

Photocatalytic Iodosulfonylation of Internal Alkynes under Green Conditions

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General information

All photocatalyzed reactions were carried out under air atmosphere at 25 °C. Thin layer chromatography was performed using pre-coated plates obtained from Merck (TLC silica gel 60 F254). TLC plates were visualized with 254 nm ultraviolet light (UV). Silica gel chromatography purifications were performed by flash chromatography using EM Science silica gel 60 (230-400 mesh).

NMR spectra were obtained on a Bruker Avance III HD (400 MHz ^1H , 101 MHz ^{13}C , 376 MHz ^{19}F). The chemical shifts are frequency referenced relative to the residual undeuterated solvent peaks.¹ Coupling constants J are given in Hertz as positive values regardless of their real individual signs. The multiplicity of the signals is indicated as “s”, “d”, “t” or “m” for singlet, doublet, triplet or multiplet, respectively. The abbreviation “br” is given for broadened signals.

High Resolution Mass Spectrometry spectra were carried out using AB Sciex TripleTOF 5600+ equipped with a TurboV and PhotoSpray ion sources. FT-IR spectra were obtained in a Bruker “Alpha-T” FTIR (KBr).

Chemicals and solvents were obtained from commercial sources and used without further purification.

Light sources and photoreactors

LED strip lights with $\lambda_{\max}=400 \text{ nm}$ (violet, “Arlight RT-B60-10mm 12V UV400”, 14.4 W/m), 470nm (blue, “Arlight RT 2-5000 24V Blue 2x2”, 19.2 W/mm), 525 nm (green, “Arlight RT 2-5000 12V Green 2x”, 14.4 W/m), 590 nm (orange, “Arlight RT 2-5000 24V Yellow 2x”, 14.4 W/m), 625 nm (red, “Arlight RT 2-5000 12V Red 2x”, 14.4 W/m) were used. A piece of LED strip was mounted onto internal surface of aluminum cup (internal diameter 90 mm, height 79 mm) with ventilation holes (diameter 8 mm) at the bottom. All led strips were powered by regulated DC power supply (violet: 12V 1.21A; blue: 24V 0.735A; green: 12V 1.25A; orange: 24V 0.635A; red: 12V 1.31A).

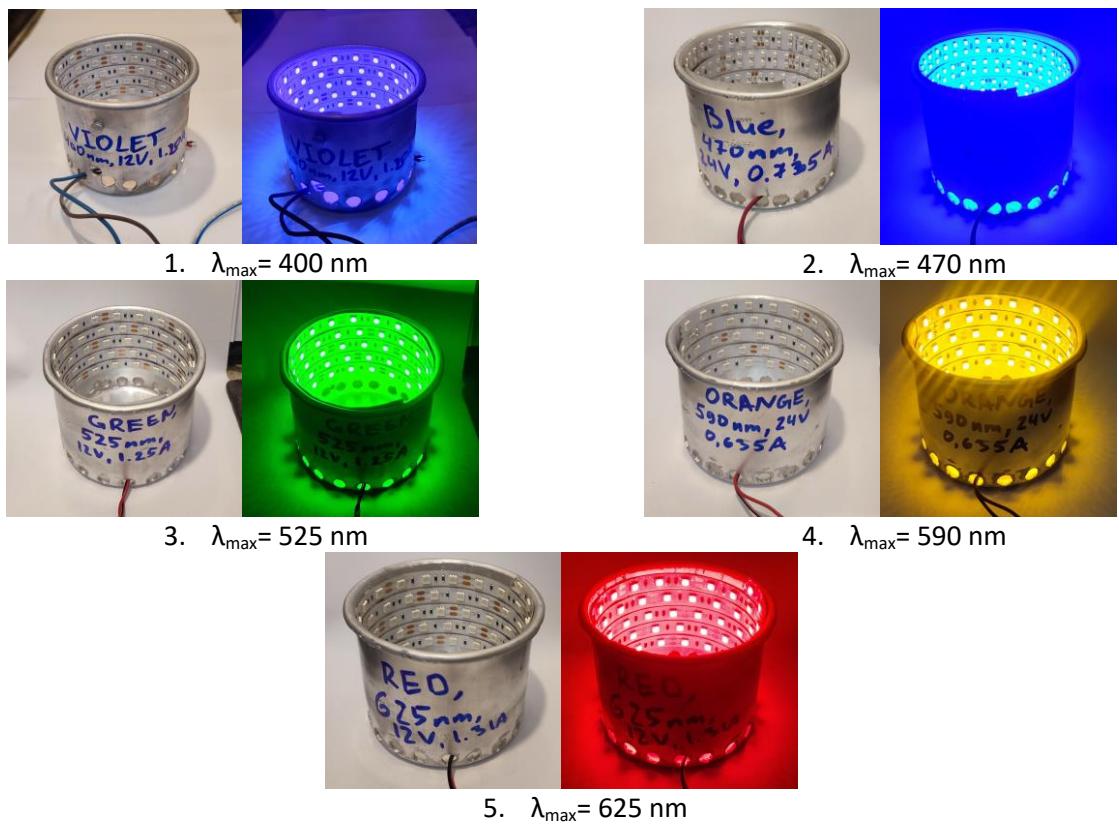


Figure S1. Photoreactors with different wavelength

The aluminum cup with LED light strip were placed in the PVC tube (internal diameter 150 mm, height 200 mm) with ventilation holes at the bottom (diameter 10 mm). Duct fan was placed on the top of PVC tube (air flow 280 m³/h) to provide sufficient cooling of reaction mixture.

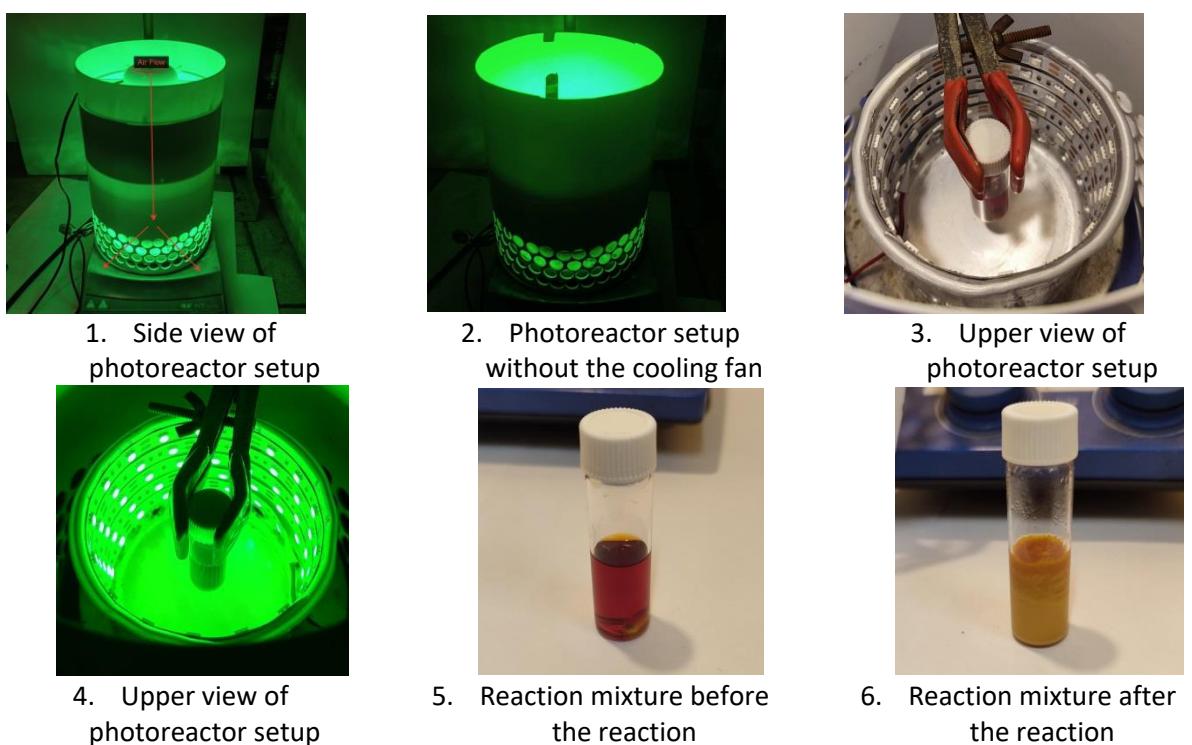


Figure S2. Reaction setup

Optimization of reaction conditions

Table S1. Optimization of reaction conditions

Entry	Solvent (0.125M)	TsI, eq.	cat (1 mol %)	Time, h	Yield TMB, %
1	EtOH, 525 nm (green)	1	BengalRose	1	99 (99^b)
2	EtOH, 525 nm (green)	1	Tetrachlorofluorescein	1	86
3	EtOH, 525 nm (green)	1	Eosyn	1	66
4	EtOH, 525 nm (green)	1	Uranine	1	85
5	EtOH, 525 nm (green)	1	Erythrosin B	1	99 (96 ^b)
6	EtOH, 525 nm (green)	1	Fluorescein	1	94

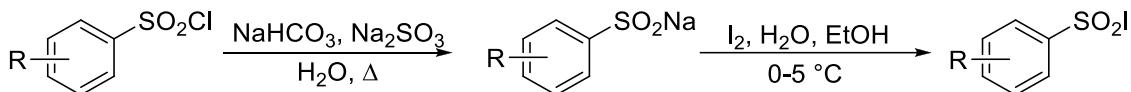
^a Reaction conditions: tolane (0.5 mmol, 1 eq.), TsI (0.5 mmol, 1 eq.), photocatalyst (1 mol%, 0.005 mmol), ethanol (4 ml, 0.125M). Yields were determined by ¹H NMR (1,3,5-trimethoxybenzene standard) ^b 0.1 mol% of photocatalyst.

Table S2. Optimization of light wavelength

Entry	Solvent (0.125M)	TsI, eq.	BengalRose (mol %)	Time, h	Yield TMB, %
1	EtOH, 400 nm (violet)	1	1	1	78
2	EtOH, 470 nm (blue)	1	1	1	89
3	EtOH, 525 nm (green)	1	1	1	99
4	EtOH, 590 nm (orange)	1	1	1	84
5	EtOH, 625 nm (red)	1	1	1	84
6	EtOH, 25°C, in the dark	1	1	1	0

Synthesis of starting materials

Starting materials such as ArSO₂I, 1,2-diarylacetyles, 1-aryl-2-alkyl-acetylens were synthesized according to our previous article². Method for synthesis of ArSO₂I was duplicated below.

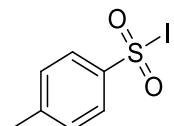


Scheme S1. Synthesis of ArSO₂I

General procedure for synthesis of ArSO₂I (GP-1)

A solution of I₂ (2.53 g, 10 mmol) in EtOH (20 ml) was added dropwise to a solution of sodium arylsulfinate (10.56 mmol) in water (40 ml) at 5 °C. A mixture was stirred at 5 °C during 0.5h. Then it was filtered and the residue was washed with cold water. The residue was dried under high vacuum in the dark (covered with aluminum foil) at room temperature for 12h to give product as a yellow or orange powder with 72-98% yield. It was found, that product was pure and it was used without further purification. The product was stored in the freezer (-25 °C) in the glass-stoppered round-bottomed flask. The flask was brought to room temperature in the dark (covered with aluminum foil) prior to stopper removal. It was found, that products were stable under this conditions up to several month.

4-Methylbenzene-1-sulfonyl iodide (2)



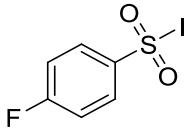
Following **GP-1**, 4-methylbenzene-1-sulfonyl iodide (13.5 g, 90%) was obtained on 53.3 mmol scale as a yellow powder. The NMR data are in agreement with previously reported.³

¹H NMR (400 MHz, CDCl₃) δ 7.75 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 7.8 Hz, 2H), 2.47 (s, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 147.7, 146.4, 129.8, 125.6, 22.0.

IR (ν/cm^{-1}): 3088 (W), 3052 (W), 2921 (VW), 1922 (W), 1647 (VW), 1588 (M), 1560 (VW), 1484 (M), 1457 (VW), 1448 (W), 1437 (VW), 1402 (W), 1379 (W), 1337 (VS), 1321 (M), 1301 (S), 1289 (M), 1209 (W), 1195 (W), 1179 (M), 1146 (VS), 1117 (M), 1069 (S), 1042 (M), 1012 (W), 969 (W), 842 (M), 807 (S), 738 (VW), 697 (M), 641 (S), 548 (VS), 514 (VS), 465 (S), 422 (W).

4-Fluorobenzene-1-sulfonyl iodide (2a)



Following **GP-1**, 4-fluorobenzene-1-sulfonyl iodide (2.8 g, 98%) was obtained as an orange powder.

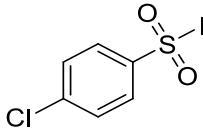
^1H NMR (400 MHz, CDCl_3) δ 8.18-7.63 (m, 2H), 7.26 (t, $J = 8.4$ Hz, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 166.02 (d, $J = 259.5$ Hz), 145.80 (d, $J = 2.9$ Hz), 128.65 (d, $J = 10.0$ Hz), 116.61 (d, $J = 23.2$ Hz).

^{19}F NMR (376 MHz, CDCl_3) δ -100.1(s, 1F, Ar-F).

IR (ν/cm^{-1}): 3099 (W), 3081 (W), 3062 (W), 1911 (VW), 1892 (VW), 1587 (S), 1488 (VS), 1406 (M), 1376 (W), 1357 (W), 1334 (S), 1305 (S), 1286 (S), 1239 (VS), 1155 (S), 1135 (VS), 1098 (M), 1071 (S), 1009 (M), 945 (W), 835 (S), 815 (M), 703 (M), 647 (S), 564 (VS), 558 (VS), 522 (M), 510 (VS), 490 (S), 441 (W).

4-Chlorobenzene-1-sulfonyl iodide (2b)



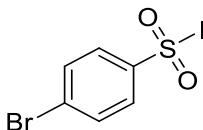
Following **GP-1**, 4-chlorobenzene-1-sulfonyl iodide (2.9 g, 95%) was obtained as an orange powder.

^1H NMR (400 MHz, CDCl_3) δ 7.86-7.74 (m, 2H), 7.59-7.48 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 148.0, 141.7, 129.6, 127.0.

IR (ν/cm^{-1}): 3086 (W), 3070 (W), 1915 (VW), 1647 (W), 1566 (M), 1523 (W), 1470 (S), 1393 (S), 1362 (W), 1337 (VS), 1321 (S), 1302 (M), 1284 (M), 1175 (M), 1163 (M), 1139 (VS), 1110 (M), 1091 (VS), 1069 (S), 1010 (S), 966 (W), 948 (W), 826 (S), 817 (M), 749 (VS), 697 (M), 581 (VS), 547 (VS), 480 (W), 455 (S), 414 (W).

4-Bromobenzene-1-sulfonyl iodide (2c)



Following **GP-1**, 4-bromobenzene-1-sulfonyl iodide (2.5 g, 72%) was obtained as a yellow powder.

¹H NMR (400 MHz, CDCl₃) δ 7.71 (s, 4H).

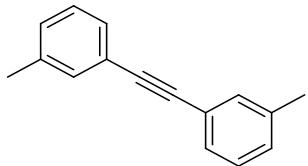
¹³C NMR (101 MHz, CDCl₃) δ 148.4, 132.6, 130.4, 126.9.

IR (ν/cm⁻¹): 3083 (M), 3063 (W), 1921 (W), 1800 (VW), 1636 (W), 1563 (S), 1473 (W), 1463 (S), 1387 (S), 1364 (W), 1339 (VS), 1323 (M), 1289 (W), 1276 (M), 1175 (M), 1155 (VS), 1125 (S), 1109 (M), 1087 (W), 1064 (VS), 1007 (S), 968 (W), 957 (W), 839 (S), 817 (S), 733 (VS), 694 (M), 620 (VW), 571 (VS), 542 (VS), 467 (M), 415 (M), 406 (M).

Synthesis of symmetrical 1,2-diarylacetylenes

Symmetrical 1,2-diarylalkynes were synthesized according to our previous article²

*1,2-Bis(*m*-tolyl)ethyne (**1b**)*

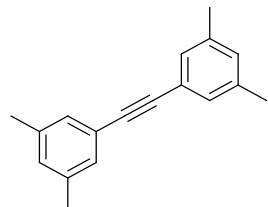


Following literature procedure², 1,2-bis(*m*-tolyl)ethyne (1.01 g, 98%) was obtained from CaC₂ and 1-iodo-3-methylbenzeneas as a white powder. The NMR data are in agreement with previously reported⁴.

¹H NMR (400 MHz, CDCl₃) δ 7.41-7.31 (m, 4H), 7.25 (t, *J* = 7.6 Hz, 2H), 7.20-7.11 (m, 2H), 2.37 (s, 6H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 138.1, 132.3, 129.2, 128.8, 128.4, 123.3, 89.4, 21.4.

*1,2-Bis(3,5-dimethylphenyl)ethyne (**1c**)*

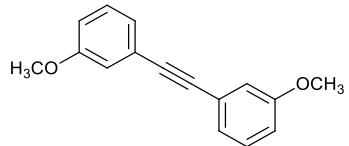


Following literature procedure², 1,2-bis(3,5-dimethylphenyl)ethyne (1.35 g, 77%) was obtained from CaC₂ and 1-iodo-3,5-dimethylbenzene as a white powder. The NMR data are in agreement with previously reported⁴.

¹H NMR (400 MHz, CDCl₃) δ 7.18 (s, 2H), 6.98 (s, 1H), 2.33 (s, 6H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 138.0, 130.2, 129.4, 123.2, 89.2, 21.3.

*1,2-Bis(3-methoxyphenyl)ethyne (**1d**)*

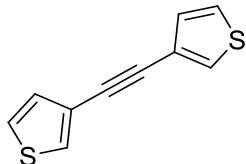


Following literature procedure², 1,2-bis(3-methoxyphenyl)ethyne (1.45 g, 40%) was obtained from CaC₂ and 1-iodo-3-methoxybenzene as a white powder. The NMR data are in agreement with previously reported⁴.

¹H NMR (400 MHz, CDCl₃) δ 7.26 (t, J = 7.9 Hz, 2H), 7.18-7.11 (m, 2H), 7.10-7.04 (m, 2H), 6.94-6.86 (m, 2H), 3.83 (s, 6H, -OCH₃).

¹³C NMR (101 MHz, CDCl₃) δ 159.5, 129.5, 124.3, 124.3, 116.5, 115.1, 89.3, 55.4.

*1,2-Di(thiophen-3-yl)ethyne (**1e**)*

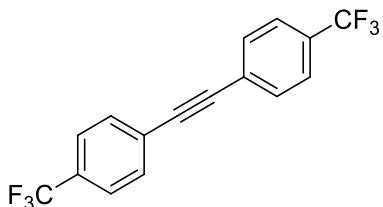


Following literature procedure², 1,2-di(thiophen-3-yl)ethyne (0.83 g, 87%) was obtained from CaC₂ and 3-iodothiopheneas as a white powder. The NMR data are in agreement with previously reported⁵.

¹H NMR (400 MHz, CDCl₃) δ 7.51 (dd, J = 3.0, 1.2 Hz, 2H), 7.33-7.27 (m, 2H), 7.19 (dd, J = 5.0, 1.2 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 129.9, 128.6, 125.5, 122.3, 84.1.

*1,2-Bis(4-(trifluoromethyl)phenyl)ethyne (**1f**)*



Following literature procedure², 1,2-bis(4-(trifluoromethyl)phenyl)ethyne (1.48 g, 94%) was obtained from CaC₂ and 1-iodo-4-(trifluoromethyl)benzene as a white powder. The NMR data are in agreement with previously reported⁶.

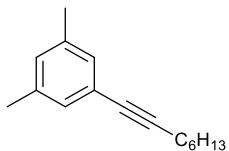
¹H NMR (400 MHz, CDCl₃) δ 7.75-7.56 (m, 8H).

¹³C NMR (101 MHz, CDCl₃) δ 132.1, 130.65 (q, J = 32.7 Hz), 126.5, 125.54 (q, J = 3.8 Hz), 123.99 (q, J = 272.2 Hz), 90.3.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.9 (s, 6F, -CF₃).

Synthesis of other acetylenes

1,3-Dimethyl-5-(oct-1-yn-1-yl)benzene (**Ii**)



Following literature procedure⁷, 1,3-dimethyl-5-(oct-1-yn-1-yl)benzene (1.22 g, 57%) was obtained from 1-iodo-3,5-dimethylbenzene and oct-1-yne as a colorless oil.

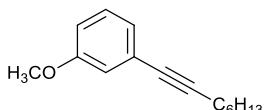
¹H NMR (400 MHz, CDCl₃) δ 7.04 (s, 2H), 6.91 (s, 1H), 2.39 (t, J = 7.1 Hz, 2H, -CH₂-), 2.28 (s, 6H, -CH₂-), 1.67-1.54 (m, 2H, -CH₂-), 1.52-1.41 (m, 2H, -CH₂-), 1.39-1.27 (m, 4H, -CH₂-), 0.92 (t, J = 6.8 Hz, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 137.8, 129.5, 129.4, 123.8, 89.8, 80.9, 31.5, 29.0, 28.8, 22.7, 21.2, 19.6, 14.2.

IR (ν/cm⁻¹): 2955 (W), 2928 (M), 2858 (W), 1737 (VW), 1599 (M), 1509 (VW), 1466 (M), 1458 (W), 1430 (W), 1377 (W), 1352 (VW), 1333 (W), 1304 (VW), 1038 (W), 894 (W), 847 (VS), 749 (VW), 724 (W), 689 (VS).

ESI-HRMS (m/z): calc. for (C₁₆H₂₃) [M+H]⁺ 215.1794; found: 215.1777.

1-Methoxy-3-(oct-1-yn-1-yl)benzene (**Ij**)



Following literature procedure⁷, 1-methoxy-3-(oct-1-yn-1-yl)benzene (2.1 g, 97%) was obtained from 1-iodo-3-methoxybenzene and oct-1-yne as a yellow oil. The NMR data are in agreement with previously reported².

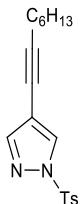
¹H NMR (400 MHz, CDCl₃) δ 7.19 (t, J = 8.0 Hz, 1H), 7.03-6.97 (m, 1H), 6.97-6.91 (m, 1H), 6.87-6.78 (m, 1H), 3.79 (s, 3H, -OCH₃), 2.40 (t, J = 7.1 Hz, 2H, -CH₂-), 1.70-1.52 (m, 2H, -CH₂-), 1.52-1.41 (m, 2H, -CH₂-), 1.41-1.23 (m, 4H, -CH₂-), 0.91 (t, J = 6.7 Hz, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 159.41, 129.35, 125.26, 124.24, 116.53, 114.20, 90.51, 80.60, 55.34, 31.51, 28.85, 28.76, 22.70, 19.54, 14.20.

IR (ν/cm⁻¹): 3000 (VW), 2954 (W), 2930 (M), 2857 (W), 2835 (W), 1604 (M), 1598 (M), 1583 (M), 1574 (M), 1489 (M), 1480 (M), 1464 (M), 1427 (M), 1378 (VW), 1316 (M), 1286 (S), 1268 (M), 1224 (VW), 1206 (S), 1200 (S), 1174 (M), 1164 (S), 1111 (VW), 1082 (VW), 1044 (S), 1014 (W), 993 (W), 942 (VW), 873 (W), 853 (M), 812 (W), 776 (S), 724 (W), 686 (VS).

ESI-HRMS (m/z): calc. for ($C_{15}H_{21}O^+$) $[M+H]^+$ 217.1587; found: 217.1574.

4-(Oct-1-yn-1-yl)-1-tosyl-1H-pyrazole (1p)



Following literature procedure⁷, 4-(oct-1-yn-1-yl)-1-tosyl-1H-pyrazole (1.49 g, 75%) was obtained from 4-iodo-1-tosyl-1H-pyrazole and oct-1-yne as a yellow oil. The NMR data are in agreement with previously reported⁸.

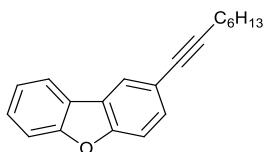
¹H NMR (400 MHz, CDCl₃) δ 8.09 (s, 1H), 7.87 (d, J = 8.0 Hz, 2H), 7.67 (s, 1H), 7.33 (d, J = 8.1 Hz, 2H), 2.42 (s, 3H, -CH₃), 2.34 (t, J = 7.1 Hz, 2H, -CH₂-), 1.58-1.51 (m, 2H, -CH₂-), 1.44-1.37 (m, 2H, -CH₂-), 1.34-1.24 (m, 4H), 0.91-0.87 (m, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 147.2, 146.3, 133.8, 132.6, 130.2, 128.4, 107.7, 93.8, 69.4, 31.5, 28.7, 28.6, 22.7, 21.9, 19.5, 14.2.

IR (v/cm⁻¹): 2954 (VW), 2928 (W), 2857 (VW), 1596 (VW), 1455 (VW), 1384 (M), 1343 (VW), 1321 (W), 1191 (M), 1172 (M), 1120 (VW), 1093 (M), 1054 (VW), 1028 (W), 1018 (W), 951 (W), 871 (VW), 811 (W), 702 (W), 670 (VS), 654 (M).

ESI-HRMS (m/z): calc. for ($C_{18}H_{23}N_2O_2S^+$) $[M+H]^+$ 331.1475; found: 331.1481.

2-(Oct-1-yn-1-yl)dibenzo[b,d]furan (1r)



Following literature procedure⁷, 2-(oct-1-yn-1-yl)dibenzo[b,d]furan (2.5 g, 92%) was obtained from 2-bromodibenzo[b,d]furan and oct-1-yne as a yellow oil.

¹H NMR (400 MHz, CDCl₃) δ 8.00 (s, 1H), 7.92 (d, J = 7.8 Hz, 1H), 7.56 (d, J = 8.2 Hz, 1H), 7.53-7.42 (m, 3H), 7.35 (t, J = 7.5 Hz, 1H), 2.45 (t, J = 7.1 Hz, 2H, -CH₂-), 1.65 (p, J = 7.2 Hz, 2H, -CH₂-), 1.54-1.44 (m, 2H, -CH₂-), 1.41-1.31 (m, 4H, -CH₂-), 0.98-0.89 (m, 3H, -CH₃).

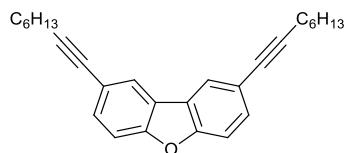
¹³C NMR (101 MHz, CDCl₃) δ 156.7, 155.5, 130.9, 127.6, 124.4, 124.0, 123.9, 123.0, 120.9, 118.8, 111.9, 111.7, 89.6, 80.6, 31.6, 29.0, 28.8, 22.7, 19.6, 14.2.

IR (v/cm⁻¹): 2954 (W), 2928 (W), 2856 (W), 1591 (VW), 1475 (M), 1449 (M), 1427 (W), 1377 (VW), 1338 (VW), 1329 (VW), 1311 (VW), 1279 (W), 1246 (W), 1230 (W), 1194 (S), 1157 (W), 1149 (W), 1120 (W), 1104 (W), 1020 (W), 1007 (VW), 969 (VW), 930 (VW), 882

(W), 864 (W), 840 (M), 813 (M), 765 (W), 746 (VS), 726 (M), 702 (W), 692 (W), 631 (W), 613 (M).

ESI-HRMS (m/z): calc. for $(C_{20}H_{21}O^+)[M+H]^+$ 277.1587; found: 277.1563.

2,8-Di(oct-1-yn-1-yl)dibenzo[b,d]furan (1s)



Following literature procedure⁷, 2,8-di(oct-1-yn-1-yl)dibenzo[b,d]furan (1.9 g, 99%) was obtained from 2,8-diiododibenzo[b,d]furan and oct-1-yne as a yellow oil.

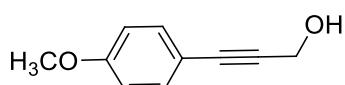
¹H NMR (400 MHz, CDCl₃) δ 7.95 (s, 2H), 7.56-7.39 (m, 4H), 2.44 (t, J = 7.1 Hz, 4H, -CH₂-), 1.75-1.58 (m, 4H, -CH₂-), 1.56-1.44 (m, 4H, -CH₂-), 1.42-1.30 (m, 8H, -CH₂-), 0.98-0.87 (m, 6H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 155.8, 131.2, 124.1, 124.0, 119.1, 111.8, 89.8, 80.5, 31.6, 28.9, 28.8, 22.7, 19.6, 14.2.

IR (ν/cm⁻¹): 2954 (W), 2928 (M), 2857 (W), 1481 (M), 1455 (M), 1430 (W), 1378 (VW), 1350 (VW), 1330 (VW), 1292 (VW), 1271 (W), 1247 (M), 1223 (VW), 1193 (VS), 1119 (W), 1023 (W), 877 (M), 842 (W), 813 (S), 731 (W), 703 (W), 685 (W), 648 (W).

ESI-HRMS (m/z): calc. for $(C_{28}H_{33}O^+)[M+H]^+$ 385.2526; found: 385.2505.

3-(4-Methoxyphenyl)prop-2-yn-1-ol (1v)

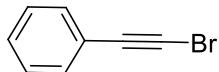


Following literature procedure⁷, 3-(4-methoxyphenyl)prop-2-yn-1-ol (715 mg, 44%) was obtained as a brown solid. The NMR data are in agreement with previously reported⁹.

¹H NMR (400 MHz, CDCl₃) δ 7.42-7.31 (m, 2H), 6.89-6.78 (m, 2H), 4.48 (s, 2H, -CH₂-OH), 3.81 (s, 3H, -OCH₃), 1.66 (s, 1H, -OH).

¹³C NMR (101 MHz, CDCl₃) δ 159.9, 133.3, 114.7, 114.1, 86.0, 85.8, 55.4, 51.9.

*(Bromoethynyl)benzene (**I'b**)*

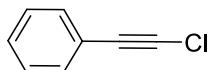


Following literature procedure¹⁰, (bromoethynyl)benzene (24 g, 94%) was obtained from ethynylbenzene and NBS (AgNO₃ was used as catalyst) as a colorless oil. The NMR data are in agreement with previously reported¹⁰.

¹H NMR (400 MHz, CDCl₃) δ 7.50-7.42 (m, 2H), 7.38-7.28 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 132.1, 128.8, 128.5, 122.8, 80.2, 49.9.

*(Chloroethynyl)benzene (**I'c**)*

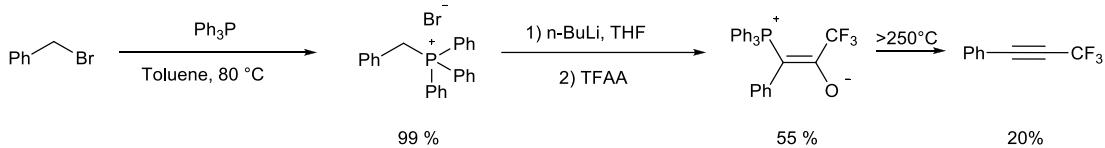


Following literature procedure¹¹, (chloroethynyl)benzene (960 mg, 58%) was obtained from ethynylbenzene and NCS (AgNO₃ was used as catalyst) as a pale yellow oil. The NMR data are in agreement with previously reported¹².

¹H NMR (400 MHz, CDCl₃) δ 7.48-7.42 (m, 2H), 7.39-7.27 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 132.1, 128.7, 128.5, 122.3, 69.5, 68.2.

*(3,3,3-Trifluoroprop-1-ynyl)benzene (**I'd**)*



Following modified literature procedure¹³ which is described below, (3,3,3-trifluoroprop-1-ynyl)benzene (394 mg, 20%) was obtained as a colorless oil.

Step 1. A mixture of the benzyl bromide (200 mmol, 34.20 g, 23.8 ml) and Ph₃P (200 mmol, 52.46 g) in a toluene (100 ml) was heated at 80 °C overnight. Then the reaction mixture was filtered and the product was washed with toluene (2x20 ml). The obtained benzyltriphenylphosphonium bromide (yield 99%) was pure enough for the next step.

¹H NMR (400 MHz, CDCl₃) δ 7.78-7.63 (m, 9H), 7.63-7.52 (m, 6H), 7.22-7.13 (m, 1H), 7.13-7.00 (m, 4H), 5.29 (d, J = 14.3 Hz, 2H).

Step 2. The solution of *n*-BuLi (2.5 M, 10 ml, 25 mmol) was added to the benzyltriphenylphosphonium bromide (20.8 mmol, 9.03 g) in THF (50 ml) at 20 °C. After stirring at 20 °C during 1h TFAA (20.83 mmol, 4.38 g) was added to the reaction mixture at the same temperature. The reaction mixture was stirred overnight, and then 1 ml of MeOH was added. Evaporation the solvent and next column chromatography using the mixture CH₂Cl₂ :

MeOH = 50:1 gave the α -(trifluoroacetyl)benzylidenetriphenylphosphorane (55%, 5.21 g) as a white powder.

^1H NMR (400 MHz, CDCl_3) δ 7.59-7.47 (m, 9H), 7.47-7.36 (m, 6H), 7.10-6.94 (m, 5H).

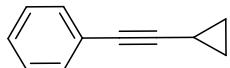
Step 3. α -(Trifluoroacetyl)benzylidenetriphenylphosphorane (11.6 mmol, 5.21 g) was heated with a heat gun at 450 °C under vacuum during 2h. The (3,3,3-trifluoroprop-1-ynyl)benzene was collected into a cooled to -78 °C trap. The collected liquid was dissolved in pentane, washed twice with NaHCO_3 (5%) and one time washed with water. The organic phase was separated, dried under Na_2SO_4 and filtered through the plug of SiO_2 using pentane as an eluent. Evaporation of the solvent gave the desire product (394 mg, 20%) as a colorless oil. The NMR data are in agreement with previously reported¹⁴.

^1H NMR (400 MHz, CDCl_3) δ 7.63-7.53 (m, 2H), 7.53-7.44 (m, 1H), 7.44-7.35 (m, 2H).

^{13}C NMR (101 MHz, CDCl_3) δ 132.58 (q, J = 1.6 Hz), 131.0, 128.8, 118.66 (q, J = 1.9 Hz), 114.99 (q, J = 256.8 Hz, - CF_3), 86.67 (q, J = 6.4 Hz, Ar-C-C- CF_3), 75.84 (q, J = 52.5 Hz, -C- CF_3).

^{19}F NMR (376 MHz, CDCl_3) δ -49.8 (s, 3F, - CF_3).

(Cyclopropylethynyl)benzene (**I'e**)



Following literature procedure¹⁵, (cyclopropylethynyl)benzene (350 mg, 30%) was obtained from (5-chloropent-1-ynyl)benzene as a colorless oil. The NMR data are in agreement with previously reported¹⁶.

^1H NMR (400 MHz, CDCl_3) δ 7.44-7.34 (m, 2H), 7.31-7.22 (m, 3H), 1.51-1.40 (m, 1H, -CH-), 0.92-0.77 (m, 4H, - CH_2 -).

^{13}C NMR (101 MHz, CDCl_3) δ 131.7, 128.3, 127.6, 124.0, 93.5, 77.2, 75.9, 8.7, 0.3.

Synthesis and further functionalization of β -iodovinyl sulfones

General procedure 2 (GP-2): iodosulfonylation of disubstituted acetylenes

A screw capped 8 ml vial equipped with a magnetic stirring bar was charged with disubstituted alkyne (0.5 mmol), EtOH (4 ml), Rose bengal (1 mol%, 0.005 mmol, 4.87 mg) followed by sulfonyl iodide (0.5 mmol). The reaction mixture was stirred under the green light irradiation (wavelength 525 nm) during 1h. The temperature of the reaction mixtures in the vials was measured during and after the completion of the reaction, and it did not exceed 27 °C. The

reaction mixture was then diluted with water (36 ml) and filtered to provide a crude product. The pure product was obtained after washing the residue with the mixture of EtOH / H₂O = 1/3 as a white, yellow, pink solid.

Notice: all operations were carried out under air atmosphere.

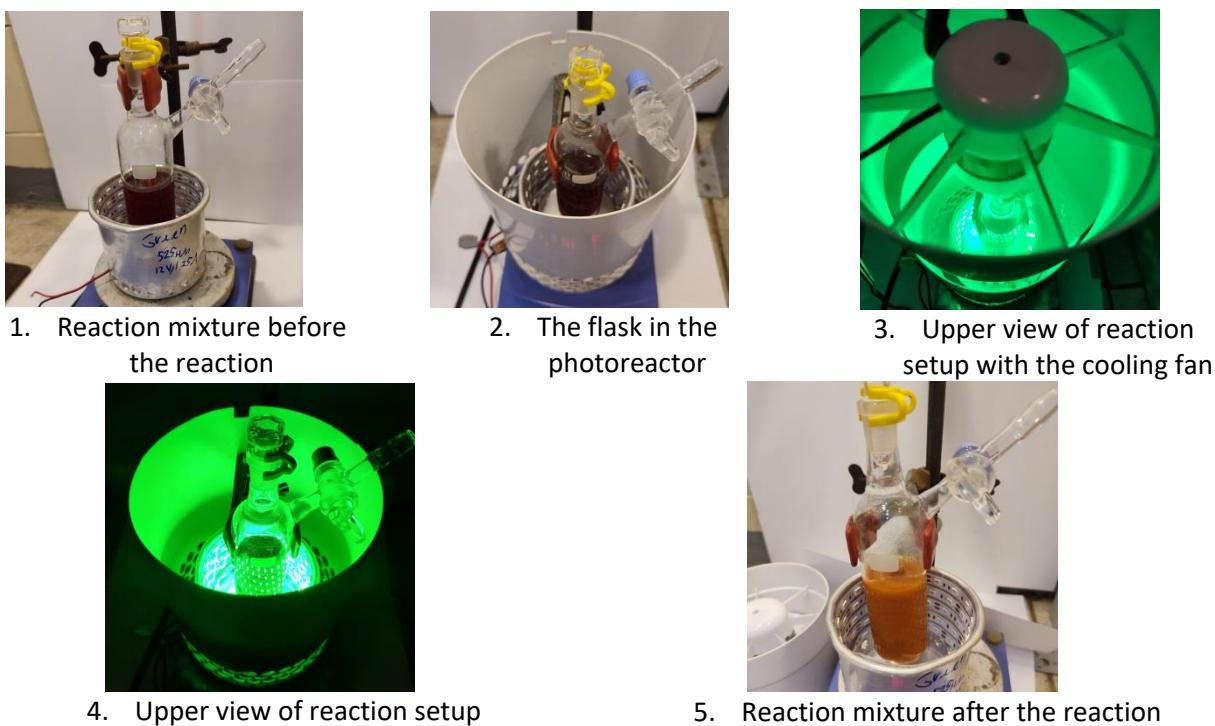


Figure S3. Reaction setup for 10-mmol scale synthesis

Procedure for 10-mmol scale synthesis of 3f:

A 100 ml Schlenk flask equipped with a magnetic stir bar was charged with but-1-ynylbenzene (10 mmol, 1.3 g), EtOH (80 ml), Rose bengal (1 mol%, 0.1 mmol, 97.4 mg) followed by sulfonyl iodide (10 mmol, 2.82 g). The solution was placed into photoreactor and stirred for 1.5h under green light irradiation. The reaction mixture was then diluted with water (720 ml) and filtered to provide a crude product. The pure product was obtained after washing the residue with the mixture of EtOH / H₂O = 1/3 as a pale pink powder with 85% (3.5 g) yield.

Notice: all operations were carried out under air atmosphere.

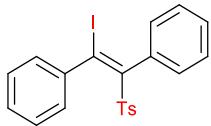
Procedure for 10-mmol scale synthesis of 3a:

A 100 ml Schlenk flask equipped with a magnetic stir bar was charged with diphenylacetylene (10 mmol, 1.78 g), EtOH (80 ml), Rose bengal (1 mol%, 0.1 mmol, 97.4 mg) followed by sulfonyl iodide (10 mmol, 2.82 g). The solution was placed into photoreactor and stirred for 1.5h under green light irradiation. The reaction mixture was then diluted with water (720 ml) and filtered to provide a crude product. The pure product was obtained after washing the residue with the mixture of EtOH / H₂O = 1/3 as a white powder with 93% (4.31 g) yield.

Notice: all operations were carried out under air atmosphere.

Characterization of Products

(*E*)-(1-*ido*-2-*tosylethene*-1,2-diyi)dibenzene (**3a**)

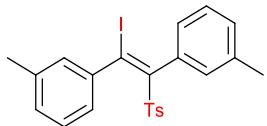


Following **GP-2**, (*E*)-(1-*ido*-2-*tosylethene*-1,2-diyi)dibenzene (213 mg, 93%) was obtained as a white powder, m.p. (EtOH) 165-167 °C. The NMR data are in agreement with previously reported.¹⁷

¹H NMR (400 MHz, CDCl₃) δ 7.46-7.30 (m, 8H, Ph-H), 7.26 (d, *J* = 8.2 Hz, 2H, Ts-H), 7.24-7.15 (m, 2H, Ph-H), 7.10 (d, *J* = 8.2 Hz, 2H, Ts-H), 2.37 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 149.2, 144.4, 142.6, 139.5, 136.9, 130.4, 129.3, 129.1, 128.7, 128.4, 128.0, 127.5, 118.2, 21.7.

(*E*)-3,3'-(1-*ido*-2-*tosylethene*-1,2-diyi)bis(methylbenzene) (**3b**)

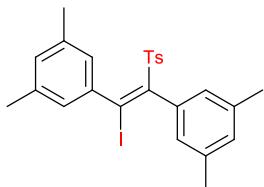


Following **GP-2**, (*E*)-3,3'-(1-*ido*-2-*tosylethene*-1,2-diyi)bis(methylbenzene) (217 mg, 89%) was obtained as a white powder, m.p. (EtOH) 197-198 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.33-7.18 (m, 6H), 7.18-7.07 (m, 4H), 7.07-7.05 (s, 1H), 7.02-6.98 (m, 2H), 2.41 (s, 3H, -CH₃), 2.39 (s, 3H, -CH₃), 2.37 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 149.5, 144.1, 142.5, 139.4, 138.2, 137.6, 137.3, 130.9, 130.1, 129.9, 129.2, 128.7, 128.3, 128.1, 127.9, 127.4, 124.8, 118.1, 21.7, 21.55, 21.51.

(*E*)-5,5'-(1-*ido*-2-*tosylethene*-1,2-diyi)bis(1,3-dimethylbenzene) (**3c**)



Following **GP-2**, (*E*)-5,5'-(1-*ido*-2-*tosylethene*-1,2-diyi)bis(1,3-dimethylbenzene) (183 mg, 71%) was obtained as a white powder, m.p. (EtOH) 204-205 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.32-7.22 (m, 2H), 7.15-7.06 (m, 2H), 7.03 (s, 1H), 6.96-6.77 (m, 5H), 2.39 (s, 3H, CH₃), 2.33 (s, 6H, -CH₃), 2.29 (s, 6H, -CH₃).

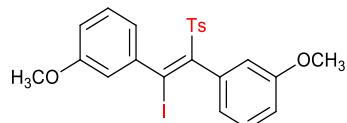
¹³C NMR (101 MHz, CDCl₃) δ 149.7, 143.9, 142.3, 139.3, 138.0, 137.6, 137.4, 130.9, 130.8, 129.0, 128.6, 128.0, 125.4, 118.0, 21.7, 21.4, 21.4.

IR (ν/cm^{-1}): 2949 (VW), 2914 (VW), 1622 (VW), 1596 (W), 1493 (VW), 1453 (W), 1404 (VW), 1377 (VW), 1324 (M), 1317 (M), 1302 (M), 1266 (VW), 1180 (W), 1154 (S), 1145 (VS), 1116 (W), 1086 (M), 1036 (W), 996 (W), 969 (W), 947 (VW), 908 (VW), 853 (M), 808 (M), 784 (M), 731 (VW), 722 (VW), 704 (VS), 696 (S), 682 (S), 667 (M), 635 (W), 622 (W).

ESI-HRMS (m/z): calc. for $(\text{C}_{25}\text{H}_{26}\text{IO}_2\text{S})^+ [\text{M}+\text{H}]^+$ 517.0693; found: 517.0690.

Elemental analysis (%) calc. for $\text{C}_{25}\text{H}_{25}\text{IO}_2\text{S}$: C, 58.14; H, 4.88; I, 24.57; O, 6.20; S, 6.21; found: C, 56.16; H, 4.89.

(E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methoxybenzene) (3d)



Following **GP-2**, *(E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methoxybenzene)* (255 mg, 98%) was obtained as a white powder, m.p. (EtOH) 127-130 °C.

^1H NMR (400 MHz, CDCl_3) δ 7.33-7.18 (m, 5H), 7.16-7.00 (m, 2H), 7.00-6.86 (m, 2H), 6.85-6.79 (m, 1H), 6.79-6.71 (m, 2H), 6.69 (s, 1H), 3.86-3.68 (m, 6H, -OCH₃), 2.34 (s, 3H, -CH₃).

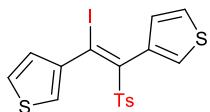
^{13}C NMR (101 MHz, CDCl_3) 159.4, 158.9, 149.4, 144.3, 143.6, 140.5, 137.1, 129.6, 129.3, 129.0, 128.7, 122.7, 120.1, 117.3, 115.5, 115.4, 115.1, 112.8, 55.5, 55.4, 21.7.

IR (ν/cm^{-1}): 1621 (VW), 1591 (M), 1483 (M), 1471 (W), 1457 (W), 1426 (W), 1326 (M), 1304 (W), 1285 (M), 1253 (S), 1202 (W), 1176 (W), 1145 (VS), 1117 (W), 1086 (M), 1038 (S), 1018 (W), 992 (W), 974 (M), 945 (W), 856 (W), 849 (W), 823 (W), 810 (M), 799 (S), 744 (M), 725 (W), 703 (M), 690 (VS), 667 (M), 657 (W), 634 (VW), 626 (W), 611 (M).

ESI-HRMS (m/z): calc. for $(\text{C}_{23}\text{H}_{22}\text{IO}_4\text{S})^+ [\text{M}+\text{H}]^+$ 521.0278; found: 521.0274.

Elemental analysis (%) calc. for $\text{C}_{23}\text{H}_{21}\text{IO}_4\text{S}$: C, 53.09; H, 4.07; I, 24.39; O, 12.30; S, 6.16; found: C, 52.97; H, 4.22.

(E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (3e)

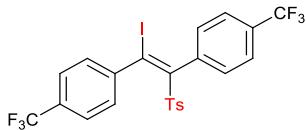


Following **GP-2**, *(E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene* (177 mg, 75%) was obtained as a white powder, m.p. (EtOH) 214-215 °C.

^1H NMR (400 MHz, CDCl_3) δ 7.51 (dd, $J = 3.0, 1.3$ Hz, 1H), 7.35-7.30 (m, 2H), 7.30-7.24 (m, 3H), 7.15-7.09 (m, 2H), 7.03 (dd, $J = 5.0, 1.3$ Hz, 1H), 6.92 (dd, $J = 4.6, 1.6$ Hz, 1H), 2.37 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 145.8, 144.3, 141.4, 138.6, 137.2, 129.4, 128.9, 128.2, 128.1, 127.6, 125.9, 125.6, 125.1, 111.8, 21.8.

(E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3f)



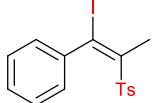
Following **GP-2**, (*E*-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (245 mg, 82%) was obtained as a white powder, m.p. (EtOH) 220-221 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.64 (t, *J* = 8.4 Hz, 4H), 7.46 (d, *J* = 8.1 Hz, 2H), 7.32 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.13 (d, *J* = 8.1 Hz, 1H), 2.39 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 149.6, 145.7, 145.3, 142.4, 136.2, 131.62 (q, *J* = 32.8 Hz, 1C, C-CF₃), 131.11 (q, *J* = 32.8 Hz, 1C, C-CF₃), 130.8, 129.7, 128.6, 127.8, 125.66 (q, *J* = 3.7 Hz, 2C, -C-C-CF₃), 125.16 (q, *J* = 3.7 Hz, 2C, -C-C-CF₃), 121.15 (q, *J* = 272.5 Hz, 1C, -CF₃), 123.14 (q, *J* = 272.4 Hz, 1C, -CF₃), 115.6, 21.8.

¹⁹F NMR (376 MHz, CDCl₃) δ -62.74 (s, 3F, -CF₃), -62.75 (s, 3F, -CF₃).

(E)-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (3g)

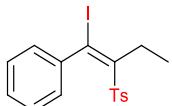


Following **GP-2**, (*E*-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (173 mg, 87%) was obtained as a pale-pink powder, m.p. (EtOH) 125-127 °C. The NMR data are in agreement with previously reported¹⁷.

¹H NMR (400 MHz, CDCl₃) δ 7.43-7.36 (m, 2H), 7.35-7.06 (m, 7H), 2.54 (s, 3H, -CH₃), 2.42 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 144.3, 144.1, 143.1, 137.4, 129.6, 128.7, 127.9, 127.8, 127.7, 115.9, 27.2, 21.7.

(E)-1-(1-iodo-1-phenylbut-1-en-2-ylsulfonyl)-4-methylbenzene (3h)

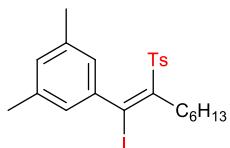


Following **GP-2**, (*E*-1-(1-iodo-1-phenylbut-1-en-2-ylsulfonyl)-4-methylbenzene (196 mg, 95%) was obtained as a pale-pink powder, m.p. (EtOH) 126-127 °C (lit. data¹⁸: m.p. 114-115 °C). The NMR data are in agreement with previously reported¹⁸.

¹H NMR (400 MHz, CDCl₃) δ 7.30-7.22 (m, 2H), 7.21-7.11 (m, 3H), 7.08 (d, *J* = 8.0 Hz, 2H), 7.04-6.97 (m, 2H), 2.95 (q, *J* = 7.4 Hz, 2H, -CH₂-CH₃), 2.36 (s, 3H, Ar-CH₃), 1.32 (t, *J* = 7.4 Hz, 3H, -CH₂-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 149.9, 143.9, 142.9, 137.9, 129.4, 128.6, 127.9, 127.73, 127.71, 115.0, 33.6, 21.7, 12.9.

(E)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3,5-dimethylbenzene (3i)



Following **GP-2**, (*E*)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3,5-dimethylbenzene (243 mg, 98%) was obtained as a yellow powder, m.p. (EtOH) 97-99 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.19 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 7.9 Hz, 2H), 6.76 (s, 1H), 6.51 (s, 2H), 3.04-2.80 (m, 2H, -CH₂-), 2.36 (s, 3H, -CH₃), 2.15 (s, 6H, -CH₃), 1.88-1.71 (m, 2H, -CH₂-), 1.53-1.29 (m, 6H, -CH₂-), 1.01-0.84 (m, 3H, -CH₃).

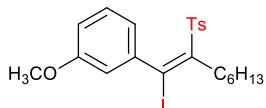
¹³C NMR (101 MHz, CDCl₃) δ 149.2, 143.4, 142.5, 138.3, 137.3, 130.3, 129.1, 127.8, 125.7, 115.4, 39.8, 31.6, 29.4, 28.5, 22.7, 21.6, 21.2, 14.2.

IR (ν/cm⁻¹): 2950 (W), 2916 (W), 2856 (W), 1621 (W), 1596 (W), 1493 (VW), 1461 (W), 1400 (VW), 1379 (W), 1320 (S), 1301 (M), 1289 (M), 1182 (W), 1151 (VS), 1132 (S), 1084 (S), 1039 (W), 1018 (W), 992 (VW), 979 (VW), 947 (W), 902 (W), 846 (S), 813 (S), 799 (W), 765 (VW), 747 (M), 731 (S), 724 (M), 715 (S), 702 (M), 683 (VS), 648 (M), 634 (W), 625 (W).

ESI-HRMS (m/z): calc. for (C₂₃H₃₀IO₂S⁺) [M+H]⁺ 497.1006; found: 497.1001.

Elemental analysis (%) calc. for C₂₃H₂₉IO₂S: C, 55.64; H, 5.89; I, 25.56; O, 6.45; S, 6.46; found: C, 55.68; H, 5.99.

(E)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3-methoxybenzene (3j)



Following **GP-2**, but the precipitate was formed after dilution of the reaction mixture with water, followed by saturation with solid NaCl (~12 g), (*E*)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3-methoxybenzene (209 mg, 84%) was obtained as a white powder, m.p. (EtOH) 83-84 °C. The NMR data are in agreement with previously reported².

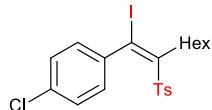
¹H NMR (400 MHz, CDCl₃) δ 7.24 (d, J = 8.1 Hz, 2H), 7.15-6.99 (m, 3H), 6.77-6.59 (m, 2H), 6.41-6.24 (m, 1H), 3.67 (s, 3H, -OCH₃), 3.02-2.81 (m, 2H, -CH₂-), 2.36 (s, 3H, -CH₃), 1.89 – 1.67 (m, 2H, -CH₂-), 1.53-1.24 (m, 6H, -CH₂-), 1.02-0.79 (m, 3H, -CH₂-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 158.7, 149.6, 143.8, 143.7, 138.0, 129.3, 128.8, 127.8, 120.7, 115.1, 114.3, 112.6, 55.1, 39.8, 31.5, 29.4, 28.4, 22.7, 21.6, 14.2.

IR (ν/cm⁻¹): 2952 (W), 2912 (W), 2870 (VW), 2849 (W), 1619 (W), 1590 (M), 1486 (W), 1461 (W), 1422 (W), 1381 (VW), 1319 (S), 1302 (M), 1275 (M), 1260 (M), 1251 (M), 1202 (W), 1181 (W), 1167 (W), 1150 (VS), 1131 (M), 1084 (S), 1037 (M), 1018 (W), 994 (W), 942 (W), 872 (W), 851 (W), 811 (M), 800 (W), 785 (M), 765 (S), 714 (S), 702 (M), 692 (VS), 669 (W), 644 (M), 634 (W), 619 (W).

ESI-HRMS (m/z): calc. for (C₂₂H₂₈IO₃S⁺) [M+H]⁺ 499.0798; found: 499.0799.

(E)-1-chloro-4-(1-iodo-2-tosyloct-1-enyl)benzene (**3k**)

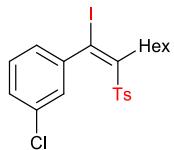


Following **GP-2**, (E)-1-chloro-4-(1-iodo-2-tosyloct-1-enyl)benzene (188 mg, 75%) was obtained as a pale-yellow powder, m.p. (EtOH) 114-115 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.34-7.24 (m, 2H), 7.15-7.10 (m, 4H), 6.98-6.89 (m, 2H), 2.93-2.78 (m, 2H), 2.38 (s, 3H), 1.73 (q, J = 8.1 Hz, 2H), 1.53-1.26 (m, 6H), 0.97-0.87 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 150.0, 144.2, 141.4, 137.8, 134.7, 129.4, 129.3, 127.9, 127.7, 113.0, 39.9, 31.5, 29.3, 28.3, 22.7, 21.7, 14.2.

(E)-1-chloro-3-(1-iodo-2-tosyloct-1-enyl)benzene (**3l**)

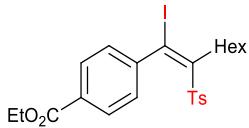


Following **GP-2**, (E)-1-chloro-3-(1-iodo-2-tosyloct-1-enyl)benzene (214 mg, 85%) was obtained as a yellow powder, m.p. (EtOH) 104-105 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.26-7.20 (m, 2H), 7.18-7.07 (m, 4H), 7.05-6.99 (m, 1H), 6.72-6.63 (m, 1H), 3.02-2.76 (m, 2H, -CH₂-), 2.39 (s, 3H, Ar-CH₃), 1.83-1.70 (m, 2H, -CH₂-), 1.57-1.28 (m, 6H, -CH₂-), 1.04-0.80 (m, 3H, -CH₂-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 150.7, 144.4, 144.2, 137.8, 133.6, 129.5, 129.1, 128.7, 127.7, 127.6, 126.4, 111.9, 39.8, 31.5, 29.4, 28.4, 22.7, 21.7, 14.2.

(E)-ethyl 4-(1-iodo-2-tosyloct-1-enyl)benzoate (3m)

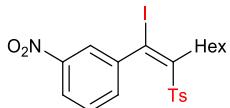


Following **GP-2**, *(E)-ethyl 4-(1-iodo-2-tosyloct-1-enyl)benzoate* (265 mg, 98%) was obtained as a white powder, m.p. (EtOH) 130-131 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.96-7.79 (m, 2H), 7.30 (d, *J* = 8.2 Hz, 2H), 7.10 (t, *J* = 8.1 Hz, 4H), 4.39 (q, *J* = 7.1 Hz, 2H), 2.99-2.75 (m, 2H), 2.38 (s, 3H), 1.77-1.68 (m, 2H), 1.54-1.27 (m, 9H), 1.01-0.87 (m, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 166.0, 149.6, 147.3, 144.4, 137.6, 130.3, 129.6, 129.0, 127.9, 127.8, 113.2, 61.2, 39.8, 31.4, 29.4, 28.3, 22.7, 21.7, 14.5, 14.2.

(E)-1-(1-iodo-2-tosyloct-1-enyl)-3-nitrobenzene (3n)

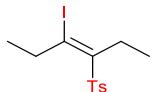


Following **GP-2**, *(E)-1-(1-iodo-2-tosyloct-1-enyl)-3-nitrobenzene* (223 mg, 87%) was obtained as a white powder, m.p. (EtOH) 118-119 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.08-8.01 (m, 1H), 7.60-7.50 (m, 2H), 7.49-7.42 (m, 1H), 7.34-7.22 (m, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 2.99-2.78 (m, 2H, -CH₂-C=C), 2.34 (s, 3H, Ar-CH₃), 1.87-1.63 (m, 2H, -CH₂-), 1.55-1.26 (m, 6H, -CH₂-), 1.01-0.85 (m, 3H, -CH₂-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 151.5, 147.4, 144.9, 144.3, 137.5, 134.0, 129.7, 128.9, 127.5, 123.2, 122.6, 110.1, 39.7, 31.4, 29.4, 28.2, 22.7, 21.5, 14.2.

(E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (3o)

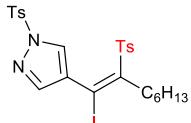


Following **GP-2**, *(E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene* (129 mg, 71%) was obtained as a white powder, m.p. (EtOH) 74-75 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.78-7.72 (m, 2H), 7.36-7.31 (m, 2H), 3.21 (q, *J* = 7.2 Hz, 2H, -CH₂-CH₃), 2.66 (q, *J* = 7.4 Hz, 2H, -CH₂-CH₃), 2.44 (s, 3H, Ar-CH₃), 1.10 (t, *J* = 7.4 Hz, 3H, -CH₂-CH₃), 1.05 (t, *J* = 7.2 Hz, 3H, -CH₂-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 145.2, 144.6, 138.6, 130.0, 129.1, 127.5, 37.6, 34.3, 21.8, 14.9, 12.8.

*(E)-4-(1-*ido*-2-tosyloct-1-en-1-yl)-1-tosyl-1H-pyrazole (3p)*



Following **GP-2**, *(E)-4-(1-*ido*-2-tosyloct-1-en-1-yl)-1-tosyl-1H-pyrazole* (275 mg, 90%) was obtained as a white powder, m.p. (EtOH) 130-131 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.16-7.75 (m, 3H), 7.40 (d, J = 8.0 Hz, 2H), 7.21 (s, 1H), 7.10 (d, J = 7.8 Hz, 2H), 6.91 (d, J = 8.0 Hz, 2H), 3.06-2.78 (m, 2H, -CH₂-), 2.46 (s, 3H, -CH₃), 2.32 (s, 3H, -CH₃), 1.86-1.64 (m, 2H, -CH₂-), 1.55-1.22 (m, 6H, -CH₂-), 1.03-0.75 (m, 3H, -CH₃).

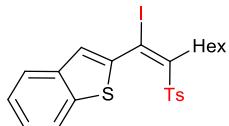
¹³C NMR (101 MHz, CDCl₃) δ 153.1, 146.6, 144.4, 144.4, 137.4, 133.8, 130.4, 130.4, 129.5, 128.7, 127.2, 125.7, 100.1, 40.0, 31.5, 29.3, 28.3, 22.7, 21.9, 21.7, 14.2.

IR (ν/cm^{-1}): 2925 (VW), 2851 (VW), 1595 (W), 1532 (VW), 1455 (VW), 1401 (VW), 1379 (M), 1320 (W), 1308 (M), 1301 (M), 1191 (W), 1185 (M), 1145 (M), 1131 (M), 1093 (M), 1083 (M), 1074 (M), 1055 (W), 1041 (W), 1015 (W), 957 (W), 876 (VW), 851 (VW), 813 (S), 800 (W), 784 (W), 711 (M), 703 (M), 695 (M), 678 (M), 667 (VS), 644 (M), 632 (VW), 607 (VW).

ESI-HRMS (m/z): calc. for (C₂₅H₃₀IN₂O₄S₂⁺) [M+H]⁺ 613.0686; found: 613.0684.

Elemental analysis (%) calc. for C₂₅H₂₉IN₂O₄S₂: C, 49.02; H, 4.77; I, 20.72; N, 4.57; O, 10.45; S, 10.47; found: C, 48.78; H, 4.90; N, 4.75.

*(E)-2-(1-*ido*-2-tosyloct-1-enyl)benzo[*b*]thiophene (3q)*

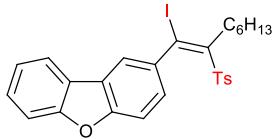


Following **GP-2**, *(E)-2-(1-*ido*-2-tosyloct-1-enyl)benzo[*b*]thiophene* (212 mg, 81%) was obtained as a pale-yellow powder, m.p. (EtOH) 100-101 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.79-7.67 (m, 1H), 7.63-7.54 (m, 1H), 7.39-7.29 (m, 3H), 7.29-7.22 (m, 2H), 6.95-6.81 (m, 2H), 3.15-2.87 (m, 2H, -CH₂-C=C-), 2.27 (s, 3H, Ar-CH₃), 1.92-1.72 (m, 2H, -CH₂-), 1.54-1.28 (m, 6H, -CH₂-), 1.02-0.85 (m, 3H, -CH₂-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 153.4, 144.3, 143.9, 141.2, 138.5, 137.2, 129.2, 128.0, 126.5, 125.6, 125.0, 124.8, 122.0, 104.5, 39.9, 31.5, 29.4, 28.4, 22.7, 21.6, 14.2.

(E)-2-(1-iodo-2-tosyloct-1-en-1-yl)dibenzod[b,d]furan (3r)



Following **GP-2**, but *n*-BuOH was used instead of EtOH and the precipitate was formed after dilution of the reaction mixture with water (150 ml), followed by saturation with solid NaCl (~36 g), (*E*)-2-(1-iodo-2-tosyloct-1-en-1-yl)dibenzod[b,d]furan (270 mg, 97%) was obtained as a white powder, m.p. (*n*-BuOH) 124-125 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.83-7.72 (m, 1H), 7.62-7.53 (m, 1H), 7.52-7.44 (m, 1H), 7.41-7.29 (m, 3H), 7.19-7.05 (m, 3H), 6.91-6.76 (m, 2H), 3.12-2.87 (m, 2H, -CH₂-), 2.01 (s, 3H, -CH₃), 1.94-1.75 (m, 2H, -CH₂-), 1.64-1.31 (m, 6H, -CH₂-), 0.99-0.89 (m, 3H, -CH₃).

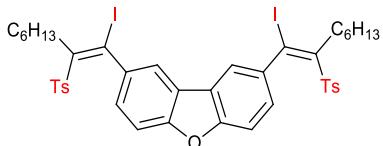
¹³C NMR (101 MHz, CDCl₃) δ 156.7, 155.8, 150.7, 143.9, 138.1, 137.6, 129.0, 127.8, 127.5, 127.5, 123.8, 123.7, 123.2, 120.9, 120.6, 114.2, 111.9, 111.0, 39.8, 31.6, 29.4, 28.5, 22.8, 21.3, 14.3.

IR (ν/cm⁻¹): 2954 (W), 2926 (W), 2855 (W), 1595 (W), 1493 (VW), 1471 (M), 1447 (M), 1427 (W), 1404 (VW), 1378 (VW), 1311 (M), 1301 (M), 1290 (M), 1244 (W), 1196 (S), 1144 (VS), 1122 (M), 1104 (W), 1082 (S), 1043 (W), 1022 (W), 979 (VW), 914 (VW), 879 (VW), 841 (W), 809 (S), 766 (W), 749 (S), 731 (M), 708 (S), 694 (M), 658 (W), 631 (W), 606 (W).

ESI-HRMS (m/z): calc. for (C₂₇H₂₈IO₃S⁺) [M+H]⁺ 559.0798; found: 559.0796.

Elemental analysis (%) calc. for C₂₇H₂₇IO₃S: C, 58.07; H, 4.87; I, 22.72; O, 8.59; S, 5.74; found: C, 57.88; H, 4.89.

2,8-Bis((E)-1-iodo-2-tosyloct-1-en-1-yl)dibenzod[b,d]furan (3s)



Following **GP-2**, but *n*-BuOH was used instead of EtOH and the precipitate was formed after dilution of the reaction mixture with water (150 ml), 2,8-bis(*E*-1-iodo-2-tosyloct-1-en-1-yl)dibenzod[b,d]furan (435 mg, 92%) was obtained as a white powder, m.p. (*n*-BuOH) 74-76 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.27 (d, J = 8.6 Hz, 2H), 7.17 (s, 2H), 7.15-6.88 (m, 7H), 6.88-6.60 (m, 3H), 3.16-2.61 (m, 4H, -CH₂-), 2.37-1.85 (m, 6H), 1.85-1.62 (m, 4H), 1.57-1.18 (m, 12H, -CH₂-), 0.98-0.69 (m, 6H, -CH₃).

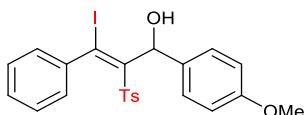
¹³C NMR (101 MHz, CDCl₃) δ 156.2, 150.9, 144.0, 138.0, 129.1, 127.9, 127.5, 123.1, 120.6, 111.1, 39.9, 31.6, 29.4, 28.5, 22.7, 21.6, 14.3.

IR (ν/cm^{-1}): 2953 (W), 2926 (W), 2855 (W), 1595 (W), 1492 (VW), 1475 (W), 1466 (W), 1448 (W), 1409 (VW), 1379 (VW), 1312 (M), 1301 (M), 1290 (M), 1247 (VW), 1195 (M), 1144 (VS), 1120 (M), 1082 (S), 1043 (W), 1025 (W), 1017 (W), 944 (VW), 870 (W), 809 (S), 761 (W), 708 (S), 695 (M), 667 (W), 655 (M), 621 (VW), 608 (VW).

ESI-HRMS (m/z): calc. for $(\text{C}_{42}\text{H}_{50}\text{I}_2\text{NO}_5\text{S}_2)^+$ [M+NH₄]⁺ 966.1214; found: 966.1212.

Elemental analysis (%) calc. for $\text{C}_{42}\text{H}_{46}\text{I}_2\text{O}_5\text{S}_2$: C, 53.17; H, 4.89; I, 26.75; O, 8.43; S, 6.76; found: C, 53.03; H, 4.93.

(E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (3t)

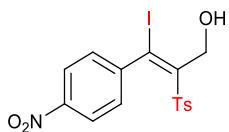


Following **GP-2**, *(E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol* (247 mg, 95%) was obtained as an orange powder, m.p. (EtOH) 156-158 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.69-7.54 (m, 2H), 7.23-6.78 (m, 11H), 6.32 (d, *J* = 12.0 Hz, 1H), 4.69 (d, *J* = 12.0 Hz, 1H), 3.85 (s, 3H, -OCH₃), 2.32 (s, 3H, Ar-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 159.4, 149.5, 144.0, 141.8, 137.8, 132.0, 129.0, 128.9, 127.6, 127.5, 126.7, 119.2, 114.1, 83.1, 55.4, 21.6.

(E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (3u)

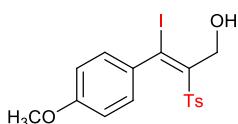


Following **GP-2**, *(E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol* (209 mg, 91%) was obtained as a white powder, m.p. (EtOH) 135-137 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.18-7.94 (m, 2H), 7.47-7.30 (m, 2H), 7.25-7.04 (m, 4H), 4.88 (s, 2H, -CH₂-OH), 3.32 (brs, -OH), 2.41 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 148.7, 148.2, 147.7, 145.5, 136.7, 129.9, 128.3, 127.9, 123.1, 115.2, 68.7, 21.8.

(E)-3-iodo-3-(4-methoxyphenyl)-2-tosylprop-2-en-1-ol (3v)



Following **GP-2**, *(E)-3-iodo-3-(4-methoxyphenyl)-2-tosylprop-2-en-1-ol* (149 mg, 67%) was obtained as a white powder, m.p. (EtOH) 120-123 °C .

¹H NMR (400 MHz, CDCl₃) δ 7.32-7.26 (m, 2H), 7.18-7.02 (m, 2H), 7.02-6.86 (m, 2H), 6.75-6.57 (m, 2H), 4.91 (d, J = 7.2 Hz, 2H, -CH₂-OH), 3.80 (s, 3H, -O-CH₃), 3.08 (t, J = 7.4 Hz, 1H, -OH), 2.36 (s, 3H, Ts-CH₃).

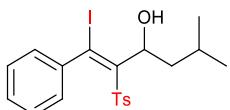
¹³C NMR (101 MHz, CDCl₃) δ 160.1, 147.2, 144.1, 137.2, 134.2, 129.4, 129.2, 127.6, 120.1, 113.0, 69.1, 55.4, 21.6.

IR (ν/cm⁻¹): 3570 (W), 2939 (VW), 2889 (VW), 2845 (VW), 1615 (W), 1601 (M), 1578 (W), 1506 (S), 1460 (W), 1446 (W), 1337 (VW), 1308 (M), 1300 (M), 1285 (M), 1248 (S), 1234 (M), 1178 (M), 1134 (S), 1112 (W), 1084 (M), 1067 (M), 1021 (M), 1015 (M), 995 (S), 977 (M), 859 (W), 832 (S), 811 (VS), 799 (M), 772 (M), 733 (S), 711 (S), 704 (M), 673 (W), 667 (VW), 638 (M), 632 (W), 625 (VW).

ESI-HRMS (m/z): calc. for (C₁₇H₁₆IO₃S⁺) [M+H]⁺ 426.9859; found: 426.9862.

Elemental analysis (%) calc. for C₁₇H₁₇IO₄S: C, 45.96; H, 3.86; I, 28.56; O, 14.40; S, 7.22; found: C, 46.13; H, 4.00; S, 7.10.

(E)-1-iodo-5-methyl-1-phenyl-2-tosylhex-1-en-3-ol (3w)

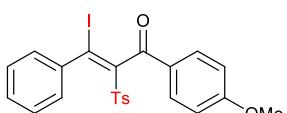


Following **GP-2**, (E)-1-iodo-5-methyl-1-phenyl-2-tosylhex-1-en-3-ol (200 mg, 85%) was obtained as a white powder, m.p. (EtOH) 145-146 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.23-6.83 (m, 8H), 6.63 (s, 1H), 5.21-4.95 (m, 1H), 3.90 (d, J = 12 Hz, 1H), 2.34 (s, 3H, Ts-CH₃), 2.31-2.17 (m, 1H, -CH₂-CH-OH), 2.17-1.96 (m, 1H, -CH-CH₃), 1.96-1.73 (m, 1H, -CH₂-CH-OH), 1.21-0.96 (m, 6H, -CH-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 150.1, 143.9, 141.9, 138.3, 129.2, 128.8, 127.6, 127.3, 116.1, 81.7, 45.2, 25.1, 23.9, 21.9, 21.7.

(E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (3x)

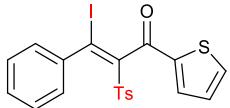


Following **GP-2**, (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (212 mg, 82%) was obtained as a white powder, m.p. (EtOH) 183-185 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.20 (d, J = 8.4 Hz, 2H), 7.39-7.22 (m, 5H), 7.22-7.01 (m, 6H), 3.93 (s, 3H, -OCH₃), 2.38 (s, 3H, Ts-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 189.3, 165.0, 149.7, 144.9, 140.3, 137.6, 132.9, 129.6, 129.5, 128.6, 128.0, 127.6, 127.1, 114.7, 113.5, 55.8, 21.8.

(E)-3-iodo-3-phenyl-1-(thiophen-2-yl)-2-tosylprop-2-en-1-one (3y)

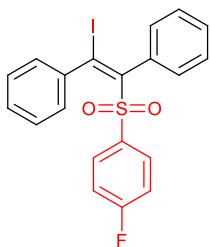


Following **GP-2**, *(E)-3-iodo-3-phenyl-1-(thiophen-2-yl)-2-tosylprop-2-en-1-one* (230 mg, 93%) was obtained as a white powder, m.p. (EtOH) 140-142 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.16-8.00 (m, 1H), 7.92-7.82 (m, 1H), 7.38-7.32 (m, 2H), 7.32-7.23 (m, 5H), 7.21-7.07 (m, 4H), 2.38 (s, 3H, Ts-CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 182.9, 149.6, 145.0, 141.2, 140.2, 137.4, 136.9, 136.5, 129.7, 129.5, 129.0, 128.6, 128.0, 127.5, 114.6, 21.8.

(E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (4a)



Following **GP-2**, *(E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene* (163 mg, 70%) was obtained as a white powder, m.p. (EtOH) 203-205 °C (lit. data¹⁹: m.p. 205-206 °C). The NMR data are in agreement with previously reported¹⁹.

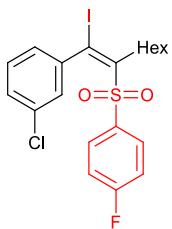
¹H NMR (400 MHz, CDCl₃) δ 7.46-7.29 (m, 10H), 7.22-7.16 (m, 2H), 7.03-6.89 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 165.58 (d, *J* = 256.5 Hz), 148.9, 142.4, 139.2, 135.92 (d, *J* = 3.2 Hz), 131.40 (d, *J* = 9.6 Hz), 130.4, 129.5, 129.3, 128.6, 128.1, 127.5, 118.6, 115.99 (d, *J* = 22.7 Hz).

166.8, 164.3, 142.4, 139.2, 135.9, 135.9, 131.4, 131.4, 130.4, 129.5, 129.3, 128.6, 128.1, 127.5, 118.6, 116.1, 115.9,

¹⁹F NMR (376 MHz, CDCl₃) δ -103.7 (s, 1F, Ar-F).

(E)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (4b)



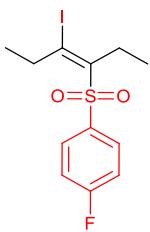
Following **GP-2**, (*E*)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (205 mg, 81%) was obtained as a white powder, m.p. (EtOH) 103-104 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.46-7.31 (m, 2H), 7.23-7.09 (m, 2H), 7.09-6.91 (m, 3H), 6.78 (s, 1H), 3.07-2.76 (m, 2H, -CH₂-), 1.89-1.66 (m, 2H, -CH₂-), 1.56-1.31 (m, 6H, -CH₂-), 1.03-0.83 (m, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 165.48 (d, *J* = 256.9 Hz), 150.4, 144.1, 136.76 (d, *J* = 3.2 Hz), 133.7, 130.41 (d, *J* = 9.6 Hz), 129.2, 128.9, 127.7, 126.4, 116.20 (d, *J* = 22.7 Hz), 112.4, 39.8, 31.5, 29.4, 28.4, 22.7, 14.2.

¹⁹F NMR (376 MHz, CDCl₃) δ -103.7 (s, 1F, Ar-F).

(E)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (4c)



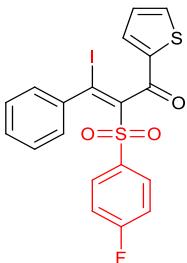
Following **GP-2**, (*E*)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (142 mg, 77%) was obtained as a yellow powder, m.p. (EtOH) 105-107 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.95-7.84 (m, 2H), 7.26-7.19 (m, 2H), 3.22 (q, *J* = 7.2 Hz, 2H), 2.66 (q, *J* = 7.4 Hz, 2H), 1.11 (t, *J* = 7.4 Hz, 3H), 1.07 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (101 MHz, CDCl₃) δ 165.67 (d, *J* = 256.4 Hz), 144.7, 137.58 (d, *J* = 3.3 Hz), 130.24 (d, *J* = 9.6 Hz), 129.7, 116.79 (d, *J* = 22.7 Hz), 37.7, 34.3, 15.0, 12.8.

¹⁹F NMR (376 MHz, CDCl₃) δ -103.5 (s, 1F, Ar-F).

(E)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (4d)



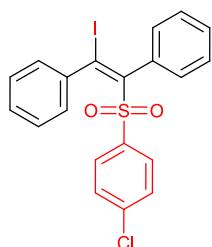
Following **GP-2**, (*E*)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (199 mg, 80%) was obtained as a pale-brown powder, m.p. (EtOH) 136-138 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.04 (d, *J* = 3.9 Hz, 1H), 7.84 (d, *J* = 5.0 Hz, 1H), 7.51-7.38 (m, 2H), 7.35-7.21 (m, 4H), 7.18-7.07 (m, 2H), 6.96 (t, *J* = 8.4 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 182.7, 165.86 (d, *J* = 257.3 Hz), 149.3, 141.1, 140.0, 137.2, 136.5, 136.30 (d, *J* = 3.0 Hz), 131.40 (d, *J* = 9.9 Hz), 129.9, 129.1, 128.2, 127.5, 116.20 (d, *J* = 22.7 Hz), 115.2.

¹⁹F NMR (376 MHz, CDCl₃) δ -102.6 (s, 1F, Ar-F).

(*E*)-(1-(4-chlorophenylsulfonyl)-2-iodoethene-1,2-diyldibenzene (**4e**)

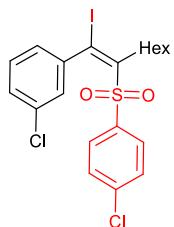


Following **GP-2**, (*E*)-(1-(4-chlorophenylsulfonyl)-2-iodoethene-1,2-diyldibenzene (204 mg, 85%) was obtained as a white powder, m.p. (EtOH) 193-195 °C (lit. data¹⁹: m.p. 197-199 °C). The NMR data are in agreement with previously reported¹⁹.

¹H NMR (400 MHz, CDCl₃) δ 7.45-7.33 (m, 8H), 7.33-7.23 (m, 4H), 7.23-7.16 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 148.8, 142.4, 140.1, 139.1, 138.4, 131.7, 130.4, 130.0, 129.6, 129.4, 129.0, 128.7, 128.1, 127.5, 118.9.

(*E*)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (**4f**)

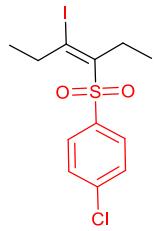


Following **GP-2**, (*E*)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (249 mg, 95%) was obtained as a white powder, m.p. (EtOH) 133-135 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.33-7.19 (m, 4H), 7.19-7.08 (m, 2H), 7.01-6.88 (m, 1H), 6.75 (s, 1H), 3.07-2.78 (m, 2H, -CH₂-), 1.87-1.65 (m, 2H, -CH₂-), 1.55-1.27 (m, 6H, -CH₂-), 1.02-0.82 (m, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 150.4, 144.0, 140.1, 139.2, 133.8, 129.2, 129.2, 129.0, 128.9, 127.8, 126.4, 112.5, 39.7, 31.5, 29.4, 28.4, 22.7, 14.2.

