Supporting Information

Nanolayered Cobalt-Molybdenum Sulphides (Co-Mo-S) Catalyse Borrowing Hydrogen C-S Bond Formation Reactions of Thiols or H_2S with Alcohols

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1. GENERAL INFORMATION

Reagents were obtained from commercial sources and were used as received. ^{1}H-NMR, ^{13}C-NMR spectra of isolated products were recorded on a Bruker AV 300 spectrometer. All chemical shifts (δ) are reported in parts per million (ppm) and coupling constants (*J*) in Hz. For ^{1}H-NMR all chemical shifts are reported relative to tetramethylsilane (δ 0.0 ppm in $CDCl_3$) or *d*-solvent peaks (δ 77.16 ppm $CDCl_3$) for ^{13}C-NMR.

The GC yields were determined by GC-FID using *n*-hexadecane as an internal standard. GC-FID analyses were performed on a Bruker 430-GC System equipped with a 25 m capillary column of 5% phenylmethylsilicone. Mass determination was carried out on a GC-Mass Agilent 6890 Network equipped with the same column as the GC and a mass selective detector. The H_2 formation was analyzed using an Agilent 490 MicroGC equipped with two columns (Pore Plot Q and MolSieve 5A) and one thermal conductivity detector (TCD).

Powder X-ray diffraction (XRD) measurements were performed in a HTPhilips X'Pert MPD diffractometer equipped with a PW3050 goniometer using CuKα radiation and a multisampling handler. Samples for electron microscopy studies were prepared by dropping a suspension of the nanolayered Co-Mo-S-based catalysts in CH_2Cl_2 directly onto the holey-carbon-coated nickel grids. All measurements were performed in a JEOL 2100F microscope operating at 200 kV both in transmission (TEM) and scanning-transmission modes (STEM). STEM images were obtained using a high-angle annular dark-field detector (HAADF), which allows Z-contrast imaging.

2. PREPARATION OF UNSUPPORTED CATALYSTS

All unsupported catalysts were prepared by a one-pot hydrothermal synthesis in a 130 mL Parr stirred reactor accordingly to literature methods.[1] Briefly, ammonium molybdate [$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$] (750 mg), elemental sulphur (283.4 mg), and variable amounts of cobalt acetate [$Co(OAc)_2\cdot 4H_2O$] (see Table S1) were placed in a stainless steel autoclave vessel. Then, distilled water (57 mL) and hydrazine monohydrate (64-65%, 5.5 mL) were added, and the autoclave was closed tightly and purged twice with nitrogen for leak testing. The mixture was heated and stirred until the internal temperature (180 °C) was

reached (approximately 60-90 min) and then maintained under static conditions at this temperature. After 22 h, the autoclave was naturally cooled down to room temperature, and the generated gas was carefully released. The resulting nanolayered catalyst was recovered by filtration and washed with distilled water, ethanol, and diethyl ether. Finally, the black solid catalyst was dried under vacuum for 1 h and stored under a nitrogen atmosphere.

Catalyst Mo-free Co_xS_y was synthesized by the above described preparation methodology but without adding the molybdenum salt precursor.[1b]

Table S1. Preparation details, phase composition (PC) and hydrogenation activity (HA) of unsupported catalysts (data reported in ref. 1).

Catalyst	$(NH_4)_6Mo_7O_{24}\cdot 4H_2O$ (mg)	$Co(OAc)_2\cdot 4H_2O$ m(g)	PC	Order of HA[c]
Co-Mo-S-0.39[a,b]	780	295.6	MoS_2; CoS_2	3
Co-Mo-S-0.58[a,b]	780	471.6	MoS_2; CoS_2	4
Co-Mo-S-0.83[a]	780	1100.4	MoS_2; Co_3S_4; CoS_2	1
Co-Mo-S-0.91[a]	780	1650.7	MoS_2; Co_9S_8	2
Mo-free Co_xS_y	-	1100.4	CoS_2; Co_3S_4; Co_9S_8	5
MoS_2	780	-	MoS_2	6

[a] All of them display Co-Mo-S-like structures which are highly active but poorly stable. [b] Apart from their Co/(Mo + Co) mole ratio, differences between both catalysts rely on the distribution/interaction of MoS_2 and CoS_2 phases: catalyst Co-Mo-S-0.39 displays more interlaced phases than catalyst Co-Mo-S-0.58, thus promoting a presumably higher formation of active Co-Mo-S-like structures. [c] Hydrogenation activity (HA) for the regioselective hydrogenation of quinoline.

3. EXTENSION DATA FOR REACTION PROFILES INVESTIGATION

General procedure for the reaction profile determination

Reaction profiles for the alkylation of thiols with alcohols and for the thioetherification with H_2S were performed following the general procedure described in the manuscript. Before taking each aliquot at different reaction times, the magnetic stirring was switched off for 1 min to collect the catalyst on the bottom, and then 50 μL of the reaction mixture was taken out for GC analysis.

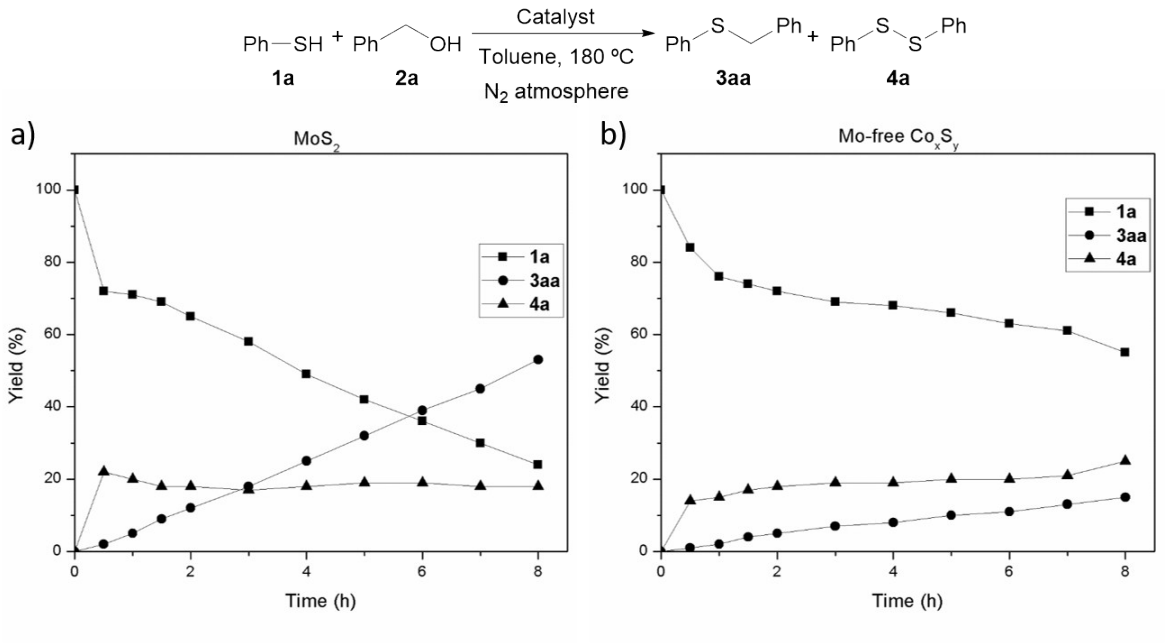


Figure S1. Yield-time diagram of benzyl phenyl sulphide (**3aa**) and diphenyl disulphide (**4a**) produced during alkylation of benzenethiol (**1a**) with benzyl alcohol (**2a**) in the presence of catalyst a) MoS_2 and b) Mo-free Co_xS_y. Reaction conditions: **1a** (0.25 mmol), **2a** (0.5 mmol), catalyst (13.1 mg), toluene (1.6 mL), 3.5 bar N_2, 180 ºC.

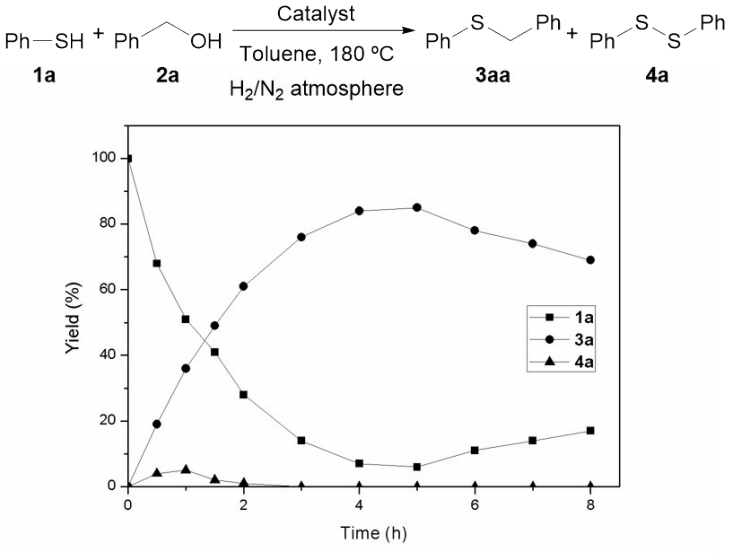


Figure S2. Yield-time diagram of benzyl phenyl sulphide (**3aa**) and diphenyl disulphide (**4a**) produced during alkylation of benzenethiol (**1a**) with benzyl alcohol (**2a**) in the presence of catalyst Co-Mo-S-0.83 with an extra supply of hydrogen gas pressure. Reaction conditions: **1a** (0.25 mmol), **2a** (0.5 mmol), catalyst (13.1 mg), toluene (1.6 mL), 2 bar N_2 and 1.5 bar H_2, 180 ºC.

As shown in Figure S2, during the alkylation reaction of benzenethiol (**1a**) with benzyl alcohol (**2a**) in the presence of catalyst Co-Mo-S-0.83 with an extra supply of hydrogen gas pressure, the concentration of benzyl phenyl sulphide (**3aa**) increased parallel to benzenethiol (**1a**) conversion. Traces (5%) of diphenyl disulphide (**4a**) are also formed, but it is rapidly consumed in 2 h. After reaching a maximum, the concentration of **3aa** starts to decrease with the concomitant formation of **1a** as result of the C-S bond cleavage, which is not surprising considering the extended used of MoS_2-based catalysts in HDS processes.

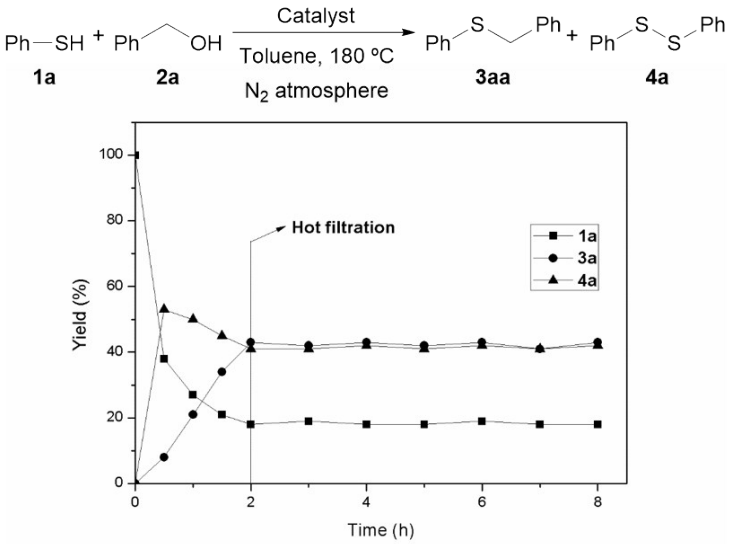


Figure S3. Yield-time diagram of benzyl phenyl sulphide (**3aa**) and diphenyl disulphide (**4a**) produced during alkylation of benzenethiol (**1a**) with benzyl alcohol (**2a**) in the presence of catalyst Co-Mo-S-0.83 when the catalyst is removed by hot filtration. Reaction conditions: **1a** (0.25 mmol), **2a** (0.5 mmol), catalyst (13.1 mg), toluene (1.6 mL), 3.5 bar N_2, 180 ºC.

4. GENERAL PROCEDURES FOR THE CATALYST RECYCLING EXPERIMENTS

For the recycling experiments, the general procedures for the alkylation of thiols with alcohols and thioetherification with H_2S were followed as described in the manuscript. After reaction time (10 or 18 h), the reactor was naturally cooled down to room temperature and the pressure was carefully release. The reaction mixture was diluted with ethyl acetate and an aliquot was taken to be analysed by gas

chromatography. Then, the catalyst was separated by centrifugation, washed with n-Et_2O and dry under vacuum before using for the next run. The combined solutions were taken to dryness and analysed by ICP-MS after each catalytic run. Table S2 summarized the ICP-MS results normalized to the initial volume used in the reaction (1.6 mL).

Table S2. Leaching experiments: ICP-MS analysis after each catalytic run.

Catalytic Run	Co content (ppm)	Mo content (ppm)
1	12.4	0.3
2	0.4	< 0.1
3	< 0.1	< 0.1
4	< 0.1	< 0.1
5	< 0.1	< 0.1
6	< 0.1	< 0.1

5. MORPHOLOGICAL CHARACTERIZATION OF REUSED Co-Mo-S CATALYSTS

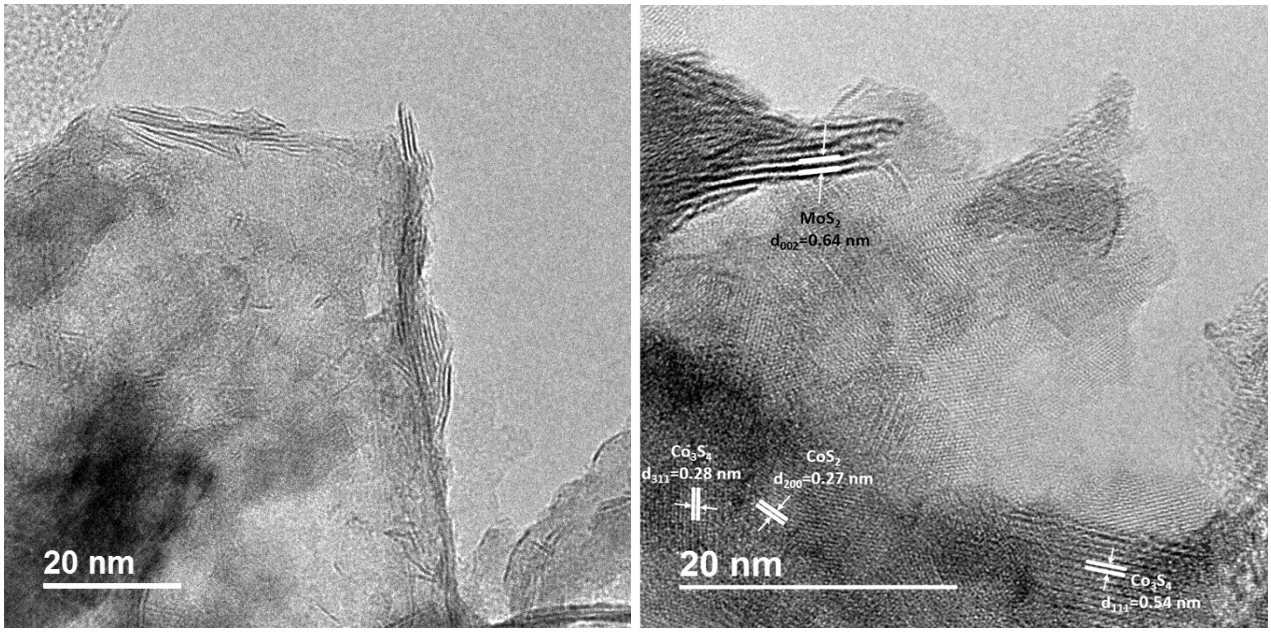


Figure S4. High-resolution transmission electron microscopy micrographs of catalyst Co-Mo-S-0.83.

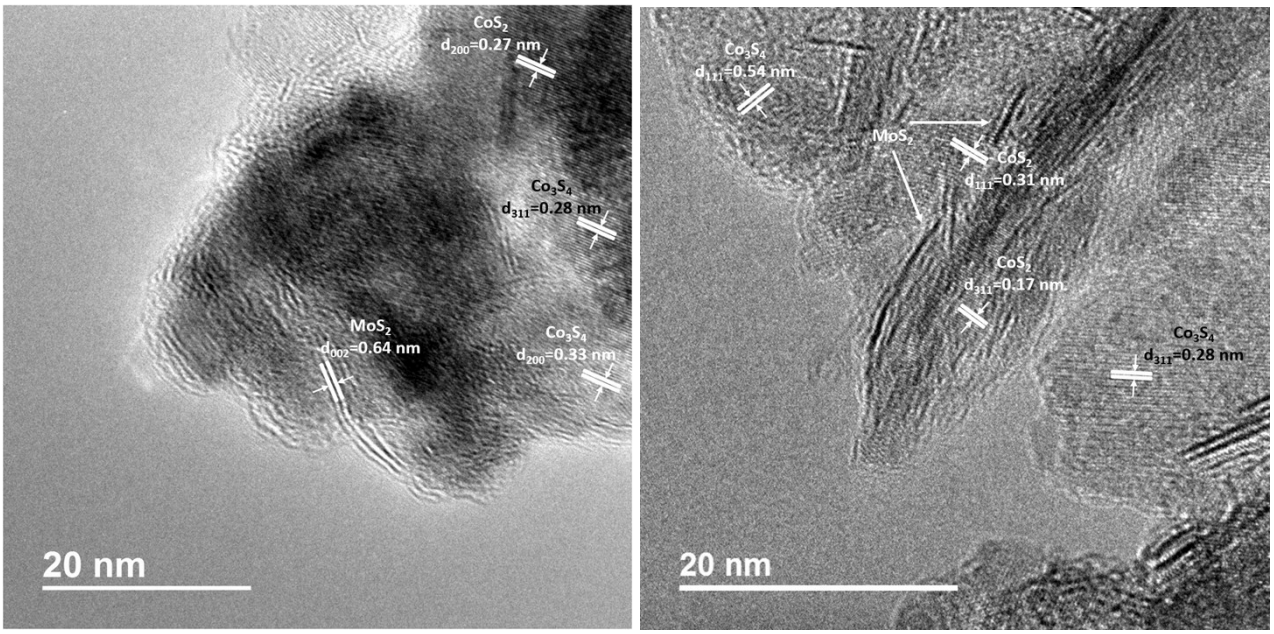


Figure S5. High-resolution transmission electron microscopy micrographs of recycled catalyst Co-Mo-S-0.83 after six runs (Co-Mo-S-0.83-R6) for the alkylation of benzenethiol (**1a**) with benzyl alcohol (**2a**).

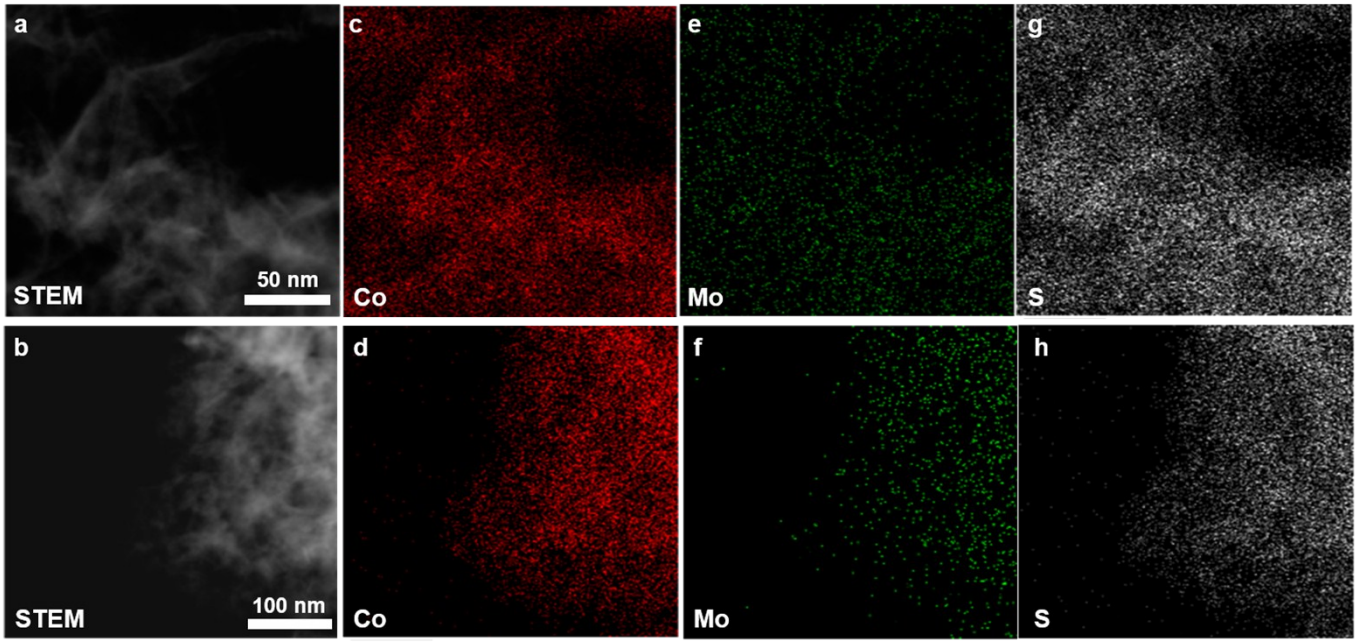


Figure S6. (a-b) STEM-HADDF images of recycled catalyst Co-Mo-S-0.83 after six runs (Co-Mo-S-0.83-R6) for the alkylation of benzenethiol (**1a**) with benzyl alcohol (**2a**). (c-h) Elemental mapping of cobalt, molybdenum and sulphur.

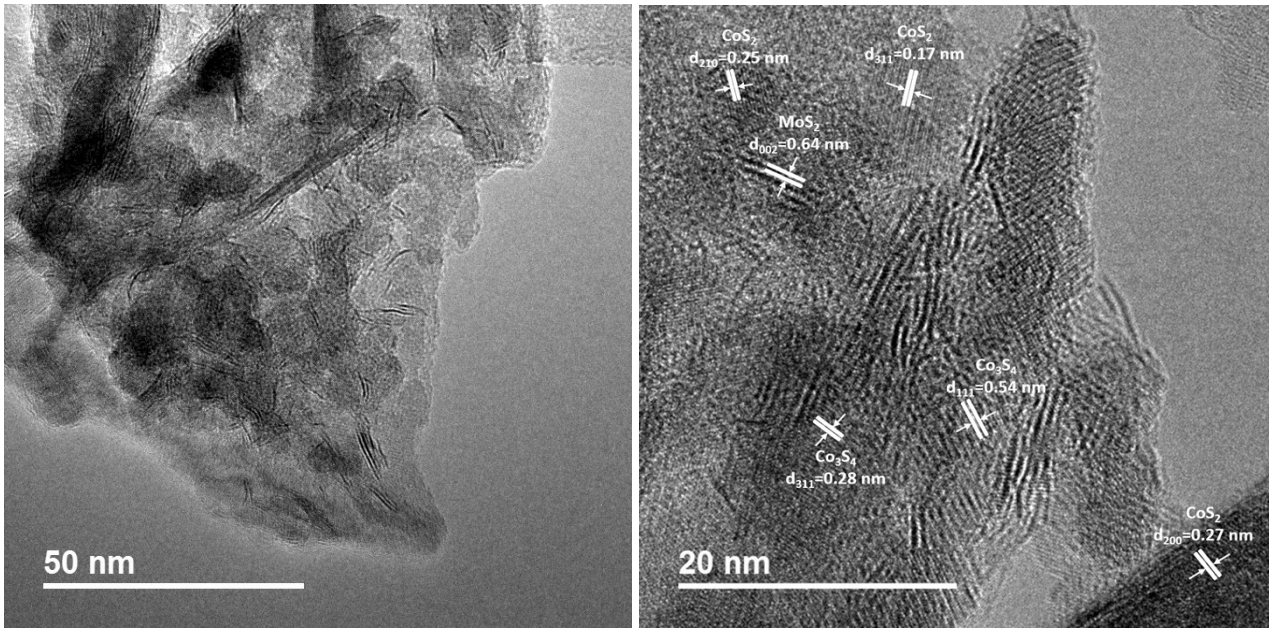


Figure S7. High-resolution transmission electron microscopy micrographs of recycled catalyst Co-Mo-S-0.83 after six runs (Co-Mo-S-0.83-R6-S) for the thioetherification of benzyl alcohol (**2a**) with H_2S.

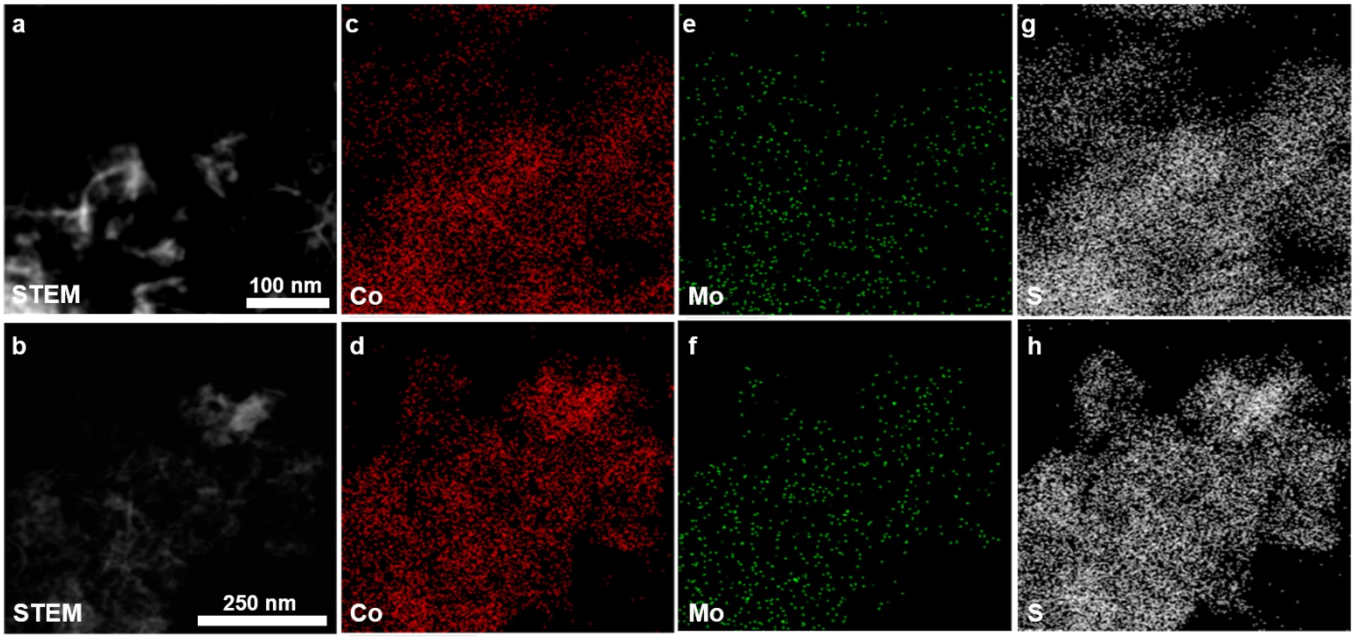
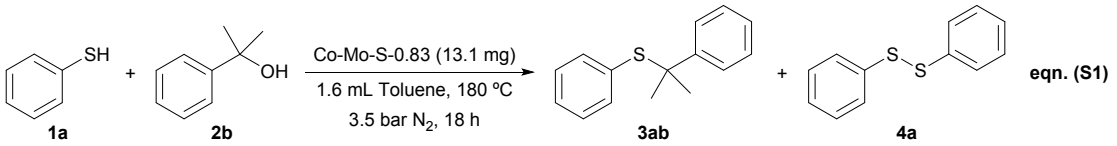


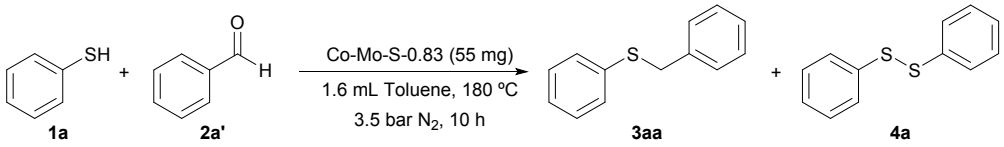
Figure S8. (a-b) STEM-HADDF images of recycled catalyst Co-Mo-S-0.83 after six runs (Co-Mo-S-0.83-R6-S) for the thioetherification of benzyl alcohol (**2a**) with H_2S. (c-h) Elemental mapping of cobalt, molybdenum and sulphur.

6. REACTION PAHTWAY DETERMINATION



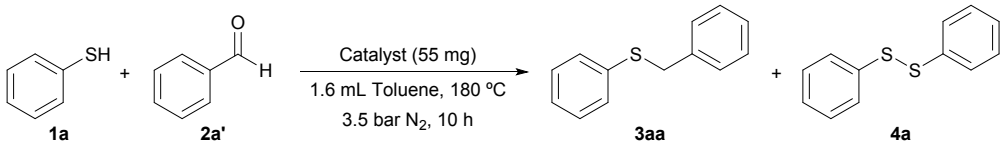
1a **2b** **3ab** **4a** **eqn. (S1)**

0.25 mmol | 2 equiv. | 79 % Conv. | 8 % yield | 70 % yield



1a **2a'** **3aa** **4a** **eqn. (S2a)***

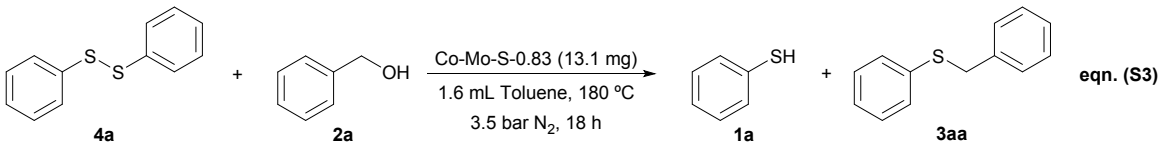
0.25 mmol | 2 equiv. | >99 % Conv. | 1 % yield | 95 % yield



1a **2a'** **3aa** **4a** **eqn. (S2b)*†**

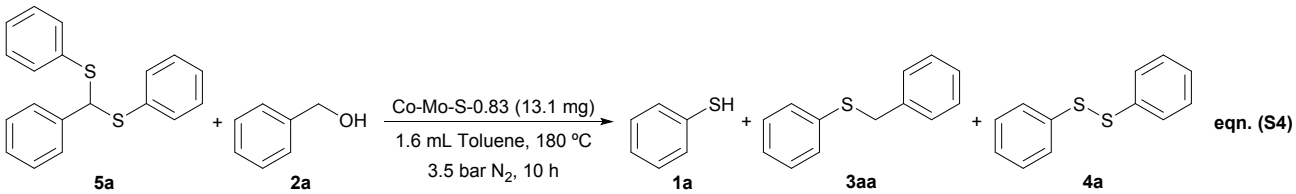
0.25 mmol | 2 equiv. | >99 % Conv. | 17 % yield | 65 % yield

*In both reactions (eqn. S2a and S2b) α,α-bis(phenylthio)toluene (**5a**) was detected as by-product. †Catalyst used for this reaction (eqn. S2b) was pre-treated as follows: Catalyst Co-Mo-S-0.83 (55 mg) was reacted with benzylalcohol (**2a**; 218 μL) under N_2 atmosphere (3.5 bar) at 180 ºC and in the absence of any solvent. After 8 h, catalyst was washed with ethyl acetate, diethyl ether and dry under vacuum.



4a **2a** **1a** **3aa** **eqn. (S3)**

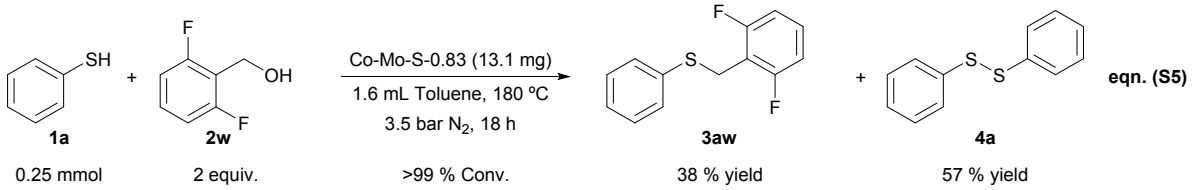
0.125 mmol | 4 equiv. | 89 % Conv. | 4 % yield | 83 % yield



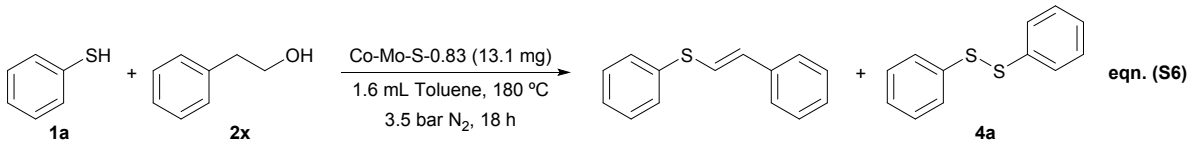
5a **2a** **1a** **3aa** **4a** **eqn. (S4)**

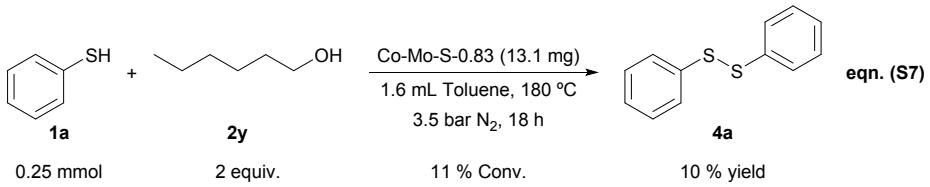
Scheme S1. Control experiments for the reaction pathway determination.

7. EXTENSION OF TABLE 3



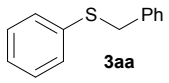
Scheme S2. Co-Mo-S catalysed alkylation of benzenethiol (**1a**) with (2,6-difluorophenyl)methanol (**2w**).



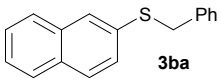


Scheme S3. Co-Mo-S catalysed alkylation of benzenethiol (**1a**) with 2-phenylethanol (**2x**) and 1-hexanol (**2y**).

8. CHARACTERIZATION DATA AND EXPERIMENTAL DETAILS OF THE ISOLATED PRODUCTS

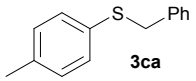


Benzyl(phenyl)sulphane (3aa): Product purified by silica gel column chromatography (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 91 % (The NMR spectrum is consistent with the reported data).[2] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.18 (m, 10H), 4.16 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.61, 136.53, 129.99, 128.95, 128.60, 127.28, 126.46, 39.21. MS (EI): *m*/*z* (rel. int.) 200.



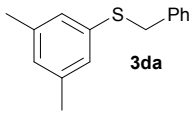
3ba

Benzyl(naphthalen-2-yl)sulphane (3ba): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 85 % (The NMR spectrum is consistent with the reported data).[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.62 – 7.54 (m, 1H), 7.54 – 7.37 (m, 3H), 7.31 – 7.16 (m, 3H), 7.16 – 7.08 (m, 2H), 7.13 – 6.96 (m, 3H), 4.01 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.72, 134.29, 134.08, 132.25, 129.19, 128.87, 128.65, 128.08, 128.03, 127.57, 127.50, 126.81, 126.07, 39.30. MS (EI): *m*/*z* (rel. int.) 250.



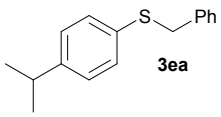
3ca

Benzyl(p-tolyl)sulphane (3ca): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 90 % (The NMR spectrum is consistent with the reported data).[4] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 7.10 (m, 7H), 6.99 (d, *J* = 7.9 Hz, 2H), 4.00 (s, 2H), 2.24 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.15, 136.86, 132.85, 131.04, 129.92, 129.15, 128.74, 127.37, 40.12, 21.34. MS (EI): *m*/*z* (rel. int.) 214.



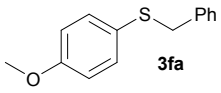
3da

Benzyl(3,5-dimethylphenyl)sulphane (3da): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 78 % (The NMR spectrum is consistent with the reported data).[5] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.16 (m, 5H), 6.93 (s, 2H), 6.81 (s, 1H), 4.10 (s, 2H), 2.25 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.53, 137.75, 136.14, 129.00, 128.57, 128.28, 127.44, 127.24, 39.12, 21.31. MS (EI): *m*/*z* (rel. int.) 228.



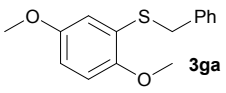
3ea

Benzyl(4-isopropylphenyl)sulphane (3ea): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 73 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 7.11 (m, 7H), 7.11 – 7.00 (m, 2H), 4.03 (s, 2H), 2.80 (hept, *J* = 6.9 Hz, 1H), 1.17 (d, *J* = 7.0 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.61, 137.91, 133.22, 130.57, 128.97, 128.57, 127.21, 127.13, 39.78, 33.84, 24.04. MS (EI): *m*/*z* (rel. int.) 242.



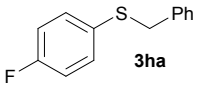
3fa

Benzyl(4-methoxyphenyl)sulphane (3fa): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 93 % (The NMR spectrum is consistent with the reported data).[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 – 7.14 (m, 7H), 6.85 – 6.73 (m, 2H), 3.98 (s, 2H), 3.77 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 159.34, 138.26, 134.16, 129.00, 128.47, 127.08, 126.25, 114.56, 55.40, 41.33. MS (EI): *m*/*z* (rel. int.) 230.



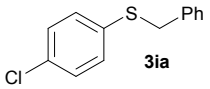
3ga

Benzyl(2,5-dimethoxyphenyl)sulphane (3ga): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99:1 → 98:2 → 97:3). Yield: 76 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40 – 7.10 (m, 5H), 7.06 – 6.63 (m, 3H), 4.13 (s, 2H), 3.87 (s, 3H), 3.72 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.88, 151.88, 137.45, 129.03, 128.56, 127.24, 125.92, 116.26, 111.93, 111.57, 56.54, 55.87, 37.32. MS (EI): *m*/*z* (rel. int.) 260.



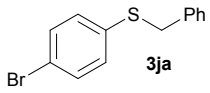
3ha

Benzyl(4-fluorophenyl)sulphane (3ha): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 96 % (The NMR spectrum is consistent with the reported data).[7] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20 (dqd, J = 8.1, 5.5, 2.2 Hz, 7H), 6.96 – 6.84 (m, 2H), 3.99 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ [163.85, 160.58 (d, $^{1}J_{C\text{-}F}$ = 246.8 Hz)], 137.64, [133.60, 133.49 (d, $^{3}J_{C\text{-}F}$ = 8.1 Hz)], [130.88, 130.83 (d, $^{4}J_{C\text{-}F}$ = 3.3 Hz)], 128.97, 128.59, 127.32, [116.17, 115.88 (d, $^{2}J_{C\text{-}F}$ = 21.8 Hz)], 40.57. MS (EI): *m*/*z* (rel. int.) 218.

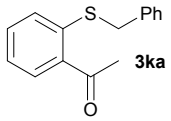


3ia

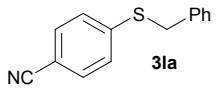
Benzyl(4-chlorophenyl)sulphane (3ia): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 93 % (The NMR spectrum is consistent with the reported data).[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 7.17 (m, 9H), 4.07 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.47, 135.04, 132.83, 131.78, 129.27, 129.12, 128.87, 127.63, 39.67. MS (EI): *m*/*z* (rel. int.) 234.



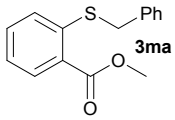
Benzyl(4-bromophenyl)sulphane (3ja): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 94 % (The NMR spectrum is consistent with the reported data).[7] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 – 7.19 (m, 2H), 7.19 – 7.07 (m, 5H), 7.06 – 6.96 (m, 2H), 3.95 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.17, 135.59, 131.97, 131.60, 128.90, 128.67, 127.44, 120.44, 39.22. MS (EI): *m*/*z* (rel. int.) 279.



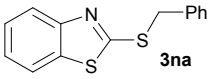
1-(2-(Benzylthio)phenyl)ethan-1-one (3ka): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2 →→90:10). Yield: 92 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.63 (dt, *J* = 7.7, 1.0 Hz, 1H), 7.31 – 7.18 (m, 5H), 7.18 – 7.09 (m, 2H), 7.08 – 6.97 (m, 1H), 3.98 (s, 2H), 2.43 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 199.55, 140.71, 136.40, 135.83, 132.14, 130.72, 129.17, 128.67, 127.39, 126.98, 124.31, 37.93, 28.55. MS (EI): *m*/*z* (rel. int.) 242.



4-(Benzylthio)benzonitrile (3la): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2 → 97:3). Yield: 90 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 – 7.49 (m, 2H), 7.46 – 7.26 (m, 7H), 4.25 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.55, 135.81, 132.29, 128.86, 128.79, 127.79, 127.41, 118.89, 108.65, 37.16. MS (EI): *m*/*z* (rel. int.) 225.

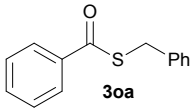


Methyl 2-(benzylthio)benzoate (3ma): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2 → 97:3). Yield: 89 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89 (ddd, *J* = 12.8, 7.8, 1.6 Hz, 1H), 7.39 – 7.13 (m, 7H), 7.11 – 6.99 (m, 1H), 4.07 (s, 2H), 3.80 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.91, 141.87, 136.14, 132.35, 131.23, 129.10, 128.61, 128.38, 127.36, 126.08, 124.11, 52.08, 37.38. MS (EI): *m*/*z* (rel. int.) 258.



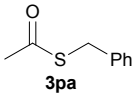
3na

2-(benzylthio)benzo[d]thiazole (3na): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 82 % (The NMR spectrum is consistent with the reported data).[9] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.87 – 7.78 (m, 1H), 7.70 – 7.61 (m, 1H), 7.44 – 7.13 (m, 7H), 4.52 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.65, 153.15, 136.26, 135.38, 129.26, 128.84, 127.90, 126.22, 124.45, 121.65, 121.13, 37.89. MS (EI): *m*/*z* (rel. int.) 257.



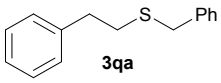
3oa

S-benzyl benzothioate (3oa): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 65 % (The NMR spectrum is consistent with the reported data).[10] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (dt, *J* = 7.2, 1.4 Hz, 2H), 7.42 – 7.31 (m, 1H), 7.30 – 6.97 (m, 7H), 4.13 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.42, 137.60, 136.91, 133.57, 129.11, 128.78, 128.76, 127.46, 127.43, 33.46. MS (EI): *m*/*z* (rel. int.) 228.



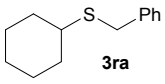
3pa

S-benzyl ethanethioate (3pa): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 45 % (The NMR spectrum is consistent with the reported data).[11] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 – 7.10 (m, 5H), 4.02 (s, 2H), 2.24 (d, *J* = 0.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.29, 137.73, 128.94, 128.77, 127.41, 33.59, 30.47. MS (EI): *m*/*z* (rel. int.) 166.



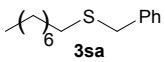
3qa

Benzyl(phenethyl)sulphane (3qa): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 89 % (The NMR spectrum is consistent with the reported data).[12] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.12 (m, 8H), 7.16 – 7.07 (m, 2H), 3.68 (s, 2H), 2.80 (dd, *J* = 9.7, 6.5 Hz, 2H), 2.63 (dd, *J* = 9.1, 6.0 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.66, 138.53, 129.00, 128.62, 128.56, 127.11, 126.44, 36.57, 36.16, 32.90. MS (EI): *m*/*z* (rel. int.) 228.



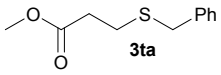
3ra

Benzyl(cyclohexyl)sulphane (3ra): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 78 % (The NMR spectrum is consistent with the reported data).[12] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 – 7.06 (m, 5H), 3.70 – 3.60 (m, 2H), 2.46 (dtq, *J* = 11.1, 7.9, 3.3 Hz, 1H), 1.87 (d, *J* = 11.4 Hz, 2H), 1.72 – 1.58 (m, 2H), 1.22 – 1.10 (m, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 139.09, 128.87, 128.55, 126.88, 43.06, 34.73, 33.51, 26.11, 26.00. MS (EI): *m*/*z* (rel. int.) 206.



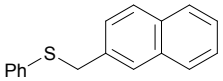
3sa

Benzyl(octyl)sulphane (3sa): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 70 % (The NMR spectrum is consistent with the reported data).[13] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.16 (m, 5H), 3.72 (s, 2H), 2.43 (t, *J* = 7.4 Hz, 2H), 1.65 – 1.49 (m, 2H), 1.29 (d, *J* = 8.5 Hz, 10H), 0.90 (t, *J* = 6.6 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.85, 128.96, 128.56, 126.98, 36.48, 31.95, 31.58, 29.39, 29.32, 29.30, 29.03, 22.78, 14.22. MS (EI): *m*/*z* (rel. int.) 236.



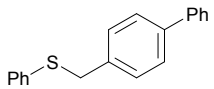
3ta

methyl 3-(benzylthio)propanoate (3ta): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 83 % (The NMR spectrum is consistent with the reported data).[14] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 – 7.07 (m, 5H), 3.65 (s, 2H), 3.59 (s, 3H), 2.60 (t, *J* = 6.4 Hz, 2H), 2.54 – 2.41 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.43, 138.18, 128.95, 128.67, 127.21, 77.58, 77.16, 76.74, 51.88, 36.45, 34.43, 26.35. MS (EI): *m*/*z* (rel. int.) 210.



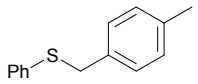
3ac

(Naphthalen-2-ylmethyl)(phenyl)sulphane (3ac): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 88 % (The NMR spectrum is consistent with the reported data).[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 – 7.57 (m, 3H), 7.52 (s, 1H), 7.36 – 7.23 (m, 3H), 7.22 – 6.96 (m, 5H), 4.11 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.36, 135.01, 133.42, 132.70, 130.12, 128.97, 128.41, 127.82, 127.77, 127.52, 127.09, 126.55, 126.24, 125.93, 39.53. MS (EI): *m*/*z* (rel. int.) 250.



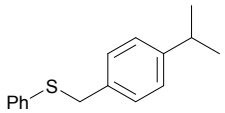
3ad

([1,1'-Biphenyl]-4-ylmethyl)(phenyl)sulphane (3ad): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 89 % (The NMR spectrum is consistent with the reported data).[15] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.57 – 7.38 (m, 4H), 7.38 – 7.02 (m, 10H), 4.07 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.87, 140.22, 136.70, 136.50, 130.04, 129.36, 129.00, 128.89, 127.41, 127.34, 127.15, 126.53, 38.94. MS (EI): *m*/*z* (rel. int.) 276.



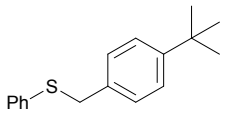
3ae

(4-Methylbenzyl)(phenyl)sulphane (3ae): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 96 % (The NMR spectrum is consistent with the reported data).[4] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.11 (m, 7H), 7.08 (d, *J* = 7.8 Hz, 2H), 4.08 (s, 2H), 2.30 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.93, 136.79, 134.44, 129.73, 129.30, 128.92, 128.82, 126.30, 38.80, 21.22. MS (EI): *m*/*z* (rel. int.) 214.



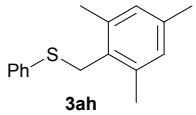
3af

(4-Isopropylbenzyl)(phenyl)sulphane (3af): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 95 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 6.94 (m, 9H), 4.02 (s, 2H), 2.79 (hept, *J* = 6.9 Hz, 1H), 1.15 (dd, *J* = 6.9, 0.7 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.00, 136.98, 134.76, 129.59, 128.94, 128.89, 126.71, 126.25, 38.77, 33.91, 24.10. MS (EI): *m*/*z* (rel. int.) 242.



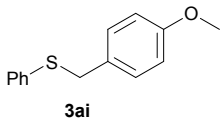
3ag

(4-(tert-Butyl)benzyl)(phenyl)sulphane (3ag): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 91 % (The NMR spectrum is consistent with the reported data).[16] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.30 – 7.21 (m, 4H), 7.21 – 7.12 (m, 4H), 7.12 – 7.03 (m, 1H), 4.03 (s, 2H), 1.22 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.30, 137.05, 134.39, 129.52, 128.95, 128.63, 126.24, 125.59, 38.63, 34.63, 31.48. MS (EI): *m*/*z* (rel. int.) 256.



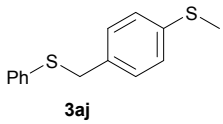
3ah

Phenyl(2,4,6-trimethylbenzyl)sulphane (3ah): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 82 % (The NMR spectrum is consistent with the reported data).[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 – 7.27 (m, 2H), 7.26 – 7.18 (m, 2H), 7.17 – 7.07 (m, 1H), 6.77 (s, 2H), 4.06 (s, 2H), 2.27 (s, 6H), 2.18 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.73, 137.37, 137.00, 130.03, 129.87, 129.18, 128.99, 126.30, 33.83, 21.08, 19.65. MS (EI): *m*/*z* (rel. int.) 242.



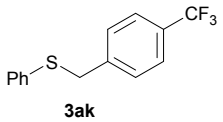
3ai

(4-Methoxybenzyl)(phenyl)sulphane (3ai): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 84 % (The NMR spectrum is consistent with the reported data).[6] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 – 7.12 (m, 7H), 6.86 – 6.75 (m, 2H), 4.07 (s, 2H), 3.76 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.89, 136.69, 130.03, 129.88, 129.50, 128.92, 126.35, 114.02, 55.35, 38.56. MS (EI): *m*/*z* (rel. int.) 230.



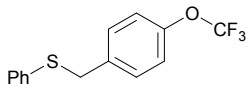
3aj

Methyl(4-((phenylthio)methyl)phenyl)sulphane (3aj): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 81 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.34 – 7.11 (m, 9H), 4.06 (s, 2H), 2.45 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.39, 136.35, 134.52, 130.10, 129.40, 128.98, 126.88, 126.55, 38.82, 16.05. MS (EI): *m*/*z* (rel. int.) 246.



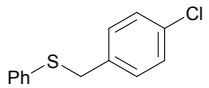
3ak

Phenyl(4-(trifluoromethyl)benzyl)sulphane (3ak): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 51 % (The NMR spectrum is consistent with the reported data).[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53 (d, J = 8.2 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 7.34 – 7.15 (m, 5H), 4.13 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.01, 135.50, 130.61, [130.21, 129.77, 129.35, 128.92 (q, $^{2}J_{C\text{-}F}$ = 32.5 Hz)], [129.68, 126.08, 122.48, 118.87 (q, $^{1}J_{C\text{-}F}$ = 271.6 Hz)], 129.22, 129.13, 127.03, [125.63, 125.58, 125.53, 125.48 (q, $^{3}J_{C\text{-}F}$ = 3.8 Hz)], 39.02. MS (EI): *m*/*z* (rel. int.) 268.



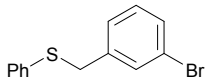
3al

Phenyl(4-(trifluoromethoxy)benzyl)sulphane (3al): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 80 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28 – 7.11 (m, 7H), 7.11 – 7.01 (m, 2H), 4.03 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 148.45, 136.53, 135.78, 130.48, 130.28, 129.08, 126.89, 121.08, [125.72, 122.31, 118.91, 115.50 (q, $^{1}J_{C\text{-}F}$ = 257.1 Hz)], 38.63. MS (EI): *m*/*z* (rel. int.) 284.



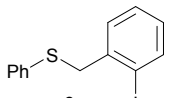
3am

(4-Chlorobenzyl)(phenyl)sulphane (3am): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 93 % (The NMR spectrum is consistent with the reported data).[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.12 (m, 9H), 4.03 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.28, 135.81, 133.06, 130.40, 130.24, 129.04, 128.72, 126.80, 38.68. MS (EI): *m*/*z* (rel. int.) 235.



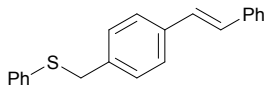
3an

(3-Bromobenzyl)(phenyl)sulphane (3an): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 76 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.50 (t, *J* = 1.9 Hz, 1H), 7.47 – 7.13 (m, 8H), 4.12 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.08, 135.71, 131.94, 130.47, 130.39, 130.08, 129.06, 127.52, 126.89, 122.56, 38.85. MS (EI): *m*/*z* (rel. int.) 279.



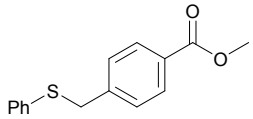
3ao

(2-Iodobenzyl)(phenyl)sulphane (3ao): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 73 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.92 (dd, *J* = 7.6, 1.0 Hz, 1H), 7.48 – 7.21 (m, 7H), 6.99 (ddd, *J* = 7.8, 5.6, 3.4 Hz, 1H), 4.27 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.02, 139.81, 135.75, 130.93, 130.14, 129.00, 128.39, 126.91, 100.72, 44.83. MS (EI): *m*/*z* (rel. int.) 326.



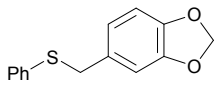
3ap

(E)-phenyl(4-styrylbenzyl)sulphane (3ap): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2 → 97:3). Yield: 79 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45 – 7.32 (m, 4H), 7.28 – 7.09 (m, 10H), 6.99 (s, 2H), 4.03 (s, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 137.45, 137.09, 136.46, 136.37, 130.19, 129.30, 128.99, 128.81, 128.40, 127.76, 127.04, 126.75, 126.63, 126.57, 39.11. MS (EI): *m*/*z* (rel. int.) 302.



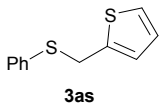
3aq

Methyl 4-((phenylthio)methyl)benzoate (3aq): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2 →→ 95:5). Yield: 86 % (The NMR spectrum is consistent with the reported data).[17] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 – 7.79 (m, 2H), 7.28 – 7.05 (m, 7H), 4.03 (s, 2H), 3.81 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.95, 143.14, 135.51, 130.62, 129.88, 129.10, 129.03, 128.91, 126.93, 52.17, 39.20. MS (EI): *m*/*z* (rel. int.) 258.



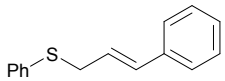
3ar

5-((Phenylthio)methyl)benzo[d][1,3]dioxole (3ar): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 86 % (The NMR spectrum is consistent with the reported data).[18] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.24 (qt, *J* = 4.9, 2.3 Hz, 3H), 7.21 – 7.07 (m, 2H), 6.77 (d, *J* = 1.4 Hz, 1H), 6.65 (d, *J* = 1.7 Hz, 2H), 5.87 (s, 2H), 3.99 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.86, 146.87, 136.42, 131.31, 130.00, 128.96, 126.49, 122.16, 109.32, 108.20, 101.14, 39.16. MS (EI): *m*/*z* (rel. int.) 244.



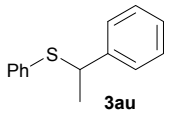
3as

2-((Phenylthio)methyl)thiophene (3as): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 81 % (The NMR spectrum is consistent with the reported data).[8] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 2H), 7.23 – 7.04 (m, 4H), 6.87 – 6.75 (m, 2H), 4.22 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.98, 135.81, 130.47, 129.02, 126.87, 126.83, 126.35, 125.04, 33.88. MS (EI): *m*/*z* (rel. int.) 206.



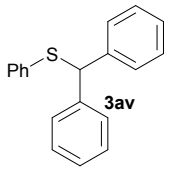
3at

Cinnamyl(phenyl)sulphane (3at): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 87 % (The NMR spectrum is consistent with the reported data).[19] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.45 – 7.35 (m, 2H), 7.35 – 7.12 (m, 8H), 6.51 – 6.36 (m, 1H), 6.26 (dt, *J* = 15.6, 7.0 Hz, 1H), 3.71 (dd, *J* = 7.0, 1.1 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.84, 135.98, 130.35, 128.94, 128.62, 127.66, 126.49, 126.44, 125.16, 37.20. MS (EI): *m*/*z* (rel. int.) 226.



