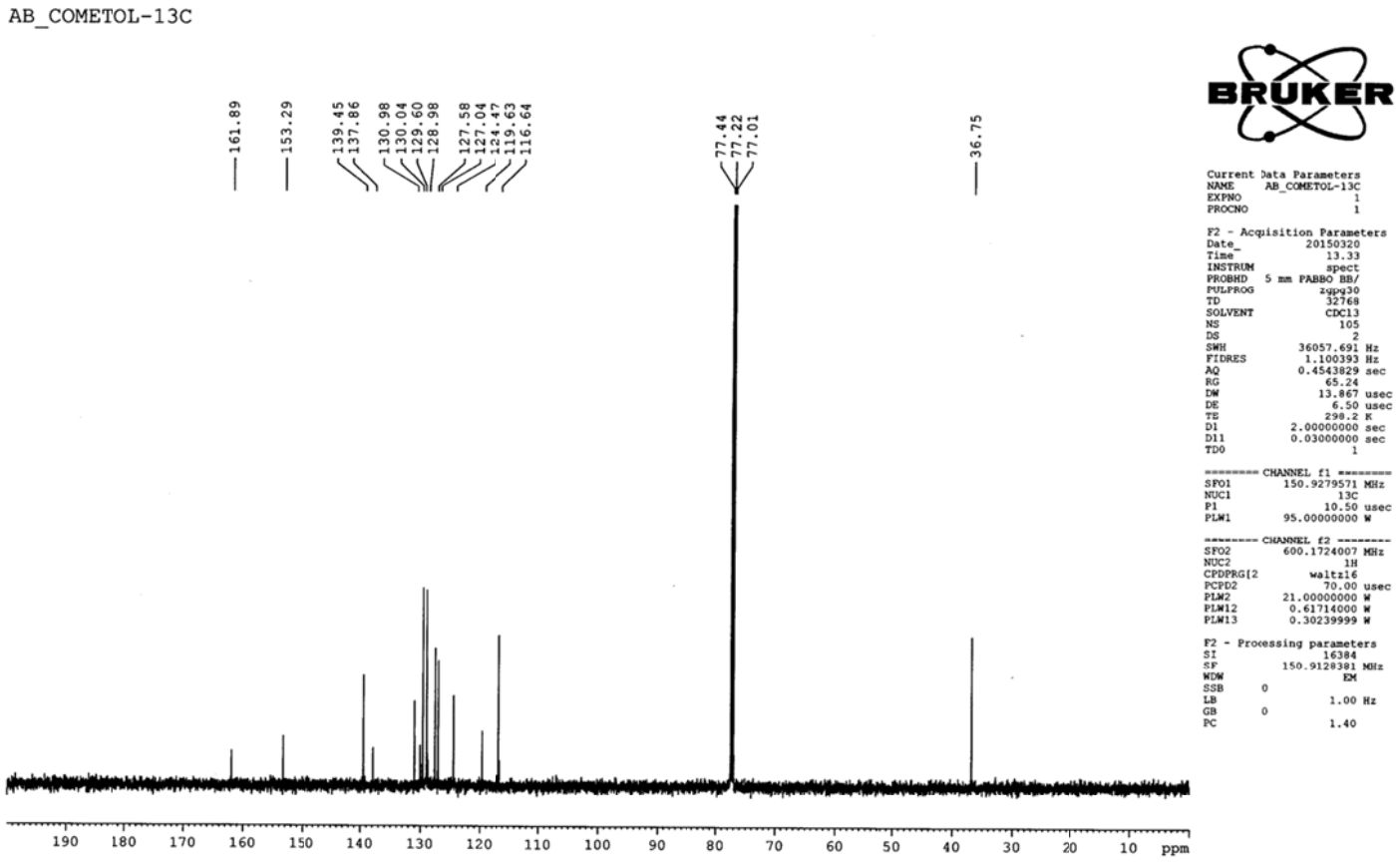
3-Cyclooctyl-2*H*-chromen-2-one (1c): ^{13}C NMR ($CDCl_3$, 150 MHz)



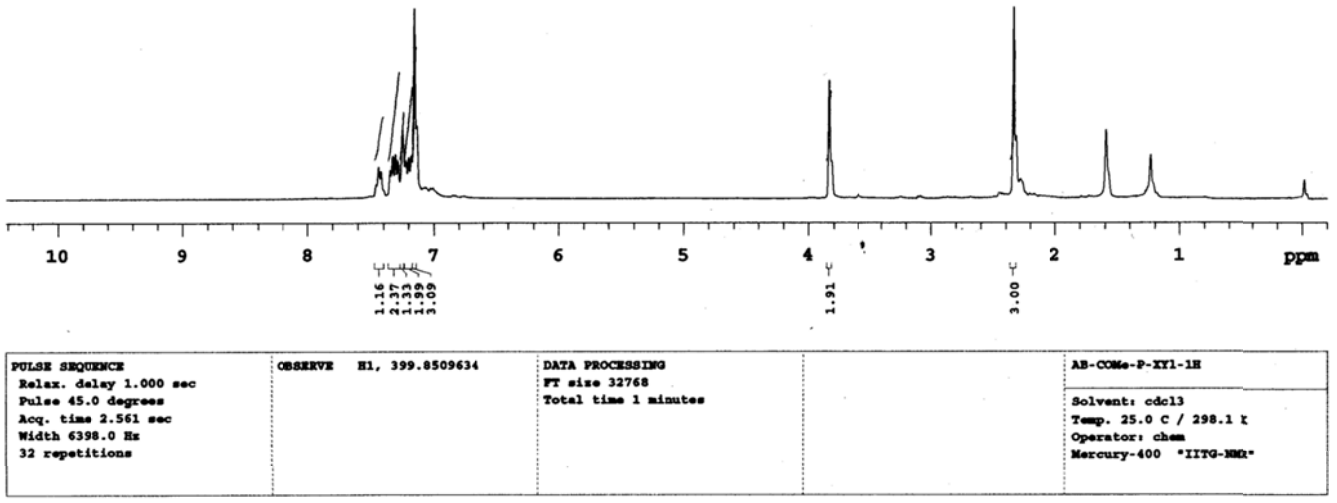
3-Benzyl-2*H*-chromen-2-one (1d): ^{1}H NMR ($CDCl_3$, 600 MHz)



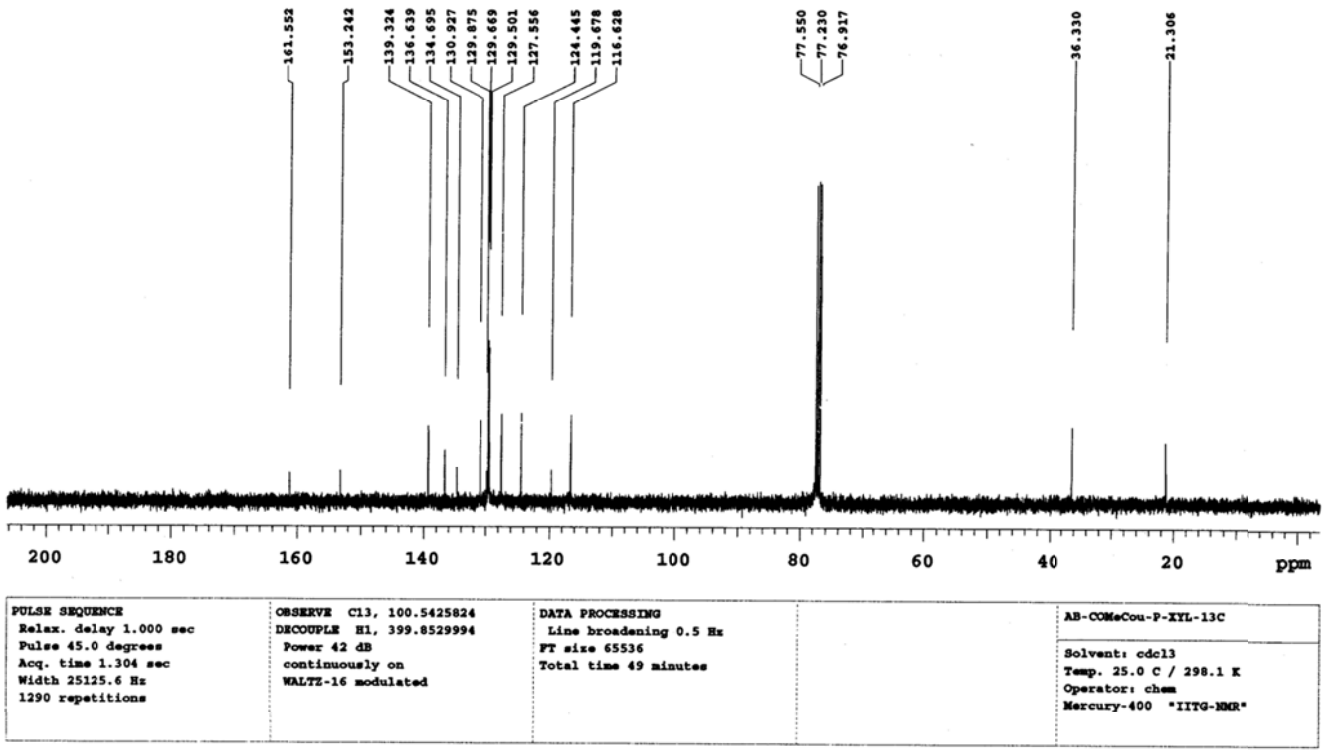
3-Benzyl-2*H*-chromen-2-one (1d): ^{13}C NMR ($CDCl_3$, 150 MHz)



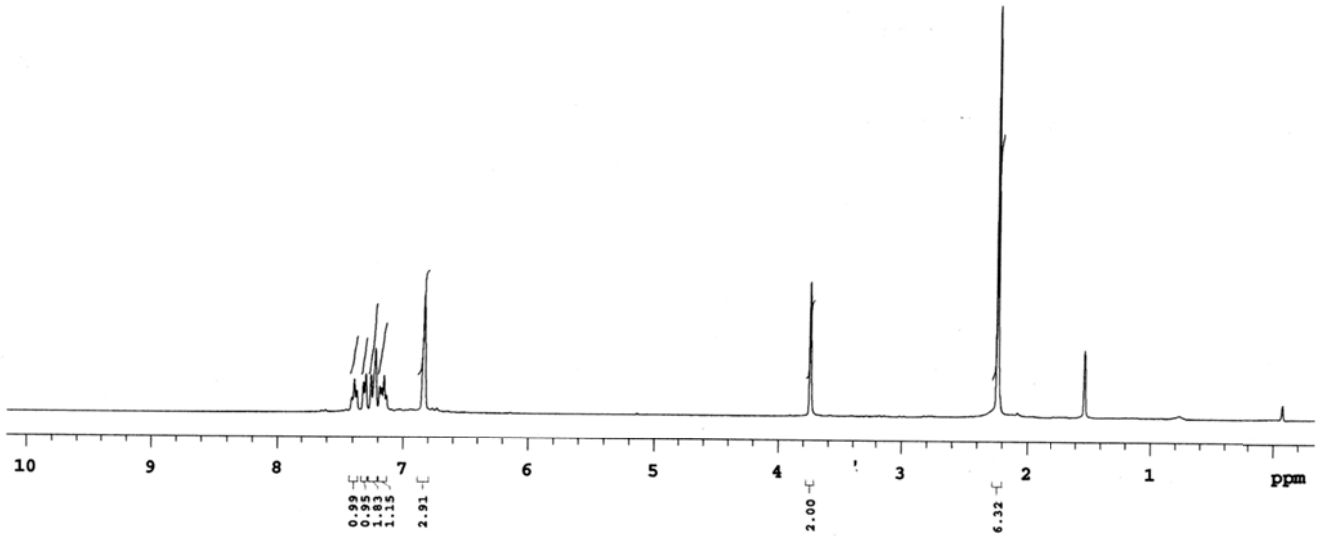
3-(4-Methylbenzyl)-2*H*-chromen-2-one (1e): ^{1}H NMR ($CDCl_3$, 400 MHz)



3-(4-Methylbenzyl)-2*H*-chromen-2-one (1e): ^{13}C NMR ($CDCl_3$, 100 MHz)

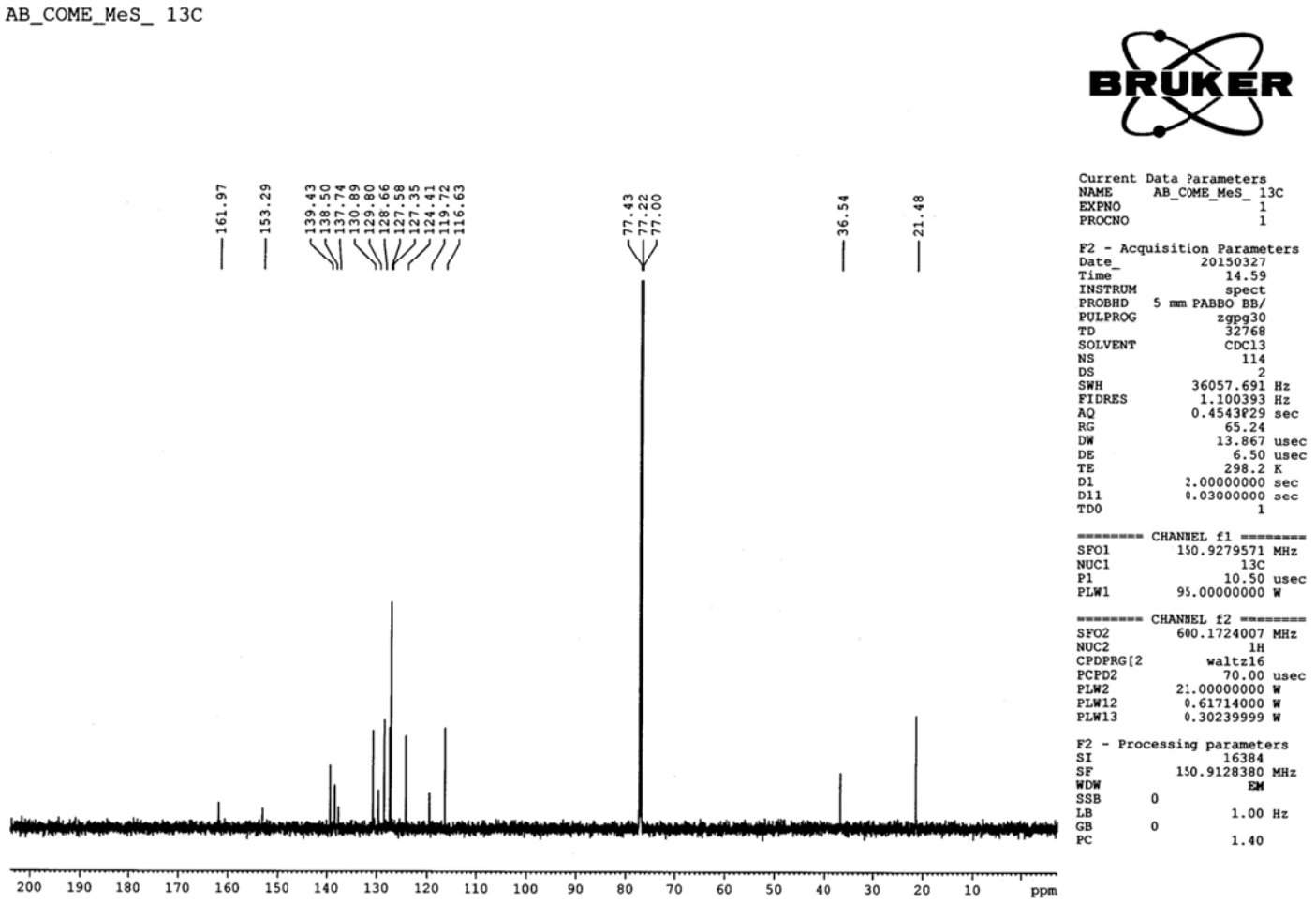


3-(3,5-Dimethylbenzyl)-2*H*-chromen-2-one (1f): ^{1}H NMR ($CDCl_3$, 400 MHz)



PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509882	DATA PROCESSING FT size 32768 Total time 1 minutes		AB-COMe-Mes-DTBP-1H Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: AB-COMe-Mes-DTBP-1H Mercury-400 "IITG-NMR"

3-(3,5-Dimethylbenzyl)-2*H*-chromen-2-one (1f): ^{13}C NMR ($CDCl_3$, 150 MHz)

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PROCNO                  1

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PULPROG          zgpg30
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SOLVENT           CDCl3
NS                  114
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SWH           36057.691 Hz
FIDRES         1.100393 Hz
AQ            0.4543929 sec
RG                65.24
DW               13.867 usec
DE                6.50 usec
TE                298.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        150.9279571 MHz
NUC1                13C
P1                10.50 usec
PLW1        95.00000000 W

