Electronic Supplementary Material (ESI) for ChemComm.

Pd-Catalyzed Stereoselective Synthesis of Chromone C-Glycosides

Manish K. Sharma[a], Bindu Tiwari[a] and Nazar Hussain*[a]

[a] M. K. Sharma, B. Tiwari, Dr. N. Hussain
Department of Medicinal Chemistry
Institute of Medical Sciences
Banaras Hindu University, Varanasi-221005
E-mail: nazar.hussain10@gmail.com, nazar10@bhu.ac.in

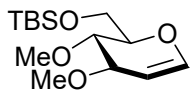
Contents

1. General Information

All reagents were used as received from commercial vendors without further purification if not stated otherwise. Thin layer chromatography (TLC) was performed on Merck Kieselgel 60 F254 aluminium plates with unmodified silica and visualized either under UV light or stained with 5% H_2SO_4 in MeOH. The products were purified by column chromatography on silica gel (100-200 mesh) using petroleum ether–ethyl acetate as the eluent to obtain the pure products. ^1H and ^{13}C NMR spectra were recorded using Avance Neo 500/600 MHz spectrometers with TMS as internal standards. Chemical shifts are expressed in parts per million (δ ppm). All product's exact masses were derived using HRMS having a QTOF analyzer. Reagents used were mostly purchased from Sigma Aldrich, TCI, and SRL

2. Starting materials used in the study[1]

Starting material of glycals[1]



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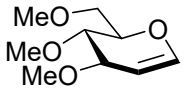

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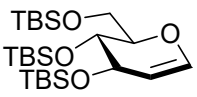

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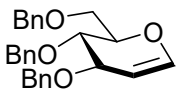

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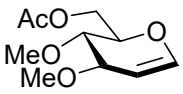

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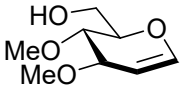

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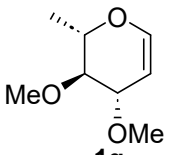

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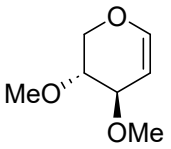

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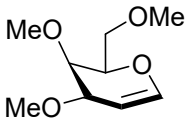

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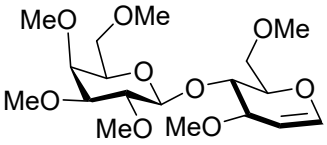

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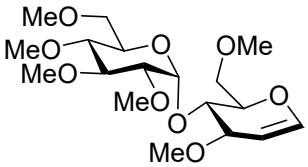

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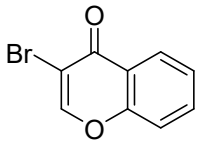
Starting material of chromones[2,3]



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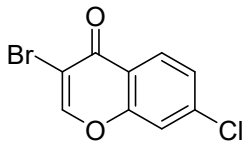

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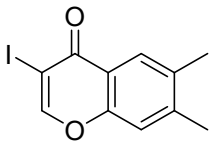

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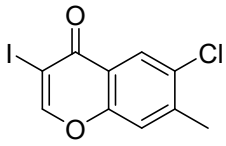

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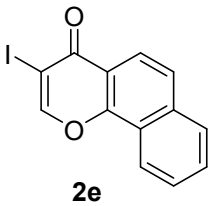

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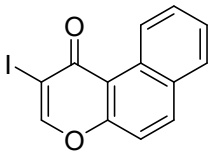

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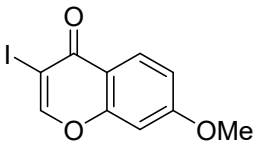

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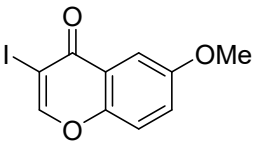

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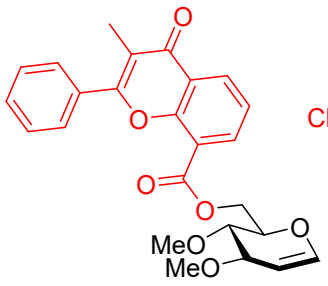
Starting material for bilogically active acids



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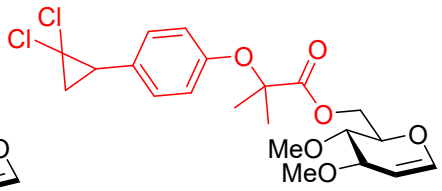

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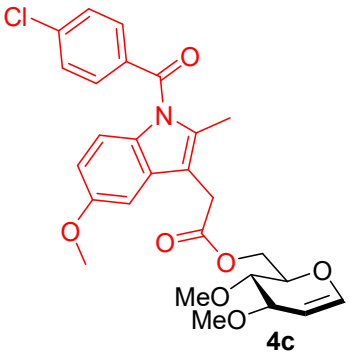

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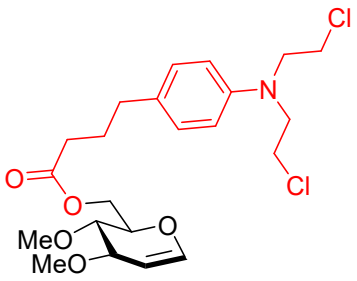

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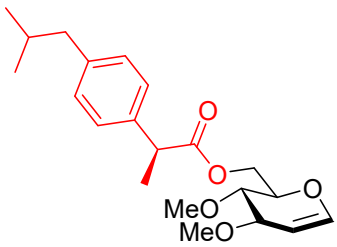

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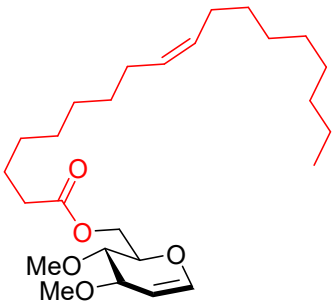
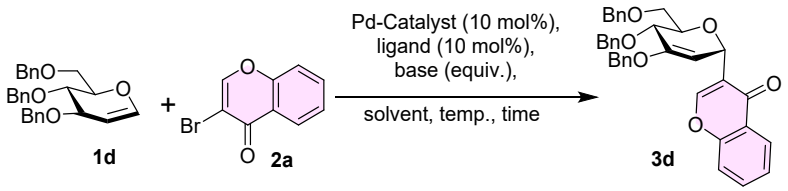


4f

3. Reaction development

3a. Random optimization

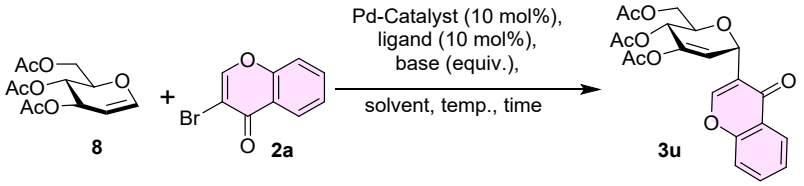


Entry	Pd-Source (10 mol%)	Ligand (10 mol%)	Base/additive (equiv.)	solvent	temperature and time	Yield (%)
1.	$Pd(OAc)_2$	PPh_3	AgOAc (2)	DMF	80 °C and 24 h	0
2.	$Pd(OAc)_2$	-	TBAB (1), K_2CO_3 (3)	DMF	100 °C and 24 h	0
3.	$Pd(OAc)_2$	-	Ag_2CO_3 (2), $Cu(OAc)_2$ (2)	AcOH	100 °C and 24 h	0
4.	$Pd(OAc)_2$	-	AgOAc	DMF:DMSO (20:1)	80 °C and 24 h	0
5.	$Pd(dba)_2$	PCy_3	K_2CO_3 (3)	Dioxane: H_2O (7:3)	50 °C and 24 h	0
6.	$Pd(TFA)_2$	PPh_3	AgOAc	DMF	80 °C and 24 h	0
7.	$Pd(dba)_2$	XantPhos	K_2CO_3 (2)	toluene	80 °C and 48 h	52
8.	$Pd(dba)_3$	XantPhos	K_2CO_3 (2)	toluene	80 °C and 48 h	40
9.	$Pd(dba)_2$	XantPhos	Cs_2CO_3 (2)	toluene	80 °C and 48 h	trace
10.	$Pd(dba)_2$	XantPhos	AgOAc (2)	toluene	80 °C and 48 h	0
11.	$Pd(dba)_2$	X-Phos	Cs_2CO_3 (2)	toluene	80 °C and 48 h	0
12.	$Pd(dba)_2$	dppf	K_2CO_3 (2)	toluene	80 °C and 48 h	13
13.	$Pd(dba)_2$	XantPhos	K_2CO_3 (2)	toluene + 100 µL water	80 °C and 48 h	37
14.	$Pd(dba)_2$	XantPhos	K_2CO_3 (2)	DCE	80 °C and 48 h	48
15.	$Pd(CH_3CN)_2Cl_2$	XantPhos	K_2CO_3 (2)	DCE	80 °C and 48 h	20
16.	$Pd(dba)_2$	XantPhos	K_2CO_3 (2)	DME	80 °C and 48 h	33
17.	$Pd(OAc)_2$	XantPhos	K_2CO_3 (2)	DME	85 °C and 48 h	38
18.	$Pd(OAc)_2$	XantPhos	K_2CO_3 (2)	DCE	85 °C and 48 h	57
19.	$Pd(OAc)_2$	XantPhos	K_2CO_3 (2)	DCM	80 °C and 48 h	43
20.	$Pd(OAc)_2$	1,10-phenonthroline	K_2CO_3 (2)	DCE	80 °C and 48 h	22
21.	$Pd(OAc)_2$	XantPhos	Cs_2CO_3 (2)	DCE	80 °C and 48 h	trace
22.	$Pd(OAc)_2$	XantPhos	K_2CO_3 (2)	toluene	90 °C and 48 h	58

23.	$Pd(OAc)_2$	XantPhos	Na_2CO_3	toluene	90 °C and 48 h	32
24.	$Pd(OAc)_2$	2,2-bipyridyl	K_2CO_3 (2)	toluene	90 °C and 48 h	trace
25.	$Pd(OAc)_2$	R-BINAP	K_2CO_3 (2)	toluene	90 °C and 48 h	43
26.	$Pd(OAc)_2$	S-BINAP	K_2CO_3 (2)	toluene	90 °C and 48 h	45
27.	$Pd(OAc)_2$	XantPhos	K_2CO_3 (2)	THF	90 °C and 48 h	48
28.	$Pd(OAc)_2$	XantPhos	K_2CO_3 (2)	Xylene	90 °C and 48 h	53
29.	$Pd(OAc)_2$	XantPhos	K_3PO_4 (2)	toluene	90 °C and 48 h	58
30.	$Pd(OAc)_2$	-	K_2CO_3 (2)	toluene	90 °C and 48 h	32

[a]Reaction conditions, unless otherwise stated: **1c** (1 equiv.), **2a** (1.2 equiv.), Pd-source (10 mol%), Ligand (10 mol%), base (2 equiv.), in solvent 1 mL (0.025M). [b]Yields are the purified products after column chromatography.

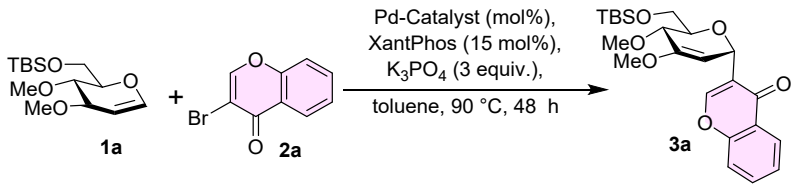
3b. Study with tri-*O*-acetyl-D-glucal



Entry	Pd-Source (10 mol%)	Ligand (mol%)	Base/additive (equiv.)	solvent	temperature and time	Yield (%)
1.	$Pd(OAc)_2$	PPh_3	AgOAc (2)	DMF	80 °C and 24 h	0
2.	$Pd(TFA)_2$	PPh_3	AgOAc (2)	DMF	80 °C and 24 h	0
3.	$Pd(dba)_2$	XantPhos	K_2CO_3 (2)	toluene	90 °C and 24 h	0
4.	$Pd(OAc)_2$	XantPhos	K_2CO_3 (2)	toluene	90 °C and 24 h	0
5.	$Ni(dppf)Cl_2$ (5)	PPh_3	Cs_2CO_3 (2)	DMF	90 °C and 24 h	0
6.	$Pd(OAc)_2$	PCy_3	K_3PO_4	toluene	90 °C and 30 h	0
7.	$Pd(OAc)_2$	XantPhos	K_3PO_4	toluene	90 °C and 30 h	0

[a]Reaction conditions, unless otherwise stated: **8** (1 equiv.), **2a** (1.2 equiv.), Pd-source (10 mol%), Ligand (10 mol%), base (2 equiv.), in solvent 1 mL, (0.035M). [b]Yields are the purified products after column chromatography.

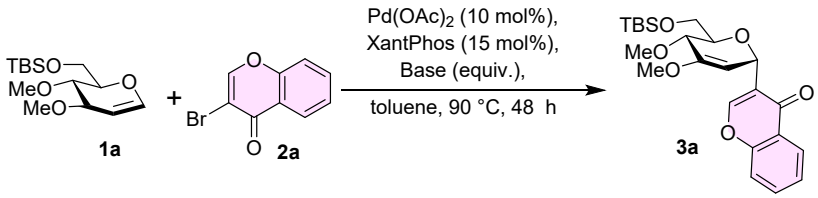
3c. Screening of Catalyst



Entry	Catalyst (mol%)	Yield (%)
1.	$Pd(dba)_2$ (10)	38
2.	$Pd(dba)_3$ (10)	51
3.	$Pd(CN)_2Cl_2$ (10)	25
4.	$Pd(OAc)_2$ (5)	47
5.	$Pd(OAc)_2$ (10)	68
6.	$Pd(OAc)_2$ (15)	65
7.	$PdCl_2$ (10)	52
8.	$Pd(TFA)_2$ (10)	56
9.	$Pd(PPh_3)_4$ (10)	43
10.	$Pd(dppf)Cl_2$ (10)	17

[a]Reaction conditions, unless otherwise stated: **1a** (1 equiv.), **2a** (1.2 equiv.), Ligand (15 mol%), base (3 equiv.), in solvent 1 mL, (0.035M), 90 °C for 48 h. [b]Yields are the purified products after column chromatography.

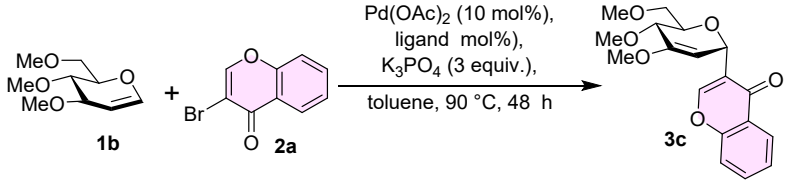
3d. Screening of bases



Entry	Base (equiv.)	Yield (%)
1.	K_2CO_3 (3)	64
2.	K_3PO_4 (2)	51
3.	K_3PO_4 (3)	68
4.	K_3PO_4 (4)	63
5.	Et_3N (3)	10
6.	NaOMe(3)	38
7.	$NaHCO_3$(3)	46
8.	KOH(3)	0
9.	DIPEA(3)	28
10.	Na_2CO_3(3)	32
11.	Cs_2CO_3(3)	20

[a]Reaction conditions, unless otherwise stated: **1a** (1 equiv.), **2a** (1.2 equiv.), Ligand (15 mol%) in solvent 1 mL, (0.035M), 90 °C for 48h. [b]Yields are the purified products after column chromatography.

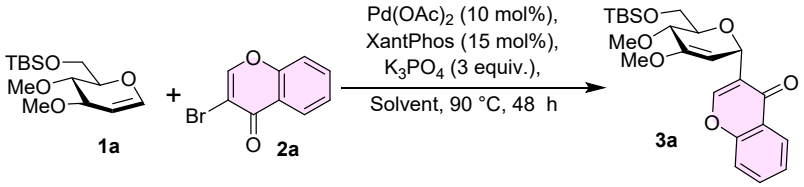
3e. Screening of ligands



Entry	ligand	Yield (%)
1.	PPh_3 (15)	47
2.	XantPhos (5)	31
3.	XantPhos (10)	52
4.	XantPhos (15)	68
5.	XantPhos (20)	64
6.	PCy_3 (15)	65
7.	R-BINAP (15)	39
8.	S-BINAP (15)	36
9.	X-Phos (15)	27
10.	Ligand free (15)	trace
11.	1,10-phenonthriline (15)	10
12.	2,2-bipydrilyl (15)	trace

[a]Reaction conditions, unless otherwise stated: **1a** (1 equiv.), **2a** (1.2 equiv.) in solvent 1 mL, (0.05M), 90 °C for 48 h. [b]Yields are the purified products after column chromatography.

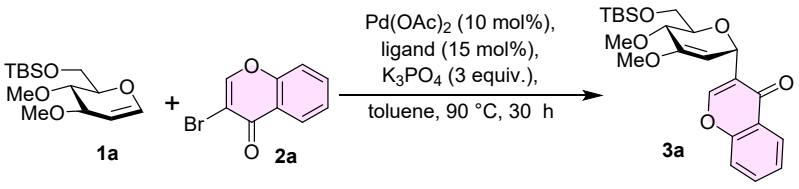
3f. Screening of Solvent



Entry	Base	Yield (%)
1.	toluene	68
2.	DCE	65
3.	Xylene	59
4.	DMF	0
5.	THF	47
6.	ACN	0
7.	DME	39
8.	DCM	43

[a]Reaction conditions, unless otherwise stated: **1a** (1 equiv.), **2a** (1.2 equiv.), Ligand (15 mol%), base (3 equiv.), in solvent 1 mL, (0.035M), 90 °C for 48 h. [b]Yields are the purified products after column chromatography.

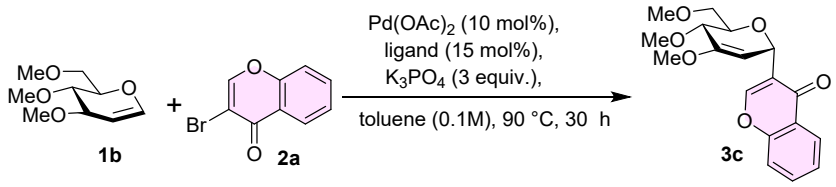
Screening of solvent concentration*



Entry	Concentration (M)	Yield (%)
1.	toluene (0.2)	53
2.	toluene (0.15)	73
3.	toluene (0.1)	78
4.	toluene (0.08)	75
5.	toluene (0.05)	71
6.	toluene (0.03)	65
7.	toluene (0.01)	48

*All the reactions were quenched after 30 h

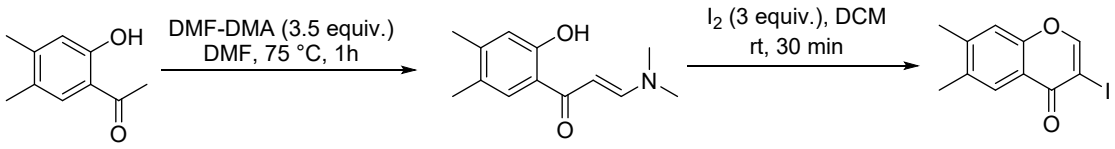
3g. Screening of reaction environment



Entry	Base	Yield (%)
1.	Seal Tube	69
2.	Open air	73
3.	Inert atmosphere	62
4.	Installed in open air and capped with glass stopper	78

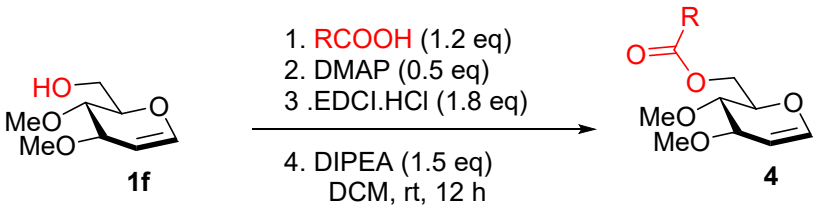
4. General procedures

4a. Preparation of 3-iodochromones[2,3]



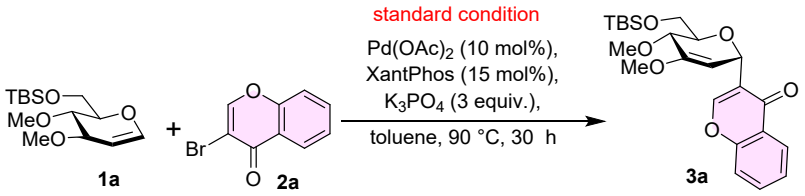
A solution of 3,4-dimethyl-*o*-acetophenols (3.05 mmol, 500 mg) and N, N-dimethylformamide dimethyl acetal (DMF-DMA) (10.7 mmol, 3.5 equiv.) in N, N -dimethylformamide (DMF, 10 mL) was stirred at 75 ºC for 45 min. After the mixture was cooled to room temperature, saturated brine (100 mL) was added and an orange precipitate appeared, which was filtered and dried to afford the corresponding 3-(dimethylamino)-1-(2-hydroxyphenyl)prop-2-enones. Then iodine (9.2 mmol, 3.0 equiv.) and 3-(dimethylamino)-1-(2-hydroxyphenyl)prop-2-enones were dissolved in 15 mL of DCM and the mixture was stirred at room temperature for 30 min. All reactions were monitored by TLC (thin layer chromatography). The solution of 5% $NaHSO_3$ was poured into the reaction mixture to quench I_2 and the mixture was stirred until the solution turned yellow. The organic phase was washed neutrally with 5% aq. $NaHCO_3$ (3 × 25 mL), dried over anhydrous Na_2SO_4, filtered and concentrated by rotary evaporation, and recrystallized from anhydrous EtOH to give the corresponding 3-iodochromones **2c-h**.

4b. Preparation of starting material of biologically active molecules



In an oven-dried round bottom flask, dimethyl protected glucal **1f** (0.29 mmol, 1 equiv.), were dissolved in DCM at room temperature, and acid (0.35 mmol,1.2 equiv.) was added. DMAP (0.14 mmol, 50 mol%) and EDCI.HCl (0.52 mmol,1.8 equiv.) and DIPEA (0.43 mmol, 1.5 equiv.) were added sequentially and the reaction mixture was stirred at room temperature for 12 h. After the starting material was converted as confirmed through TLC, the reaction mixture was quenched with 1N HCl (10 mL), and the organic layer was extracted with DCM (10 × 2 ml). The organic layer was dried over sodium sulfate and the residue left was purified by column chromatography over silica gel (100-200 mesh) using hexane/ethyl acetate as eluent.

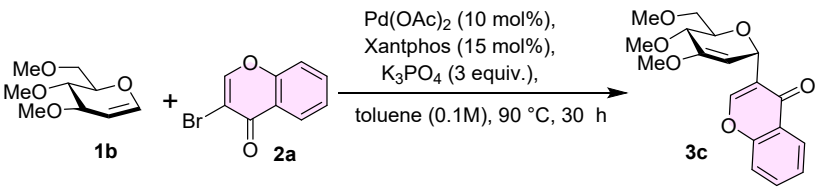
4c. Preparation of chromones *C*-glycosides



In an oven-dried round bottom flask glycal **1a** (0.10 mmol, 1 equiv.), $Pd(OAc)_2$ (0.01 mmol, 0.1 equiv,) Xantphos (0.015 mmol, 0.15 equiv.), K_3PO_4 (0.31 mmol, 3 equiv.) and 3-bromo chromone (0.13 mmol, 1.2 equiv.) were added successively and dissolved in 1.04 mL, (0.1M) of toluene at room temperature. Finally, the round bottom flask was capped with the glass stopper and reaction mixture was stirred at preheated silicon oil bath for 30 h at 90 °C. After the starting material was converted as confirmed through TLC, the mixture was cooled at room temperature, diluted with ethyl acetate (10 mL×2), and passed through the celite. The reaction mixture was washed with brine solution (10 mL) and the organic layer was dried over sodium sulfate and the residue left was purified by column chromatography over silica gel (100-200 mesh) using hexane/ethyl acetate as eluent.

4d. Time profile of $Pd(OAc)_2$ catalyzed condition

In an oven-dried round bottom flask glycal **1b** (47 mg, 0.25 mmol, 1 equiv.), $Pd(OAc)_2$ (0.025 mmol, 0.1 equiv,) Xantphos (0.0375 mmol, 0.15 equiv.), K_3PO_4 (0.75 mmol, 3 equiv.) and 3-bromo chromone (0.3 mmol, 1.2 equiv.) were added successively and dissolved in 2.5 mL, (0.1M) of toluene at room temperature. Finally, the round bottom flask was capped with the glass stopper and reaction mixture was stirred at preheated silicon oil bath at 90 °C. The conversion of **1b** into **3c** was monitored by ^{1}H NMR spectroscopy. A 400 µL aliquot was taken from the reaction solution . The aliquots were concentrated under reduced pressure, dissolved in $CDCl_3$ and internal standard (dibromomethane 0.50 mmol, 2 equiv.), and analyzed by ^{1}H NMR spectroscopy.



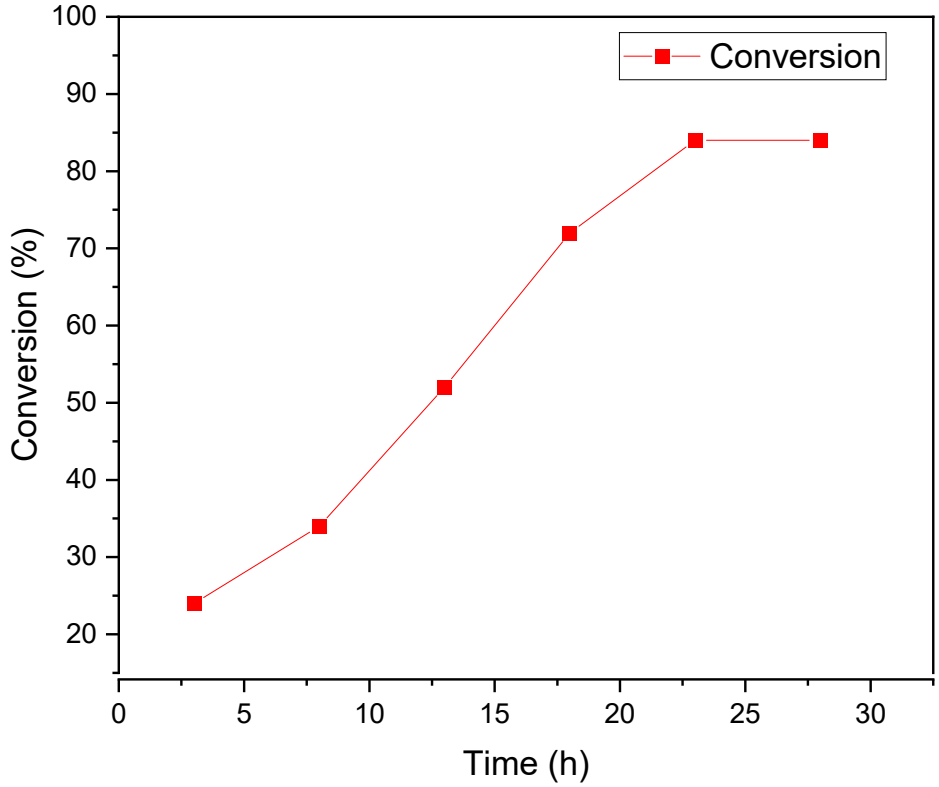


Fig. S1 Time profile of $Pd(OAc)_2$ catalyzed reaction

5. 2D-NMR analysis

5a. HSQC analysis of compound 3a

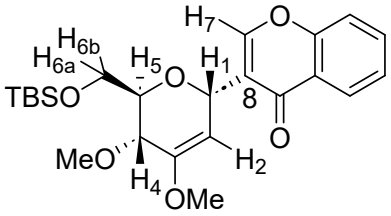


Fig. S2 HSQC analysis of compound **3a**

Table S1 HSQC analysis

	^{1}H	^{13}C
H-1/C-1	5.50 (s, 1H)	65.9
H-2/C-2	5.03 (s, 1H)	96.4
C-3		151.1
H-4/C-4	3.65 (s, 1H)	72.3
H-5/C-5	4.04 (d, J = 5.8 Hz, 1H)	74.7
H-$6_{a,b}$/C-6	3.78 – 3.72 (m, 2H),	61.4
H-7/C-7	8.07	153.4
C-8		125.0

5b. COSY Correlations of 3a

Table S2 COSY analysis

	^{1}H	**^{1}H**
H-1	5.50 (s, 1H)	5.03 (s, 1H, H-2)
H-2	5.03 (s, 1H)	5.50 (s, 1H, H-1)
H-4	3.65 (s, 1H)	4.04 (d, J = 5.8 Hz, 1H, H-5)
H-5	4.04 (d, J = 5.8 Hz, 1H)	3.65 (s, 1H, H-4) and 3.78 – 3.72 (m, 2H, H-6)
H-$6_{a,b}$	3.78 – 3.72 (m, 2H),	4.04 (d, J = 5.8 Hz, 1H, H-5)

5c. NOESY correlation analysis of compound 3a

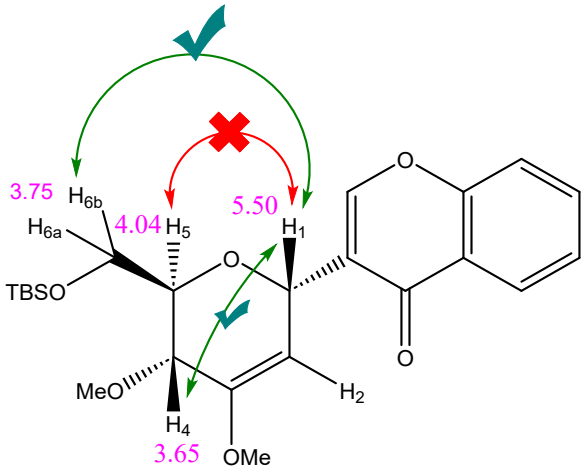
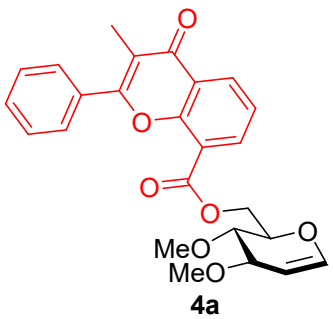


Fig. S3 NOESY correlation analysis

6. Characterization data of starting materials and products



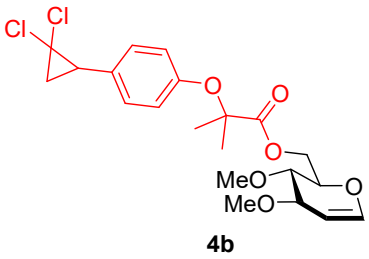
4a

The compound **4a** was synthesized according to the general procedure **4b** and isolated through column chromatography on silica gel. R_f = 0.45 (30%, n-hexane/ ethyl acetate), eluent 18% (n-hexane/ethyl acetate), pale yellow solid (104.0 mg, 83%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.40 (d, J = 7.9 Hz, 1H), 8.21 (d, J = 7.5 Hz, 1H), 7.74 (d, J = 7.1 Hz, 2H), 7.45 (t, J = 6.8 Hz, 3H), 7.38 (t, J = 7.6 Hz, 1H), 6.26 (d, J = 6.1 Hz, 1H), 4.87 – 4.80 (m, 1H), 4.55 (dt, J = 12.0, 9.4 Hz, 2H)), 4.12 (t, J = 7.0 Hz, 1H), 3.80 (s, 1H), 3.43 (s, 3H), 3.37 (t, J = 6.6 Hz, 1H), 3.33 (s, 3H), 2.17 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 178.3, 164.1, 161.1, 154.5, 144.2, 136.2, 133.0, 131.0, 130.5, 129.4 (*2C*), 128.5 (*2C*), 124.0, 123.3, 120.3, 117.7, 99.5, 76.1, 75.9, 74.6, 63.9, 59.2, 55.9, 11.8.

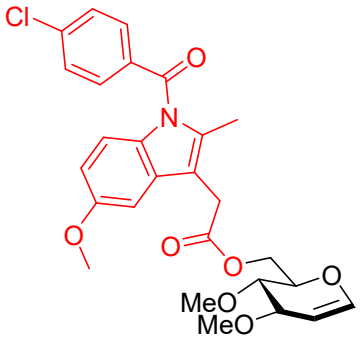
HRMS (ESI) calcd for $[C_{25}H_{24}O_7+H]^+$ 437.1595, found 437.1599.



4b

The compound **4b** was synthesized according to the general procedure **4b** and isolated through column chromatography on silica gel. R_f = 0.57 (20%, n-hexane/ ethyl acetate), eluent 10% (n-hexane/ethyl acetate), pale yellow gummy (111.0 mg, 87%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.03 (d, *J* = 7.8 Hz, 2H), 6.77 (d, *J* = 7.6 Hz, 2H), 6.24 (d, *J* = 6.0 Hz, 1H), 4.80 – 4.74 (m, 1H), 4.43 (d, *J* = 11.9 Hz, 1H), 4.33 (dd, *J* = 11.9, 5.5 Hz, 1H), 3.95 (t, *J* = 6.6 Hz, 1H), 3.77 (s, 1H), 3.36 (s, 3H), 3.30 (s, 3H), 3.28 (s, 1H, *bs*), 2.75 (t, *J* = 9.4 Hz, 1H), 1.89 – 1.83 (m, 1H), 1.69 (t, *J* = 7.8 Hz, 1H), 1.54 (s, 6H).



4c

^{13}C NMR (151 MHz, $CDCl_3$) δ 174.0, 154.9, 144.2, 129.6 (*2C*), 118.9 (*2C*), 99.6, 79.2, 76.2, 75.7, 74.6, 63.2, 60.9, 59.2, 55.8, 34.8, 25.9, 25.6, 25.3.

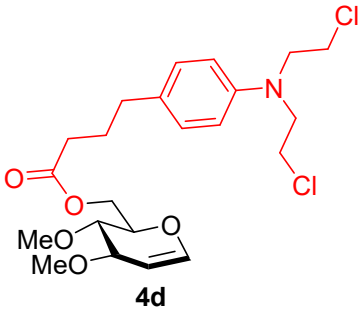
HRMS (ESI) calculated for $[C_{21}H_{26}Cl_2O_6+Na]^+$ 467.0999, found 467.1015

The compound **4c** was synthesized according to the general procedure **4b** and isolated through column chromatography on silica gel. R_f = 0.63 (30%, n-hexane/ ethyl acetate), eluent 12% (n-hexane/ethyl acetate), yellow gummy (129.7 mg, 88%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.59 (d, *J* = 7.3 Hz, 2H), 7.39 (d, *J* = 7.7 Hz, 2H), 6.90 (s, 1H), 6.79 (d, *J* = 8.9 Hz, 1H), 6.59 (d, *J* = 8.9 Hz, 1H), 6.24 (d, *J* = 6.0 Hz, 1H), 4.78 – 4.76 (m, 1H), 4.34 (d, *J* = 12.1 Hz, 1H), 4.27 (dd, *J* = 12.0, 5.5 Hz, 1H), 3.93 (t, *J* = 6.7 Hz, 1H), 3.76 (s, 4H), 3.64 (s, 2H), 3.30 (s, 3H), 3.27 (s, 3H), 3.20 (t, *J* = 6.9 Hz, 1H), 2.31 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 169.5, 167.3, 155.1, 143.1, 138.2, 134.9, 132.9, 130.2, 129.8, 129.6, 128.1, 128.1, 113.9, 111.5, 110.8, 100.2, 98.7, 75.2, 74.7, 73.7, 62.0, 58.1, 54.68, 54.7, 29.3, 12.3.

HRMS (ESI) calcd for $[C_{27}H_{28}ClNO_7+Na]^+$ 536.1447, found 536.1459



4d

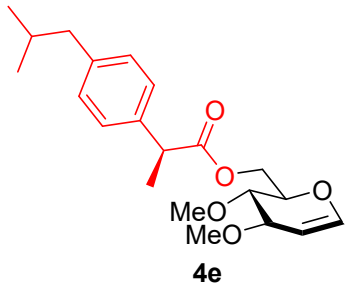
The compound **4d** was synthesized according to the general procedure **4b** and isolated through column chromatography on

silica gel. R_f = 0.7 (30%, n-hexane/ ethyl acetate), eluent 9% (n-hexane/ethyl acetate), pale yellow gummy (112.1 mg, 85%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.00 (d, *J* = 8.5 Hz, 2H), 6.57 (d, *J* = 8.4 Hz, 2H), 6.30 (d, *J* = 6.2 Hz, 1H), 4.81 (dd, *J* = 6.2, 2.9 Hz, 1H), 4.33 (dd, *J* = 12.1, 2.4 Hz, 1H), 4.25 (dd, *J* = 12.1, 6.0 Hz, 1H), 4.00 – 3.95 (m, 1H), 3.81 (dd, *J* = 5.0, 2.2 Hz, 1H), 3.63 (t, *J* = 7.1 Hz, 4H), 3.55 (t, *J* = 7.0 Hz, 4H), 3.46 (s, 3H), 3.35 (dd, *J* = 6.3, 3.9 Hz, 1H), 3.33 (s, 3H), 2.50 (t, *J* = 7.5 Hz, 2H), 2.30 (t, *J* = 7.5 Hz, 2H), 1.90 – 1.81 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 173.3, 144.2, 130.7, 129.8 (*2C*), 129.7, 112.3 (*2C*), 99.6, 76.3, 76.0, 74.9, 62.6, 59.3, 55.9, 53.7 (*2C*), 40.5 (*2C*), 33.9, 33.5, 26.7.

HRMS (ESI) calcd for $[C_{22}H_{31}Cl_2O_5+H]^+$ 460.1652, found 460.1646.



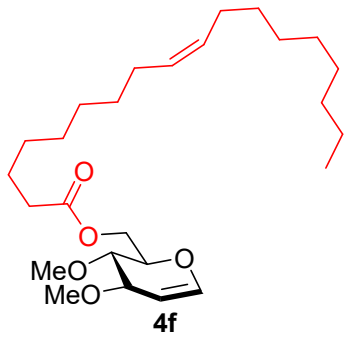
4e

The compound **4e** was synthesized according to the general procedure **4b** and isolated through column chromatography on silica gel. R_f = 0.63 (30%, n-hexane/ ethyl acetate), eluent 12% (n-hexane/ethyl acetate), pale yellow gummy (94.7 mg, 91%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.14 (d, *J* = 7.3 Hz, 2H), 7.01 (d, *J* = 7.3 Hz, 2H), 6.27 (d, *J* = 6.0 Hz, 1H), 4.80 – 4.71 (m, 1H), 4.29 (d, *J* = 12.1 Hz, 1H), 4.21 (dd, *J* = 12.0, 4.2 Hz, 1H), 3.92 – 3.85 (m, 1H), 3.76 (d, *J* = 5.4 Hz, 1H), 3.69 (d, *J* = 7.0 Hz, 1H), 3.28 (s, 3H), 3.23 (d, *J* = 7.3 Hz, 1H), 3.13 (s, 3H), 2.35 (d, *J* = 7.0 Hz, 2H), 1.82 – 1.69 (m, 1H), 1.43 (d, *J* = 7.1 Hz, 3H), 0.81 (d, *J* = 6.4 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 173.4, 143.3, 139.6, 136.6, 128.3 (*2C*), 126.2 (*2C*), 98.8, 75.9, 74.4, 73.8, 61.5, 58.1, 54.8, 44.0 (*2C*), 29.2, 21.3 (*2C*), 17.3.

HRMS (ESI) calcd for $[C_{21}H_{30}O_5+Na]^+$ 385.1985, found 385.1991.



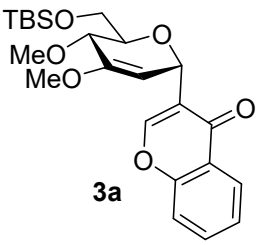
4f

The compound **4f** was synthesized according to the general procedure **4b** and isolated through column chromatography on silica gel. R_f = 0.73 (10%, n-hexane/ ethyl acetate), eluent 4% (n-hexane/ethyl acetate), pale yellow gummy (105.3 mg, 84%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 6.30 (d, *J* = 6.0 Hz, 1H), 5.27 (d, *J* = 1.3 Hz, 2H), 4.82 – 4.78 (m, 1H), 4.33 (d, *J* = 12.1 Hz, 1H), 4.24 (dd, *J* = 12.0, 5.8 Hz, 1H), 3.97 (t, *J* = 6.7 Hz, 1H), 3.81 (s, 1H, *bs*), 3.46 (s, 3H), 3.35 (s, 1H, *bs*), 3.33 (s, 3H), 2.28 (t, *J* = 7.4 Hz, 2H), 1.94 – 1.92 (m, 4H), 1.59 – 1.54 (m, 2H), 1.23 – 1.20 (m, 20H), 0.81 (t, *J* = 6.7 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 172.6, 143.2, 129.0, 128.7, 98.6, 75.4, 74.9, 73.9, 61.5, 58.3, 54.8, 33.2, 30.9, 28.8, 28.7, 28.5, 28.3 (*2C*), 28.2, 28.1, 28.1, 26.2, 26.2, 23.9, 21.7, 13.1.

HRMS (ESI) calcd for $[C_{26}H_{46}O_5+Na]^+$ 461.3239, found 461.3237.



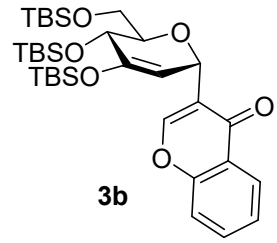
3a

The compound **3a** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.57 (20%, n-hexane/ ethyl acetate), eluent 10% (n-hexane/ethyl acetate), pale yellow solid (35.1 mg, 78%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.8 Hz, 1H), 8.07 (s, 1H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.32 (t, *J* = 7.5 Hz, 1H), 5.50 (s, 1H), 5.03 (d, *J* = 2.2 Hz, 1H), 4.04 (dd, *J* = 9.0, 6.2 Hz, 1H), 3.79 – 3.71 (m, 2H), 3.65 (d, *J* = 1.8 Hz, 1H), 3.50 (s, 3H), 3.41 (s, 3H), 0.82 (s, 9H), -0.00 (d, *J* = 5.5 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.7, 155.4, 153.1, 151.0, 132.6, 124.7, 124.0, 123.3, 123.0, 117.2, 96.2, 74.7, 72.4, 64.9, 61.1, 56.9, 53.5, 24.9 (*3C*), 17.2, -6.4, -6.4.

HRMS (ESI) calcd for $[C_{23}H_{32}O_6Si+H]^+$ 433.2041, found 433.2039.



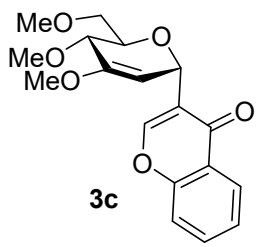
3b

The compound **3b** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.65 (10%, n-hexane/ ethyl acetate), eluent 3% (n-hexane/ethyl acetate), colourless gummy (28.36 mg, 73%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.9 Hz, 1H), 8.11 (s, 1H), 7.58 (t, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.32 (t, *J* = 7.5 Hz, 1H), 5.46 (s, 1H, *bs*), 5.04 (s, 1H), 3.97 (s, 1H, *bs*), 3.92 (t, *J* = 6.4 Hz, 1H), 3.73 (d, *J* = 6.8 Hz, 2H), 0.86 (s, 18H), 0.83 (s, 9H), 0.11 (s, 3H), 0.08 (s, 3H), 0.06 (s, 3H), 0.04 (s, 3H), -0.00 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.5, 155.4, 153.9, 147.3, 132.4, 124.7, 124.0, 123.8, 123.0, 117.2, 103.7, 66.2, 64.4, 61.1, 59.4, 24.9 (*6C*), 24.8 (*3C*), 17.3, 17.2, 17.1, -5.1, -5.2 (*2C*), -5.6, -6.4, -6.5.

HRMS (ESI) calcd for $[C_{33}H_{56}O_6Si_3+H]^+$ 633.3457, found 633.3433.



3c

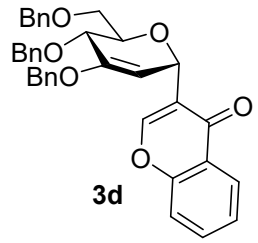
The compound **3c** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.45 (30%, n-hexane/ ethyl acetate), eluent 18% (n-hexane/ethyl acetate), brownish gummy (35.5 mg, 67%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.23 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.15 (d, *J* = 0.9 Hz, 1H), 7.67 (ddd, *J* = 8.6, 7.2, 1.6 Hz, 1H), 7.46 (d, *J* = 8.4 Hz, 1H), 7.44 – 7.37 (m,

1H), 5.73 – 5.42 (m, 1H), 5.11 (d, *J* = 2.7 Hz, 1H), 4.31 – 4.17 (m, 1H), 3.69 – 3.63 (m, 2H), 3.58 (s, 3H), 3.54 (dd, *J* = 11.0, 6.2 Hz, 1H), 3.51 (s, 3H), 3.41 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.8, 156.4, 154.2, 152.5, 133.6, 125.7, 125.1, 124.0, 123.9, 118.2, 97.1, 74.0, 73.5, 71.0, 65.7, 59.4, 58.2, 54.6.

HRMS (ESI) calcd for $[C_{18}H_{20}O_6+Na]^+$ 355.1152, found 355.1154.



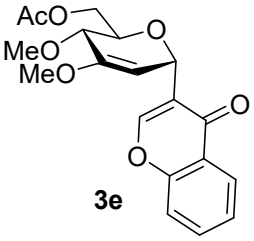
3d

The compound **3d** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel.R_f = 0.40 (20%, n-hexane/ ethyl acetate), eluent 12% (n-hexane/ethyl acetate), as a white solid (27.9 mg, 69%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.24 (d, *J* = 7.8 Hz, 1H), 8.11 (s, 1H), 7.66 (t, *J* = 7.6 Hz, 1H), 7.45 (d, *J* = 8.4 Hz, 1H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.37 – 7.26 (m, 15H), 5.67 (s, 1H, *bs*), 5.19 (s, 1H, *bs*), 4.81 (m, *J* = 9.8 Hz, 3H), 4.63 (d, *J* = 11.5 Hz, 1H), 4.55 (s, 2H), 4.22 (d, *J* = 2.9 Hz, 1H), 4.03 (s, 1H, *bs*), 3.75 – 3.67 (m, 1H), 3.64 – 3.56 (m, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.8, 156.4, 154.4, 152.4, 138.3, 138.0, 136.7, 133.7, 128.5 (*2C*), 128.4 (*2C*), 128.3 (*2C*), 128.1 (*2C*), 127.9, 127.8 (*2C*), 127.7, 127.7, 127.5 (*2C*), 125.8, 125.1, 124.1, 123.6, 118.2, 97.9, 74.4, 73.5, 72.97, 71.5, 69.1, 68.5, 66.1.

HRMS (ESI) calcd for $[C_{36}H_{32}O_6+Na]^+$ 583.2091, found 583.2060.



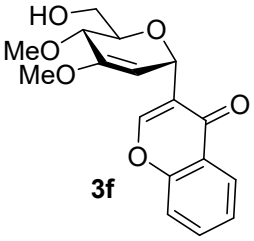
3e

The compound **3e** was synthesized according to the general procedure **3e** and isolated through column chromatography on silica gel. R_f = 0.35 (30%, n-hexane/ ethyl acetate), eluent 17% (n-hexane/ethyl acetate), brownish gummy (32.5 mg, 65%).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.21 (d, *J* = 7.9 Hz, 1H), 8.09 (s, 1H), 7.65 (t, *J* = 8.0 Hz, 1H), 7.44 (d, *J* = 8.6 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 5.64 (s, 1H), 5.06 (d, *J* = 2.6 Hz, 1H), 4.29 (td, *J* = 9.8, 4.2 Hz, 1H), 4.25 – 4.15 (m, 2H), 3.61 (d, *J* = 1.9 Hz, 1H), 3.57 (s, 3H), 3.49 (s, 3H), 2.07 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 176.3, 170.4, 156.2, 153.9, 152.4, 133.4, 125.6, 124.9, 123.9, 123.7, 117.9, 96.8, 73.4, 72.7, 65.7, 62.6, 58.1, 54.3, 20.5.

