Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information for:

Mild Synthesis of Silyl Ethers via Potassium Carbonate Catalyzed Reactions between Alcohols and Hydrosilanes

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Contents

I. Spectroscopic Results

Triethyl(phenoxy)silane (1)

95% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.23 (t, 2H), 6.95 (t, 1H), 6.86 (d, 2H), 1.01 (t, 9H), 0.75 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 155.60, 129.40, 121.24, 119.98, 6.62, 5.00. Data agrees with literature reports.[1]

Triethyl(2-methylphenoxy)silane (2a)

78% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.06 (d, 1H, J = 7.4 Hz), 7.02 (t, 1H, J = 7.6 Hz), 6.85 (t, 1H, J = 7.4 Hz), 6.76 (d, 1H, J = 7.6 Hz), 2.21 (s, 3H), 0.99 (t, 9H, J = 7.8 Hz), 0.76 (q, 6H, J = 7.8 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 153.97, 130.85, 128.80, 126.58, 120.96, 118.40, 16.61, 6.69, 5.33. ^{29}Si NMR (80 MHz, $CDCl_3$) ∂ 19.99. MS: *m/z* 222 (M^+, 63%), 193 (100), 165 (24), 135 (7), 91 (10). HRMS(EI) Calcd for $C_{13}H_{22}OSi$ (M+): 222.1440; found: 222.1447.

Triethyl(3-methylphenoxy)silane (2b)

87% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.11 (t, 1H), 6.78–6.65 (m, 3H), 2.31 (s, 3H), 1.01 (t, 9H), 0.77 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 155.54, 139.38, 129.06, 122.05, 120.74, 116.83, 21.40, 6.66, 5.03. Data agrees with literature reports.[2]

Triethyl(4-methylphenoxy)silane (2c)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.03 (d, 2H), 6.75 (d, 2H), 2.29 (s, 3H), 1.01 (t, 9H), 0.73 (q, 6H). ^{13}C NMR (75MHz, $CDCl_3$) ∂ 153.24, 130.42, 129.67, 119.67, 20.58, 6.65, 4.97. Data agrees with literature reports.[1]

(2-bromophenoxy)triethylsilane (3a)

84% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.52 (d, 1H), 7.13(t, 1H), 6.88-6.78 (m, 2H), 0.99 (t, 9H), 0.78 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 152.71, 133.30, 128.26, 122.37, 120.27, 115.40, 6.64, 5.23. Data agree with literature reports.[3]

(3-bromophenoxy)triethylsilane (3b)

91% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.10–7.08 (m, 2H), 7.03 (s, 1H), 6.81–6.77 (m, 1H), 1.01 (t, 9H), 0.75 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 156.52, 130.43, 124.42, 123.36, 122.49, 118.66, 6.81, 4.94. Data agrees with literature reports.[4]

(4-bromophenoxy)triethylsilane (3c)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.32 (d, 2H), 6.73 (d,2H), 0.97 (t, 9H), 0.72 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 154.82, 132.31, 121.74, 113.54, 6.81, 4.93. Data agrees with literature reports.[5]

Triethyl(2-methoxyphenoxy)silane (4a)

87% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 6.95–6.79 (m, 4H), 3.82 (s, 3H), 1.00 (t, 9H), 0.74 (t, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 151.03, 145.03, 121.68, 120.84, 112.13, 55.48, 6.80, 5.11. Data agrees with literature reports.[6]

Triethyl(3-methoxyphenoxy)silane (4b)

67% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.11 (t, 1H, J = 8.1 Hz), 6.53–6.42 (m, 3H) 3.77 (s, 3H), 0.98 (q, 9H, J = 7.8Hz), 0.76 (t, 6H, J = 7.8 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 160.72, 156.79, 129.69, 112.41, 106.81, 106.16, 55.20, 6.81, 5.01. ^{29}Si NMR (80 MHz, $CDCl_3$) ∂ 20.97. MS: *m/z* 238 (M^+, 60%), 209 (100), 181 (46), 153 (26), 121 (4), 107 (7), 91 (21), 77 (29). HRMS(EI) Calcd for $C_{13}H_{22}O_2Si$ (M+): 238.1389; found: 238.1398.

Triethyl(4-methoxyphenoxy)silane (4c)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 6.78–6.74 (m, 4H), 3.76 (s, 3H), 0.99 (t, 9H), 0.70 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 154.07, 149.29, 120.50, 114.48, 55.62, 6.63, 4.92. Data agrees with literature reports.[1]

Triethyl(4-nitrophenoxy)silane (5)

98% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 8.21–8.14 (m, 2H), 6.94-6.90 (m, 2H), 1.01 (t, 9H), 0.79 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 161.57, 146.41, 125.89, 119.90, 6.49, 5.00. Data agrees with literature reports.[5]

Dimethylphenyl(phenoxy)silane (6)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.65–7.13 (m, 10H), 0.34 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 153.70, 133.47, 133.00, 131.16, 130.91, 129.90, 129.25, 128.97, 127.96, 127.71, 120.05, 118.27, 0.86. Data agrees with literature reports.[6]

(Phenoxy)triphenylsilane (7)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.72–7.70 (m, 6H), 7.52–7.39 (m, 9H), 7.19–7.14 (m, 2H), 6.97–6.90 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 155.07, 135.54, 135.22 133.60, 130.33, 129.82, 129.34, 127.98, 127.72, 121.58, 120.08. Data agrees with literature reports.[7]

Triisopropyl(phenoxy)silane (8)

67% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.23–7.19 (m, 2H), 6.95–6.87 (m, 3H), 1.31–1.26 (m, 3H), 1.21–1.08 (m, 18H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 156.07, 129.34, 120.97, 119.94, 17.92, 12.07. Data agrees with literature reports.[8]

Dimethyl*tert*-butyl(phenoxy)silane (9)

99% yield; colorless oil; ^{1}H NMR (300 MHZ, $CDCl_3$) ∂ 7.23 (t, 2H), 6.95 (t, 1H), 6.84 (d, 2H), 0.99 (s, 9H), 0.21 (s, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 155.64, 129.38, 121.27, 120.12, 25.69, 18.21, -4.42. Data agrees with literature reports.[9]

(Benzyloxy)triphenylsilane (10)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.62–7.37 (m, 20H), 5.49 (s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 135.83, 135.46, 134.01, 130.12, 128.25, 127.93, 127.07, 126.38, 65.58. Data agrees with literature reports.[10]

Triethyl(4-*tert*butylbenzyloxy)silane (11)

98% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.39–7.28 (m, 4H), 4.72 (s, 2H), 1.34 (s, 9H), 1.00 (t, 9H), 0.70 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 149.89, 138.28, 126.10, 125.14,

64.54, 34.47, 31.41, 6.81, 4.51. Data agree with literature reports.[11]

(2-phenylethoxy)triphenylsilane (12)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.61–7.12 (m, 20H), 3.93 (t, 2H), 2.81 (t, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 138.82, 135.40, 134.19, 130.02, 129.21, 128.27, 127.87, 126.18, 65.09, 39.25. Data agrees with literature reports.[10]

Triethyl[(3-phenyl-2-propenyl)oxy]silane (13)

87% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.30–7.21 (m, 5H), 6.60 (d, 1H), 6.36–6.28 (m, 1H), 4.37 (dd, 2H), 1.02 (t, 9H), 0.68 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 137.14, 129.75, 128.50, 127.34, 126.41, 63.60, 6.80, 4.54. Data agrees with literature reports.[12]

(Cyclohexyloxy)triphenylsilane (14)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.52–7.13 (m, 15H), 3.79–3.71 (m, 1H), 1.65–1.03 (m, 10H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 135.48, 135.20, 129.84, 127.87, 127.78, 71.82, 35.61, 25.64, 23.96. Data agrees with literature reports.[10]

(3-chloropropyl)triethylsilane (15)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 3.76 (t, 2H), 3.65 (t, 2H), 1.98 (qu, 2H), 0.97 (t, 9H), 0.58 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 58.87, 41.34, 34.78, 7.87, 2.30. Data agrees with literature reports.[13]

Triethyl(4-methoxypyridyl)silane (16)

83% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 8.57 (d, 2H), 7.29 (d, 2H), 4.76 (s, 2H), 1.00 (t, 9H), 0.69 (q, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 150.55, 149.62, 120.69, 63.19, 6.74, 4.42. Data agree with literature reports.[14]

Triethyl(furfuryloxy)silane (17)

56% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.39–7.38 (m, 1H), 6.31–6.24 (m, 2H), 4.63 (s, 2H), 0.96 (q, 9H), 0.63 (t, 6H). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 142.13, 110.21, 107.41, 57.69, 6.60, 4.40. Data agree with literature reports.[14]

N(2-((triethylsilyl)oxy)ethyl)phthalimide (18)

99% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.88–7.85 (m, 2H), 7.74–7.71 (m, 2H), 3.86 (s, 4H), 0.91 (t, 9H, J = 7.9 Hz), 0.57 (q, 6H, J = 7.9 Hz). ^{13}C NMR (75 MHz, $CDCl_3$) ∂ 168.33, 133.89, 132.16, 123.17, 59.73, 40.16, 6.60, 4.30. ^{29}Si NMR (80 MHz, $CDCl_3$) ∂ 19.60. MS: *m/z* 276 (M – Et, 100%), 232 (16), 204 (3), 160 (6), 130 (18), 87 (4). HRMS(EI) Calcd for $C_{16}H_{23}NO_3Si$ (M+): 276.1056; found: 276.1056.

(3-ethynylphenoxy)triethylsilane (19)

81% yield; colorless oil; ^{1}H NMR (300 MHz, $CDCl_3$) ∂ 7.09 (t, 1H, J = 7.8 Hz), 6.98 (d, 1H, J = 7.6 Hz), 6.87 (s, 1H), 6.71 (m, 1H), 3.05 (s, 1H), 1.00 (t, 9H, J = 7.8 Hz), 0.71 (q, 6H, J = 7.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$) ∂ 155.41, 129.42, 125.27, 123.45, 123.05, 121.03, 83.54, 6.59, 4.96. ^{29}Si NMR (80 MHz, $CDCl_3$) ∂ 21.74. MS: *m/z* 232 (M^+, 57%), 203 (100), 175 (49), 147 (42), 115 (5), 101 (22), 88 (18), 74 (17). HRMS (EI) Calcd for $C_{14}H_{20}OSi$ (M+): 232.1283; found: 232.1293