(E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (4g)

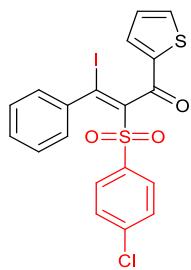


Following **GP-2**, *(E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene* (150 mg, 78%) was obtained as a white powder, m.p. (EtOH) 87-89 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.85-7.77 (m, 2H), 7.59-7.48 (m, 2H), 3.21 (q, *J* = 7.2 Hz, 2H, -CH₂-), 2.65 (q, *J* = 7.5 Hz, 2H, -CH₂-), 1.12 (t, *J* = 7.4 Hz, 3H, -CH₃), 1.07 (t, *J* = 7.2 Hz, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 144.5, 140.3, 140.0, 130.1, 129.8, 128.8, 37.7, 34.3, 15.0, 12.9.

(E)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (4h)

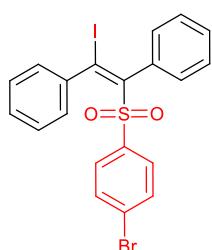


Following **GP-2**, *(E)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one* (211 mg, 82%) was obtained as a pale-brown powder, m.p. (EtOH) 140-142 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.07 (d, *J* = 3.8 Hz, 1H), 7.88 (d, *J* = 4.9 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.37-7.22 (m, 6H), 7.15 (d, *J* = 7.2 Hz, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 182.6, 149.1, 141.1, 140.7, 140.0, 138.8, 137.2, 136.6, 130.0, 129.9, 129.2, 129.1, 128.2, 127.5, 115.6.

(E)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (4i)



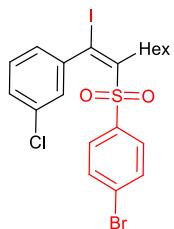
Following **GP-2**, (*E*)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (197 mg, 75%) was obtained as a white powder, m.p. (EtOH) 210-212 °C (lit. data¹⁹: m.p. 186-188 °C)..

¹H NMR (400 MHz, CDCl₃) δ 7.46-7.33 (m, 10H), 7.25-7.16 (m, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 148.8, 142.4, 139.0, 139.0, 132.0, 130.4, 130.0, 129.6, 129.3, 128.7, 128.7, 128.1, 127.5, 118.9.

The NMR data are in agreement with previously reported¹⁹

(*E*)-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (4j)

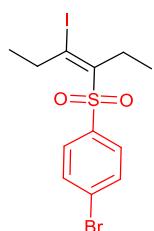


Following **GP-2**, (*E*-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (264 mg, 93%) was obtained as a white powder, m.p. (EtOH) 143-144 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.52-7.40 (m, 2H), 7.25-7.09 (m, 4H), 7.02 – 6.91 (m, 1H), 6.82-6.70 (m, 1H), 3.10-2.74 (m, 2H, -CH₂-), 1.85-1.66 (m, 2H, -CH₂-), 1.56-1.28 (m, 6H, -CH₂-), 1.01-0.84 (m, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 150.4, 143.9, 139.8, 133.8, 132.2, 129.2, 129.0, 128.9, 128.6, 127.8, 126.4, 112.6, 39.7, 31.5, 29.4, 28.4, 22.7, 14.2.

(*E*)-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (4k)

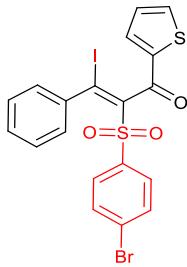


Following **GP-2**, (*E*-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (170 mg, 79%) was obtained as a white powder, m.p. (EtOH) 95-97 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.78-7.66 (m, 4H), 3.20 (q, *J* = 7.2 Hz, 2H, -CH₂-), 2.65 (q, *J* = 7.4 Hz, 2H, -CH₂-), 1.12 (t, *J* = 7.4 Hz, 3H, -CH₃), 1.08 (t, *J* = 7.2 Hz, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 144.5, 140.6, 132.8, 130.1, 128.9, 128.8, 37.7, 34.3, 15.0, 12.9.

(E)-2-(4-bromophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (4l)

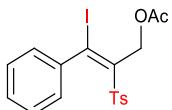


Following **GP-2**, *(E)-2-(4-bromophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one* (274 mg, 98%) was obtained as a pale-pink powder, m.p. (EtOH) 157-158 °C.

¹H NMR (400 MHz, CDCl₃) δ 8.07 (dd, J = 3.8, 1.2 Hz, 1H), 7.88 (dd, J = 4.9, 1.2 Hz, 1H), 7.51-7.43 (m, 2H), 7.37-7.24 (m, 6H), 7.18-7.09 (m, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 182.6, 149.1, 141.1, 140.0, 139.3, 137.3, 136.6, 132.2, 130.0, 129.9, 129.3, 129.1, 128.2, 127.5, 115.6.

(E)-3-iodo-3-phenyl-2-tosylallyl acetate (5a)



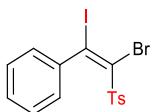
Following **GP-2**, *(E)-3-iodo-3-phenyl-2-tosylallyl acetate* (214 mg, 94%) was obtained as a white powder, m.p. (EtOH) 172-174 °C (lit. data²⁰: m.p. 163-164 °C). The NMR data are in agreement with previously reported²⁰.

¹H NMR (400 MHz, CDCl₃) δ 7.35-7.28 (m, 2H), 7.26-7.17 (m, 3H), 7.12 (d, J = 8.1 Hz, 2H), 7.09-7.03 (m, 2H), 5.29 (s, 2H, -CH₂-OAc), 2.38 (s, 3H, Ts-CH₃), 2.11 (s, 3H, -OC(=O)CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 170.3, 144.4, 143.8, 142.3, 137.4, 129.5, 129.4, 128.0, 127.8, 127.4, 123.7, 69.2, 21.7, 20.8.

IR (ν/cm⁻¹): 1733 (VS), 1619 (W), 1594 (M), 1489 (W), 1455 (W), 1441 (W), 1384 (W), 1361 (W), 1325 (S), 1301 (W), 1233 (S), 1182 (W), 1153 (VS), 1111 (S), 1079 (M), 1029 (M), 1014 (M), 996 (W), 969 (W), 923 (M), 916 (M), 853 (W), 845 (M), 814 (M), 812 (M), 799 (W), 759 (VS), 719 (S), 701 (M), 694 (VS), 673 (W), 653 (W), 625 (M), 606 (M).

(Z)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (5b)



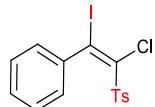
Following **GP-2**, (*Z*)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (174 mg, 75%) was obtained as an orange powder, m.p. (EtOH) 160-162 °C (lit. data²⁰: m.p. 144-145 °C). The NMR data are in agreement with previously reported²⁰.

¹H NMR (400 MHz, CDCl₃) δ 7.56 (d, J = 8.1 Hz, 2H), 7.39-7.15 (m, 7H), 2.43 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 145.3, 142.6, 136.1, 132.6, 129.8, 129.5, 128.9, 128.0, 127.2, 120.1, 21.9.

IR (ν/cm⁻¹): 1593 (W), 1577 (VW), 1548 (W), 1487 (W), 1442 (W), 1401 (VW), 1377 (VW), 1321 (S), 1304 (W), 1292 (W), 1196 (W), 1183 (W), 1159 (M), 1150 (VS), 1119 (W), 1082 (M), 1031 (W), 1016 (W), 1000 (W), 986 (VW), 923 (W), 900 (M), 838 (W), 813 (M), 762 (S), 689 (VS), 644 (S), 632 (W), 621 (VW), 603 (VW).

(Z)-1-(1-chloro-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (5c)



Following **GP-2**, (*Z*)-1-(1-chloro-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (174 mg, 83%) was obtained as a white powder, m.p. (EtOH) 156-158 °C.

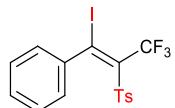
¹H NMR (400 MHz, CDCl₃) δ 7.57 (d, J = 8.3 Hz, 2H), 7.36-7.28 (m, 3H), 7.28-7.16 (m, 5H), 2.41 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 145.5, 141.4, 138.6, 135.7, 129.9, 129.6, 128.9, 128.1, 127.6, 115.5, 21.9.

IR (ν/cm⁻¹): 1593 (W), 1578 (VW), 1561 (W), 1487 (W), 1442 (W), 1398 (VW), 1377 (VW), 1323 (S), 1304 (M), 1293 (W), 1201 (W), 1183 (W), 1160 (S), 1151 (VS), 1118 (W), 1083 (S), 1031 (W), 1016 (W), 1000 (W), 985 (VW), 940 (M), 909 (M), 845 (W), 835 (M), 813 (M), 763 (VS), 708 (W), 699 (W), 690 (VS), 667 (W), 661 (W), 646 (S), 632 (W), 622 (W), 611 (M).

ESI-HRMS (m/z): calc. for (C₁₅H₁₃ClIO₂S⁺) [M+H]⁺ 418.9364; found: 418.9366.

(E)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (5d)



Following **GP-2**, (*E*)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (127 mg, 56%) was obtained as a white powder, m.p. (EtOH) 129-131 °C.

¹H NMR (400 MHz, CDCl₃) δ 7.52 (d, J = 8.1 Hz, 2H), 7.38-7.28 (m, 3H), 7.28-7.15 (m, 4H), 2.42 (s, 3H, -CH₃).

¹³C NMR (101 MHz, CDCl₃) δ 145.3, 143.7, 139.63 (q, J = 31.3 Hz, -C=C-CF₃), 137.2, 130.0, 129.8, 128.4, 128.0, 126.8, 122.43 (q, J = 3.5 Hz, -C=C-CF₃), 120.13 (q, J = 278.0 Hz, -CF₃), 21.8.

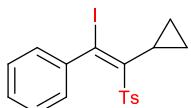
¹⁹F NMR (376 MHz, CDCl₃) δ -57.0 (s, 3F, -CF₃).

IR (ν/cm⁻¹): 1604 (W), 1596 (W), 1586 (M), 1490 (VW), 1482 (VW), 1442 (W), 1406 (VW), 1384 (VW), 1338 (M), 1306 (W), 1292 (VW), 1243 (M), 1212 (M), 1185 (W), 1154 (S), 1138 (VS), 1084 (M), 1042 (W), 1030 (W), 1020 (W), 1000 (VW), 962 (M), 917 (W), 842 (W), 817 (M), 801 (W), 770 (M), 730 (M), 703 (M), 691 (S), 682 (M), 667 (W), 632 (M).

ESI-HRMS (m/z): calc. for (C₁₆H₁₃F₃IO₂S⁺) [M+H]⁺ 452.9628; found: 452.9627.

Elemental analysis (%) calc. for C₁₆H₁₂F₃IO₂S: C, 42.49; H, 2.67; F, 12.60; I, 28.06; O, 7.08; S, 7.09; found: C, 42.73; H, 2.97; S, 7.17.

(E)-1-(1-cyclopropyl-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5e**)



Following **GP-2**, (E)-1-(1-cyclopropyl-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (199 mg, 94%) was obtained as a pale-yellow powder, m.p. (EtOH) 166-168 °C (lit. data²⁰: m.p. 159-160 °C). The NMR data are in agreement with previously reported²⁰.

¹H NMR (400 MHz, CDCl₃) δ 7.47 (d, J = 8.2 Hz, 2H), 7.25-7.06 (m, 7H), 2.37 (s, 3H), 1.35 (p, J = 7.0 Hz, 1H), 1.08 (d, J = 7.0 Hz, 4H).

¹³C NMR (101 MHz, CDCl₃) δ 147.8, 143.8, 143.0, 137.8, 129.3, 129.1, 128.4, 128.0, 127.8, 120.0, 21.7, 20.8, 12.2.

IR (ν/cm⁻¹): 1613 (W), 1595 (W), 1588 (W), 1488 (W), 1462 (VW), 1442 (W), 1427 (VW), 1398 (VW), 1384 (VW), 1312 (M), 1301 (S), 1288 (M), 1217 (VW), 1191 (W), 1184 (W), 1153 (S), 1133 (S), 1083 (S), 1072 (W), 1065 (W), 1035 (M), 1016 (W), 1004 (W), 998 (W), 933 (W), 914 (VW), 869 (W), 848 (VW), 832 (VW), 814 (M), 802 (W), 766 VS 718 (M), 705 (M), 691 (VS), 667 (W), 651 (M), 634 (M), 622 (M), 616 (VW), 605 (W).

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NMR spectra of substrates

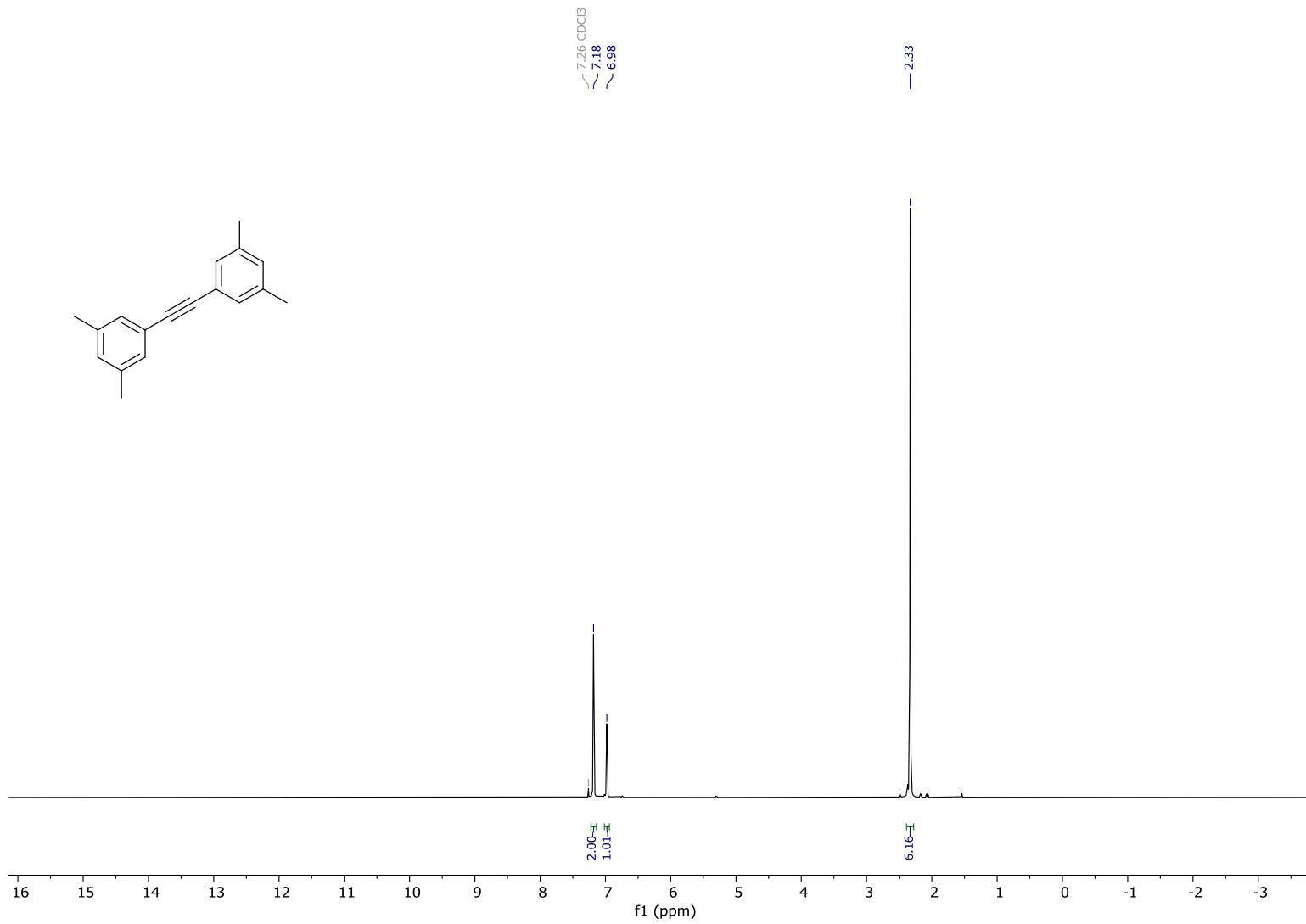


Figure S4. ¹H NMR (600 MHz, Chloroform-d) of 1,2-bis(3,5-dimethylphenyl)ethyne (1c).

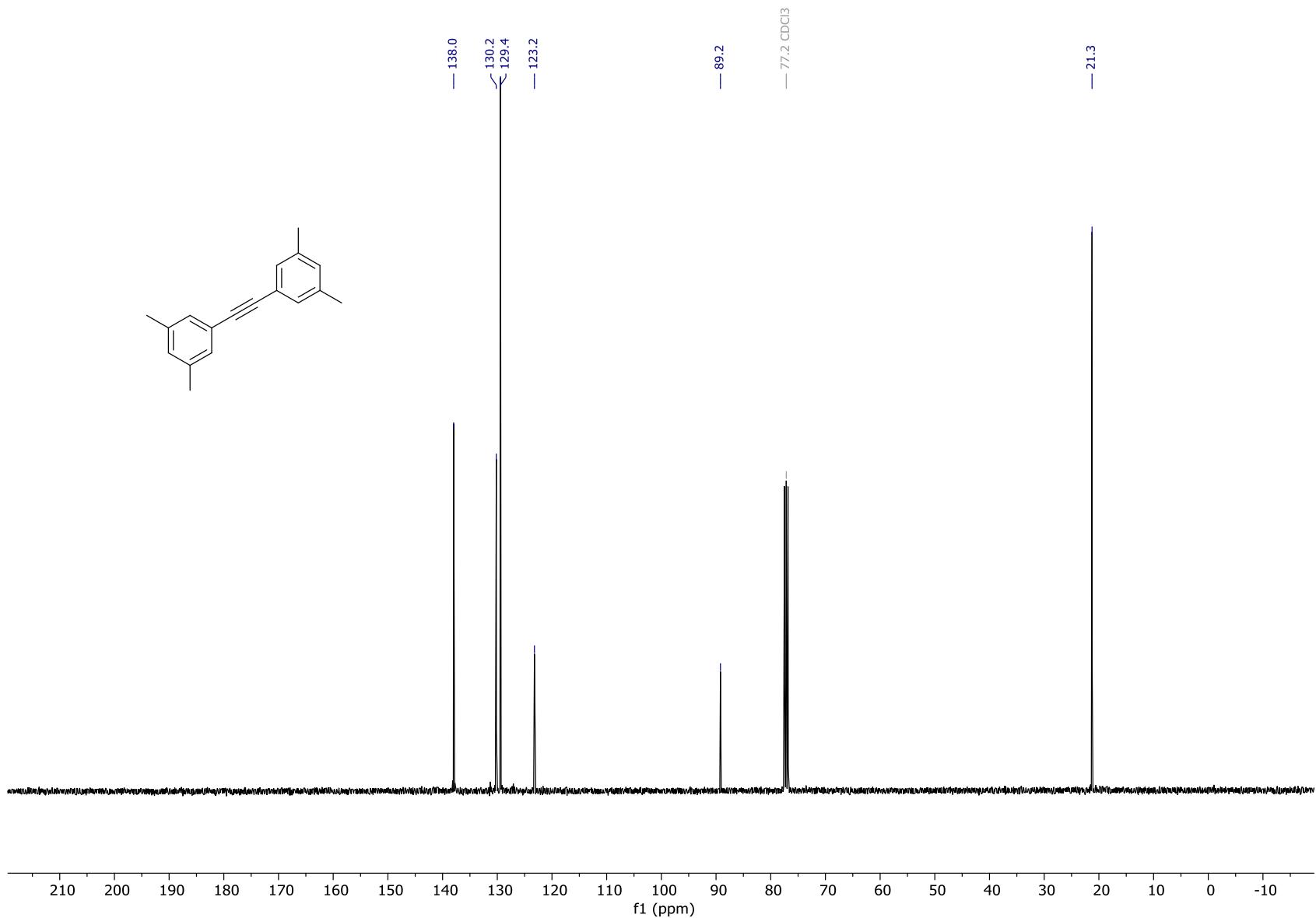


Figure S5. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of 1,2-bis(3,5-dimethylphenyl)ethyne (**1c**).

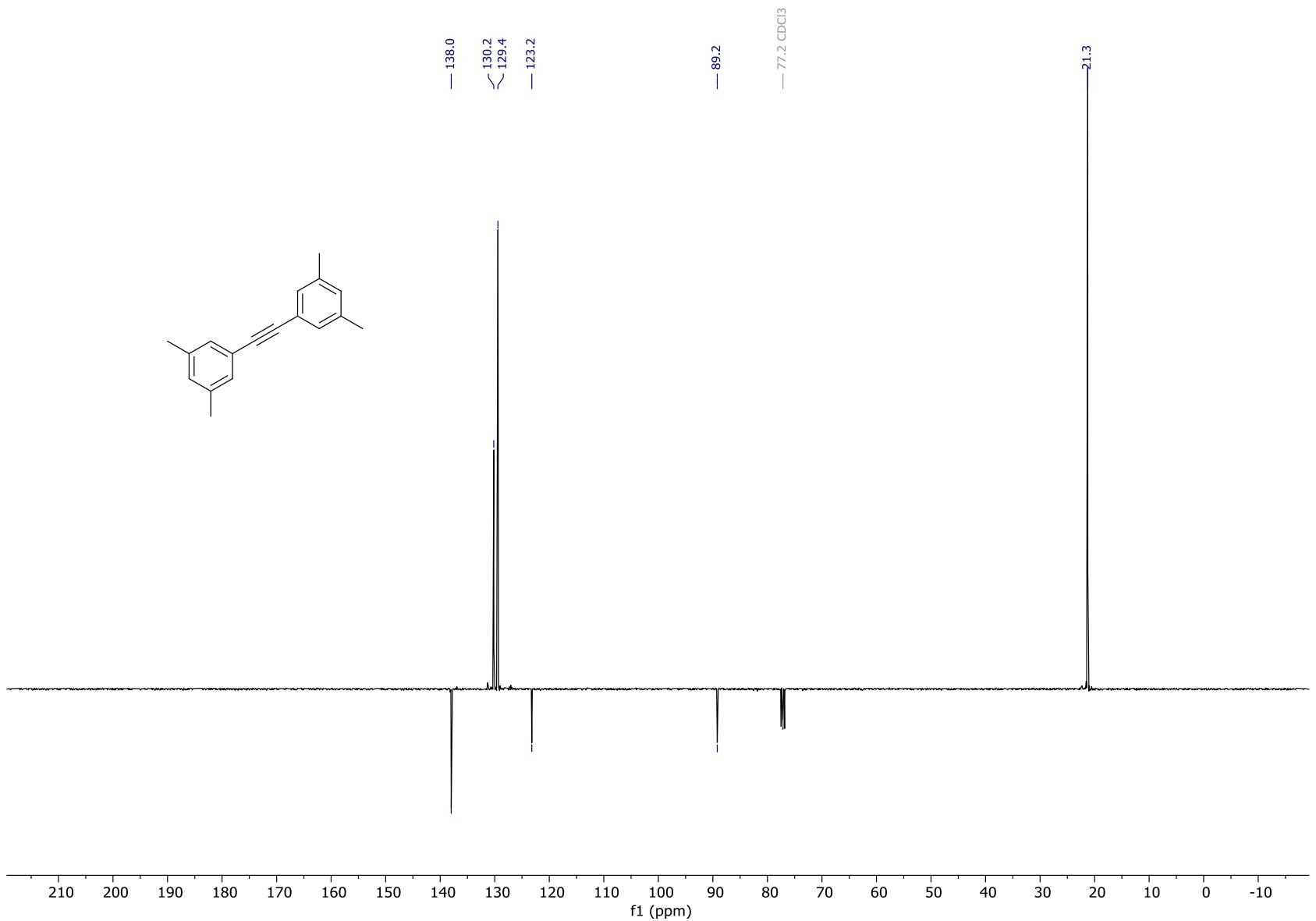


Figure S6. ^{13}C DEPTQ-135 NMR 1,2-bis(3,5-dimethylphenyl)ethyne (**1c**).

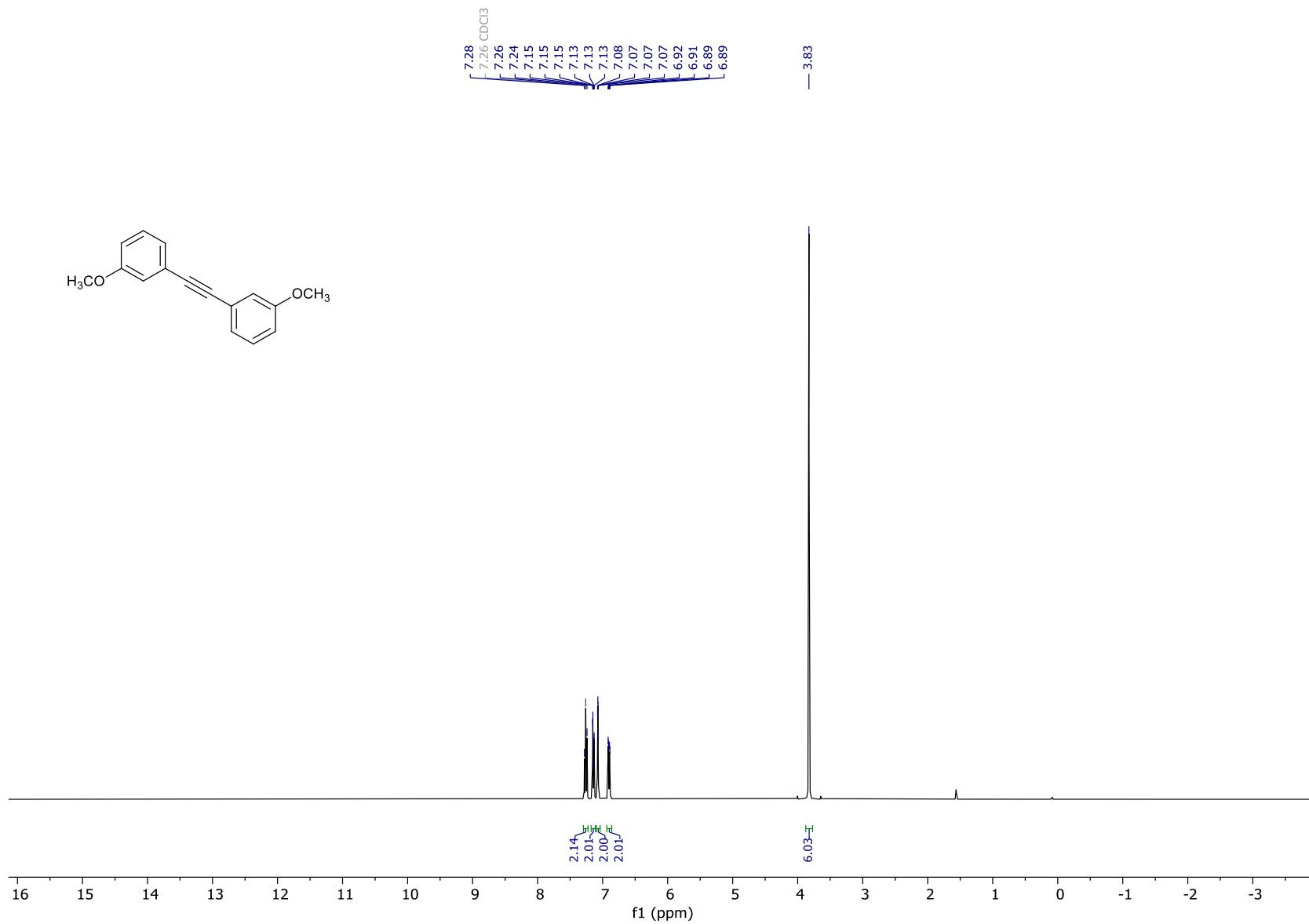


Figure S7. ¹H NMR (600 MHz, Chloroform-d) of 1,2-bis(3-methoxyphenyl)ethyne (**1d**).

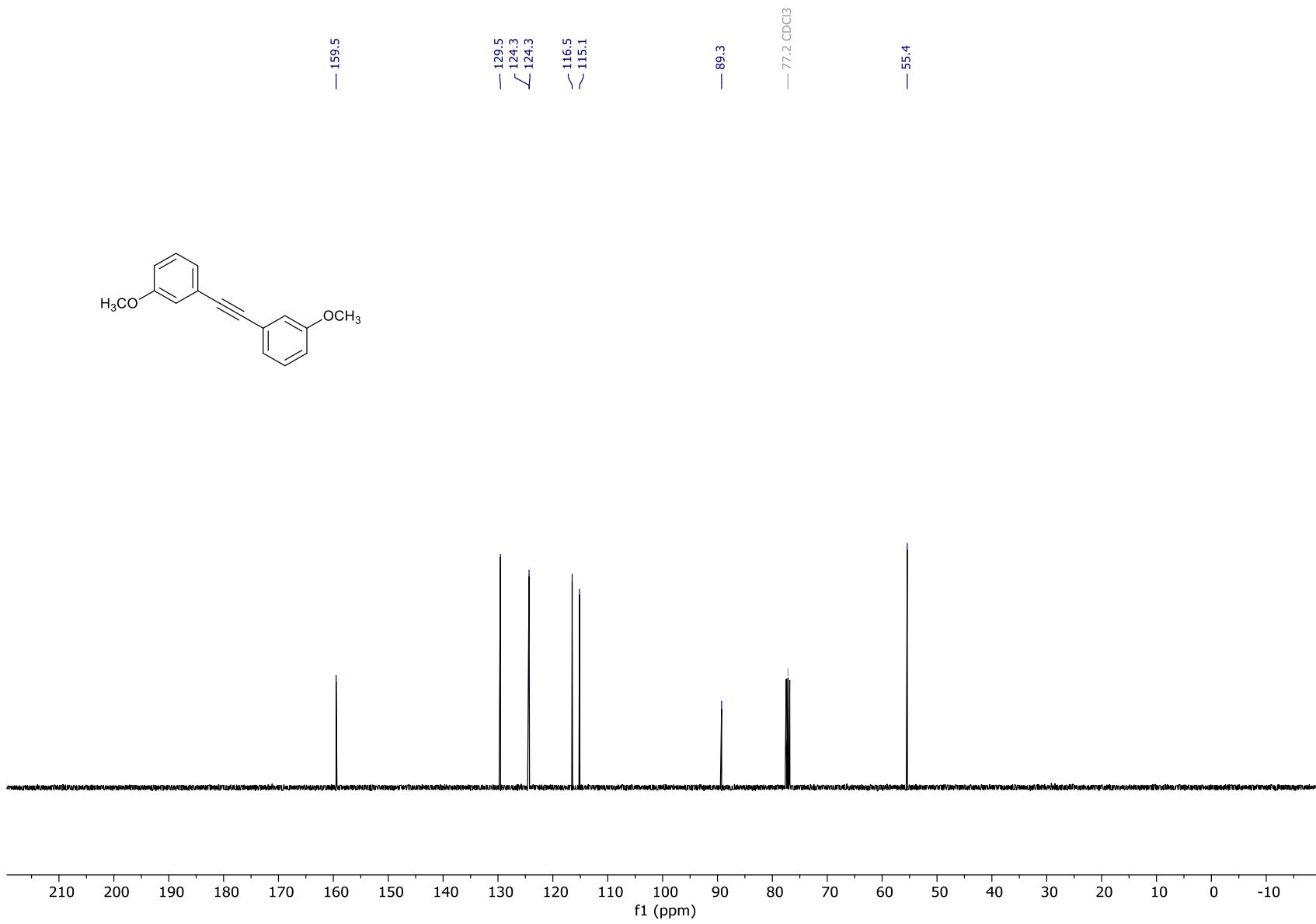


Figure S8. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of 1,2-bis(3-methoxyphenyl)ethyne (**1d**).

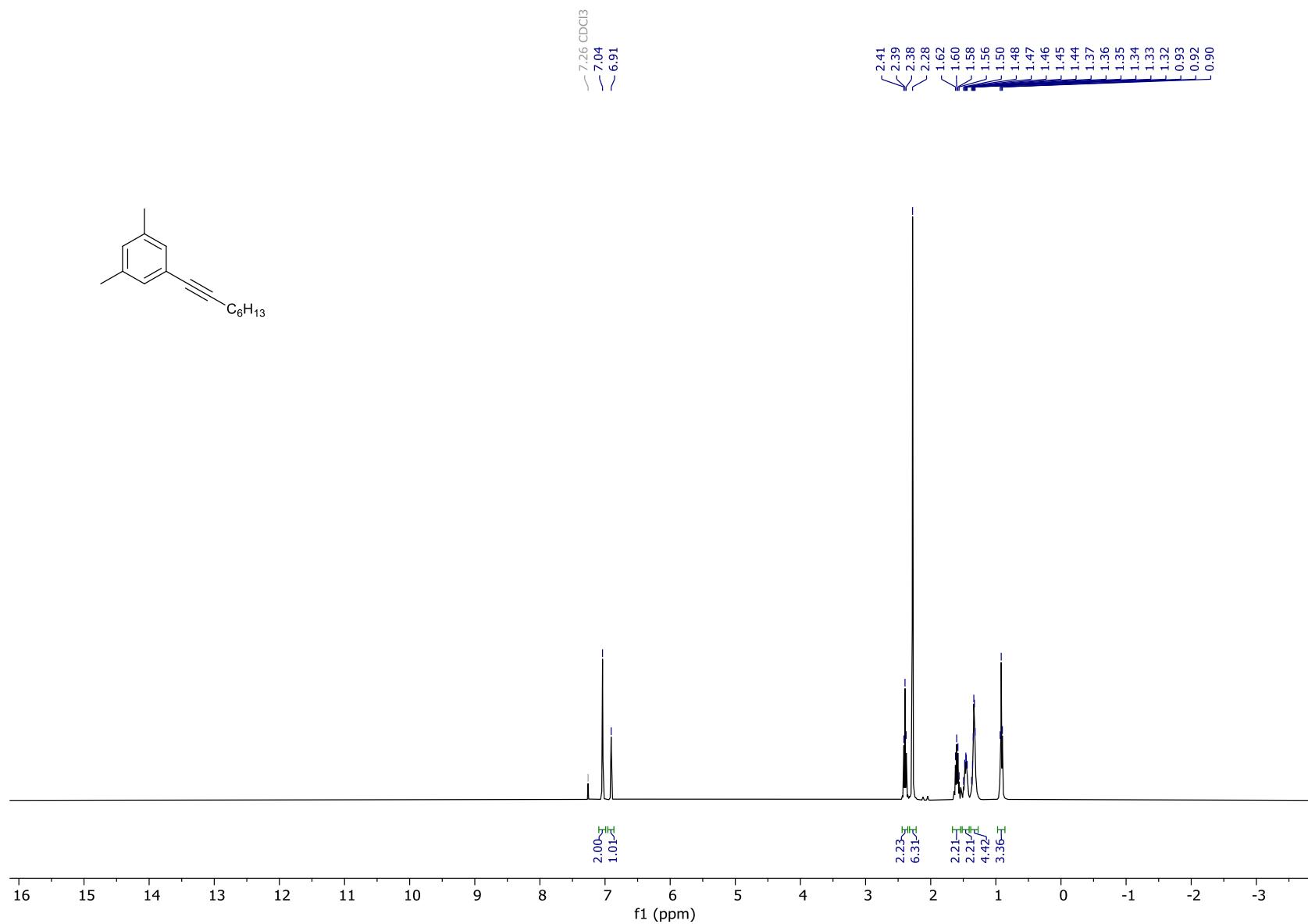


Figure S9. ¹H NMR (600 MHz, Chloroform-d) of 1,3-dimethyl-5-(oct-1-yn-1-yl)benzene (1i).

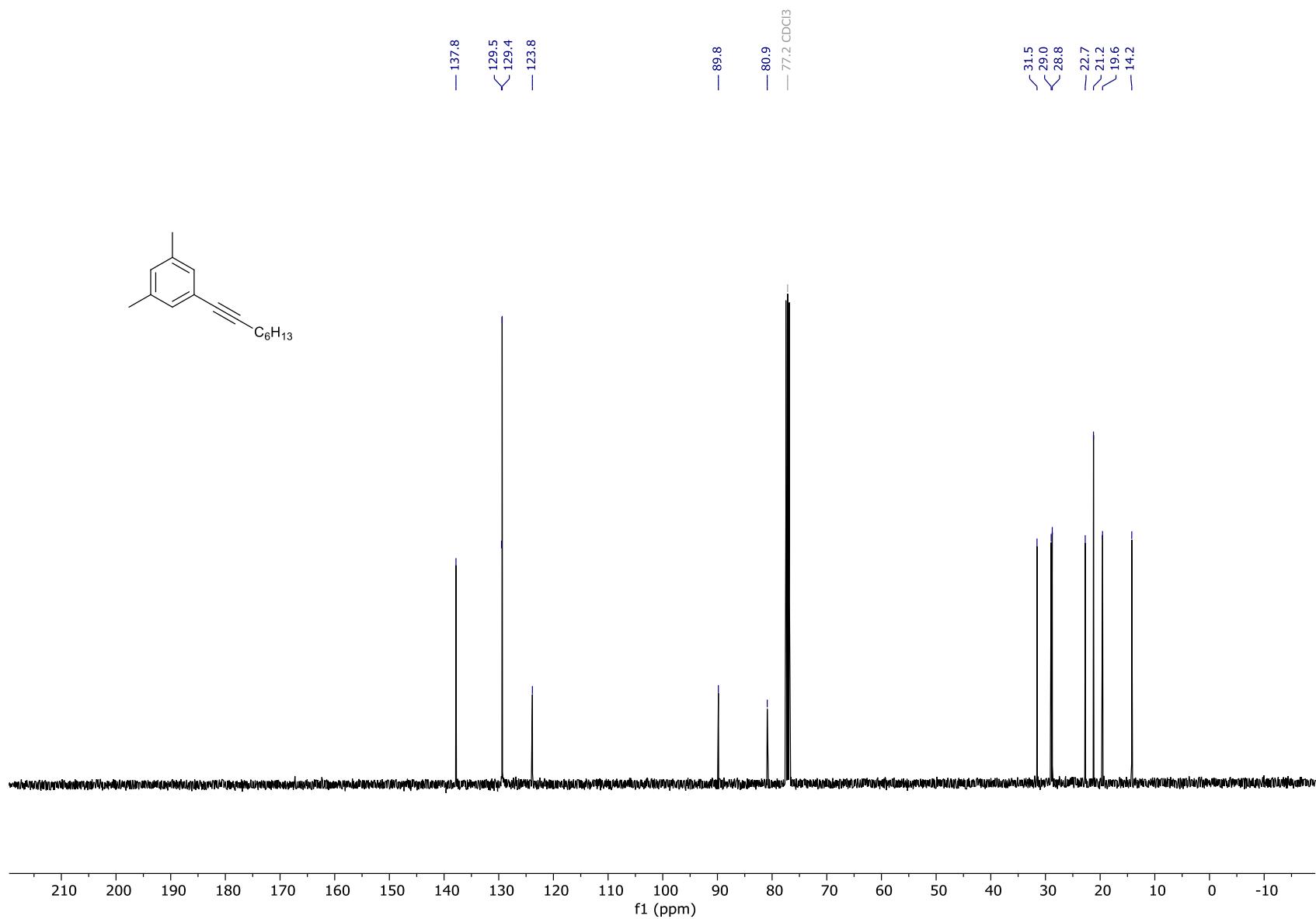


Figure S10. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of 1,3-dimethyl-5-(oct-1-yn-1-yl)benzene (**1i**).

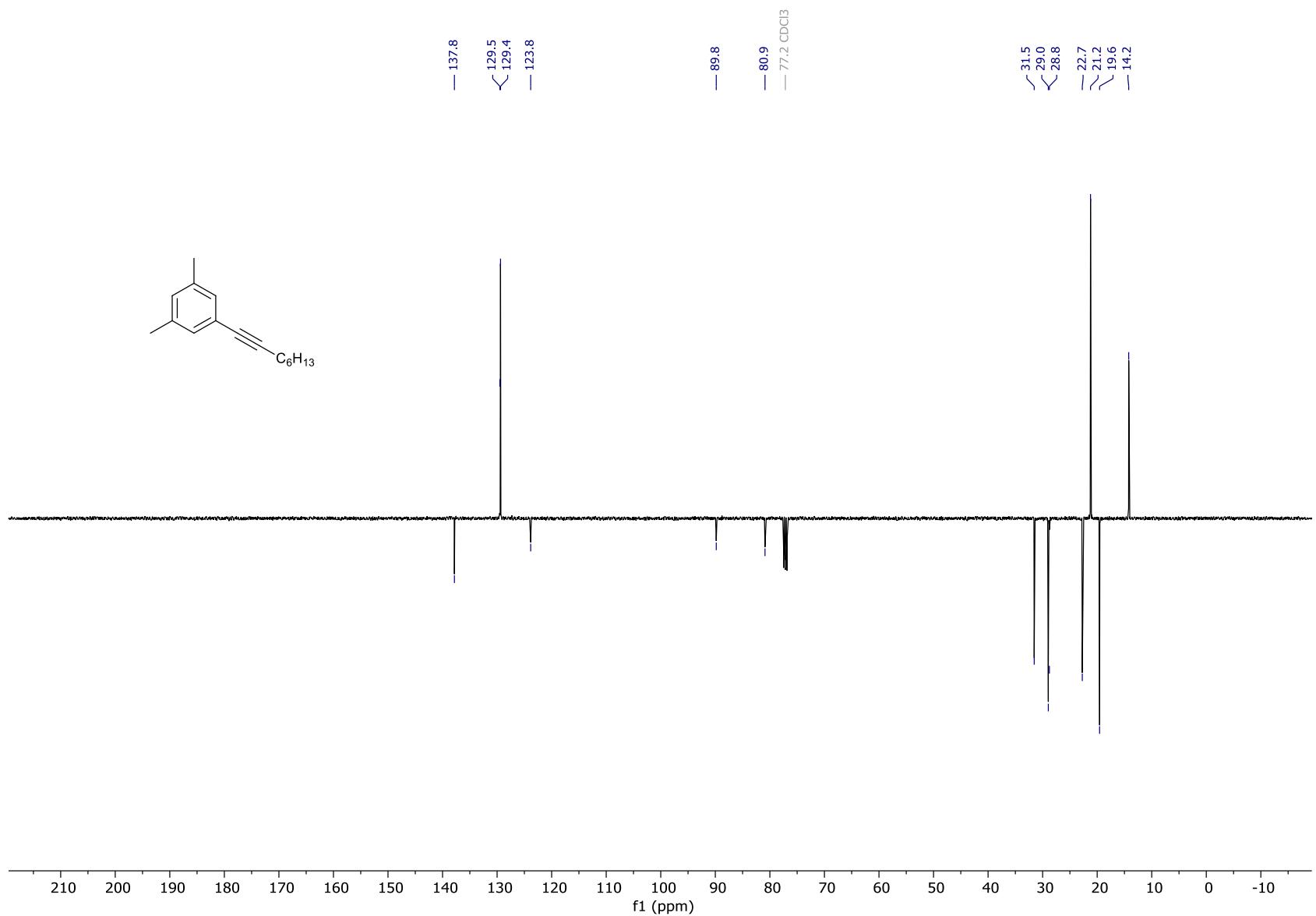


Figure S11. ^{13}C DEPTQ-135 NMR 1,3-dimethyl-5-(oct-1-yn-1-yl)benzene (**1i**).

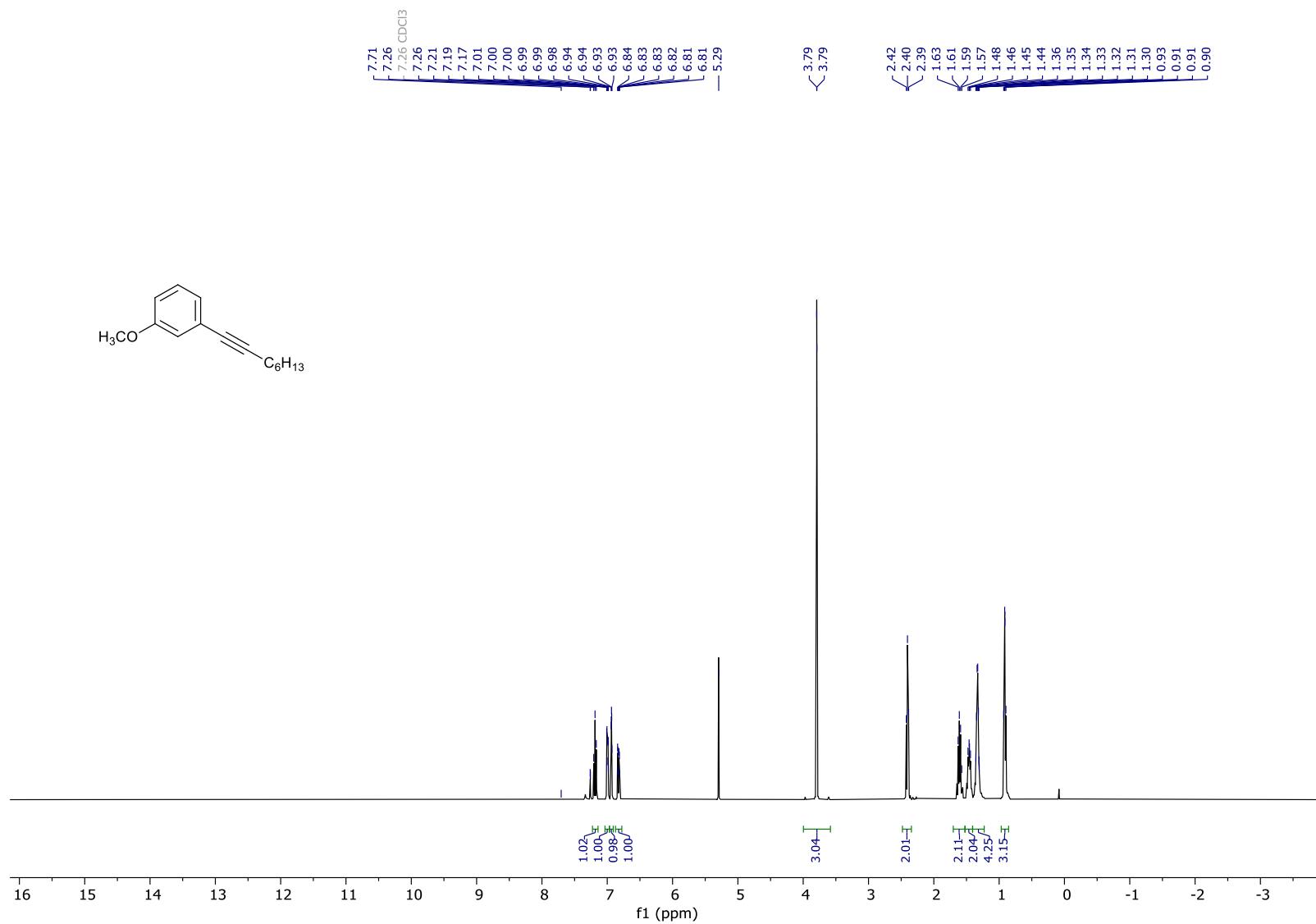


Figure S12. ¹H NMR (600 MHz, Chloroform-d) of 1-methoxy-3-(oct-1-yn-1-yl)benzene (1j).

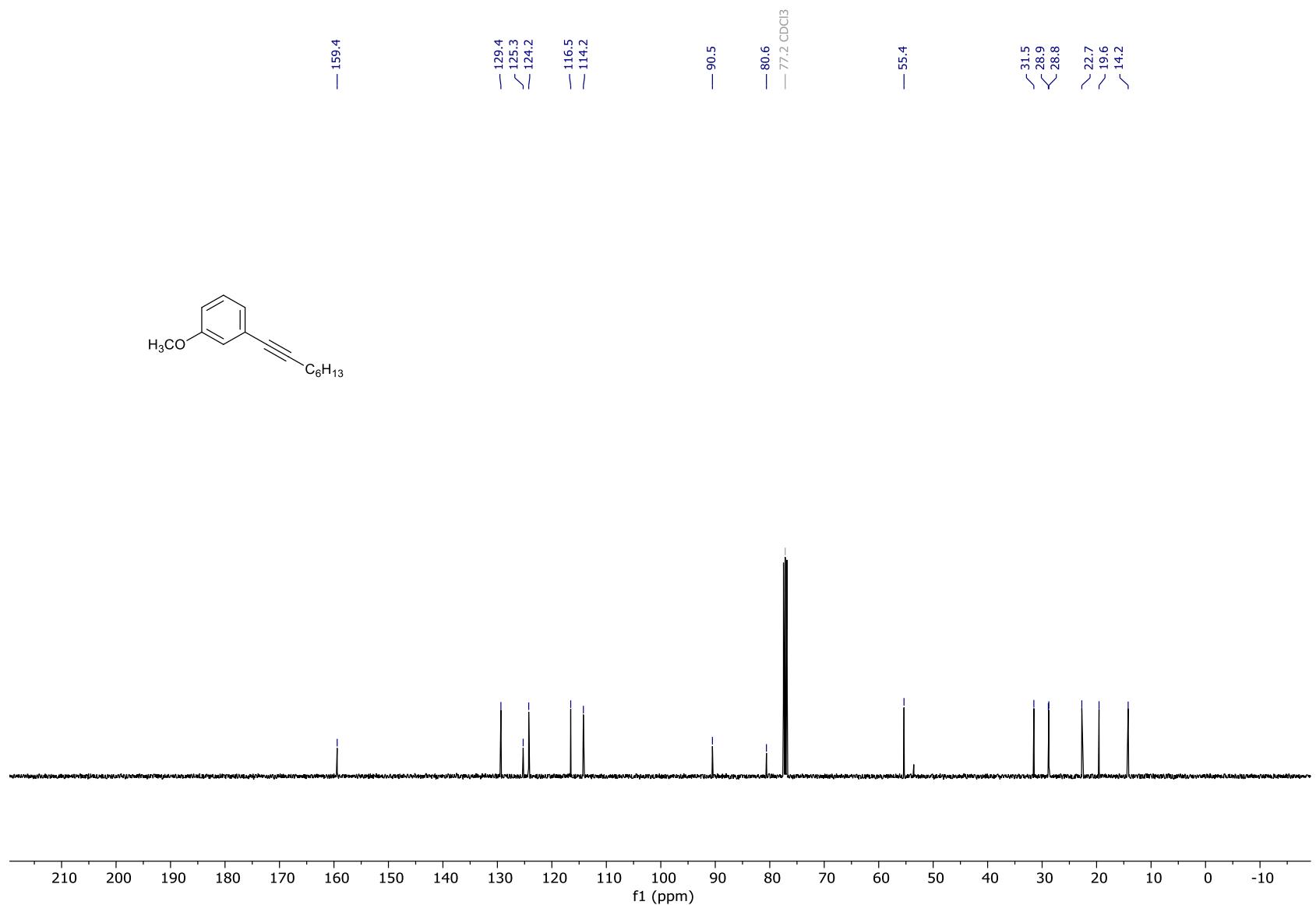


Figure S13. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of 1-methoxy-3-(oct-1-yn-1-yl)benzene (**1j**).

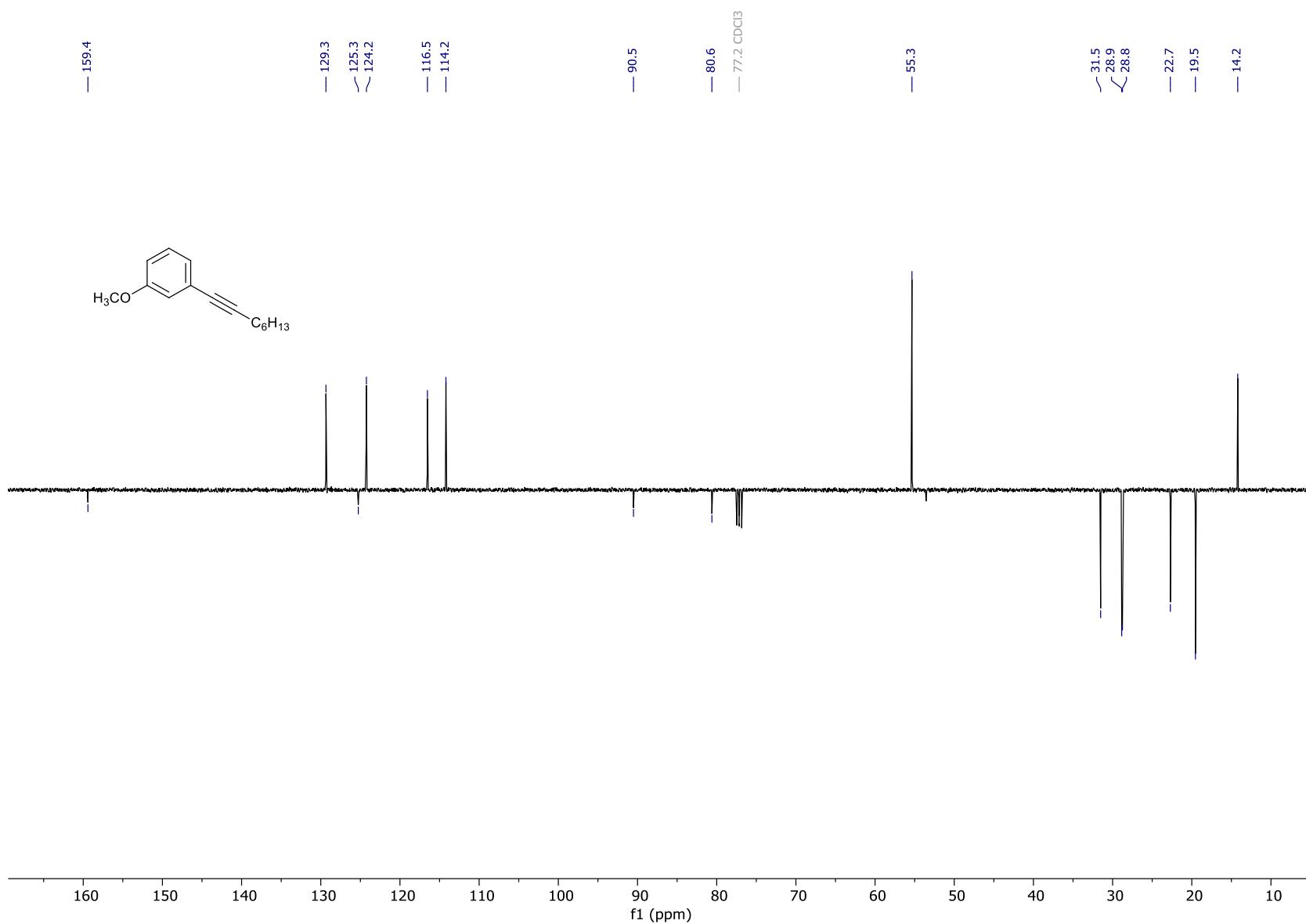


Figure S14. ^{13}C DEPTQ-135 NMR 1-methoxy-3-(oct-1-yn-1-yl)benzene (**1j**).

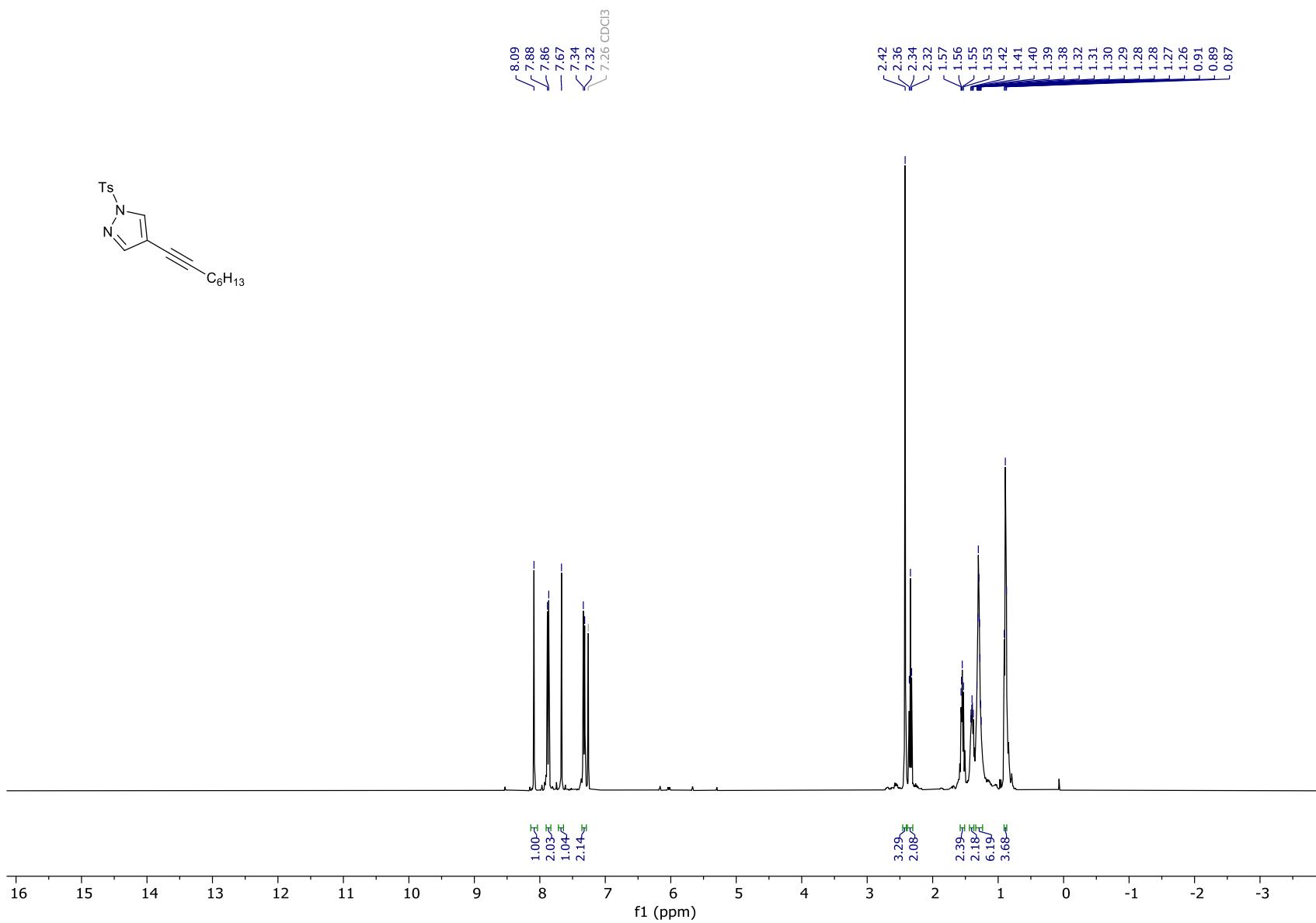


Figure S15. ¹H NMR (600 MHz, Chloroform-d) of 4-(oct-1-yn-1-yl)-1-tosyl-1H-pyrazole (1p).

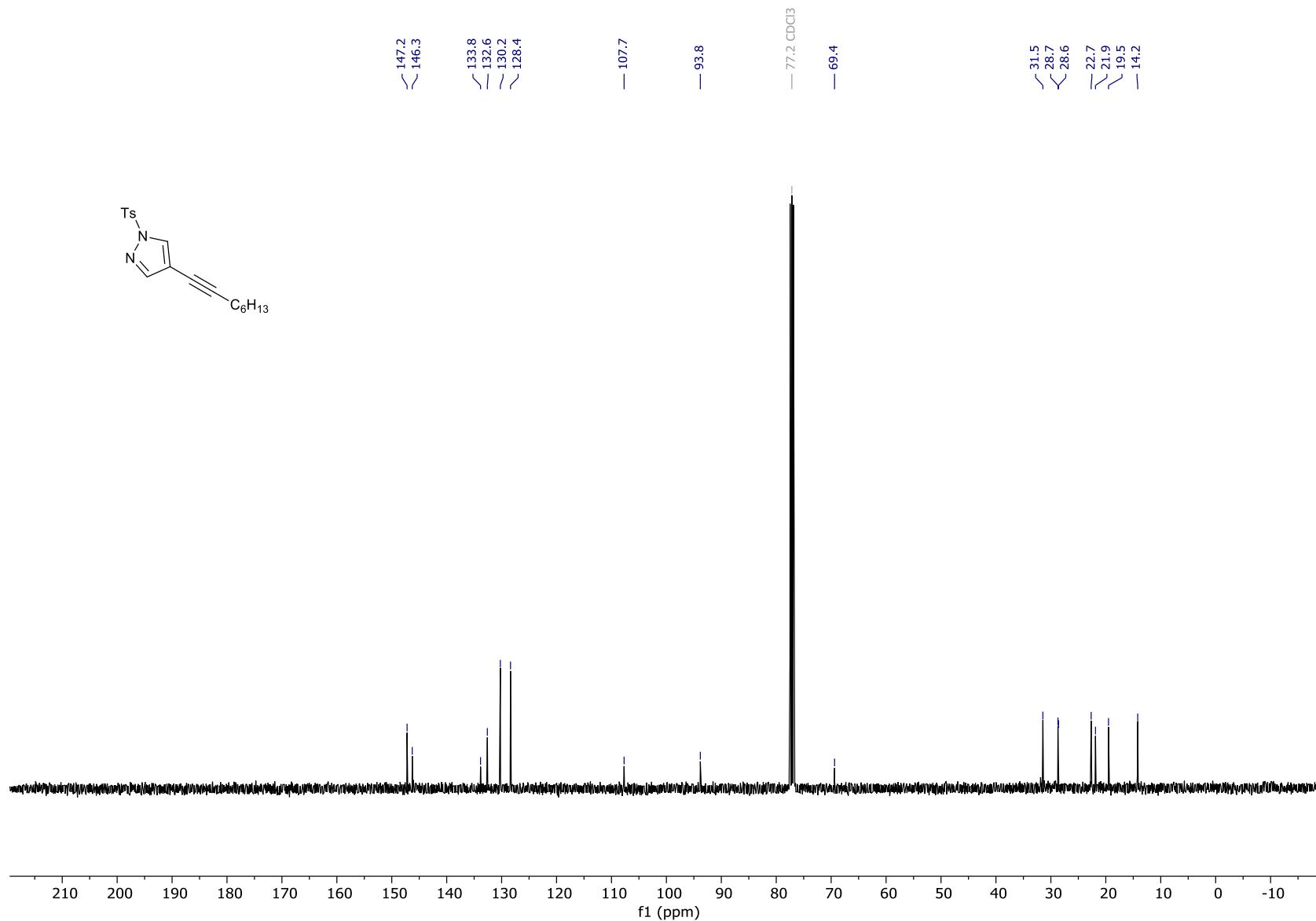


Figure S16. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of 4-(oct-1-yn-1-yl)-1-tosyl-1H-pyrazole (**1p**).

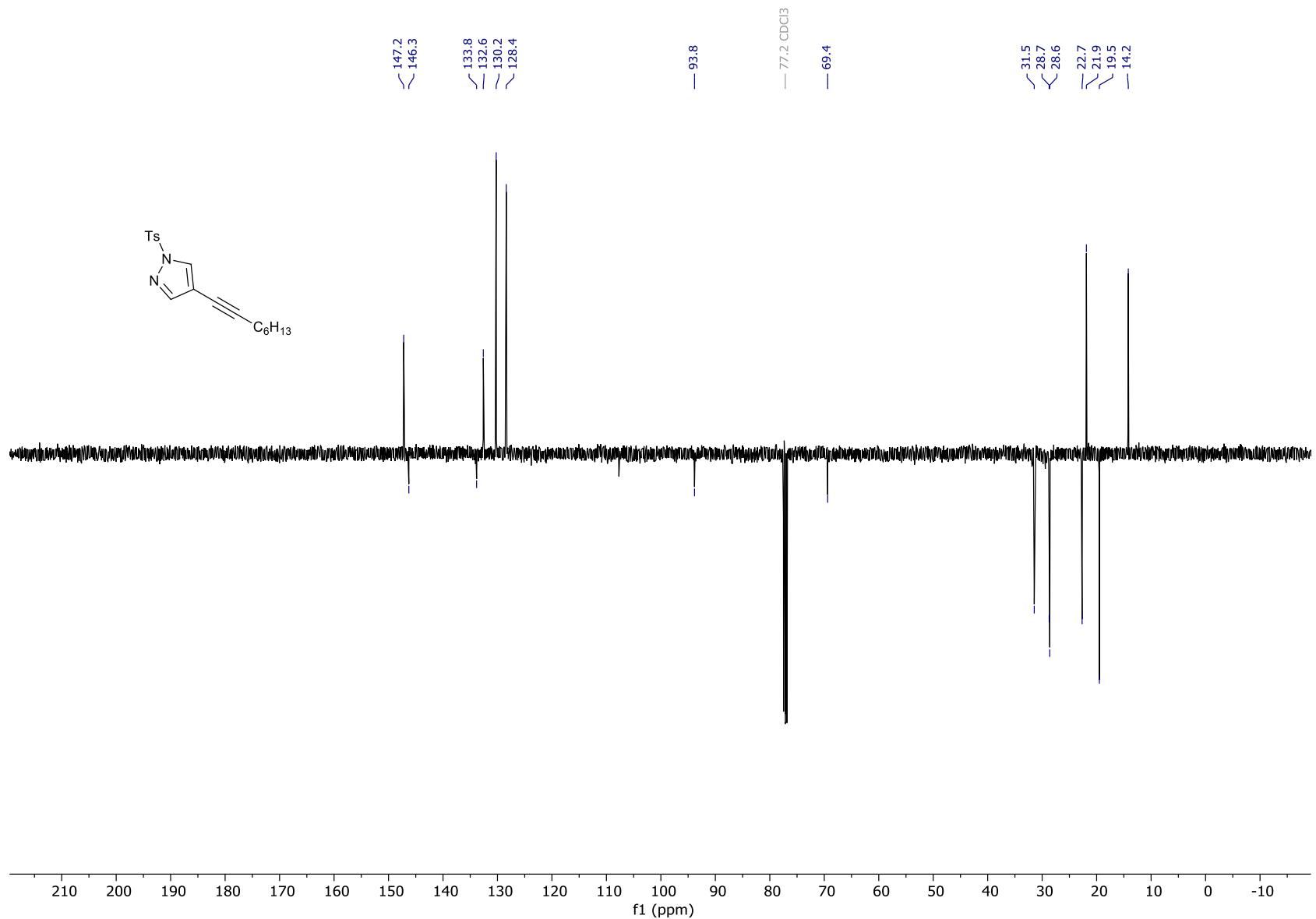


Figure S17. ^{13}C DEPTQ-135 NMR 4-(oct-1-yn-1-yl)-1-tosyl-1H-pyrazole (1p).

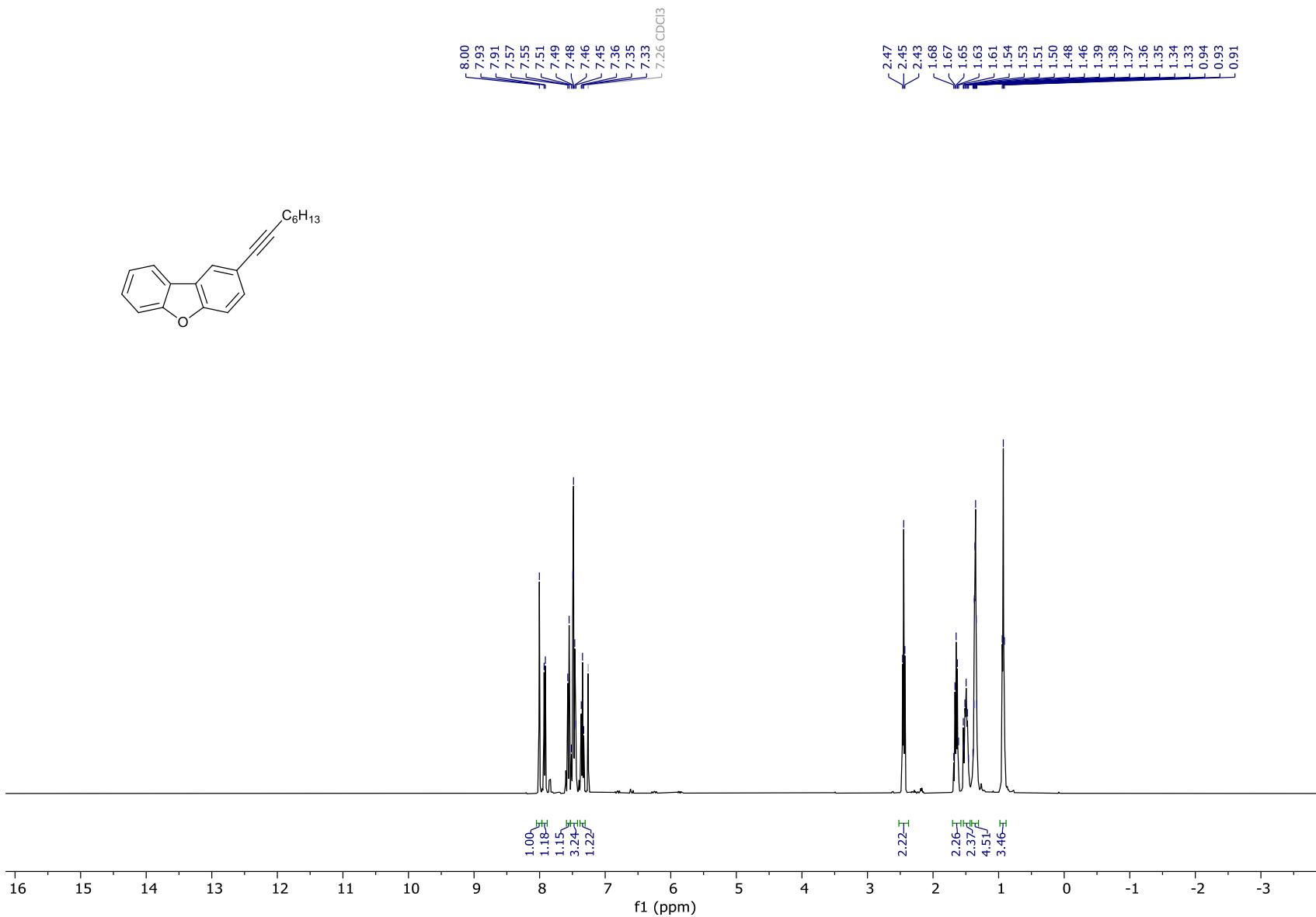


Figure S18. ^1H NMR (600 MHz, Chloroform-d) of 2-(oct-1-yn-1-yl)dibenzo[b,d]furan (**1r**).

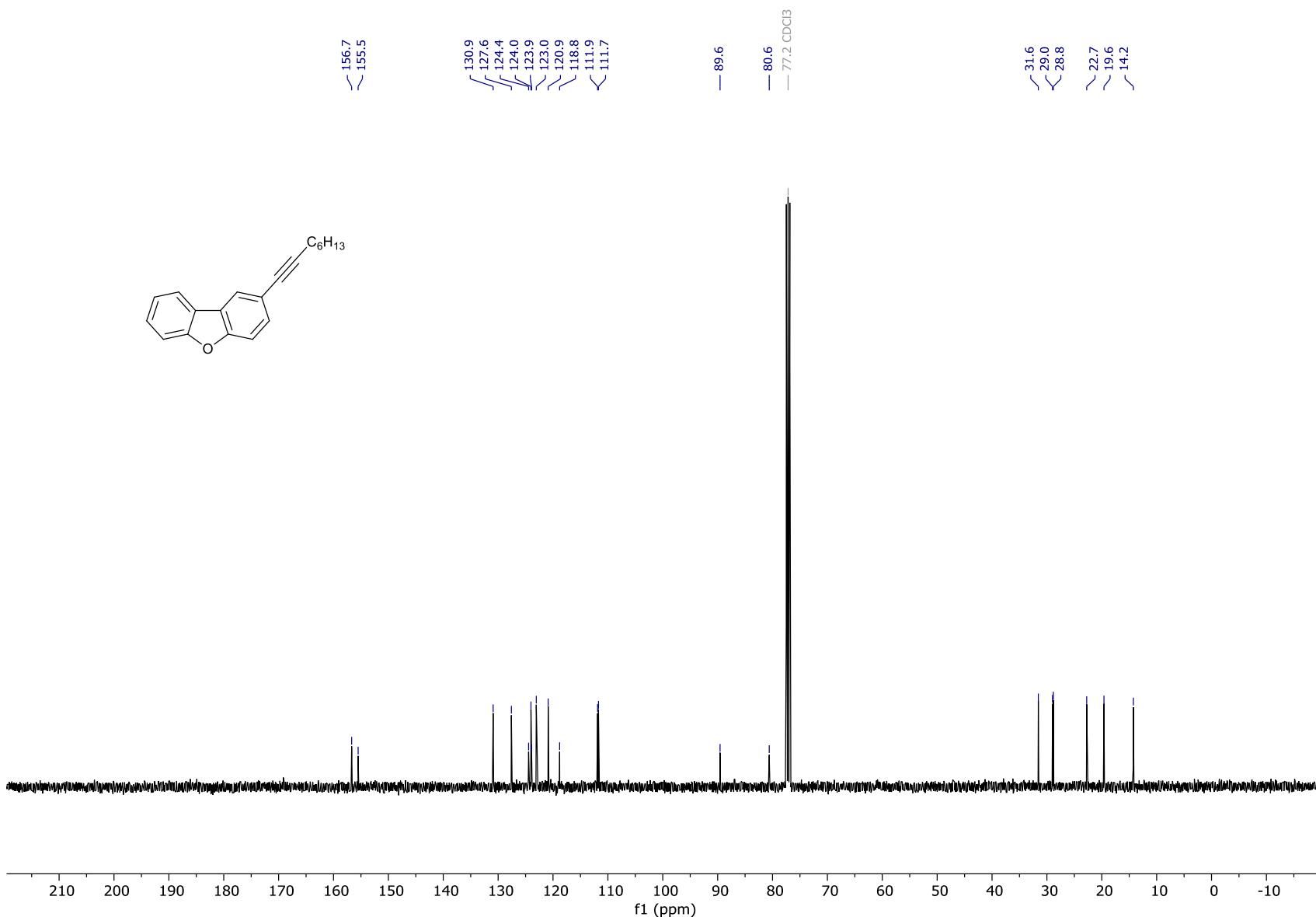


Figure S19. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of 2-(oct-1-yn-1-yl)dibenzo[b,d]furan (**1r**).

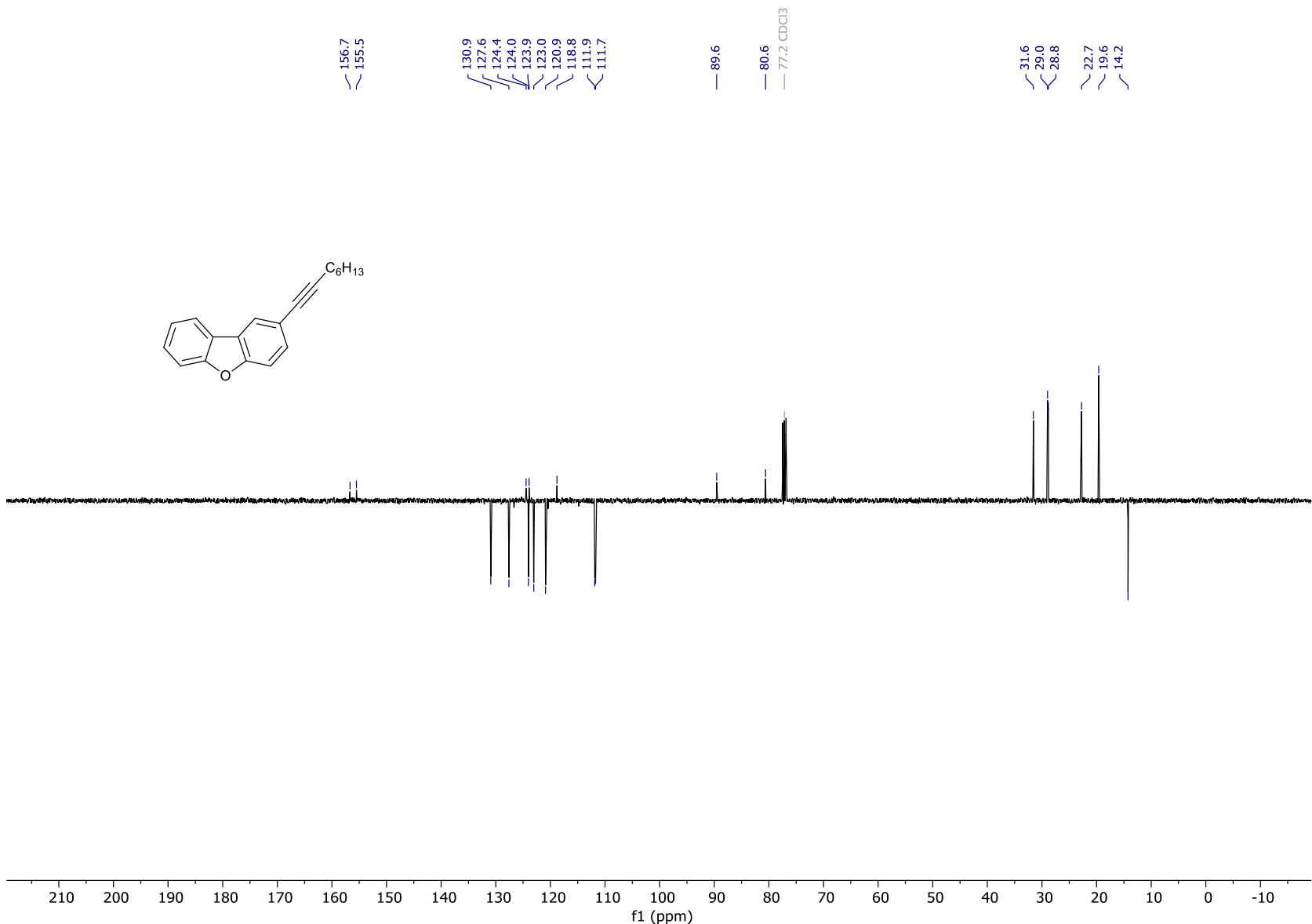


Figure S`20. ^{13}C DEPTQ-135 NMR 2-(oct-1-yn-1-yl)dibenzo[b,d]furan (**1r**).

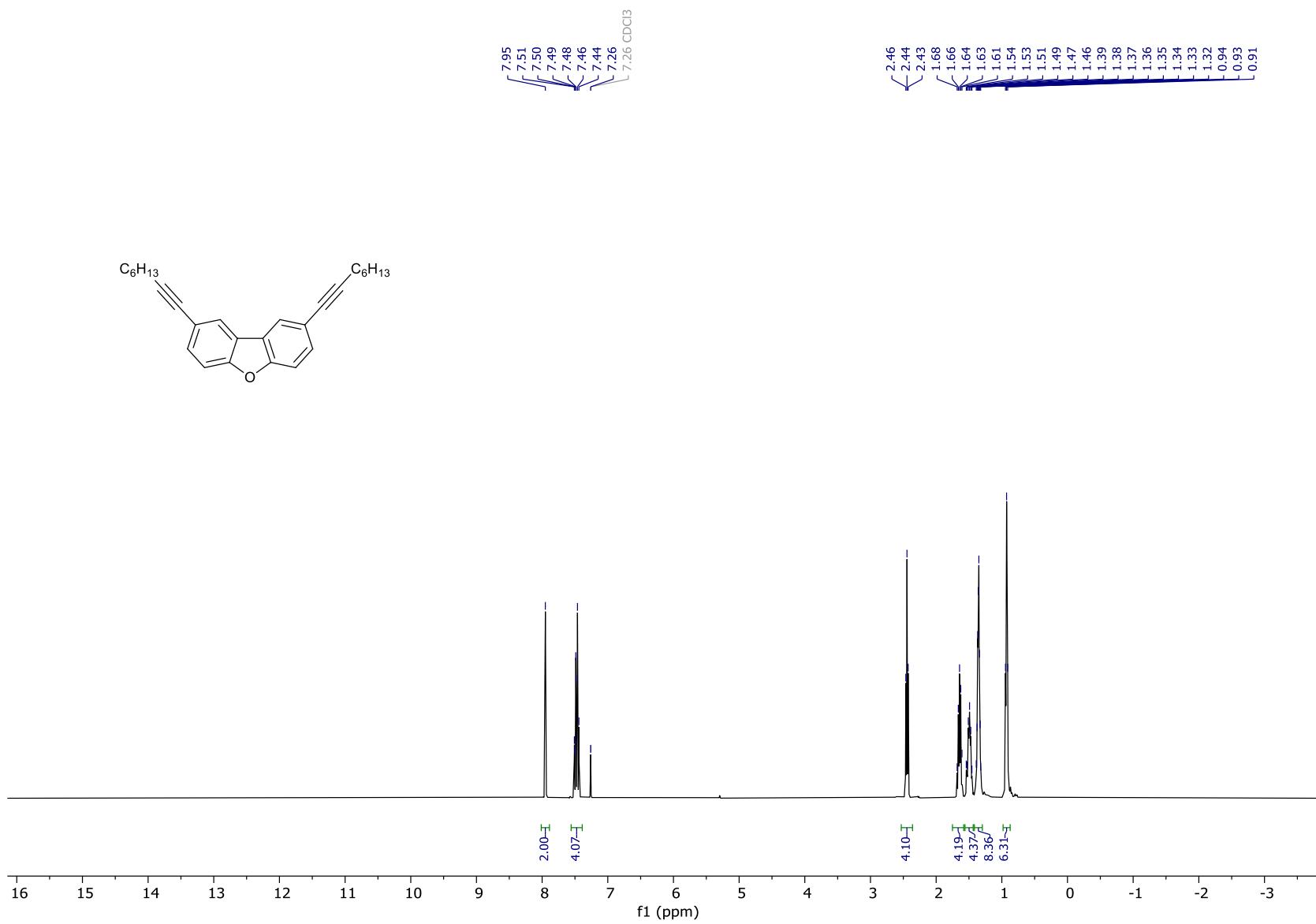


Figure S21. ^1H NMR (600 MHz, Chloroform-d) of 2,8-di(oct-1-yn-1-yl)dibenzo[b,d]furan (1s).