HRMS (ESI) calcd for $[C_{19}H_{20}O_7+H]^+$ 361.1282, found 361.1290



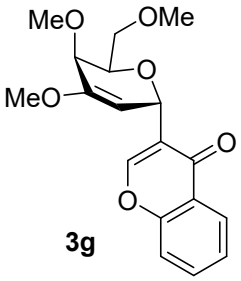
3f

The compound **3f** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.30 (60%, n-hexane/ ethyl acetate), eluent 43% (n-hexane/ethyl acetate), brownish gummy (22.5 mg, 41%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (dd, *J* = 8.0, 1.2 Hz, 1H), 8.01 (s, 1H), 7.62 – 7.57 (m, 1H), 7.37 (d, *J* = 8.4 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 5.64 (d, *J* = 2.8 Hz, 1H), 4.85 (d, *J* = 3.2 Hz, 1H), 3.82 (td, *J* = 6.1, 4.1 Hz, 1H), 3.73 (dd, *J* = 11.7, 6.7 Hz, 1H), 3.68 – 3.62 (m, 2H), 3.54 (s, 3H), 3.46 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.9, 155.4, 153.8, 153.5, 132.8, 124.9, 124.2, 123.0, 122.0, 117.2, 94.5, 73.1, 72.1, 64.7, 60.5, 58.1, 53.6.

HRMS (ESI) calcd for $[C_{17}H_{18}O_6+Na]^+$ 341.0996, found 341.1003.



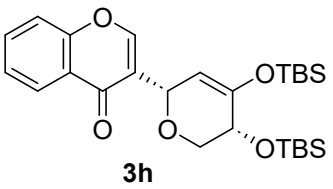
3g

The compound **3g** was synthesized according to the general procedure xx and isolated through column chromatography on silica gel. R_f = 0.43 (30%, n-hexane/ ethyl acetate), eluent 18% (n-hexane/ethyl acetate), as a pale yellow solid (37.6 mg, 71%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.22 (d, *J* = 7.9 Hz, 1H), 8.12 (s, 1H), 7.67 (t, *J* = 7.7 Hz, 1H), 7.46 (d, *J* = 8.5 Hz, 1H), 7.40 (t, *J* = 7.5 Hz, 1H), 5.75 (d, *J* = 3.1 Hz, 1H), 5.38 (d, *J* = 3.4 Hz, 1H), 4.00 (td, *J* = 6.0, 1.9 Hz, 1H), 3.67 (d, *J* = 6.0 Hz, 2H), 3.63 (s, 3H), 3.61 (d, *J* = 1.5 Hz, 1H), 3.50 (s, 3H), 3.43 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.8, 156.4, 154.2, 152.5, 133.6, 125.7, 125.1, 124.0, 123.9, 118.2, 97.1, 74.0, 73.5, 71.0, 65.7, 59.4, 58.2, 54.6.

HRMS (ESI) calcd for $[C_{18}H_{20}O_6+H]^+$ 333.1333, found 333.1324.

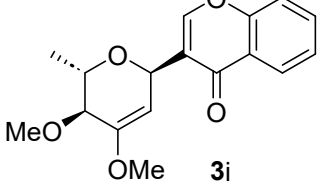


3h

The compound **3h** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.57 (20%, n-hexane/ ethyl acetate), eluent 10% (n-hexane/ethyl acetate), white gummy (26.8 mg, 63%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12 (d, *J* = 7.9 Hz, 1H), 7.99 (s, 1H), 7.54 (t, *J* = 7.7 Hz, 1H), 7.35 (d, *J* = 8.4 Hz, 1H), 7.28 (t, *J* = 7.5 Hz, 1H), 5.36 (s, 1H, *bs*), 4.95 (s, 1H, *bs*), 3.86 – 3.79 (m, 2H), 3.72 – 3.67 (m, 1H), 0.81 (s, 18H), 0.06 (s, 3H), 0.03 (s, 3H), -0.00 (s, 3H), -0.05 (s, 3H). **^{13}C NMR (151 MHz, $CDCl_3$)** δ 175.4, 155.4, 153.6, 148.7, 132.5, 124.8, 124.0, 123.5, 123.0, 117.2, 104.8, 70.2, 67.3, 66.3, 24.9 (*3C*), 24.8 (*3C*), 17.2, 17.1, -5.2 (*2C*), -5.5(*2C*).

HRMS (ESI) calcd for $[C_{26}H_{40}O_5Si_2+Na]^+$ 511.2306, found 511.2302



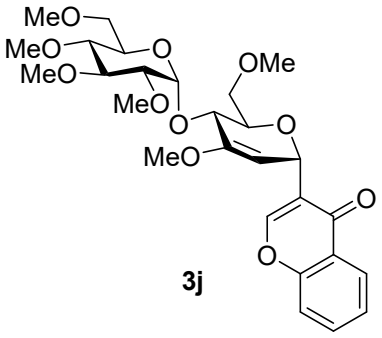
3i

The compound **3i** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.50 (20%, n-hexane/ ethyl acetate), eluent 12% (n-hexane/ethyl acetate), pale yellow solid (43.0 mg, 75%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 (d, *J* = 7.9 Hz, 1H), 8.03 (s, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 5.53 (s, 1H, *bs*), 4.99 (d, *J* = 1.8 Hz, 1H), 4.19 (qd, *J* = 6.6, 2.2 Hz, 1H), 3.50 (s, 3H), 3.43 (s, 3H), 3.36 (s, 1H, *bs*), 1.26 (d, *J* = 6.7 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.7, 155.4, 153.2, 150.8, 132.6, 124.7, 124.1, 123.3, 123.0, 117.2, 96.3, 76.7, 70.1, 63.2, 56.8, 53.4, 15.3.

HRMS (ESI) calcd for $[C_{17}H_{18}O_5+H]^+$ 303.1227, found 303.1226

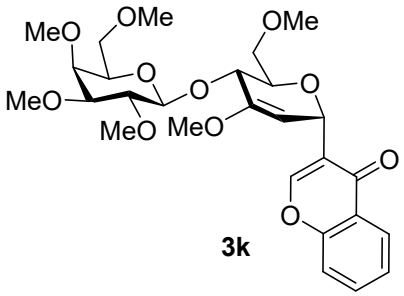


3j

The compound **3j** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.27 (40%, n-hexane/ ethyl acetate), eluent 35% (n-hexane/ethyl acetate), brownish gummy (23.4 mg, 57%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 (dd, *J* = 8.0, 1.5 Hz, 1H), 8.14 (d, *J* = 0.8 Hz, 1H), 7.60 (ddd, *J* = 8.6, 7.2, 1.6 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 1H), 7.35 – 7.32 (m, 1H), 5.58 – 5.54 (m, 1H), 5.38 (d, *J* = 3.8 Hz, 1H), 5.02 (d, *J* = 2.7 Hz, 1H), 4.26 – 4.18 (m, 1H), 4.02 (dd, *J* = 3.1, 1.0 Hz, 1H), 3.63 (ddd, *J* = 10.2, 3.2, 2.2 Hz, 1H), 3.61 – 3.58 (m, 1H), 3.57 (s, 3H), 3.54 (dd, *J* = 10.5, 3.6 Hz, 1H), 3.50 (d, *J* = 2.0 Hz, 1H), 3.48 (s, 3H), 3.46 (s, 3H), 3.45 (d, *J* = 4.7 Hz, 1H), 3.44 – 3.41 (m, 1H), 3.34 (s, 3H), 3.33 (s, 3H), 3.32 (s, 3H), 3.19 – 3.12 (m, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.8, 156.4, 154.9, 152.3, 133.6, 125.7, 125.1, 124.0, 123.9, 118.3, 97.5, 96.2, 82.7, 81.4, 79.3, 75.4, 70.9, 70.8, 70.5, 69.4, 65.4, 60.8, 60.5, 59.2 (*2C*), 57.6, 54.4.



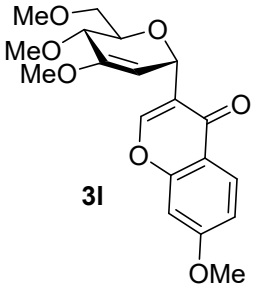
3k

HRMS (ESI) calcd for $[C_{27}H_{36}O_{11}+Na]^+$ 559.2150, found 559.2167

The compound **3k** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.35 (50%, n-hexane/ ethyl acetate), eluent 38% (n-hexane/ethyl acetate), pale yellow gummy (22.6 mg, 55%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 (dd, *J* = 8.0, 1.1 Hz, 1H), 8.07 (s, 1H), 7.62 – 7.58 (m, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 5.53 (s, 1H, *bs*), 5.04 (d, *J* = 2.3 Hz, 1H), 4.41 – 4.35 (m, 1H), 4.32 (d, *J* = 7.7 Hz, 1H), 3.97 (s, 1H), 3.62 (dd, *J* = 10.0, 7.5 Hz,

1H), 3.58 (d, *J* = 3.3 Hz, 2H), 3.57 (s, 3H), 3.48 (s, 3H), 3.45 (s, 6H), 3.44 (s, 1H, *bs*), 3.43 (d, *J* = 4.1 Hz, 1H), 3.42 (s, 1H, *bs*), 3.32 (d, *J* = 2.2 Hz, 6H), 3.29 (dd, *J* = 9.5, 7.9 Hz, 1H), 3.07 (dd, *J* = 9.7, 3.0 Hz, 1H).



3l

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.7, 155.4, 153.3, 149.5, 132.6, 124.7, 124.1, 123.3, 123.0, 117.2, 101.2, 97.2, 83.0, 79.4, 73.9, 73.7, 72.2, 70.9, 69.7, 69.4, 64.1, 60.2, 59.9, 58.3, 58.2, 57.3, 53.5.

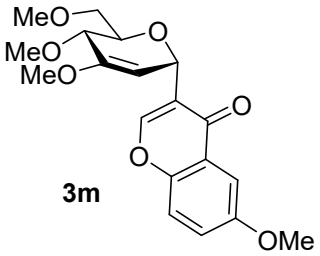
HRMS (ESI) calcd for $[C_{27}H_{36}O_{11}+H]^+$ 537.2330, found 537.2308

The compound **3l** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.30 (30%, n-hexane/ ethyl acetate), eluent 24% (n-hexane/ethyl acetate), pale yellow solid (36.4 mg, 63%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.9 Hz, 1H), 7.99 (d, *J* = 0.9 Hz, 1H), 6.90 (dd, *J* = 8.9, 2.3 Hz, 1H), 6.77 (d, *J* = 2.3 Hz, 1H), 5.57 – 5.50 (m, 1H), 5.04 (d, *J* = 2.7 Hz, 1H), 4.14 (dt, *J* = 7.0, 4.6 Hz, 1H), 3.83 (s, 3H), 3.59 – 3.57 (m, 2H), 3.51 (s, 3H), 3.47 – 3.45 (m, 1H), 3.44 (s, 3H), 3.34 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.2, 163.1, 157.2, 152.7, 151.3, 126.1, 122.6, 116.9, 113.7, 99.1, 96.2, 72.9, 72.5, 69.9, 64.6, 58.3, 57.2, 54.8, 53.5.

HRMS (ESI) calcd for $[C_{19}H_{22}O_7+H]^+$ 363.1438, found 363.1436.



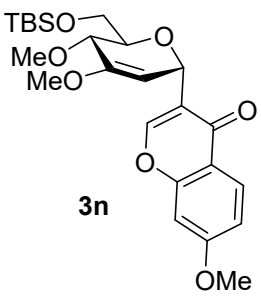
3m

The compound **3m** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.33 (30%, n-hexane/ ethyl acetate), eluent 22% (n-hexane/ethyl acetate), brownish gummy (37.5 mg, 65%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.51 (d, *J* = 2.7 Hz, 1H), 7.33 (d, *J* = 9.1 Hz, 1H), 7.20 (s, 1H), 5.56 (s, 1H, *bs*), 5.03 (d, *J* = 2.2 Hz, 1H), 4.16 – 4.12 (m, 1H), 3.83 (s, 3H), 3.62 – 3.57 (m, 2H), 3.51 (s, 3H), 3.46 (s, 1H, *bs*), 3.44 (s, 3H), 3.34 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.6, 155.9, 153.0, 151.5, 150.3, 123.5, 122.8, 122.0, 118.6, 103.6, 96.0, 72.8, 72.4, 70.0, 64.7, 58.3, 57.2, 54.9, 53.5.

HRMS (ESI) calcd for $[C_{19}H_{22}O_7+H]^+$ 363.1438, found 363.1452.



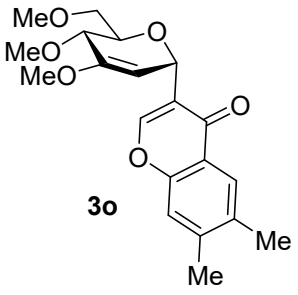
3n

The compound **3n** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.35 (20%, n-hexane/ ethyl acetate), eluent 17% (n-hexane/ethyl acetate), white solid (35.6 mg, 74%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.9 Hz, 1H), 7.99 (s, 1H), 6.89 (dd, *J* = 8.9, 2.3 Hz, 1H), 6.76 (d, *J* = 2.3 Hz, 1H), 5.48 (s, 1H, *bs*), 5.03 (d, *J* = 2.5 Hz, 1H), 4.04 (dd, *J* = 6.2, 3.1 Hz, 1H), 3.82 (s, 3H), 3.75 (m, 2H), 3.65 (d, *J* = 1.9 Hz, 1H), 3.50 (s, 3H), 3.42 (s, 3H), 0.82 (s, 9H), -0.00 (d, *J* = 1.1 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.1, 164.0, 158.2, 153.6, 151.9, 127.1, 124.1, 117.9, 114.7, 100.1, 97.4, 75.7, 73.4, 65.9, 62.1, 57.9, 55.8, 54.5, 25.9 (*3C*), 18.2, -5.4, -5.4.

HRMS (ESI) calcd for $[C_{24}H_{34}O_7Si + H]^+$ 463.2147, found 463.2161.



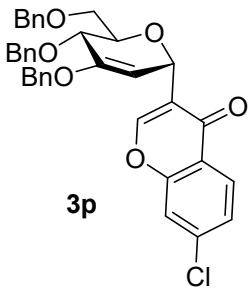
3o

The compound **3o** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.30 (30%, n-hexane/ ethyl acetate), eluent 25% (n-hexane/ethyl acetate), yellow solid (35.0 mg, 61%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.88 (s, 1H), 7.16 (s, 1H), 5.54 (d, *J* = 1.1 Hz, 1H), 5.03 (d, *J* = 2.6 Hz, 1H), 4.14 (dt, *J* = 7.0, 4.5 Hz, 1H), 3.62 – 3.56 (m, 2H), 3.50 (s, 3H), 3.47 – 3.44 (m, 1H), 3.43 (s, 3H), 3.34 (s, 3H), 2.31 (s, 3H), 2.28 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.7, 154.0, 152.8, 151.3, 143.1, 133.4, 124.2, 122.4, 120.8, 117.2, 96.3, 72.8, 72.4, 69.9, 64.6, 58.3, 57.1, 53.5, 19.4, 18.3.

HRMS (ESI) calcd for $[C_{20}H_{24}O_6+H]^+$ 361.1646, found 361.1641.



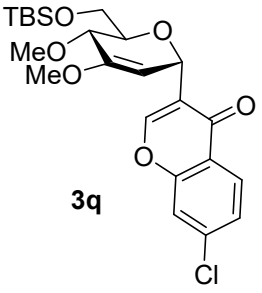
3p

The compound **3p** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.52 (20%, n-hexane/ ethyl acetate), eluent 9% (n-hexane/ethyl acetate), yellow gummy (27.0 mg, 63%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11 (d, *J* = 2.5 Hz, 1H), 8.02 (s, 1H), 7.52 (dd, *J* = 8.9, 2.5 Hz, 1H), 7.34 (d, *J* = 8.9 Hz, 1H), 7.30 – 7.19 (m, 15H), 5.56 (s, 1H, *bs*), 5.09 (d, *J* = 2.9 Hz, 1H), 4.77 – 4.68 (m, 3H), 4.55 (d, *J* = 11.5 Hz, 1H), 4.47 (s, 2H, *bs*), 4.14 (dd, *J* = 10.6, 4.6 Hz, 1H), 3.93 (d, *J* = 4.3 Hz, 1H), 3.61 (dd, *J* = 10.2, 6.4 Hz, 1H), 3.52 (dd, *J* = 10.2, 4.6 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.7, 154.7, 154.6, 152.5, 138.2, 137.9, 136.6, 133.9, 131.1, 128.5 (*2C*), 128.4, 128.4, 128.3 (*2C*), 128.1 (*2C*), 127.9, 127.8 (*2C*), 127.7, 127.7, 127.5 (*2C*), 125.2, 124.9, 123.8, 120.0, 97.6, 74.4, 73.5, 73.0, 71.5, 69.2, 68.5, 66.0.

HRMS (ESI) calcd for $[C_{19}H_{22}O_7+H]^+$ 595.1882, found 595.1930.



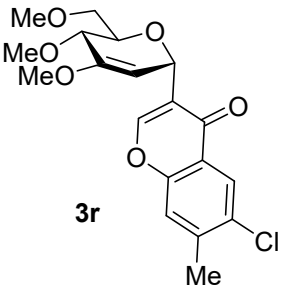
3q

The compound **3q** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.50 (20%, n-hexane/ ethyl acetate), eluent 12% (n-hexane/ethyl acetate), yellow solid (31.1 mg, 64%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.11 (d, *J* = 2.5 Hz, 1H), 8.08 (s, 1H), 7.52 (dd, *J* = 8.9, 2.5 Hz, 1H), 7.35 (d, *J* = 8.9 Hz, 1H), 5.47 (s, 1H, *bs*), 5.00 (d, *J* = 2.5 Hz, 1H), 4.03 (td, *J* = 6.2, 3.2 Hz, 1H), 3.74 (m, 2H), 3.65 – 3.62 (m, 1H), 3.50 (s, 3H), 3.41 (s, 3H), 0.82 (s, 9H), -0.00 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.6, 154.7, 154.3, 152.3, 133.9, 131.1, 125.1, 124.9, 124.5, 120.0, 96.8, 75.8, 73.4, 65.9, 62.1, 58.0, 54.5, 25.9 (*3C*), 18.2, -5.4, -5.4.

HRMS (ESI) calcd for $[C_{23}H_{31}ClO_6Si + Na]^+$ 489.1471, found 489.1487.



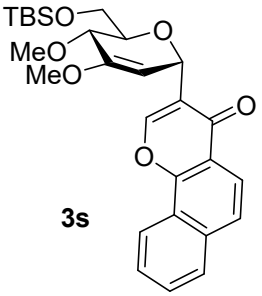
3r

The compound **3r** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.57 (20%, n-hexane/ ethyl acetate), eluent 10% (n-hexane/ethyl acetate), yellow solid (41.2 mg, 68%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.10 (s, 1H), 8.03 (s, 1H), 7.28 (s, 1H), 5.52 (s, 1H, *bs*), 5.01 (d, *J* = 2.2 Hz, 1H), 4.19 – 4.11 (m, 1H), 3.60 – 3.55 (m, 2H), 3.51 (s, 3H), 3.47 – 3.43 (m, 1H), 3.43 (s, 3H), 3.34 (s, 3H), 2.42 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 174.6, 153.7, 153.1, 151.5, 141.9, 130.9, 124.3, 122.8, 122.0, 119.0, 95.9, 72.9, 72.5, 69.9, 64.6, 58.3, 57.2, 53.5, 19.8.

HRMS (ESI) calcd for $[C_{19}H_{21}ClO_6 + H]^+$ 381.1099, found 381.1096.



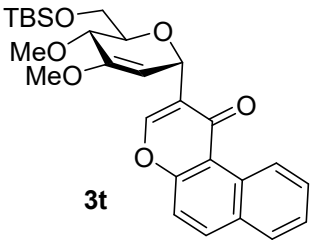
3s

The compound **3s** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.6 (20%, n-hexane/ ethyl acetate), eluent 8% (n-hexane/ethyl acetate), yellow gummy (37.7 mg, 75%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.39 (d, *J* = 8.2 Hz, 1H), 8.27 (s, 1H), 8.07 (d, *J* = 8.7 Hz, 1H), 7.84 (d, *J* = 8.0 Hz, 1H), 7.68 (d, *J* = 8.7 Hz, 1H), 7.64 – 7.56 (m, 1H), 7.16 (s, 1H, *bs*), 5.54 (d, *J* = 1.1 Hz, 1H), 5.09 (d, *J* = 2.6 Hz, 1H), 4.07 (td, *J* = 6.2, 3.2 Hz, 1H), 3.81 – 3.71 (m, 2H), 3.68 – 3.64 (m, 1H), 3.50 (s, 3H), 3.42 (s, 3H), 0.82 (s, 9H), -0.00 (d, *J* = 1.5 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.6, 153.9, 153.3, 152.1, 135.8, 129.3, 128.1, 127.2, 125.8, 125.3, 124.1, 122.4, 120.7, 120.3, 97.2, 75.8, 73.4, 66.1, 62.2, 57.9, 54.5, 25.9 (*3C*), 18.3, -5.4, -5.4.

HRMS (ESI) calcd for $[C_{27}H_{34}O_6Si + H]^+$ 483.2197, found 483.2195.



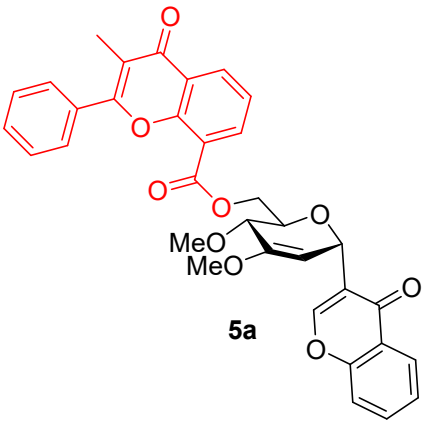
3t

The compound **3t** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel R_f = 0.57 (20%, n-hexane/ ethyl acetate), eluent 10% (n-hexane/ethyl acetate), brownish solid (37.1 mg, 74%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 9.98 (d, *J* = 8.6 Hz, 1H), 8.12 (s, 1H), 7.99 (d, *J* = 9.0 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H), 7.67 (t, *J* = 7.8 Hz, 1H), 7.53 (t, *J* = 7.4 Hz, 1H), 7.42 (d, *J* = 9.0 Hz, 1H), 5.59 (s, 1H, *bs*), 5.10 (d, *J* = 2.0 Hz, 1H), 4.08 (td, *J* = 6.4, 3.0 Hz, 1H), 3.81 – 3.74 (m, 2H), 3.67 (d, *J* = 1.3 Hz, 1H), 3.51 (s, 3H), 3.42 (s, 3H), 0.82 (s, 9H), 0.00 (d, *J* = 2.4 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 177.4, 156.6, 151.0, 150.6, 134.4, 129.6, 128.2, 127.2 (*2C*), 126.0, 126.0, 125.6, 116.7, 116.2, 96.3, 74.8, 72.5, 65.0, 61.0, 56.8, 53.5, 24.8 (*3C*), 17.2, -6.4, -6.4.

HRMS (ESI) calcd for $[C_{27}H_{34}O_6Si + H]^+$ 483.2197, found 483.2194.



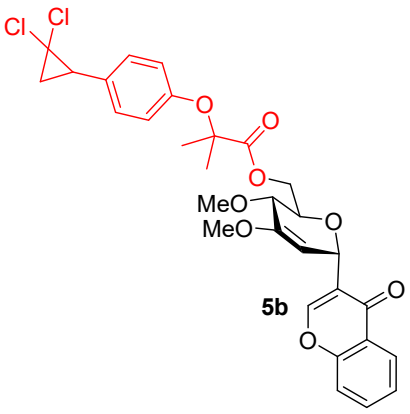
5a

The compound **5a** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.4 (40%, n-hexane/ ethyl acetate), eluent 27% (n-hexane/ethyl acetate), brownish solid (25.0 mg, 62%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.38 (d, *J* = 7.8 Hz, 1H), 8.17 (d, *J* = 7.4 Hz, 1H), 8.13 (d, *J* = 7.9 Hz, 1H), 7.98 (s, 1H), 7.72 (d, *J* = 7.1 Hz, 2H), 7.59 (t, *J* = 7.6 Hz, 1H), 7.50 – 7.41 (m, 3H), 7.35 (dd, *J* = 15.1, 6.6 Hz, 3H), 5.61 (s, 1H, *bs*), 5.02 (s, 1H, *bs*), 4.57 – 4.49 (m, 1H), 4.42 (d, *J* = 11.6 Hz, 1H), 4.24 (d, *J* = 3.6 Hz, 1H), 3.60 (s, 1H, *bs*), 3.53 (s, 3H), 3.41 (s, 3H), 2.16 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 177.3, 175.6, 162.9, 160.1, 155.3, 153.5, 153.1, 151.8, 135.3, 132.7, 131.9, 130.0, 129.5, 128.4 (*2C*), 127.4 (*2C*), 124.7, 124.2, 123.0, 122.9, 122.5, 122.2, 119.1, 117.1, 116.6, 95.6, 72.5, 71.4, 64.9, 62.7, 57.6, 53.6, 10.8.

HRMS (ESI) calcd for $[C_{34}H_{28}O_9 + H]^+$, 581.1806, found 581.1819.

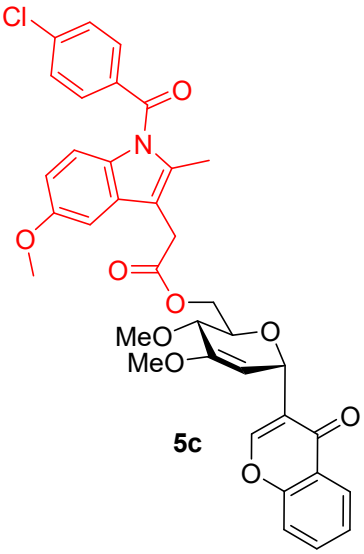


The compound **5b** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.47 (30%, n-hexane/ ethyl acetate), eluent 18% (n-hexane/ethyl acetate), pale yellow gummy (26.6 mg, 67%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (d, *J* = 7.9 Hz, 1H), 7.98 (s, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.38 (d, *J* = 8.4 Hz, 1H), 7.33 (t, *J* = 7.4 Hz, 1H), 6.98 (d, *J* = 7.7 Hz, 2H), 6.73 (d, *J* = 7.7 Hz, 2H), 5.52 (s, 1H, *bs*), 5.01 (s, 1H, *bs*), 4.38 – 4.29 (m, 1H), 4.21 (d, *J* = 11.6 Hz, 1H), 4.13 (d, *J* = 3.7 Hz, 1H), 3.50 (s, 3H), 3.46 (s, 1H, *bs*), 3.35 (s, 3H), 2.67 (d, *J* = 9.7 Hz, 1H), 1.80 – 1.74 (m, 1H), 1.66 – 1.62 (m, 1H), 1.52 (s, 3H), 1.49 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.6, 174.0, 156.4, 154.8, 154.0, 152.5, 133.7, 129.6 (*2C*), 128.2, 125.8, 125.2, 124.0, 123.5, 118.8 (*2C*), 118.2, 96.7, 79.1, 73.3, 72.3, 65.9, 63.7, 60.8, 58.5, 54.6, 34.8, 25.8, 25.5, 25.3.

HRMS (ESI) calcd for $[C_{30}H_{30}Cl_2O_8+H]^+$ 589.1390, found 589.1394.

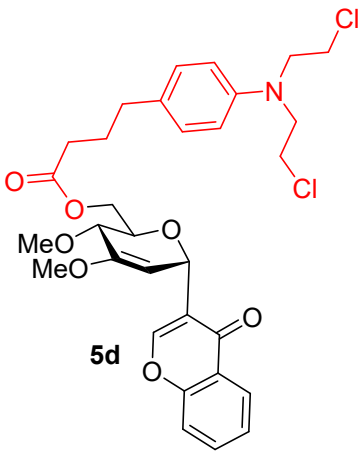


The compound **5c** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.3 (30%, n-hexane/ ethyl acetate), eluent 25% (n-hexane/ethyl acetate), yellow gummy (25.0 mg, 65%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.21 (d, *J* = 7.9 Hz, 1H), 8.00 (s, 1H), 7.67 (t, *J* = 7.8 Hz, 1H), 7.64 (d, *J* = 7.5 Hz, 2H), 7.47 – 7.44 (m, 3H), 7.41 (t, *J* = 7.5 Hz, 1H), 6.96 (s, 1H), 6.81 (d, *J* = 9.0 Hz, 1H), 6.57 (d, *J* = 9.0 Hz, 1H), 5.56 (s, 1H, *bs*), 5.05 (s, 1H, *bs*), 4.38 – 4.30 (m, 1H), 4.25 – 4.21 (m, 1H), 4.19 (d, *J* = 3.3 Hz, 1H), 3.80 (s, 3H), 3.70 (d, *J* = 2.9 Hz, 2H), 3.56 (s, 3H), 3.51 (d, *J* = 3.1 Hz, 1H), 3.40 (s, 3H), 2.34 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.5, 170.6, 168.3, 156.4, 156.1, 154.0, 152.6, 139.2, 136.0, 133.9, 133.7, 131.2 (*2C*), 130.7, 130.5, 129.1 (*2C*), 125.8, 125.2, 124.0, 123.5, 118.2, 114.9, 112.3, 111.8, 101.2, 96.7, 73.2, 72.4, 65.7, 63.1, 58.5, 55.7, 54.6, 30.3, 13.3.

HRMS (ESI) calcd for $[C_{36}H_{32}ClNO_9+H]^+$ 658.1838, found 658.1856.

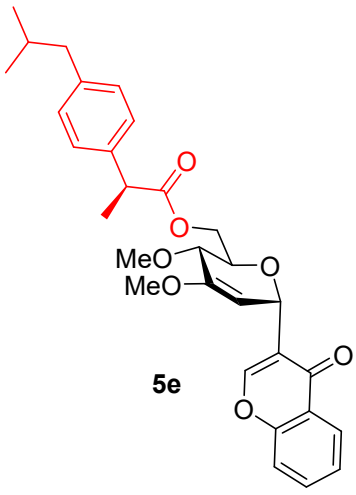


The compound **5d** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.4 (40%, n-hexane/ ethyl acetate), eluent 27% (n-hexane/ethyl acetate), brownish solid (27.2 mg, 69%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 (d, *J* = 7.9 Hz, 1H), 8.04 (s, 1H), 7.61 (t, *J* = 7.8 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.34 (t, *J* = 7.5 Hz, 1H), 6.97 (d, *J* = 8.4 Hz, 2H), 6.52 (d, *J* = 8.4 Hz, 2H), 5.59 (d, *J* = 1.0 Hz, 1H), 5.02 (d, *J* = 2.6 Hz, 1H), 4.26 – 4.23 (m, 1H), 4.20 – 4.12 (m, 2H), 3.61 (t, *J* = 7.1 Hz, 4H), 3.55 (s, 1H, *bs*), 3.54 – 3.52 (t, 4H), 3.52 (s, 3H), 3.43 (s, 3H), 2.46 (t, *J* = 7.5 Hz, 2H), 2.28 (t, *J* = 7.4 Hz, 2H), 1.82 (p, *J* = 7.5 Hz, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.6, 173.4, 156.4, 154.2, 152.5, 144.3, 133.7, 130.5, 129.7 (*2C*), 125.8, 125.2, 124.0, 123.7, 118.2, 112.2 (*2C*), 96.9, 73.4, 72.7, 65.8, 62.5, 58.4, 54.6, 53.6 (*2C*) , 40.5 (*2C*), 33.9, 33.5, 26.7.

HRMS (ESI) calcd for $[C_{31}H_{35}Cl_2NO_7+H]^+$ 604.1863, found:604.1855

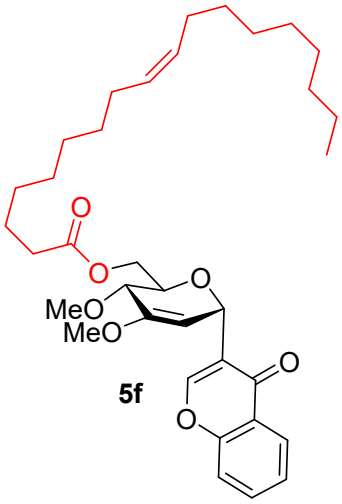


The compound **5e** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.27 (20%, n-hexane/ ethyl acetate), eluent 16% (n-hexane/ethyl acetate), pale yellow gummy (29.8 mg, 71%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.16 (d, *J* = 7.8 Hz, 1H), 7.94 (s, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 8.3 Hz, 1H), 7.34 (t, *J* = 7.3 Hz, 1H), 7.11 (d, *J* = 7.6 Hz, 2H), 6.95 (d, *J* = 7.5 Hz, 2H), 5.51 (s, 1H, *bs*), 4.98 (s, 1H, *bs*), 4.25 – 4.22 (m, 1H), 4.11 – 4.10 (m, 1H), 4.04 (d, *J* = 2.5 Hz, 1H), 3.68 – 3.65 (m, 1H), 3.49 (s, 3H), 3.44 (d, *J* = 1.0 Hz, 1H), 3.25 (s, 3H), 2.29 (d, *J* = 6.9 Hz, 2H), 1.74 – 1.65 (m, 1H), 1.40 (d, *J* = 7.0 Hz, 3H), 0.77 (d, *J* = 6.5 Hz, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 176.6, 174.4, 156.4, 154.1, 152.7, 140.6, 137.5, 133.7, 129.3 (*2C*), 127.2 (*2C*), 125.8, 125.2, 124.0, 123.5, 118.2, 96.4, 73.2, 72.1, 66.0, 62.9, 58.5, 55.0, 45.0, 45.0, 30.1, 22.3 (*2C*), 18.2.

HRMS (ESI) calcd for $[C_{30}H_{34}O_7+H]^+$ 507.2377, found 507.2375.

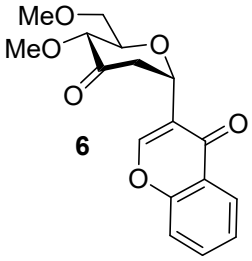


The compound **5f** was synthesized according to the general procedure **4c** and isolated through column chromatography on silica gel. R_f = 0.45 (10%, n-hexane/ ethyl acetate), eluent 4% (n-hexane/ethyl acetate), pale yellow gummy (22.7 mg, 57%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.0 Hz, 1H), 8.04 (s, 1H), 7.60 (t, *J* = 7.7 Hz, 1H), 7.39 (d, *J* = 8.3 Hz, 1H), 7.34 (t, *J* = 7.3 Hz, 1H), 5.58 (s, 1H, *bs*), 5.26 (d, *J* = 4.1 Hz, 2H), 5.02 (s, 1H, *bs*), 4.27 – 4.21 (m, 1H), 4.16 (s, 2H, *bs*), 3.56 (s, 1H, *bs*), 3.52 (s, 3H), 3.44 (s, 3H), 2.26 (t, *J* = 7.4 Hz, 2H), 1.97 – 1.88 (m, 4H), 1.59 – 1.48 (m, 2H), 1.19 (s, 20H, *bs*), 0.80 (t, *J* = 6.5 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 175.6, 172.6, 155.4, 153.1, 151.5, 132.7, 129.0, 128.7, 124.8, 124.1, 123.0, 122.7, 117.2, 95.8, 72.4, 71.6, 64.8, 61.5, 57.4, 53.6, 33.1, 30.9, 28.7, 28.7, 28.5, 28.3 (*3C, bs*), 28.1, 28.1, 26.2, 26.1, 23.9, 21.7, 13.1.

HRMS (ESI) calcd for $[C_{35}H_{50}O_7+H]^+$ 583.3629, found 583.3628.

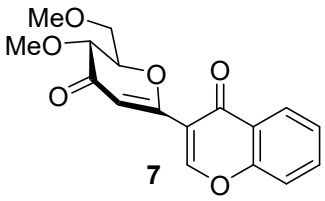


The compound **6** was synthesized according to the scheme **1B** and isolated through column chromatography on silica gel. R_f = 0.30 (30%, n-hexane/ ethyl acetate), eluent 20% (n-hexane/ethyl acetate), pale yellow gummy (21.0 mg, 73%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.14 (d, *J* = 6.9 Hz, 1H), 8.00 (s, 1H), 7.65 – 7.57 (m, 1H), 7.39 (d, *J* = 7.7 Hz, 1H), 7.34 (t, *J* = 6.3 Hz, 1H), 5.45 (s, 1H, *bs*), 4.00 (d, *J* = 3.0 Hz, 1H), 3.85 (d, *J* = 6.5 Hz, 1H), 3.64 – 3.54 (m, 2H), 3.43 (s, 3H), 3.32 (s, 3H), 3.07 – 3.02 (m, 1H), 2.64 (dd, *J* = 15.5, 7.2 Hz, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 205.7, 176.3, 156.2, 154.0, 133.9, 125.9, 125.3, 123.9, 123.4, 118.2, 81.2, 76.6, 72.1, 68.5, 59.5, 59.1, 44.3.

HRMS (ESI) calcd for $[C_{17}H_{18}O_6+H]^+$ 319.1176, found 319.1172.



The compound **7** was synthesized according to scheme **1B** and isolated through column chromatography on silica gel. R_f = 0.35 (30%, n-hexane/ ethyl acetate), eluent 20% (n-hexane/ethyl acetate), yellow solid (24.5 mg, 86%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.51 (s, 1H), 8.20 (d, *J* = 7.9 Hz, 1H), 7.64 (t, *J* = 7.7 Hz, 1H), 7.45 – 7.36 (m, 2H), 6.88 (s, 1H), 4.39 (dd, *J* = 11.2, 2.7 Hz, 1H), 3.91 (d, *J* = 11.2 Hz, 1H), 3.75 (s, 2H, *bs*), 3.61 (s, 3H), 3.41 (s, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 193.9, 174.1, 161.7, 157.7, 155.4, 134.2, 126.5, 126.1, 124.3, 118.1, 116.2, 105.5, 80.5, 76.2, 70.6, 60.6, 59.5.

HRMS (ESI) calcd for $[C_{17}H_{16}O_6+H]^+$ 317.1020, found 317.1021.

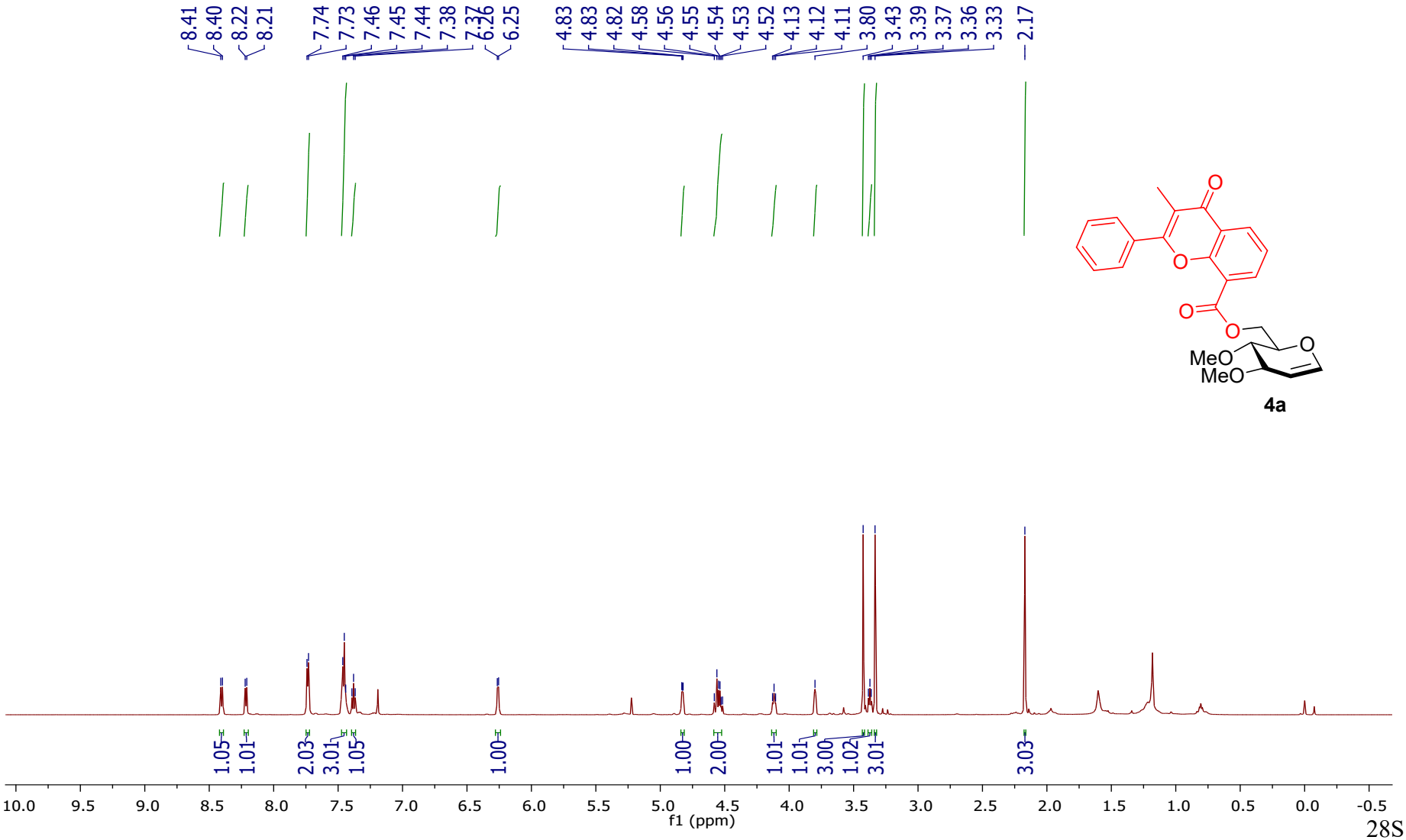
References

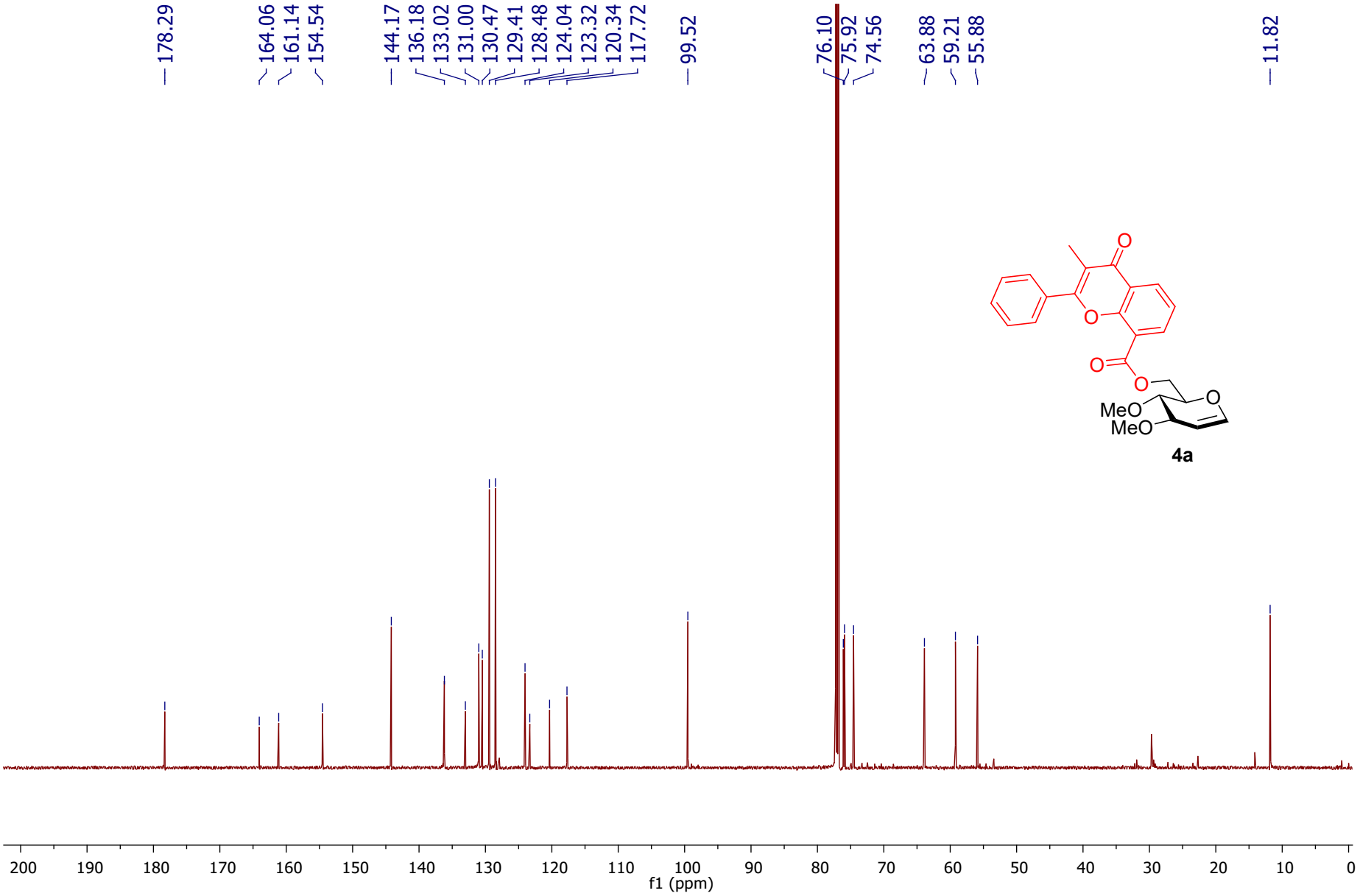
1 J. Zhao, S. Wei, X. Ma and H. Shao, *Carbohydr. Res.*, 2010, **345**, 168–171.

2 Q. Tong, R.-F. Xiu, J.-H. Chen, Y. Zhang, B.-D. Cui, N.-W. Wan, Y.-Z. Chen and W.-Y. Han, *ACS Catal.*, 2023, **13**, 12692–12699.