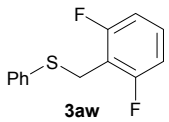
3au

Phenyl(1-phenylethyl)sulphane (3au): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 81 % (The NMR spectrum is consistent with the reported data).[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32 – 7.23 (m, 5H), 7.18 (dtd, *J* = 9.1, 4.1, 2.5 Hz, 5H), 4.31 (q, *J* = 7.0 Hz, 1H), 1.60 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.36, 135.27, 132.62, 128.80, 128.51, 127.40, 127.25, 127.23, 48.14, 22.46. MS (EI): *m*/*z* (rel. int.) 214.



3av

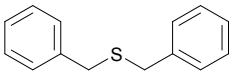
Benzhydryl(phenyl)sulphane (3av): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 82 % (The NMR spectrum is consistent with the reported data).[3] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.38 – 7.28 (m, 4H), 7.26 – 6.96 (m, 11H), 5.46 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 141.17, 136.28, 130.71, 128.85, 128.67, 128.56, 127.37, 126.72, 57.63. MS (EI): *m*/*z* (rel. int.) 276.



3aw

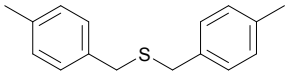
(2,6-Difluorobenzyl)(phenyl)sulphane (3aw): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 38 % (The NMR spectrum is consistent with the reported data).[7] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.35 (dq, *J* = 6.6, 3.0, 2.5 Hz, 2H), 7.28 – 7.18 (m, 3H), 7.14 (tt, *J* = 8.4, 4.8 Hz, 1H), 6.86 – 6.72 (m, 2H), 4.09 (d, *J* = 1.3 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ [163.07, 162.97, 159.77, 159.66 (dd, $^{1}J_{C\text{-}F}$ = 249.5, 7.8 Hz)], 135.20, 132.00, [129.06, 128.92, 128.79 (t,

$^{3}J_{C\text{-}F}$ = 10.2 Hz)], 128.96, 127.44, [114.80, 114.55, 114.29 (t, $^{2}J_{C\text{-}F}$ = 19.2 Hz)], [111.50, 111.48, 111.40, 111.26, 111.18, 111.16 (m)], [26.98, 26.94, 26.91 (t, $^{3}J_{C\text{-}F}$ = 2.6 Hz)]. MS (EI): *m*/*z* (rel. int.) 236.



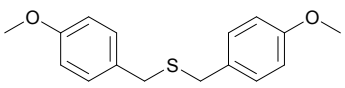
7a

Dibenzyl sulphide (7a): Product purified by silica gel column chromatography (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 66 % (The NMR spectrum is consistent with the reported data).[20] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 – 7.26 (m, 10H), 3.68 (s, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 138.28, 129.12, 128.59, 127.08, 35.76. MS (EI): *m*/*z* (rel. int.) 214.



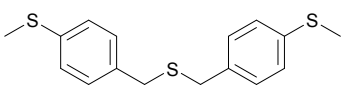
7e

Bis(4-methylbenzyl)sulphane (7e): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1). Yield: 75 % (The NMR spectrum is consistent with the reported data).[21] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.26 – 7.07 (m, 8H), 3.57 (s, 4H), 2.34 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 136.66, 135.28, 129.27, 129.03, 35.43, 21.23. MS (EI): *m*/*z* (rel. int.) 242.



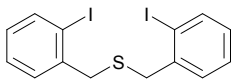
7i

Bis(4-methoxybenzyl)sulphane (7i): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2 →→ 96:4). Yield: 70 % (The NMR spectrum is consistent with the reported data).[22] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.21 – 7.05 (m, 4H), 6.87 – 6.72 (m, 4H), 3.72 (s, 6H), 3.48 (s, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.59, 130.20, 130.03, 113.87, 55.28, 34.95. MS (EI): *m*/*z* (rel. int.) 274.



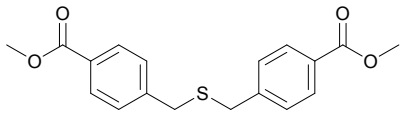
7j

Bis(4-(methylthio)benzyl)sulphane (7j): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5). Yield: 64 % (The NMR spectrum is consistent with the reported data).[23] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.27 – 7.13 (m, 8H), 3.55 (s, 4H), 2.48 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 137.14, 135.10, 129.61, 126.93, 35.24, 16.12. MS (EI): *m*/*z* (rel. int.) 306.



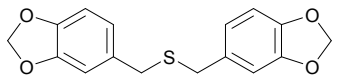
7o

Bis(2-iodobenzyl)sulphane (7o): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2). Yield: 54 % (The NMR spectrum is consistent with the reported data).[21] ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.76 (dd, *J* = 7.9, 1.2 Hz, 2H), 7.33 – 7.16 (m, 4H), 6.85 (td, *J* = 7.6, 1.9 Hz, 2H), 3.72 (s, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 140.28, 139.84, 130.10, 128.78, 128.35, 100.86, 41.46. MS (EI): *m*/*z* (rel. int.) 466.



7q

Dimethyl 4,4'-(thiobis(methylene))dibenzoate (7q): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture = 99.5:0.5 → 99:1 → 98:2 →→ 90:10). Yield: 56 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.91 (dq, *J* = 8.4, 1.9 Hz, 4H), 7.30 – 7.13 (m, 4H), 3.84 (d, *J* = 1.5 Hz, 6H), 3.54 (d, *J* = 1.7 Hz, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.96, 143.28, 129.99, 129.50, 129.13, 52.26, 35.45. MS (EI): *m*/*z* (rel. int.) 330.



7r

Bis(benzo[d][1,3]dioxol-5-ylmethyl)sulphane (7r): Product purified by TLC plate of silica gel (*n*-hexane/ethyl acetate mixture 99.5:0.5 → 99:1 → 98:2 →→ 96:4). Yield: 70 %. ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.82 (d, *J* = 1.6 Hz, 2H), 6.78 – 6.65 (m, 4H), 5.95 (s, 4H), 3.53 (s, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 147.81, 146.59, 131.91, 122.10, 109.28, 107.95, 101.00, 35.50. MS (EI): *m*/*z* (rel. int.) 302.

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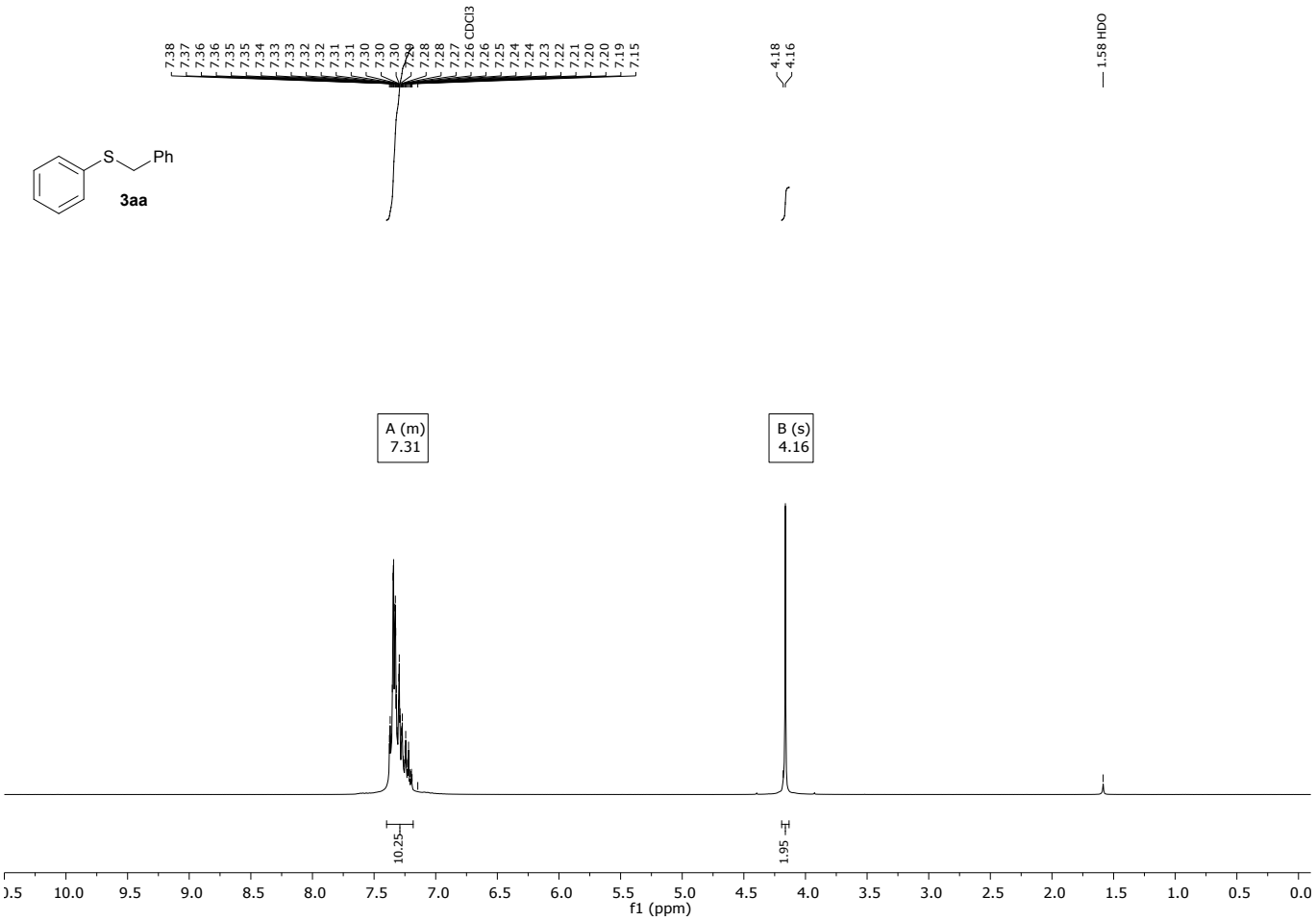
10. ^{1}H NMR, ^{13}C NMR AND ^{19}F NMR SPECTRA OF THE ISOLATED PRODUCTS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7.15
4.18
4.16
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Ph
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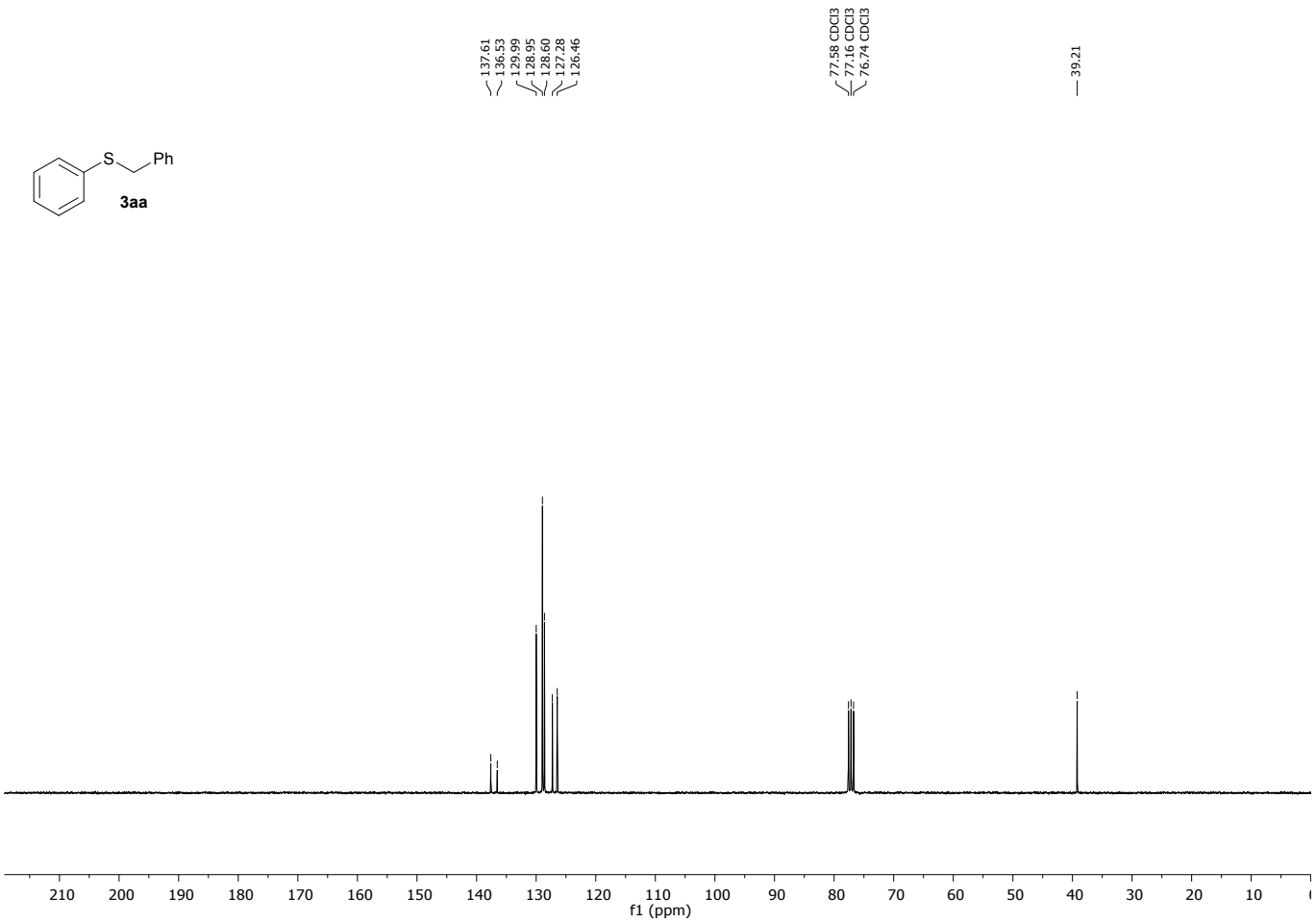
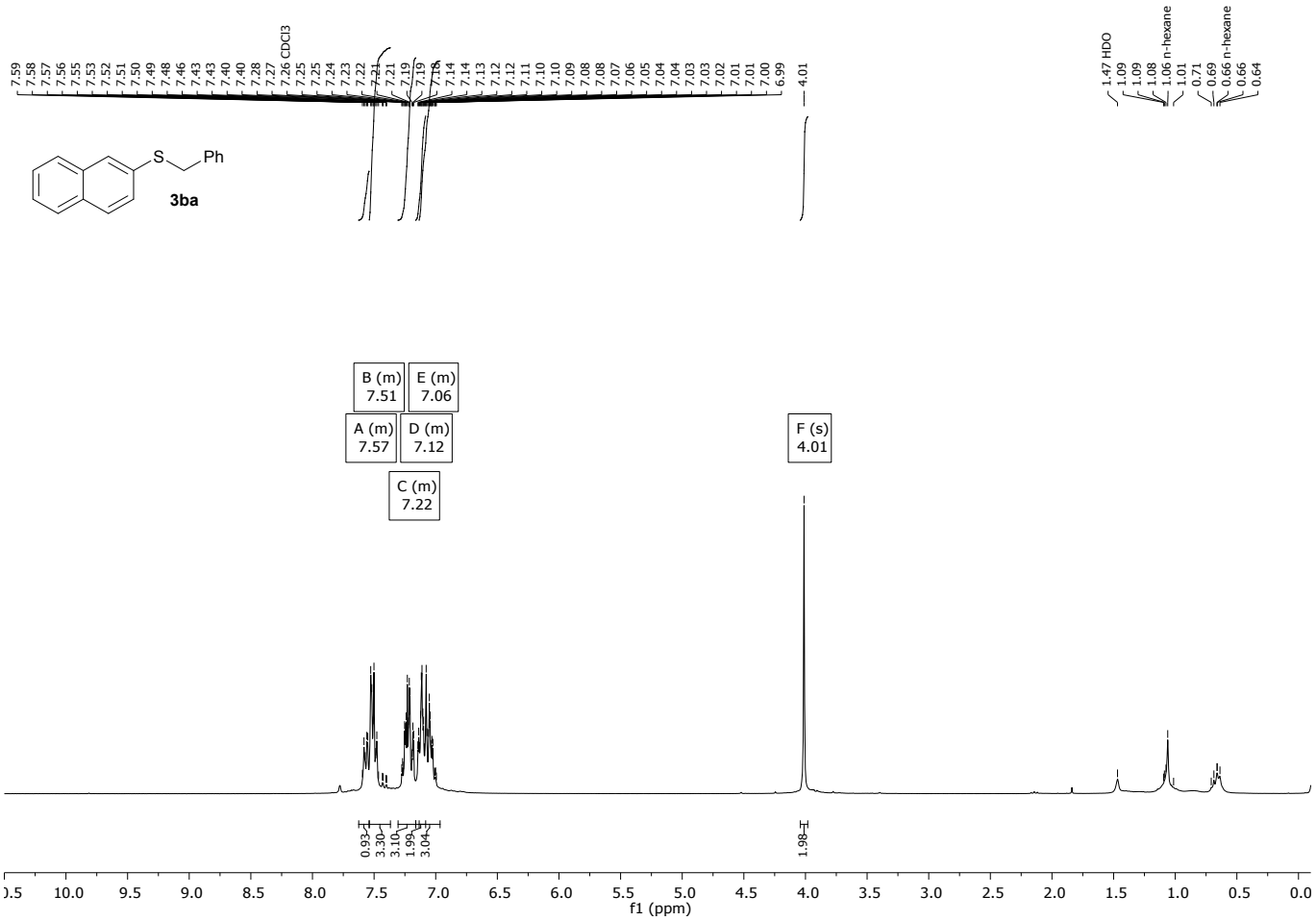
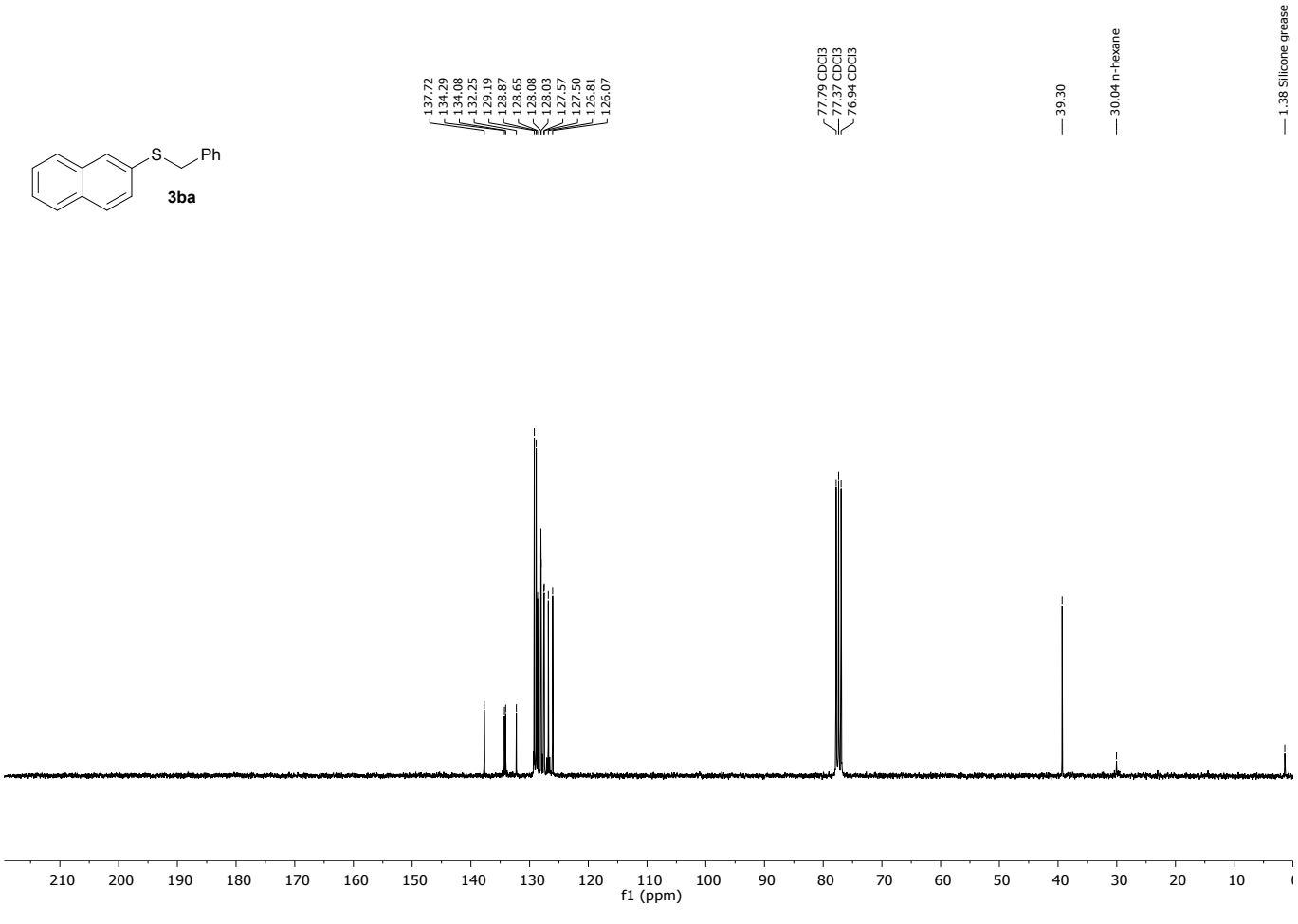
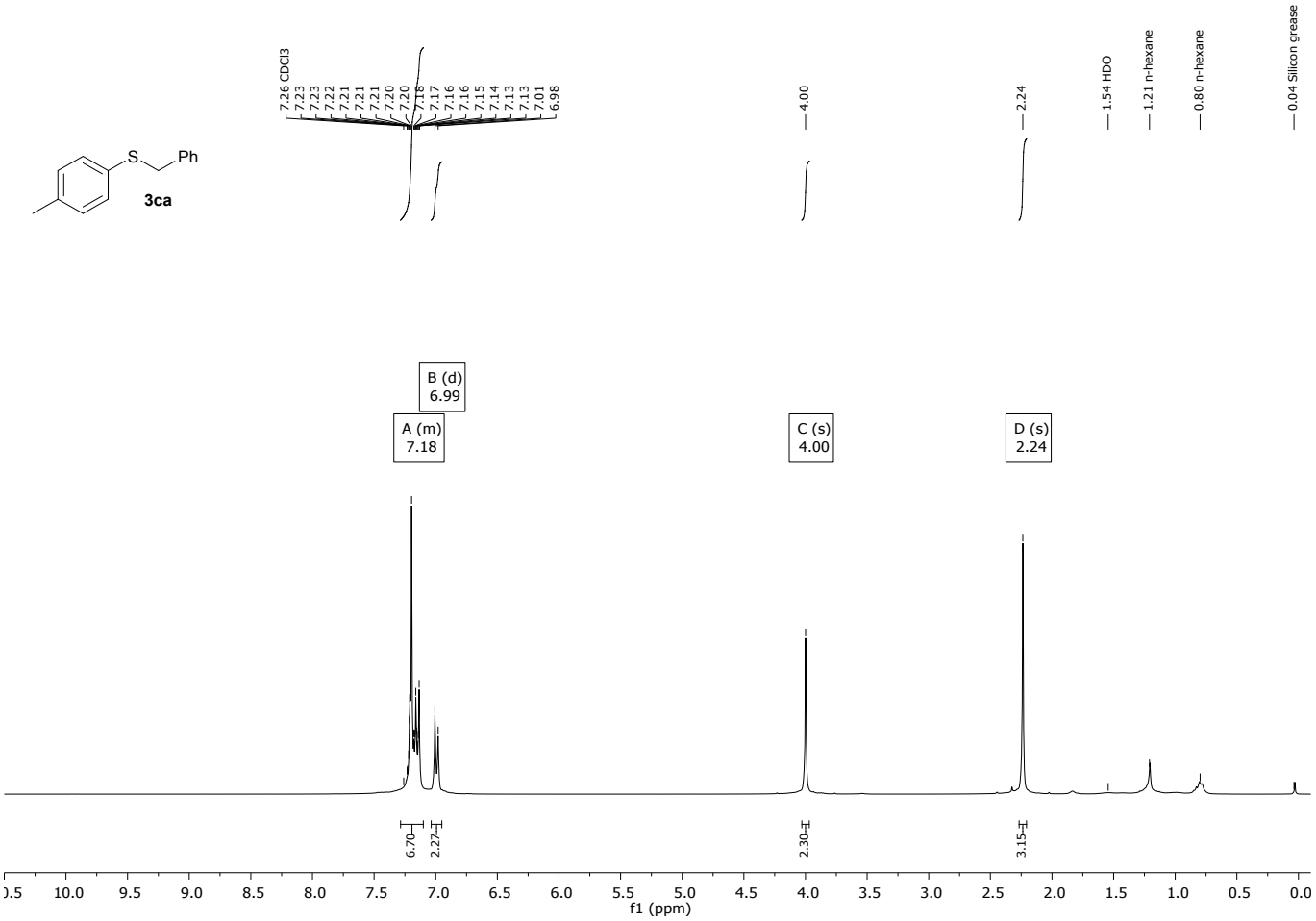
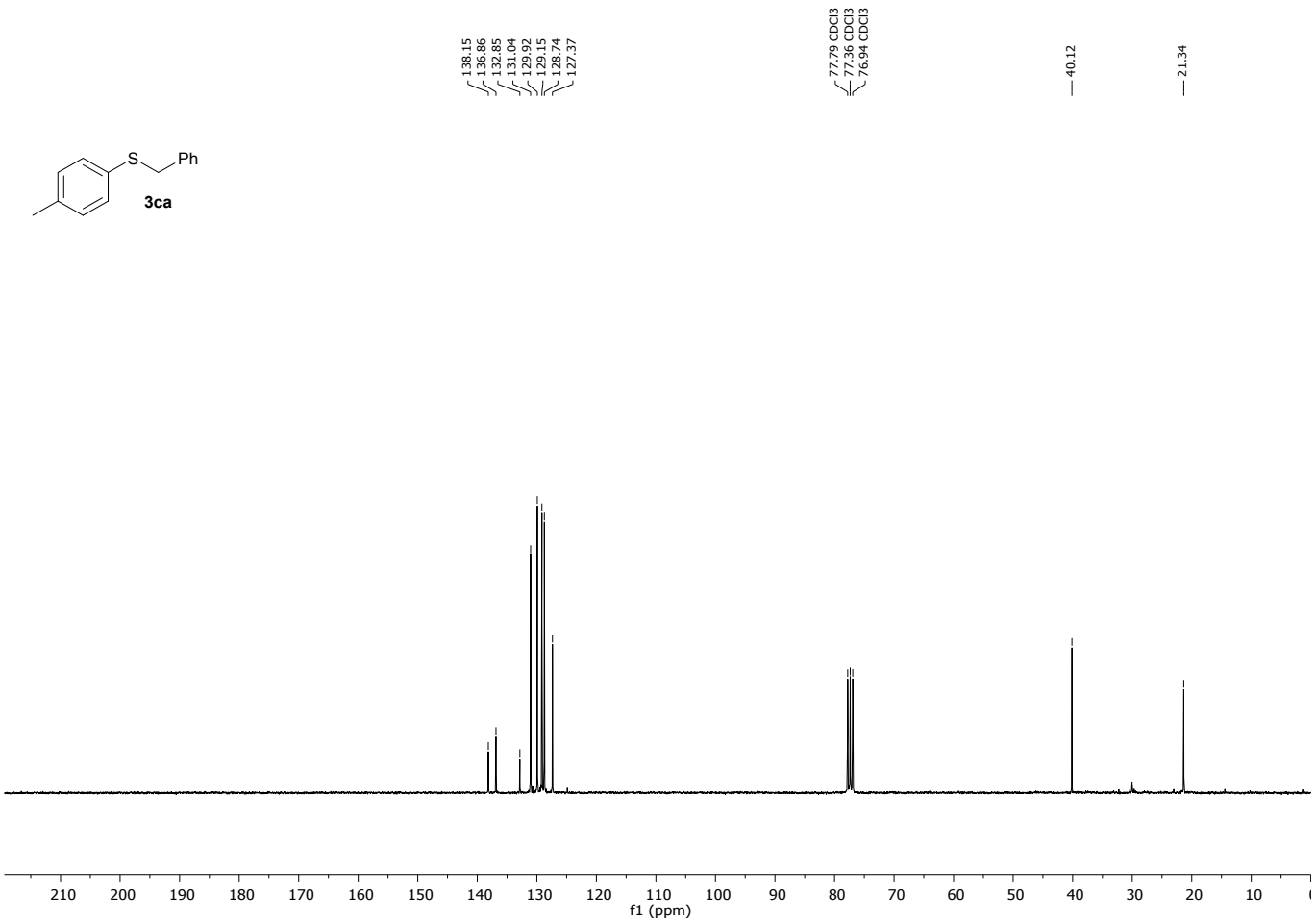
B (s)
4.16
10.25
1.95
f1 (ppm)

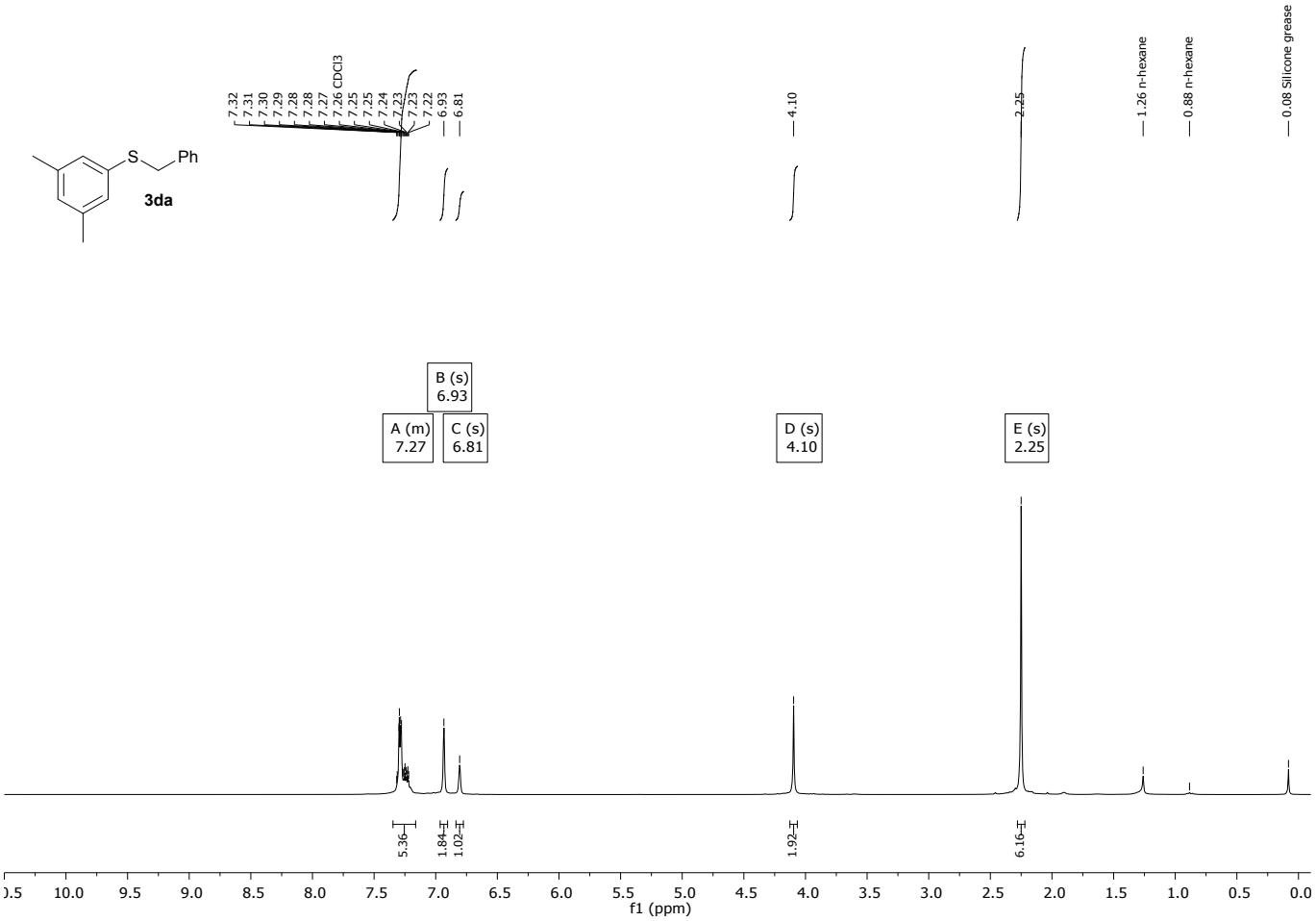

137.61
136.53
129.99
128.95
128.60
127.28
126.46
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
39.21
S
Ph
3aa
f1 (ppm)

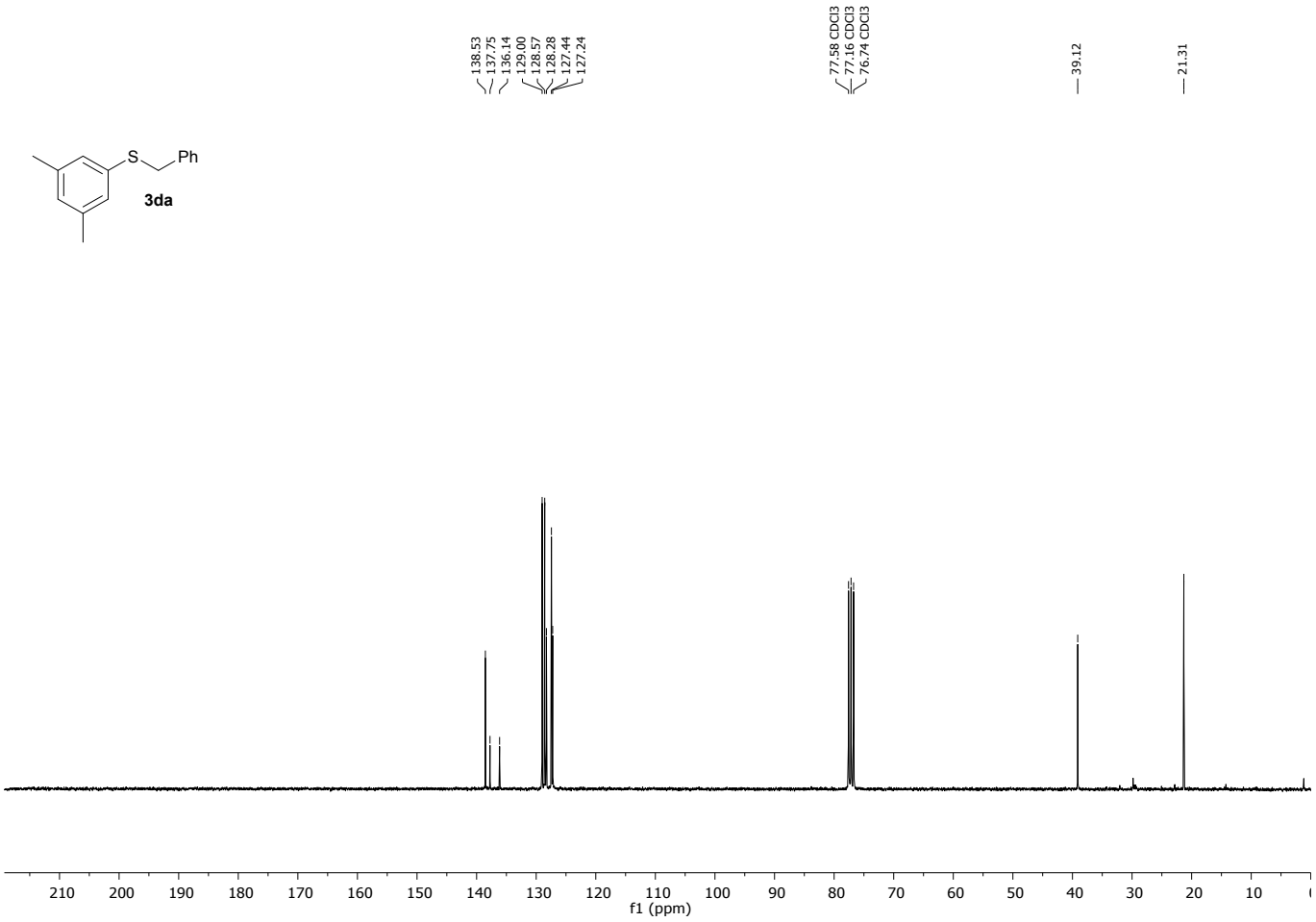

7.59 7.58 7.57 7.56 7.55 7.53 7.52 7.51 7.50 7.49 7.48 7.46 7.43 7.43 7.40 7.40 7.28 7.27 7.26 CDCl3 7.25 7.24 7.23 7.22 7.21 7.21 7.19 7.19 7.16 7.14 7.13 7.12 7.12 7.11 7.10 7.10 7.09 7.08 7.08 7.07 7.06 7.05 7.04 7.04 7.03 7.03 7.02 7.01 7.01 7.00 6.99 4.01
1.47 HDO 1.09 1.09 1.08 1.06 n-hexane 1.01 0.71 0.69 0.66 n-hexane 0.66 0.64
S Ph
3ba
B (m) 7.51
E (m) 7.06
A (m) 7.57
D (m) 7.12
C (m) 7.22
F (s) 4.01
0.93 3.30 3.10 1.99 3.04 1.98
f1 (ppm)

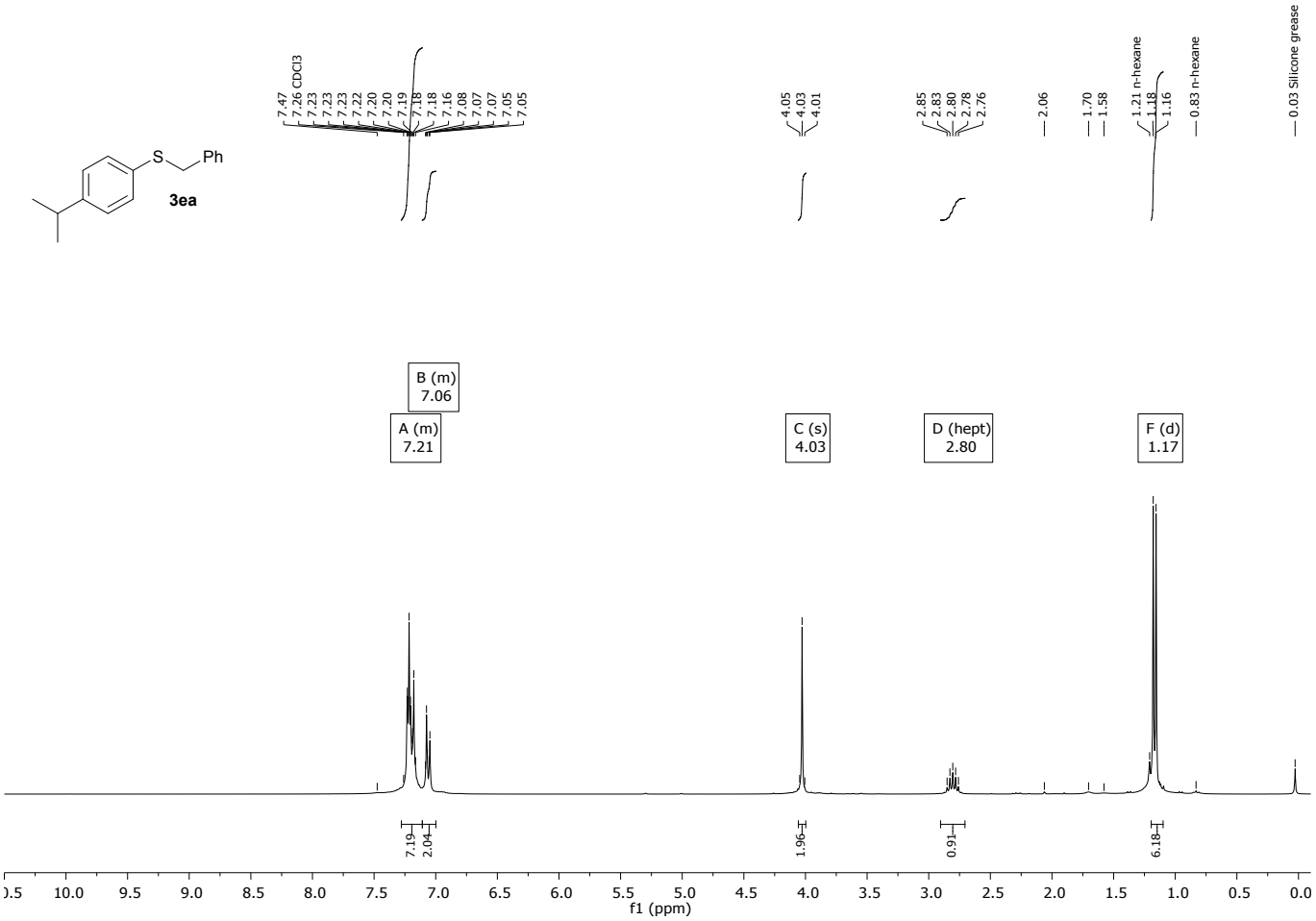

137.72 134.29 134.08 132.25 129.19 128.87 128.65 128.08 128.03 127.57 127.50 126.81 126.07
77.79 CDCl3 77.37 CDCl3 76.94 CDCl3
39.30
30.04 n-hexane
1.38 Silicone grease
S Ph
3ba
f1 (ppm)

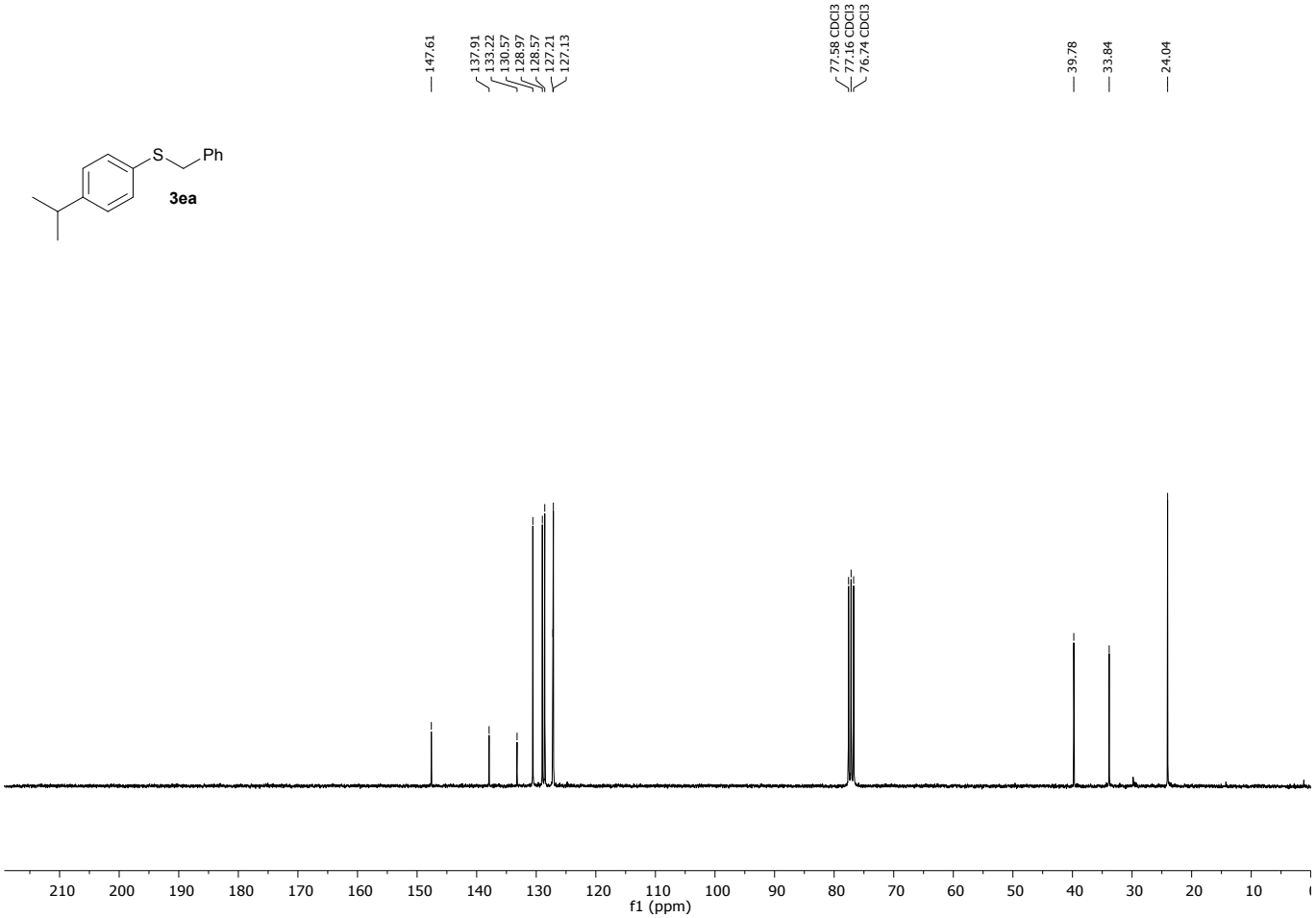

7.26 CDCl3
7.23
7.23
7.22
7.21
7.21
7.21
7.20
7.20
7.18
7.17
7.16
7.16
7.15
7.14
7.13
7.13
7.01
6.98
4.00
2.24
1.54 HDO
1.21 n-hexane
0.80 n-hexane
0.04 Silicon grease
S
Ph
3ca
B (d)
6.99
A (m)
7.18
C (s)
4.00
D (s)
2.24
6.70
2.27
2.30
3.15
f1 (ppm)

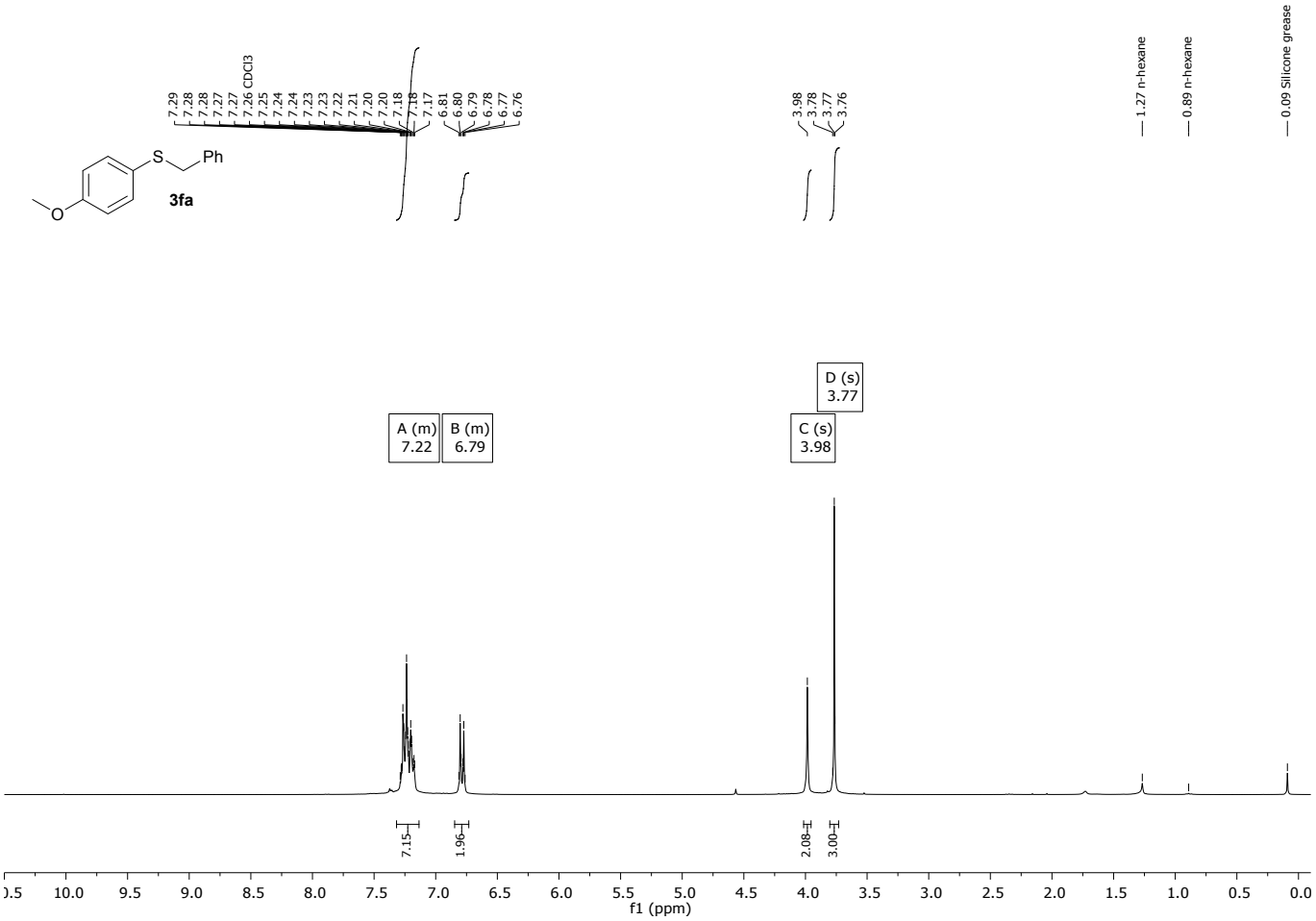

138.15
136.86
132.85
131.04
129.92
129.15
128.74
127.37
77.79 CDCl3
77.36 CDCl3
76.94 CDCl3
40.12
21.34
S
Ph
3ca
f1 (ppm)

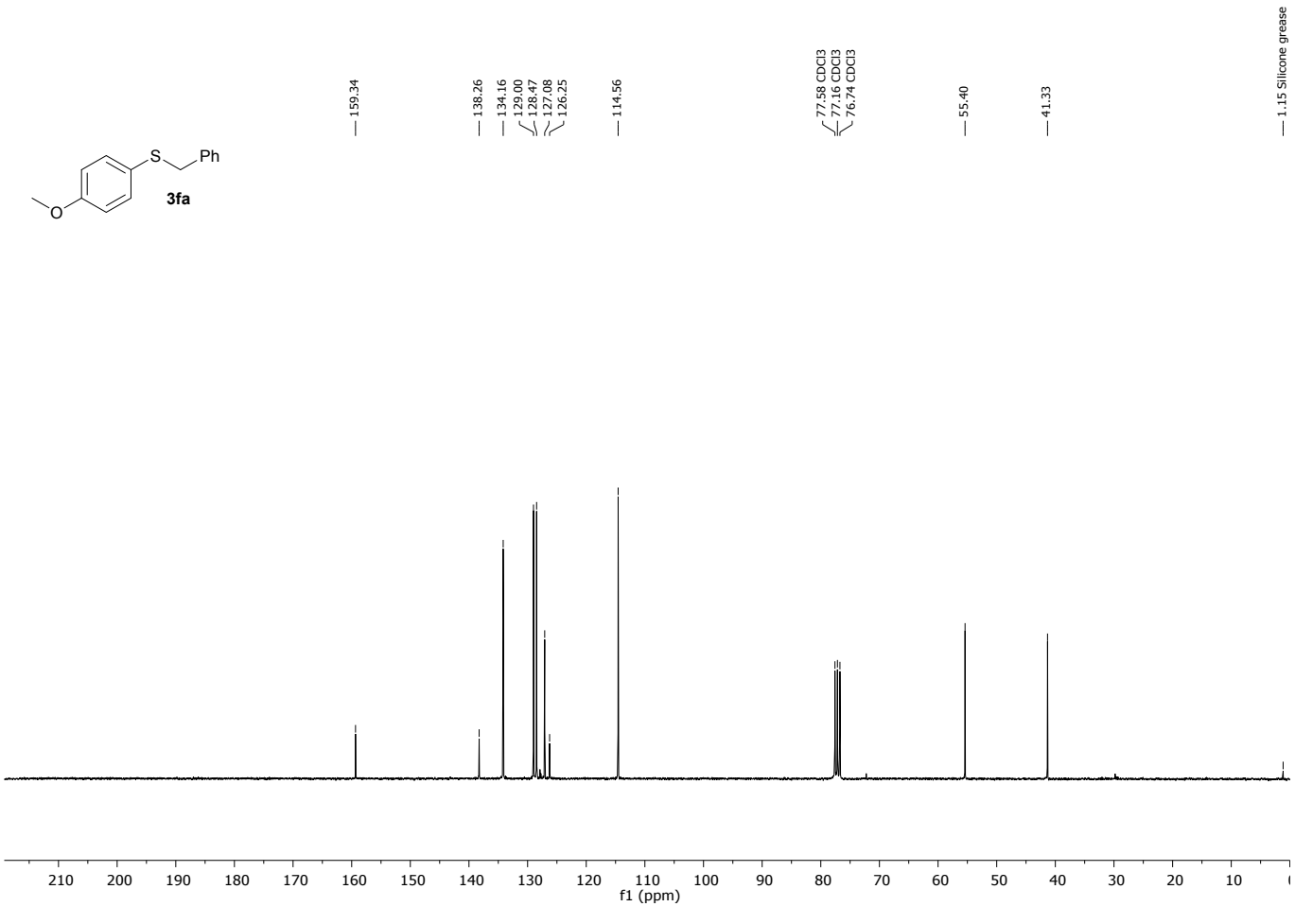

S
Ph
3da
7.32
7.31
7.30
7.29
7.28
7.28
7.27
7.26 CDCl3
7.25
7.25
7.24
7.23
7.23
7.22
6.93
6.81
4.10
2.25
1.26 n-hexane
0.88 n-hexane
0.08 Silicone grease
B (s)
6.93
A (m)
7.27
C (s)
6.81
D (s)
4.10
E (s)
2.25
5.36
1.84
1.02
1.92
6.16
f1 (ppm)