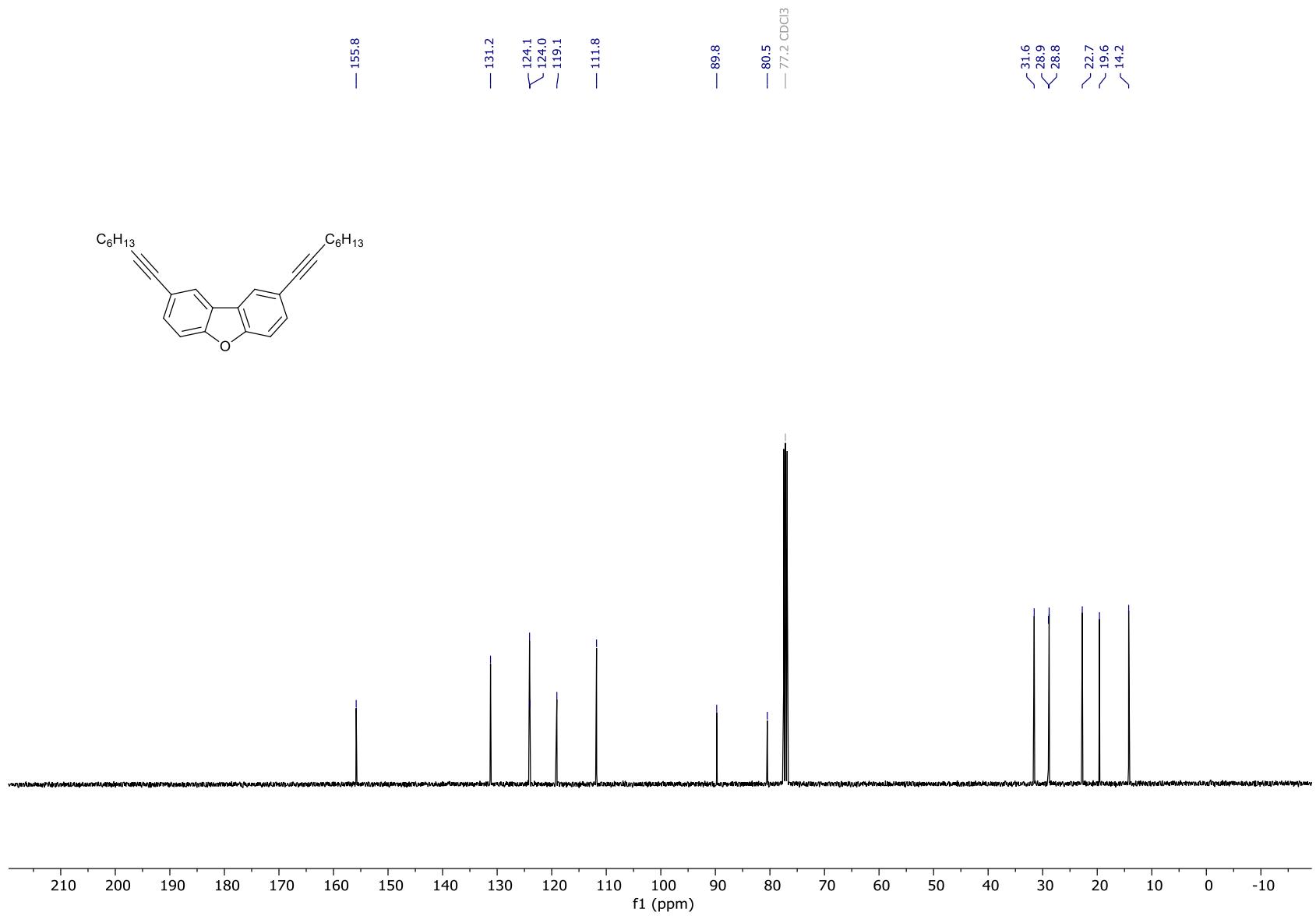


Figure S22. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of 2,8-di(oct-1-yn-1-yl)dibenzo[b,d]furan (**1s**).

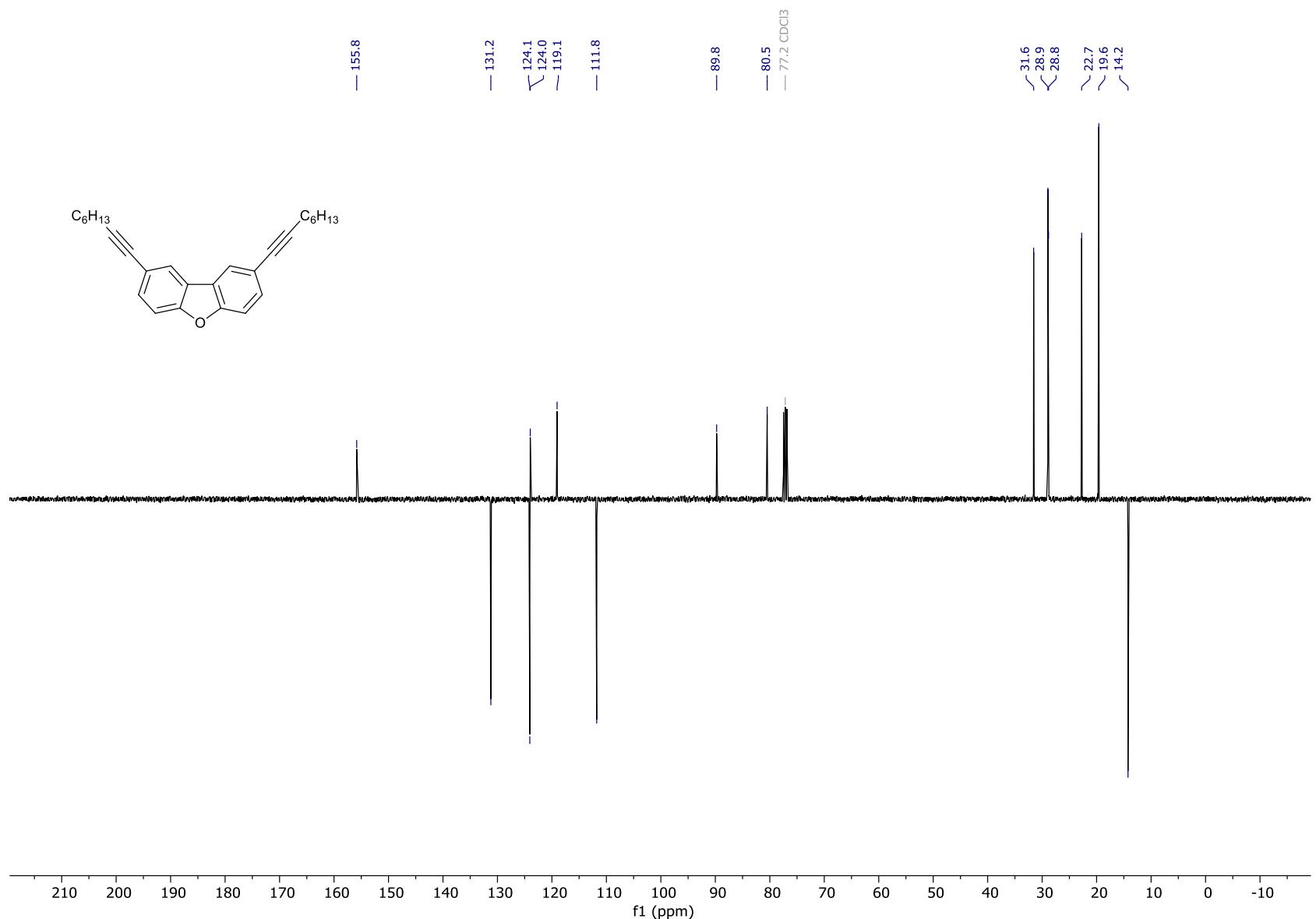


Figure S23. ^{13}C DEPTQ-135 NMR 2,8-di(oct-1-yn-1-yl)dibenzo[b,d]furan (**1s**).

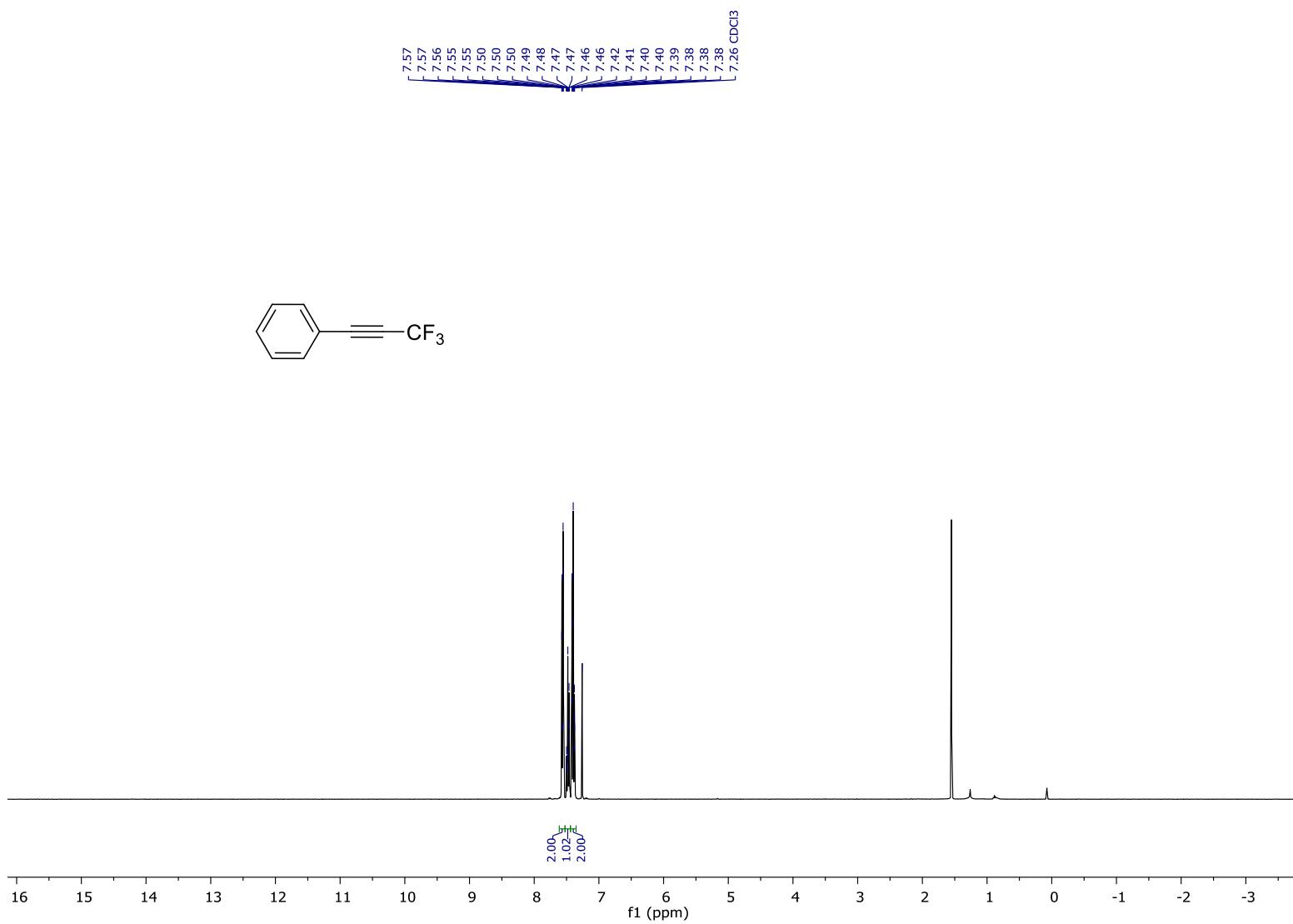


Figure S24. ¹H NMR (600 MHz, Chloroform-d) of (3,3,3-trifluoroprop-1-ynyl)benzene (1'd).

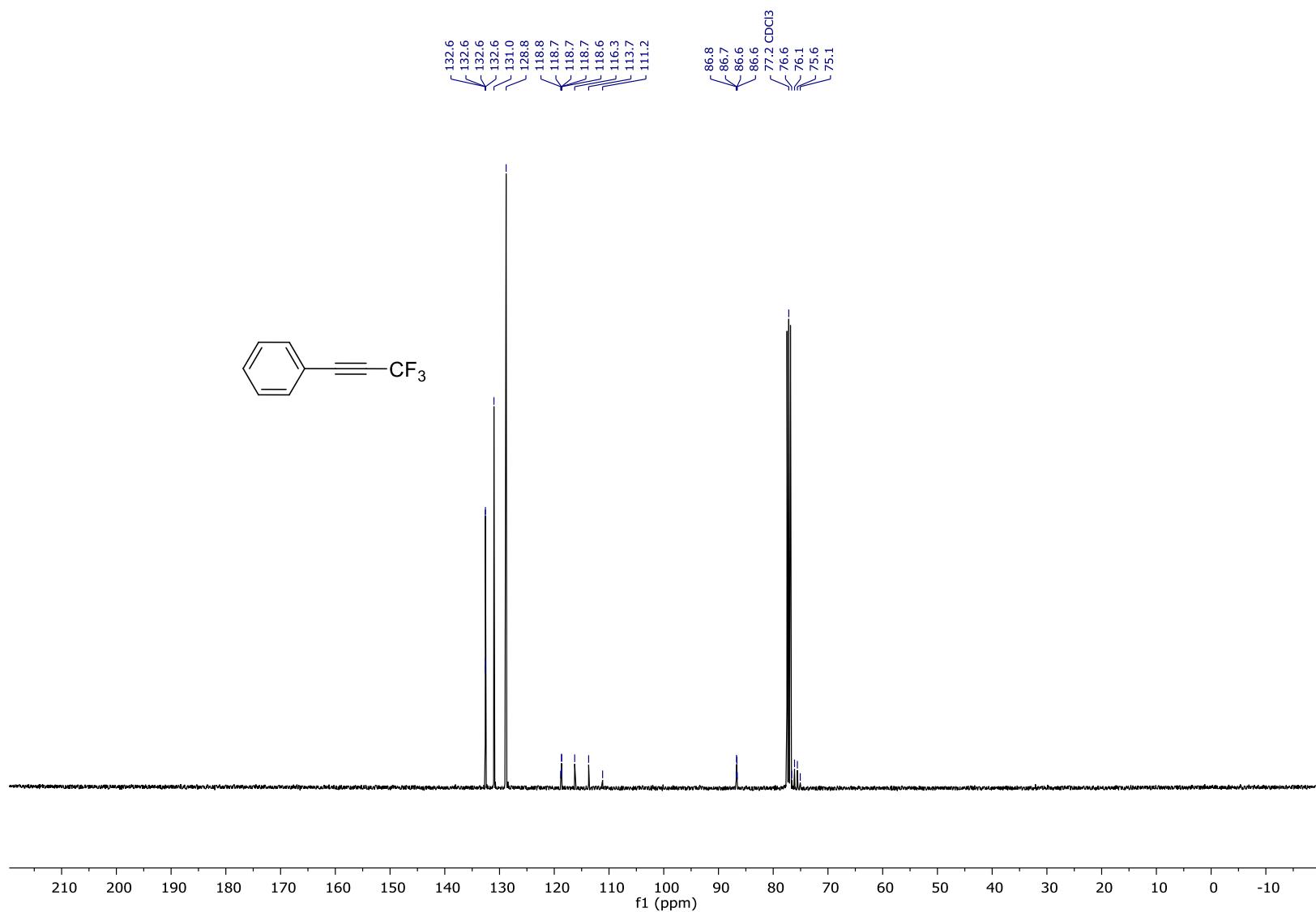


Figure S25. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (3,3,3-trifluoroprop-1-ynyl)benzene (1'd).

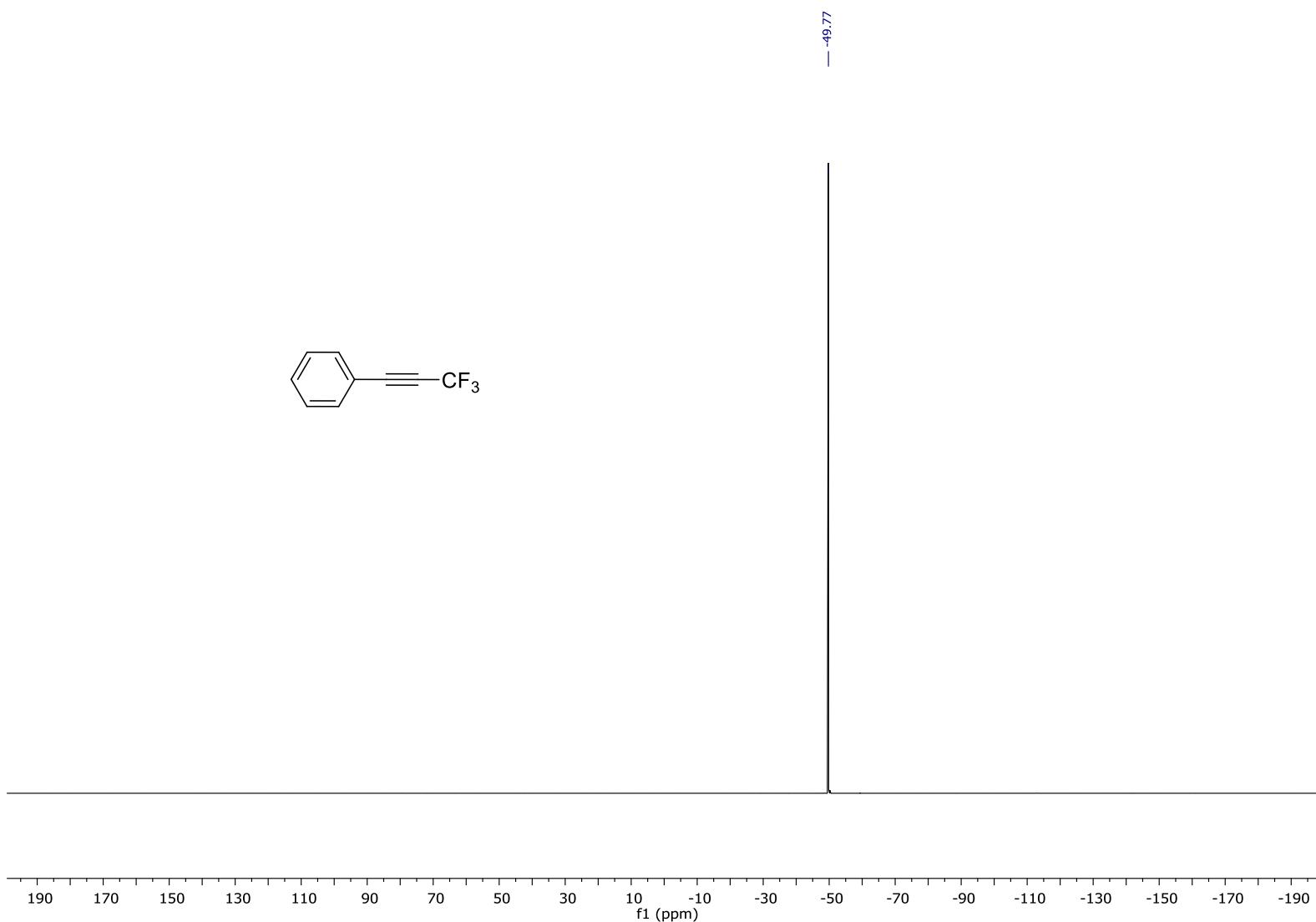


Figure S26. ^{19}F NMR (188 MHz, Chloroform-*d*) of (3,3,3-trifluoroprop-1-ynyl)benzene (1'd).

NMR spectra of products

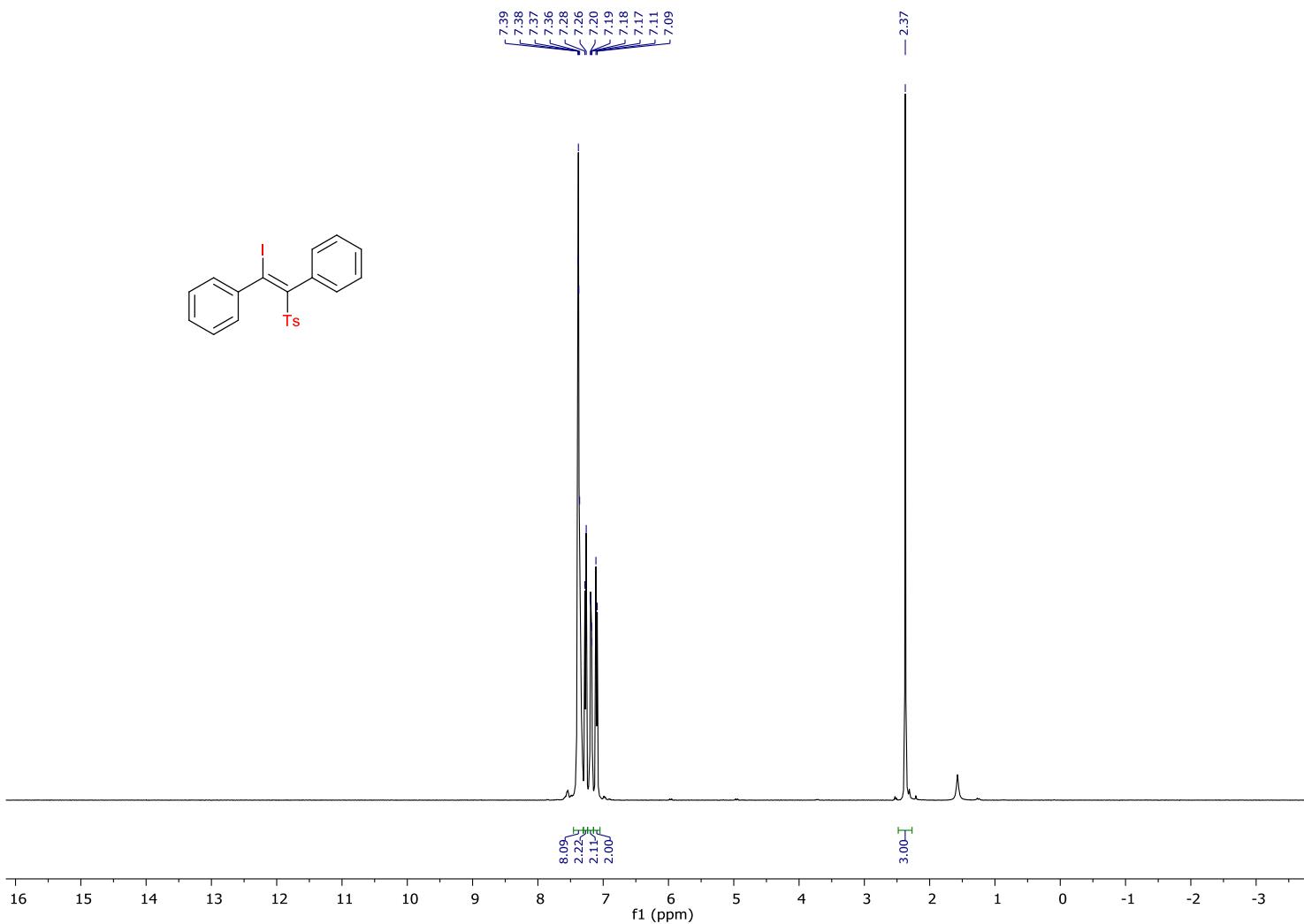


Figure S27. ¹H NMR (600 MHz, Chloroform-d) of (E)-(1-iodo-2-tosylethene-1,2-diy) dibenzene (3a).

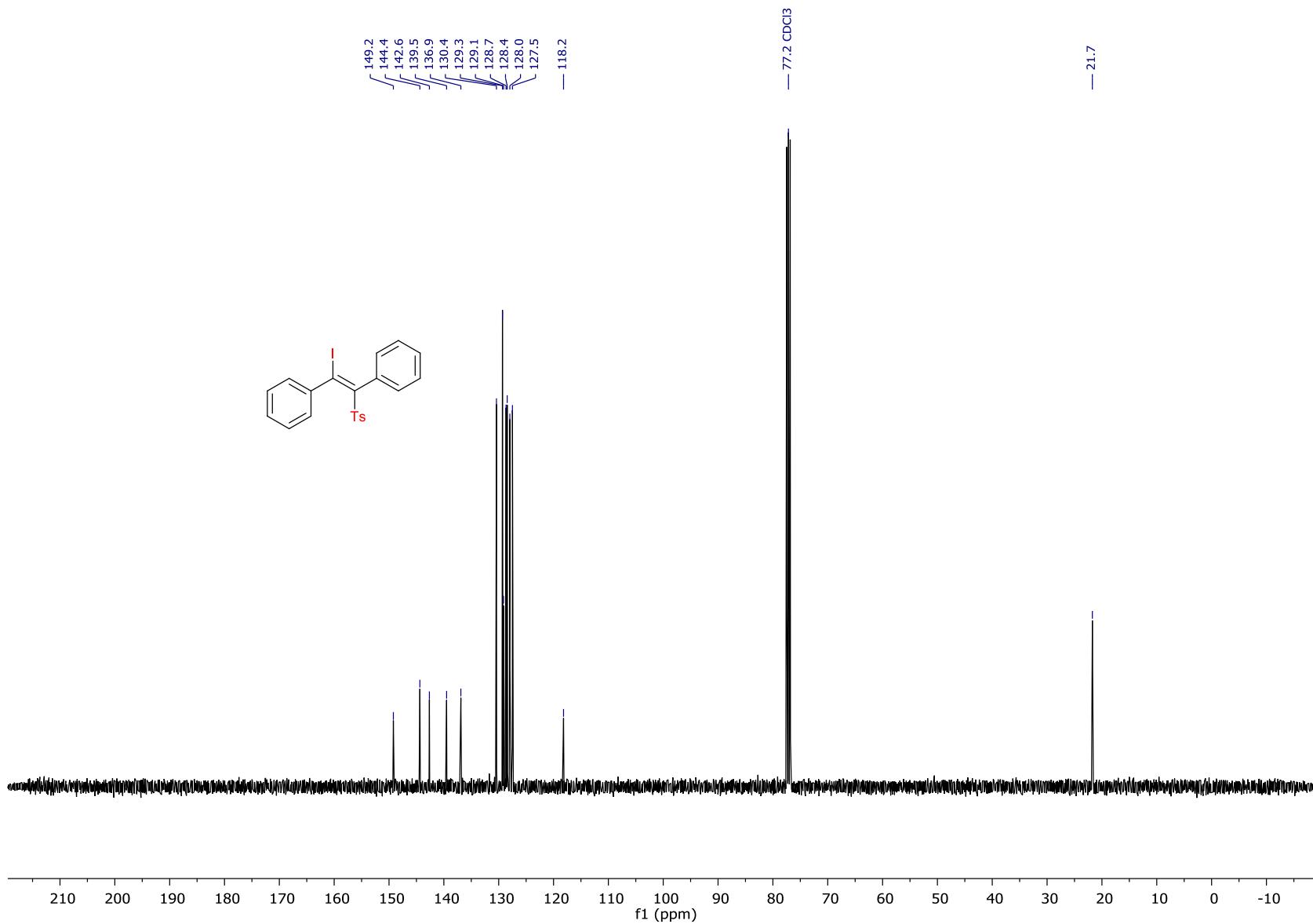


Figure S28. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-(1-iodo-2-tosylethene-1,2-diyl)dibenzene (3a).

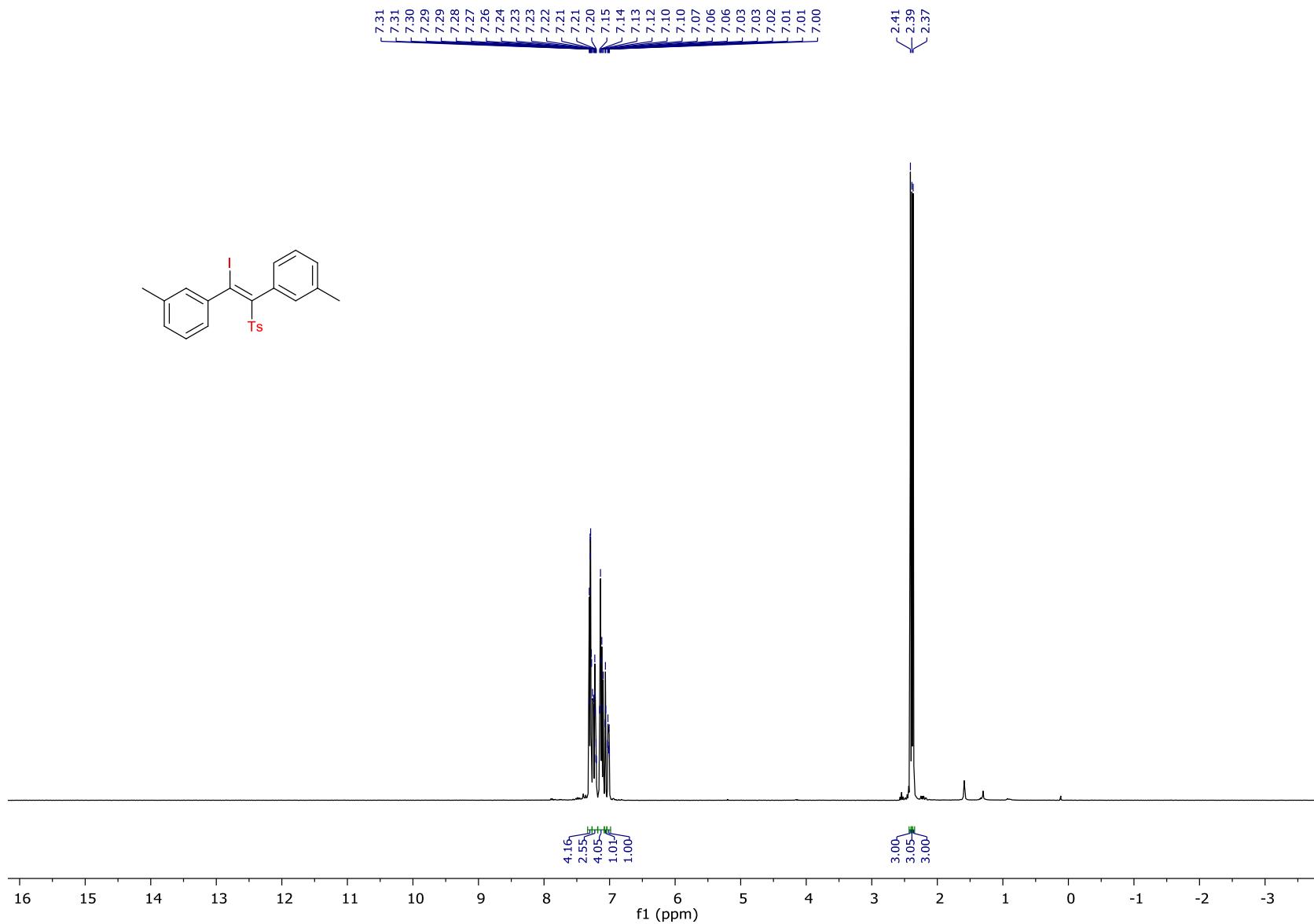


Figure S29. ^1H NMR (600 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3b).

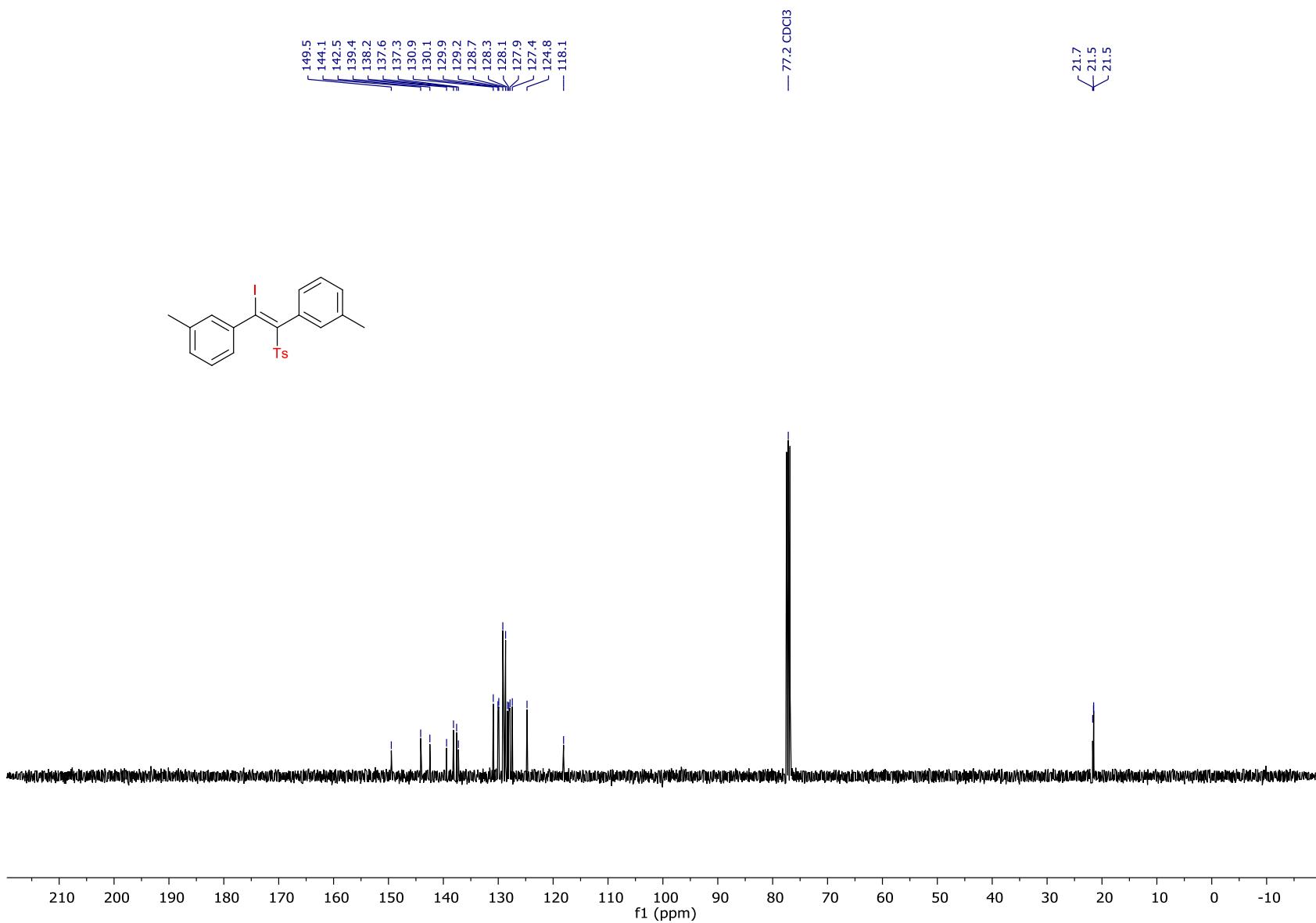


Figure S30. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (*E*)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methylbenzene) (3b).

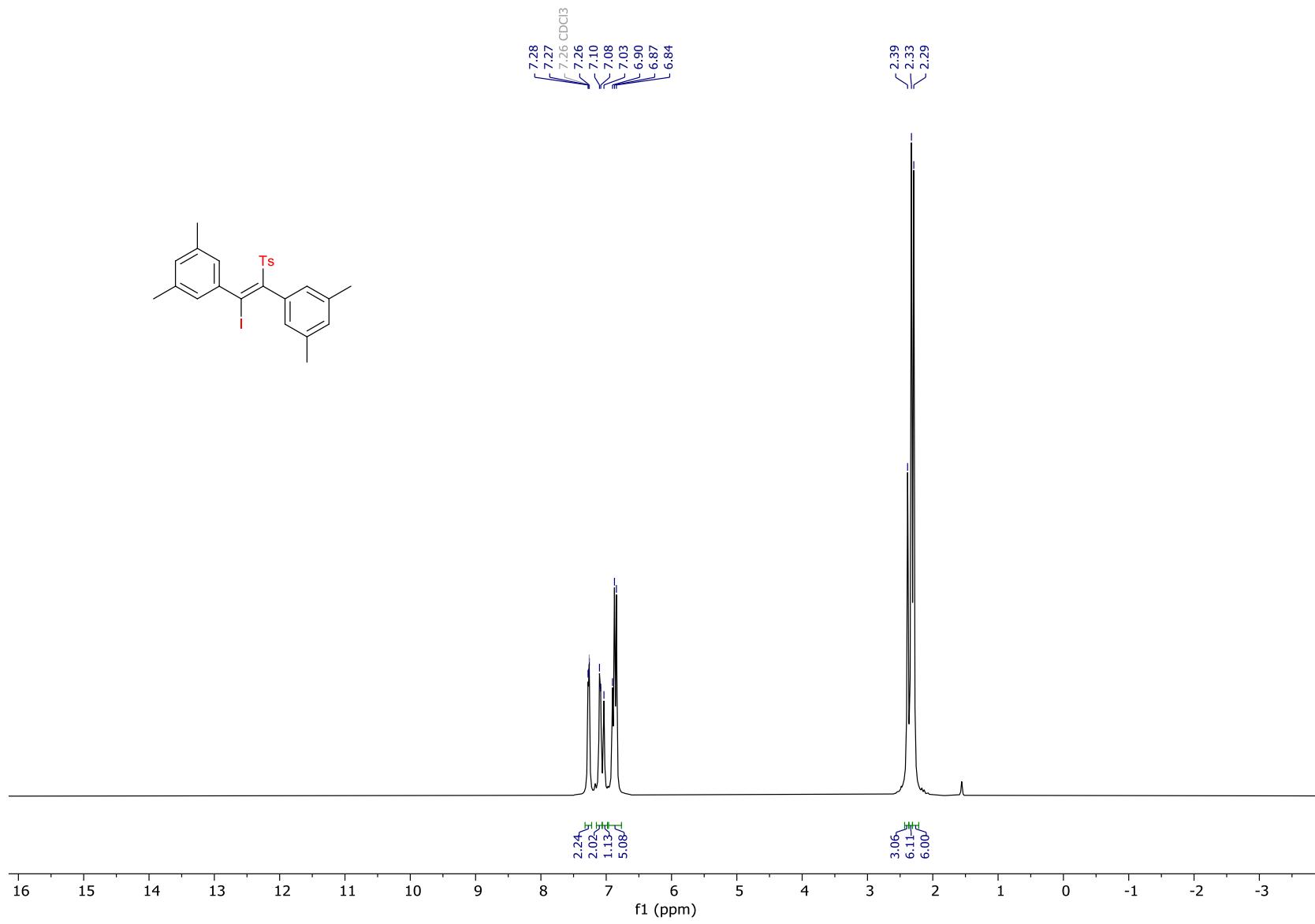


Figure S31. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-5,5'-(1-iodo-2-tosylethene-1,2-diyl)bis(1,3-dimethylbenzene) (**3c**).

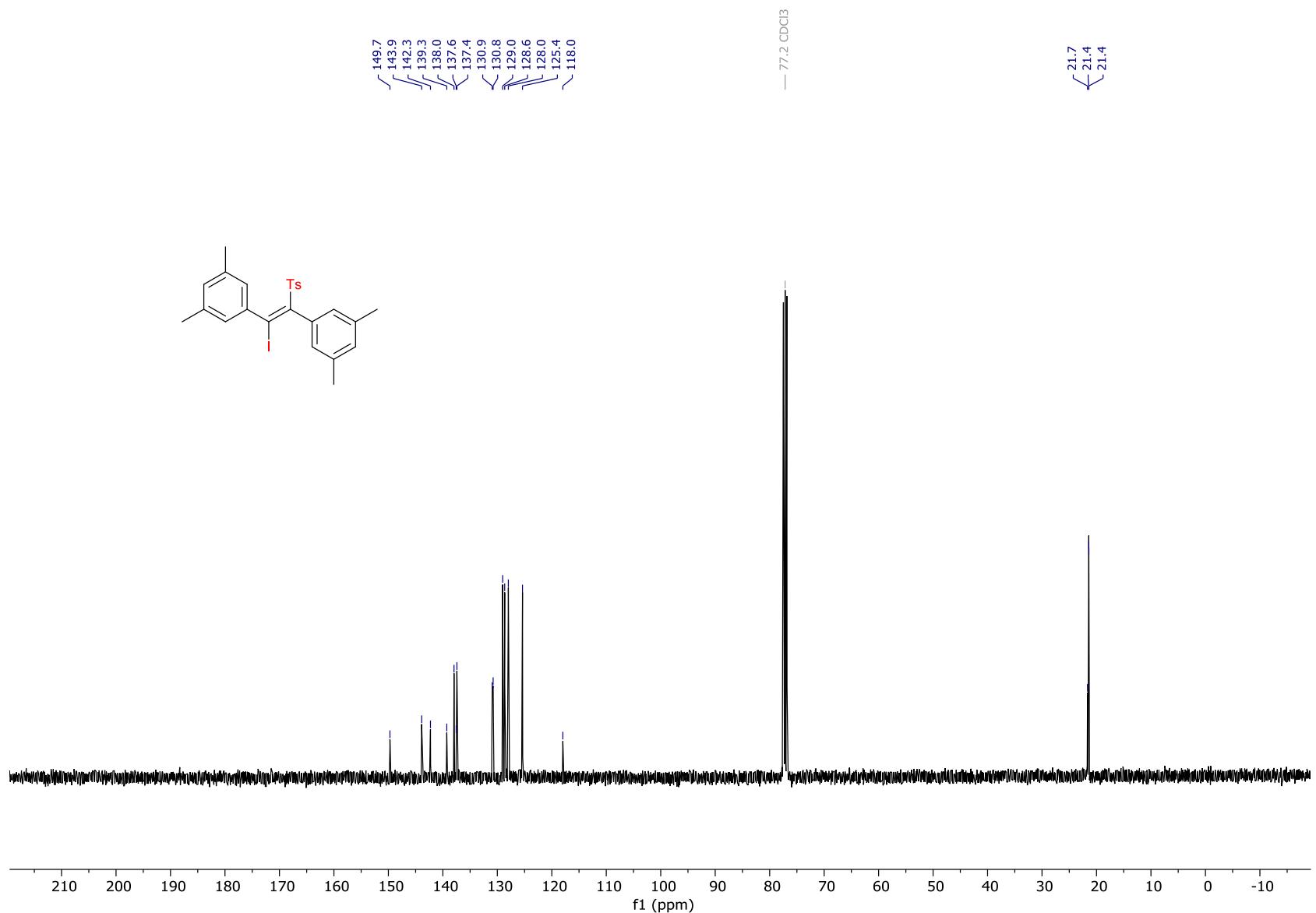


Figure S32. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-5,5'-(1-iodo-2-tosylethene-1,2-diyl)bis(1,3-dimethylbenzene) (3c).

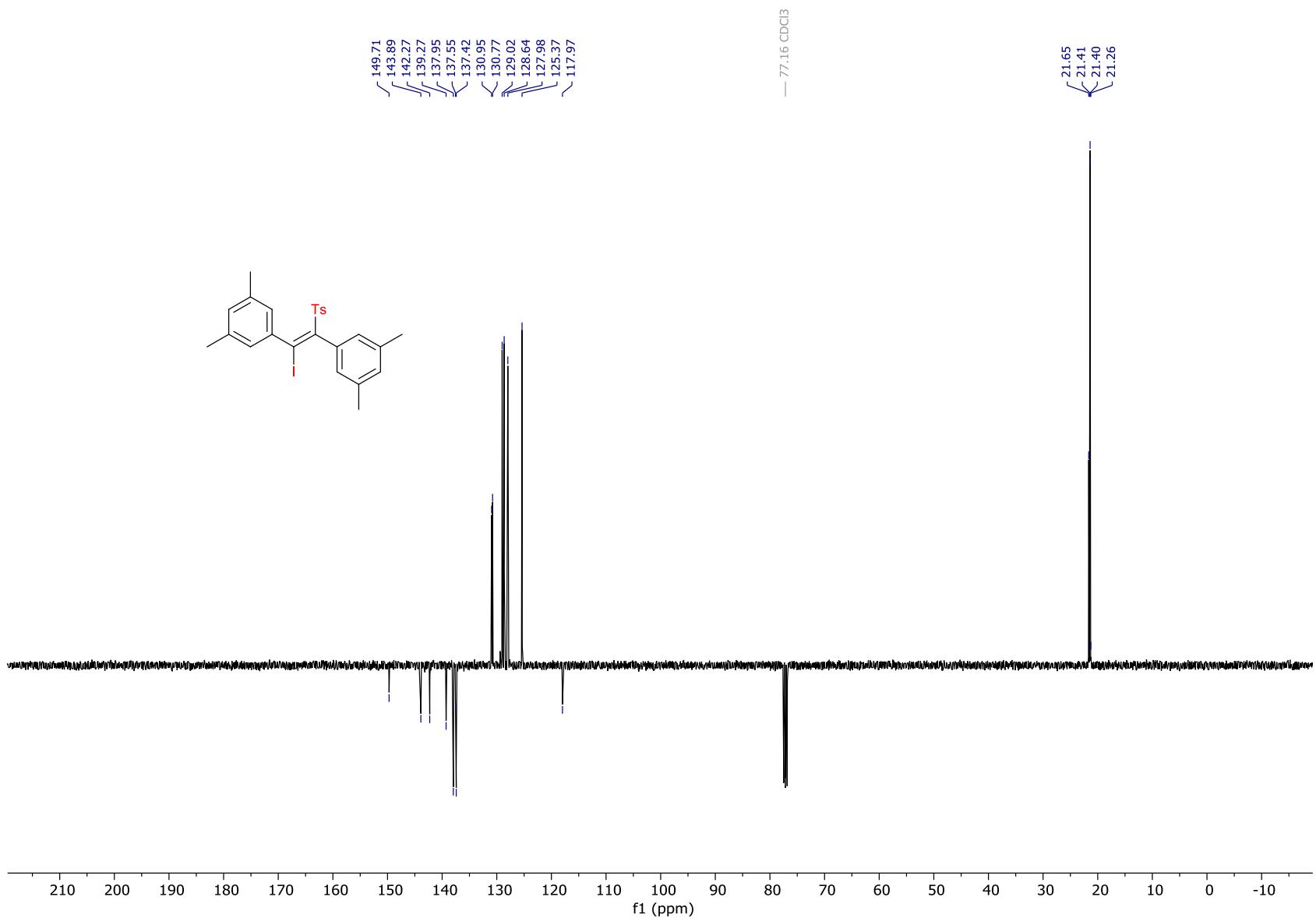


Figure S33. ^{13}C DEPTQ-135 NMR (*E*)-5,5'-(1-iodo-2-tosylethene-1,2-diyl)bis(1,3-dimethylbenzene) (**3c**).

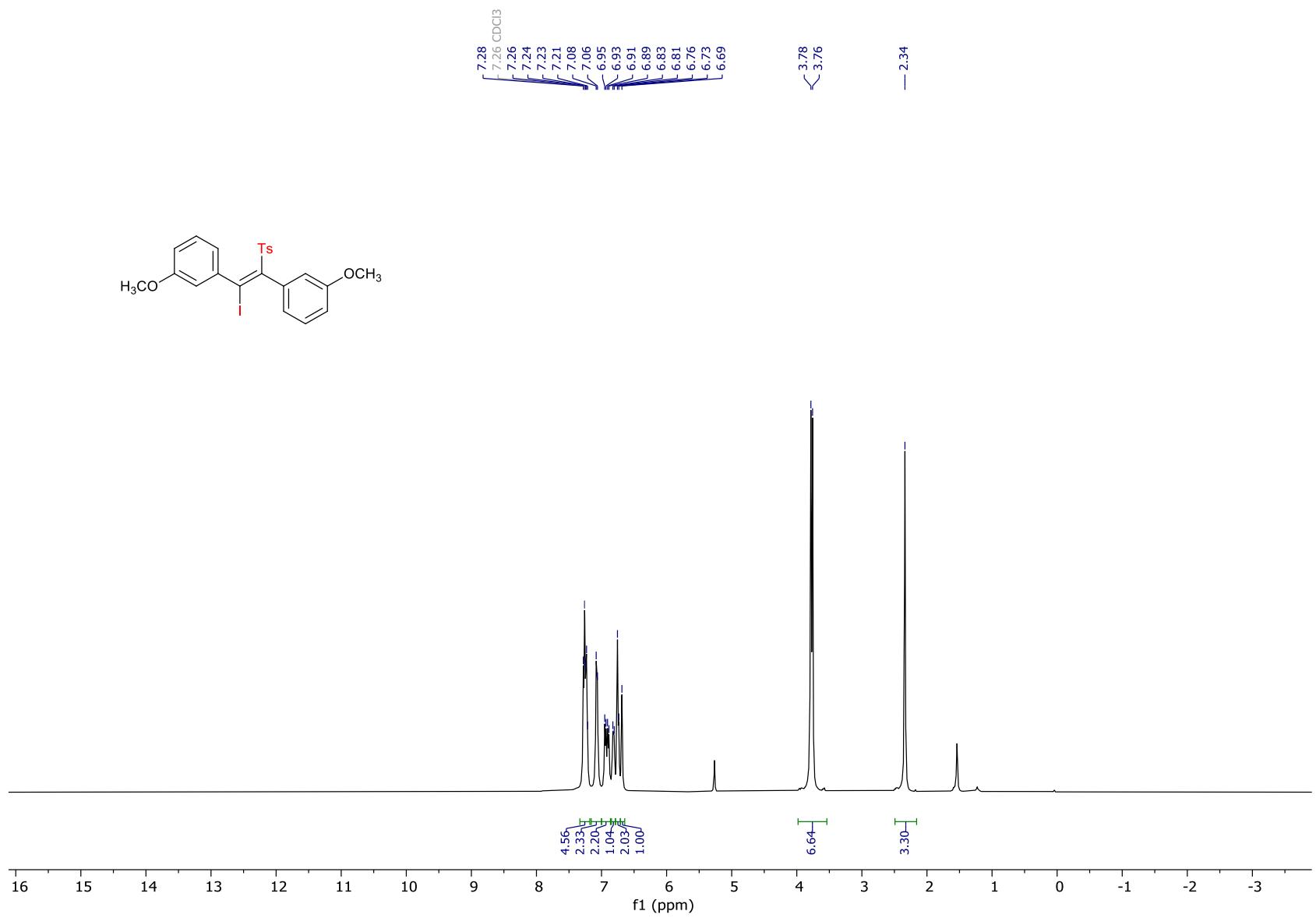


Figure S34. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methoxybenzene) (3d).

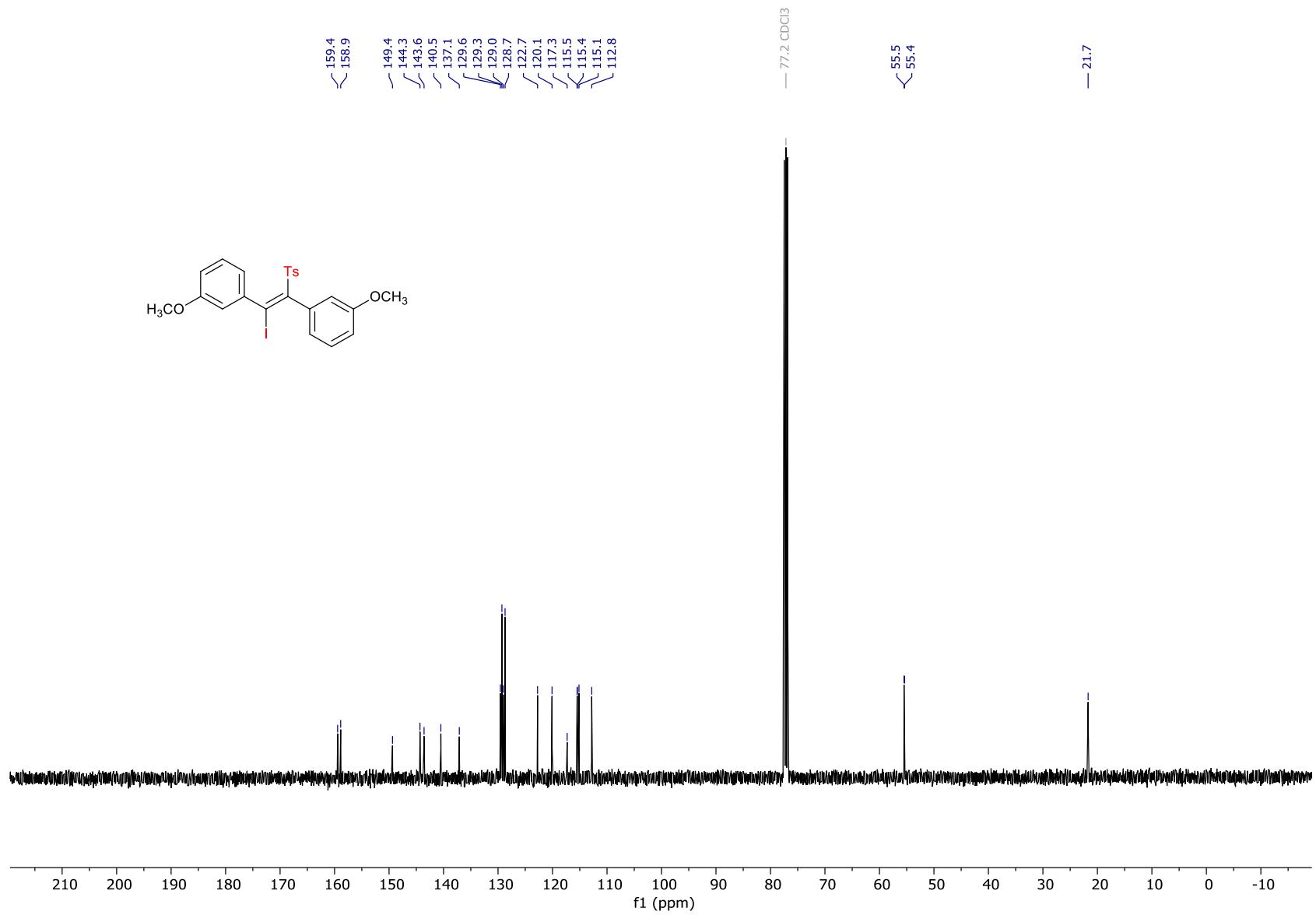
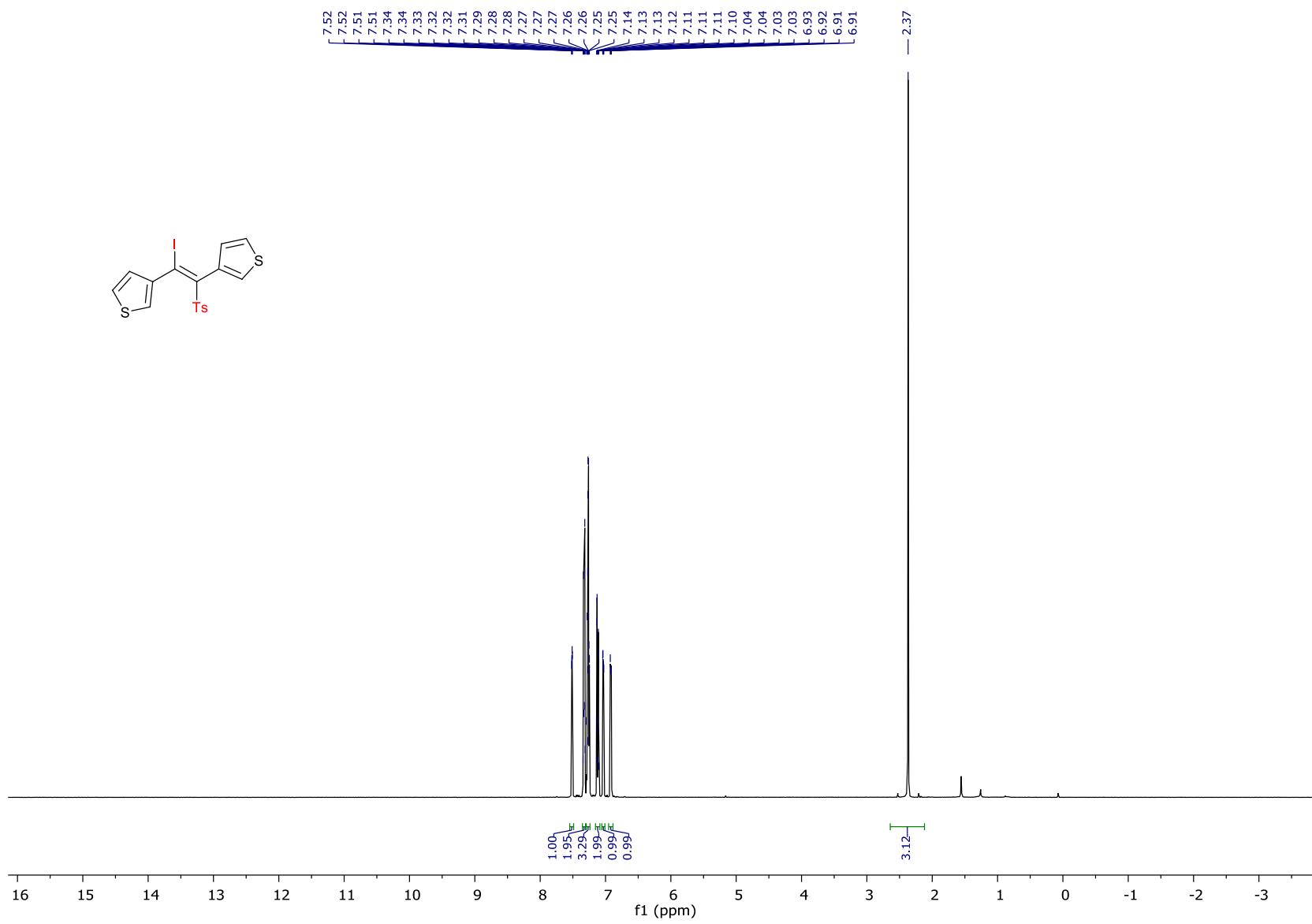


Figure S35. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (*E*)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)bis(methoxybenzene) (**3d**).



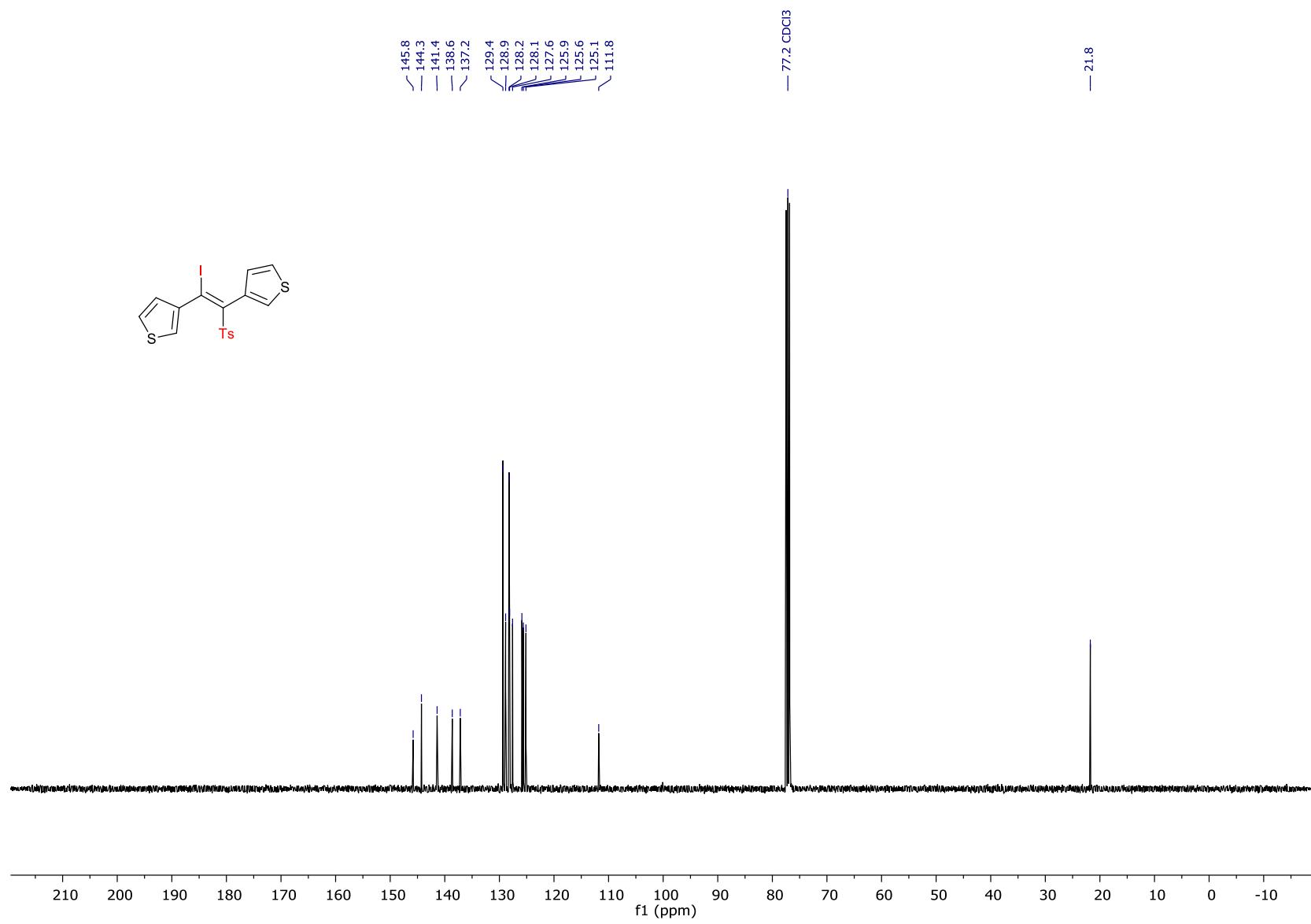


Figure S37. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-3,3'-(1-iodo-2-tosylethene-1,2-diyl)dithiophene (3e).

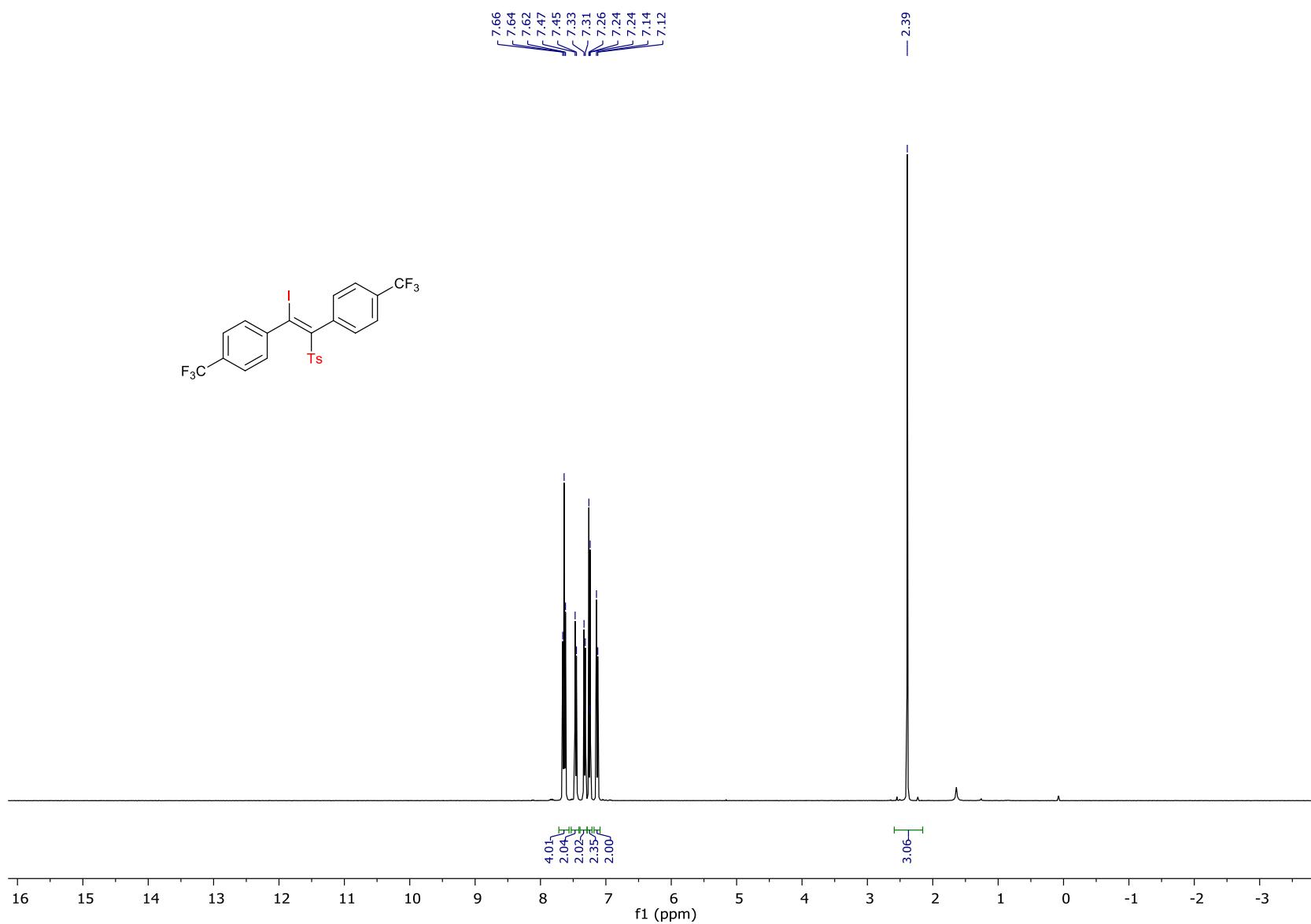


Figure S38. ^1H NMR (600 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (**3f**).

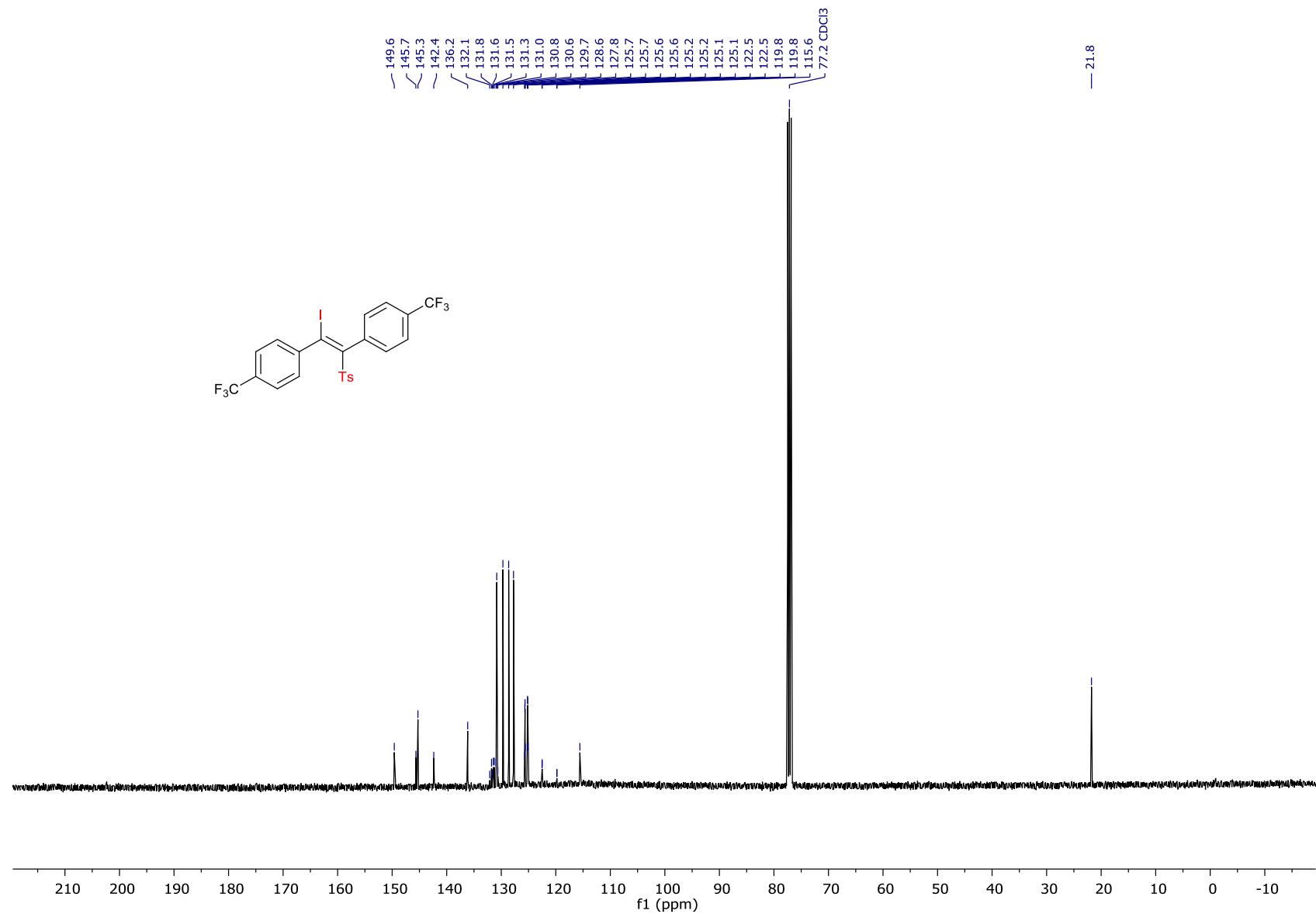


Figure S39. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (3f).

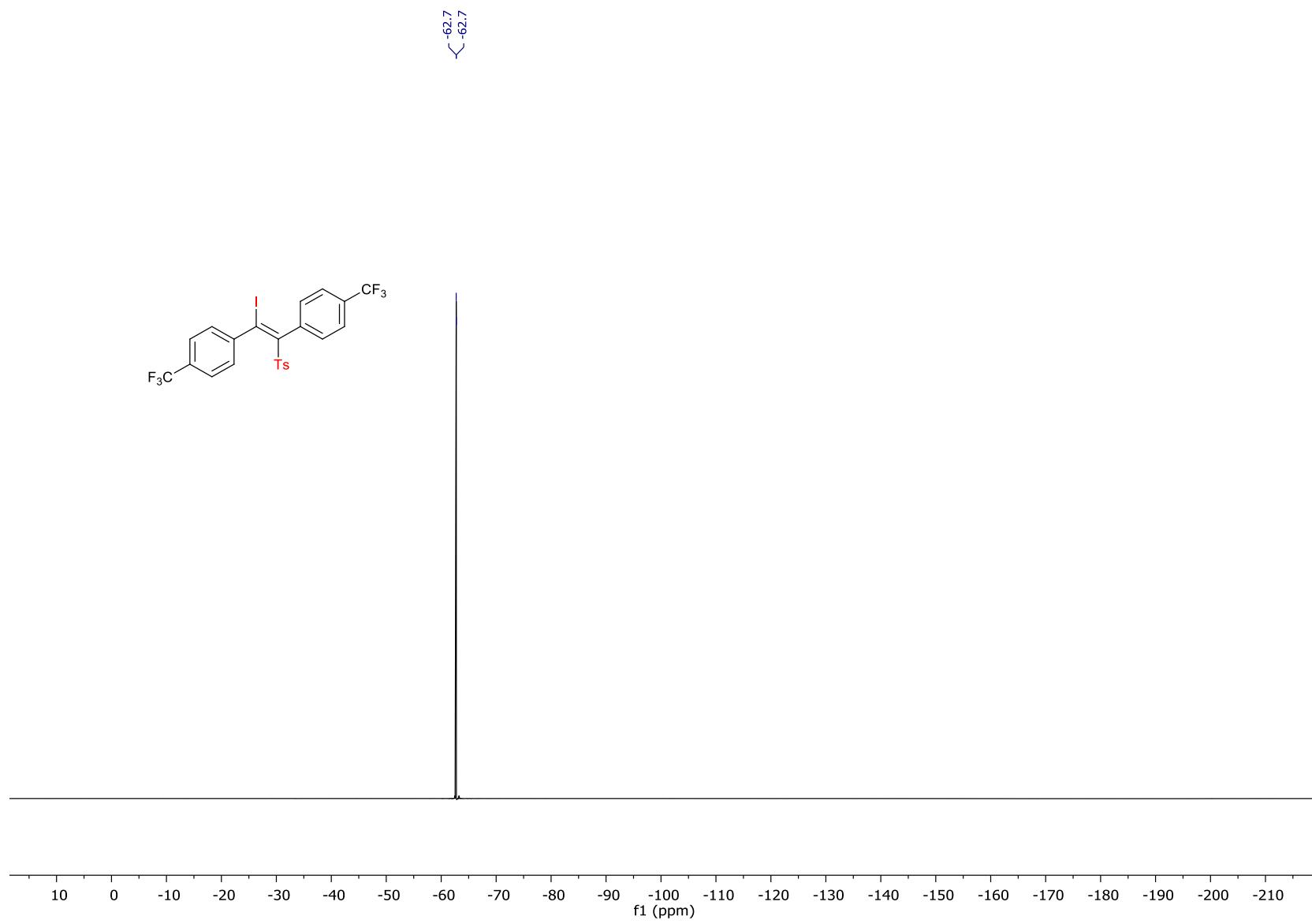


Figure S40. ^{19}F NMR (188 MHz, Chloroform-*d*) of (*E*)-4,4'-(1-iodo-2-tosylethene-1,2-diyl)bis((trifluoromethyl)benzene) (**3f**).

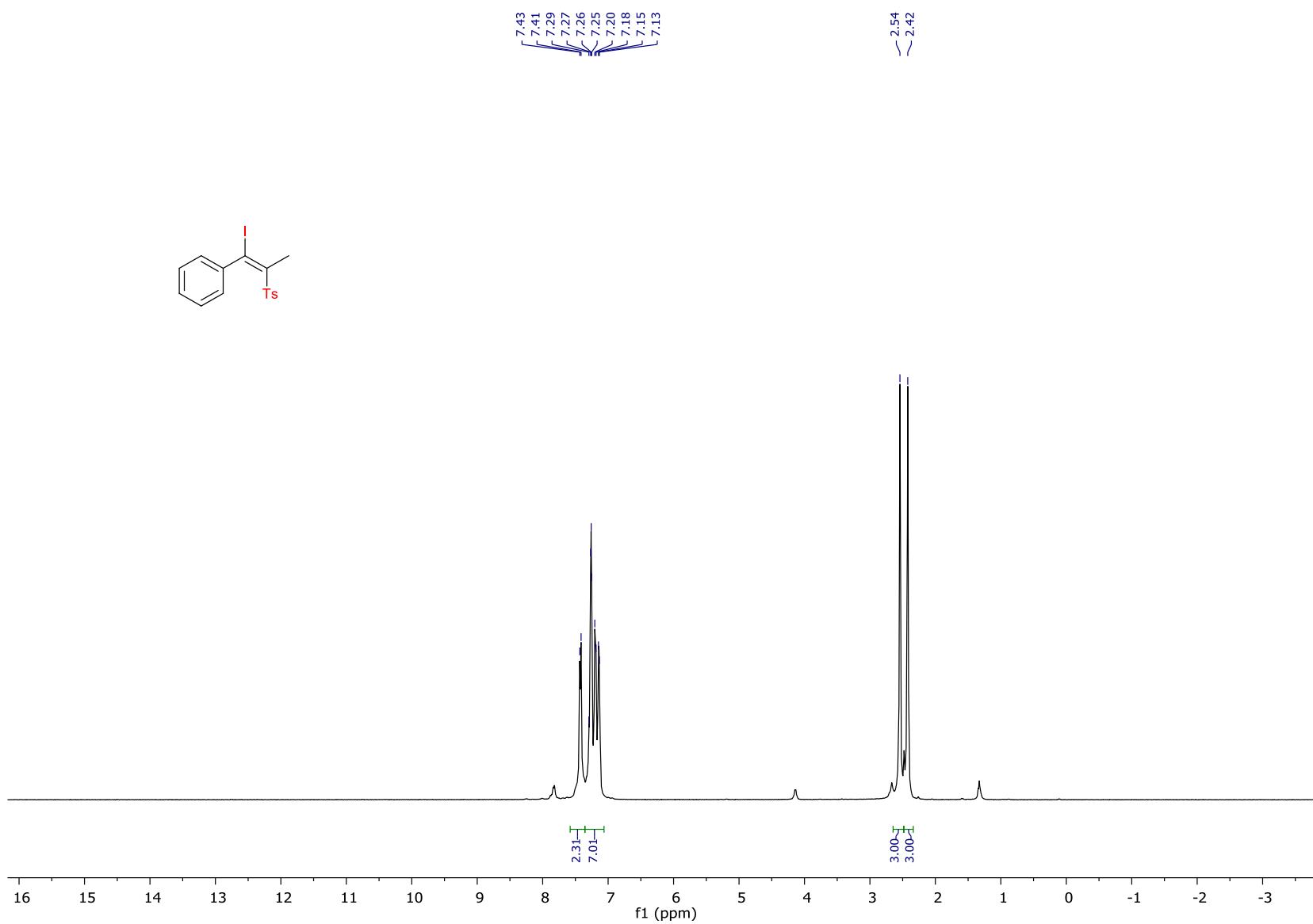


Figure S41. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (**3g**).