3 Q. Yang, Y. He, T. Wang, L. Zeng and Z. Zhang, *Mol. Divers*, 2016, **20**, 9–16.

7. NMR spectrum of compounds

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120.34
117.72
99.52
76.10
75.92
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63.88
59.21
55.88
11.82
O
O
O
O
O
MeO
MeO
4a
200
190
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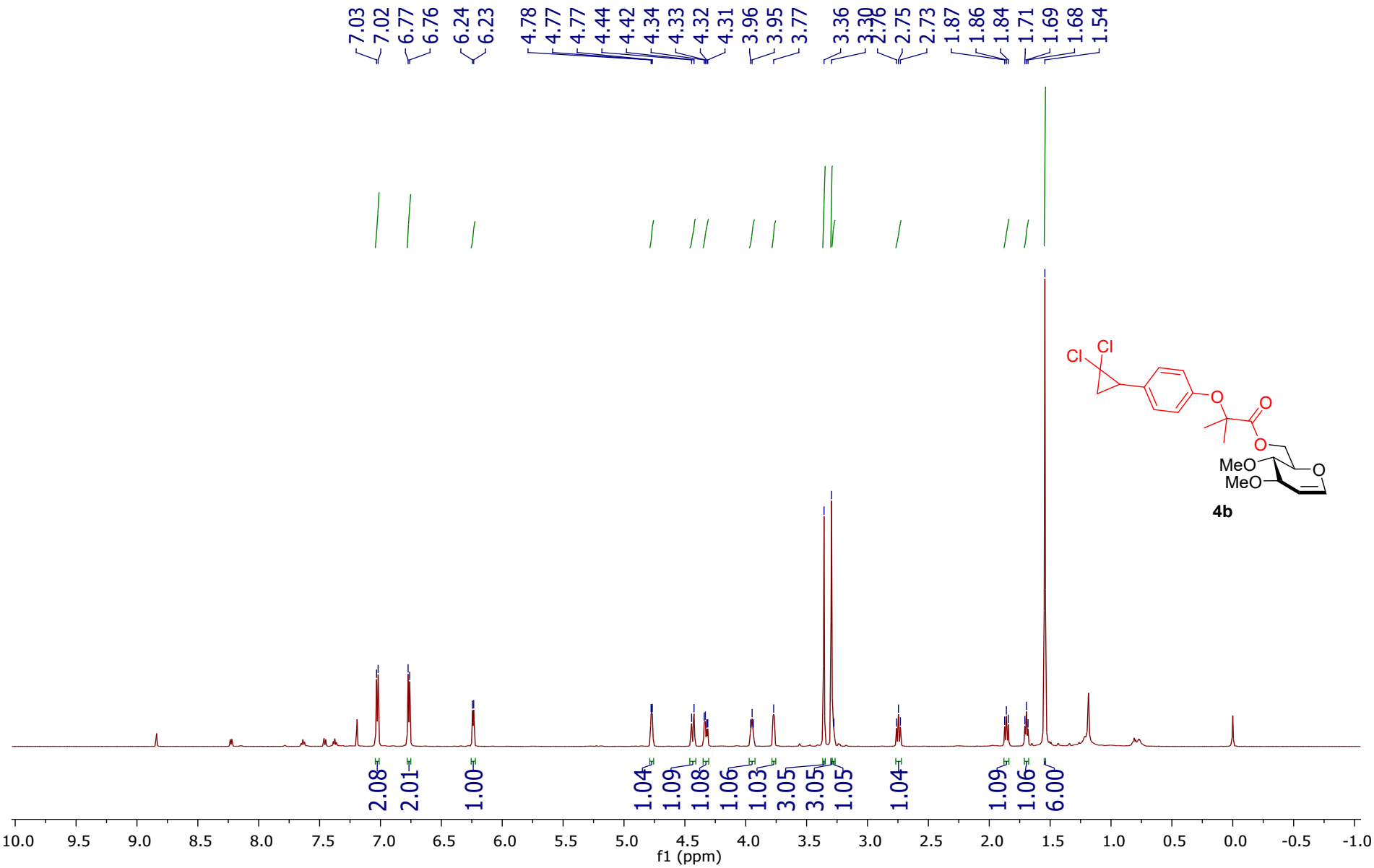
f1 (ppm)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f1 (ppm)
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O
O
O
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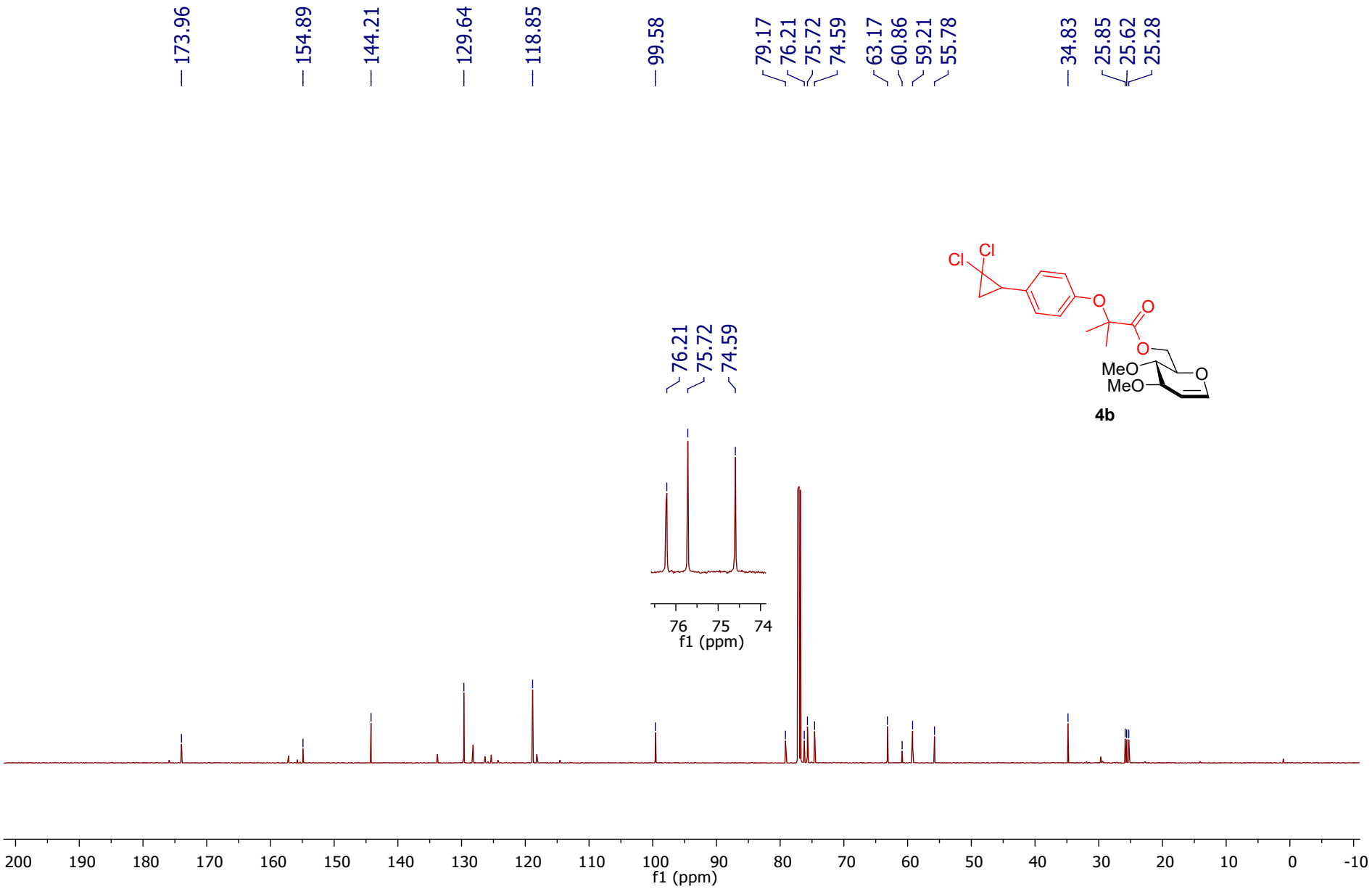
MeO
O

4b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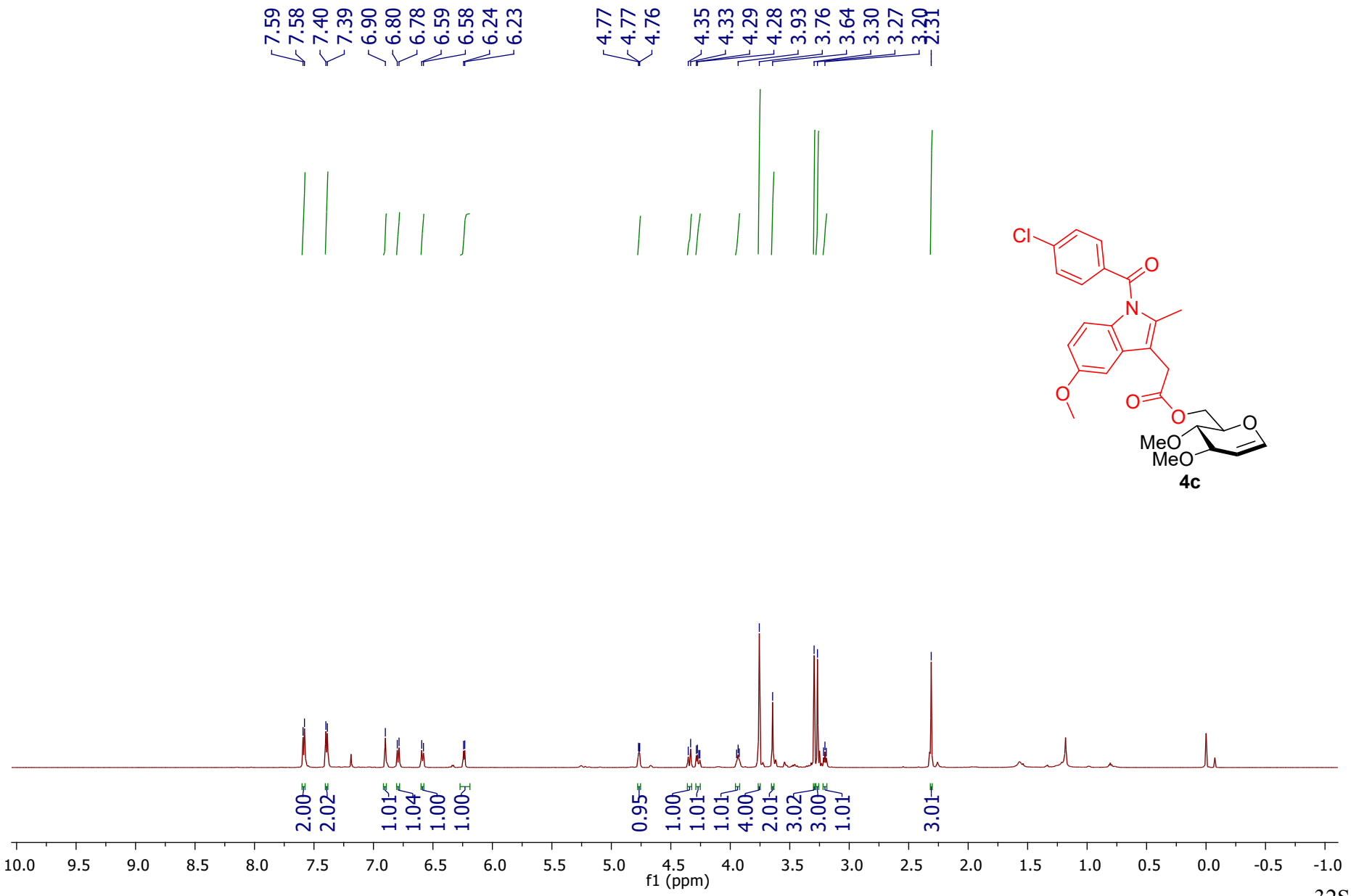
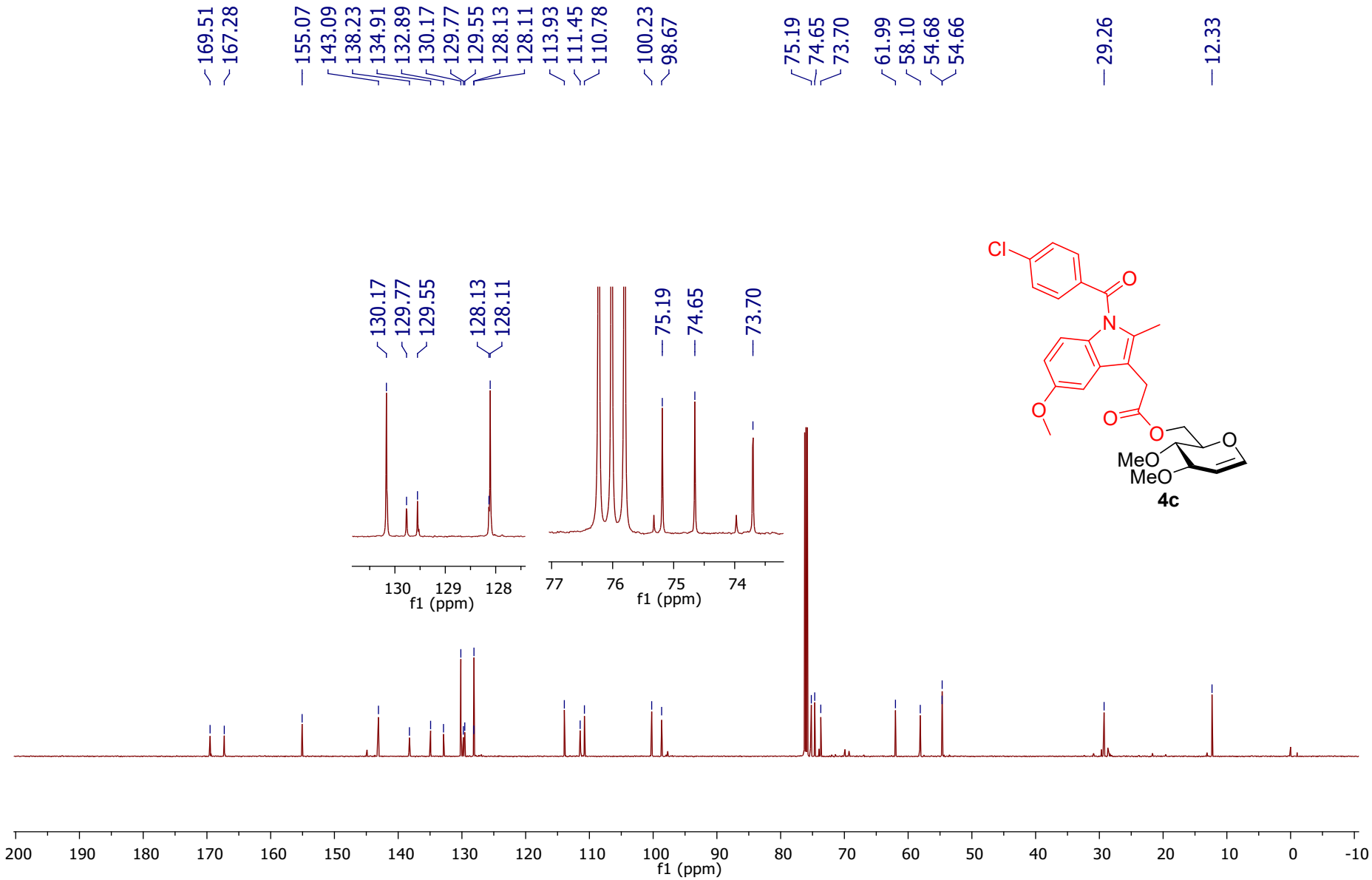
60.86
59.21
55.78
34.83
25.85
25.62
25.28
Cl
Cl
O
O
O
MeO
MeO
O
4b
76.21
75.72
74.59
76
75
74
f1 (ppm)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


7.59
7.58
7.40
7.39
6.90
6.80
6.78
6.59
6.58
6.24
6.23
4.77
4.77
4.76
4.35
4.33
4.29
4.28
3.93
3.76
3.64
3.30
3.27
3.20
2.31
Cl
O
N
O
O
O
O
MeO
MeO
4c
2.00
2.02
1.01
1.04
1.00
1.00
0.95
1.00
1.01
1.01
4.00
2.01
3.02
3.00
1.01
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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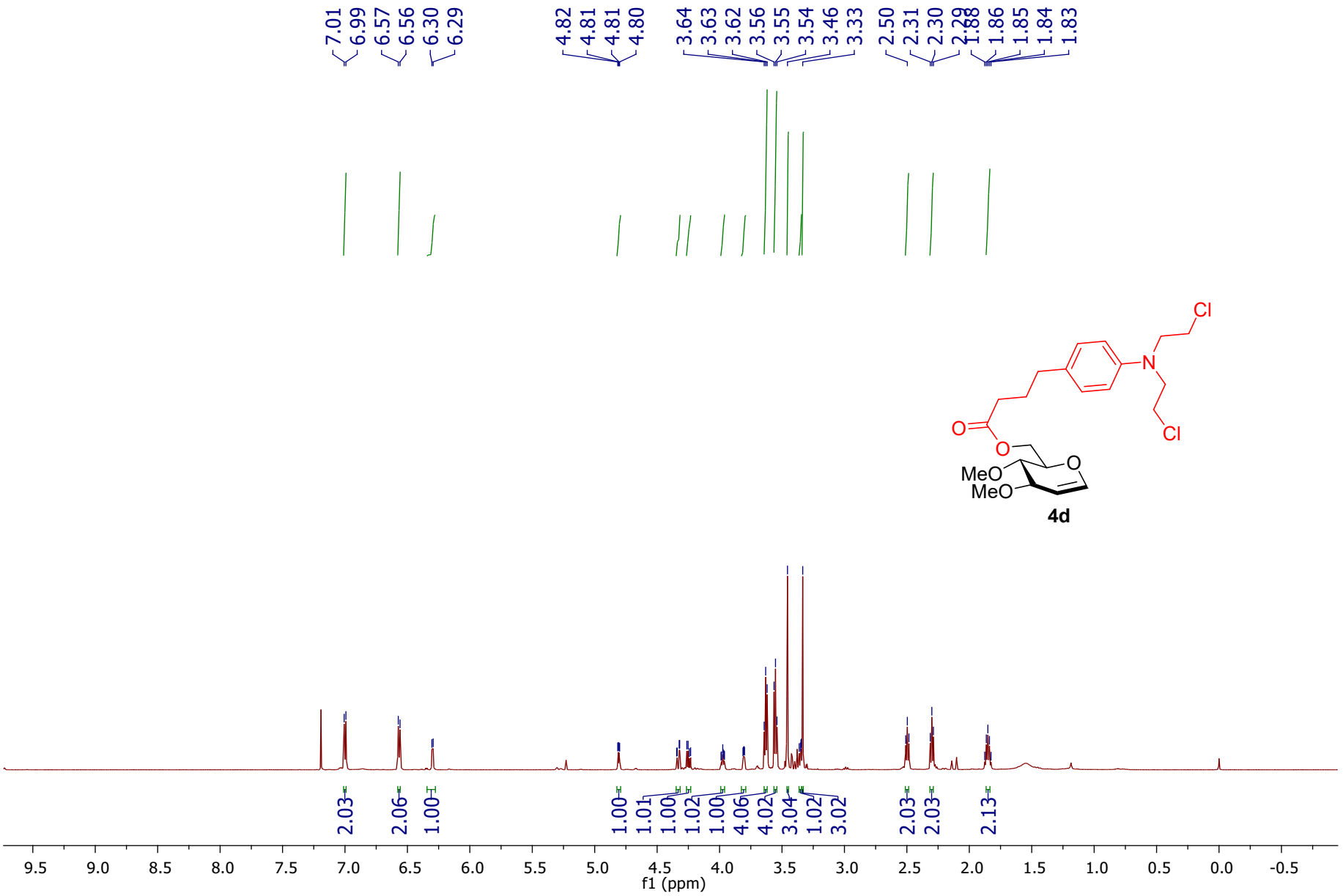
167.28
155.07
143.09
138.23
134.91
132.89
130.17
129.77
129.55
128.13
128.11
113.93
111.45
110.78
100.23
98.67
75.19
74.65
73.70
61.99
58.10
54.68
54.66
29.26
12.33
130.17
129.77
129.55
128.13
128.11
75.19
74.65
73.70
130 129 128
f1 (ppm)
77 76 75 74
f1 (ppm)
Cl
O
N
O
O
O
O
O
MeO
MeO
4c
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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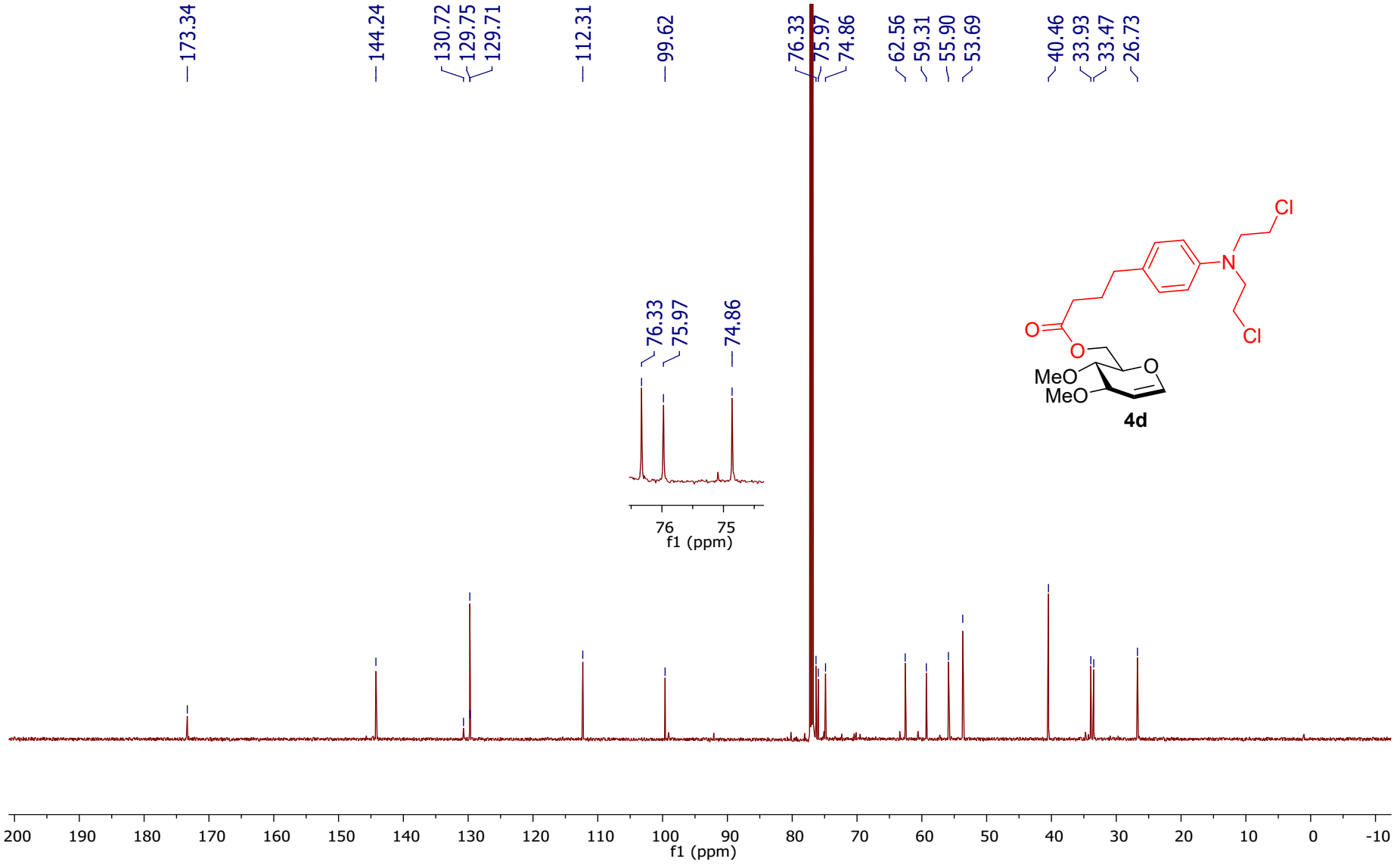
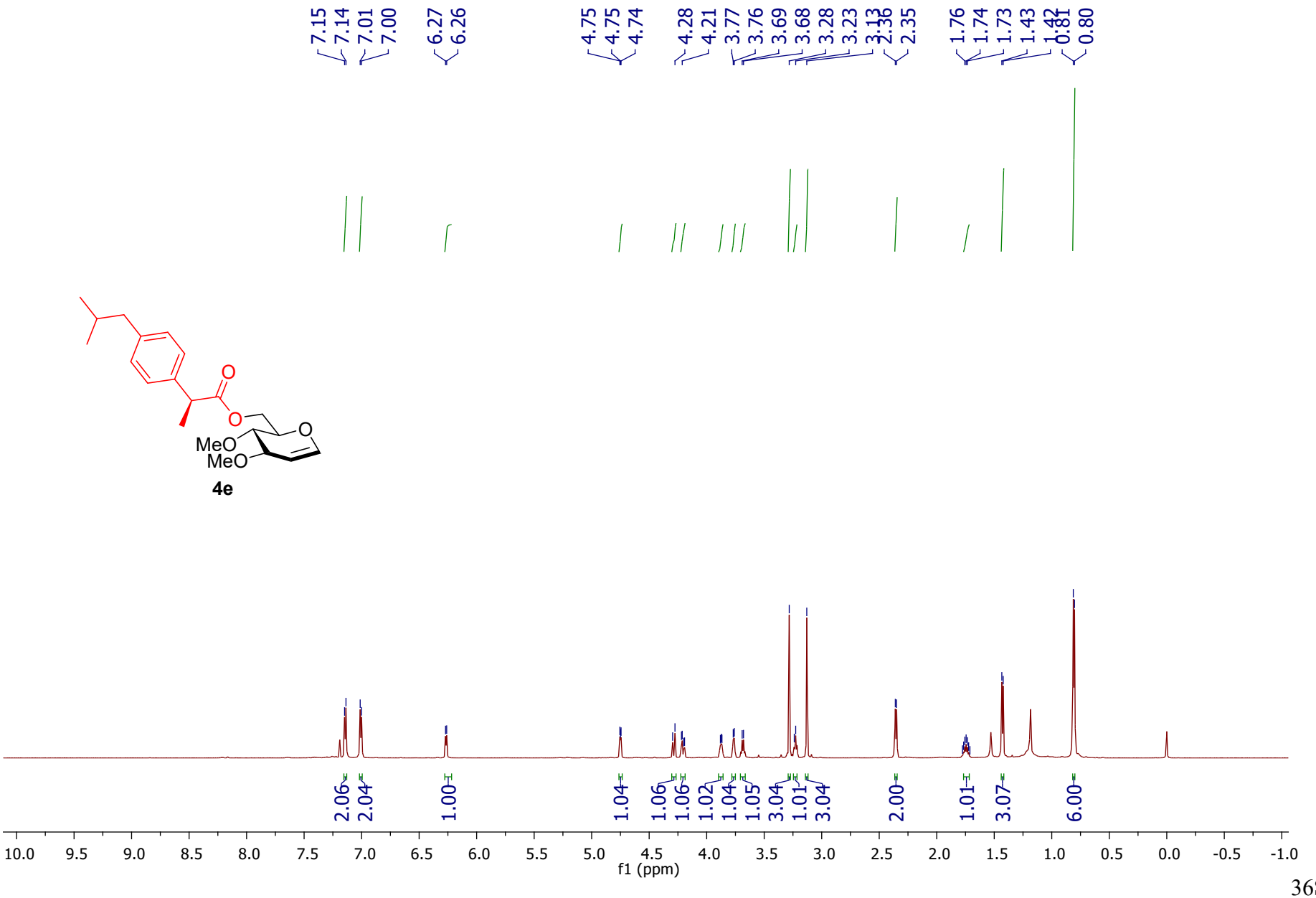
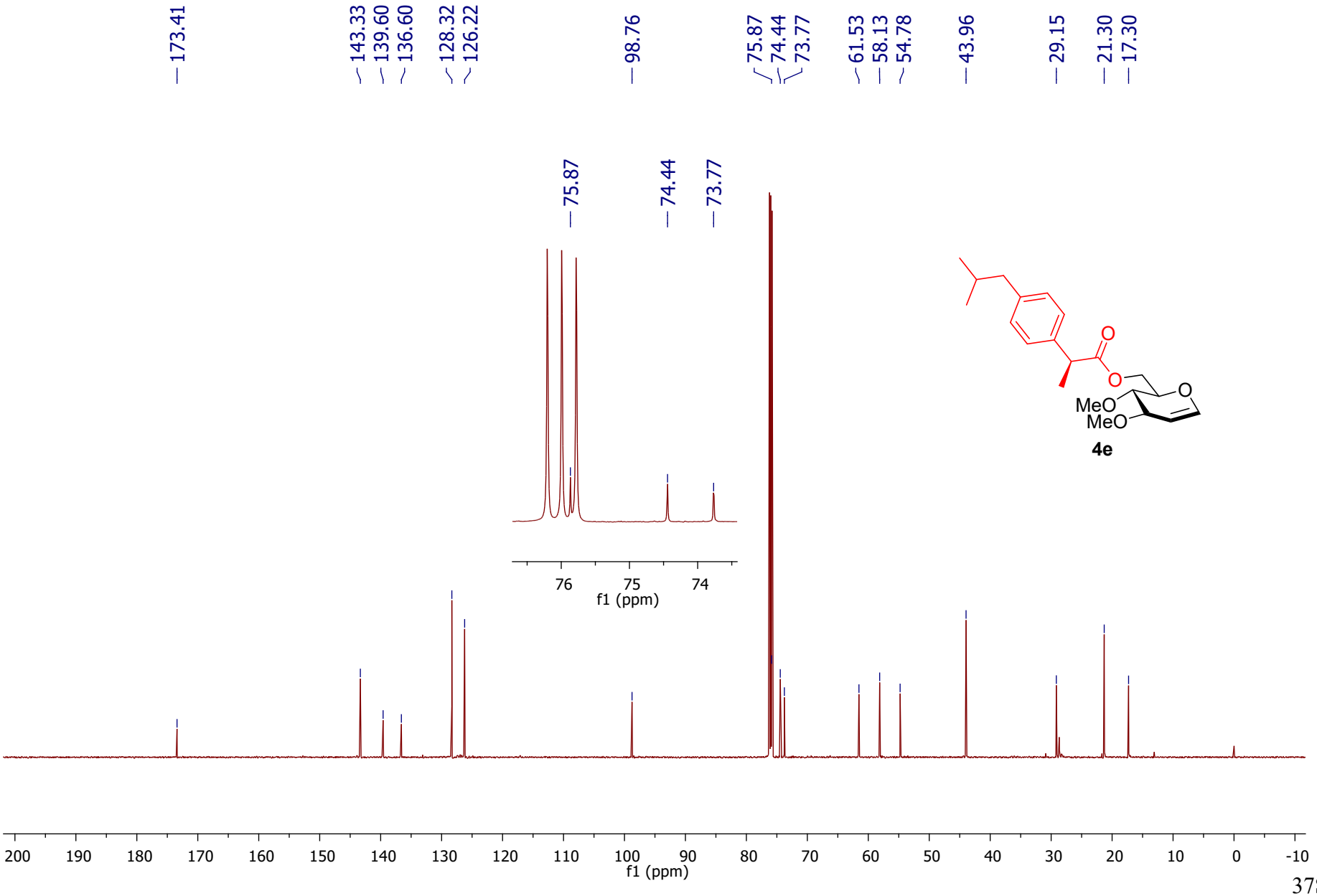
6.99
6.57
6.56
6.30
6.29
4.82
4.81
4.81
4.80
3.64
3.63
3.62
3.56
3.55
3.54
3.46
3.33
2.50
2.31
2.30
2.29
1.88
1.86
1.85
1.84
1.83
Cl
N
Cl
O
O
O
MeO
MeO
4d
2.03
2.06
1.00
1.00
1.01
1.00
1.02
1.00
4.06
4.02
3.04
1.02
3.02
2.03
2.03
2.13
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


173.34
144.24
130.72
129.75
129.71
112.31
99.62
76.33
75.97
74.86
62.56
59.31
55.90
53.69
40.46
33.93
33.47
26.73
f1 (ppm)
Cl
N
Cl
O
O
MeO
MeO
O
4d


7.15
7.14
7.01
7.00
6.27
6.26
4.75
4.75
4.74
4.28
4.21
3.77
3.76
3.69
3.68
3.28
3.23
3.13
2.38
2.35
1.76
1.74
1.73
1.43
1.42
0.81
0.80
O
O
O
MeO
MeO
2.06
2.04
1.00
1.04
1.06
1.06
1.02
1.04
1.05
3.04
1.01
3.04
2.00
1.01
3.07
6.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

4e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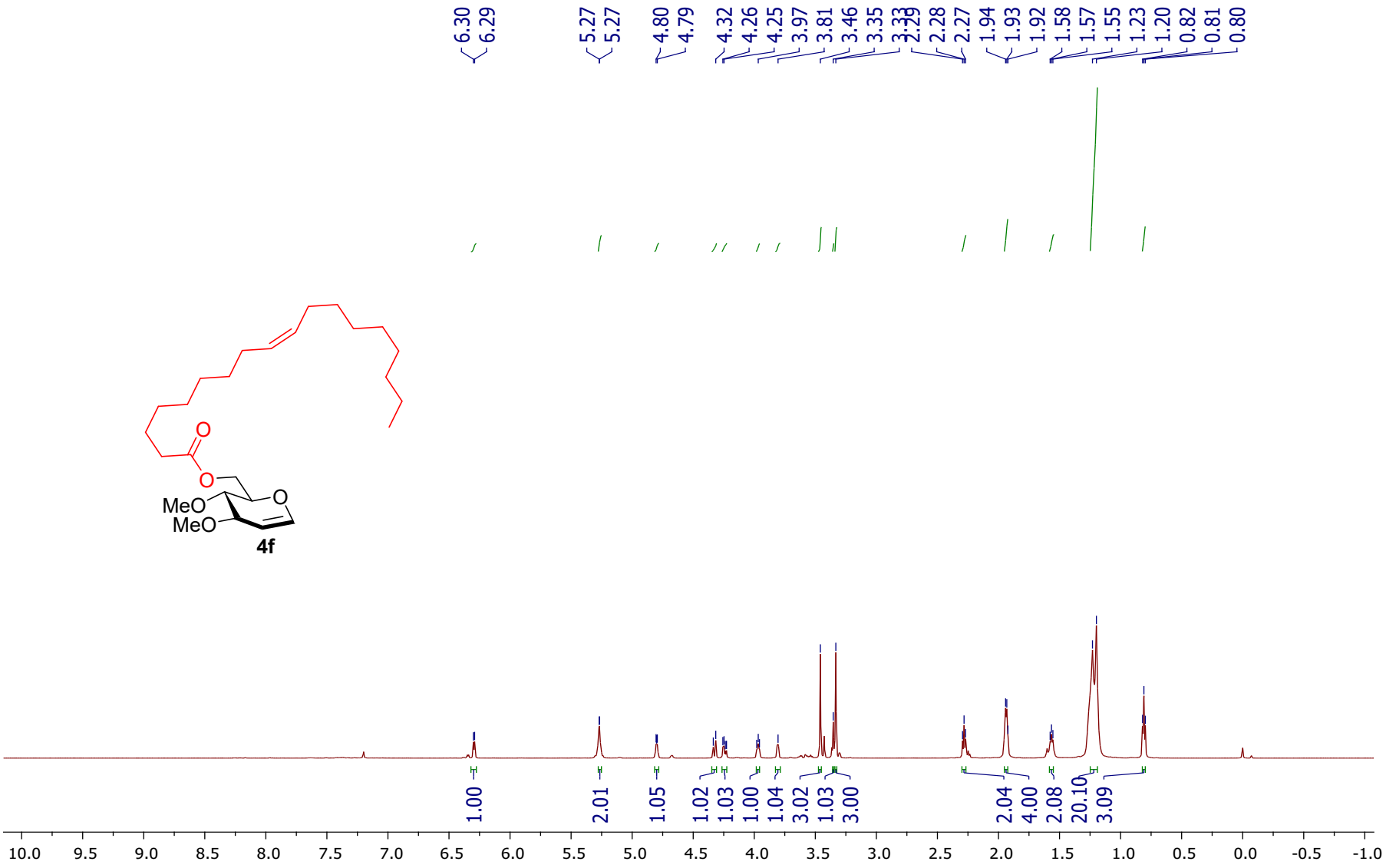
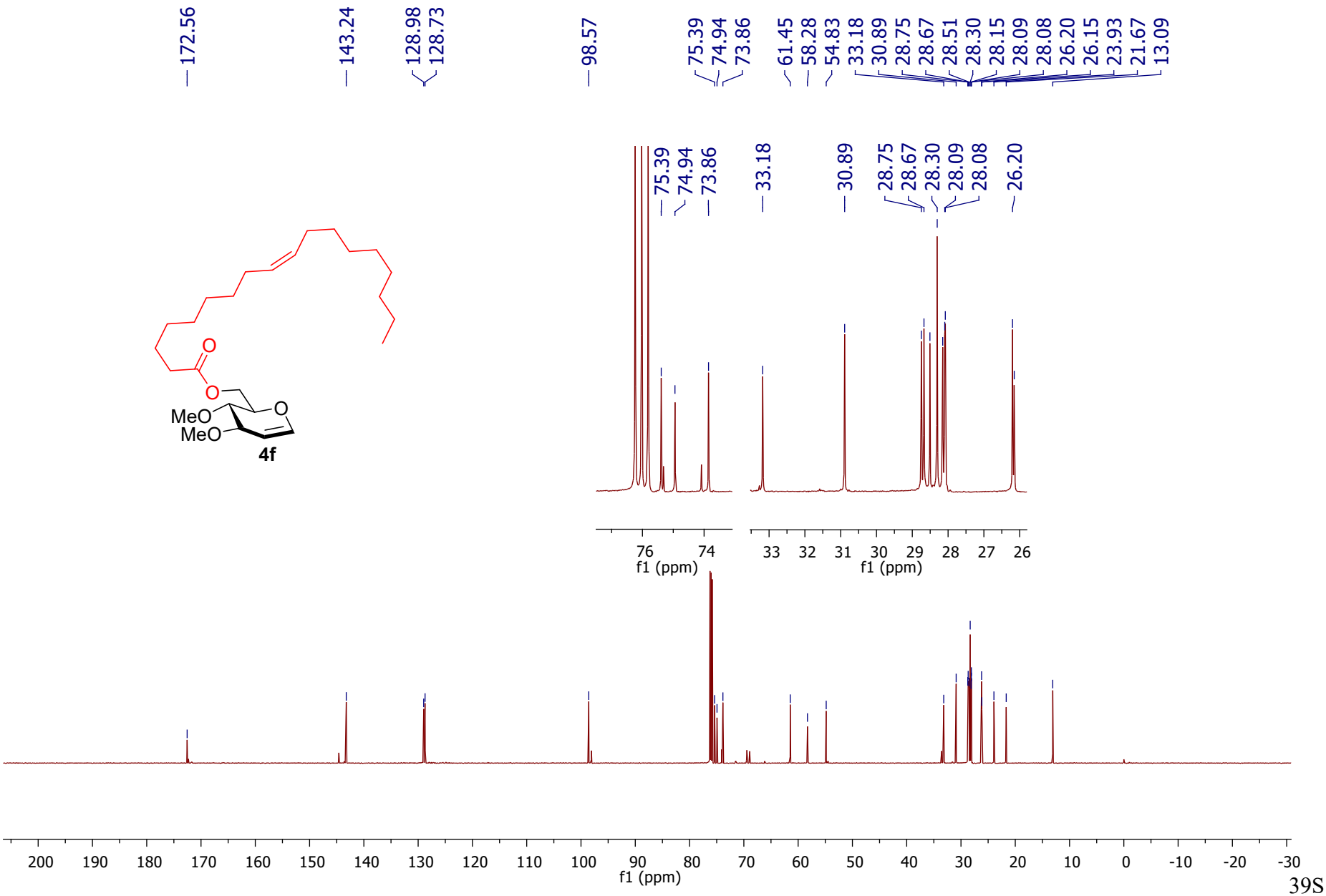
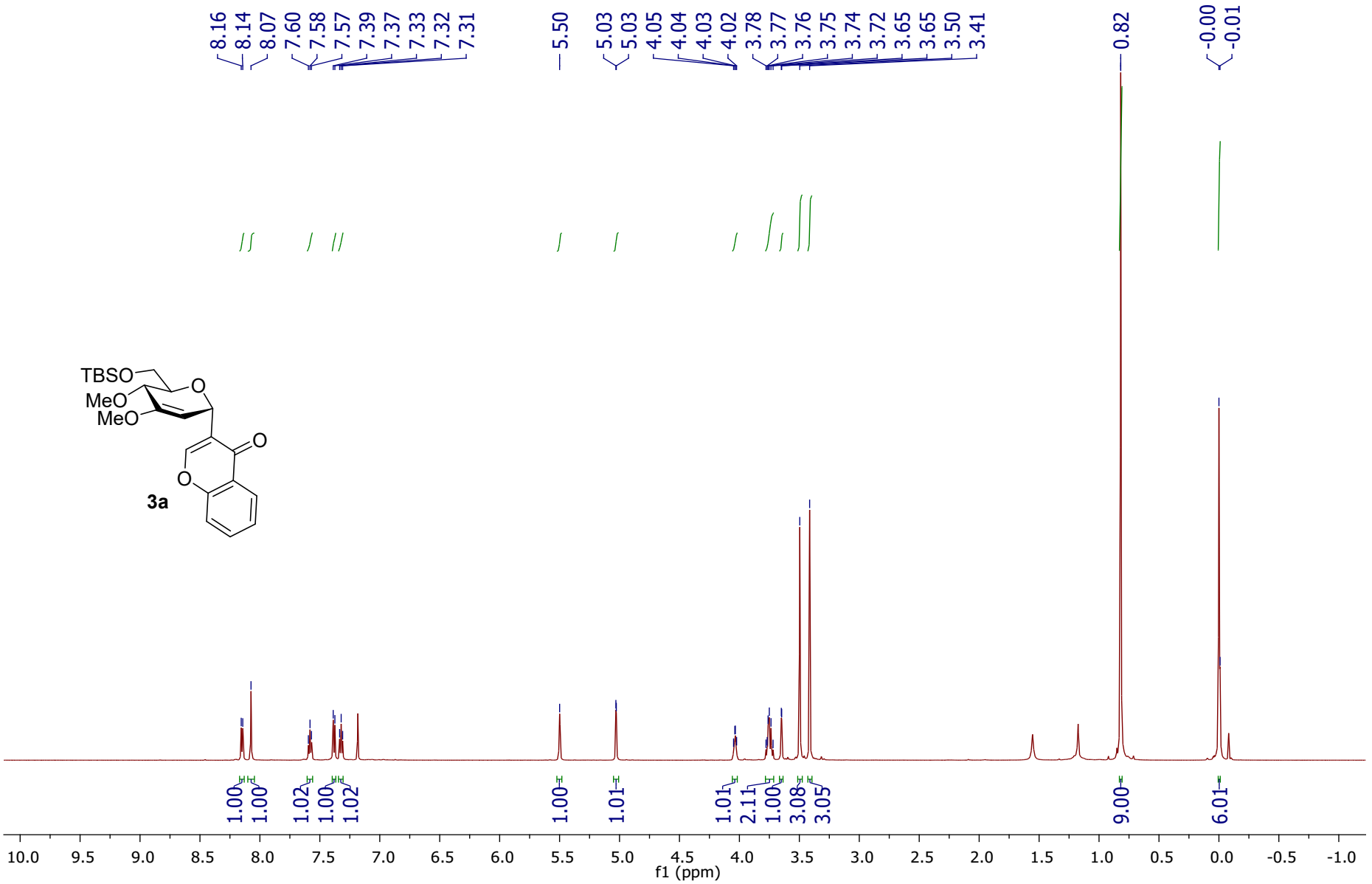
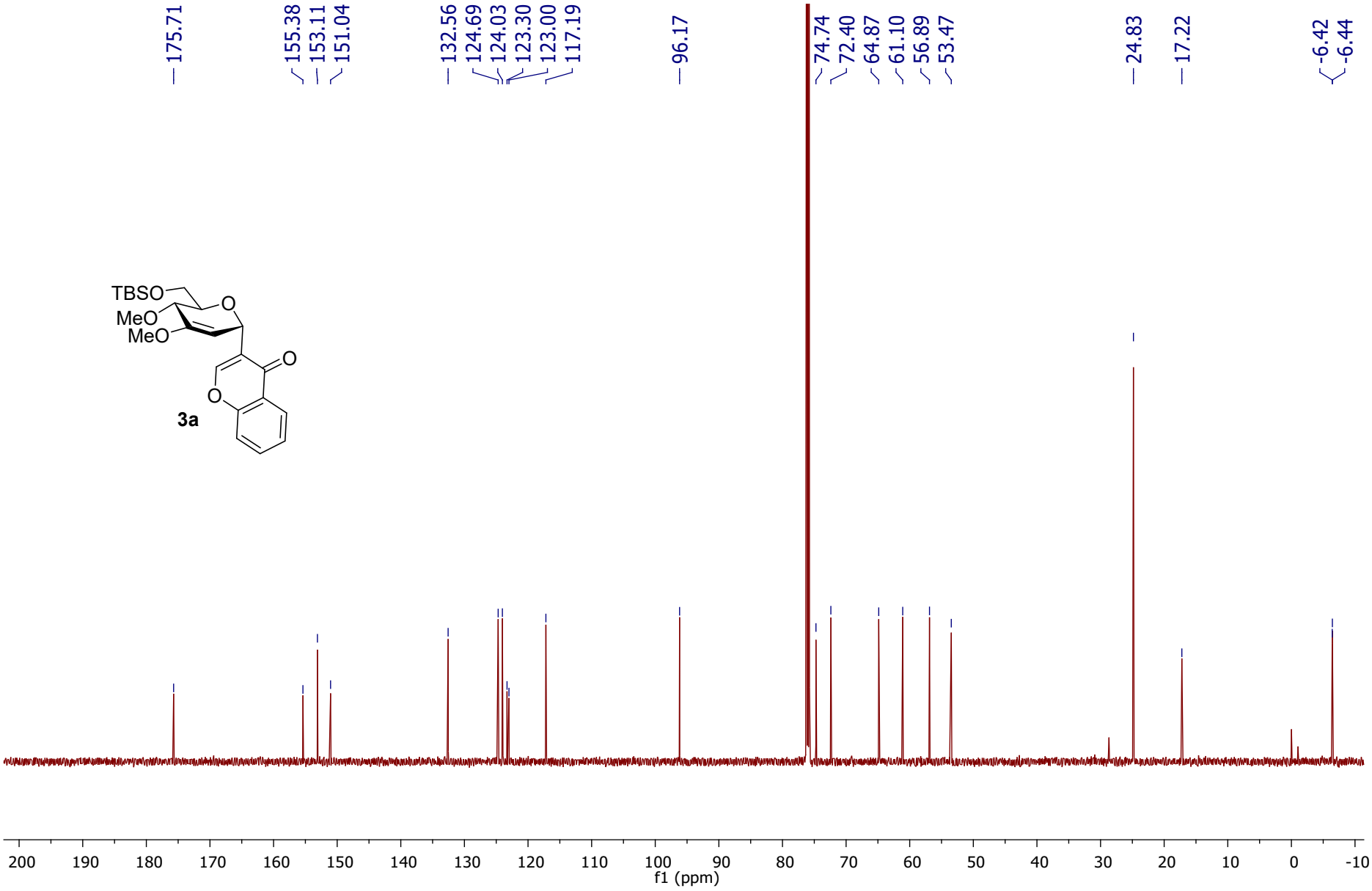
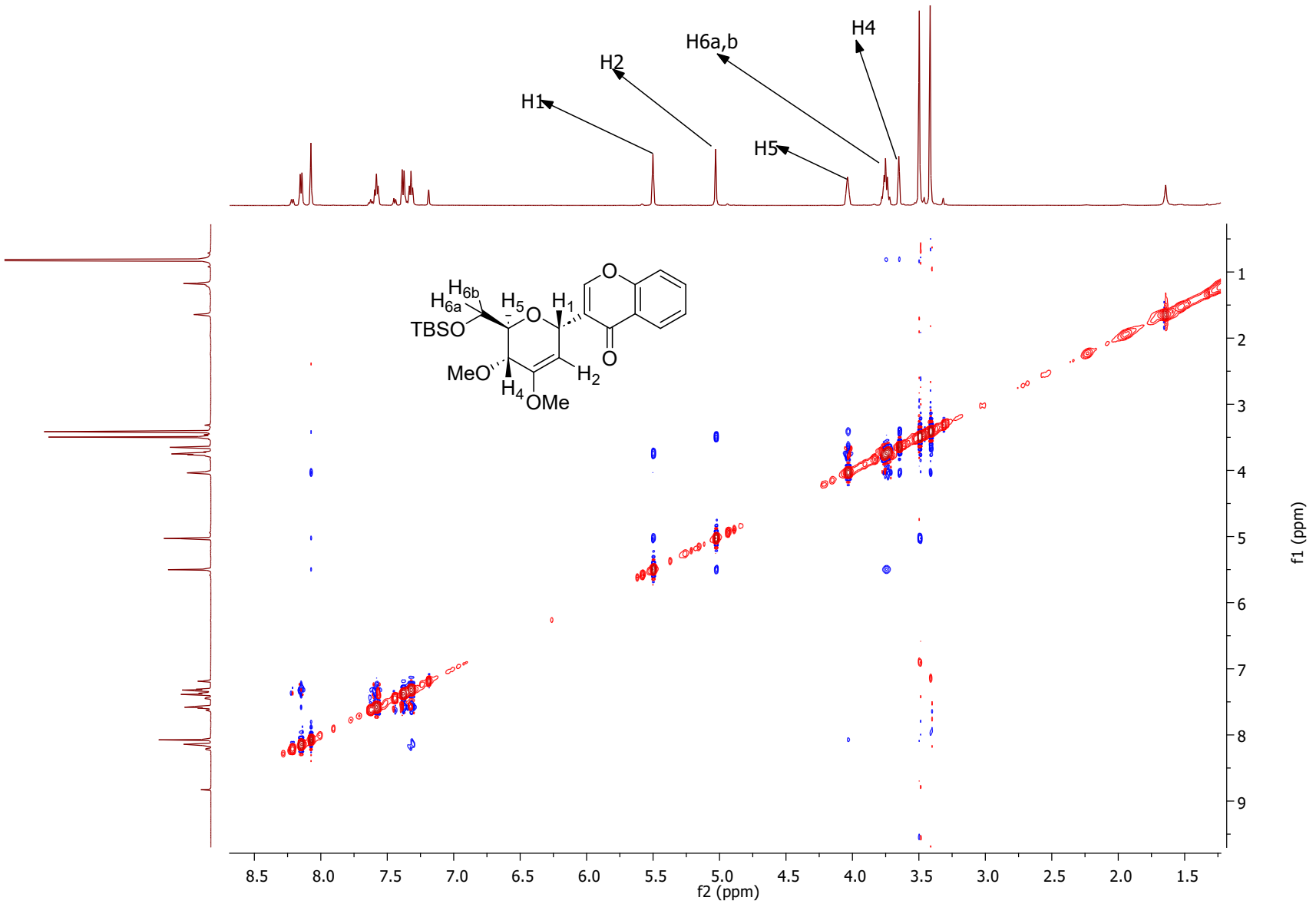
143.33
139.60
136.60
128.32
126.22
98.76
75.87
74.44
73.77
61.53
58.13
54.78
43.96
29.15
21.30
17.30
MeO
MeO
O
O
O
4e
f1 (ppm)
f1 (ppm)