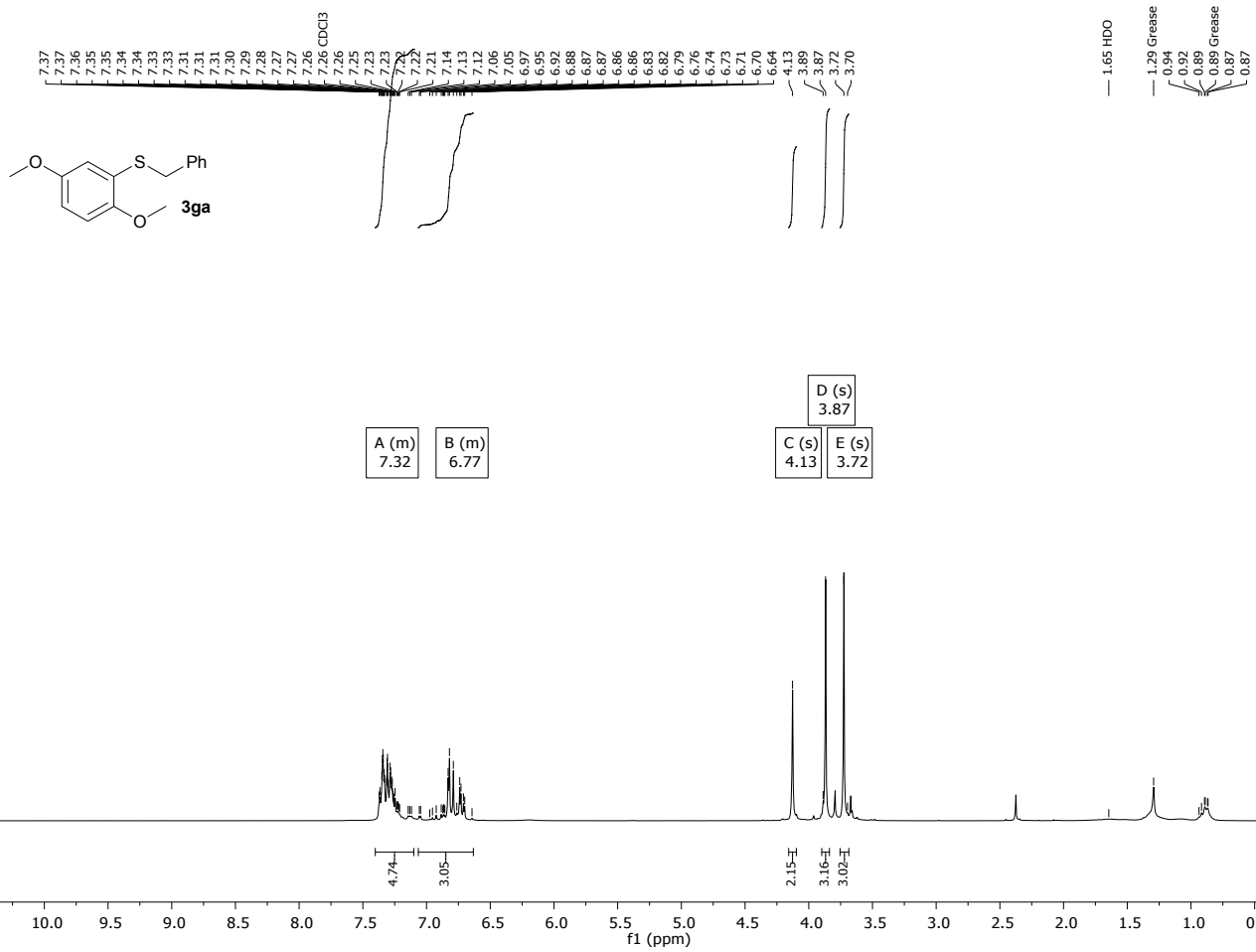

138.53
137.75
136.14
129.00
128.57
128.28
127.44
127.24
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
39.12
21.31
S
Ph
3da
f1 (ppm)

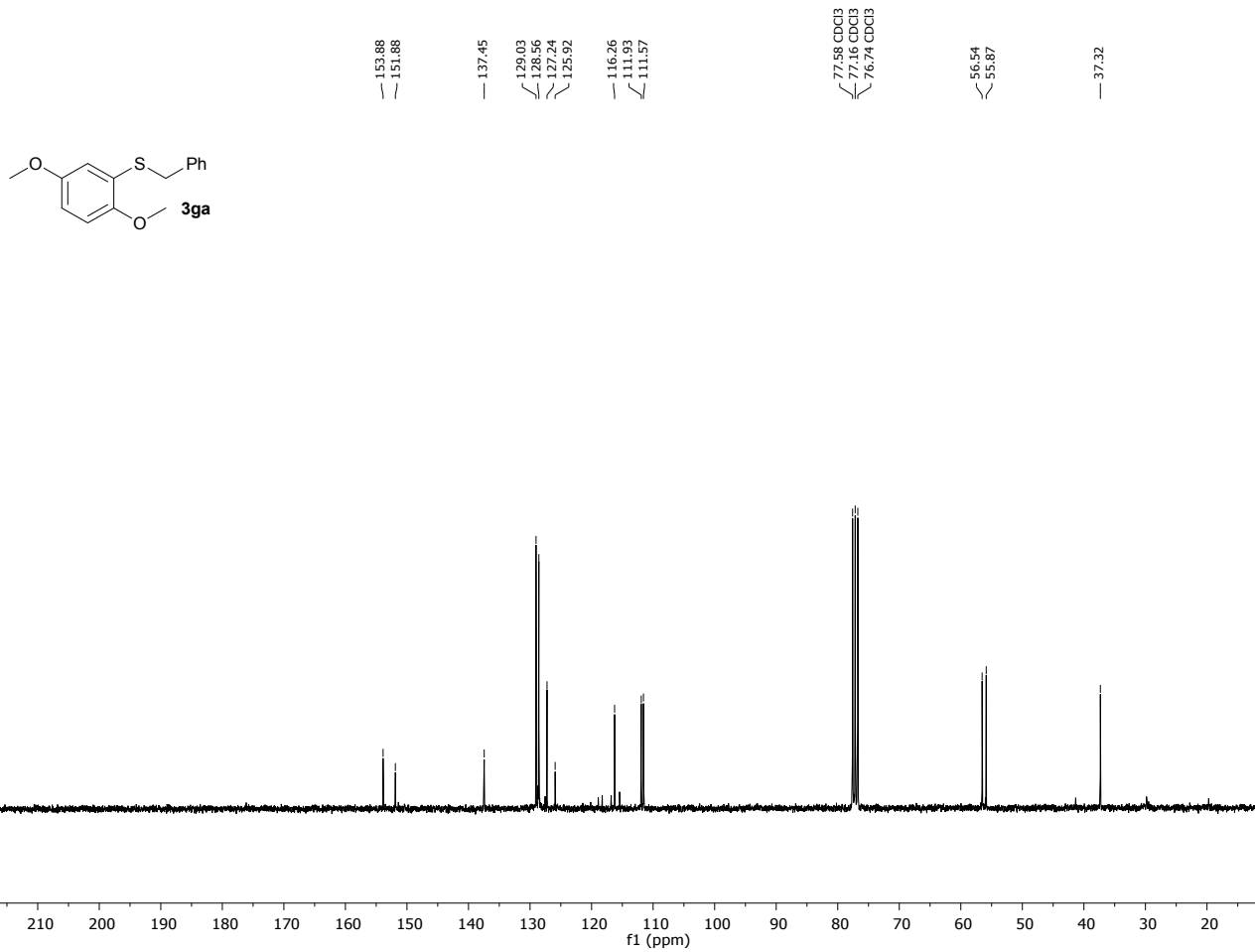

3ea
7.47
7.26 CDCl3
7.23
7.23
7.23
7.22
7.20
7.20
7.19
7.18
7.18
7.16
7.08
7.07
7.07
7.05
7.05
4.05
4.03
4.01
2.85
2.83
2.80
2.78
2.76
2.06
1.70
1.58
1.21 n-hexane
1.18
1.16
0.83 n-hexane
0.03 Silicone grease
B (m)
7.06
A (m)
7.21
C (s)
4.03
D (hept)
2.80
F (d)
1.17
7.19
2.04
1.96
0.91
6.18
f1 (ppm)

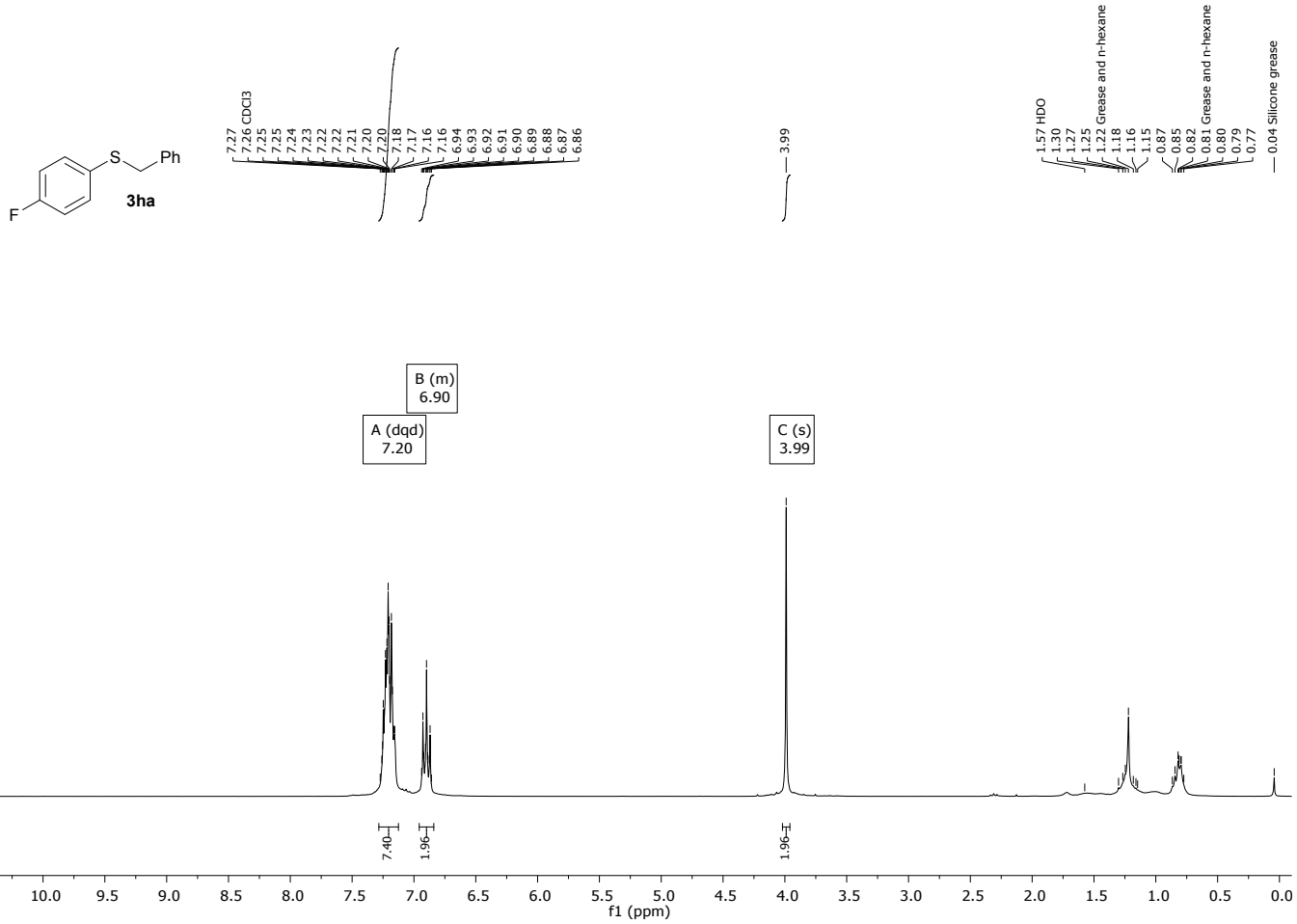

3ea
147.61
137.91
133.22
130.57
128.97
128.57
127.21
127.13
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
39.78
33.84
24.04
f1 (ppm)

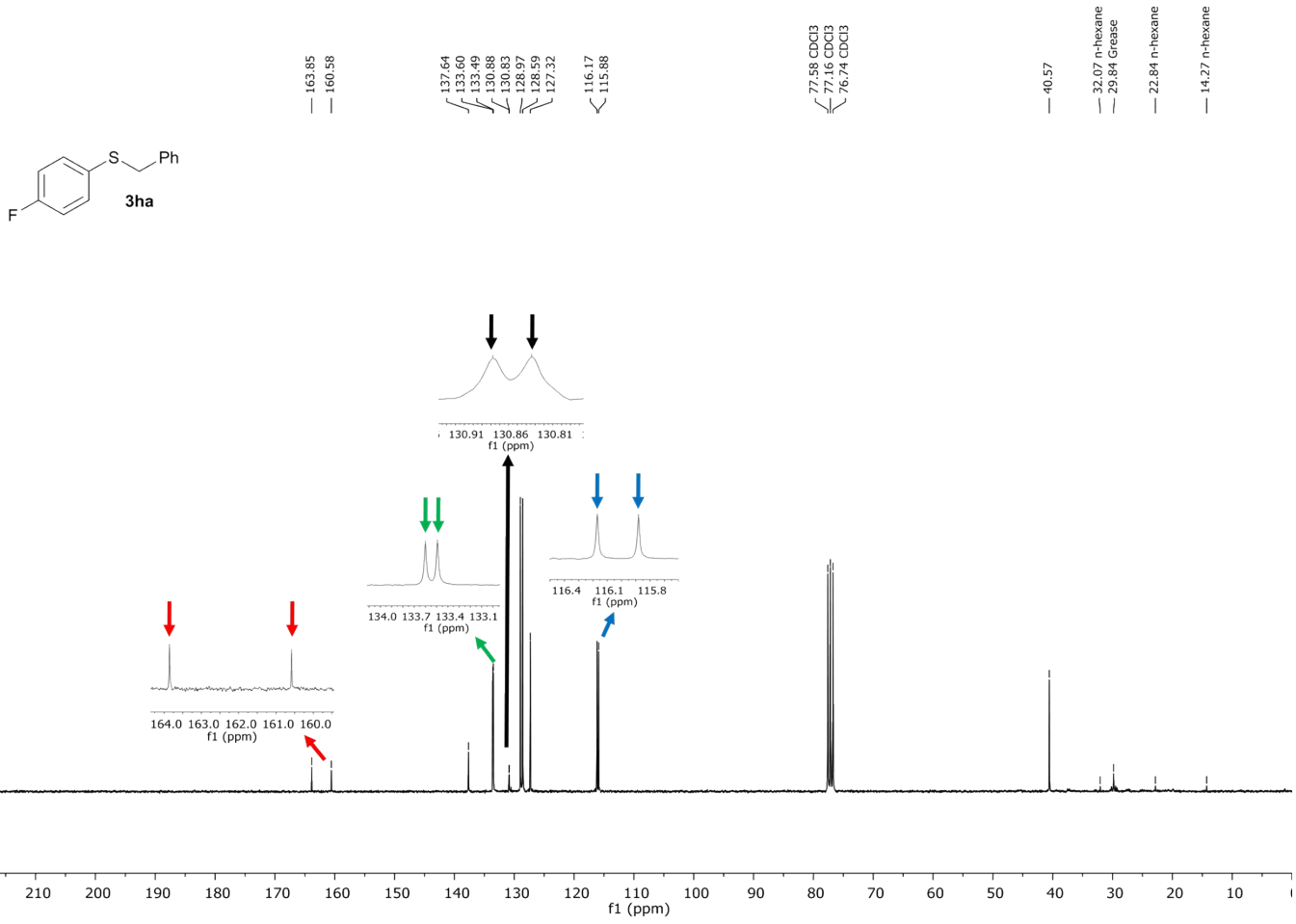

7.29
7.28
7.28
7.27
7.27
7.26 CDCl3
7.25
7.24
7.24
7.23
7.23
7.22
7.21
7.20
7.20
7.18
7.18
7.17
6.81
6.80
6.79
6.78
6.77
6.76
3.98
3.78
3.77
3.76
1.27 n-hexane
0.89 n-hexane
0.09 Silicone grease
S
Ph
O
3fa
A (m)
7.22
B (m)
6.79
C (s)
3.98
D (s)
3.77
7.15
1.96
2.08
3.00
f1 (ppm)

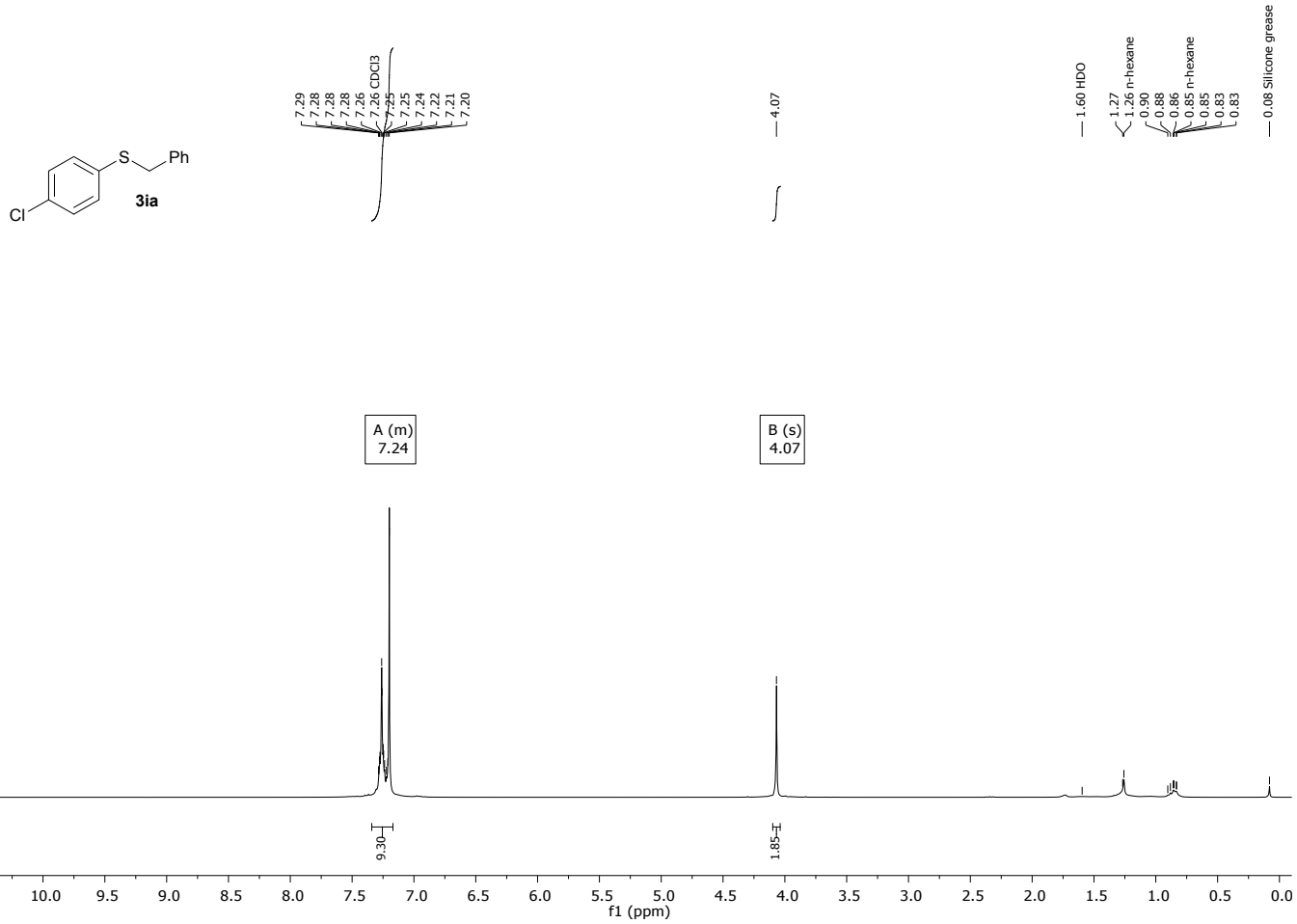

159.34
138.26
134.16
129.00
128.47
127.08
126.25
114.56
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
55.40
41.33
1.15 Silicone grease
S
Ph
O
3fa
f1 (ppm)

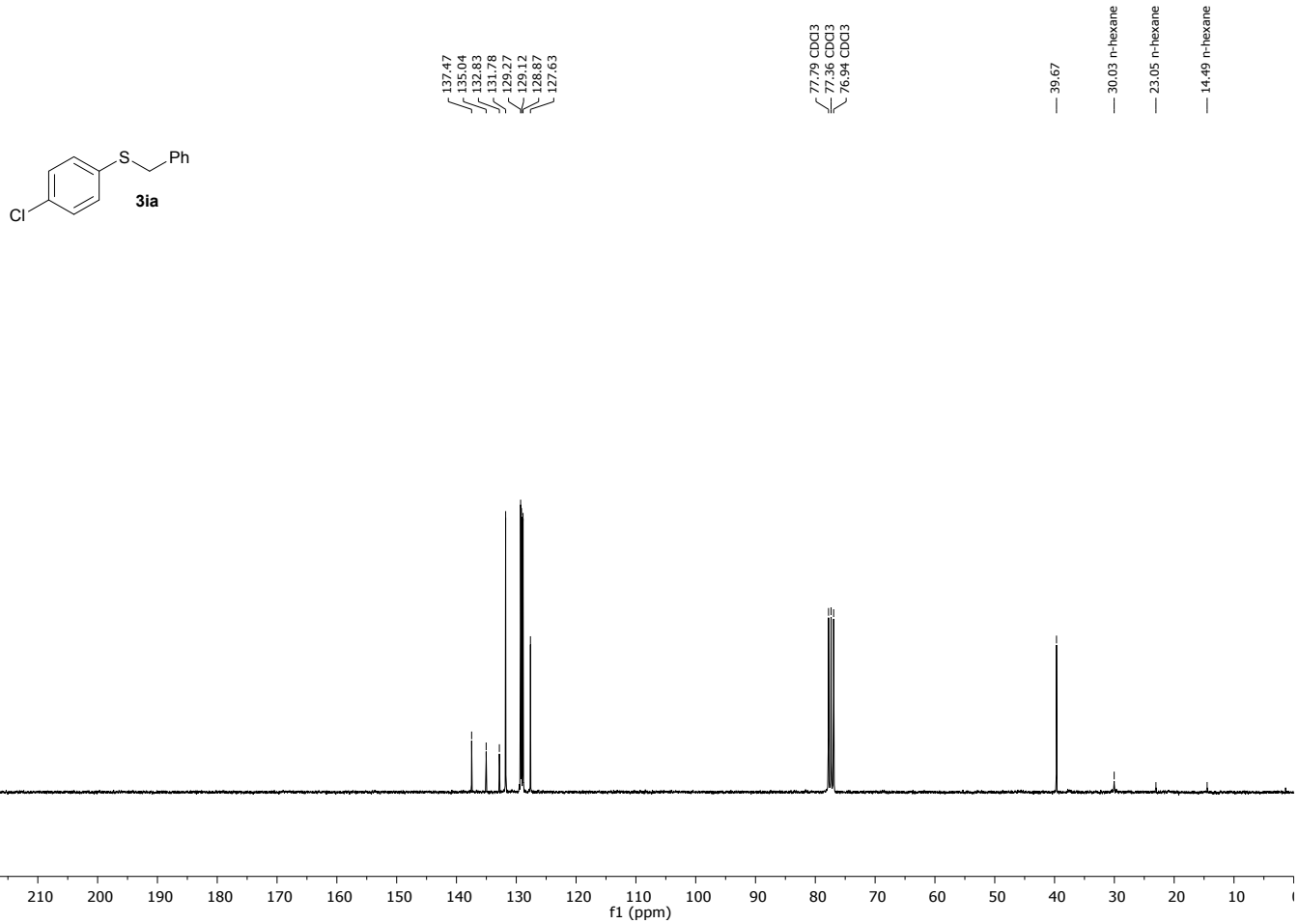

7.37 7.37 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.31 7.31 7.31 7.30 7.29 7.28 7.27 7.27 7.26 7.26 CDCl3 7.26 7.25 7.23 7.23 7.22 7.22 7.21 7.14 7.13 7.12 7.06 7.05 6.97 6.95 6.92 6.88 6.87 6.87 6.86 6.86 6.83 6.82 6.79 6.76 6.74 6.73 6.71 6.70 6.64 4.13 3.89 3.87 3.72 3.70
1.65 HDO
1.29 Grease 0.94 0.92 0.89 0.89 Grease 0.87 0.87
O S Ph O
3ga
A (m) 7.32
B (m) 6.77
D (s) 3.87
C (s) 4.13
E (s) 3.72
4.74 3.05 2.15 3.16 3.02
f1 (ppm)

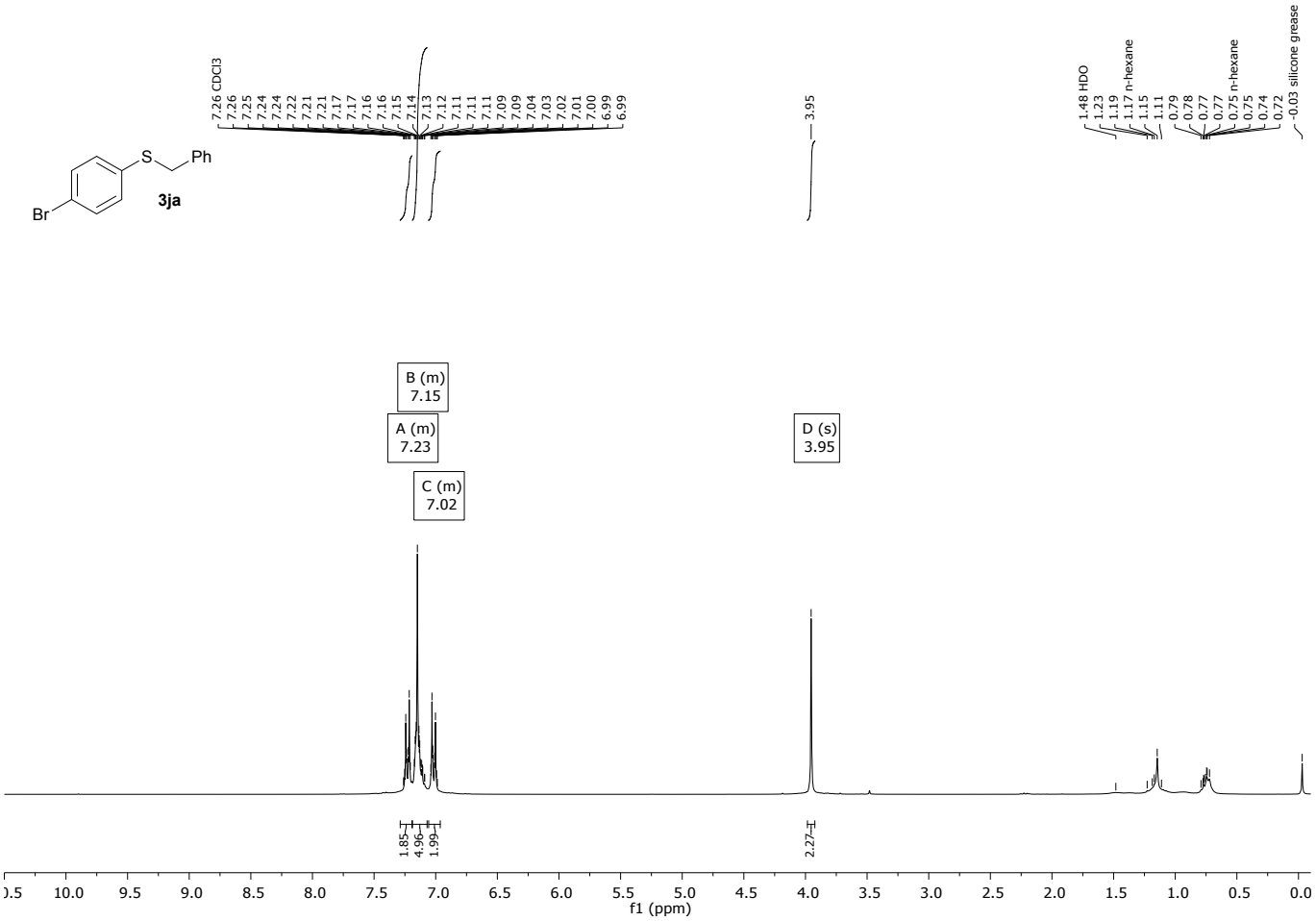

153.88 151.88 137.45 129.03 128.56 127.24 125.92 116.26 111.93 111.57 77.58 CDCl3 77.16 CDCl3 76.74 CDCl3 56.54 55.87 37.32
O S Ph O
3ga
f1 (ppm)

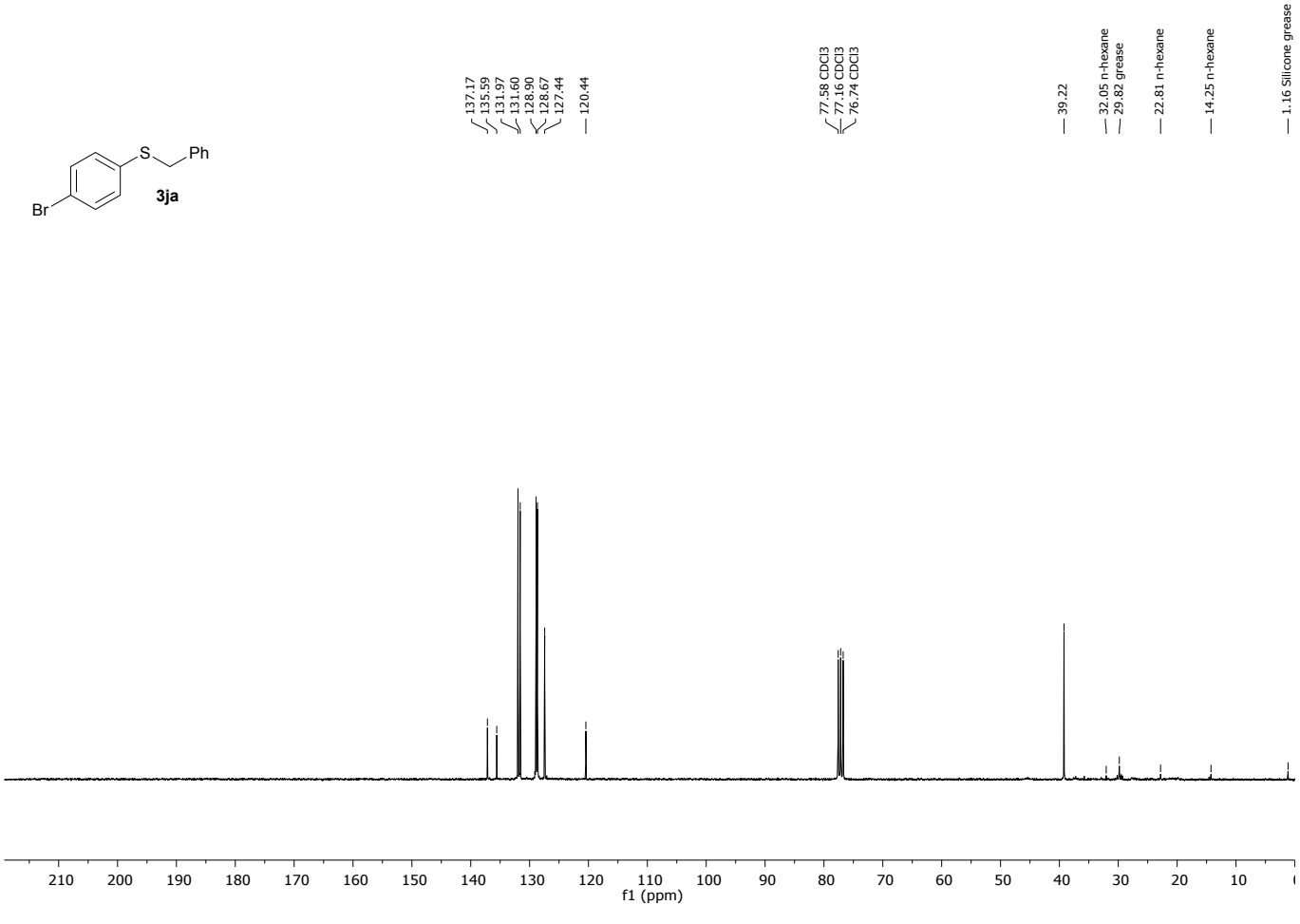

S
Ph
F
3ha
7.27
7.26 CDCl3
7.25
7.24
7.23
7.22
7.21
7.20
7.18
7.17
7.16
6.94
6.93
6.92
6.91
6.90
6.89
6.88
6.87
6.86
3.99
1.57 HDO
1.30
1.27
1.25
1.22 Grease and n-hexane
1.18
1.16
1.15
0.87
0.85
0.82
0.81 Grease and n-hexane
0.80
0.79
0.77
0.04 Silicone grease
B (m)
6.90
A (dqd)
7.20
C (s)
3.99
7.40
1.96
1.96
f1 (ppm)

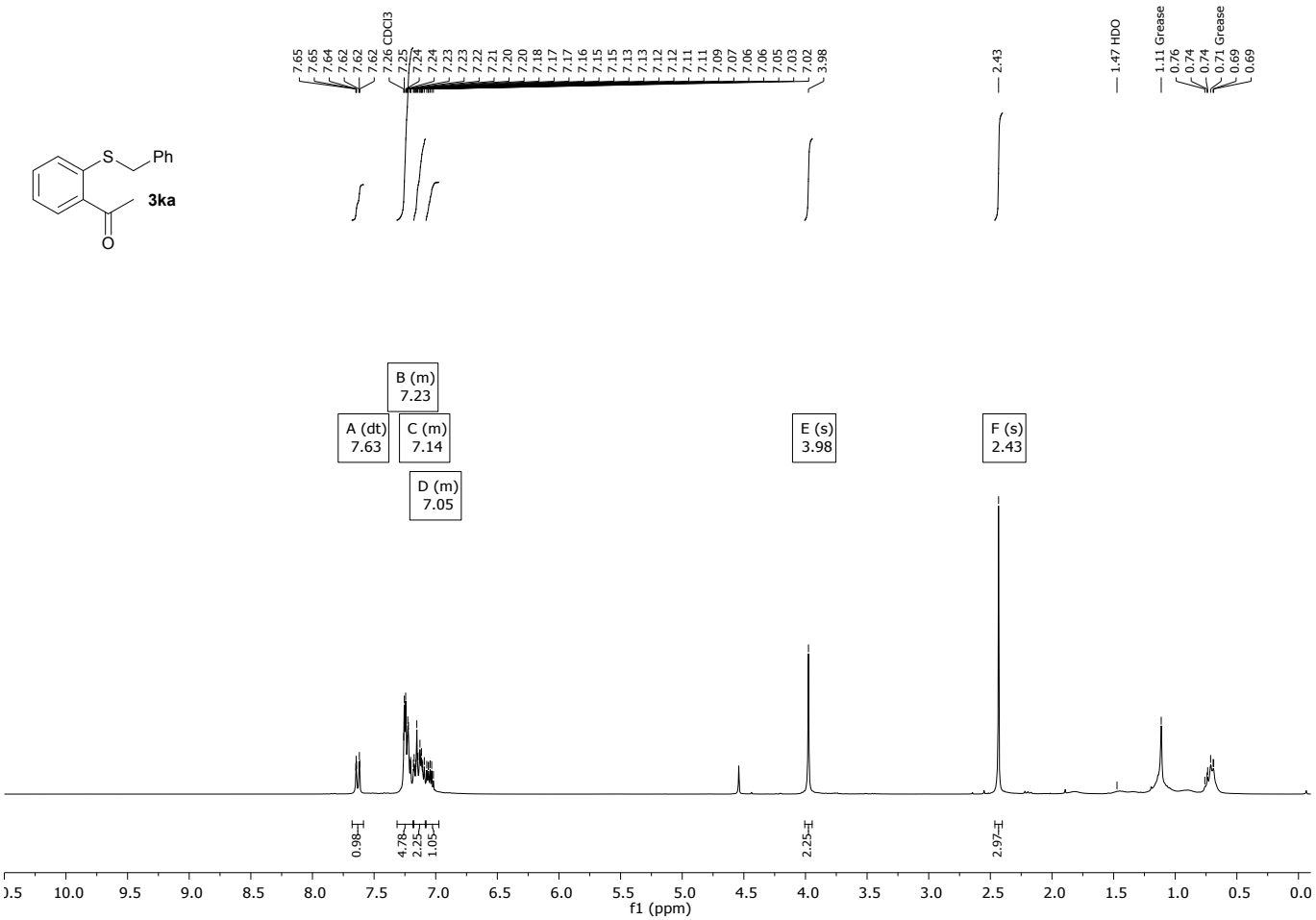

163.85
160.58
137.64
133.60
133.49
130.88
130.83
128.97
128.59
127.32
116.17
115.88
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
40.57
32.07 n-hexane
29.84 Grease
22.84 n-hexane
14.27 n-hexane
S
Ph
F
3ha
130.91 130.86 130.81
f1 (ppm)
134.0 133.7 133.4 133.1
f1 (ppm)
116.4 116.1 115.8
f1 (ppm)
164.0 163.0 162.0 161.0 160.0
f1 (ppm)
f1 (ppm)

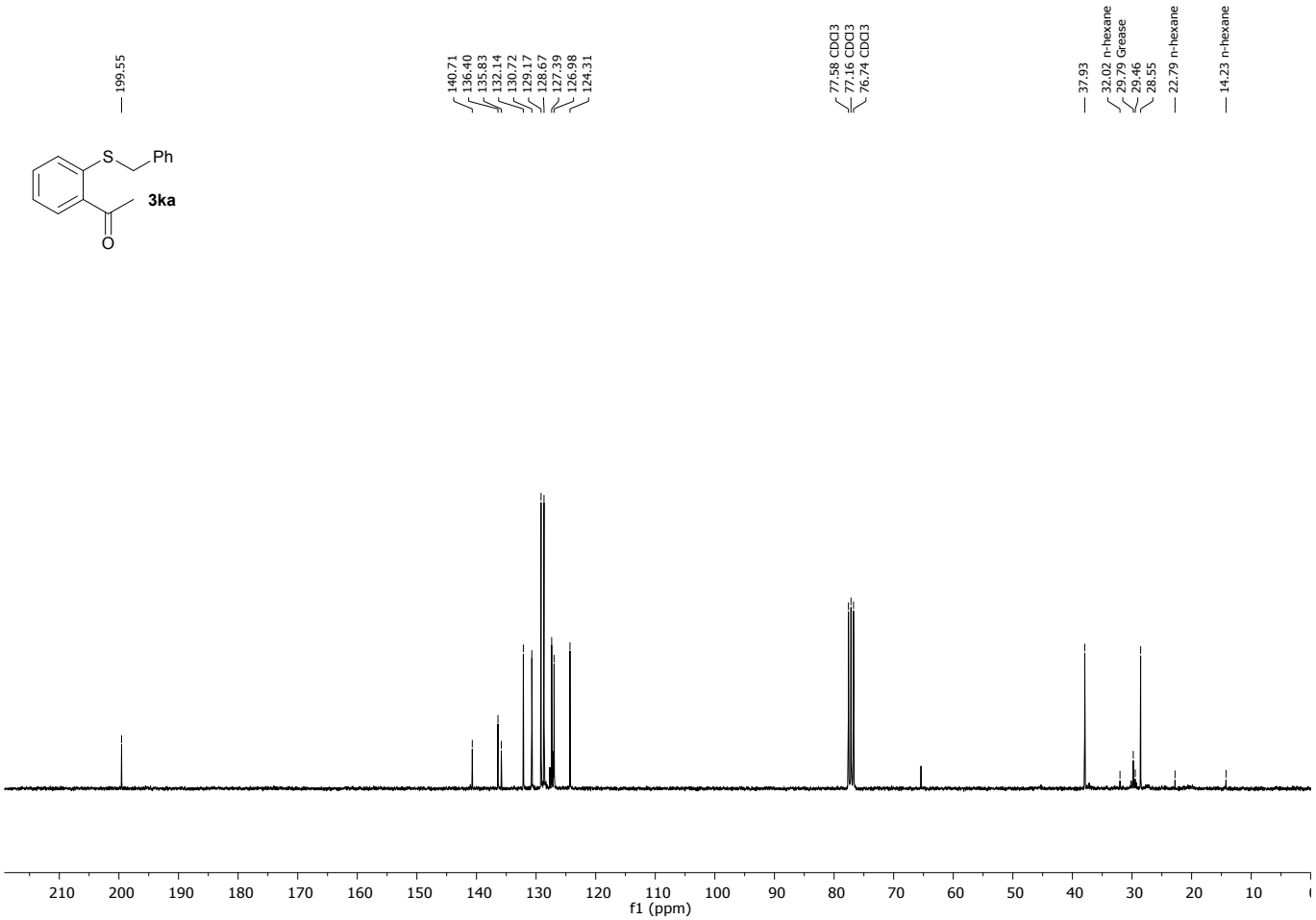

S
Ph
Cl
3ia
7.29
7.28
7.28
7.28
7.26
7.26 CDCl3
7.25
7.25
7.24
7.22
7.21
7.20
4.07
1.60 HDO
1.27
1.26 n-hexane
0.90
0.88
0.86
0.85 n-hexane
0.85
0.83
0.83
0.08 Silicone grease
A (m)
7.24
B (s)
4.07
9.30
1.85
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

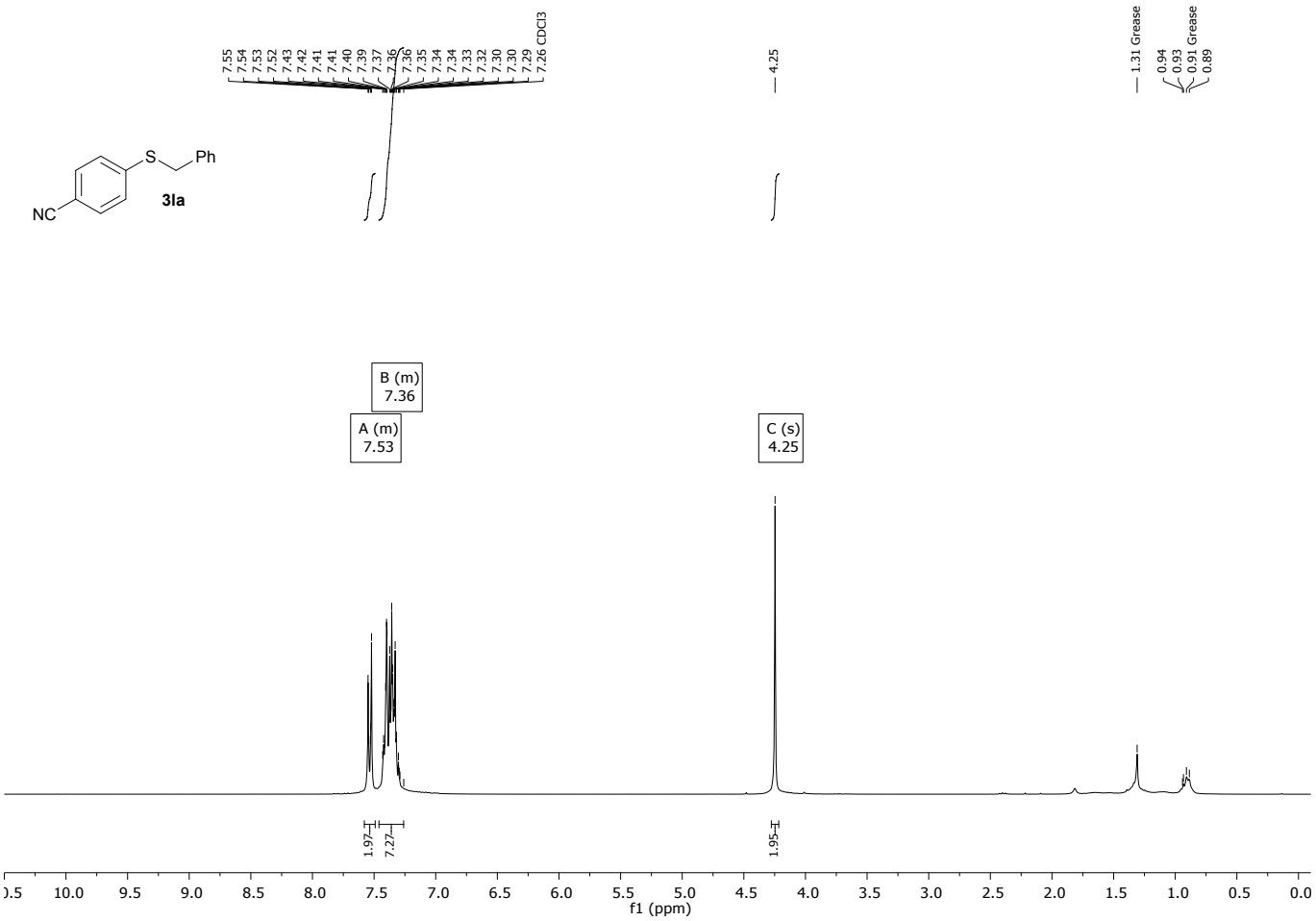

137.47
135.04
132.83
131.78
129.27
129.12
128.87
127.63
77.79 CDCl3
77.36 CDCl3
76.94 CDCl3
39.67
30.03 n-hexane
23.05 n-hexane
14.49 n-hexane
S
Ph
Cl
3ia
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

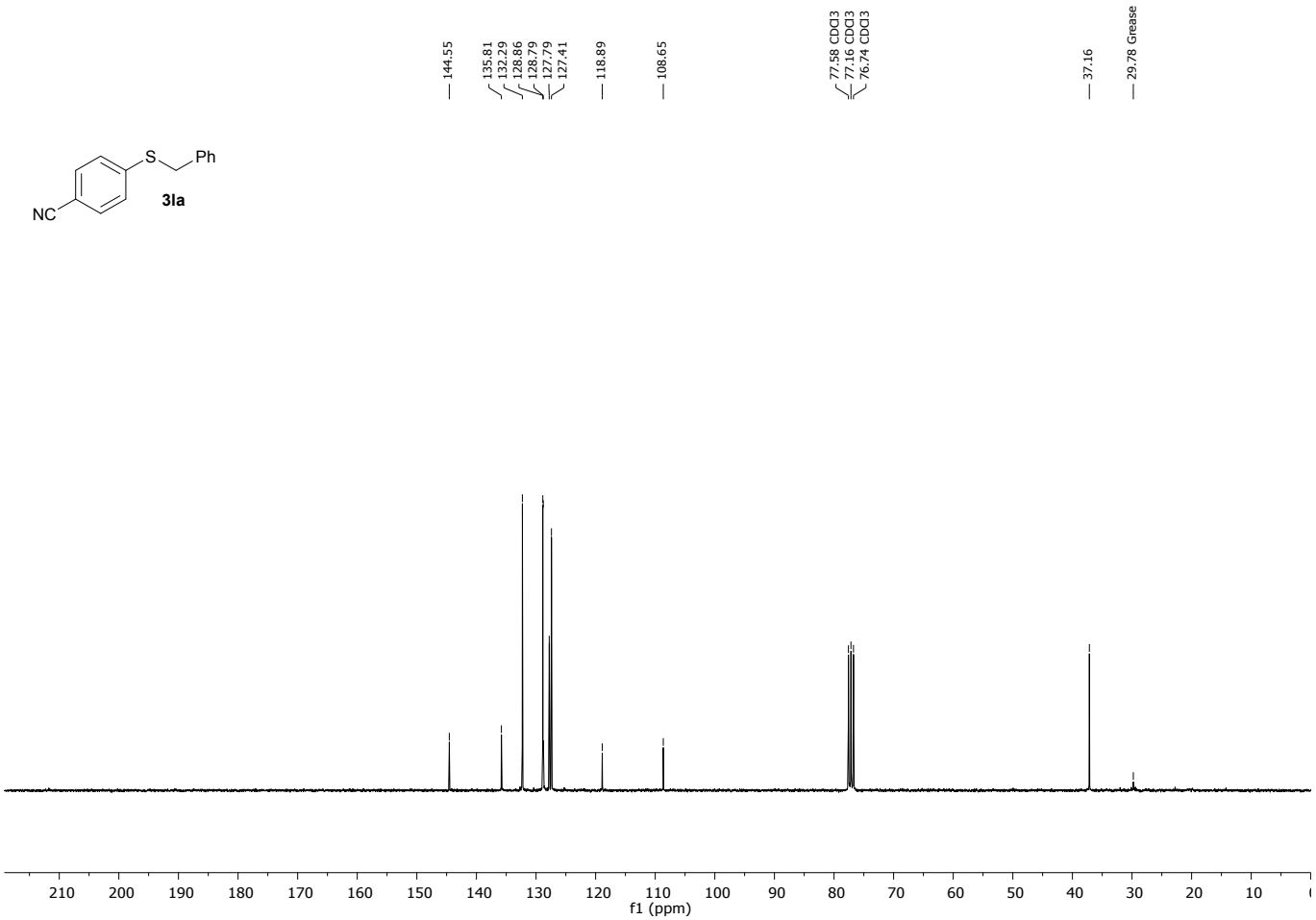

7.26 CDCl3
7.26
7.25
7.24
7.24
7.22
7.21
7.21
7.17
7.17
7.16
7.16
7.15
7.14
7.13
7.12
7.11
7.11
7.11
7.09
7.09
7.04
7.03
7.02
7.01
7.00
6.99
6.99
3.95
1.48 HDO
1.23
1.19
1.17 n-hexane
1.15
1.11
0.79
0.78
0.77
0.77
0.75 n-hexane
0.75
0.74
0.72
-0.03 silicone grease
S
Ph
Br
3ja
B (m)
7.15
A (m)
7.23
C (m)
7.02
D (s)
3.95
1.85
4.96
1.99
2.27
f1 (ppm)

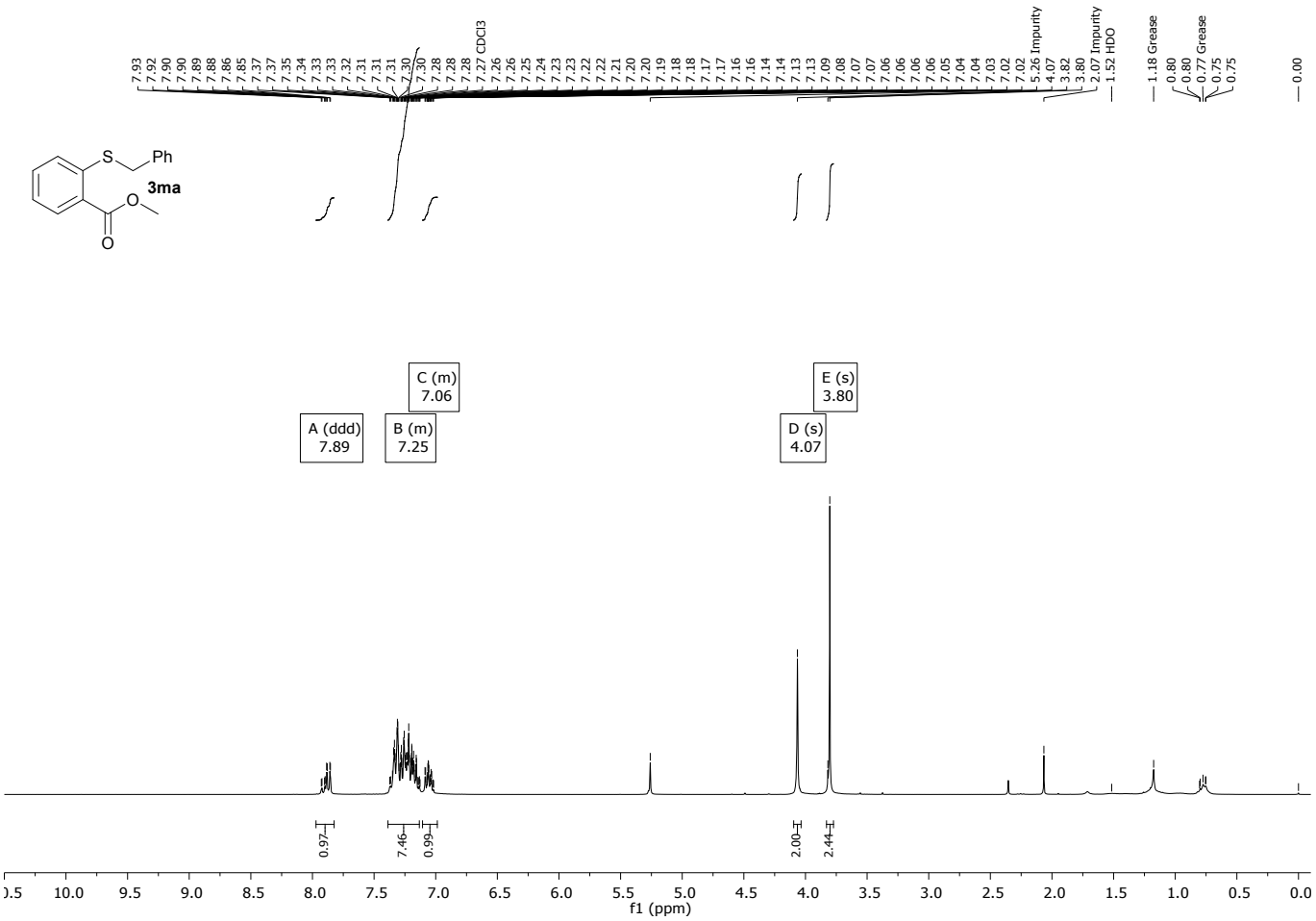

137.17
135.59
131.97
131.60
128.90
128.67
127.44
120.44
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
39.22
32.05 n-hexane
29.82 grease
22.81 n-hexane
14.25 n-hexane
1.16 Silicone grease
S
Ph
Br
3ja
f1 (ppm)

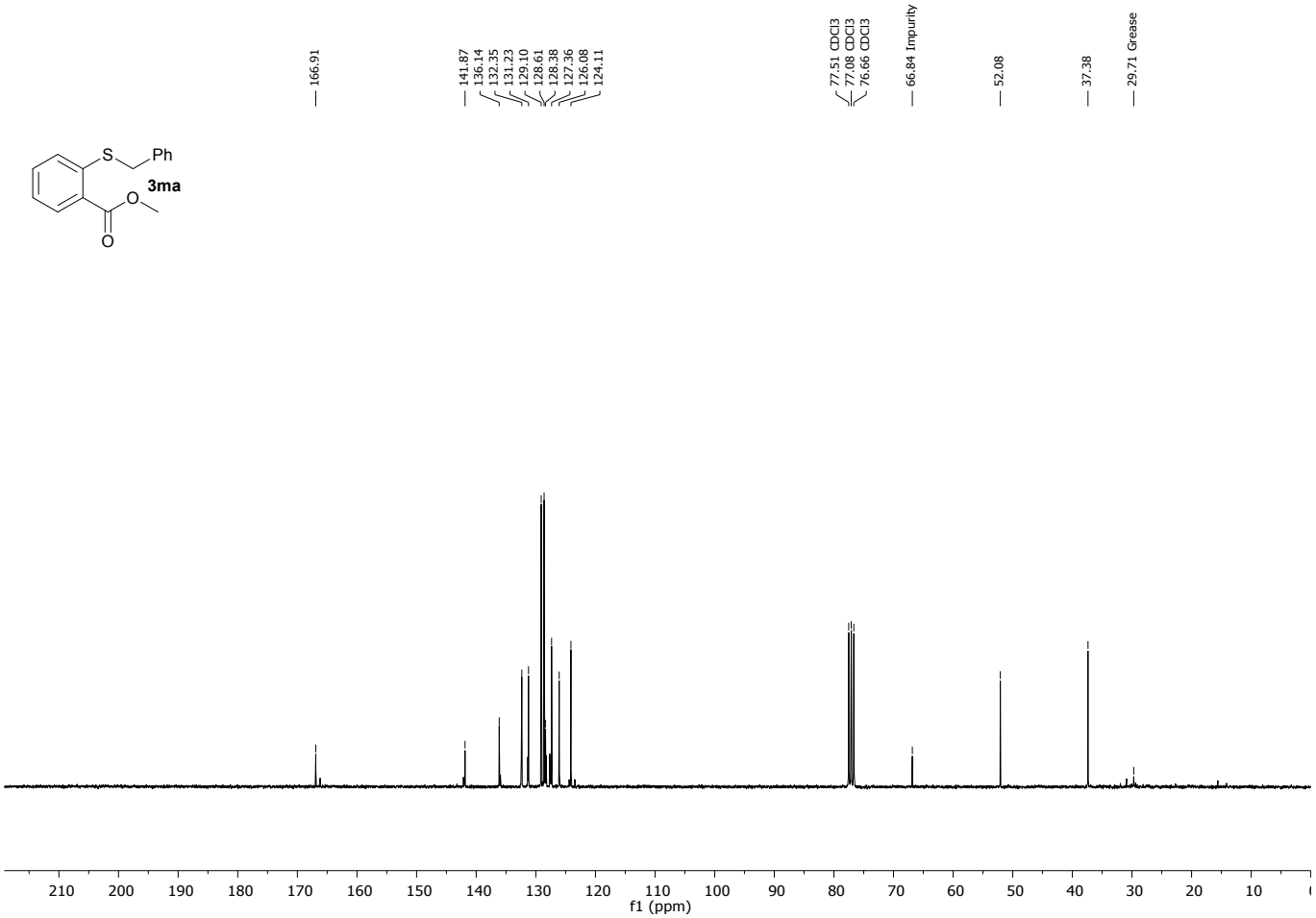

7.65
7.65
7.64
7.62
7.62
7.62
7.26 CDCl3
7.25
7.24
7.24
7.23
7.23
7.22
7.21
7.20
7.20
7.18
7.17
7.17
7.16
7.15
7.15
7.13
7.13
7.12
7.12
7.11
7.11
7.09
7.07
7.06
7.06
7.05
7.03
7.02
3.98
2.43
1.47 HDO
1.11 Grease
0.76
0.74
0.74
0.71 Grease
0.69
0.69
S
Ph
3ka
O
B (m)
7.23
A (dt)
7.63
C (m)
7.14
D (m)
7.05
E (s)
3.98
F (s)
2.43
0.98
4.78
2.25
1.05
2.25
2.97
f1 (ppm)