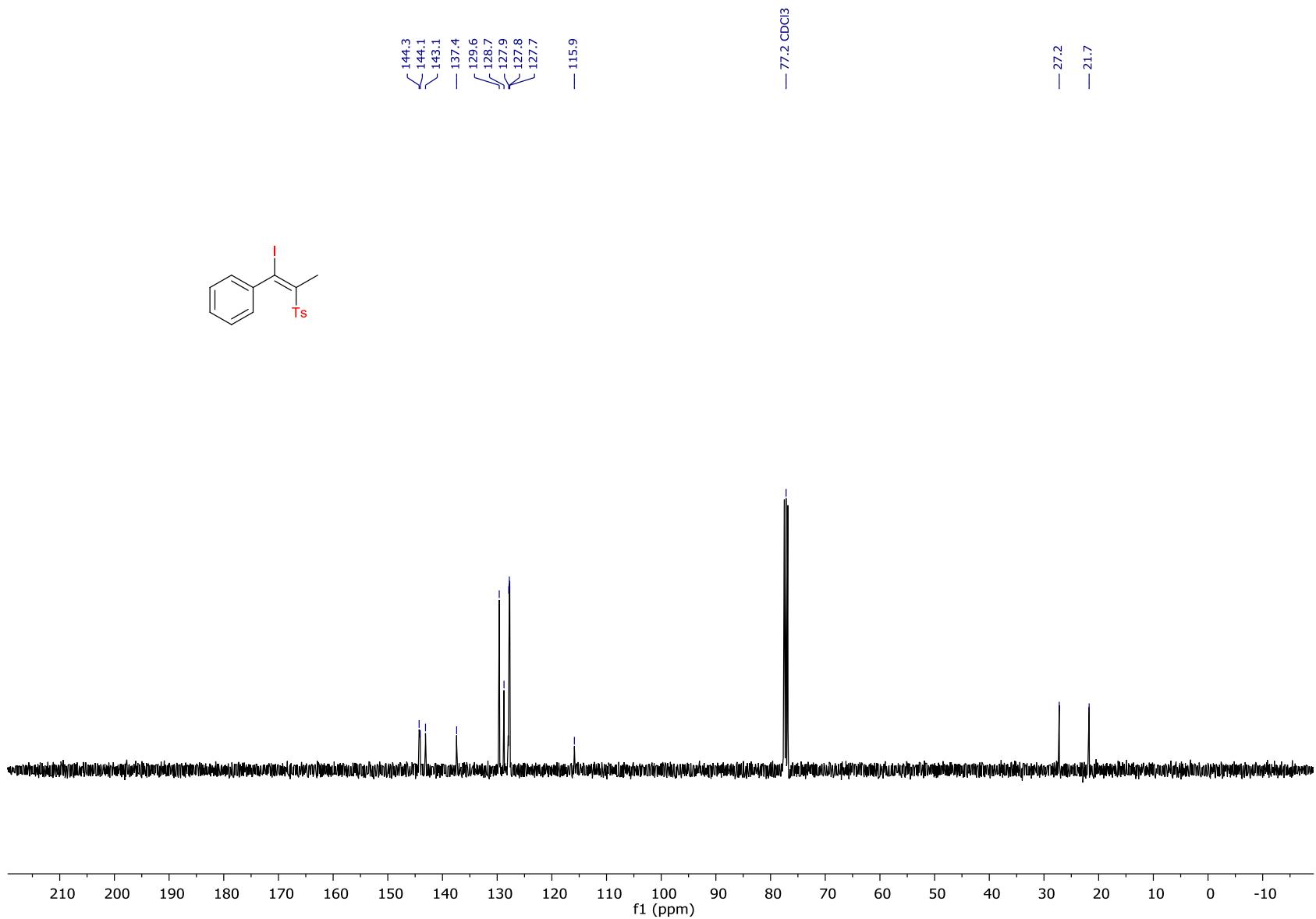


Figure S42. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylprop-1-en-2-ylsulfonyl)-4-methylbenzene (3g).

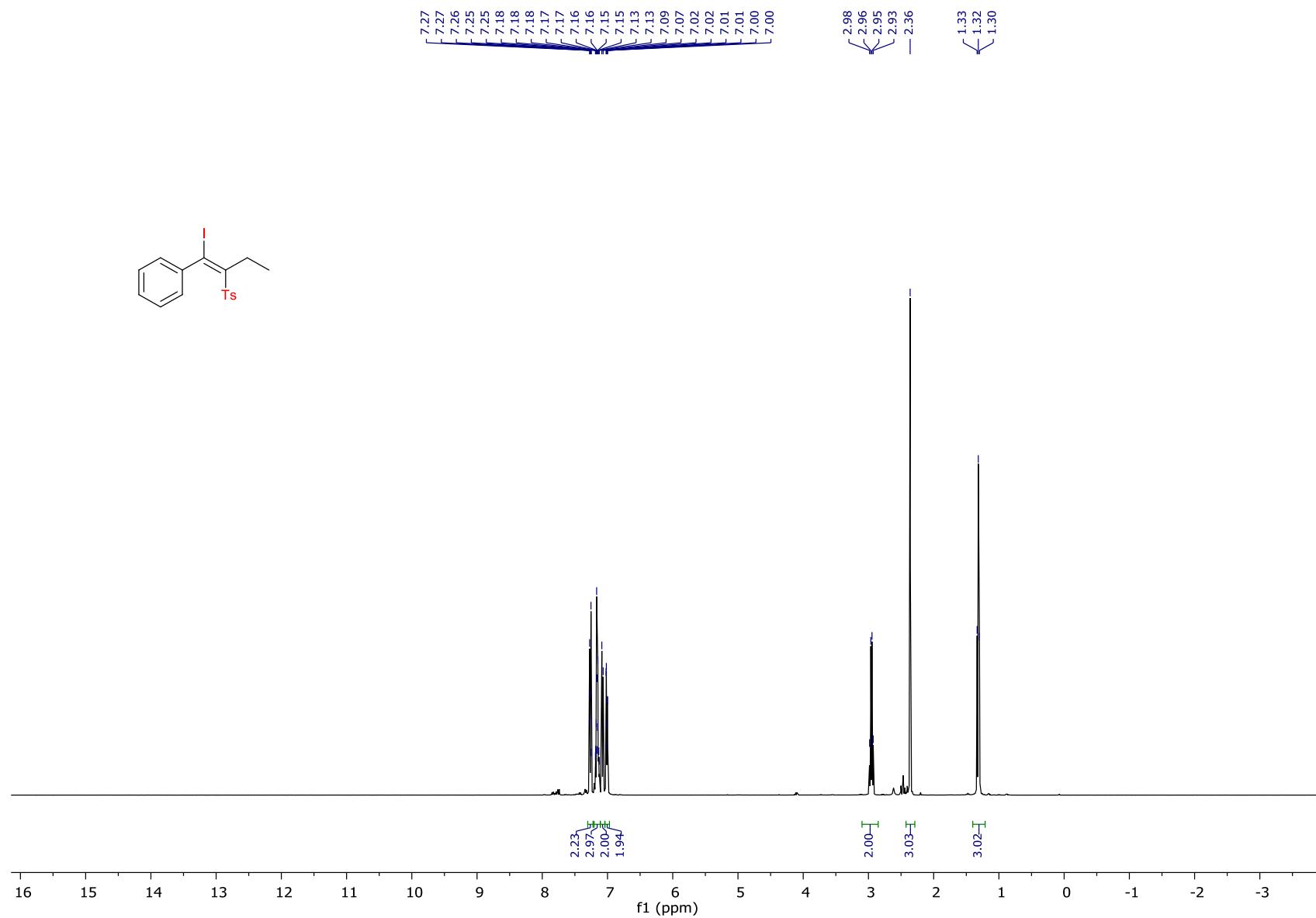


Figure S43. ^1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylbut-1-en-2-ylsulfonyl)-4-methylbenzene (3h).

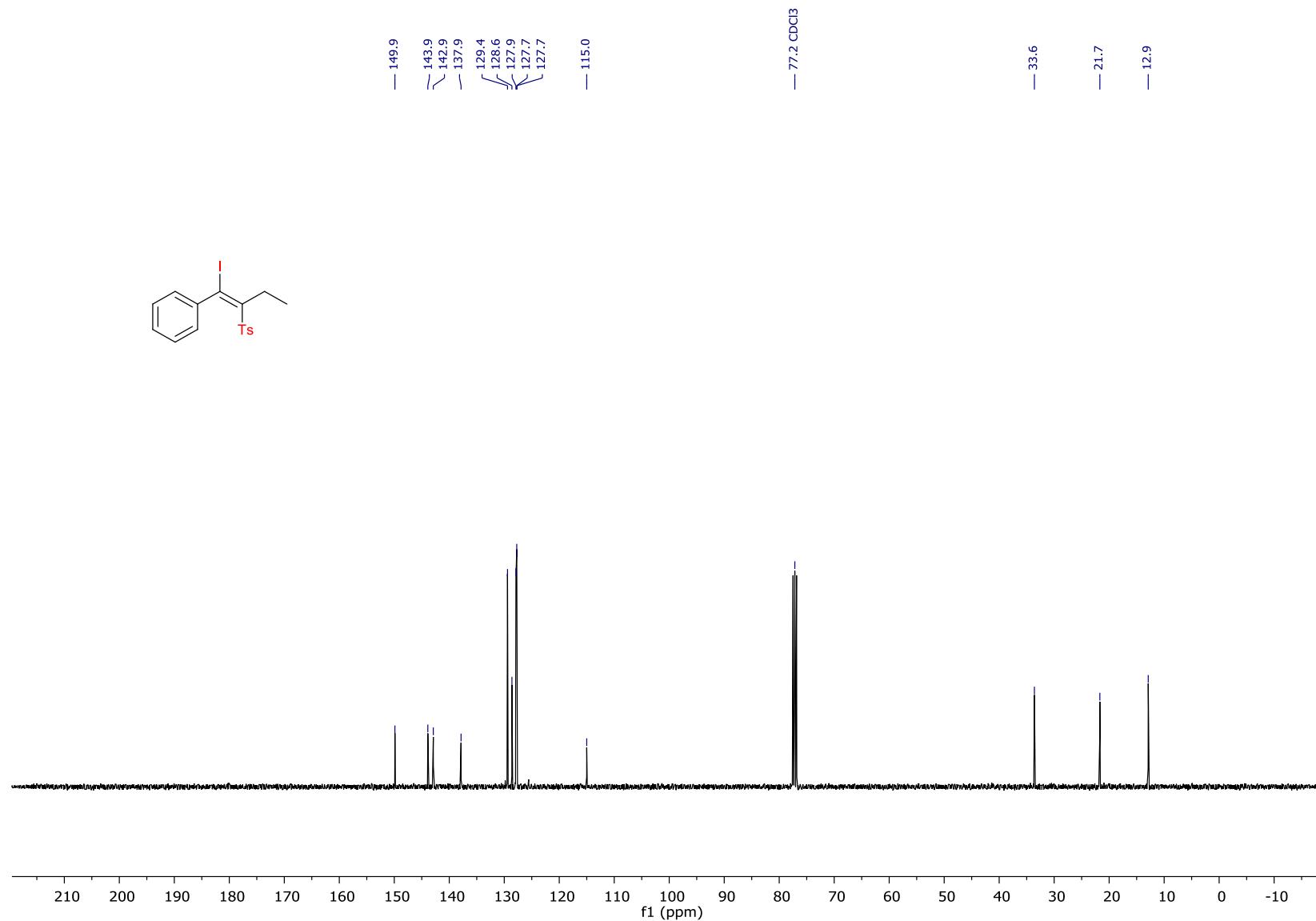


Figure S44. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-1-phenylbut-1-en-2-ylsulfonyl)-4-methylbenzene (3g).

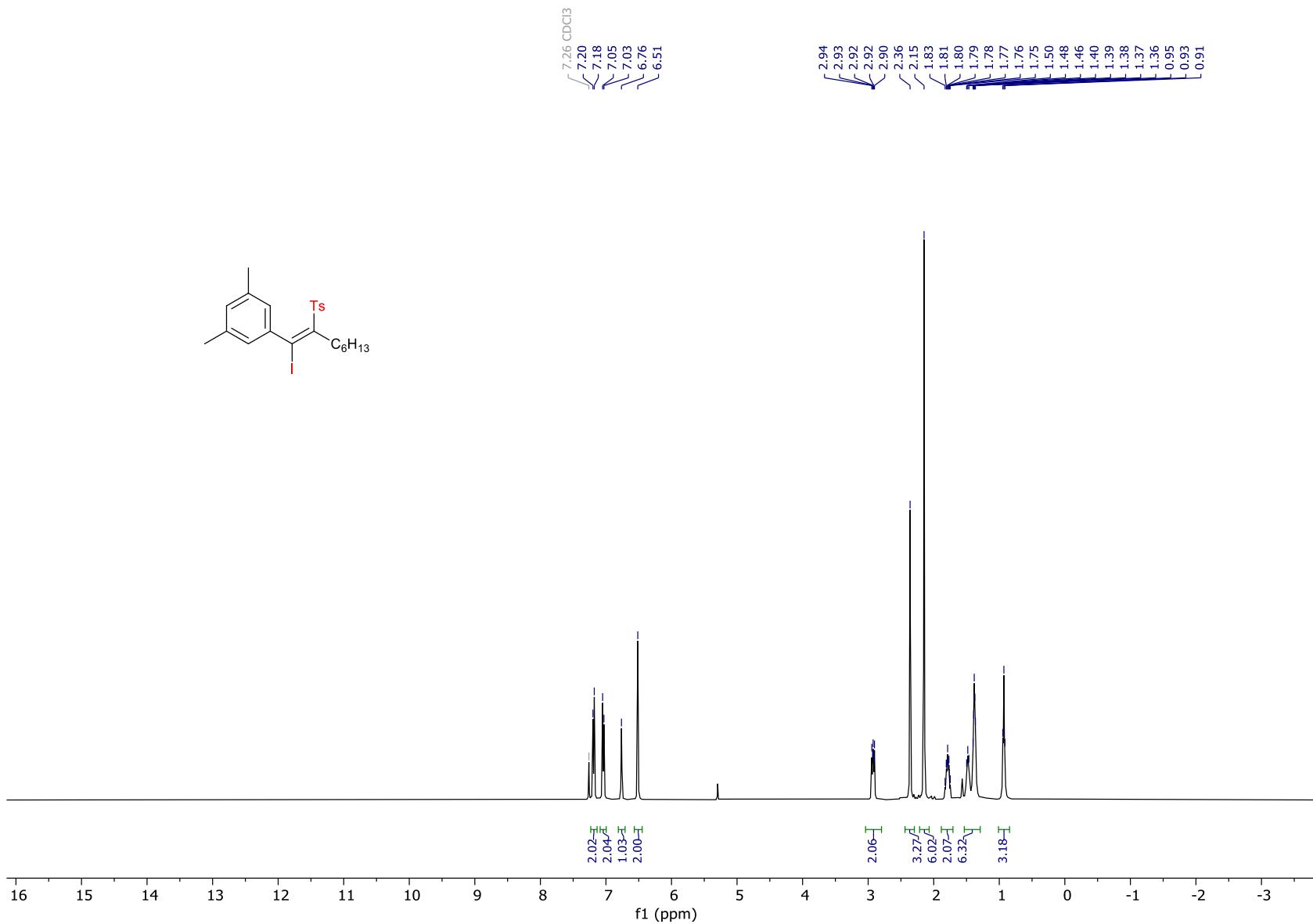


Figure S45. **1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3,5-dimethylbenzene (3i).**

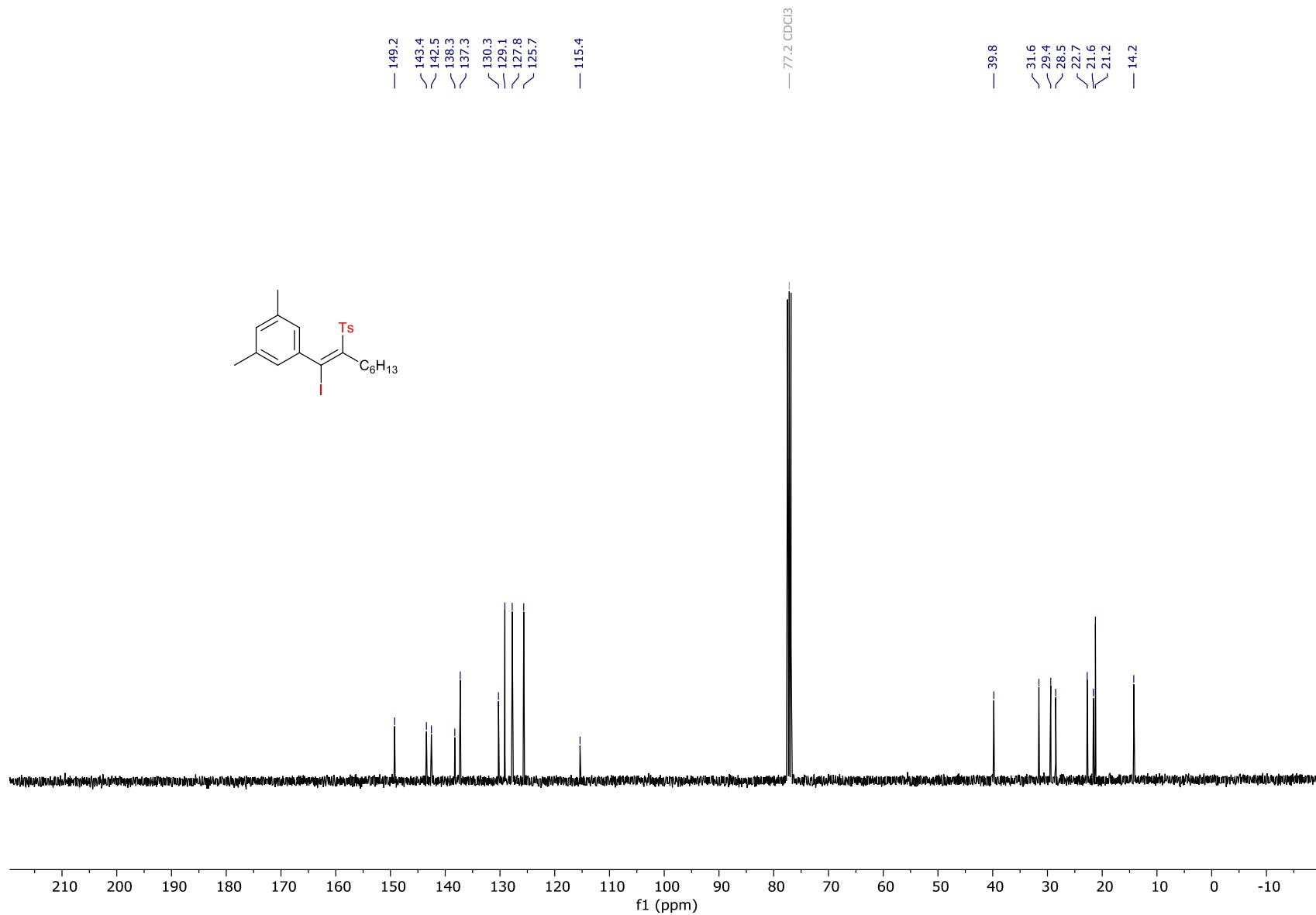


Figure S46. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3,5-dimethylbenzene (3i).

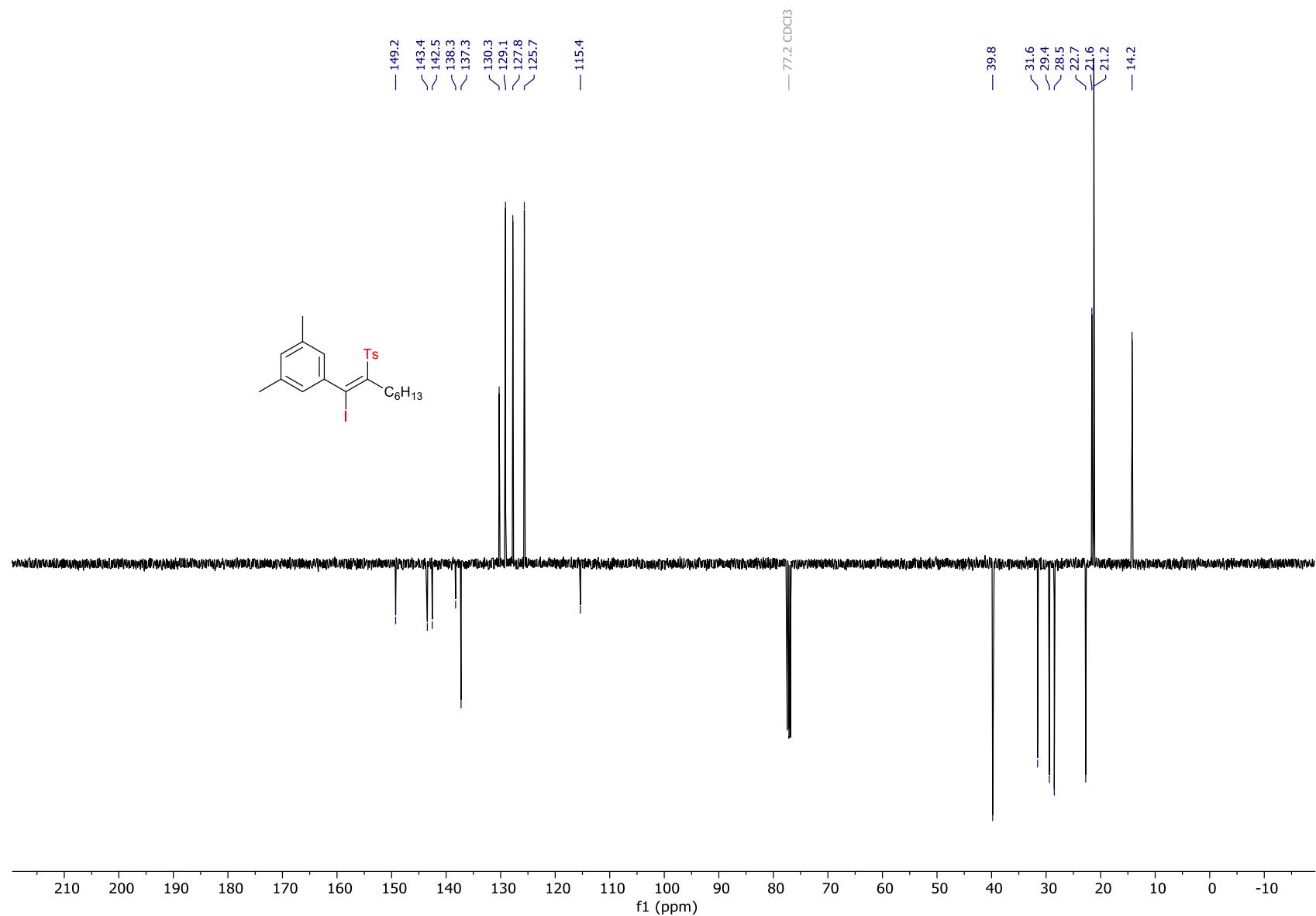


Figure S47. ^{13}C DEPTQ-135 NMR (*E*)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3,5-dimethylbenzene (**3i**).

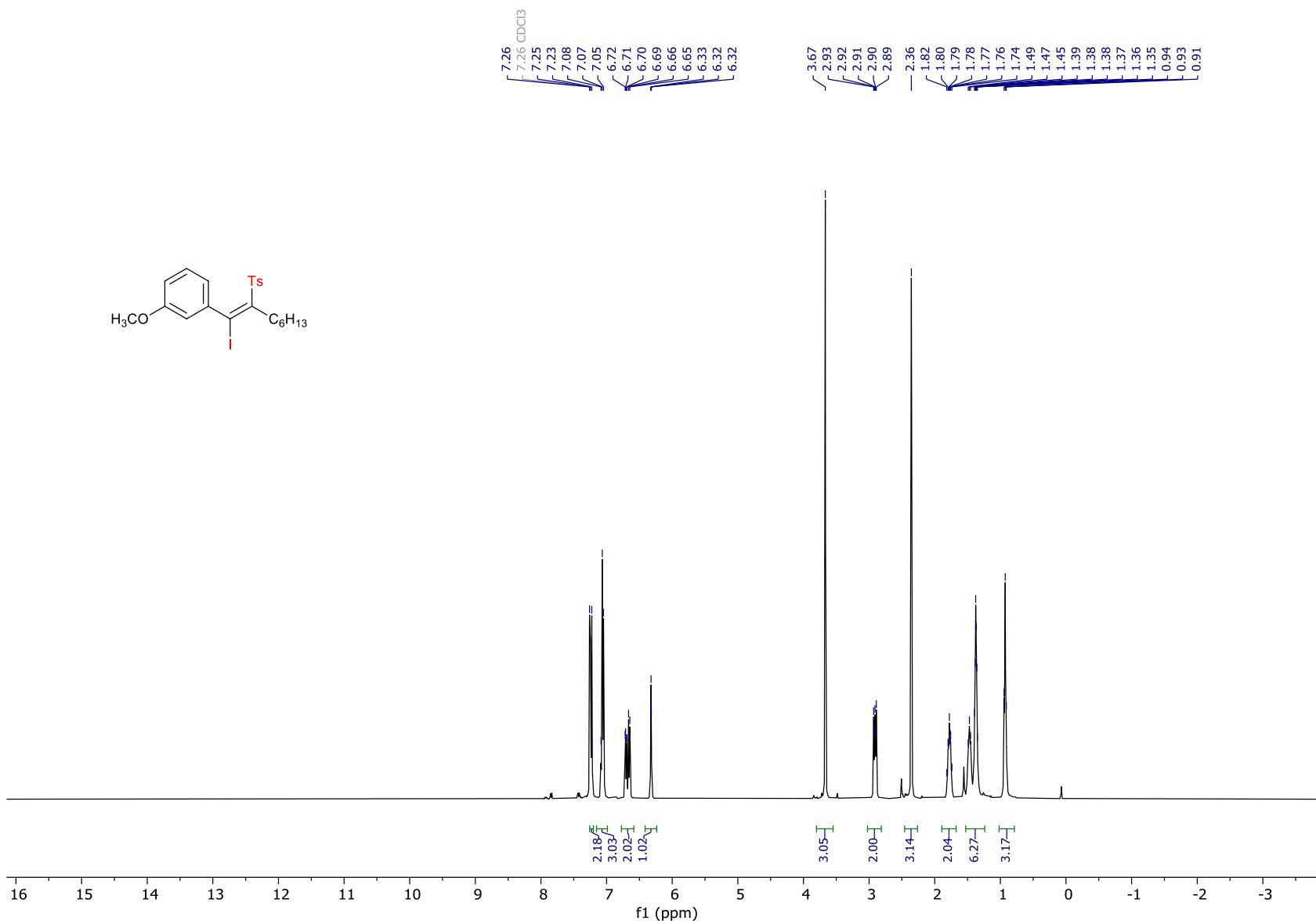


Figure S48. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3-methoxybenzene (3j).

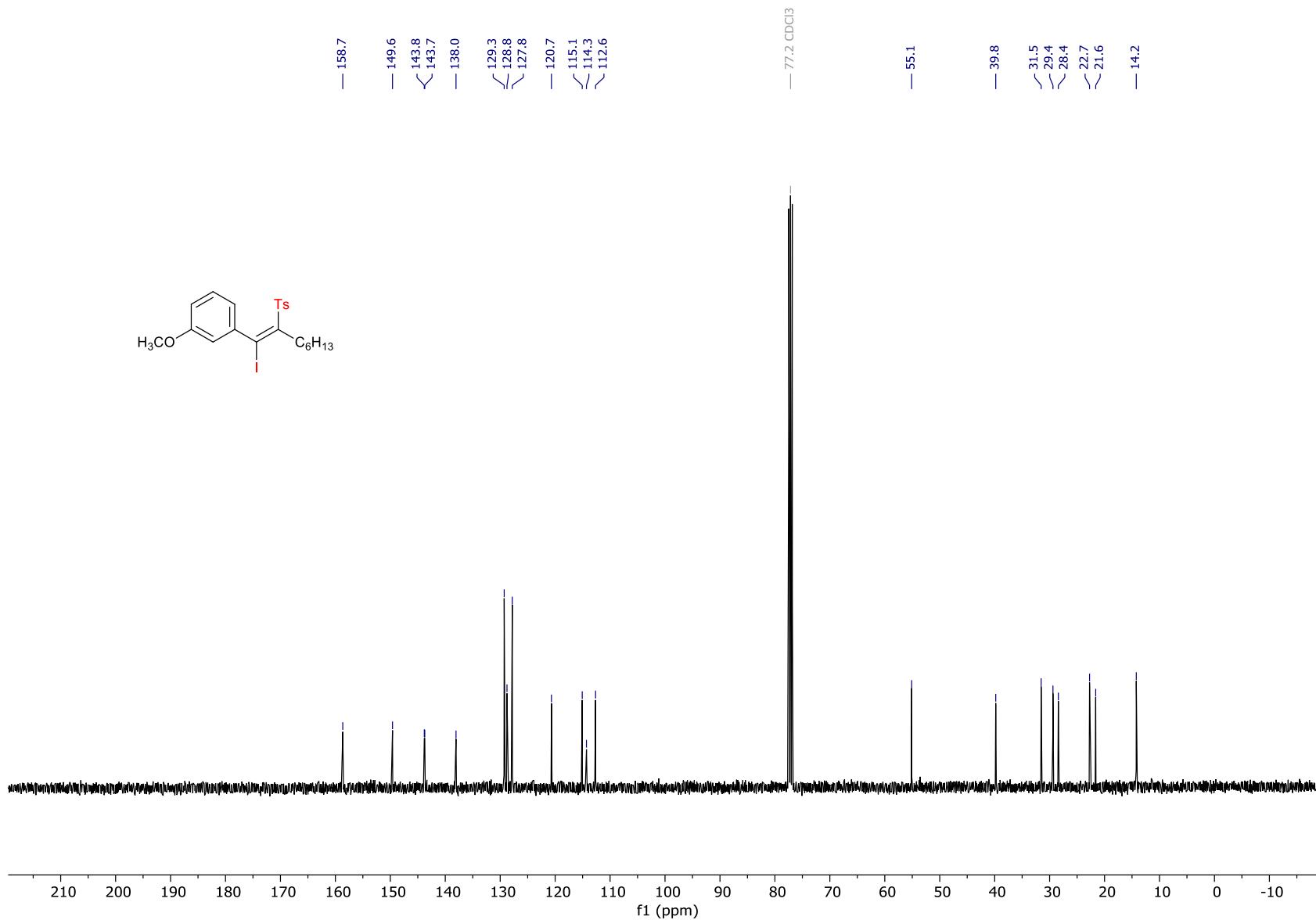


Figure S49. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (*E*)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3-methoxybenzene (3j).

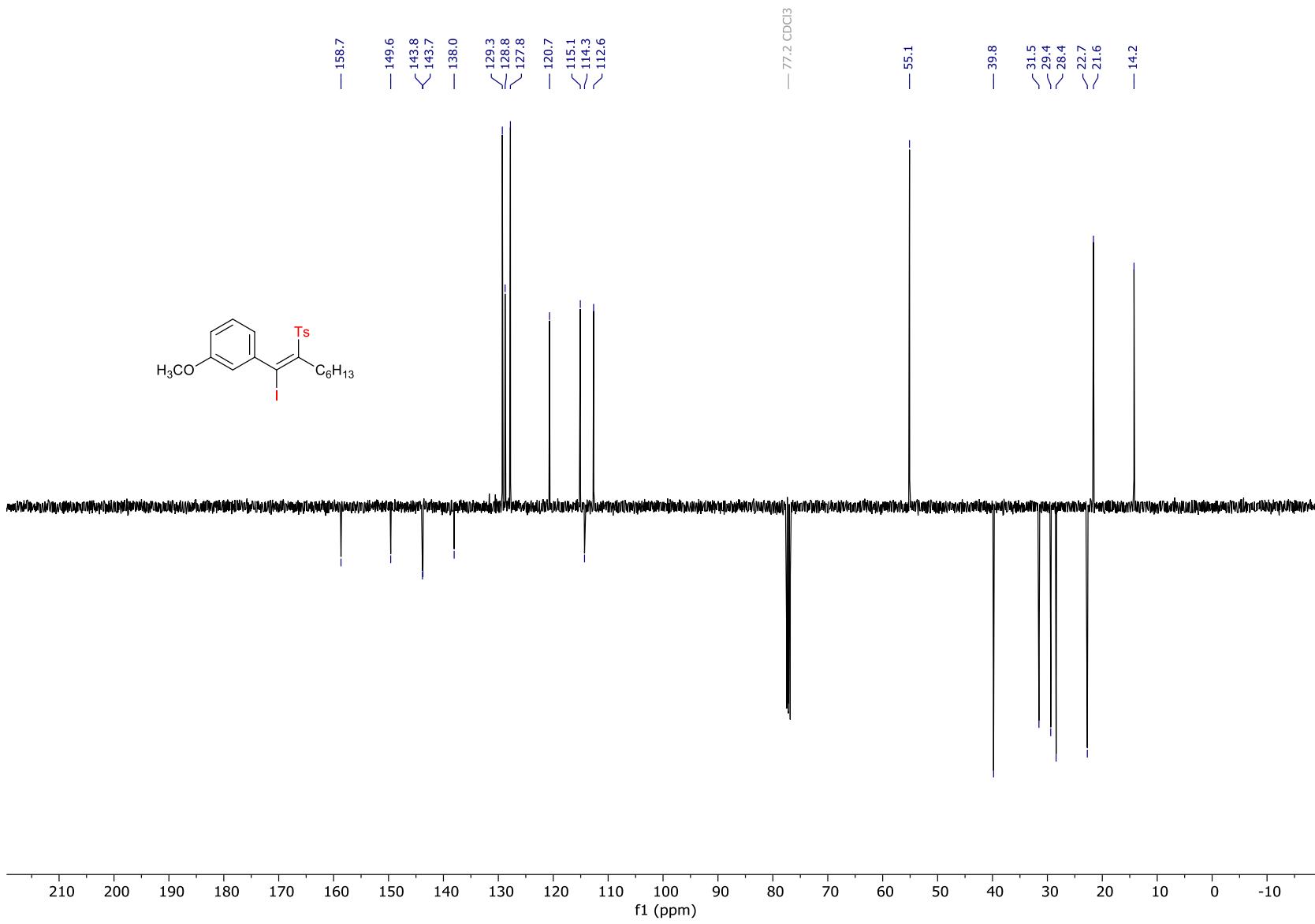


Figure S50. ^{13}C DEPTQ-135 NMR (*E*)-1-(1-iodo-2-tosyloct-1-en-1-yl)-3-methoxybenzene (**3j**).

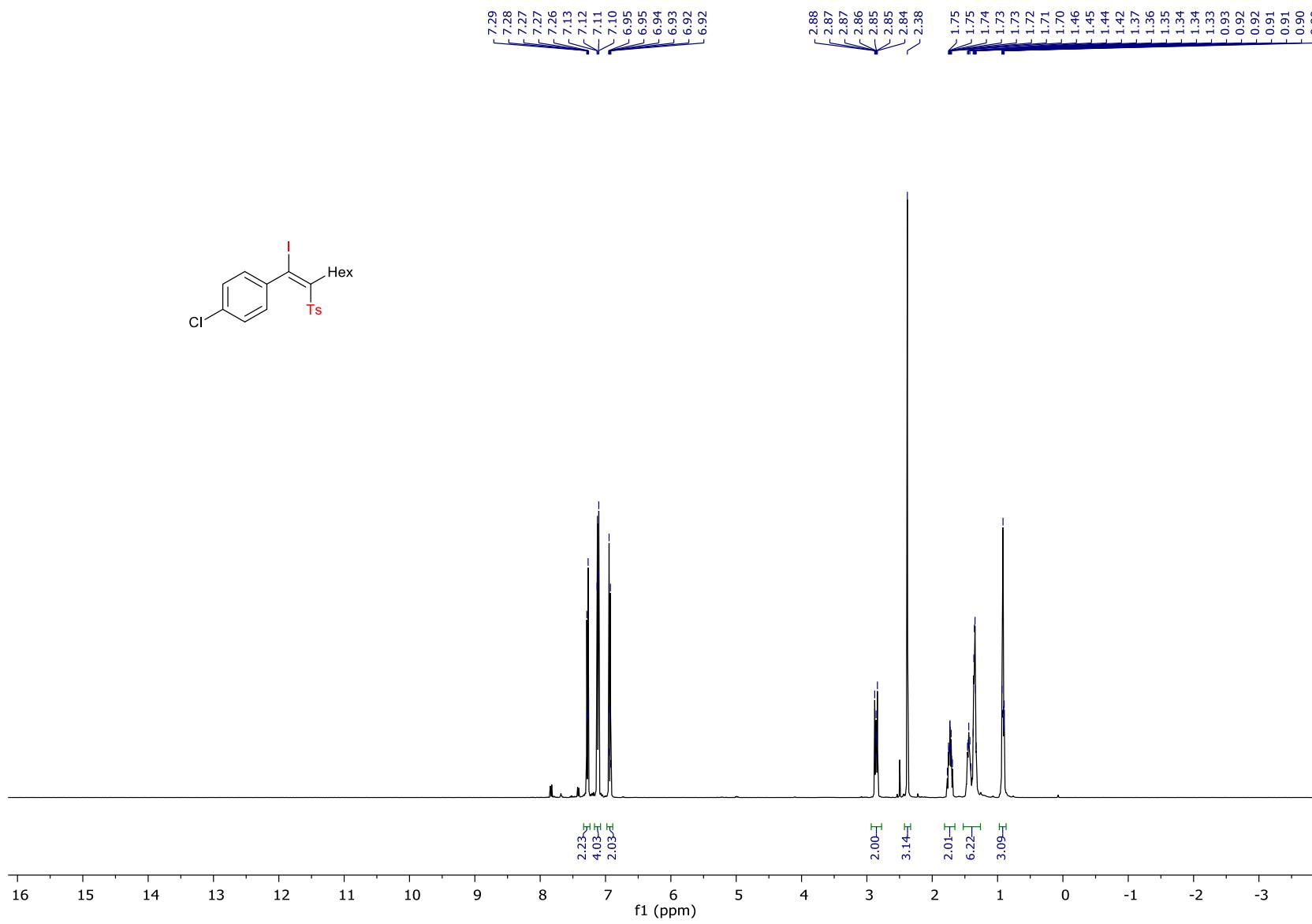


Figure S51. ^1H NMR (600 MHz, Chloroform-d) of (E)-1-chloro-4-(1-iodo-2-tosyloct-1-enyl)benzene (3k).

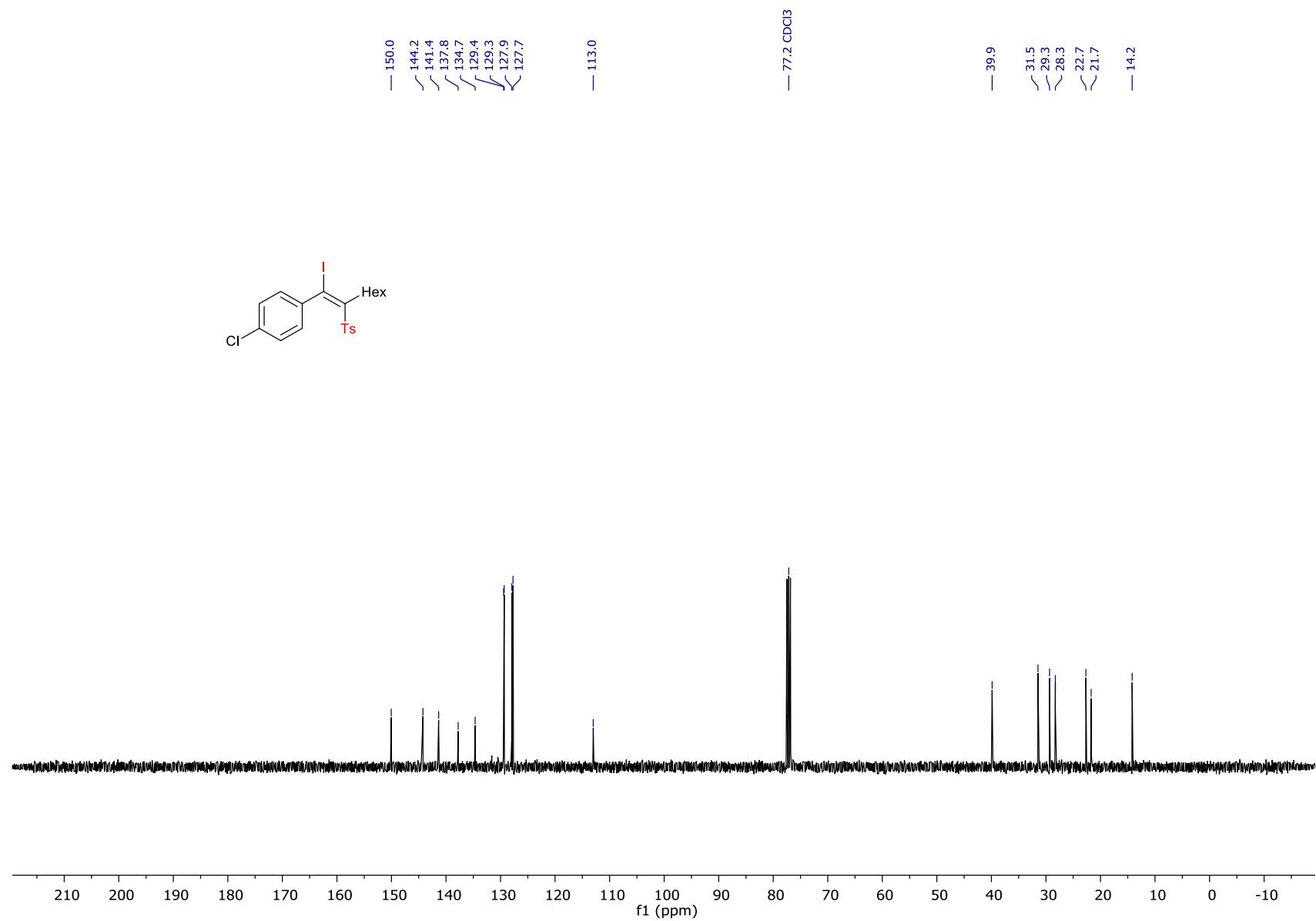


Figure S52. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-1-chloro-4-(1-iodo-2-tosyloct-1-enyl)benzene (3k).

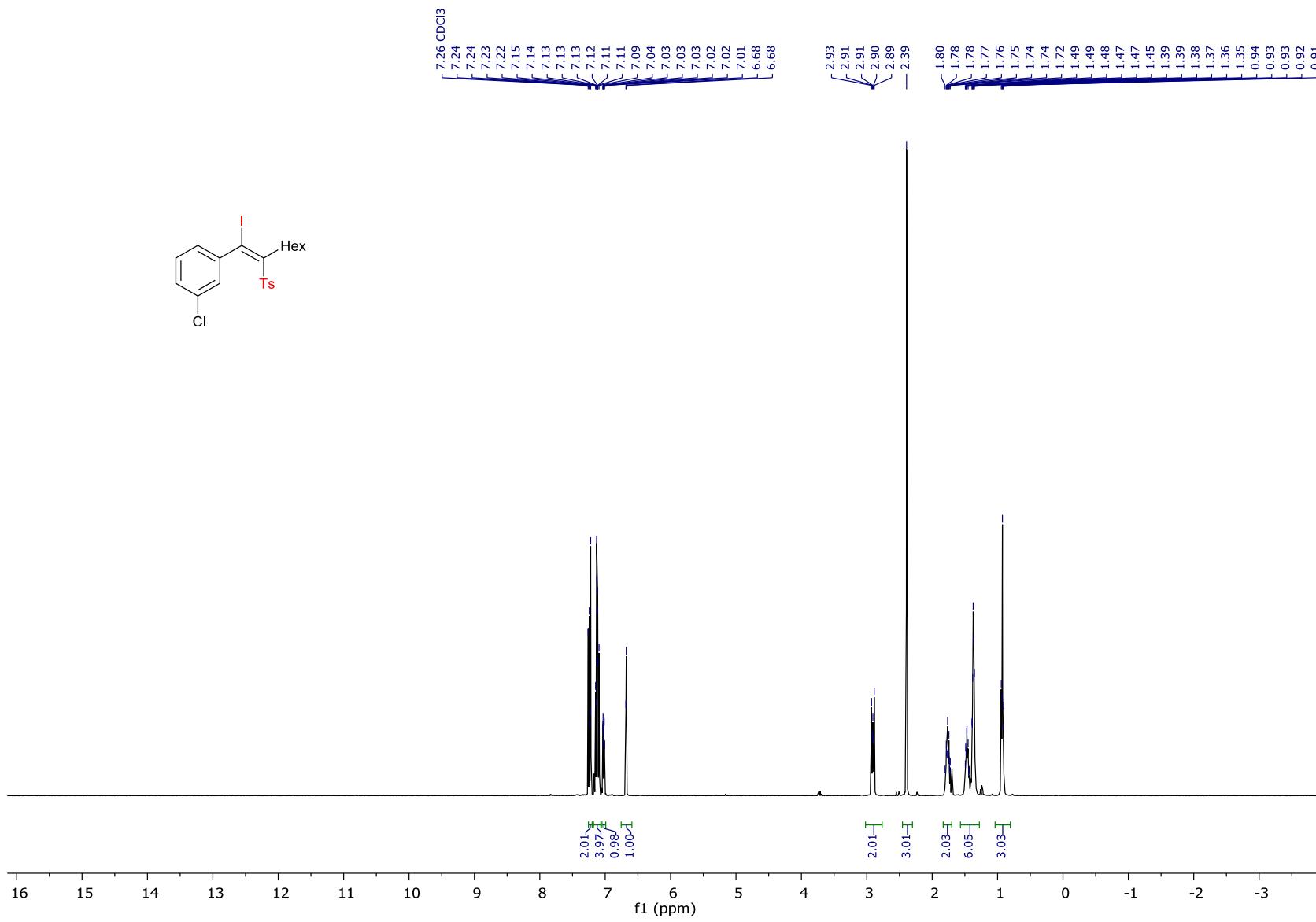


Figure S53. ¹H NMR (600 MHz, Chloroform-d) of (E)-1-chloro-3-(1-iodo-2-tosyloct-1-enyl)benzene (3l).

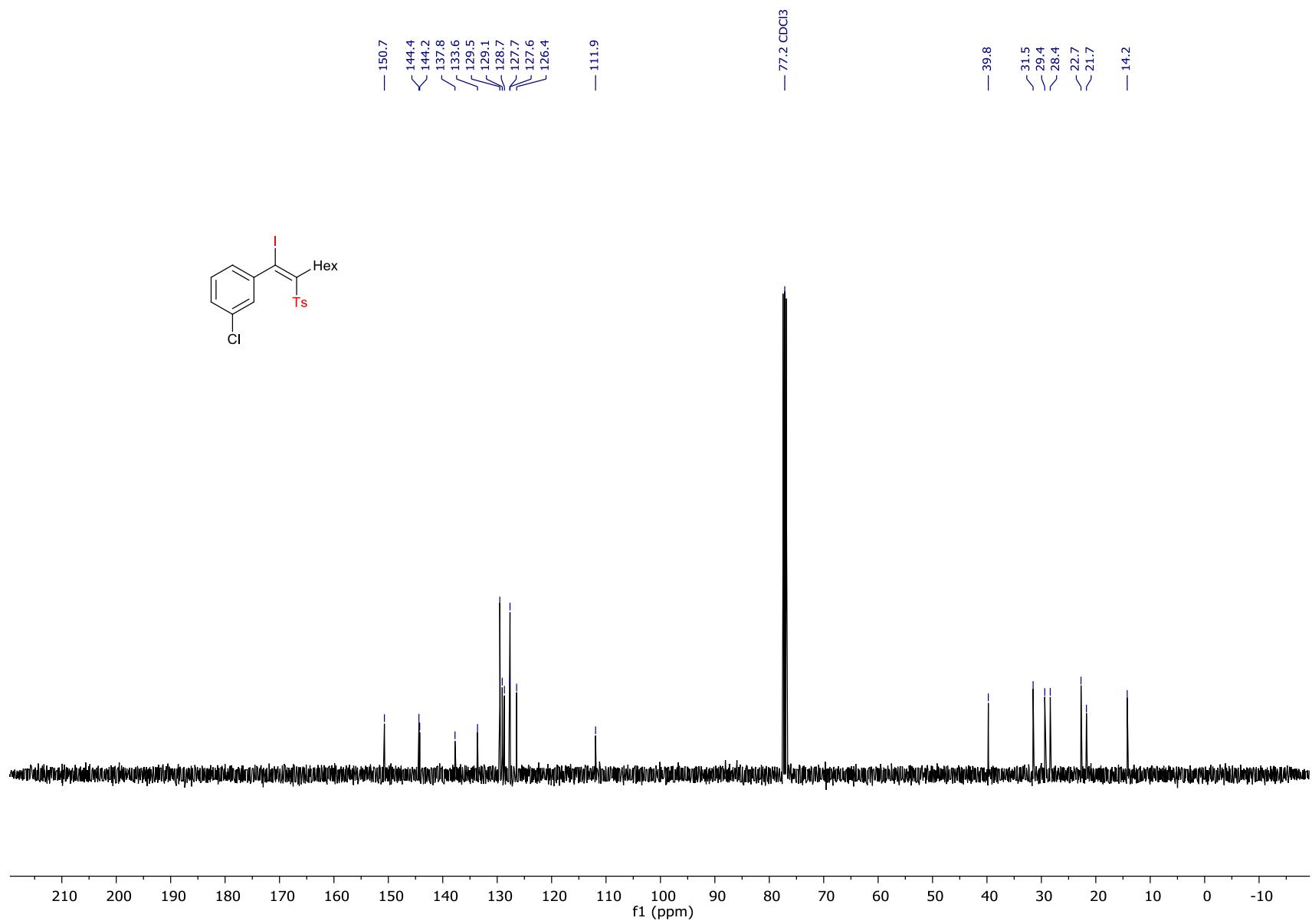


Figure S54. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-1-chloro-3-(1-iodo-2-tosyloct-1-enyl)benzene (3l).

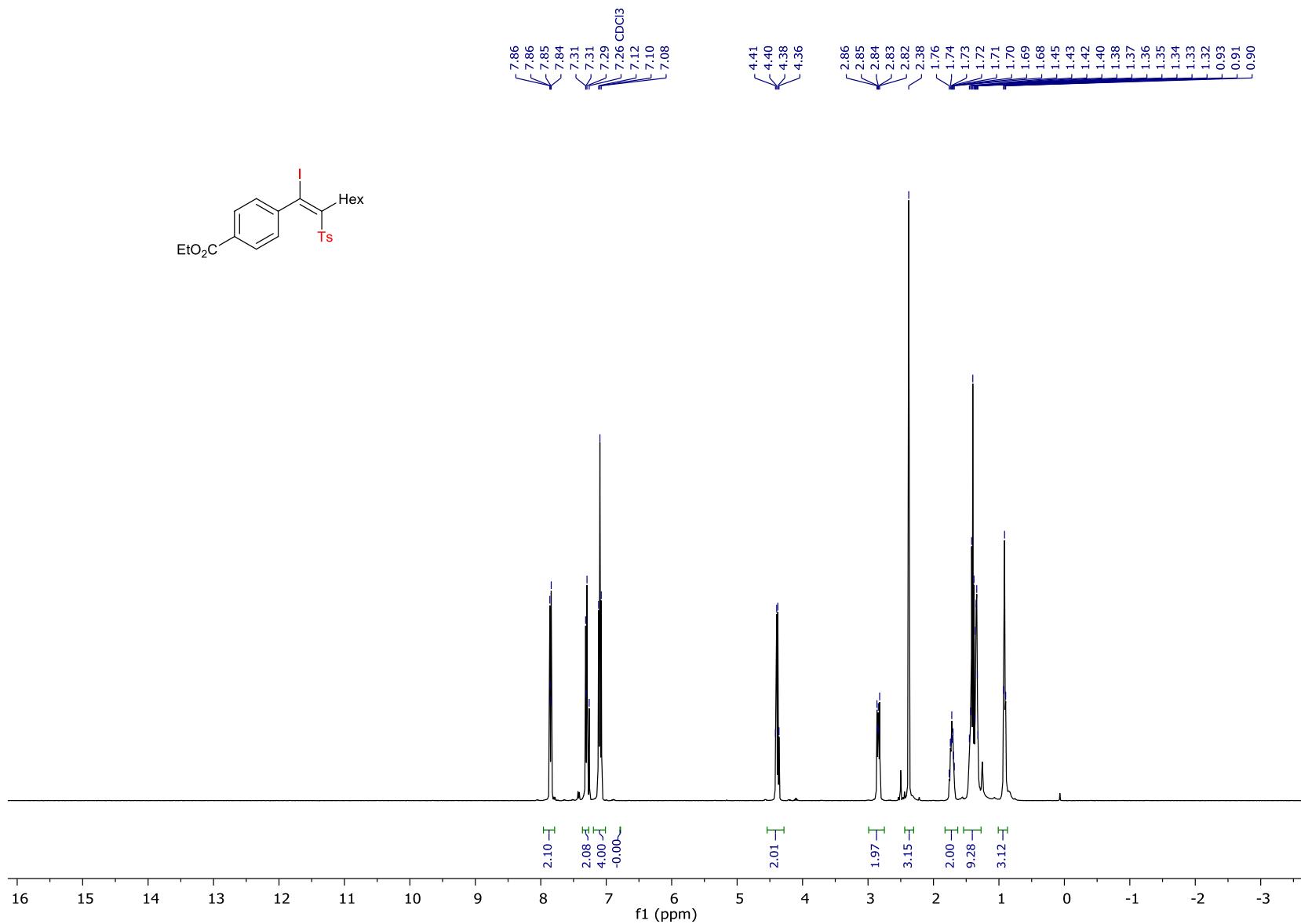


Figure S55. ^1H NMR (600 MHz, Chloroform-d) of (E)-ethyl-4-(1-iodo-2-tosyloct-1-enyl)benzoate (3m).

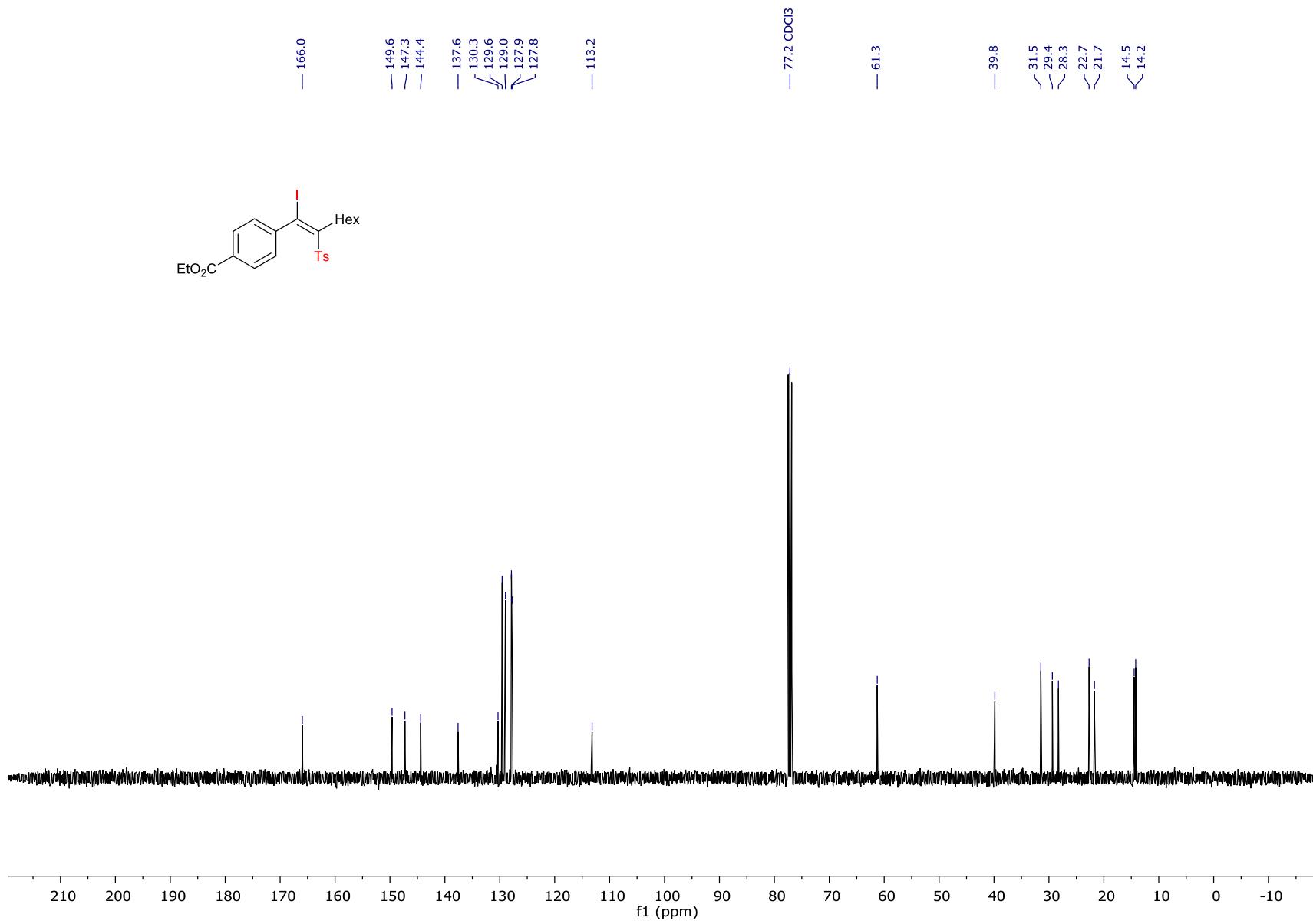


Figure S56. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-ethyl-4-(1-iodo-2-tosyloct-1-enyl)benzoate (**3m**).

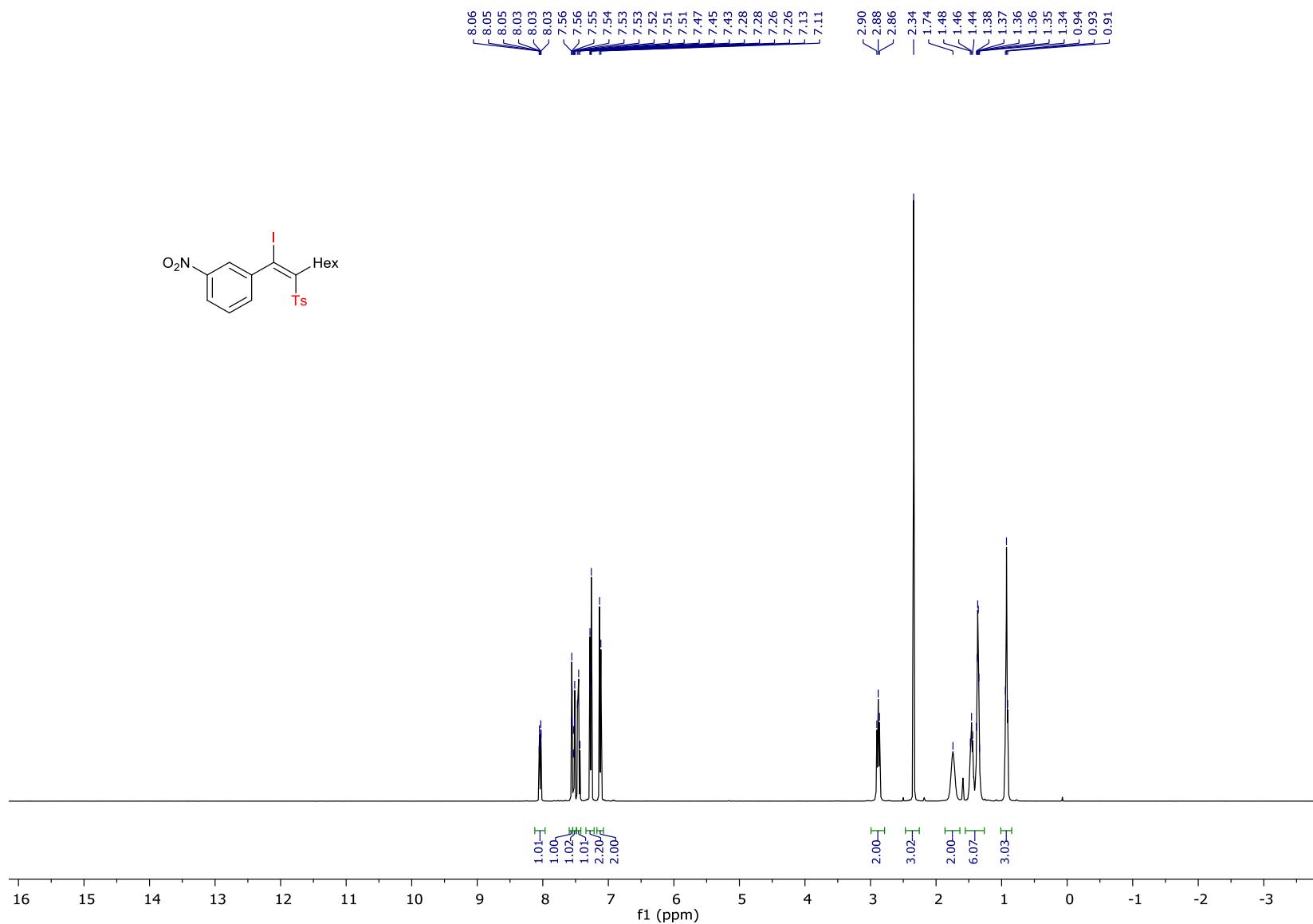


Figure S57. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-1-(1-iodo-2-tosyloct-1-enyl)-3-nitrobenzene (**3n**).

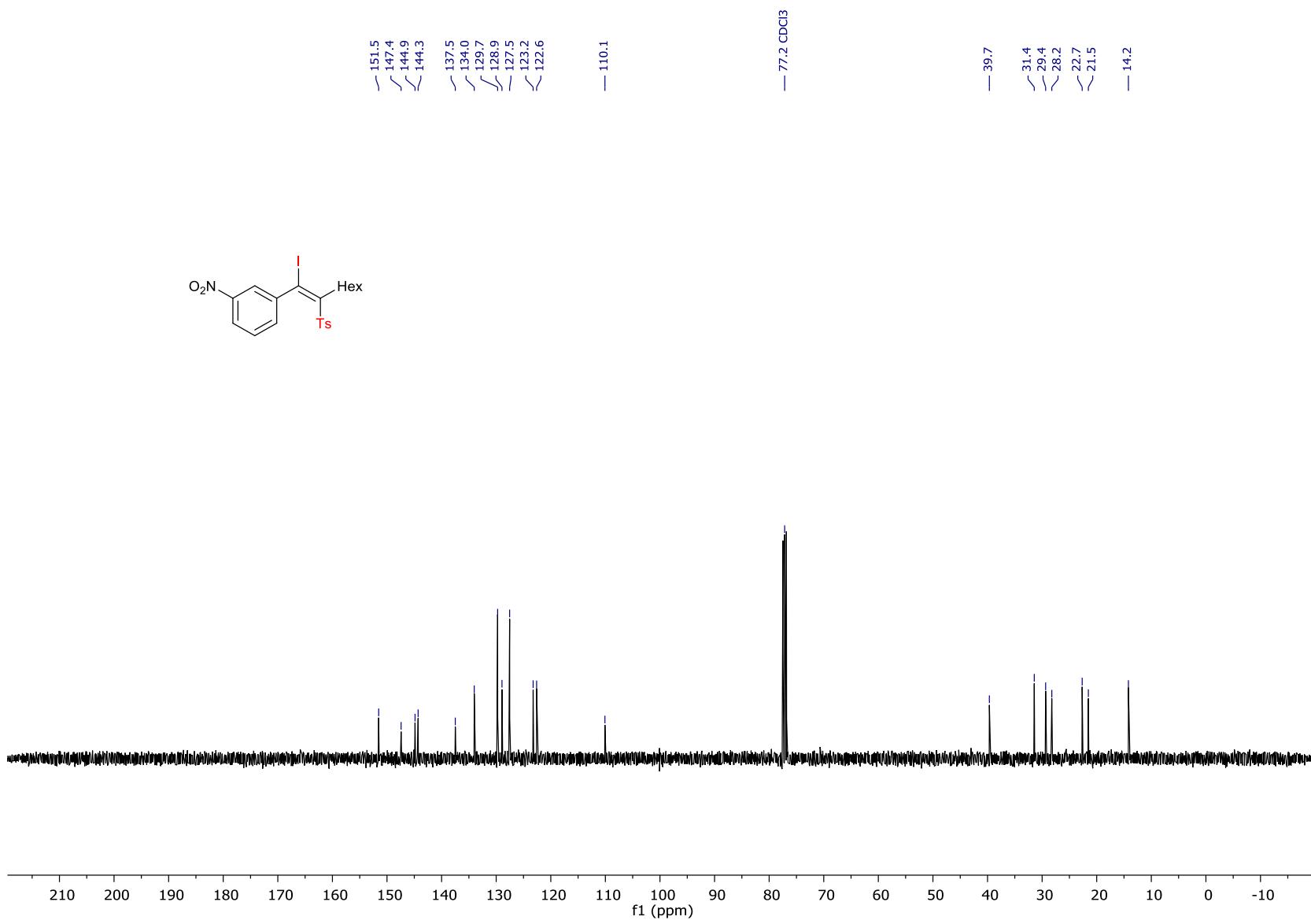


Figure S58. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-1-(1-iodo-2-tosyloct-1-enyl)-3-nitrobenzene (**3n**).

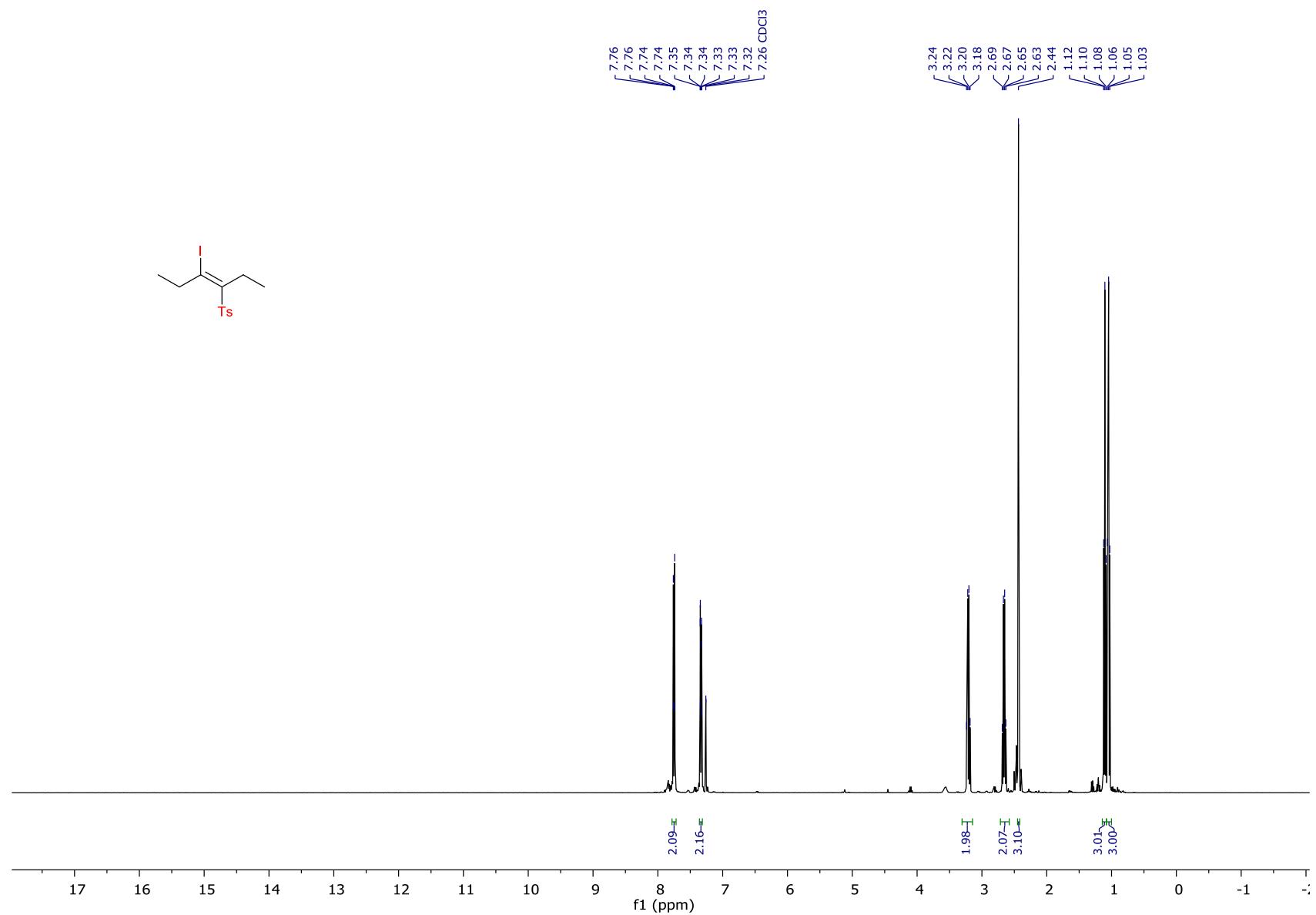


Figure S59. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (**3o**).

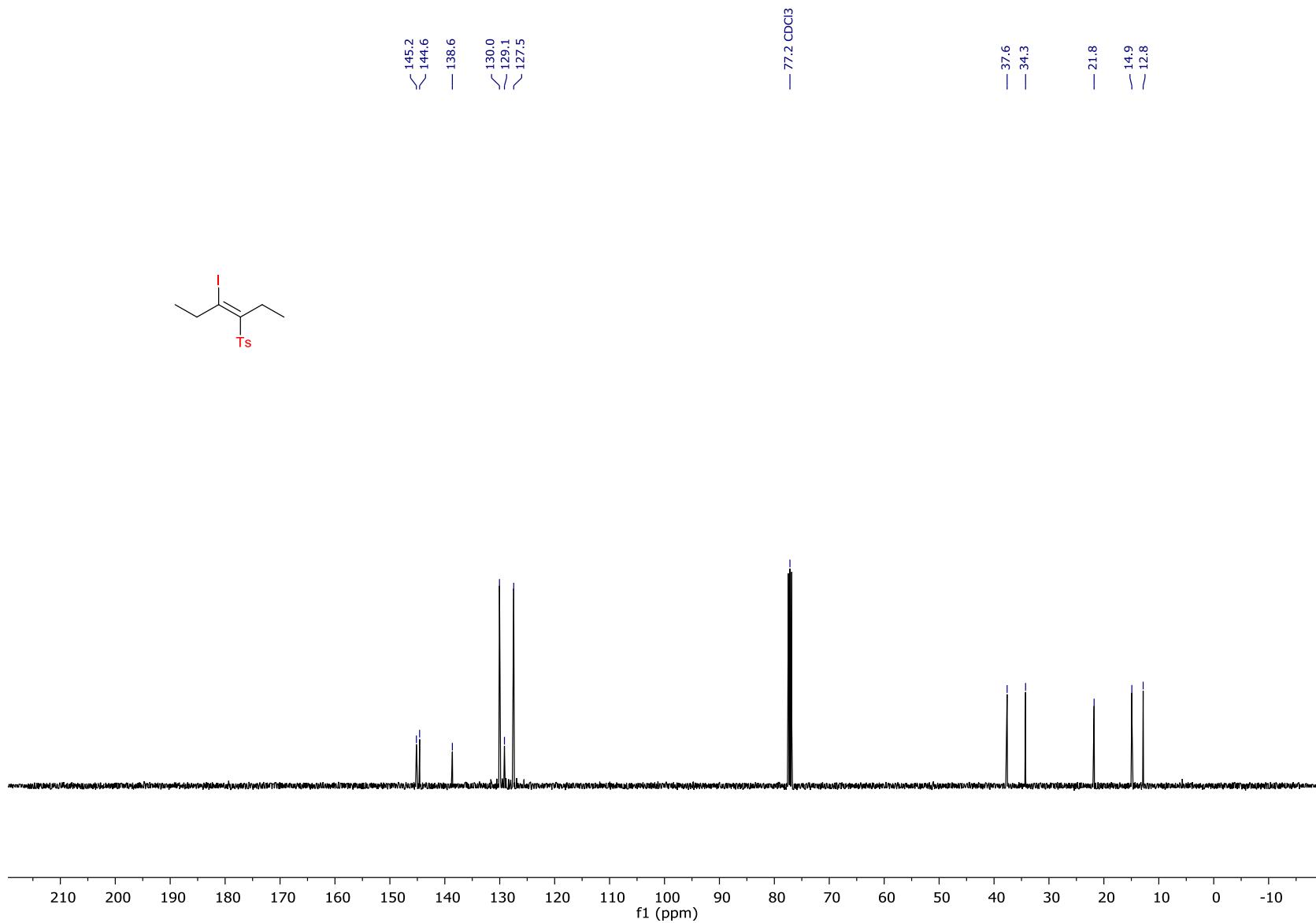


Figure S60. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-1-(4-iodohex-3-en-3-ylsulfonyl)-4-methylbenzene (3o).

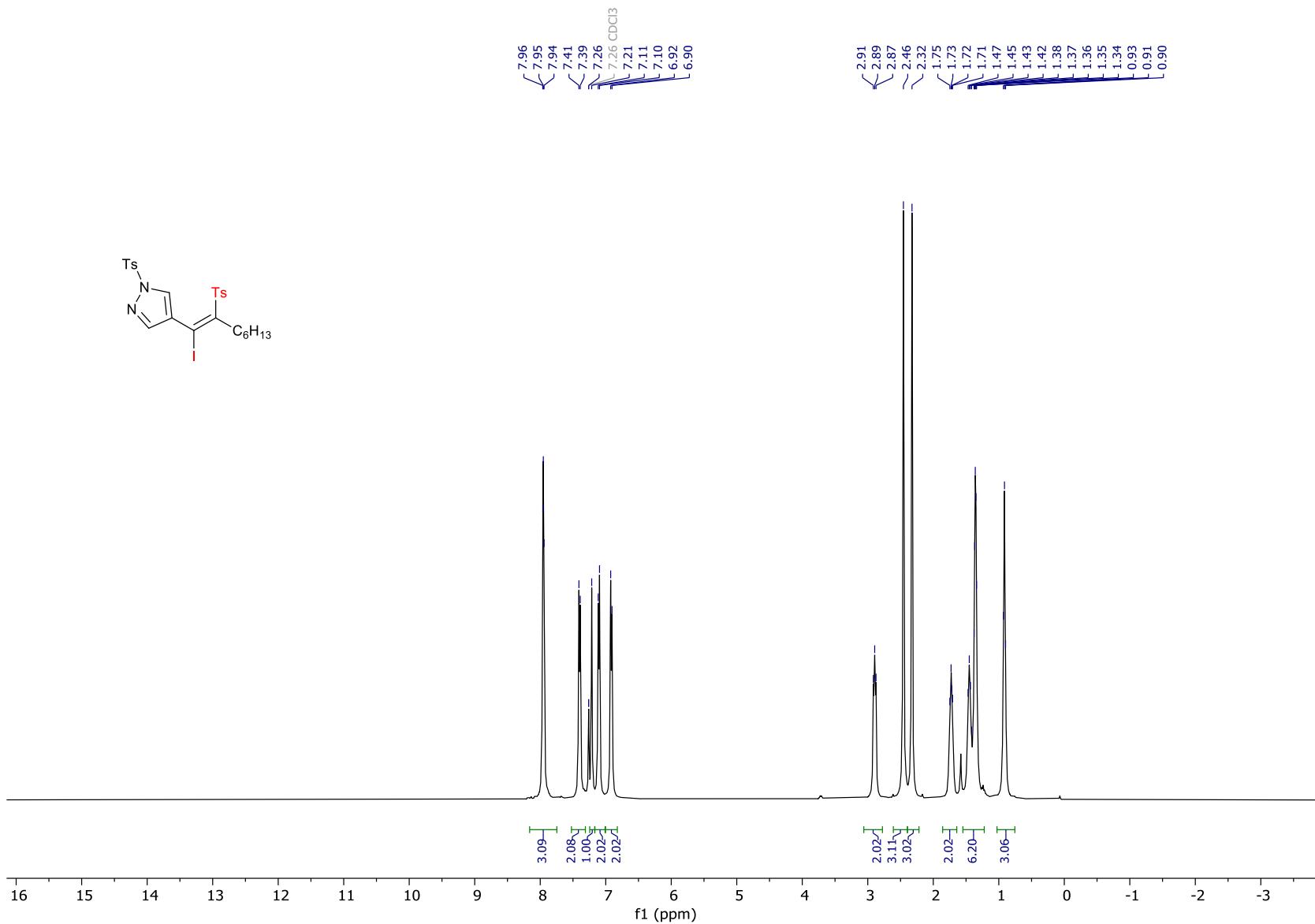


Figure S61. ^1H NMR (600 MHz, Chloroform-d) of (E)-4-(1-iodo-2-tosyloct-1-en-1-yl)-1-tosyl-1H-pyrazole. (3p).

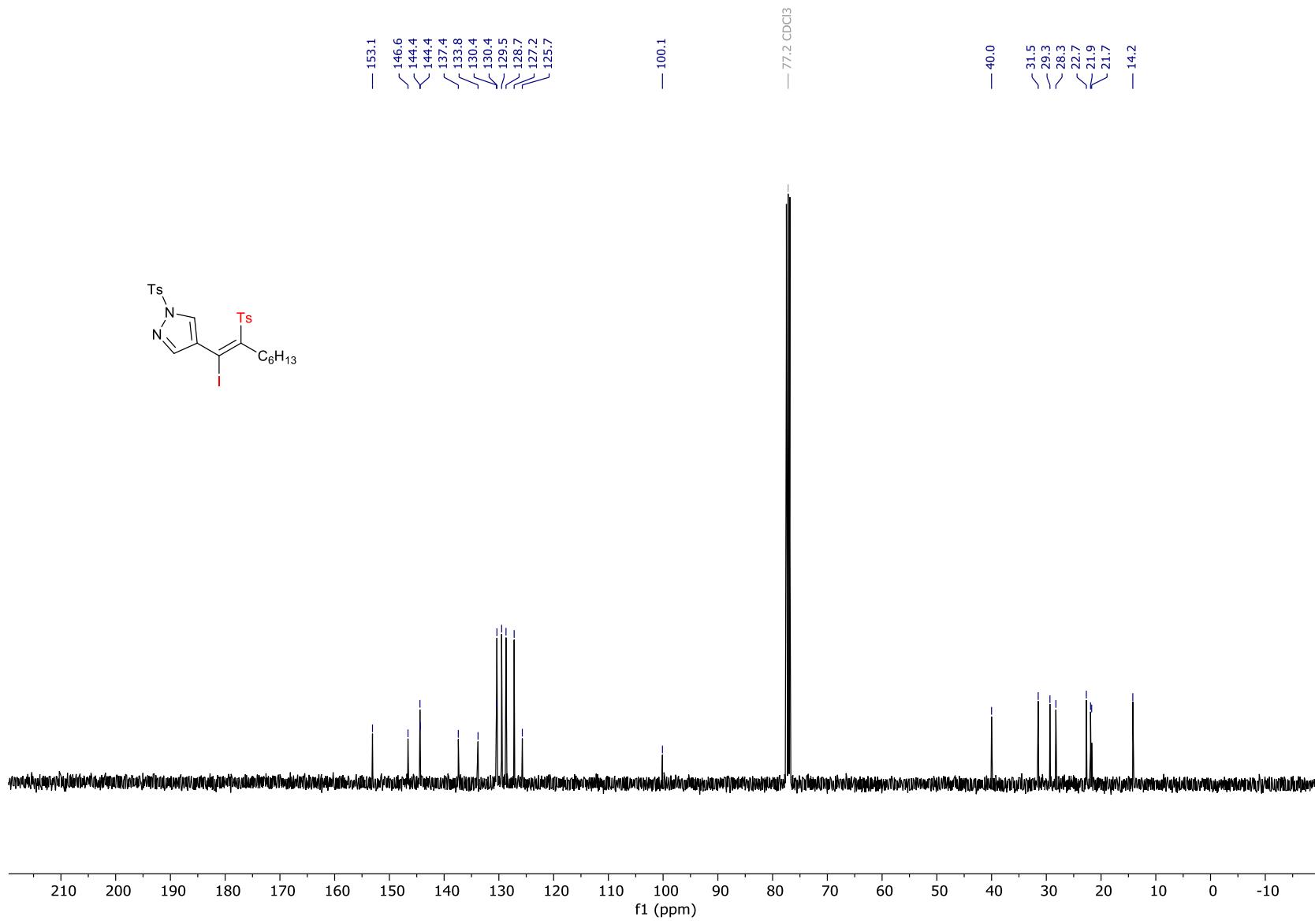


Figure S62. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-4-(1-iodo-2-tosyloct-1-en-1-yl)-1-tosyl-1H-pyrazole (3p).