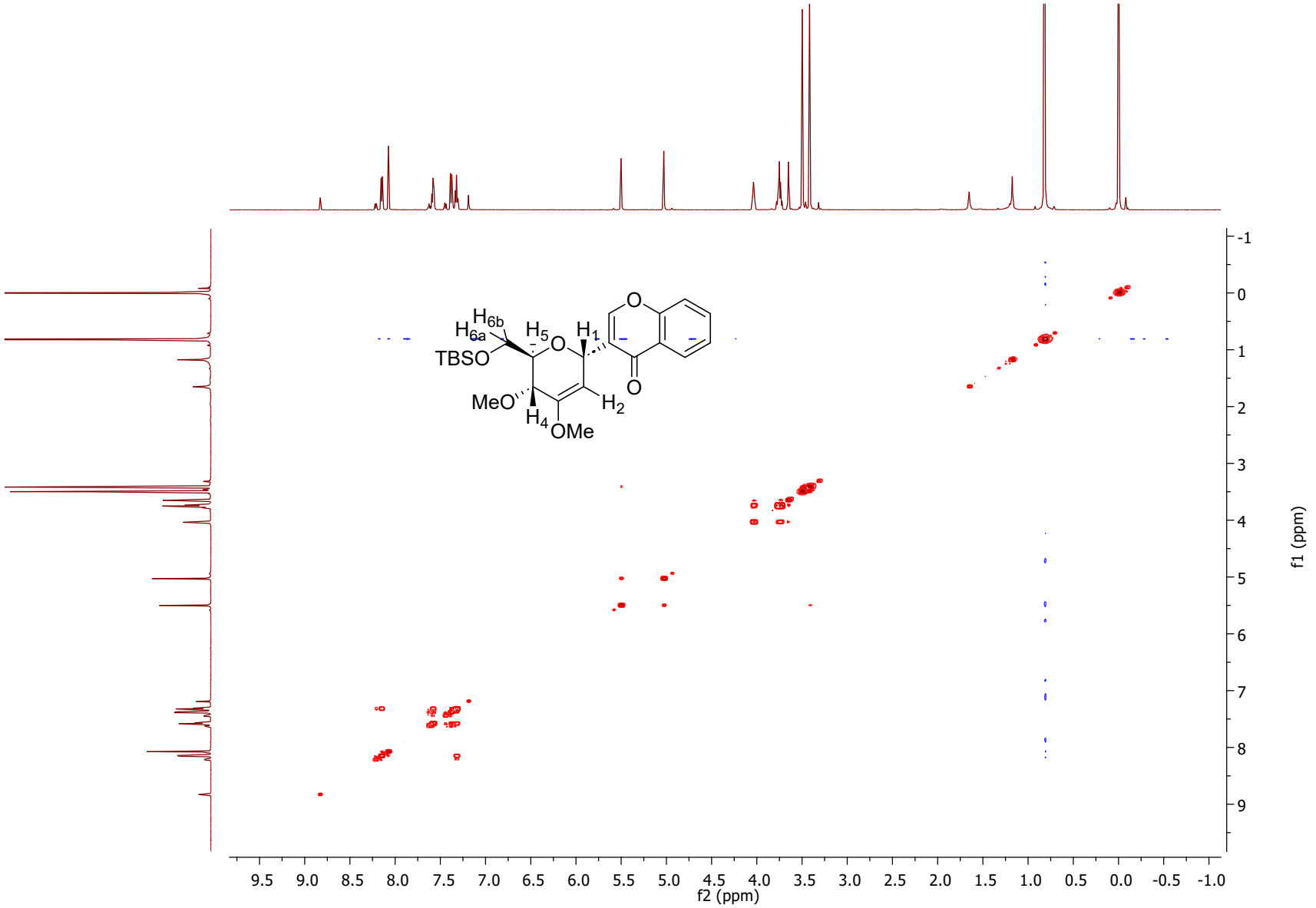

6.30
6.29
5.27
5.27
4.80
4.79
4.32
4.26
4.25
3.97
3.81
3.46
3.35
3.33
2.29
2.28
2.27
1.94
1.93
1.92
1.58
1.57
1.55
1.23
1.20
0.82
0.81
0.80
O
O
MeO
MeO
O
4f
1.00
2.01
1.05
1.02
1.03
1.00
1.04
3.02
1.03
3.00
2.04
4.00
2.08
20.10
3.09
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0

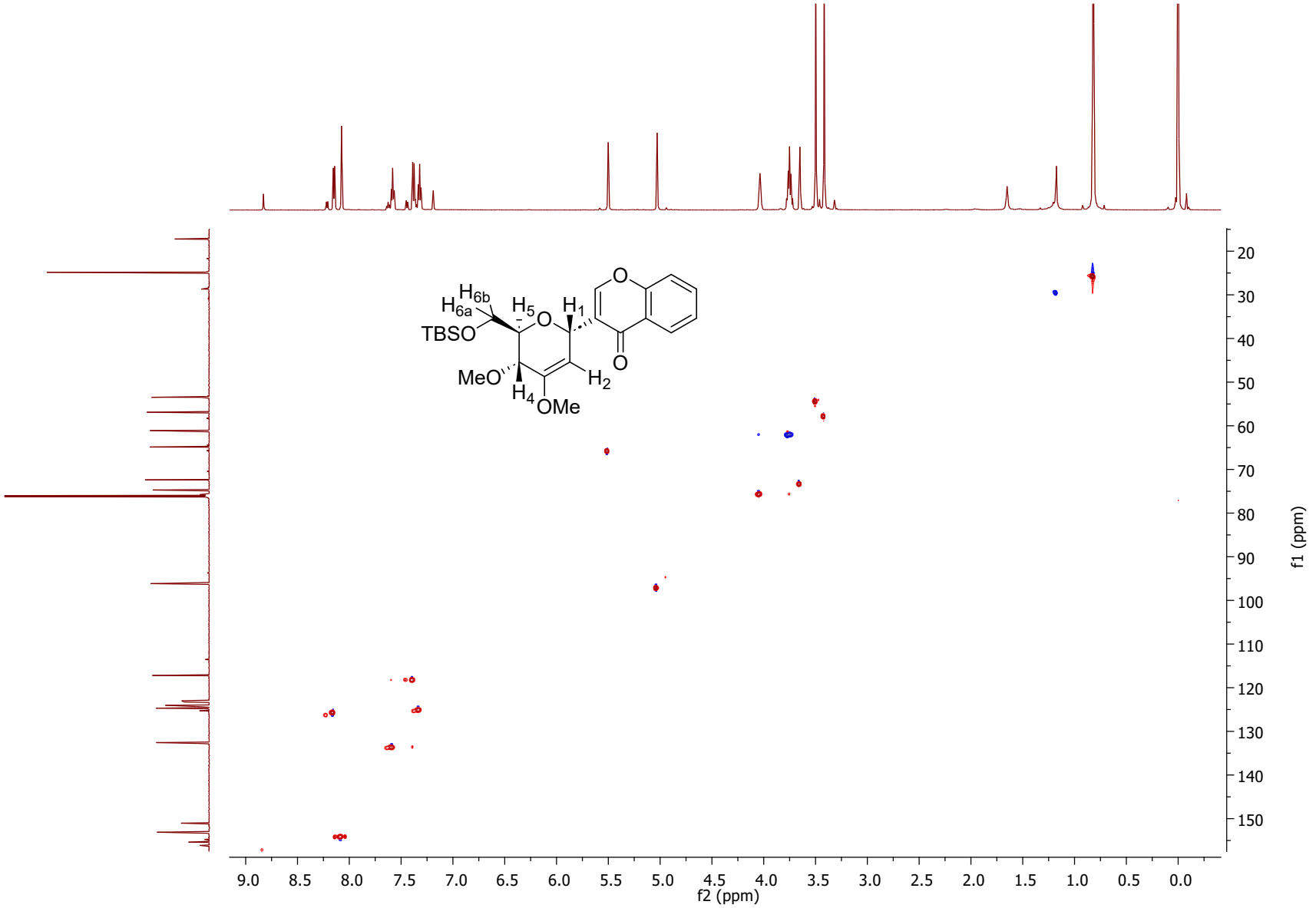

172.56
143.24
128.98
128.73
98.57
75.39
74.94
73.86
61.45
58.28
54.83
33.18
30.89
28.75
28.67
28.51
28.30
28.15
28.09
28.08
26.20
26.15
23.93
21.67
13.09
MeO
MeO
O
O
O
4f
f1 (ppm)
f1 (ppm)
f1 (ppm)

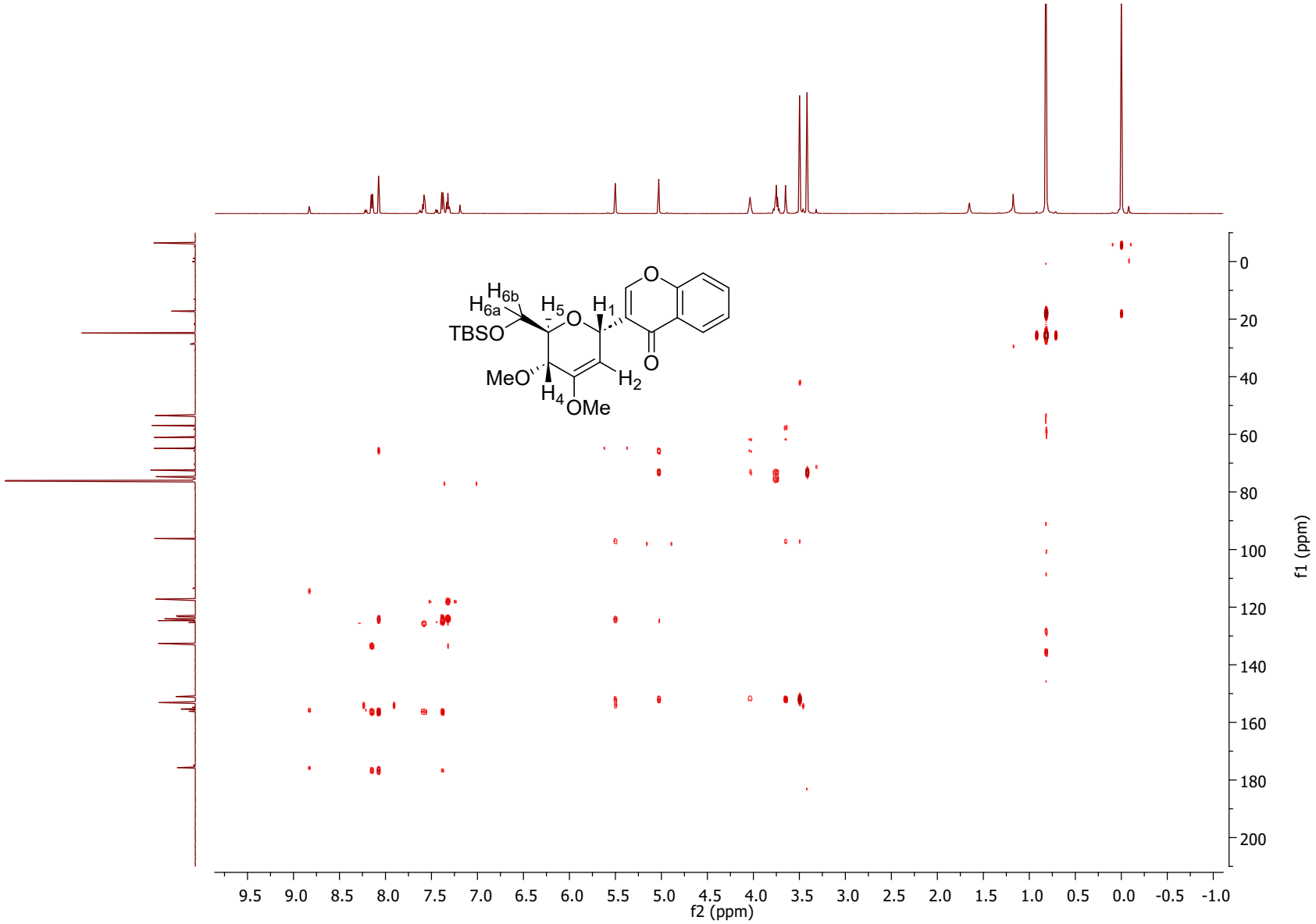

8.16
8.14
8.07
7.60
7.58
7.57
7.39
7.37
7.33
7.32
7.31
5.50
5.03
5.03
4.05
4.04
4.03
4.02
3.78
3.77
3.76
3.75
3.74
3.72
3.65
3.65
3.50
3.41
0.82
-0.00
-0.01
TBSO
MeO
MeO
O
O
O
3a
1.00
1.00
1.02
1.00
1.02
1.00
1.01
1.01
2.11
1.00
3.08
3.05
9.00
6.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

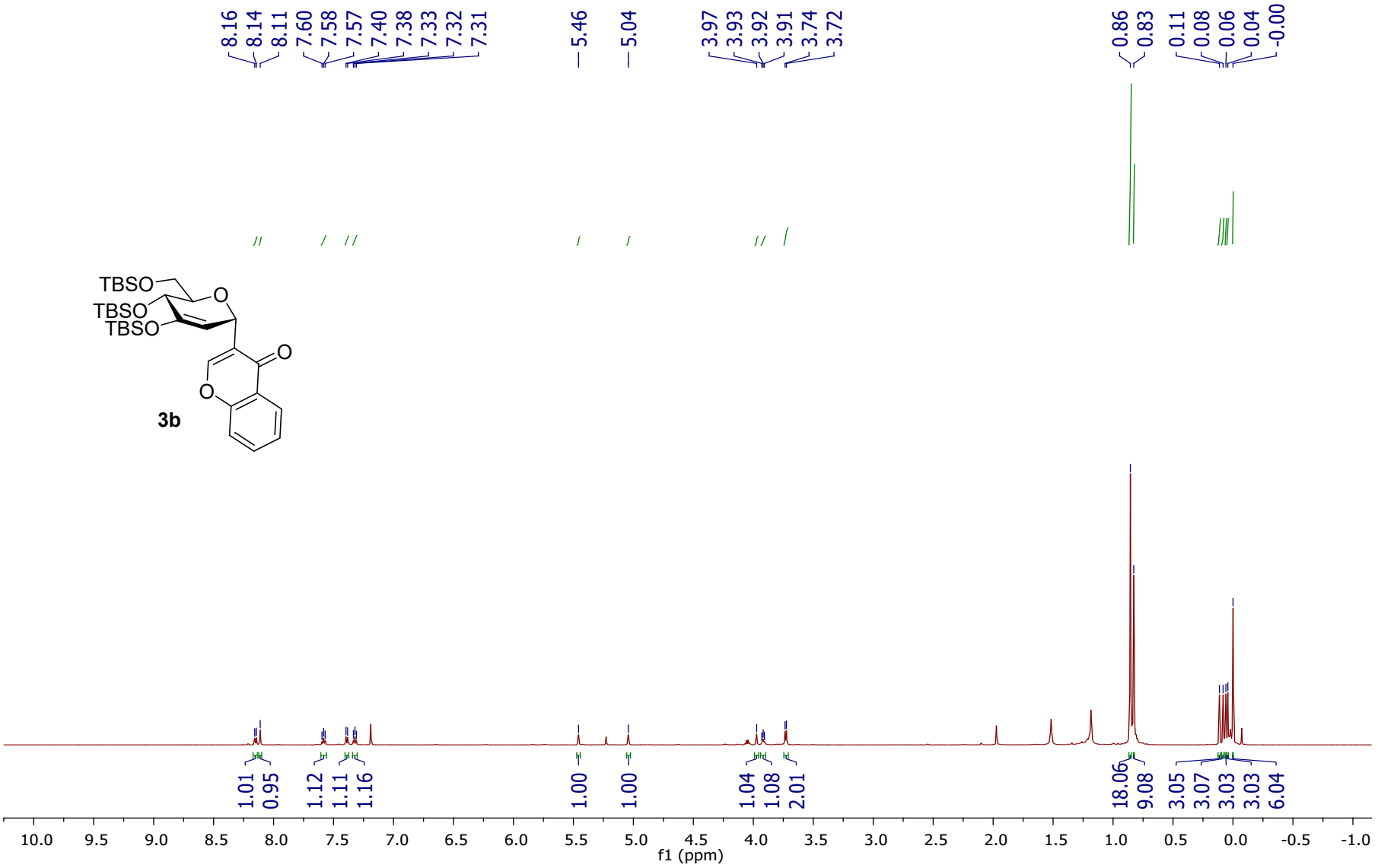

175.71
155.38
153.11
151.04
132.56
124.69
124.03
123.30
123.00
117.19
96.17
74.74
72.40
64.87
61.10
56.89
53.47
24.83
17.22
-6.42
-6.44
TBSO
MeO
MeO
O
O
O
3a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

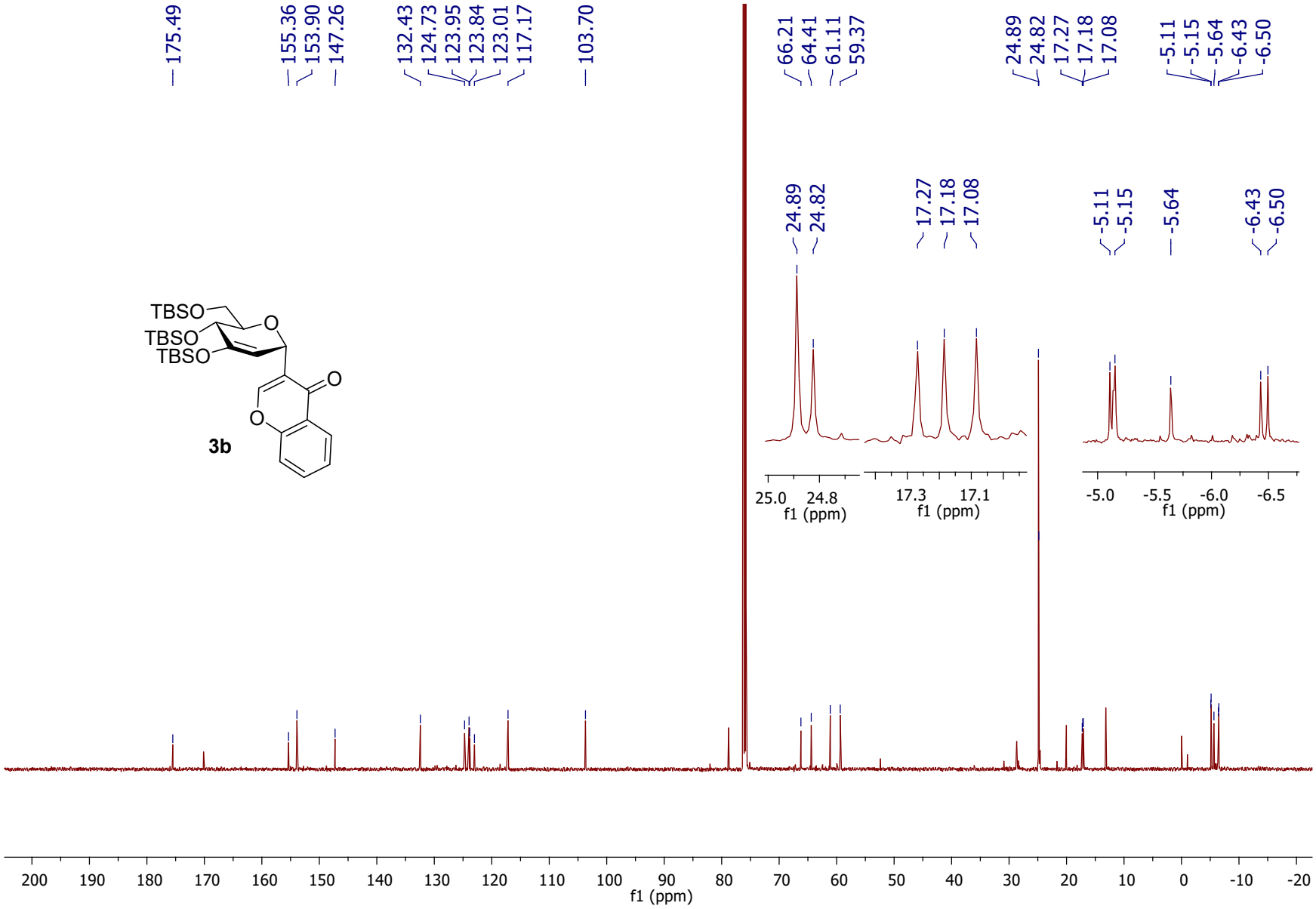

H1
H2
H6a,b
H4
H5
H6b
H6a
H5
H1
TBSO
MeO
H4
OMe
H2
O
O
O
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f2 (ppm)
1
2
3
4
5
6
7
8
9
f1 (ppm)


H6b
H6a
H5
H1
TBSO
O
O
MeO
H4
OMe
H2
f2 (ppm)
f1 (ppm)


H6b
H6a
H5
O
H1
TBSO
O
O
MeO
H2
H4
OMe
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f2 (ppm)
20
30
40
50
60
70
80
90
100
110
120
130
140
150
f1 (ppm)


H6b
H6a
H5
H1
TBSO
O
O
MeO
H2
H4
OMe
f2 (ppm)
f1 (ppm)


8.16
8.14
8.11
7.60
7.58
7.57
7.40
7.38
7.33
7.32
7.31
5.46
5.04
3.97
3.93
3.92
3.91
3.74
3.72
0.86
0.83
0.11
0.08
0.06
0.04
-0.00
TBSO
TBSO
TBSO
O
O
O
3b
1.01
0.95
1.12
1.11
1.16
1.00
1.00
1.04
1.08
2.01
18.06
9.08
3.05
3.07
3.03
3.03
6.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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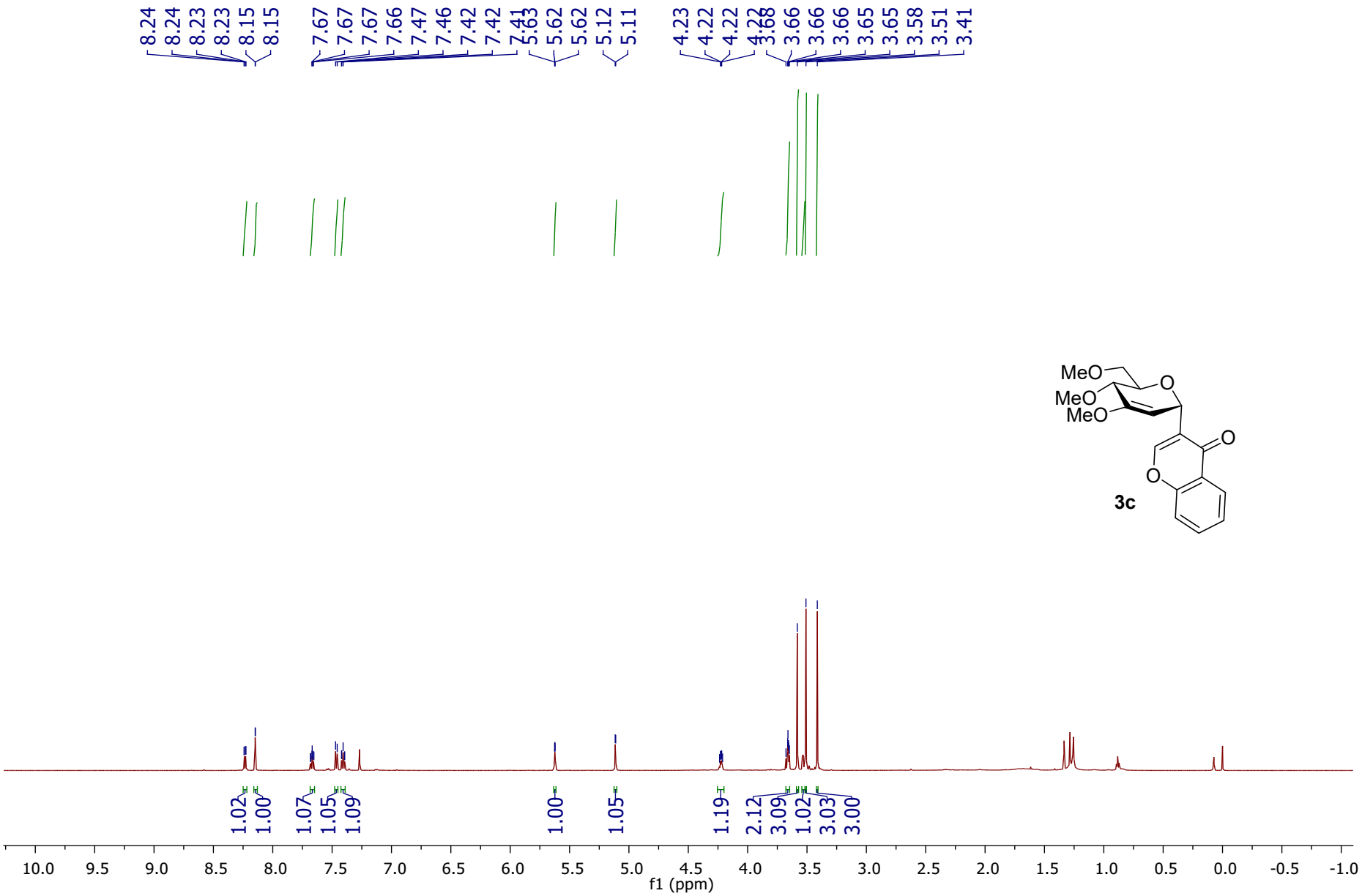
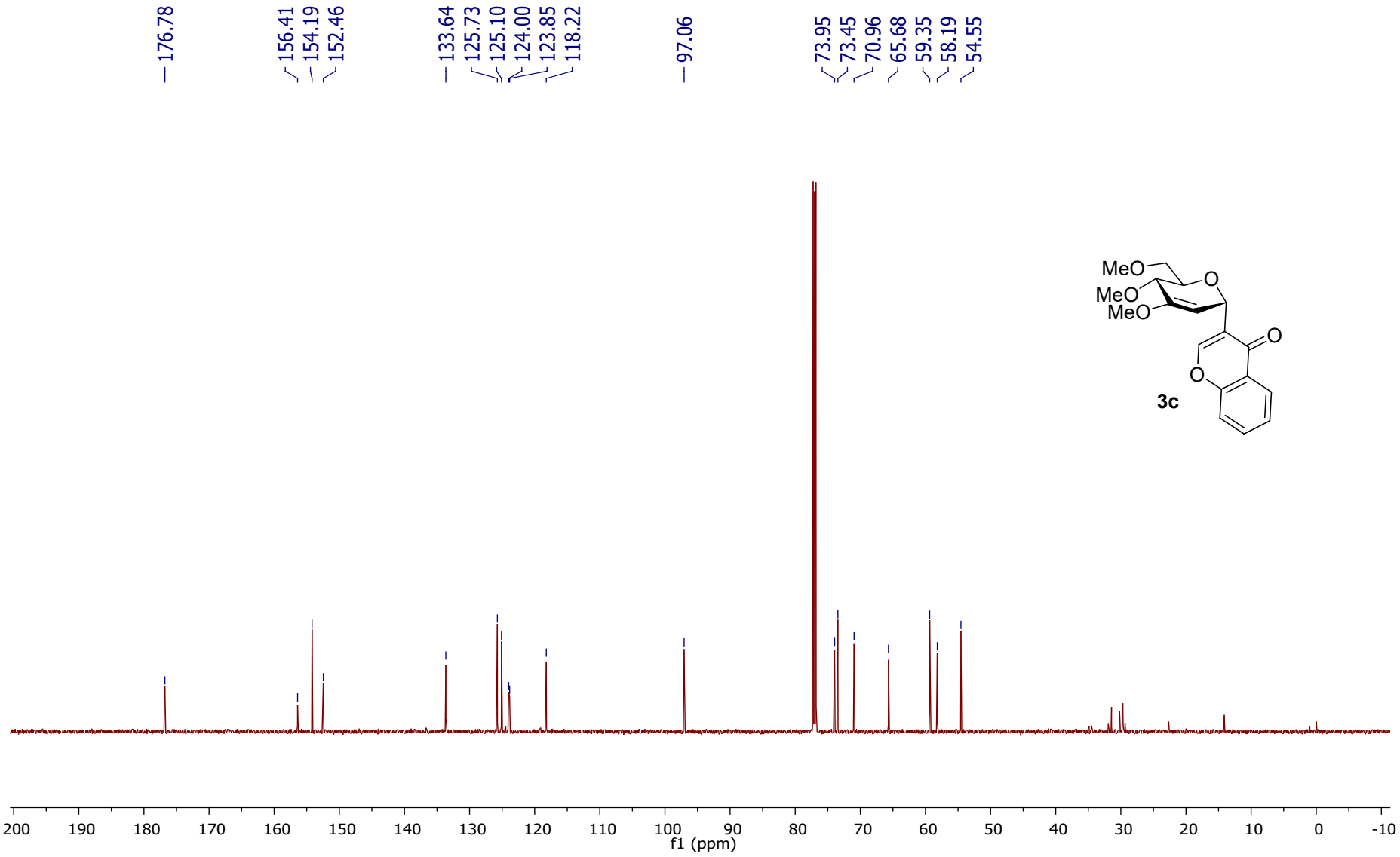
155.36
153.90
147.26
132.43
124.73
123.95
123.84
123.01
117.17
103.70
66.21
64.41
61.11
59.37
24.89
24.82
17.27
17.18
17.08
-5.11
-5.15
-5.64
-6.43
-6.50
TBSO
TBSO
TBSO
O
O
O
3b
f1 (ppm)


8.24
8.24
8.23
8.23
8.15
8.15
7.67
7.67
7.67
7.66
7.47
7.46
7.42
7.42
7.41
5.63
5.62
5.62
5.12
5.11
4.23
4.22
4.22
4.22
3.68
3.66
3.66
3.66
3.65
3.65
3.58
3.51
3.41
MeO
MeO
MeO
O
O
O
3c
1.02
1.00
1.07
1.05
1.09
1.00
1.05
1.19
2.12
3.09
1.02
3.03
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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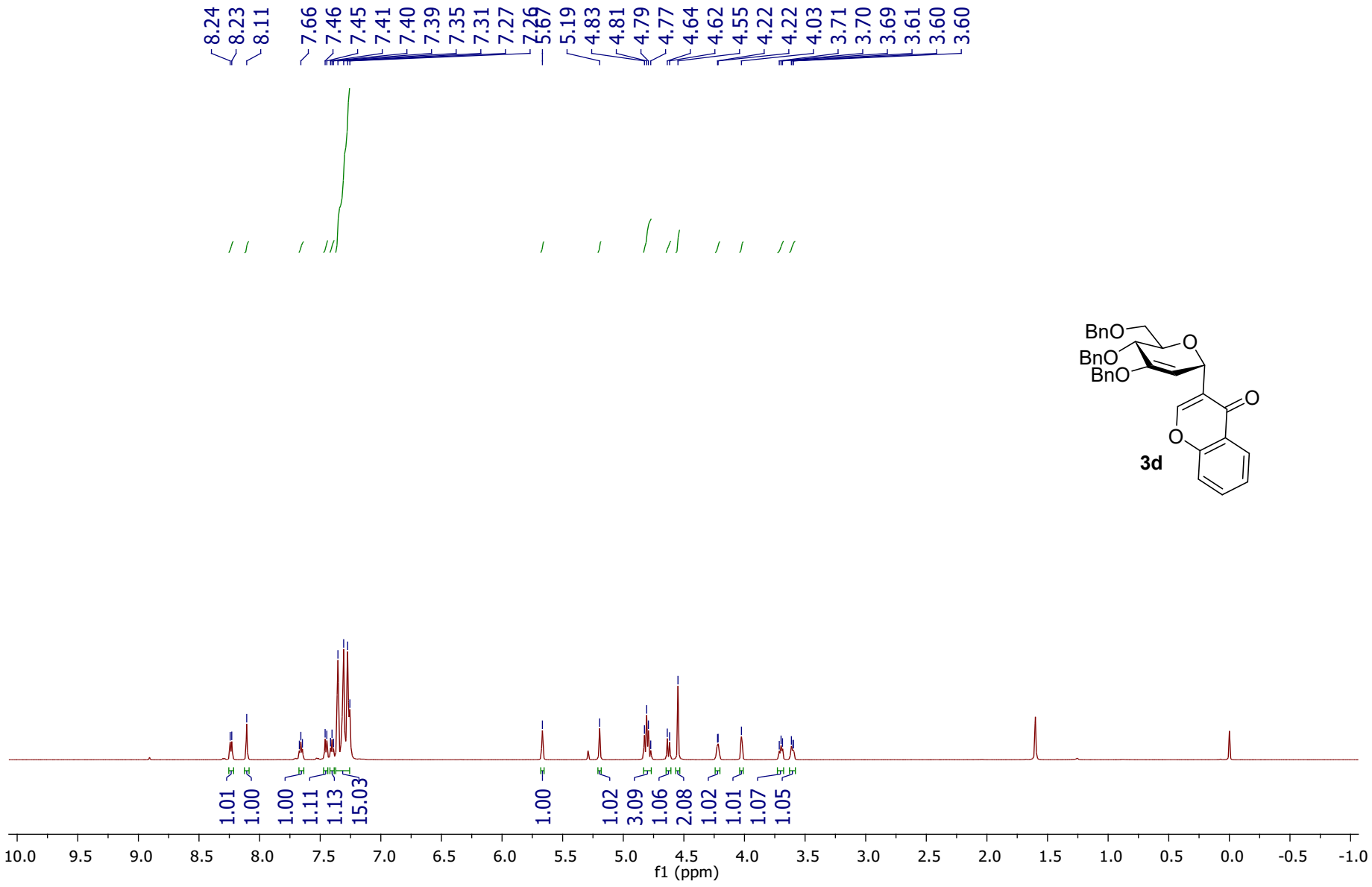
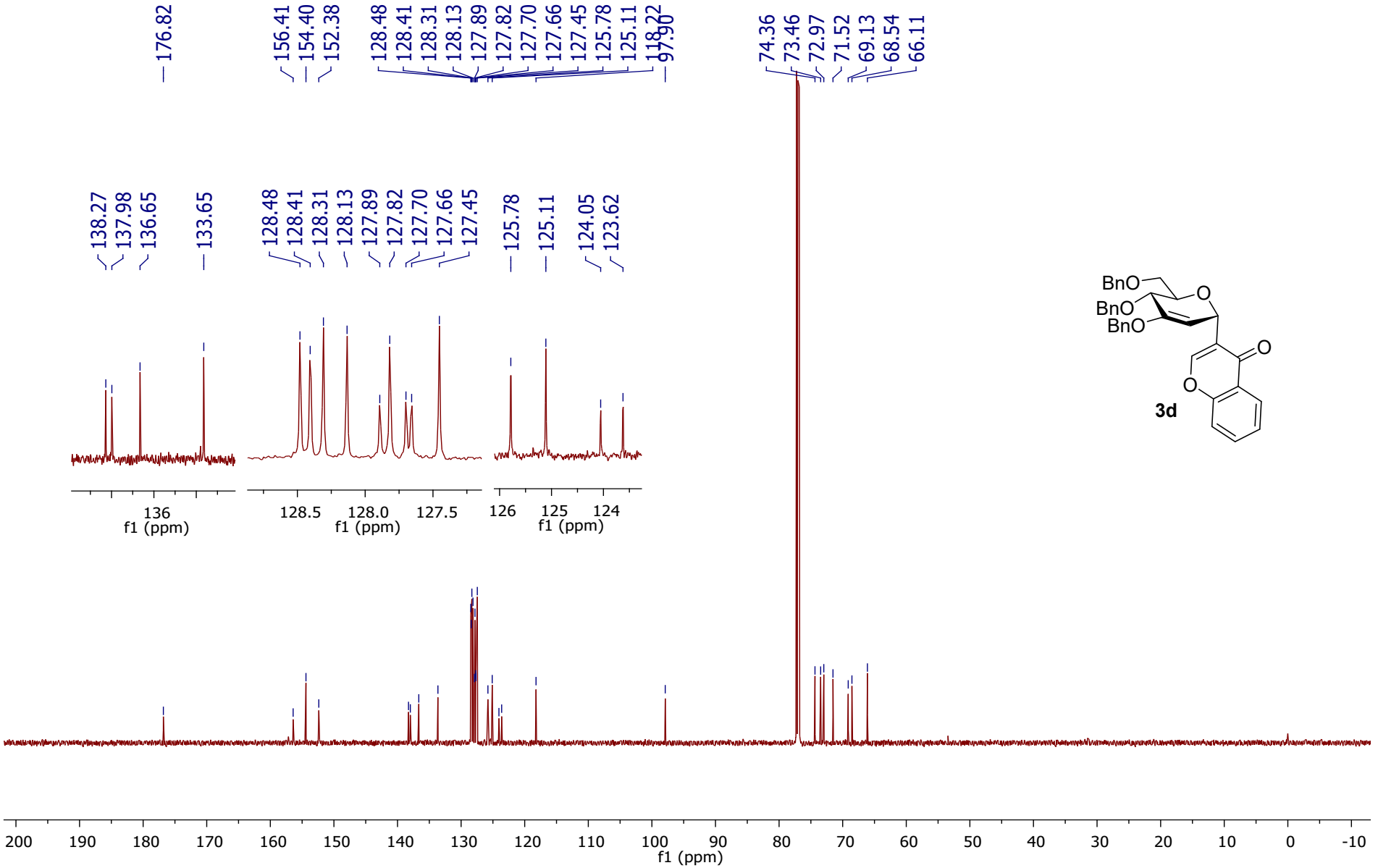
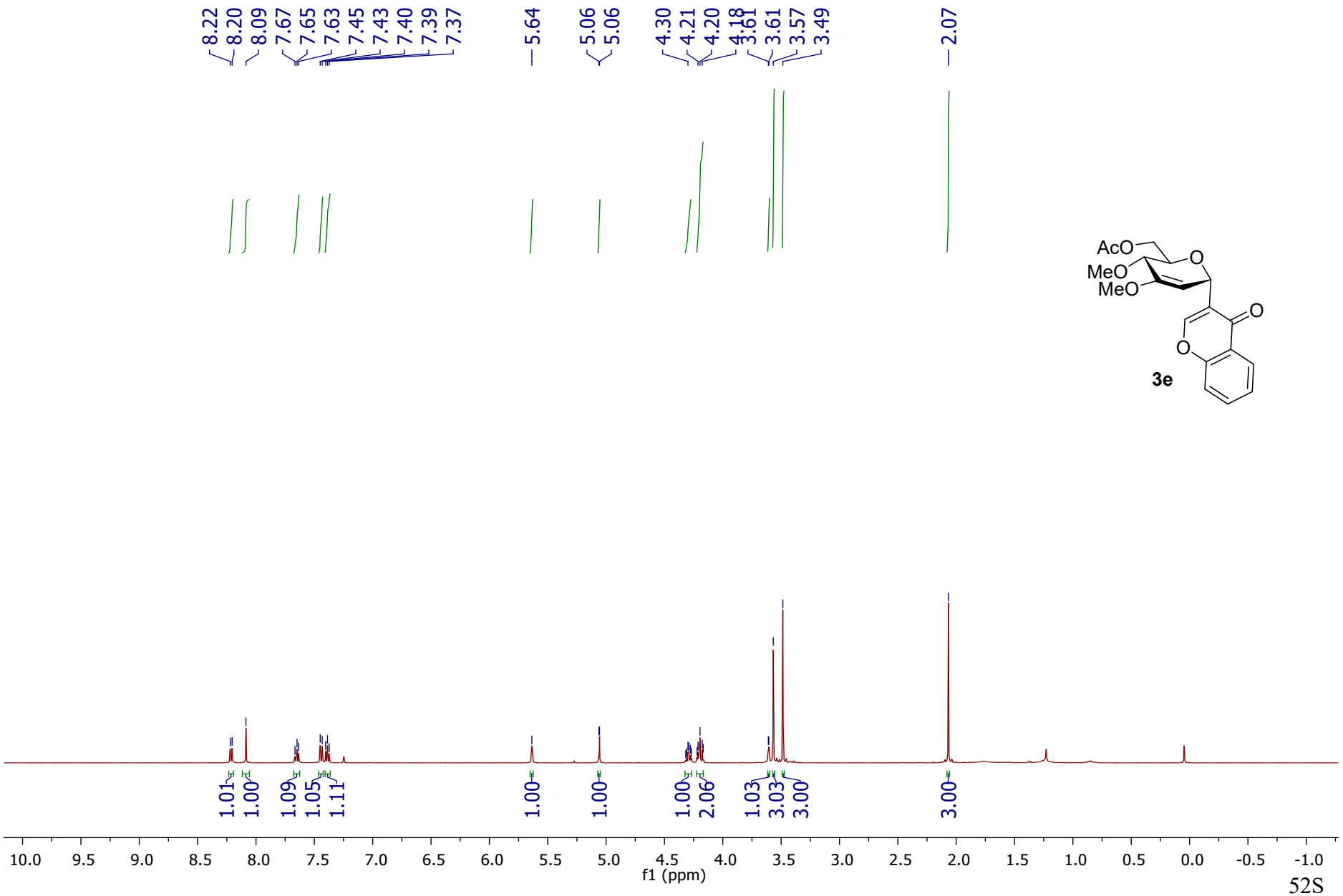
154.19
152.46
133.64
125.73
125.10
124.00
123.85
118.22
97.06
73.95
73.45
70.96
65.68
59.35
58.19
54.55
MeO
MeO
MeO
O
O
O
3c
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.24
8.23
8.11
7.66
7.46
7.45
7.41
7.40
7.39
7.35
7.31
7.27
7.26
5.67
5.19
4.83
4.81
4.79
4.77
4.64
4.62
4.55
4.22
4.22
4.03
3.71
3.70
3.69
3.61
3.60
3.60
BnO
BnO
BnO
O
O
O
3d
1.01
1.00
1.00
1.11
1.13
15.03
1.00
1.02
3.09
1.06
2.08
1.02
1.01
1.07
1.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