======== CHANNEL f2 ========
SFO2        600.1724007 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             70.00 usec
PLW2        21.00000000 W
PLW12        0.61714000 W
PLW13        0.30239999 W

F2 - Processing parameters
SI                16384
SF          150.9128380 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```
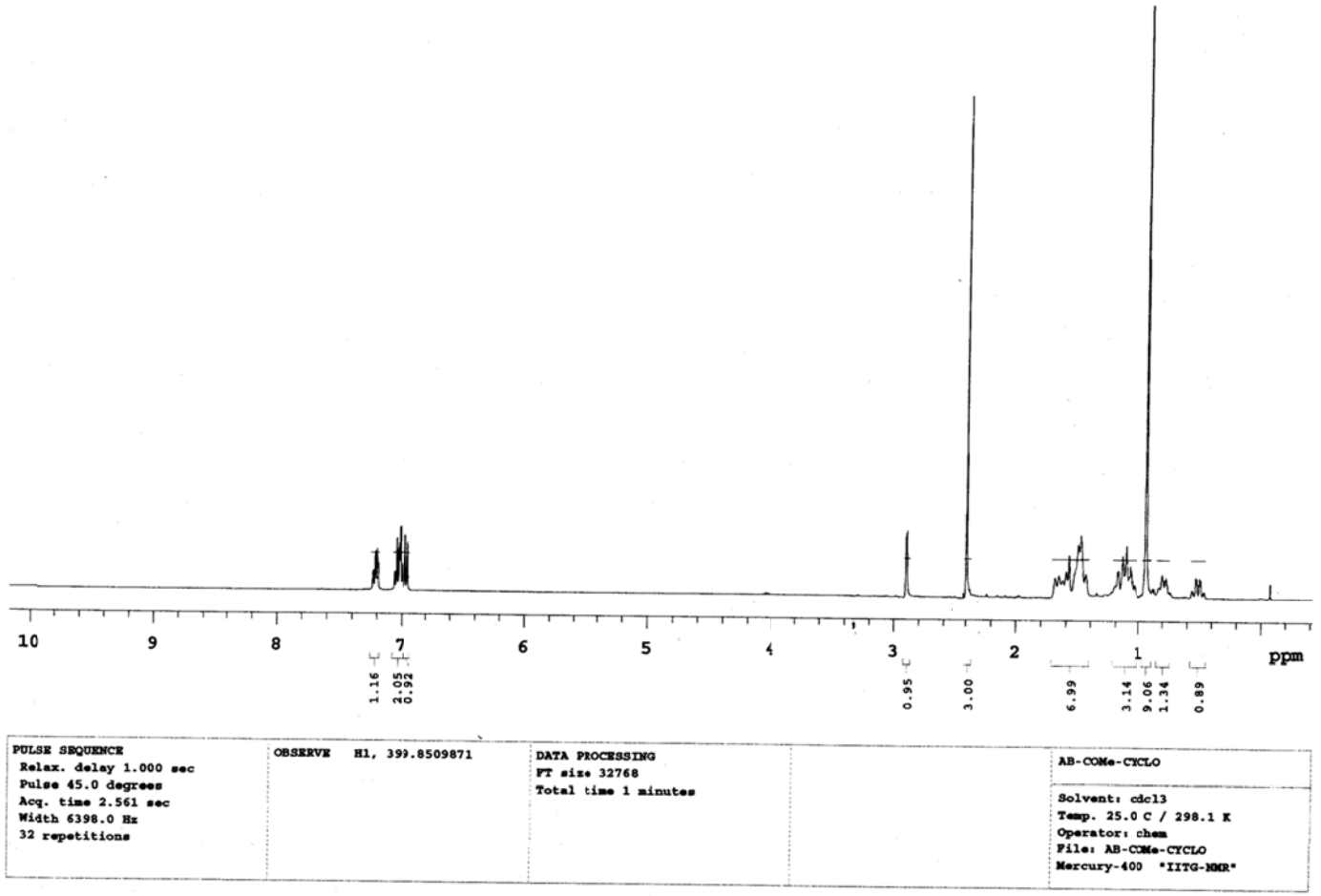
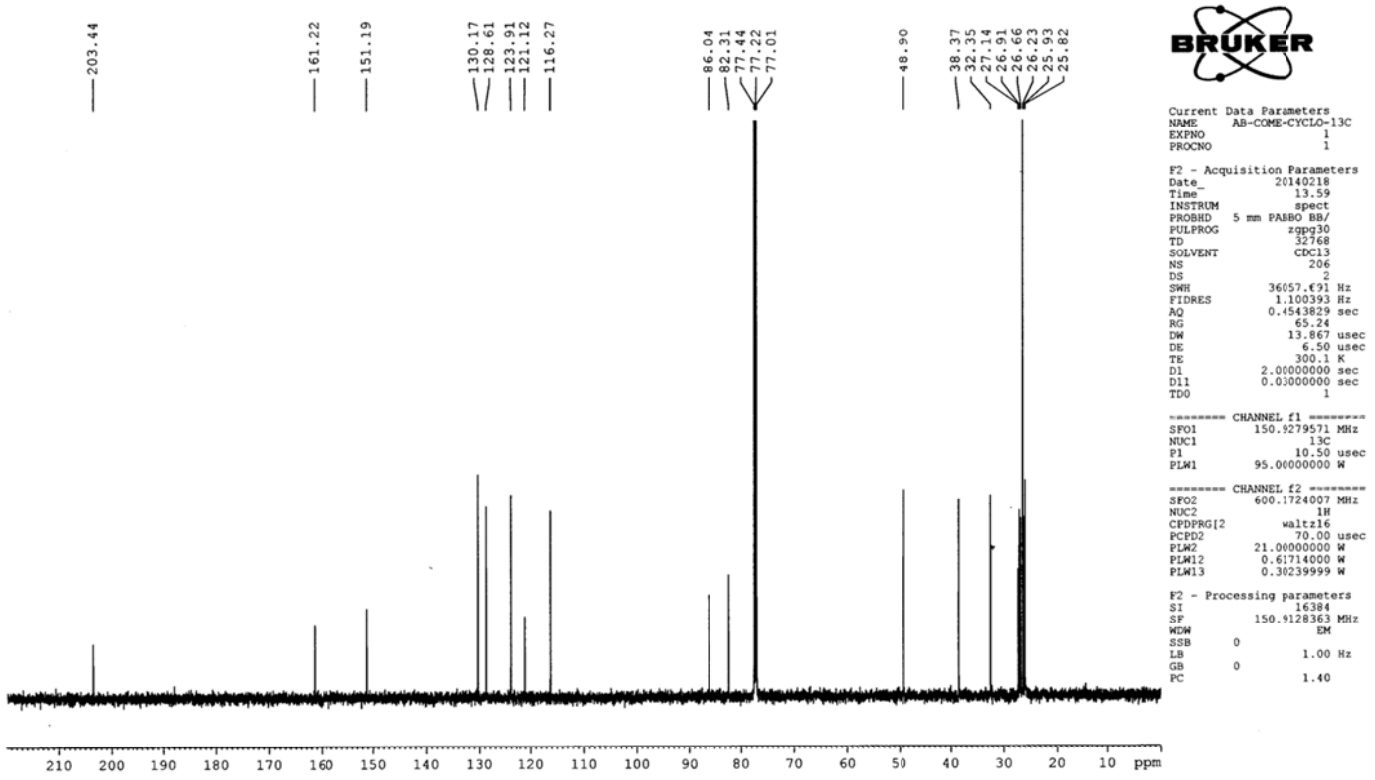

3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (1a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (1a'): ^{13}C NMR ($CDCl_3$, 150 MHz)

AB-COME-CYCLO-13C



BRUKER

Current Data Parameters
NAME AB-COME-CYCLO-13C
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PROCNO 1

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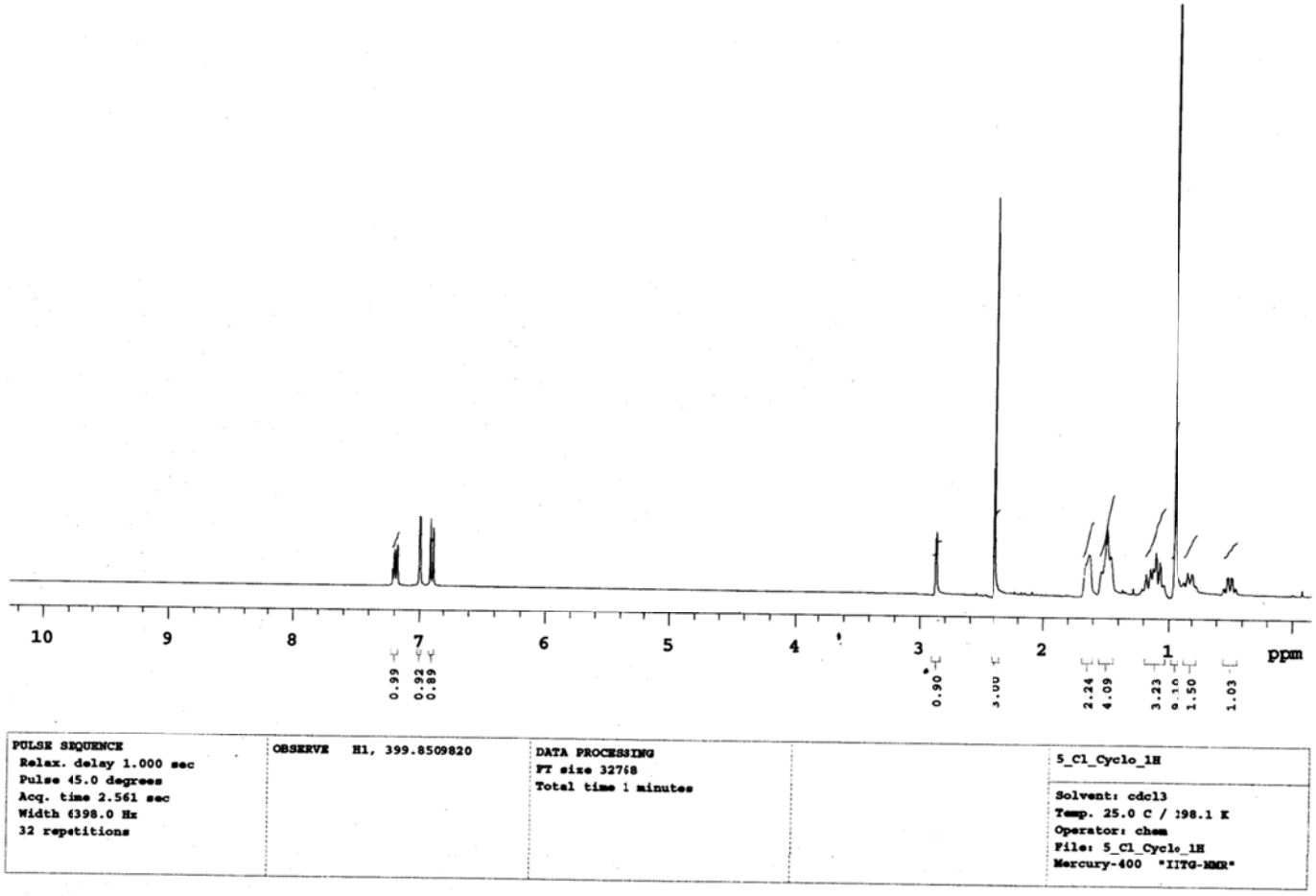
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 206
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

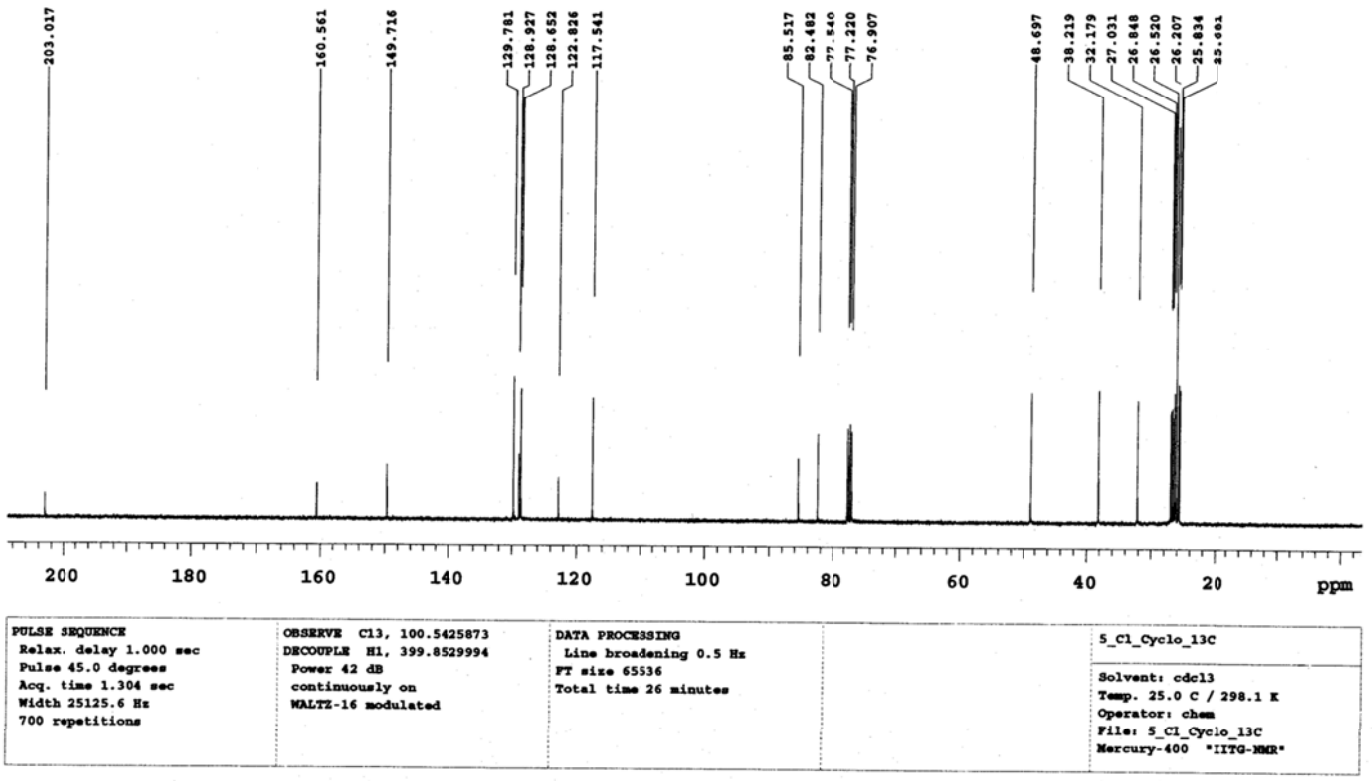
======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.9128363 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

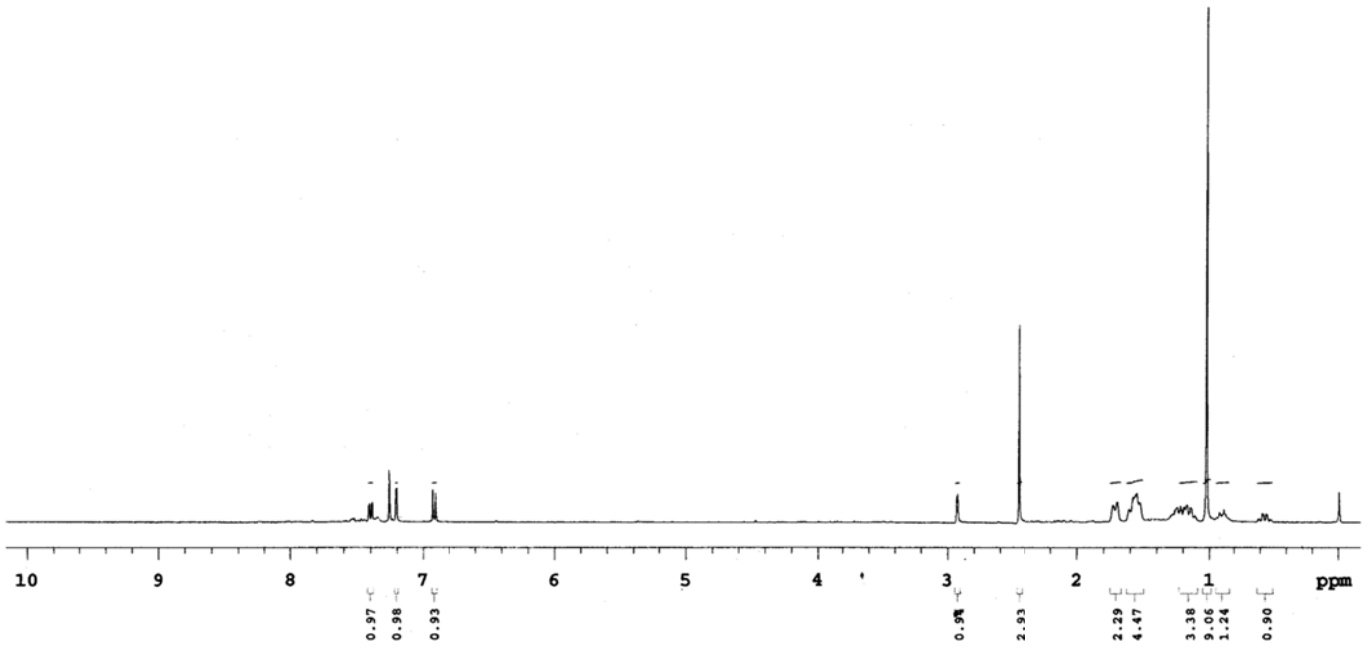
3-Acetyl-3-(*tert*-butylperoxy)-6-chloro-4-cyclohexylchroman-2-one (13a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