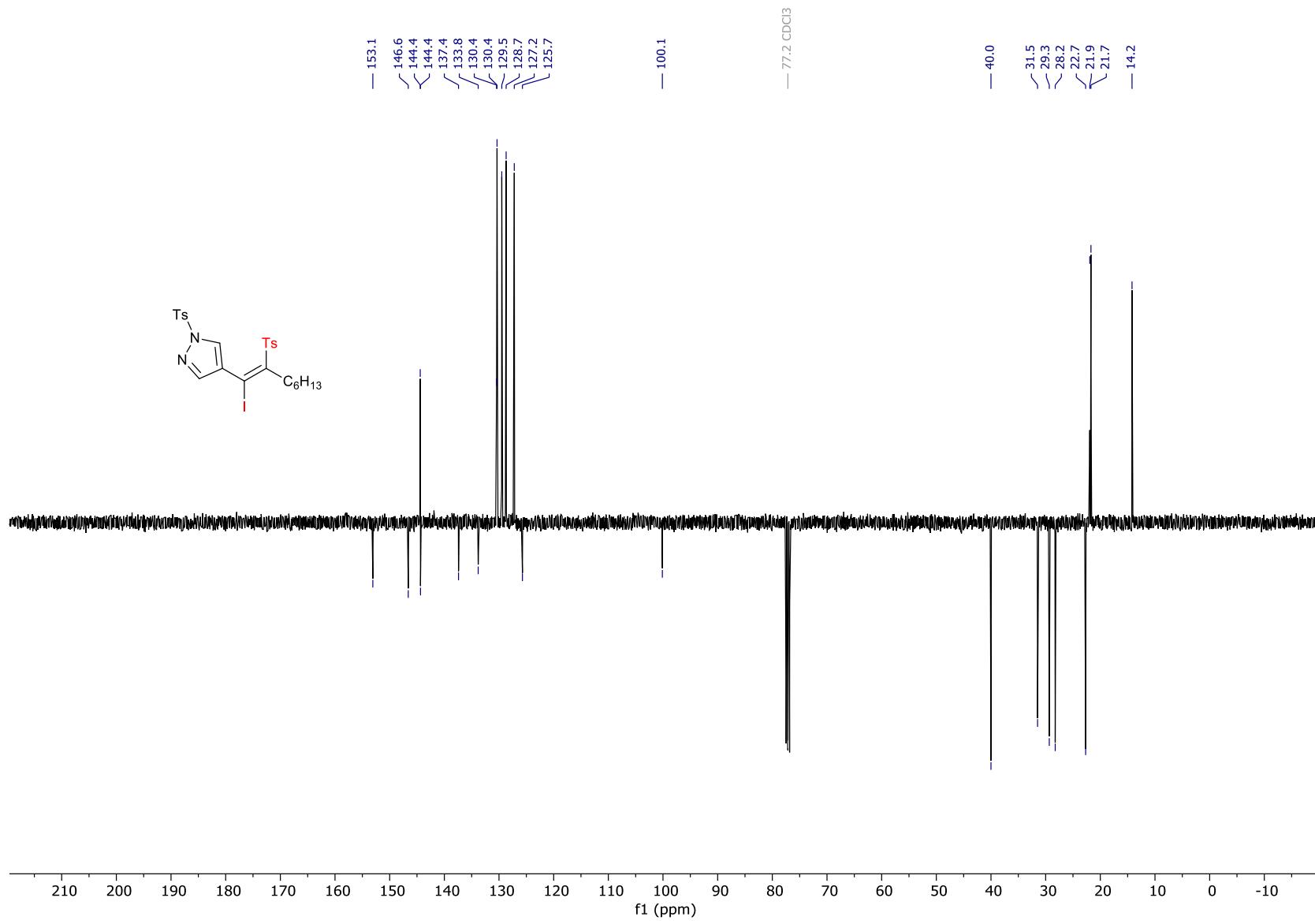


Figure S63. ^{13}C DEPTQ-135 NMR (*E*)-4-(1-iodo-2-tosyloct-1-en-1-yl)-1-tosyl-1H-pyrazole (**3p**).

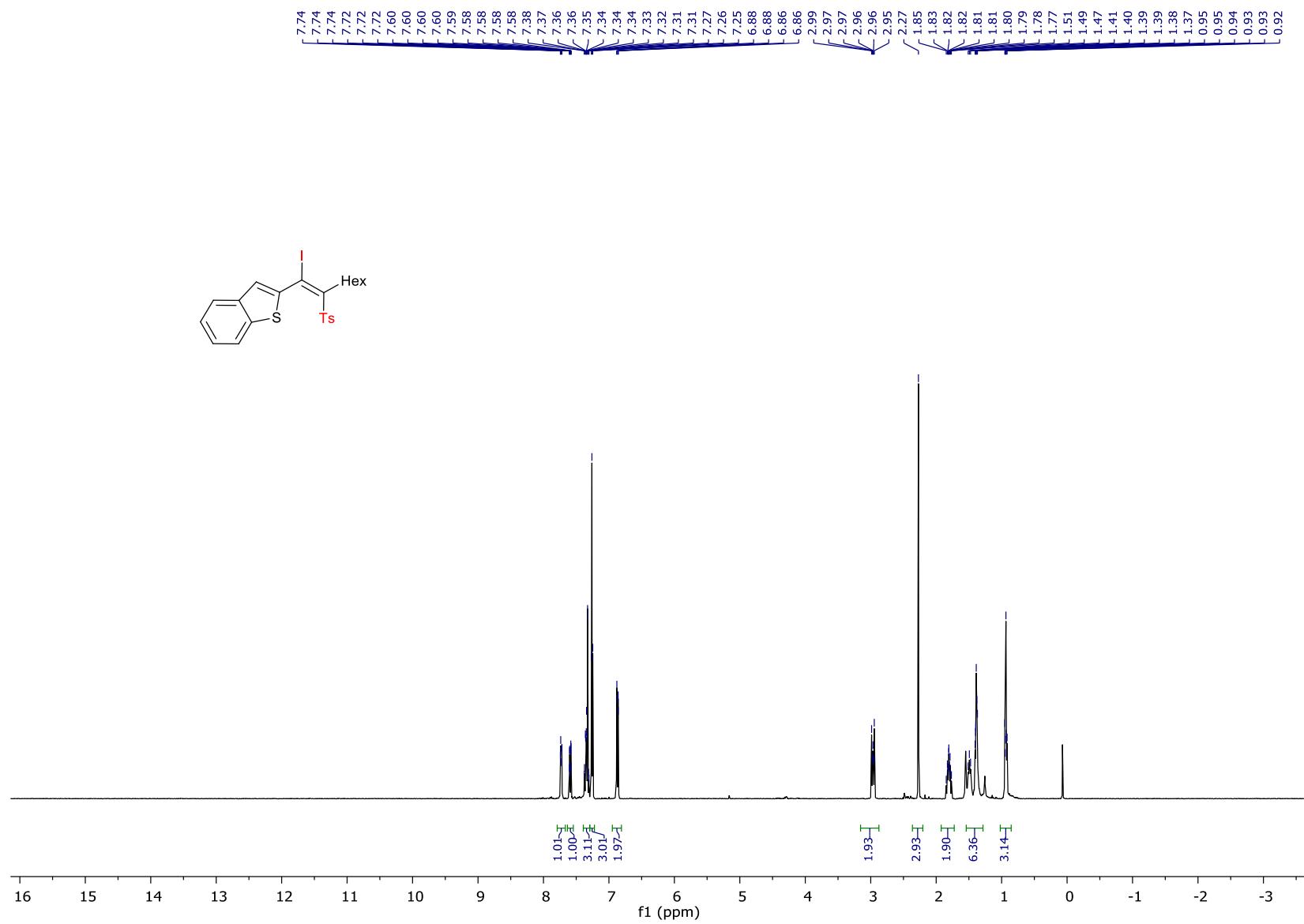


Figure S64. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-2-(1-*iodo*-2-tosyloct-1-enyl)benzo[b]thiophene (3q).

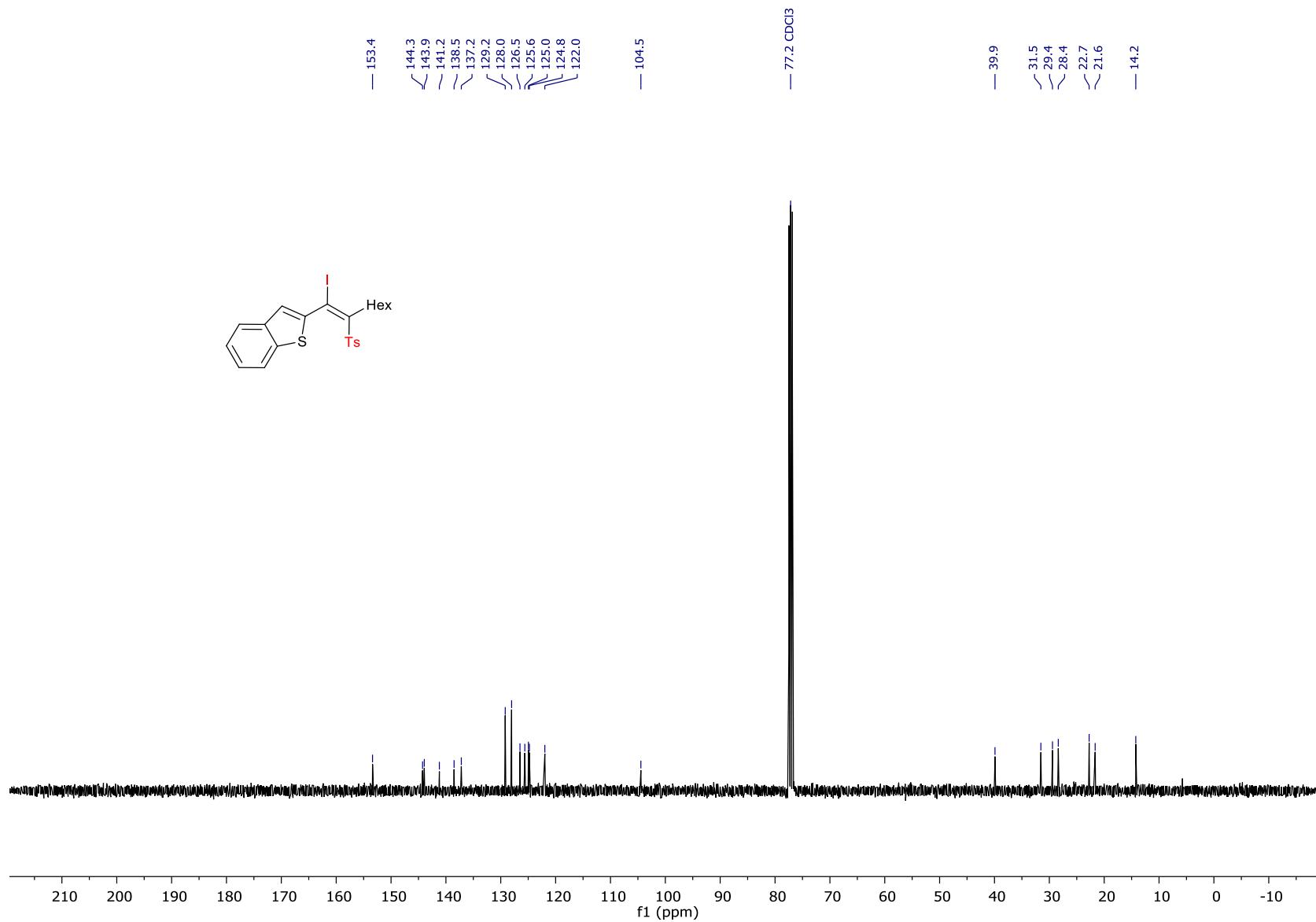


Figure S65. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-2-(1-iodo-2-tosyloct-1-enyl)benzo[b]thiophene (**3q**).

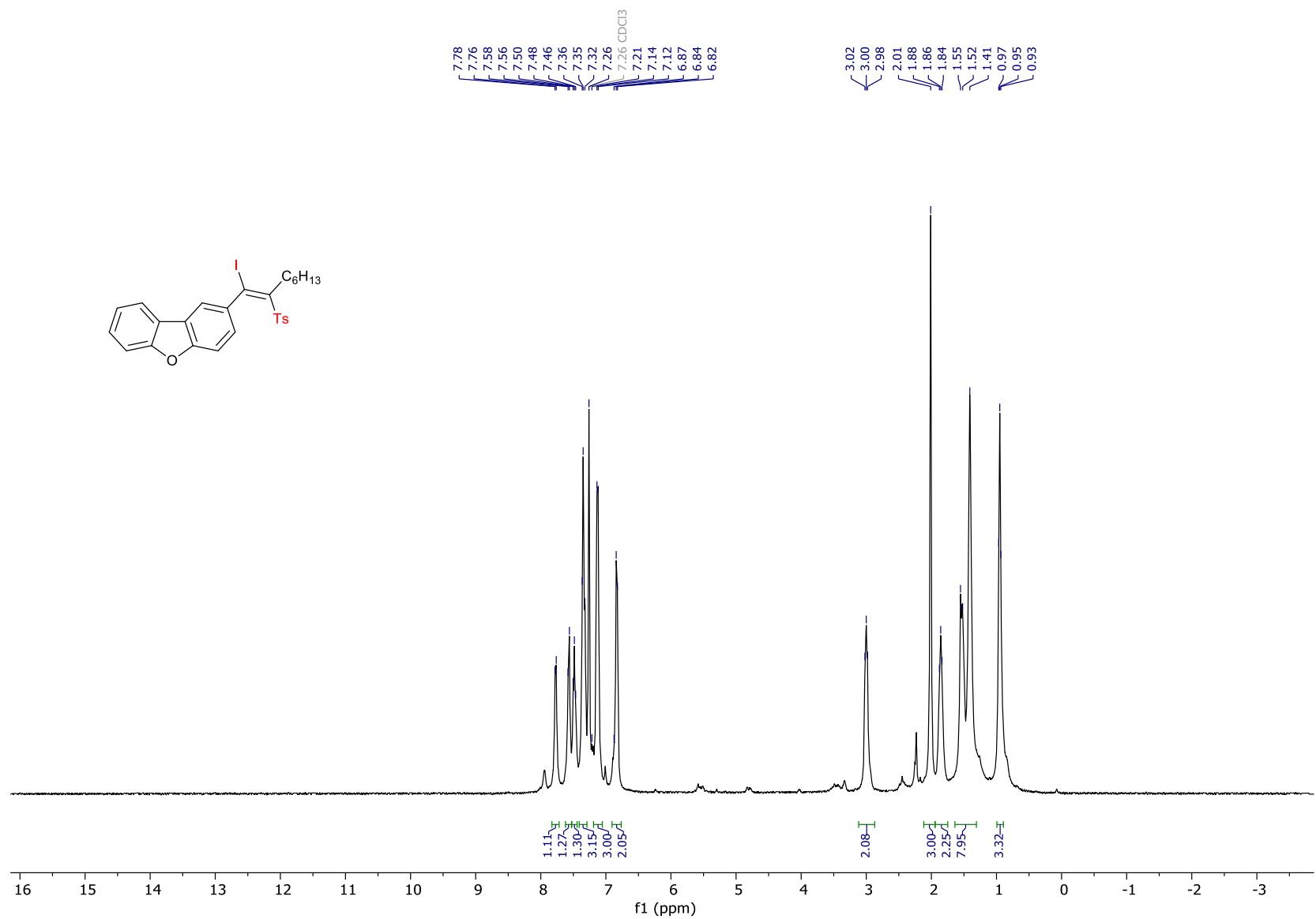


Figure S66. ^1H NMR (600 MHz, Chloroform-d) of (E)-2-(1-iodo-2-tosyloct-1-en-1-yl)dibenzo[b,d]furan (3r).

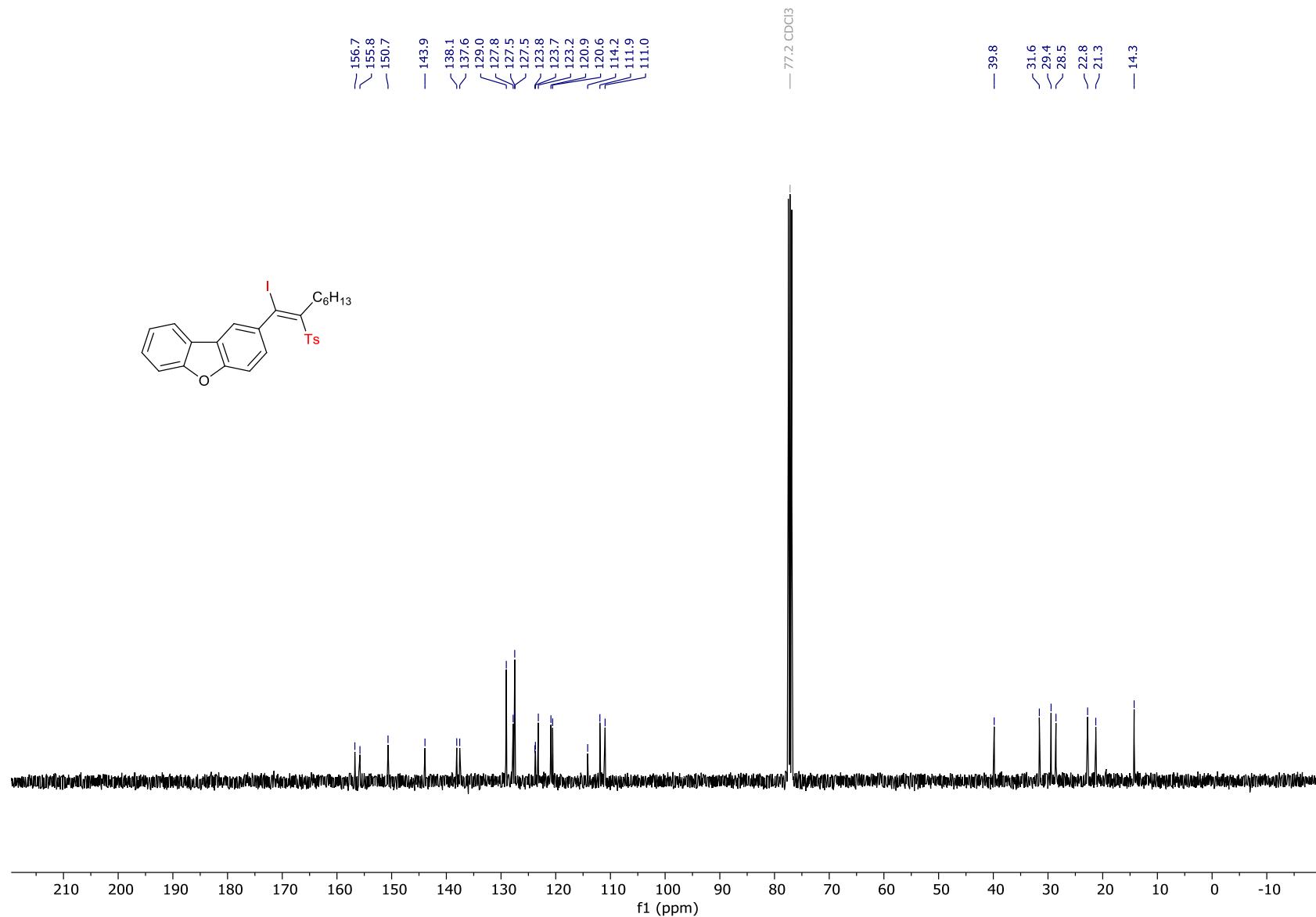


Figure S67. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (*E*)-2-(1-iodo-2-tosyloct-1-en-1-yl)dibenzo[b,d]furan (3r).

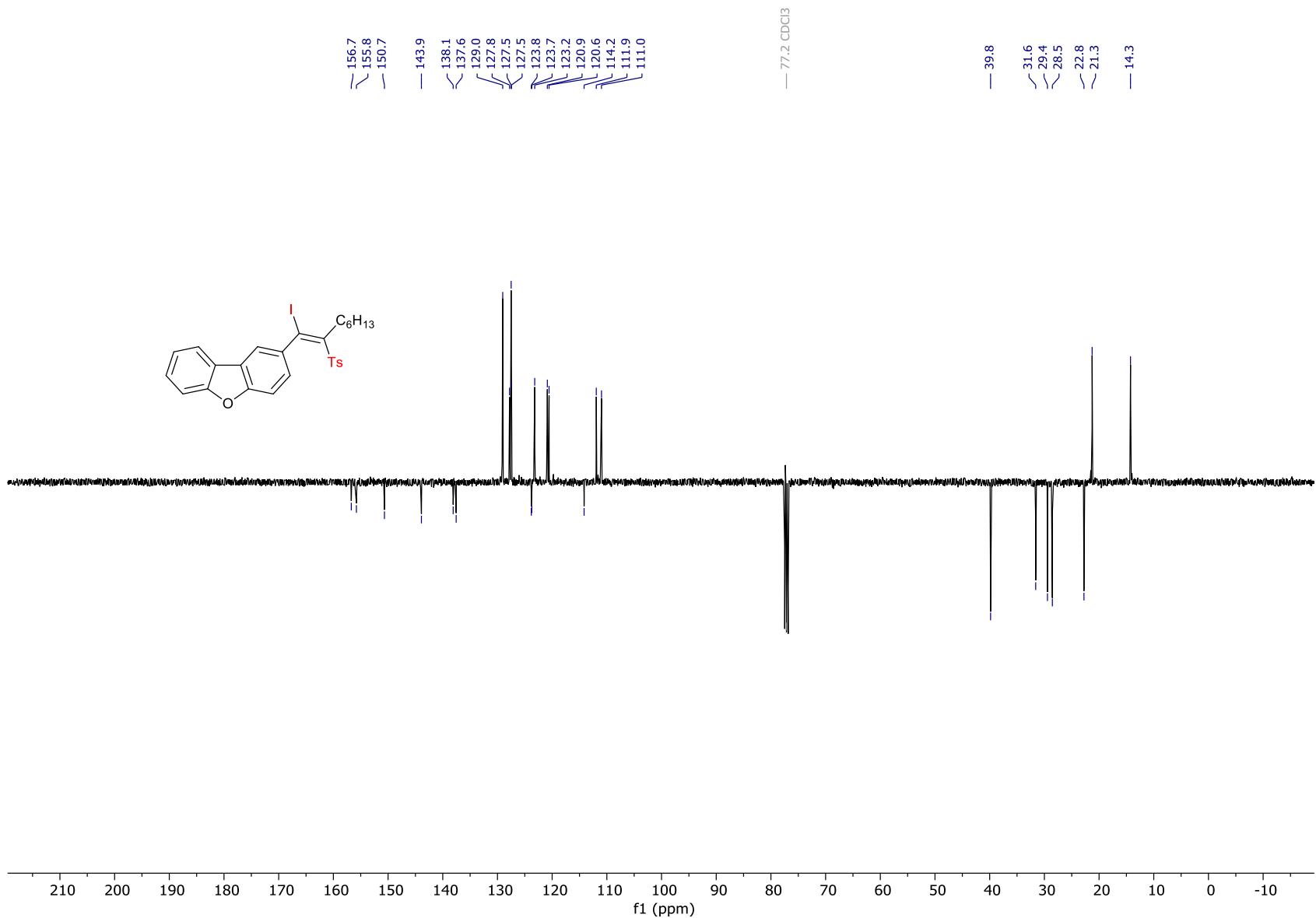


Figure S68. ^{13}C DEPTQ-135 NMR (*E*)-2-(1-iodo-2-tosyloct-1-en-1-yl)dibenzo[b,d]furan (3r).

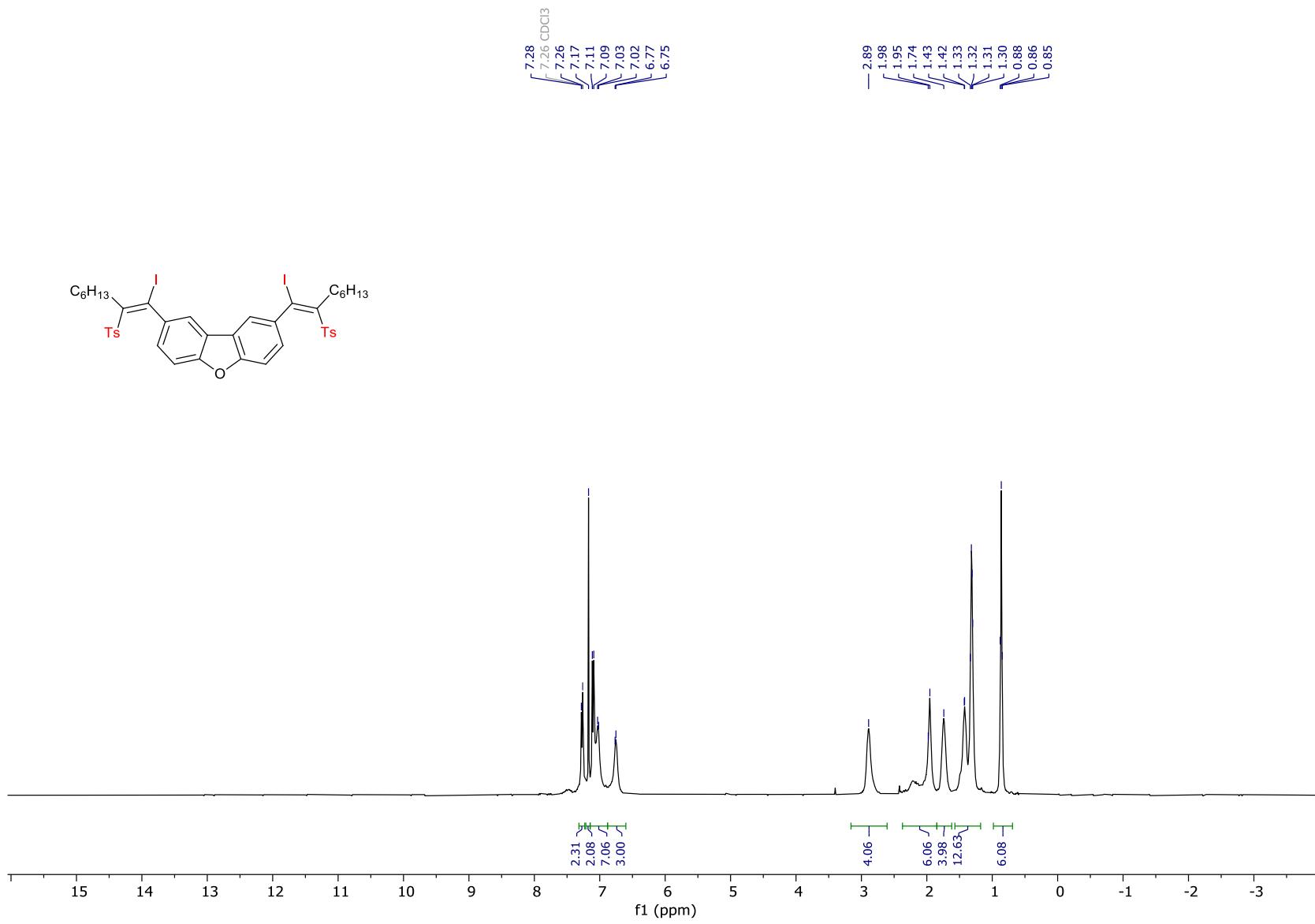


Figure S69. ¹H NMR (600 MHz, Chloroform-d) of 2,8-bis((*E*)-1-iodo-2-tosyloct-1-en-1-yl)dibenzo[b,d]furan (3s).

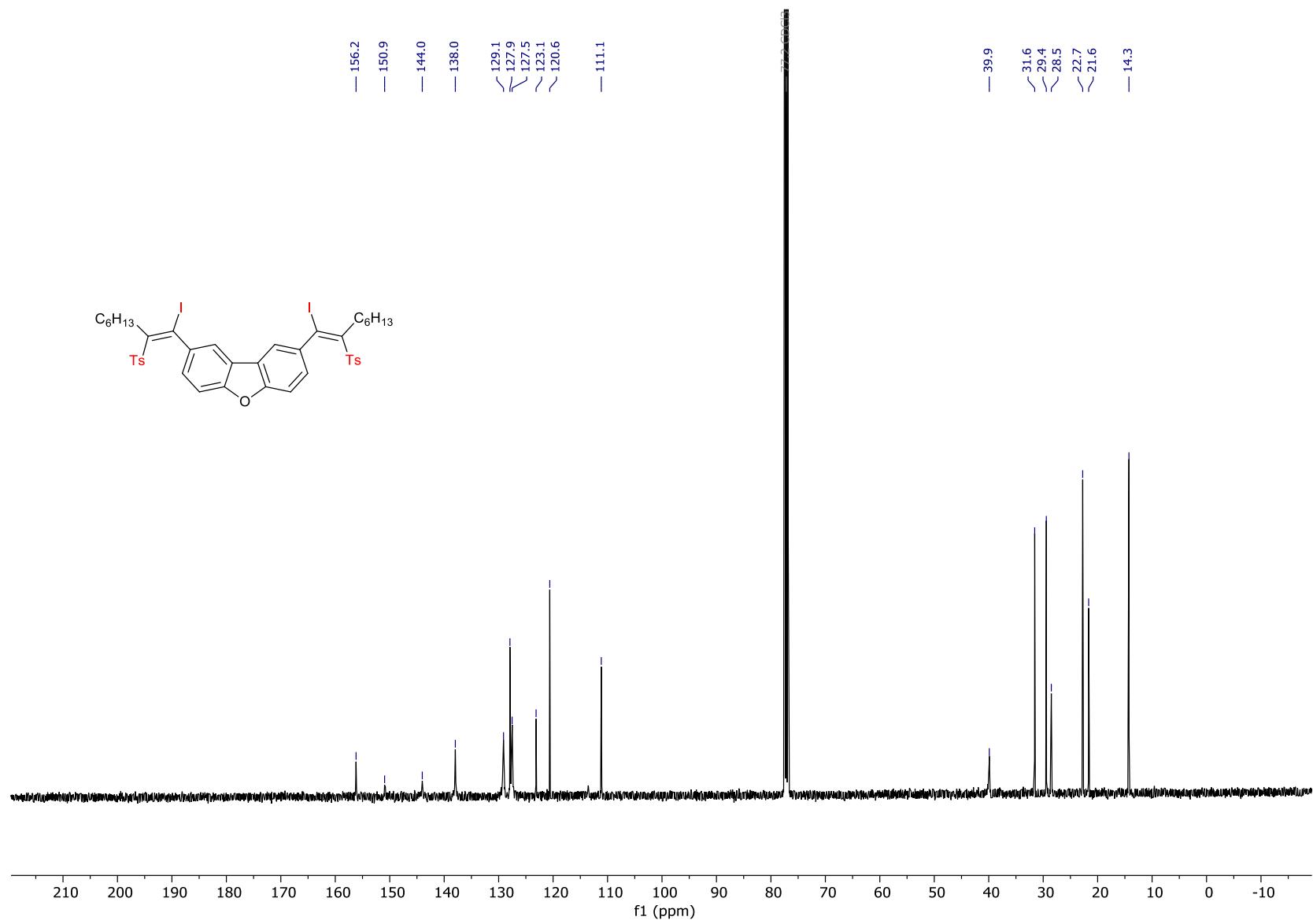


Figure S70. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of 2,8-bis(*E*-1-iodo-2-tosyloct-1-en-1-yl)dibenzo[b,d]furan (**3s**).

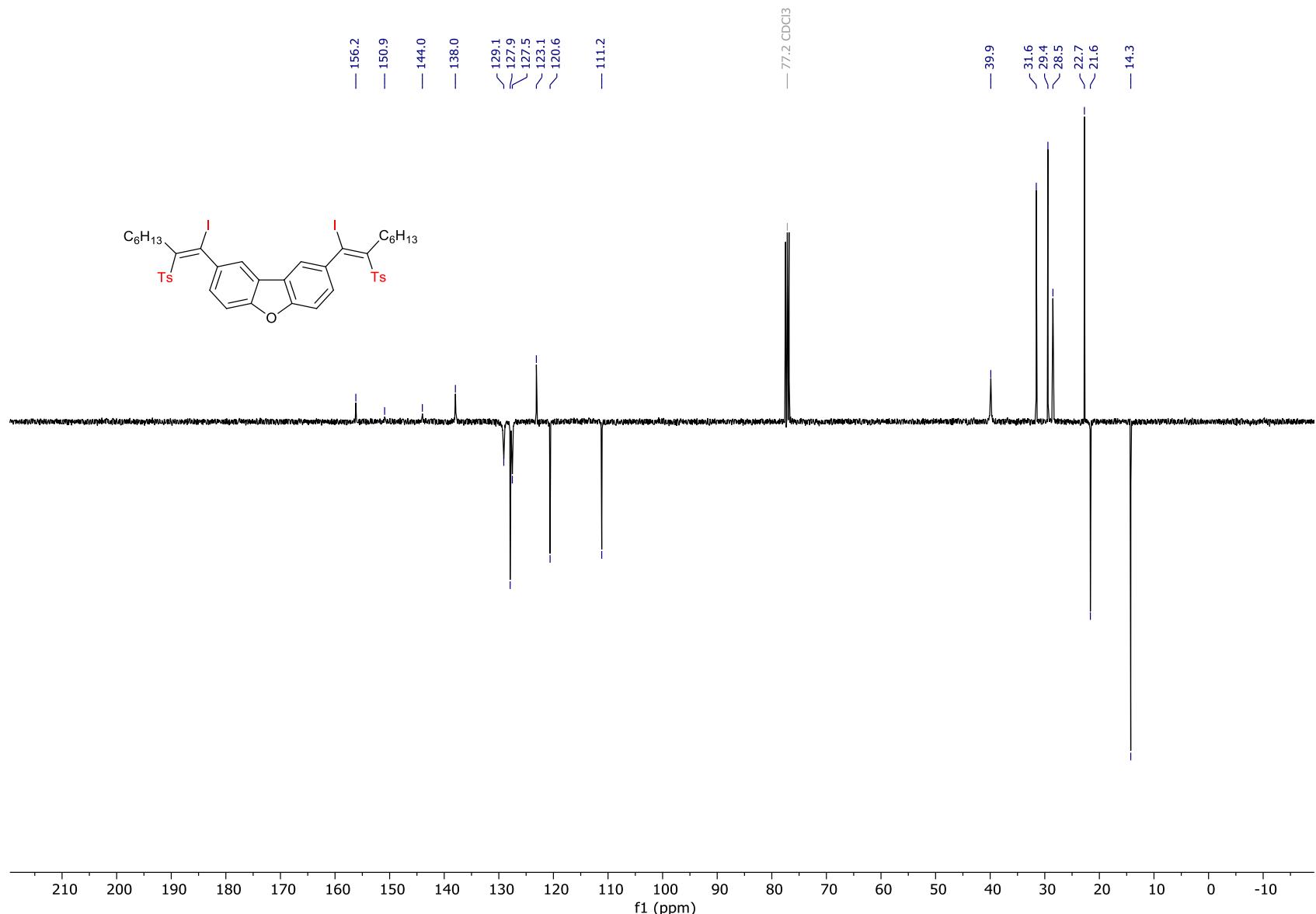


Figure S71. ^{13}C DEPTQ-135 NMR 2,8-bis((E)-1-iodo-2-tosyloct-1-en-1-yl)dibenzo[b,d]furan (3s).

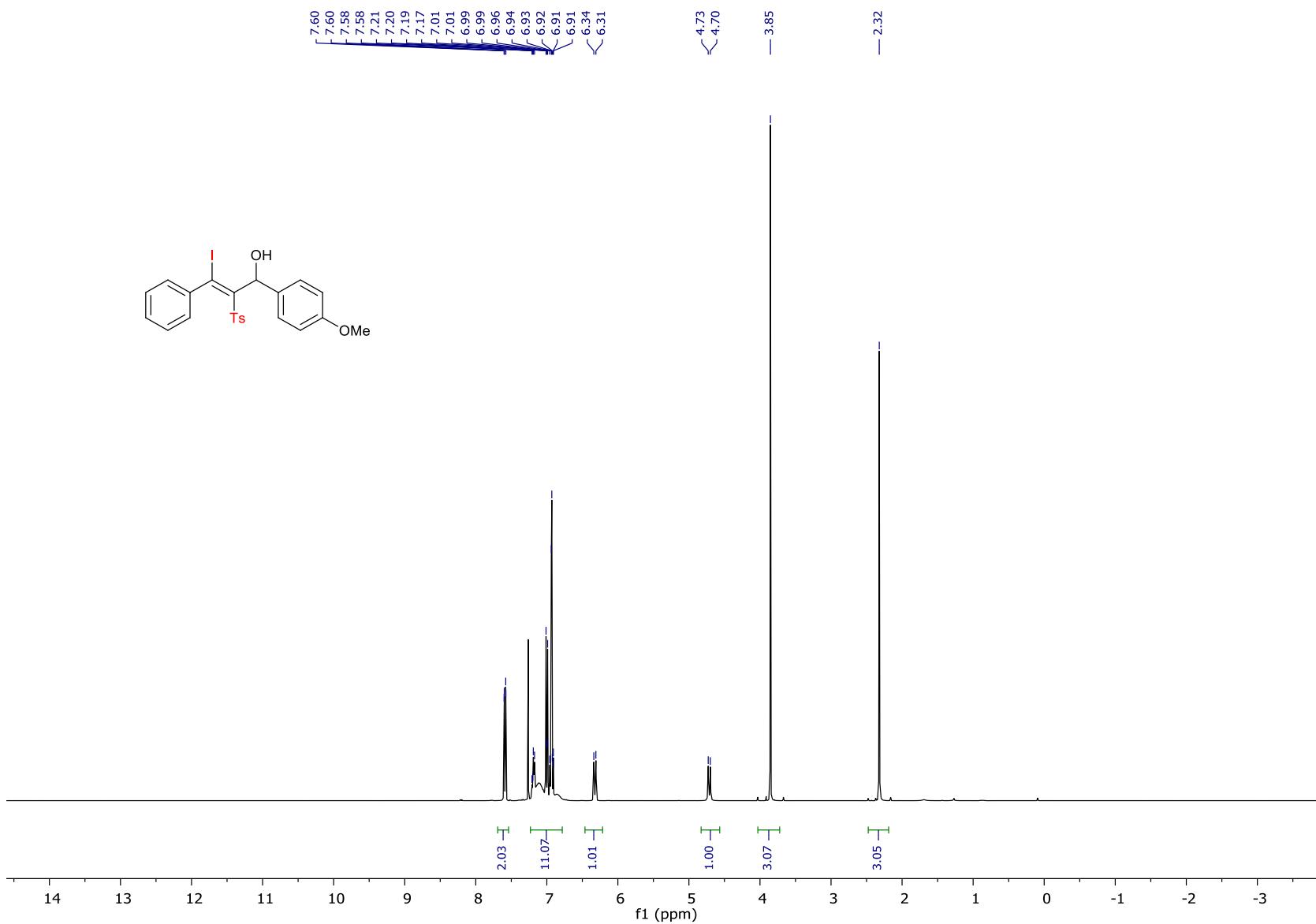


Figure S72. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (**3t**).

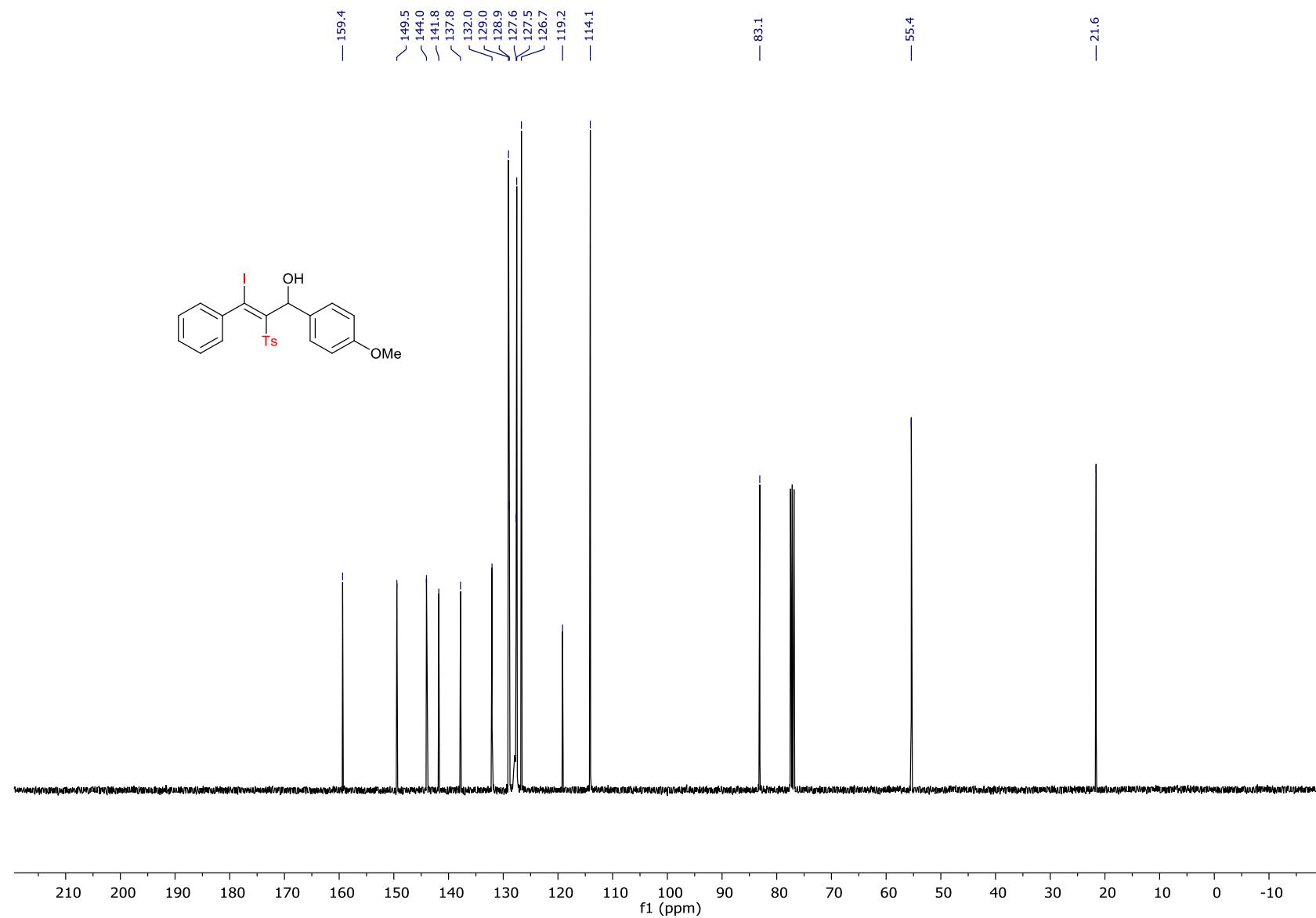


Figure S73. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-ol (3t).

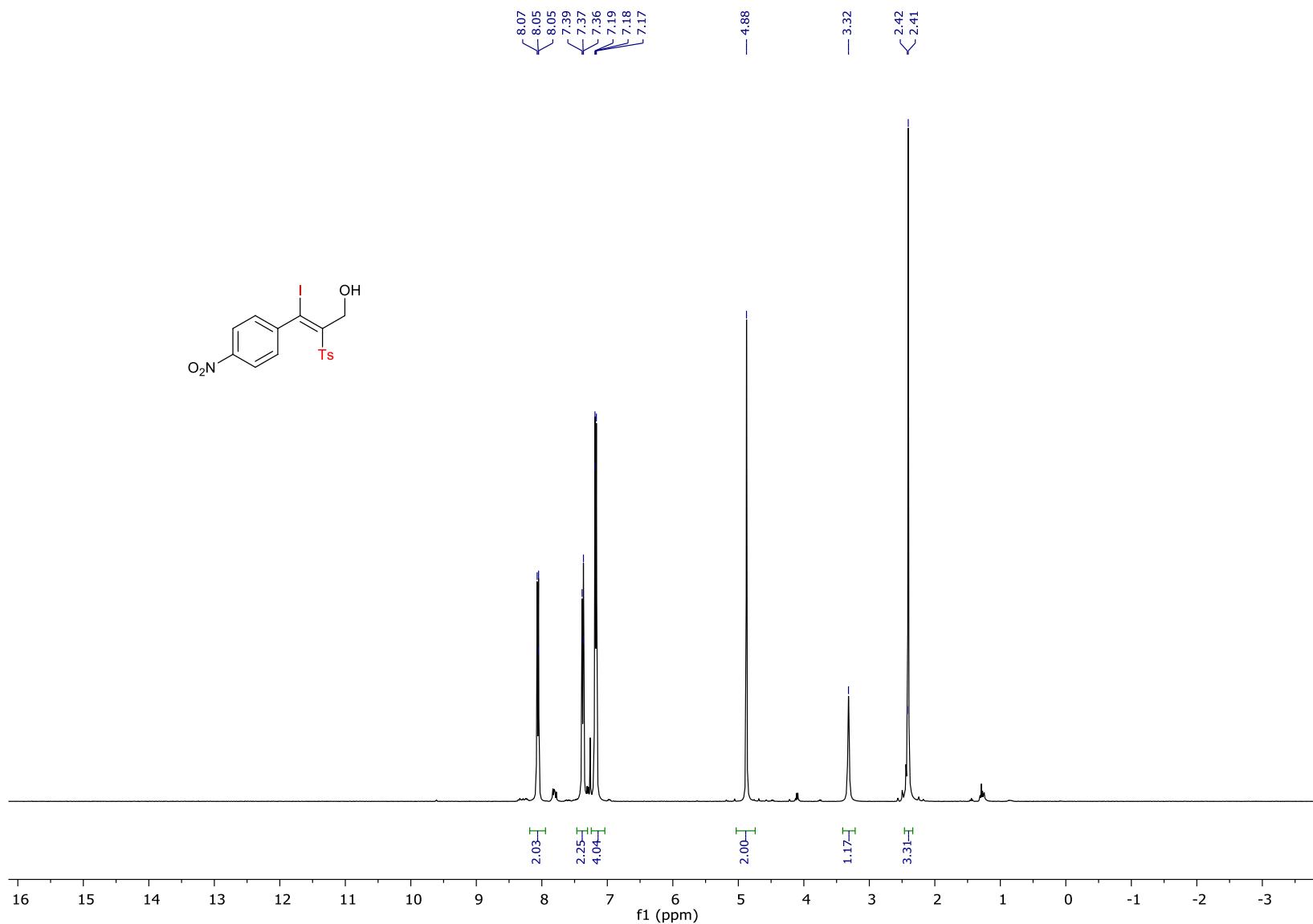


Figure S74. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (**3u**).

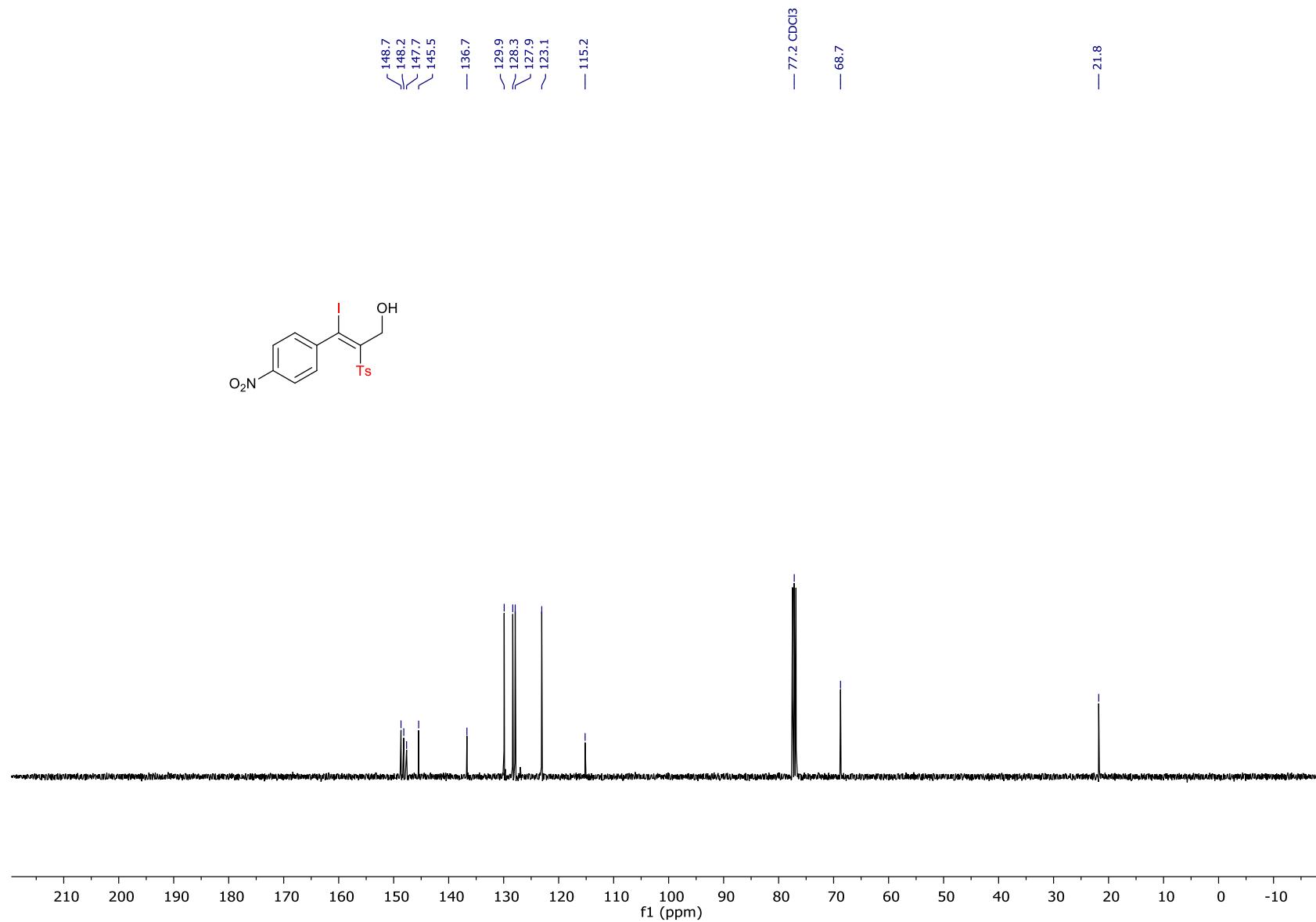


Figure S75. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-3-iodo-3-(4-nitrophenyl)-2-tosylprop-2-en-1-ol (**3u**).

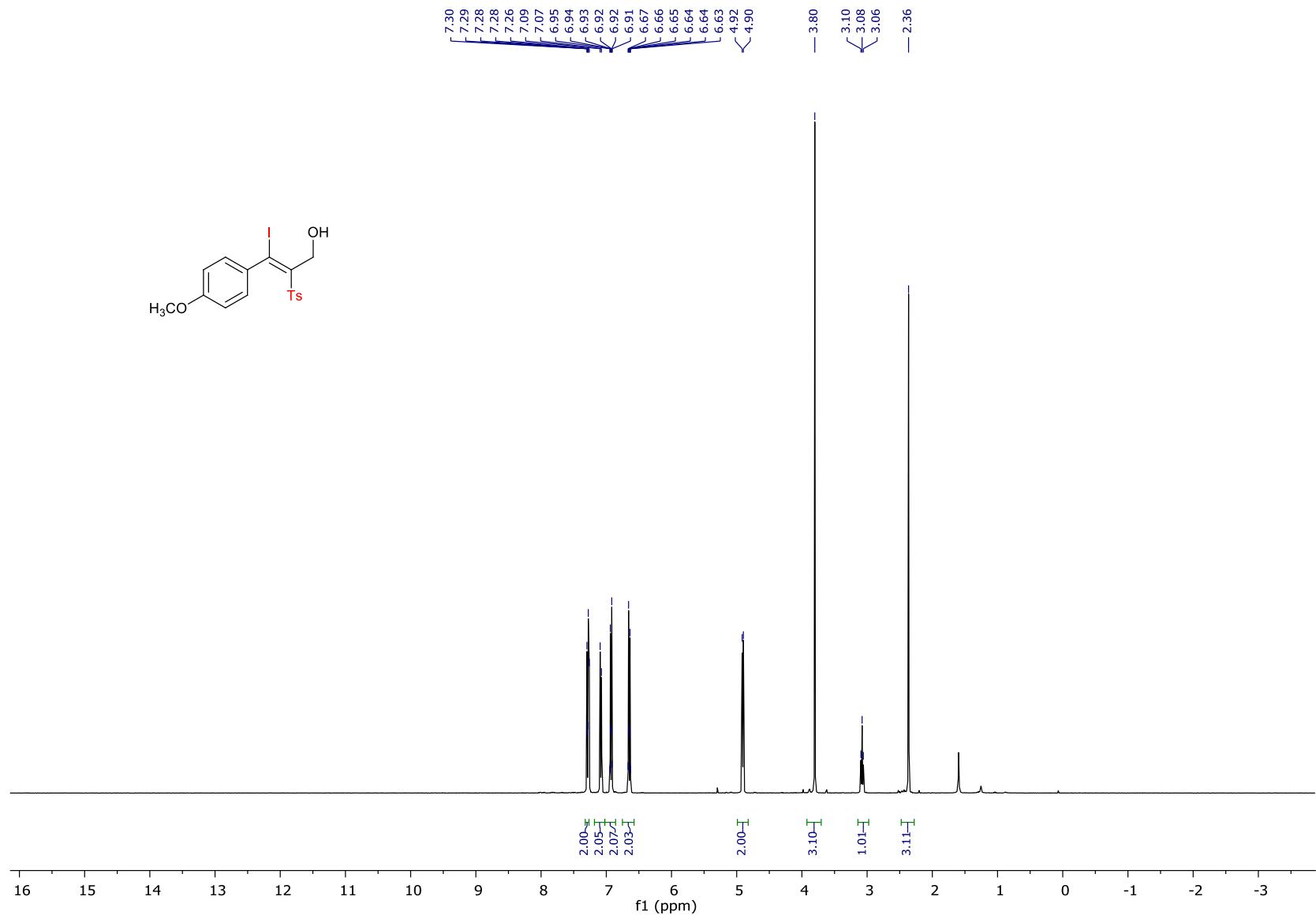


Figure S76. ¹H NMR (600 MHz, Chloroform-d) of (E)-3-iodo-3-(4-methoxyphenyl)-2-tosylprop-2-en-1-ol (3v).

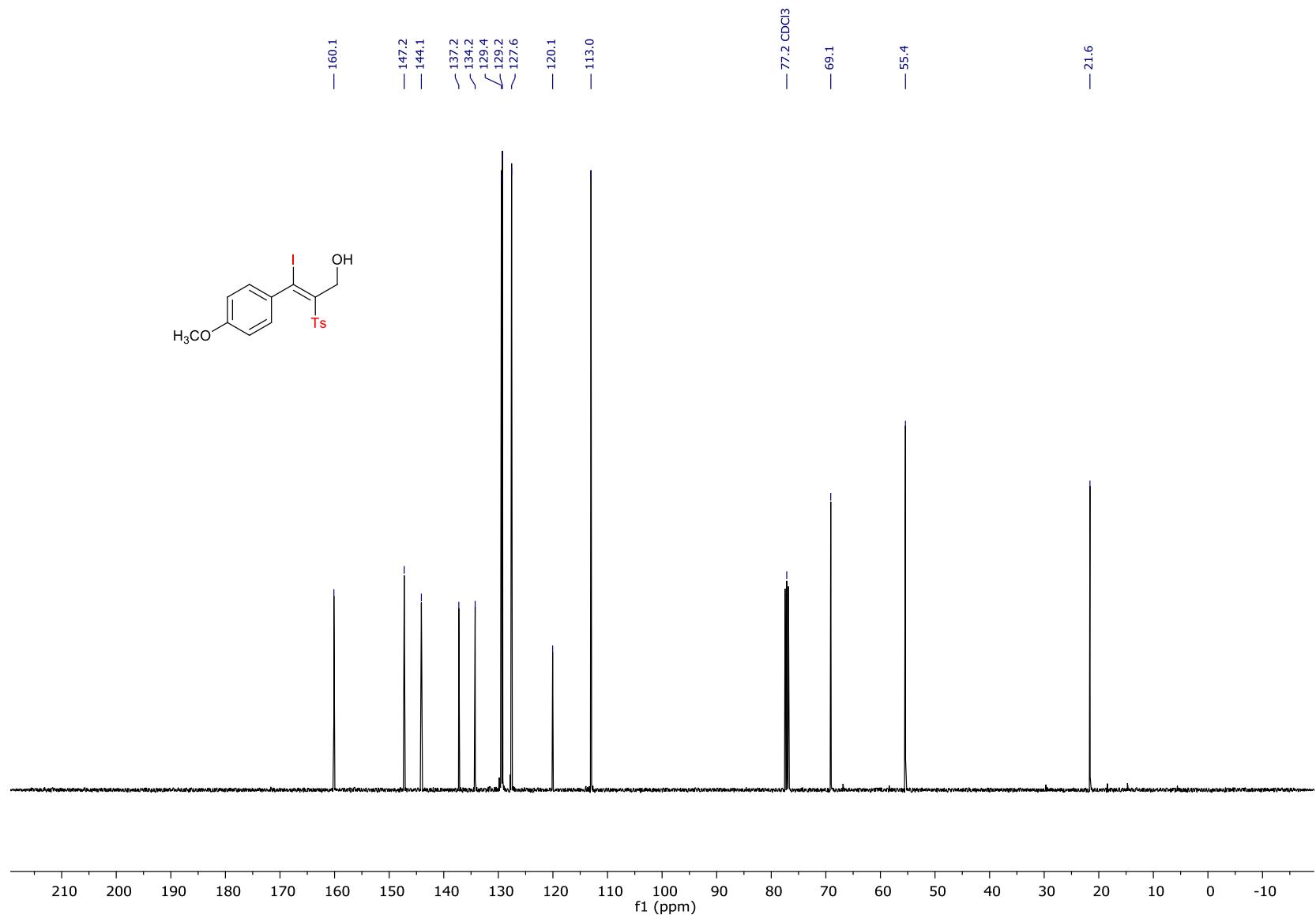


Figure S77. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-3-iodo-3-(4-methoxyphenyl)-2-tosylprop-2-en-1-ol (**3v**).

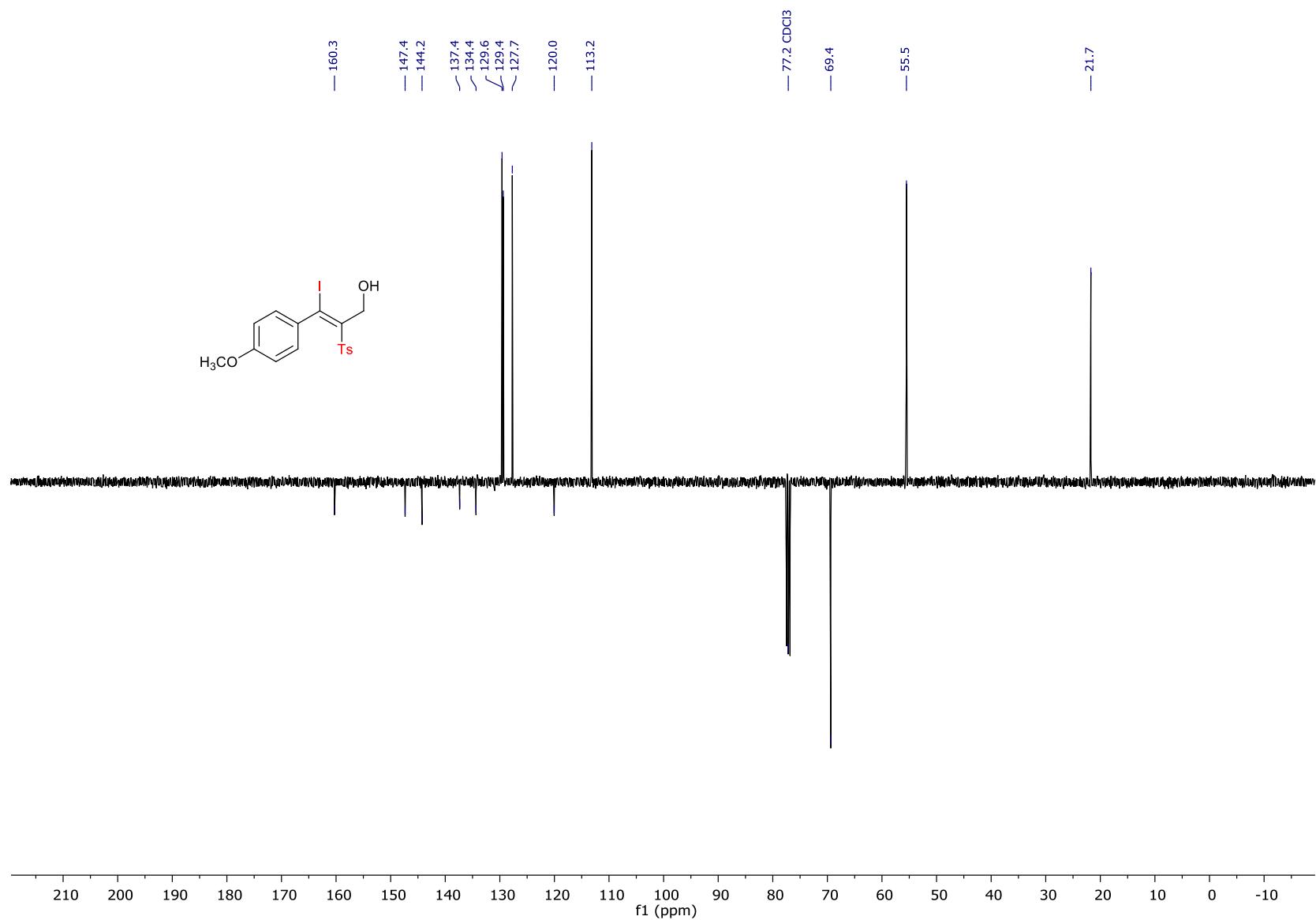


Figure S78. ^{13}C DEPTQ-135 NMR (*E*)-3-iodo-3-(4-methoxyphenyl)-2-tosylprop-2-en-1-ol (**3v**).

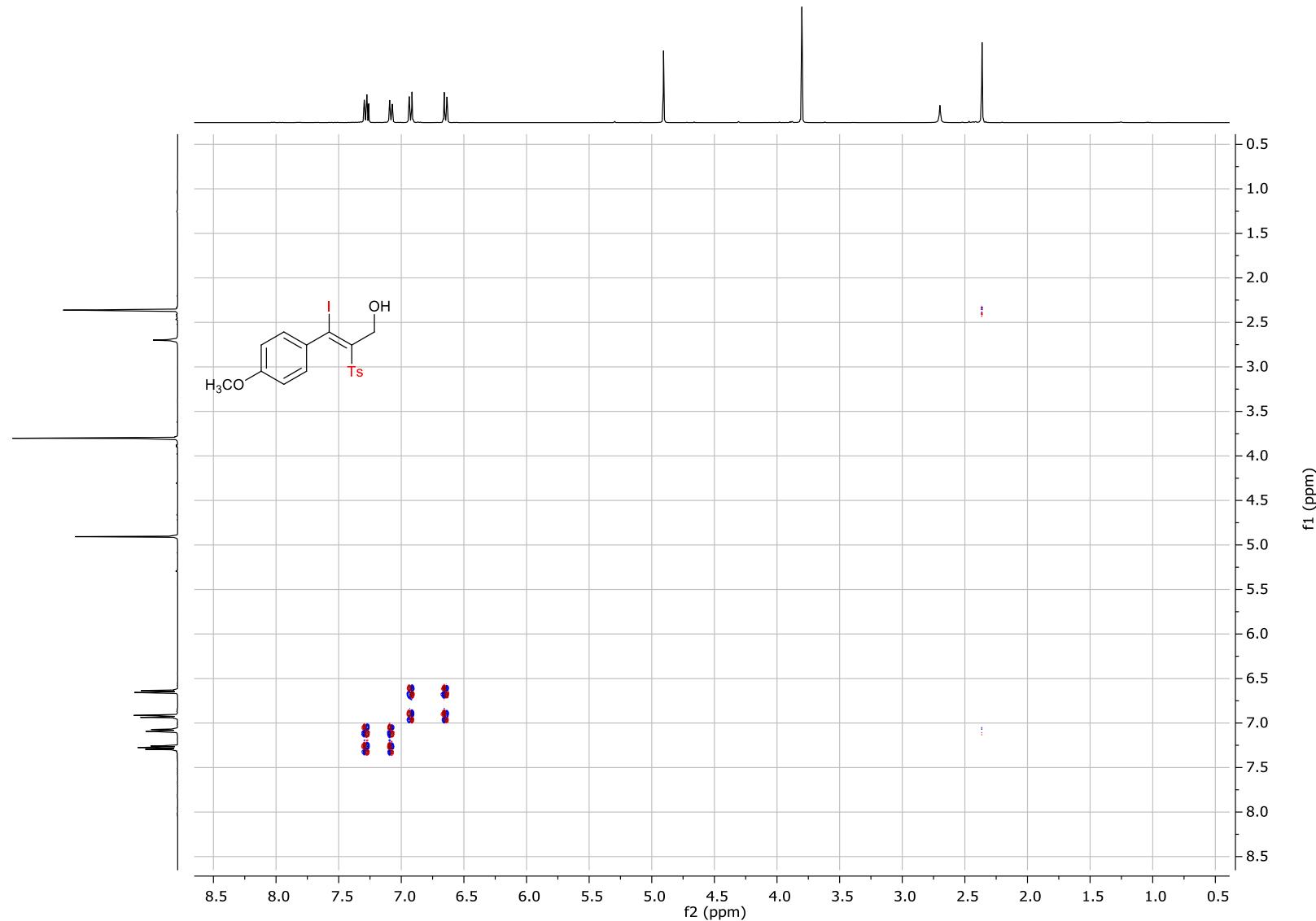


Figure S79. ^1H - ^1H COSY (*E*)-3-iodo-3-(4-methoxyphenyl)-2-tosylprop-2-en-1-ol (3v).

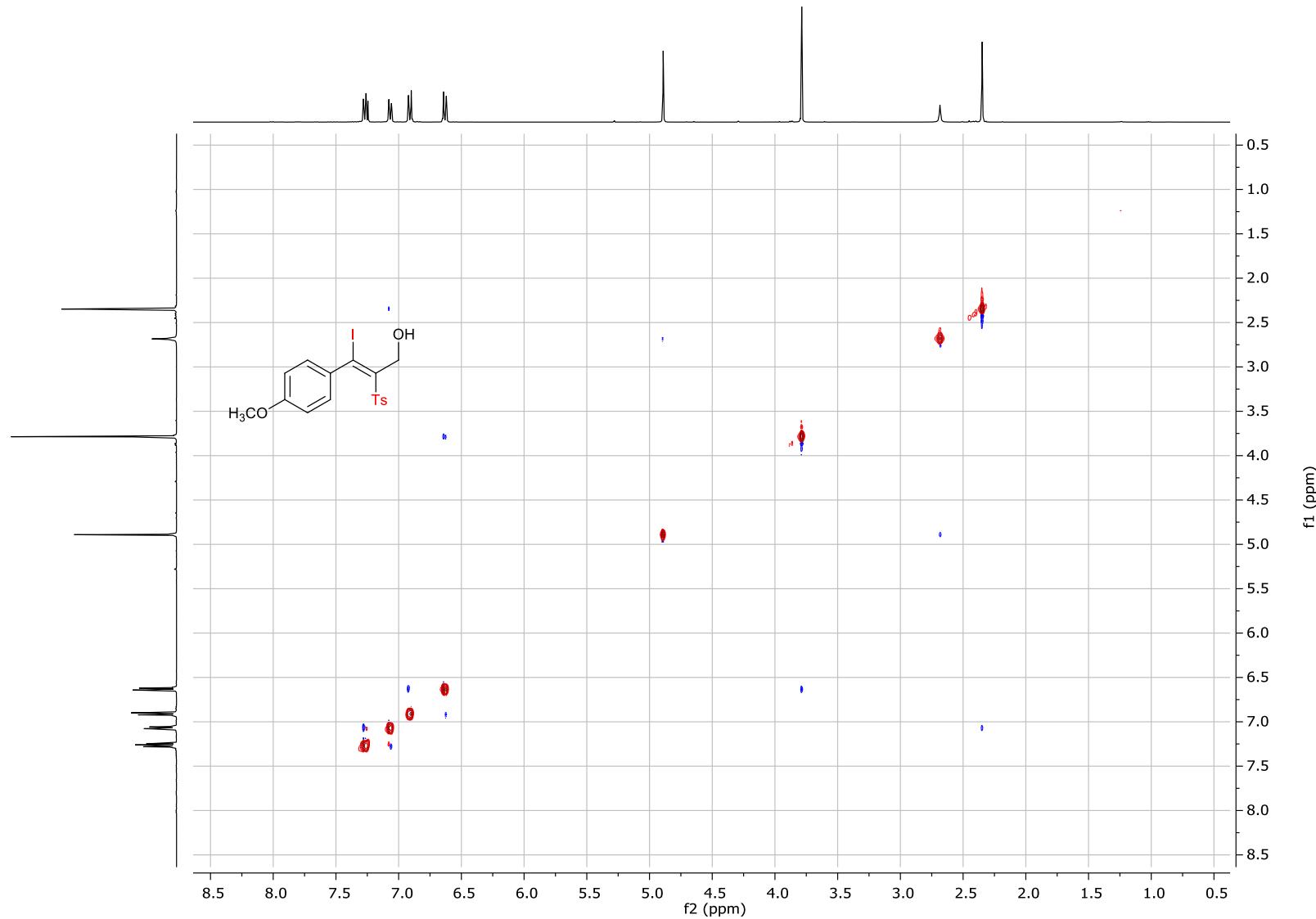
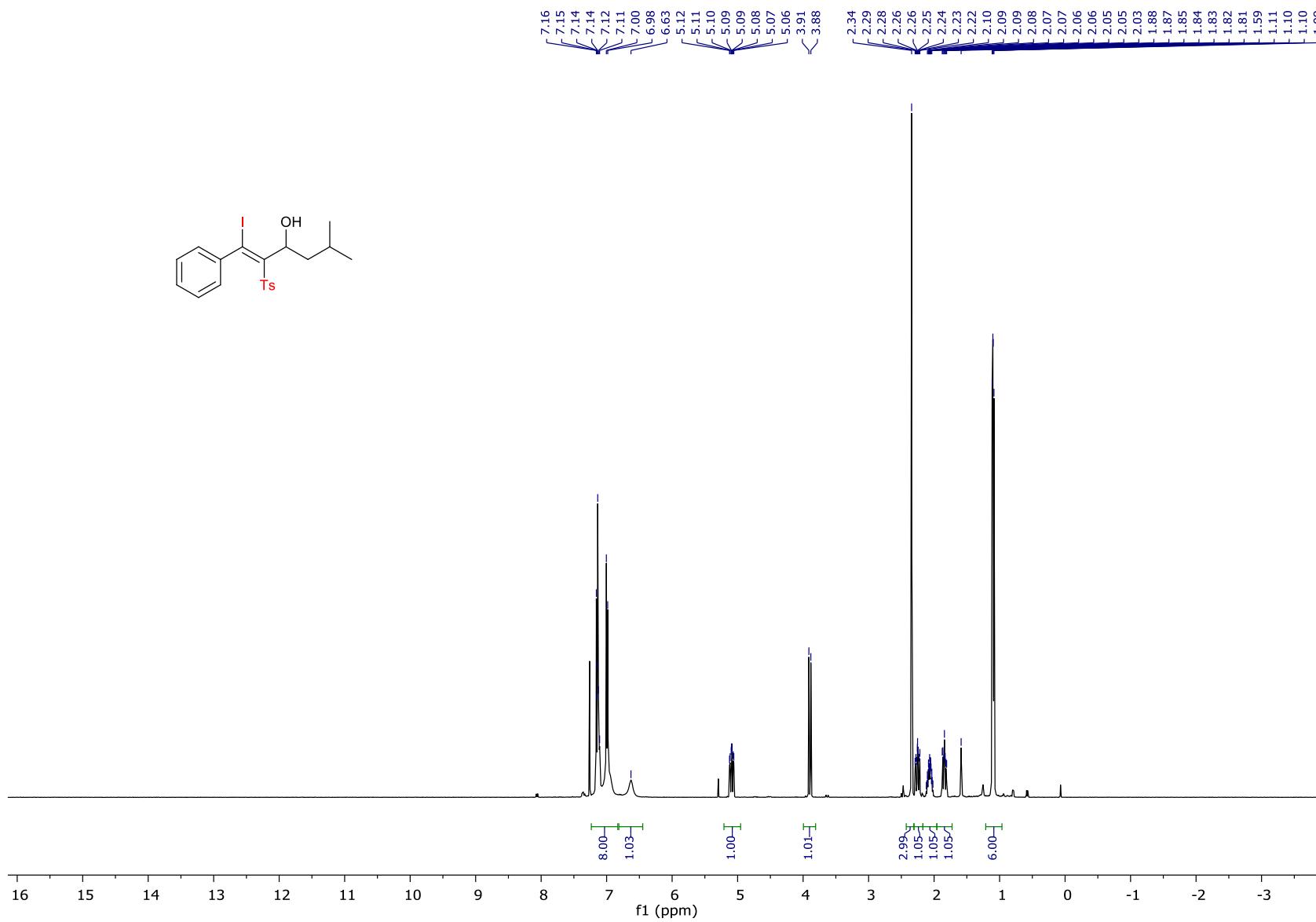


Figure S80. NOESY (*E*)-3-iodo-3-(4-methoxyphenyl)-2-tosylprop-2-en-1-ol (**3v**).



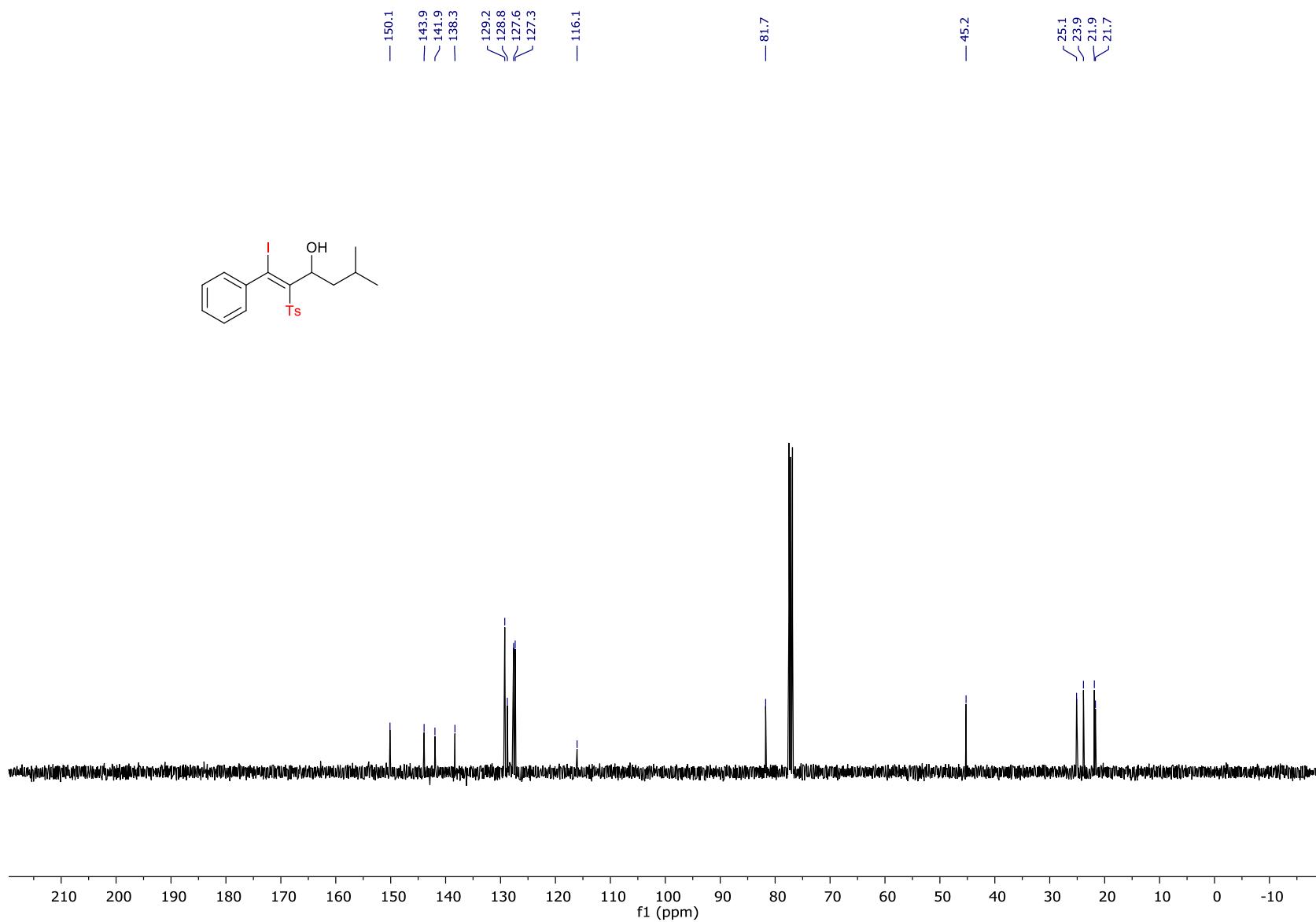


Figure S82. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-1-iodo-5-methyl-1-phenyl-2-tosylhex-1-en-3-ol (**3w**).

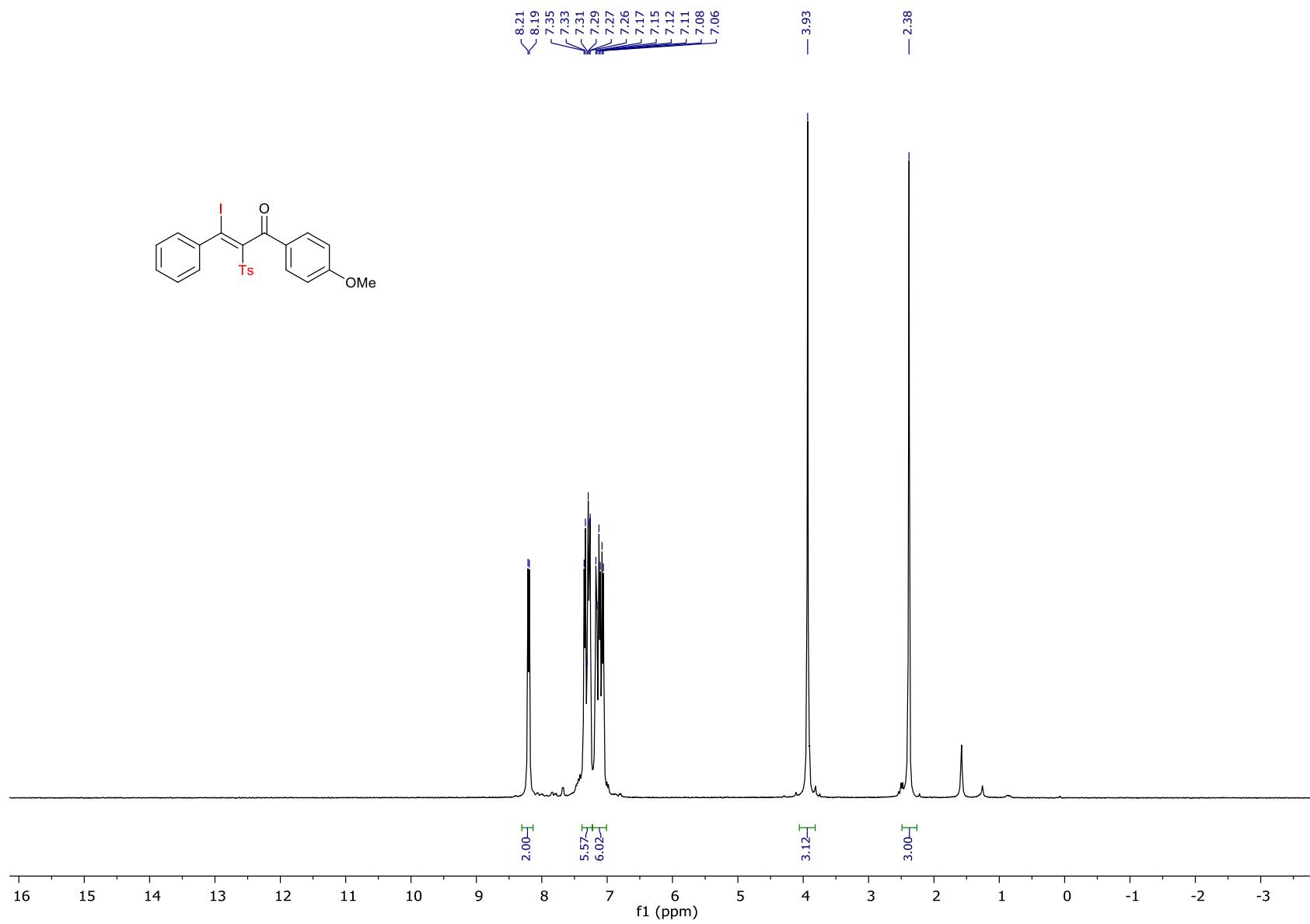


Figure S83. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (**3x**).