176.82
156.41
154.40
152.38
128.48
128.41
128.31
128.13
127.89
127.82
127.70
127.66
127.45
125.78
125.11
118.22
97.90
74.36
73.46
72.97
71.52
69.13
68.54
66.11
138.27
137.98
136.65
133.65
128.48
128.41
128.31
128.13
127.89
127.82
127.70
127.66
127.45
125.78
125.11
124.05
123.62
136
f1 (ppm)
128.5
128.0
127.5
f1 (ppm)
126
125
124
f1 (ppm)
BnO
BnO
BnO
O
O
O
3d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.22
8.20
8.09
7.67
7.65
7.63
7.45
7.43
7.40
7.39
7.37
5.64
5.06
5.06
4.30
4.21
4.20
4.18
3.61
3.61
3.57
3.49
2.07
AcO
MeO
MeO
O
O
O
3e
1.01
1.00
1.09
1.05
1.11
1.00
1.00
1.00
2.06
1.03
3.03
3.00
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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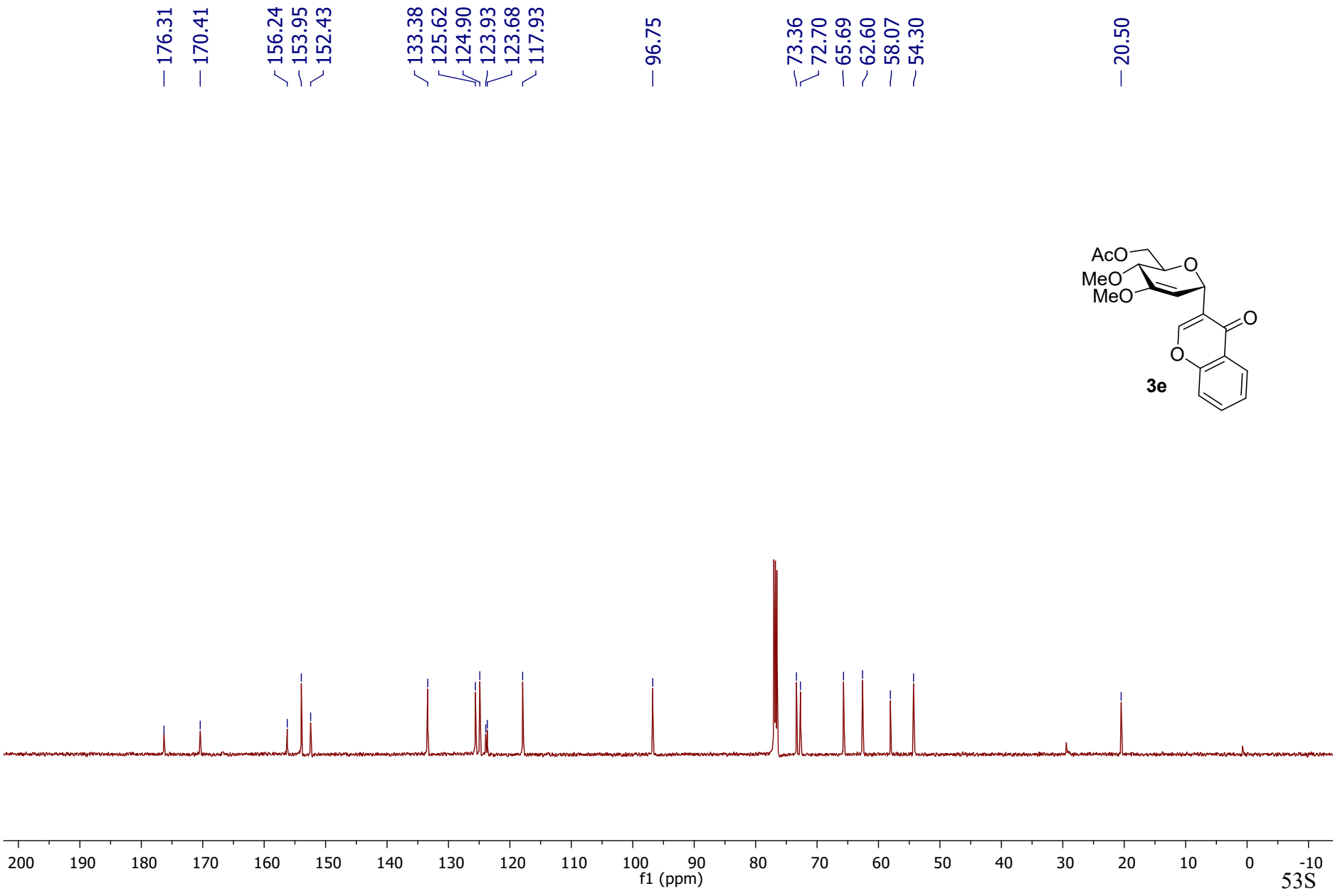
-1.0
f1 (ppm)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156.24
153.95
152.43
133.38
125.62
124.90
123.93
123.68
117.93
96.75
73.36
72.70
65.69
62.60
58.07
54.30
20.50
AcO
MeO
MeO
O
O
O
3e
200
190
180
170
160
150
140
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120
110
100
90
80
70
60
50
40
30
20
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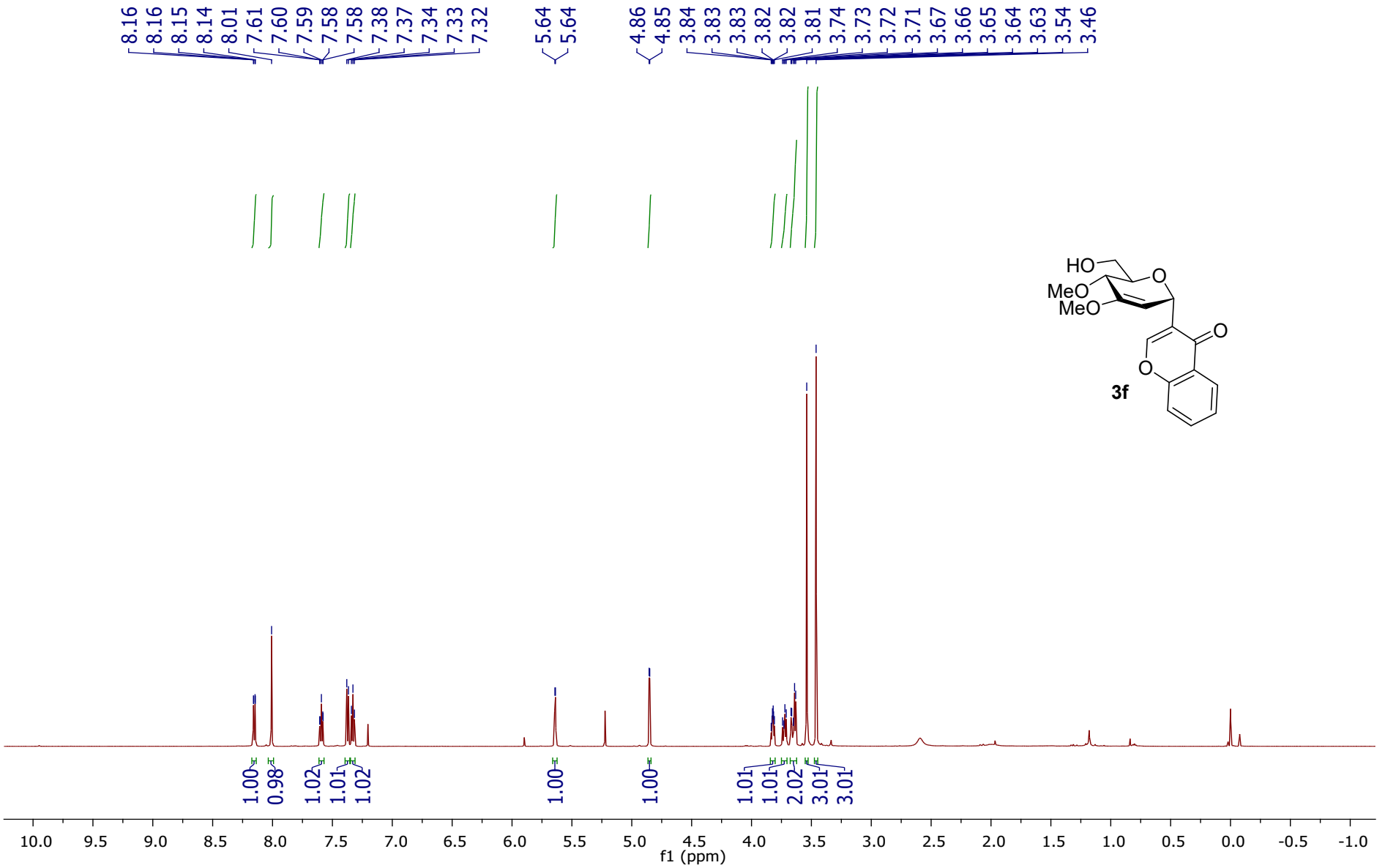
-10
f1 (ppm)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8.15
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7.60
7.59
7.58
7.58
7.38
7.37
7.34
7.33
7.32
5.64
5.64
4.86
4.85
3.84
3.83
3.83
3.82
3.82
3.81
3.74
3.73
3.72
3.71
3.67
3.66
3.65
3.64
3.63
3.54
3.46
HO
MeO
MeO
O
O
O
3f
1.00
0.98
1.02
1.01
1.02
1.00
1.00
1.01
1.01
2.02
3.01
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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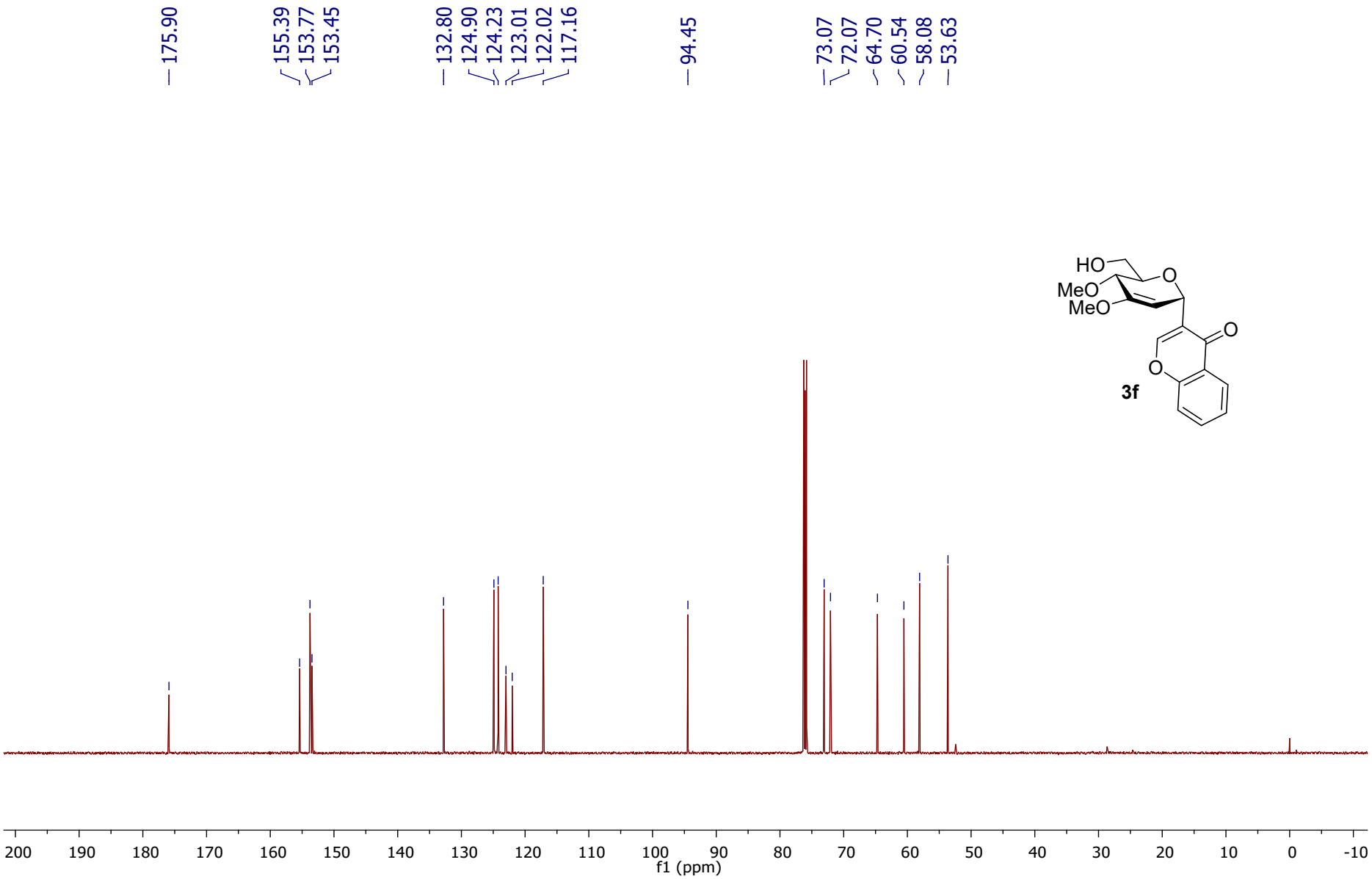
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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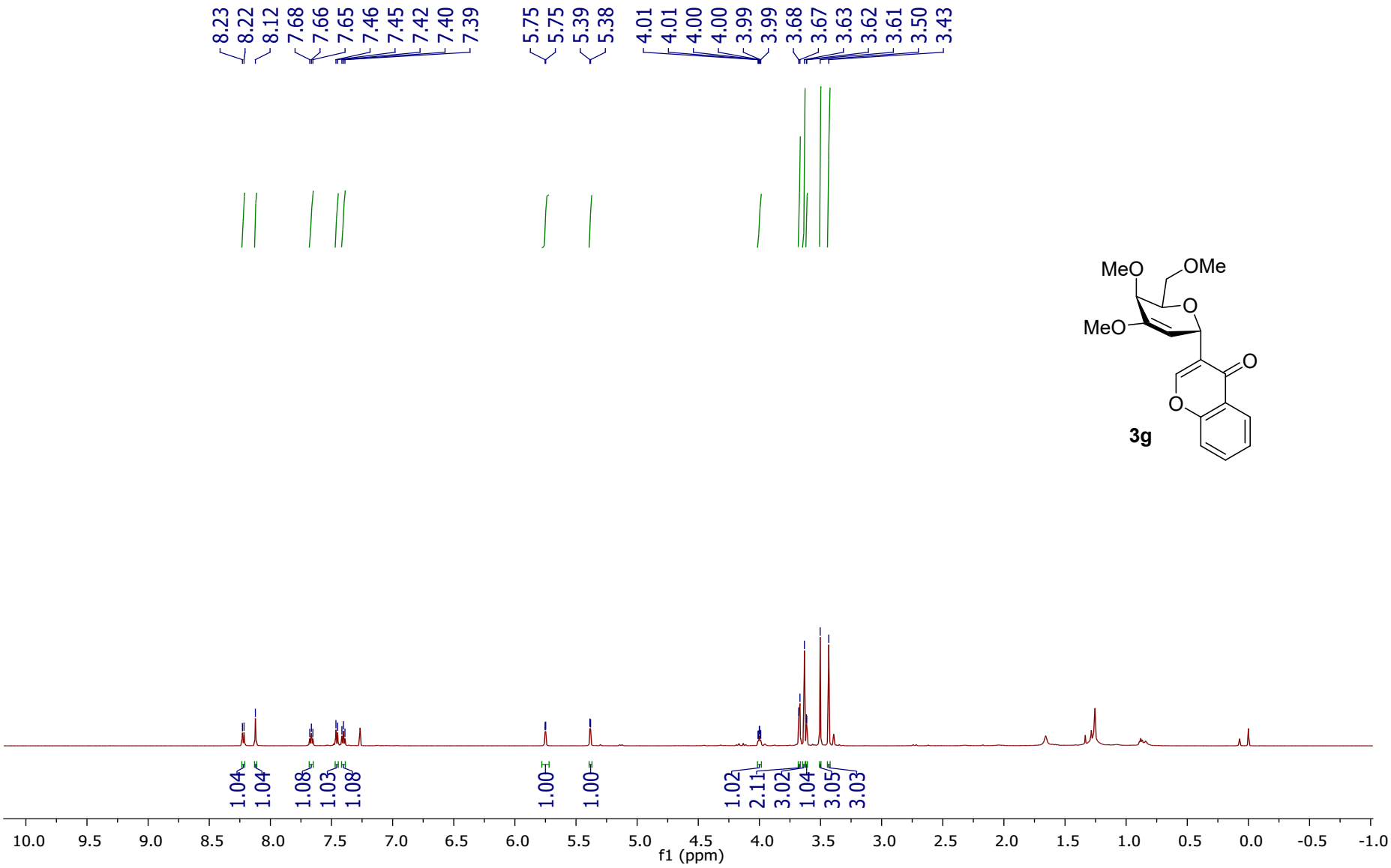
124.23
123.01
122.02
117.16
94.45
73.07
72.07
64.70
60.54
58.08
53.63
HO
MeO
MeO
O
O
O
3f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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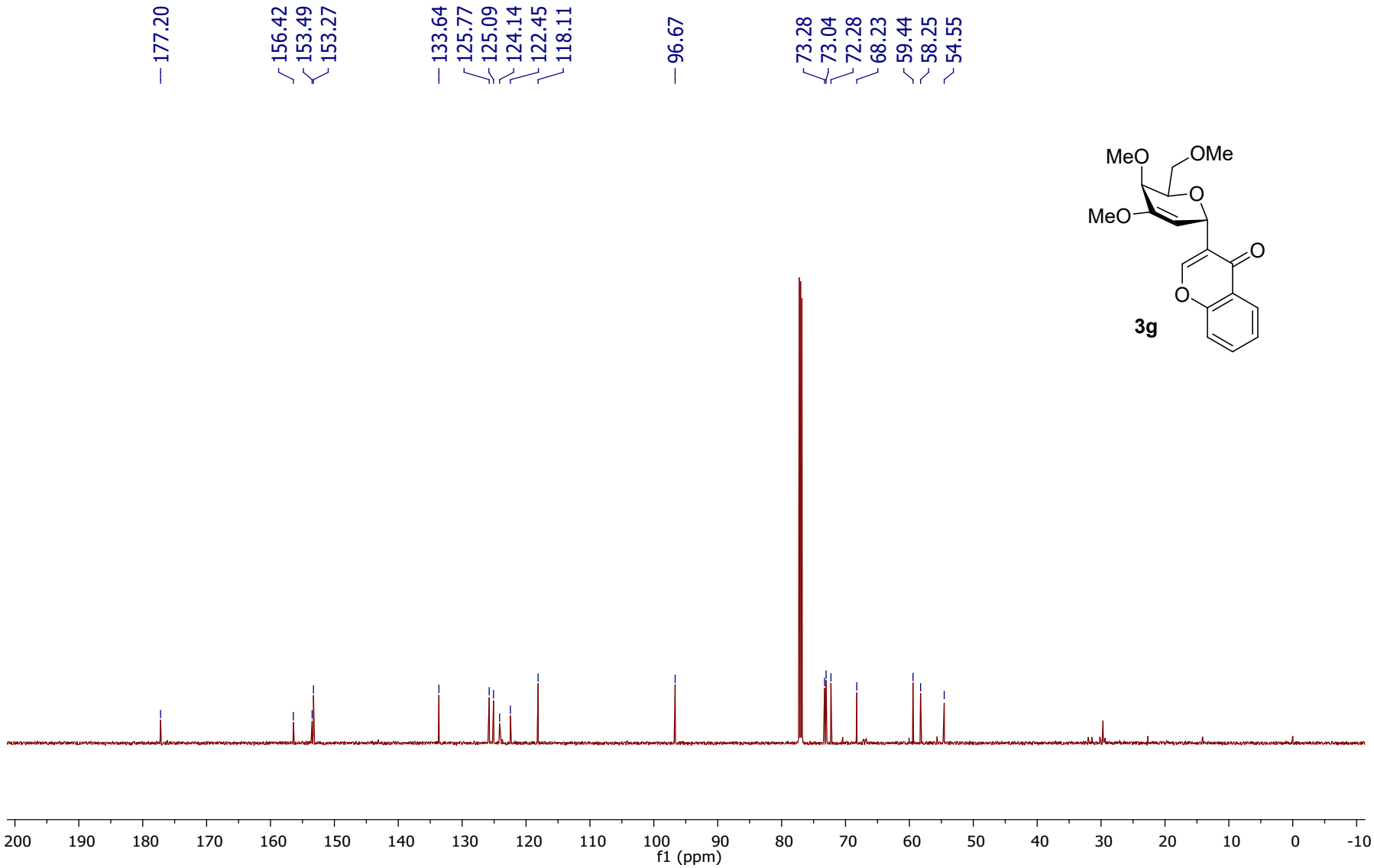
7.65
7.46
7.45
7.42
7.40
7.39
5.75
5.75
5.39
5.38
4.01
4.01
4.00
4.00
3.99
3.99
3.68
3.67
3.63
3.62
3.61
3.50
3.43
MeO
OMe
O
MeO
O
O
3g
1.04
1.04
1.08
1.03
1.08
1.00
1.00
1.02
2.11
3.02
1.04
3.05
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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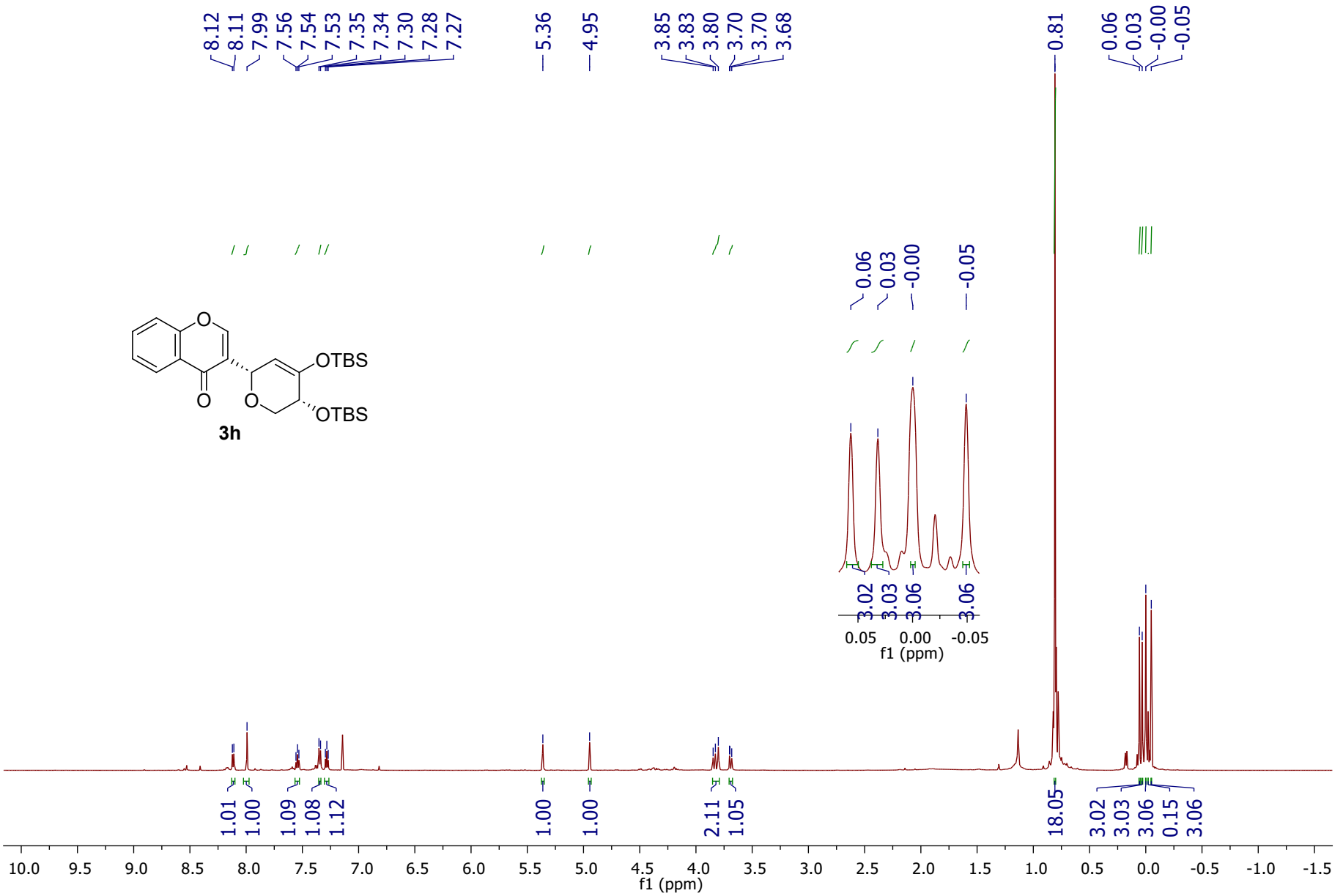
125.77
125.09
124.14
122.45
118.11
96.67
73.28
73.04
72.28
68.23
59.44
58.25
54.55
MeO
OMe
O
MeO
O
O
3g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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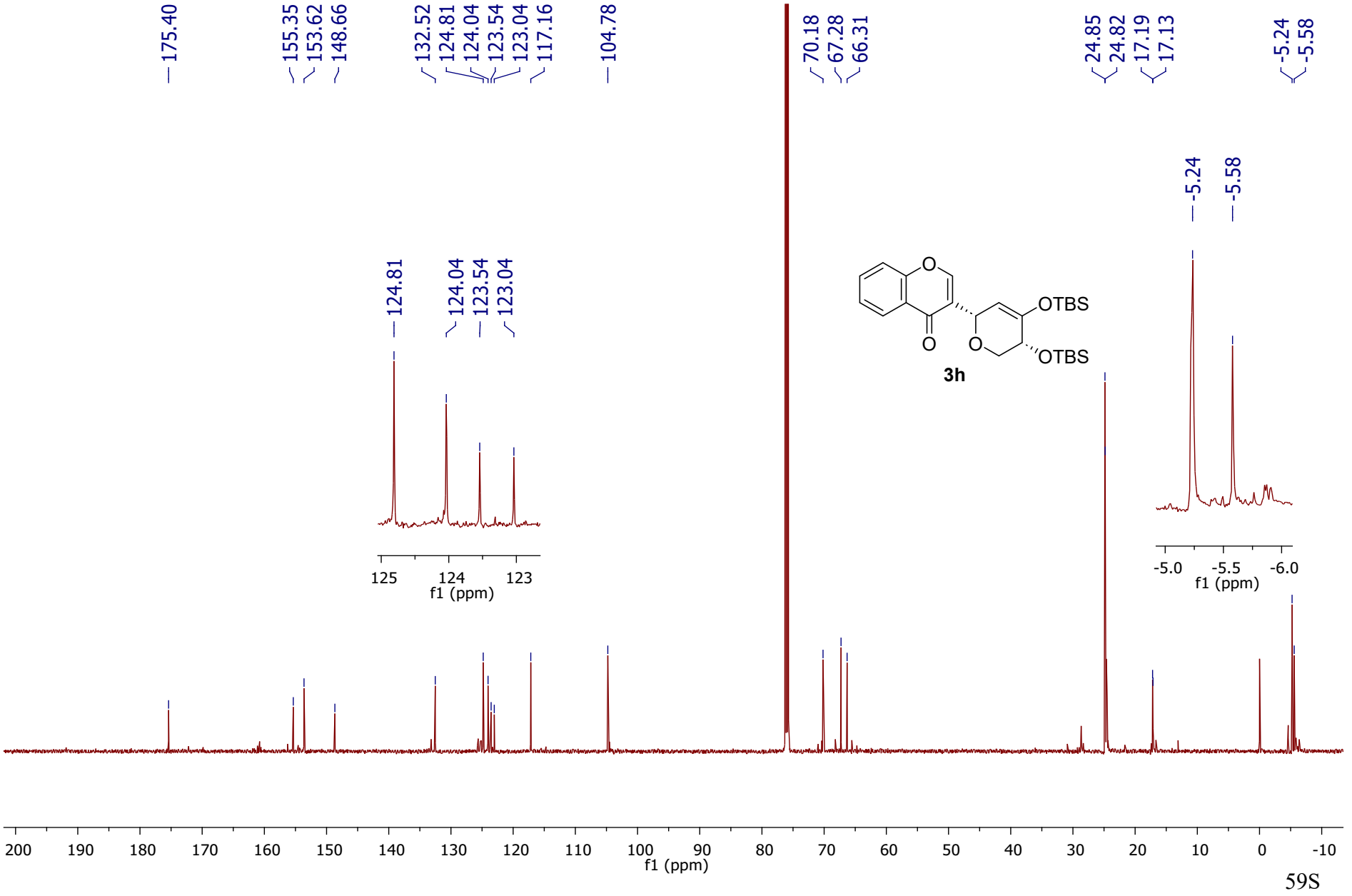
8.11
7.99
7.56
7.54
7.53
7.35
7.34
7.30
7.28
7.27
5.36
4.95
3.85
3.83
3.80
3.70
3.70
3.68
0.81
0.06
0.03
-0.00
-0.05
OTBS
OTBS
3h
0.06
0.03
-0.00
-0.05
3.02
3.03
3.06
3.06
0.05
0.00
-0.05
f1 (ppm)
1.01
1.00
1.09
1.08
1.12
1.00
1.00
2.11
1.05
18.05
3.02
3.03
3.06
0.15
3.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
f1 (ppm)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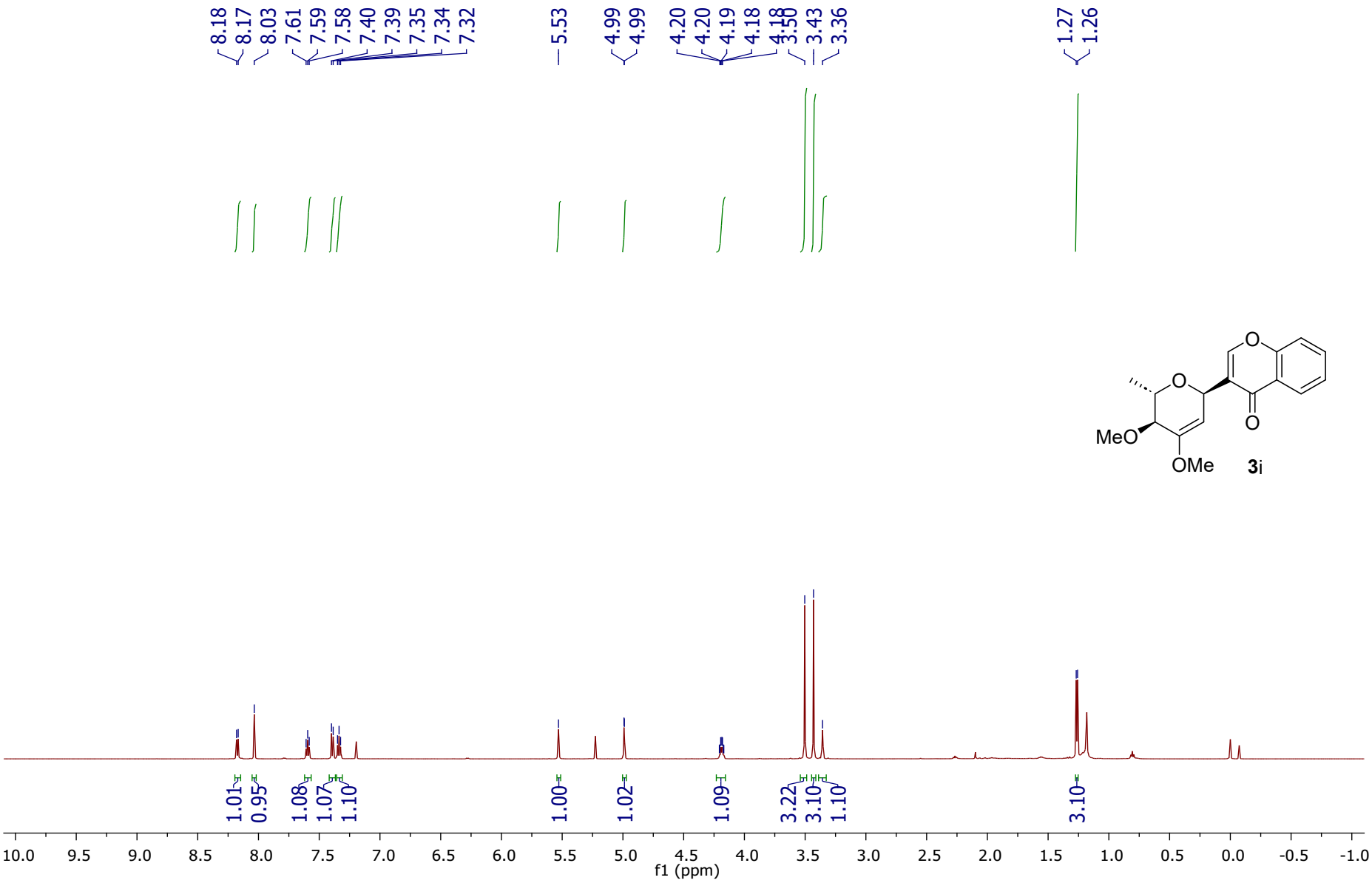
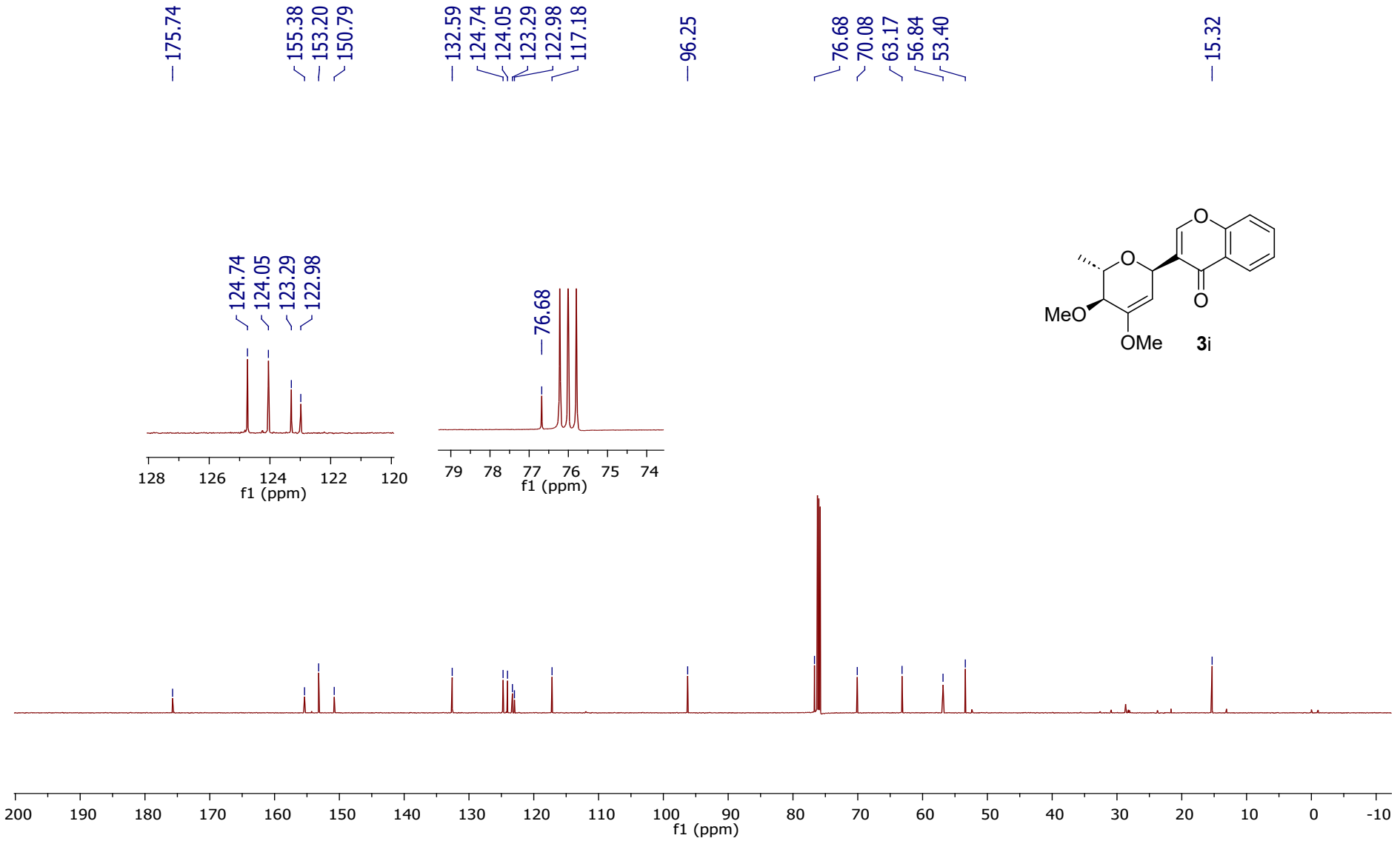
155.35
153.62
148.66
132.52
124.81
124.04
123.54
123.04
117.16
104.78
70.18
67.28
66.31
24.85
24.82
17.19
17.13
-5.24
-5.58
OTBS
OTBS
3h
f1 (ppm)
f1 (ppm)
f1 (ppm)


8.18
8.17
8.03
7.61
7.59
7.58
7.40
7.39
7.35
7.34
7.32
5.53
4.99
4.99
4.20
4.20
4.19
4.18
3.50
3.43
3.36
1.27
1.26
MeO
OMe
3i
1.01
0.95
1.08
1.07
1.10
1.00
1.02
1.09
3.22
3.10
1.10
3.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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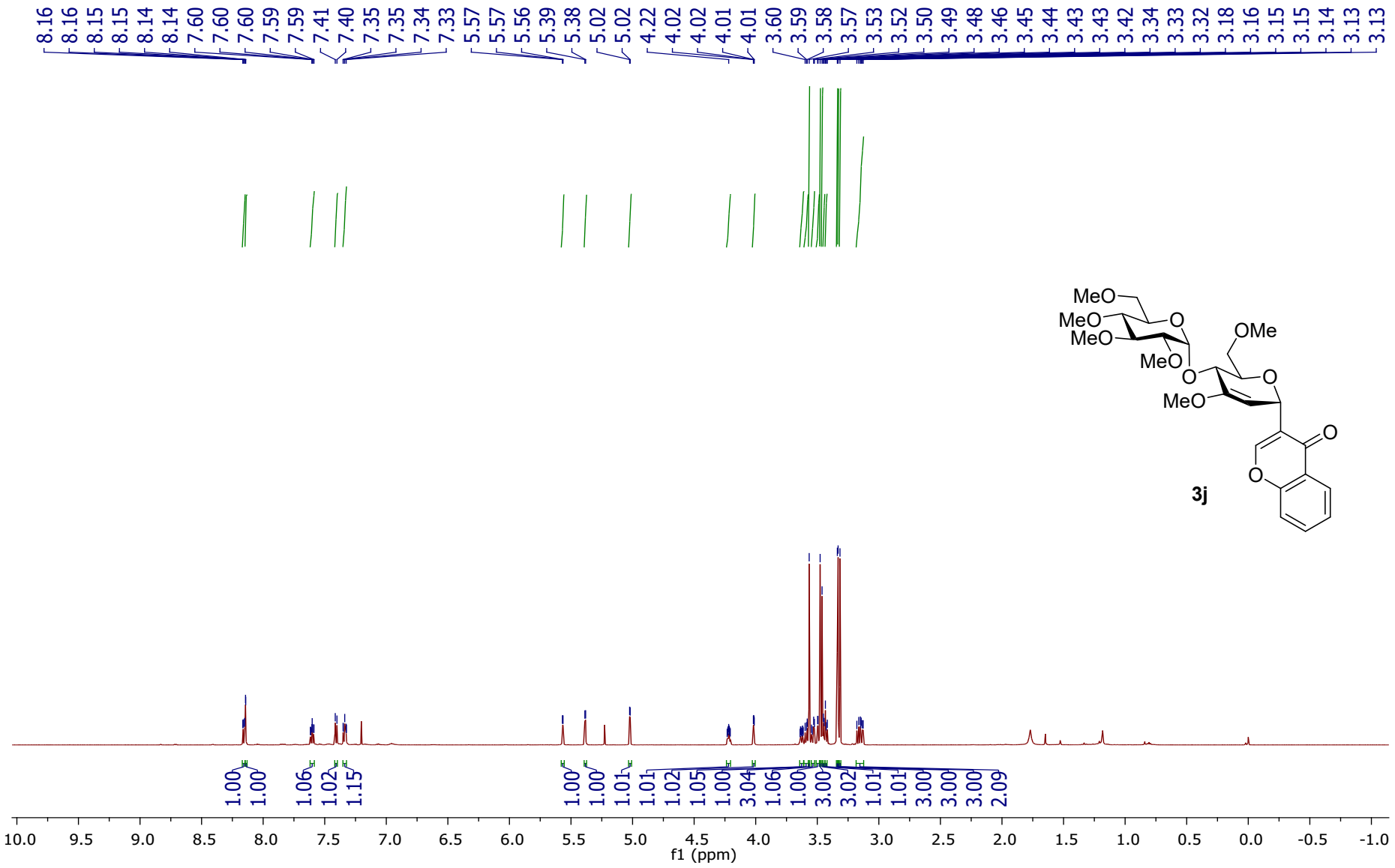
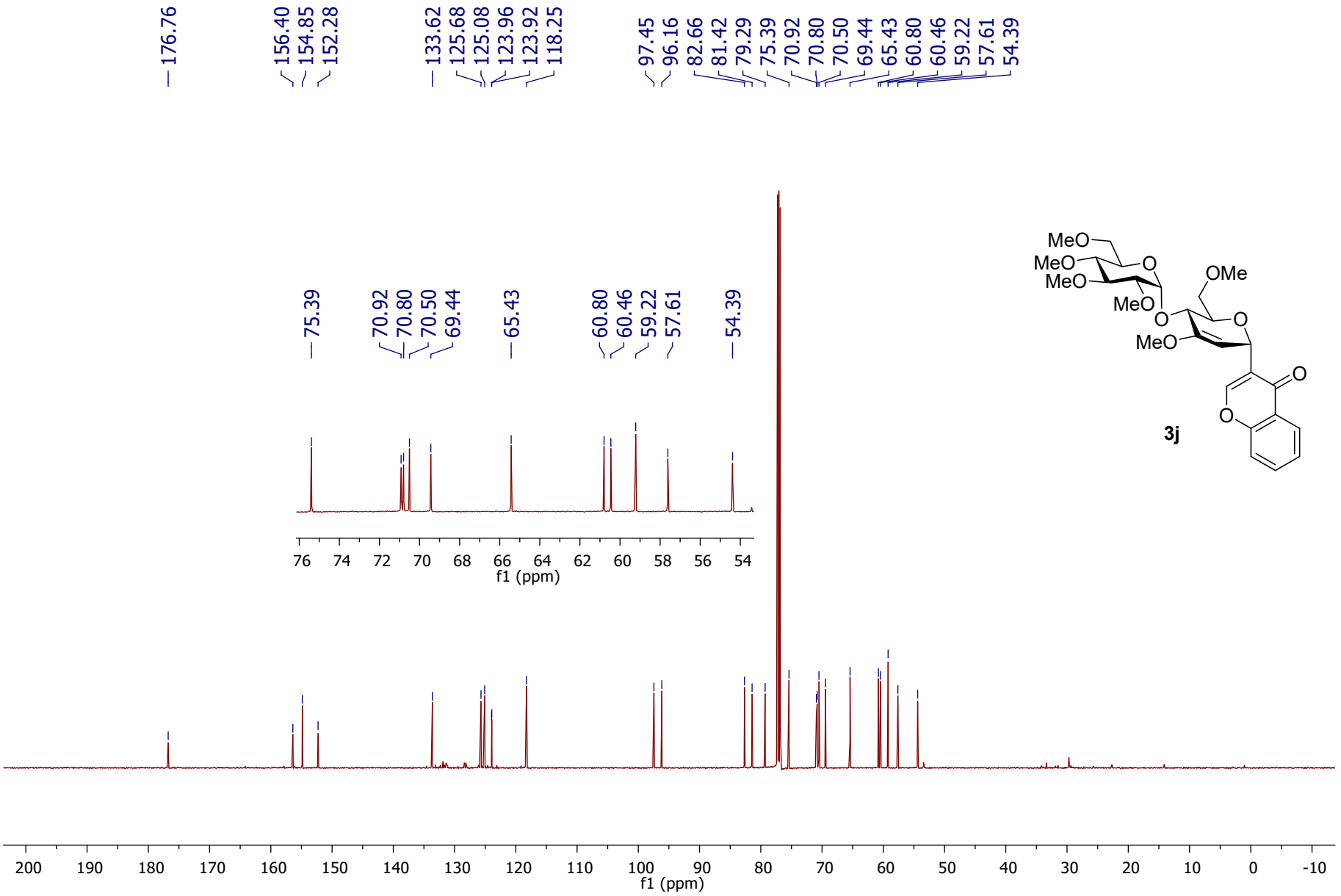
155.38
153.20
150.79
132.59
124.74
124.05
123.29
122.98
117.18
96.25
76.68
70.08
63.17
56.84
53.40
15.32
MeO
OMe
3i
f1 (ppm)


8.16 8.16 8.15 8.15 8.14 8.14 7.60 7.60 7.60 7.59 7.59 7.41 7.40 7.35 7.35 7.34 7.33 5.57 5.57 5.56 5.39 5.38 5.02 5.02 4.22 4.02 4.02 4.01 4.01 3.60 3.59 3.58 3.57 3.53 3.52 3.50 3.49 3.48 3.46 3.45 3.44 3.43 3.43 3.42 3.34 3.33 3.32 3.18 3.16 3.15 3.15 3.14 3.13 3.13
MeO
MeO
MeO
MeO
MeO
OMe
O
O
O
O
O
O
3j
1.00 1.00 1.06 1.02 1.15 1.00 1.00 1.01 1.01 1.02 1.05 1.00 3.04 1.06 1.00 3.00 3.02 1.01 1.01 3.00 3.00 3.00 2.09
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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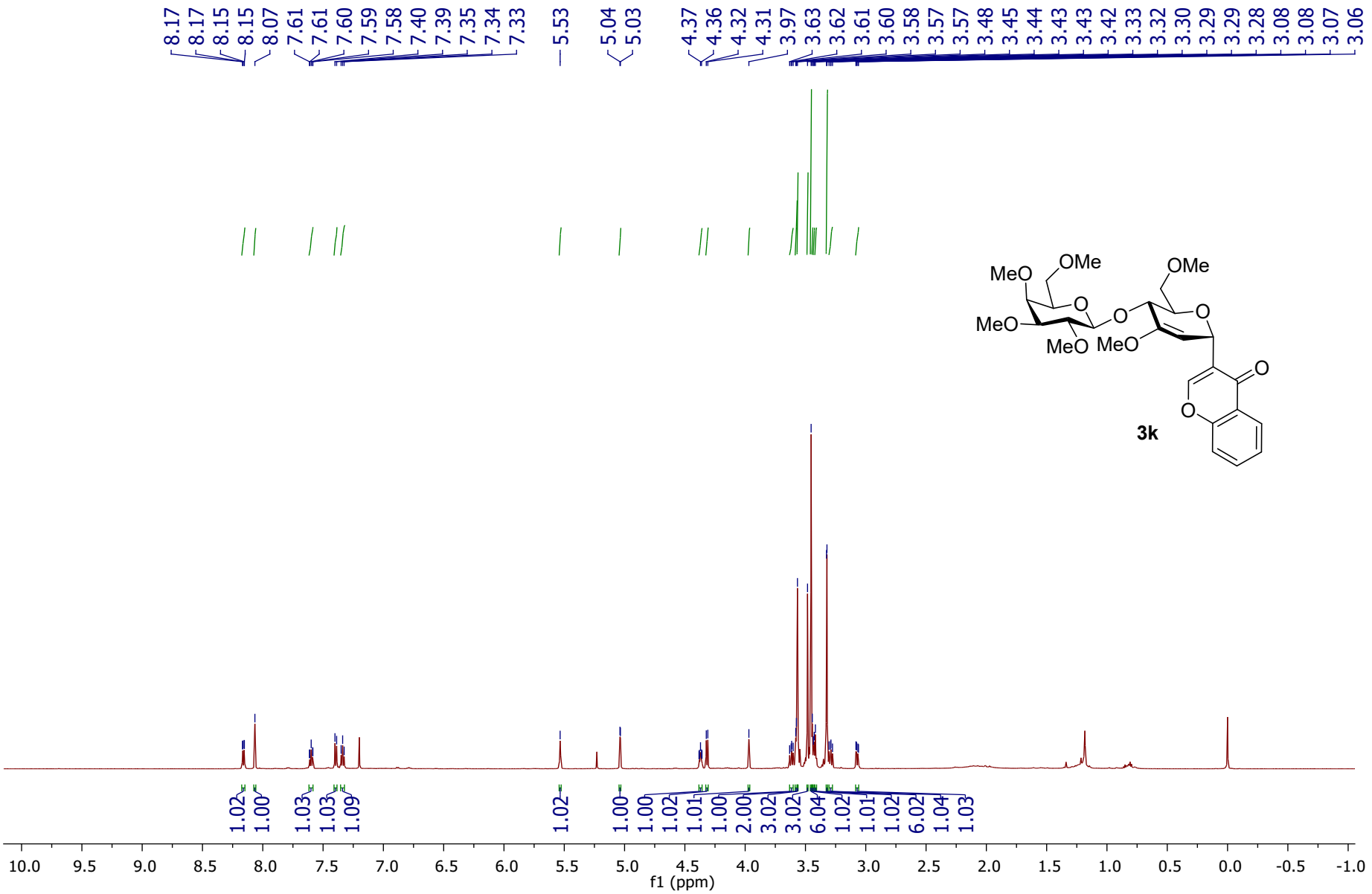
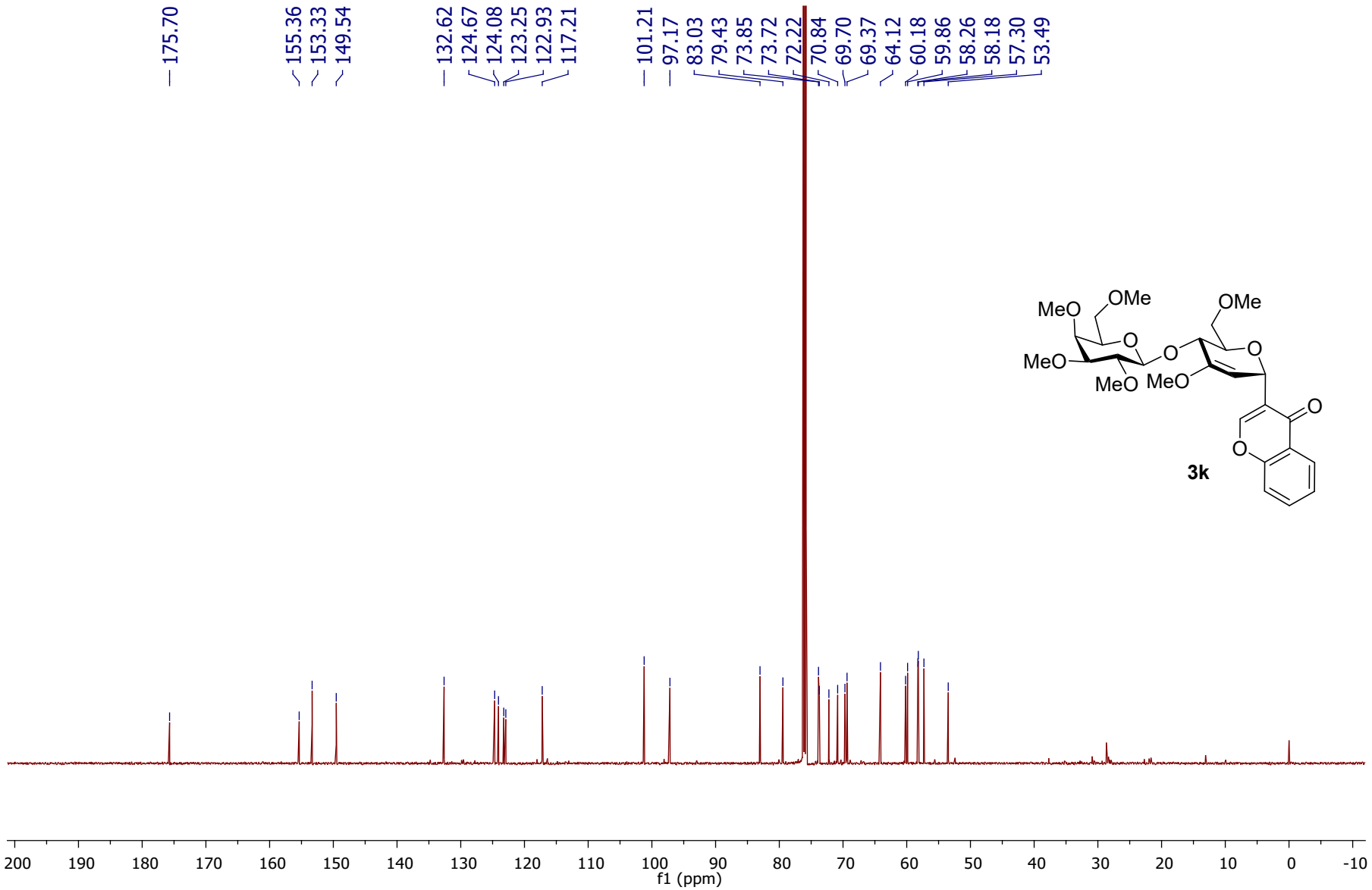
154.85
152.28
133.62
125.68
125.08
123.96
123.92
118.25
97.45
96.16
82.66
81.42
79.29
75.39
70.92
70.80
70.50
69.44
65.43
60.80
60.46
59.22
57.61
54.39
f1 (ppm)
MeO
MeO
MeO
MeO
MeO
OMe
O
O
O
O
O
3j