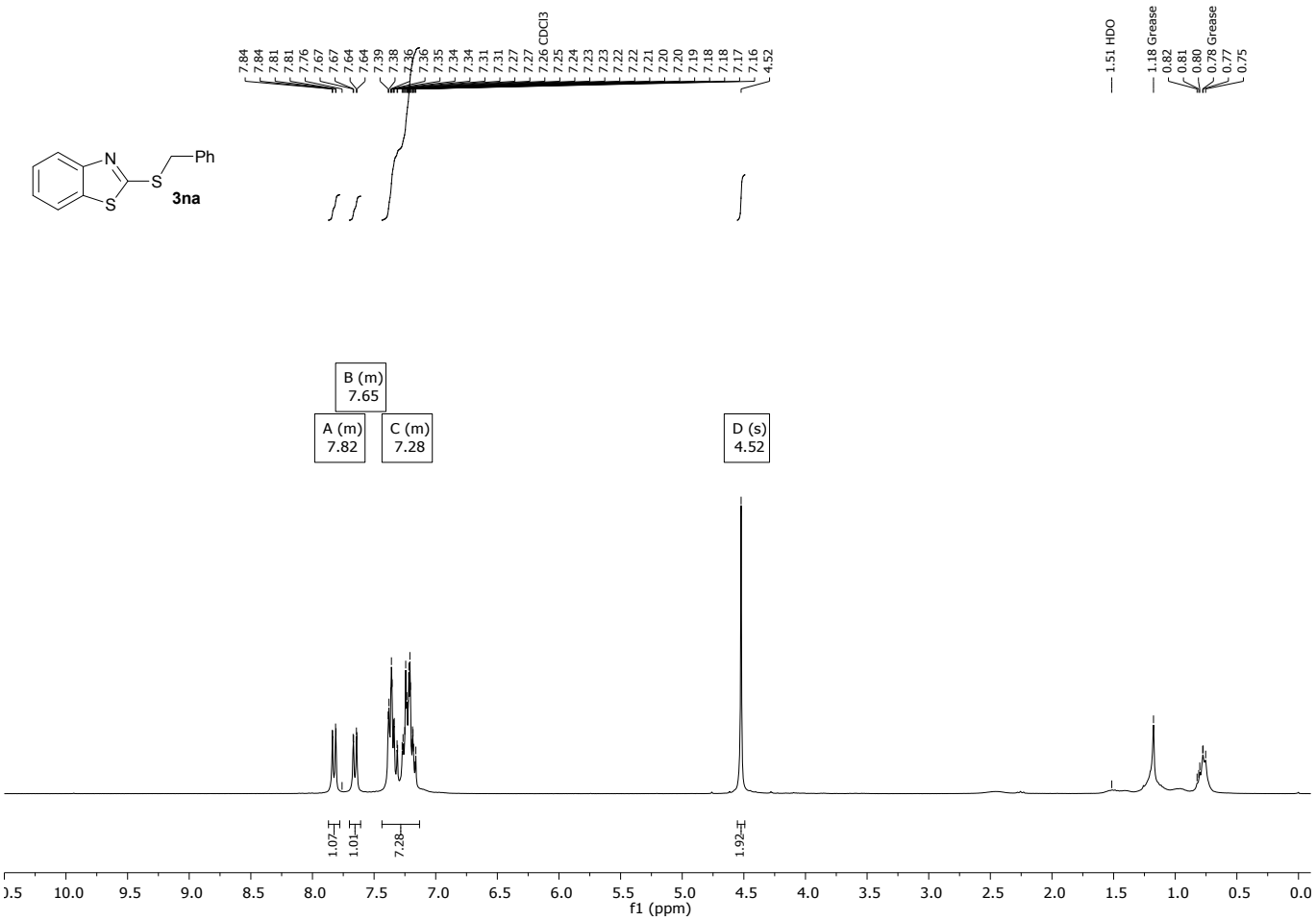

199.55
140.71
136.40
135.83
132.14
130.72
129.17
128.67
127.39
126.98
124.31
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
37.93
32.02 n-hexane
29.79 Grease
29.46
28.55
22.79 n-hexane
14.23 n-hexane
S
Ph
3ka
O
f1 (ppm)

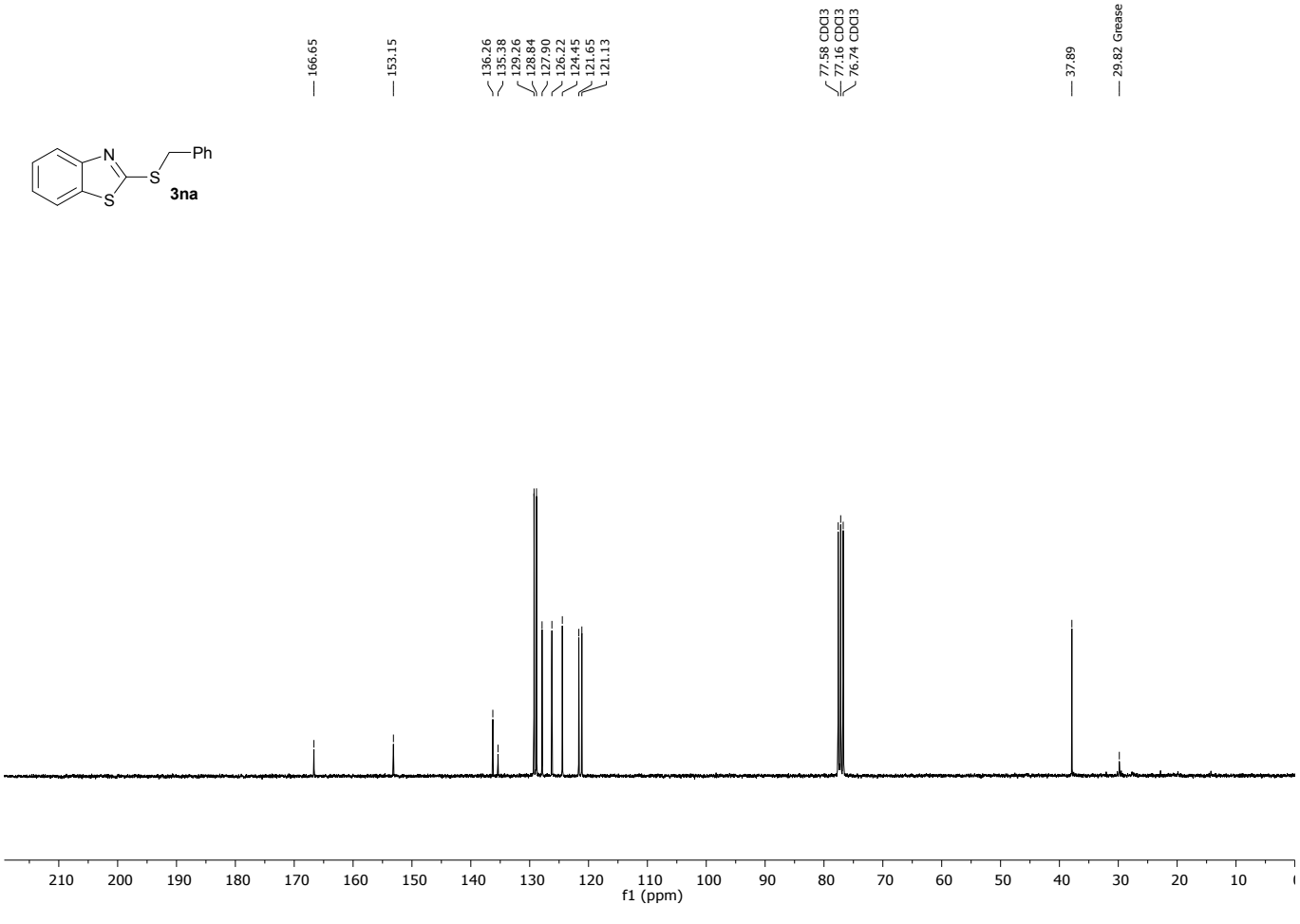

7.55
7.54
7.53
7.52
7.43
7.42
7.41
7.41
7.40
7.39
7.37
7.36
7.36
7.35
7.34
7.34
7.33
7.32
7.30
7.30
7.29
7.26 CDCl3
4.25
1.31 Grease
0.94
0.93
0.91 Grease
0.89
S
Ph
NC
3la
B (m)
7.36
A (m)
7.53
C (s)
4.25
1.97
2.27
1.95
f1 (ppm)


144.55
135.81
132.29
128.86
128.79
127.79
127.41
118.89
108.65
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
37.16
29.78 Grease
S
Ph
NC
3la
f1 (ppm)


3ma
A (ddd) 7.89
B (m) 7.25
C (m) 7.06
D (s) 4.07
E (s) 3.80
f1 (ppm)


3ma
f1 (ppm)


7.84 7.84 7.81 7.81 7.76 7.67 7.64 7.64 7.39 7.38 7.36 7.36 7.35 7.34 7.31 7.31 7.27 7.27 7.26 CDCl3 7.25 7.24 7.23 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.18 7.18 7.17 7.16 4.52
1.51 HDO
1.18 Grease
0.82
0.81
0.80
0.78 Grease
0.77
0.75
N
S
S
Ph
3na
B (m) 7.65
A (m) 7.82
C (m) 7.28
D (s) 4.52
1.07
1.01
7.28
1.92
f1 (ppm)


166.65
153.15
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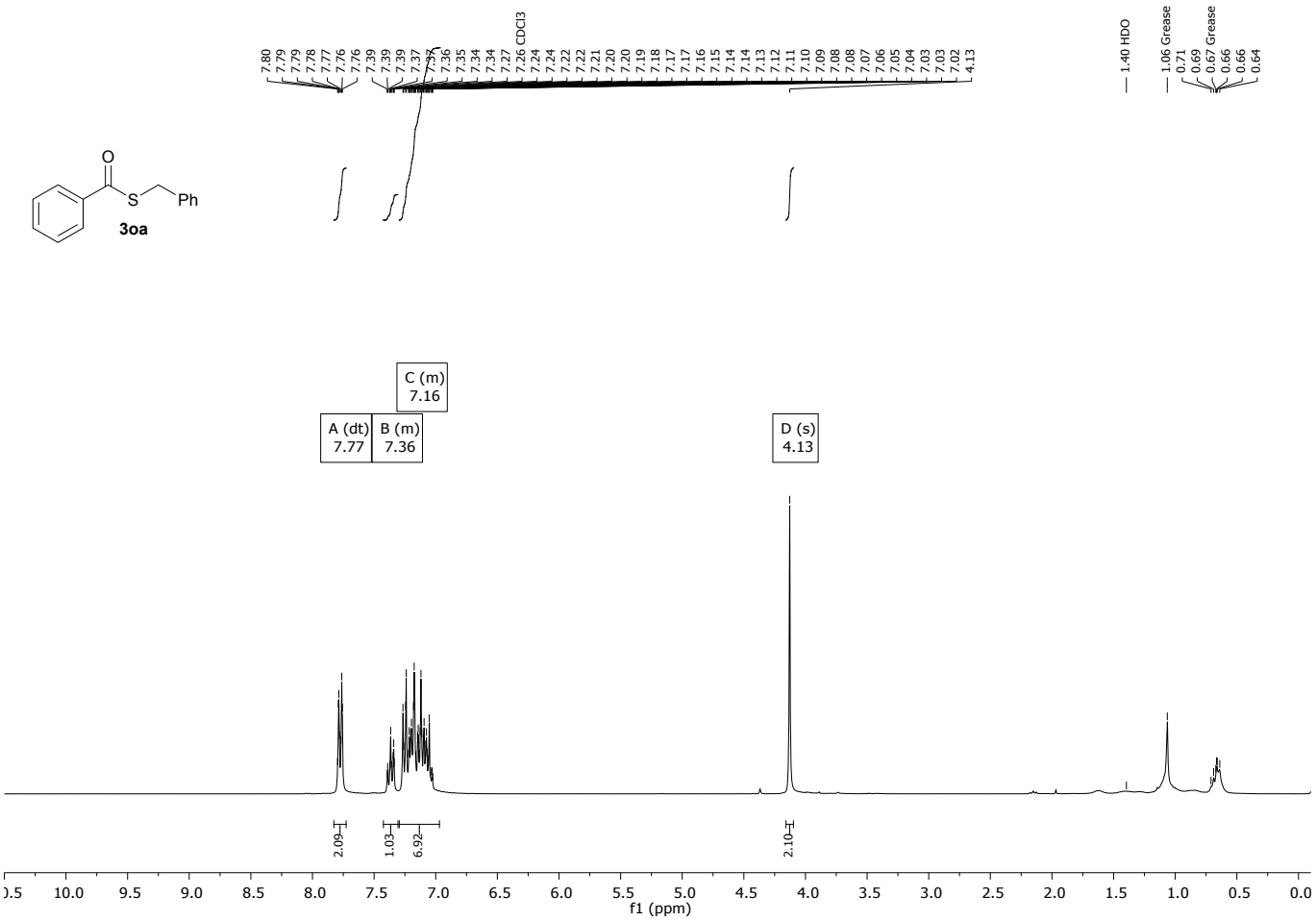
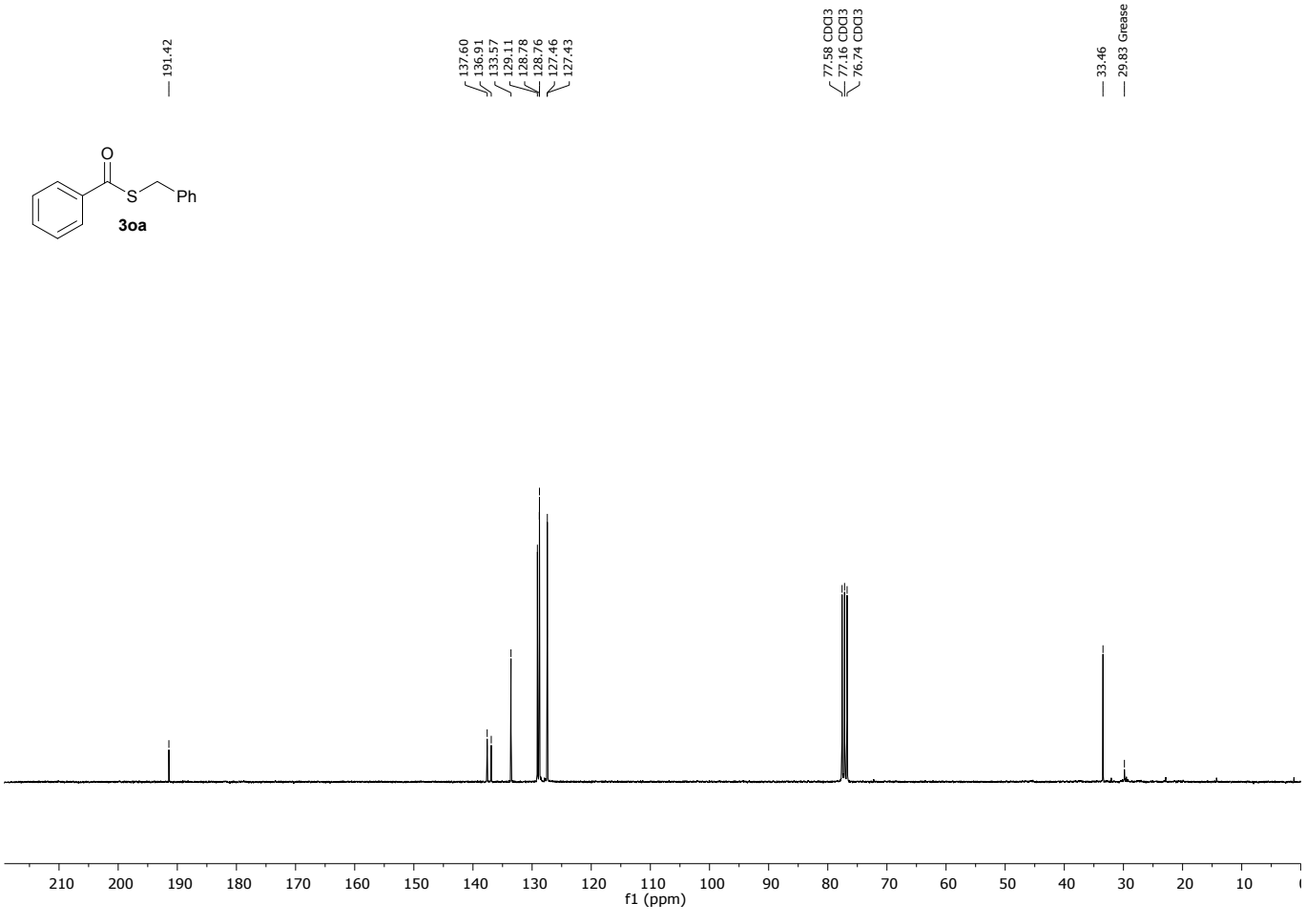
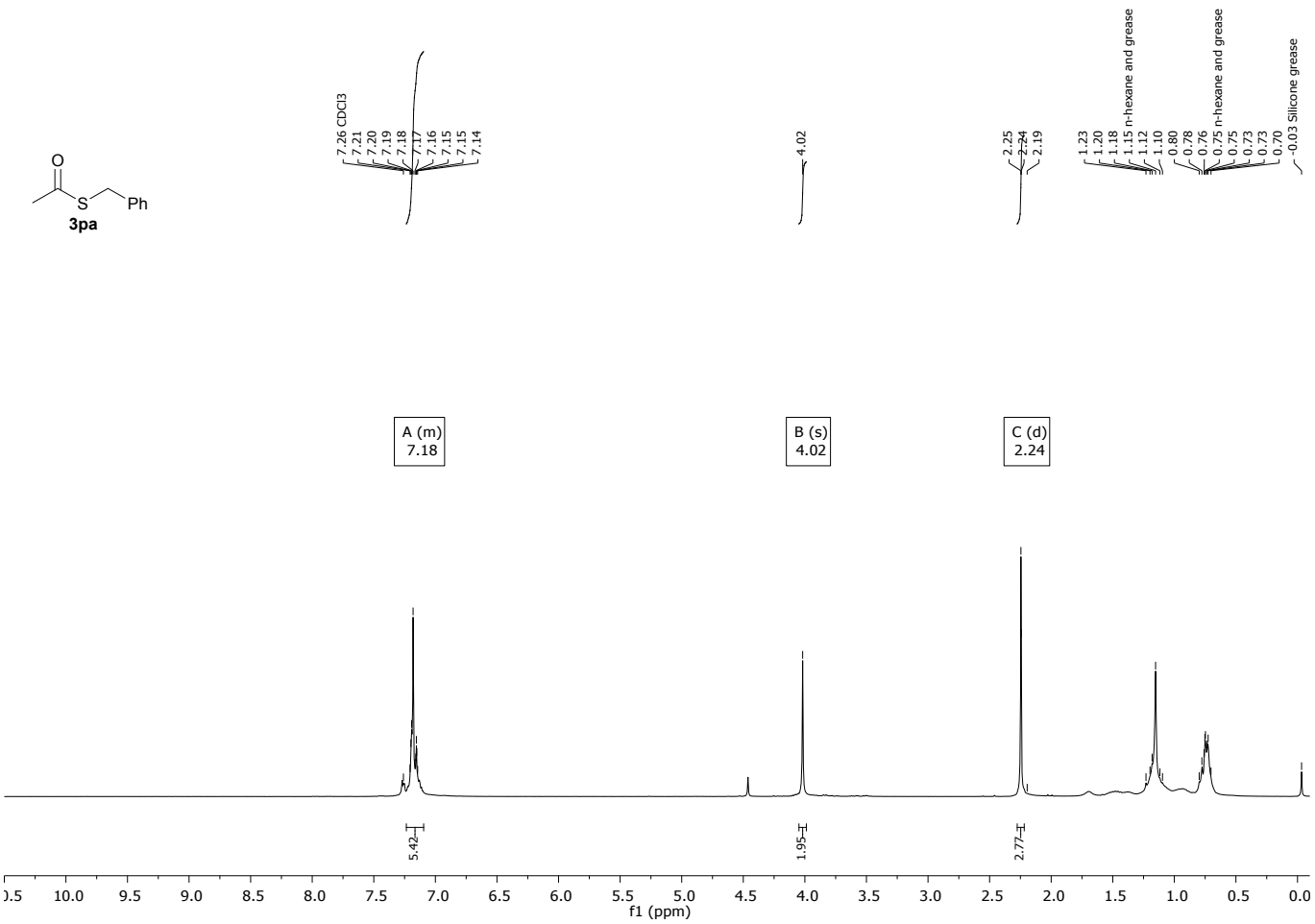
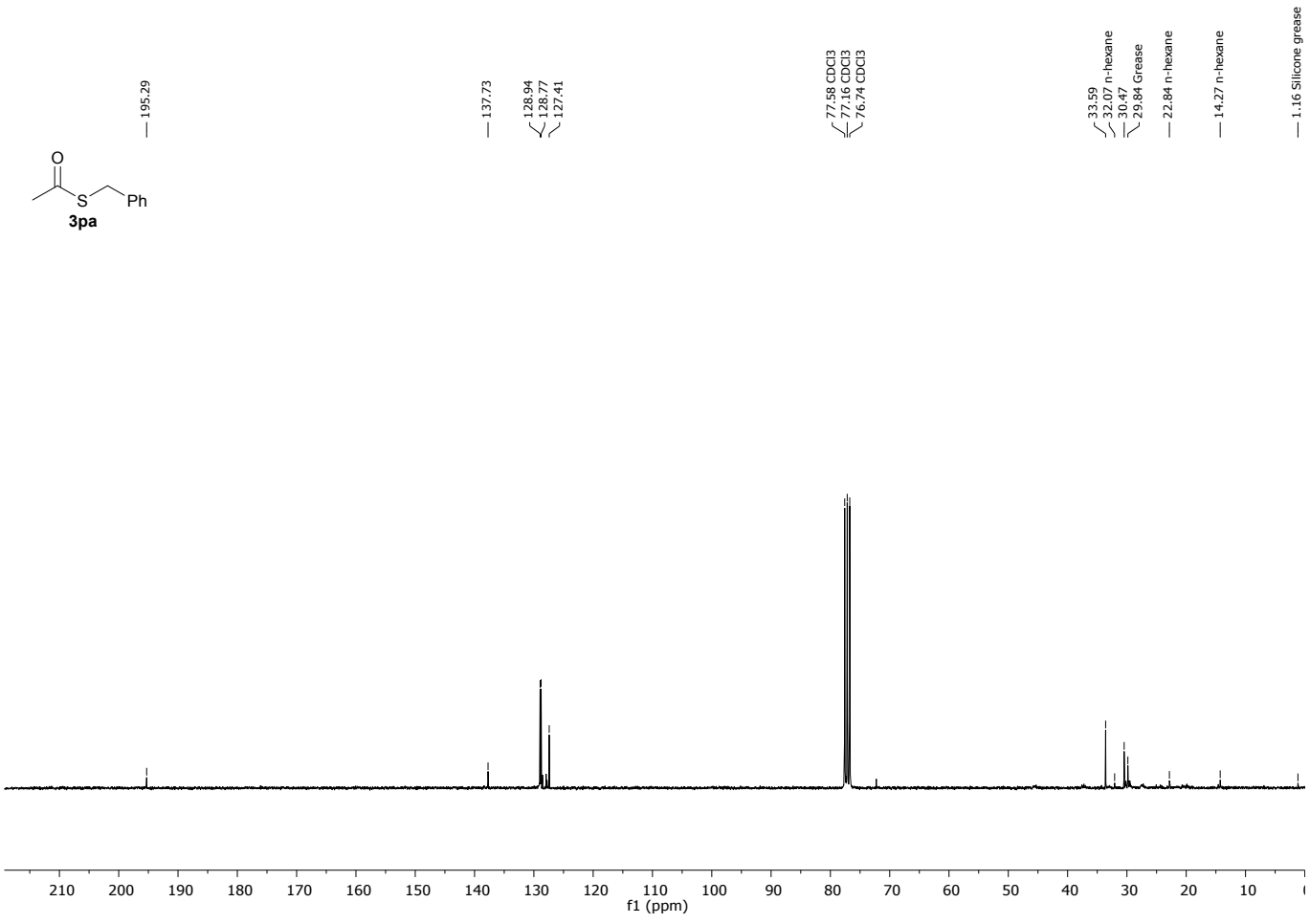
135.38
129.26
128.84
127.90
126.22
124.45
121.65
121.13
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
37.89
29.82 Grease
N
S
S
Ph
3na
f1 (ppm)


7.80 7.79 7.79 7.78 7.77 7.76 7.76 7.39 7.39 7.37 7.37 7.36 7.35 7.34 7.34 7.27 7.26 CDCl3 7.24 7.24 7.22 7.22 7.21 7.20 7.20 7.19 7.18 7.17 7.17 7.16 7.15 7.14 7.14 7.13 7.12 7.11 7.10 7.09 7.08 7.08 7.07 7.06 7.05 7.04 7.03 7.03 7.02 4.13
1.40 HDO
1.06 Grease 0.71 0.69 0.67 Grease 0.66 0.66 0.64
O
S
Ph
3oa
C (m) 7.16
A (dt) 7.77
B (m) 7.36
D (s) 4.13
2.09
1.03
6.92
2.10
f1 (ppm)


191.42
137.60 136.91 133.57 129.11 128.78 128.76 127.46 127.43
77.58 CDCl3 77.16 CDCl3 76.74 CDCl3
33.46
29.83 Grease
O
S
Ph
3oa
f1 (ppm)


3pa
7.26 CDCl3
7.21
7.20
7.19
7.18
7.17
7.16
7.15
7.15
7.14
4.02
2.25
2.24
2.19
1.23
1.20
1.18
1.15 n-hexane and grease
1.12
1.10
0.80
0.78
0.76
0.75 n-hexane and grease
0.75
0.73
0.73
0.70
-0.03 Silicone grease
A (m)
7.18
B (s)
4.02
C (d)
2.24
5.42
1.95
2.77
f1 (ppm)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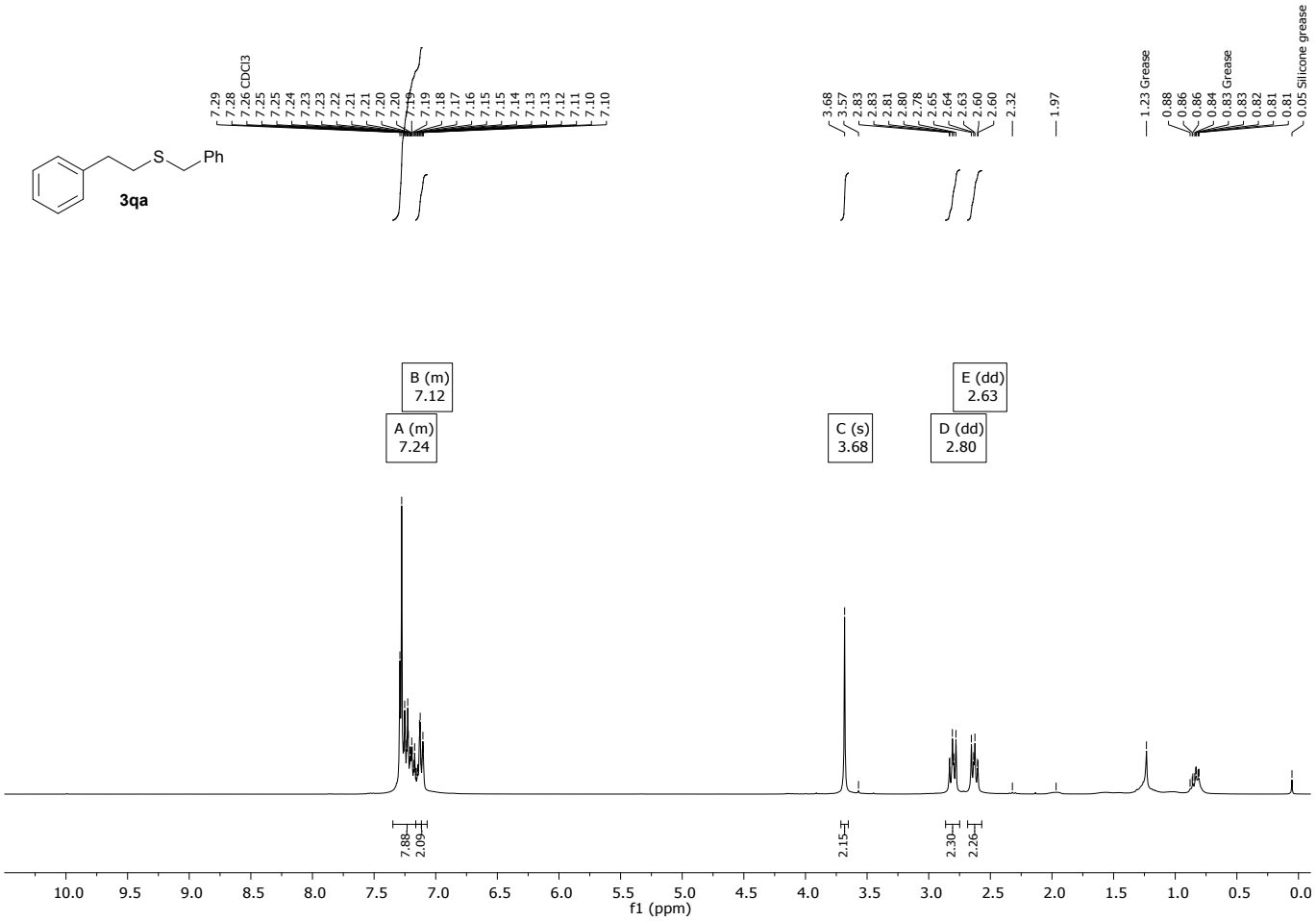
195.29
137.73
128.94
128.77
127.41
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
33.59
32.07 n-hexane
30.47
29.84 Grease
22.84 n-hexane
14.27 n-hexane
1.16 Silicone grease
f1 (ppm)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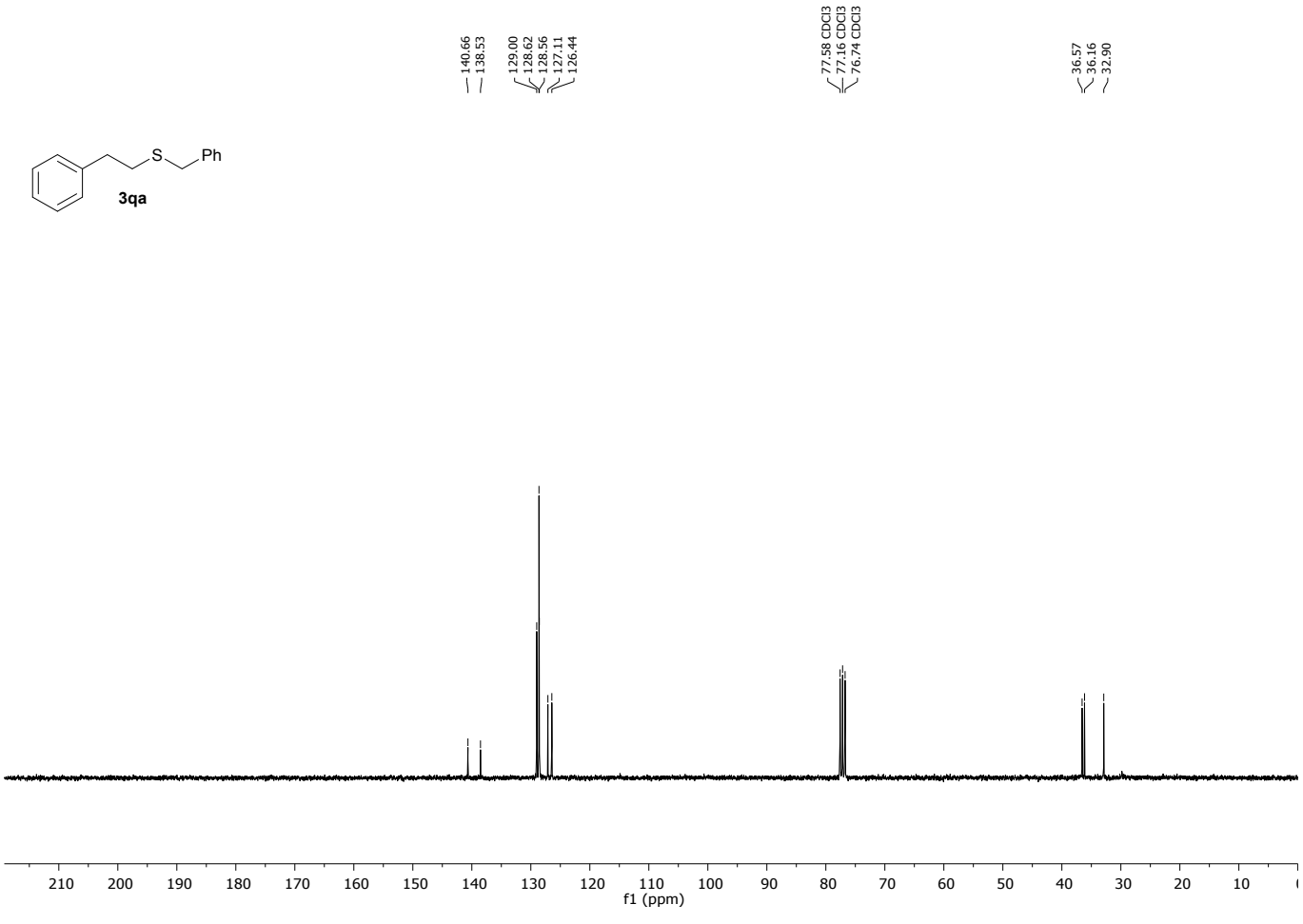
7.28
7.26 CDCl3
7.25
7.25
7.24
7.23
7.22
7.21
7.21
7.20
7.20
7.19
7.19
7.18
7.17
7.16
7.15
7.15
7.14
7.13
7.13
7.12
7.11
7.10
7.10
3.68
3.57
2.83
2.83
2.81
2.80
2.78
2.65
2.64
2.63
2.60
2.60
2.32
1.97
1.23 Grease
0.88
0.86
0.86
0.84
0.83 Grease
0.83
0.82
0.81
0.81
0.05 Silicone grease
S
Ph
3qa
B (m)
7.12
A (m)
7.24
C (s)
3.68
E (dd)
2.63
D (dd)
2.80
7.88
2.09
2.15
2.30
2.26
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)


140.66
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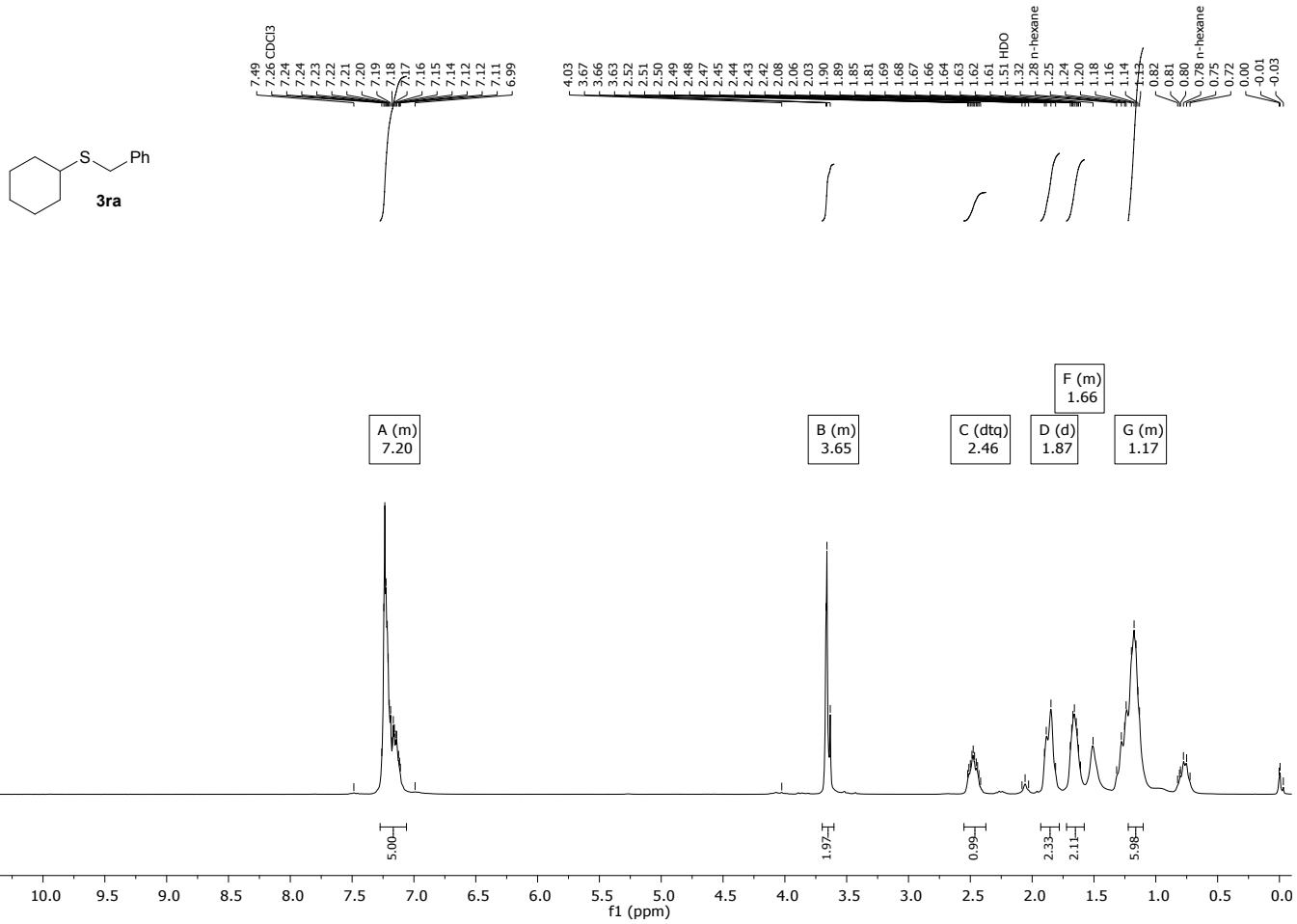
129.00
128.62
128.56
127.11
126.44
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
36.57
36.16
32.90
S
Ph
3qa
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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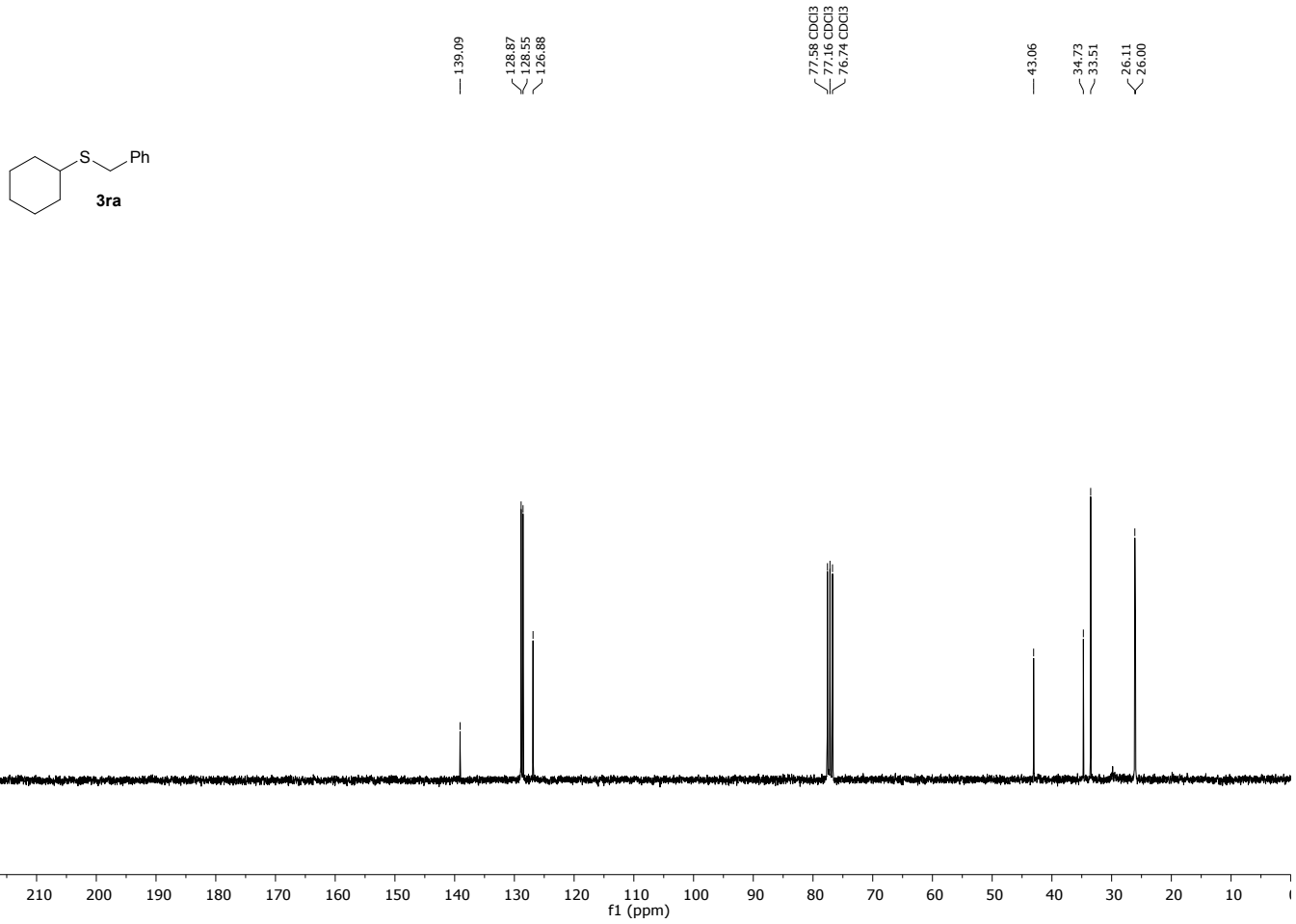
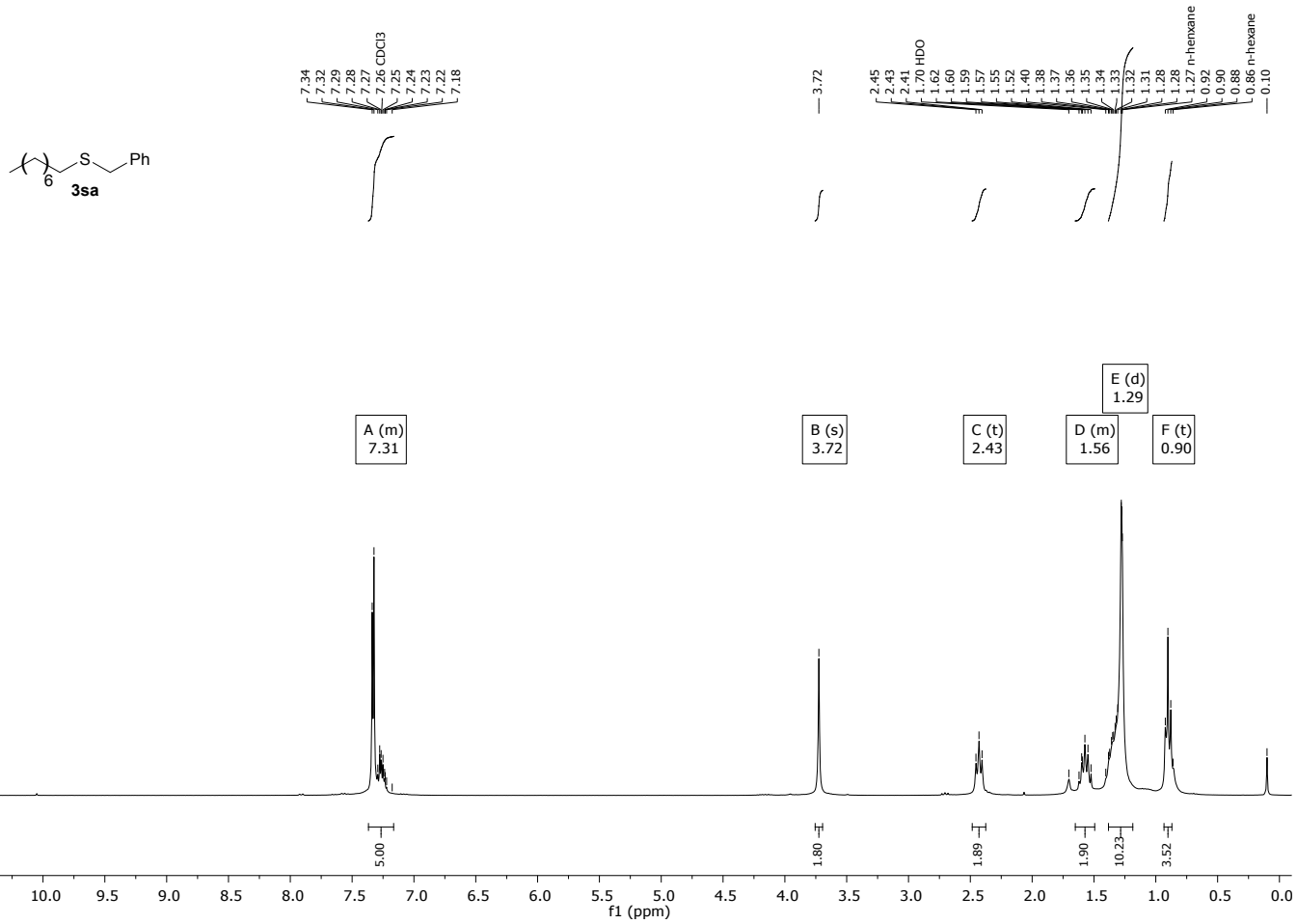
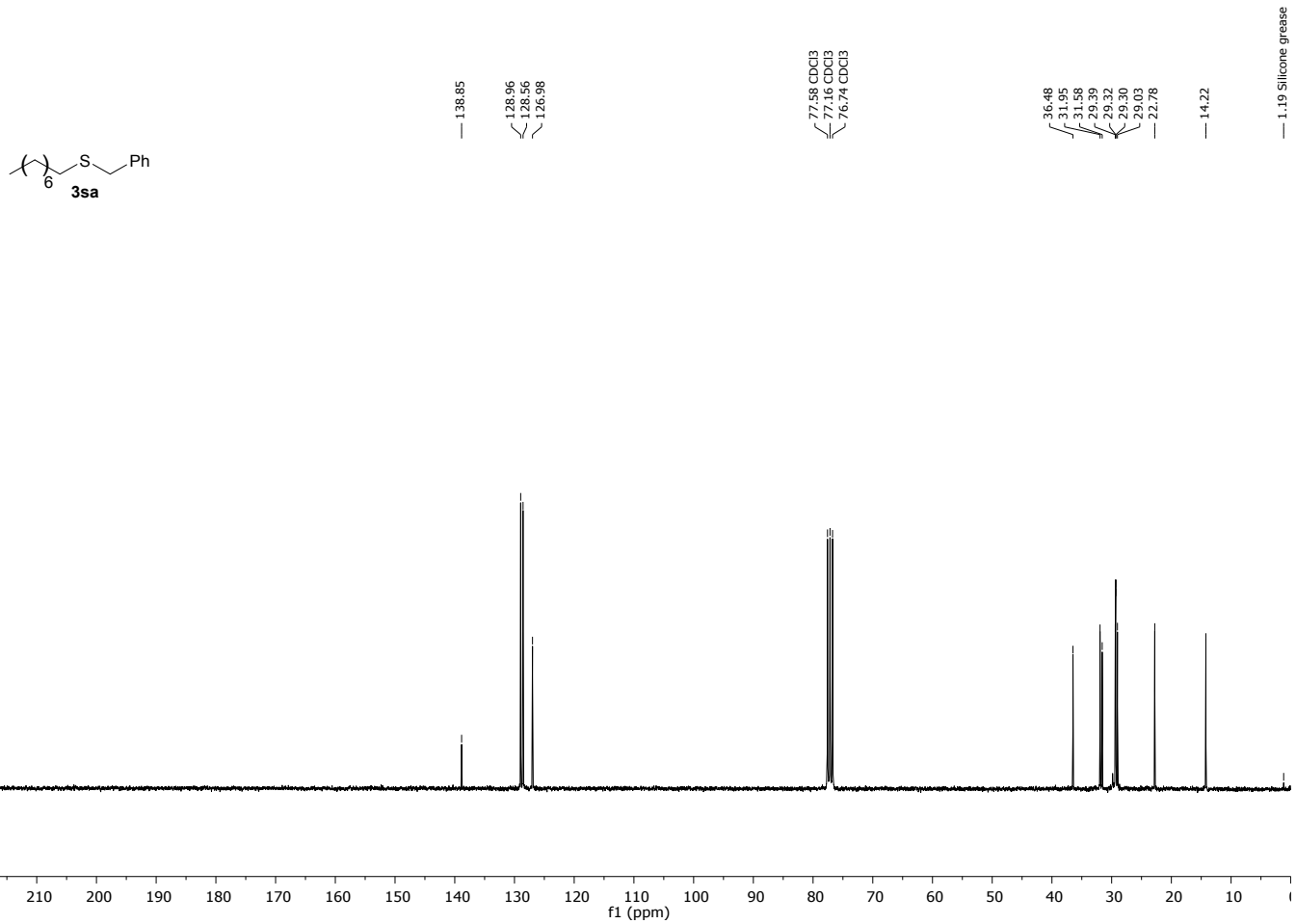
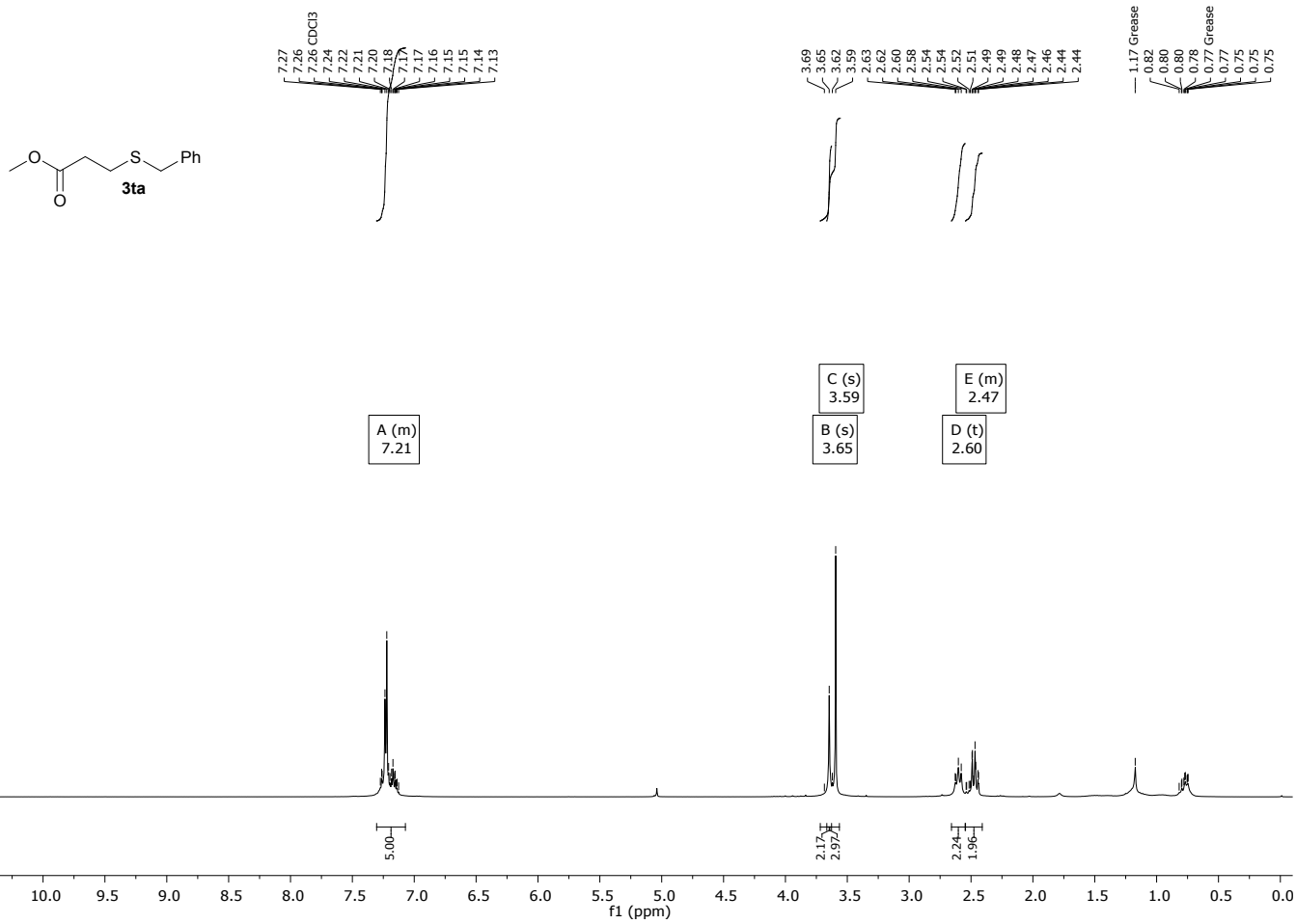
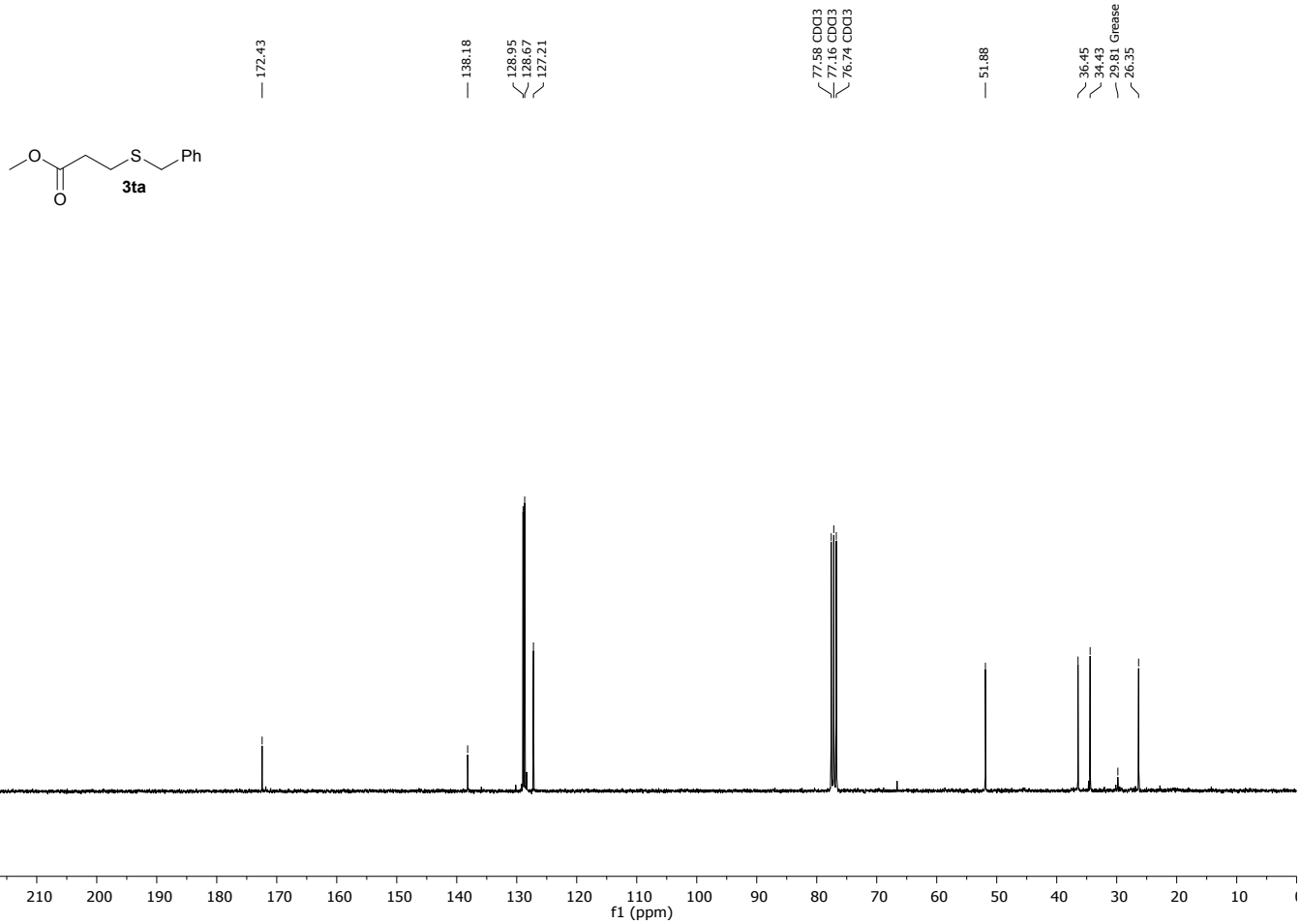
7.26 CDCl3
7.24
7.24
7.23
7.22
7.21
7.20
7.19
7.18
7.17
7.16
7.15
7.14
7.12
7.12
7.11
6.99
4.03
3.67
3.66
3.63
3.52
2.51
2.50
2.49
2.48
2.47
2.45
2.44
2.43
2.42
2.08
2.06
2.03
1.90
1.89
1.85
1.81
1.69
1.68
1.67
1.66
1.64
1.63
1.62
1.61
1.51 HDO
1.32
1.28 n-hexane
1.25
1.24
1.20
1.18
1.16
1.14
1.13
0.82
0.81
0.80
0.78 n-hexane
0.75
0.72
0.00
-0.01
-0.03
S
Ph
3ra
A (m)
7.20
B (m)
3.65
C (dtq)
2.46
D (d)
1.87
F (m)
1.66
G (m)
1.17
5.00
1.97
0.99
2.33
2.11
5.98
f1 (ppm)