II. NMR Spectra

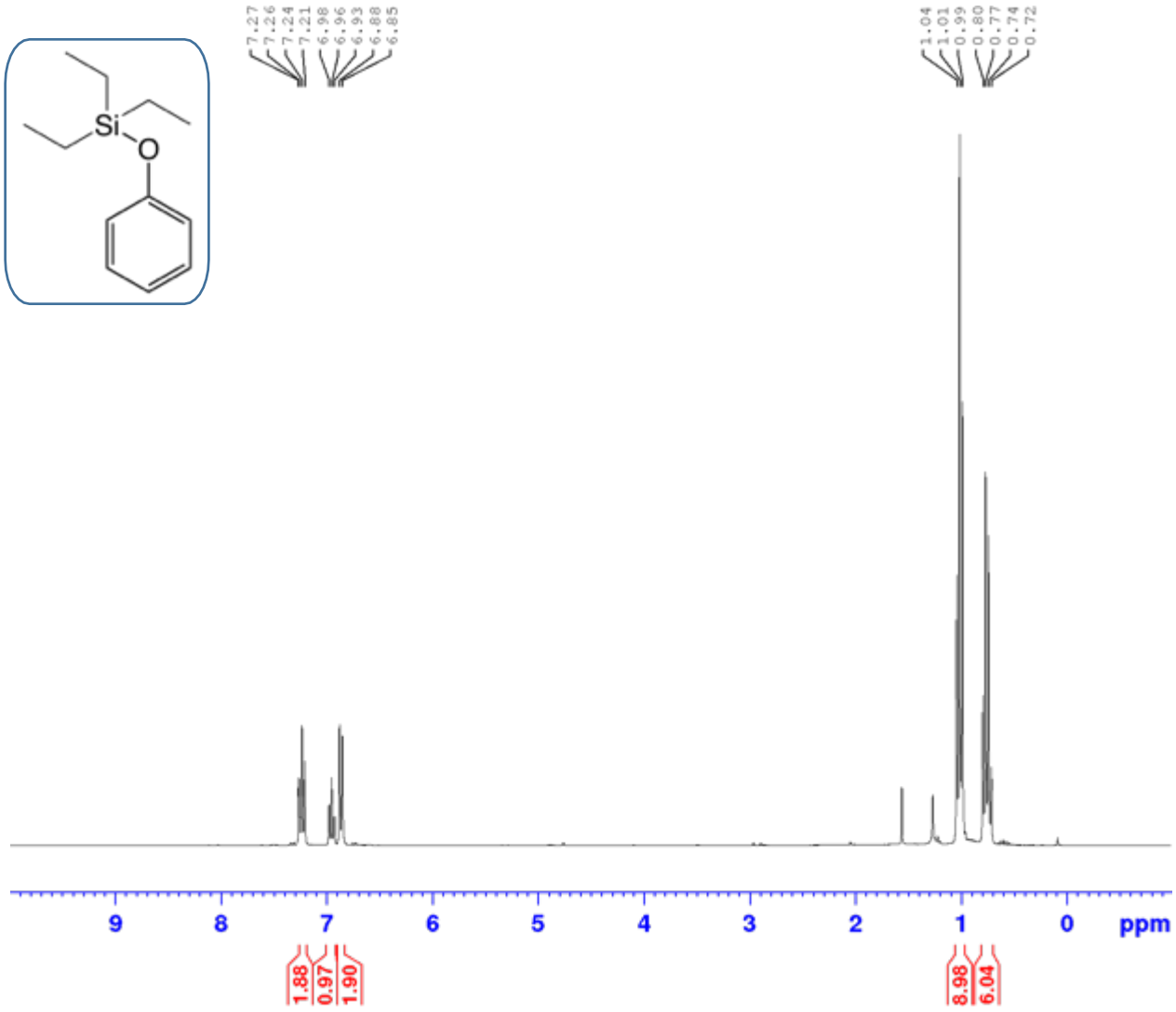

Fig. S1. ^{1}H NMR spectrum of product **1** in $CDCl_3$

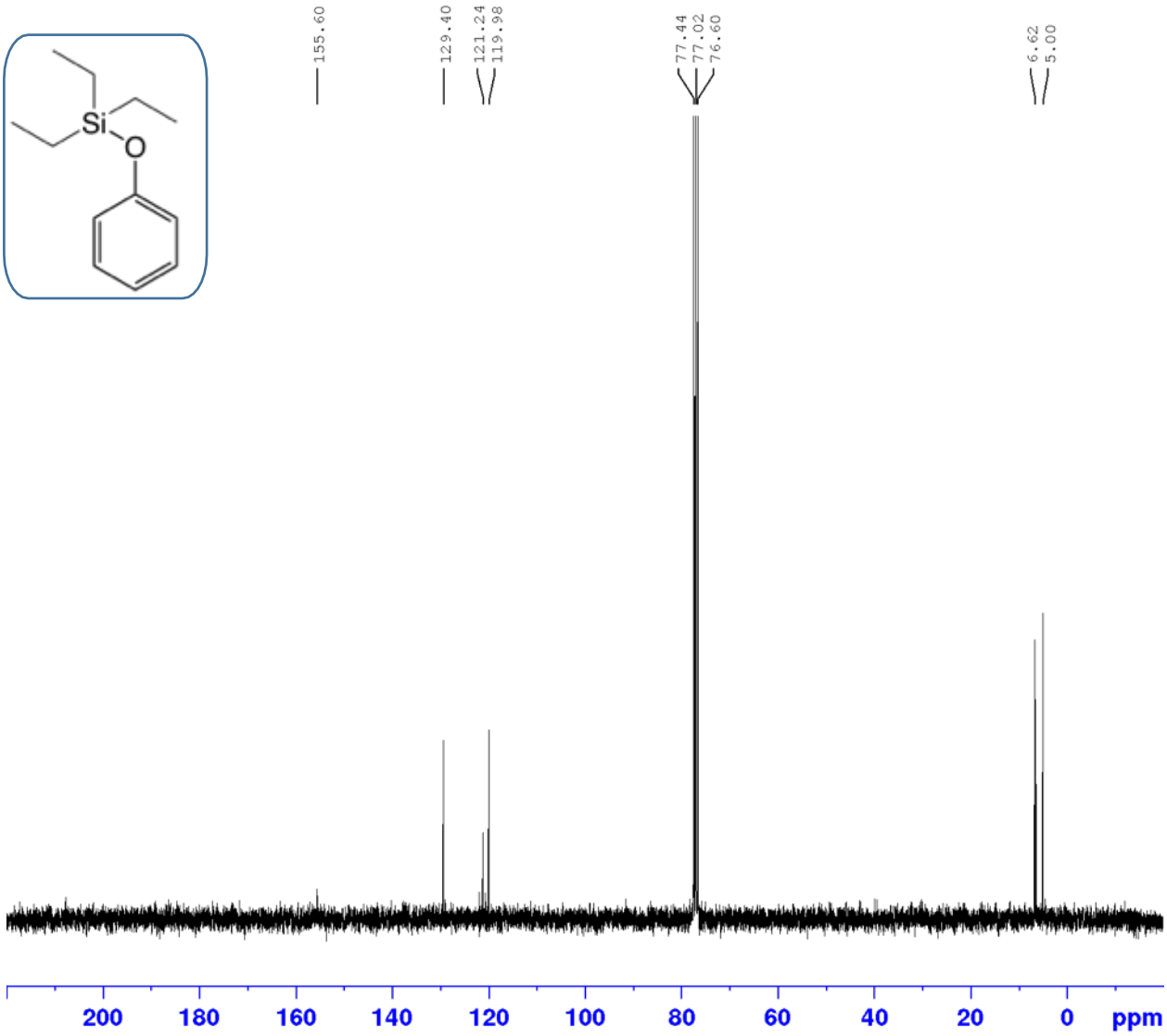

Fig. S2. ^{13}C NMR spectrum of product **1** in $CDCl_3$

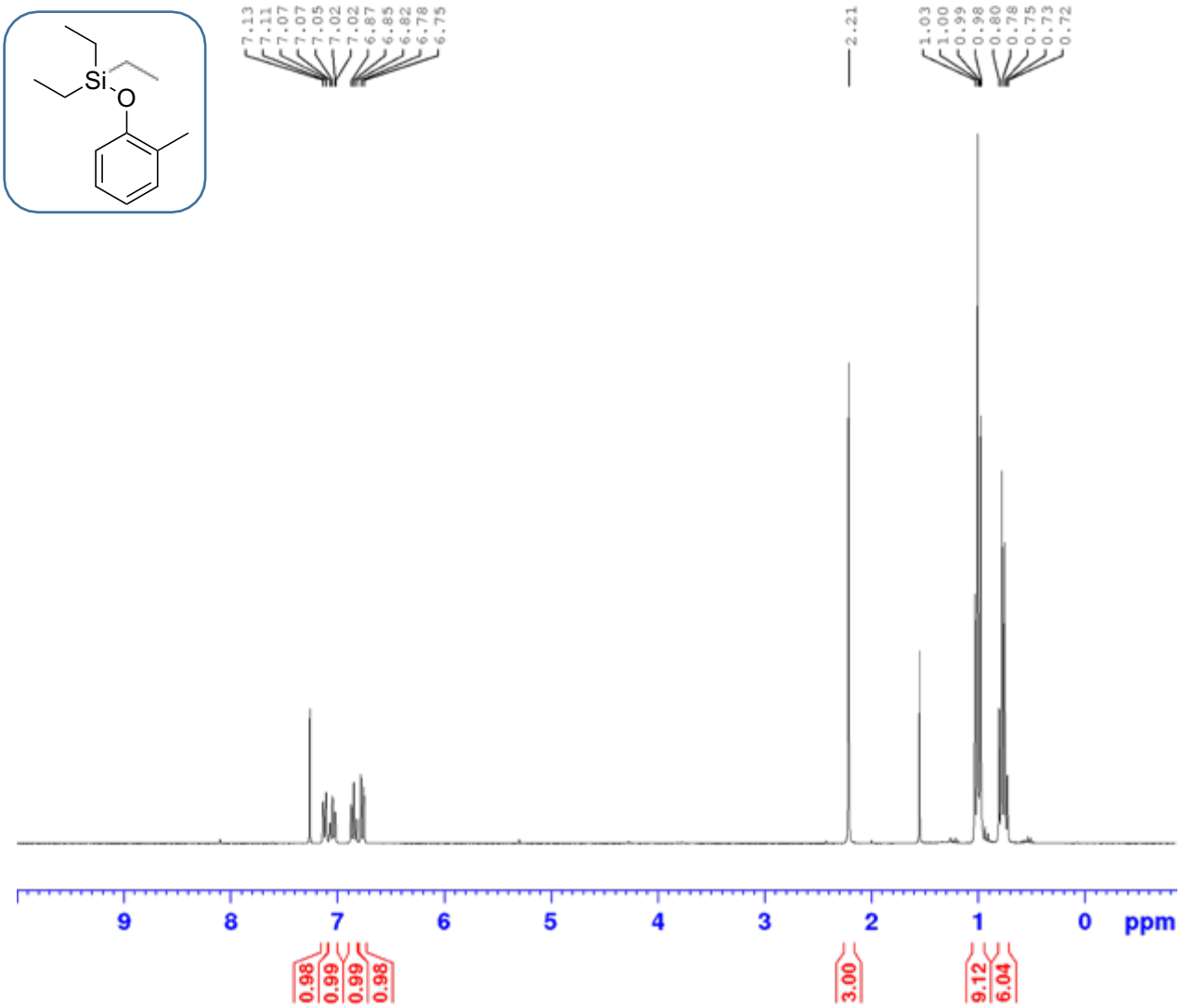

Fig. S3. ^{1}H NMR spectrum of compound **2a** in $CDCl_3$

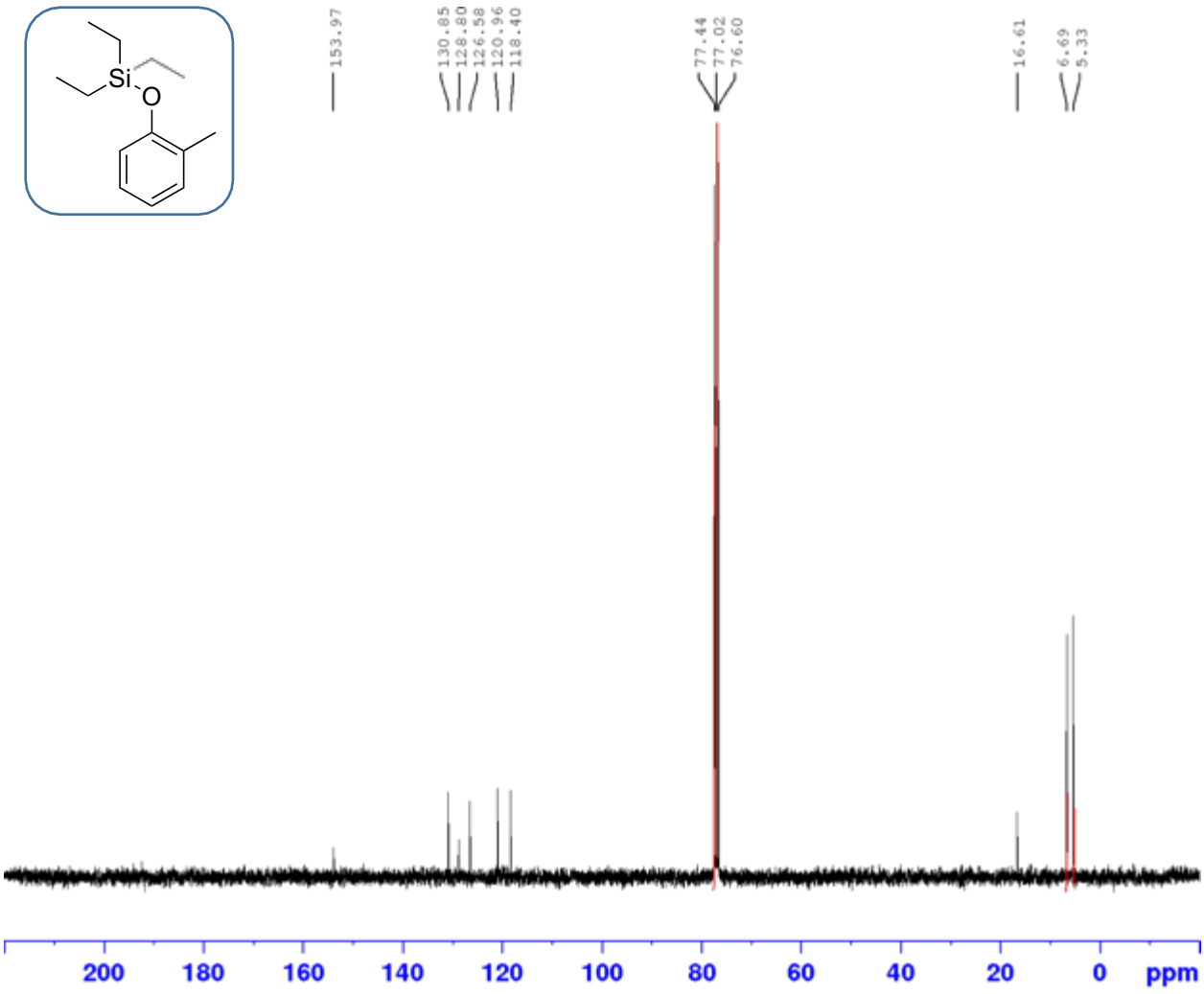

Fig. S4. ^{13}C NMR spectrum of compound **2a** in $CDCl_3$

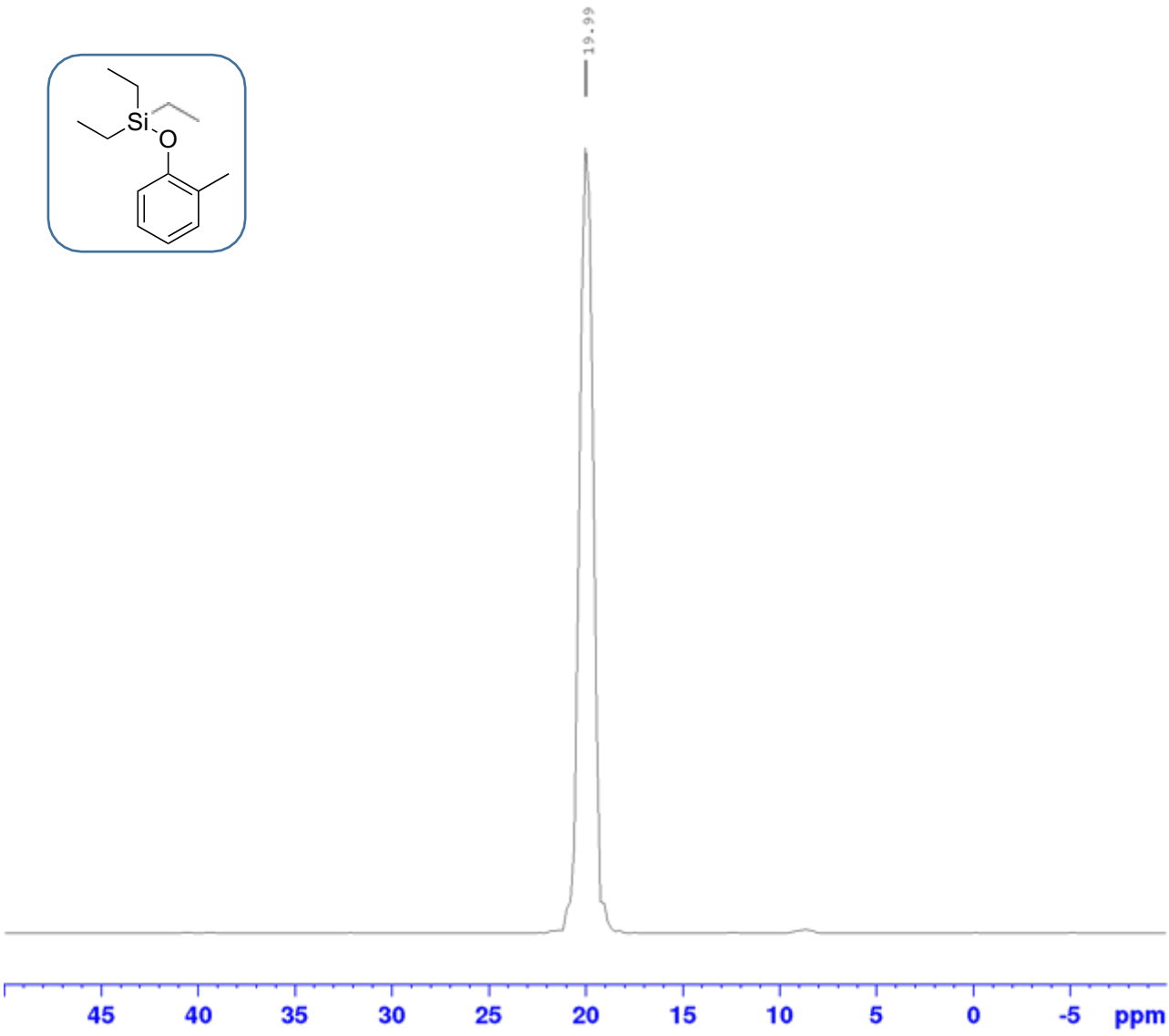

Fig. S5. ^{29}Si NMR spectrum of compound **2a** in $CDCl_3$

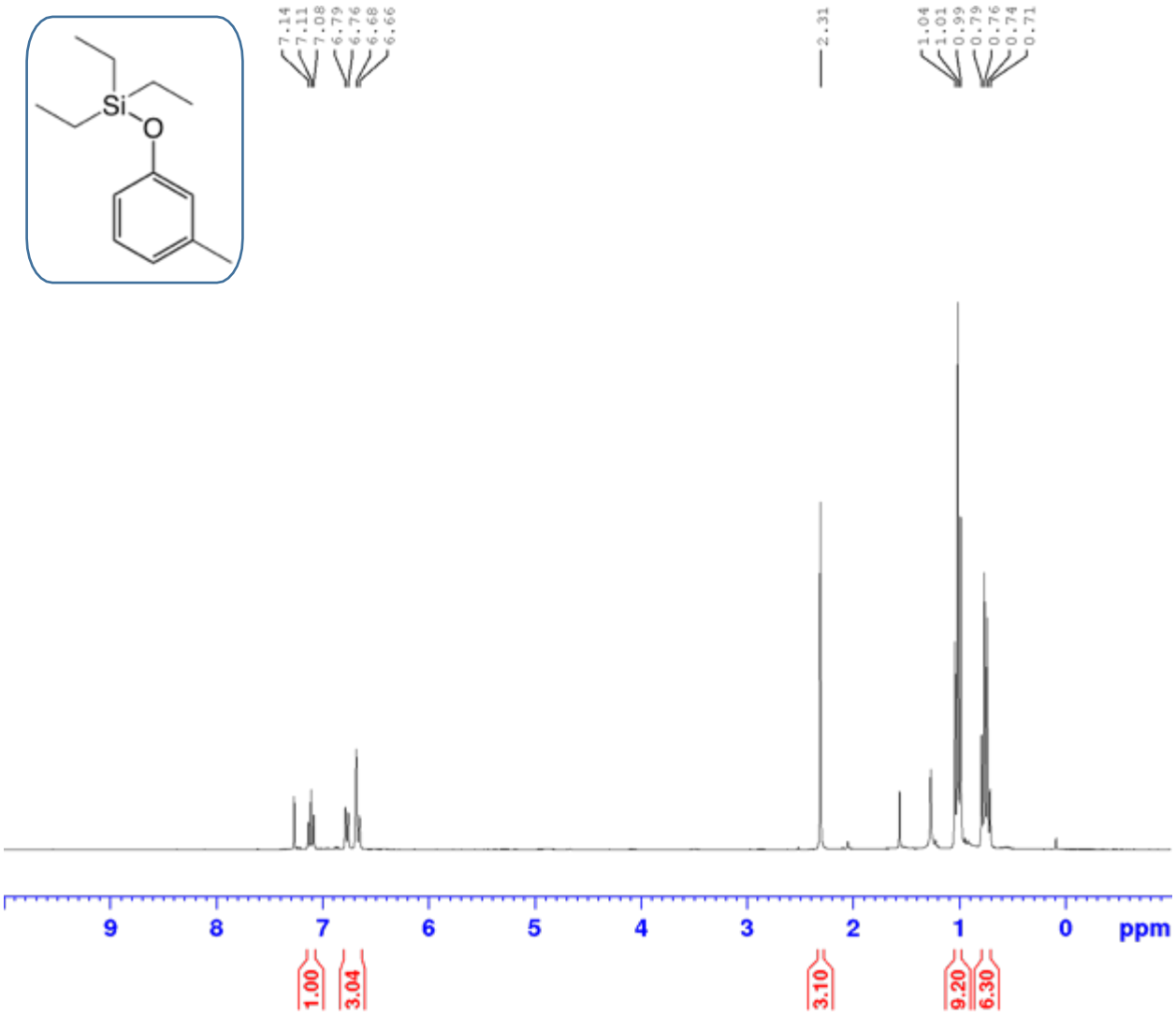

Fig. S6. ^{1}H NMR spectrum of product **2b** in $CDCl_3$

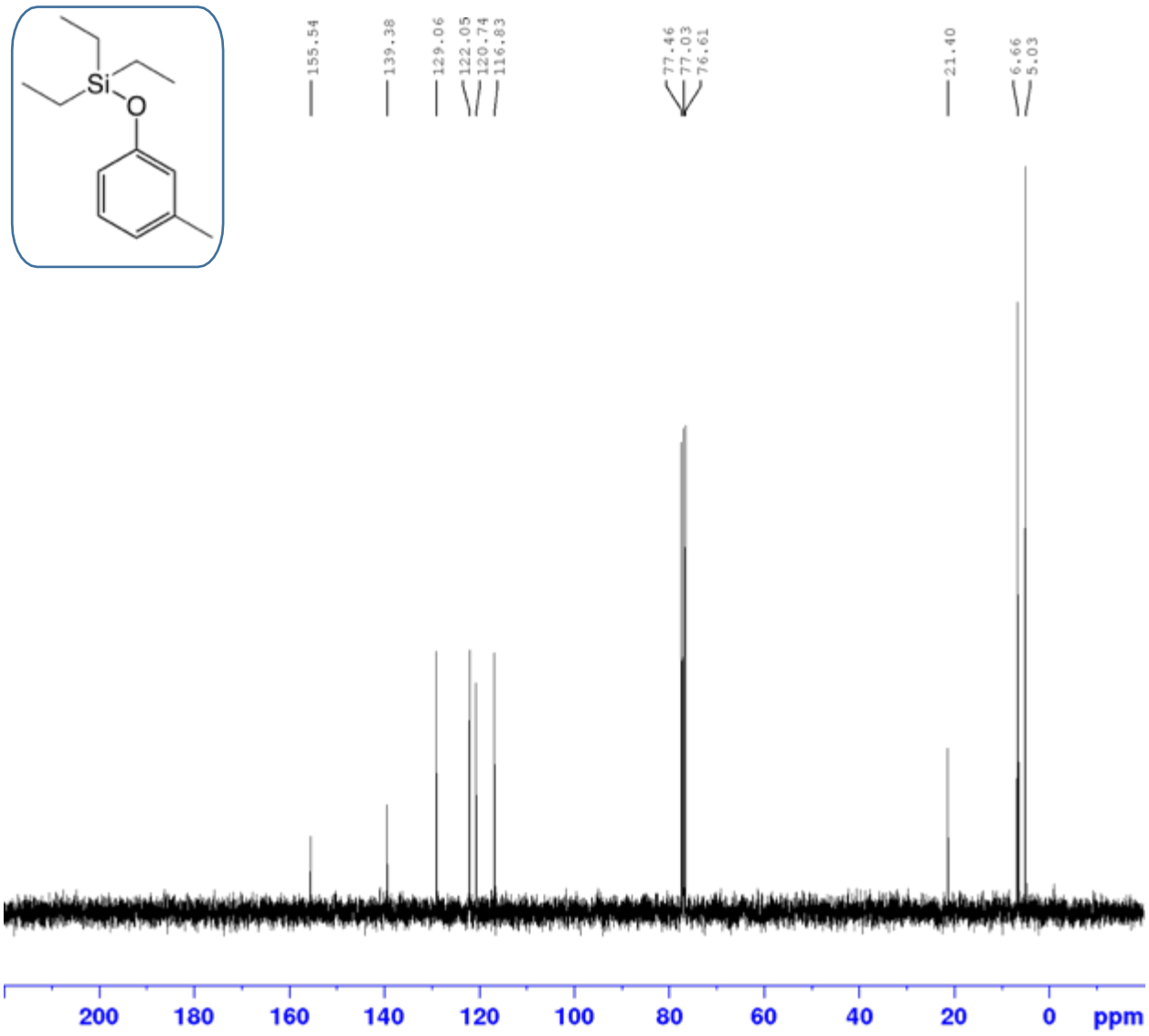