3-Acetyl-3-(*tert*-butylperoxy)-6-chloro-4-cyclohexylchroman-2-one (13a'): ^{13}C NMR ($CDCl_3$, 100 MHz)

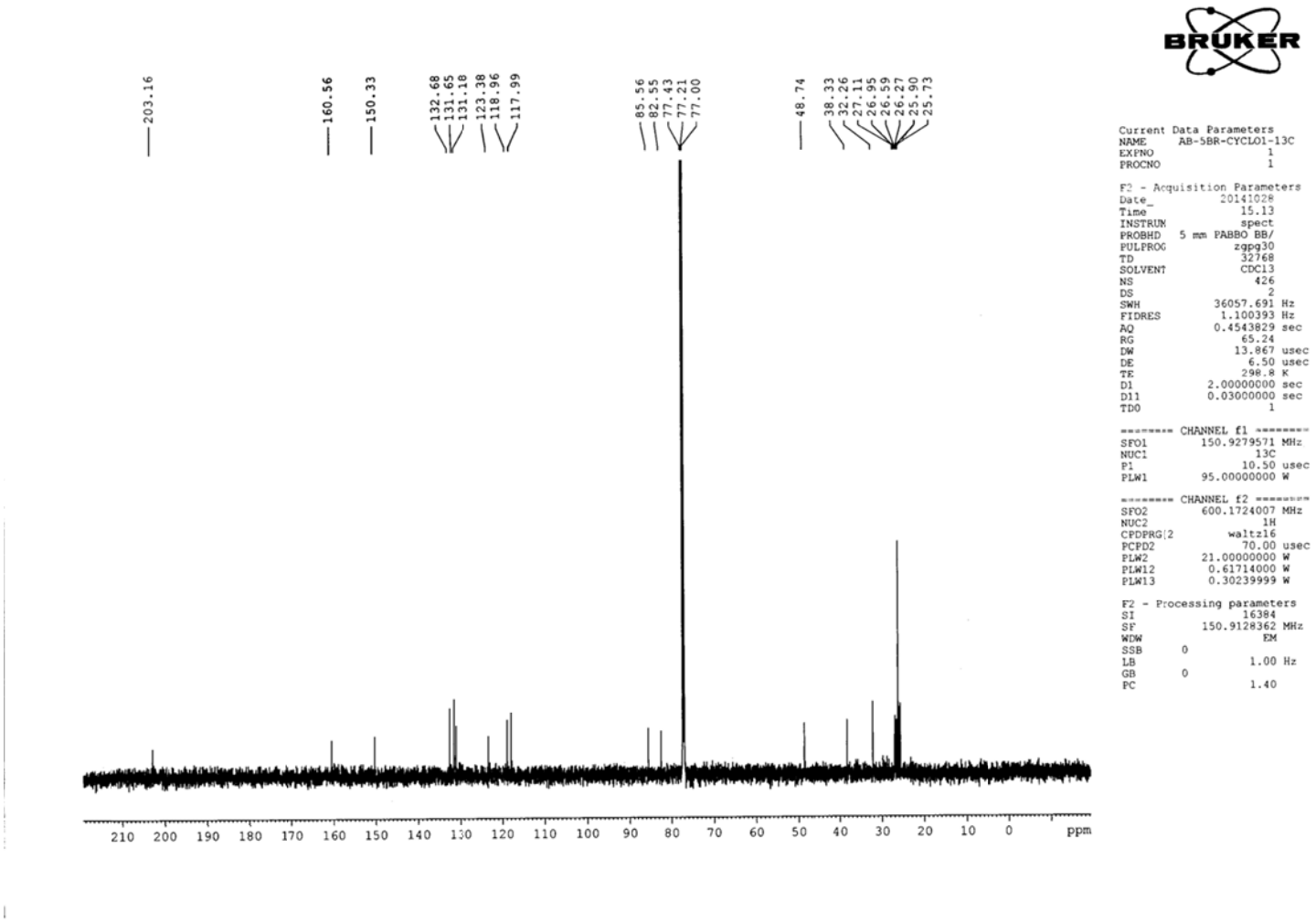


3-Acetyl-6-bromo-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (14a'): ^{1}H NMR ($CDCl_3$, 400 MHz)

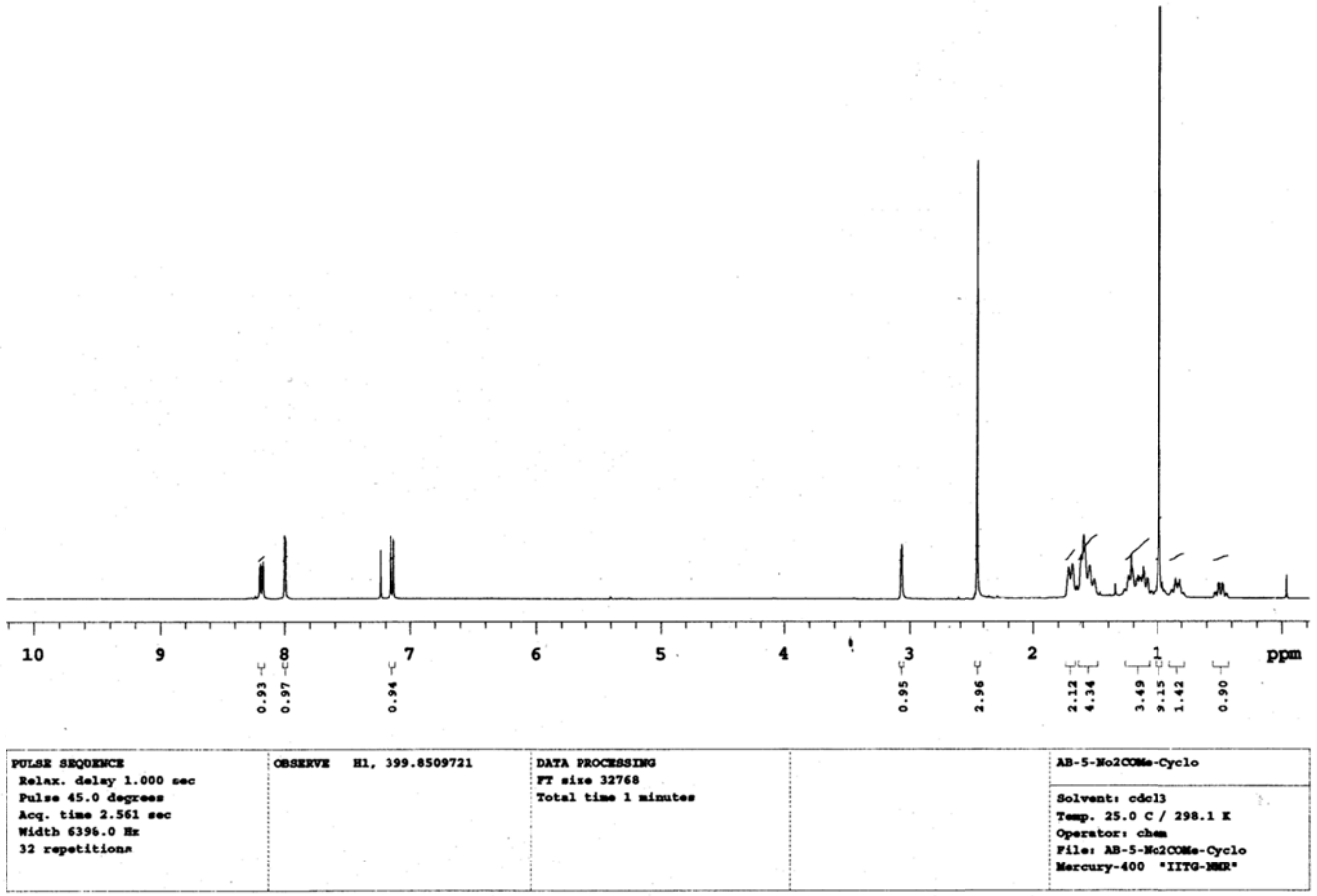


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509641	DATA PROCESSING FT size 32768 Total time 1 minutes		AB_5_Br_COMe_Cyclo_1H Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: AB_5_Br_COMe_Cyclo_1H Mercury-400 "IITG-NMR"

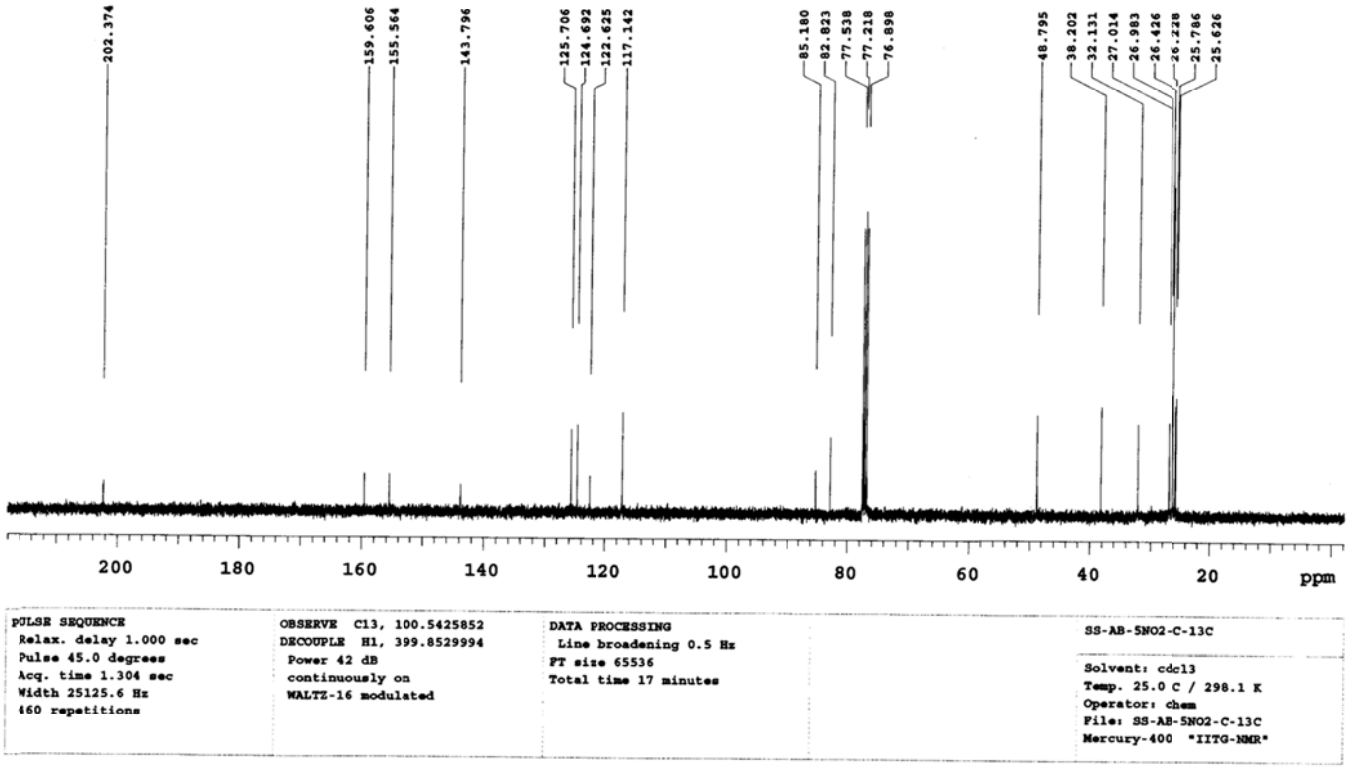
3-Acetyl-6-bromo-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (14a'): ^{13}C NMR ($CDCl_3$, 150 MHz)



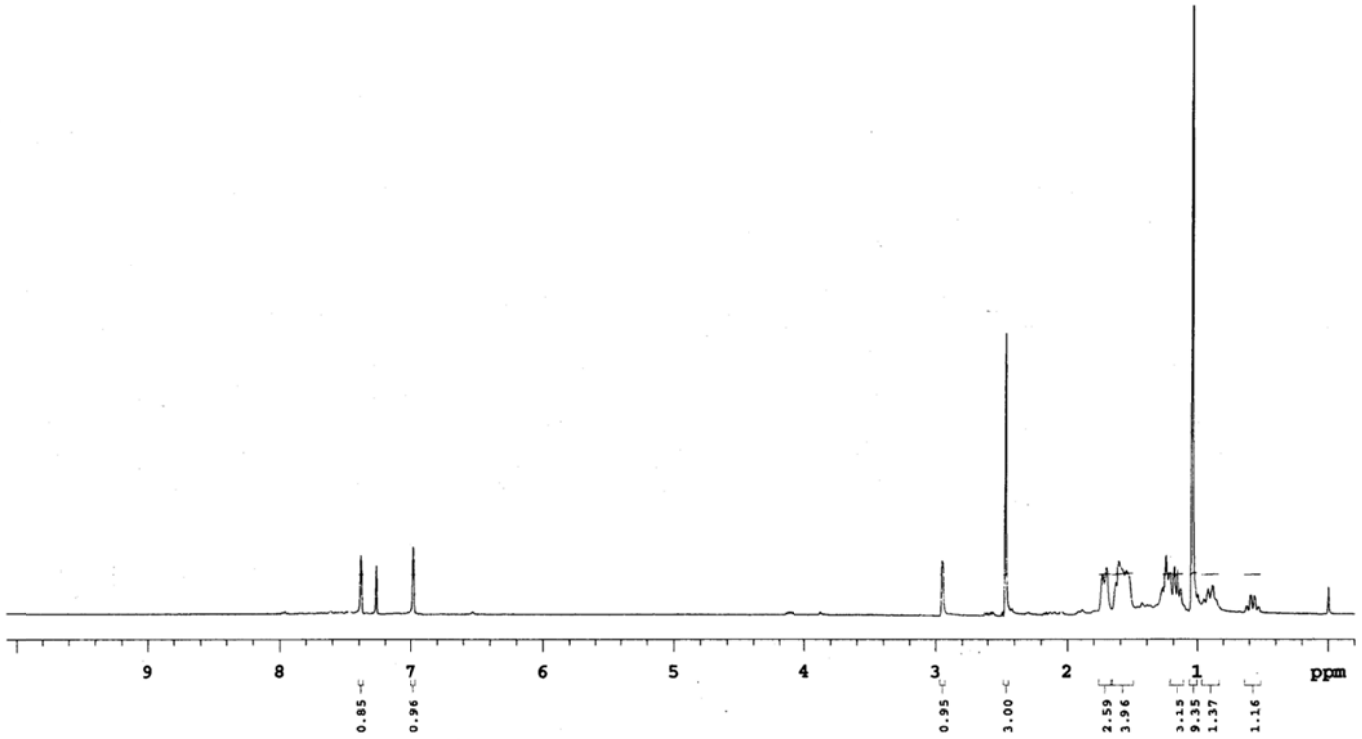
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexyl-6-nitrochroman-2-one (5a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexyl-6-nitrochroman-2-one (5a'): ^{13}C NMR ($CDCl_3$, 100 MHz)