8.17 8.17 8.15 8.15 8.07 7.61 7.61 7.60 7.59 7.58 7.40 7.39 7.35 7.34 7.33 5.53 5.04 5.03 4.37 4.36 4.32 4.31 3.97 3.63 3.62 3.61 3.60 3.58 3.57 3.57 3.48 3.45 3.44 3.43 3.43 3.42 3.33 3.32 3.30 3.29 3.29 3.28 3.08 3.08 3.07 3.06
MeO
OMe
MeO
O
O
MeO
MeO
OMe
O
O
O
O
3k
1.02 1.00 1.03 1.03 1.09 1.02 1.00 1.00 1.02 1.01 1.00 2.00 3.02 3.02 6.04 1.02 1.01 1.02 6.02 1.04 1.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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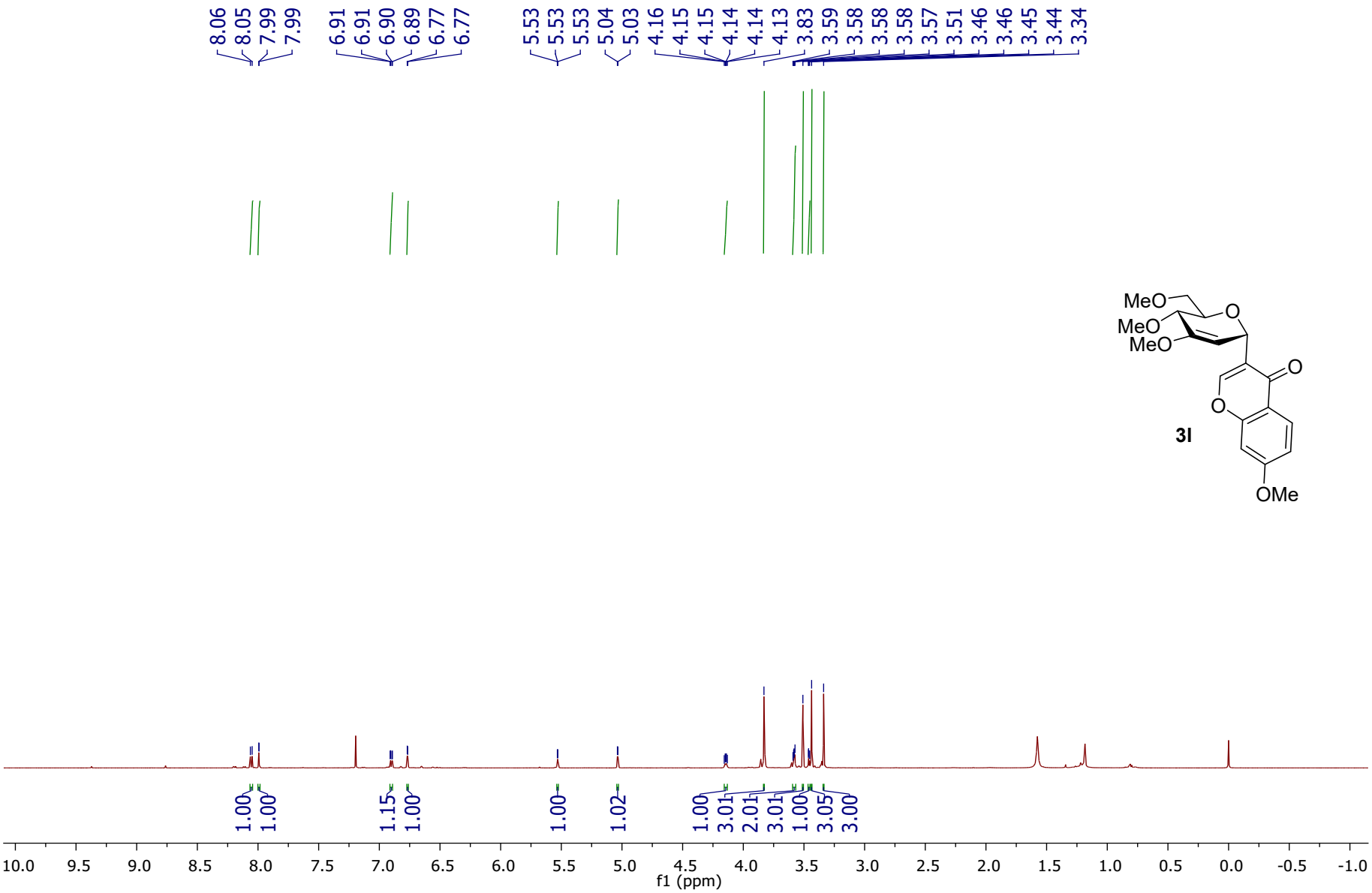
153.33
149.54
132.62
124.67
124.08
123.25
122.93
117.21
101.21
97.17
83.03
79.43
73.85
73.72
72.22
70.84
69.70
69.37
64.12
60.18
59.86
58.26
58.18
57.30
53.49
MeO
OMe
MeO
MeO
MeO
OMe
O
O
O
O
O
O
3k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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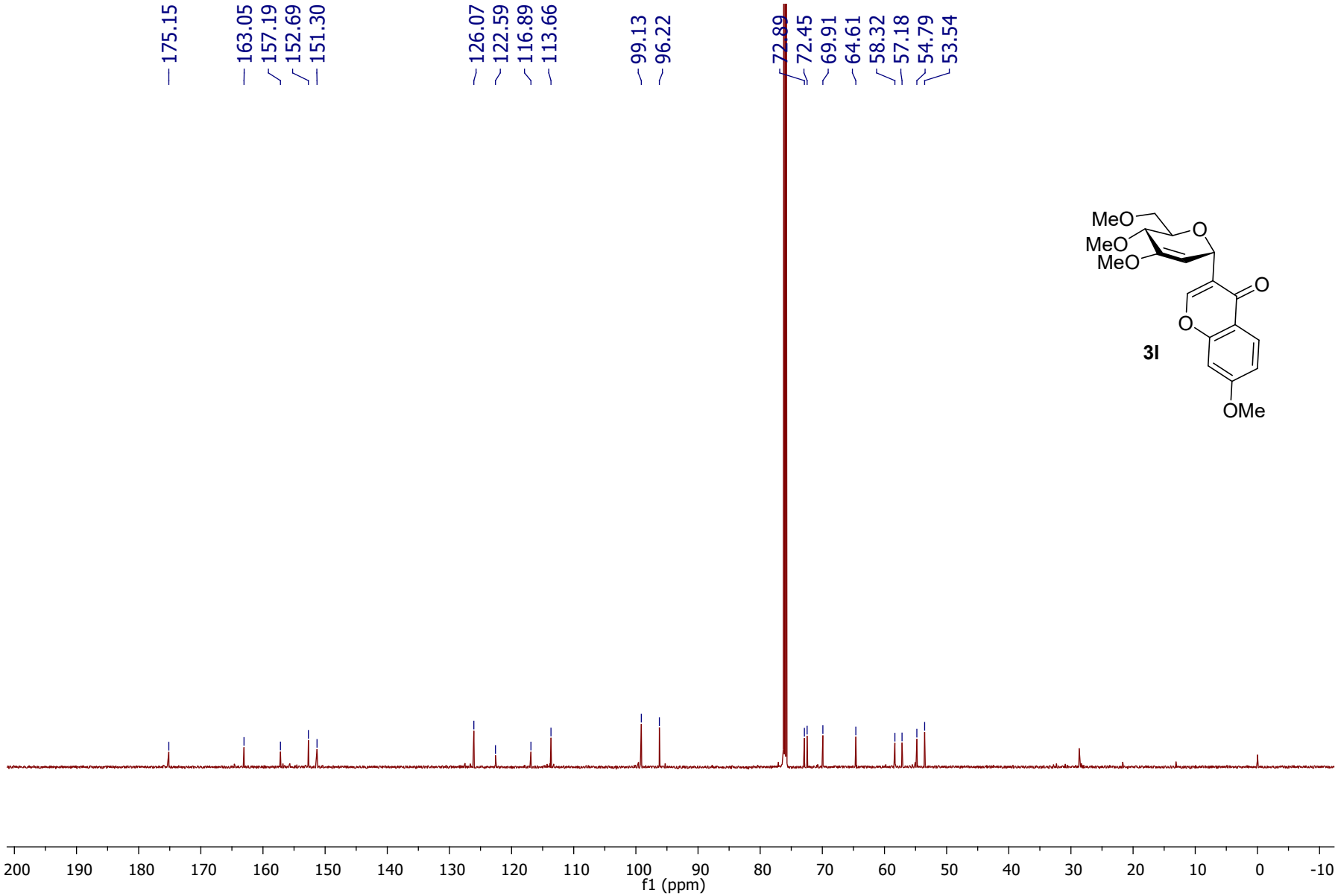
8.05
7.99
7.99
6.91
6.91
6.90
6.89
6.77
6.77
5.53
5.53
5.53
5.04
5.03
4.16
4.15
4.15
4.14
4.14
4.13
3.83
3.59
3.58
3.58
3.58
3.57
3.51
3.46
3.46
3.45
3.44
3.34
MeO
MeO
MeO
O
O
O
OMe
3l
1.00
1.00
1.15
1.00
1.00
1.02
1.00
3.01
2.01
3.01
1.00
3.05
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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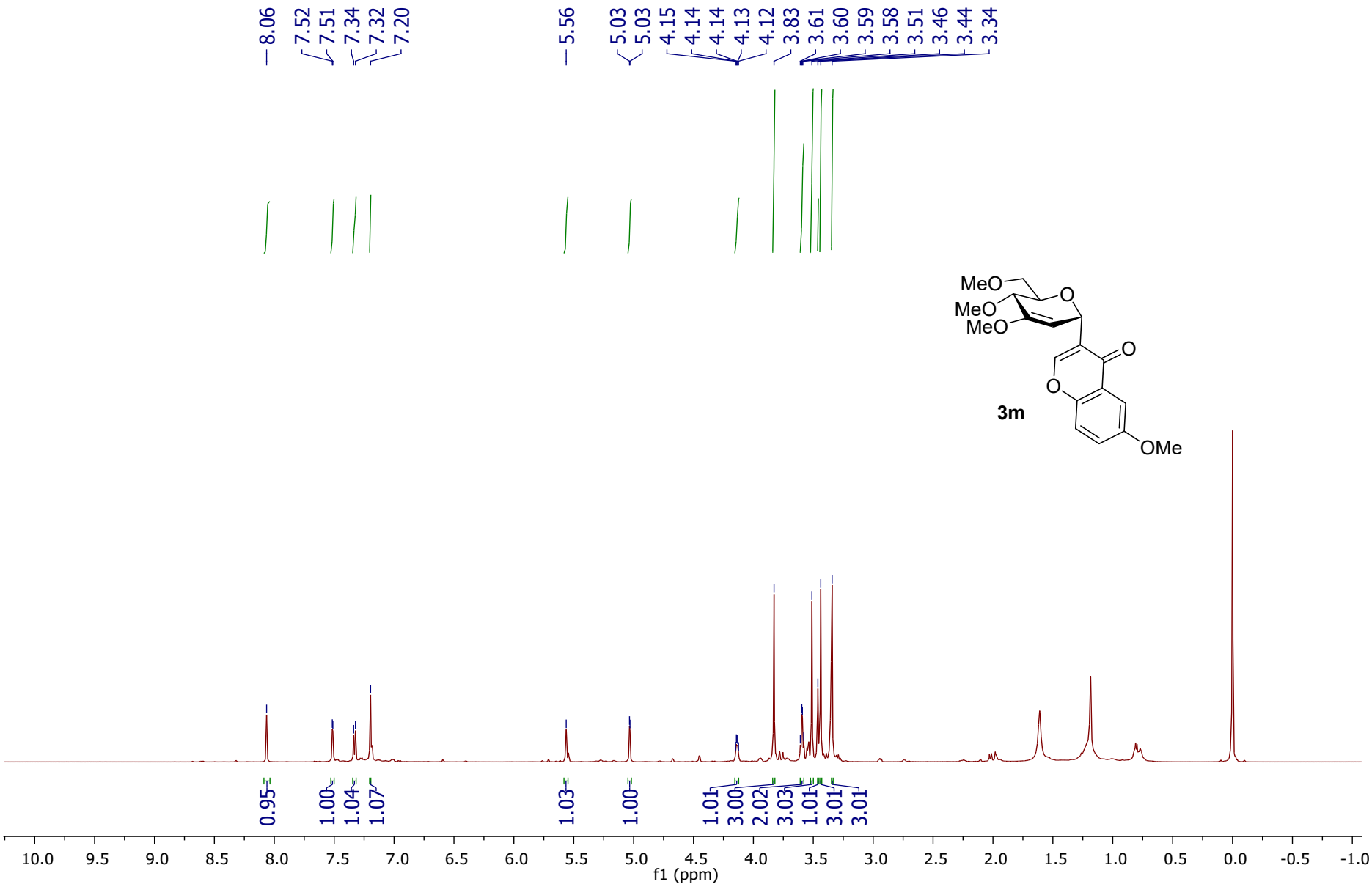
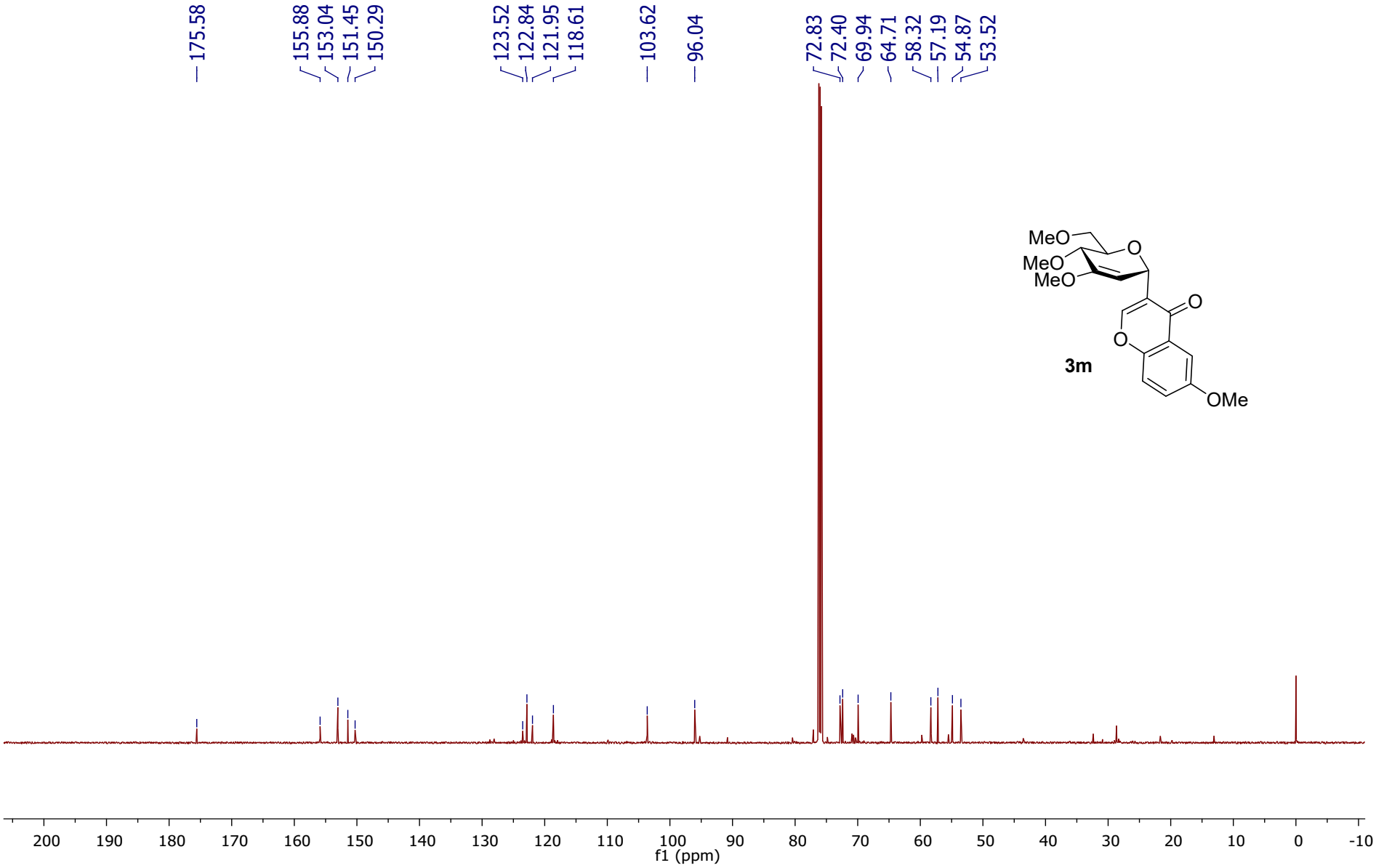
151.30
126.07
122.59
116.89
113.66
99.13
96.22
72.89
72.45
69.91
64.61
58.32
57.18
54.79
53.54
MeO
MeO
MeO
O
O
O
OMe
3l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.06
7.52
7.51
7.34
7.32
7.20
5.56
5.03
5.03
4.15
4.14
4.14
4.13
4.12
3.83
3.61
3.60
3.59
3.58
3.51
3.46
3.44
3.34
MeO
MeO
MeO
O
O
O
3m
OMe
0.95
1.00
1.04
1.07
1.03
1.00
1.01
3.00
2.02
3.03
1.01
3.01
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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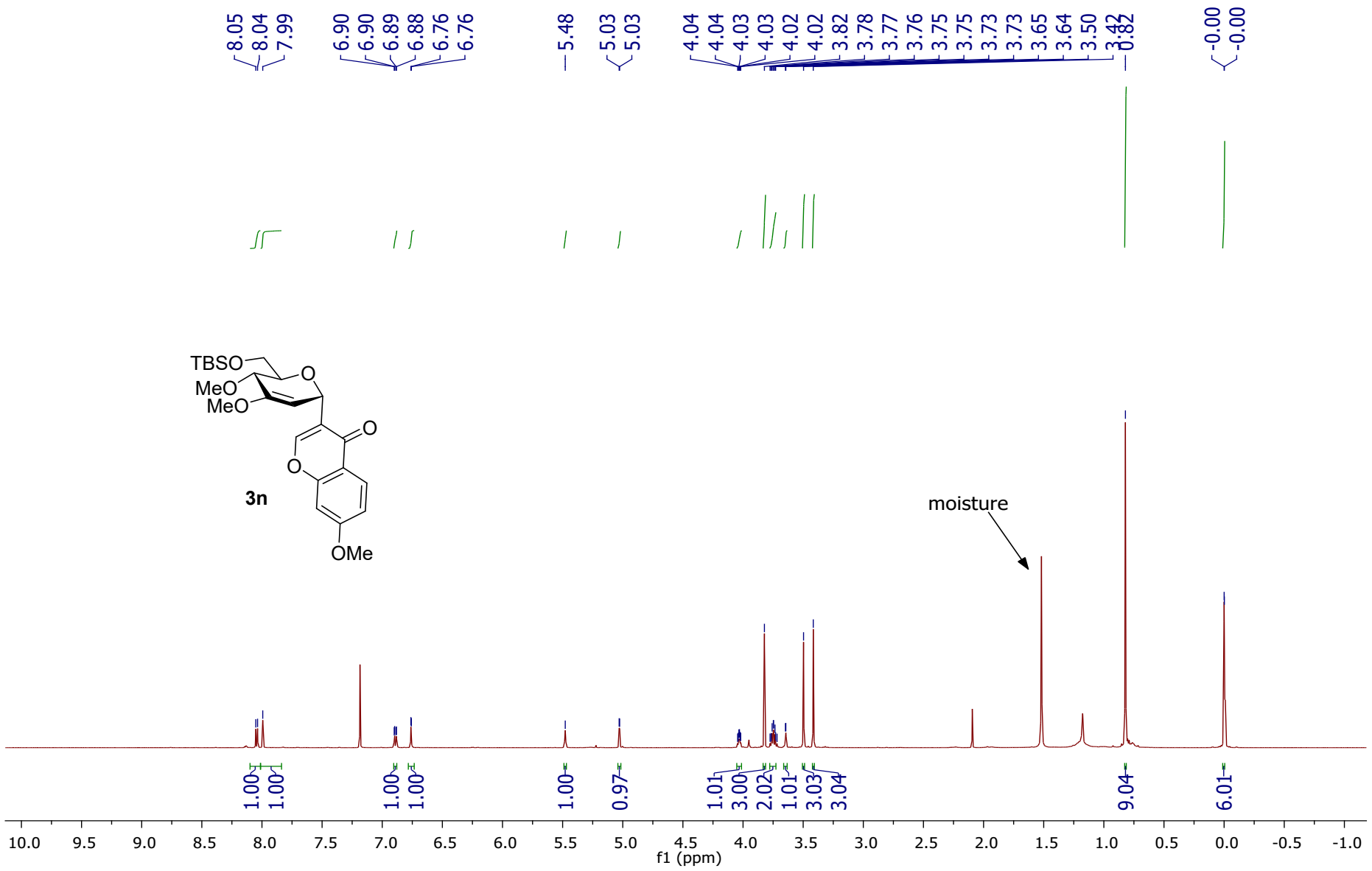
123.52
122.84
121.95
118.61
103.62
96.04
72.83
72.40
69.94
64.71
58.32
57.19
54.87
53.52
MeO
MeO
MeO
O
O
O
OMe
3m
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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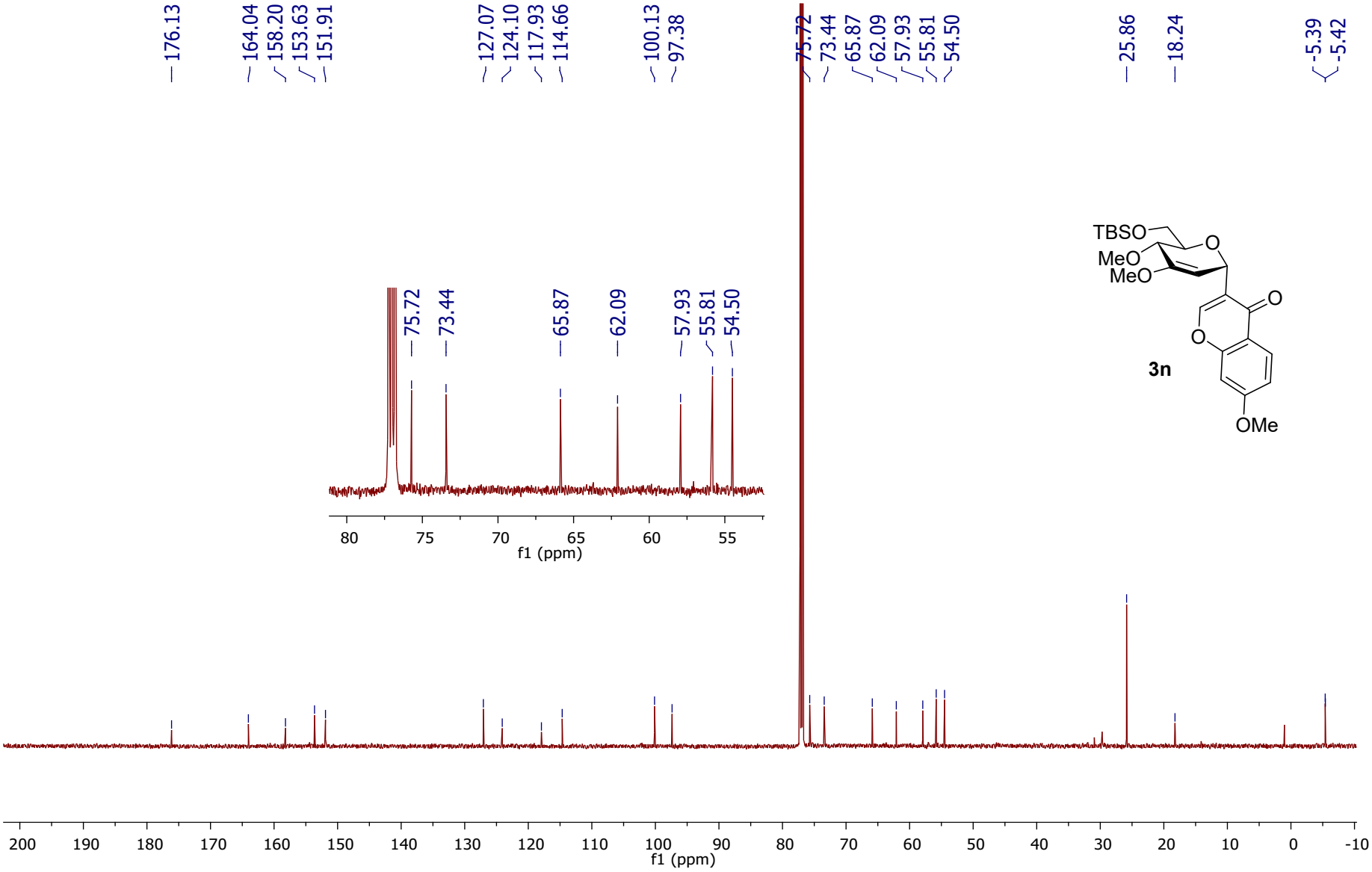
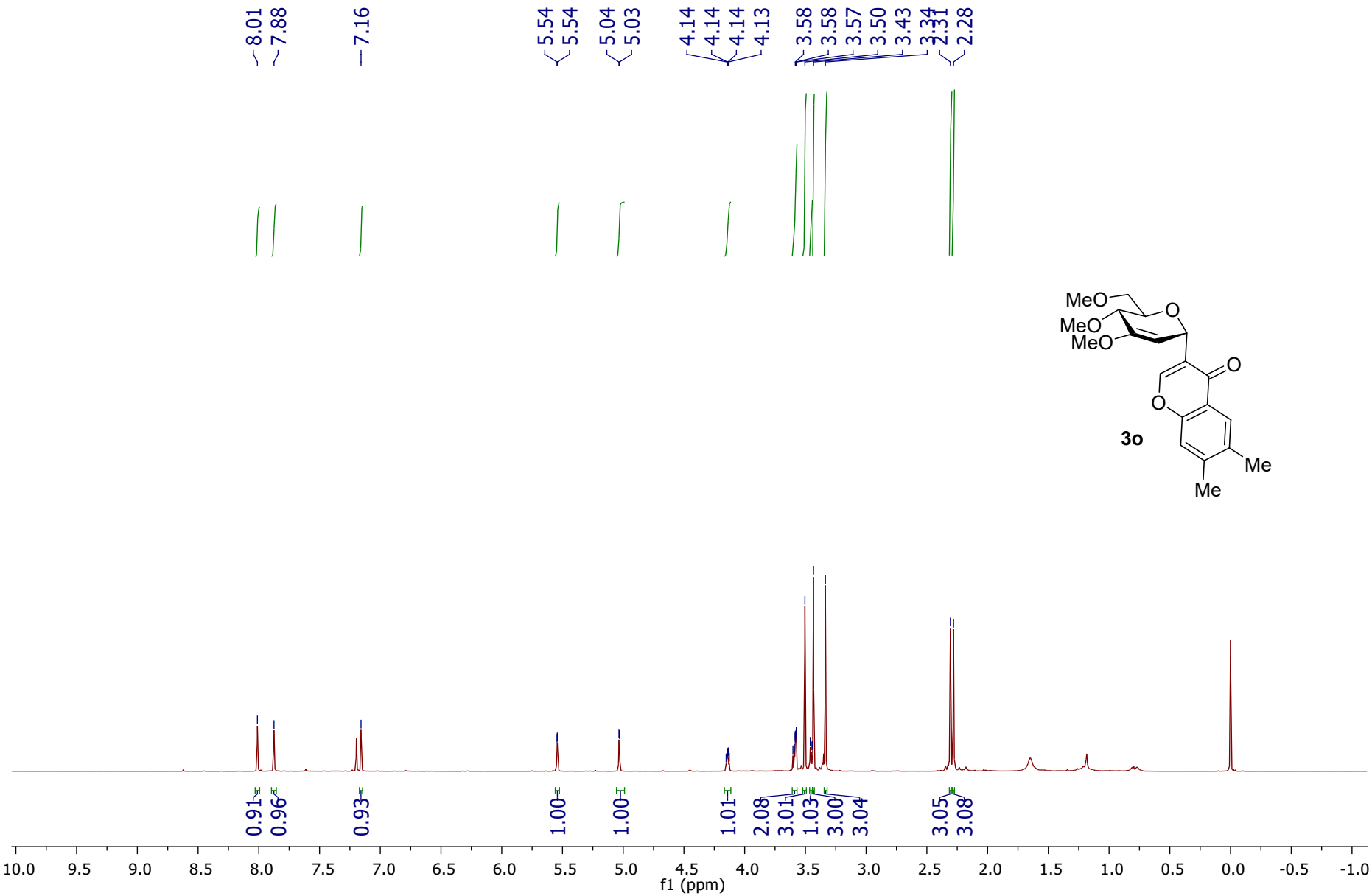
6.76
5.48
5.03
5.03
4.04
4.04
4.03
4.03
4.02
4.02
3.82
3.78
3.77
3.76
3.75
3.75
3.73
3.73
3.65
3.64
3.50
3.42
0.82
-0.00
-0.00
TBSO
MeO
MeO
O
O
O
OMe
3n
moisture
1.00
1.00
1.00
1.00
1.00
0.97
1.01
3.00
2.02
1.01
3.03
3.04
9.04
6.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