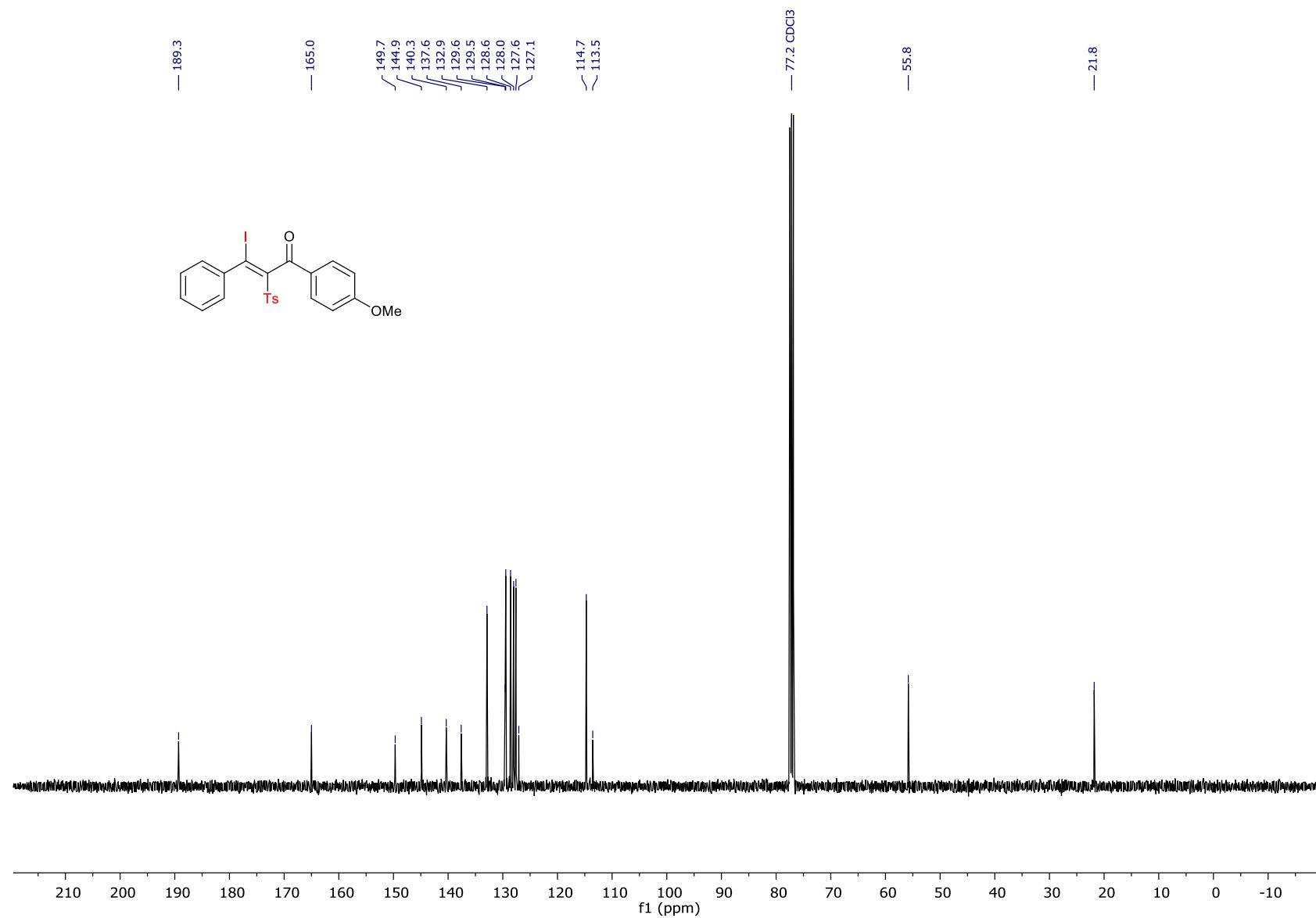


Figure S84. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (*E*)-3-iodo-1-(4-methoxyphenyl)-3-phenyl-2-tosylprop-2-en-1-one (3x).

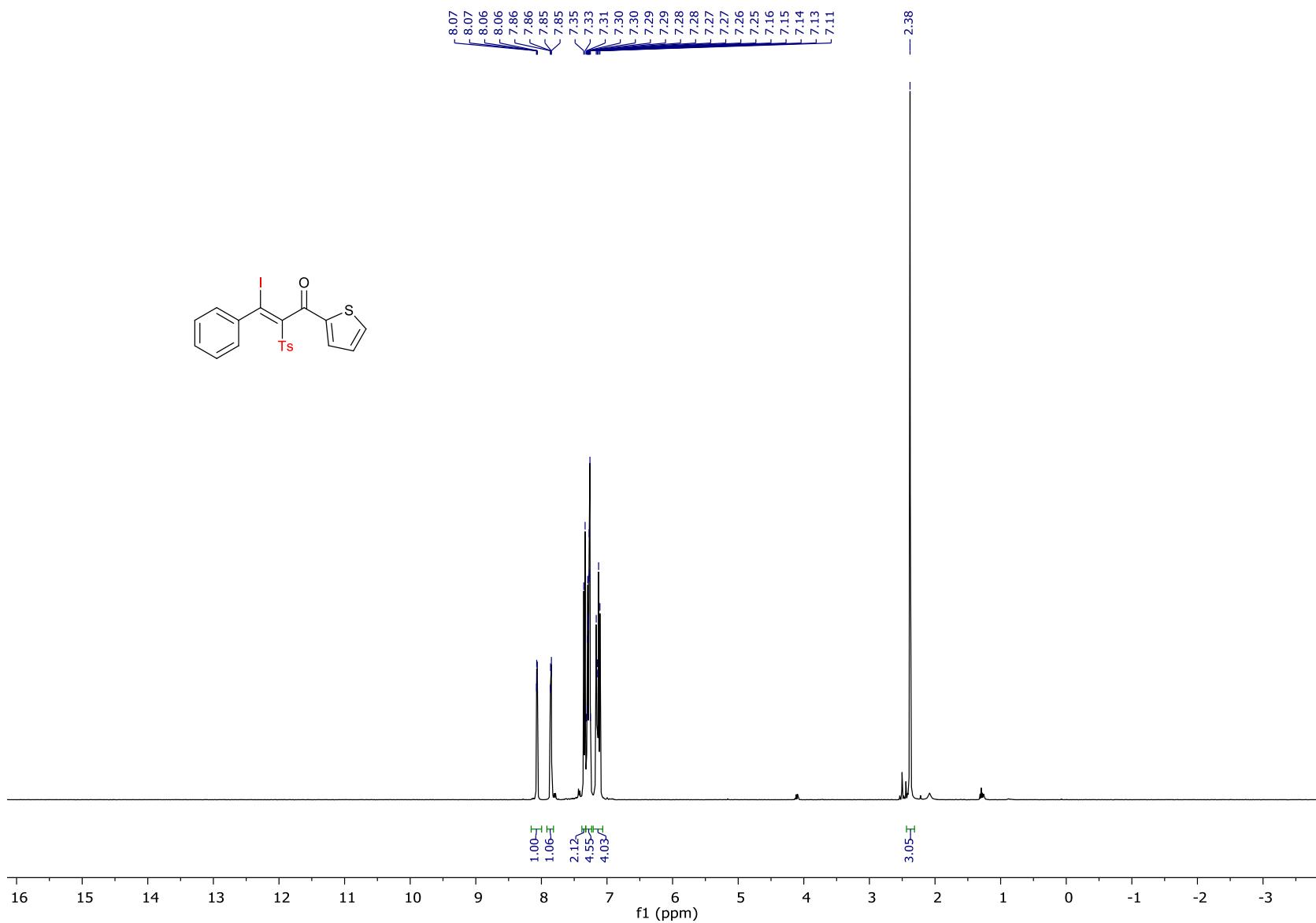


Figure S85. ^1H NMR (600 MHz, Chloroform-d) of (*E*)-3-iodo-3-phenyl-1-(thiophen-2-yl)-2-tosylprop-2-en-1-one (**3y**).

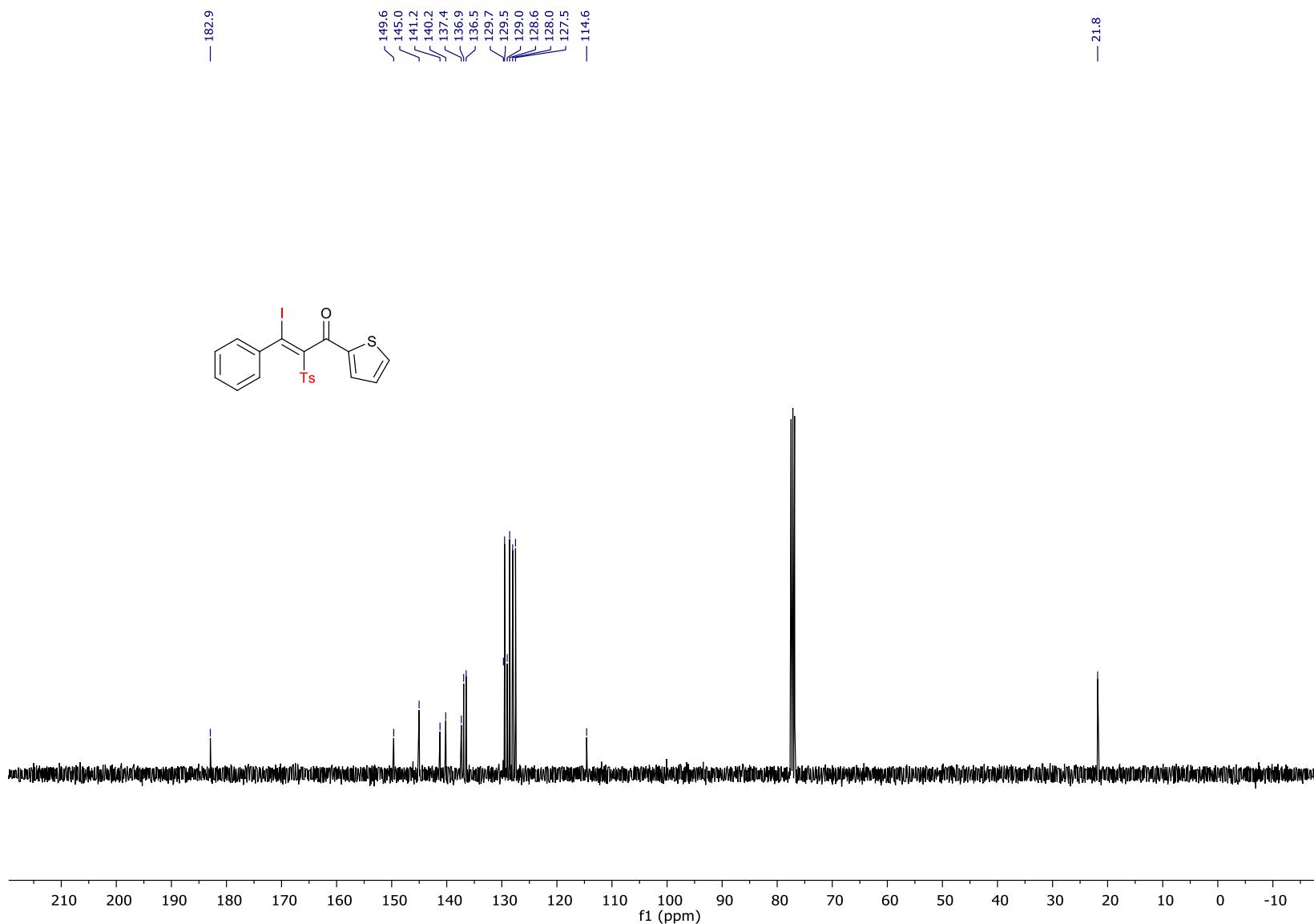


Figure S86. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (*E*)-3-iodo-3-phenyl-1-(thiophen-2-yl)-2-tosylprop-2-en-1-one (**3y**).

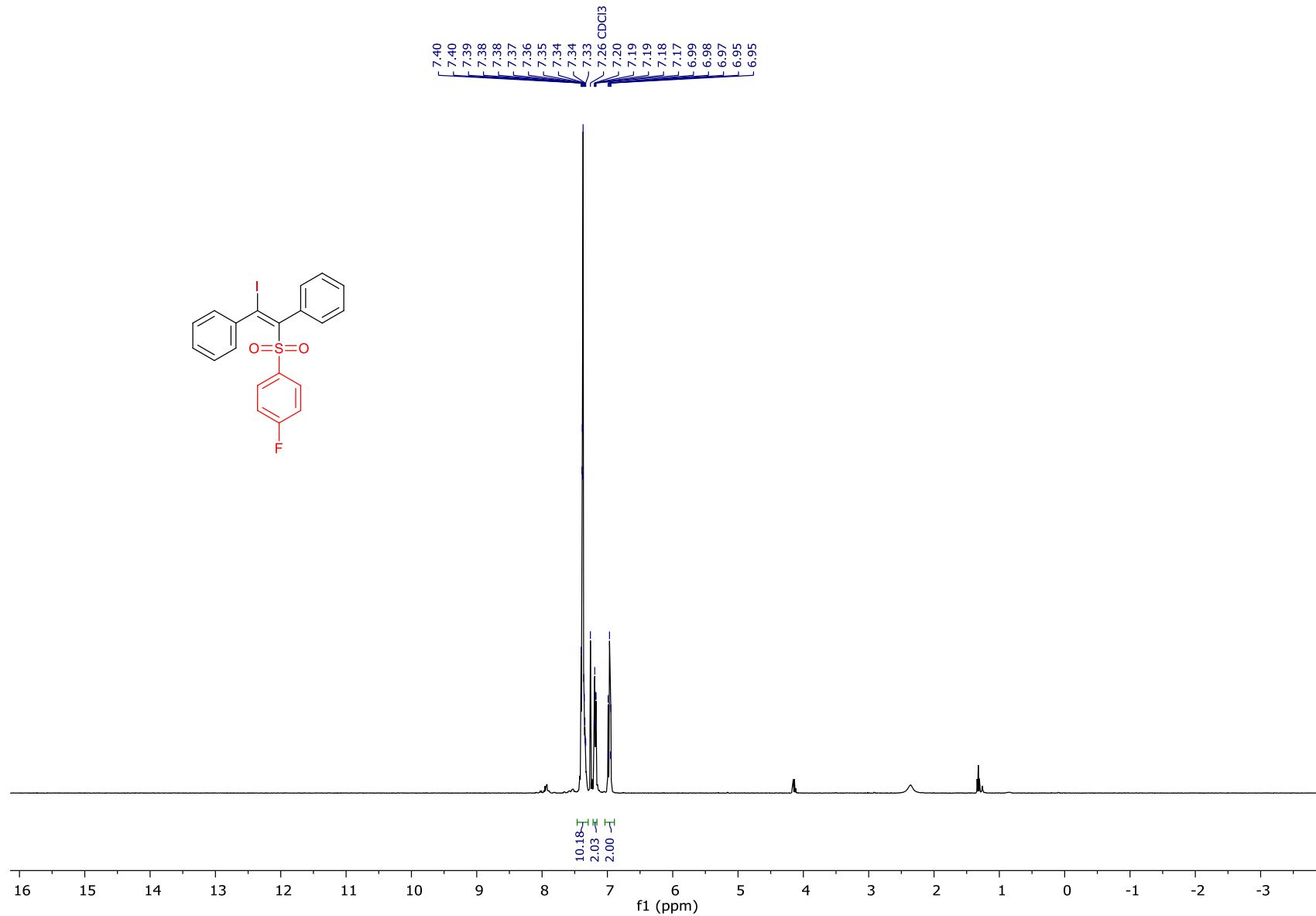
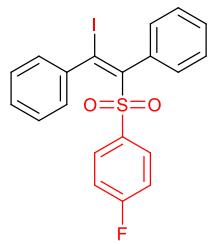


Figure S87. ^1H NMR (600 MHz, CDCl_3) of (*E*)-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (**4a**).

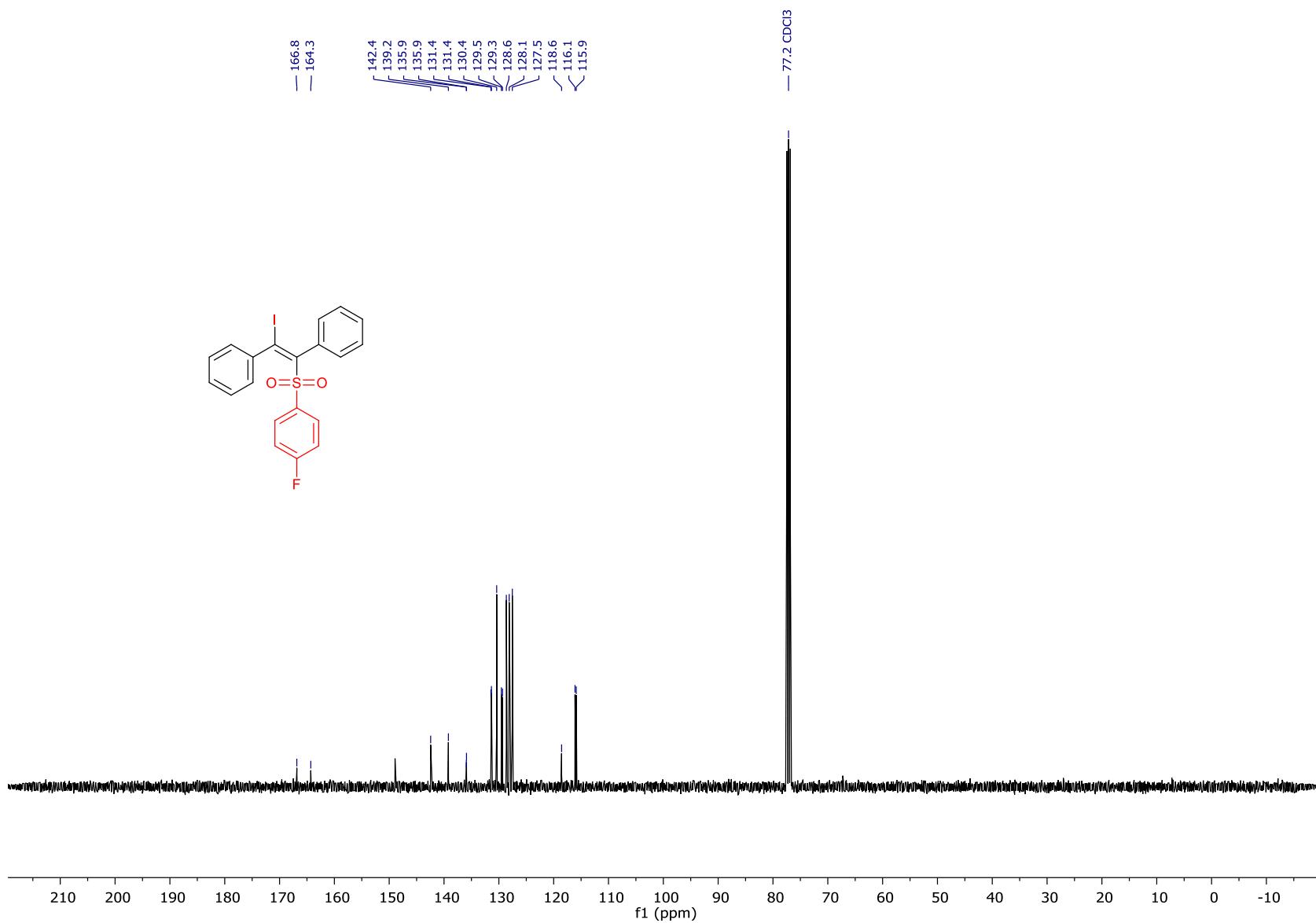


Figure S88. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl₃) of (E)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (4a).

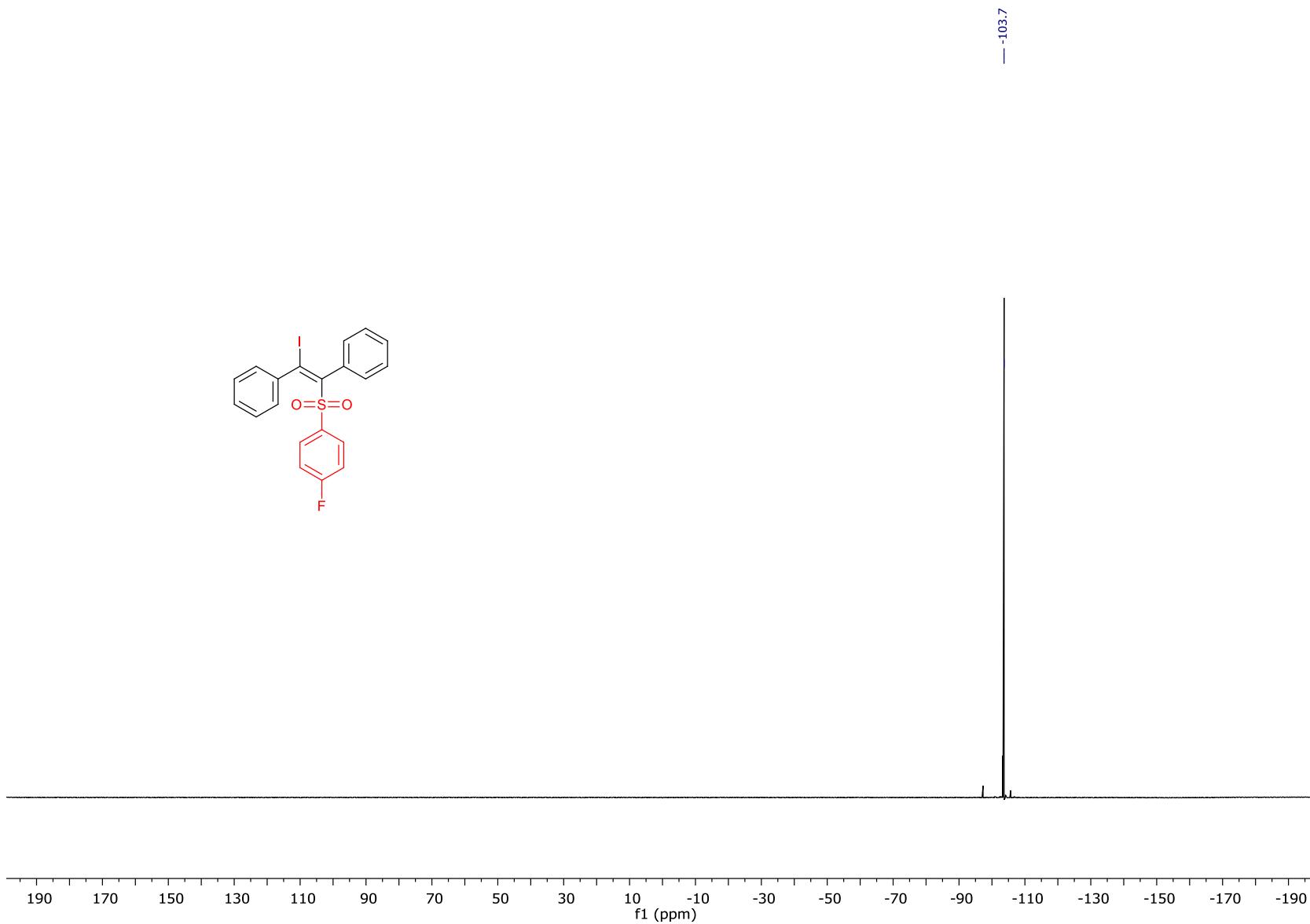


Figure S89. ^{19}F NMR (188 MHz, Chloroform-*d*) of (*E*)-(1-(4-fluorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (**4a**).

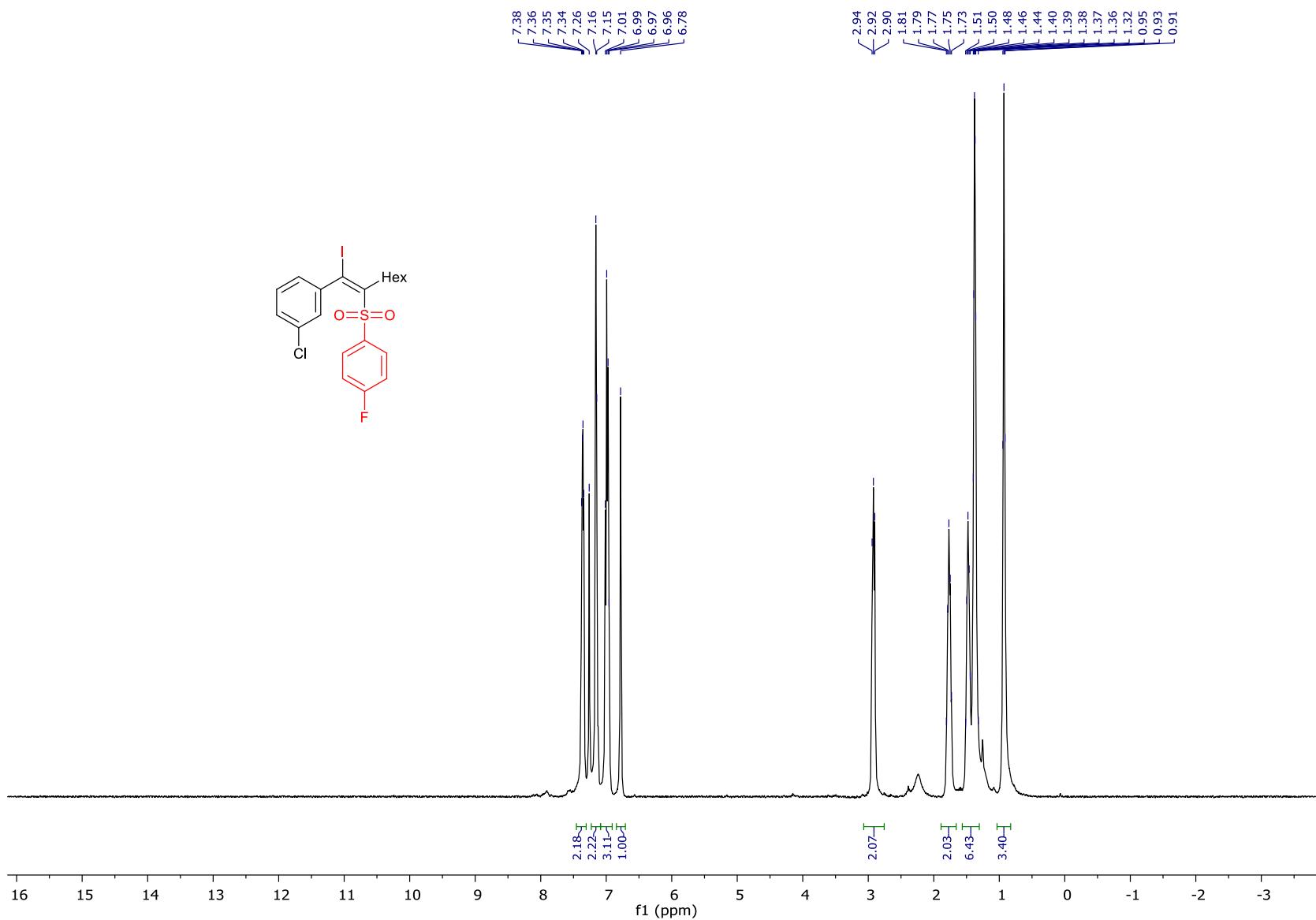


Figure S90. ^1H NMR (600 MHz, CDCl_3) of (*E*)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (**4b**).

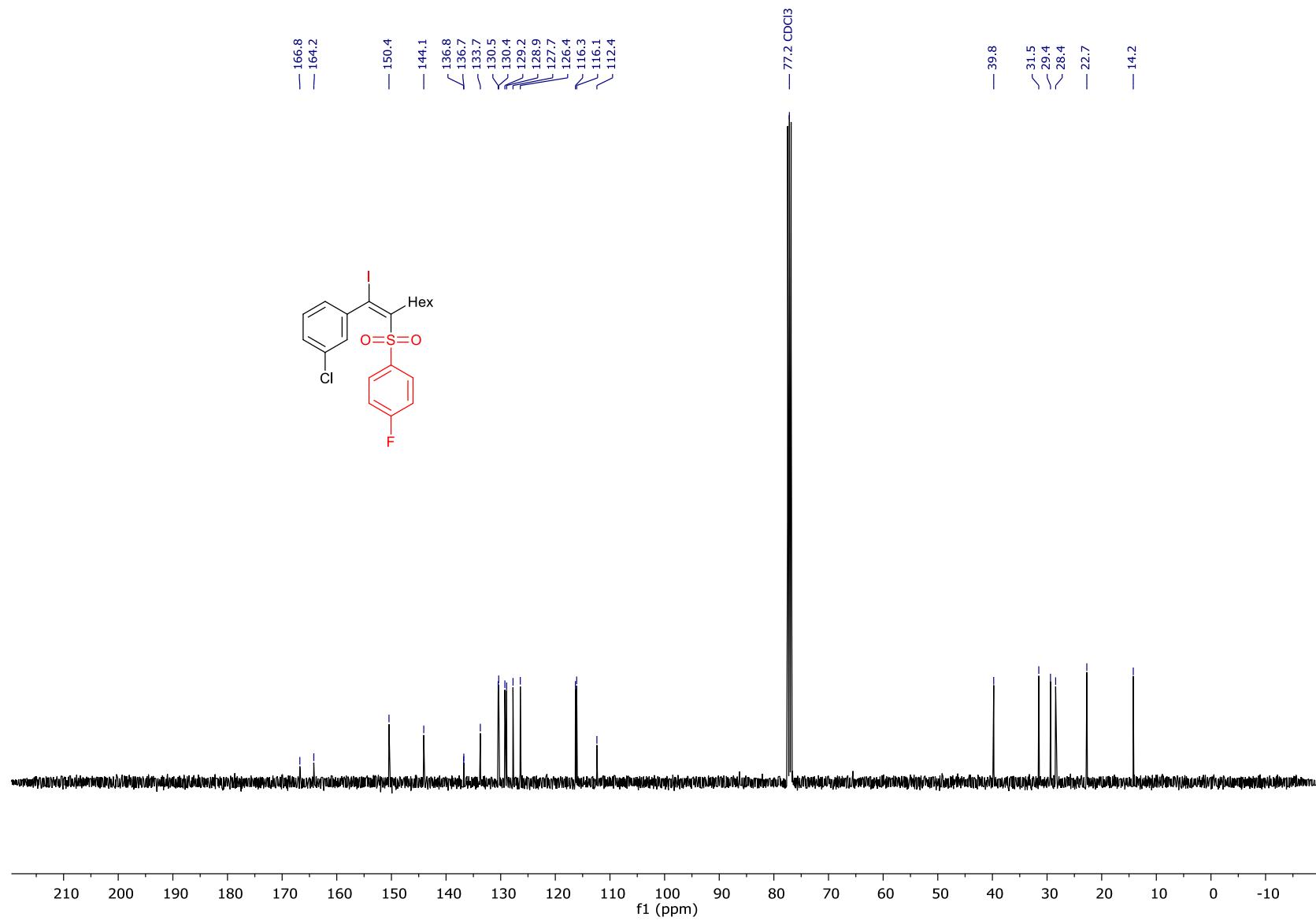


Figure S91. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl_3) of (*E*)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (4b).

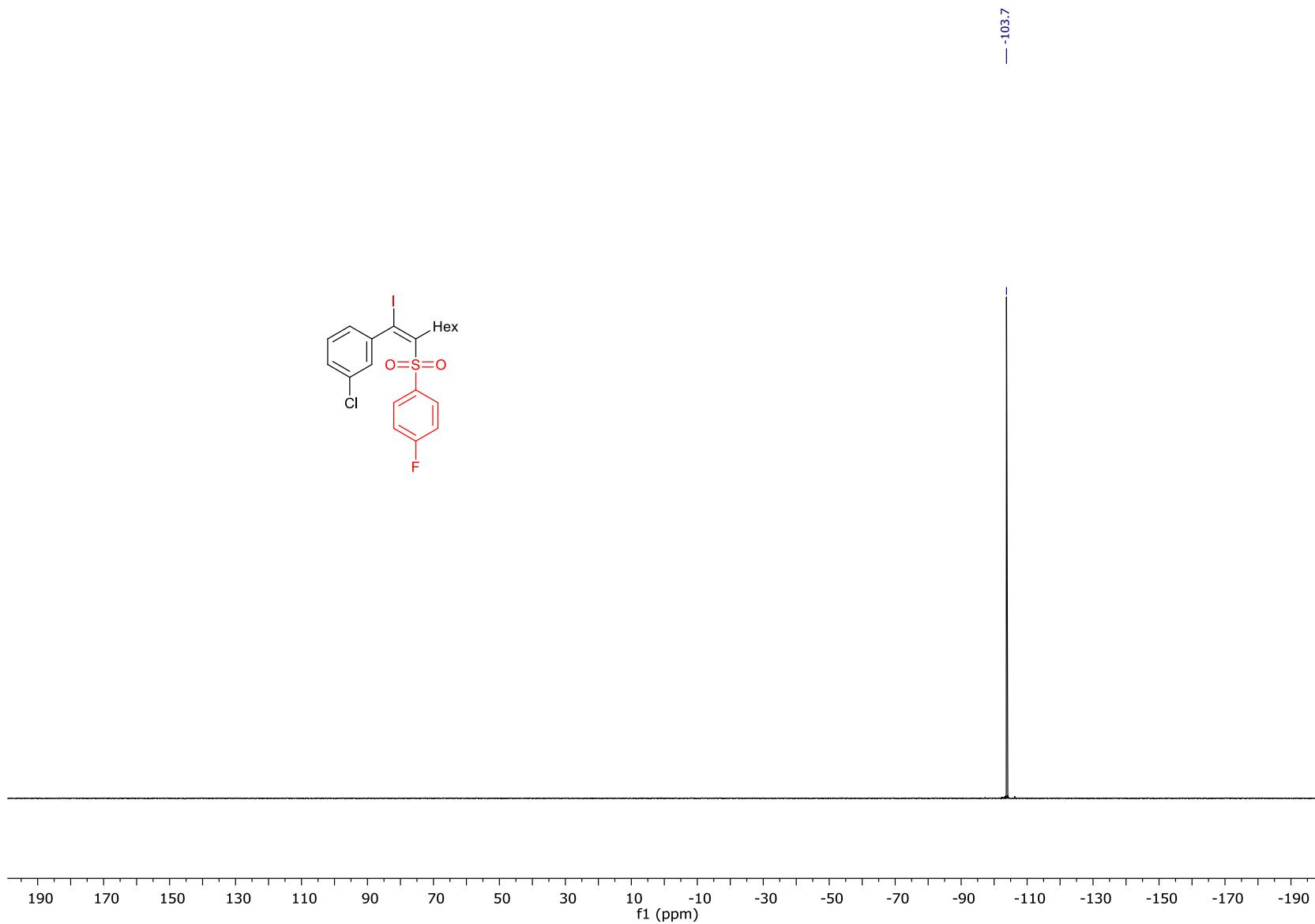


Figure S92. ^{19}F NMR (188 MHz, Chloroform-*d*) of (*E*)-1-chloro-3-(2-(4-fluorophenylsulfonyl)-1-iodooct-1-enyl)benzene (**4b**).

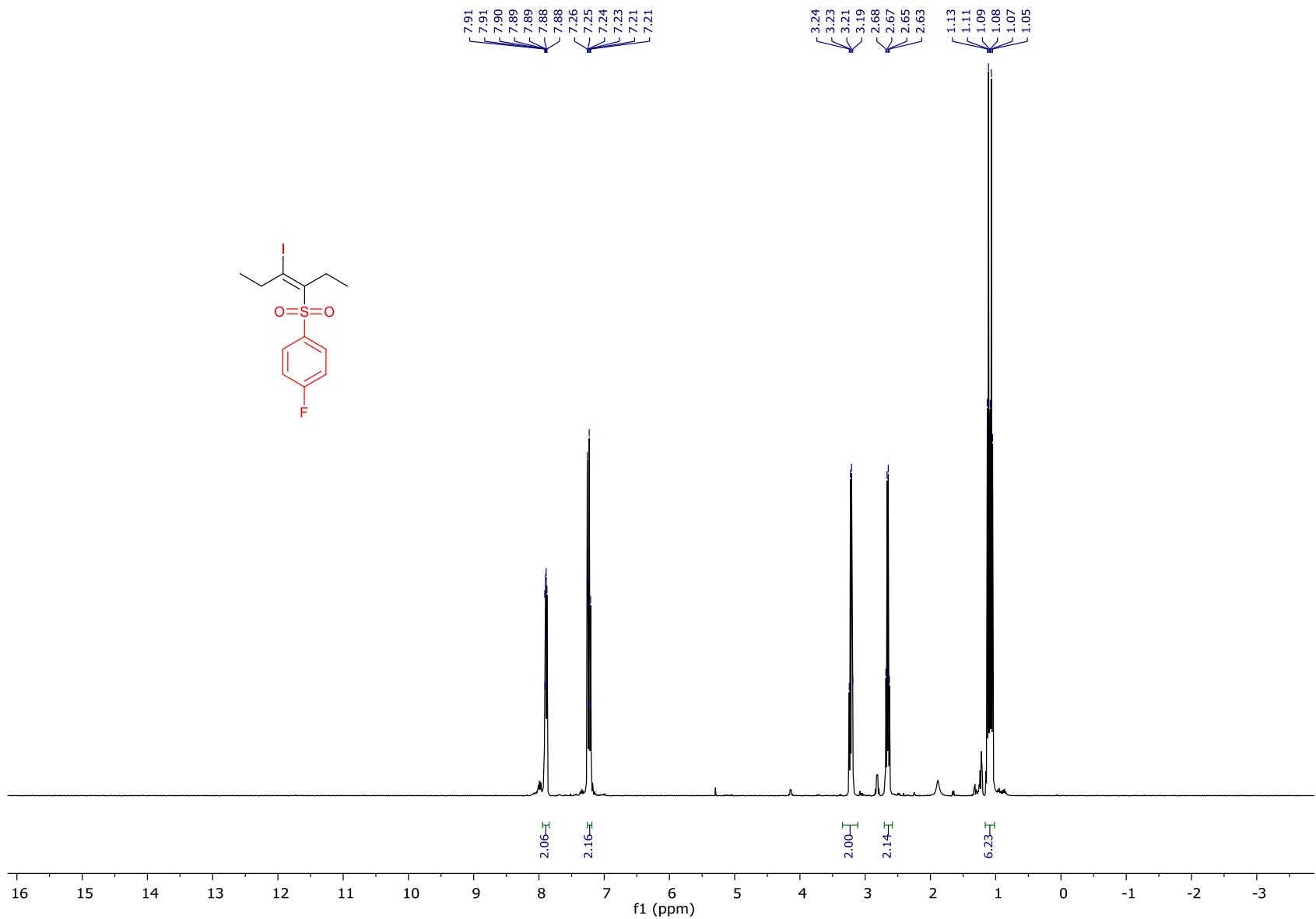


Figure S93. ^1H NMR (600 MHz, CDCl_3) of (*E*)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (**4c**).

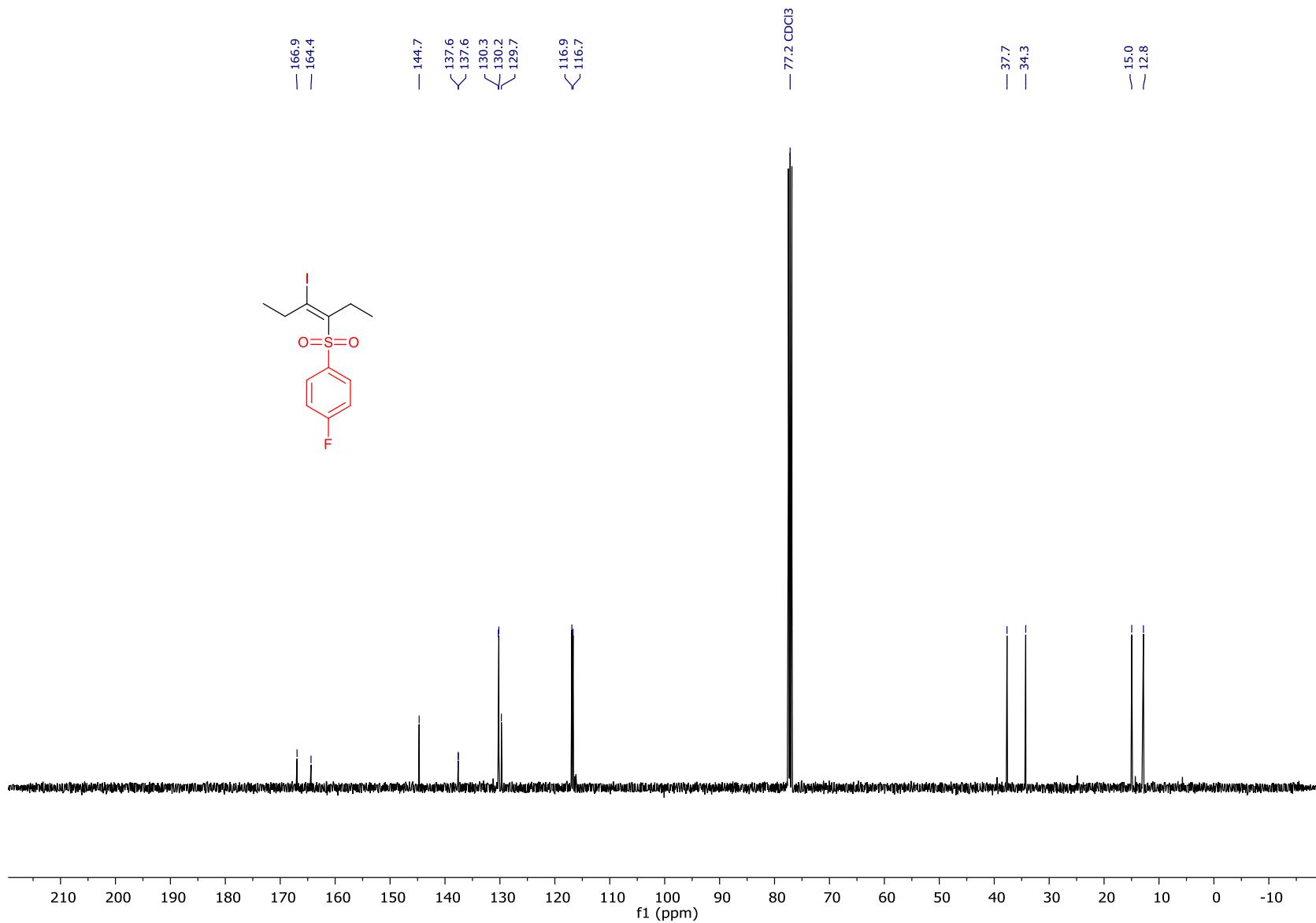


Figure S94. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, CDCl_3) of (E)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (4c).

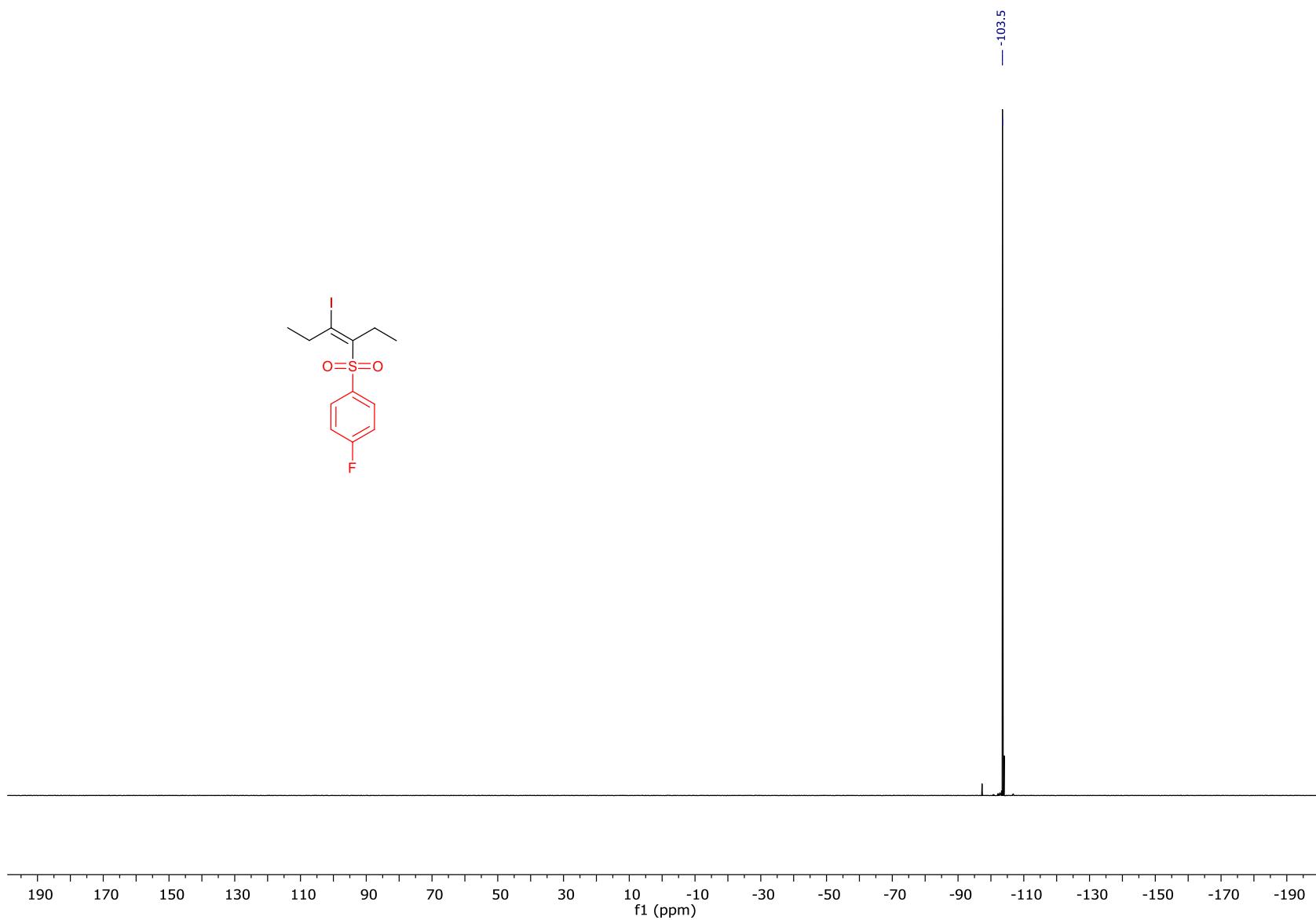


Figure S95. ^{19}F NMR (188 MHz, Chloroform-*d*) of (*E*)-1-fluoro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (**4c**).

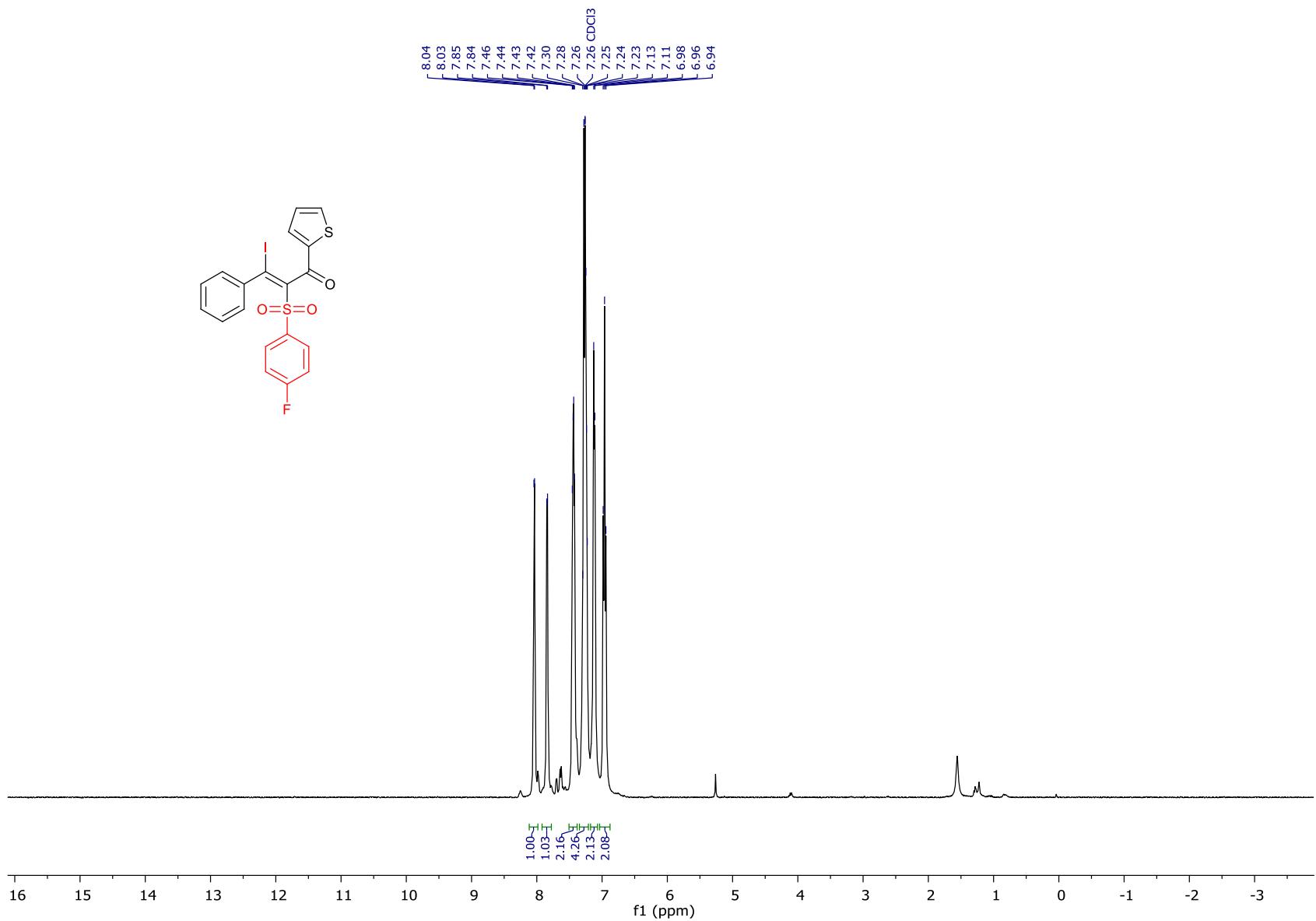


Figure S96. ^1H NMR (600 MHz, CDCl_3) of (*E*)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (**4d**).

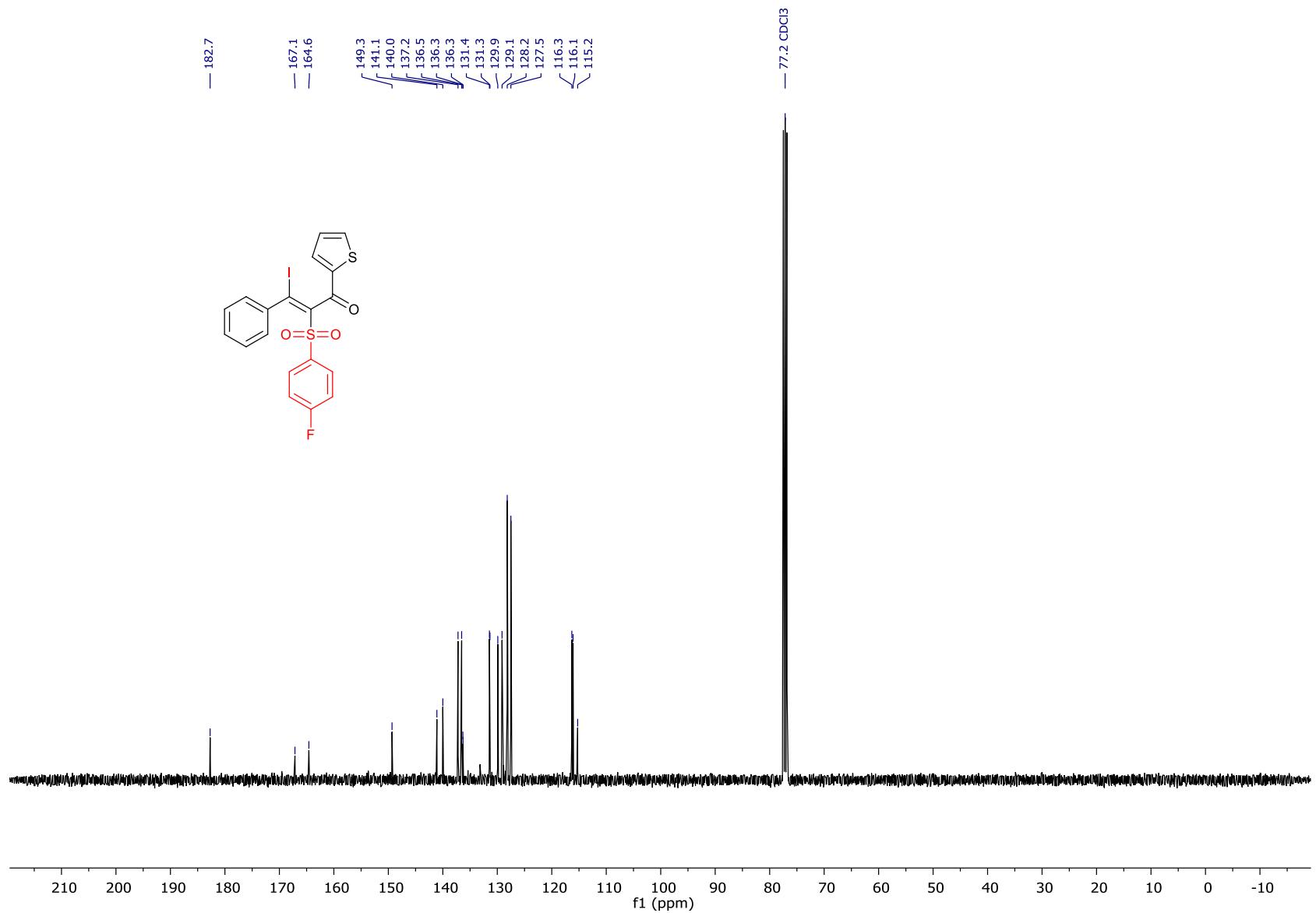


Figure S97. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl_3) of (*E*)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (**4d**).

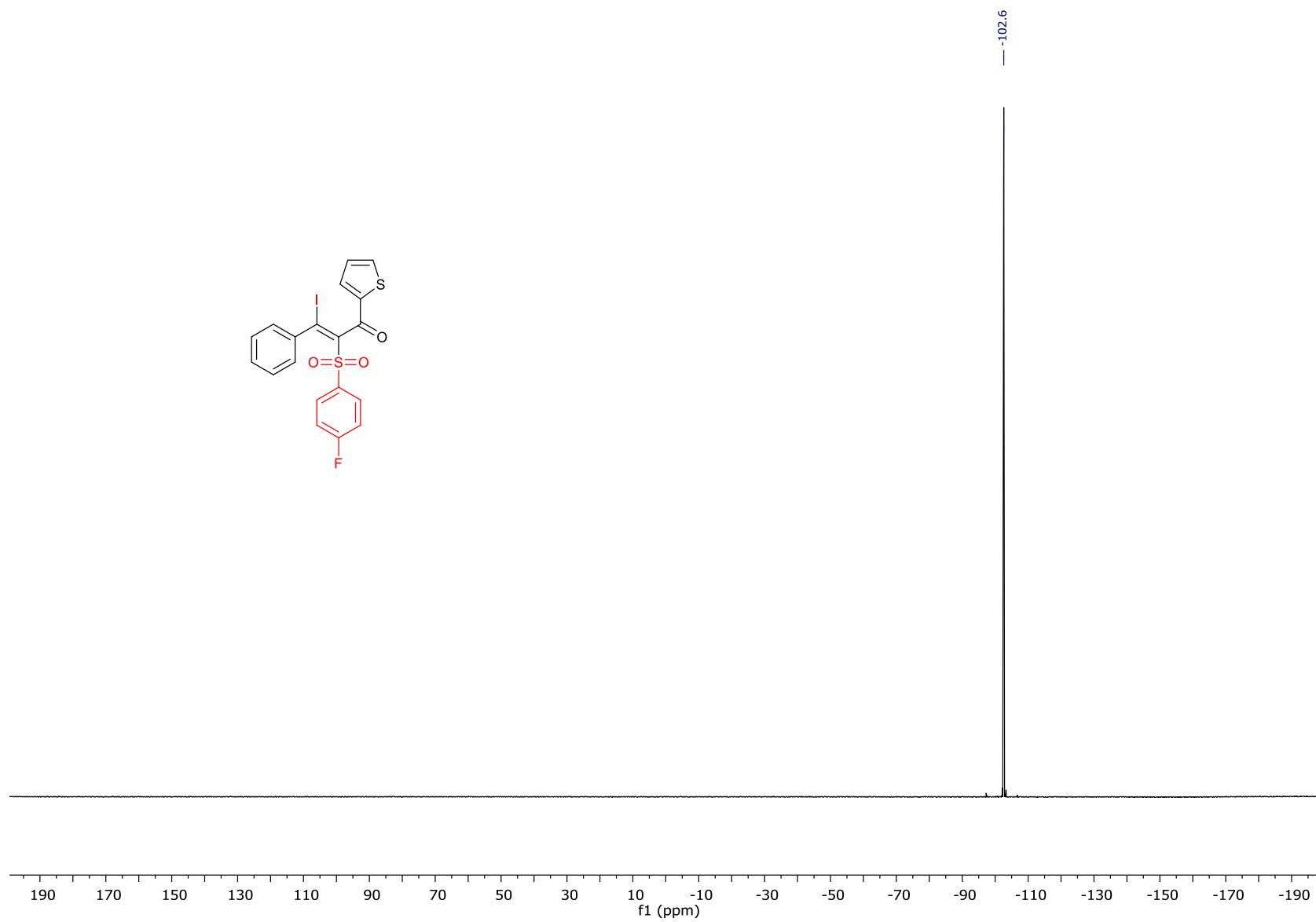


Figure S98. ^{19}F NMR (188 MHz, Chloroform-*d*) of (*E*)-2-(4-fluorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (**4d**).

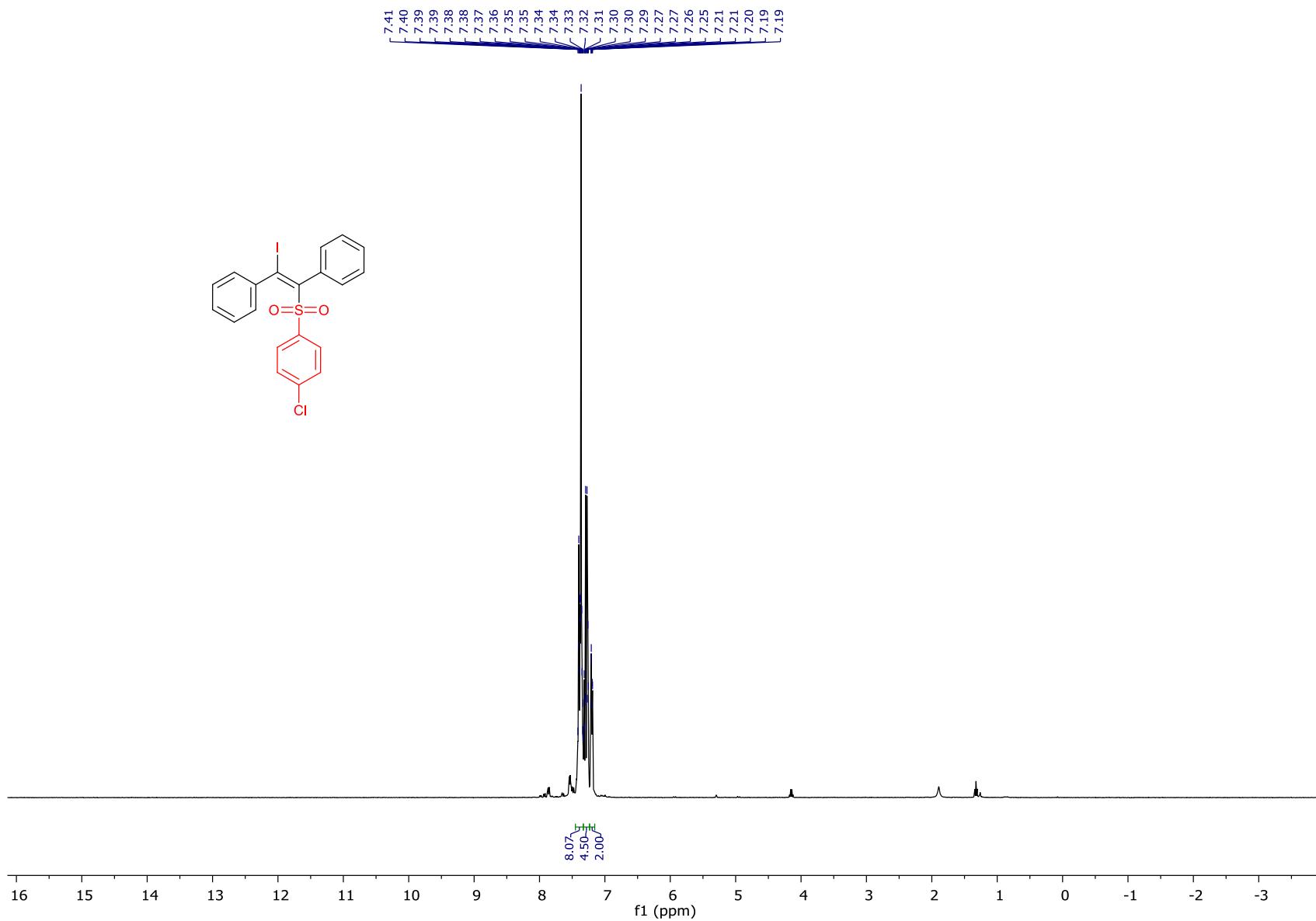


Figure S99. ^1H NMR (600 MHz, CDCl_3) of (*E*)-(1-(4-chlorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (**4e**).

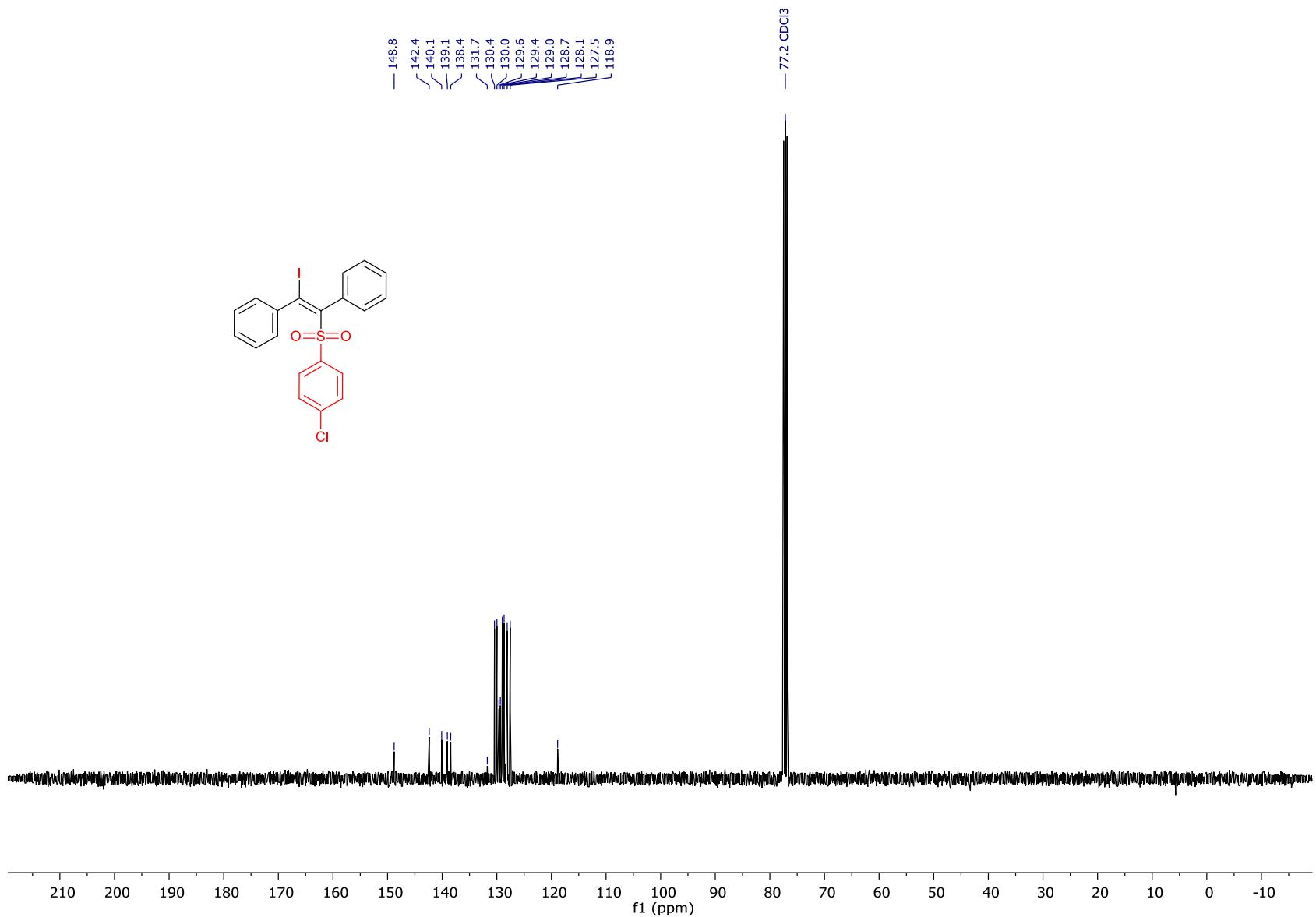


Figure S100. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, CDCl_3) of (E)-(1-(4-chlorophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (**4e**).

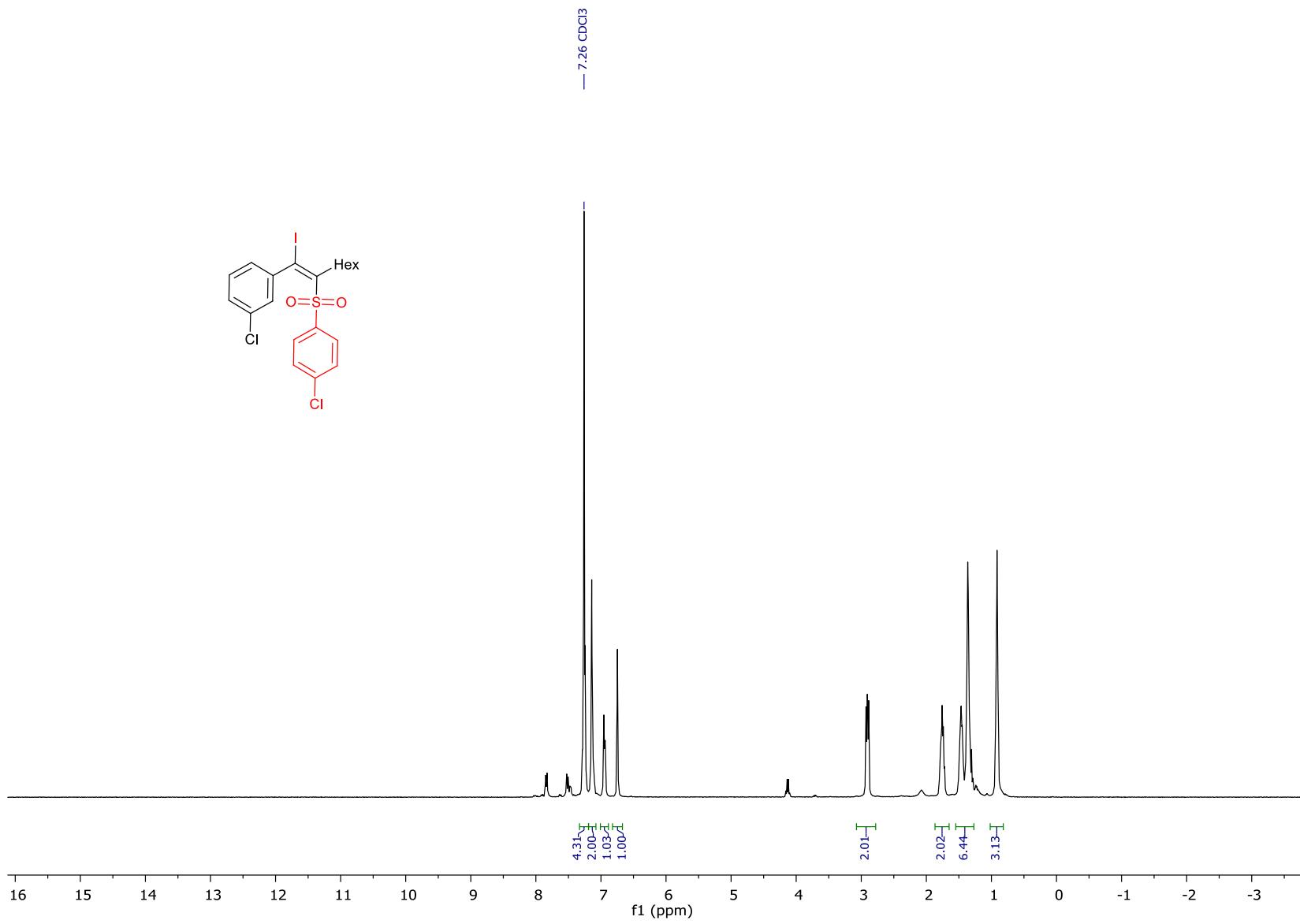
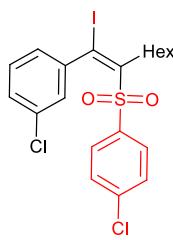


Figure S101. ^1H NMR (600 MHz, CDCl_3) of (*E*)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (**4f**).

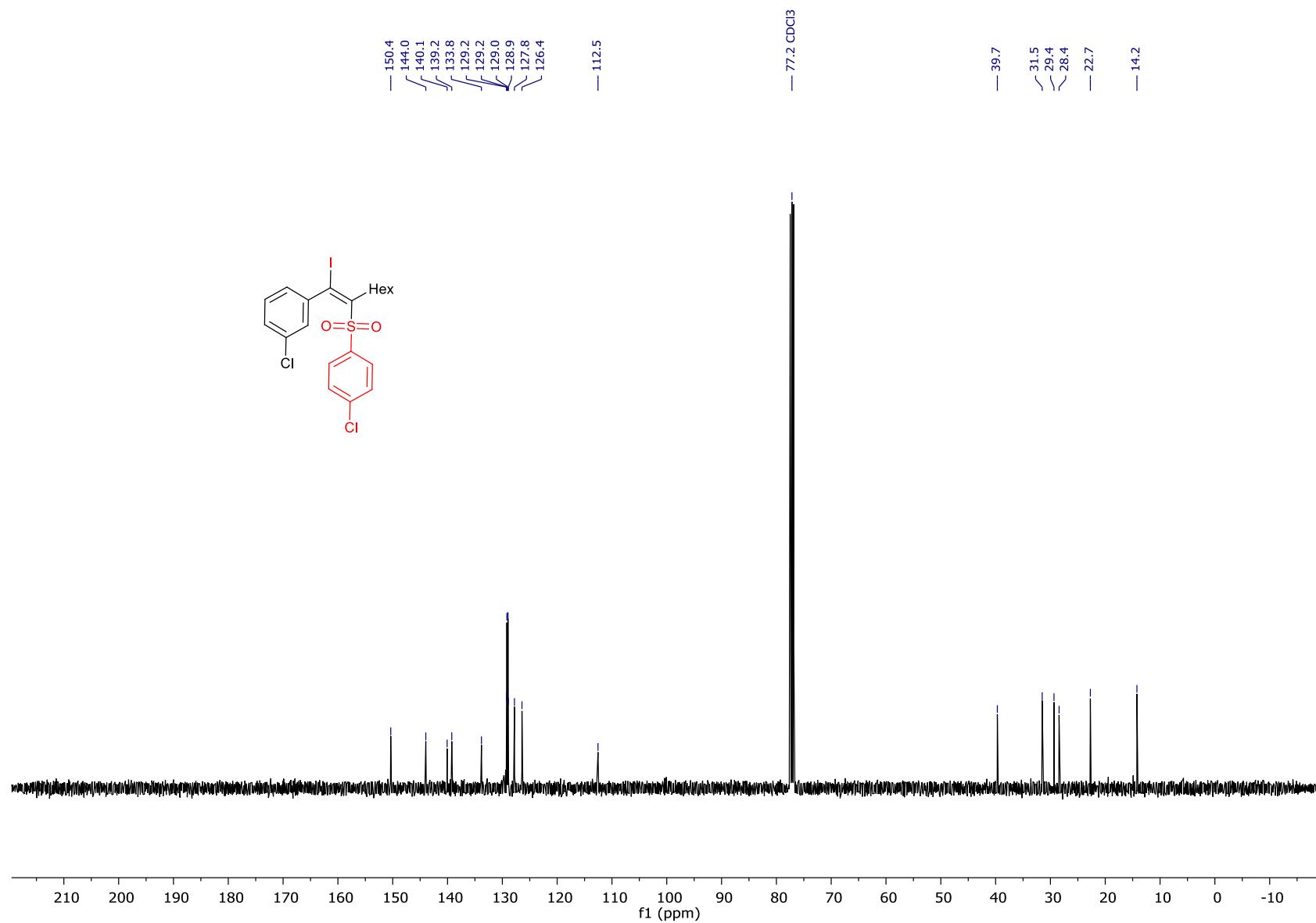


Figure S102. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, CDCl₃) of (*E*)-1-chloro-3-(2-(4-chlorophenylsulfonyl)-1-iodooct-1-enyl)benzene (**4f**).

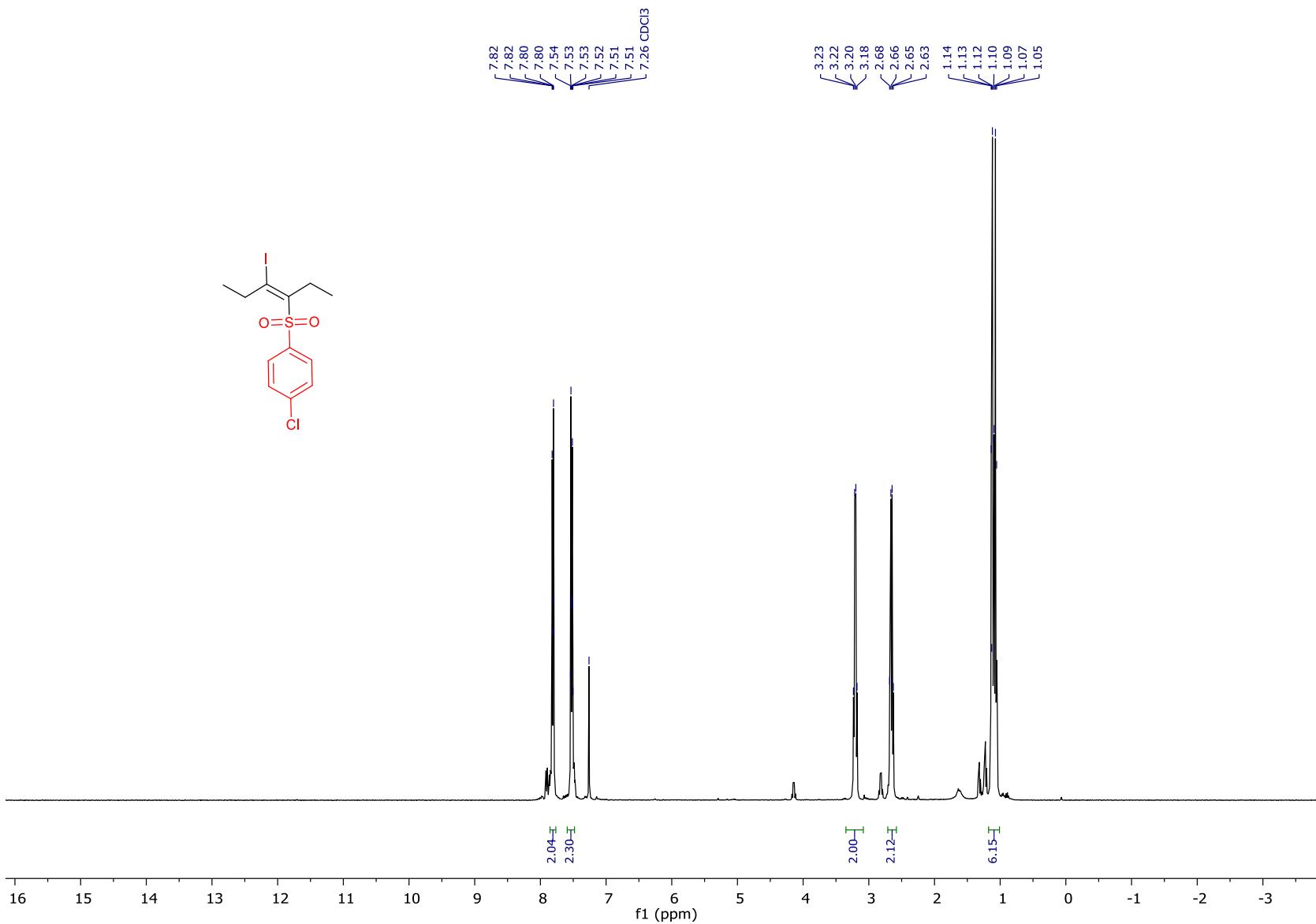


Figure S103. ^1H NMR (600 MHz, CDCl₃) of (E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (4g).