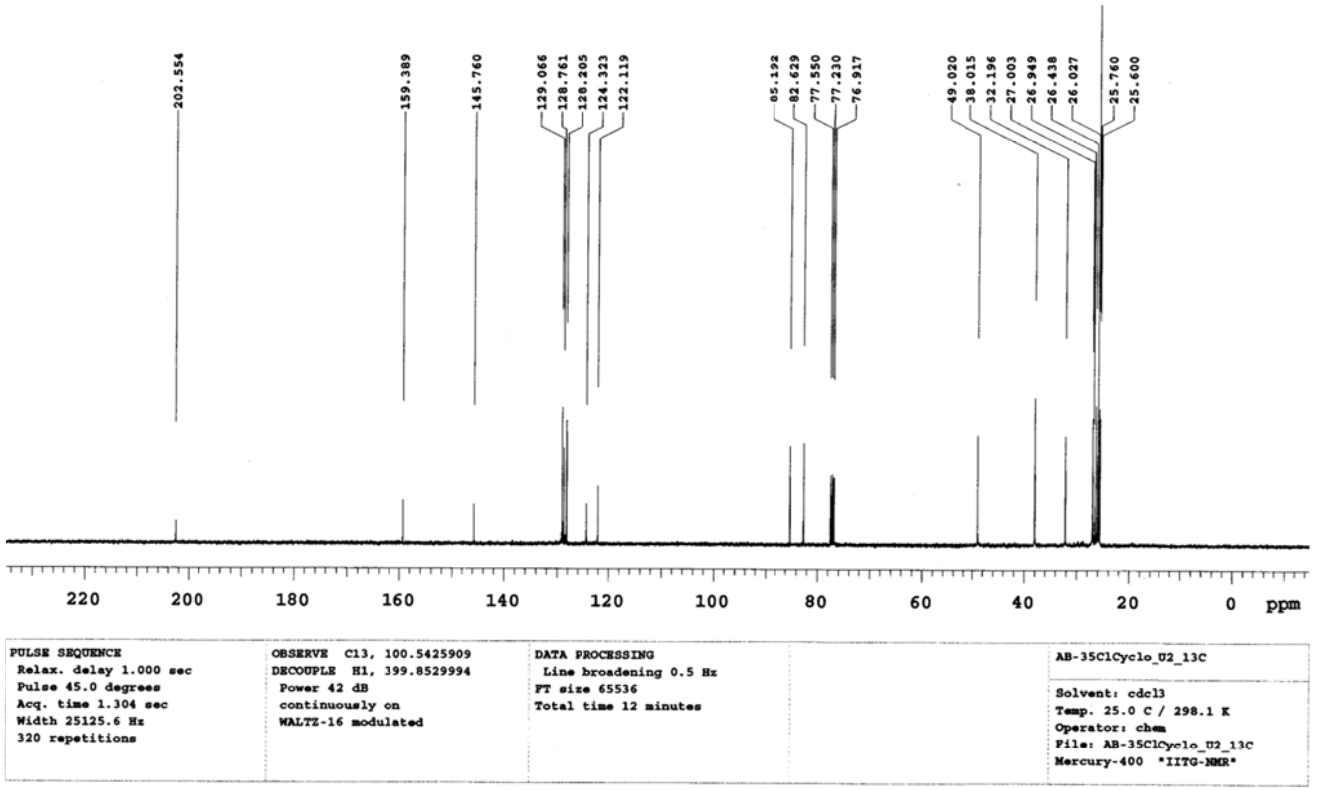


3-Acetyl-3-(*tert*-butylperoxy)-6,8-dichloro-4-cyclohexylchroman-2-one (6a'): ^{1}H NMR ($CDCl_3$, 400 MHz)

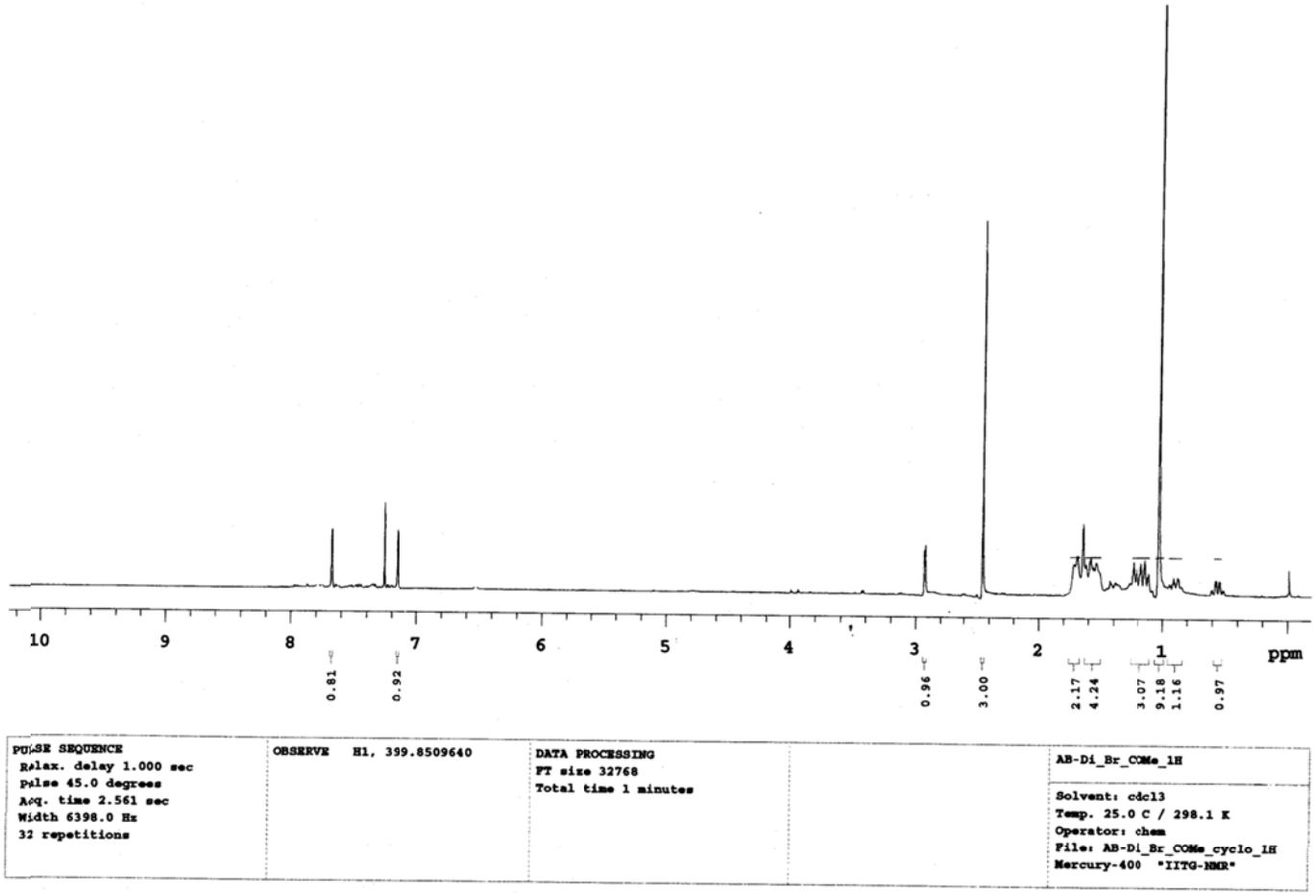


PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509613	DATA PROCESSING FT size 32768 Total time 1 minutes		AB-68Cl-1H Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem File: AB-68Cl-1H Mercury-400 *IITG-NMR*

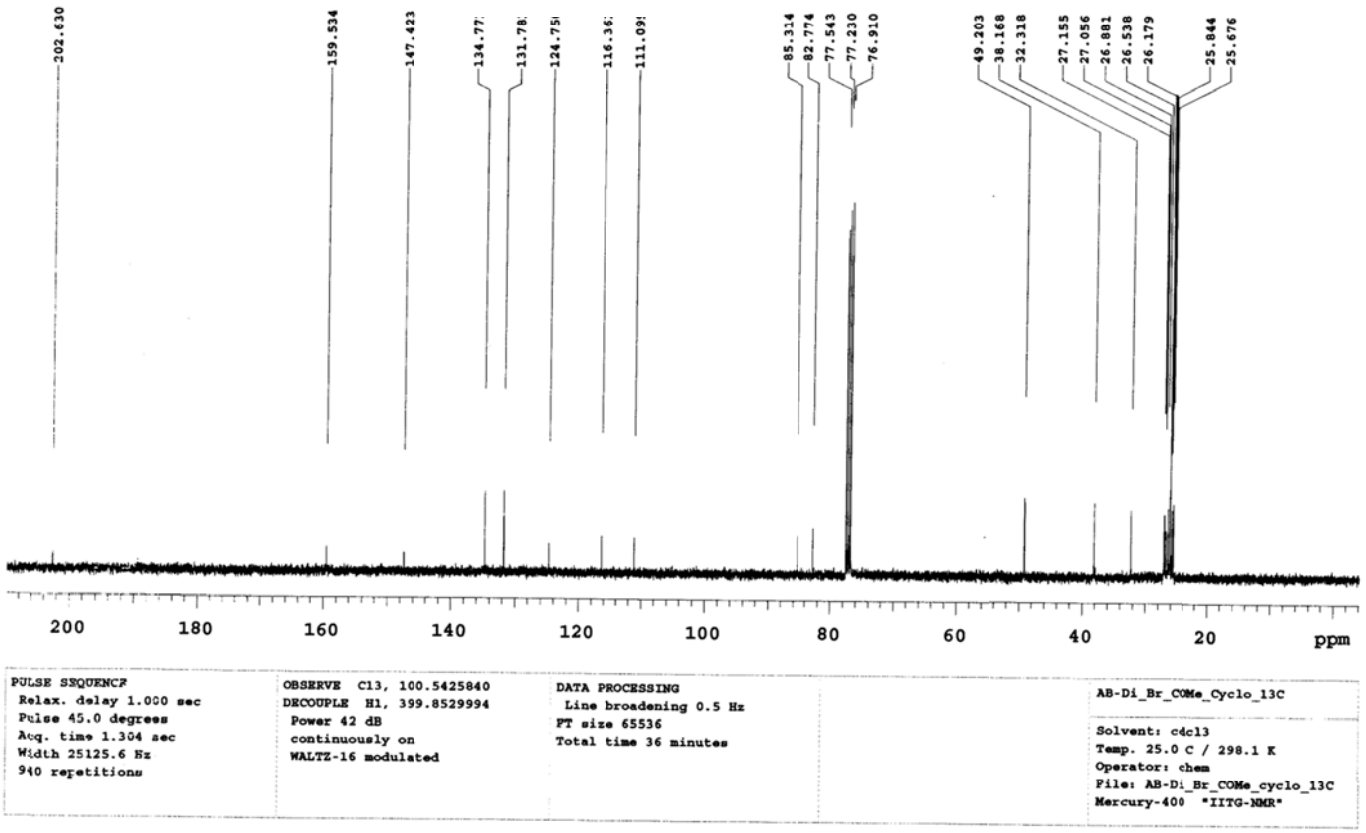
3-Acetyl-3-(*tert*-butylperoxy)-6,8-dichloro-4-cyclohexylchroman-2-one (6a'): ^{13}C NMR ($CDCl_3$, 100 MHz)



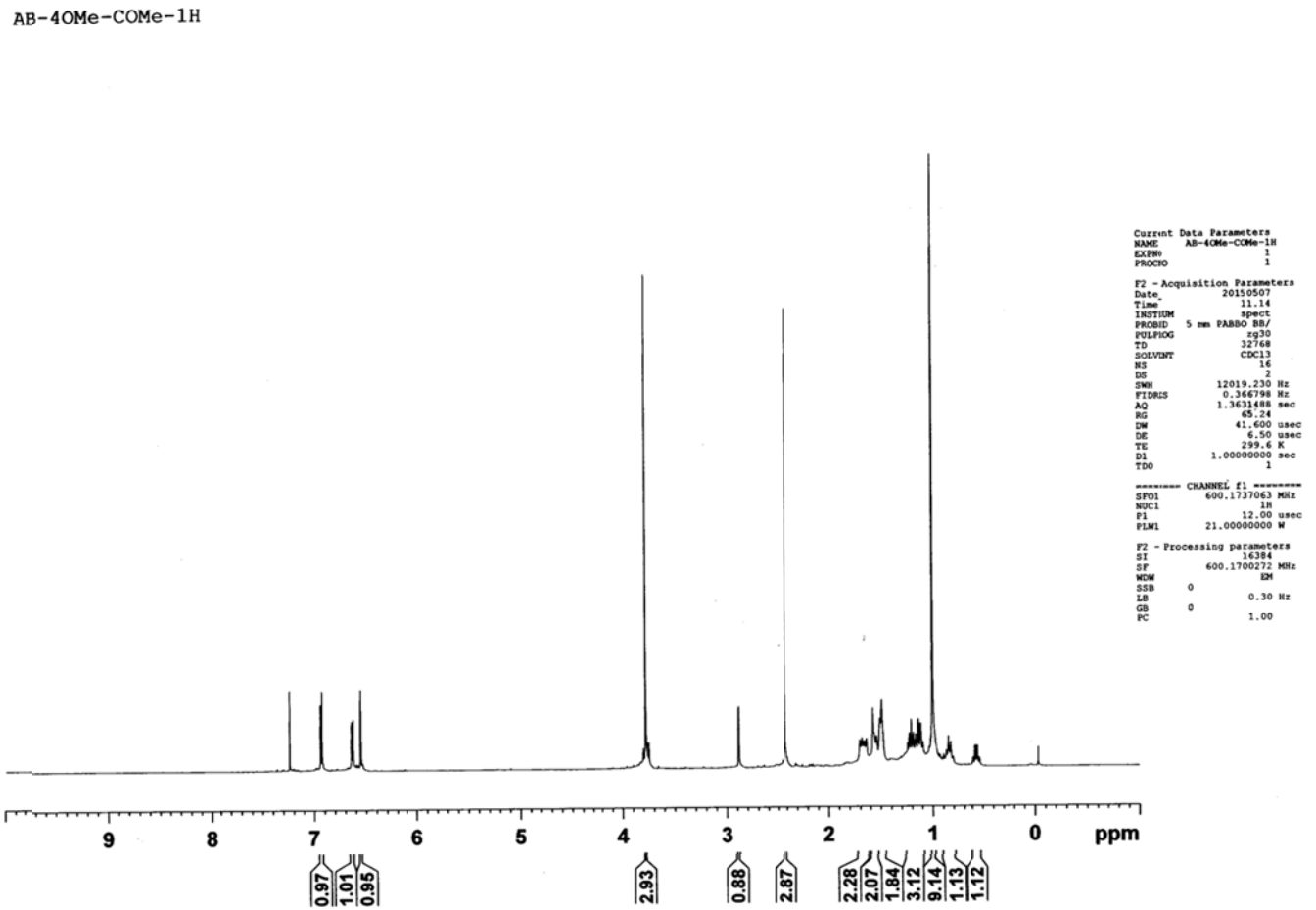
3-Acetyl-6,8-dibromo-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (7a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



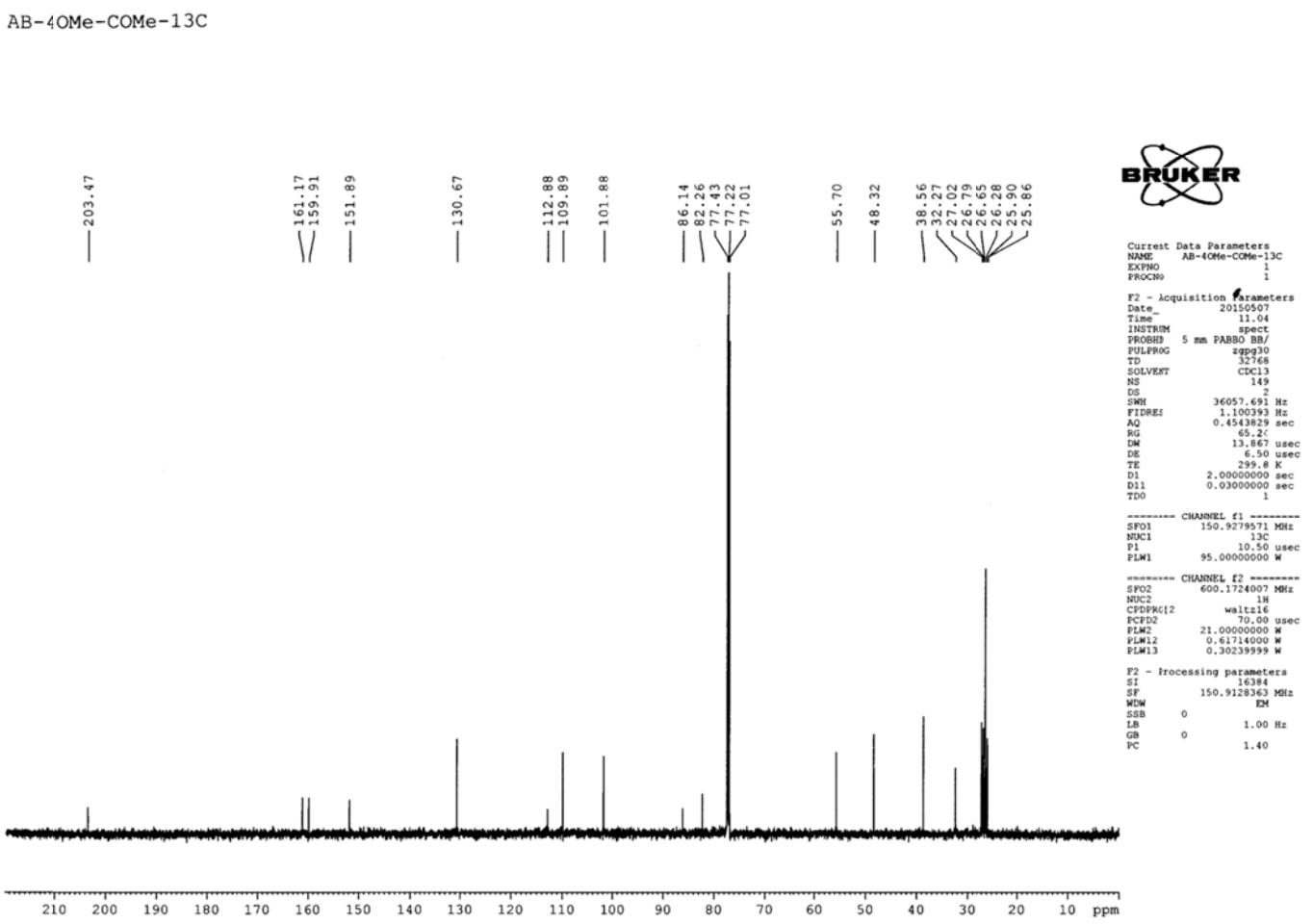
3-Acetyl-6,8-dibromo-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (7a'): ^{13}C NMR ($CDCl_3$, 100 MHz)