Fig. S7. ^{13}C NMR spectrum of product **2b** in $CDCl_3$

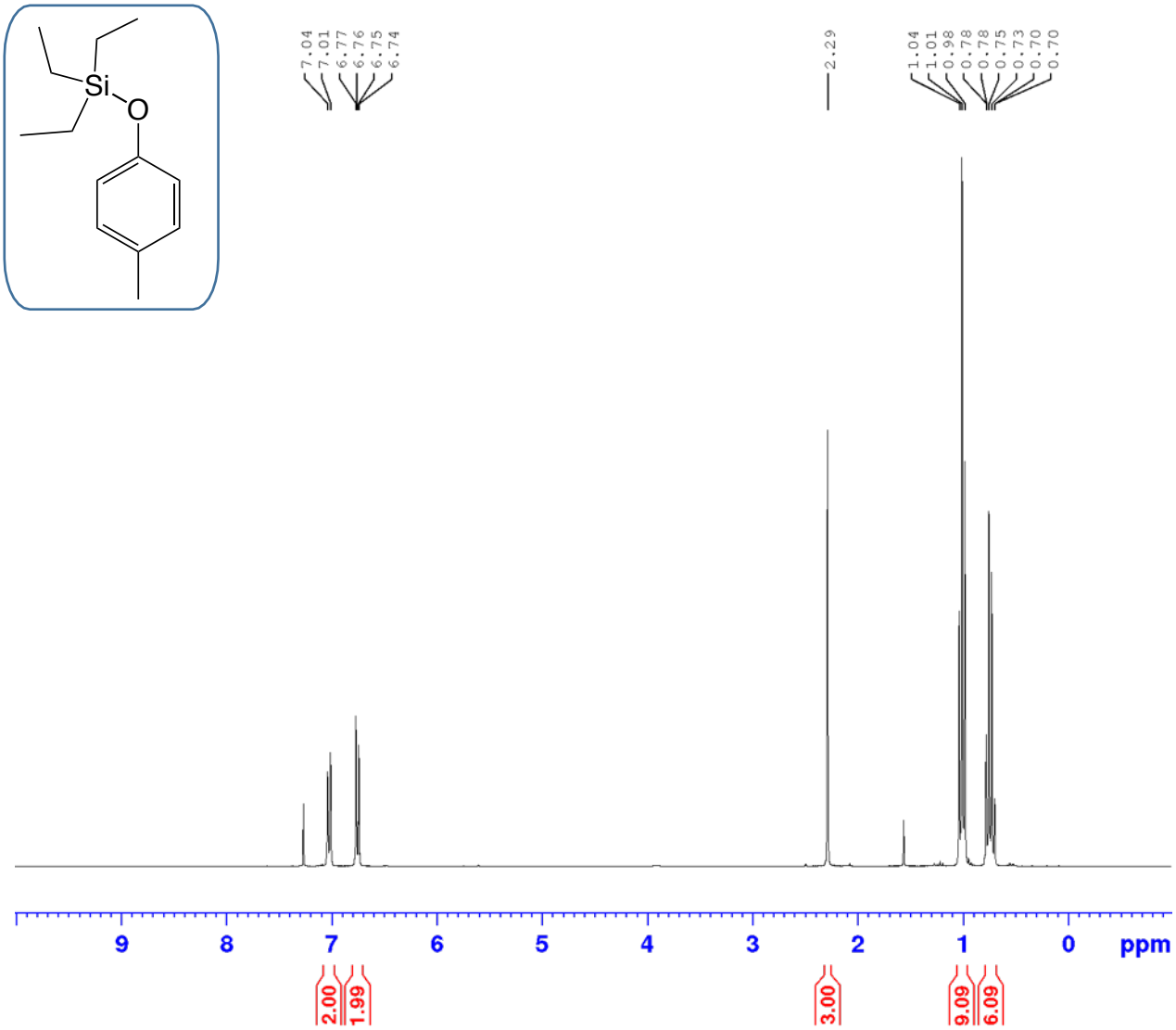

Fig. S8. ^{1}H NMR spectrum of product **2c** in $CDCl_3$

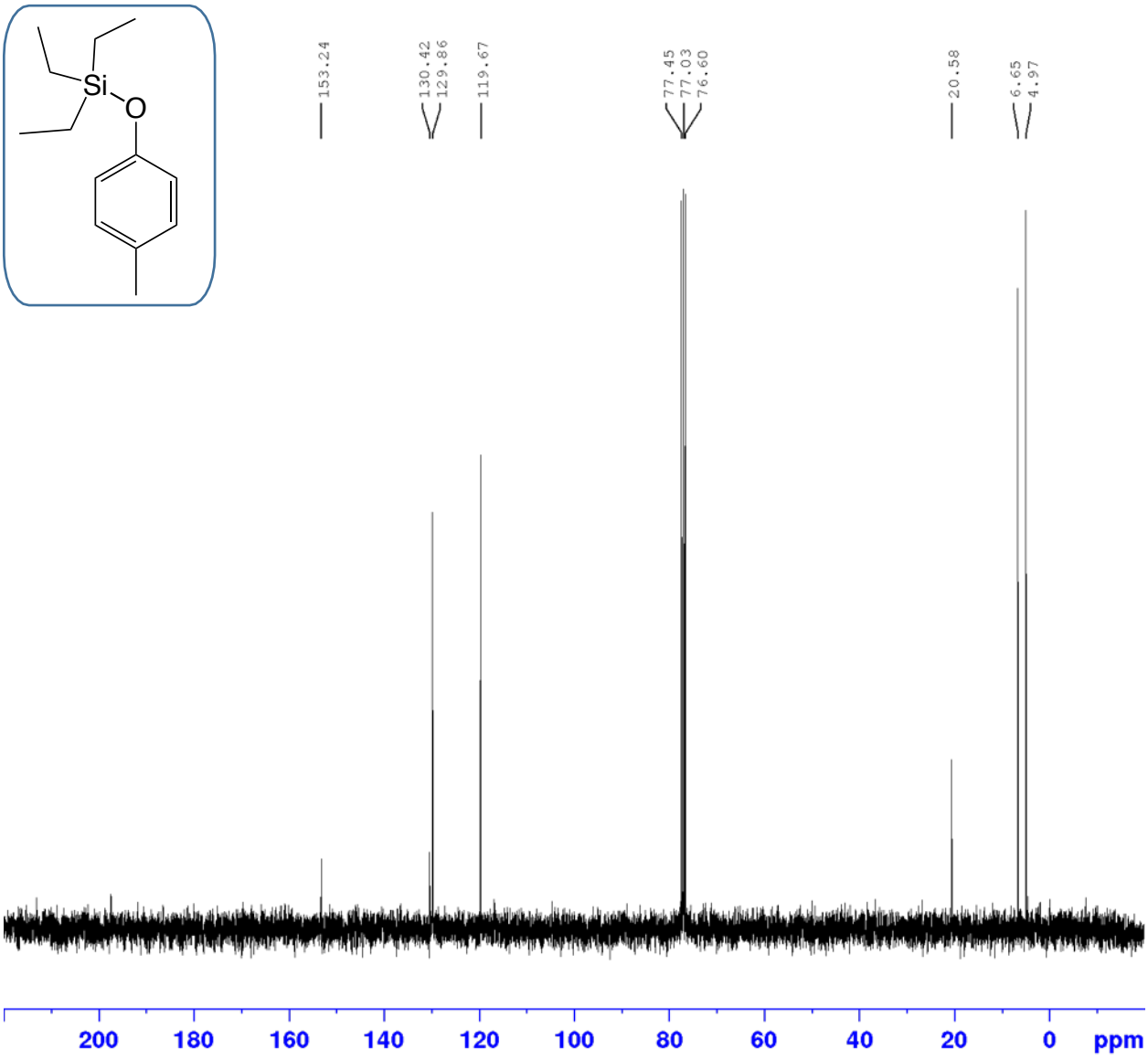

Fig. S9. ^{13}C NMR spectrum of product **2c** in $CDCl_3$

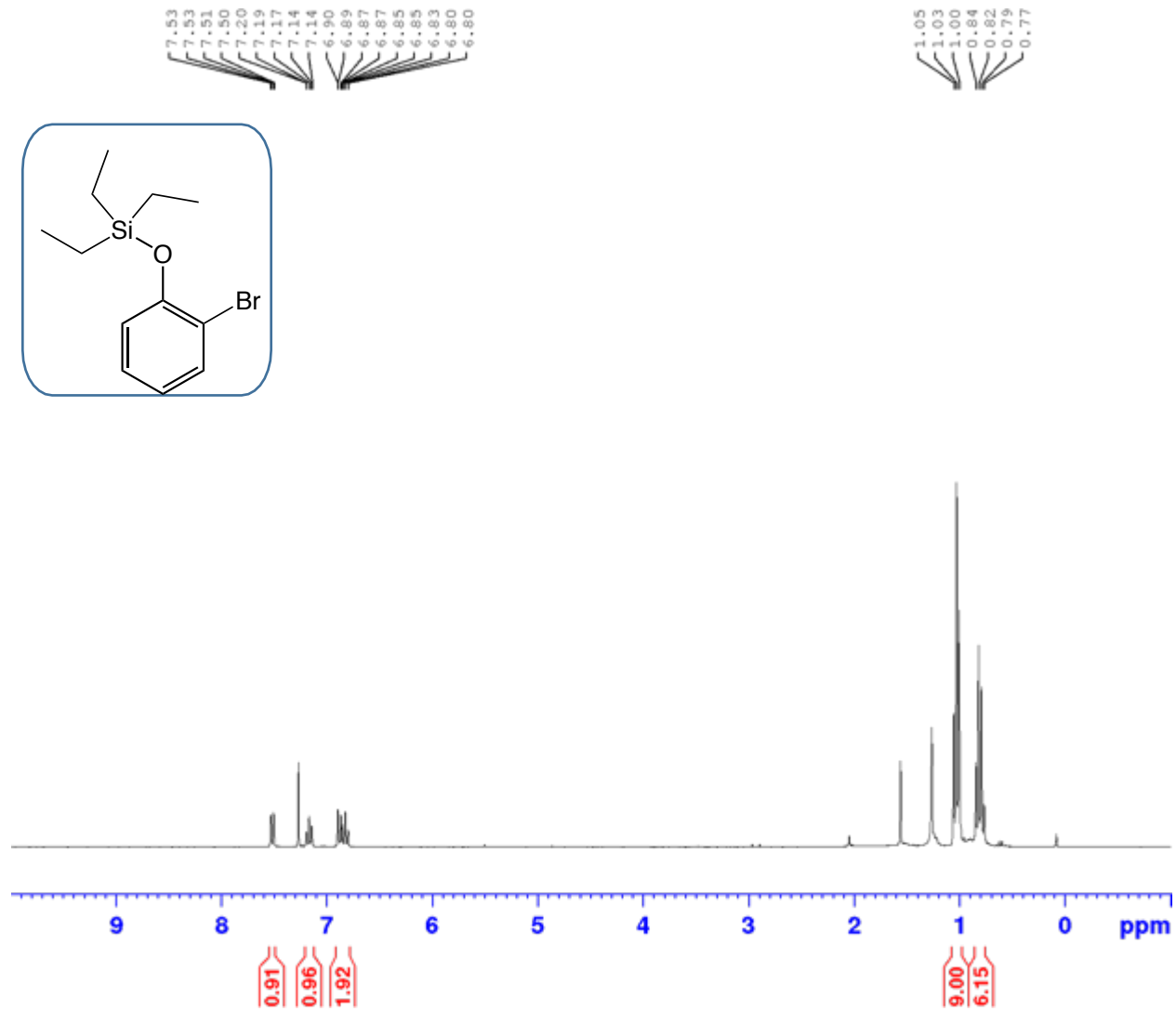

Fig. S10. ^{1}H NMR spectrum of product **3a** in $CDCl_3$

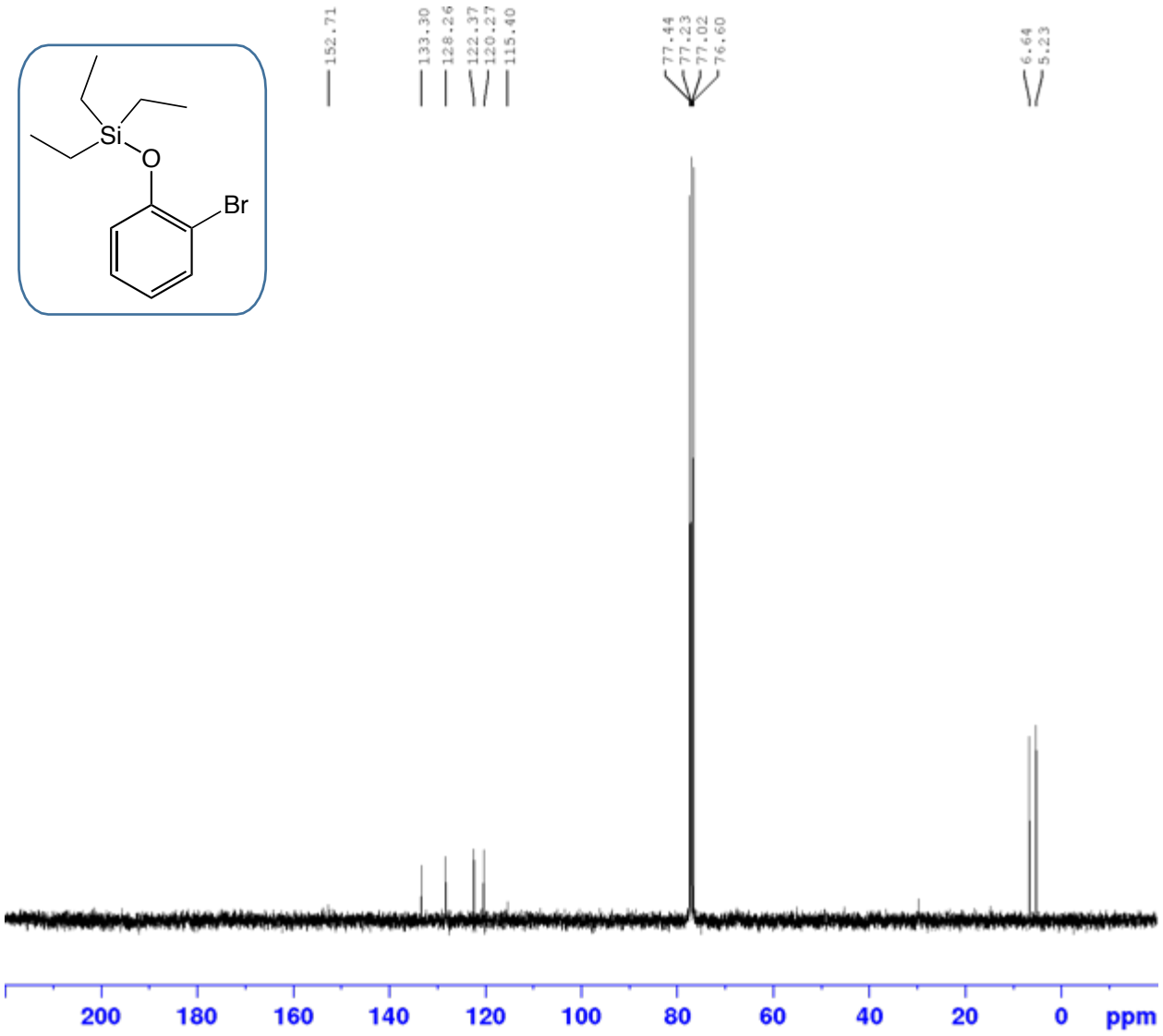

Fig. S11. ^{13}C NMR spectrum of product **3a** in $CDCl_3$

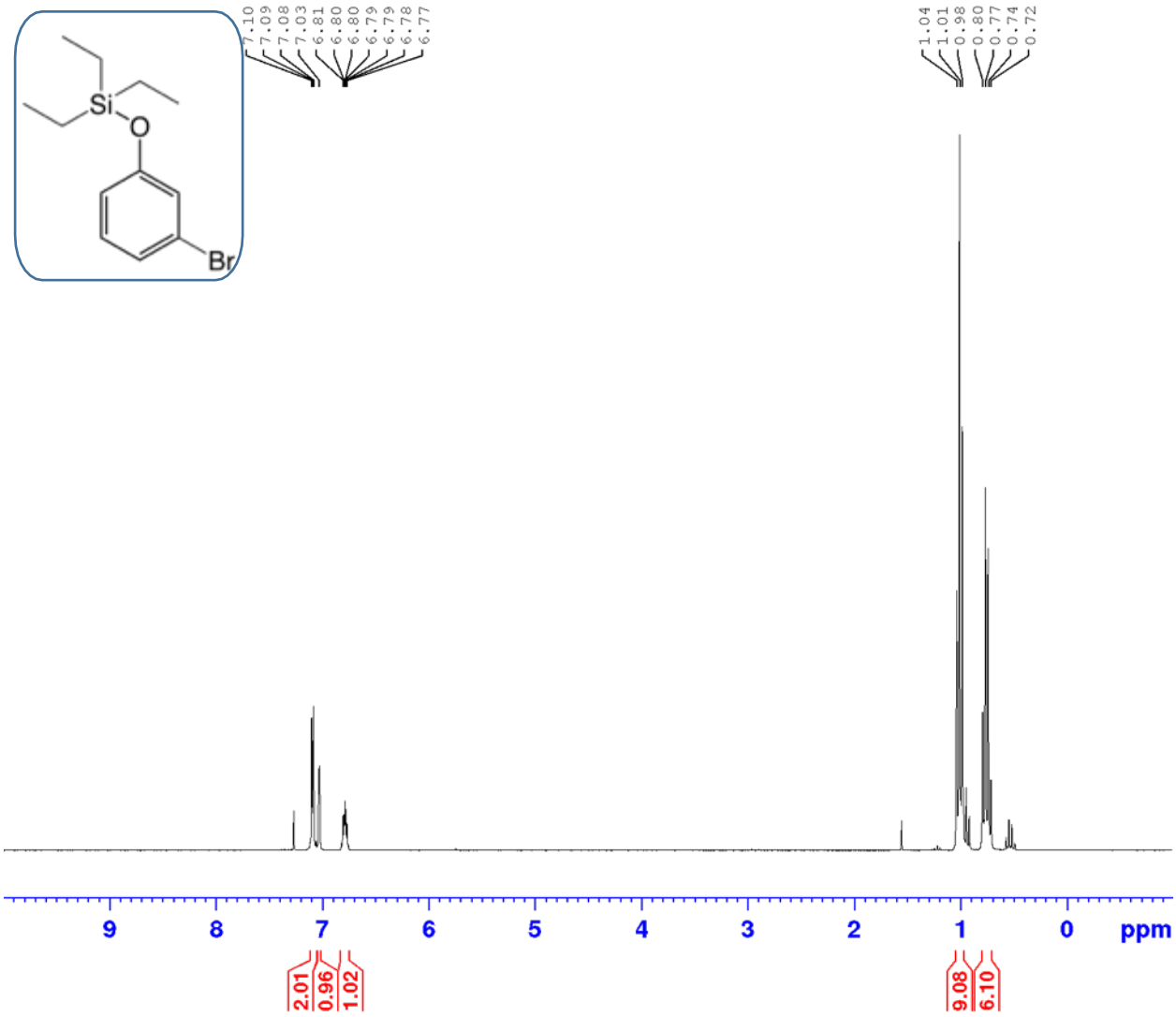

Fig. S12. ^{1}H NMR spectrum of product **3b** in $CDCl_3$

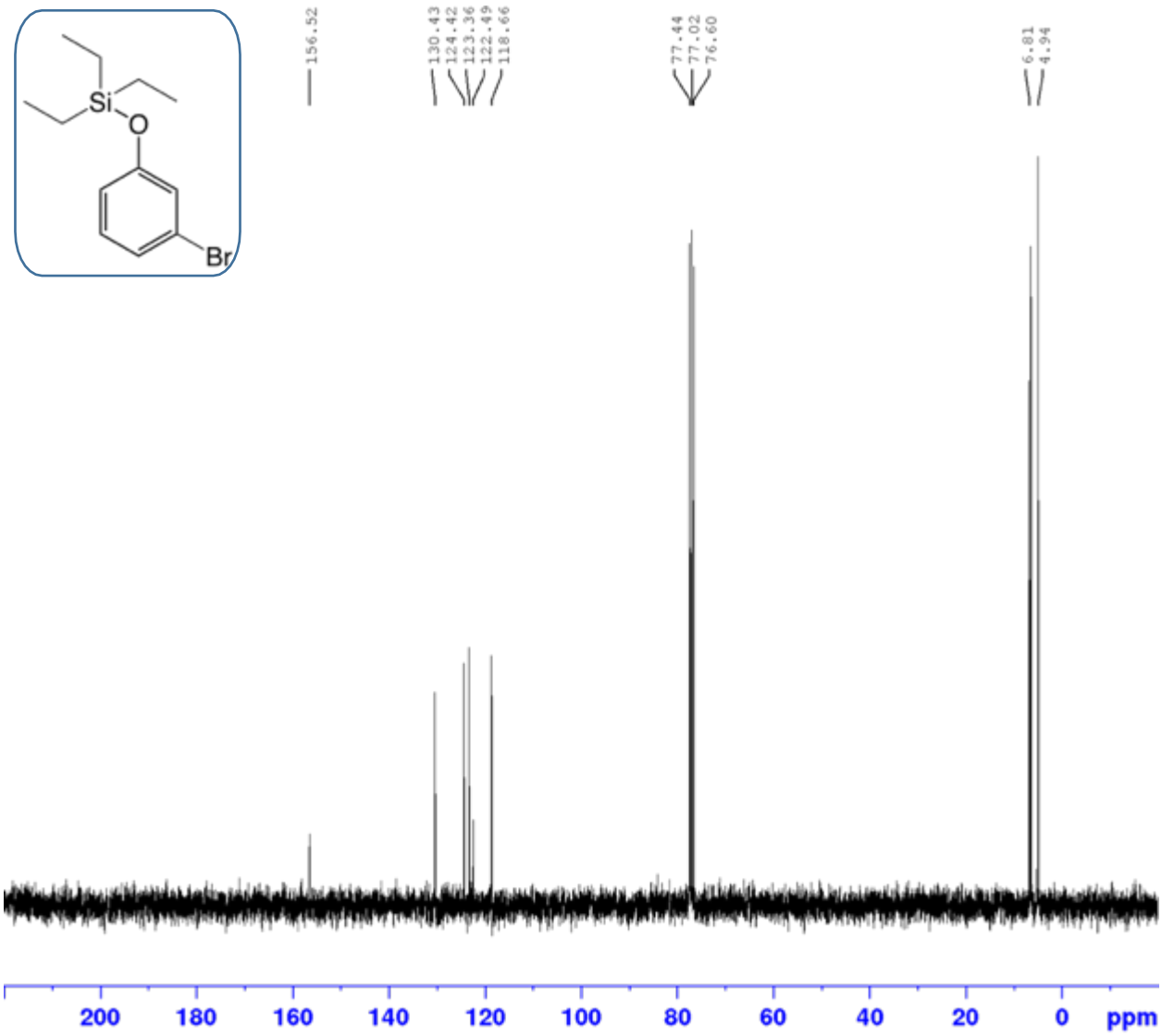

Fig. S13. ^{13}C NMR spectrum of product **3b** in $CDCl_3$