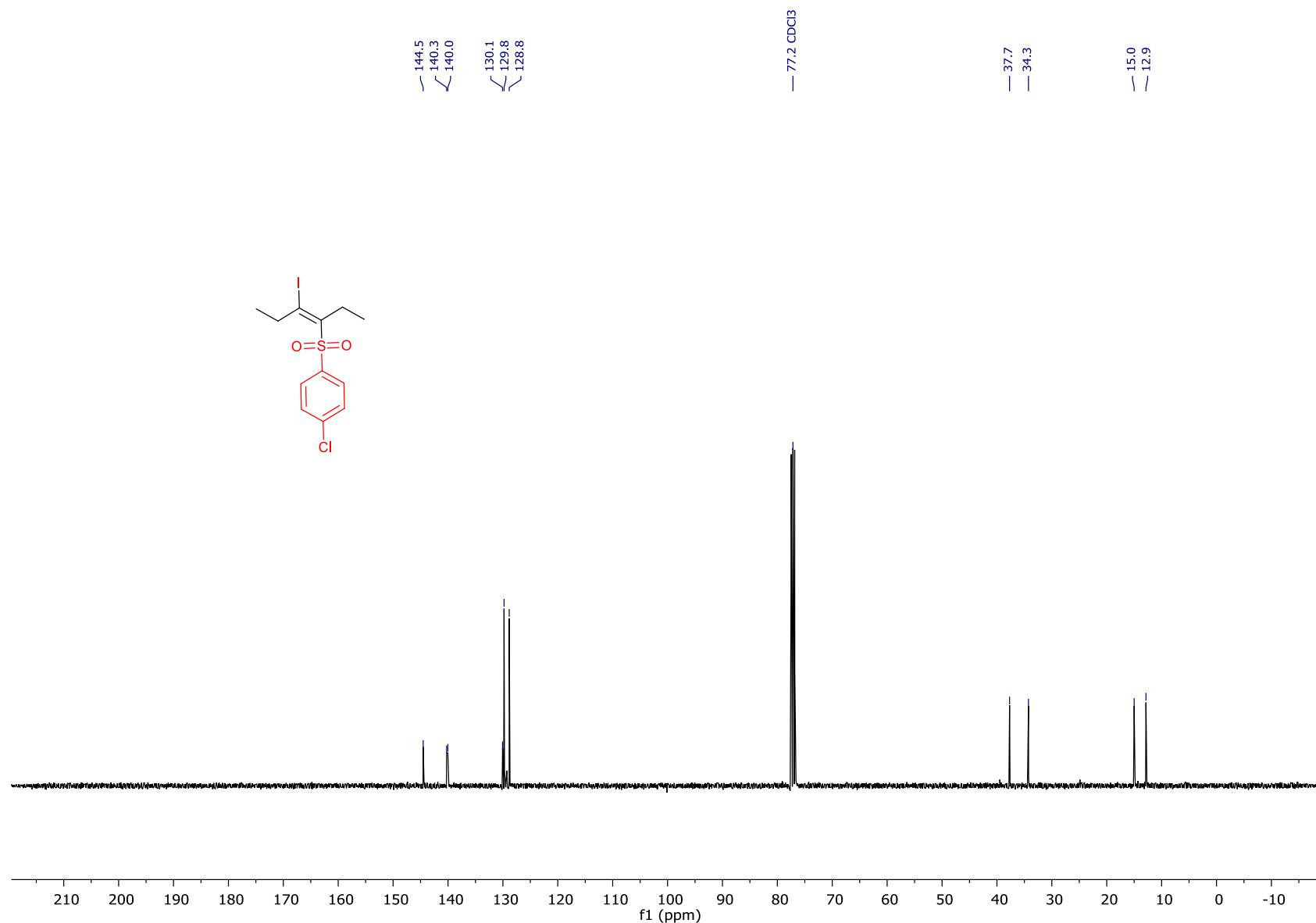


Figure S104. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl_3) of (E)-1-chloro-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (**4g**).

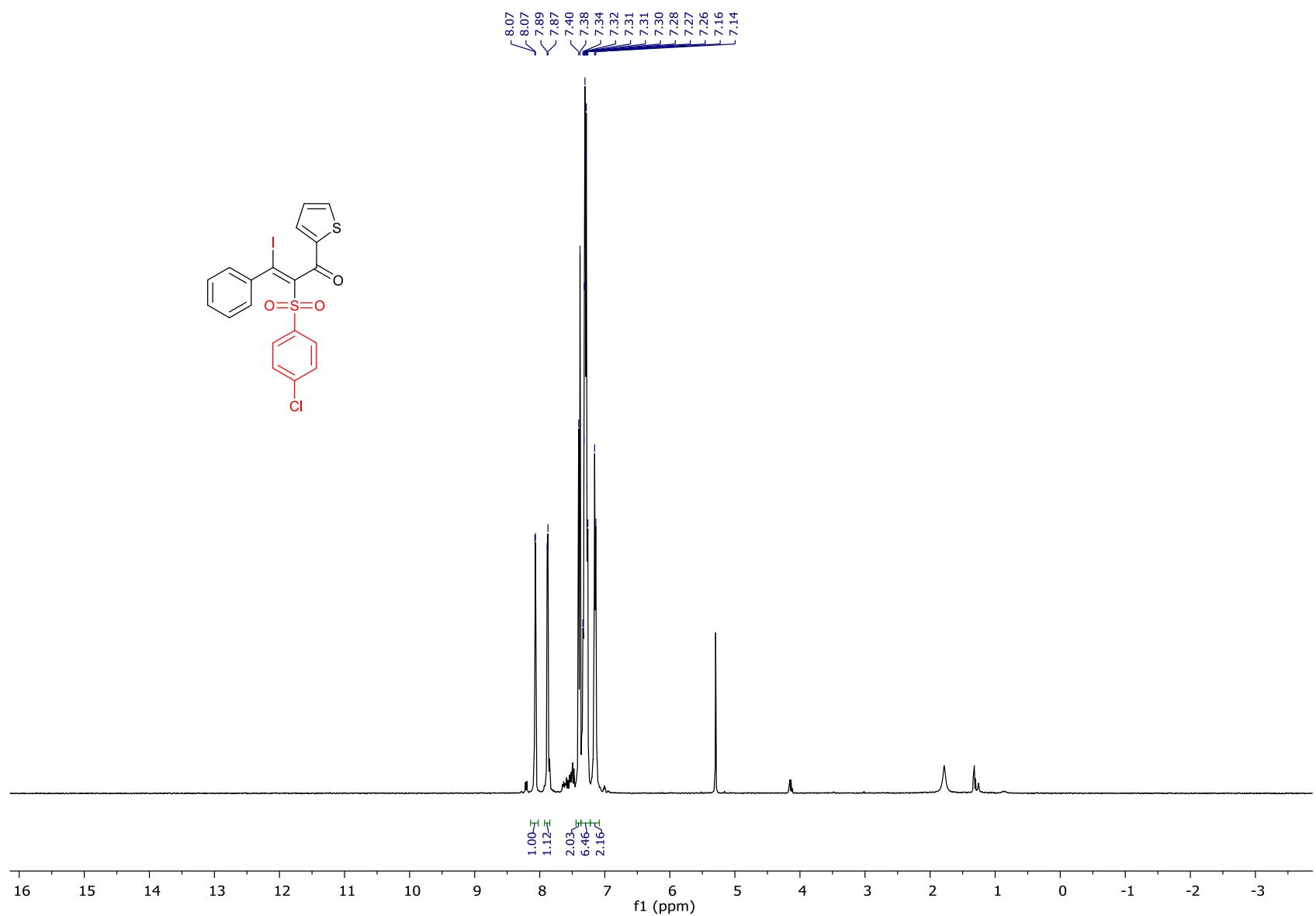


Figure S105. ^1H NMR (600 MHz, CDCl_3) of (*E*)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (**4h**).

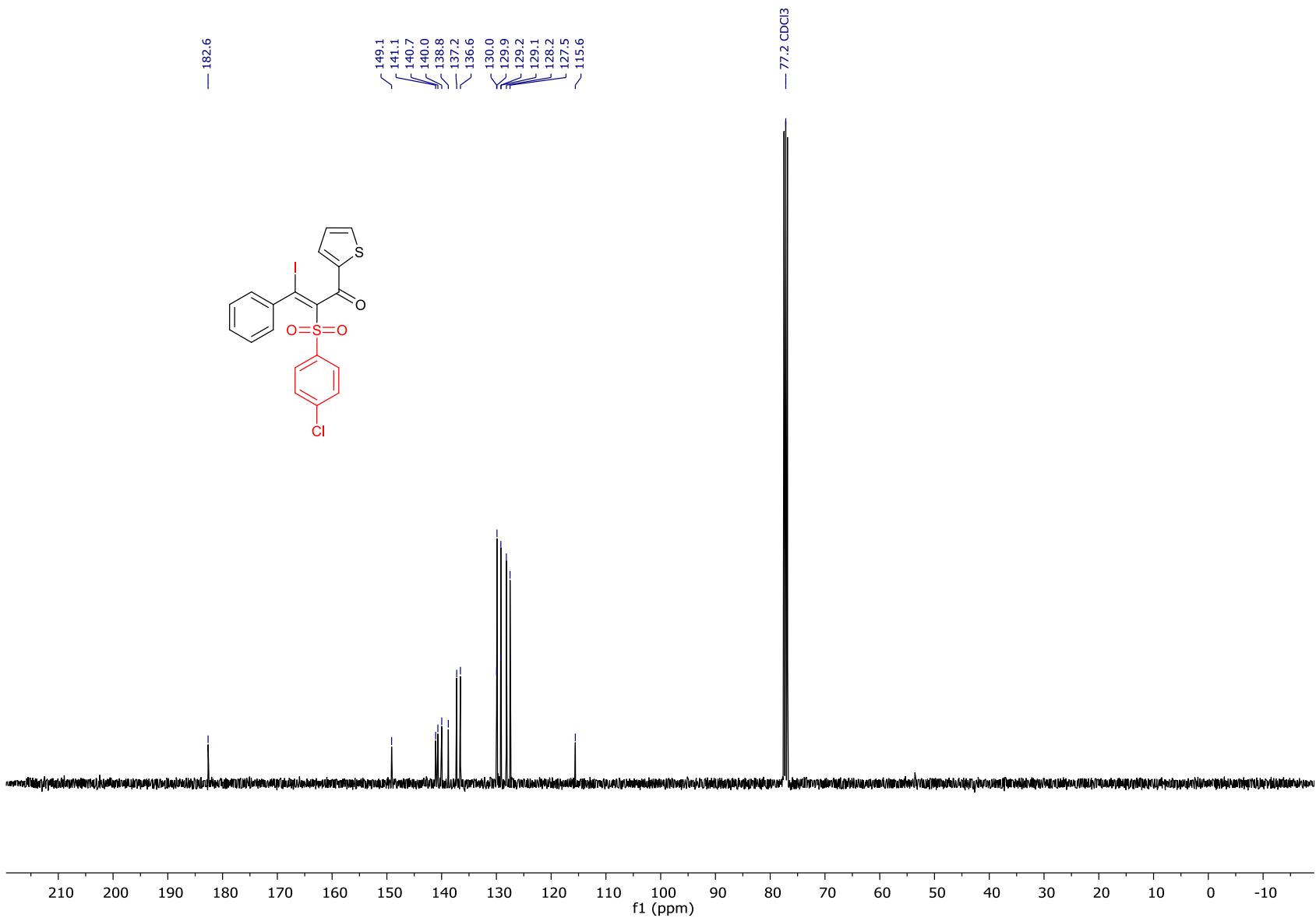


Figure S106. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl_3) of (*E*)-2-(4-chlorophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (**4h**).

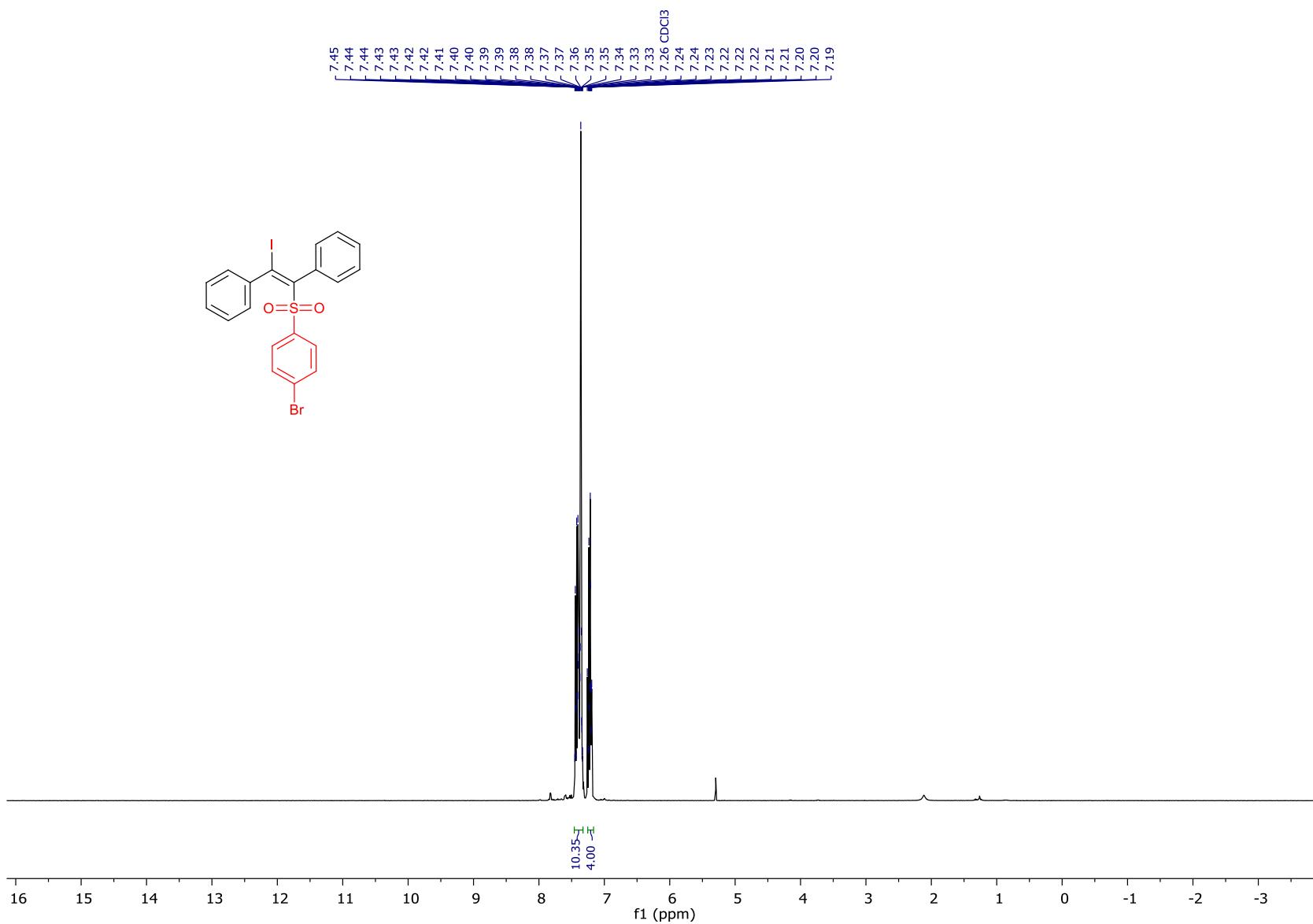


Figure S107. ^1H NMR (600 MHz, CDCl_3) of (*E*)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyl)dibenzene (**4i**).

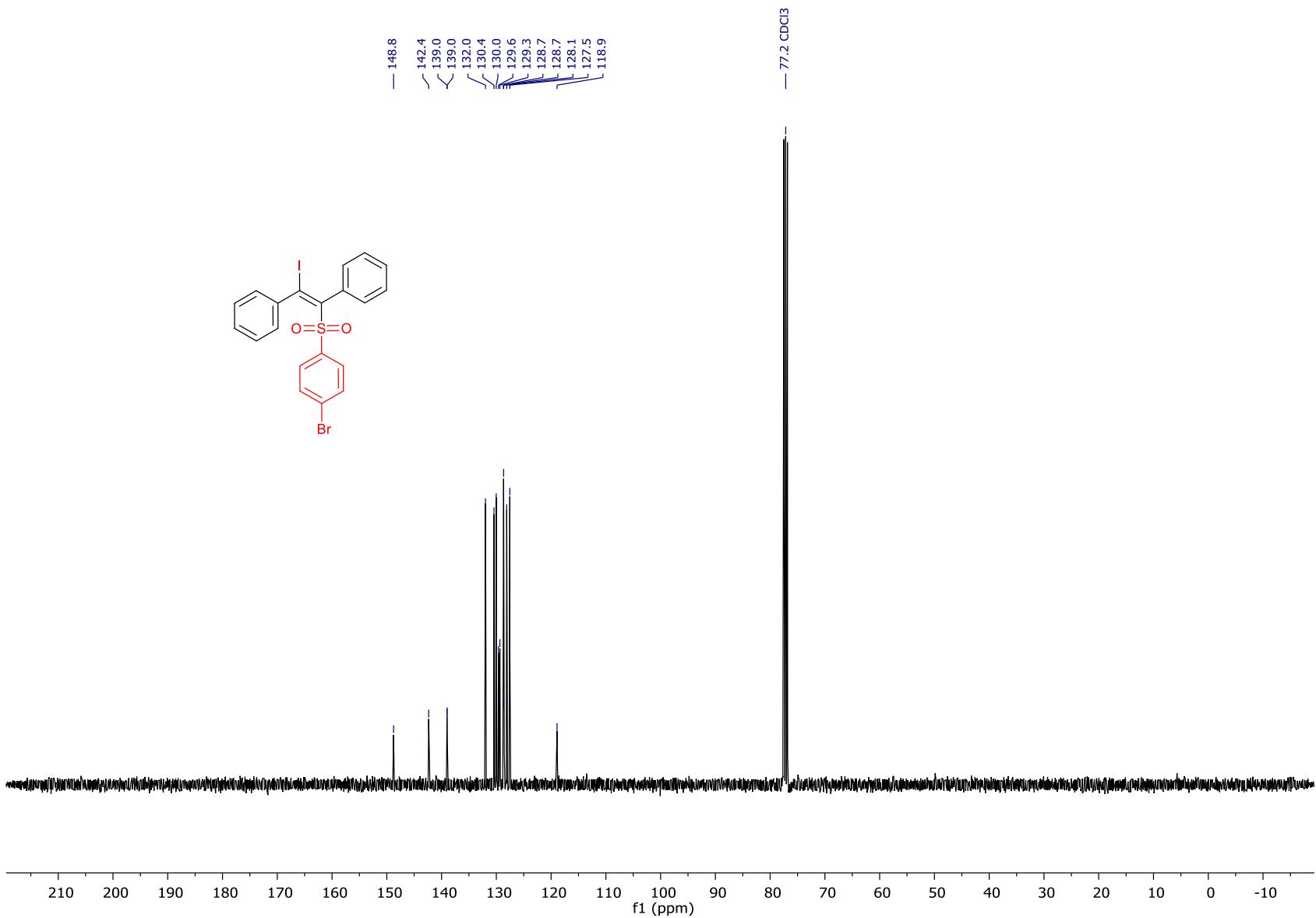


Figure S108. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl_3) of (*E*)-(1-(4-bromophenylsulfonyl)-2-iodoethene-1,2-diyldibenzene (**4i**).

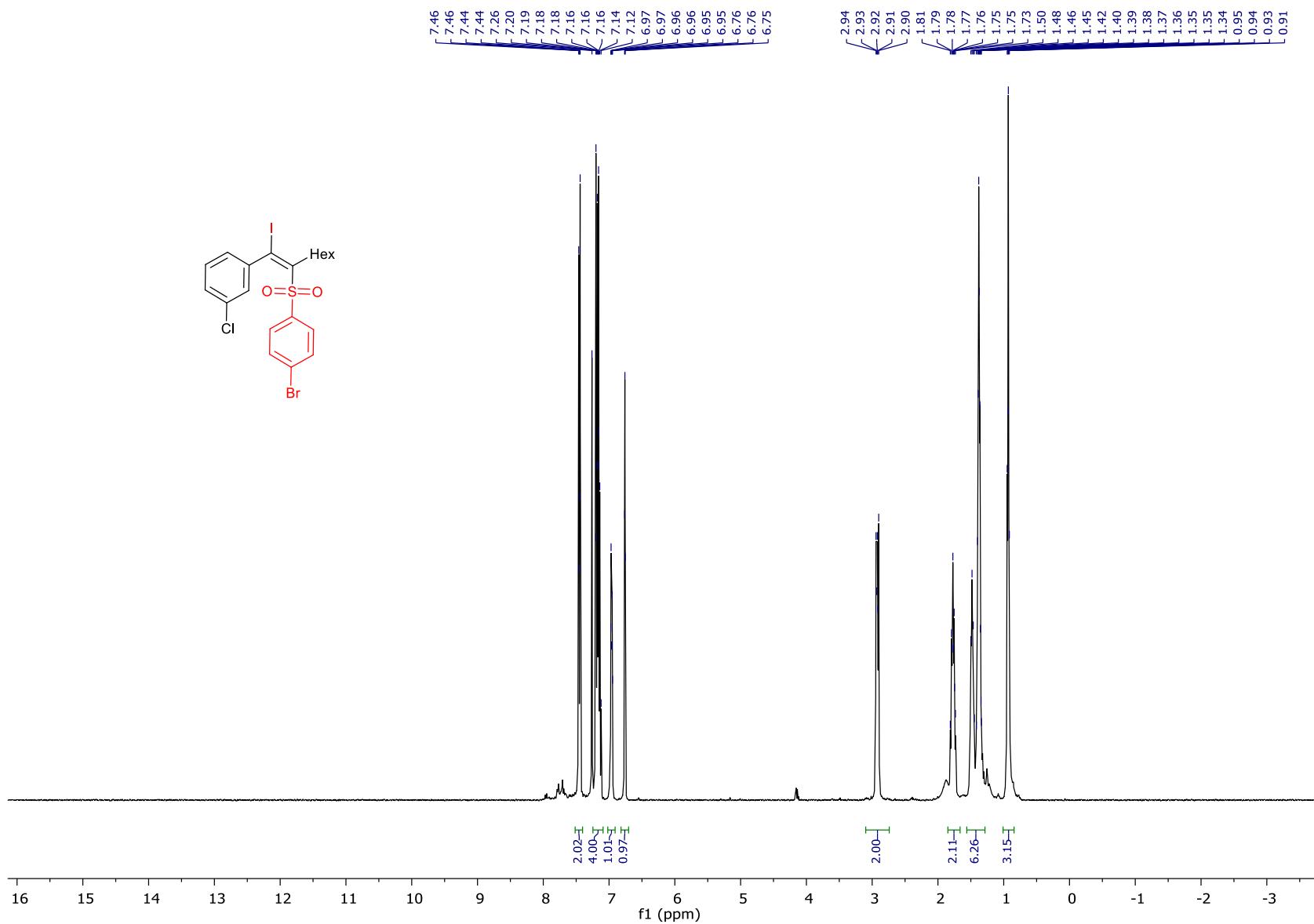


Figure S109. ^1H NMR (600 MHz, CDCl_3) of (*E*)-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (**4j**).

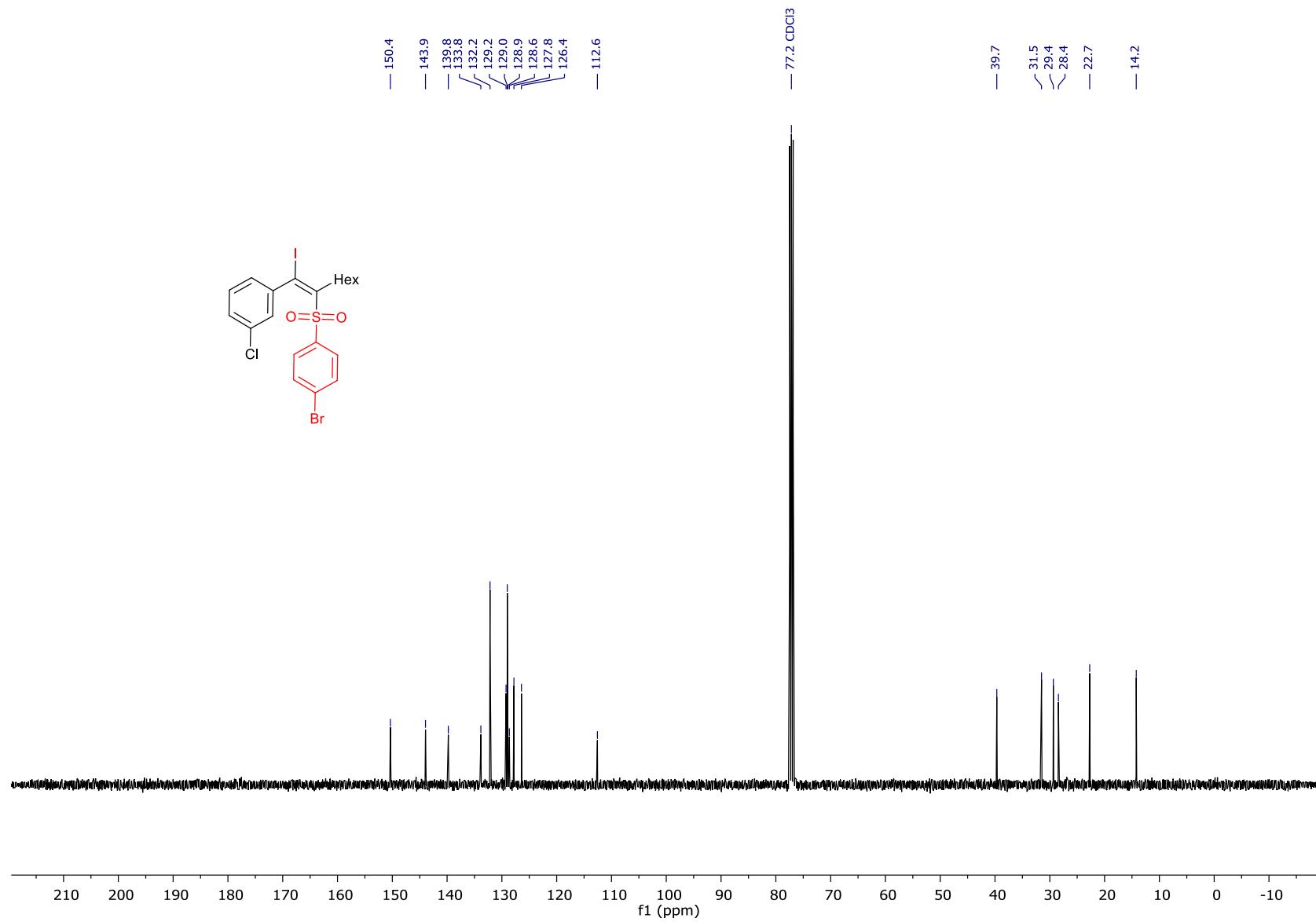


Figure S110. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, CDCl₃) of (*E*)-1-(2-(4-bromophenylsulfonyl)-1-iodooct-1-enyl)-3-chlorobenzene (**4j**).

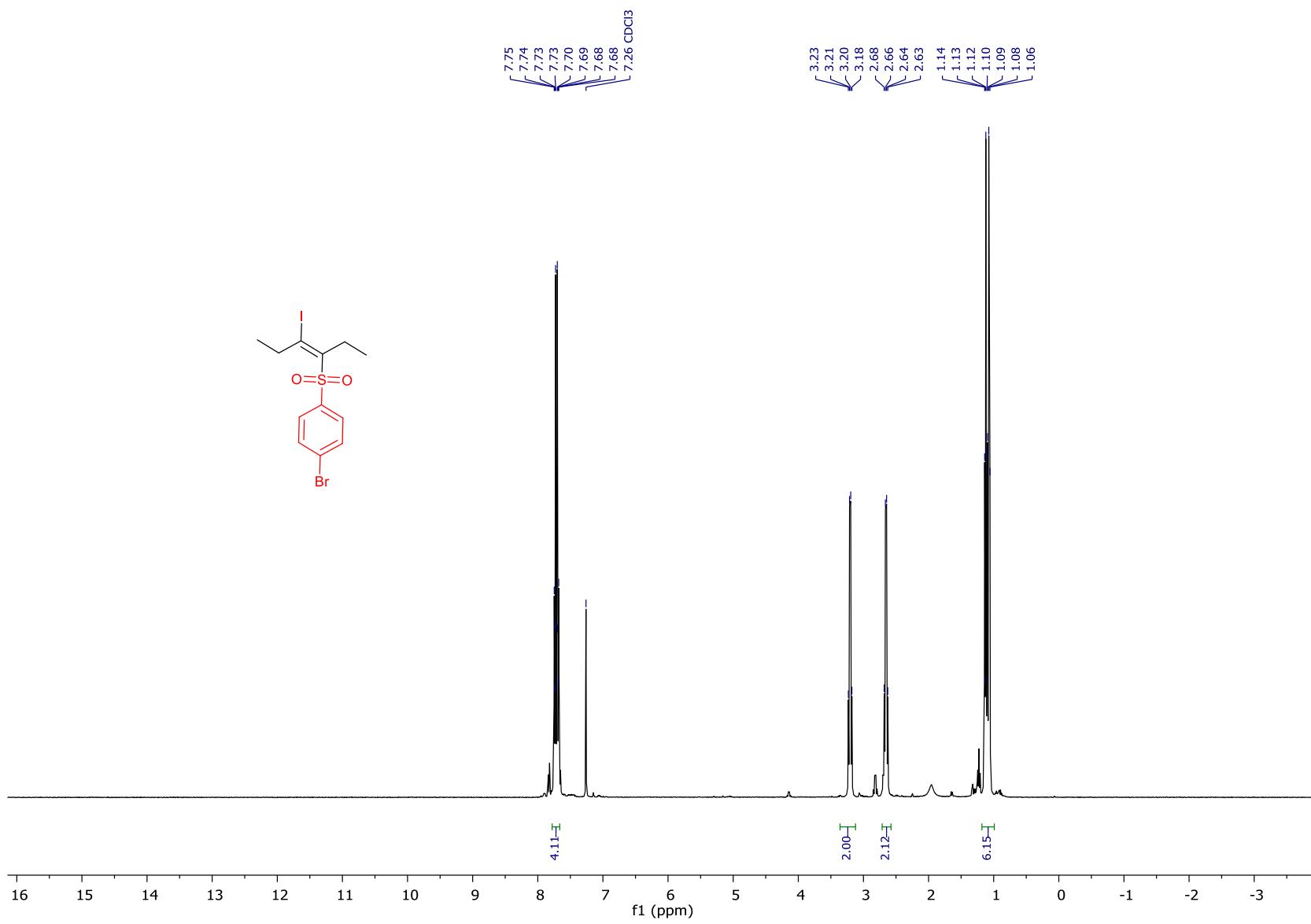


Figure S111. ^1H NMR (600 MHz, CDCl_3) of (*E*)-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (**4k**).

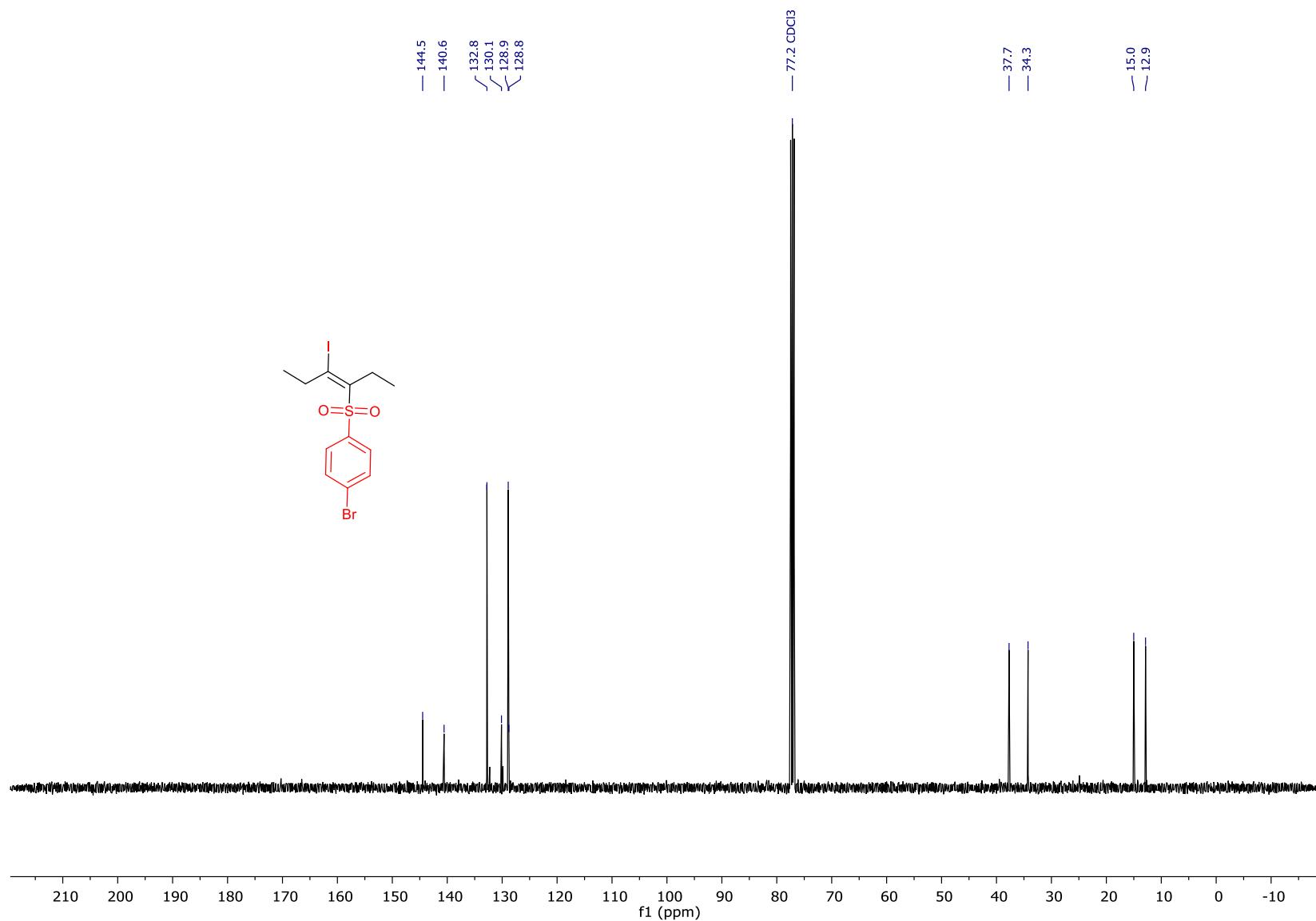


Figure S112. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl₃) of (E)-1-bromo-4-(4-iodohex-3-en-3-ylsulfonyl)benzene (**4k**).

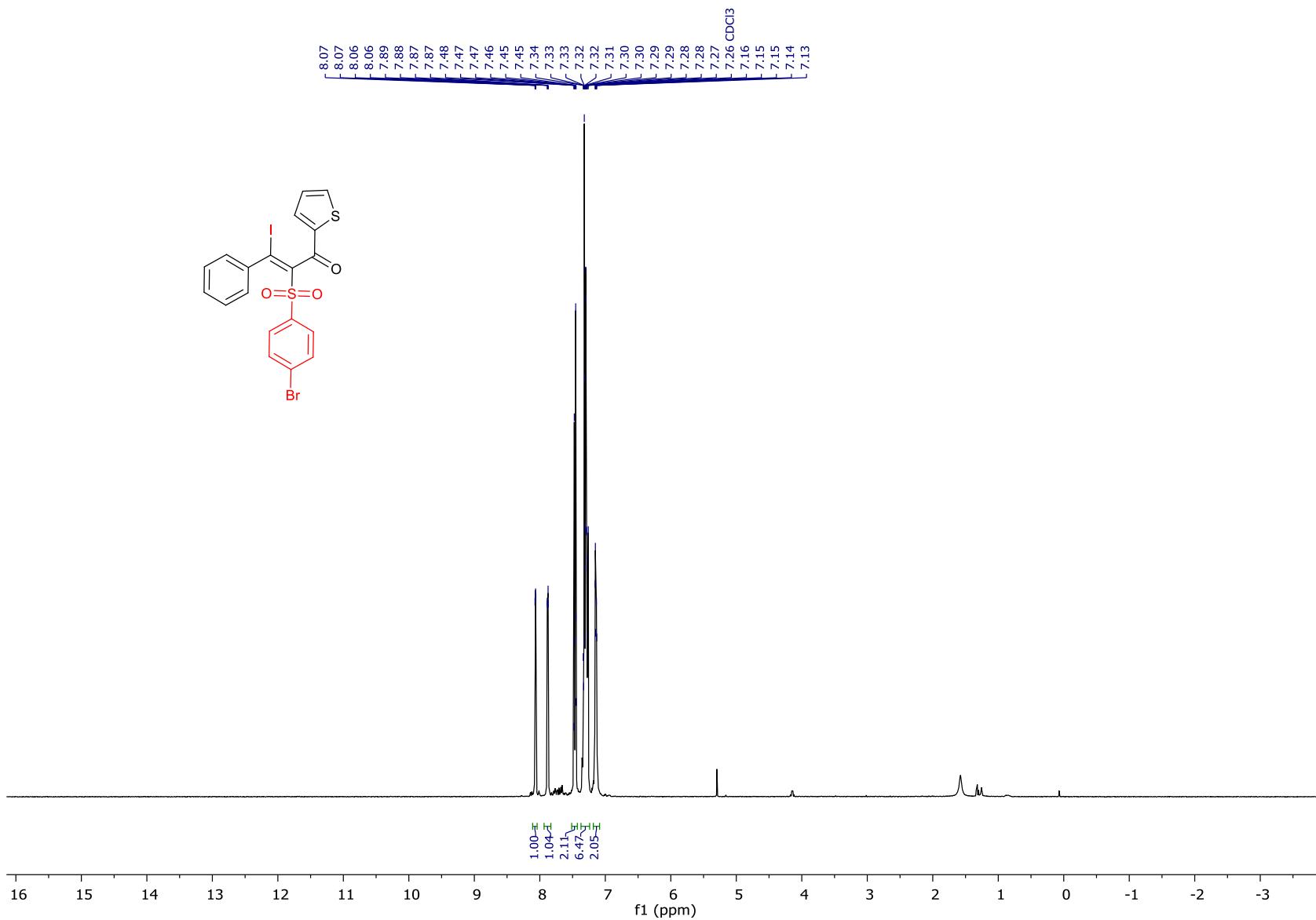


Figure S113. ^1H NMR (600 MHz, CDCl_3) of (*E*)-2-(4-bromophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (**4l**).

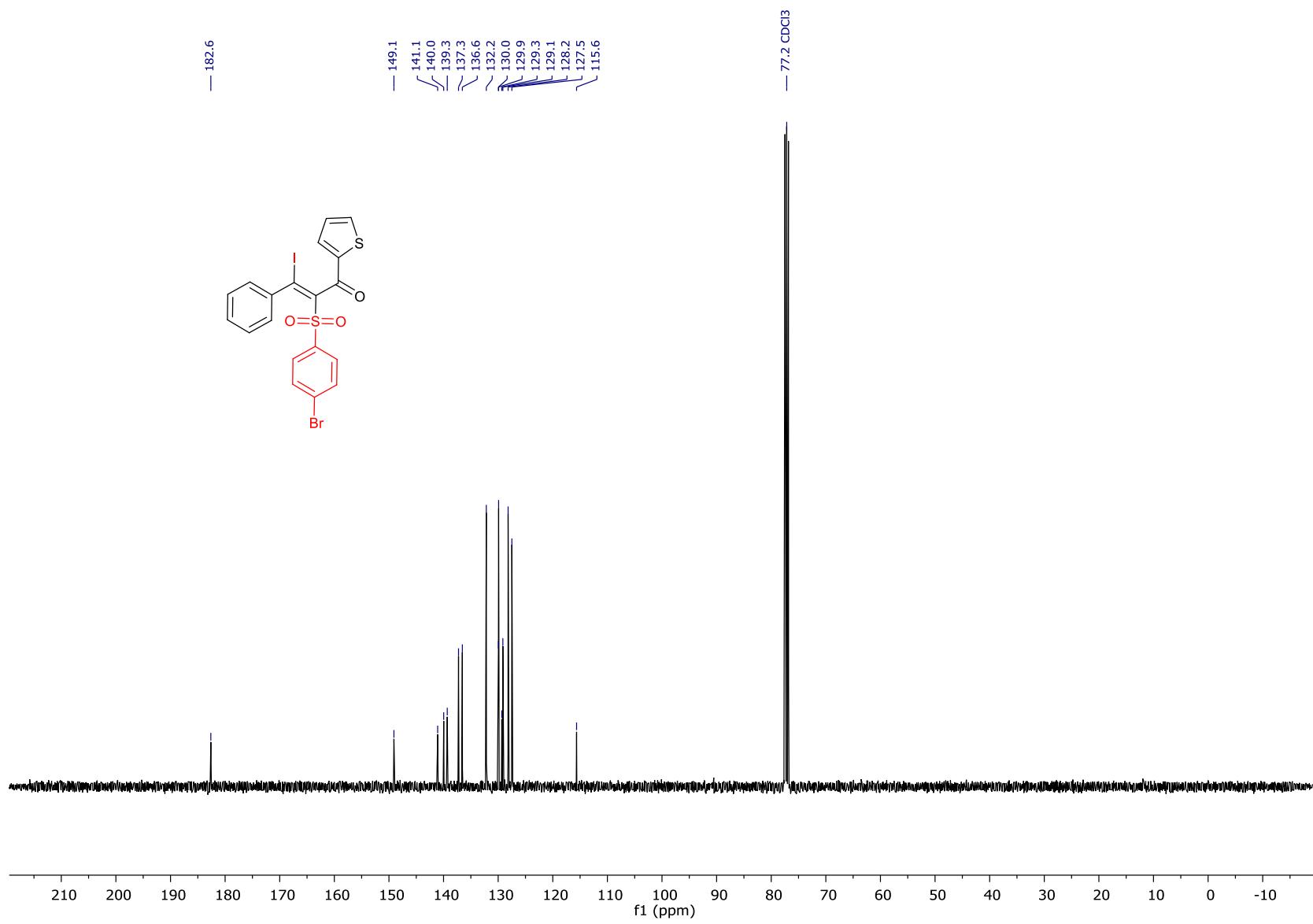


Figure S114. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, CDCl₃) of (*E*)-2-(4-bromophenylsulfonyl)-3-iodo-3-phenyl-1-(thiophen-2-yl)prop-2-en-1-one (**4l**).

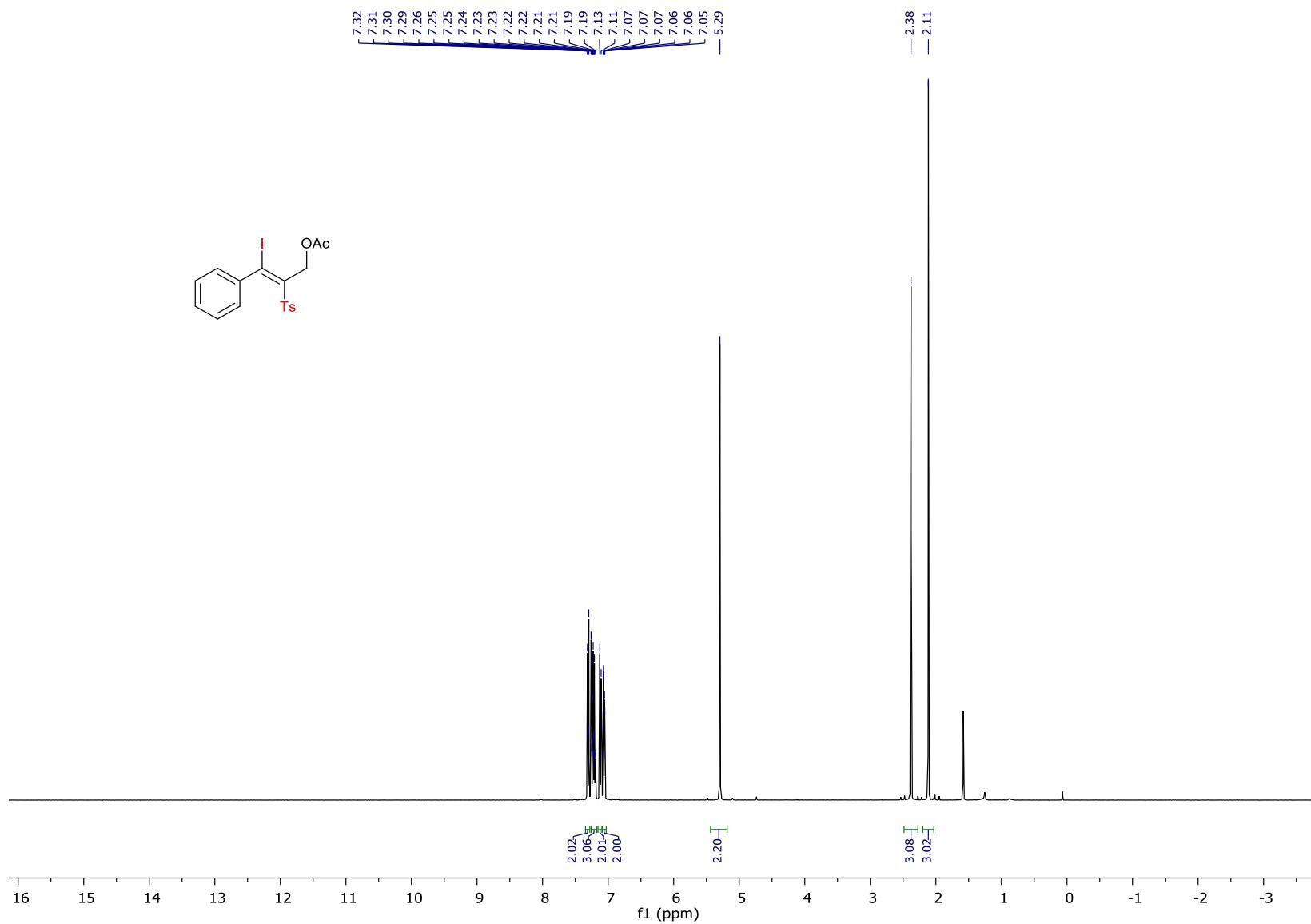


Figure S115. ¹H NMR (600 MHz, Chloroform-d) of (E)-3-iodo-3-phenyl-2-tosylallyl acetate (5a).

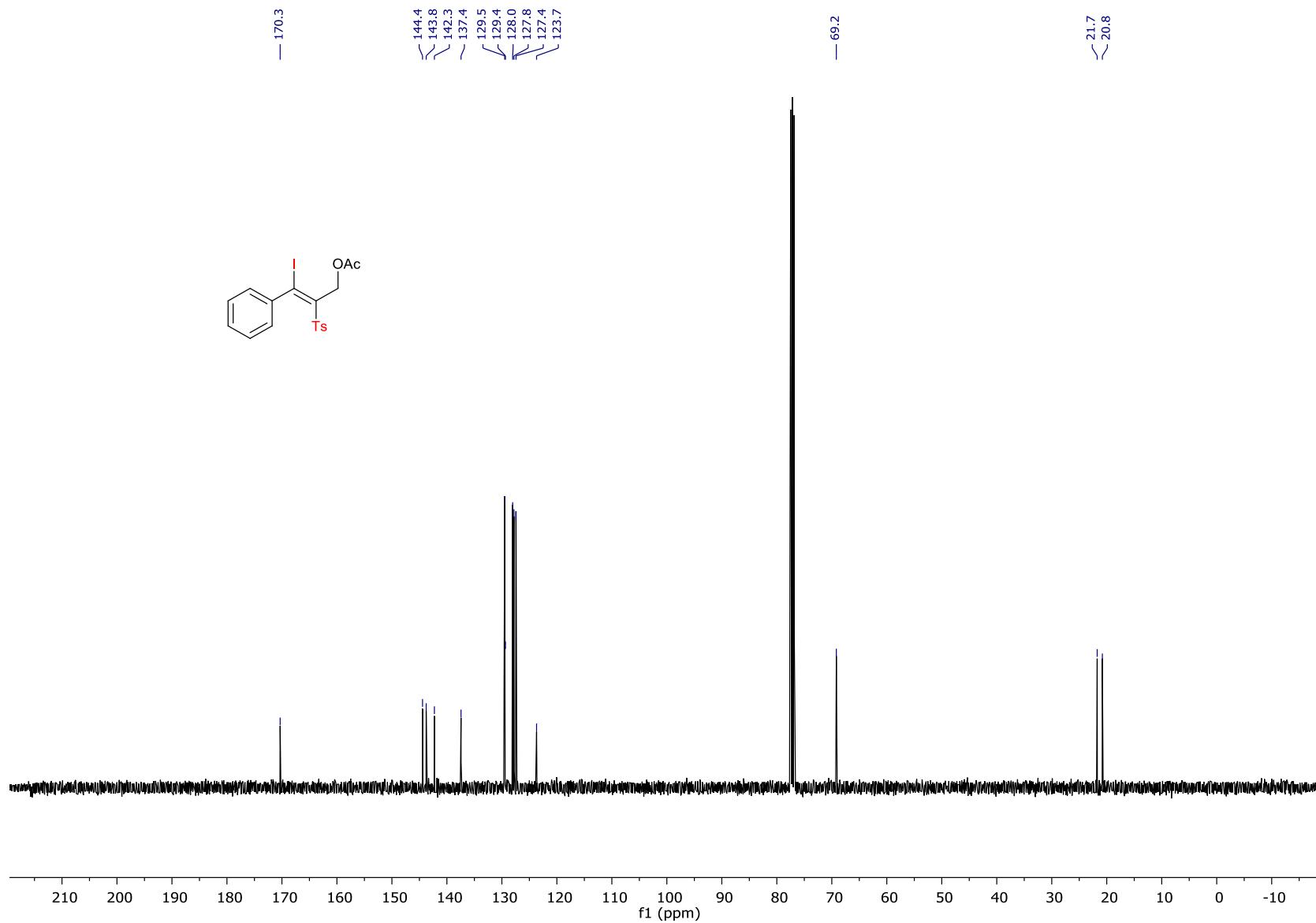


Figure S116. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E)-3-iodo-3-phenyl-2-tosylallyl acetate (**5a**).

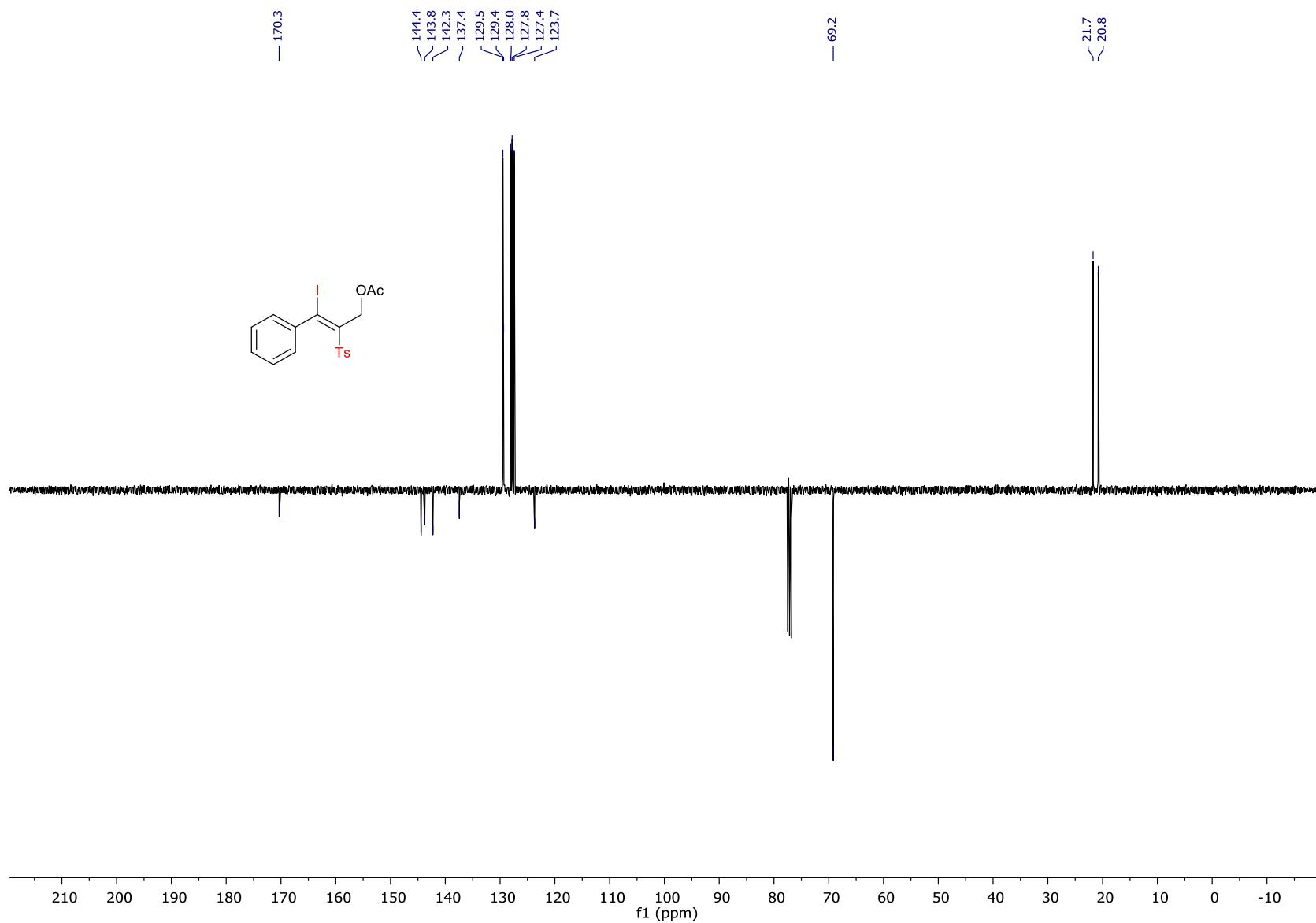


Figure S117. ^{13}C DEPTQ-135 NMR (*E*)-3-iodo-3-phenyl-2-tosylallyl acetate (**5a**).

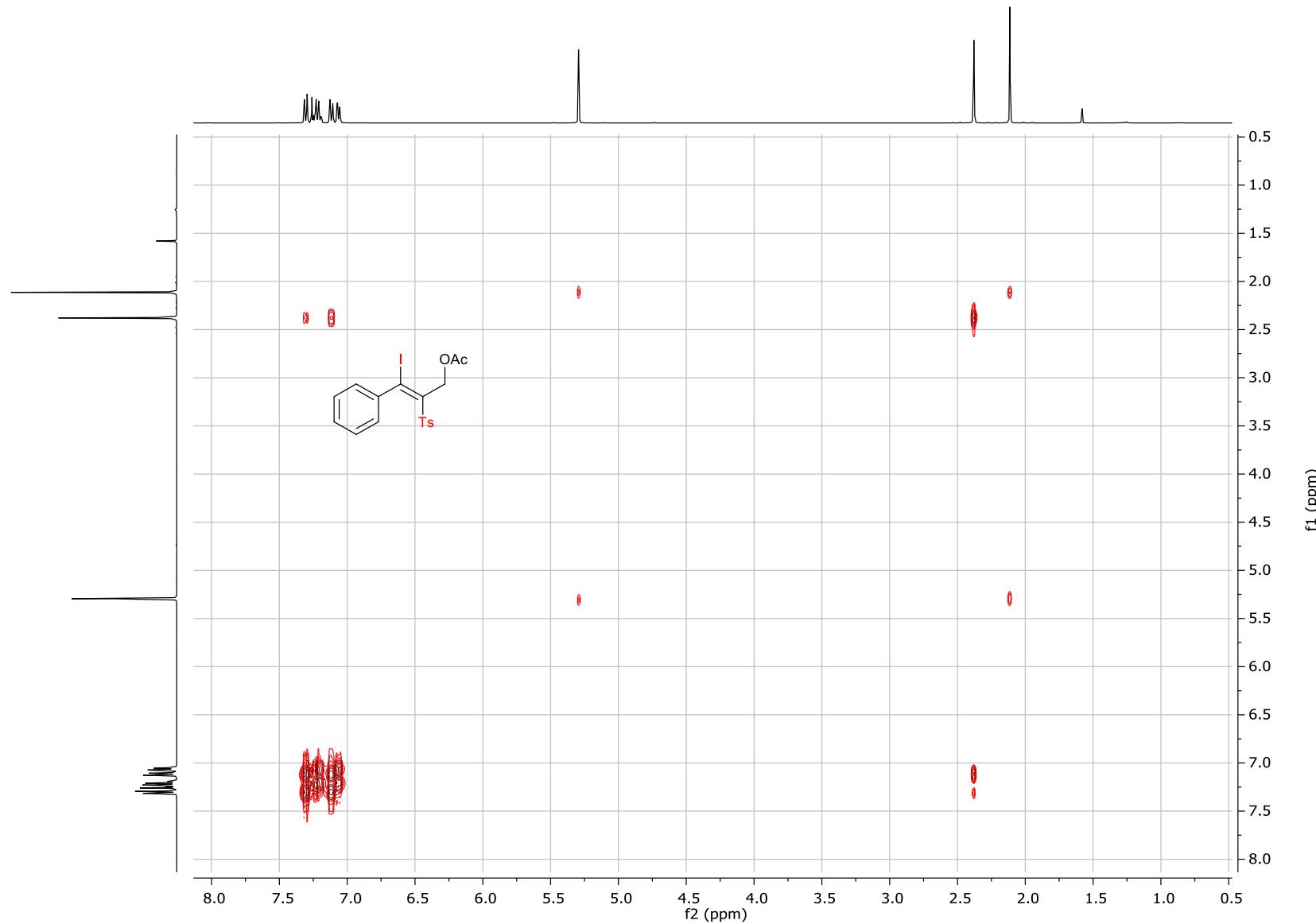


Figure S118. ^1H - ^1H COSY (*E*)-3-iodo-3-phenyl-2-tosylallyl acetate (5a).

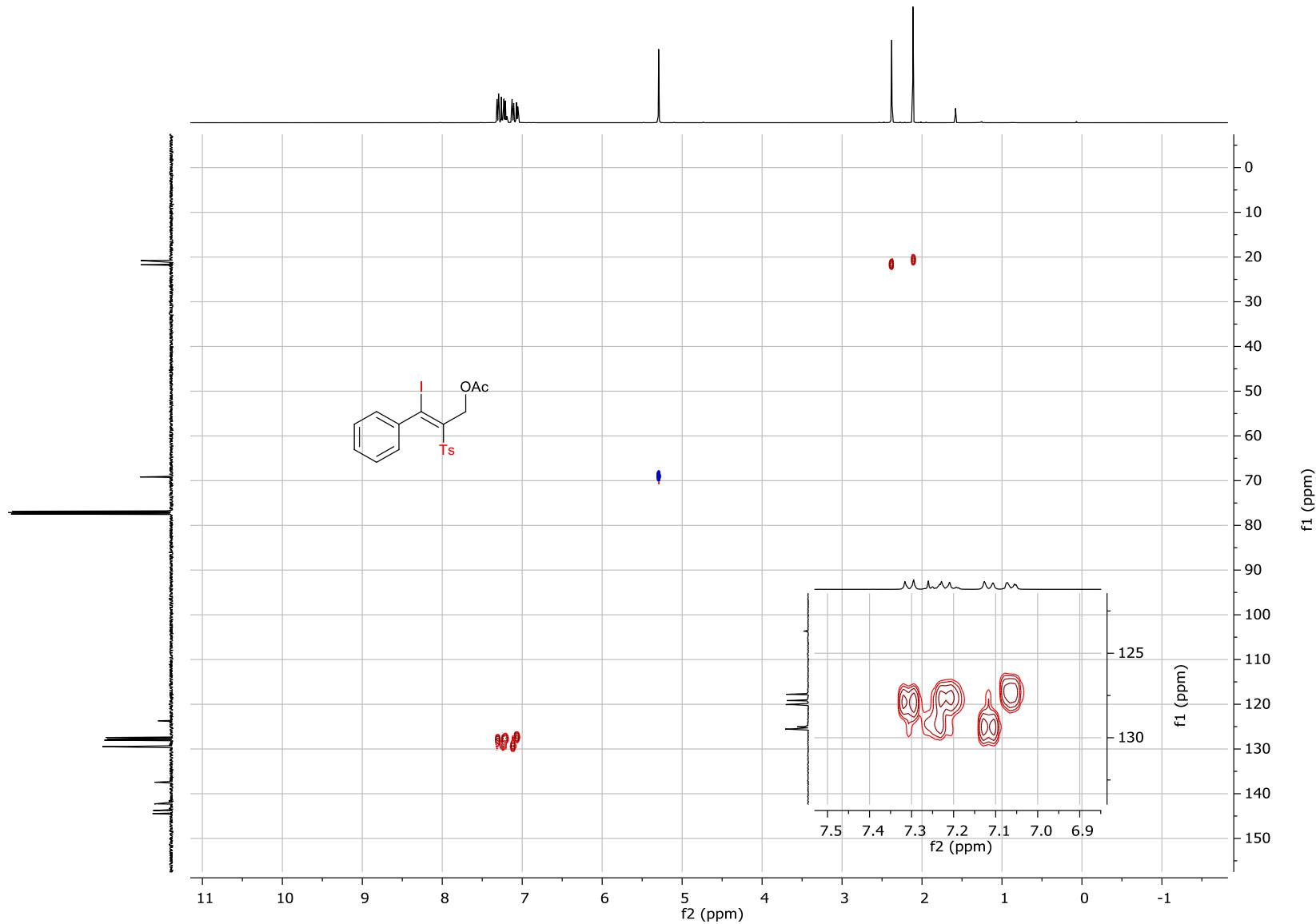


Figure S119. ^1H - ^{13}C HSQC (*E*)-3-iodo-3-phenyl-2-tosylallyl acetate (**5a**).

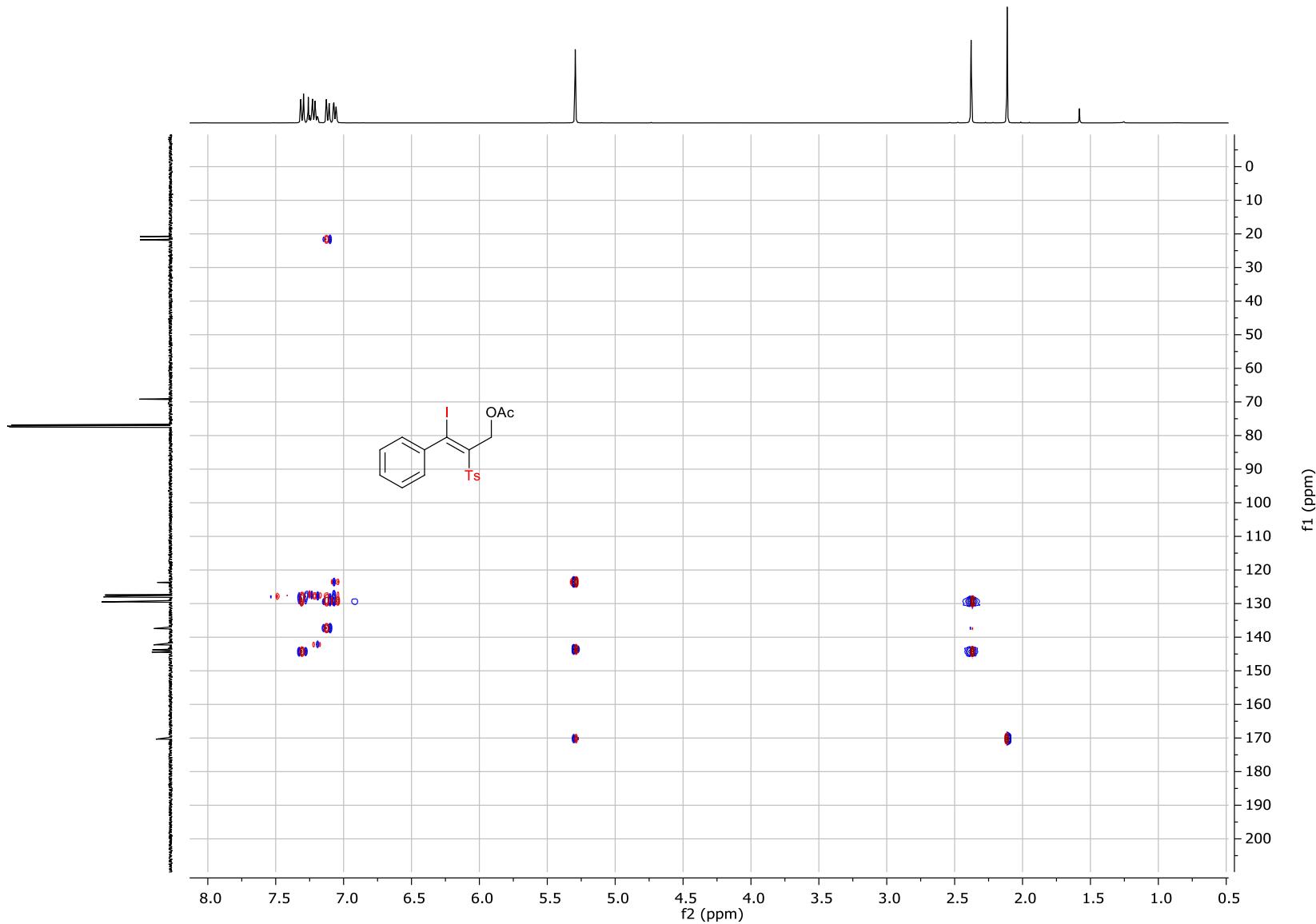


Figure S120. ^1H - ^{13}C HMBC (*E*)-3-iodo-3-phenyl-2-tosylallyl acetate (**5a**).

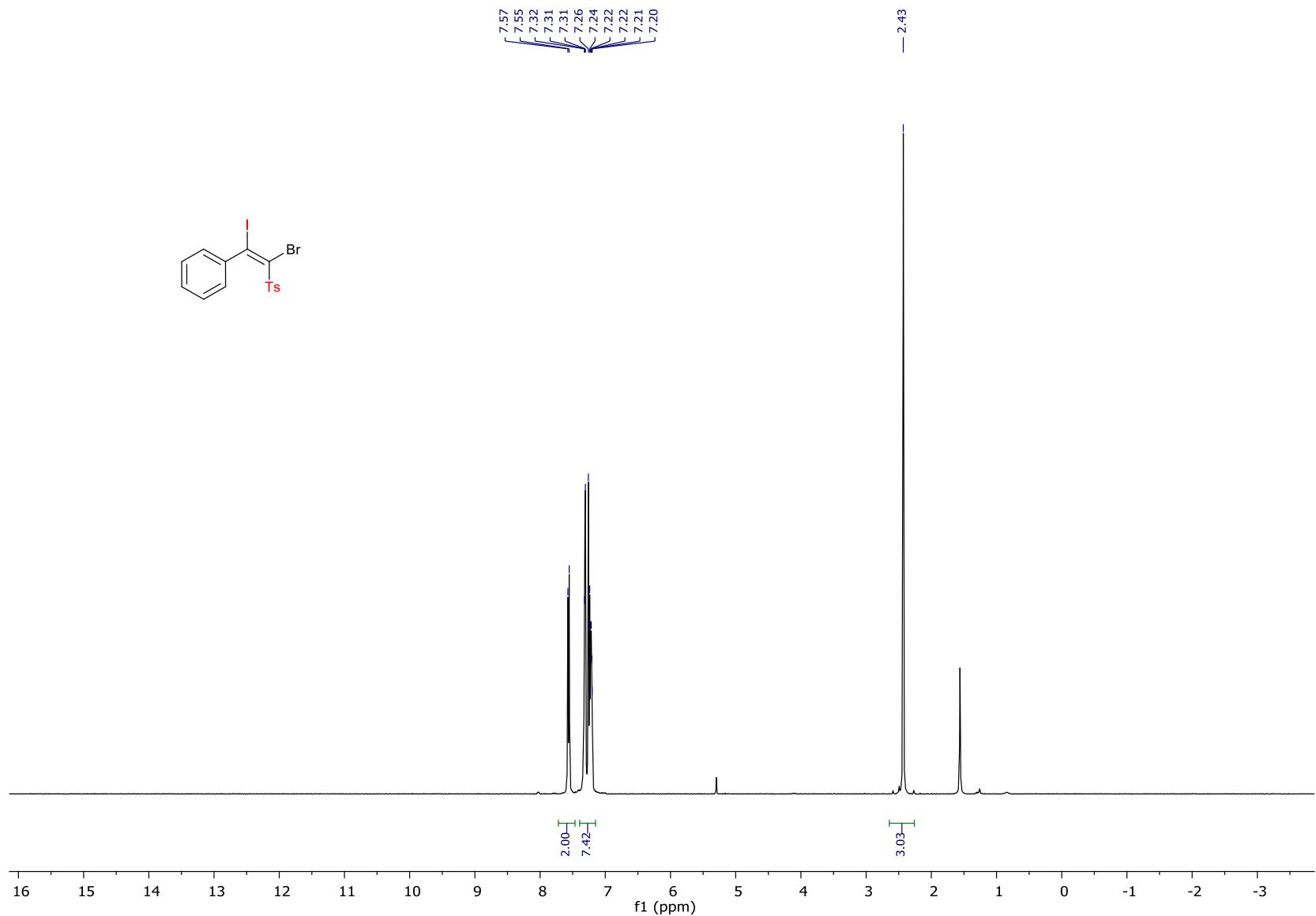


Figure S121. ^1H NMR (600 MHz, Chloroform-d) of (Z)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5b**).

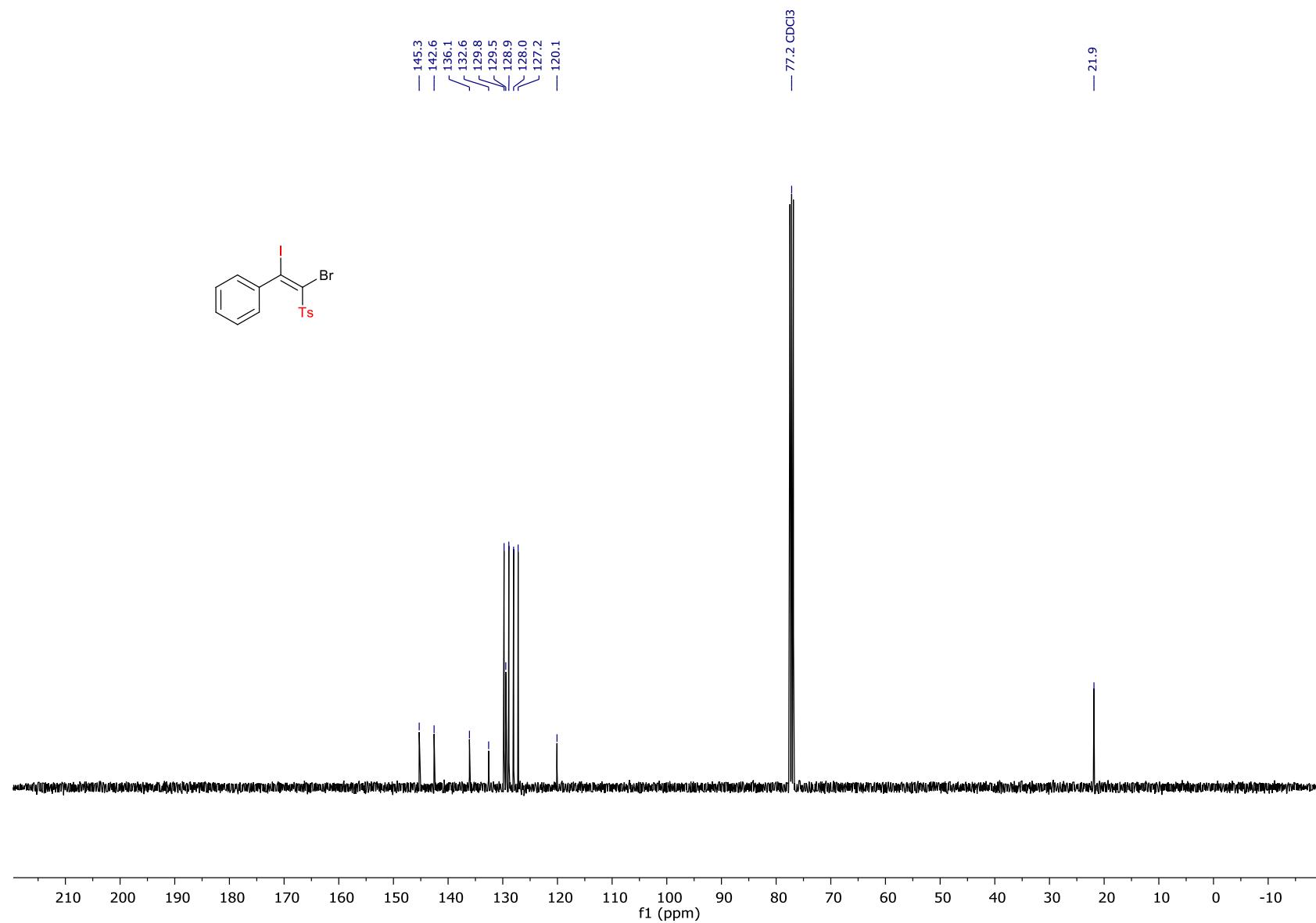


Figure S122. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (Z)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5b**).

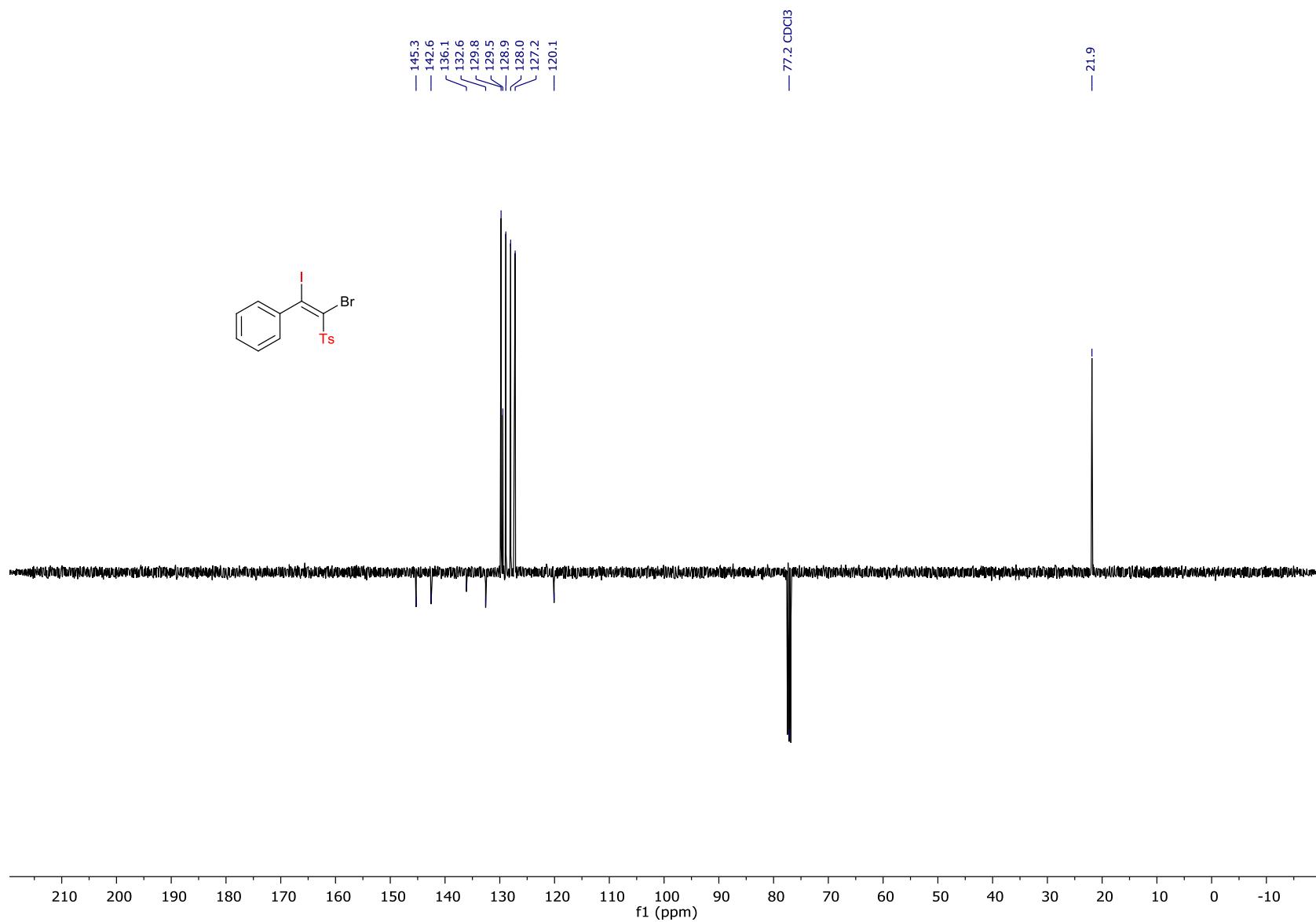


Figure S123. ^{13}C DEPTQ-135 NMR (Z)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5b**).

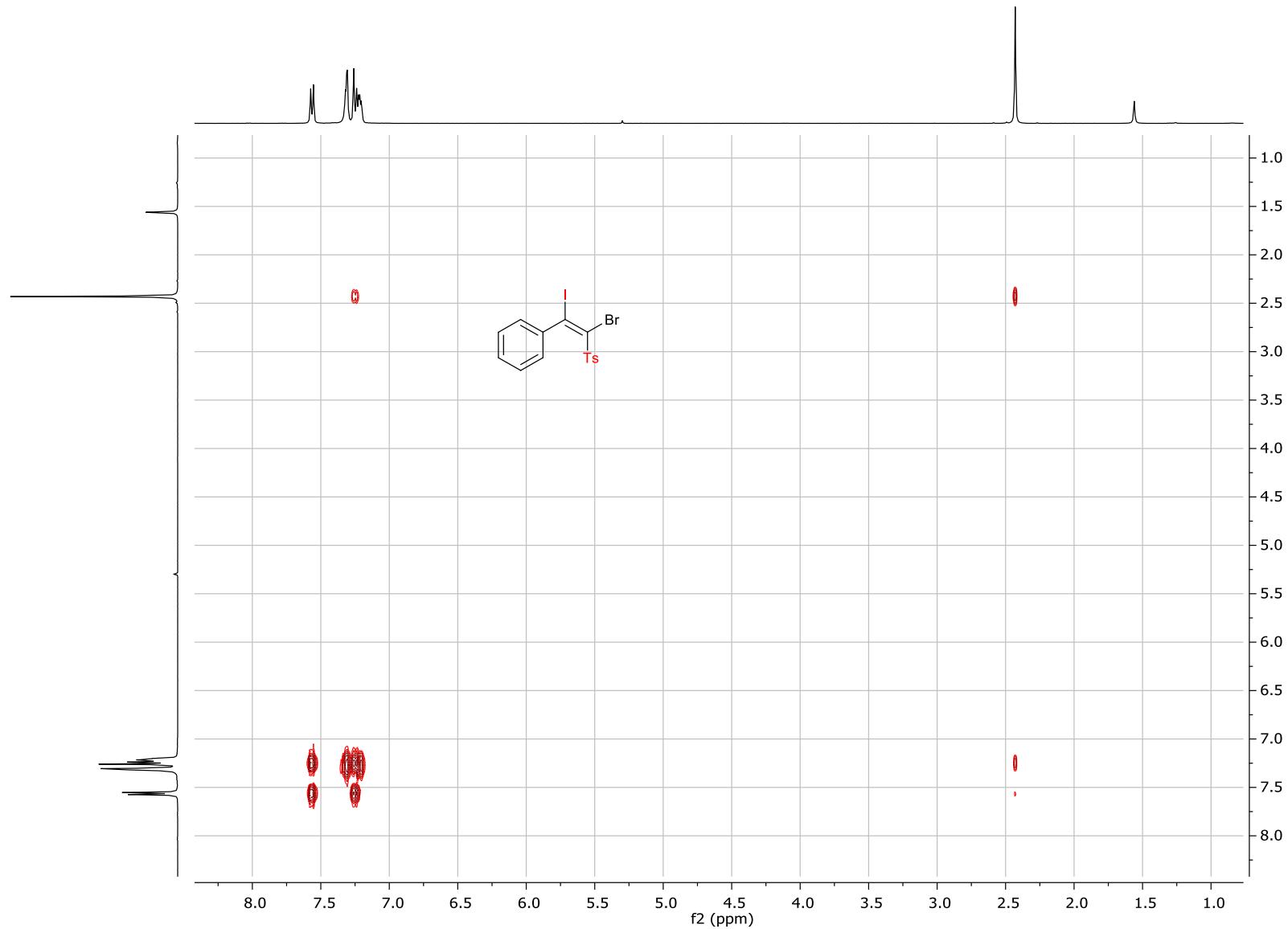


Figure S124. ^1H - ^1H COSY (*Z*)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5b**).