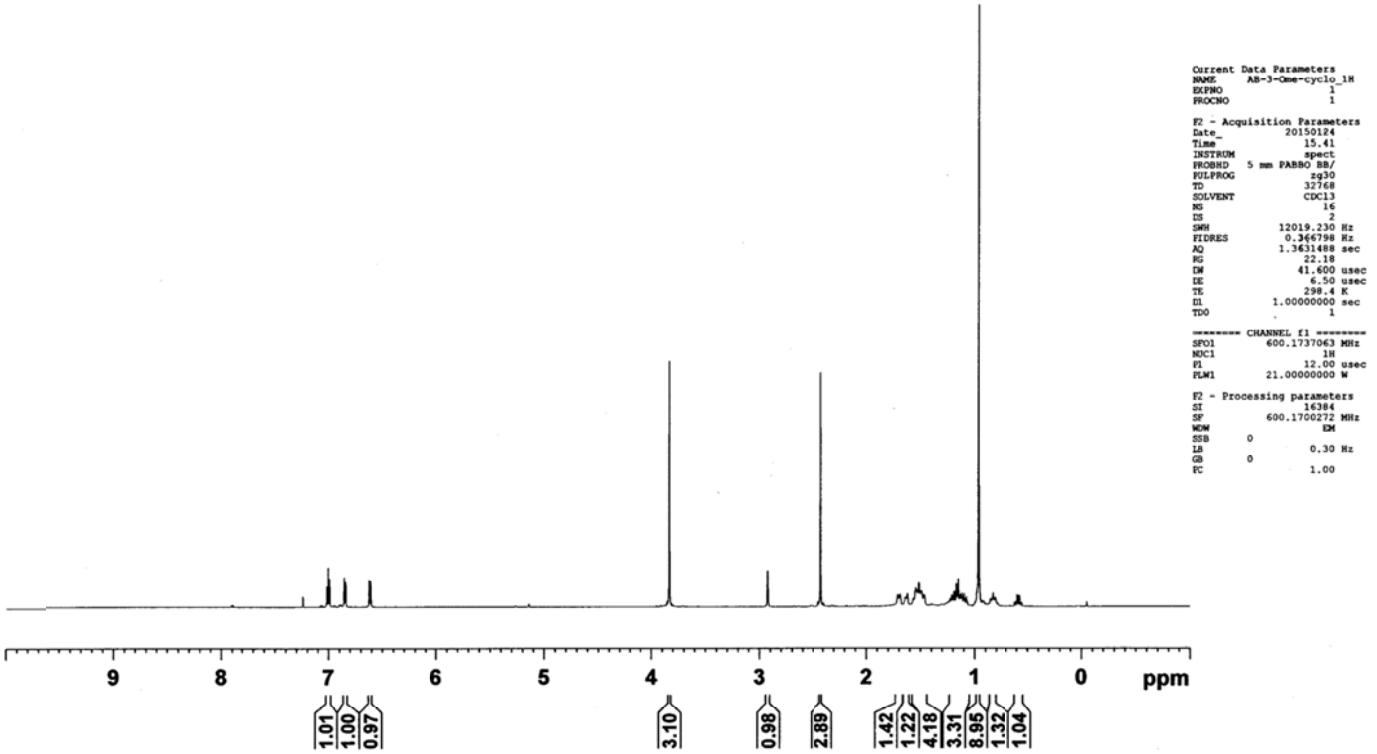
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexyl-7-methoxychroman-2-one (2a'): ^{1}H NMR ($CDCl_3$, 600 MHz)



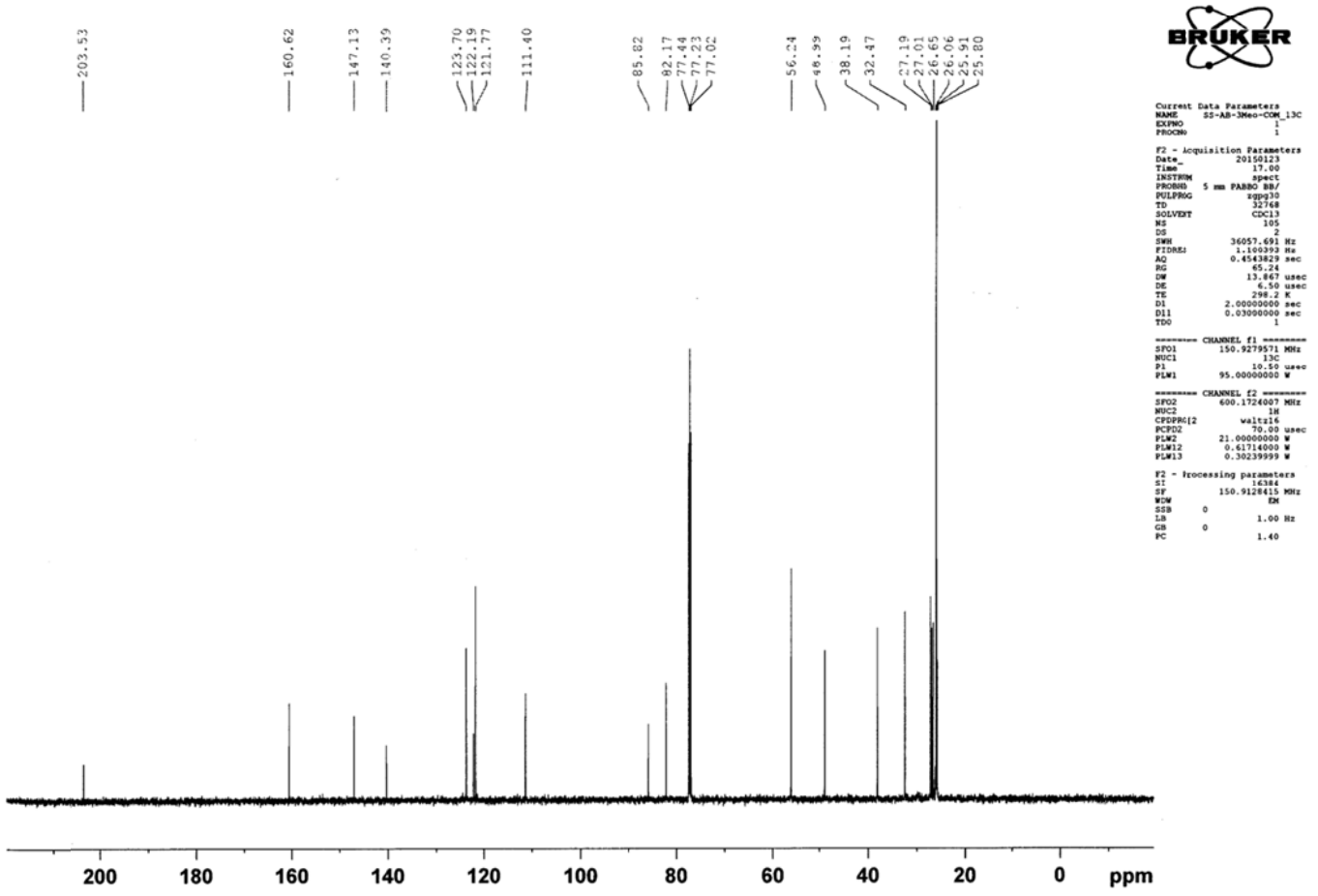
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexyl-7-methoxychroman-2-one (2a'): ^{13}C NMR ($CDCl_3$, 150 MHz)



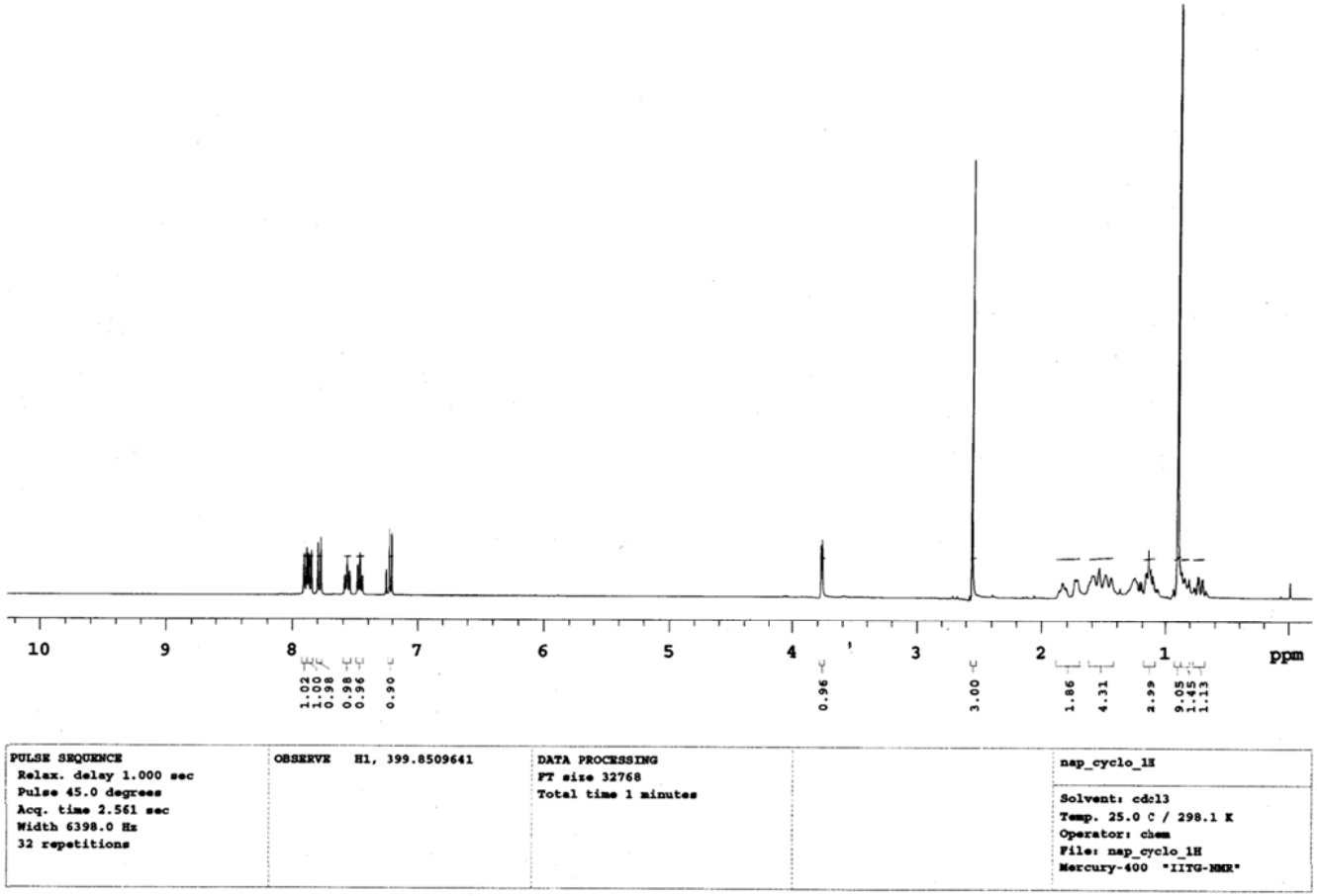
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexyl-8-methoxychroman-2-one (15a'): ^{1}H NMR ($CDCl_3$, 600 MHz)



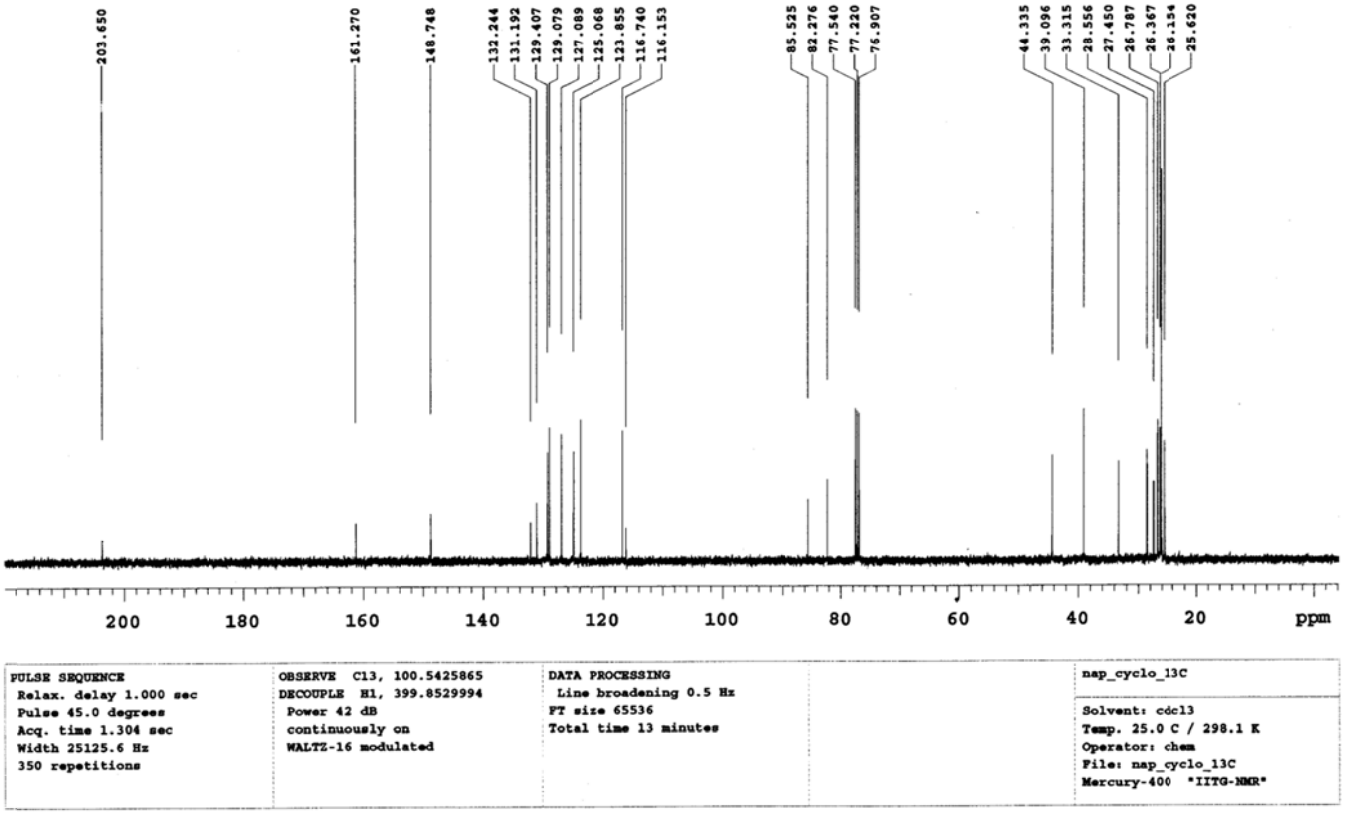
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclohexyl-8-methoxychoman-2-one (15a'): ^{13}C NMR ($CDCl_3$, 150 MHz)