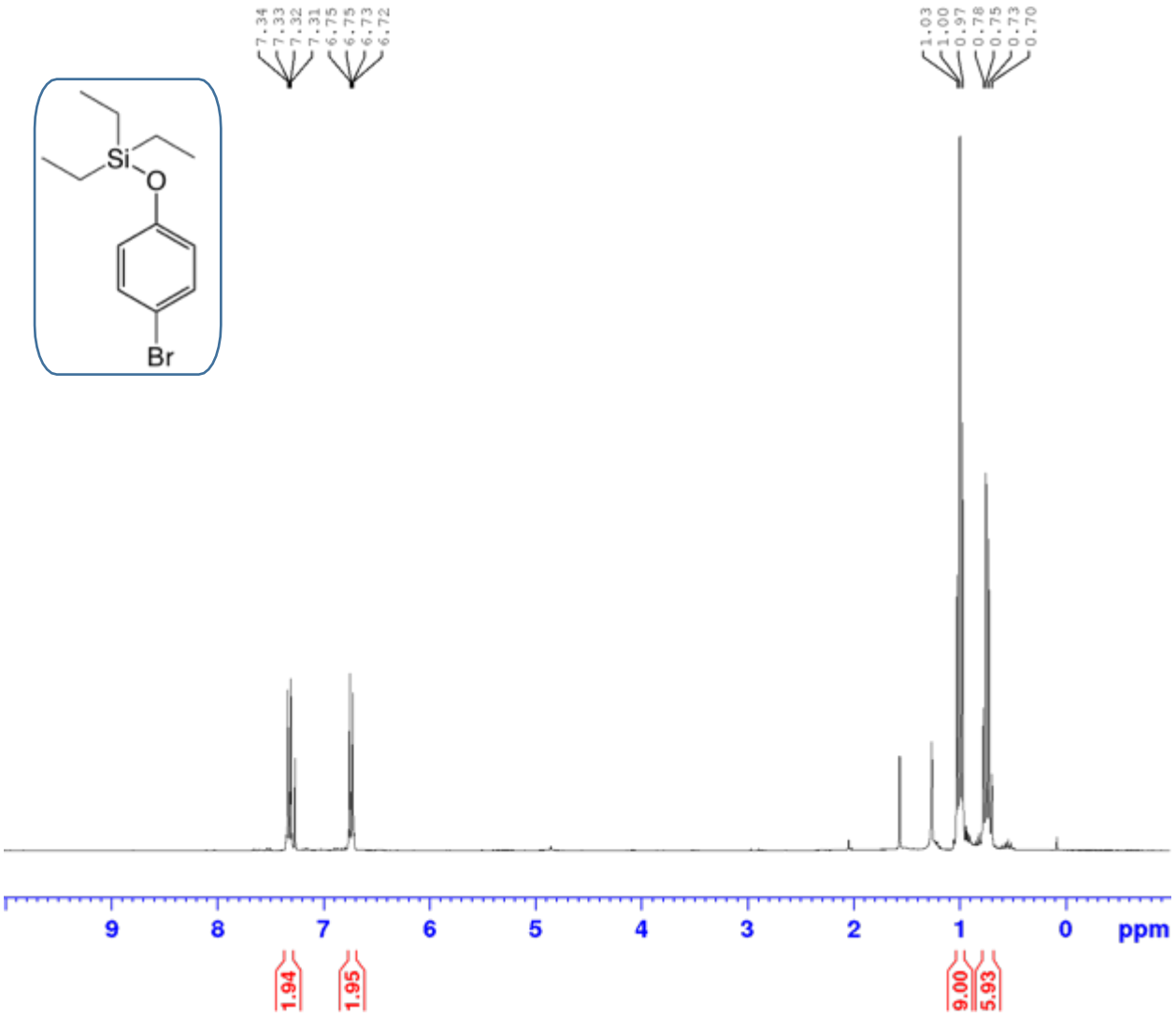

Fig. S14. ^{1}H NMR spectrum of product **3c** in $CDCl_3$

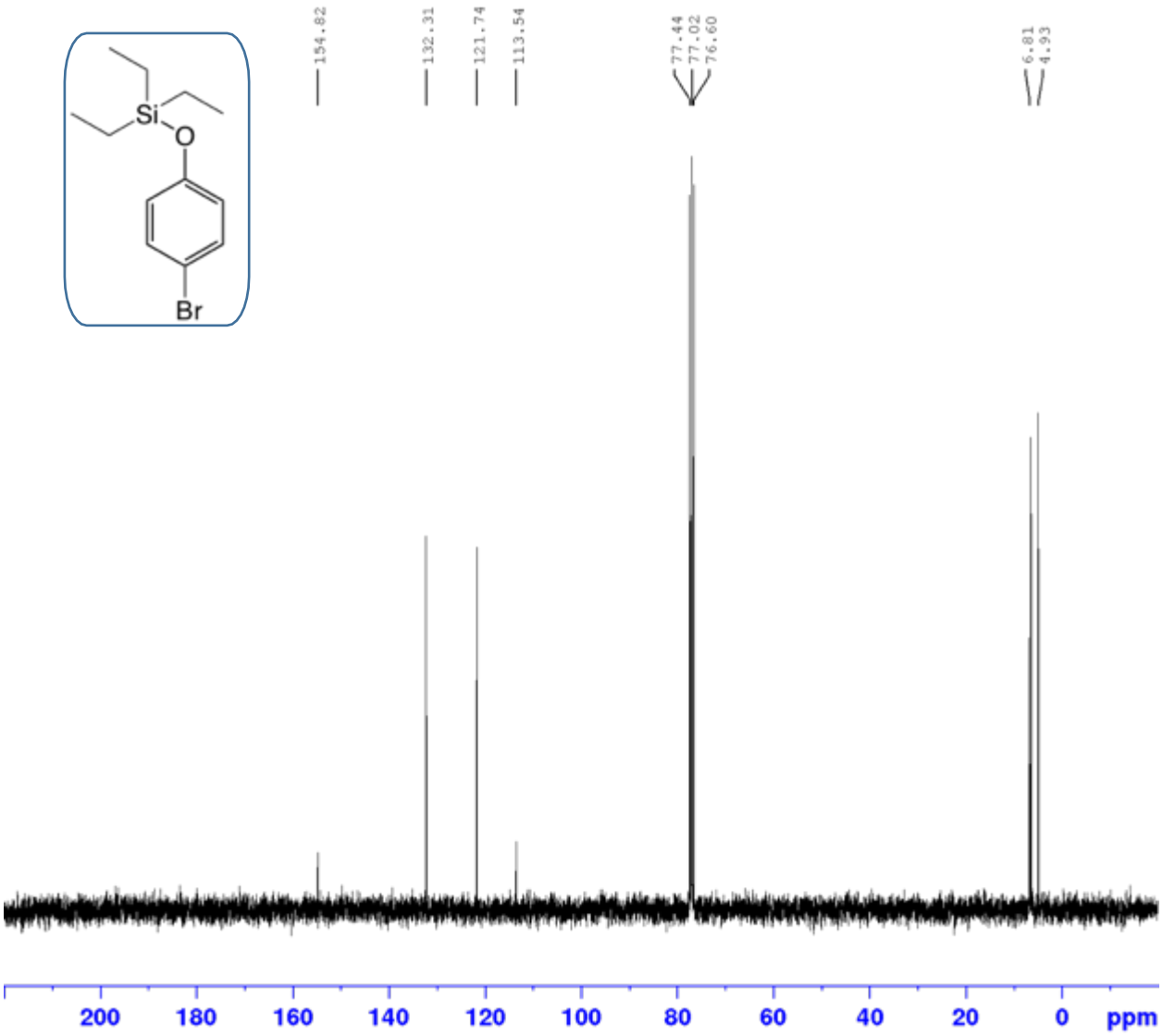

Fig. S15. ^{13}C NMR spectrum of product **3c** in $CDCl_3$

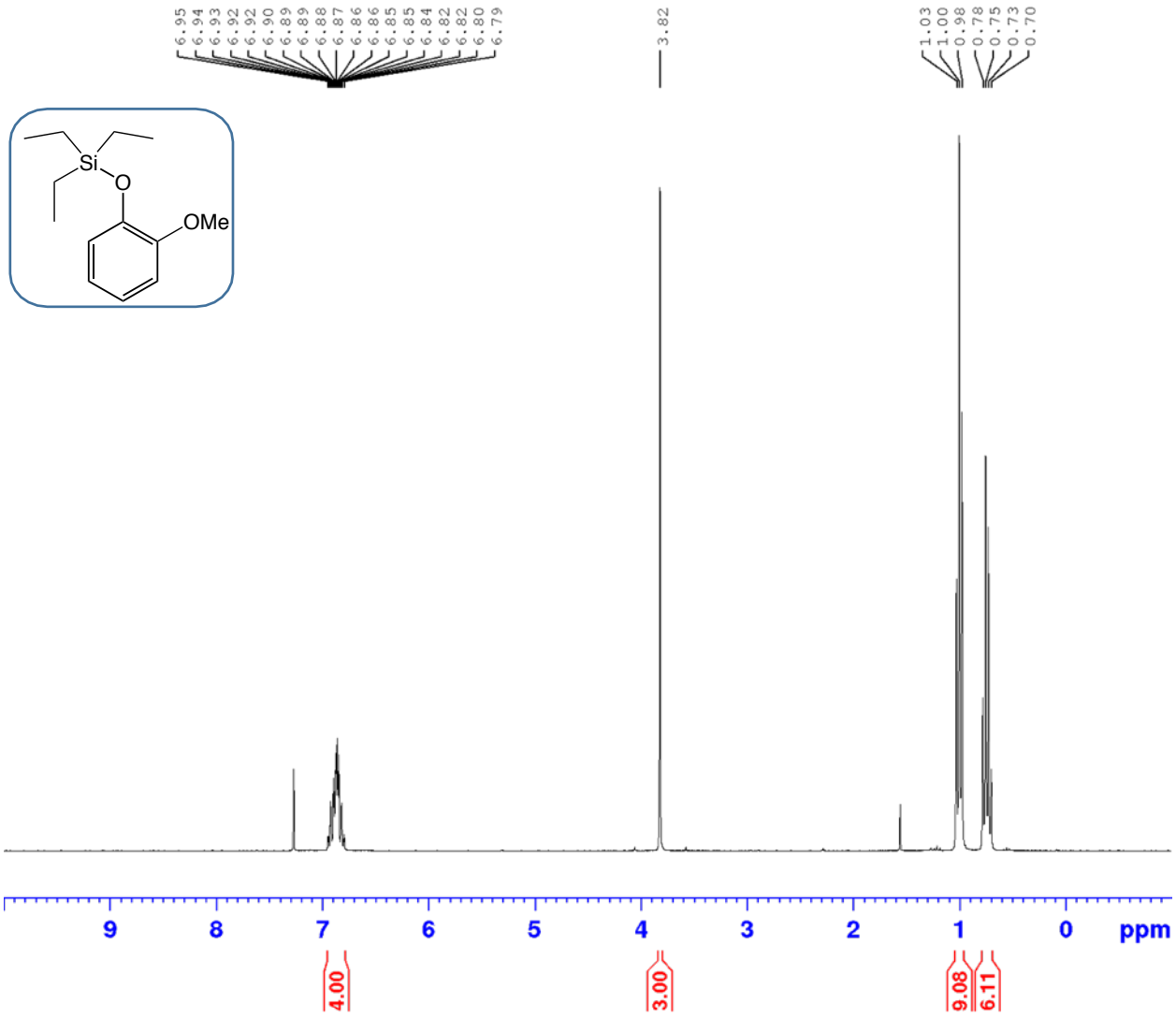

Fig. S16. ^{1}H NMR spectrum of product **4a** in $CDCl_3$

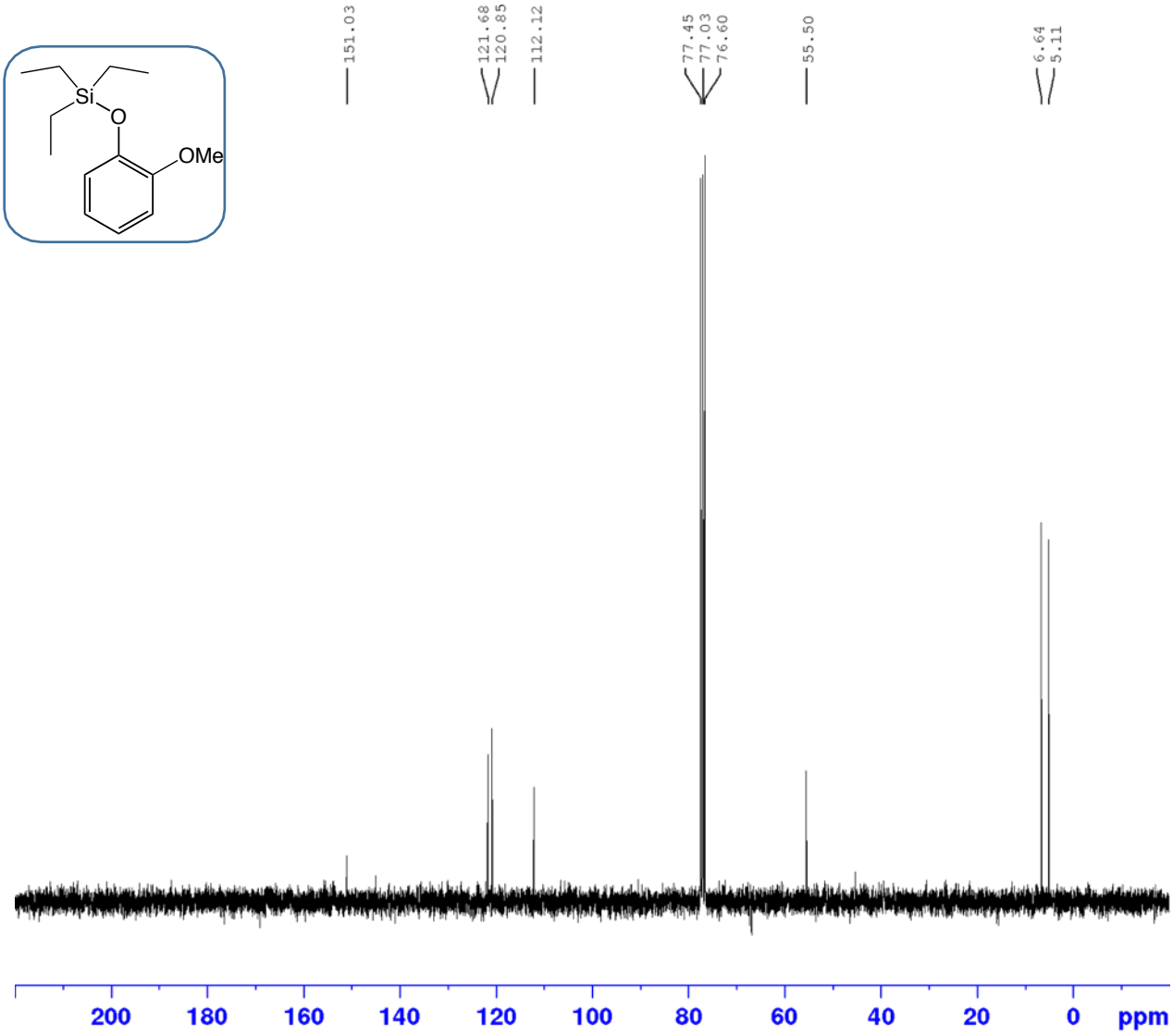

Fig. S17. ^{13}C NMR spectrum of product **4a** in $CDCl_3$

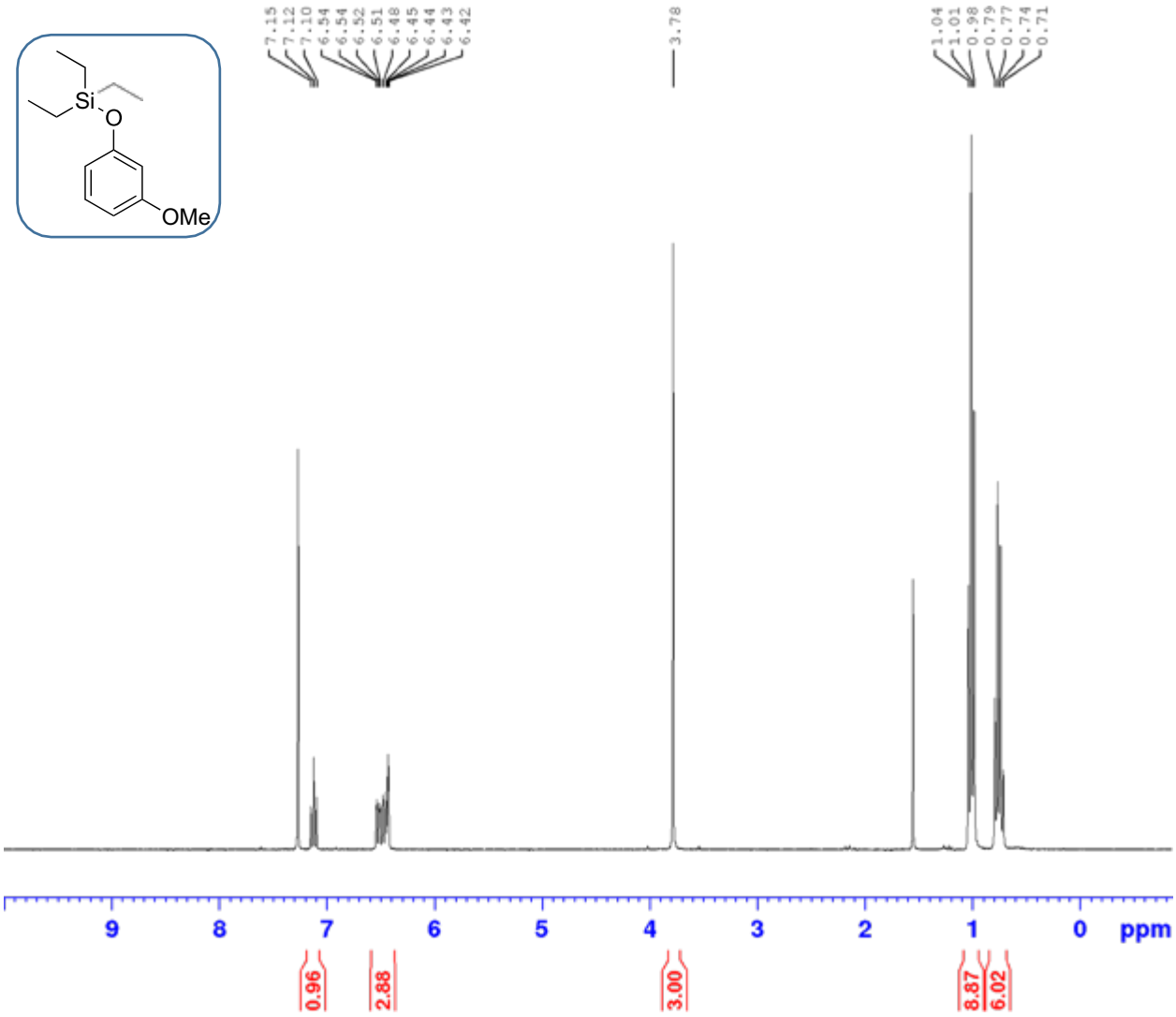

Fig. S18. ^{1}H NMR spectrum of product **4b** in $CDCl_3$

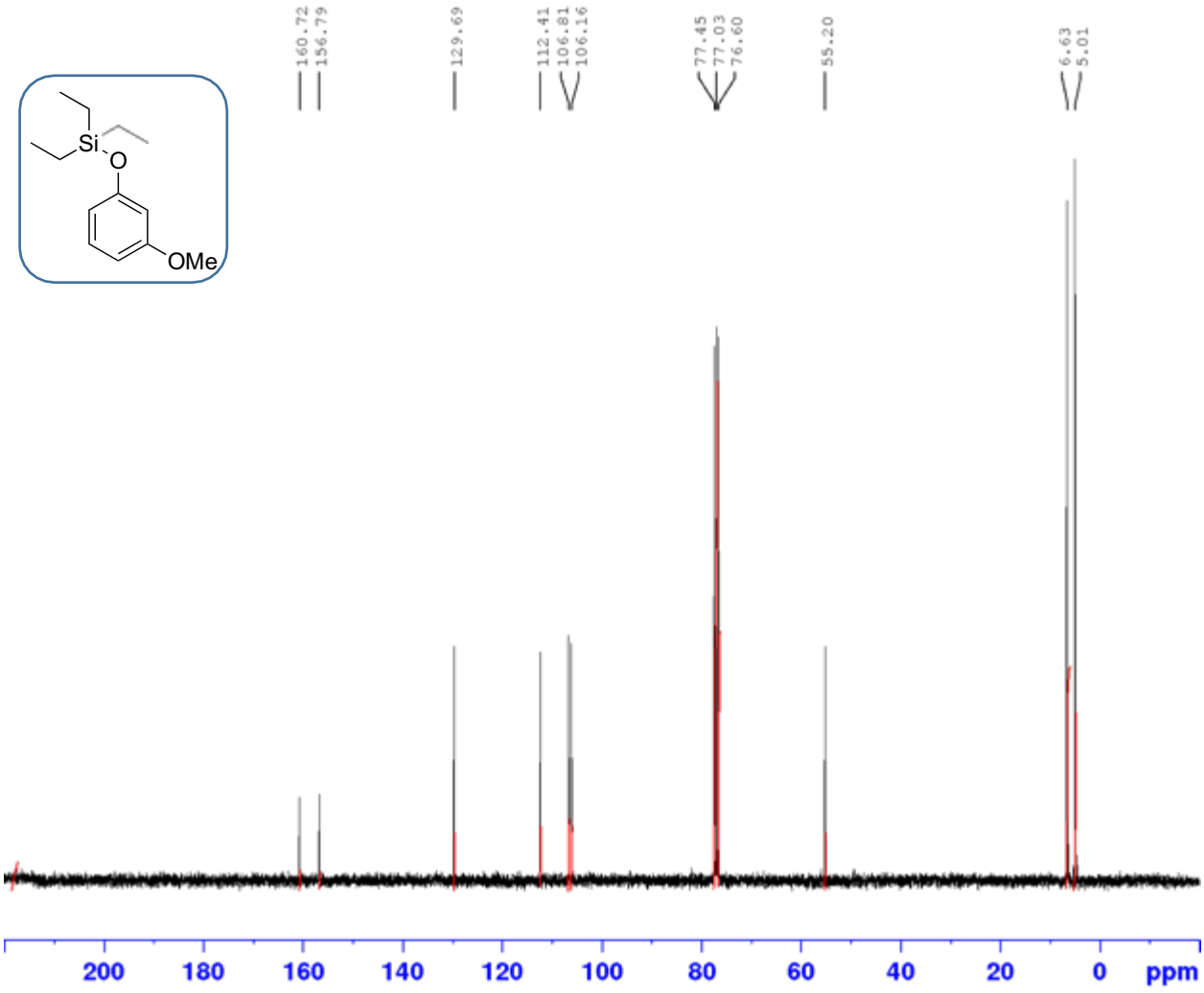

Fig. S19. ^{13}C NMR spectrum of product **4b** in $CDCl_3$