176.13
164.04
158.20
153.63
151.91
127.07
124.10
117.93
114.66
100.13
97.38
75.72
73.44
65.87
62.09
57.93
55.81
54.50
25.86
18.24
-5.39
-5.42
TBSO
MeO
MeO
O
O
O
OMe
3n
f1 (ppm)
f1 (ppm)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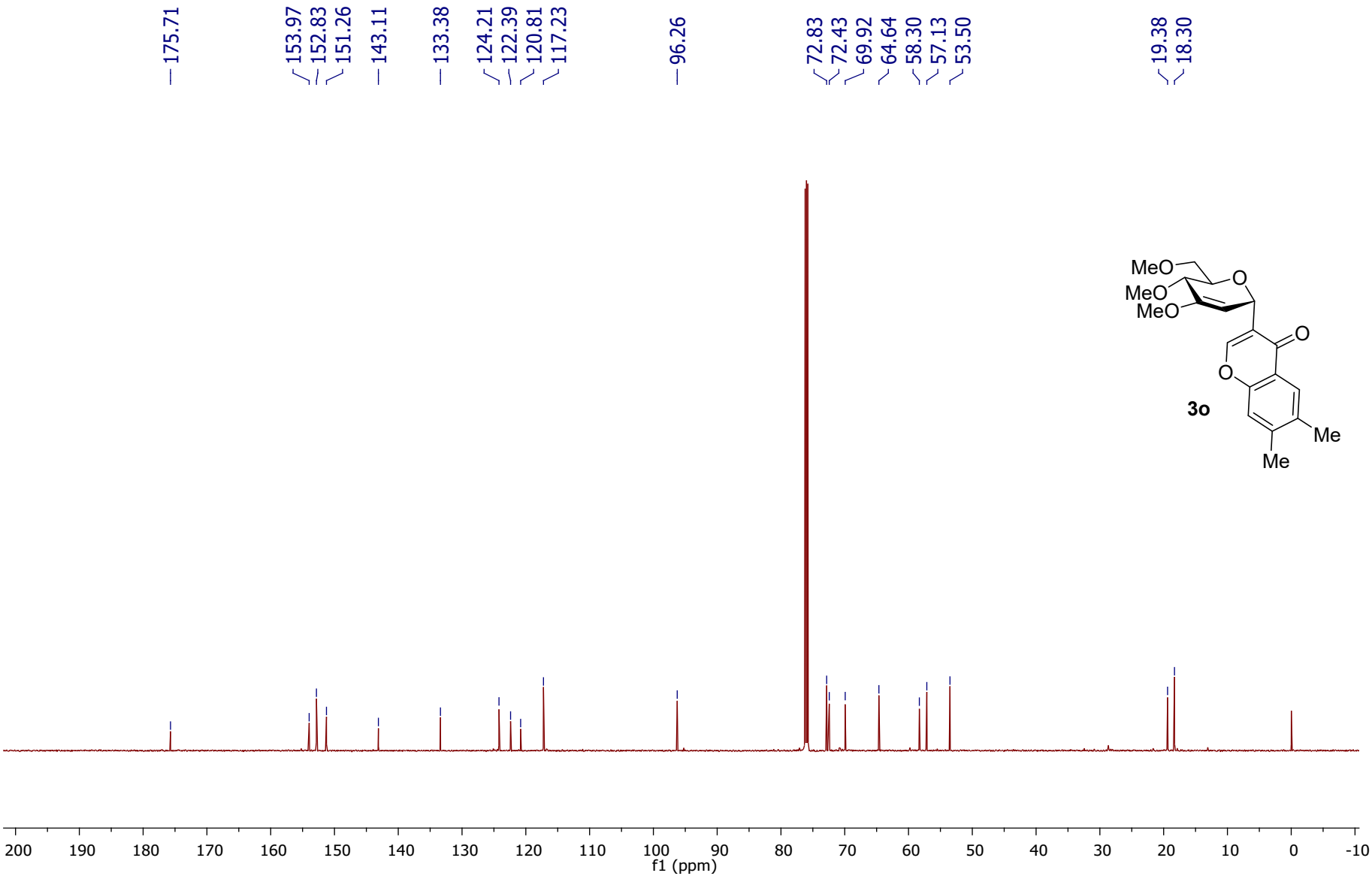
5.54
5.04
5.03
4.14
4.14
4.14
4.13
3.58
3.58
3.57
3.50
3.43
3.34
2.31
2.28
MeO
MeO
MeO
O
O
O
3o
Me
Me
0.91
0.96
0.93
1.00
1.00
1.01
2.08
3.01
1.03
3.00
3.04
3.05
3.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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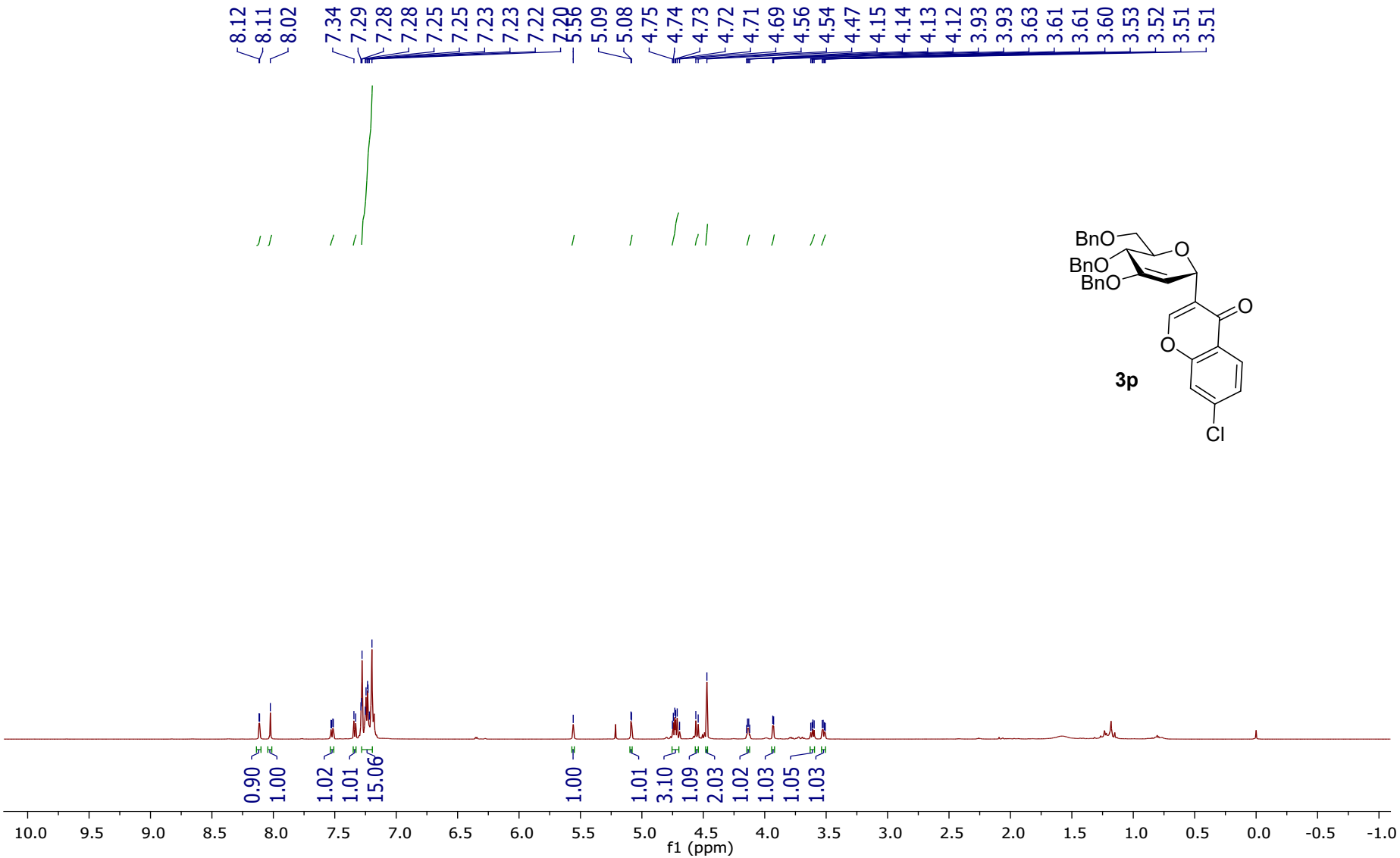
153.97
152.83
151.26
143.11
133.38
124.21
122.39
120.81
117.23
96.26
72.83
72.43
69.92
64.64
58.30
57.13
53.50
19.38
18.30
MeO
MeO
MeO
O
O
O
Me
Me
3o
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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8.02
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7.28
7.28
7.25
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7.23
7.23
7.22
7.20
5.56
5.09
5.08
4.75
4.74
4.73
4.72
4.71
4.69
4.56
4.54
4.47
4.15
4.14
4.13
4.12
3.93
3.93
3.63
3.61
3.61
3.60
3.53
3.52
3.51
3.51
BnO
BnO
BnO
O
O
O
Cl
3p
0.90
1.00
1.02
1.01
15.06
1.00
1.01
3.10
1.09
2.03
1.02
1.03
1.05
1.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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2.5
2.0
1.5
1.0
0.5
0.0
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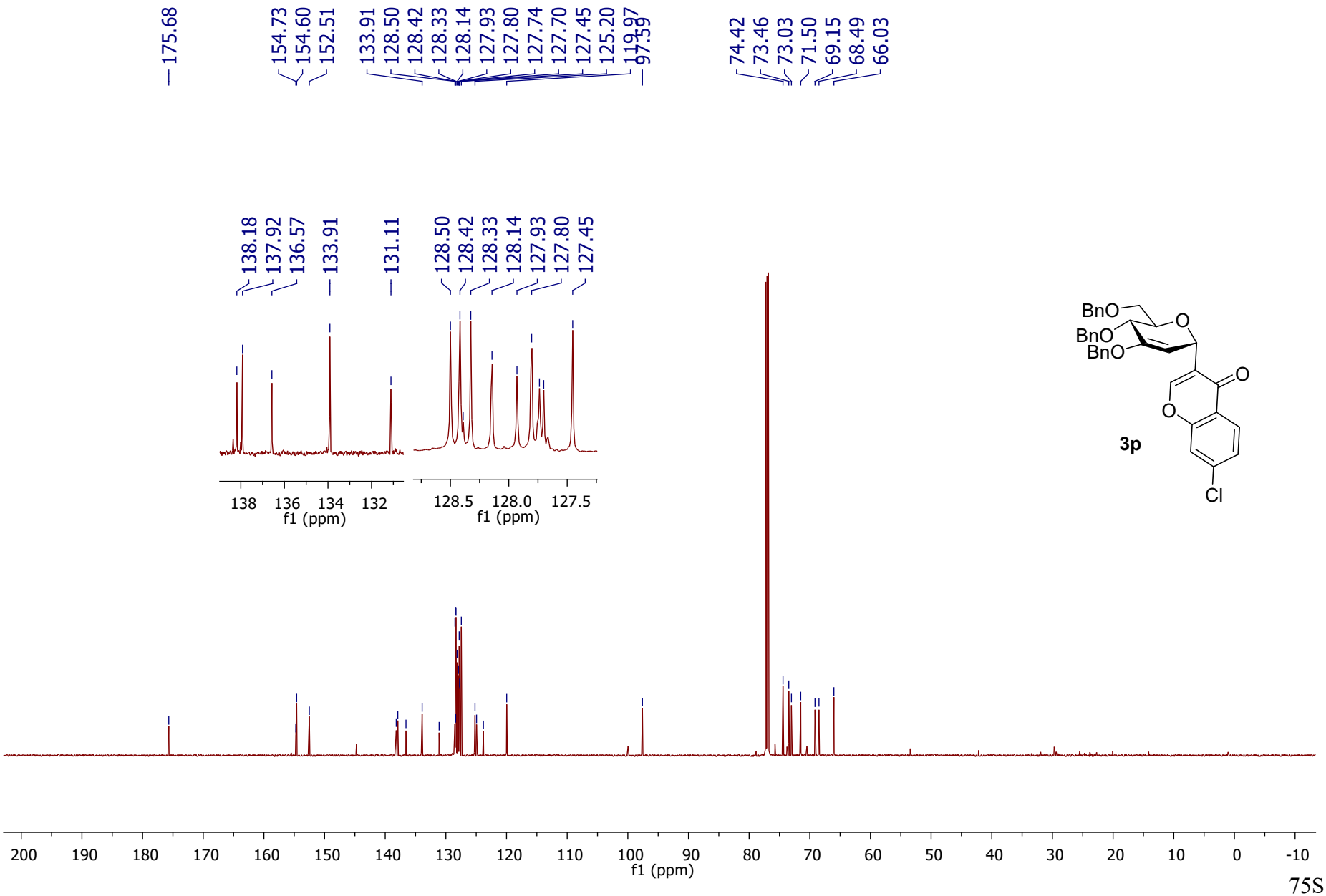
-1.0
f1 (ppm)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154.60
152.51
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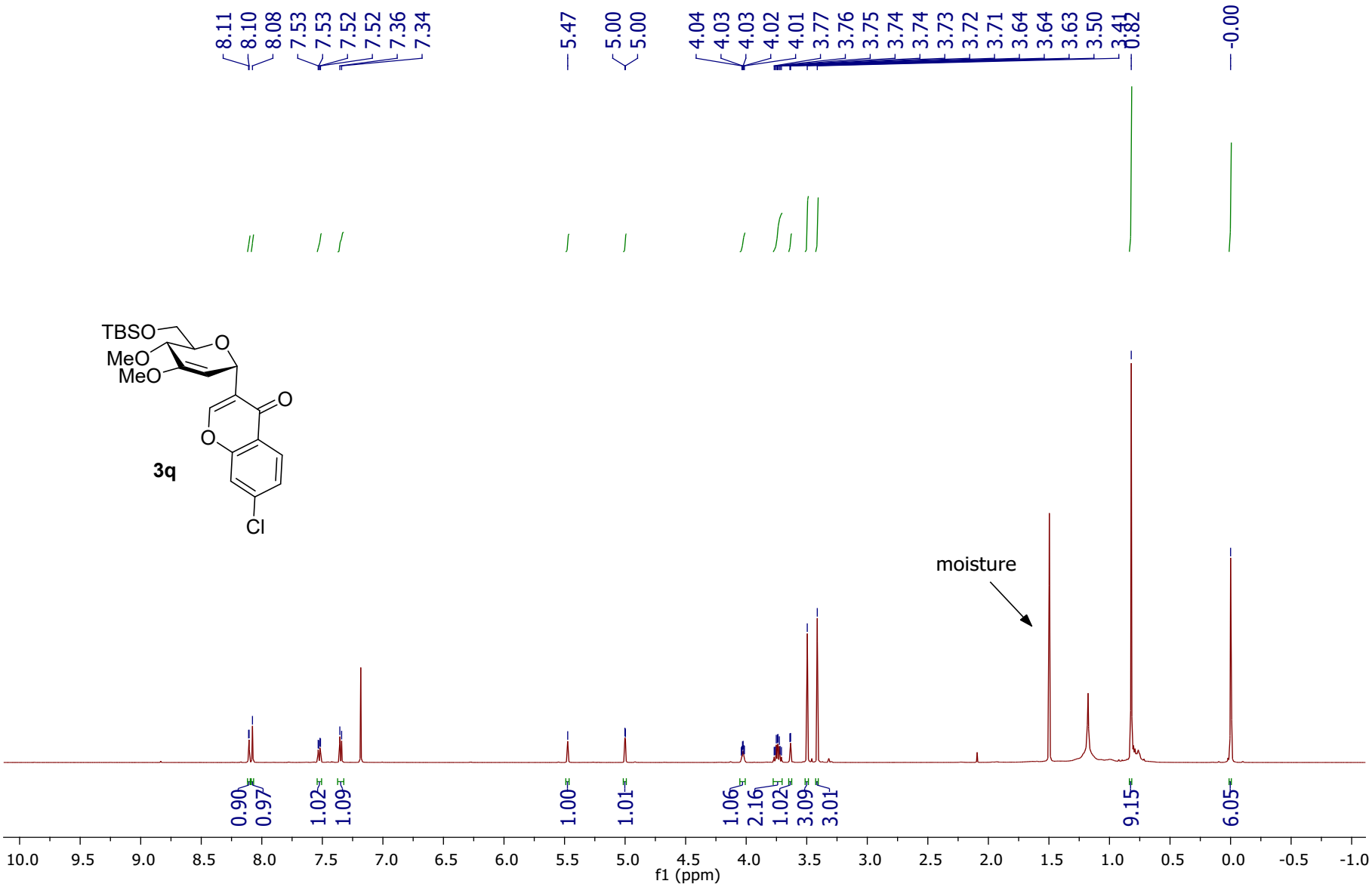
128.42
128.33
128.14
127.93
127.80
127.74
127.70
127.45
125.20
119.97
97.59
74.42
73.46
73.03
71.50
69.15
68.49
66.03
138.18
137.92
136.57
133.91
131.11
128.50
128.42
128.33
128.14
127.93
127.80
127.45
138
136
134
132
f1 (ppm)
128.5
128.0
127.5
f1 (ppm)
BnO
BnO
BnO
O
O
O
Cl
3p
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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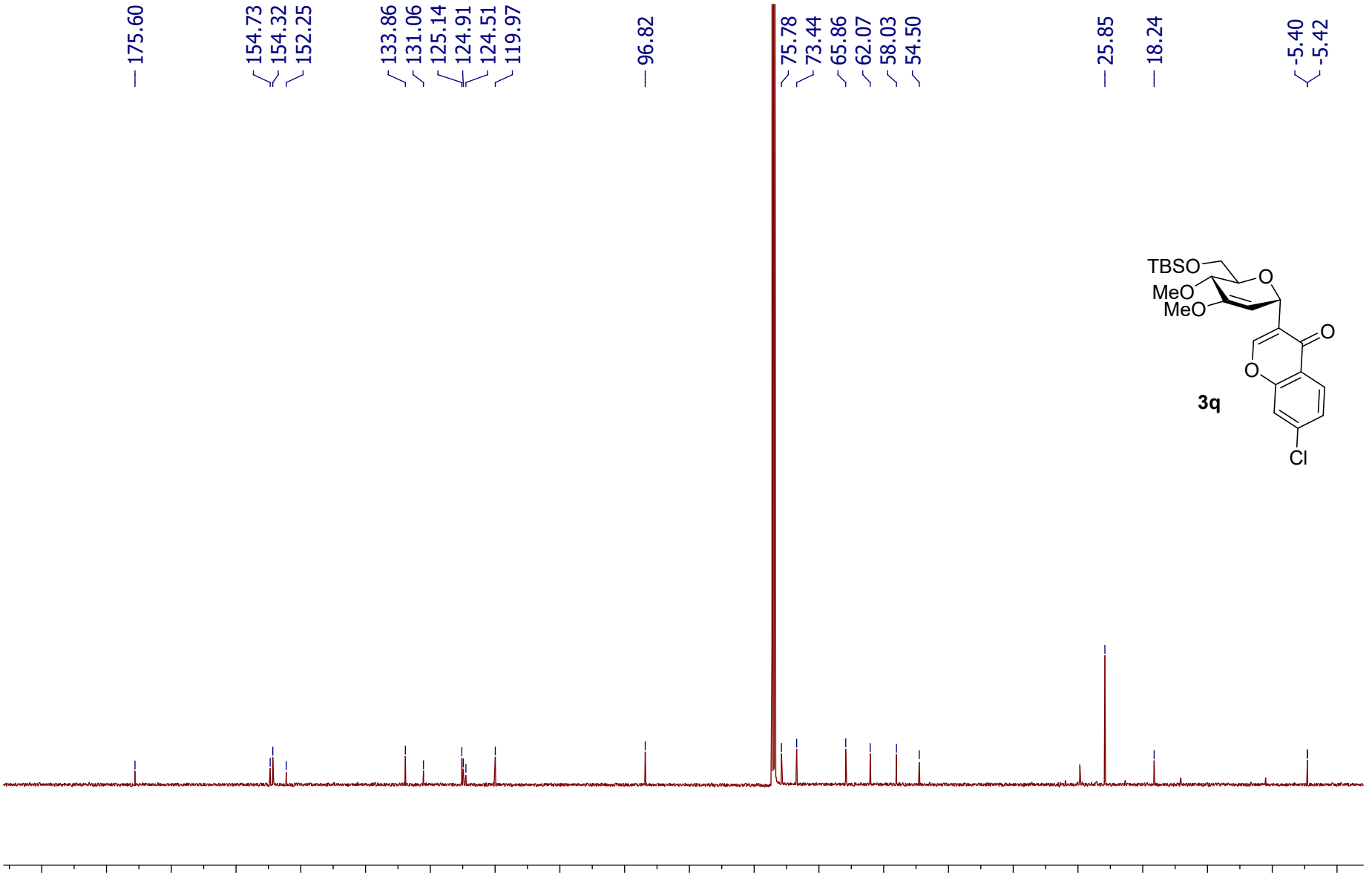
8.10
8.08
7.53
7.53
7.52
7.52
7.36
7.34
5.47
5.00
5.00
4.04
4.03
4.03
4.02
4.01
3.77
3.76
3.75
3.74
3.74
3.73
3.72
3.71
3.64
3.64
3.63
3.50
3.41
0.82
-0.00
TBSO
MeO
MeO
O
O
O
Cl
3q
moisture
0.90
0.97
1.02
1.09
1.00
1.01
1.06
2.16
1.02
3.09
3.01
9.15
6.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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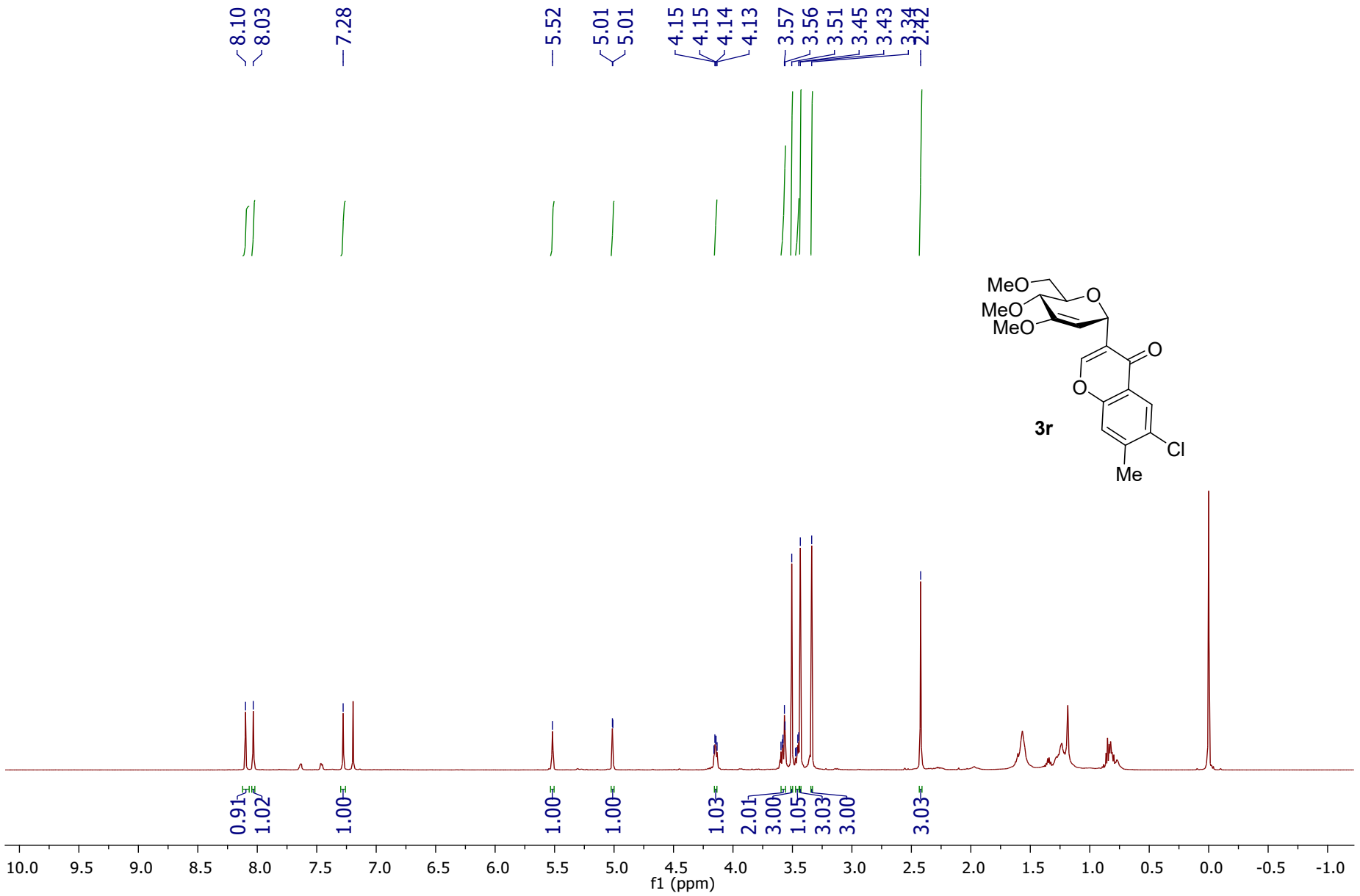
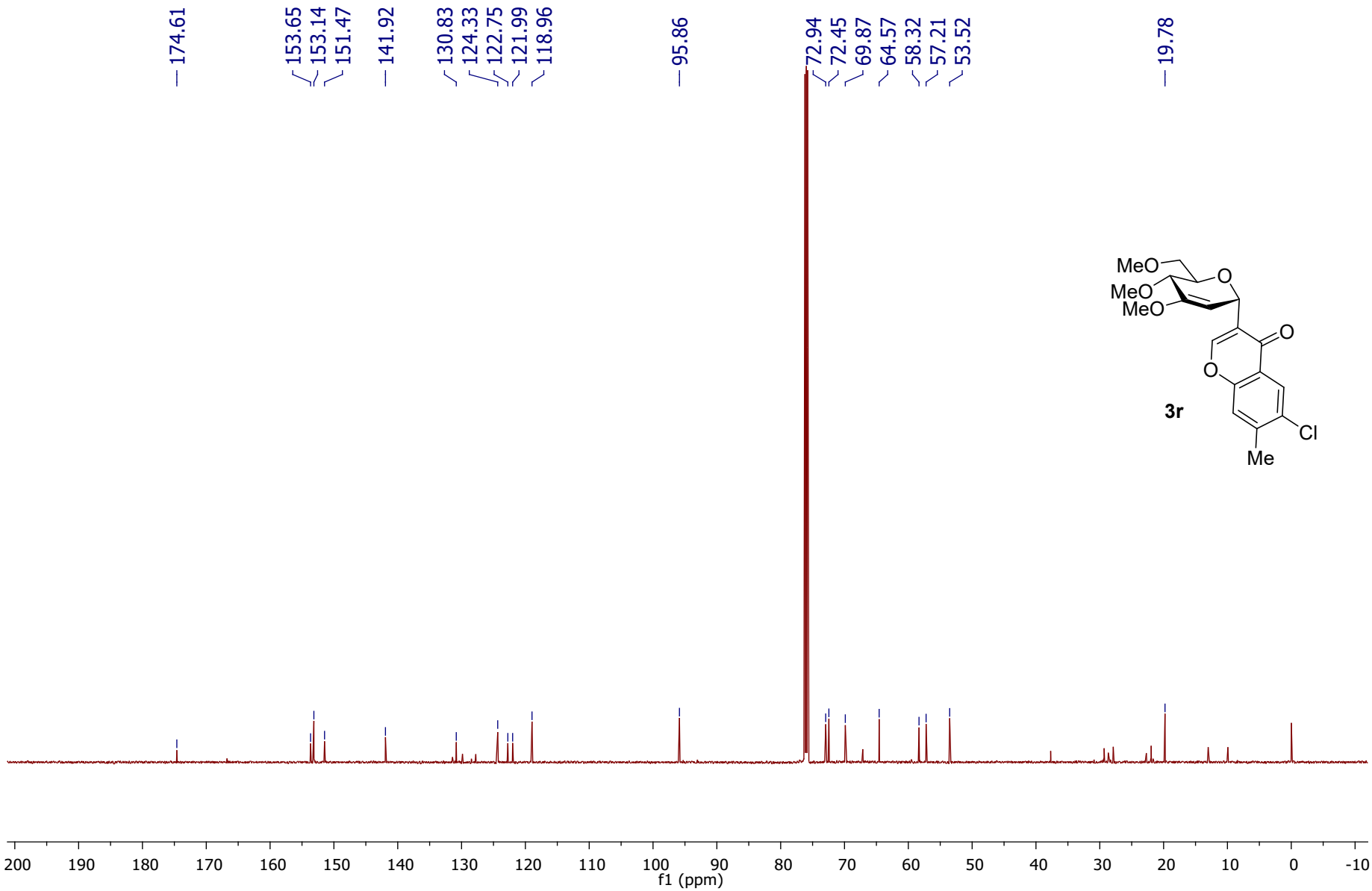
154.73
154.32
152.25
133.86
131.06
125.14
124.91
124.51
119.97
96.82
75.78
73.44
65.86
62.07
58.03
54.50
25.85
18.24
-5.40
-5.42
TBSO
MeO
MeO
O
O
O
Cl
3q
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.10
8.03
7.28
5.52
5.01
5.01
4.15
4.15
4.14
4.13
3.57
3.56
3.51
3.45
3.43
3.34
2.42
MeO
MeO
MeO
O
O
O
Cl
Me
3r
0.91
1.02
1.00
1.00
1.00
1.03
2.01
3.00
1.05
3.03
3.00
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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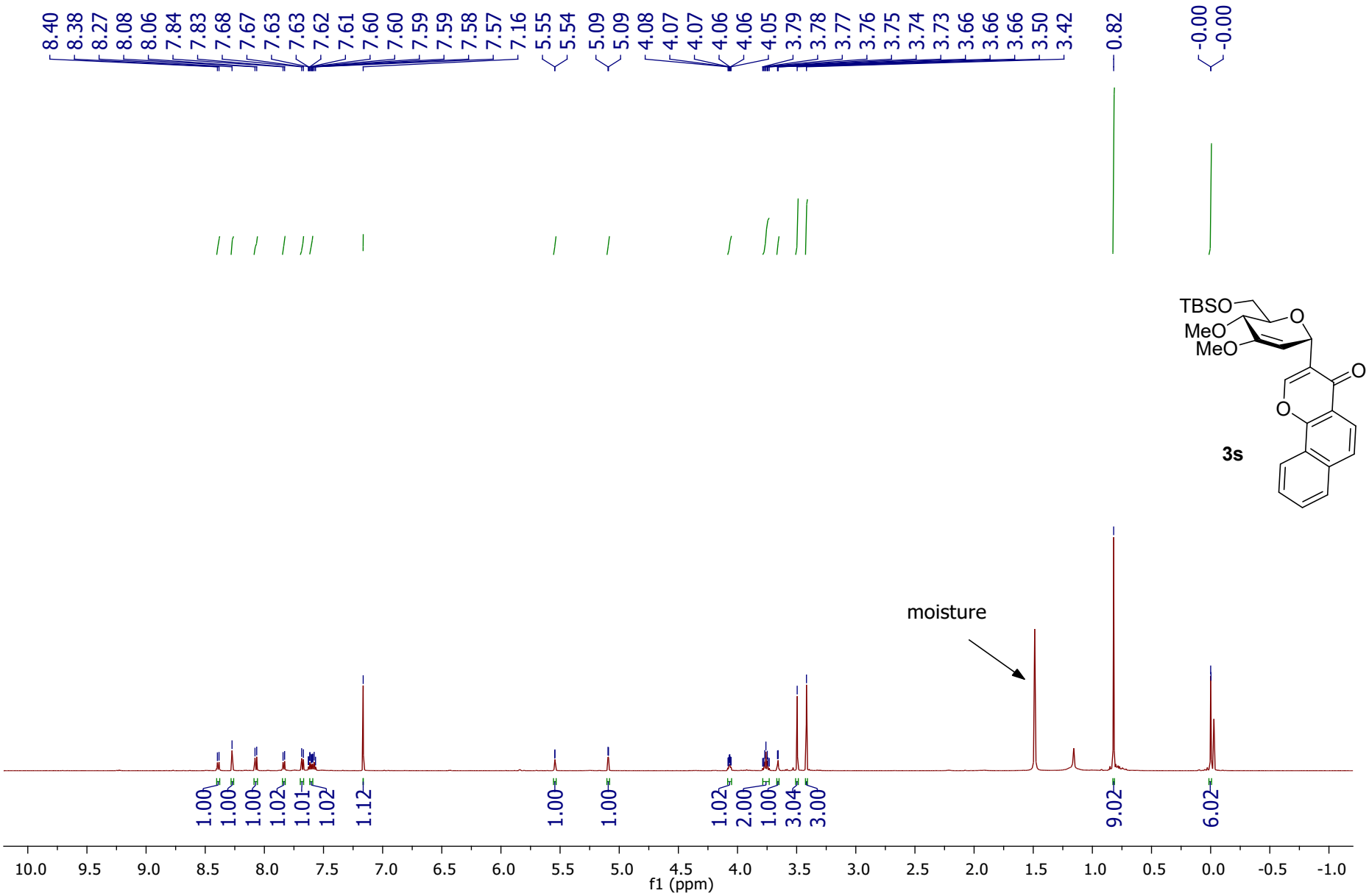
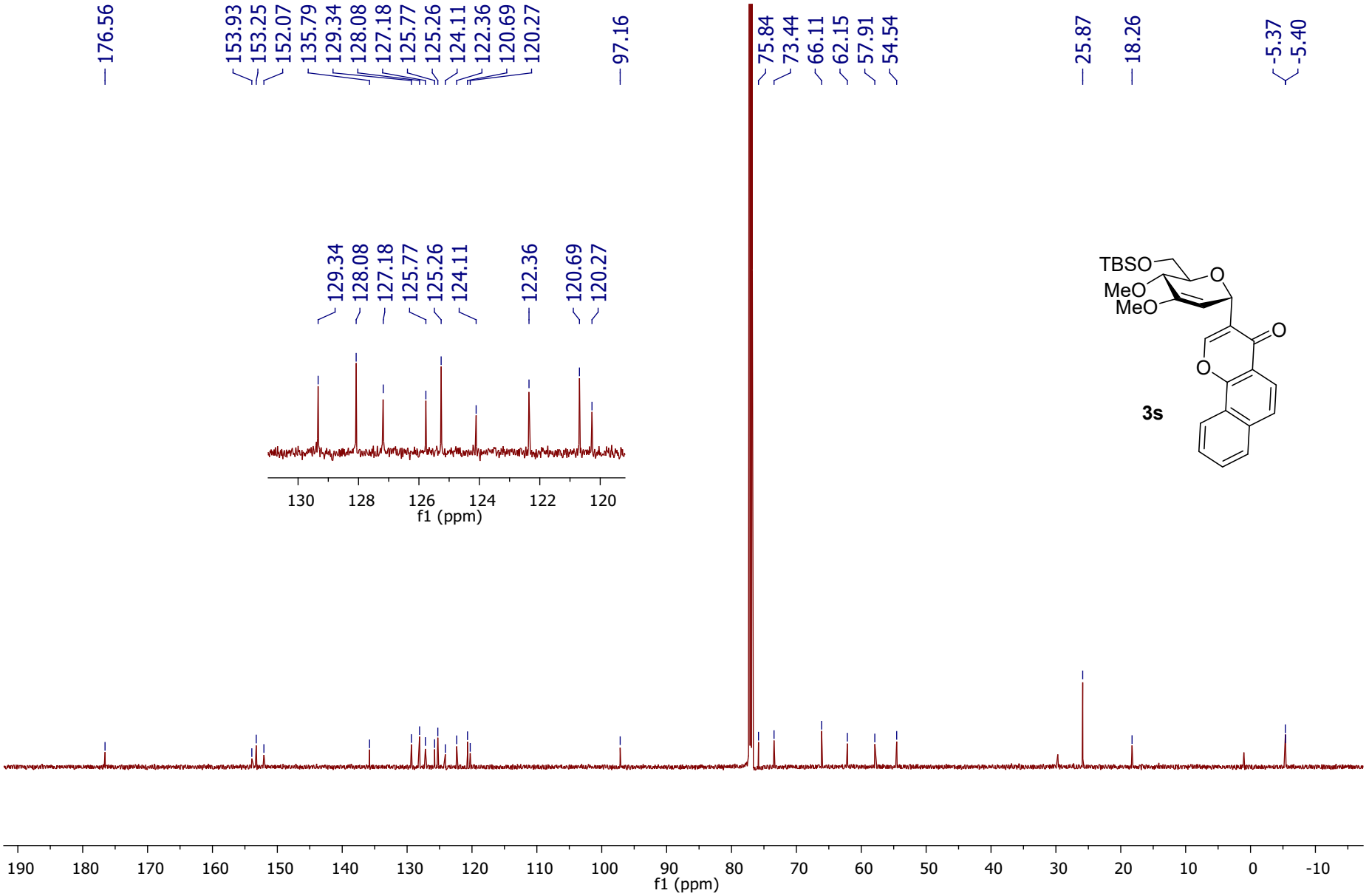
153.65
153.14
151.47
141.92
130.83
124.33
122.75
121.99
118.96
95.86
72.94
72.45
69.87
64.57
58.32
57.21
53.52
19.78
MeO
MeO
MeO
O
O
O
Cl
Me
3r
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.40
8.38
8.27
8.08
8.06
7.84
7.83
7.68
7.67
7.63
7.63
7.62
7.61
7.60
7.60
7.59
7.59
7.58
7.57
7.16
5.55
5.54
5.09
5.09
4.08
4.07
4.07
4.06
4.06
4.05
3.79
3.78
3.77
3.76
3.75
3.74
3.73
3.66
3.66
3.66
3.50
3.42
0.82
-0.00
-0.00
TBSO
MeO
MeO
O
O
O
3s
moisture
1.00
1.00
1.00
1.02
1.01
1.02
1.12
1.00
1.00
1.02
2.00
1.00
3.04
3.00
9.02
6.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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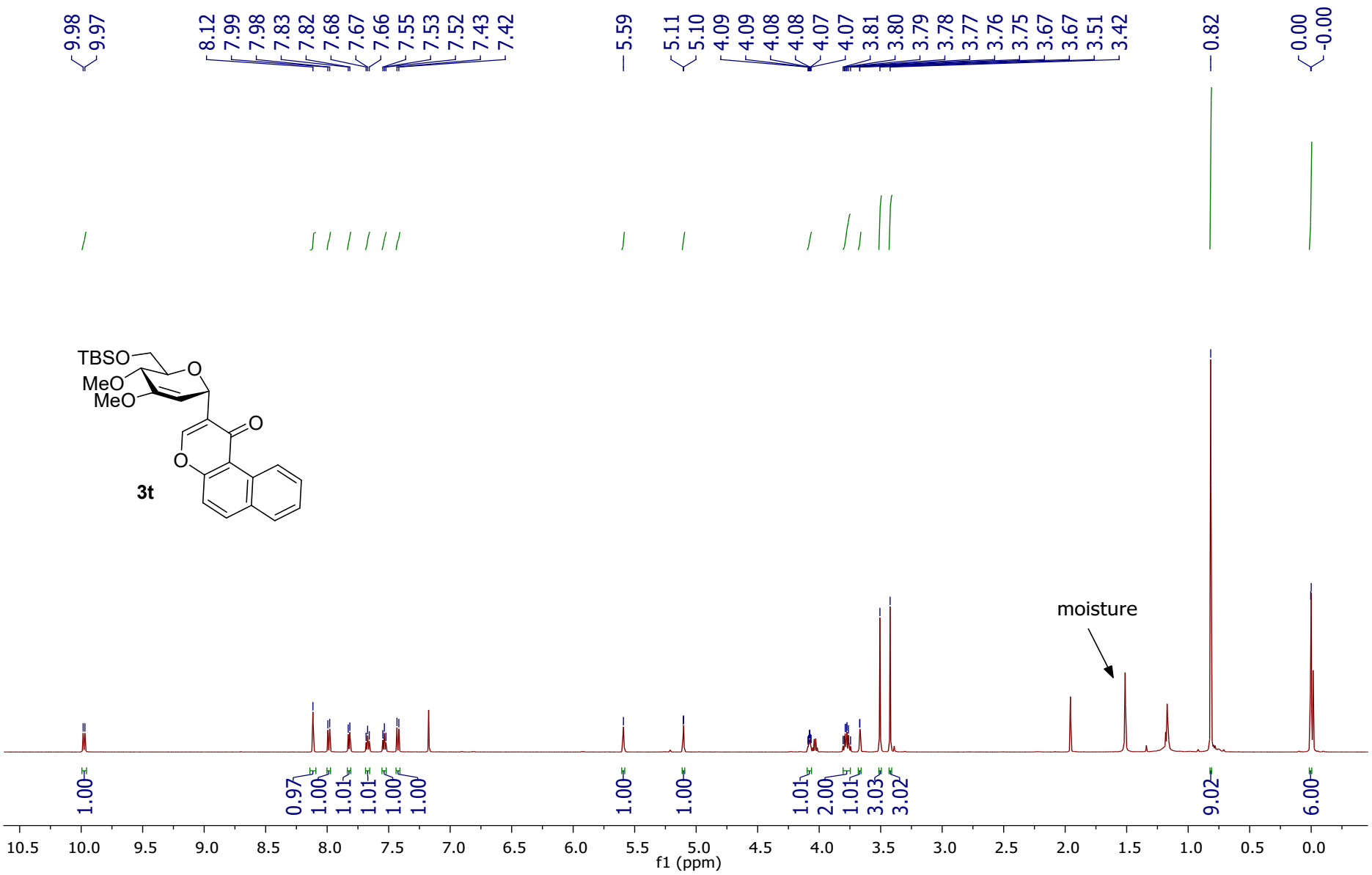
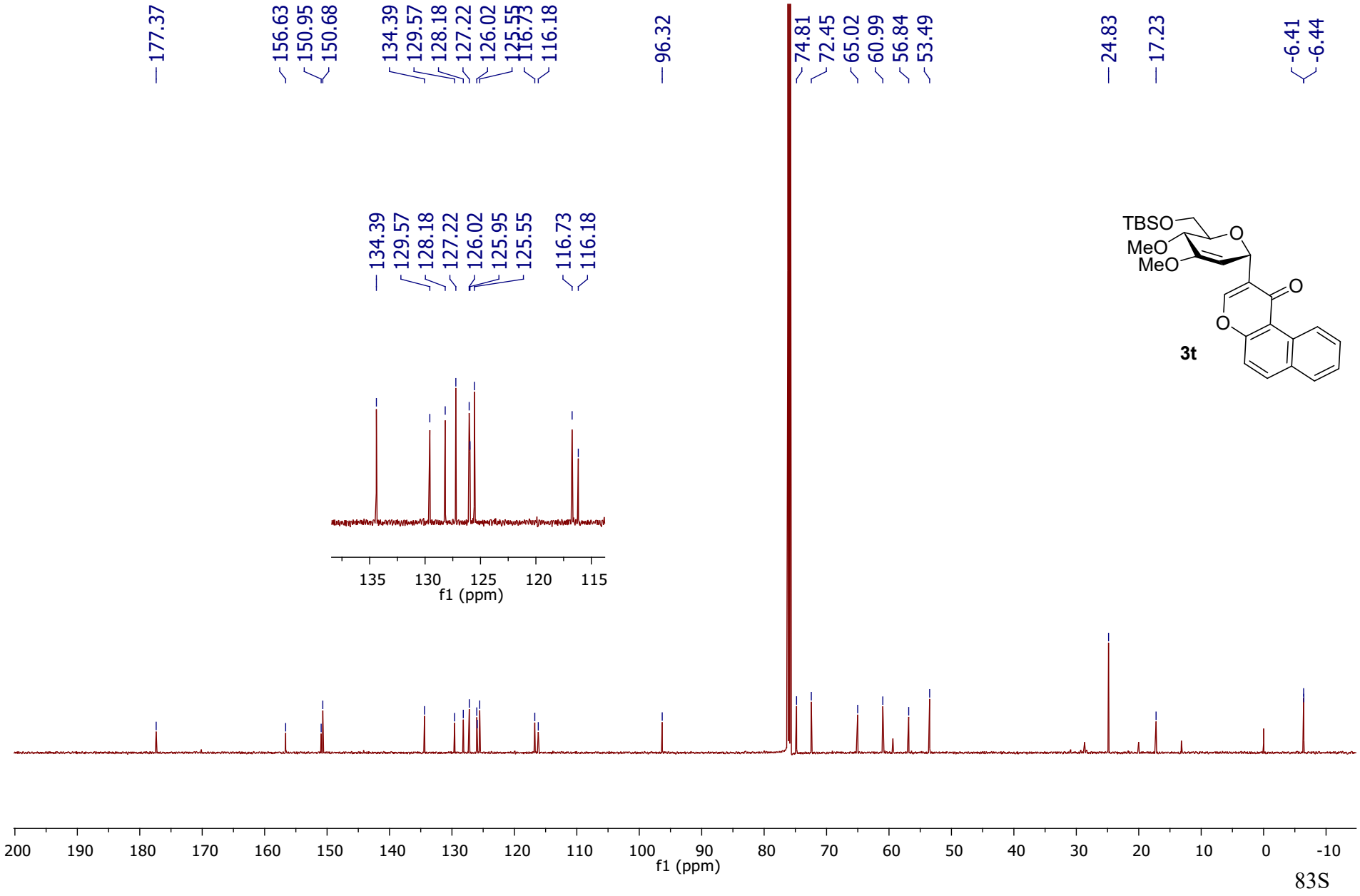
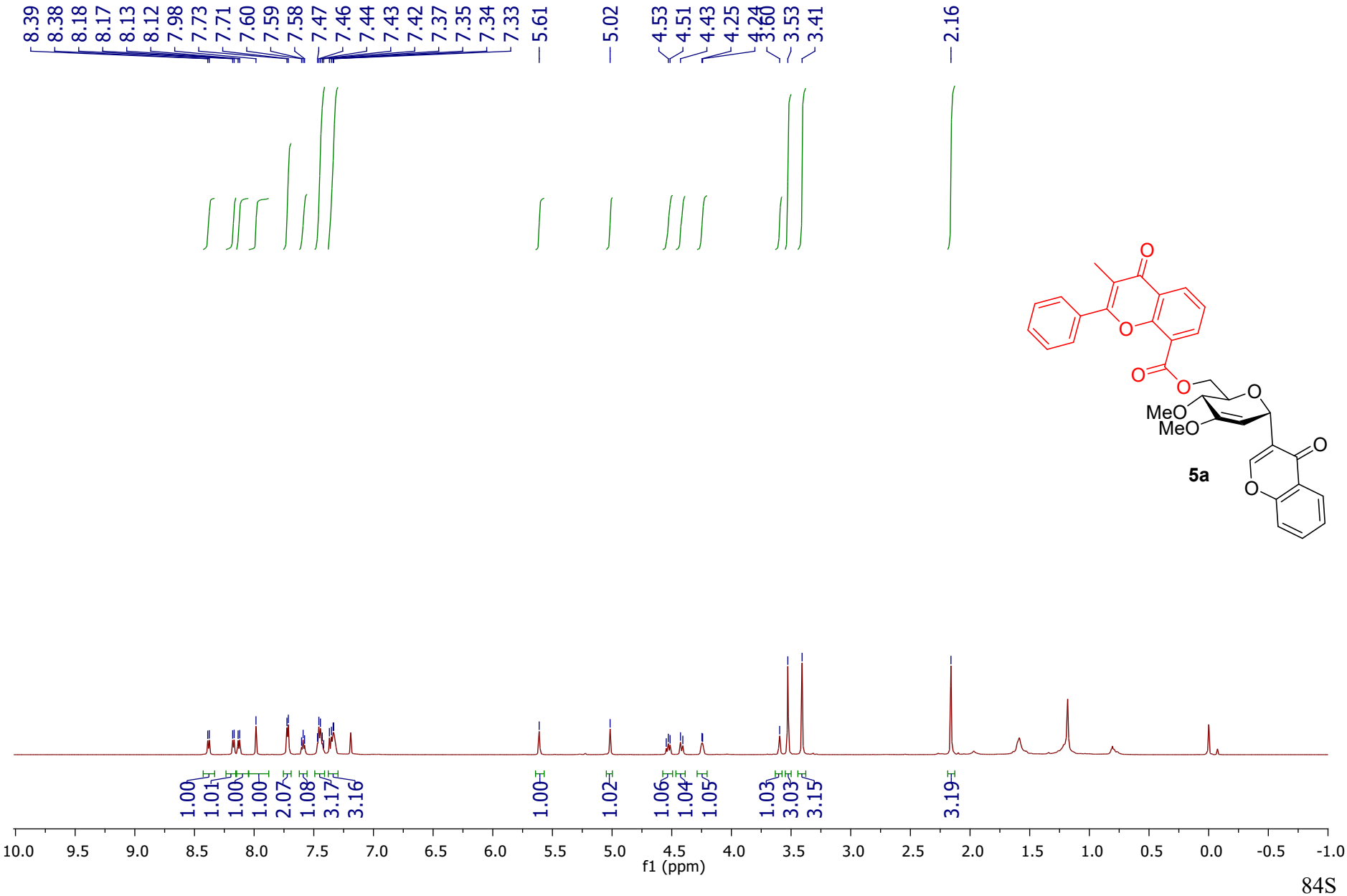
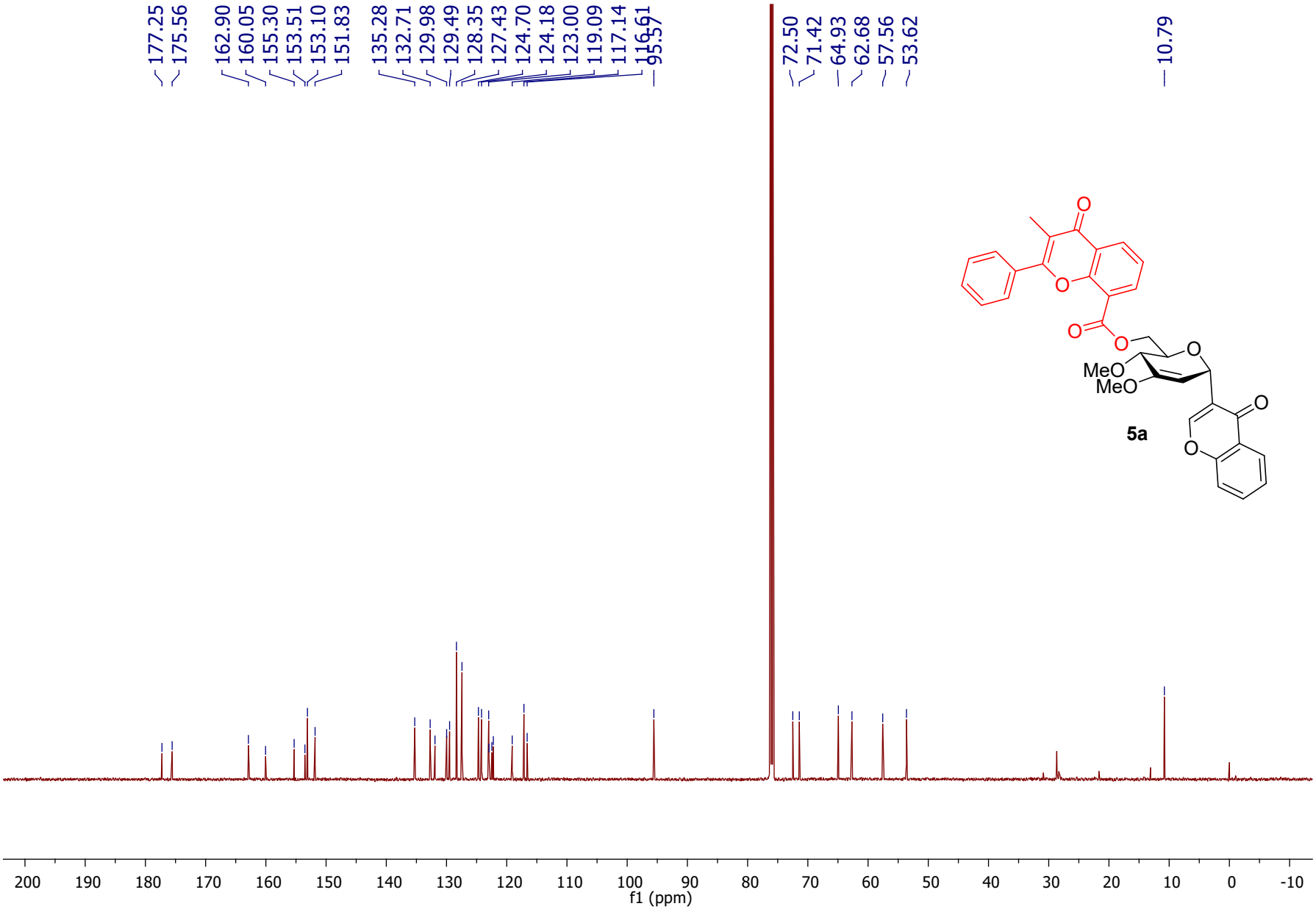
153.93
153.25
152.07
135.79
129.34
128.08
127.18
125.77
125.26
124.11
122.36
120.69
120.27
97.16
75.84
73.44
66.11
62.15
57.91
54.54
25.87
18.26
-5.37
-5.40
TBSO
MeO
MeO
O
O
O
3s
f1 (ppm)


9.98
9.97
8.12
7.99
7.98
7.83
7.82
7.68
7.67
7.66
7.55
7.53
7.52
7.43
7.42
5.59
5.11
5.10
4.09
4.09
4.08
4.08
4.07
4.07
3.81
3.80
3.79
3.78
3.77
3.76
3.75
3.67
3.67
3.51
3.42
0.82
0.00
-0.00
TBSO
MeO
MeO
O
O
O
3t
moisture
1.00
0.97
1.00
1.01
1.01
1.00
1.00
1.00
1.00
1.01
2.00
1.01
3.03
3.02
9.02
6.00
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


177.37
156.63
150.95
150.68
134.39
129.57
128.18
127.22
126.02
125.95
125.55
116.73
116.18
96.32
74.81
72.45
65.02
60.99
56.84
53.49
24.83
17.23
-6.41
-6.44
TBSO
MeO
MeO
O
O
O
3t
f1 (ppm)
f1 (ppm)


8.39
8.38
8.18
8.17
8.13
8.12
7.98
7.73
7.71
7.60
7.59
7.58
7.47
7.46
7.44
7.43
7.42
7.37
7.35
7.34
7.33
5.61
5.02
4.53
4.51
4.43
4.25
4.24
3.60
3.53
3.41
2.16
MeO
MeO
5a
1.00
1.01
1.00
1.00
2.07
1.08
3.17
3.16
1.00
1.02
1.06
1.04
1.05
1.03
3.03
3.15
3.19
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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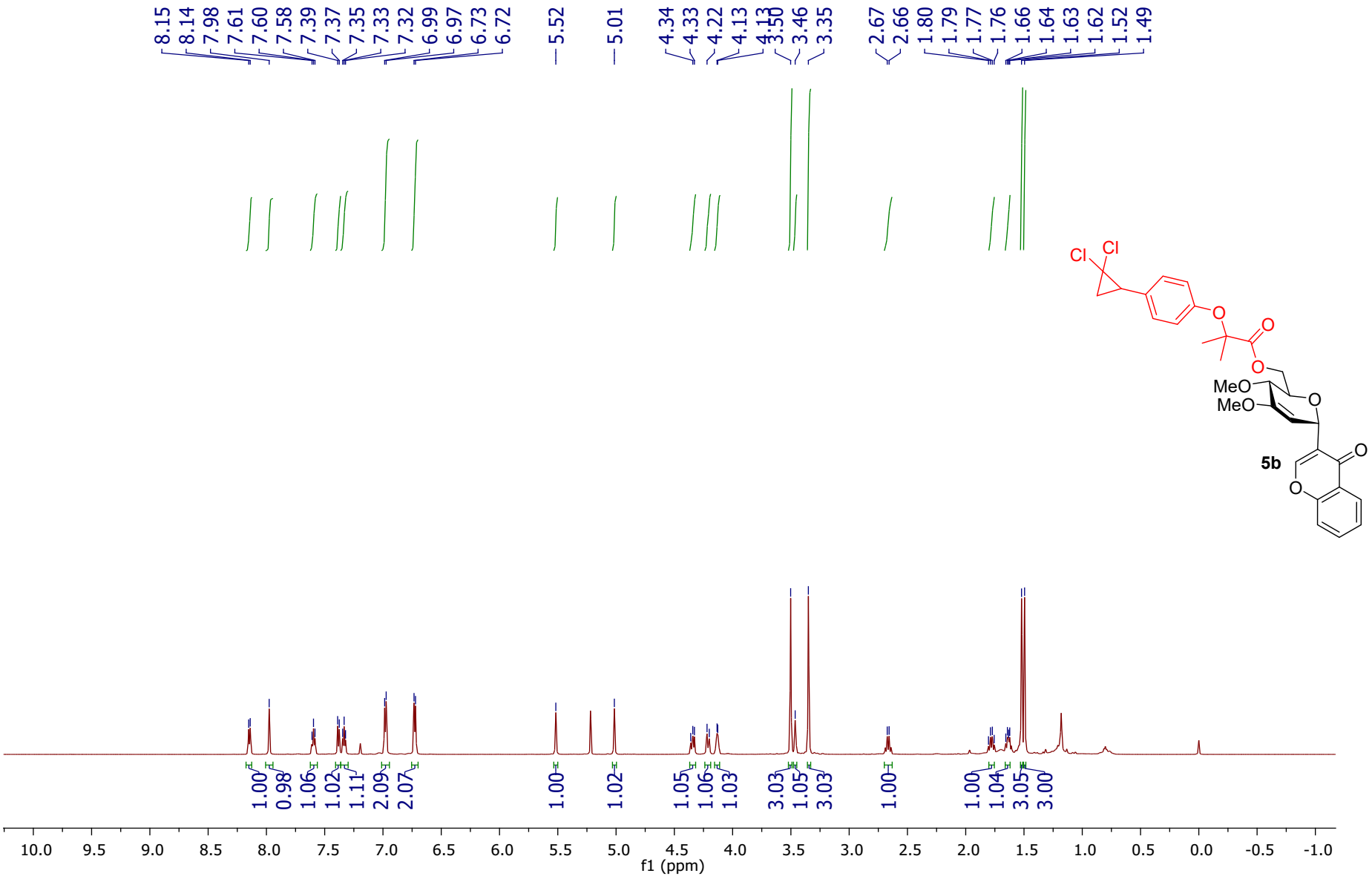
175.56
162.90
160.05
155.30
153.51
153.10
151.83
135.28
132.71
129.98
129.49
128.35
127.43
124.70
124.18
123.00
119.09
117.14
116.61
95.57
72.50
71.42
64.93
62.68
57.56
53.62
10.79
MeO
MeO
5a
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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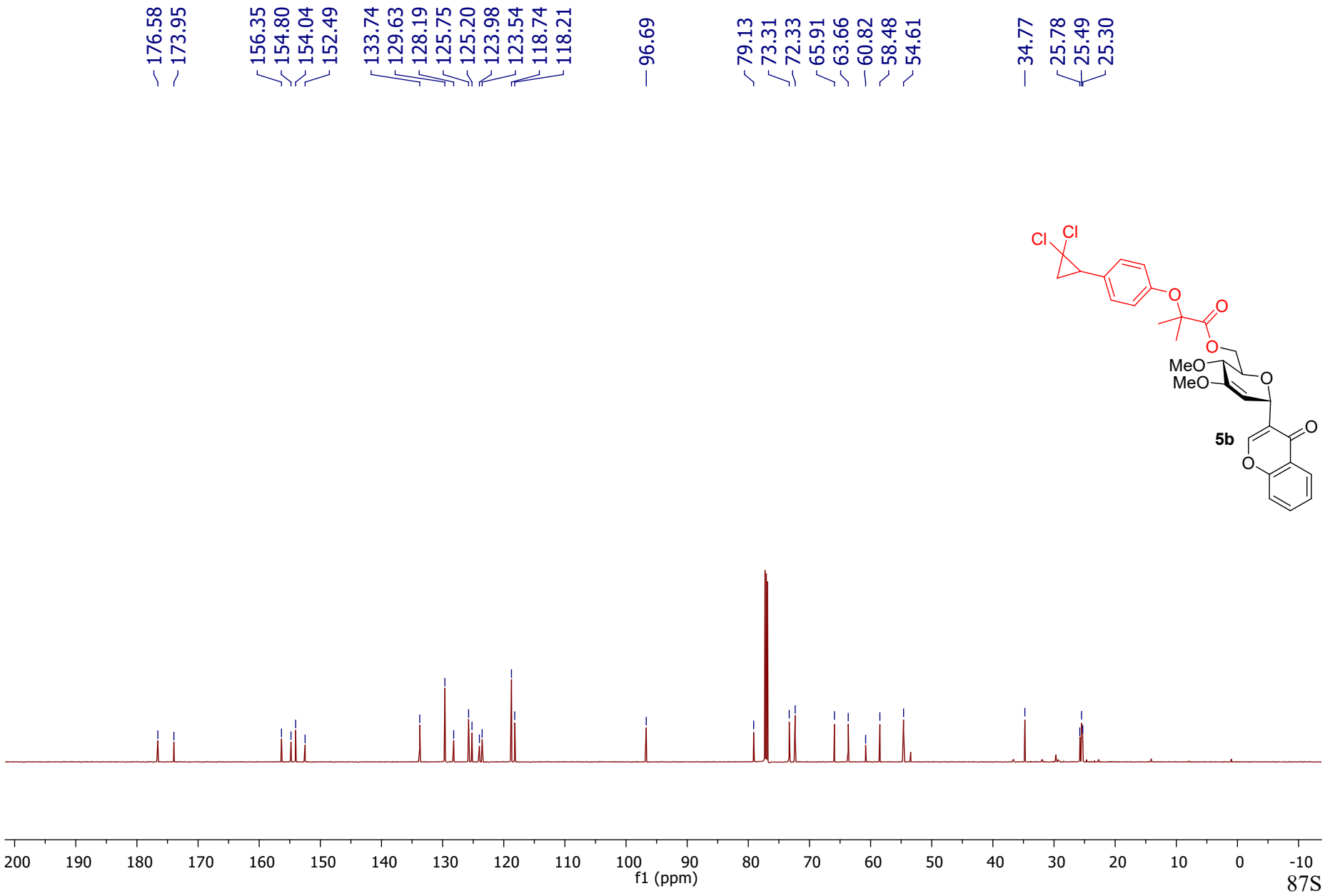
8.14
7.98
7.61
7.60
7.58
7.39
7.37
7.35
7.33
7.32
6.99
6.97
6.73
6.72
5.52
5.01
4.34
4.33
4.22
4.13
4.13
3.50
3.46
3.35
2.67
2.66
1.80
1.79
1.77
1.76
1.66
1.64
1.63
1.62
1.52
1.49
Cl
Cl
O
O
O
MeO
MeO
O
O
O
5b
1.00
0.98
1.06
1.02
1.11
2.09
2.07
1.00
1.02
1.05
1.06
1.03
3.03
1.05
3.03
1.00
1.00
1.04
3.05
3.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