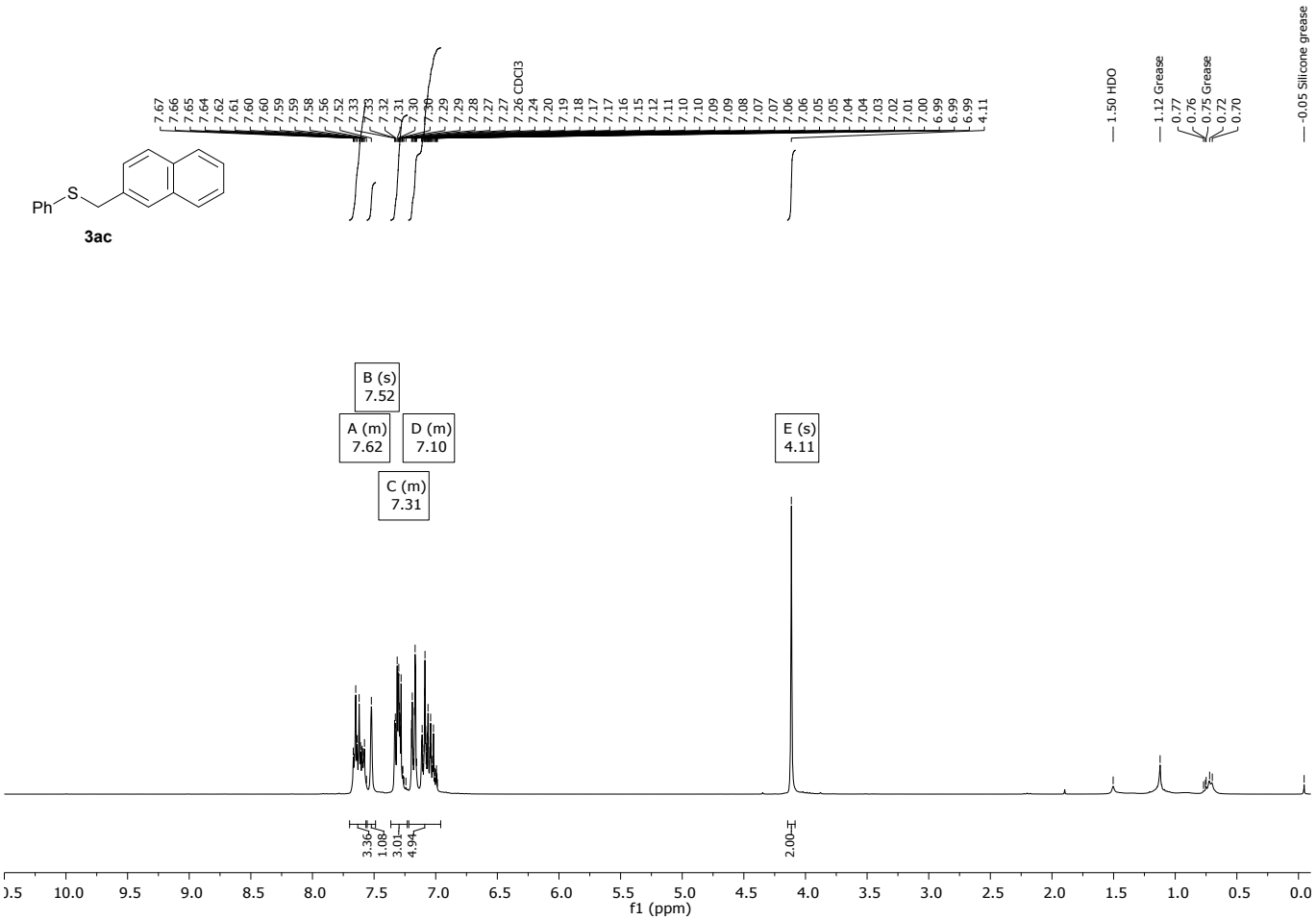

139.09
128.87
128.55
126.88
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
43.06
34.73
33.51
26.11
26.00
S
Ph
3ra
f1 (ppm)

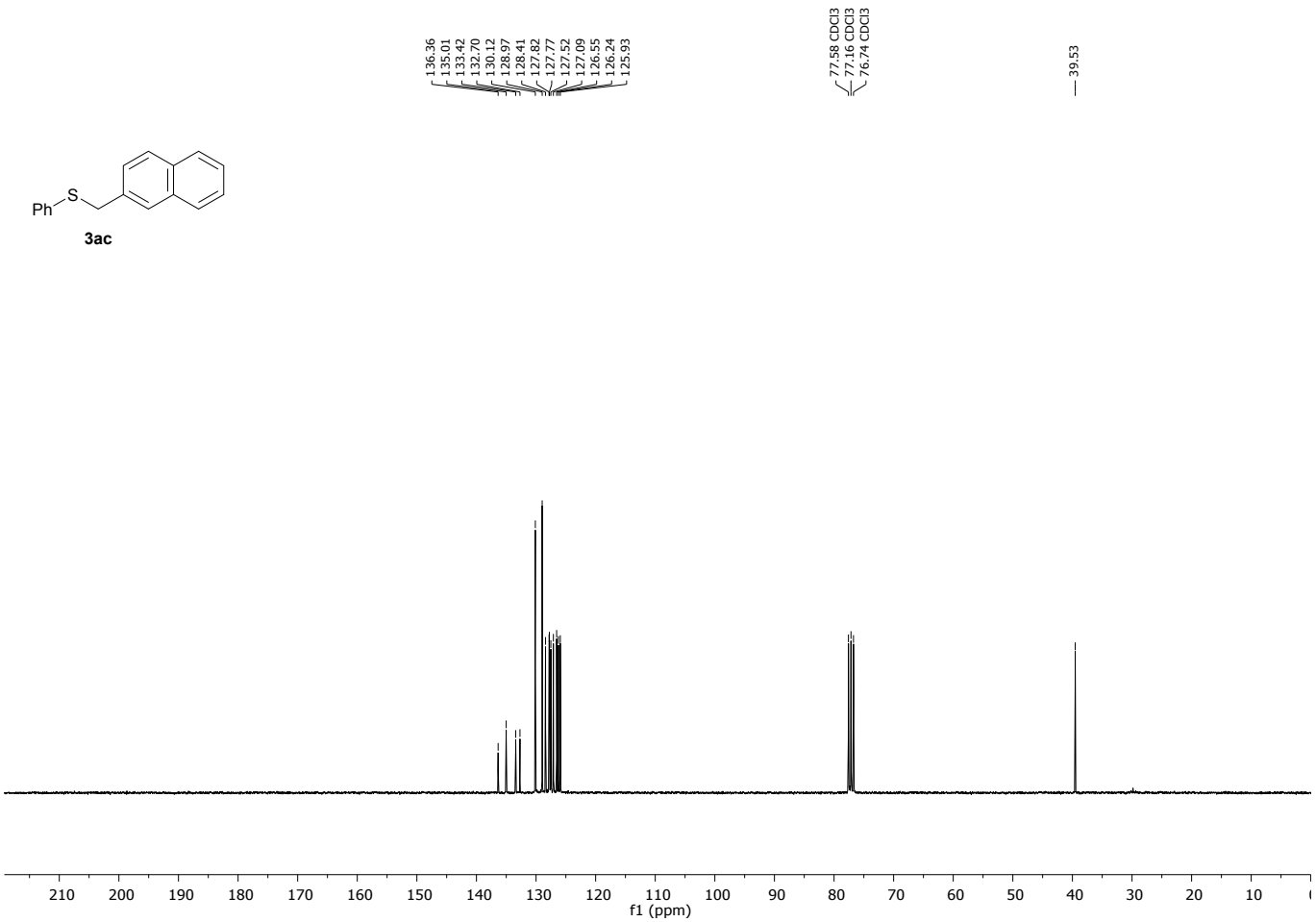

7.34
7.32
7.29
7.28
7.27
7.26 CDCl3
7.25
7.24
7.23
7.22
7.18
3.72
2.45
2.43
2.41
1.70 HDO
1.62
1.60
1.59
1.57
1.55
1.52
1.40
1.38
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.28
1.28
1.27 n-hexane
0.92
0.90
0.88
0.86 n-hexane
0.10
S
Ph
6
3sa
A (m)
7.31
B (s)
3.72
C (t)
2.43
D (m)
1.56
E (d)
1.29
F (t)
0.90
5.00
1.80
1.89
1.90
10.23
3.52
f1 (ppm)

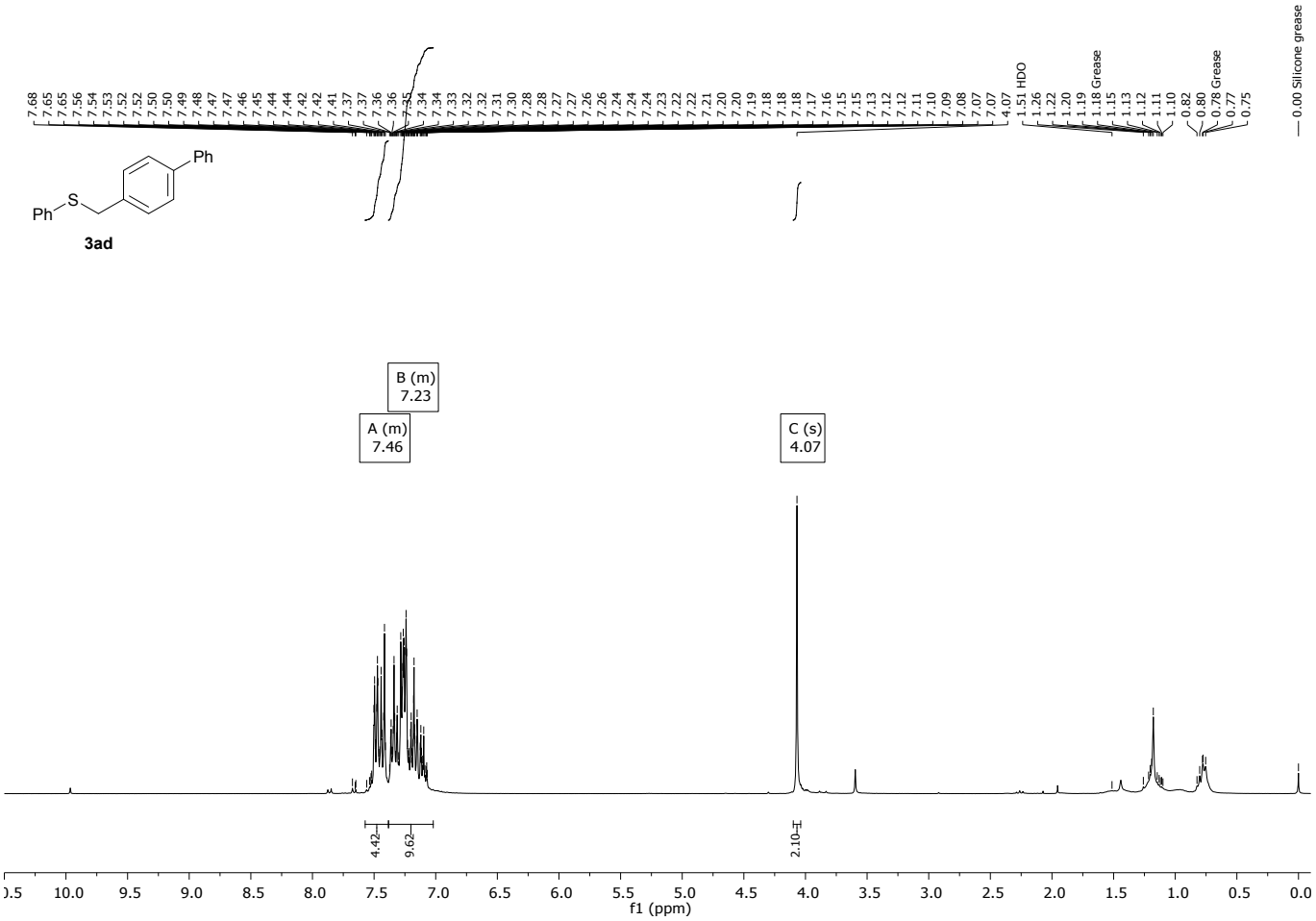

138.85
128.96
128.56
126.98
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
36.48
31.95
31.58
29.39
29.32
29.30
29.03
22.78
14.22
1.19 Silicone grease
S
Ph
6
3sa
f1 (ppm)

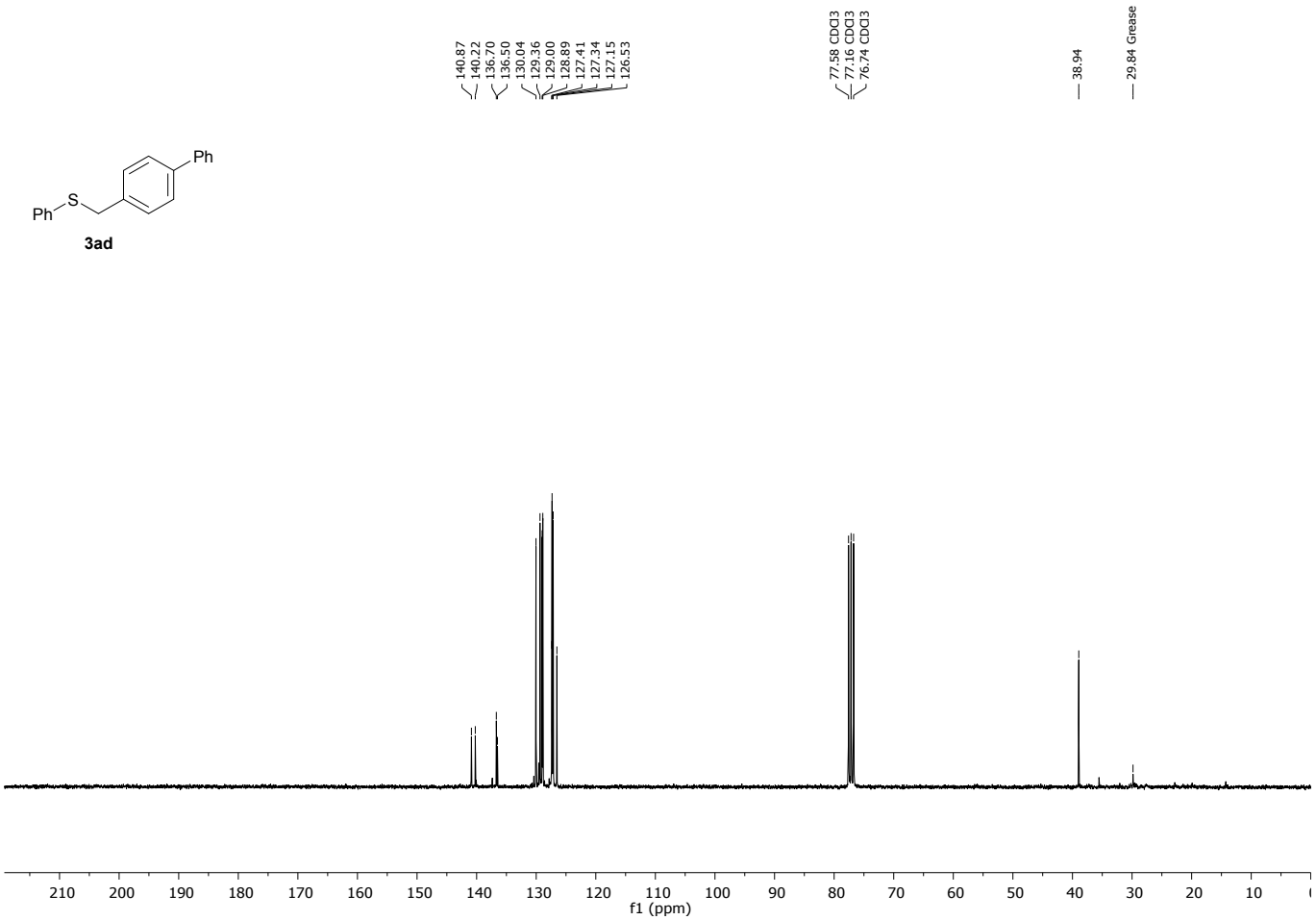

3ta
A (m) 7.21
B (s) 3.65
C (s) 3.59
D (t) 2.60
E (m) 2.47
f1 (ppm)

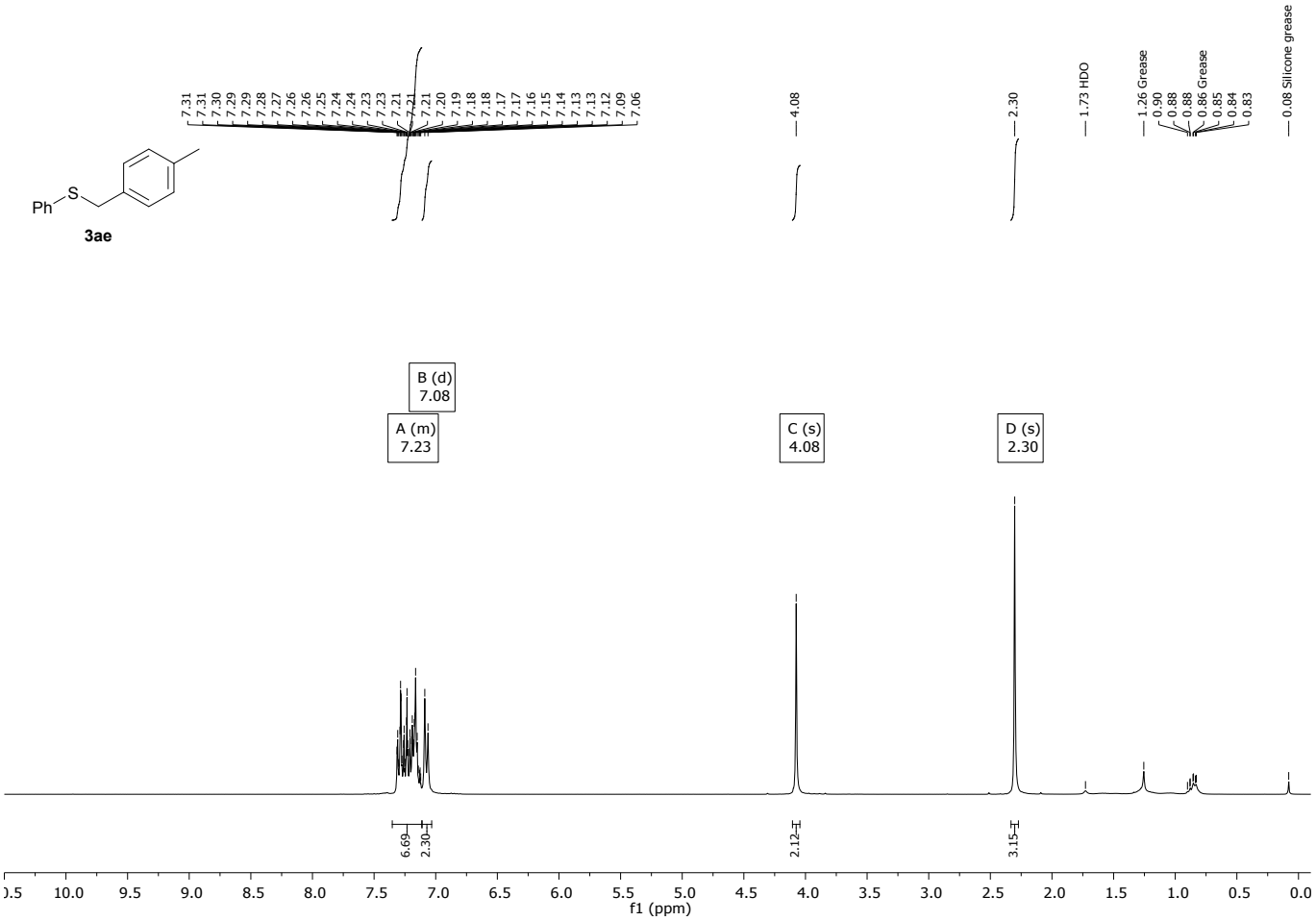

3ta
f1 (ppm)


7.67 7.66 7.65 7.64 7.62 7.61 7.60 7.60 7.59 7.59 7.58 7.56 7.52 7.33 7.33 7.32 7.31 7.30 7.30 7.29 7.29 7.28 7.27 7.27 7.26 CDCl3 7.24 7.20 7.19 7.18 7.17 7.17 7.16 7.15 7.12 7.11 7.10 7.10 7.09 7.09 7.08 7.07 7.07 7.06 7.06 7.05 7.05 7.04 7.04 7.03 7.02 7.01 7.00 6.99 6.99 6.99 4.11
1.50 HDO
1.12 Grease
0.77
0.76
0.75 Grease
0.72
0.70
-0.05 Silicone grease
Ph
S
3ac
B (s) 7.52
A (m) 7.62
D (m) 7.10
C (m) 7.31
E (s) 4.11
3.36
1.08
3.01
4.94
2.00
f1 (ppm)


136.36 135.01 133.42 132.70 130.12 128.97 128.41 127.82 127.77 127.52 127.09 126.55 126.24 125.93
77.58 CDCl3 77.16 CDCl3 76.74 CDCl3
39.53
Ph
S
3ac
f1 (ppm)


7.68 7.65 7.65 7.56 7.54 7.53 7.52 7.52 7.50 7.50 7.49 7.48 7.47 7.47 7.46 7.45 7.44 7.44 7.42 7.42 7.41 7.37 7.37 7.36 7.36 7.35 7.34 7.34 7.33 7.32 7.32 7.31 7.30 7.28 7.28 7.27 7.26 7.26 7.24 7.24 7.24 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.18 7.18 7.18 7.17 7.16 7.15 7.15 7.13 7.12 7.12 7.11 7.10 7.09 7.08 7.07 7.07 4.07 1.51 HDO 1.26 1.22 1.20 1.19 1.18 Grease 1.15 1.13 1.12 1.11 1.10 0.82 0.80 0.78 Grease 0.77 0.75 0.00 Silicone grease
Ph
S
Ph
3ad
B (m) 7.23
A (m) 7.46
C (s) 4.07
4.42
9.62
2.10
f1 (ppm)


140.87 140.22 136.70 136.50 130.04 129.36 129.00 128.89 127.41 127.34 127.15 126.53
77.58 CDCl3 77.16 CDCl3 76.74 CDCl3
38.94
29.84 Grease
Ph
S
Ph
3ad
f1 (ppm)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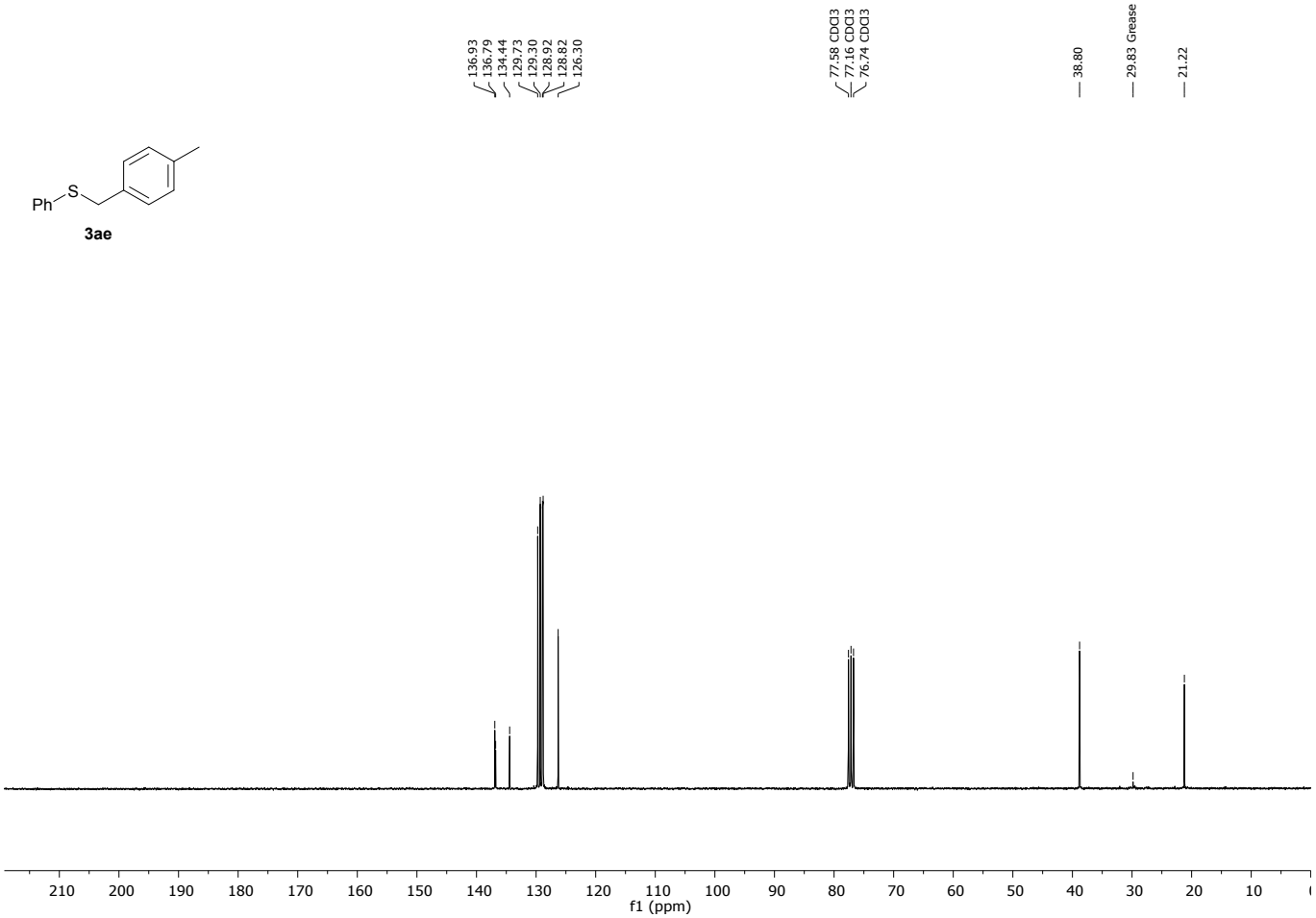
7.24
7.24
7.23
7.23
7.21
7.21
7.21
7.20
7.19
7.18
7.18
7.17
7.17
7.16
7.15
7.14
7.13
7.12
7.09
7.06
4.08
2.30
1.73 HDO
1.26 Grease
0.90
0.88
0.88
0.86 Grease
0.85
0.84
0.83
0.08 Silicone grease
Ph
S
3ae
B (d)
7.08
A (m)
7.23
C (s)
4.08
D (s)
2.30
6.69
2.30
2.12
3.15
f1 (ppm)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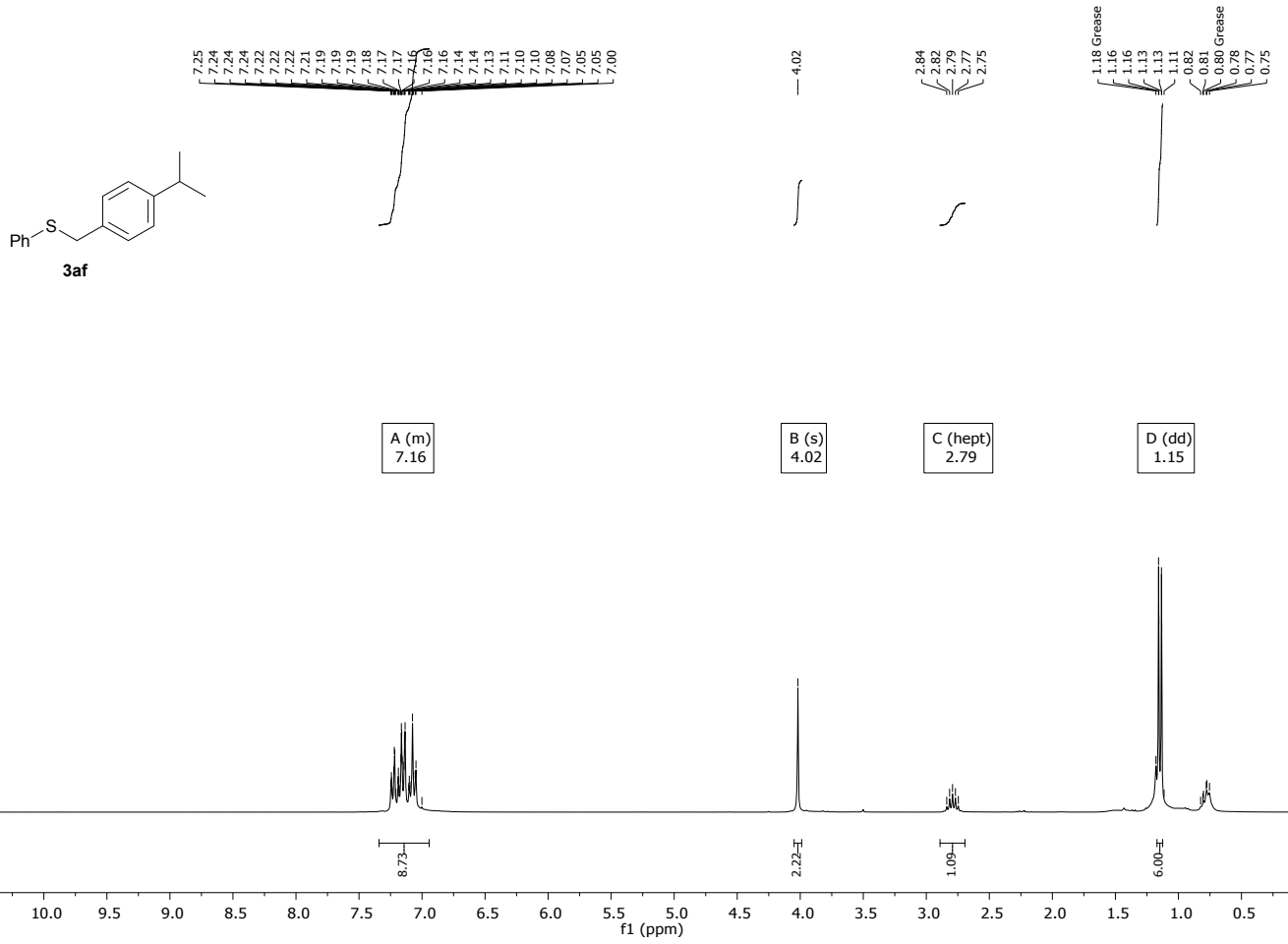
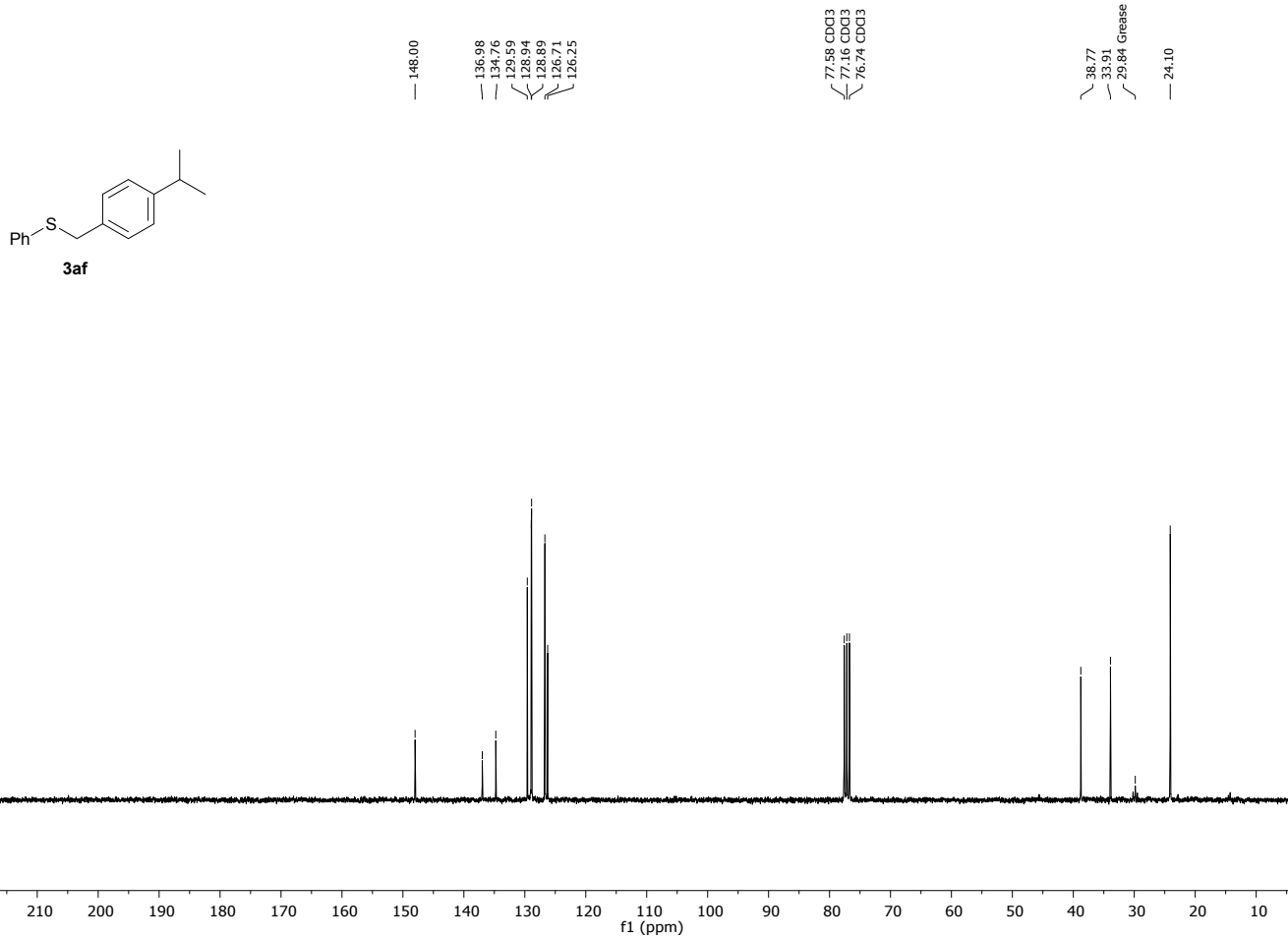
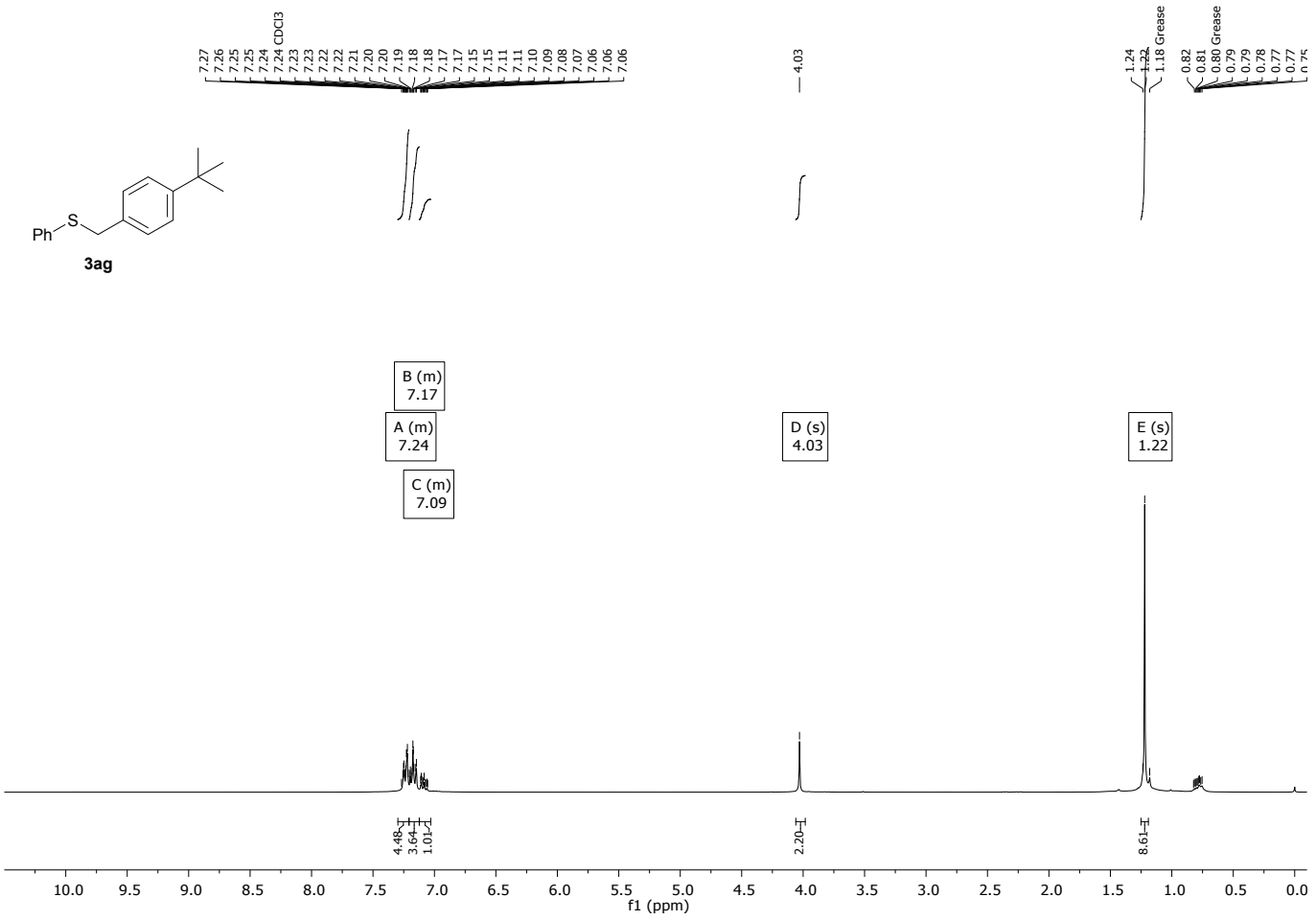
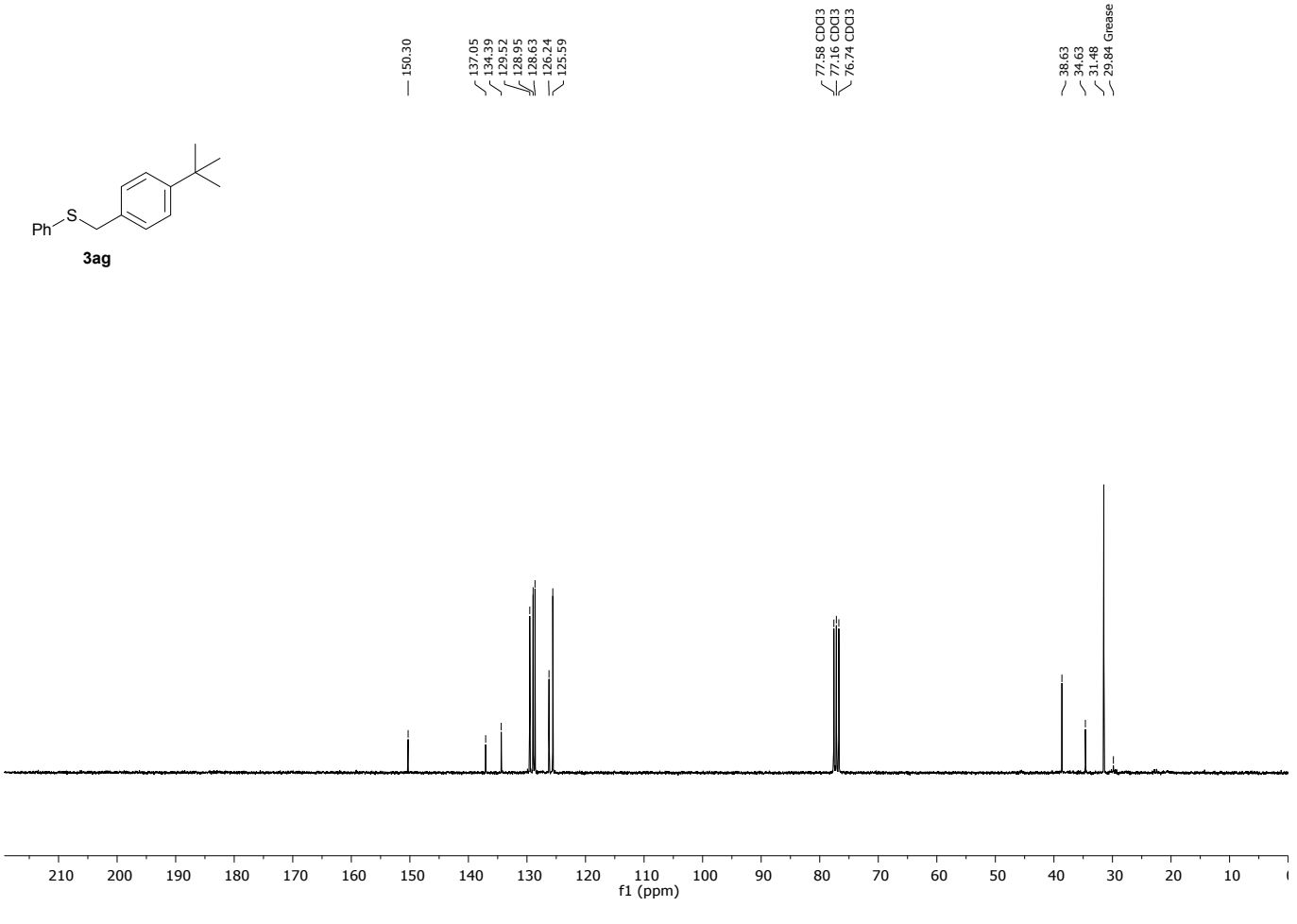
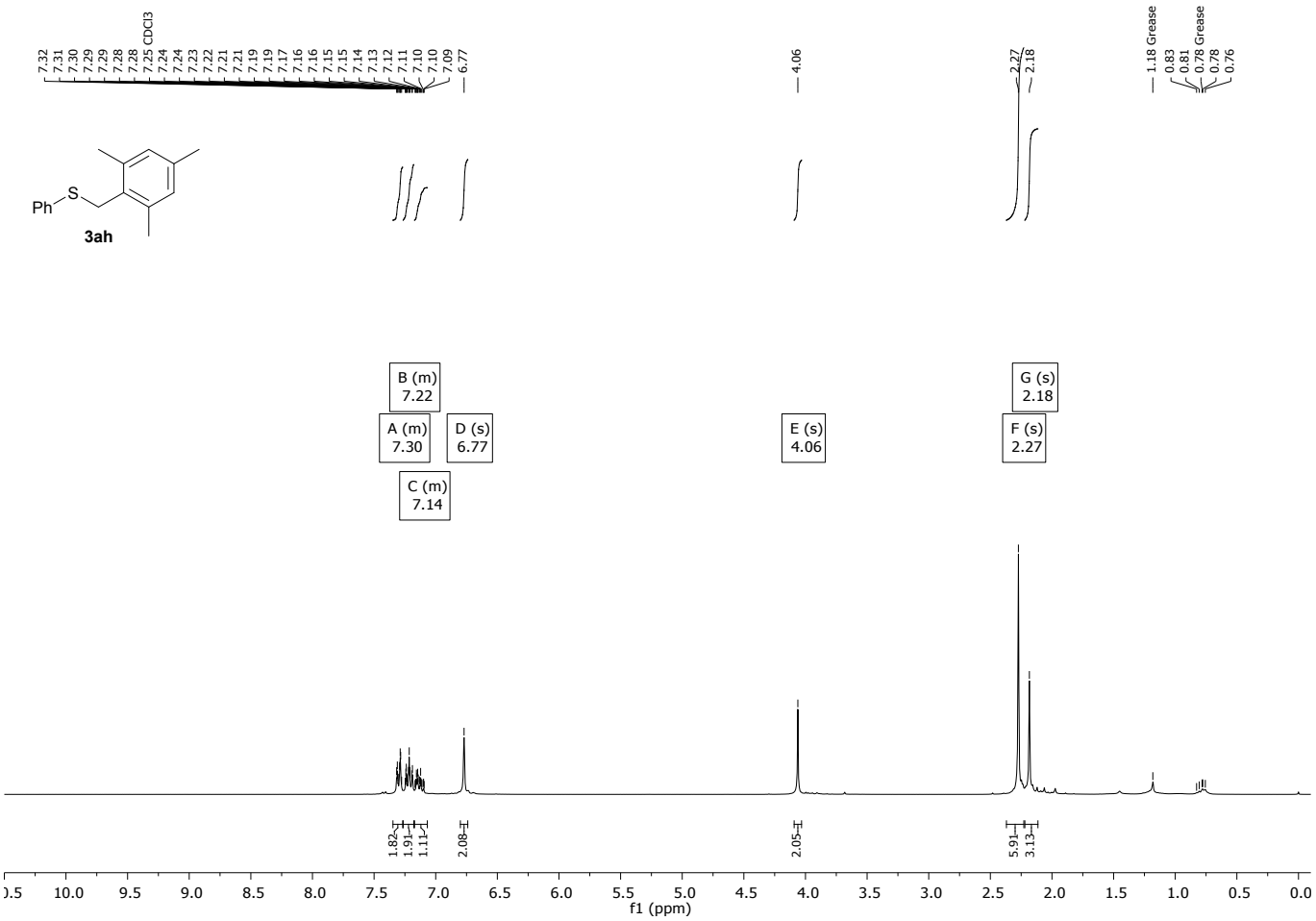
134.44
129.73
129.30
128.92
128.82
126.30
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
38.80
29.83 Grease
21.22
Ph
S
3ae
f1 (ppm)


7.25 7.24 7.24 7.24 7.22 7.22 7.22 7.21 7.19 7.19 7.19 7.18 7.17 7.17 7.16 7.16 7.16 7.14 7.14 7.13 7.11 7.10 7.10 7.08 7.07 7.05 7.05 7.00
4.02
2.84 2.82 2.79 2.77 2.75
1.18 Grease 1.16 1.16 1.13 1.13 1.11 0.82 0.81 0.80 Grease 0.78 0.77 0.75
Ph S
3af
A (m) 7.16
B (s) 4.02
C (hept) 2.79
D (dd) 1.15
8.73
2.22
1.09
6.00
f1 (ppm)


148.00
136.98 134.76 129.59 128.94 128.89 126.71 126.25
77.58 CDCl3 77.16 CDCl3 76.74 CDCl3
38.77 33.91 29.84 Grease 24.10
Ph S
3af
f1 (ppm)


7.27 7.26 7.25 7.24 7.24 CDCl3 7.23 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.18 7.18 7.17 7.17 7.15 7.15 7.11 7.11 7.10 7.09 7.08 7.07 7.06 7.06 7.06
4.03
1.24 1.22 1.18 Grease
0.82 0.81 0.80 Grease 0.79 0.79 0.78 0.77 0.77
Ph S
3ag
B (m) 7.17
A (m) 7.24
C (m) 7.09
D (s) 4.03
E (s) 1.22
4.48 3.64 1.01 2.20 8.61
f1 (ppm)


150.30
137.05 134.39 129.52 128.95 128.63 126.24 125.59
77.58 CDCl3 77.16 CDCl3 76.74 CDCl3
38.63 34.63 31.48 29.84 Grease
Ph S
3ag
f1 (ppm)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7.28
7.28
7.25 CDCl3
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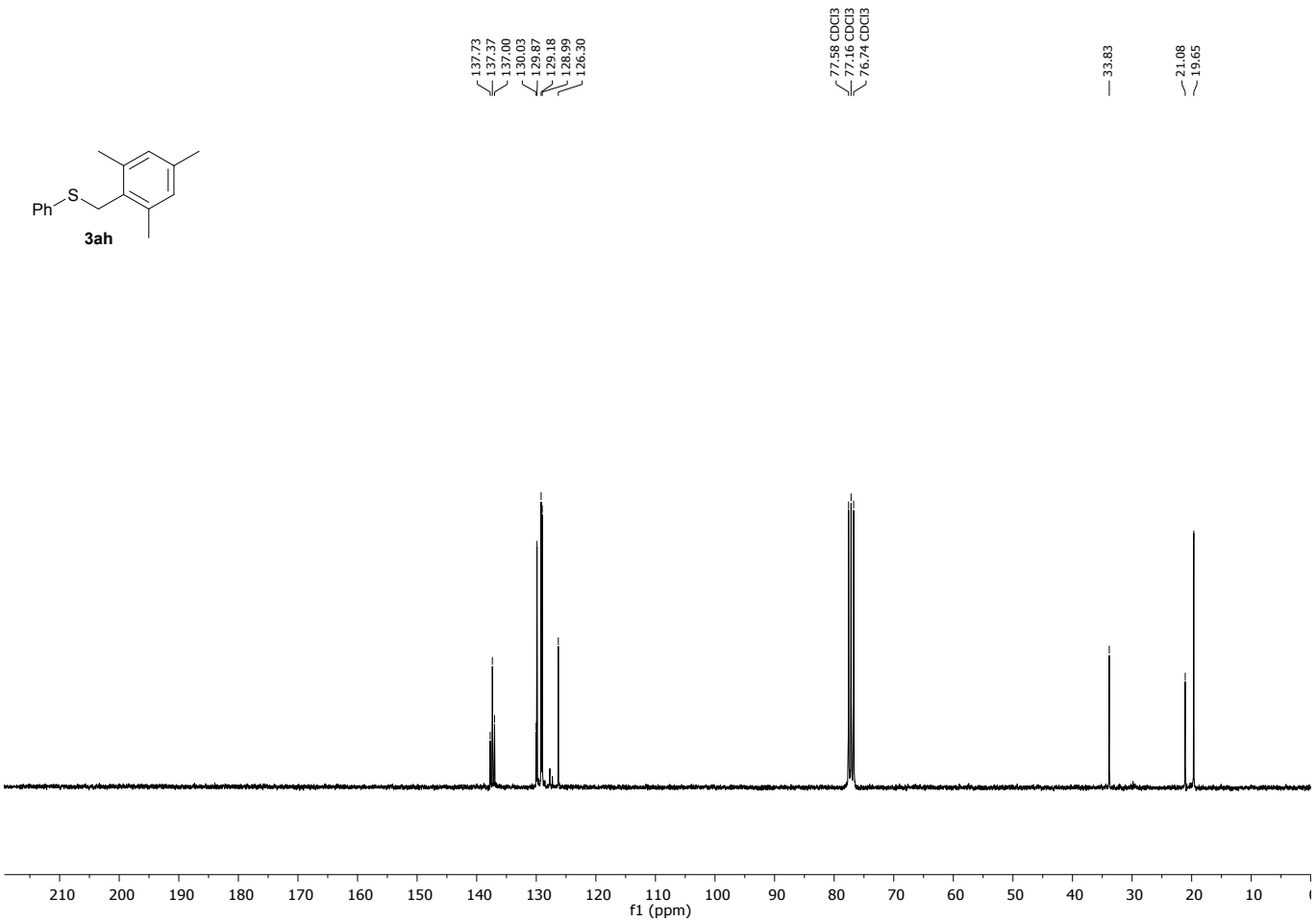
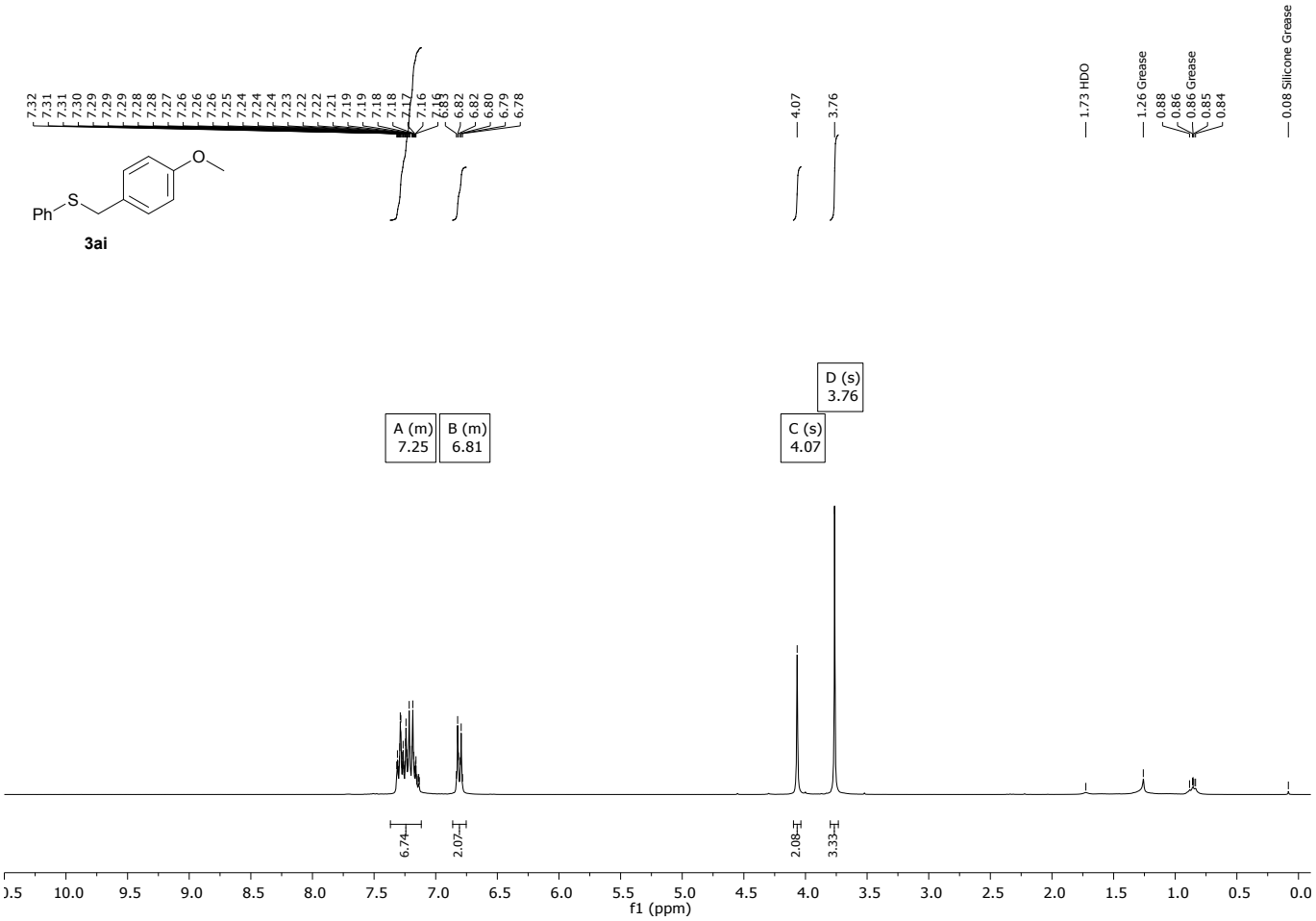
7.21
7.21
7.19
7.19
7.17
7.16
7.16
7.15
7.15
7.14
7.13
7.12
7.11
7.10
7.10
7.09
6.77
4.06
2.27
2.18
1.18 Grease
0.83
0.81
0.78 Grease
0.78
0.76
Ph
S
3ah
B (m)
7.22
A (m)
7.30
D (s)
6.77
C (m)
7.14
E (s)
4.06
G (s)
2.18
F (s)
2.27
1.82
1.91
1.11
2.08
2.05
5.91
3.13
f1 (ppm)