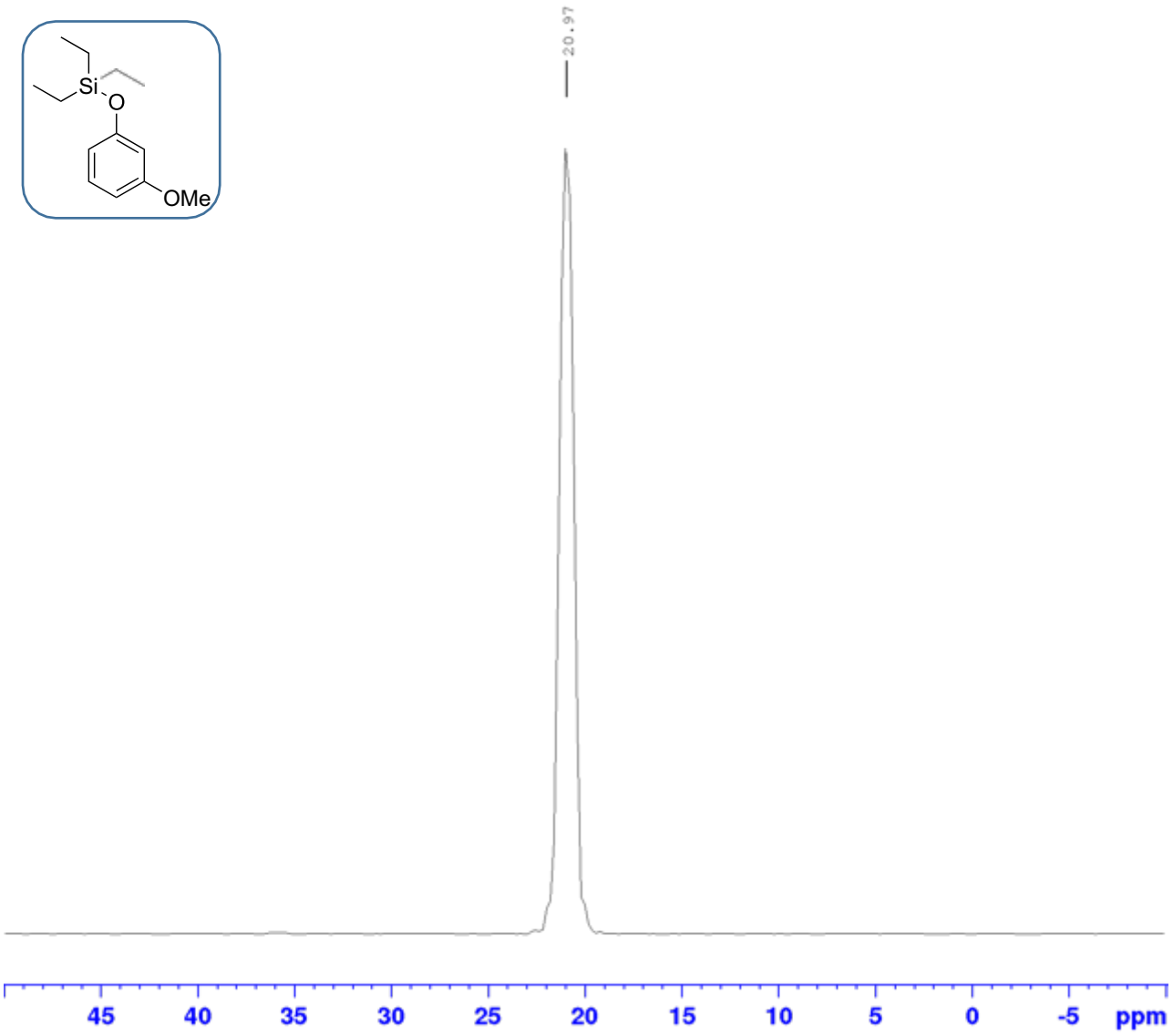

Fig. S20. ^{29}Si NMR spectrum of product **4b** in $CDCl_3$

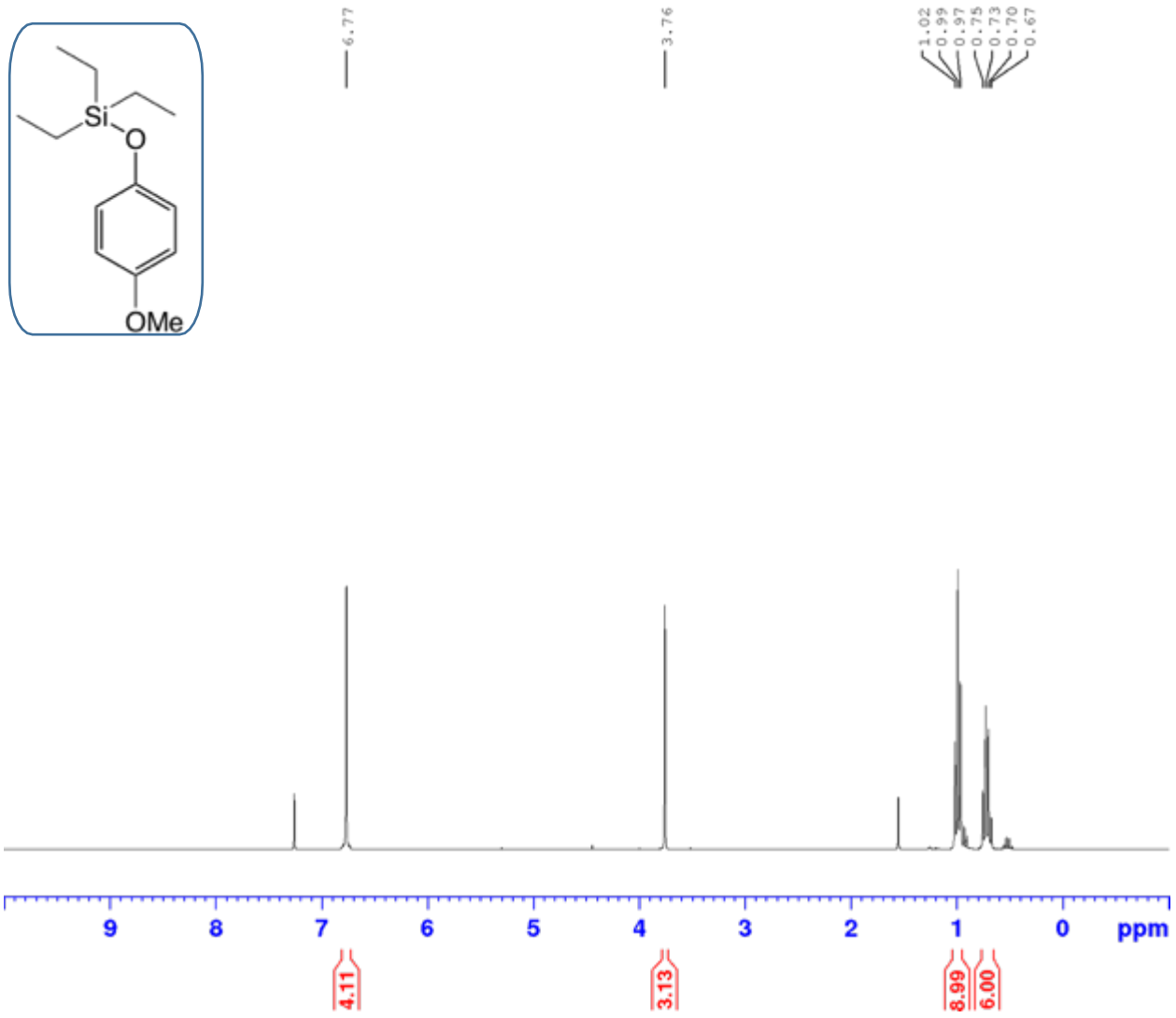

Fig. S21. ^{1}H NMR spectrum of product **4c** in $CDCl_3$

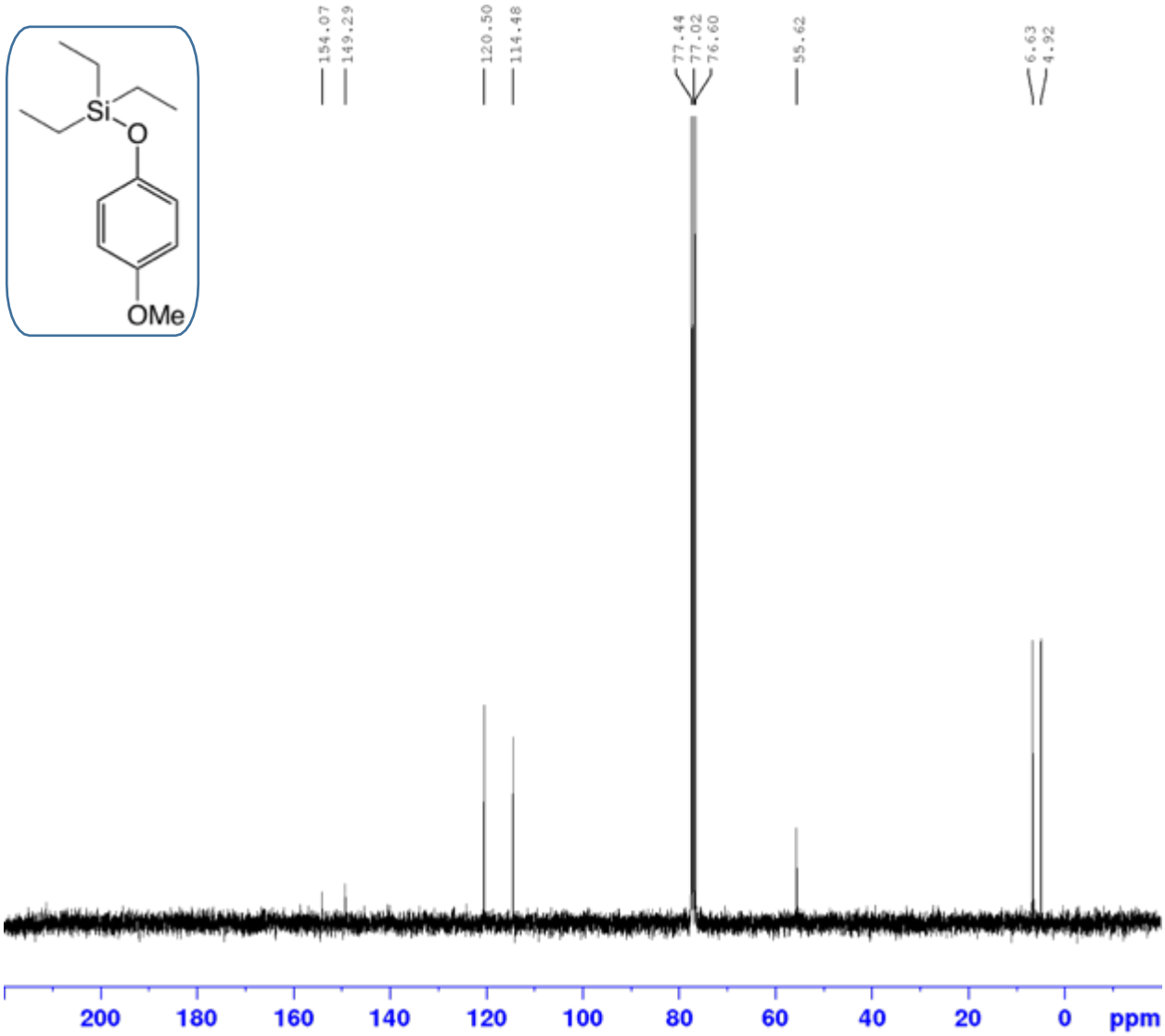

Fig. S22. ^{13}C NMR spectrum of product **4c** in $CDCl_3$

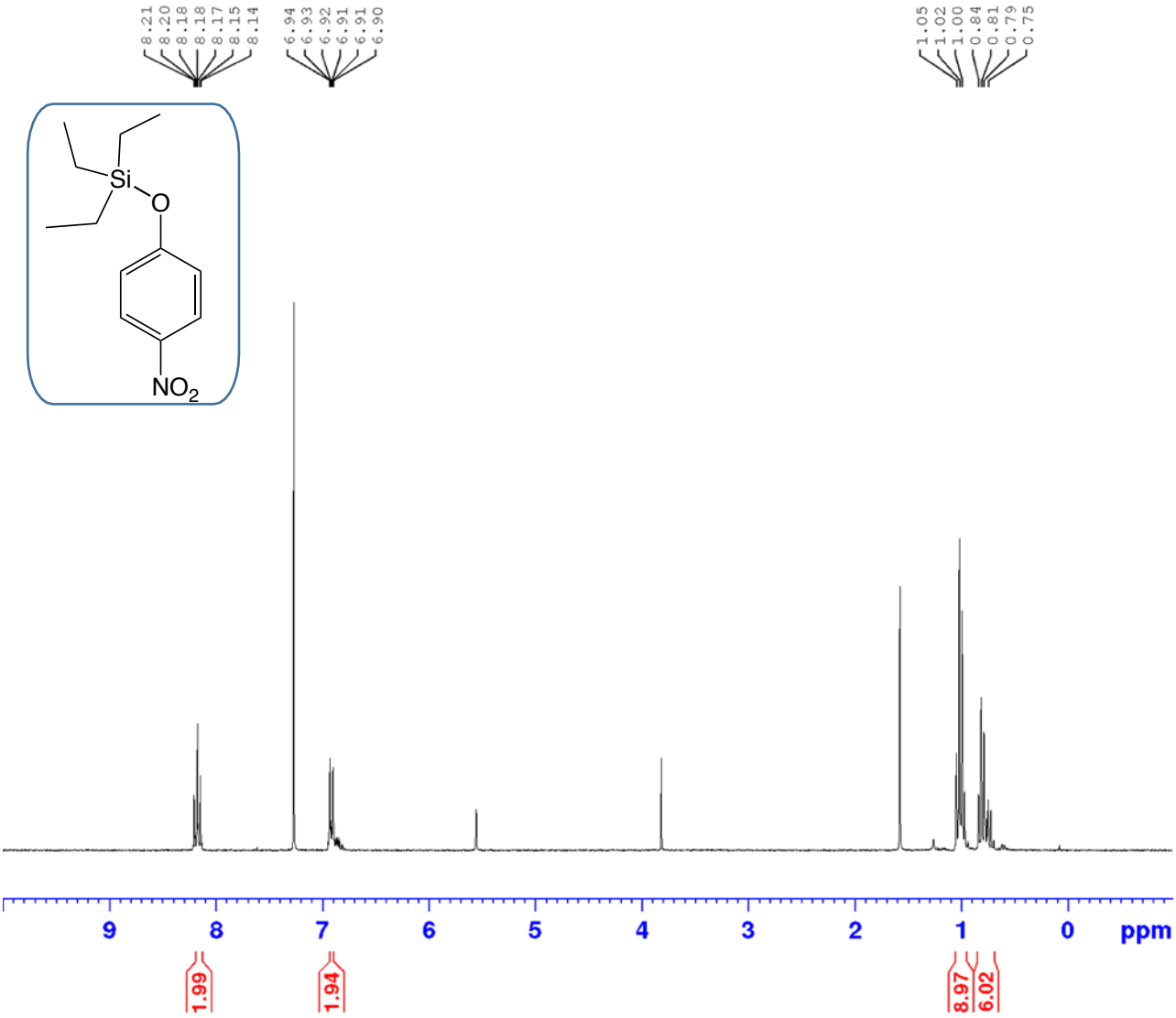

Fig. S23. ^{1}H NMR spectrum of product **5** in $CDCl_3$

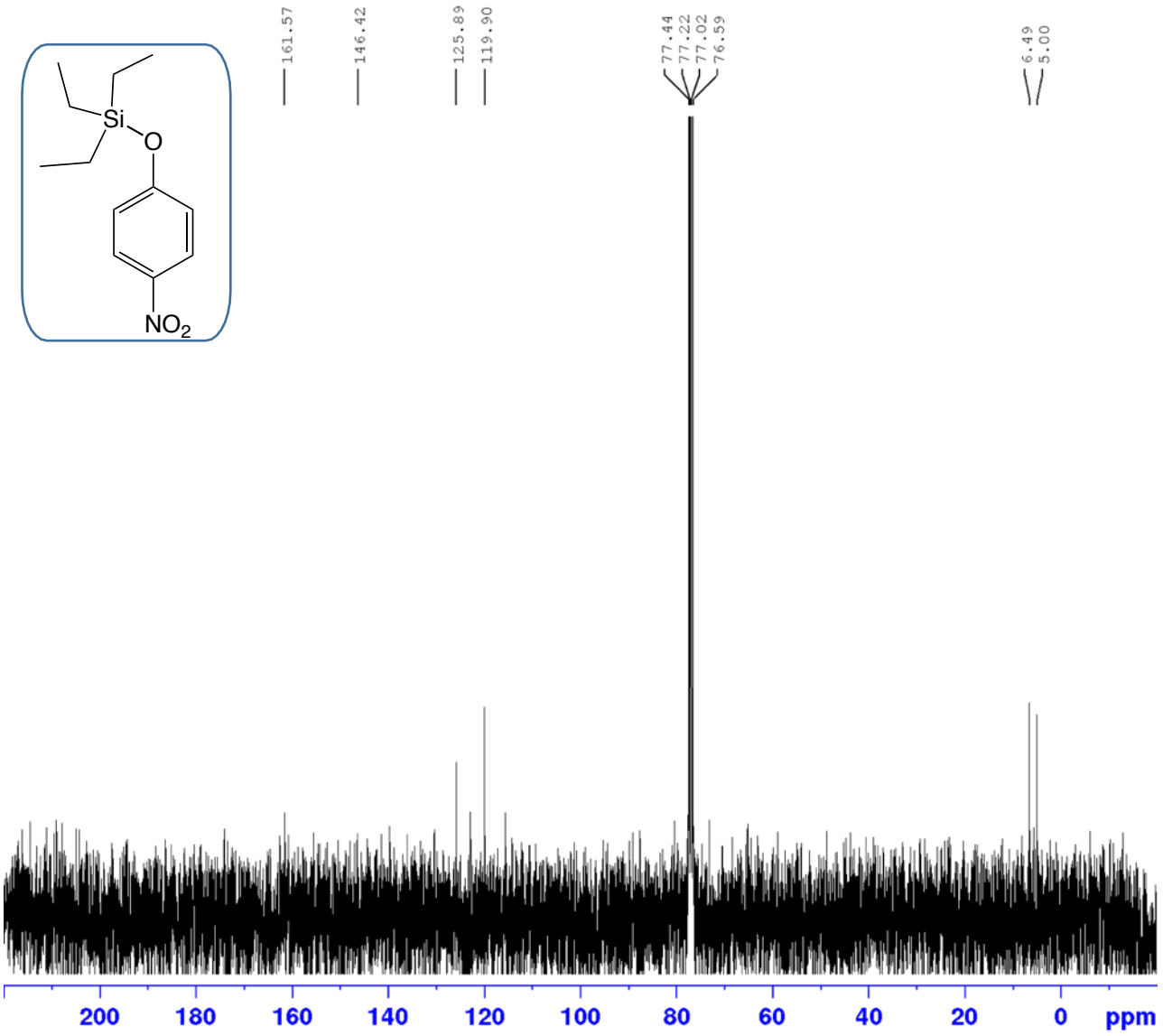

Fig. S24. ^{13}C NMR spectrum of product **5** in $CDCl_3$

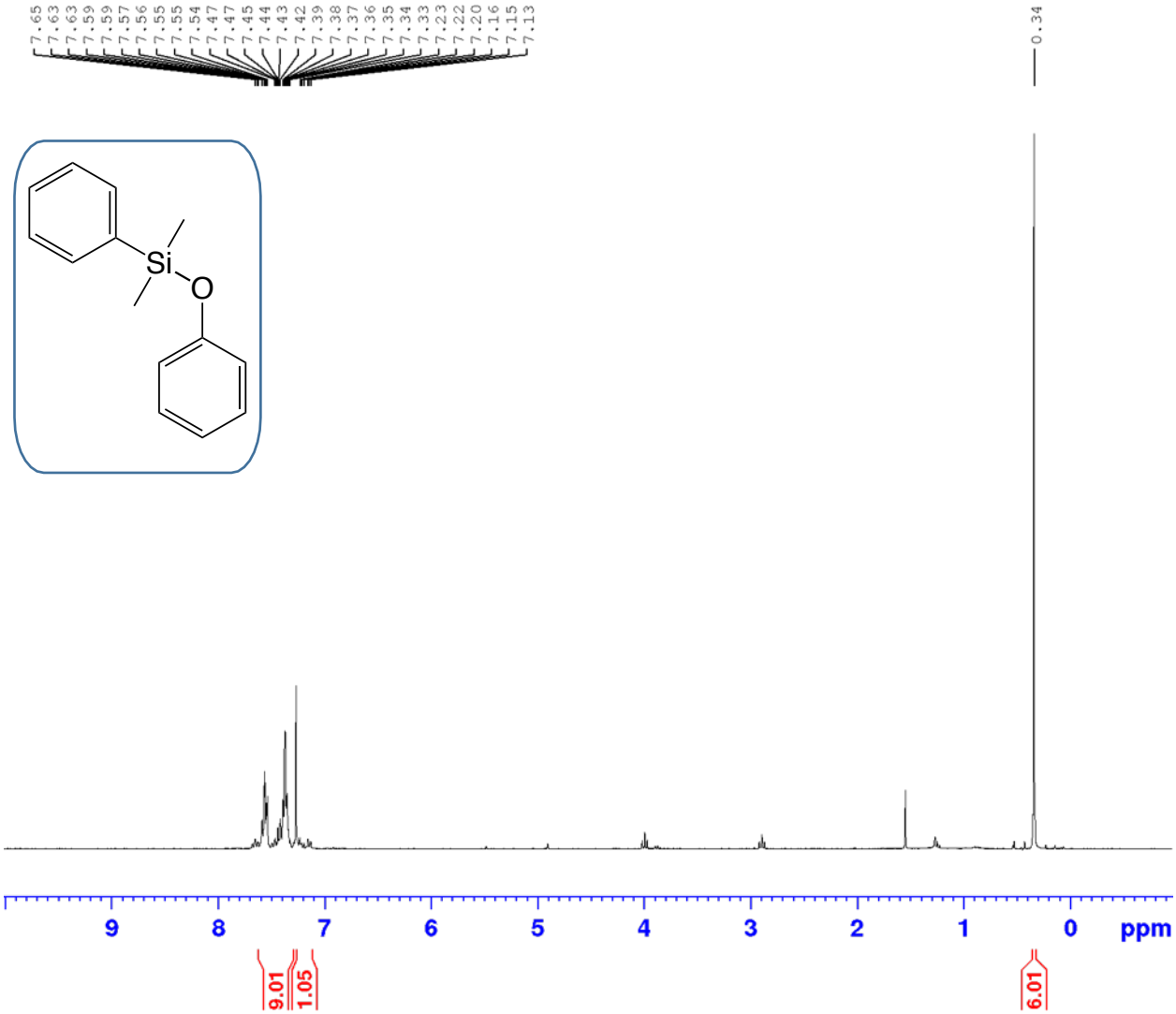

Fig. S25. ^{1}H spectrum of product **6** in $CDCl_3$

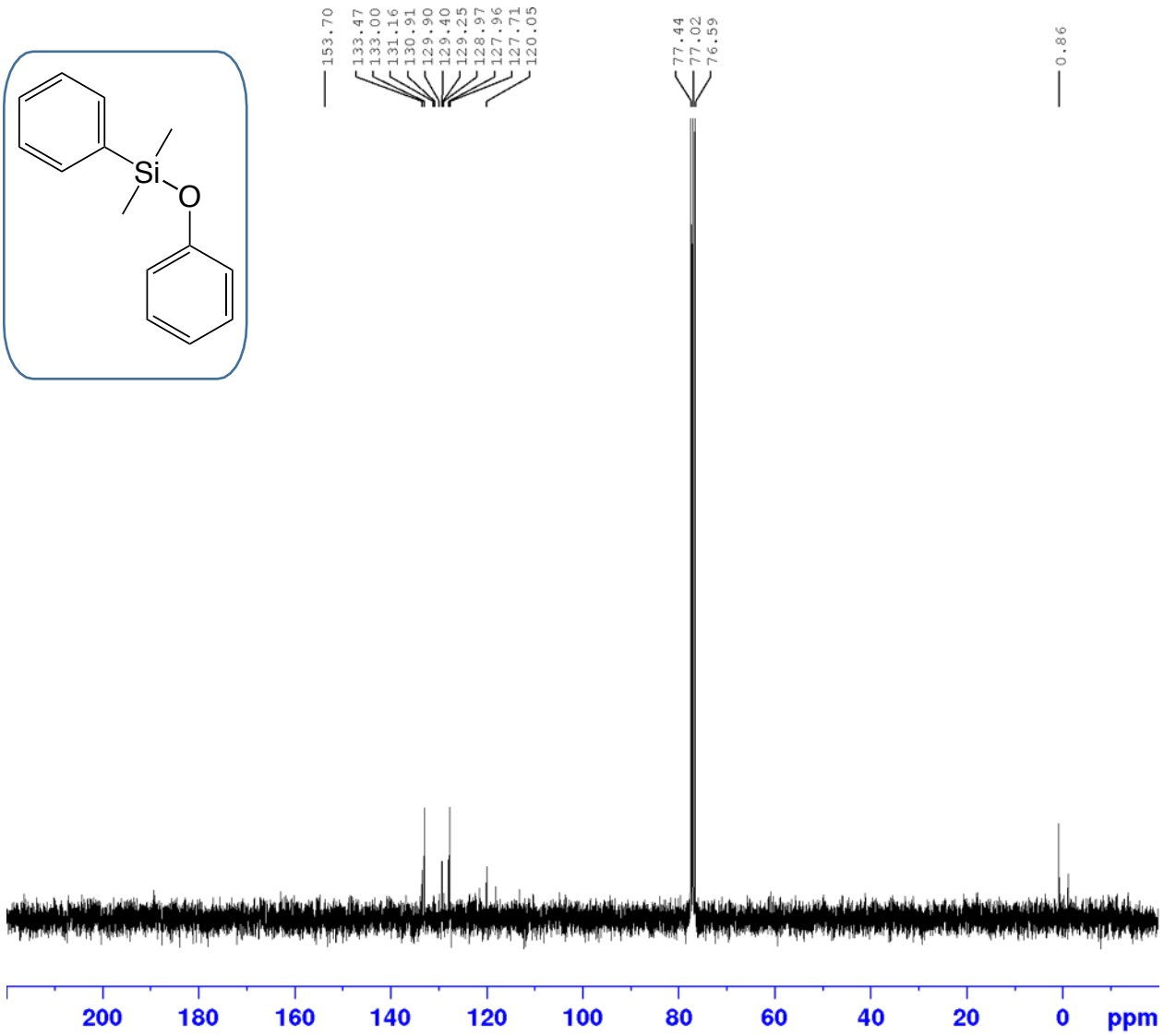