176.58
173.95
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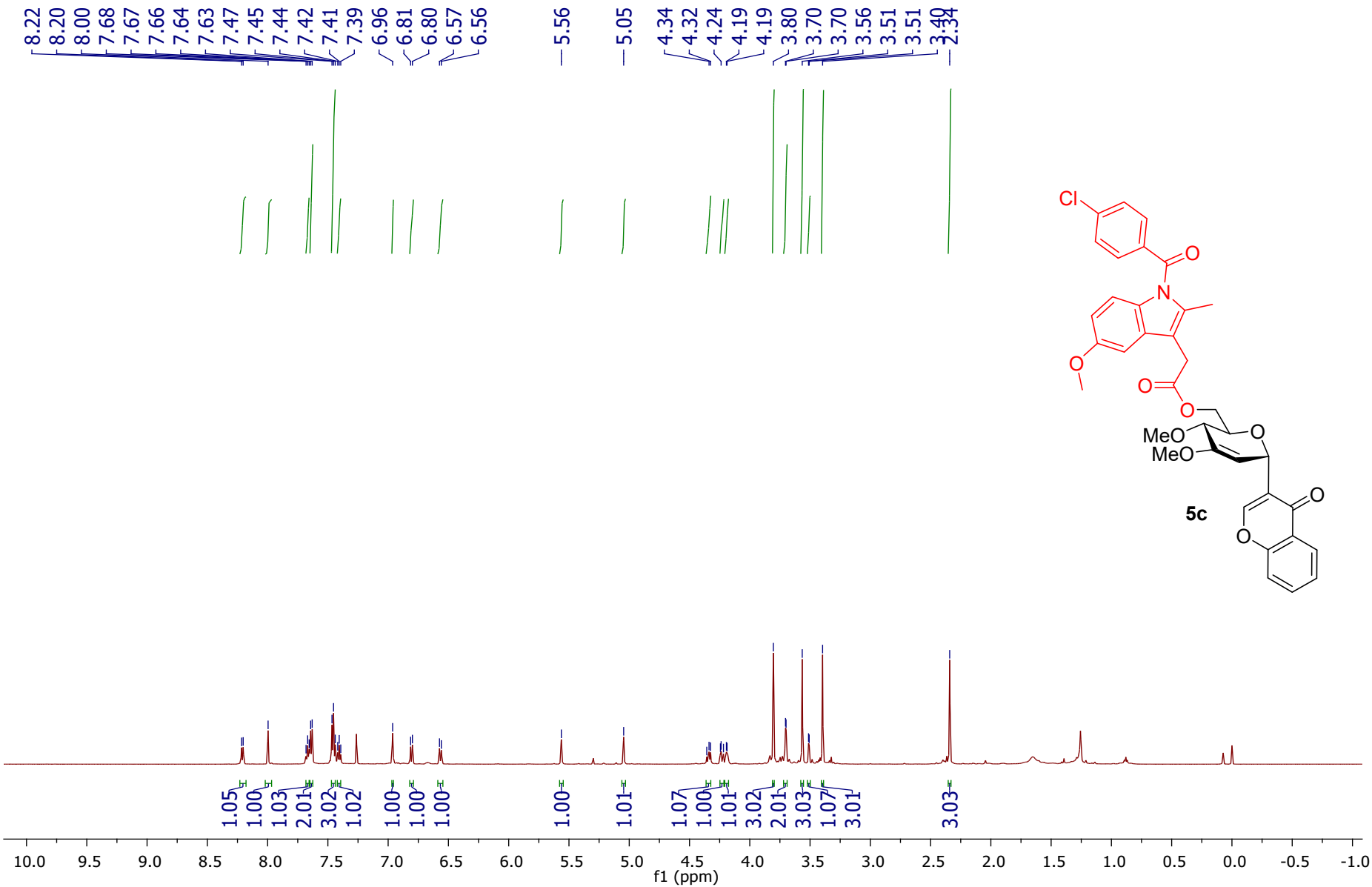
154.80
154.04
152.49
133.74
129.63
128.19
125.75
125.20
123.98
123.54
118.74
118.21
96.69
79.13
73.31
72.33
65.91
63.66
60.82
58.48
54.61
34.77
25.78
25.49
25.30
Cl
Cl
O
O
O
MeO
MeO
O
O
O
5b
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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8.20
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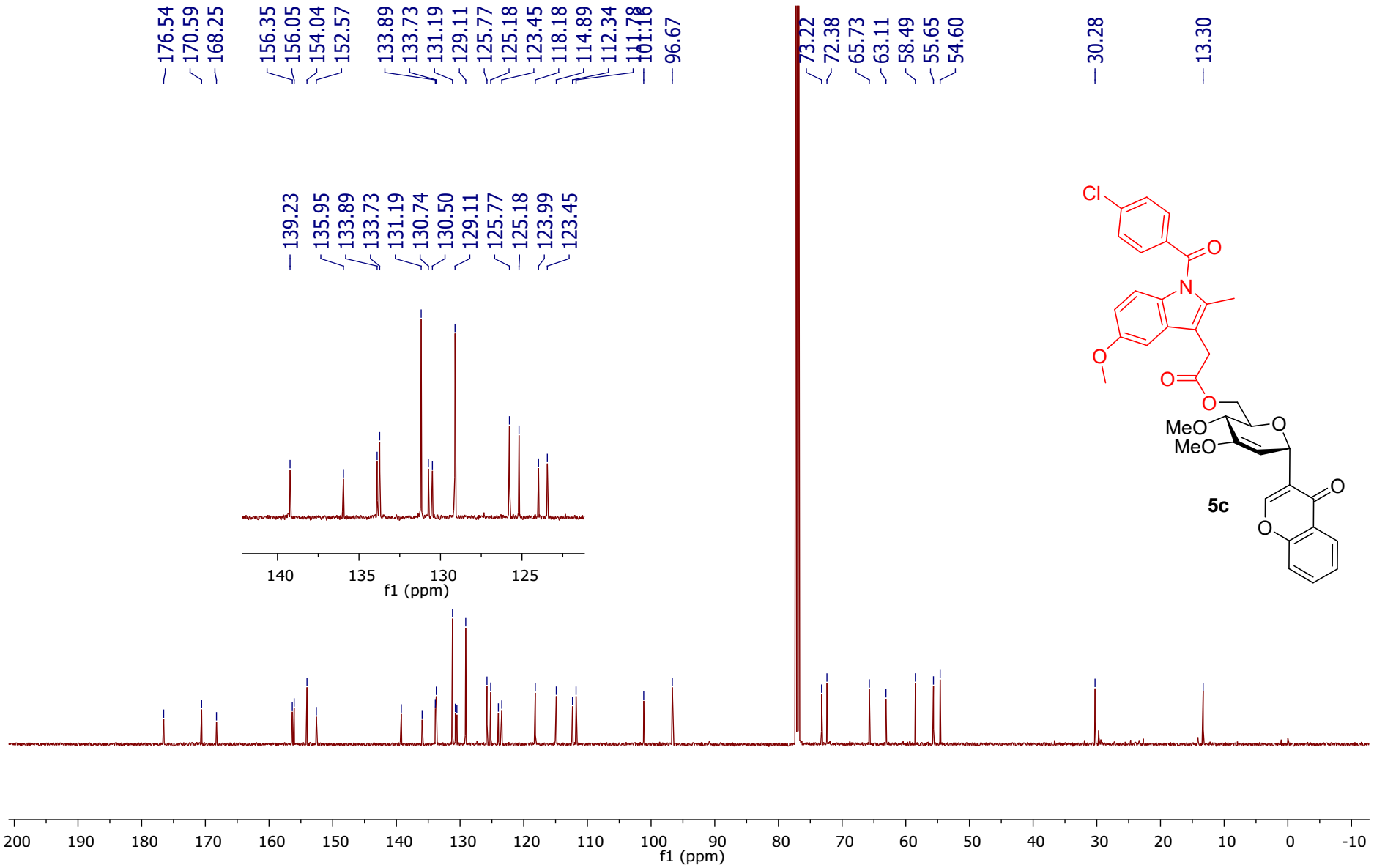
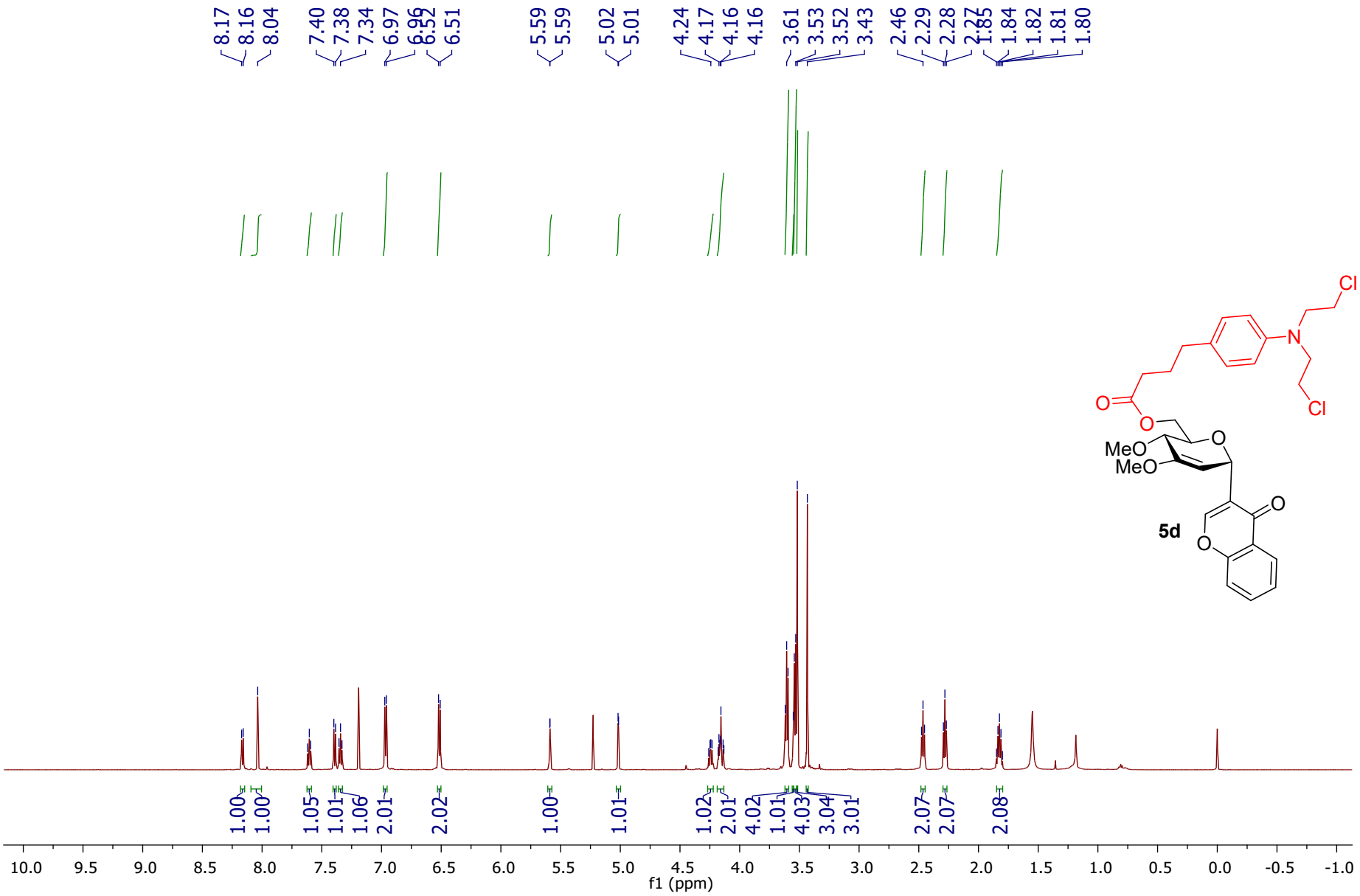
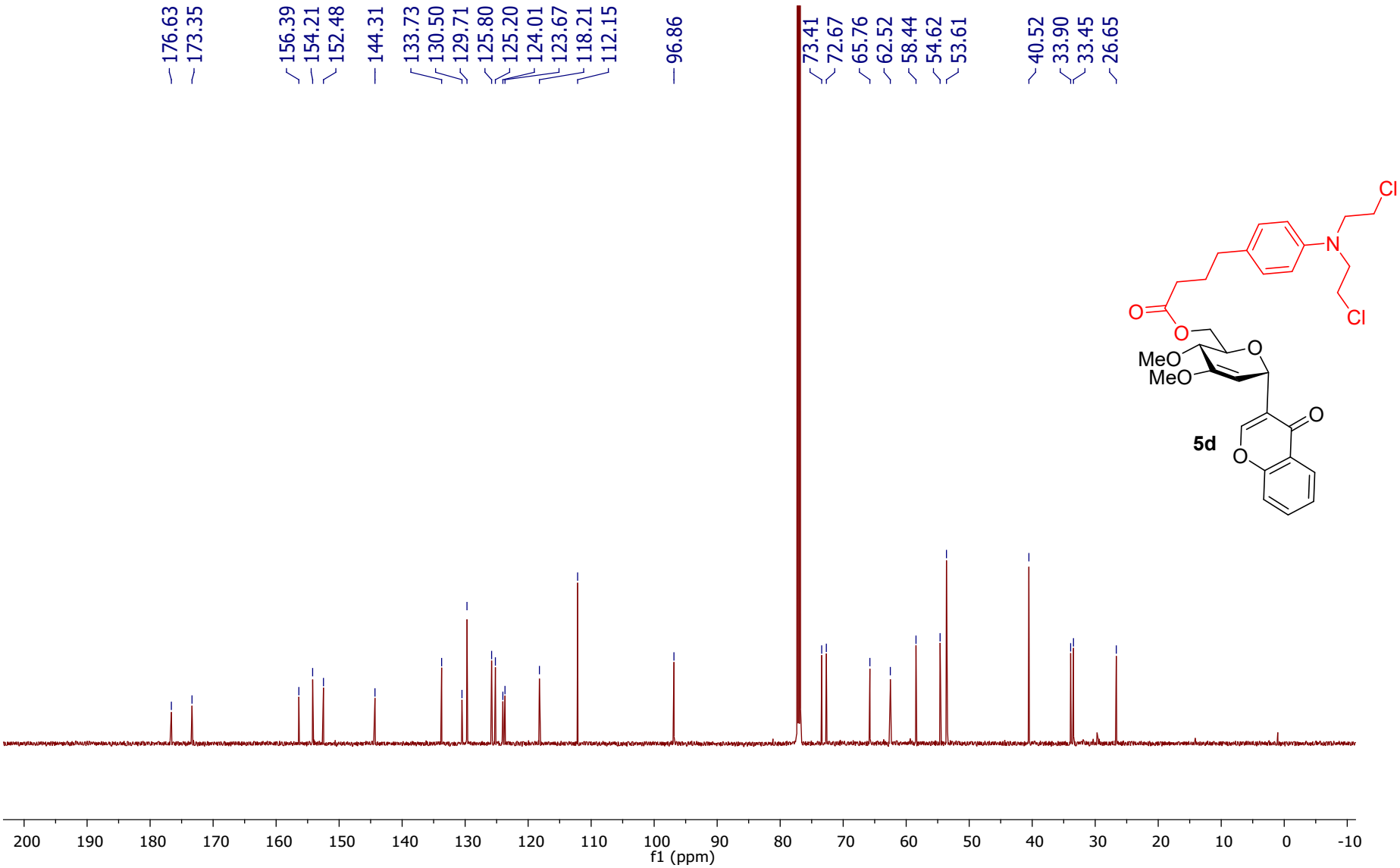
7.67
7.66
7.64
7.63
7.47
7.45
7.44
7.42
7.41
7.39
6.96
6.81
6.80
6.57
6.56
5.56
5.05
4.34
4.32
4.24
4.19
4.19
3.80
3.70
3.70
3.56
3.51
3.51
3.40
2.34
Cl
O
N
O
O
O
MeO
MeO
O
O
O
5c
1.05
1.00
1.03
2.01
3.02
1.02
1.00
1.00
1.00
1.00
1.01
1.07
1.00
1.01
3.02
2.01
3.03
1.07
3.01
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


176.54
170.59
168.25
156.35
156.05
154.04
152.57
133.89
133.73
131.19
129.11
125.77
125.18
123.45
118.18
114.89
112.34
111.78
101.16
96.67
73.22
72.38
65.73
63.11
58.49
55.65
54.60
30.28
13.30
139.23
135.95
133.89
133.73
131.19
130.74
130.50
129.11
125.77
125.18
123.99
123.45
140
135
130
125
f1 (ppm)
Cl
O
N
O
O
O
O
MeO
MeO
O
O
O
5c
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.17
8.16
8.04
7.40
7.38
7.34
6.97
6.96
6.52
6.51
5.59
5.59
5.02
5.01
4.24
4.17
4.16
4.16
3.61
3.53
3.52
3.43
2.46
2.29
2.28
2.27
1.85
1.84
1.82
1.81
1.80
Cl
N
Cl
O
O
O
MeO
MeO
O
O
5d
1.00
1.00
1.05
1.01
1.06
2.01
2.02
1.00
1.01
1.02
2.01
4.02
1.01
4.03
3.04
3.01
2.07
2.07
2.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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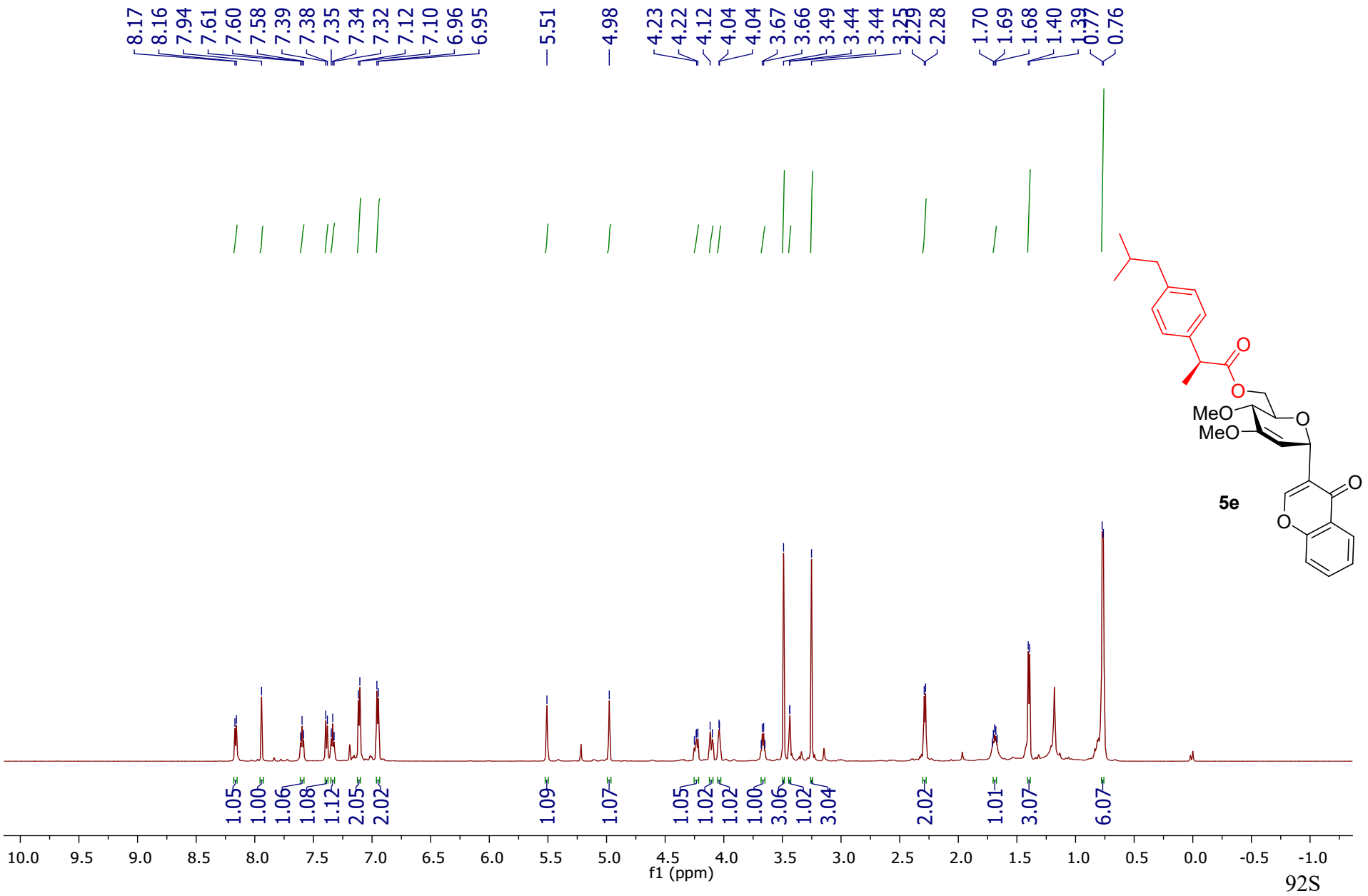
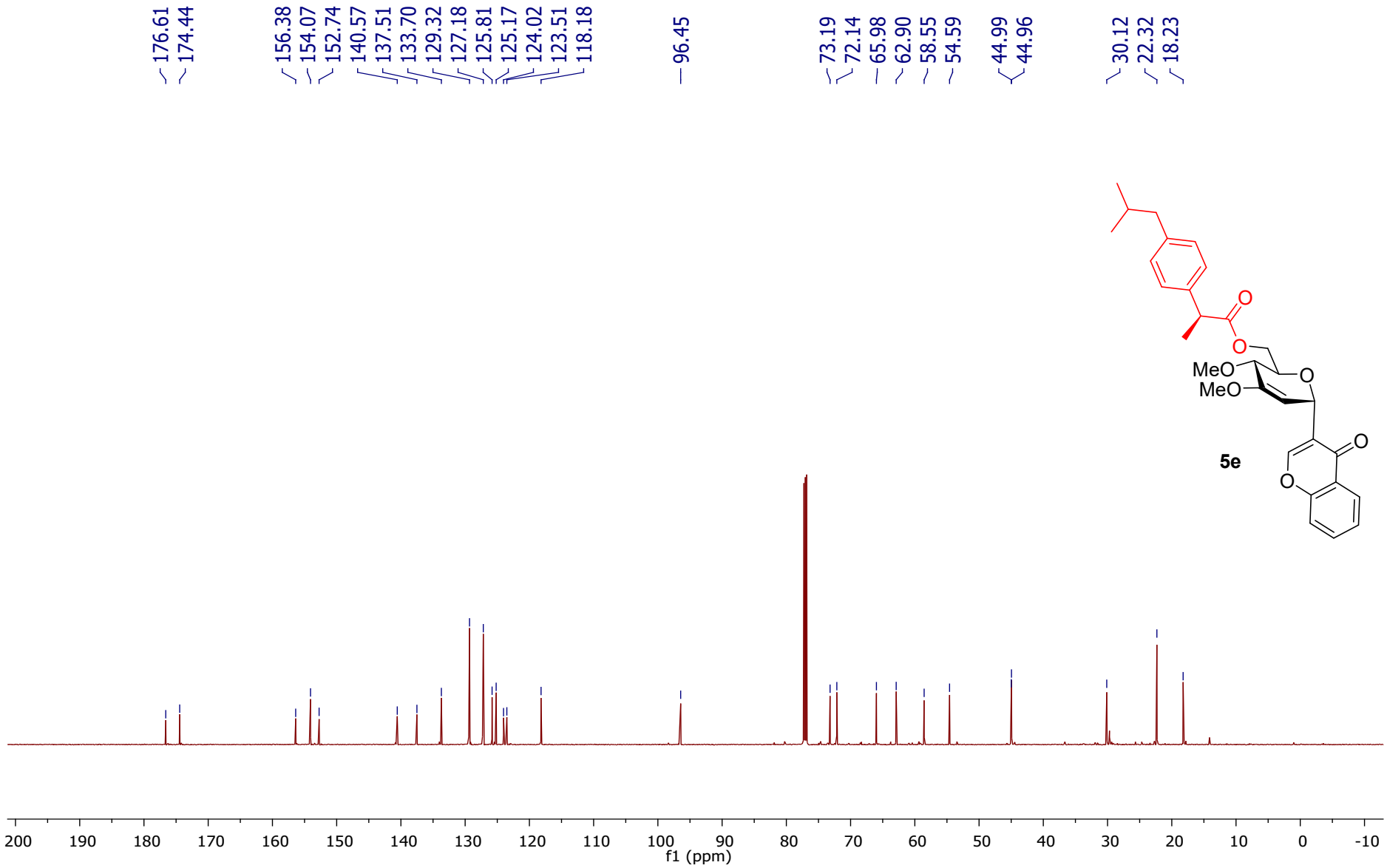
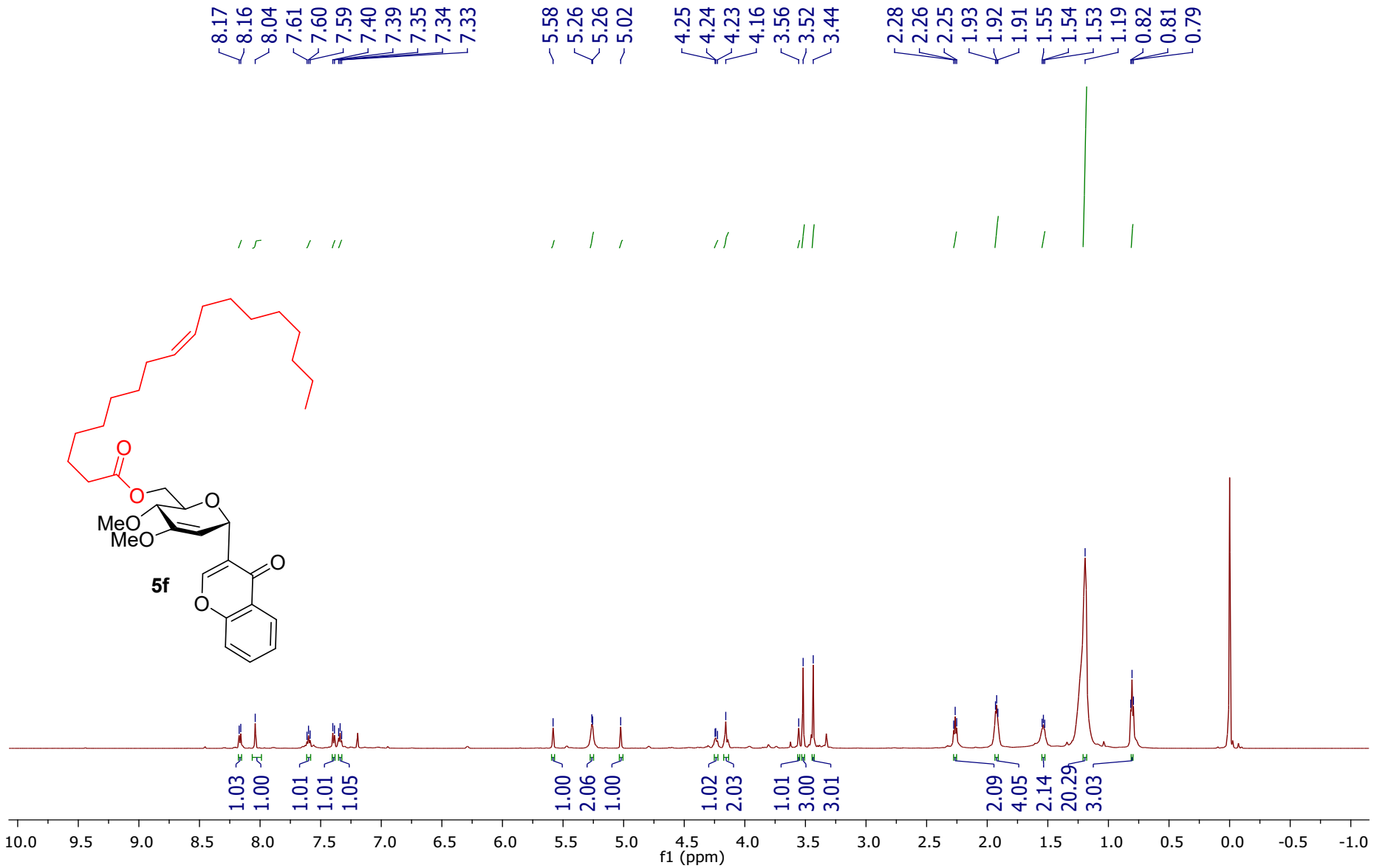
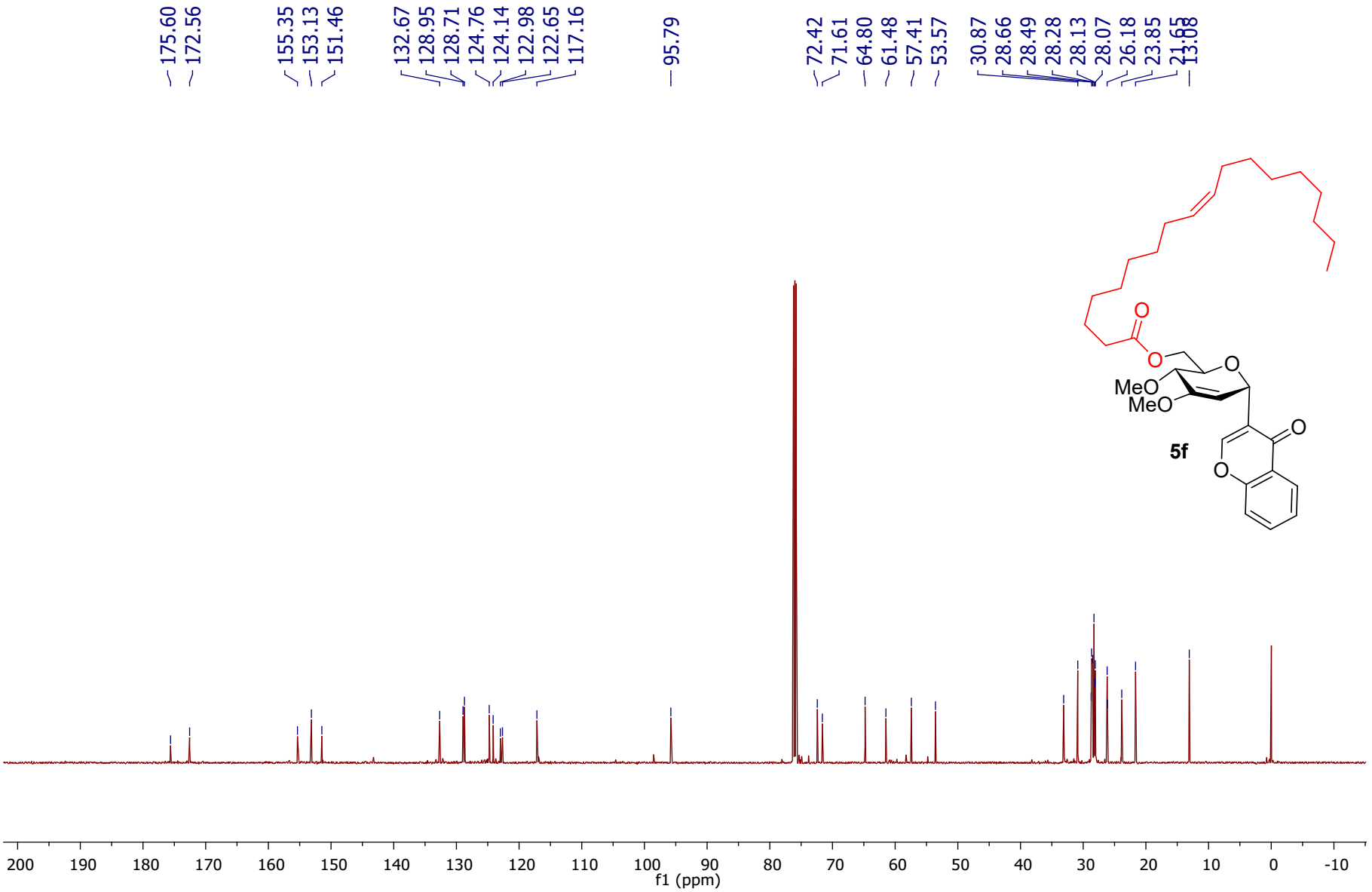
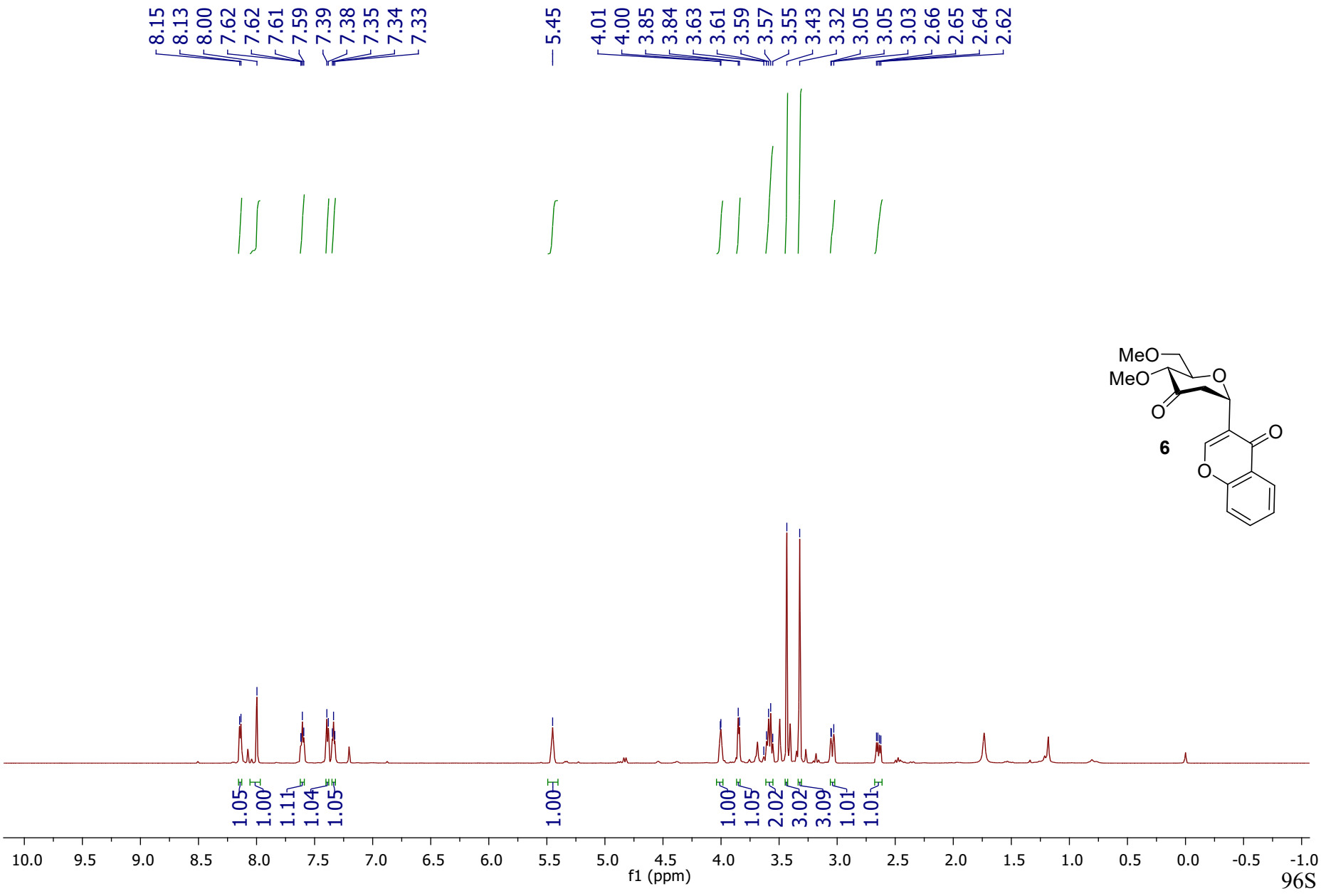
173.35
156.39
154.21
152.48
144.31
133.73
130.50
129.71
125.80
125.20
124.01
123.67
118.21
112.15
96.86
73.41
72.67
65.76
62.52
58.44
54.62
53.61
40.52
33.90
33.45
26.65
Cl
N
Cl
O
O
MeO
MeO
O
O
O
5d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

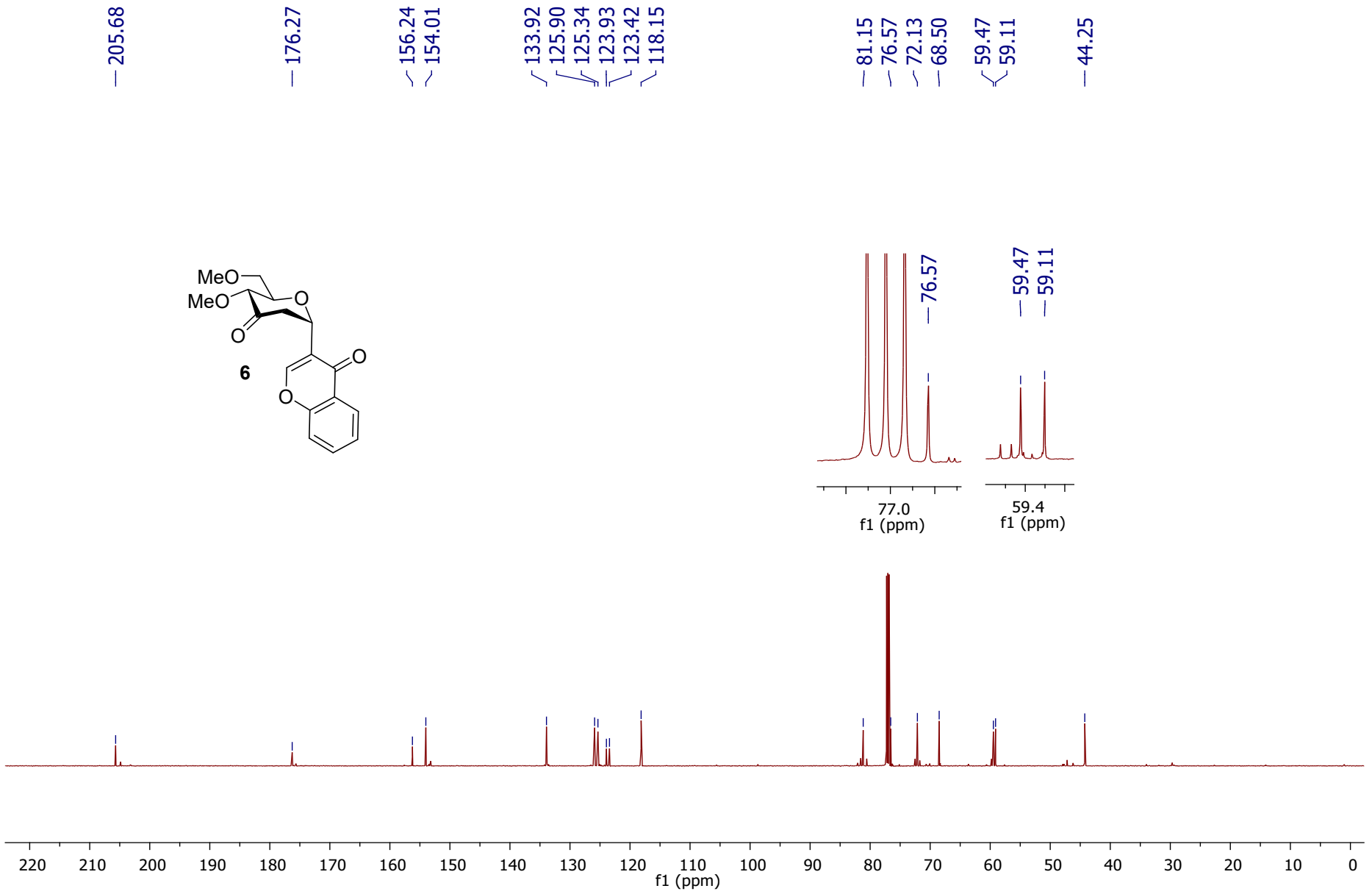

8.17
8.16
7.94
7.61
7.60
7.58
7.39
7.38
7.35
7.34
7.32
7.12
7.10
6.96
6.95
5.51
4.98
4.23
4.22
4.12
4.04
4.04
3.67
3.66
3.49
3.44
3.44
3.25
2.29
2.28
1.70
1.69
1.68
1.40
1.39
0.77
0.76
MeO
MeO
O
O
O
O
O
5e
1.05
1.00
1.06
1.08
1.12
2.05
2.02
1.09
1.07
1.05
1.02
1.02
1.00
3.06
1.02
3.04
2.02
1.01
3.07
6.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)

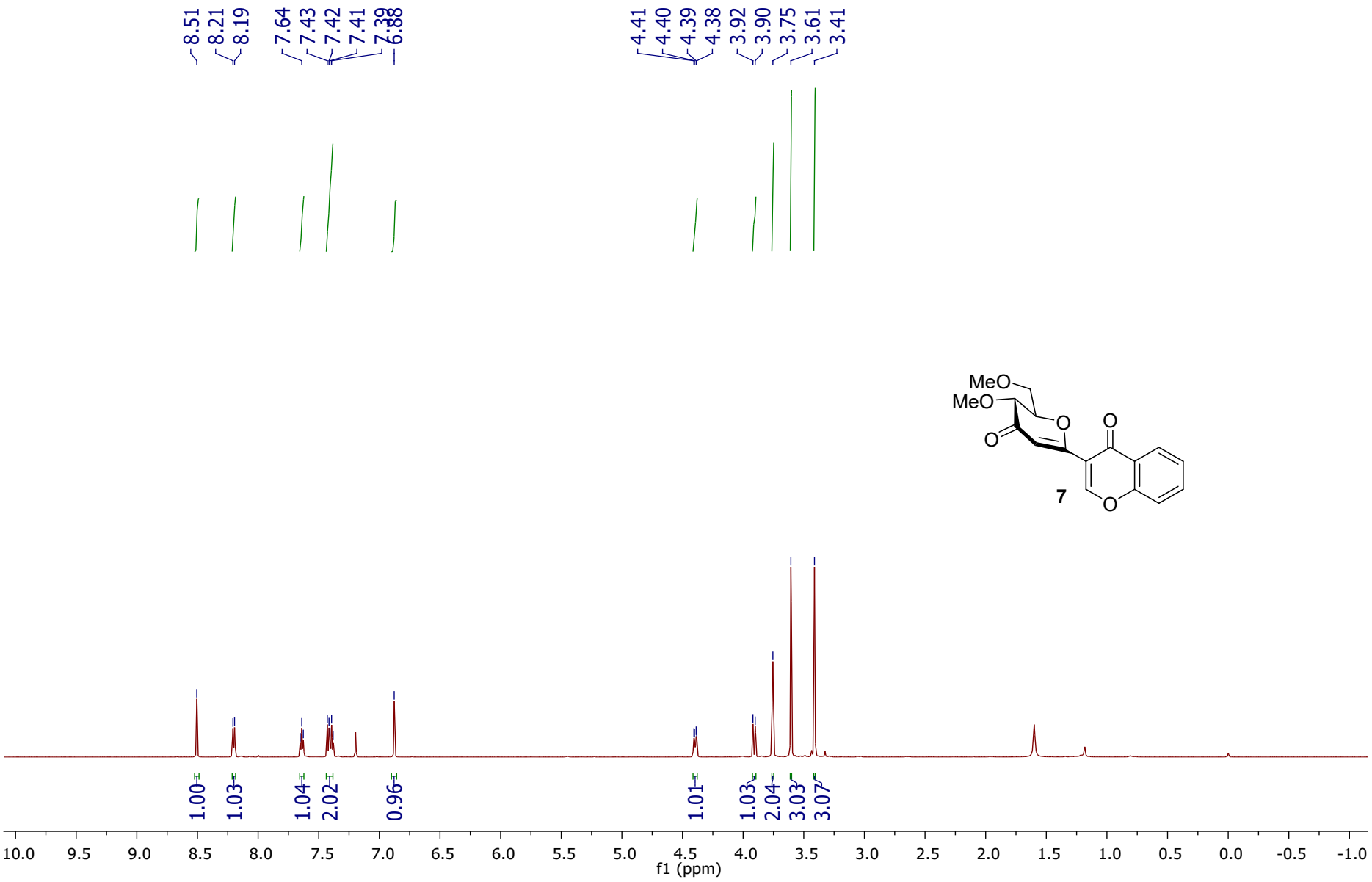

176.61
174.44
156.38
154.07
152.74
140.57
137.51
133.70
129.32
127.18
125.81
125.17
124.02
123.51
118.18
96.45
73.19
72.14
65.98
62.90
58.55
54.59
44.99
44.96
30.12
22.32
18.23
MeO
MeO
O
O
O
O
O
5e
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

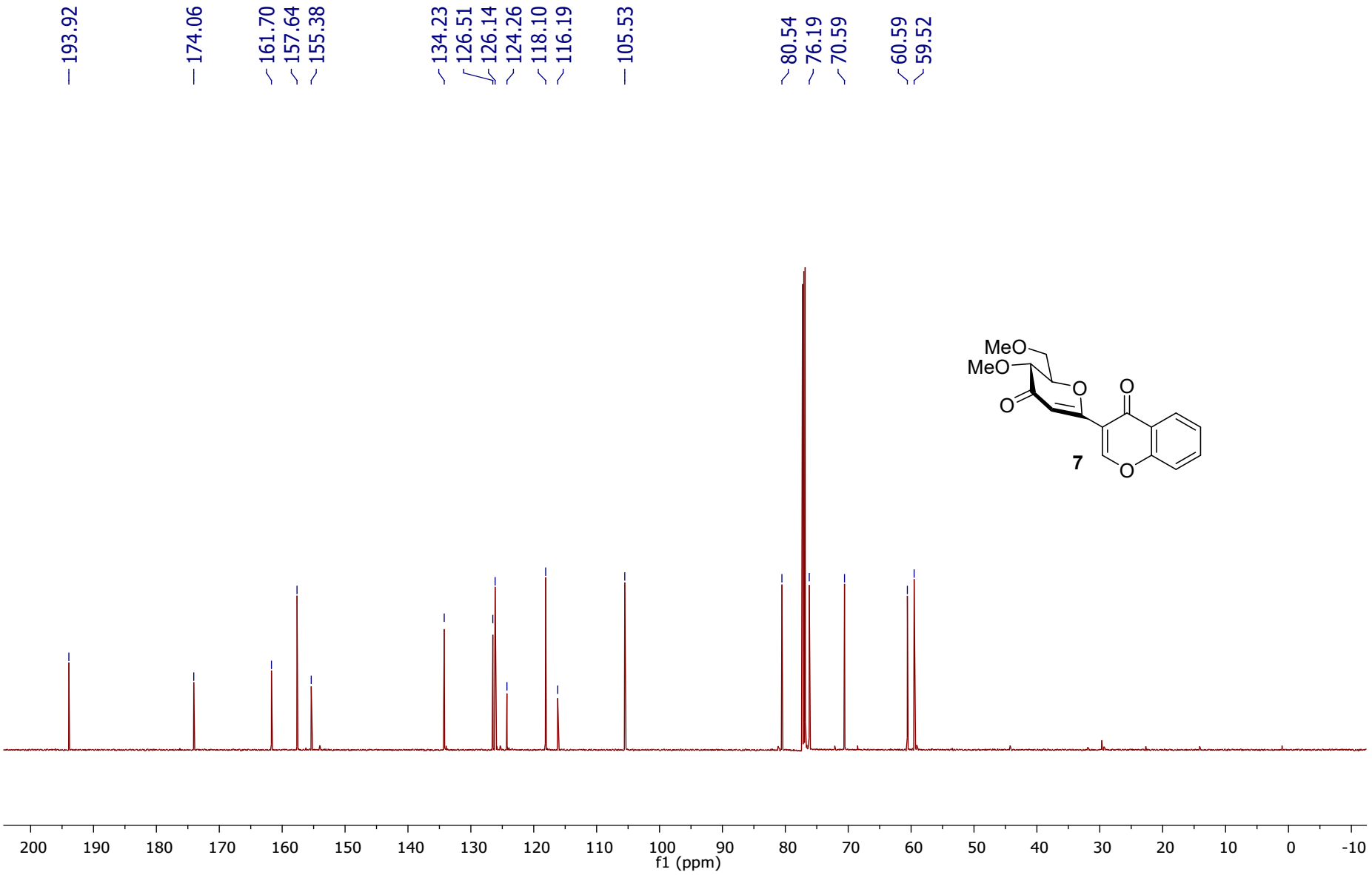

8.17
8.16
8.04
7.61
7.60
7.59
7.40
7.39
7.35
7.34
7.33
5.58
5.26
5.26
5.02
4.25
4.24
4.23
4.16
3.56
3.52
3.44
2.28
2.26
2.25
1.93
1.92
1.91
1.55
1.54
1.53
1.19
0.82
0.81
0.79
MeO
MeO
O
O
O
O
O
5f
1.03
1.00
1.01
1.01
1.05
1.00
2.06
1.00
1.02
2.03
1.01
3.00
3.01
2.09
4.05
2.14
20.29
3.03
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


175.60
172.56
155.35
153.13
151.46
132.67
128.95
128.71
124.76
124.14
122.98
122.65
117.16
95.79
72.42
71.61
64.80
61.48
57.41
53.57
30.87
28.66
28.49
28.28
28.13
28.07
26.18
23.85
21.65
13.88
MeO
MeO
O
O
O
O
O
5f
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


8.15
8.13
8.00
7.62
7.62
7.61
7.59
7.39
7.38
7.35
7.34
7.33
5.45
4.01
4.00
3.85
3.84
3.63
3.61
3.59
3.57
3.55
3.43
3.32
3.05
3.05
3.03
2.66
2.65
2.64
2.62
MeO
MeO
O
O
O
O
6
1.05
1.00
1.11
1.04
1.05
1.00
1.00
1.05
2.02
3.02
3.09
1.01
1.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


205.68
176.27
156.24
154.01
133.92
125.90
125.34
123.93
123.42
118.15
81.15
76.57
72.13
68.50
59.47
59.11
44.25
MeO
MeO
O
O
O
O
6
76.57
59.47
59.11
77.0
f1 (ppm)
59.4
f1 (ppm)
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.51
8.21
8.19
7.64
7.43
7.42
7.41
7.39
7.38
4.41
4.40
4.39
4.38
3.92
3.90
3.75
3.61
3.41
MeO
MeO
O
O
O
O
7
1.00
1.03
1.04
2.02
0.96
1.01
1.03
2.04
3.03
3.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


193.92
174.06
161.70
157.64
155.38
134.23
126.51
126.14
124.26
118.10
116.19
105.53
80.54
76.19
70.59
60.59
59.52
MeO
MeO
O
O
O
O
7
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)