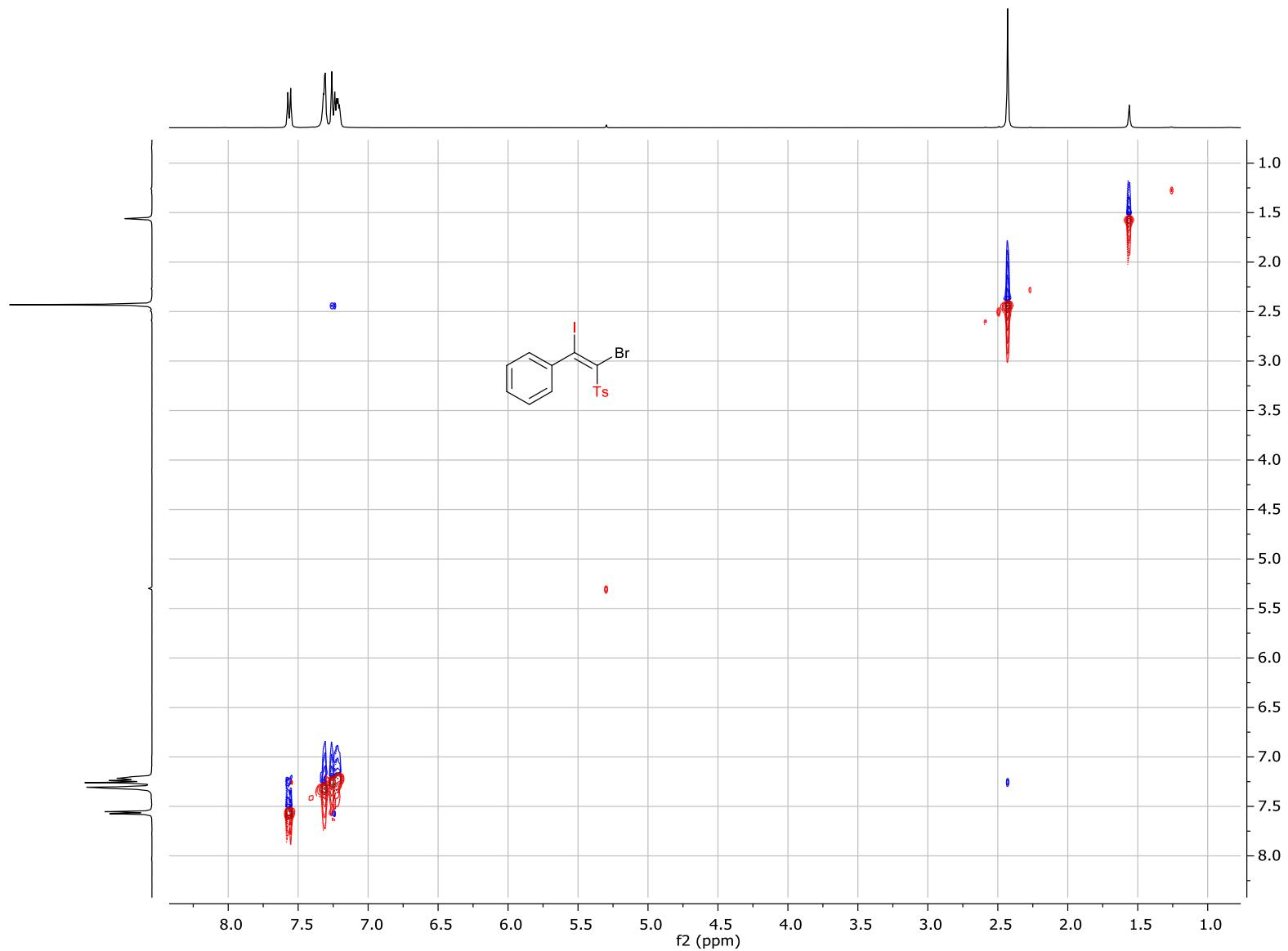


Figure S125. NOESY (*Z*)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5b**).

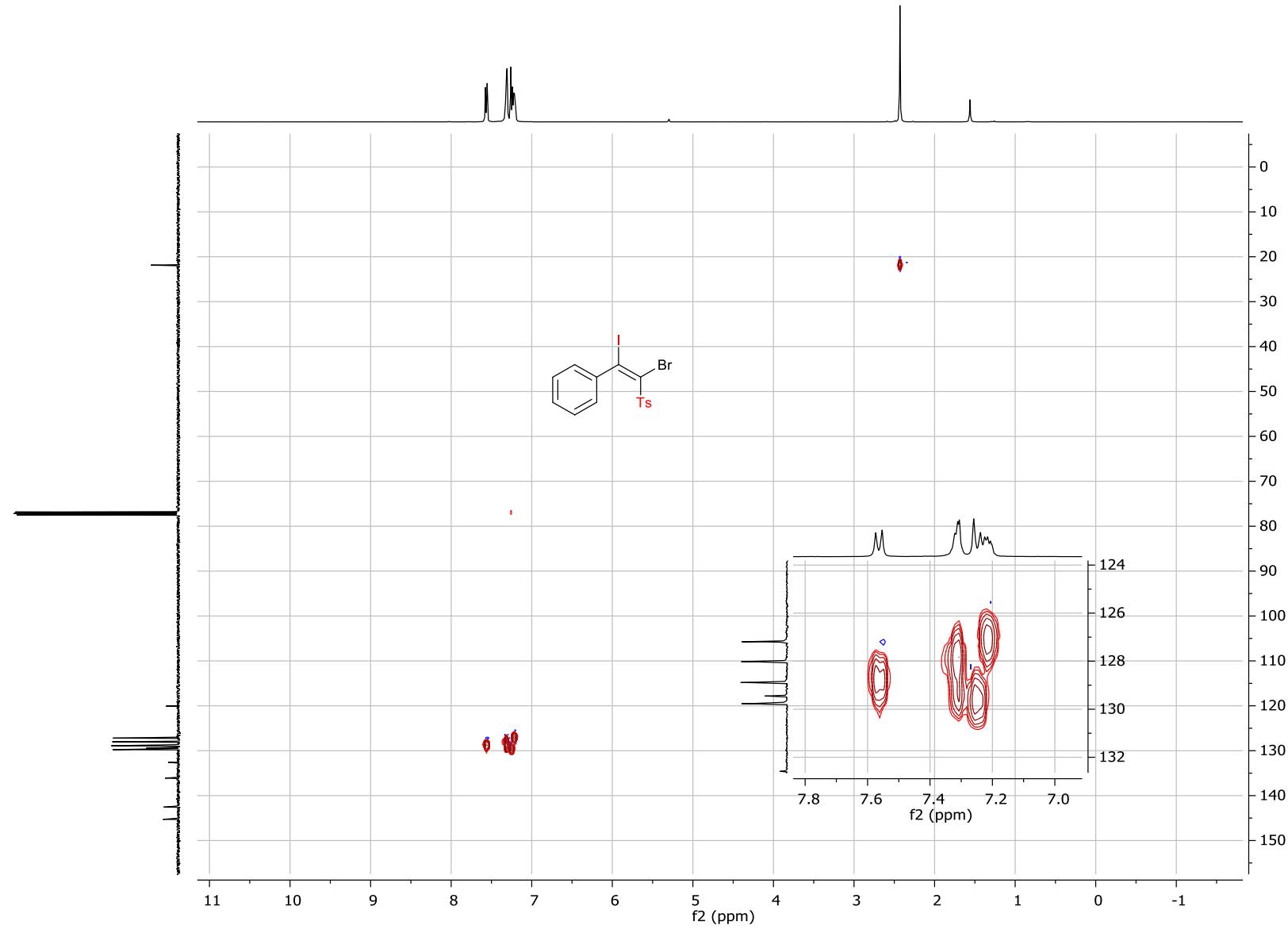


Figure S126. ^1H - ^{13}C HSQC (*Z*)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5b**).

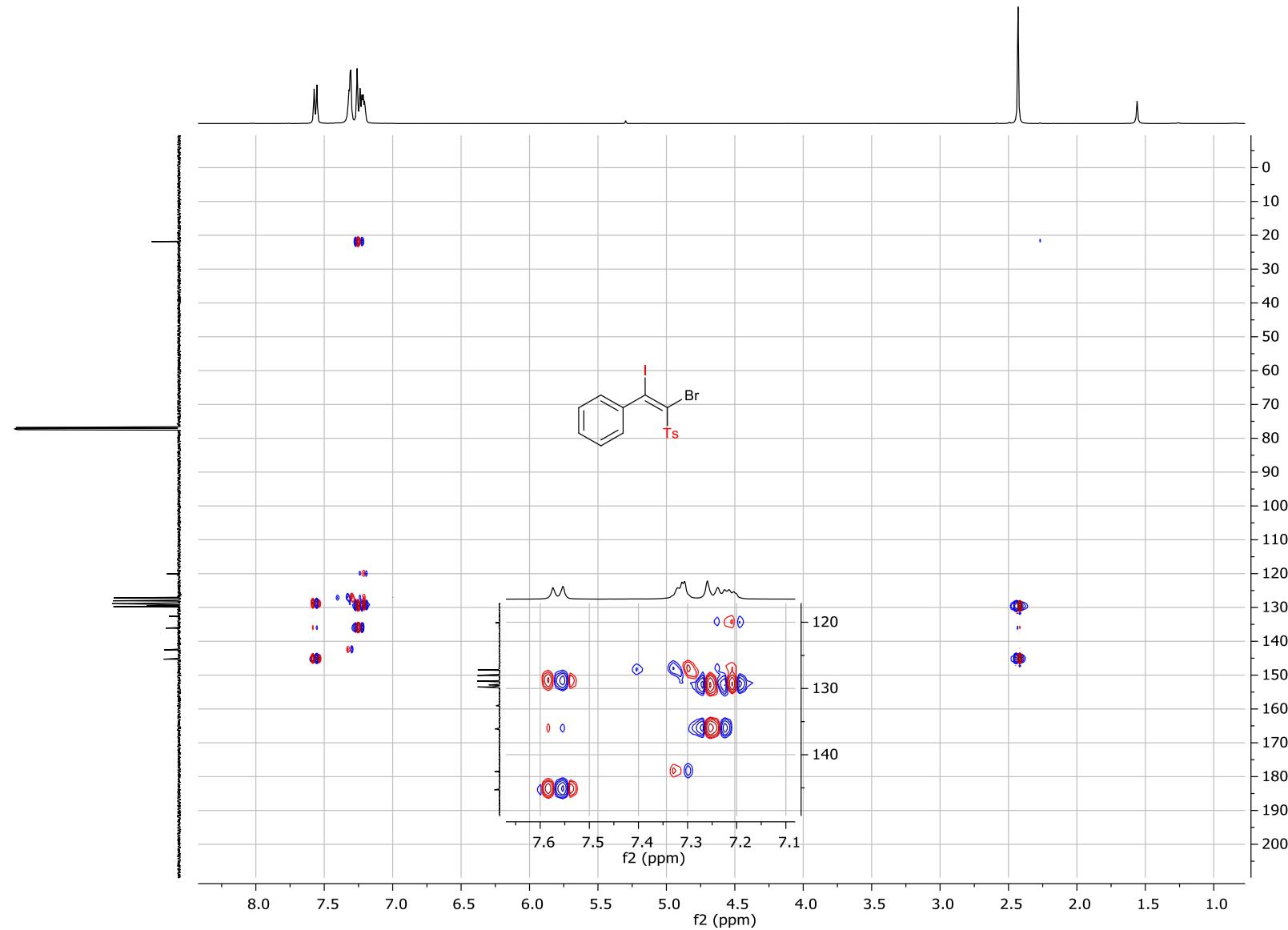


Figure S127. ^1H - ^{13}C HMBC (Z)-1-(1-bromo-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5b**).

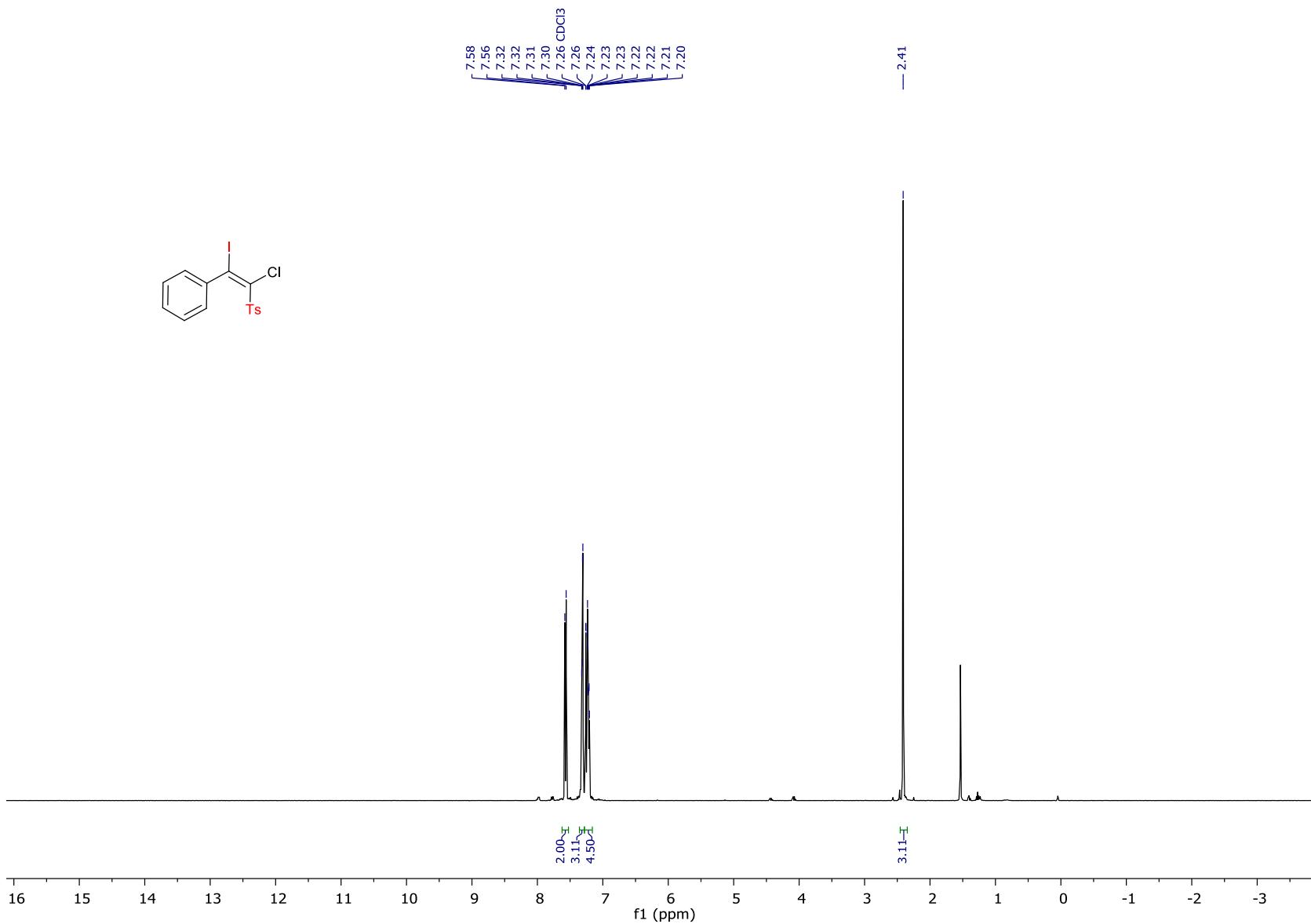


Figure S128. ^1H NMR (600 MHz, Chloroform-d) of (Z)-1-(1-chloro-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5c**).

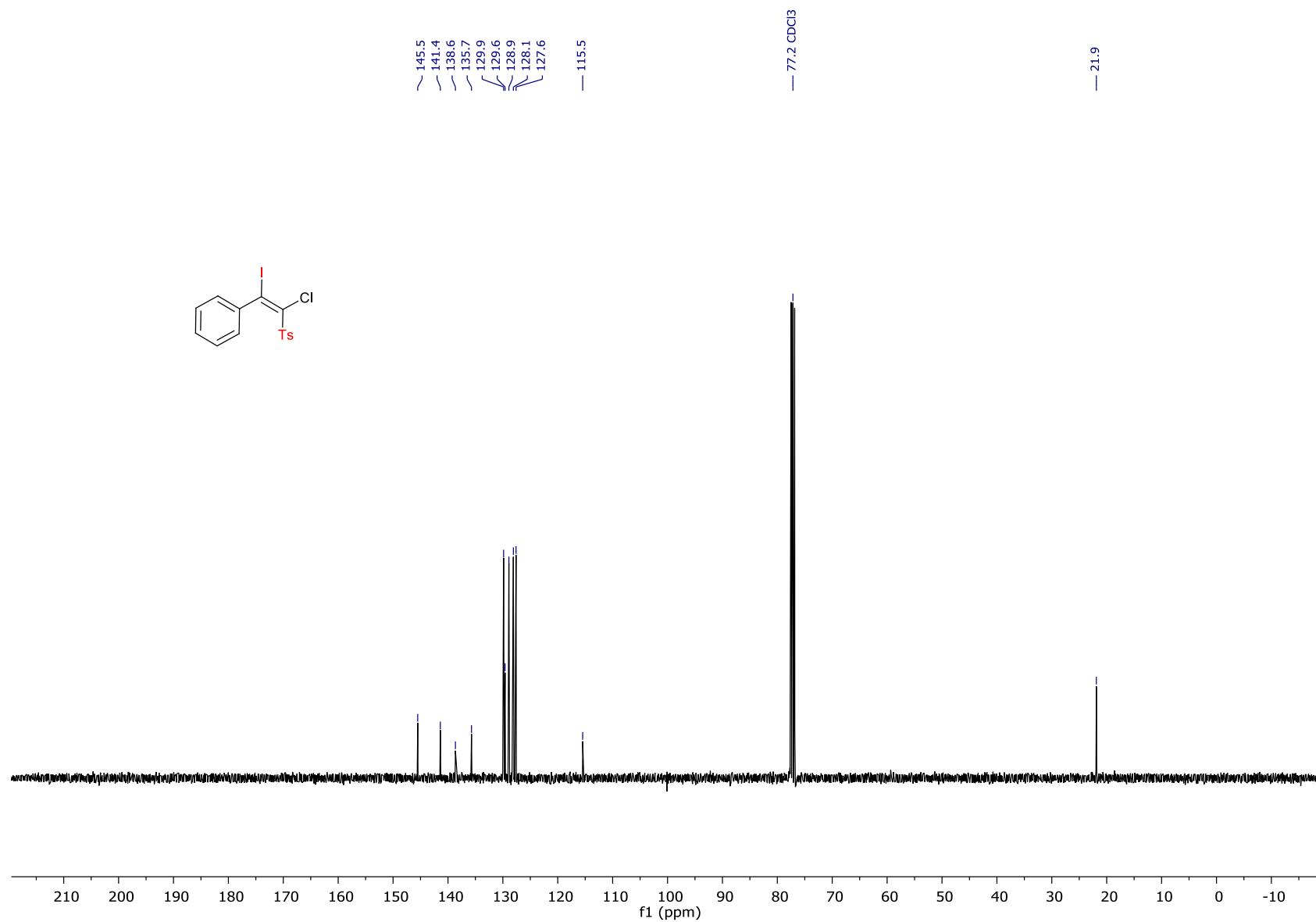


Figure S129. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (Z)-1-(1-chloro-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5c**).

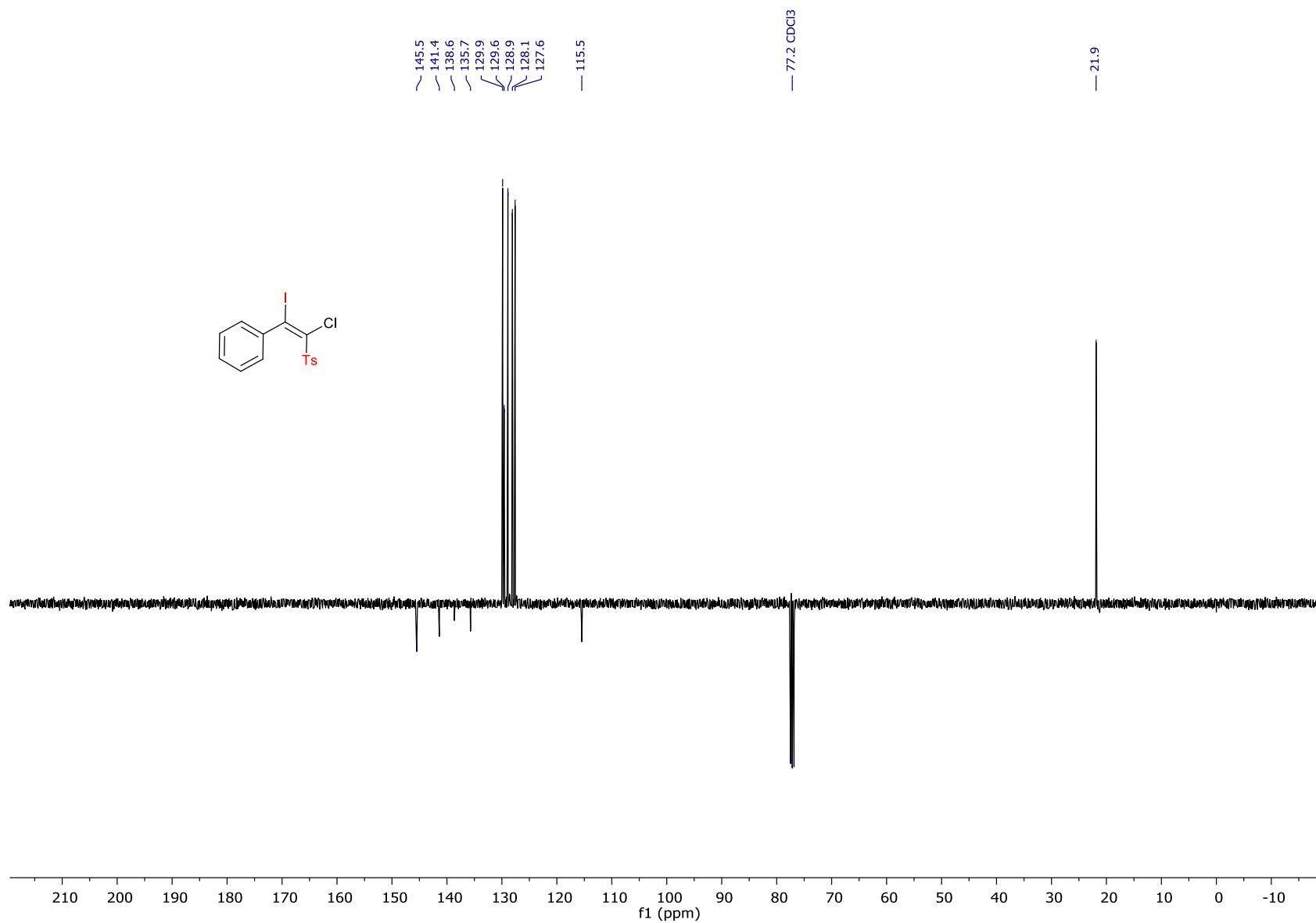


Figure S130. ^{13}C DEPTQ-135 NMR (Z)-1-(1-chloro-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5c**).

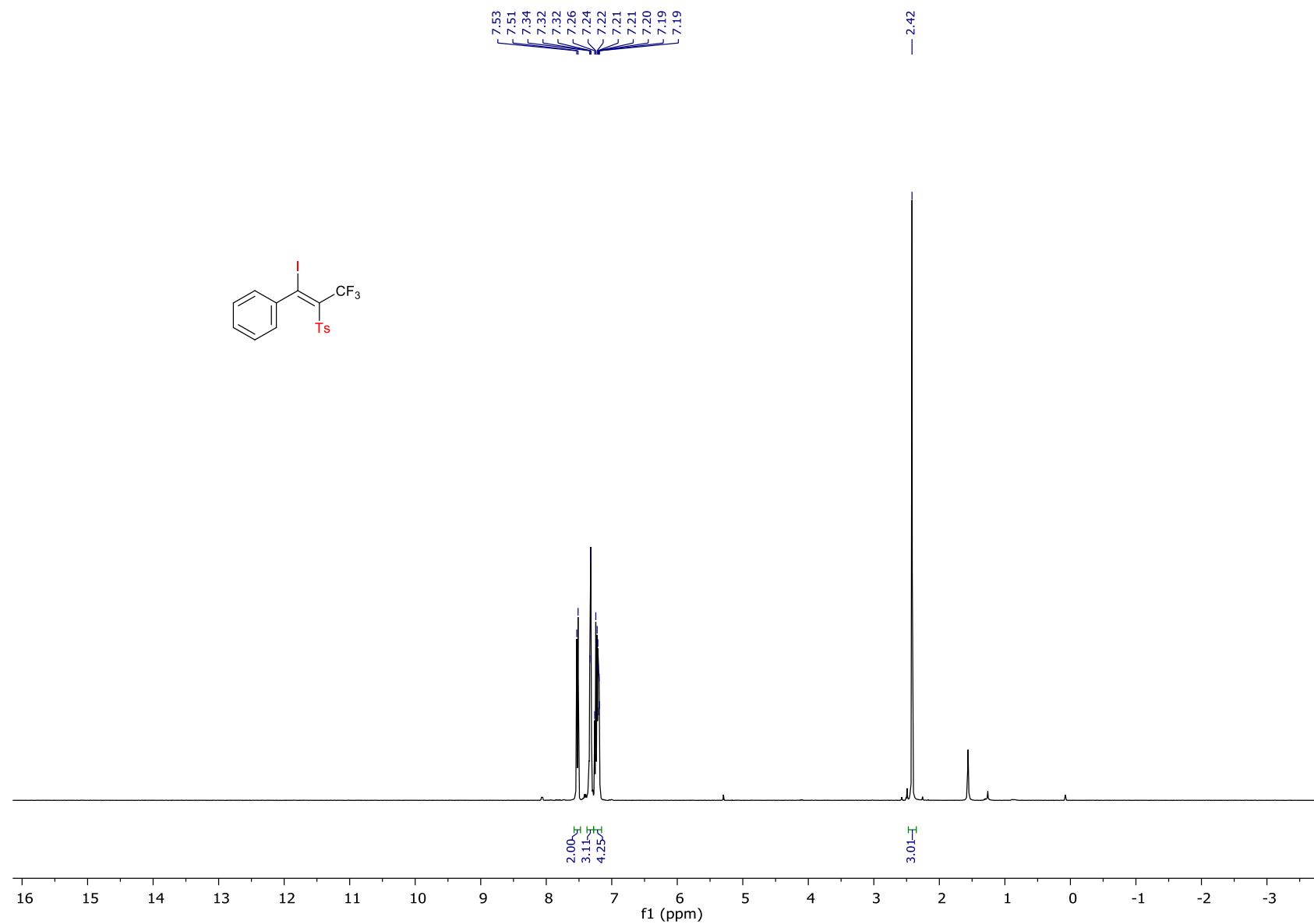


Figure S131. ^1H NMR (600 MHz, Chloroform-d) of (E)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (5d).

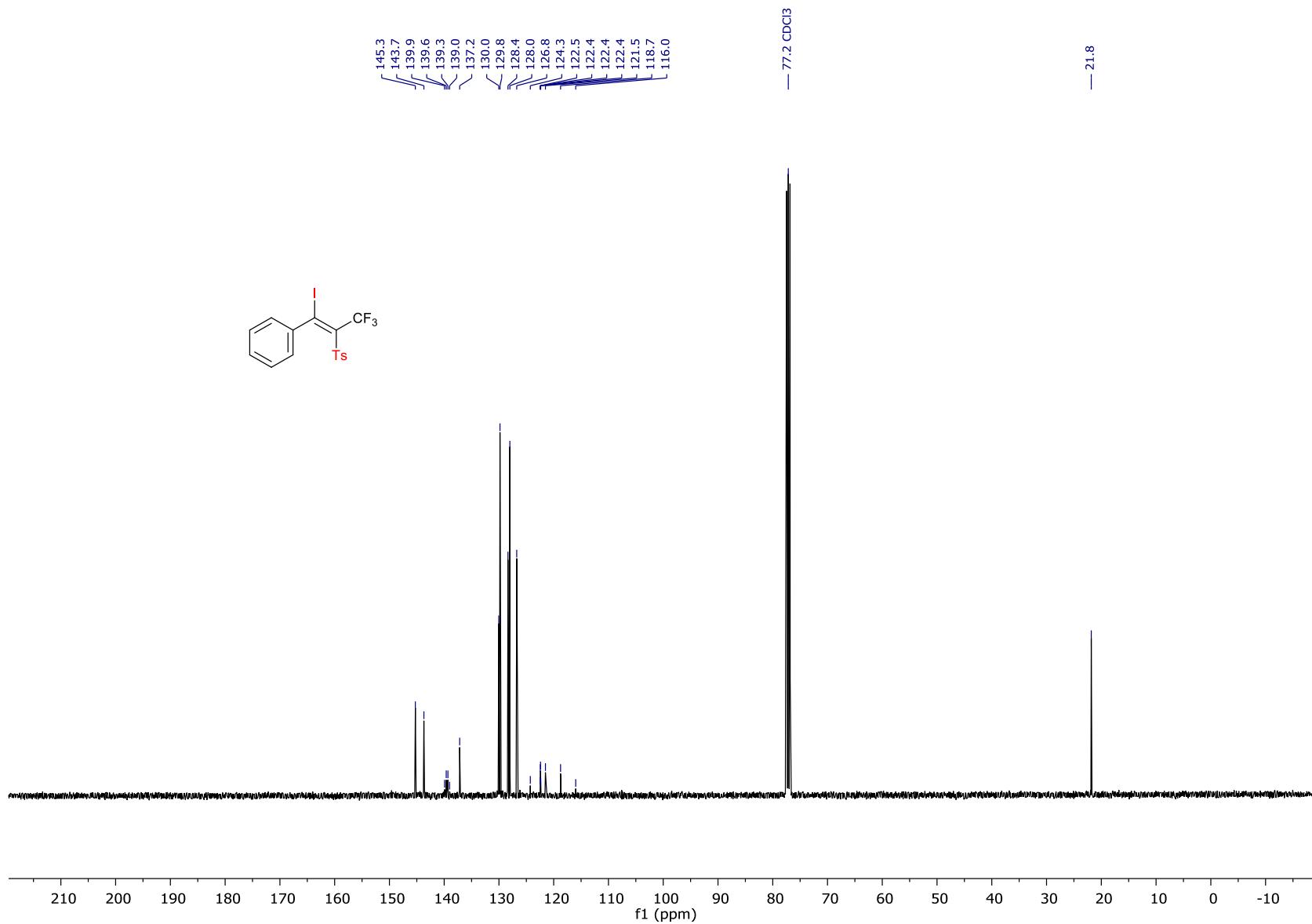


Figure S132. $^{13}\text{C}\{^1\text{H}\}$ NMR (151 MHz, Chloroform-d) of (*E*)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (**5d**).

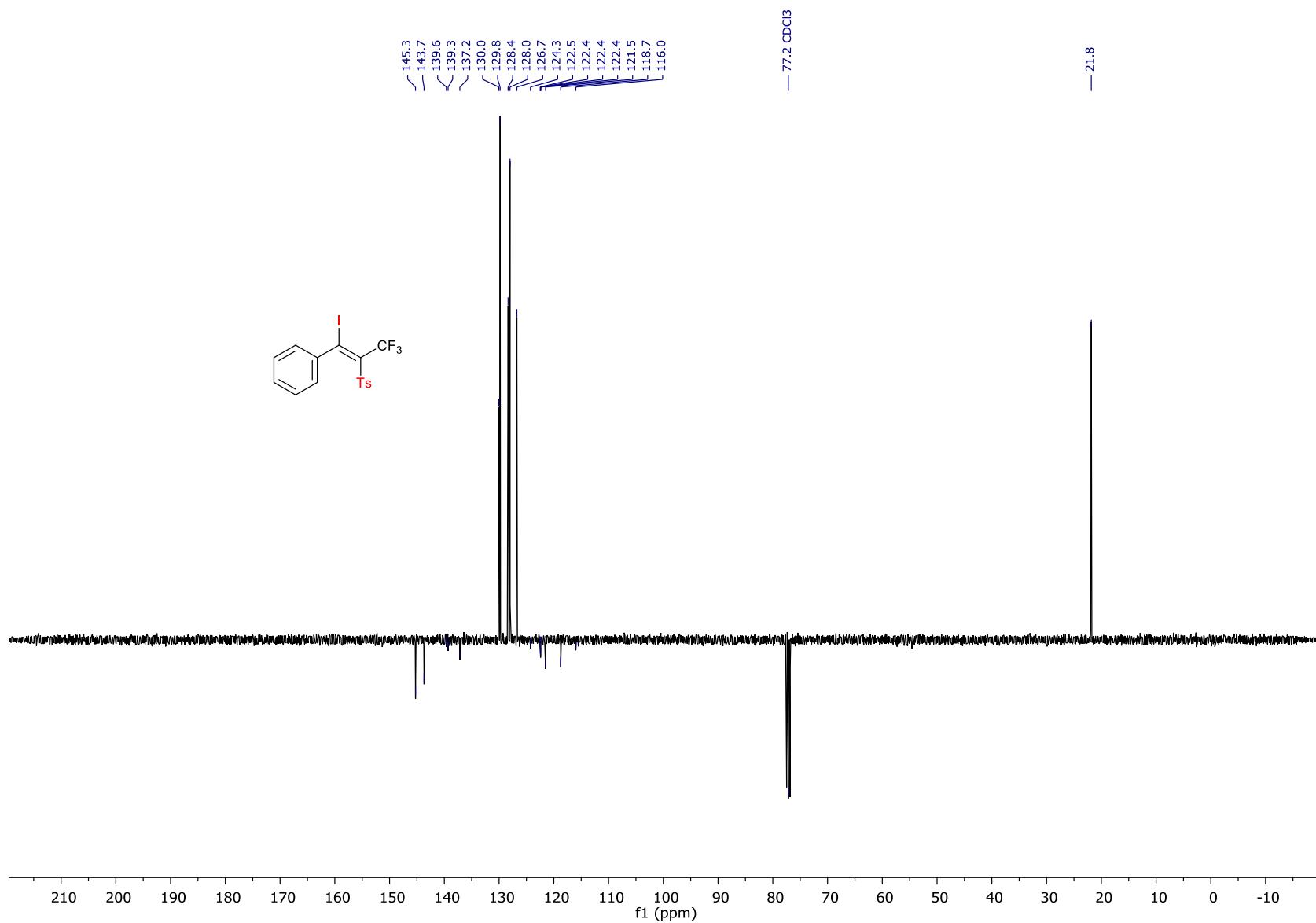


Figure S133. ^{13}C DEPTQ-135 NMR (*E*)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (**5d**).

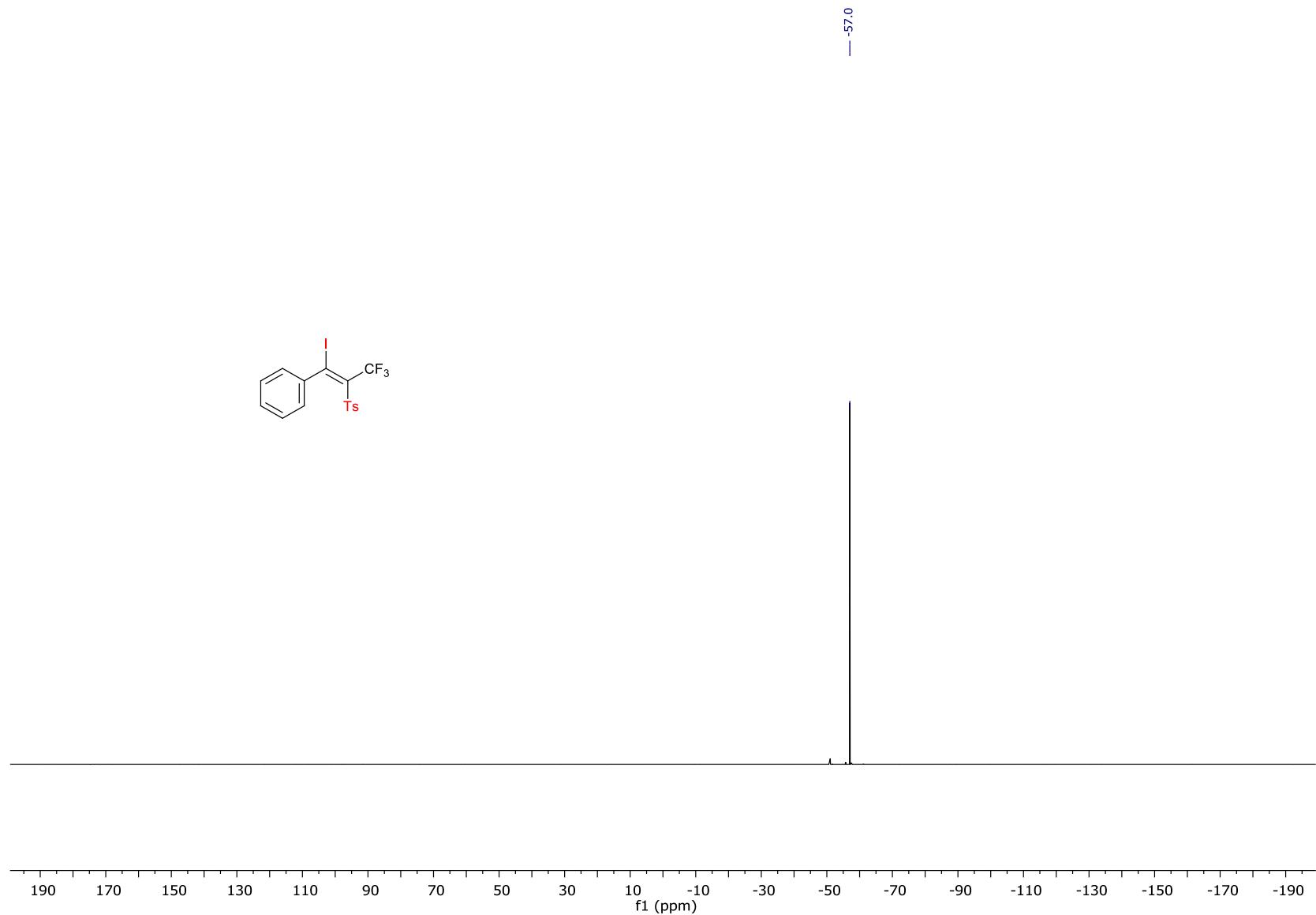


Figure S134. ^{19}F NMR (188 MHz, Chloroform-*d*) of (*E*)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (**5d**).

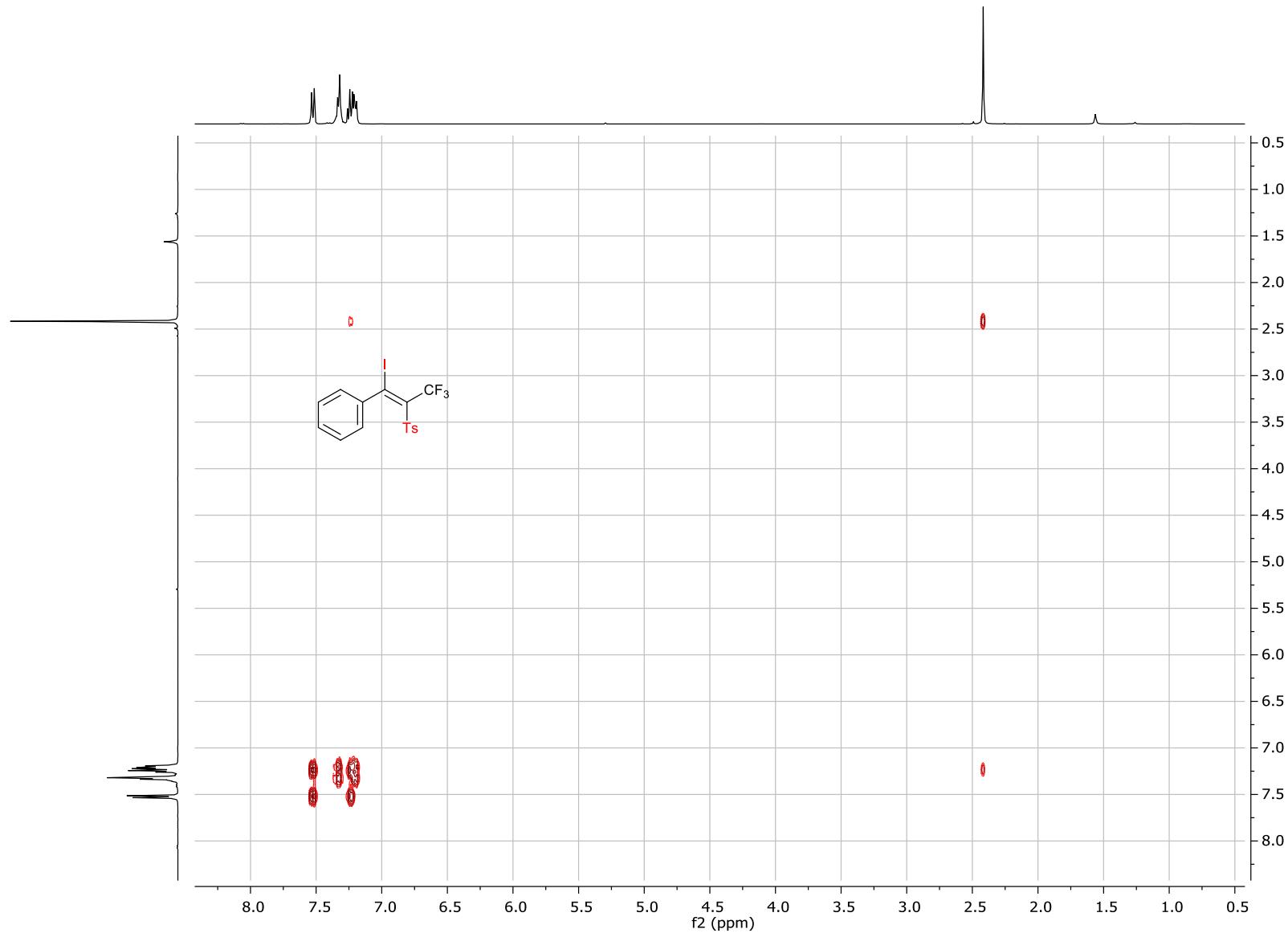


Figure S135. ^1H - ^1H COSY (*E*)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (**5d**).

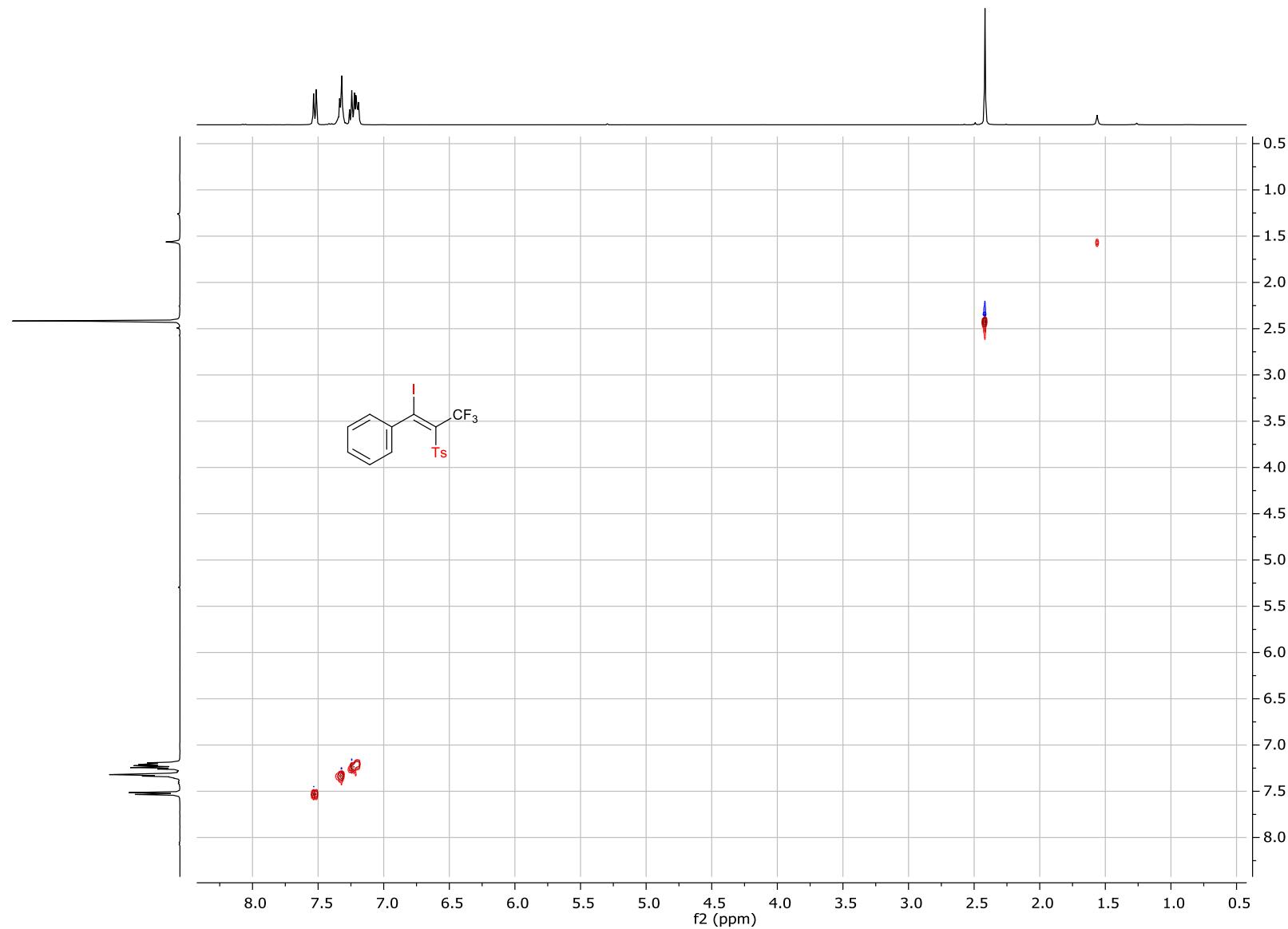


Figure S136. NOESY (*E*)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (**5d**).



Figure S137. ^1H - ^{13}C HSQC (*E*-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (**5d**).

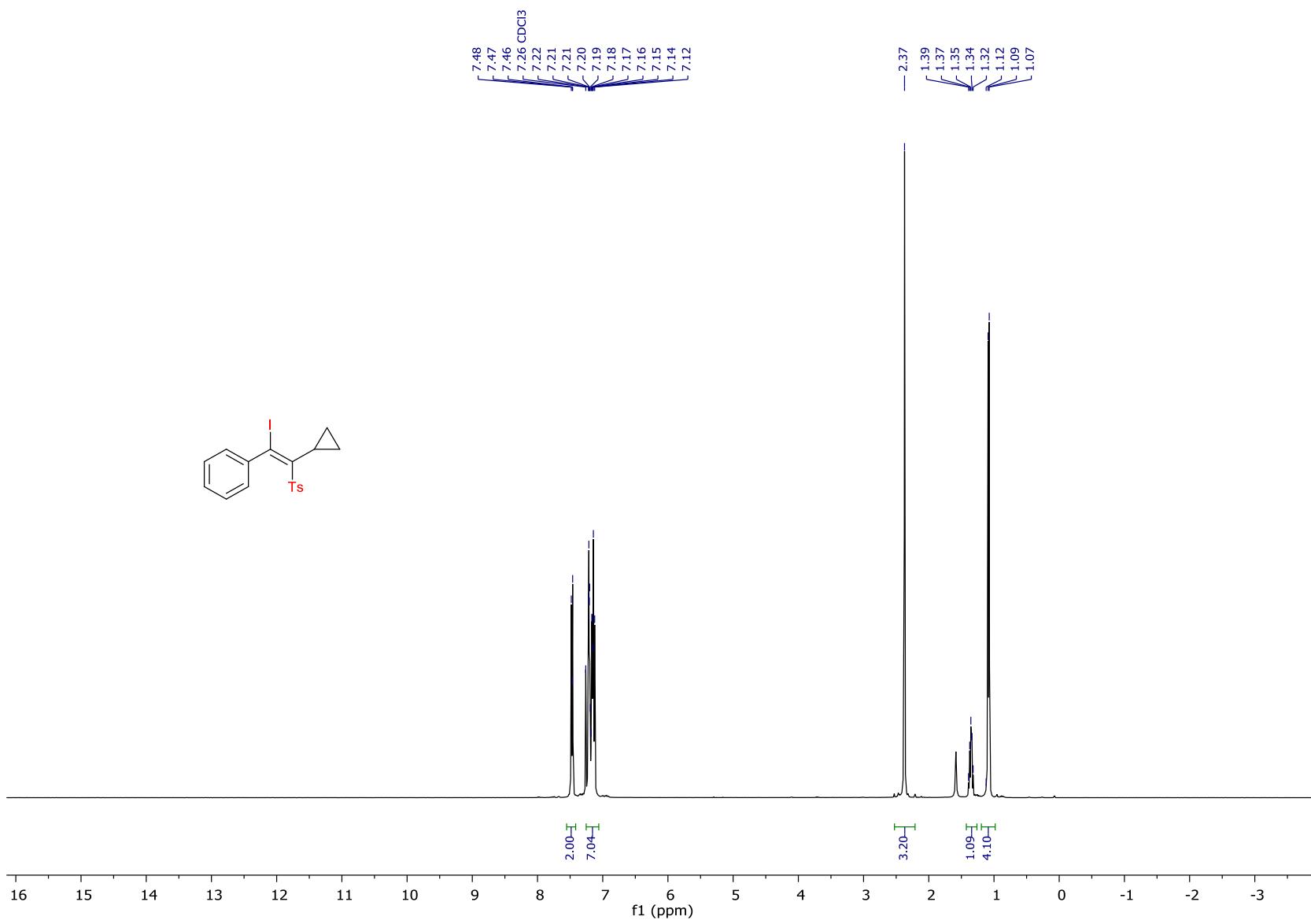


Figure S138. ^1H NMR (600 MHz, Chloroform-d) of (E)-1-(1-cyclopropyl-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5e**).

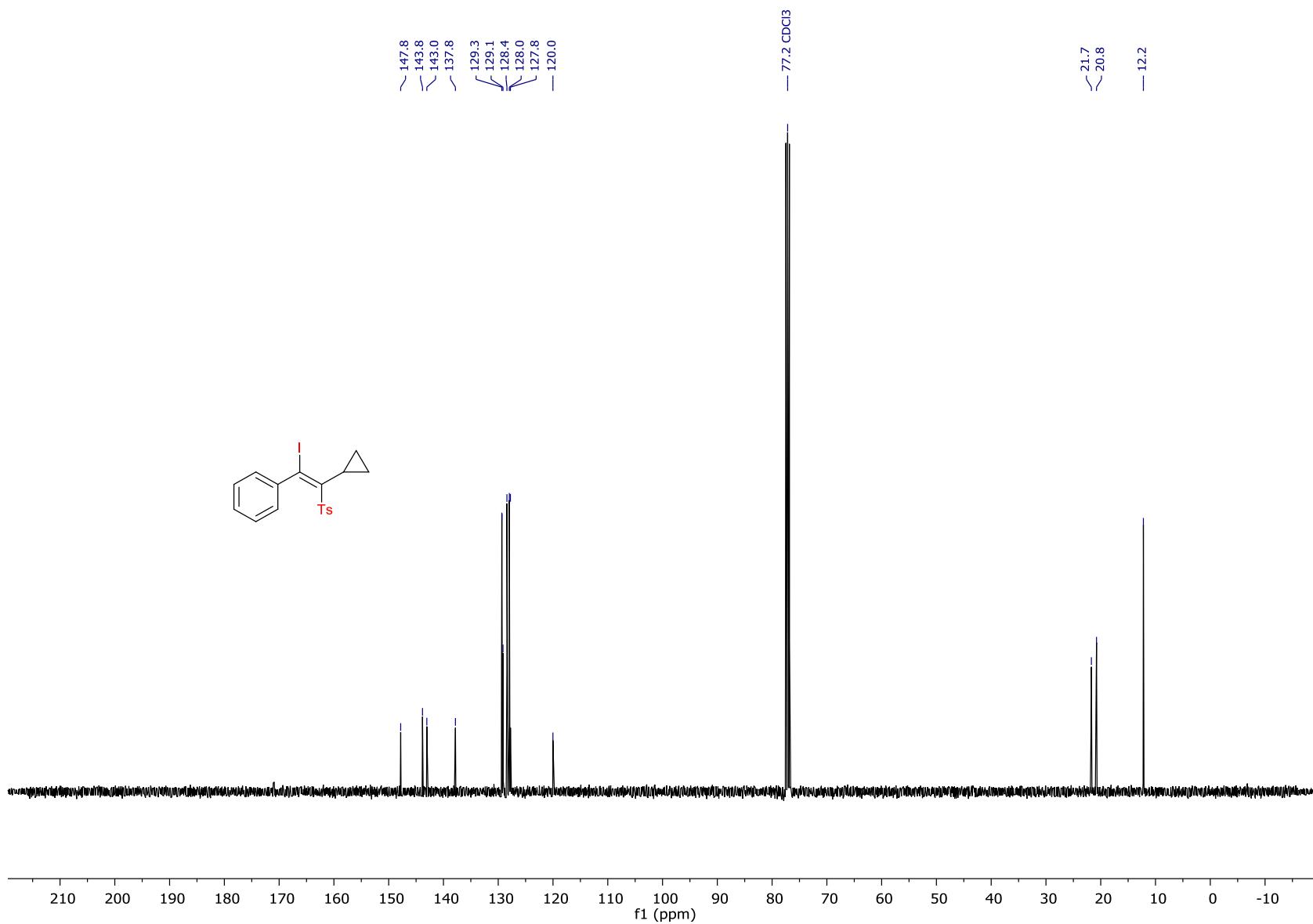


Figure S139. $^{13}\text{C}\{\text{H}\}$ NMR (151 MHz, Chloroform-d) of (E) -1-(1-cyclopropyl-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5e**).

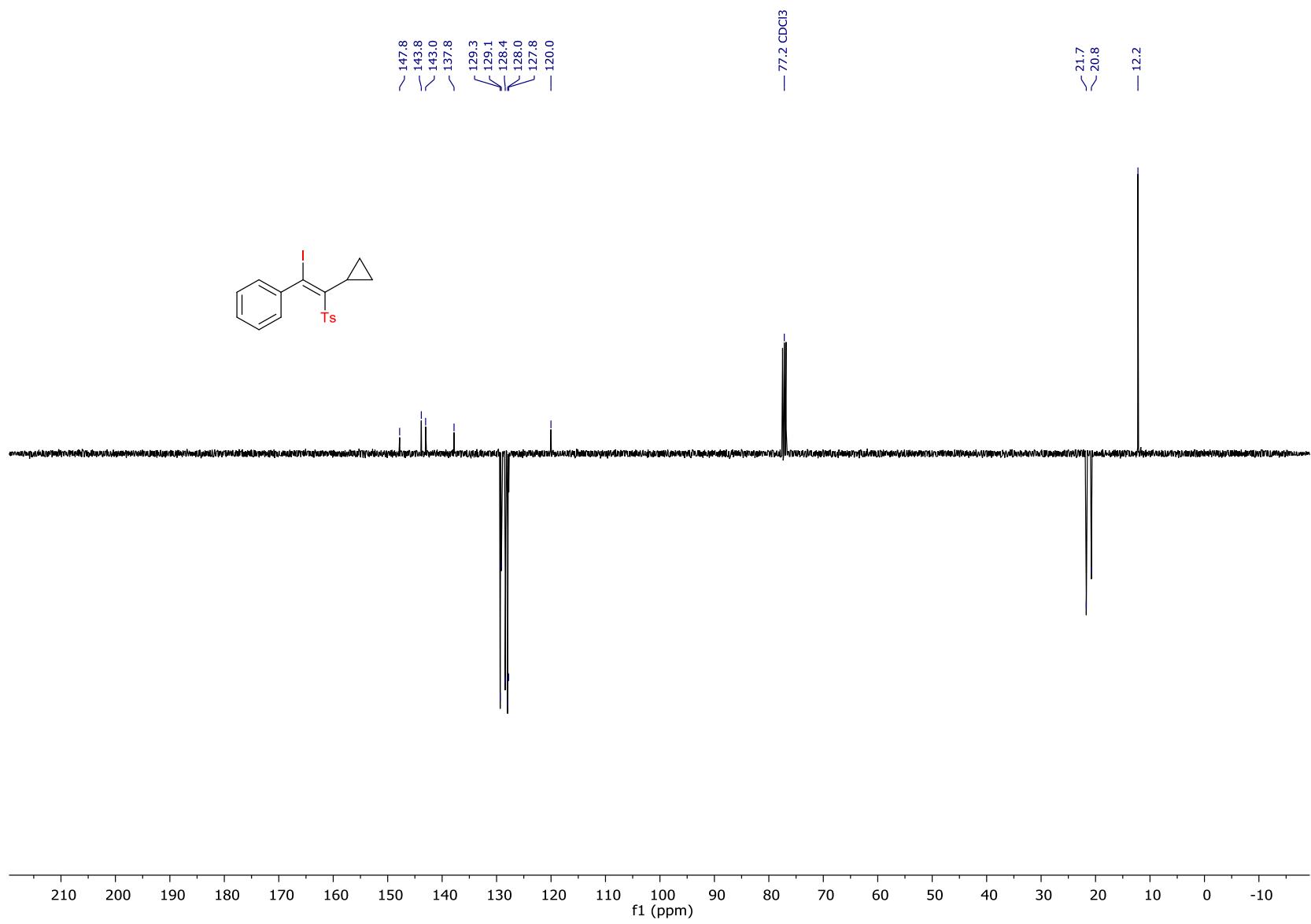


Figure S140. ^{13}C DEPTQ-135 NMR (*E*)-1-(1-cyclopropyl-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (5e).

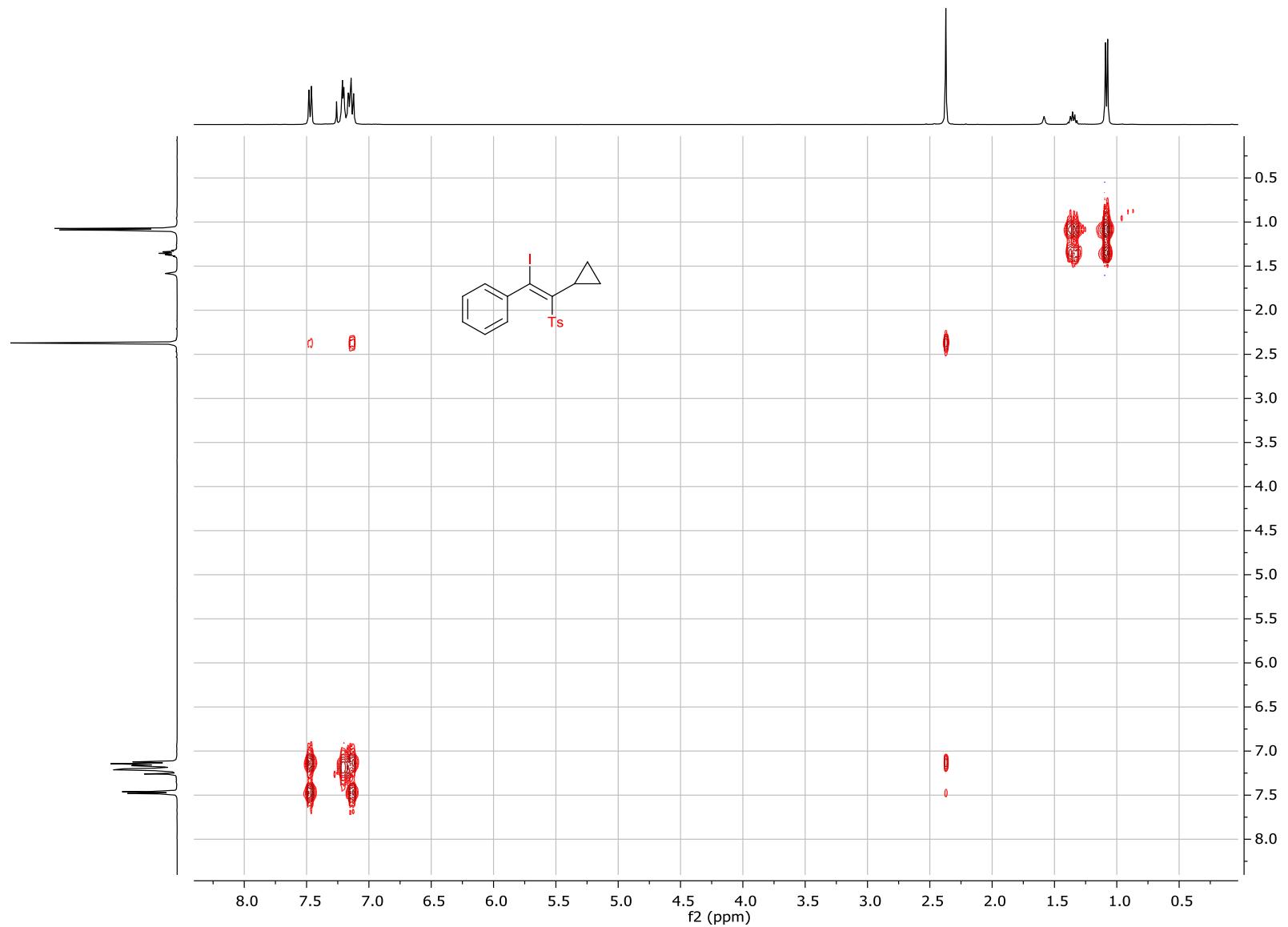


Figure S141. ^1H - ^1H COSY (*E*)-1-(1-cyclopropyl-2-iodo-2-phenylvinylsulfonyl)-4-methylbenzene (**5e**).

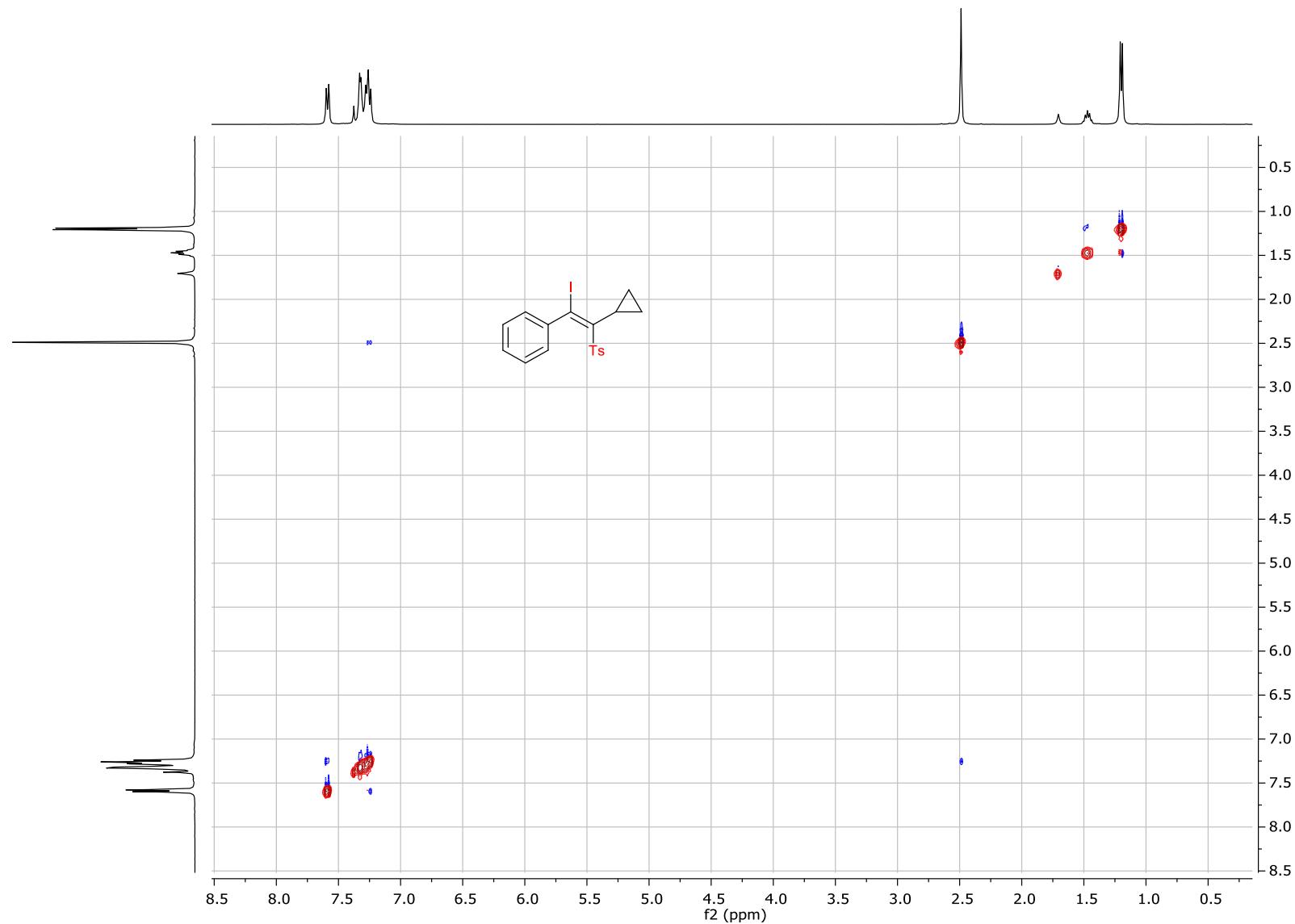


Figure S142. NOESY (*E*)-1-methyl-4-(3,3,3-trifluoro-1-iodo-1-phenylprop-1-en-2-ylsulfonyl)benzene (5e).

IR spectra of ArSO₂I

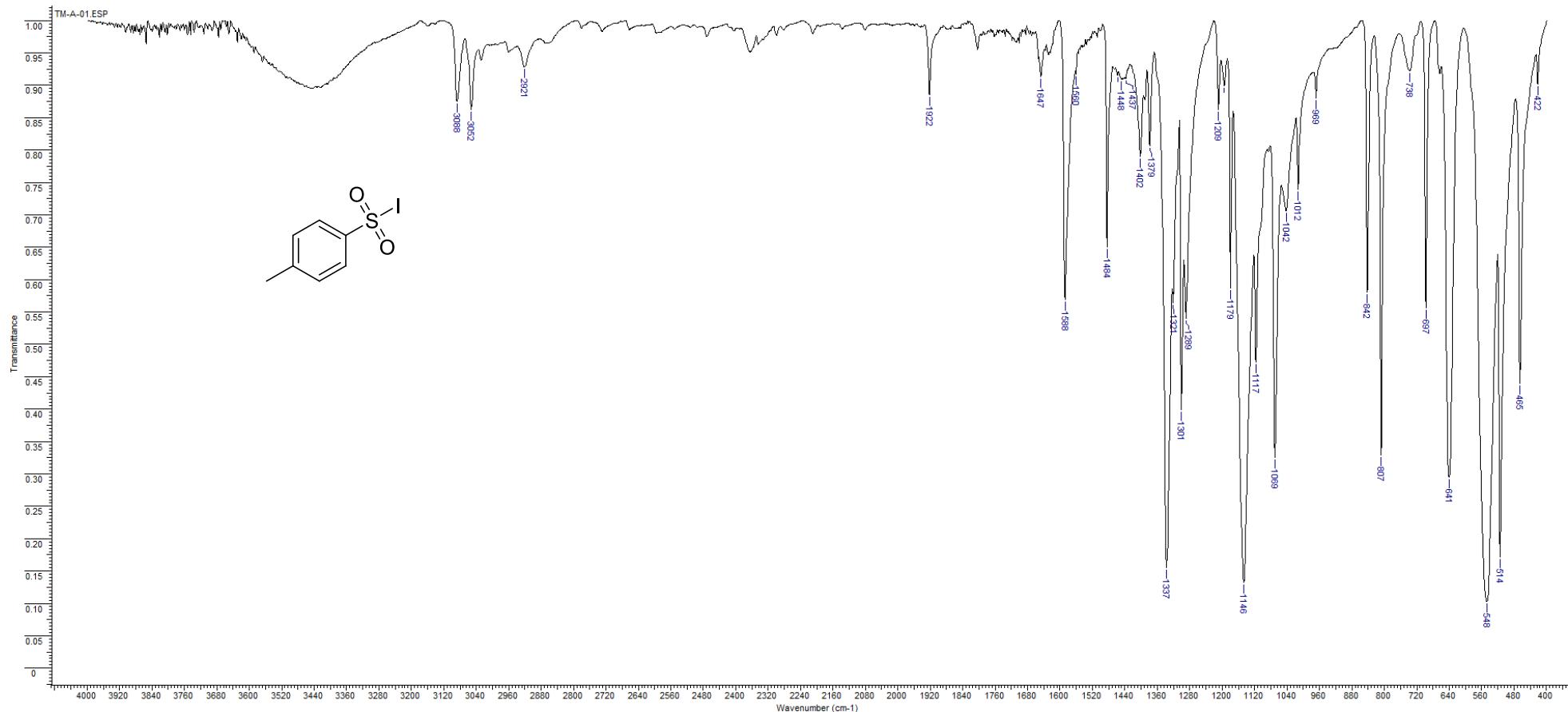


Figure S143. IR spectra of 4-methylbenzene-1-sulfonyl iodide (2)

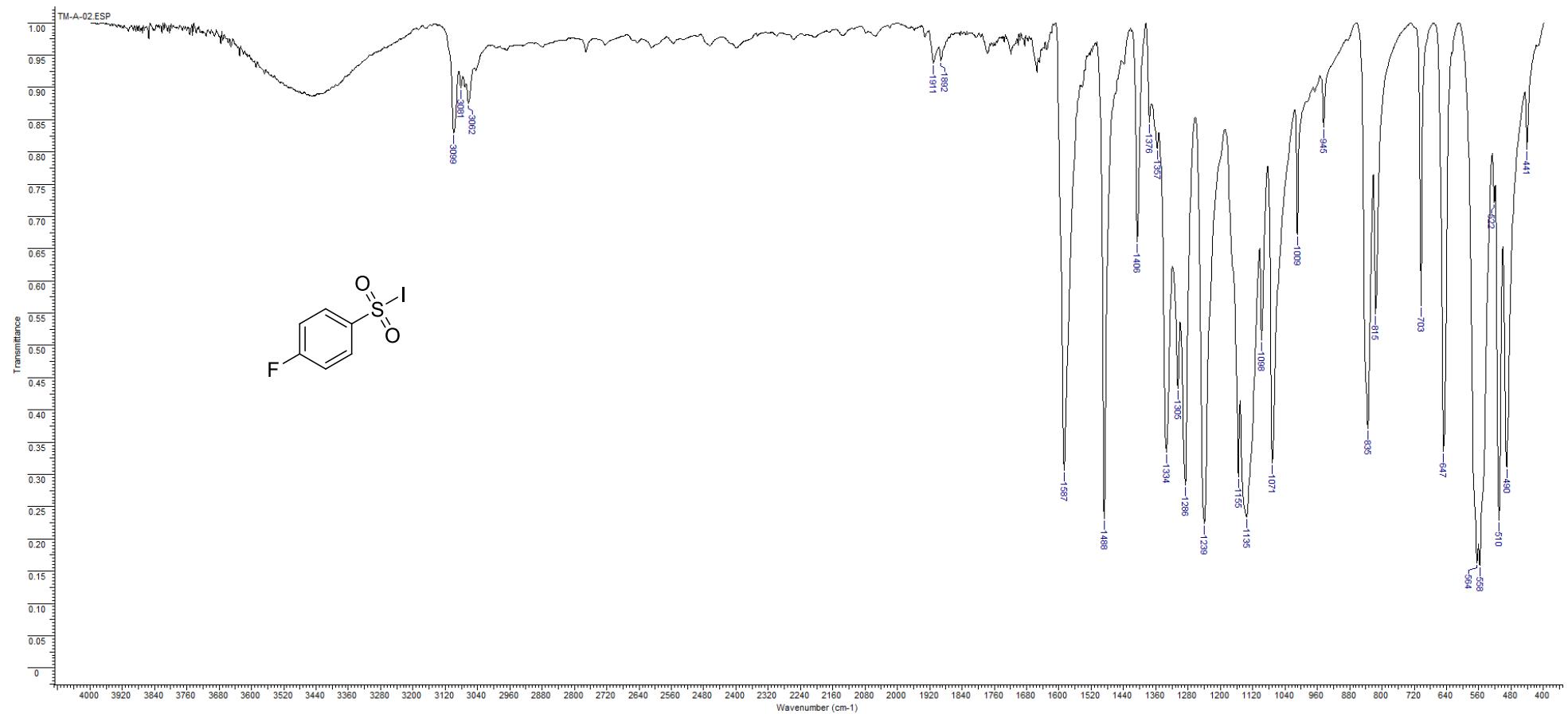


Figure S144. IR spectra of 4-fluorobenzene-1-sulfonyl iodide (2a)

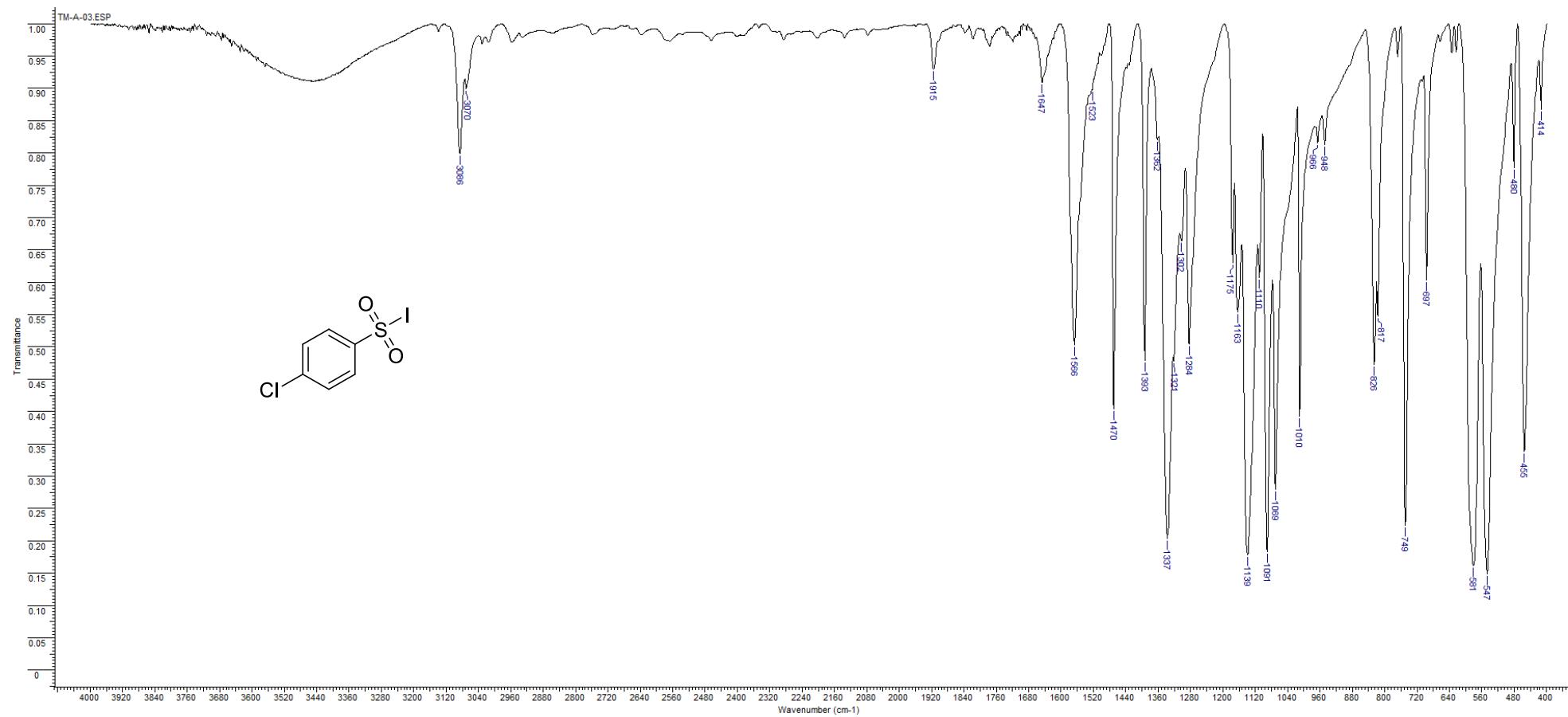


Figure S145. IR spectra of 4-chlorobenzene-1-sulfonyl iodide (2b)

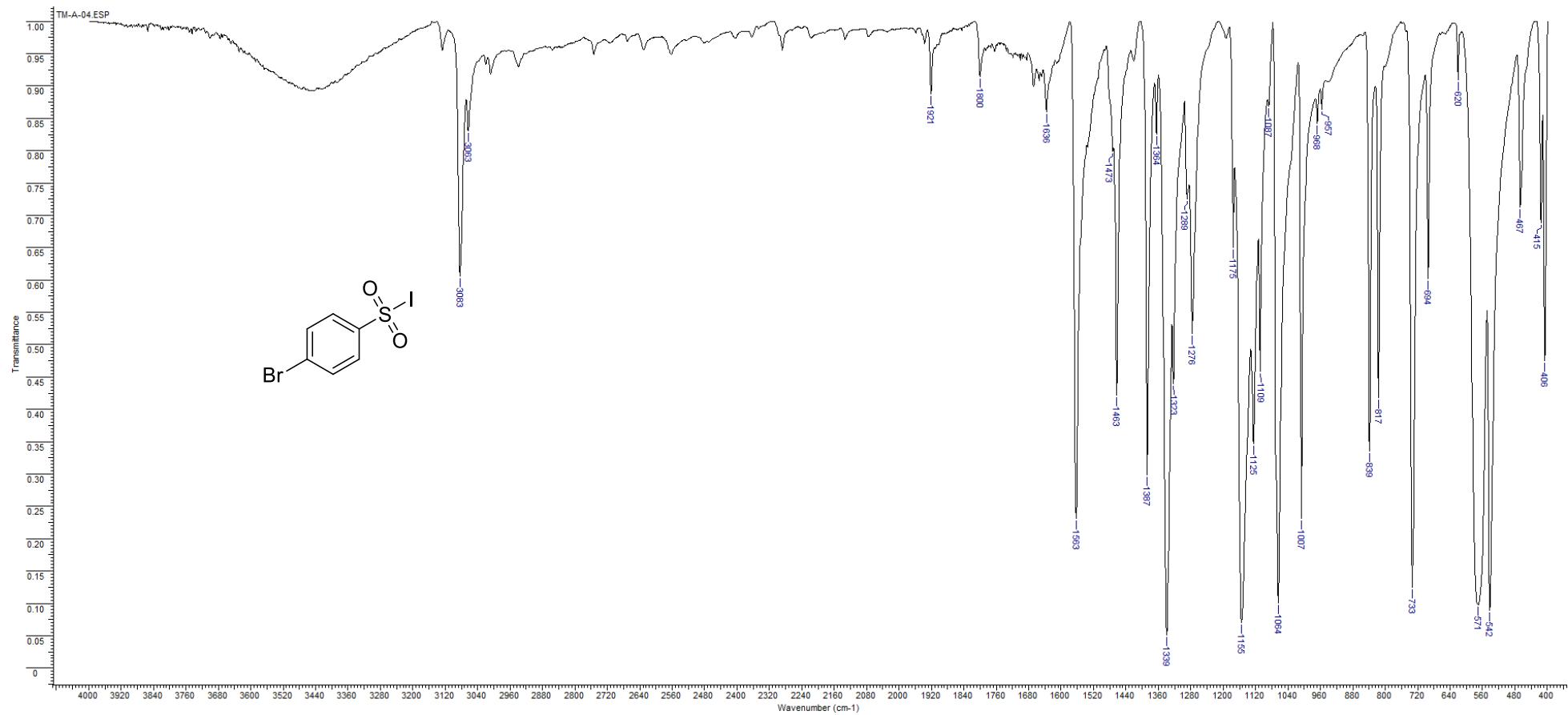


Figure S146. IR spectra of 4-bromobenzene-1-sulfonyl iodide (2c)