Fig. S26. ^{13}C NMR spectrum of product **6** in $CDCl_3$

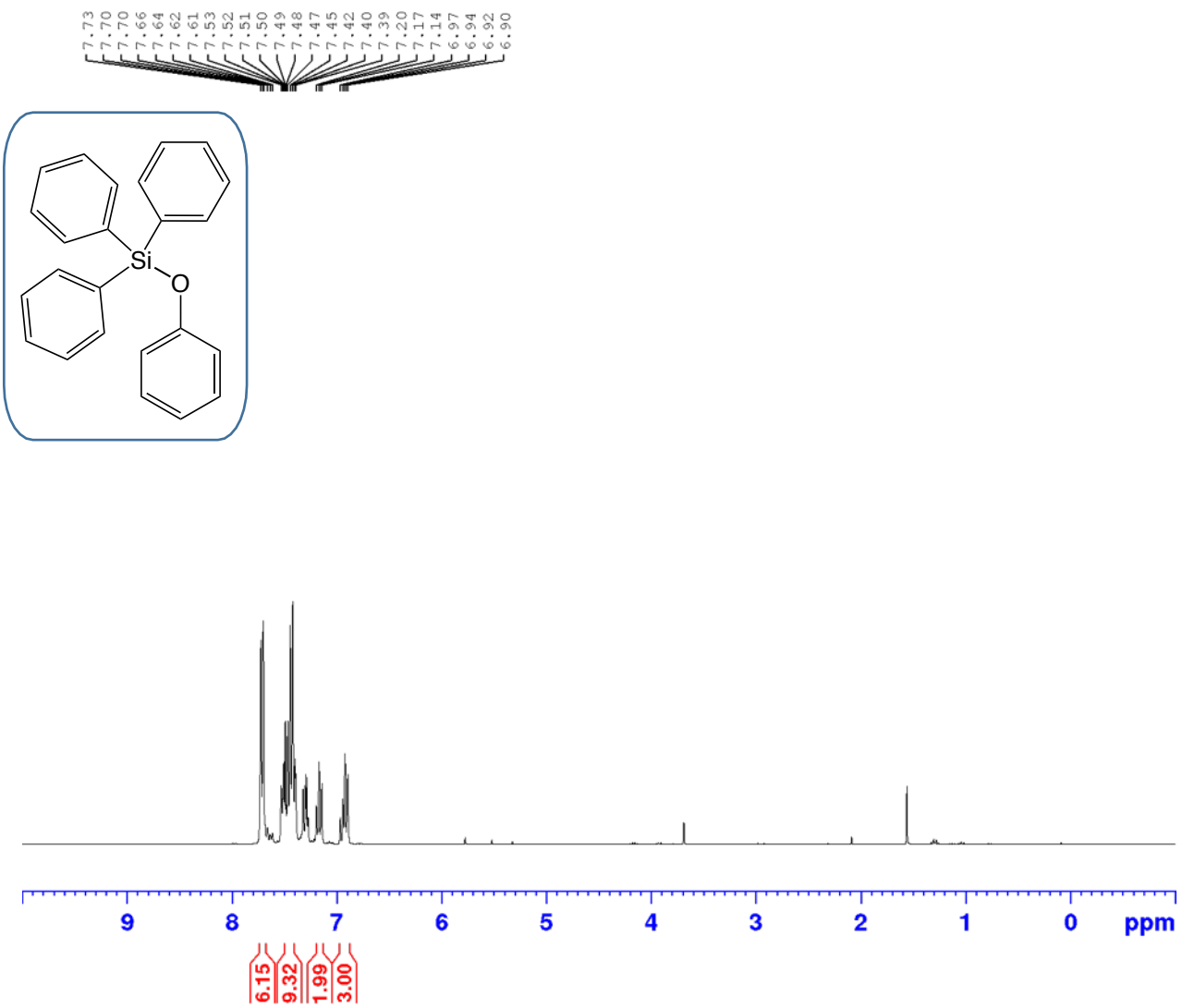

Fig. S27. ^{1}H NMR spectrum of product **7** in $CDCl_3$

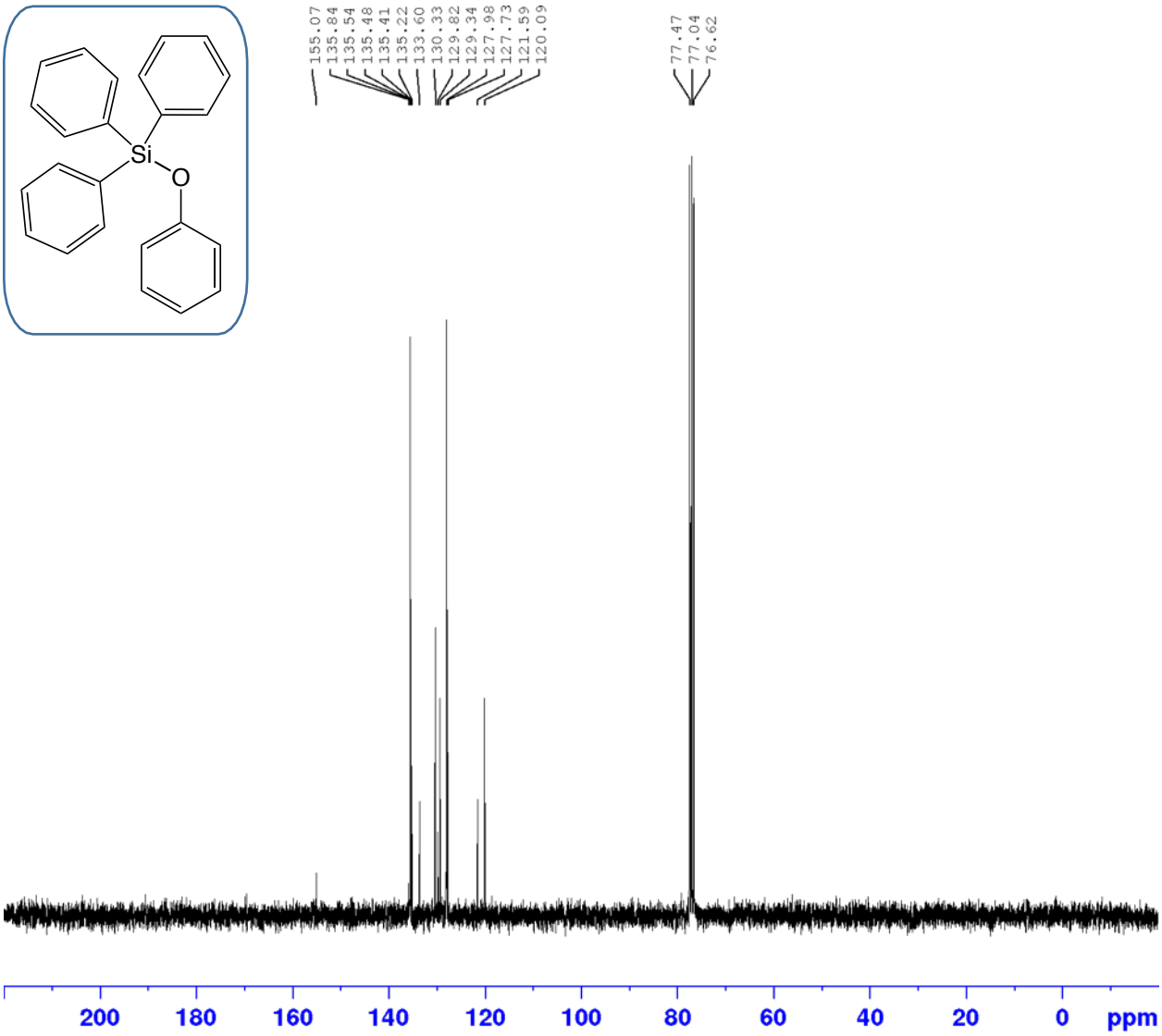

Fig. S28. ^{13}C NMR spectrum of product **7** in $CDCl_3$

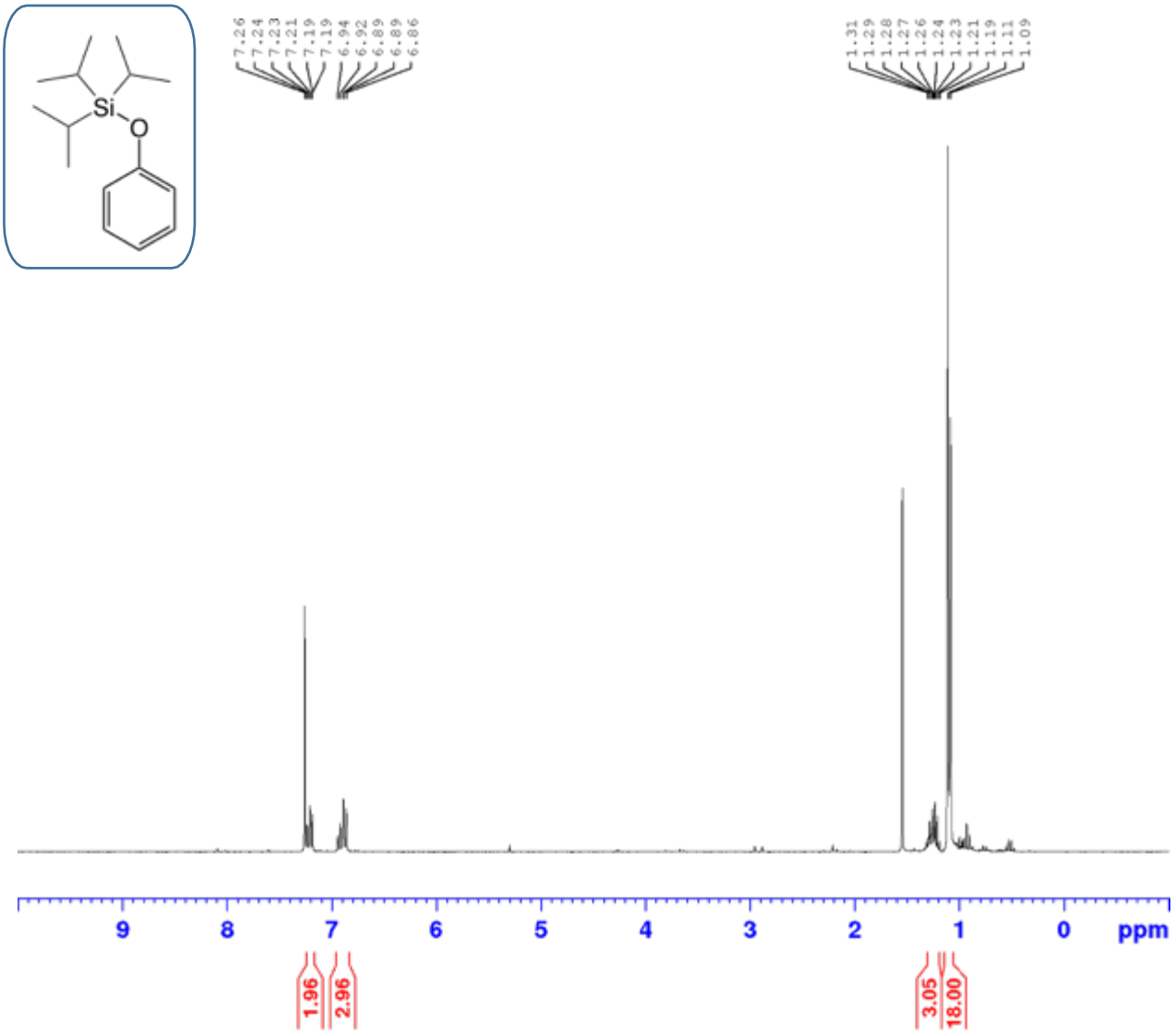

Fig. S29. ^{1}H NMR spectrum of product **8** in $CDCl_3$

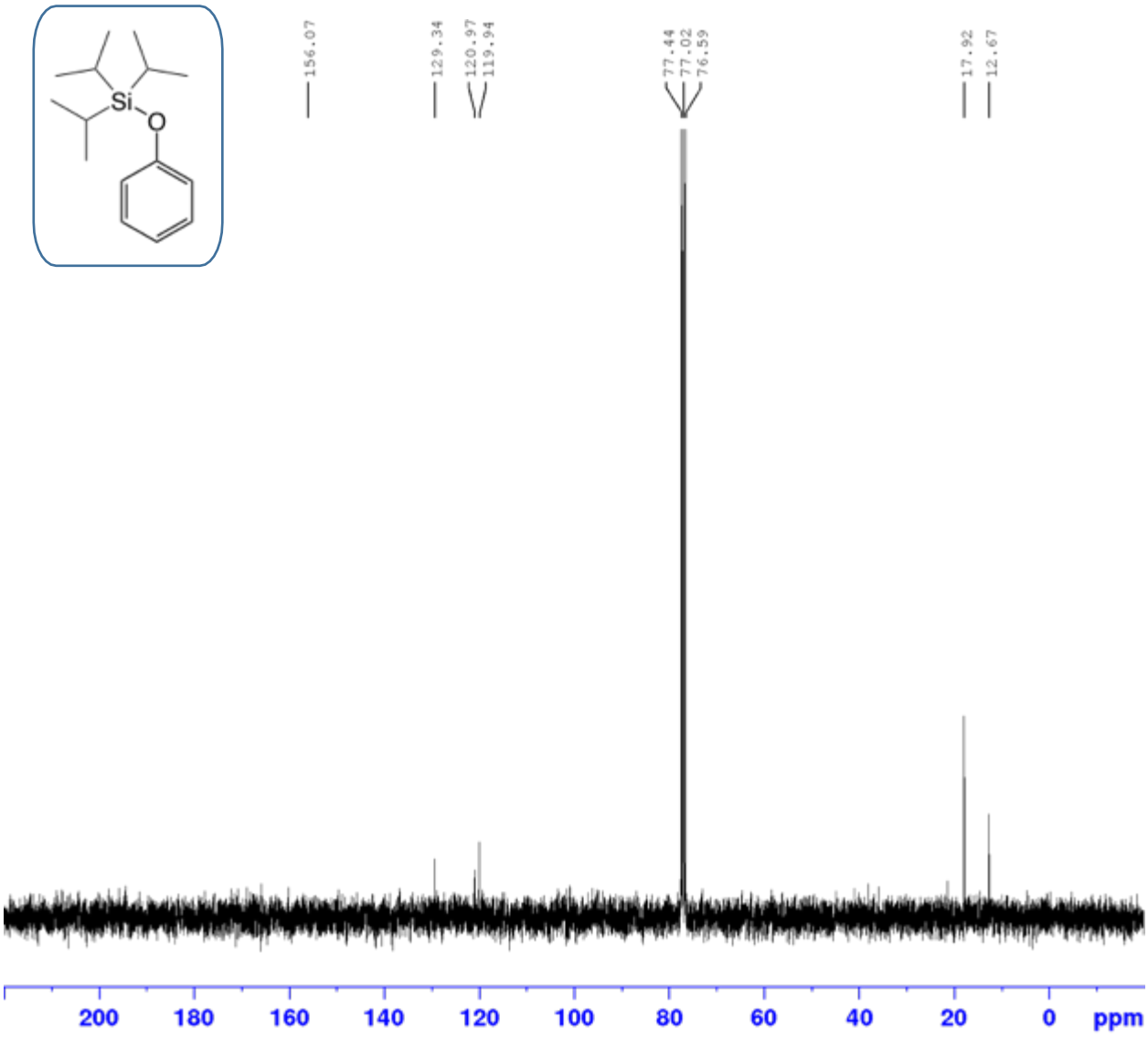

Fig. S30. ^{13}C NMR spectrum of product **8** in $CDCl_3$

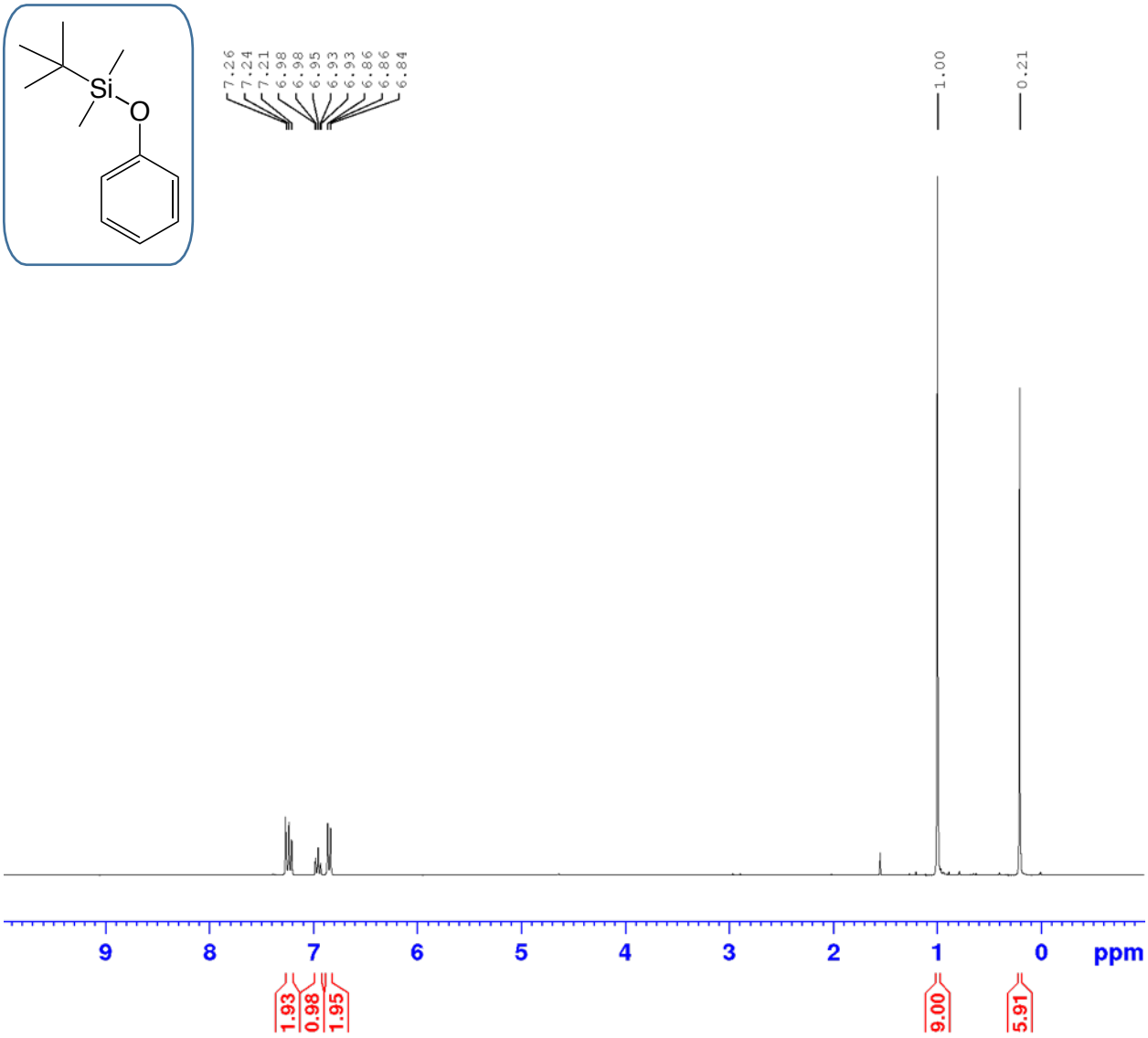

Fig. S31. ^{1}H NMR spectrum of product **9** in $CDCl_3$

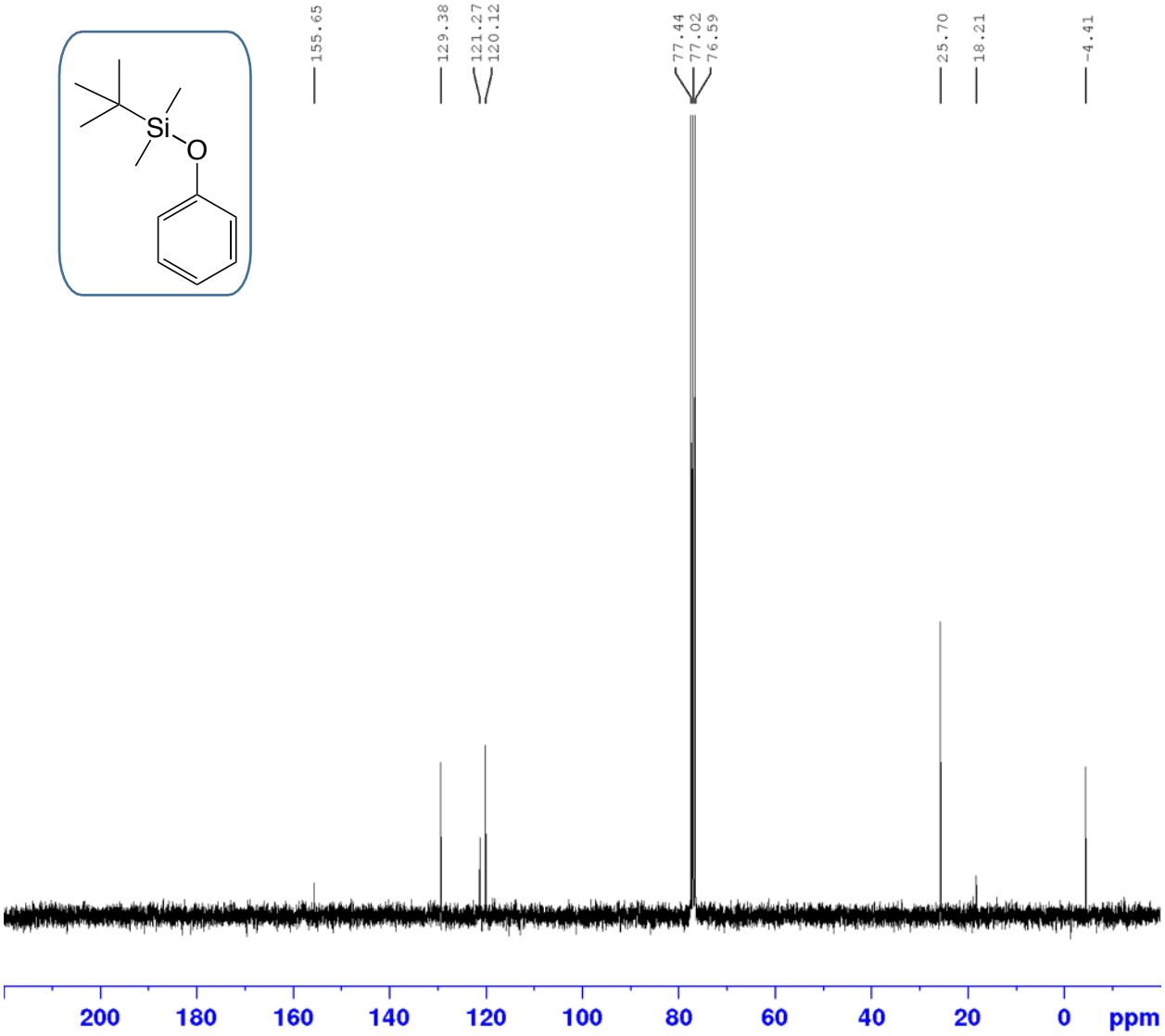

Fig. S32. ^{13}C NMR spectrum of product **9** in $CDCl_3$