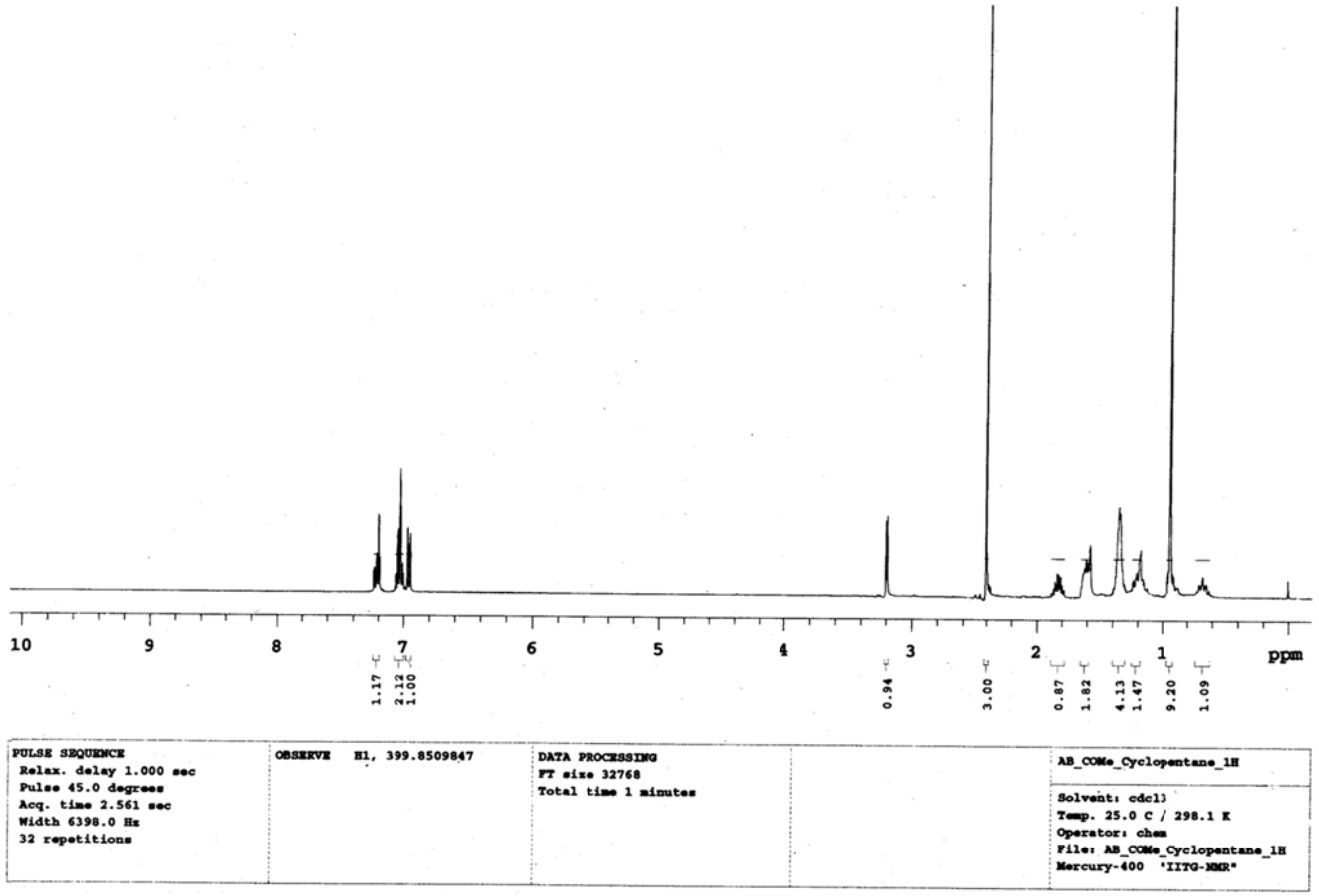
2-Acetyl-2-(*tert*-butylperoxy)-1-cyclohexyl-1H-benzo[*f*]chromen-3(2H)-one (8a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



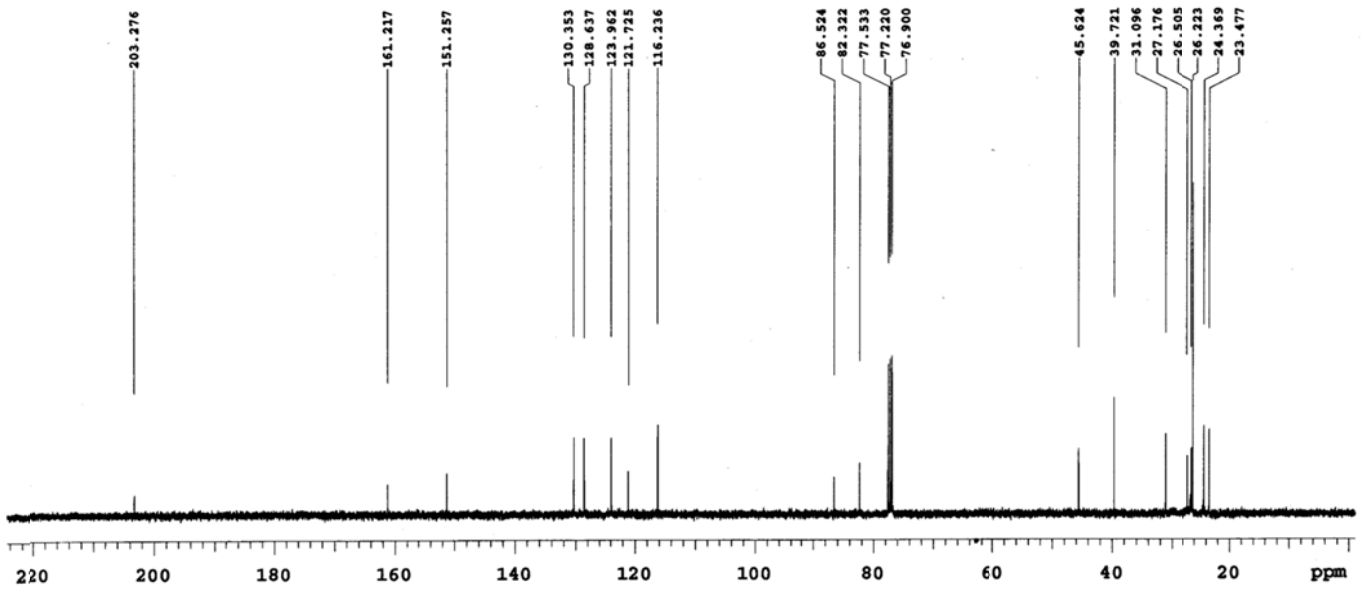
2-Acetyl-2-(*tert*-butylperoxy)-1-cyclohexyl-1H-benzo[*f*]chromen-3(2H)-one (8a'): ^{13}C NMR ($CDCl_3$, 100 MHz)



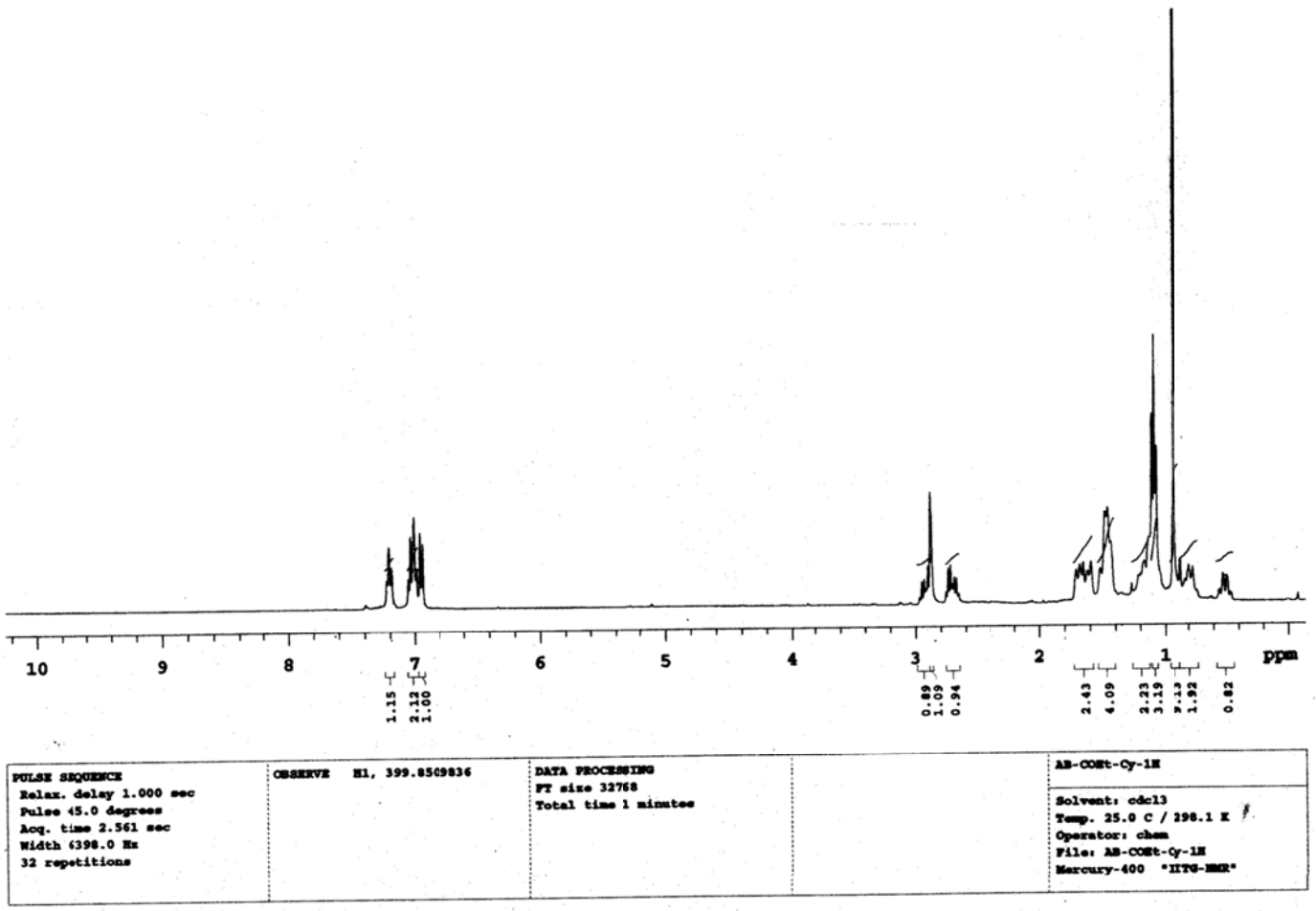
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclopentylchroman-2-one (1b'): ^{1}H NMR ($CDCl_3$, 400 MHz)



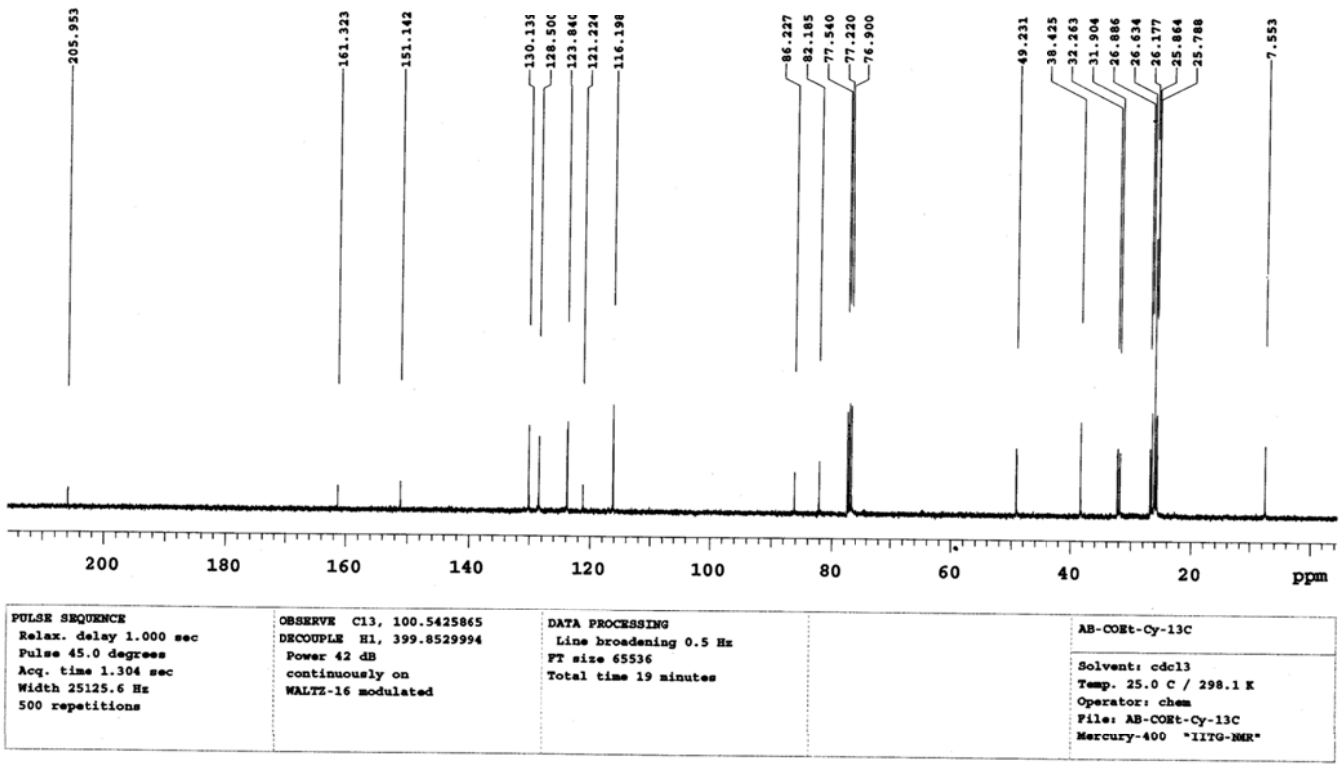
3-Acetyl-3-(*tert*-butylperoxy)-4-cyclopentylchroman-2-one (1b'): ^{13}C NMR ($CDCl_3$, 100 MHz)



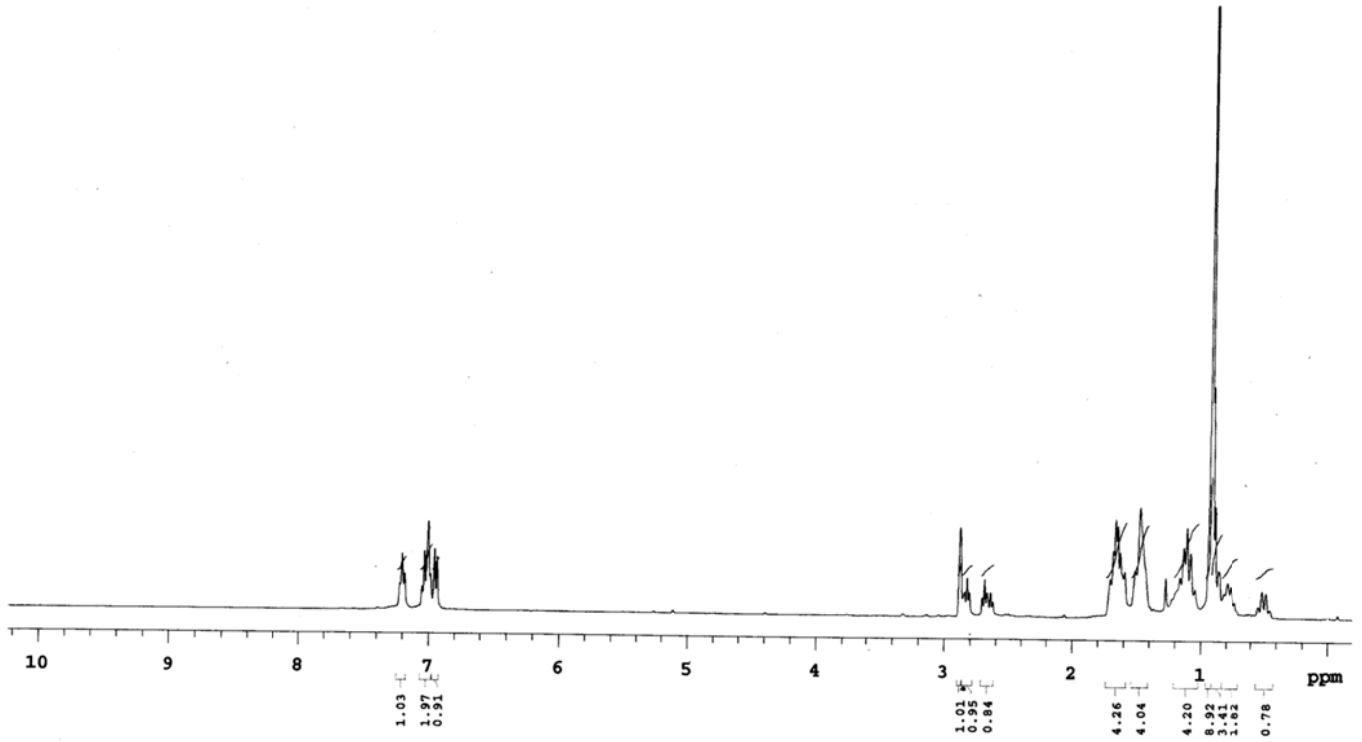
3-(*tert*-Butylperoxy)-4-cyclohexyl-3-propionylchroman-2-one (9a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