137.73
137.37
137.00
130.03
129.87
129.18
128.99
126.30
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
33.83
21.08
19.65
Ph
S
3ah
f1 (ppm)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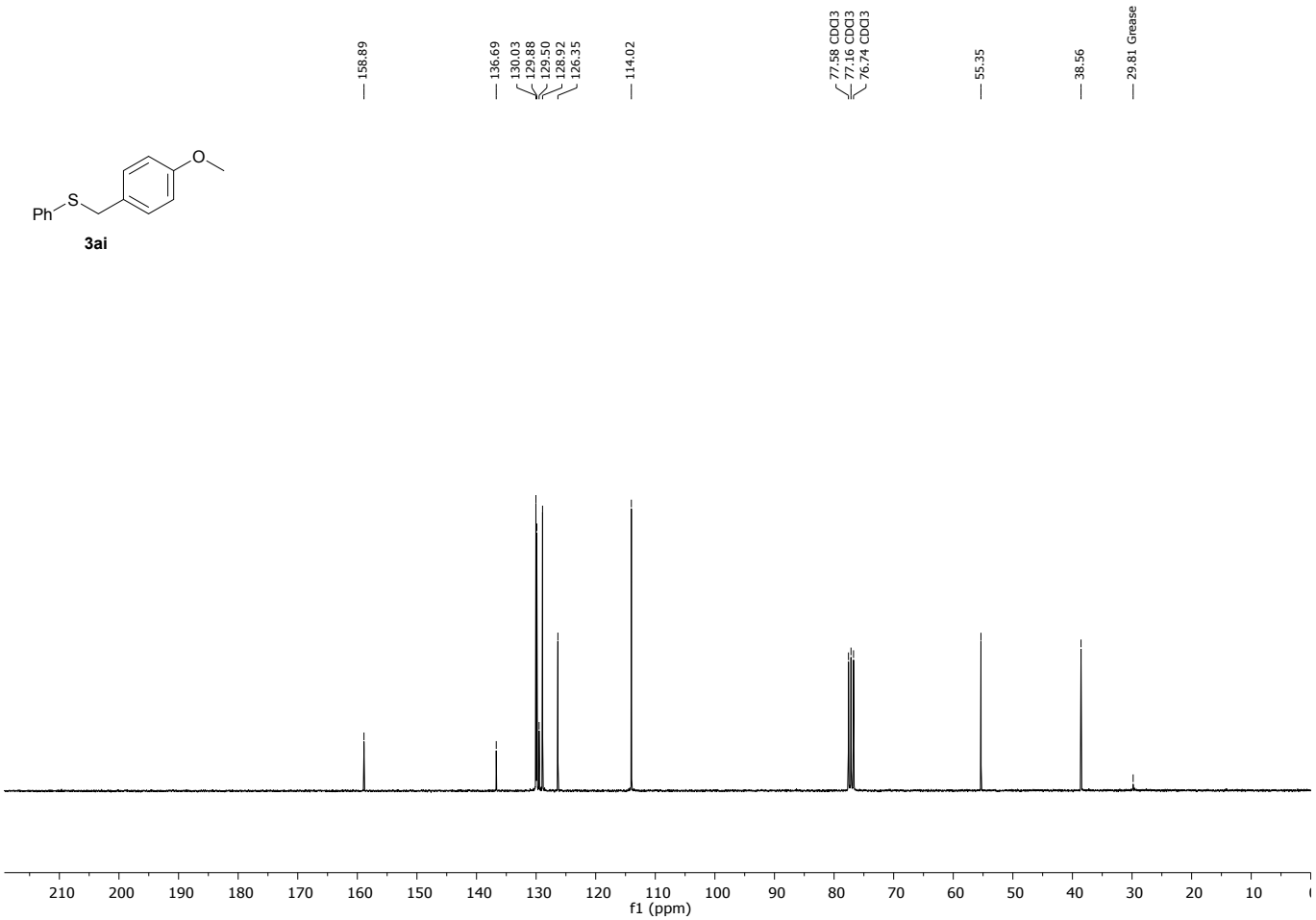
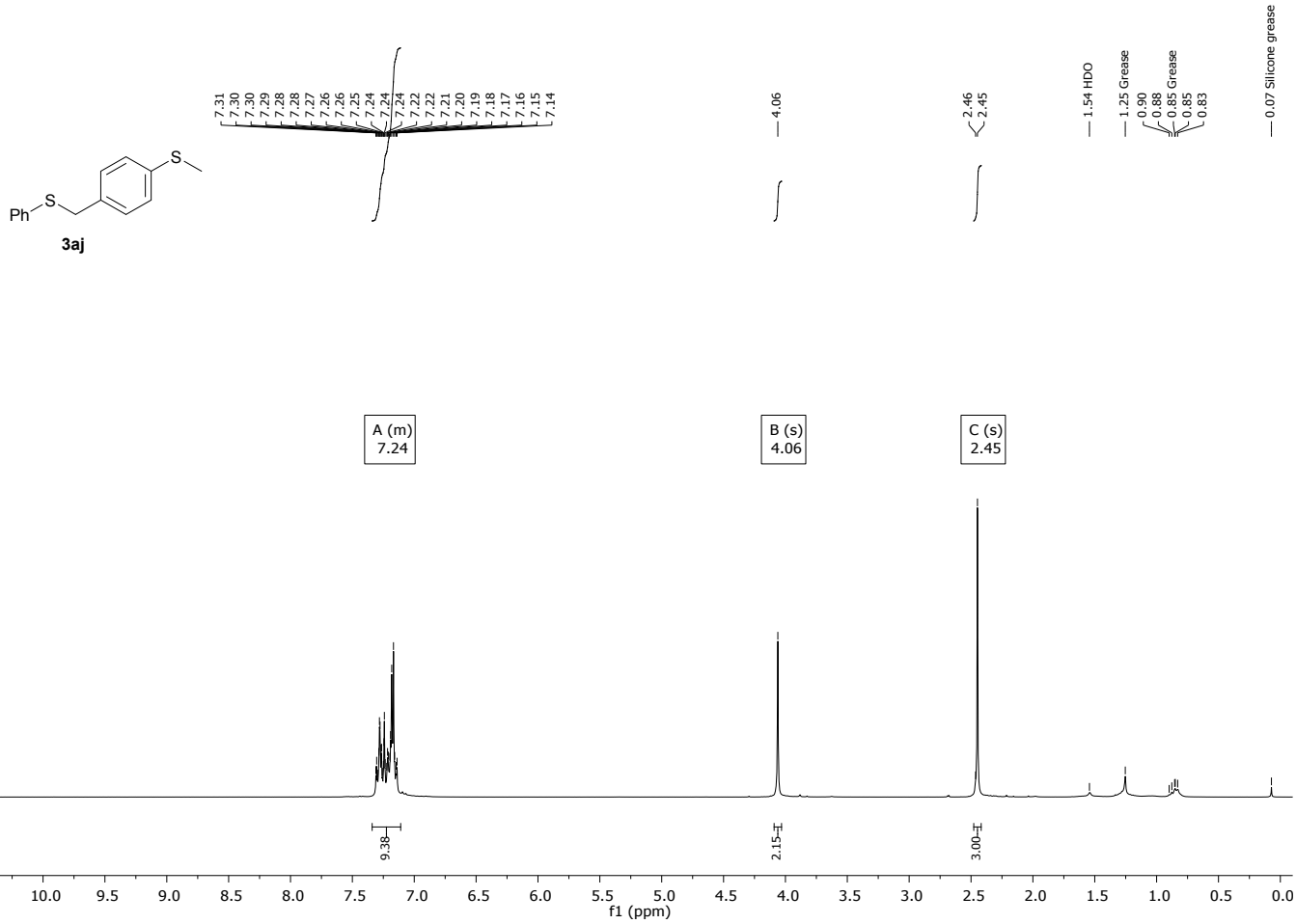
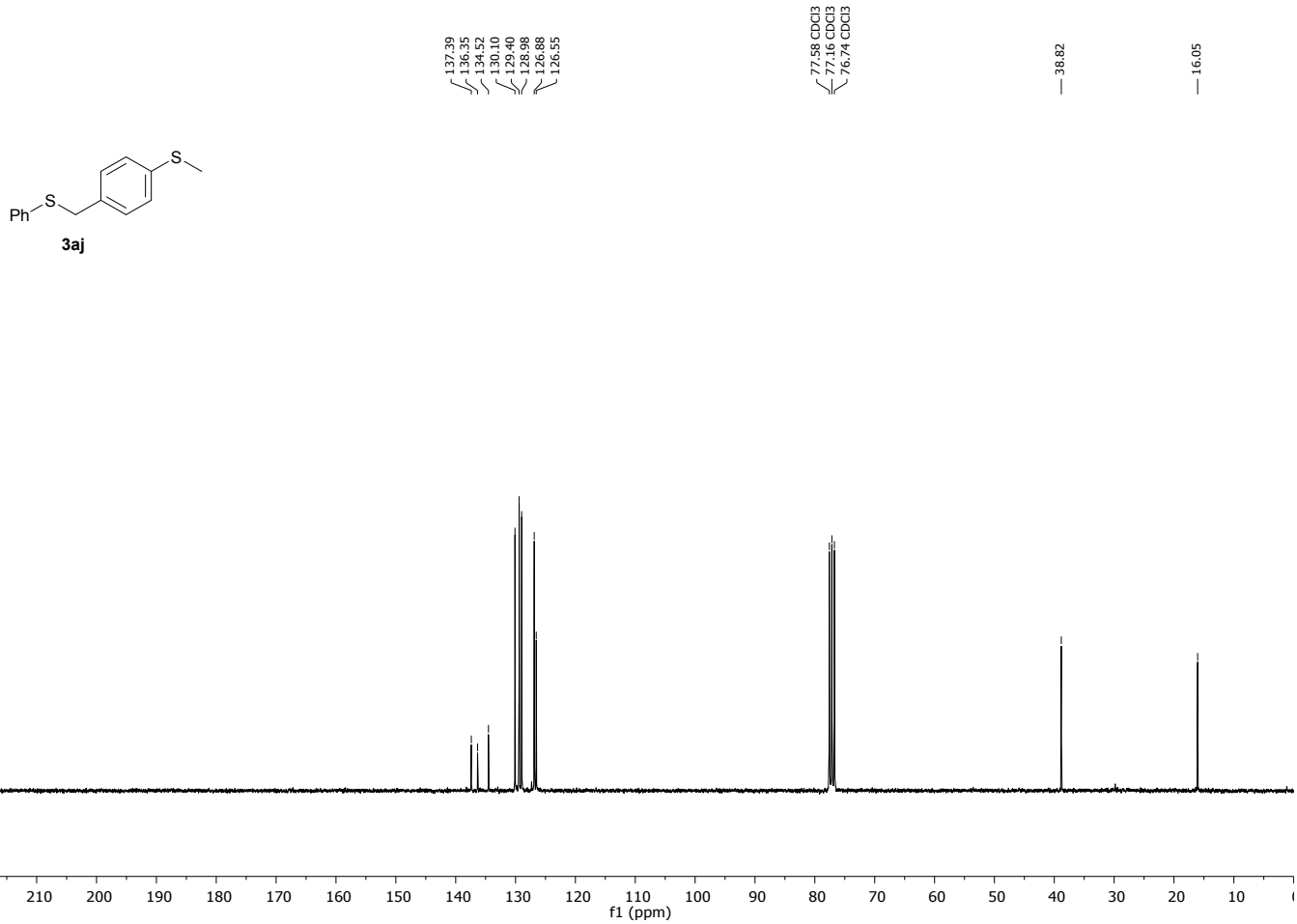
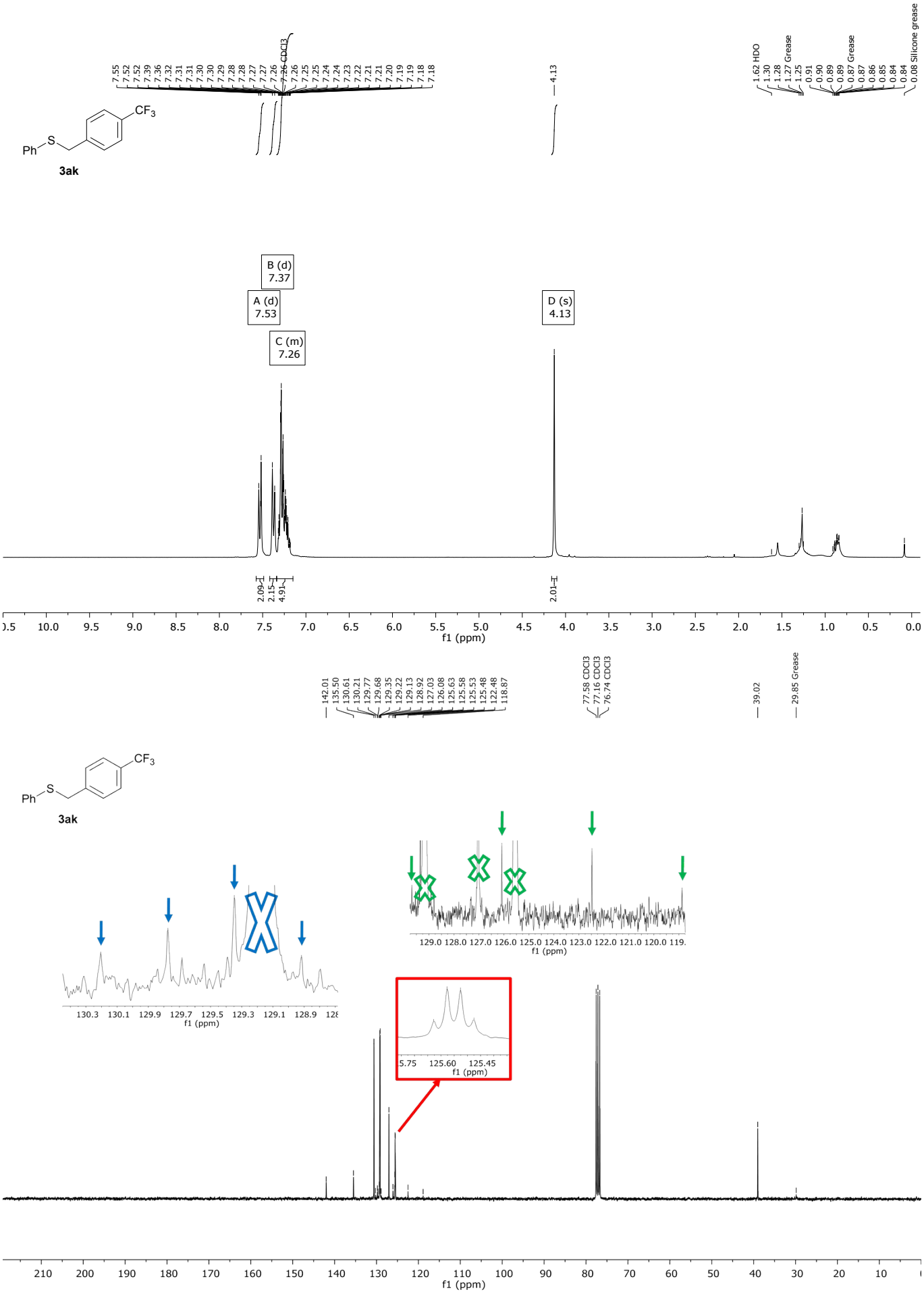
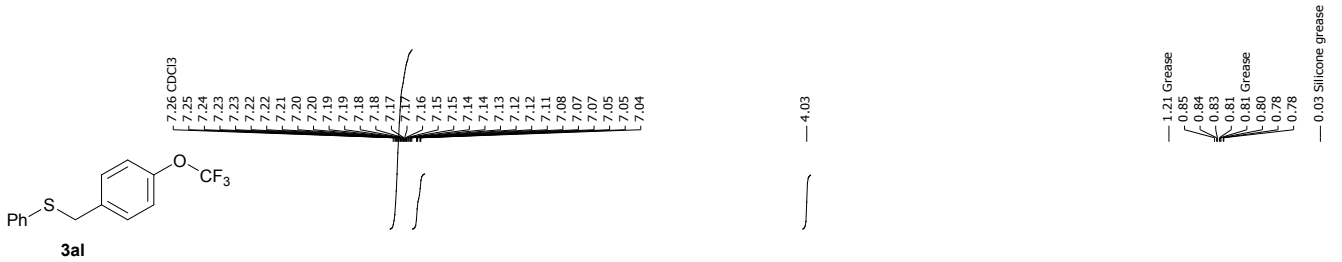
7.19
7.18
7.18
7.17
7.16
6.83
6.82
6.82
6.80
6.79
6.78
4.07
3.76
1.73 HDO
1.26 Grease
0.88
0.86
0.86 Grease
0.85
0.84
0.08 Silicone Grease
Ph
S
O
3ai
A (m)
7.25
B (m)
6.81
C (s)
4.07
D (s)
3.76
6.74
2.07
2.08
3.33
f1 (ppm)


158.89
136.69
130.03
129.88
129.50
128.92
126.35
114.02
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
55.35
38.56
29.81 Grease
Ph
S
O
3ai
f1 (ppm)

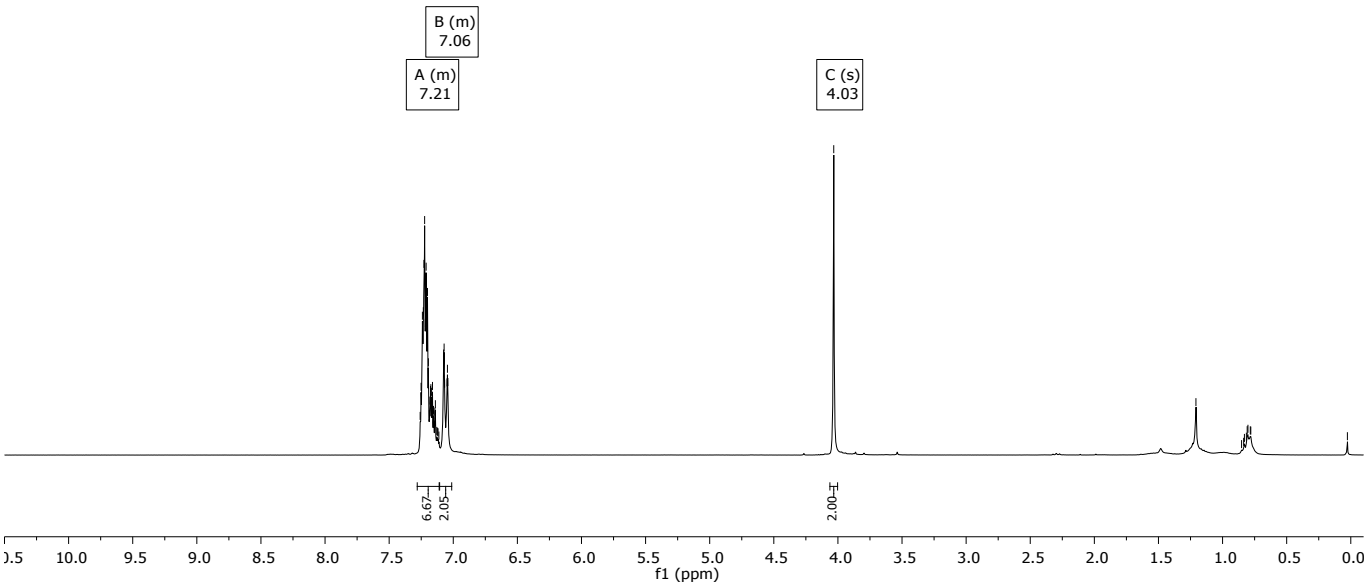

7.31
7.30
7.30
7.29
7.28
7.28
7.27
7.26
7.26
7.25
7.24
7.24
7.24
7.22
7.21
7.20
7.19
7.18
7.17
7.16
7.15
7.14
4.06
2.46
2.45
1.54 HDO
1.25 Grease
0.90
0.88
0.85 Grease
0.85
0.83
0.07 Silicone grease
S
S
Ph
3aj
A (m)
7.24
B (s)
4.06
C (s)
2.45
9.38
2.15
3.00
f1 (ppm)

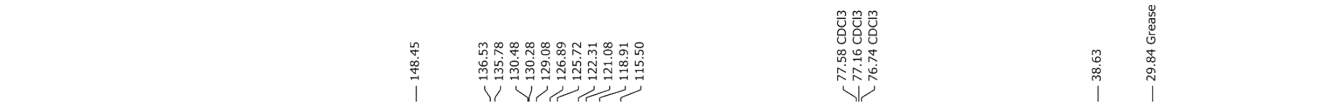

137.39
136.35
134.52
130.10
129.40
128.98
126.88
126.55
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
38.82
16.05
S
S
Ph
3aj
f1 (ppm)

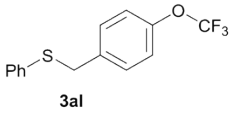

3ak
B (d) 7.37
A (d) 7.53
C (m) 7.26
D (s) 4.13
f1 (ppm)
3ak
f1 (ppm)


7.26 CDCl3
7.25
7.24
7.23
7.23
7.22
7.22
7.21
7.20
7.20
7.19
7.19
7.18
7.18
7.17
7.17
7.16
7.15
7.15
7.14
7.14
7.13
7.12
7.12
7.11
7.08
7.07
7.07
7.05
7.05
7.04
4.03
1.21 Grease
0.85
0.84
0.83
0.81
0.81 Grease
0.80
0.78
0.78
0.03 Silicone grease
O
CF3
Ph
S

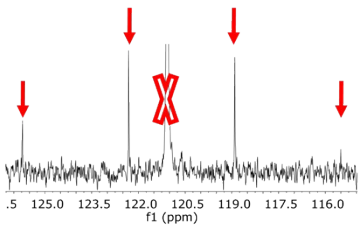
3al

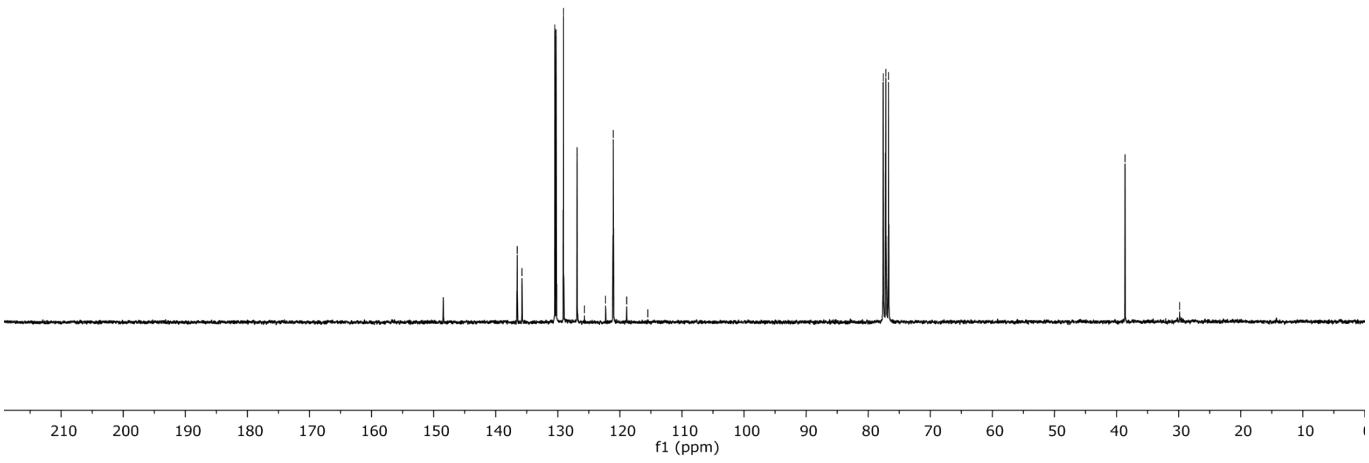

B (m)
7.06
A (m)
7.21
C (s)
4.03
6.67
2.05
2.00
f1 (ppm)

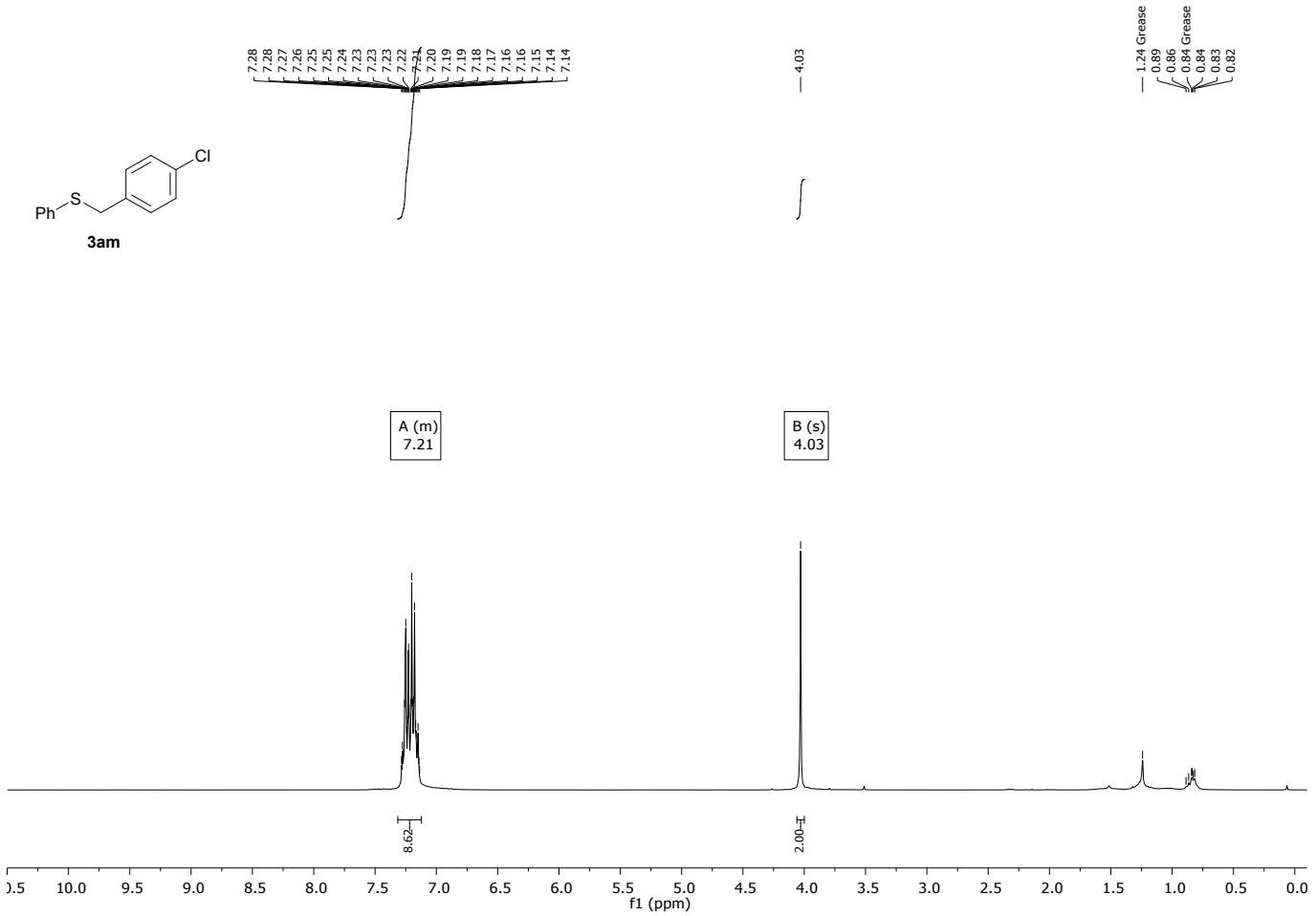

148.45
136.53
135.78
130.48
130.28
129.08
126.89
125.72
122.31
121.08
118.91
115.50
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
38.63
29.84 Grease


O
CF3
Ph
S

3al


125.0
123.5
122.0
120.5
119.0
117.5
116.0
f1 (ppm)


f1 (ppm)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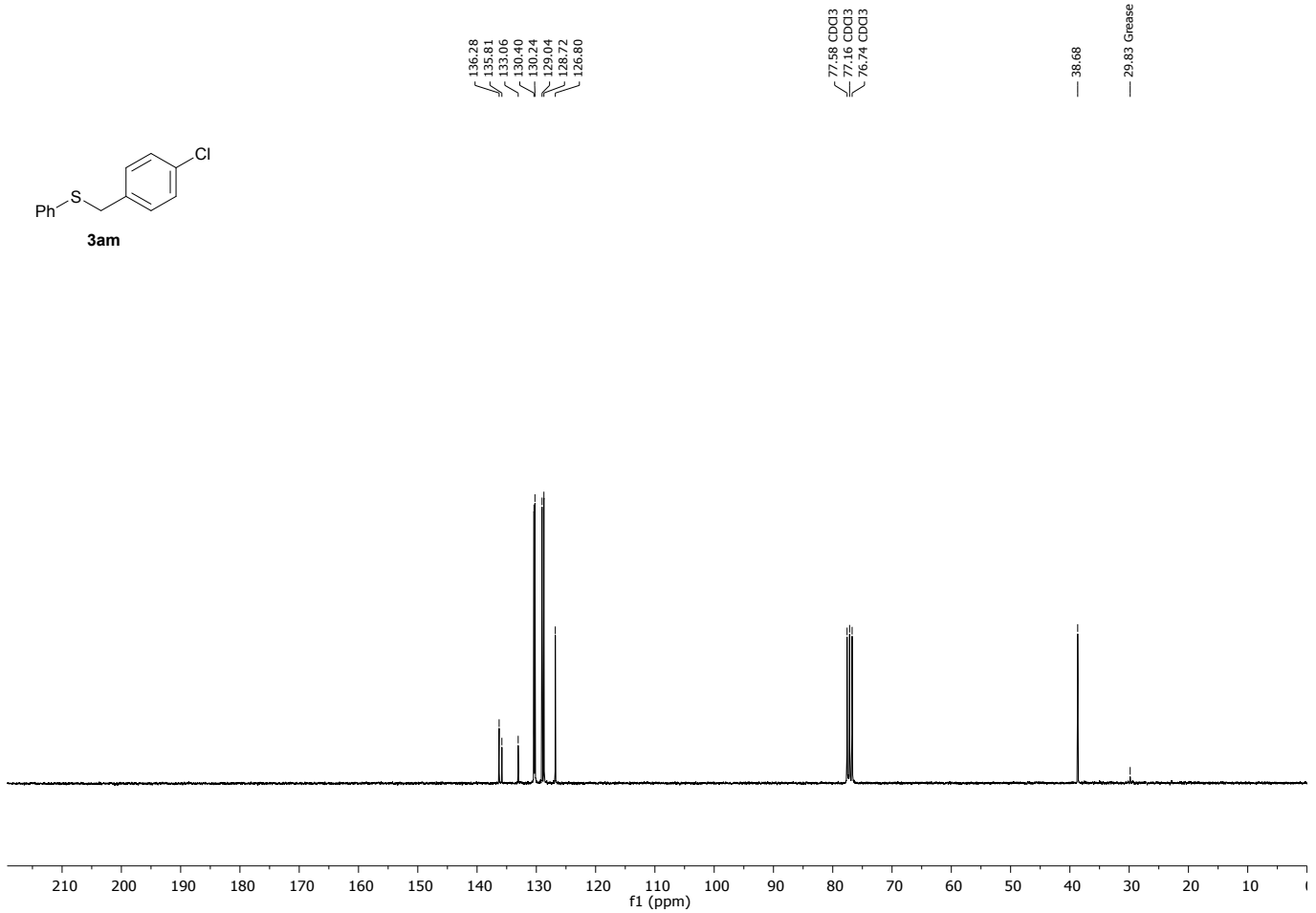
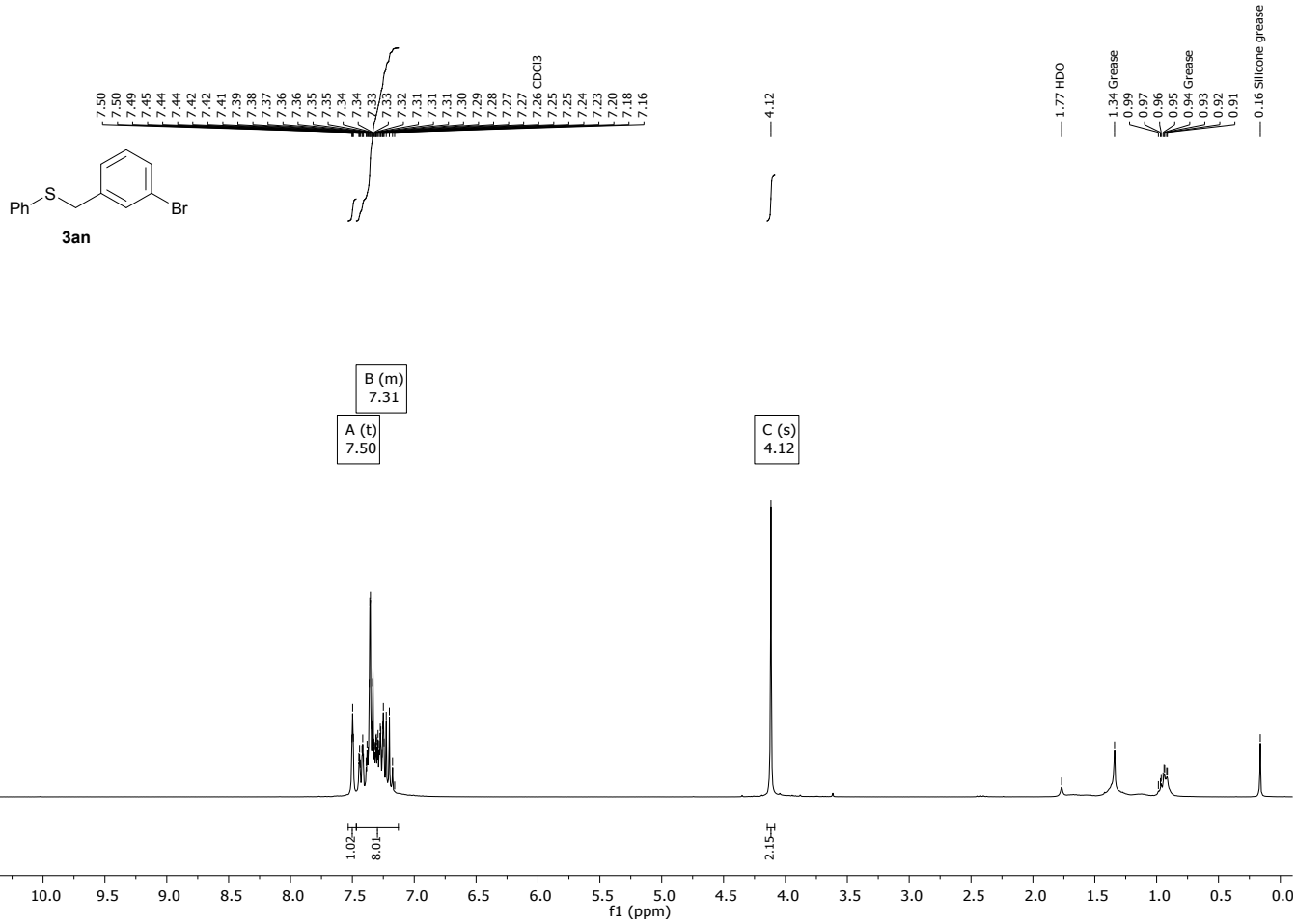
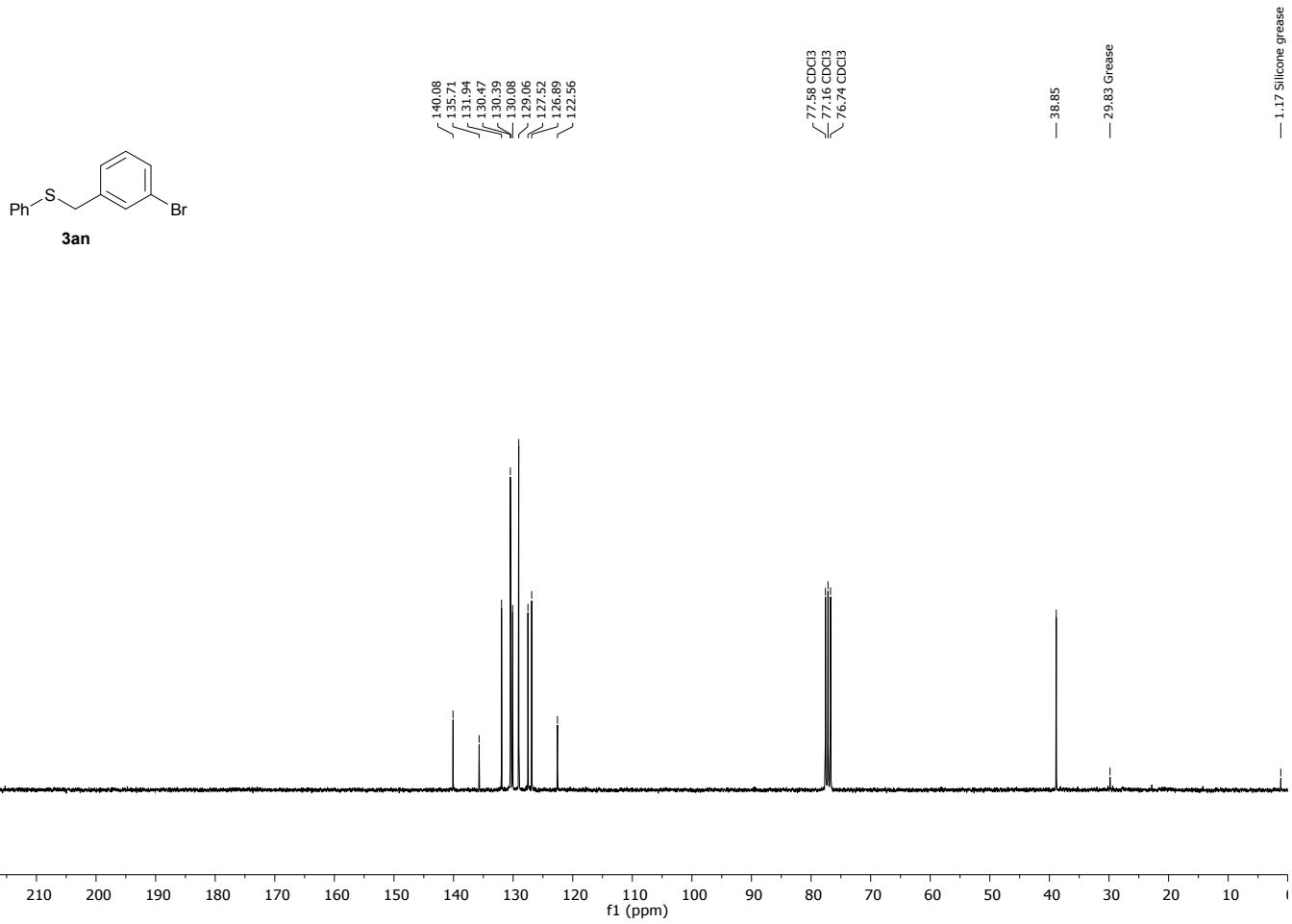
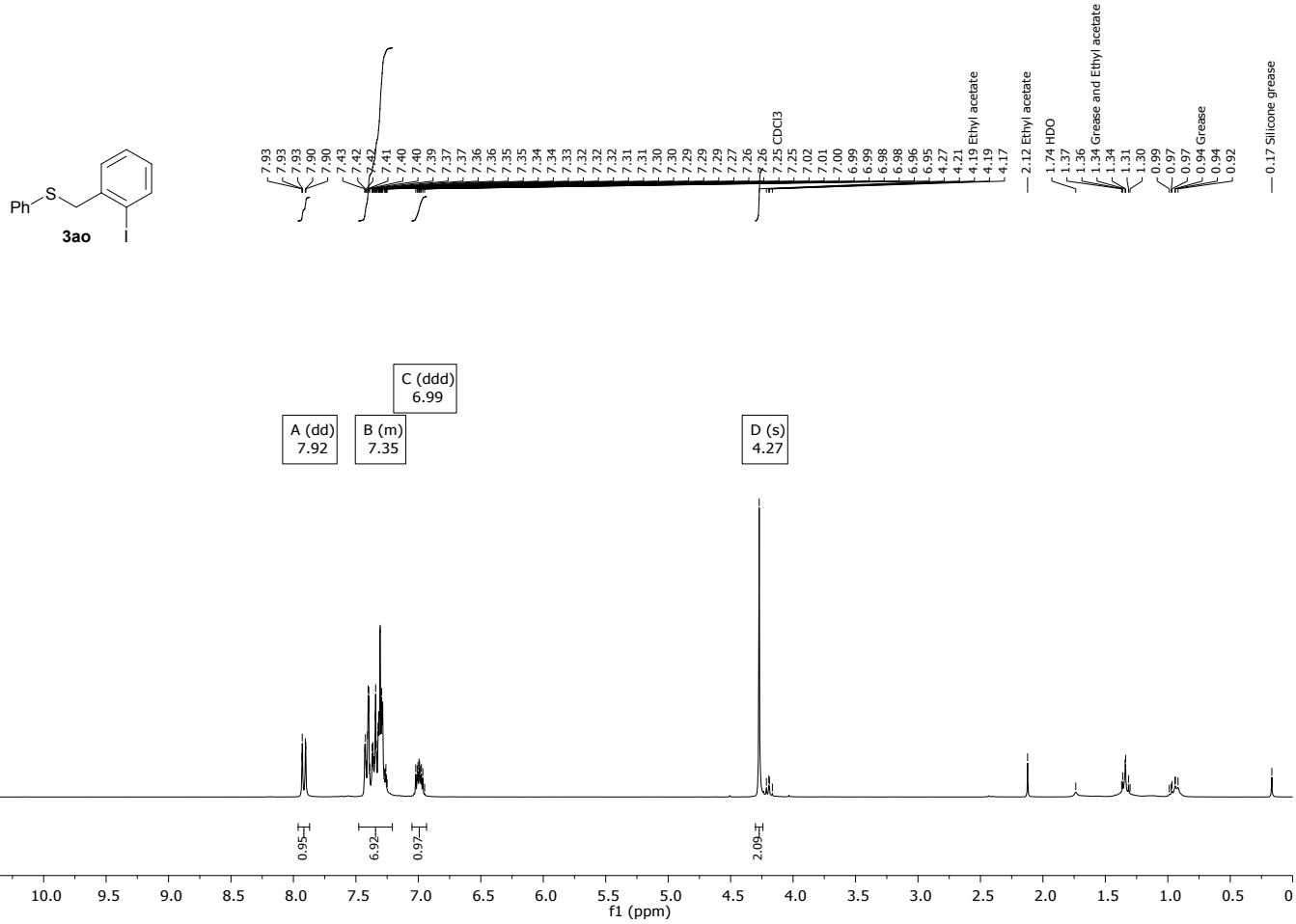
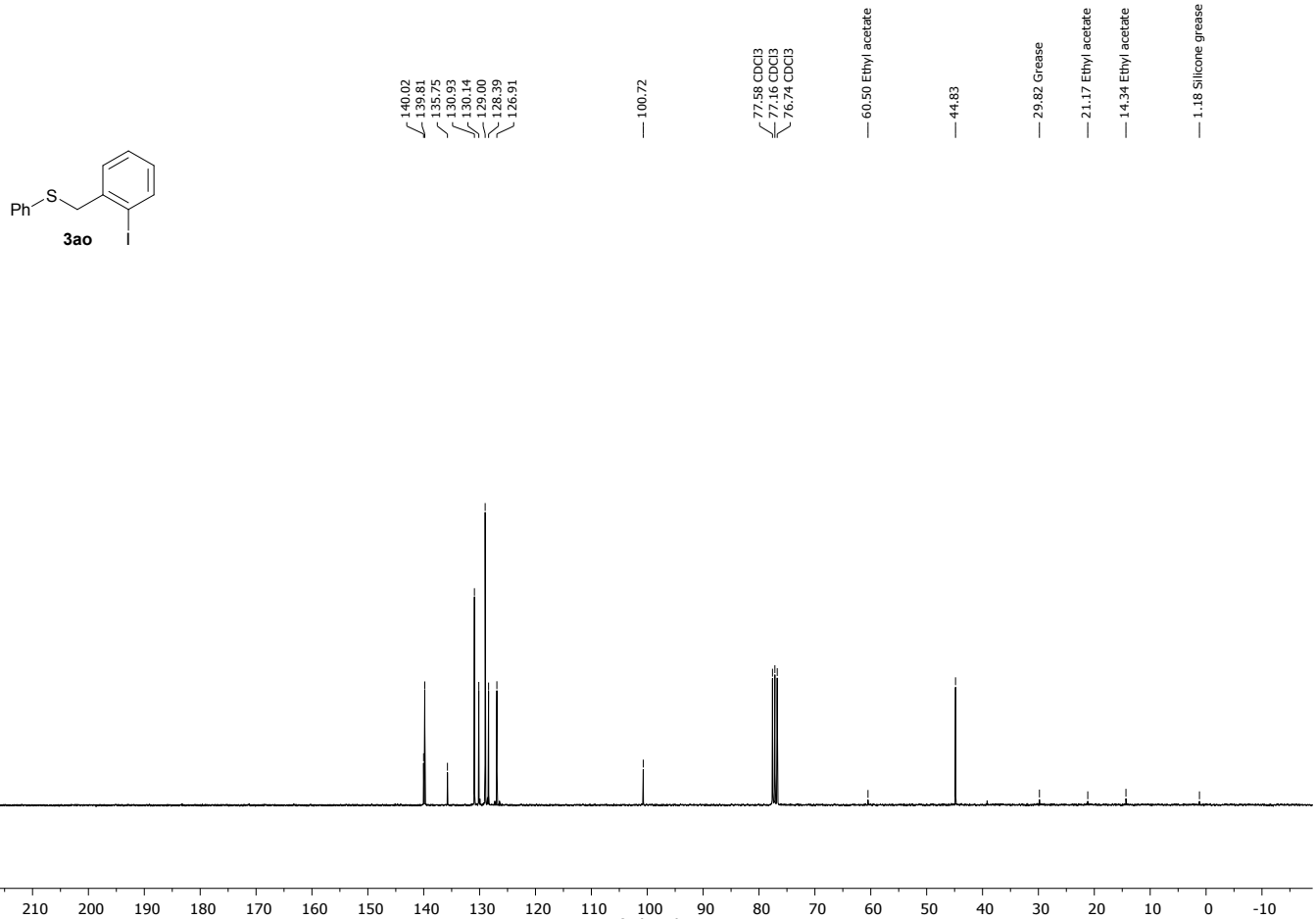
7.28
7.27
7.26
7.25
7.24
7.23
7.23
7.23
7.22
7.21
7.20
7.19
7.18
7.17
7.16
7.16
7.15
7.14
7.14
4.03
1.24 Grease
0.89
0.86
0.84 Grease
0.84
0.83
0.82
Cl
S
Ph
3am
A (m)
7.21
B (s)
4.03
8.62
2.00
f1 (ppm)

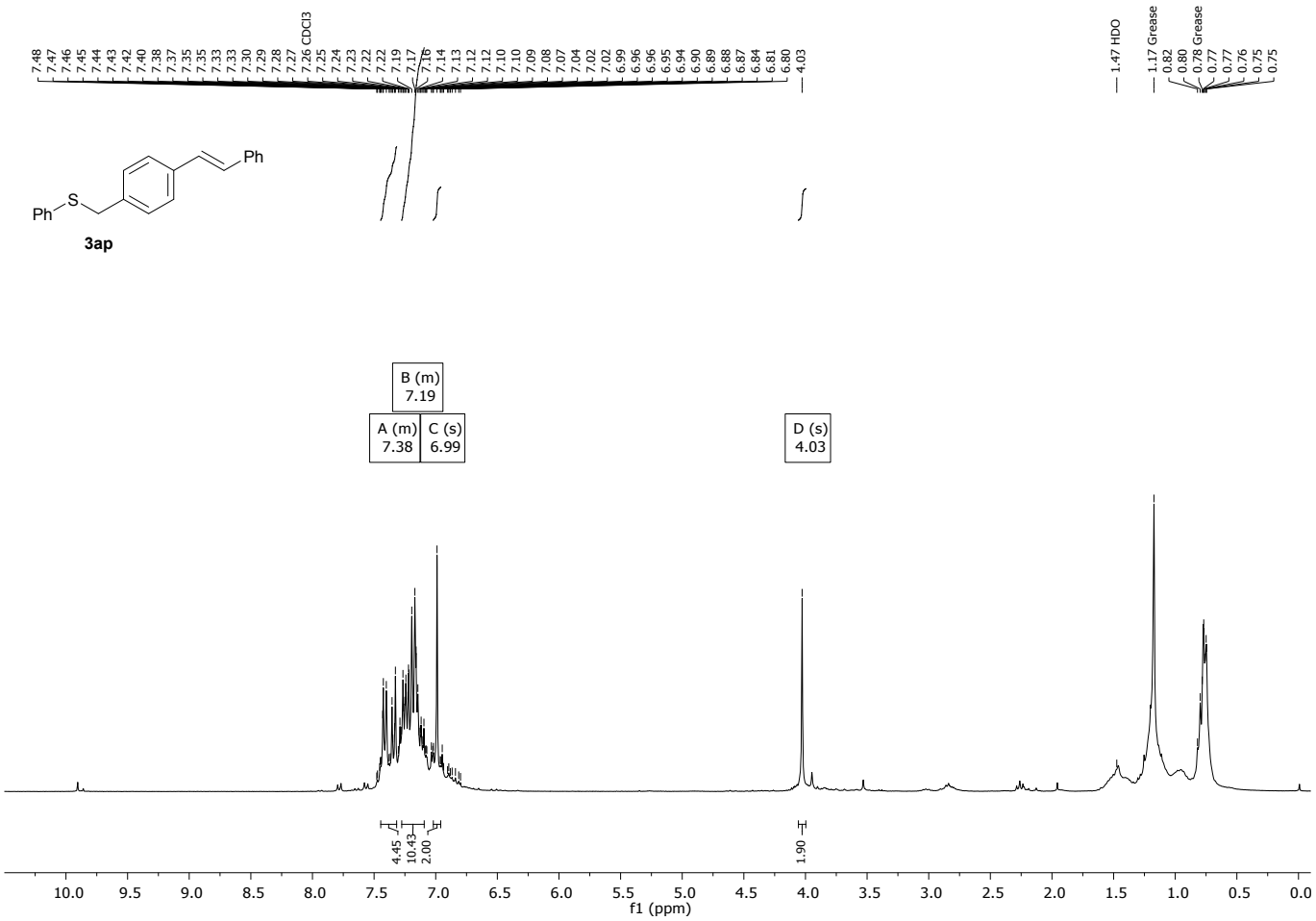

136.28
135.81
133.06
130.40
130.24
129.04
128.72
126.80
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
38.68
29.83 Grease
Cl
S
Ph
3am
f1 (ppm)