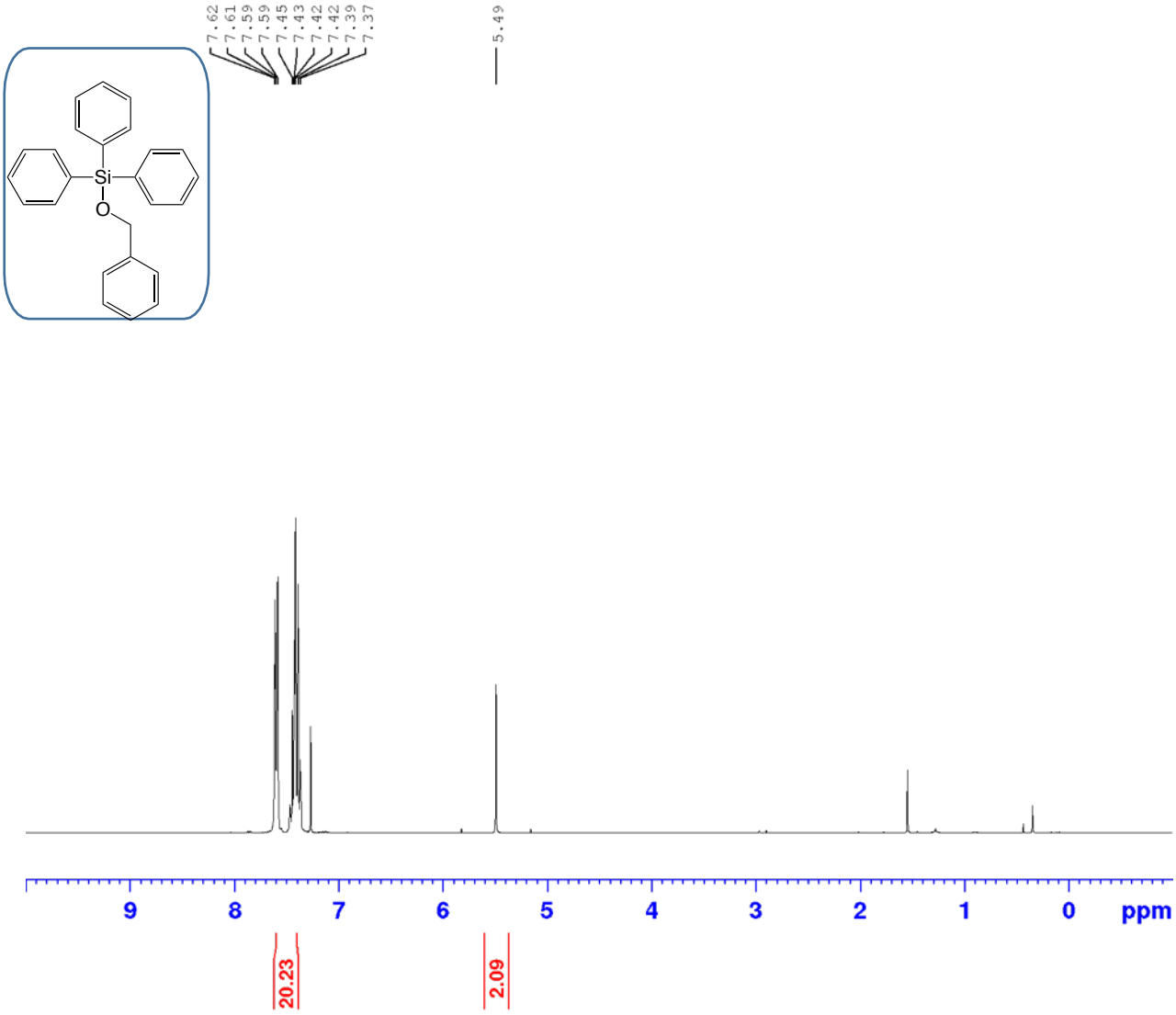

Fig. S33. ^{1}H NMR spectrum of product **10** in $CDCl_3$

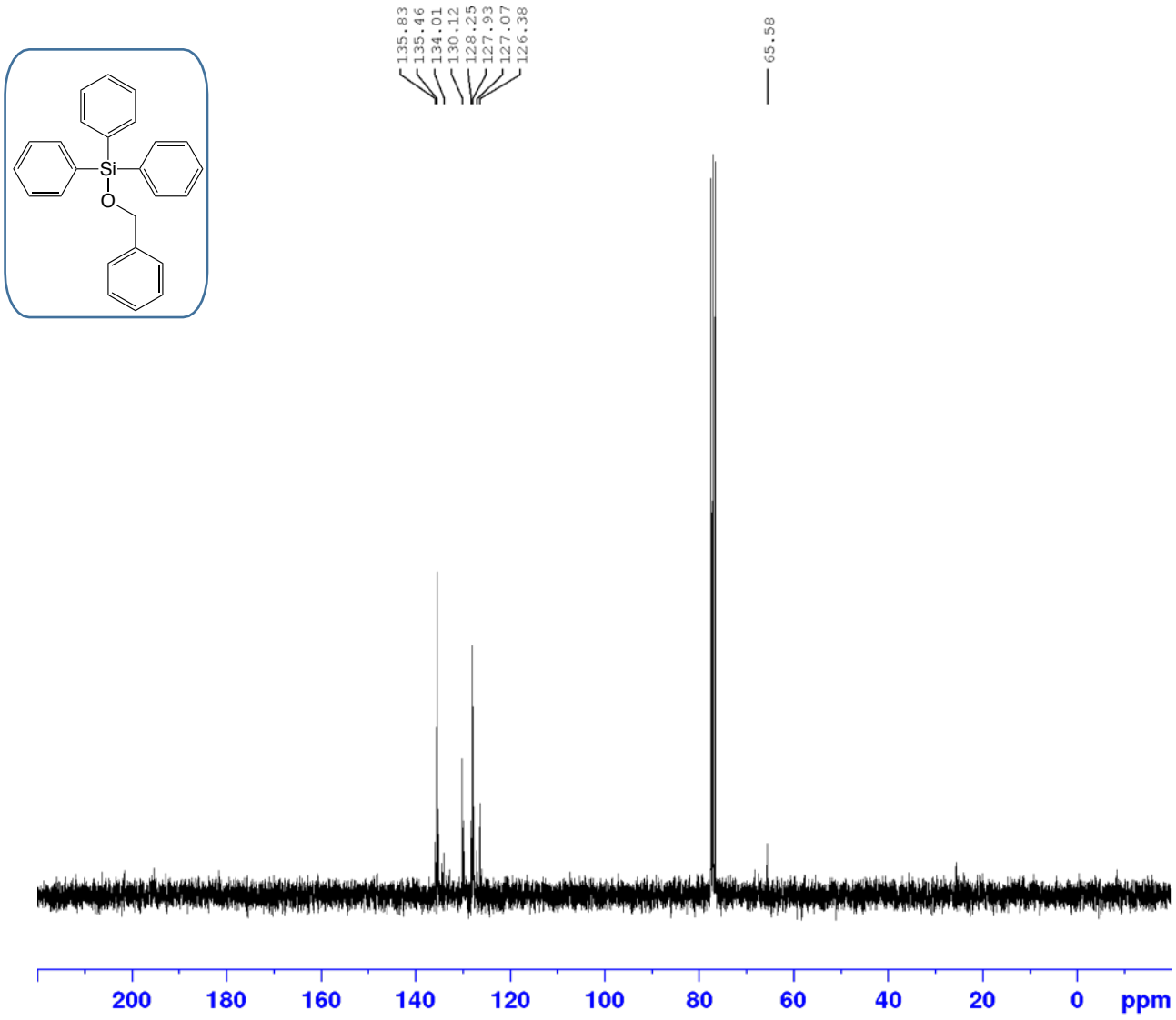

Fig. S34. ^{13}C NMR spectrum of product **10** in $CDCl_3$

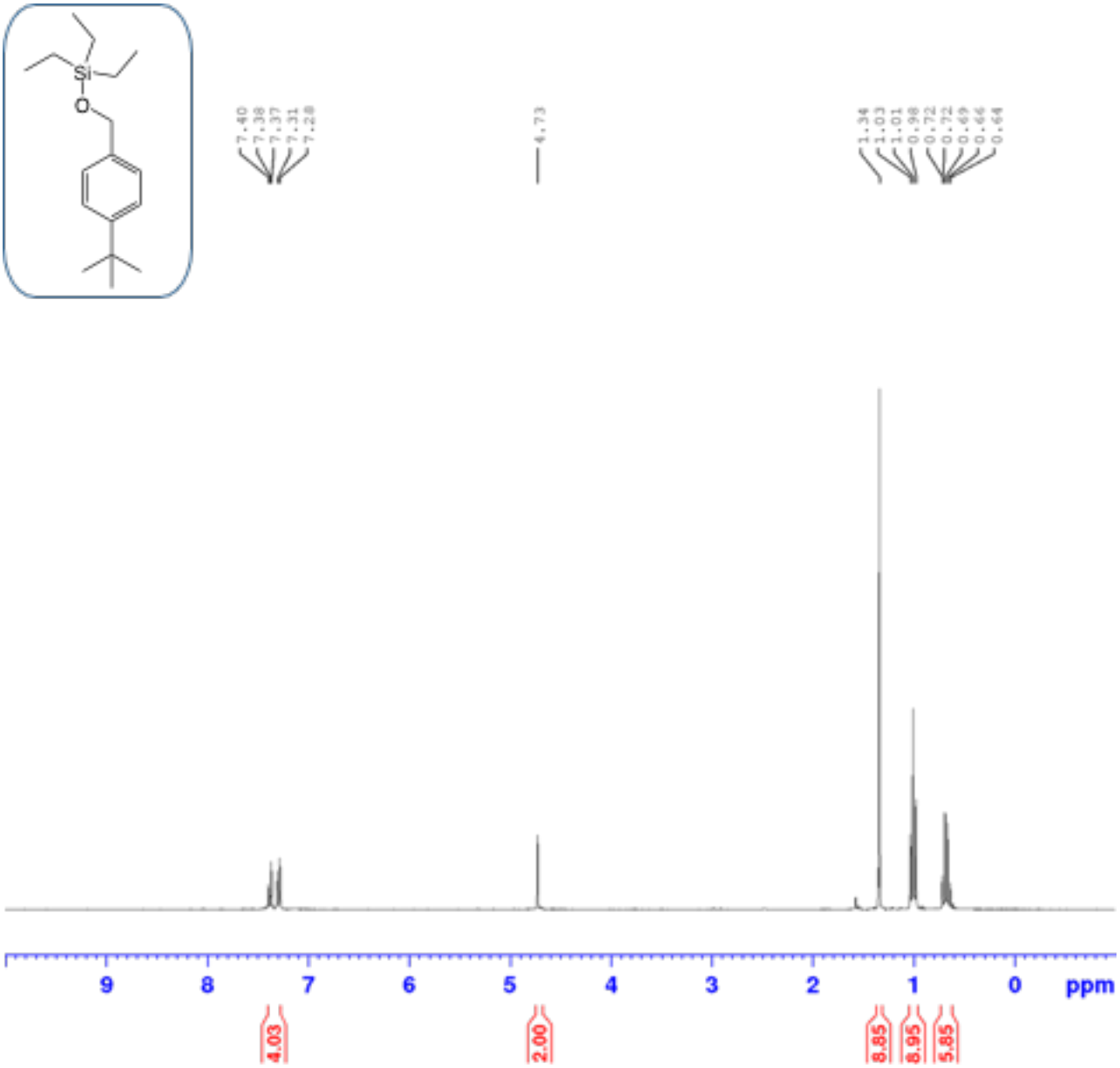

Fig. S35: ^{1}H NMR spectrum of product **11** in $CDCl_3$

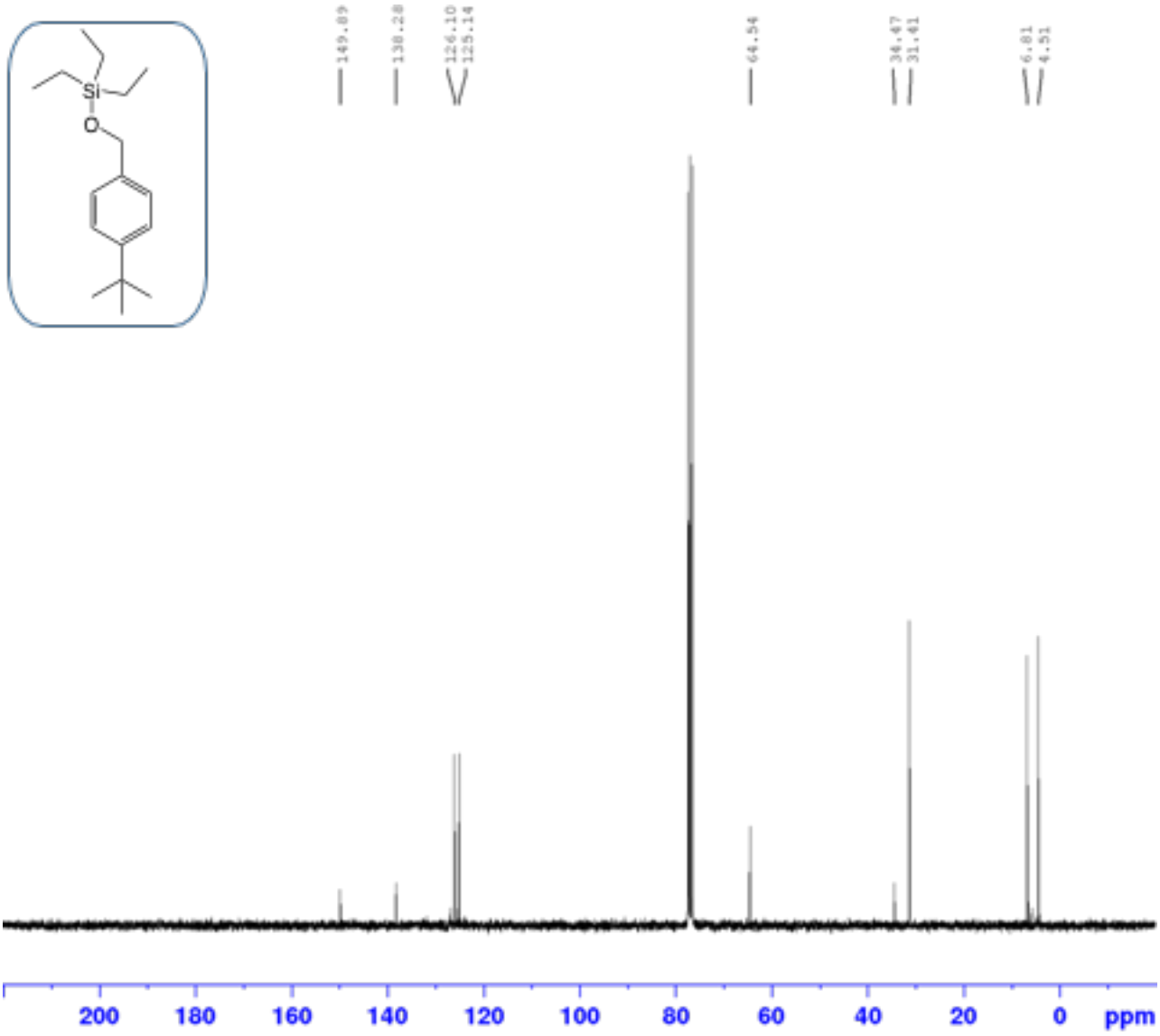

Fig. S36: ^{13}C NMR spectrum of product **11** in $CDCl_3$

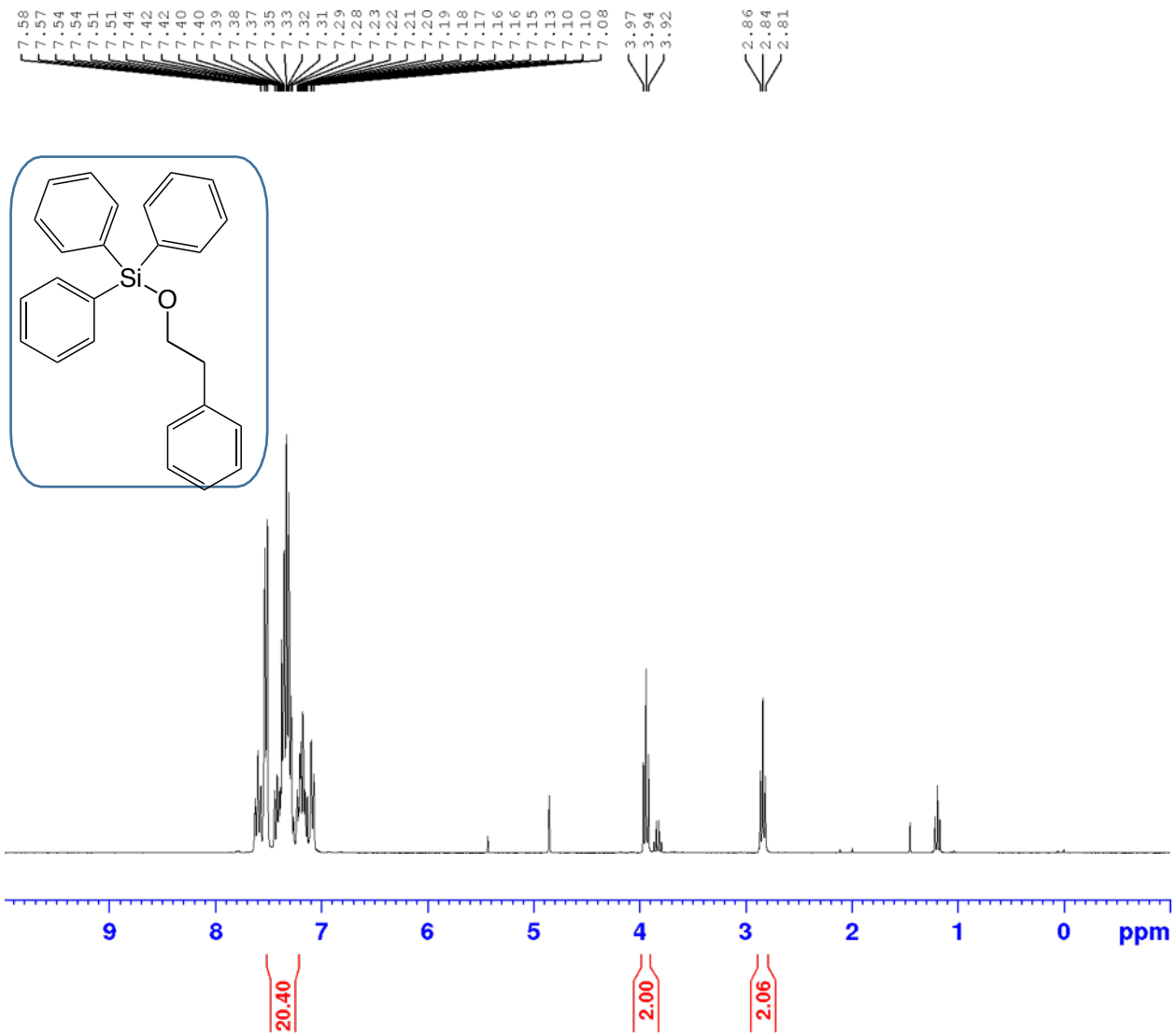

Fig. S37. ^{1}H NMR spectrum of product **12** in $CDCl_3$

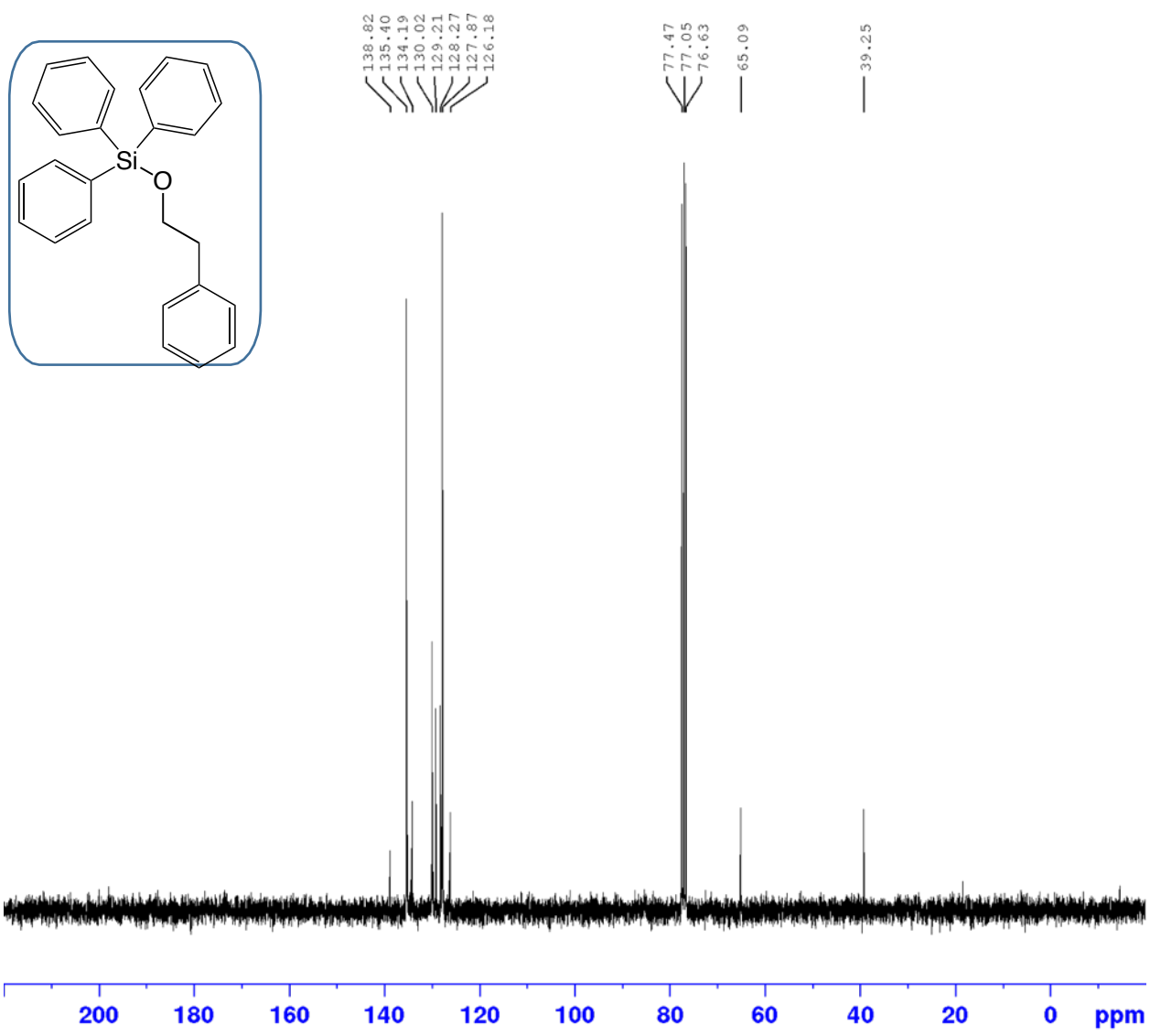

Fig. S38. ^{1}H NMR spectrum of product **12** in $CDCl_3$

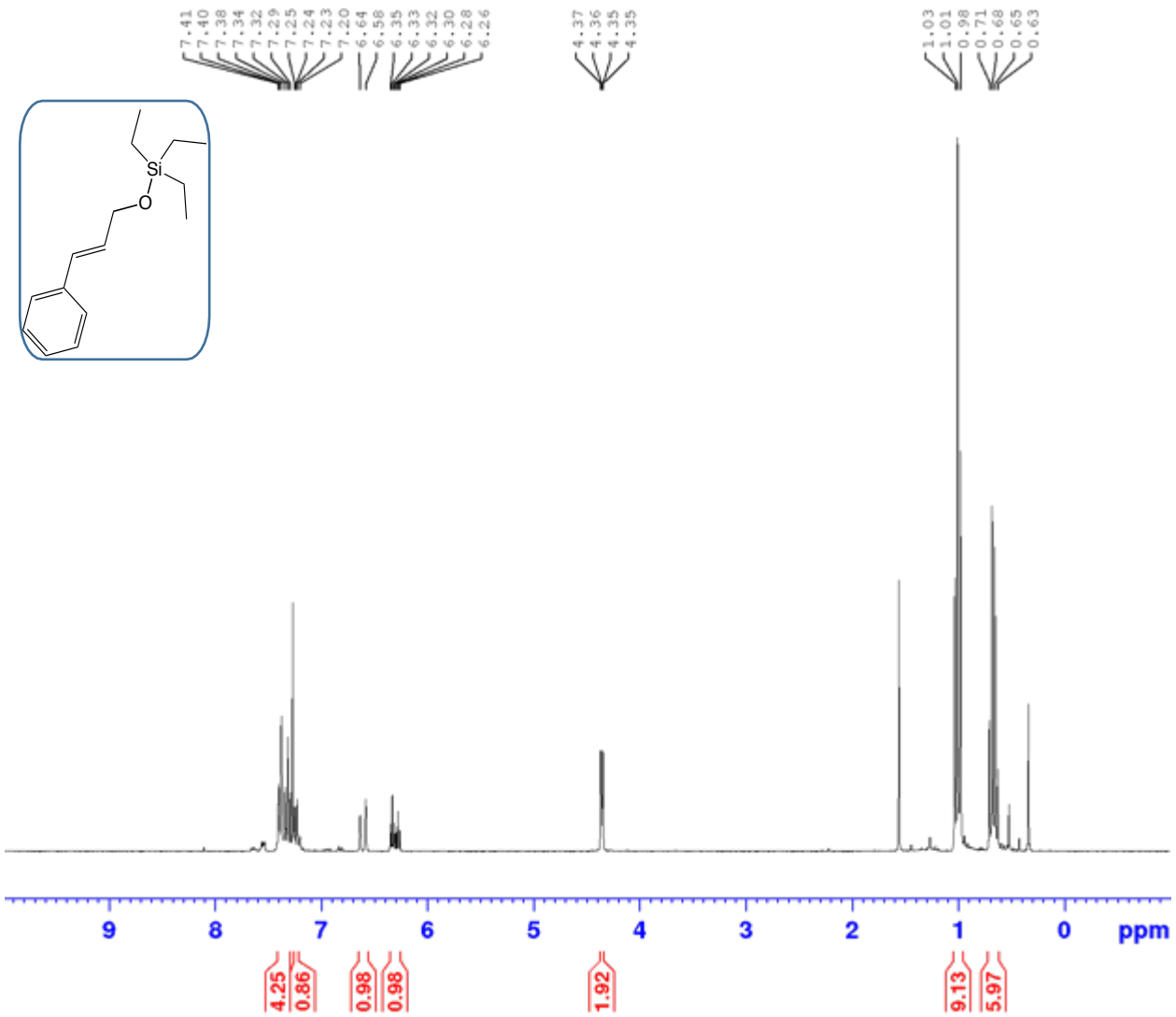