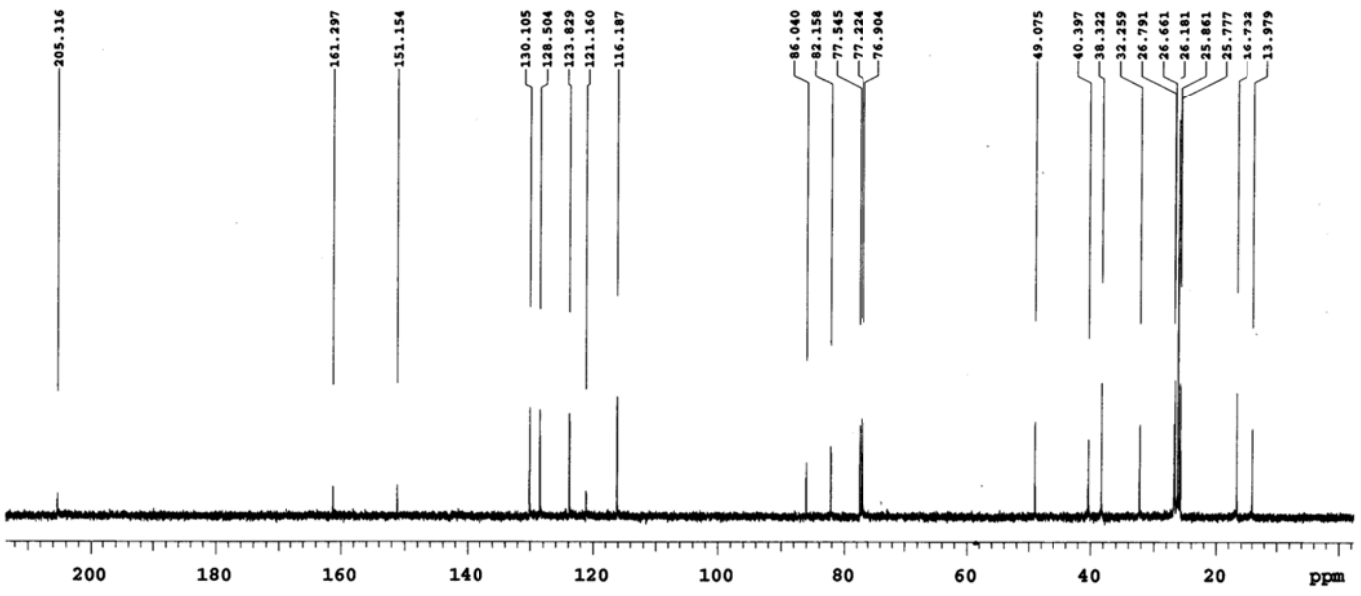
3-(*tert*-Butylperoxy)-4-cyclohexyl-3-propionylchroman-2-one (9a'): ^{13}C NMR ($CDCl_3$, 100 MHz)



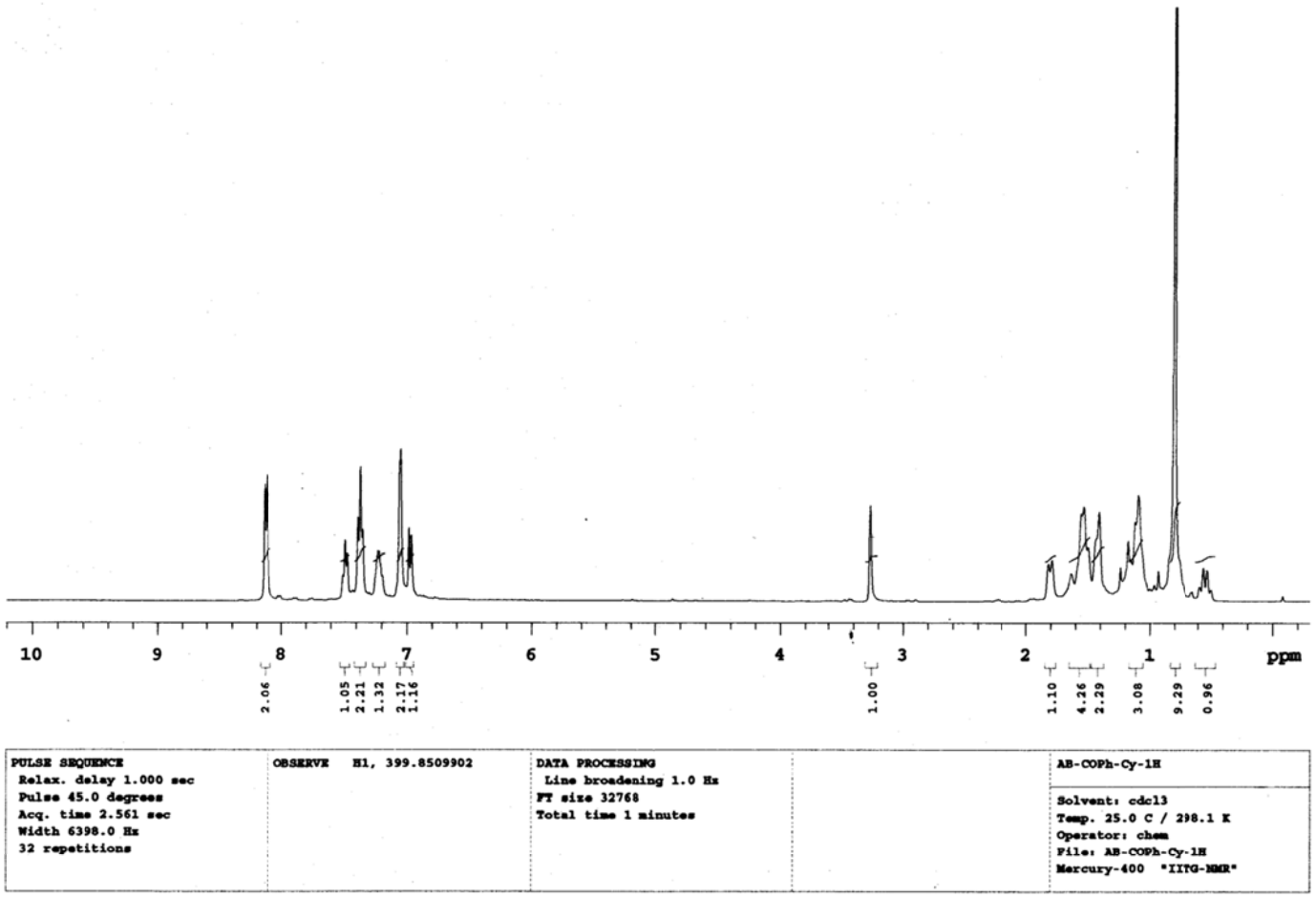
3-(*tert*-Butylperoxy)-4-cyclohexyl-3-pentanoylchroman-2-one (10a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



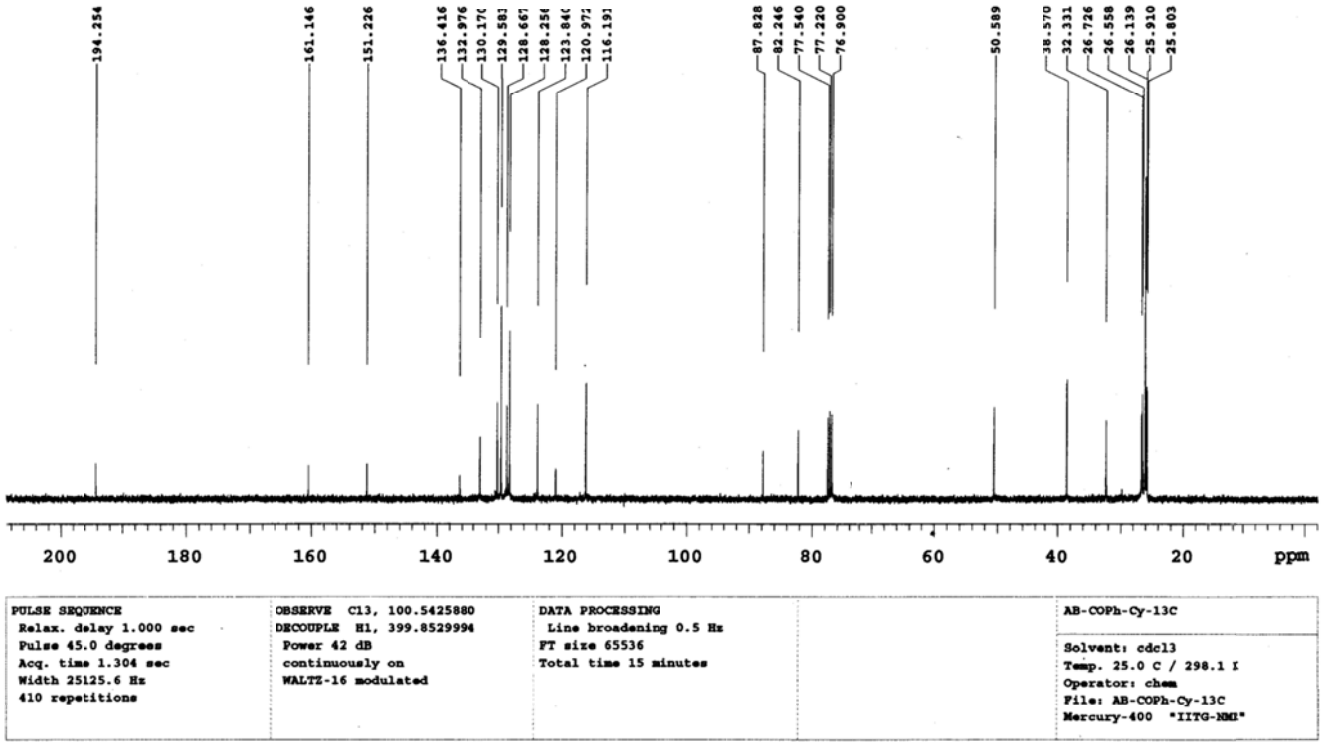
3-(*tert*-Butylperoxy)-4-cyclohexyl-3-pentanoylchroman-2-one (10a'): ^{13}C NMR ($CDCl_3$, 100 MHz)



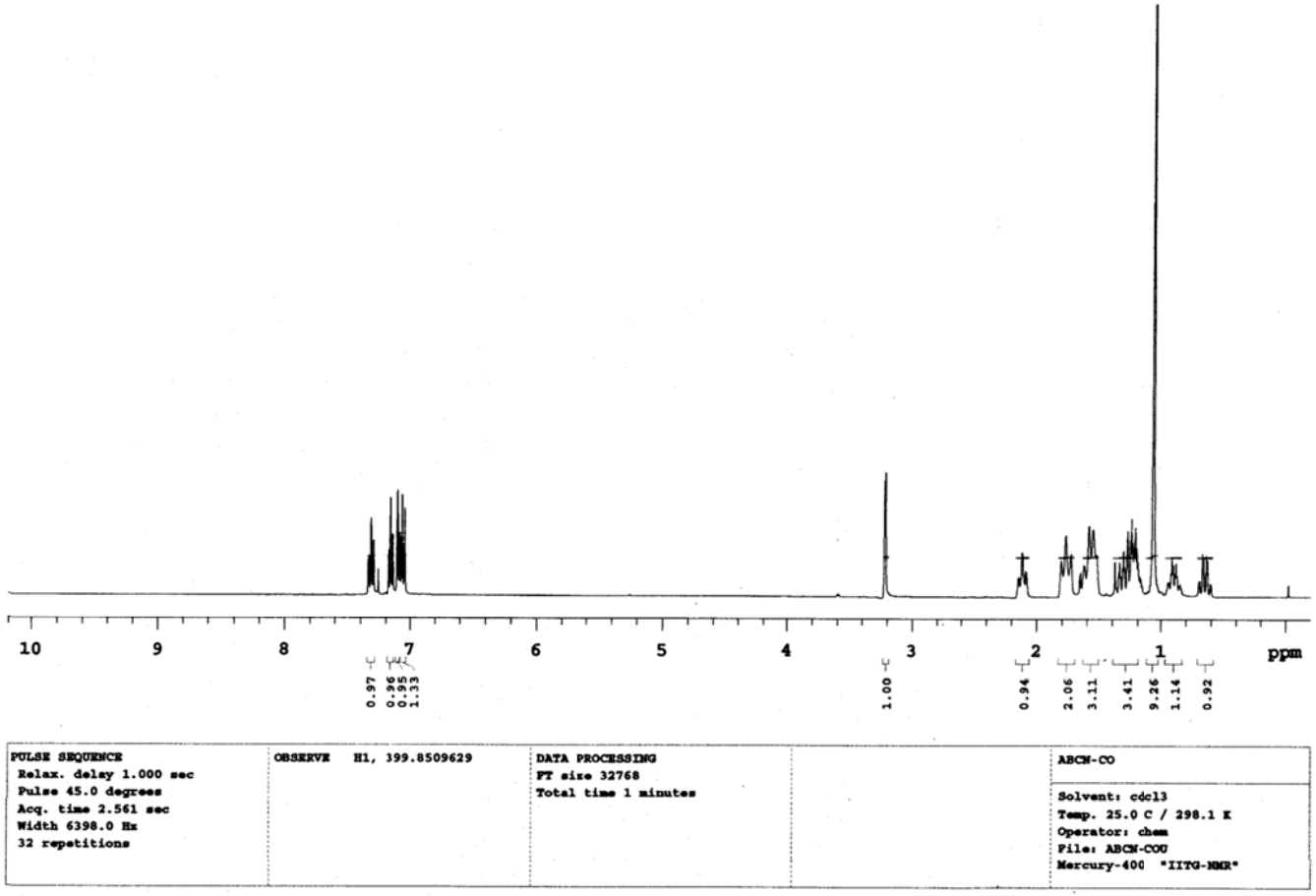
3-Benzoyl-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (11a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