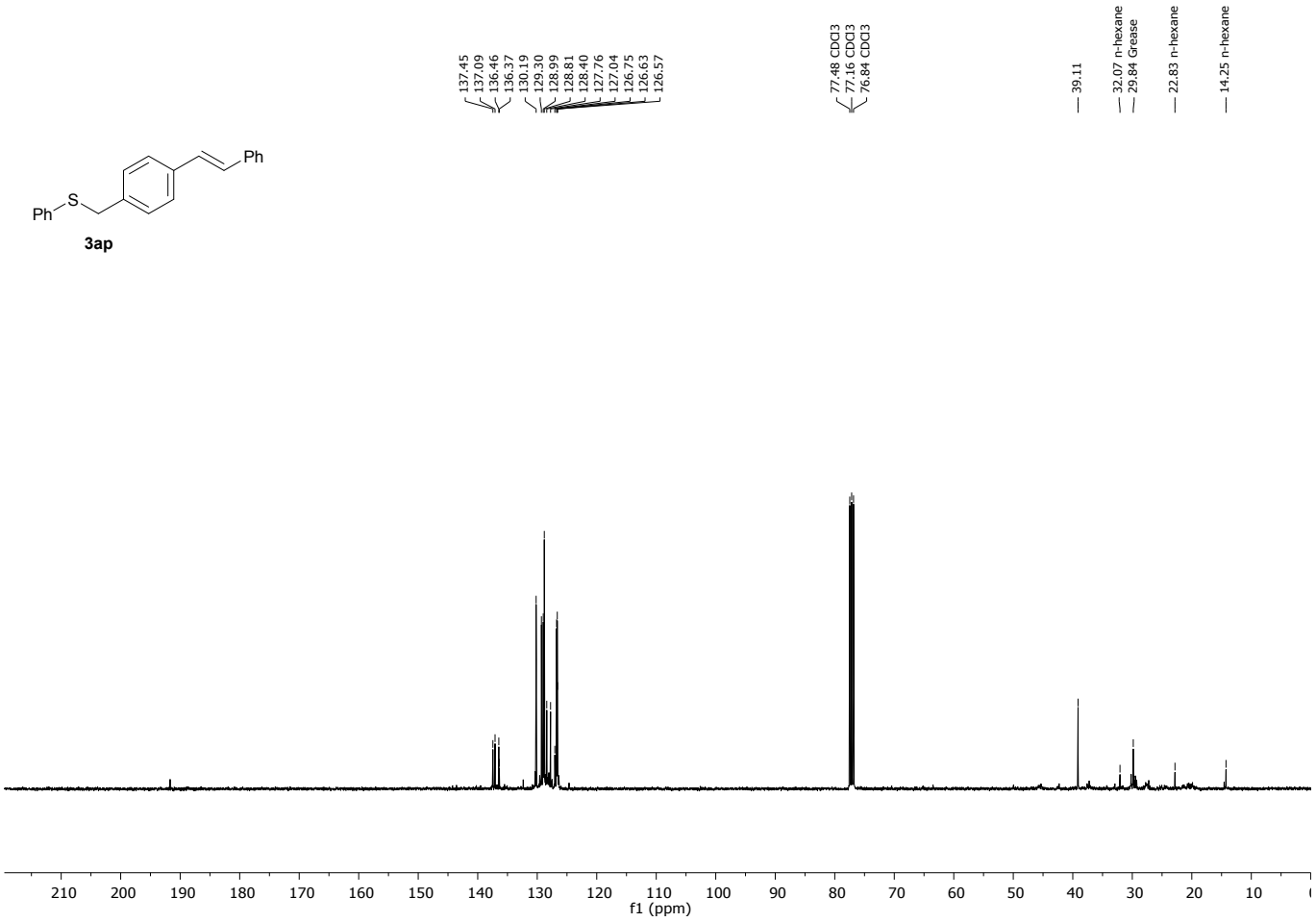

7.50
7.50
7.49
7.45
7.44
7.44
7.42
7.42
7.41
7.39
7.38
7.37
7.36
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.31
7.31
7.31
7.30
7.29
7.28
7.27
7.27
7.26 CDCl3
7.25
7.25
7.24
7.23
7.20
7.18
7.16
4.12
1.77 HDO
1.34 Grease
0.99
0.97
0.96
0.95
0.94 Grease
0.93
0.92
0.91
0.16 Silicone grease
Ph
S
Br
3an
B (m)
7.31
A (t)
7.50
C (s)
4.12
1.02
8.01
2.15
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)

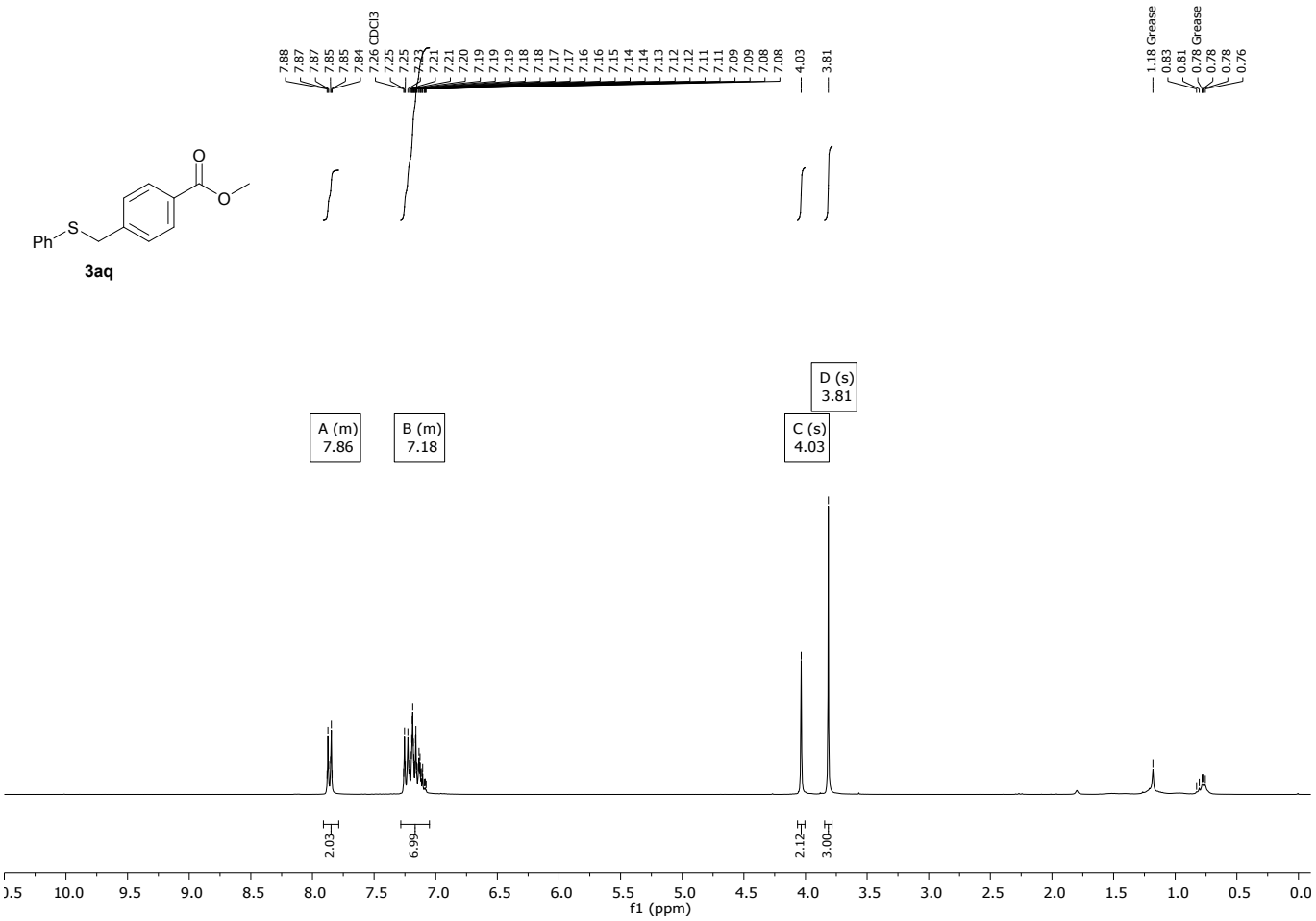

140.08
135.71
131.94
130.47
130.39
130.08
129.06
127.52
126.89
122.56
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
38.85
29.83 Grease
1.17 Silicone grease
Ph
S
Br
3an
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

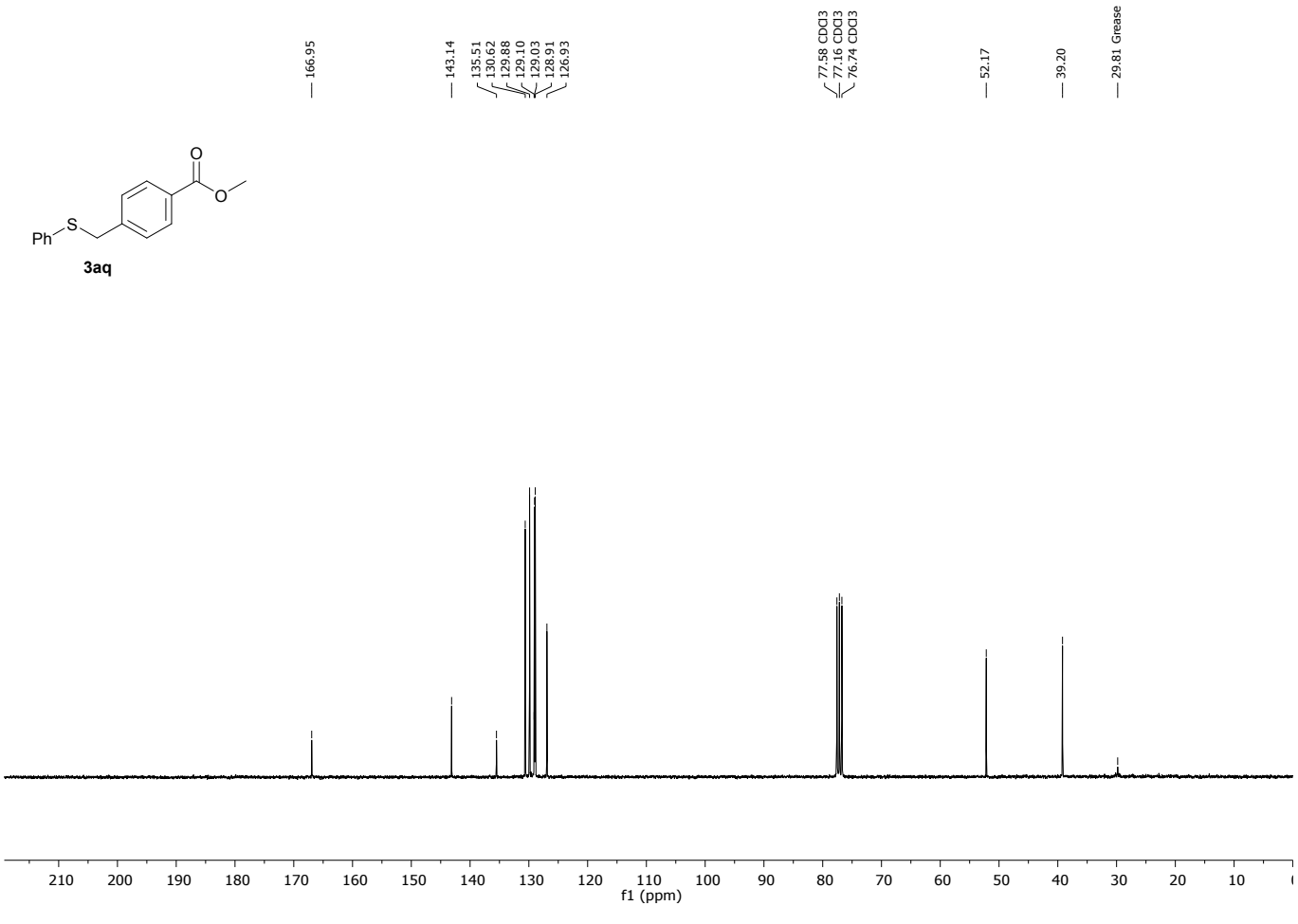

Ph
S
I
3ao
7.93 7.93 7.93 7.90 7.90 7.43 7.42 7.42 7.41 7.40 7.40 7.39 7.37 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.32 7.32 7.31 7.31 7.30 7.30 7.29 7.29 7.29 7.27 7.26 7.26 CDCl3 7.25 7.25 7.02 7.01 7.00 6.99 6.99 6.98 6.98 6.96 6.95 4.27 4.21 4.19 Ethyl acetate 4.19 4.17
2.12 Ethyl acetate
1.74 HDO 1.37 1.36 1.34 Grease and Ethyl acetate 1.34 1.31 1.30
0.99 0.97 0.97 0.94 Grease 0.94 0.92
0.17 Silicone grease
A (dd) 7.92
B (m) 7.35
C (ddd) 6.99
D (s) 4.27
0.95
6.92
0.97
2.09
f1 (ppm)

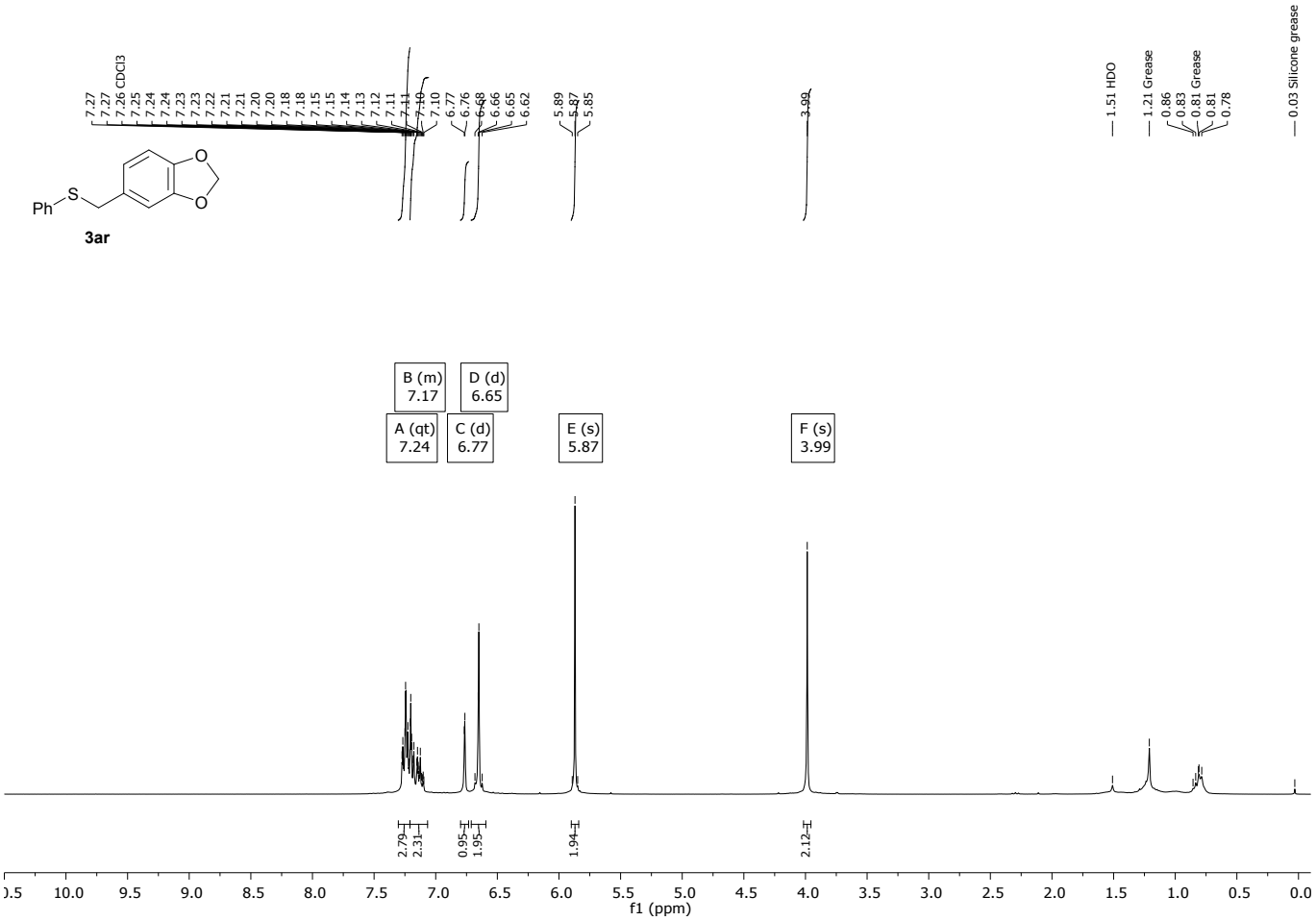

Ph
S
I
3ao
140.02 139.81 135.75 130.93 130.14 129.00 128.39 126.91
100.72
77.58 CDCl3 77.16 CDCl3 76.74 CDCl3
60.50 Ethyl acetate
44.83
29.82 Grease
21.17 Ethyl acetate
14.34 Ethyl acetate
1.18 Silicone grease
f1 (ppm)

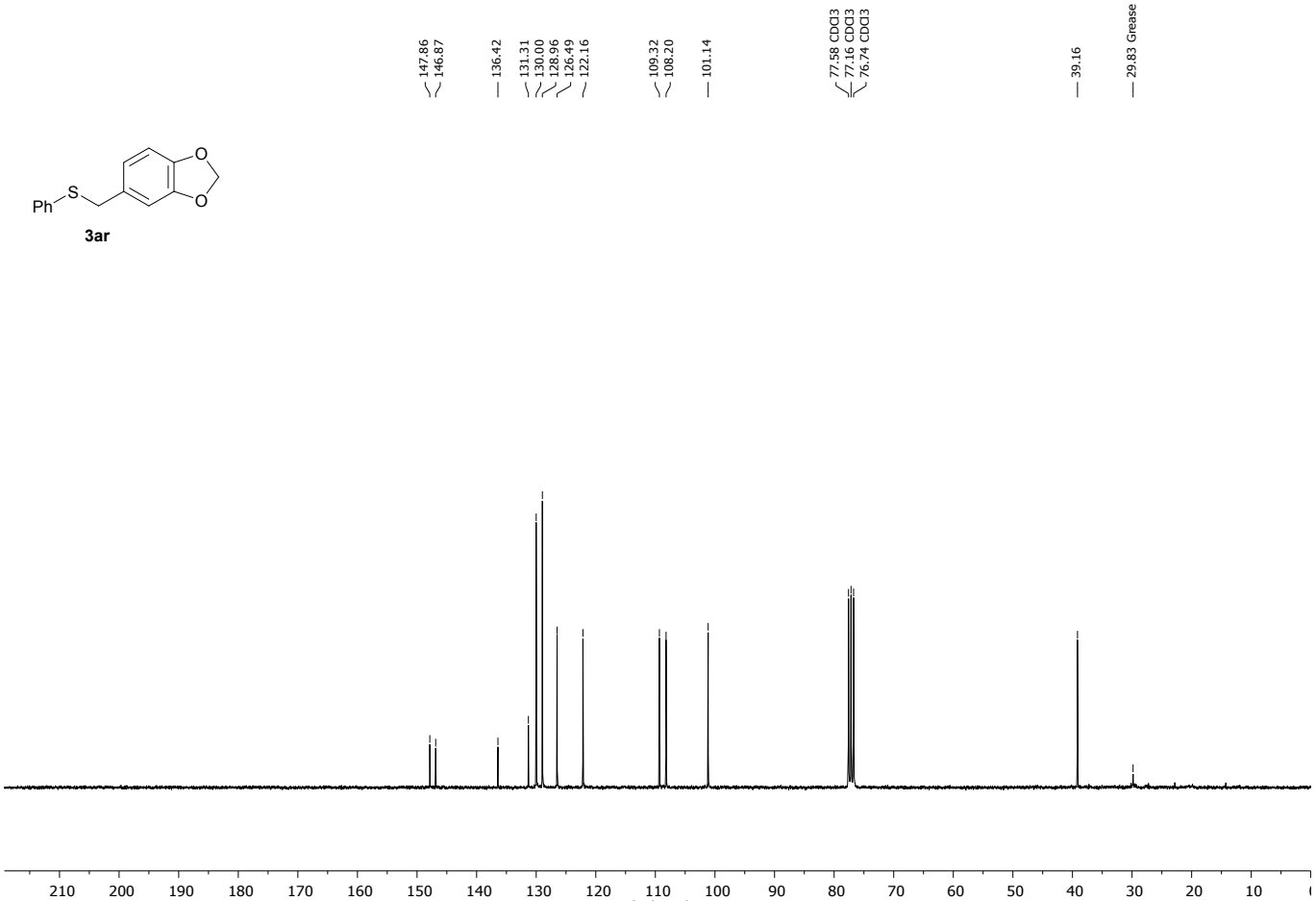

7.48 7.47 7.46 7.45 7.44 7.43 7.42 7.40 7.38 7.37 7.35 7.33 7.33 7.30 7.29 7.28 7.27 7.26 CDCl3 7.25 7.24 7.23 7.22 7.22 7.19 7.17 7.16 7.14 7.13 7.12 7.12 7.10 7.10 7.09 7.08 7.07 7.04 7.02 7.02 6.99 6.96 6.96 6.95 6.94 6.90 6.89 6.88 6.87 6.84 6.81 6.80 4.03
1.47 HDO
1.17 Grease
0.82
0.80
0.78 Grease
0.77
0.77
0.76
0.75
0.75
Ph
S
Ph
3ap
B (m) 7.19
A (m) 7.38
C (s) 6.99
D (s) 4.03
4.45
10.43
2.00
1.90
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)

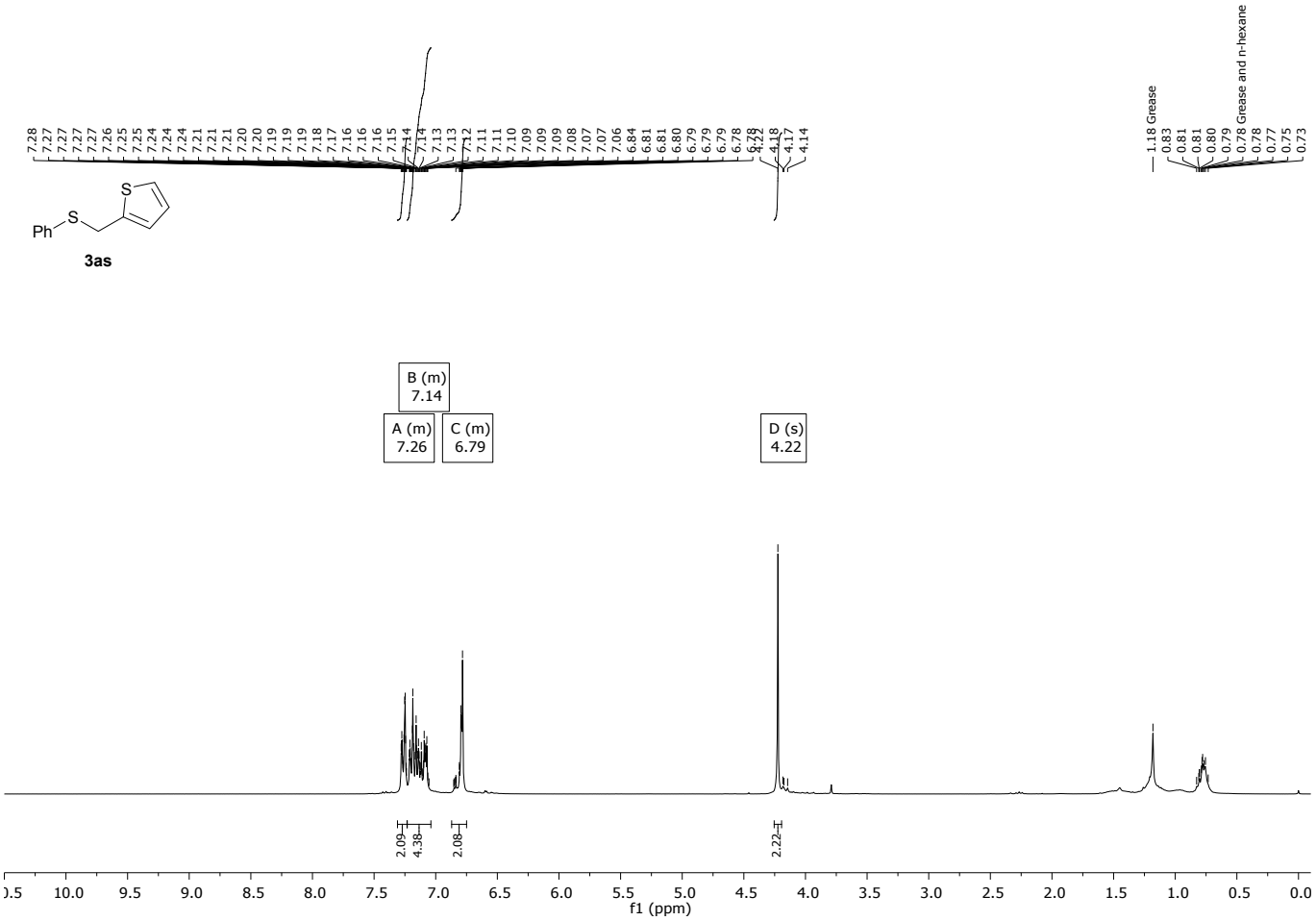

137.45
137.09
136.46
136.37
130.19
129.30
128.99
128.81
128.40
127.76
127.04
126.75
126.63
126.57
77.48 CDCl3
77.16 CDCl3
76.84 CDCl3
39.11
32.07 n-hexane
29.84 Grease
22.83 n-hexane
14.25 n-hexane
Ph
S
Ph
3ap
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

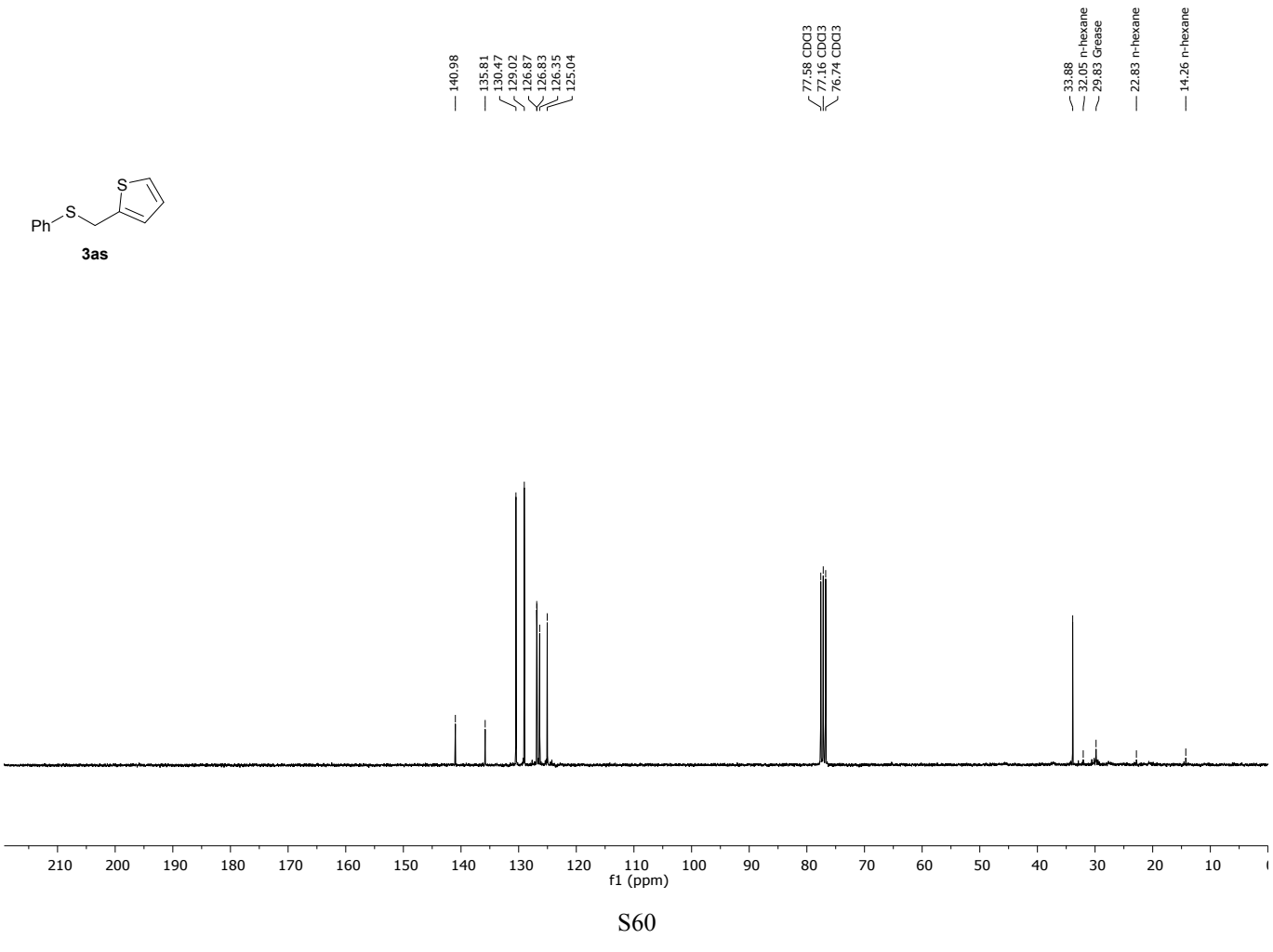

7.88
7.87
7.87
7.85
7.85
7.84
7.26 CDCl3
7.25
7.25
7.23
7.21
7.21
7.20
7.19
7.19
7.18
7.18
7.17
7.16
7.16
7.15
7.14
7.14
7.13
7.12
7.12
7.11
7.11
7.09
7.09
7.08
7.08
4.03
3.81
1.18 Grease
0.83
0.81
0.78 Grease
0.78
0.78
0.76
O
O
Ph
S
3aq
A (m)
7.86
B (m)
7.18
C (s)
4.03
D (s)
3.81
2.03
6.99
2.12
3.00
f1 (ppm)

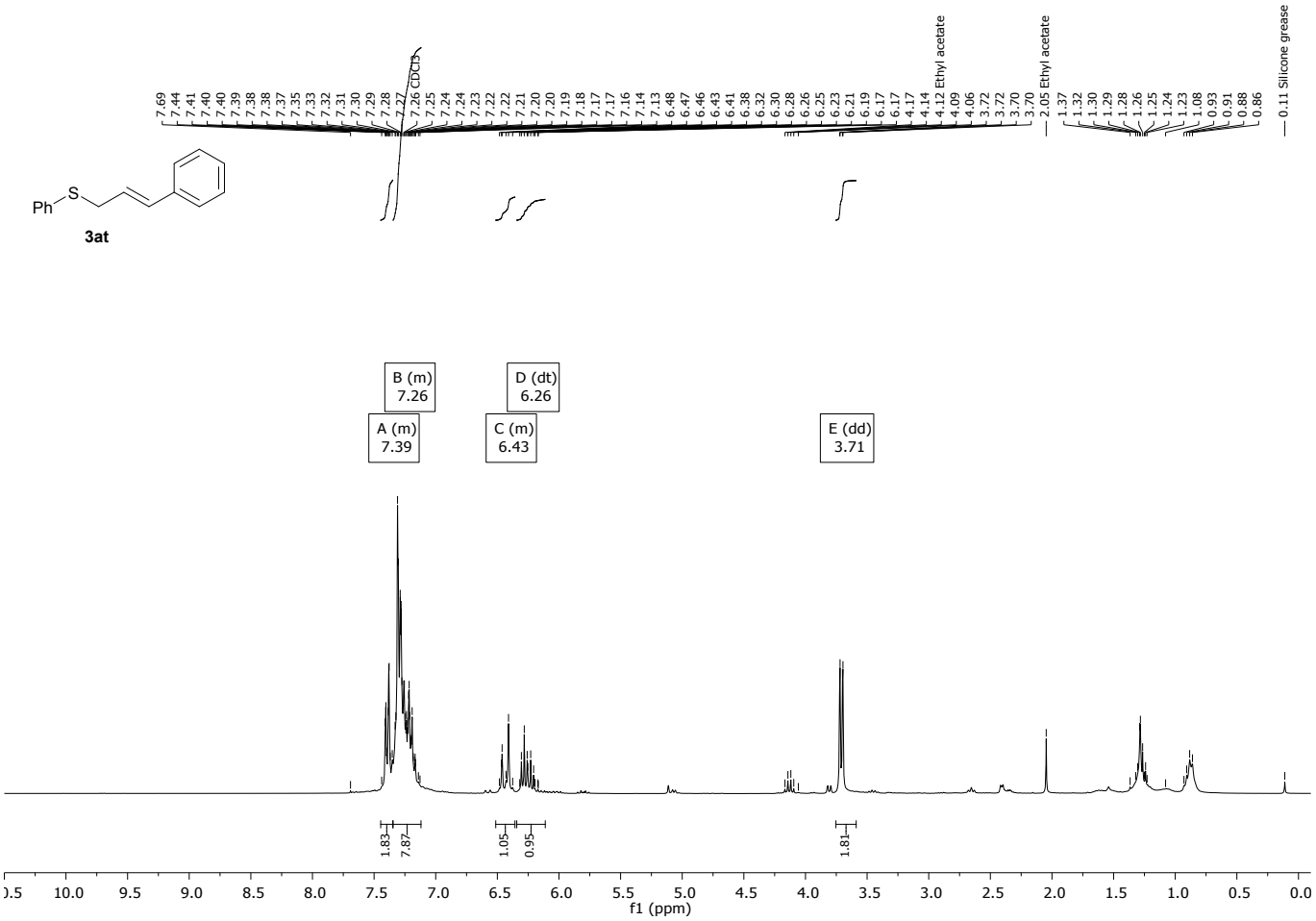

166.95
143.14
135.51
130.62
129.88
129.10
129.03
128.91
126.93
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
52.17
39.20
29.81 Grease
O
O
Ph
S
3aq
f1 (ppm)

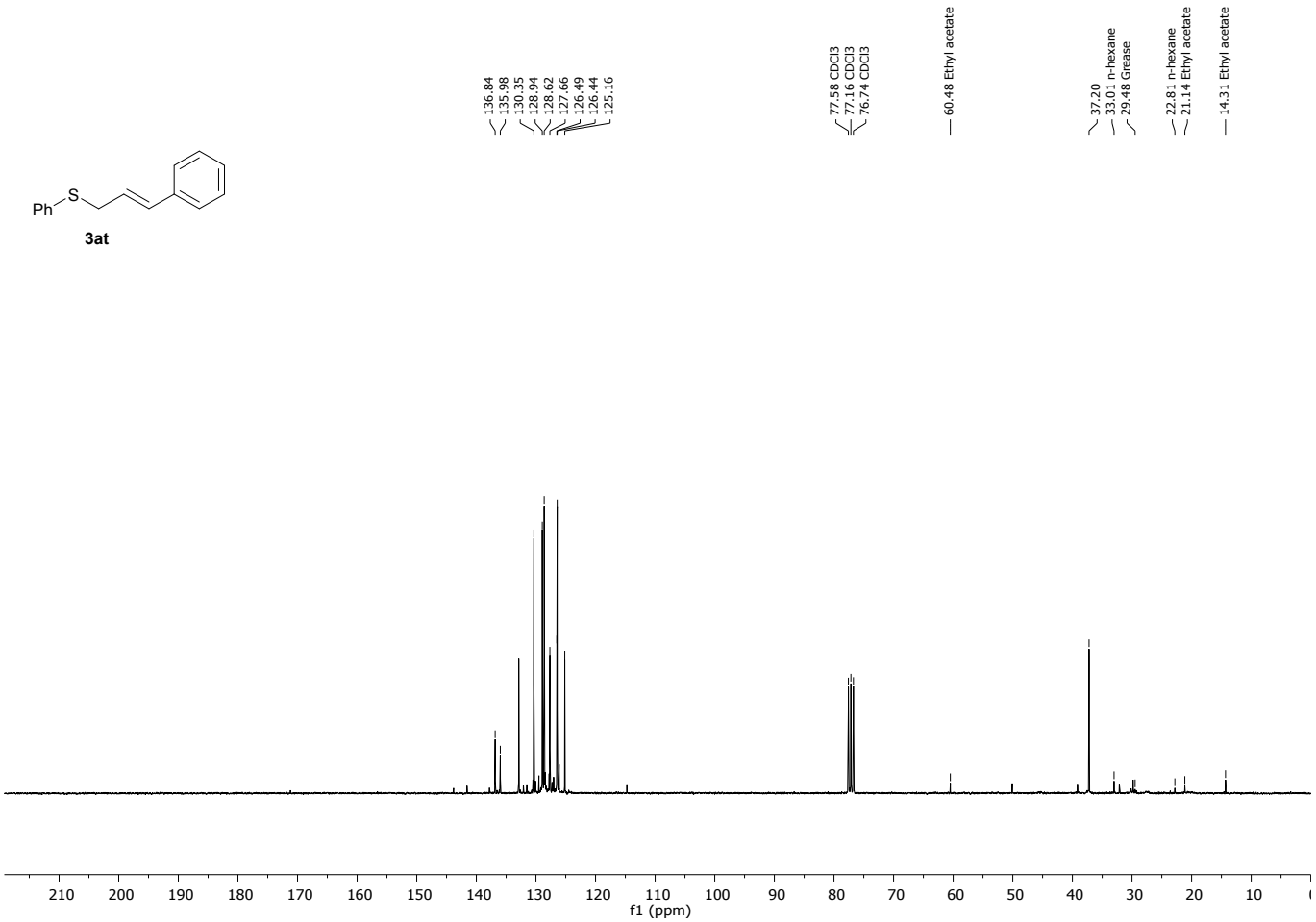

7.27
7.27
7.26 CDCl3
7.25
7.24
7.24
7.23
7.23
7.22
7.21
7.21
7.20
7.20
7.18
7.18
7.15
7.15
7.14
7.13
7.12
7.11
7.11
7.10
6.77
6.76
6.68
6.66
6.65
6.62
5.89
5.87
5.85
3.99
1.51 HDO
1.21 Grease
0.86
0.83
0.81 Grease
0.81
0.78
0.03 Silicone grease
Ph
S
O
O
3ar
B (m) 7.17
D (d) 6.65
A (qt) 7.24
C (d) 6.77
E (s) 5.87
F (s) 3.99
2.79
2.31
0.95
1.95
1.94
2.12
f1 (ppm)

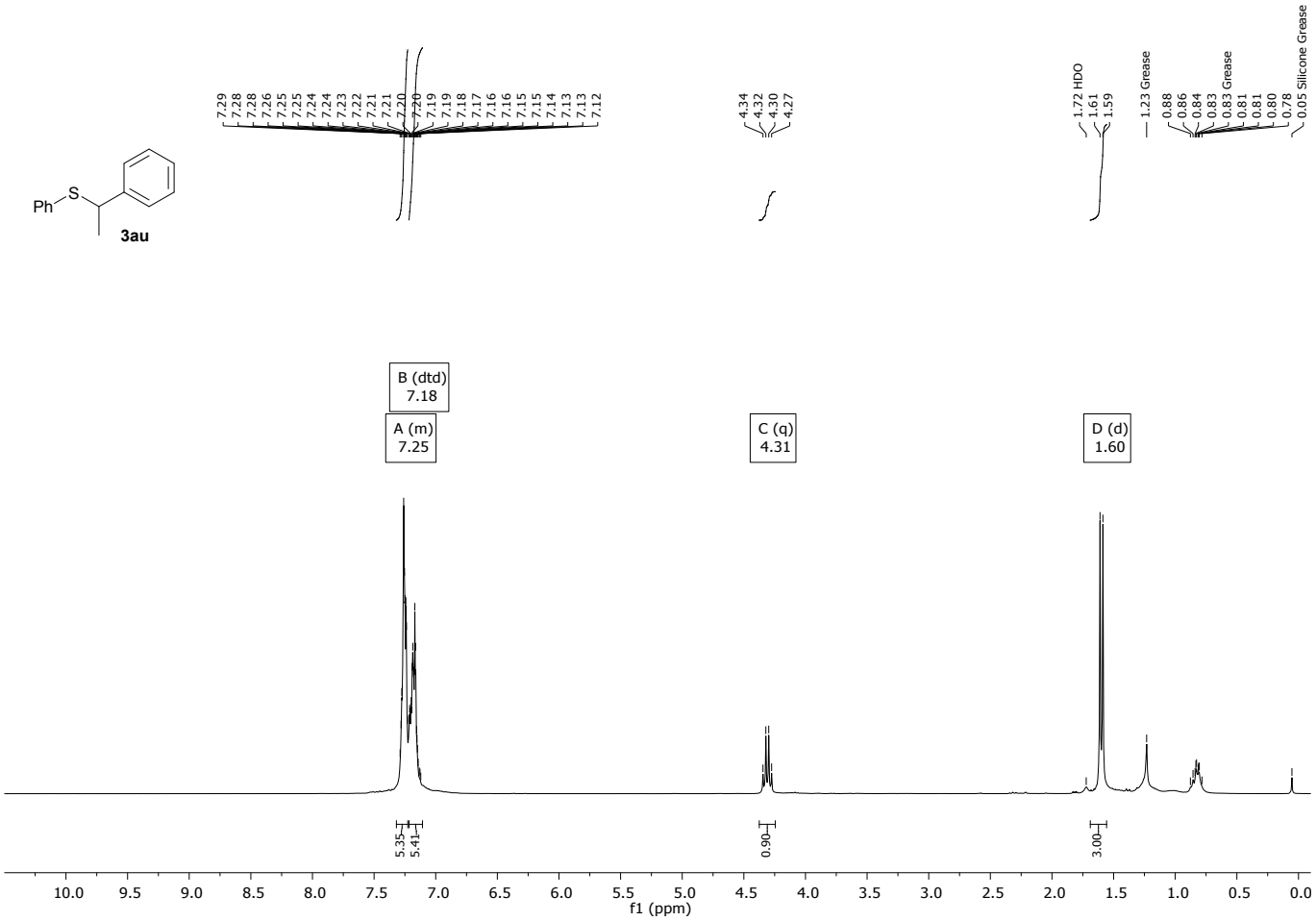

147.86
146.87
136.42
131.31
130.00
128.96
126.49
122.16
109.32
108.20
101.14
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
39.16
29.83 Grease
Ph
S
O
O
3ar
f1 (ppm)


7.28 7.27 7.27 7.27 7.27 7.26 7.25 7.24 7.24 7.24 7.21 7.21 7.21 7.20 7.20 7.19 7.19 7.19 7.18 7.17 7.16 7.16 7.16 7.15 7.14 7.14 7.13 7.13 7.12 7.11 7.11 7.10 7.09 7.09 7.09 7.08 7.07 7.07 7.06 6.84 6.81 6.81 6.80 6.79 6.79 6.79 6.78 6.78 4.22 4.18 4.17 4.14
1.18 Grease
0.83
0.81
0.81
0.80
0.79 Grease and n-hexane
0.78
0.78
0.77
0.75
0.73
S
Ph
S
3as
B (m) 7.14
A (m) 7.26
C (m) 6.79
D (s) 4.22
2.09
4.38
2.08
2.22
f1 (ppm)


140.98
135.81
130.47
129.02
126.87
126.83
126.35
125.04
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
33.88
32.05 n-hexane
29.83 Grease
22.83 n-hexane
14.26 n-hexane
S
Ph
S
3as
f1 (ppm)


7.69 7.44 7.41 7.40 7.40 7.39 7.38 7.38 7.37 7.35 7.33 7.32 7.31 7.30 7.29 7.28 7.27 7.26 CDCl3 7.25 7.24 7.24 7.23 7.22 7.22 7.21 7.20 7.20 7.19 7.18 7.17 7.17 7.16 7.14 7.13 6.48 6.47 6.46 6.43 6.41 6.38 6.32 6.30 6.28 6.26 6.25 6.23 6.21 6.19 6.17 6.17 4.17 4.14 4.12 Ethyl acetate 4.09 4.06 3.72 3.72 3.70 3.70 2.05 Ethyl acetate 1.37 1.32 1.30 1.29 1.28 1.26 1.25 1.24 1.23 1.08 0.93 0.91 0.88 0.86 0.11 Silicone grease
Ph S
3at
B (m) 7.26
D (dt) 6.26
A (m) 7.39
C (m) 6.43
E (dd) 3.71
1.83 7.87 1.05 0.95 1.81
f1 (ppm)


136.84 135.98 130.35 128.94 128.62 127.66 126.49 126.44 125.16
77.58 CDCl3 77.16 CDCl3 76.74 CDCl3
60.48 Ethyl acetate
37.20 33.01 n-hexane 29.48 Grease
22.81 n-hexane 21.14 Ethyl acetate
14.31 Ethyl acetate
Ph S
3at
f1 (ppm)


7.29
7.28
7.28
7.26
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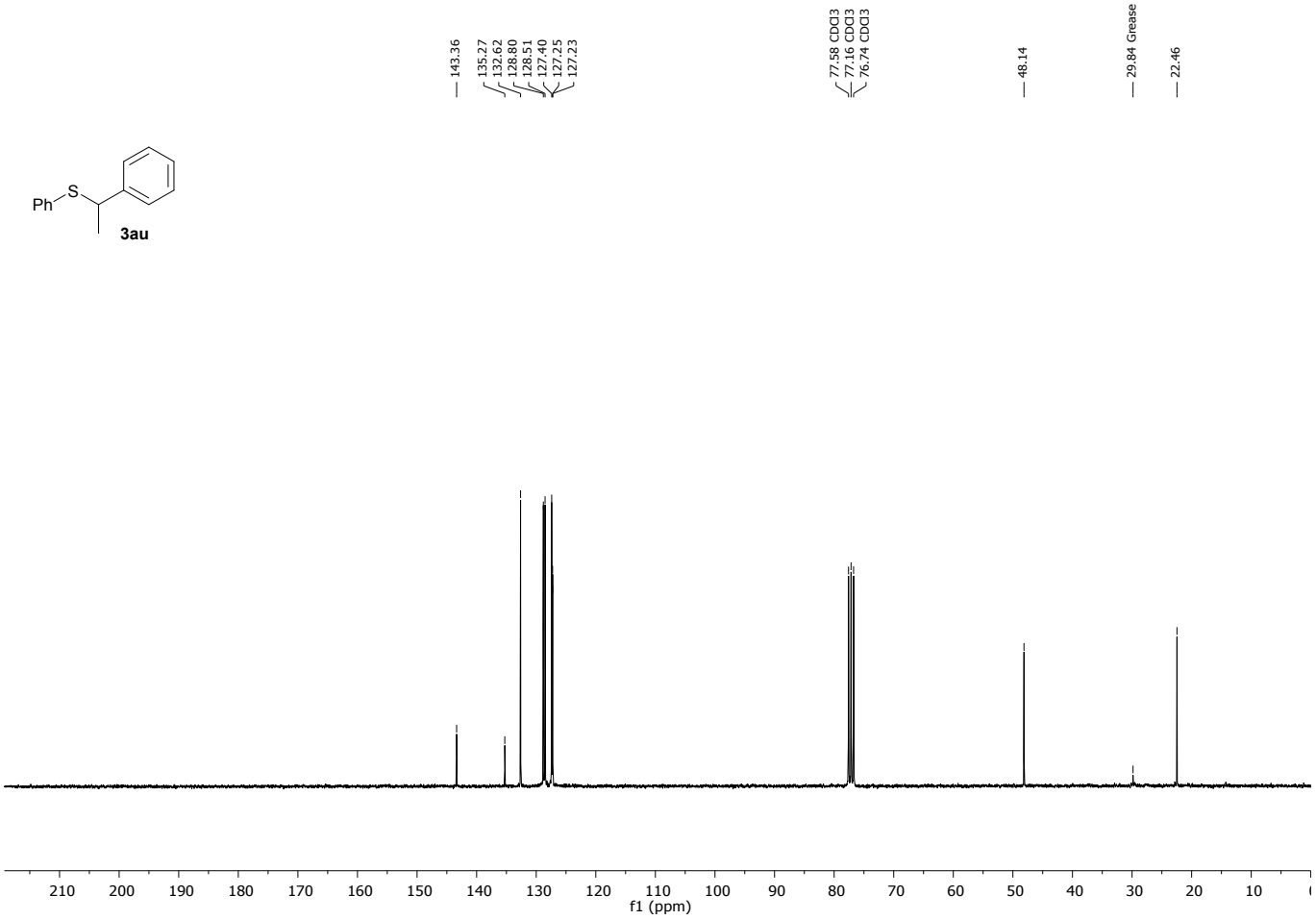
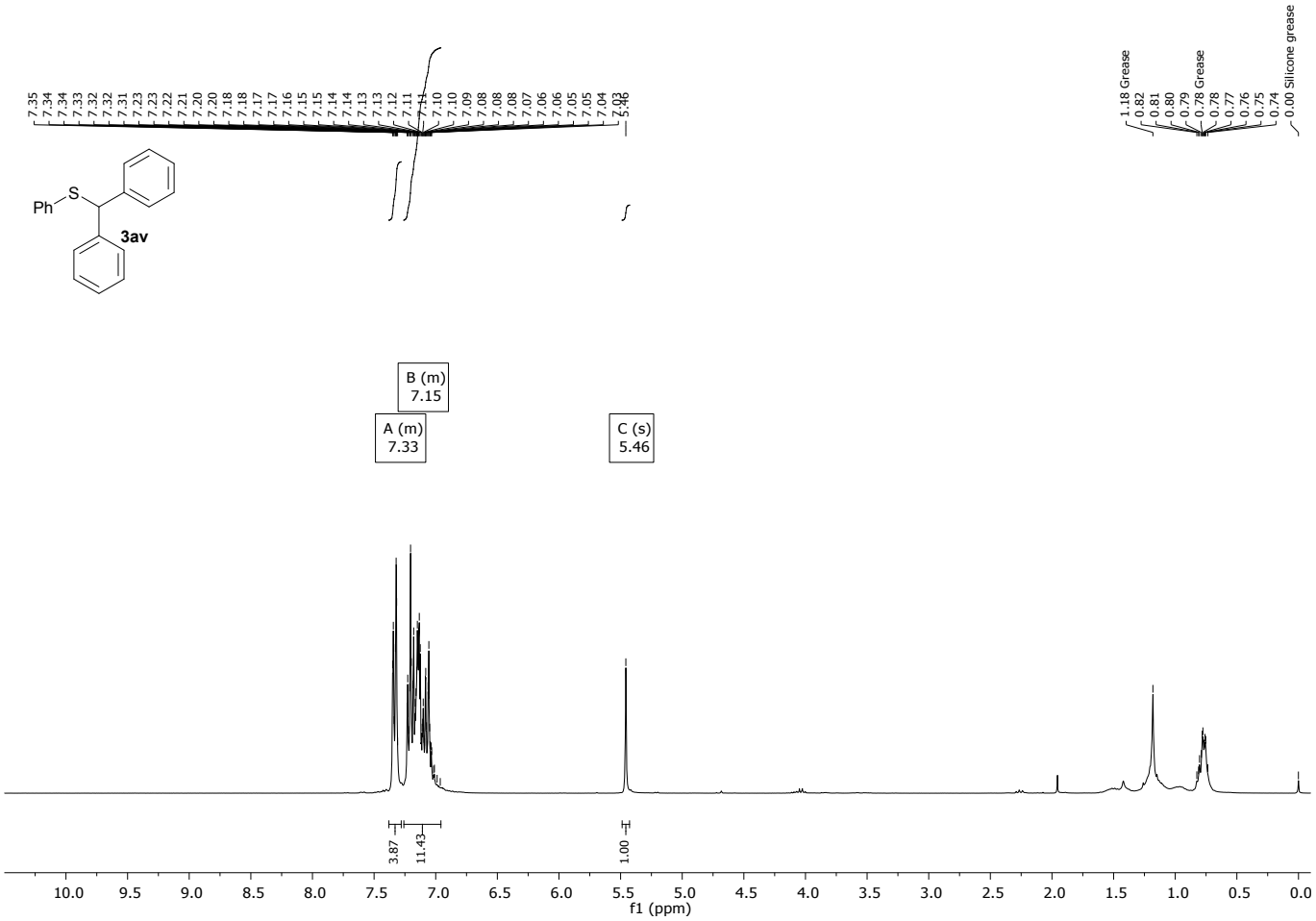
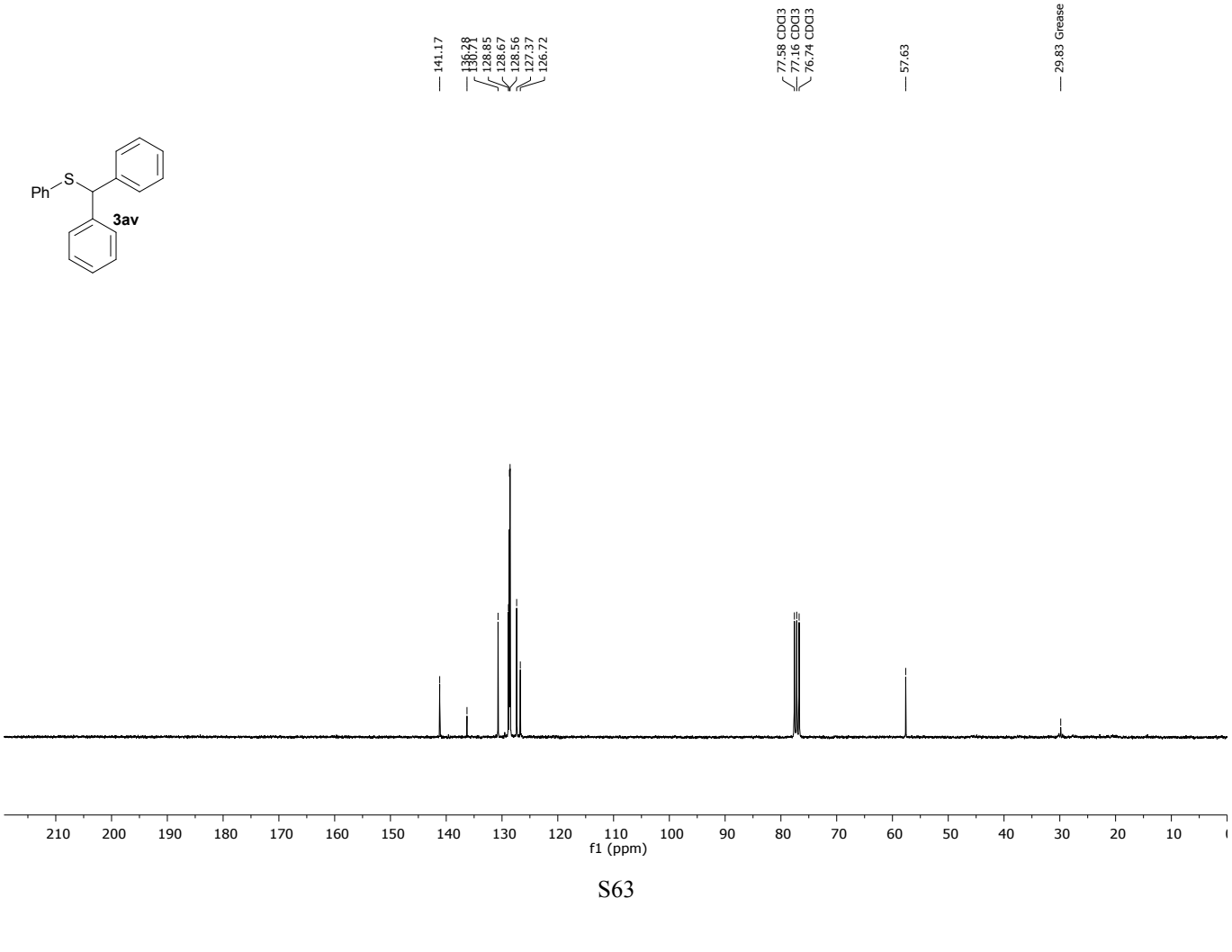
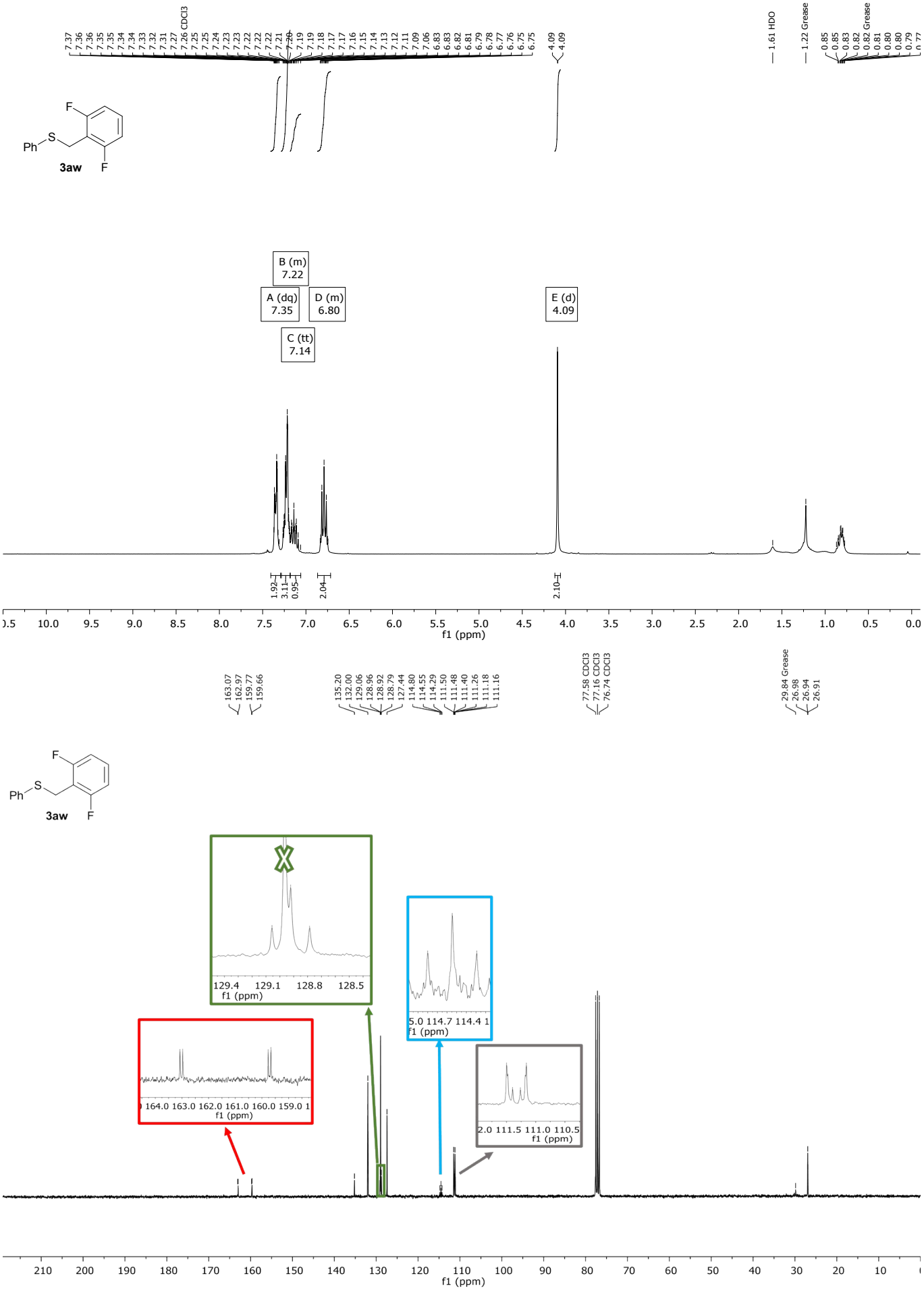
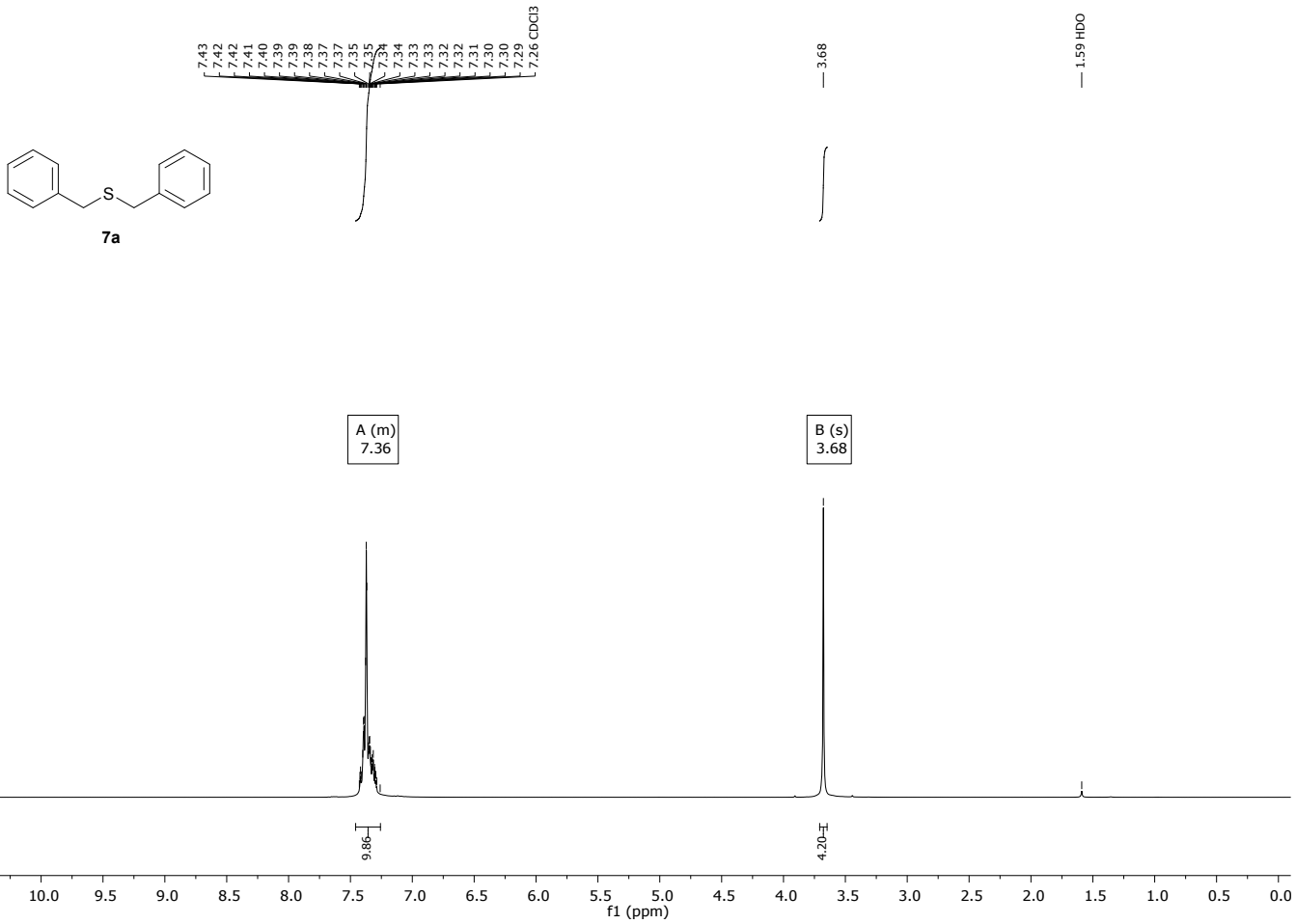
7.25
7.24
7.24
7.23
7.22
7.21
7.21
7.20
7.20
7.19
7.19
7.18
7.17
7.16
7.16
7.15
7.15
7.14
7.13
7.13
7.12
4.34
4.32
4.30
4.27
1.72 HDO
1.61
1.59
1.23 Grease
0.88
0.86
0.84
0.83
0.83 Grease
0.81
0.81
0.80
0.78
0.05 Silicone Grease
Ph
S
3au
B (dtd)
7.18
A (m)
7.25
C (q)
4.31
D (d)
1.60
5.35
5.41
0.90
3.00
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
f1 (ppm)