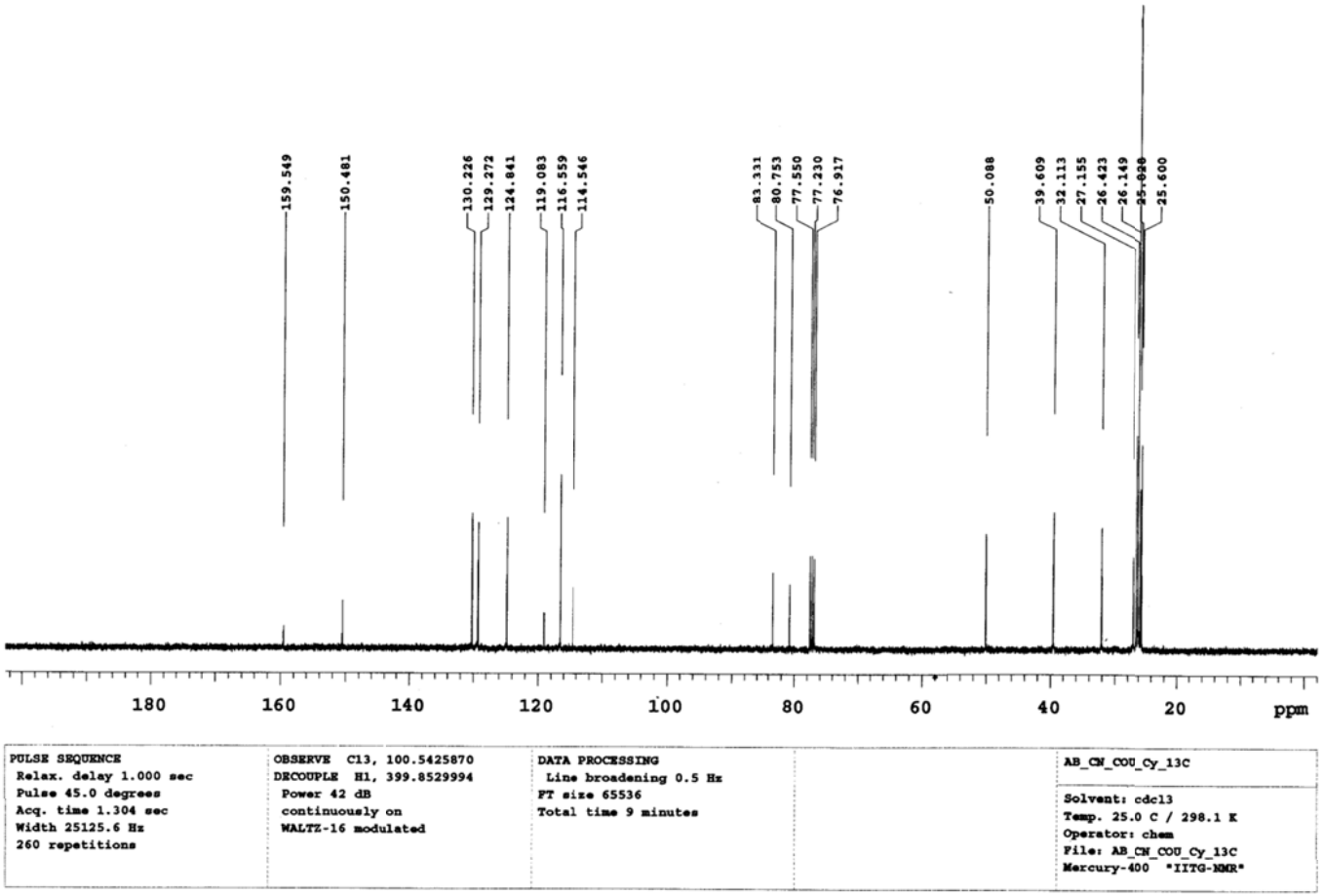
3-Benzoyl-3-(*tert*-butylperoxy)-4-cyclohexylchroman-2-one (11a'): ^{13}C NMR ($CDCl_3$, 100 MHz)



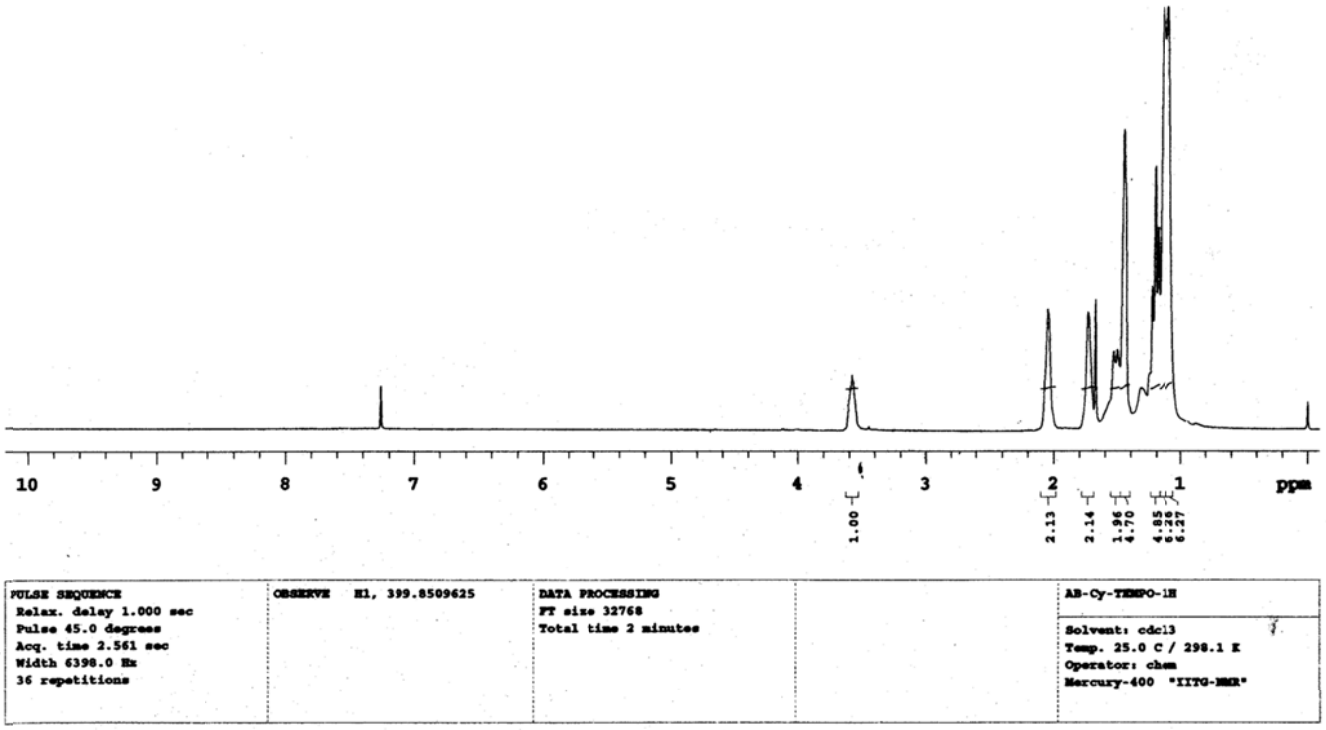
3-(*tert*-Butylperoxy)-4-cyclohexyl-2-oxochroman-3-carbonitrile (12a'): ^{1}H NMR ($CDCl_3$, 400 MHz)



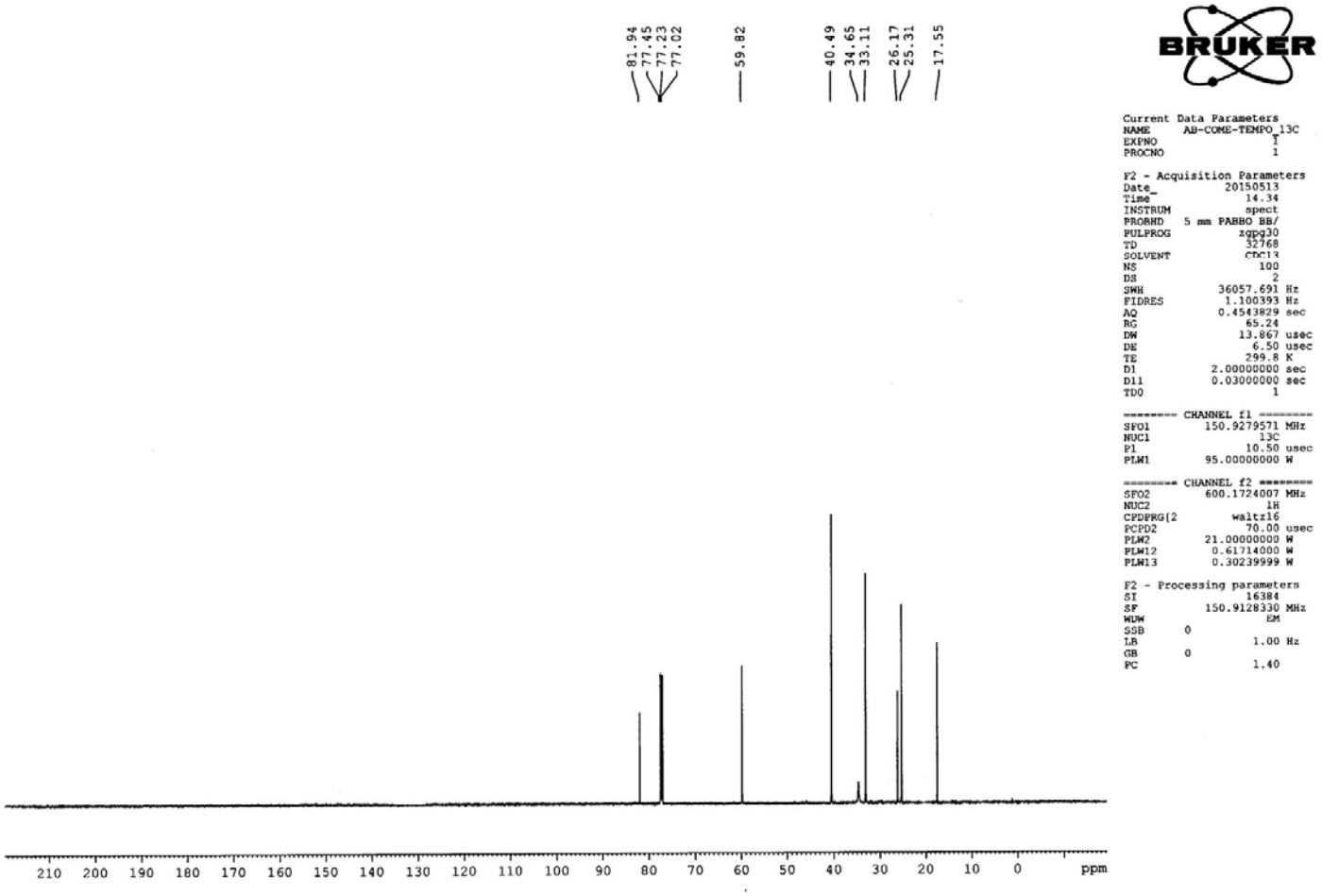
3-(*tert*-Butylperoxy)-4-cyclohexyl-2-oxochroman-3-carbonitrile (12a'): ^{13}C NMR ($CDCl_3$, 100 MHz)



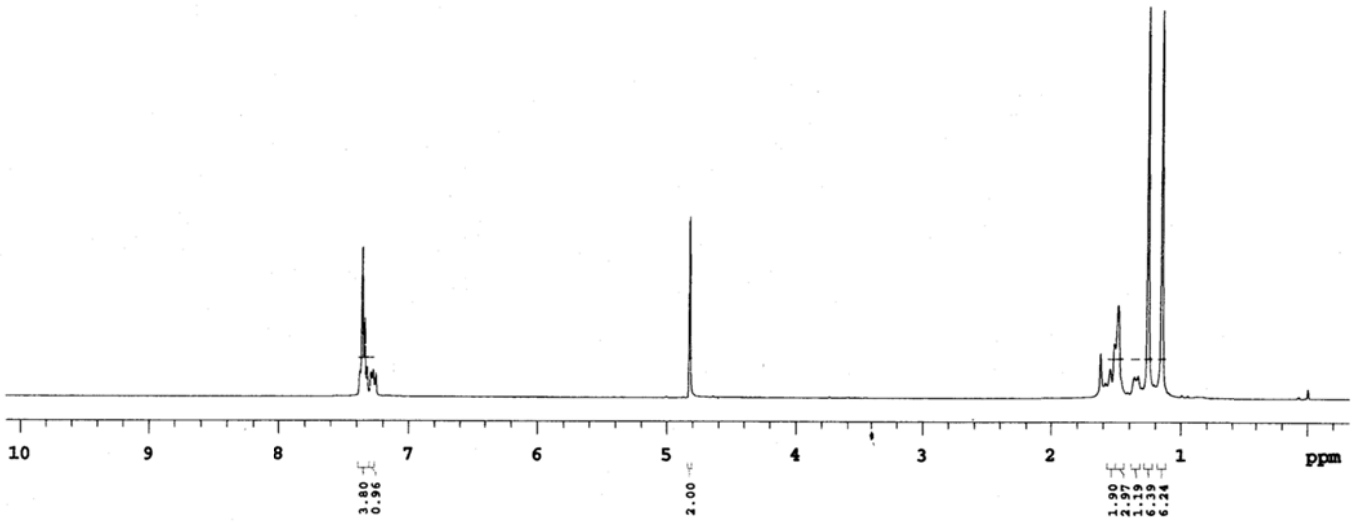
1-(Cyclohexyloxy)-2,2,6,6-tetramethylpiperidine (1A): ^{1}H NMR ($CDCl_3$, 400 MHz)



1-(Cyclohexyloxy)-2,2,6,6-tetramethylpiperidine (1A): ^{13}C NMR ($CDCl_3$, 150 MHz)

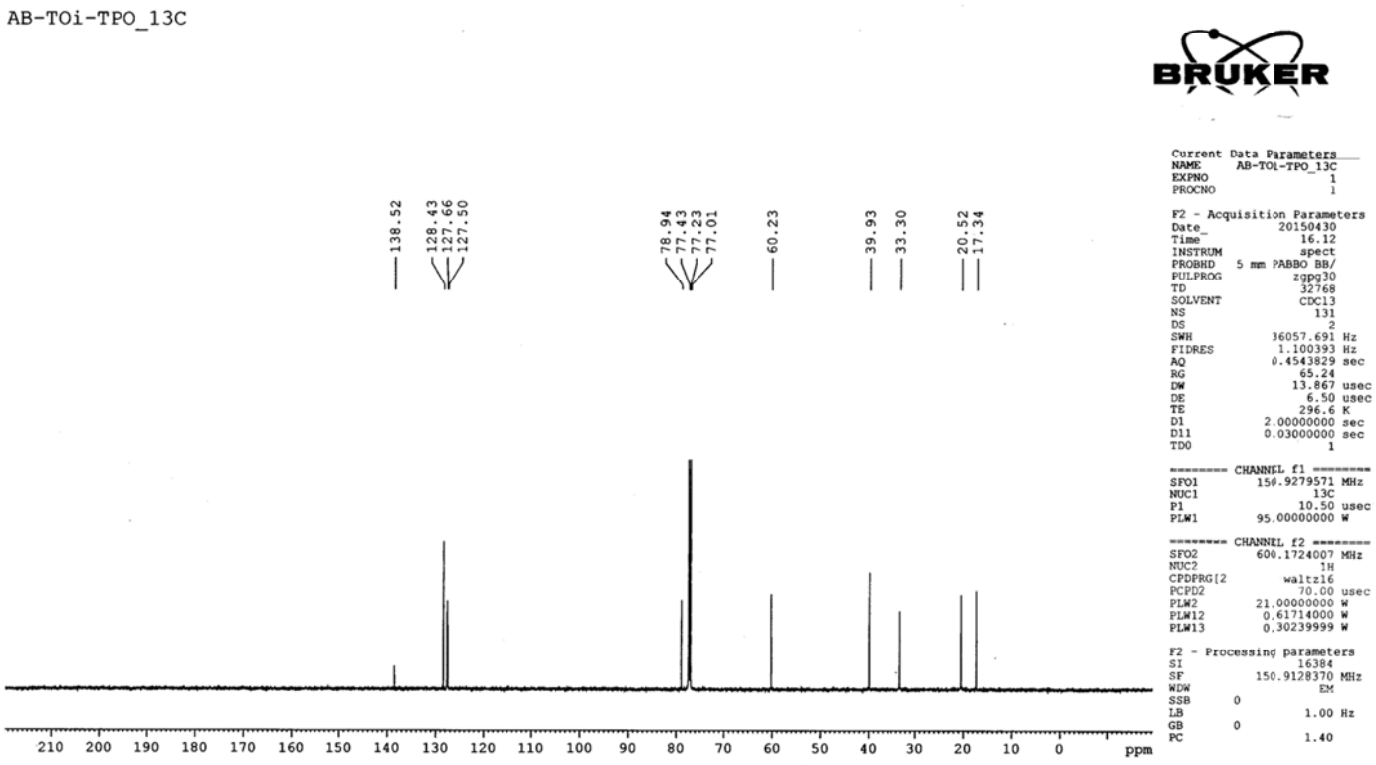


1-(Benzyloxy)-2,2,6,6-tetramethylpiperidine (1B): ^{1}H NMR ($CDCl_3$, 400 MHz)



PULSE SEQUENCE Relax. delay 1.000 sec Pulse 45.0 degrees Acq. time 2.561 sec Width 6398.0 Hz 32 repetitions	OBSERVE H1, 399.8509672	DATA PROCESSING FT size 32768 Total time 1 minutes		AB-Tol-TEMPO-1H Solvent: cdcl3 Temp. 25.0 C / 298.1 K Operator: chem Mercury-400 "IITG-NMR"

1-(Benzyloxy)-2,2,6,6-tetramethylpiperidine (1B): ^{13}C NMR ($CDCl_3$, 150 MHz)



BRUKER

Current Data Parameters
NAME AB-TOi-TPO_13C
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150430
Time 16.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 32768
SOLVENT CDCl3
NS 131
DS 2
SWH 36057.691 Hz
FIDRES 1.100393 Hz
AQ 0.4543829 sec
RG 65.24
DW 13.867 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 150.9279571 MHz
NUC1 13C
P1 10.50 usec
PLW1 95.00000000 W

======== CHANNEL f2 ========
SFO2 600.1724007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 70.00 usec
PLW2 21.00000000 W
PLW12 0.61714000 W
PLW13 0.30239999 W

F2 - Processing parameters
SI 16384
SF 150.9128370 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40