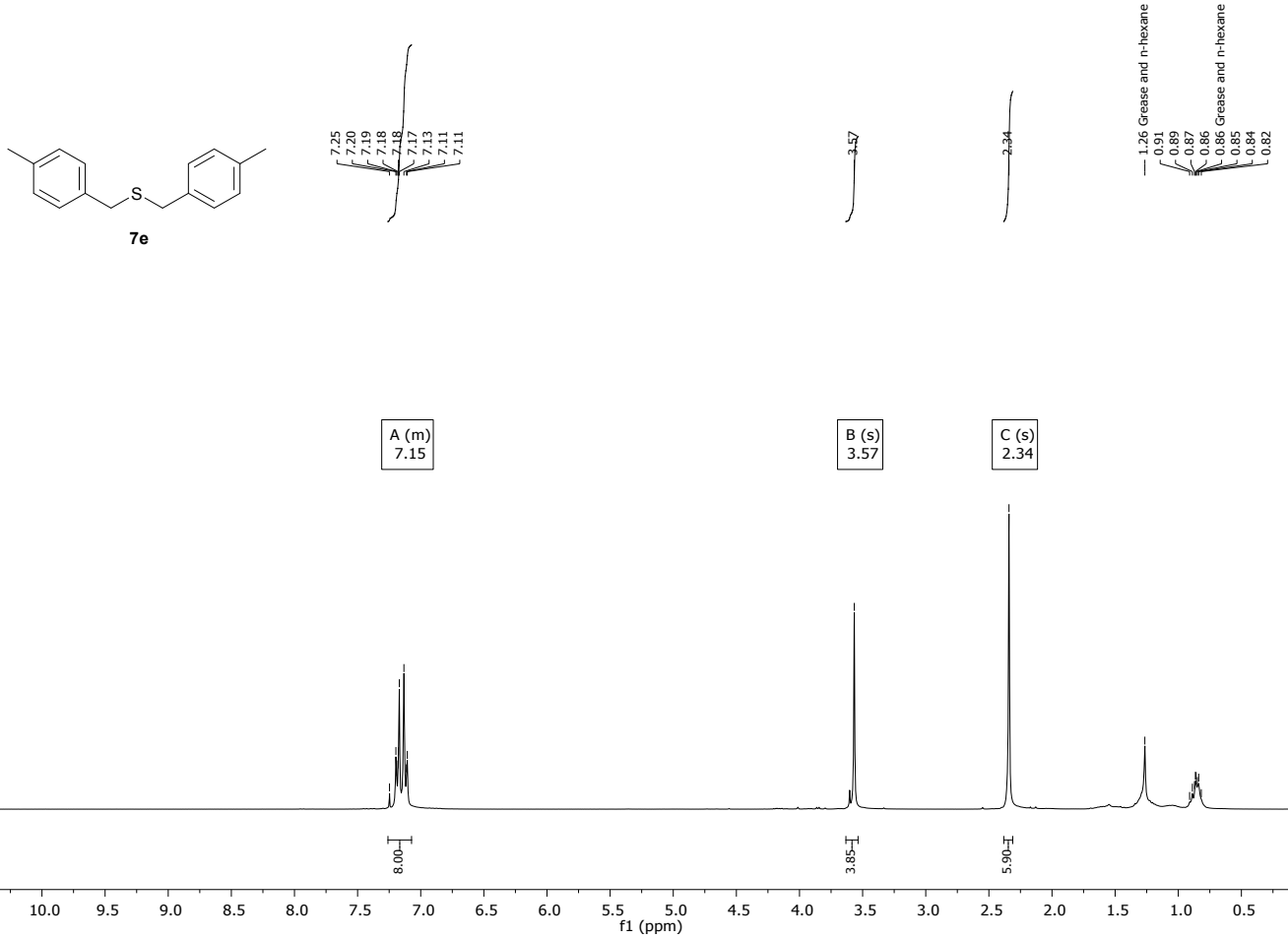

143.36
135.27
132.62
128.80
128.51
127.40
127.25
127.23
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
48.14
29.84 Grease
22.46
Ph
S
3au
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

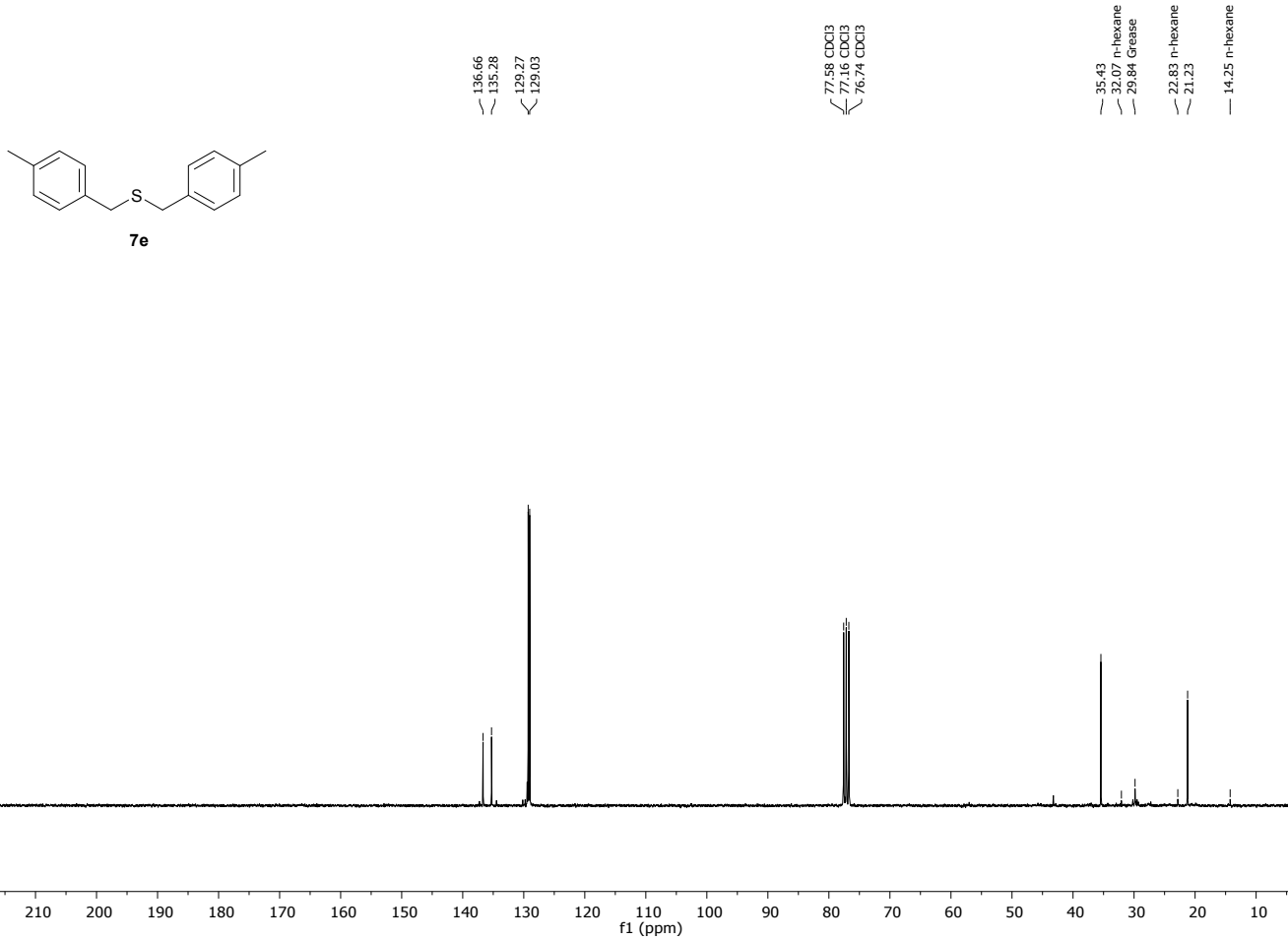

7.35 7.34 7.34 7.33 7.32 7.32 7.31 7.23 7.23 7.22 7.21 7.20 7.20 7.18 7.18 7.17 7.17 7.16 7.15 7.15 7.14 7.14 7.13 7.13 7.12 7.11 7.11 7.10 7.10 7.09 7.08 7.08 7.08 7.07 7.06 7.06 7.05 7.05 7.04 7.03 5.46
1.18 Grease 0.82 0.81 0.80 0.79 0.78 Grease 0.78 0.77 0.76 0.75 0.74 0.00 Silicone grease
Ph S 3av
B (m) 7.15
A (m) 7.33
C (s) 5.46
3.87 11.43 1.00
f1 (ppm)

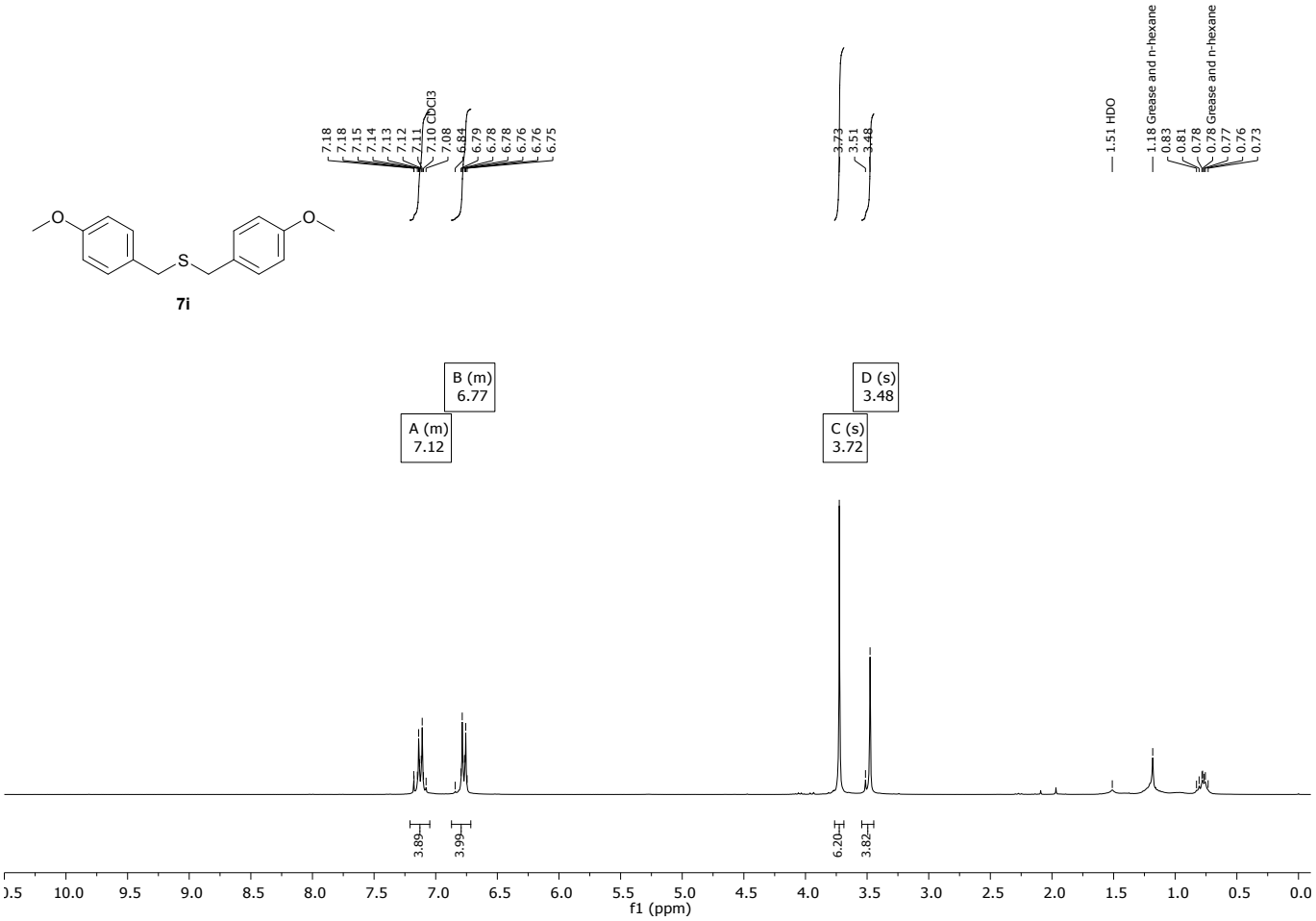

141.17 136.28 130.71 128.85 128.67 128.56 127.37 126.72
77.58 CDCl3 77.16 CDCl3 76.74 CDCl3
57.63
29.83 Grease
Ph S 3av
f1 (ppm)

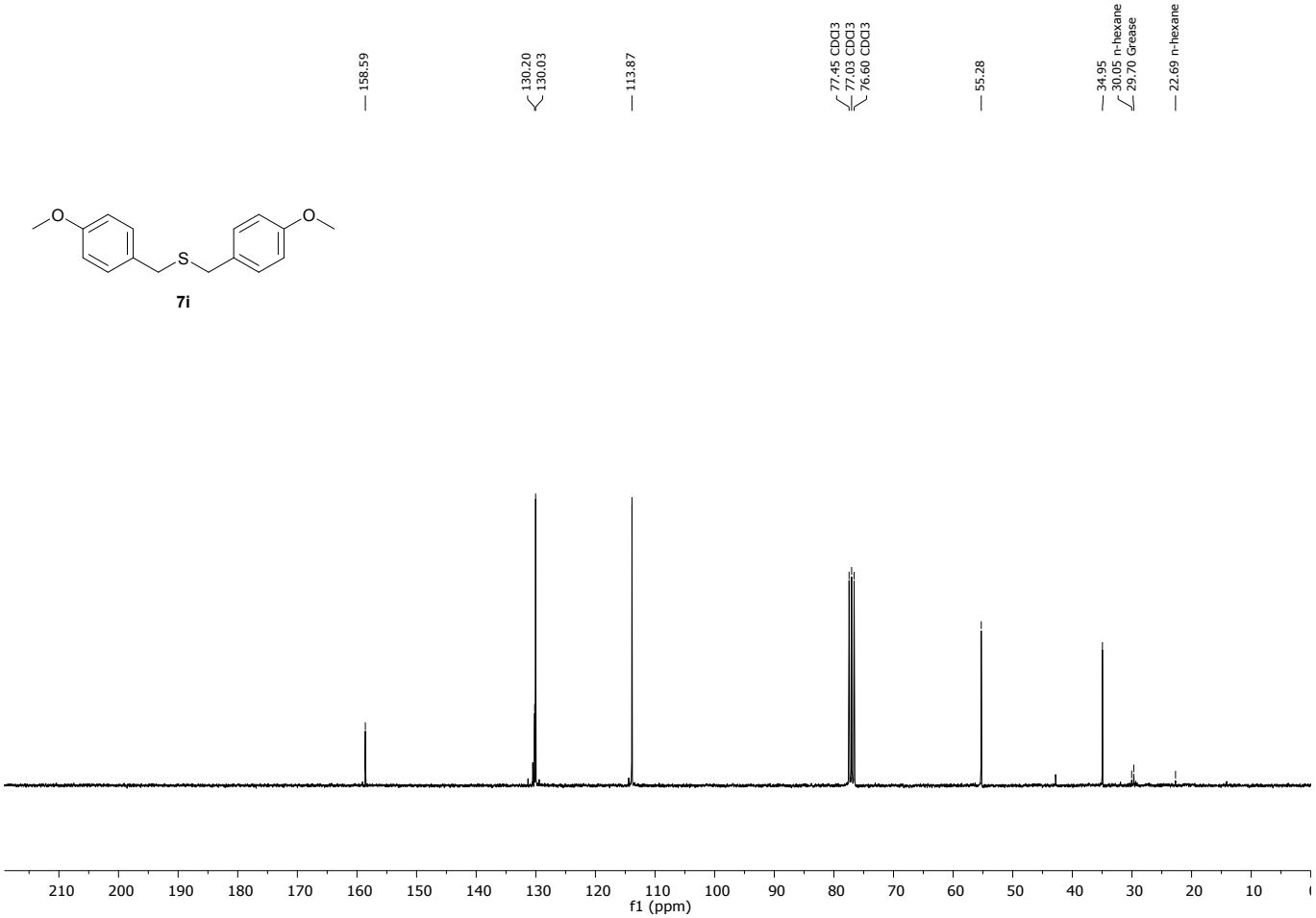

3aw
B (m) 7.22
A (dq) 7.35
D (m) 6.80
C (tt) 7.14
E (d) 4.09
f1 (ppm)

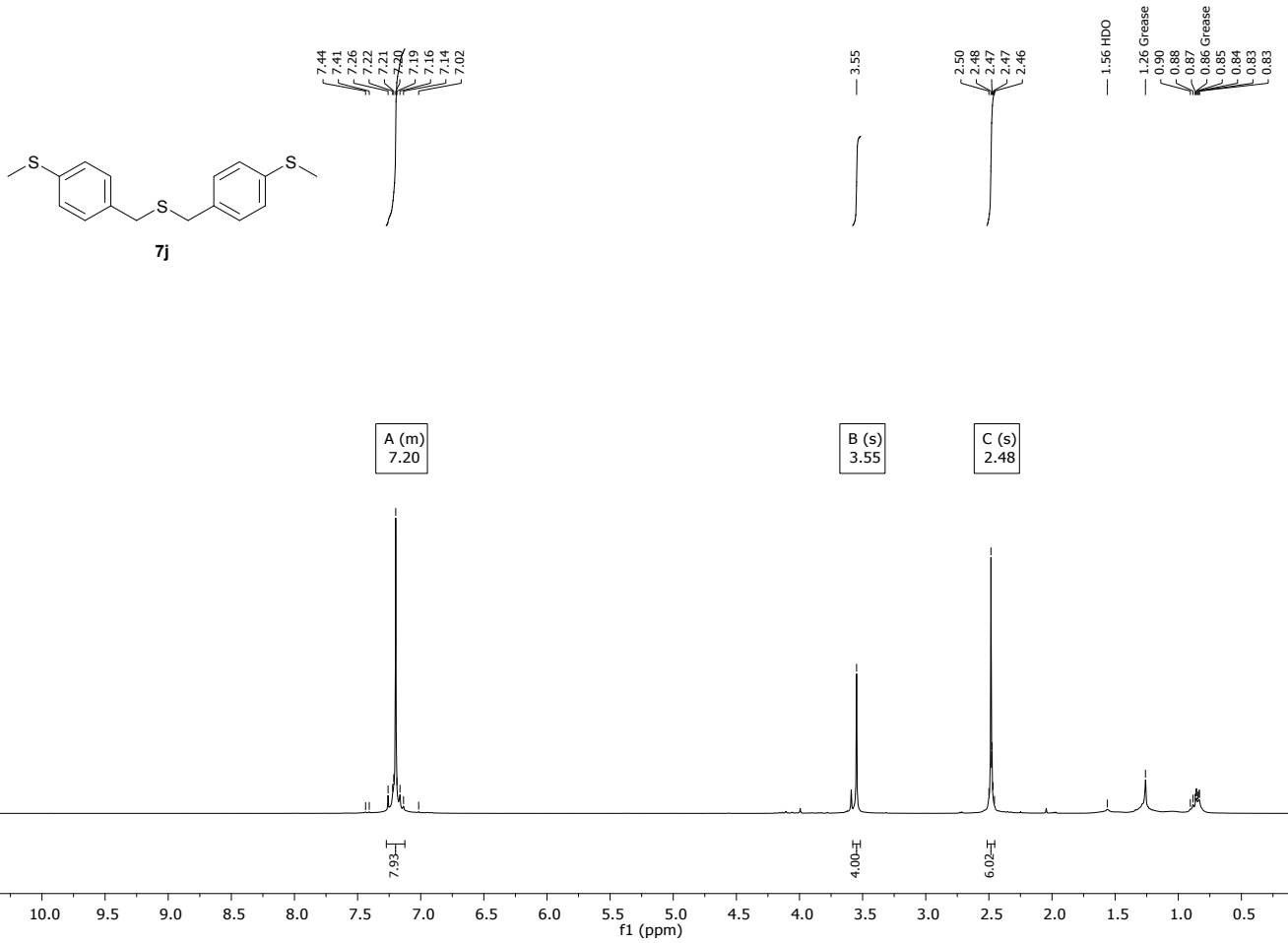

7.43
7.42
7.42
7.41
7.40
7.39
7.39
7.38
7.37
7.37
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.31
7.30
7.30
7.29
7.26 CDCl3
3.68
1.59 HDO
S
7a
A (m)
7.36
B (s)
3.68
9.86
4.20
f1 (ppm)
138.28
129.12
128.59
127.08
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
35.76
S
7a
f1 (ppm)

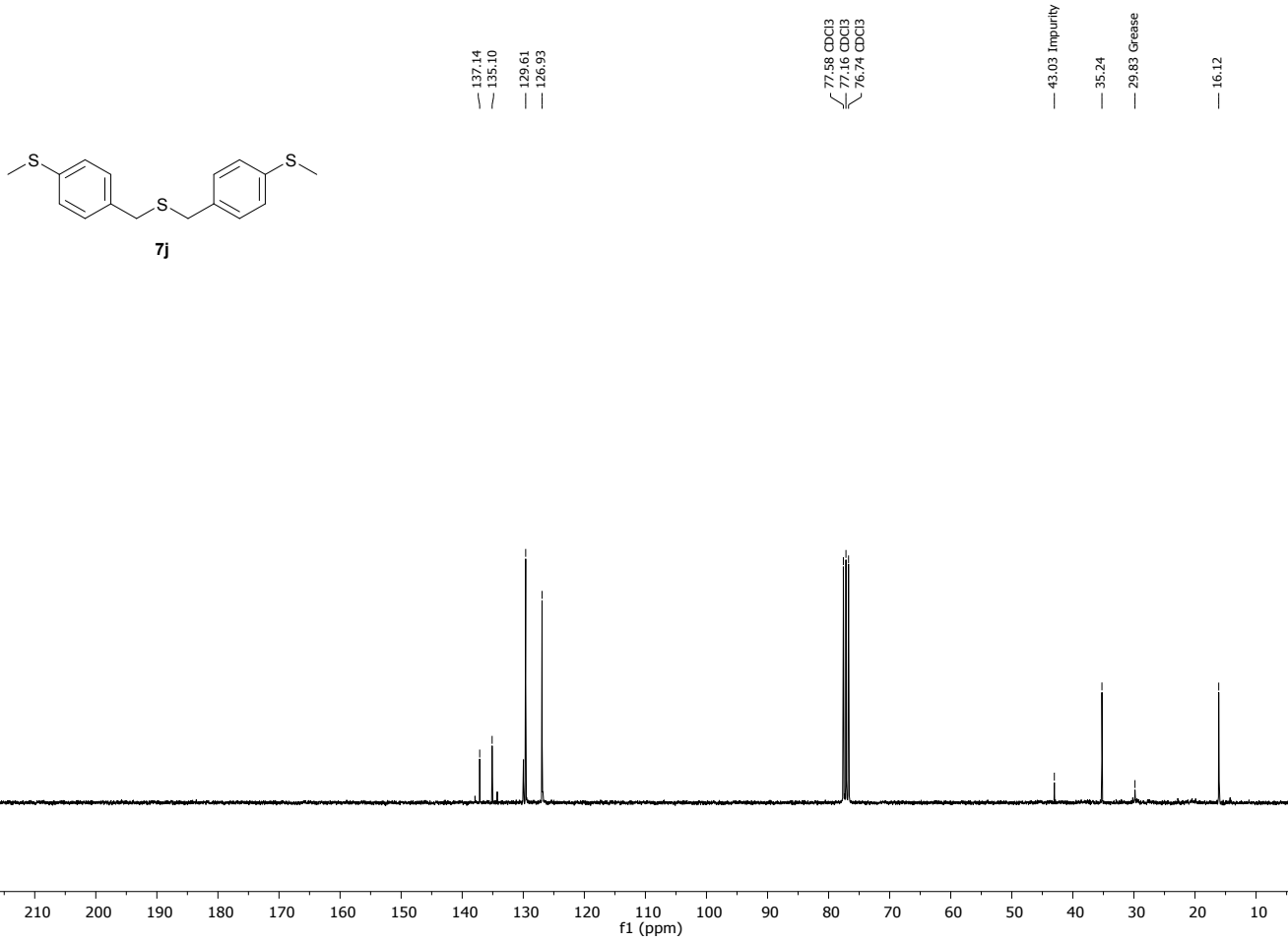

7e
7.25
7.20
7.19
7.18
7.18
7.17
7.13
7.11
7.11
3.57
2.34
1.26 Grease and n-hexane
0.91
0.89
0.87
0.86
0.86 Grease and n-hexane
0.85
0.84
0.82
A (m)
7.15
B (s)
3.57
C (s)
2.34
8.00
3.85
5.90
f1 (ppm)

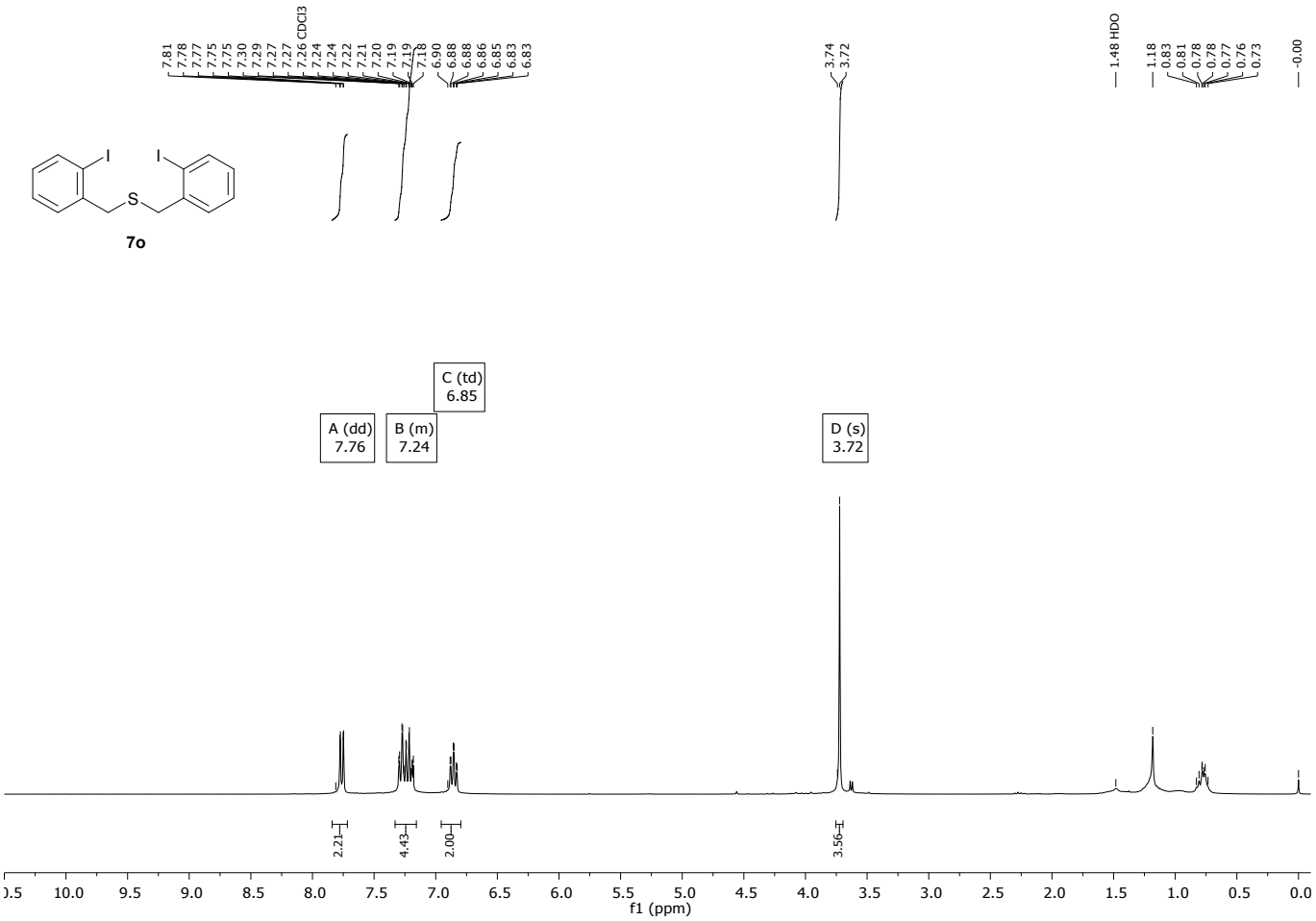

136.66
135.28
129.27
129.03
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
35.43
32.07 n-hexane
29.84 Grease
22.83 n-hexane
21.23
14.25 n-hexane
7e
f1 (ppm)

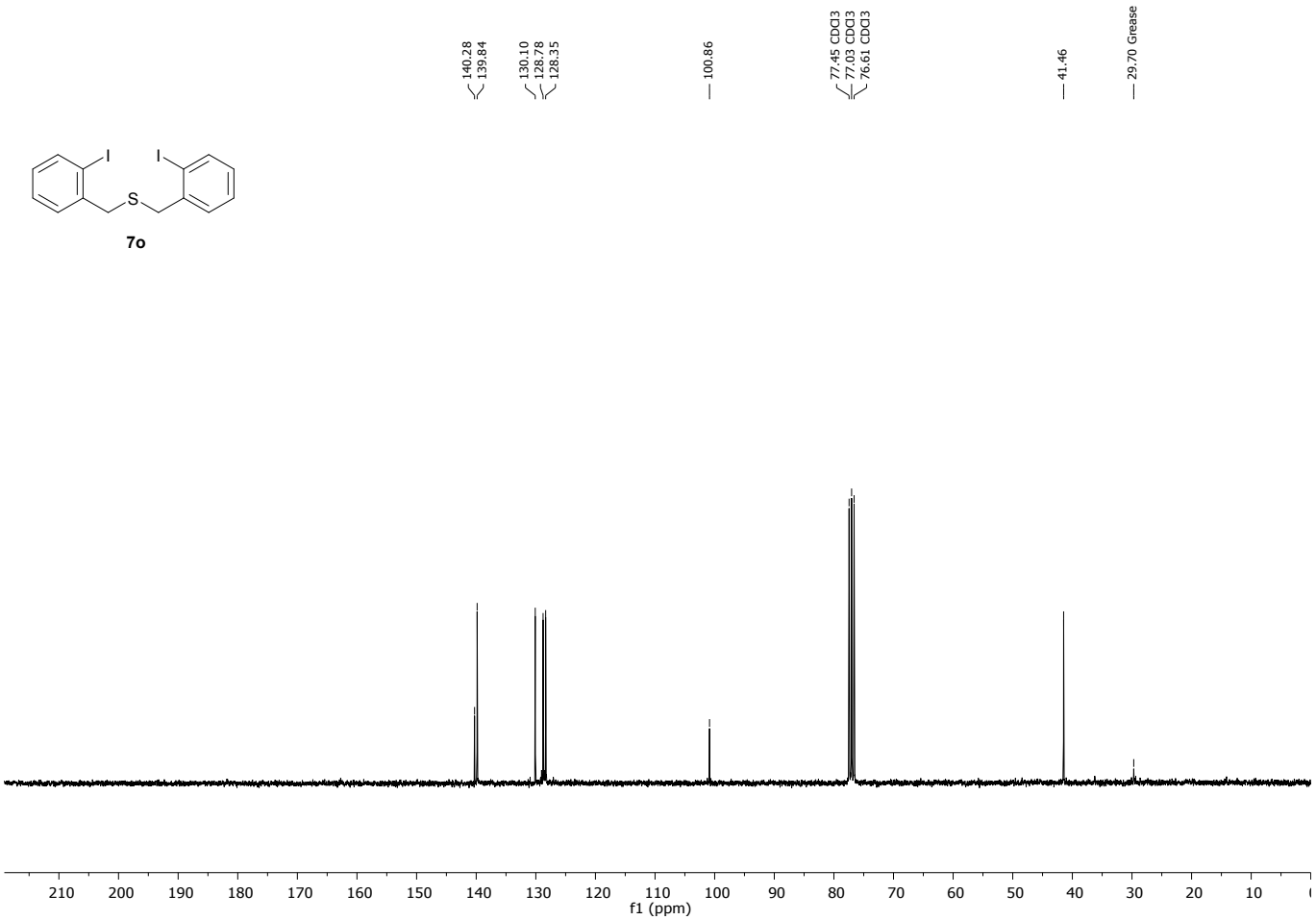

7.18
7.18
7.15
7.14
7.13
7.12
7.11
7.10 CDCl3
7.08
6.84
6.79
6.78
6.78
6.76
6.76
6.75
3.73
3.51
3.48
1.51 HDO
1.18 Grease and n-hexane
0.83
0.81
0.78
0.78 Grease and n-hexane
0.77
0.76
0.73
O
O
S
7i
B (m)
6.77
A (m)
7.12
D (s)
3.48
C (s)
3.72
3.89
3.99
6.20
3.82
f1 (ppm)

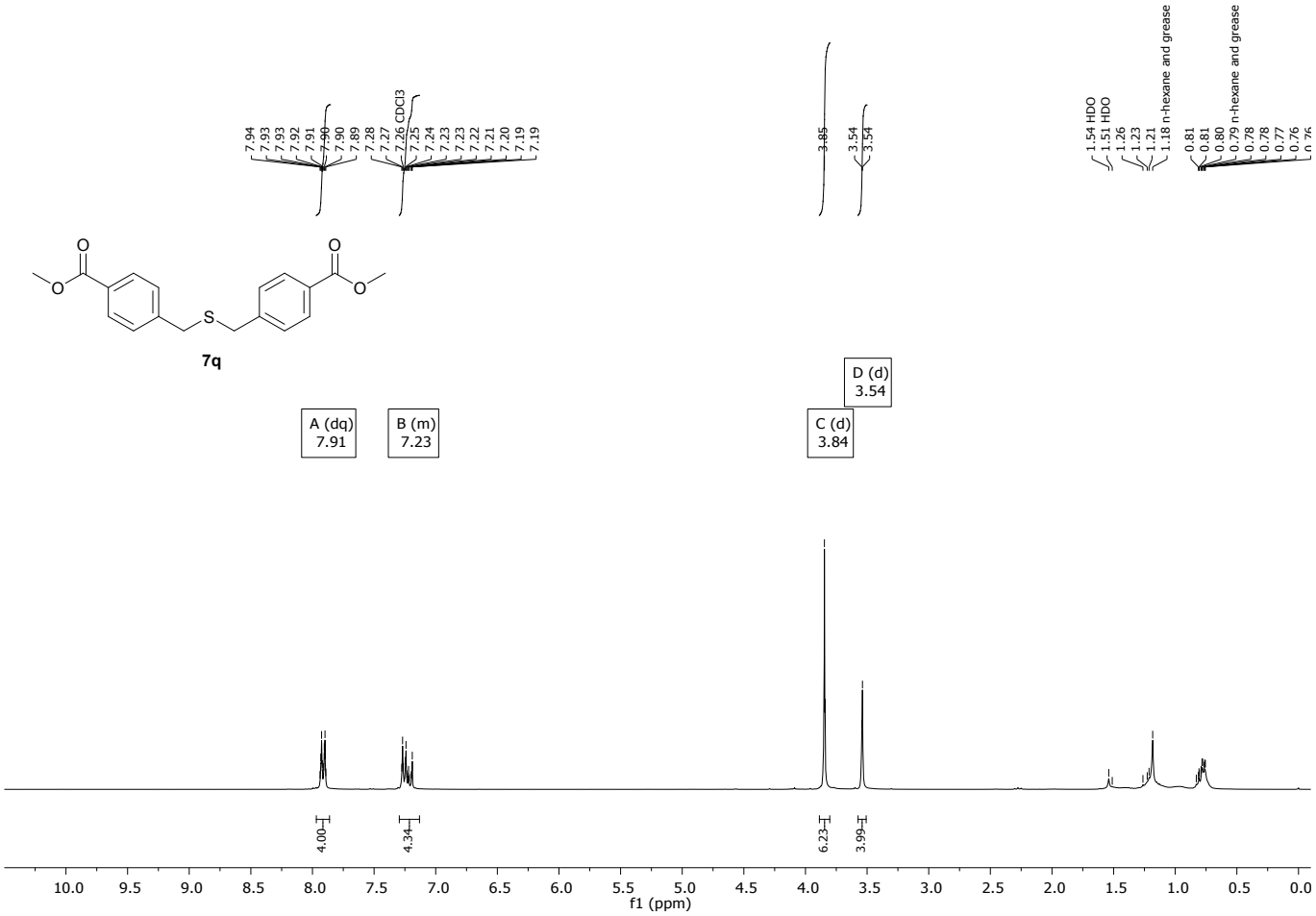

158.59
130.20
130.03
113.87
77.45 CDCl3
77.03 CDCl3
76.60 CDCl3
55.28
34.95
30.05 n-hexane
29.70 Grease
22.69 n-hexane
O
O
S
7i
f1 (ppm)

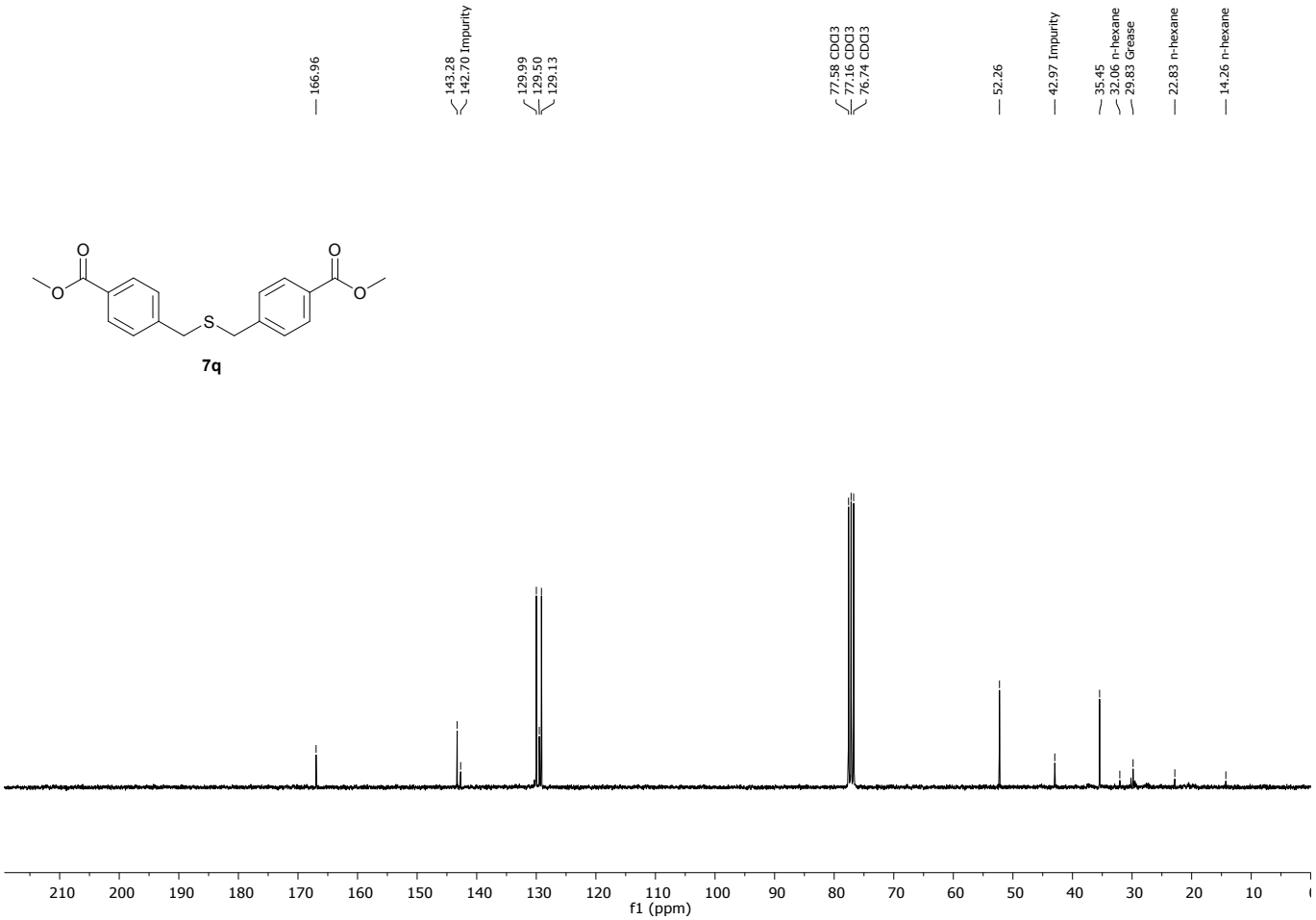

7.44
7.41
7.26
7.22
7.21
7.20
7.19
7.16
7.14
7.02
3.55
2.50
2.48
2.47
2.47
2.46
1.56 HDO
1.26 Grease
0.90
0.88
0.87
0.86 Grease
0.85
0.84
0.83
0.83
S
S
S
7j
A (m)
7.20
B (s)
3.55
C (s)
2.48
7.93
4.00
6.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

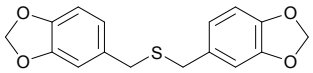

137.14
135.10
129.61
126.93
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
43.03 Impurity
35.24
29.83 Grease
16.12
S
S
S
7j
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.81
7.78
7.77
7.75
7.75
7.30
7.29
7.27
7.27
7.26 CDCl3
7.24
7.24
7.22
7.21
7.20
7.19
7.19
7.18
6.90
6.88
6.88
6.86
6.85
6.83
6.83
3.74
3.72
1.48 HDO
1.18
0.83
0.81
0.78
0.78
0.77
0.76
0.73
-0.00
I
I
S
7o
C (td)
6.85
A (dd)
7.76
B (m)
7.24
D (s)
3.72
2.21
4.43
2.00
3.56
f1 (ppm)

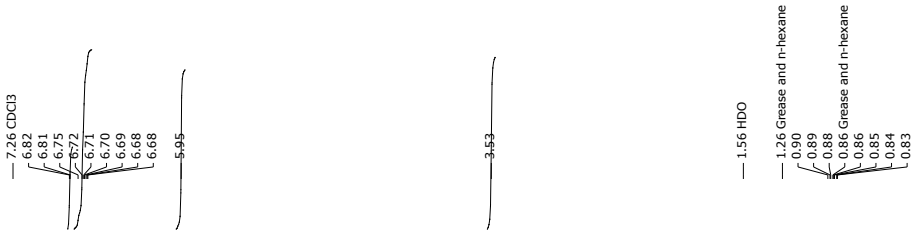

140.28
139.84
130.10
128.78
128.35
100.86
77.45 CDCl3
77.03 CDCl3
76.61 CDCl3
41.46
29.70 Grease
I
I
S
7o
f1 (ppm)

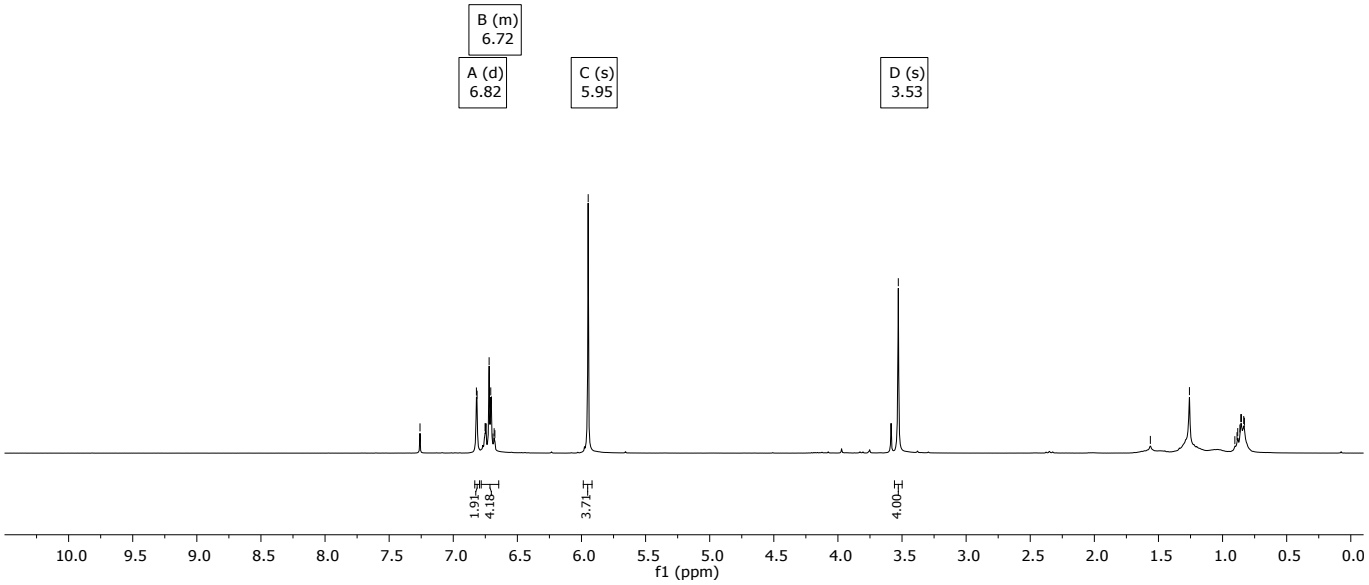

7.94
7.93
7.93
7.92
7.91
7.90
7.90
7.89
7.28
7.27
7.26 CDCl3
7.25
7.24
7.23
7.23
7.22
7.21
7.20
7.19
7.19
3.85
3.54
3.54
1.54 HDO
1.51 HDO
1.26
1.23
1.21
1.18 n-hexane and grease
0.81
0.81
0.80
0.79 n-hexane and grease
0.78
0.78
0.78
0.77
0.76
O
O
O
O
S
7q
A (dq)
7.91
B (m)
7.23
C (d)
3.84
D (d)
3.54
4.00
4.34
6.23
3.99
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)

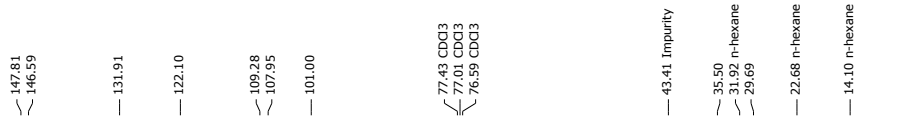

166.96
143.28
142.70 Impurity
129.99
129.50
129.13
77.58 CDCl3
77.16 CDCl3
76.74 CDCl3
52.26
42.97 Impurity
35.45
32.06 n-hexane
29.83 Grease
22.83 n-hexane
14.26 n-hexane
O
O
O
O
S
7q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

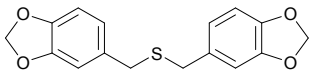


7r

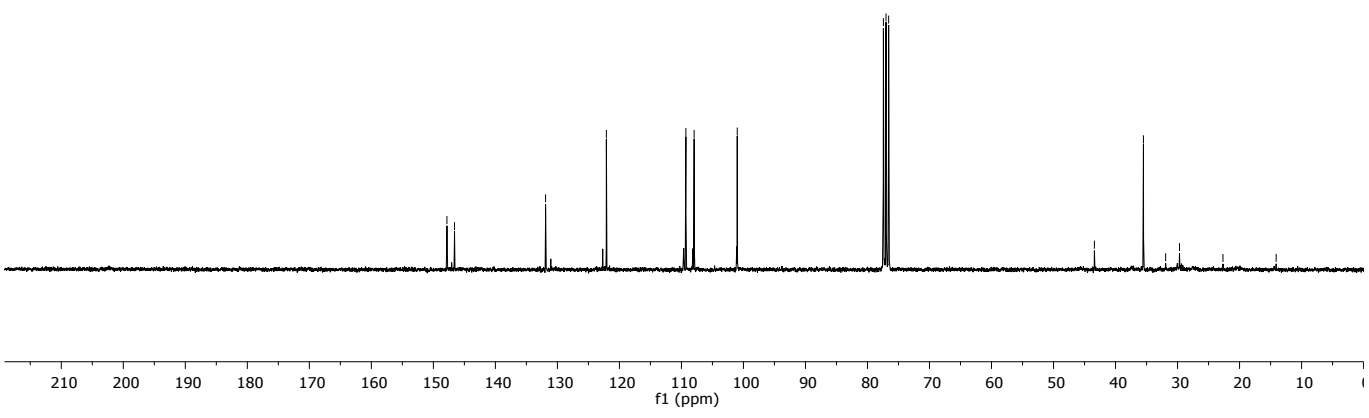

7.26 CDCl3
6.82
6.81
6.75
6.72
6.71
6.70
6.69
6.68
6.68
5.95
3.53
1.56 HDO
1.26 Grease and n-hexane
0.90
0.89
0.88
0.86 Grease and n-hexane
0.86
0.85
0.84
0.83


B (m)
6.72
A (d)
6.82
C (s)
5.95
D (s)
3.53
1.91
4.18
3.71
4.00
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
f1 (ppm)


147.81
146.59
131.91
122.10
109.28
107.95
101.00
77.43 CDCl3
77.01 CDCl3
76.59 CDCl3
43.41 Impurity
35.50
31.92 n-hexane
29.69
22.68 n-hexane
14.10 n-hexane



7r


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)