Fig. S39. ^{1}H NMR spectrum of product **13** in $CDCl_3$

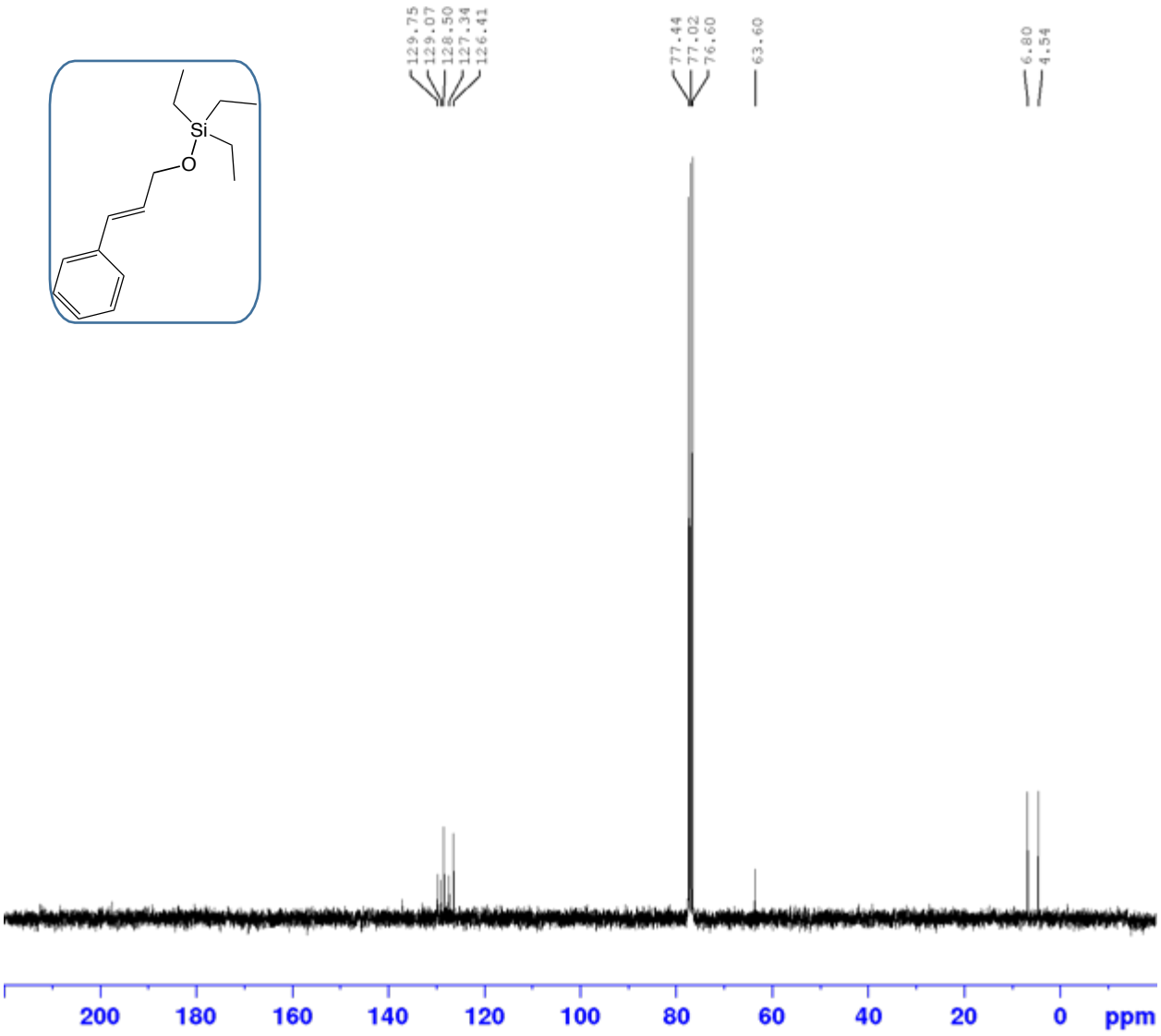

Fig. S40. ^{13}NMR spectrum of product **13** in $CDCl_3$

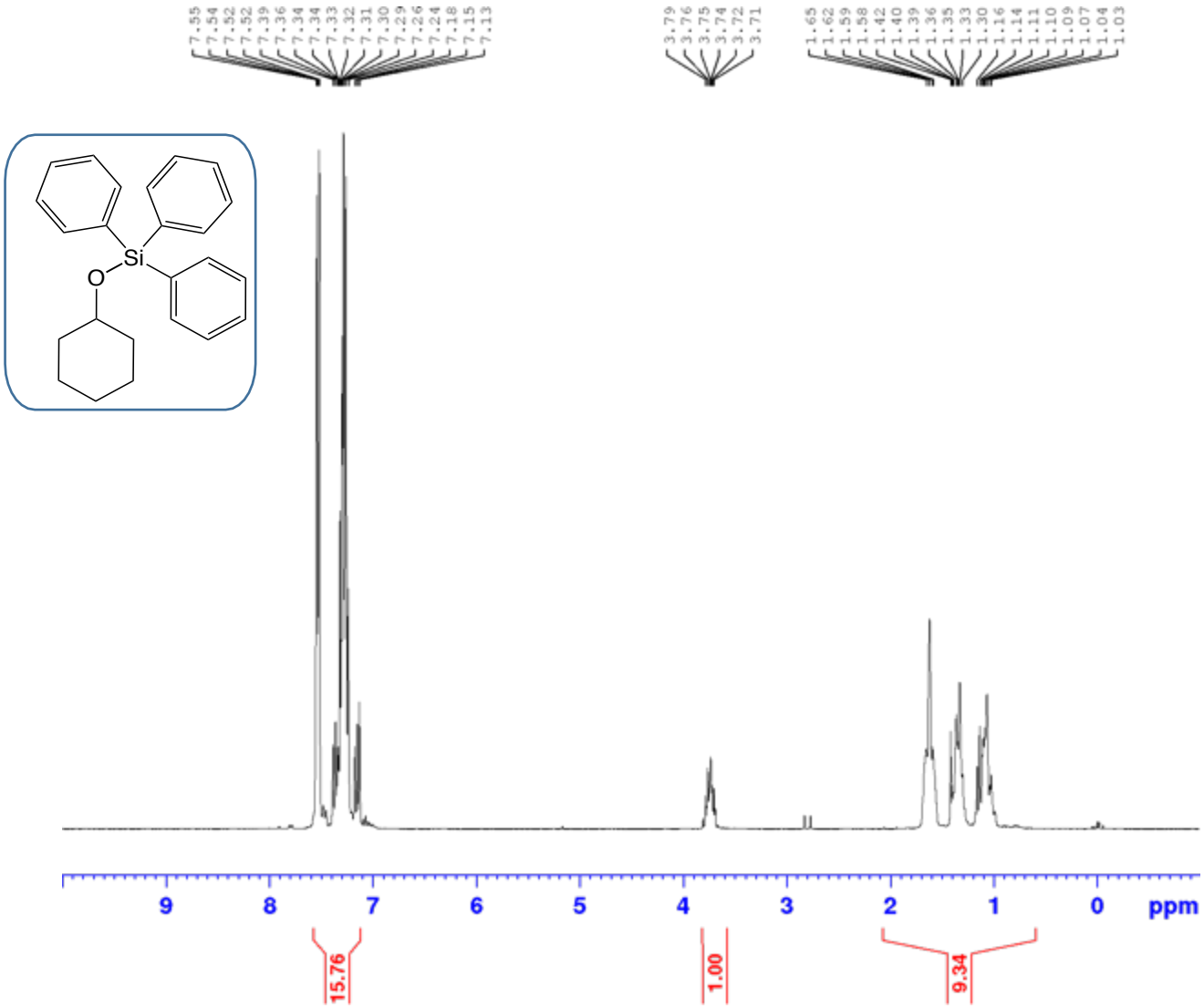

Fig. S41. ^{1}H NMR spectrum of product **14** in $CDCl_3$

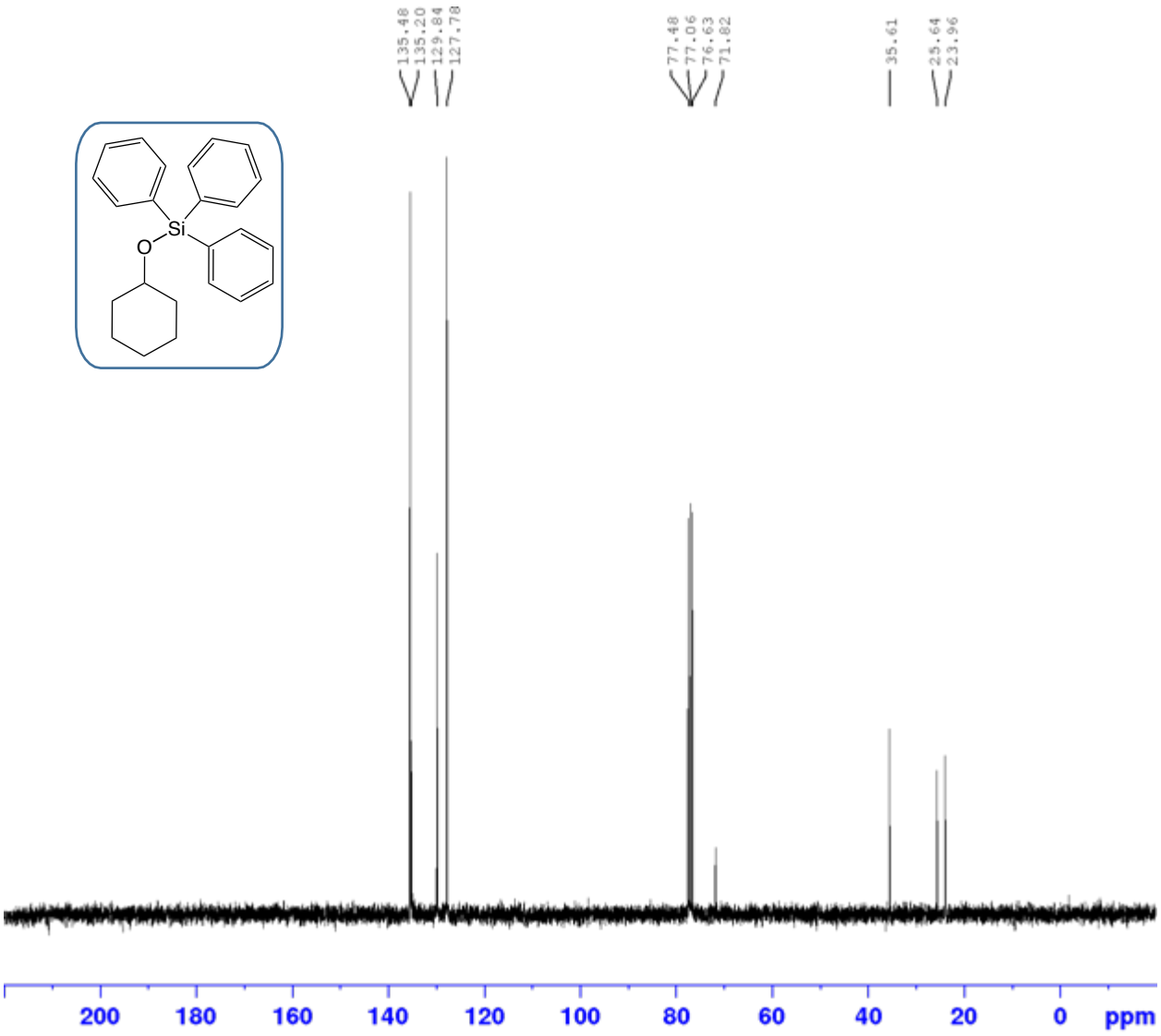

Fig. S42. ^{13}C NMR spectrum of product **14** in $CDCl_3$

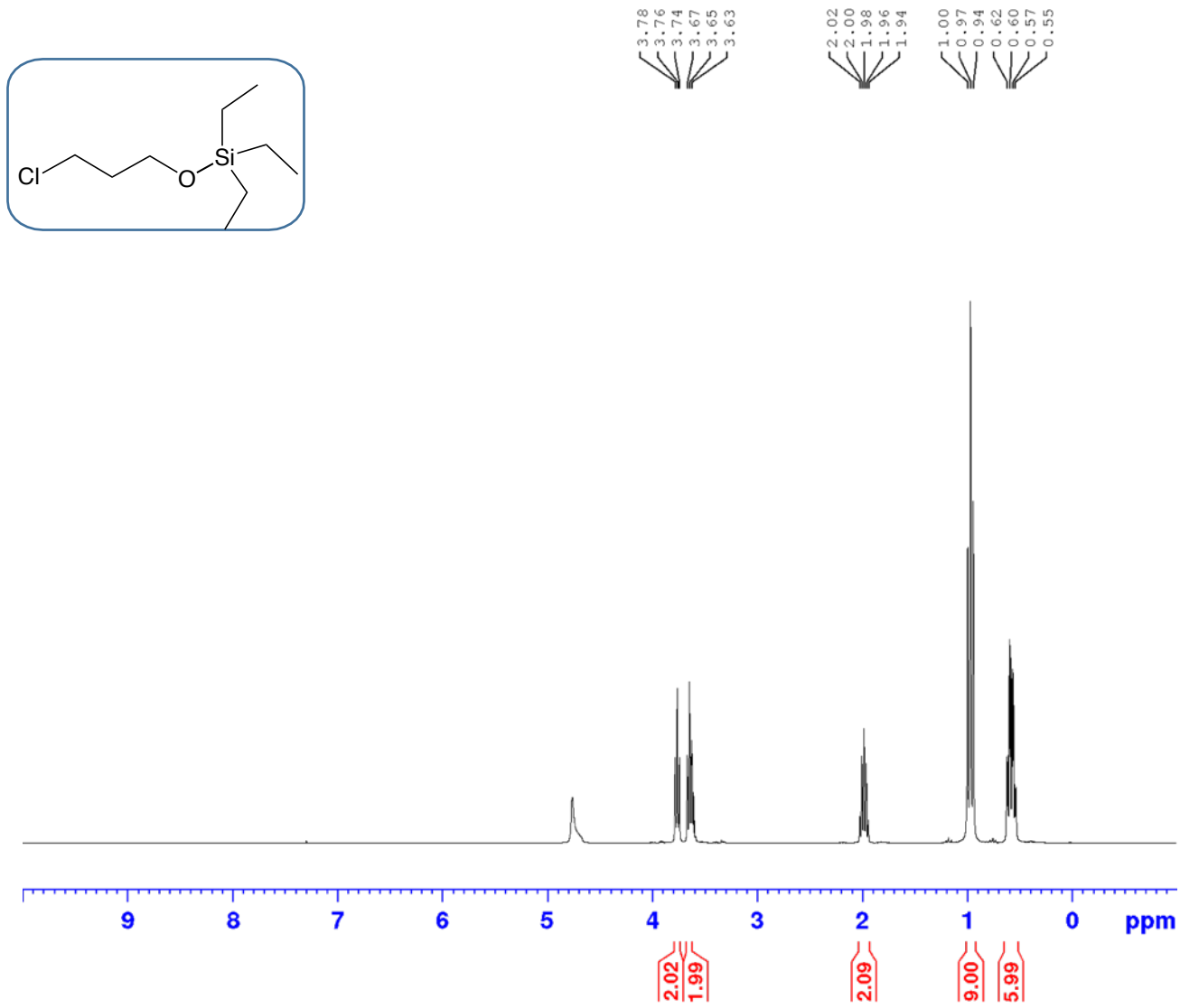

Fig. S43. ^{1}H NMR spectrum of product **15** in $CDCl_3$

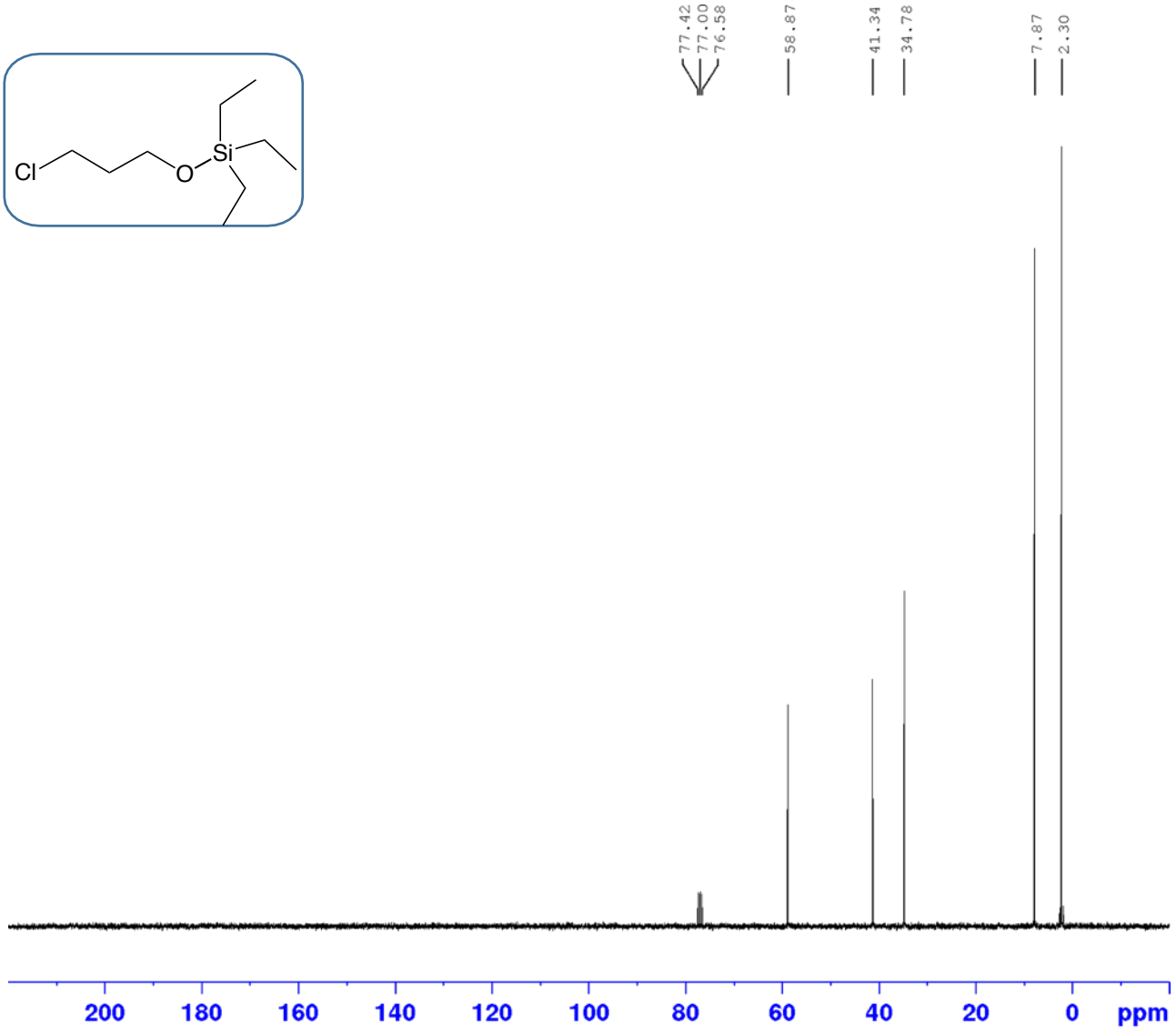

Fig. S44. ^{1}H NMR spectrum of product **15** in $CDCl_3$

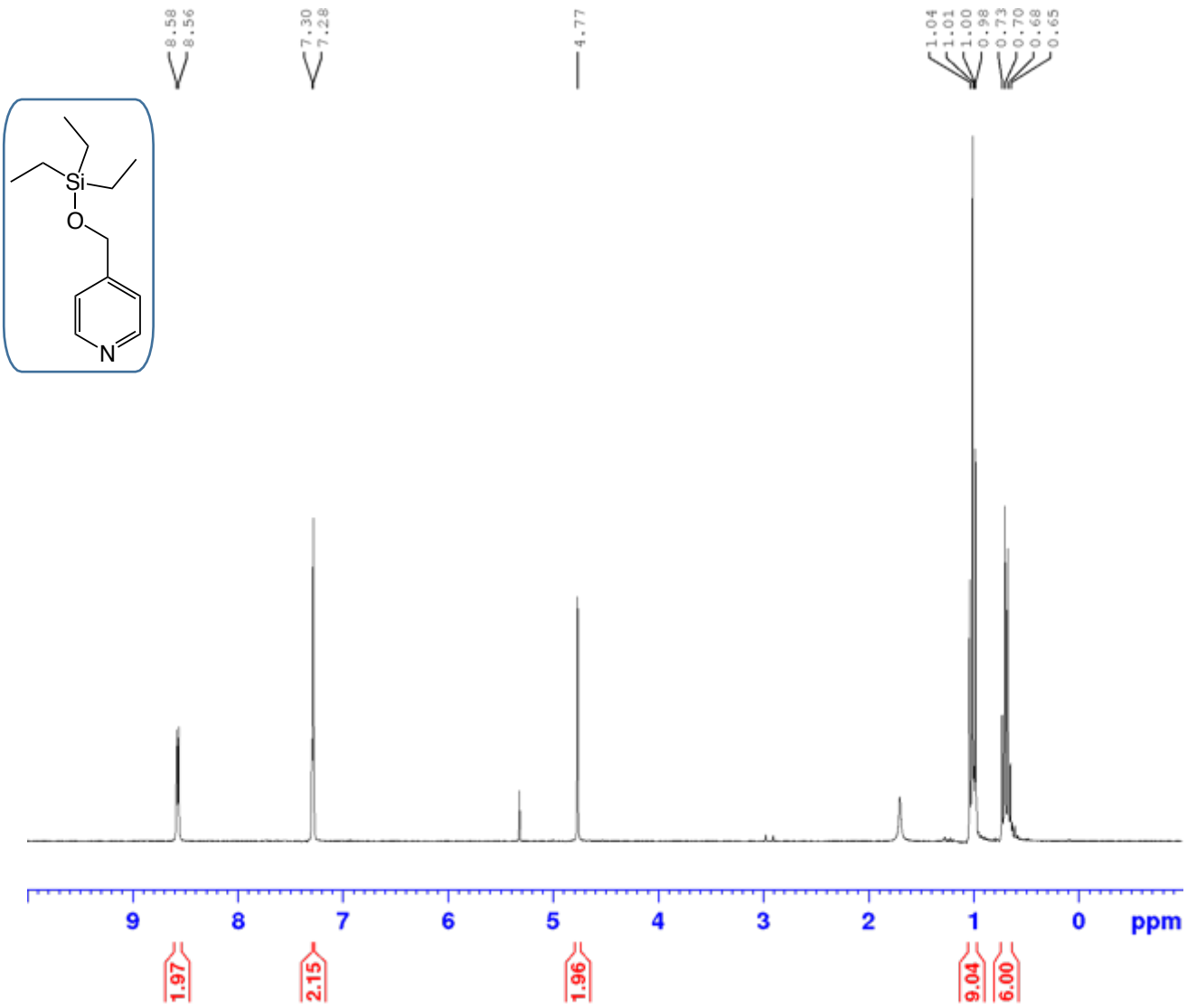

Fig. S45: ^{1}H NMR spectrum of compound **16** in $CDCl_3$

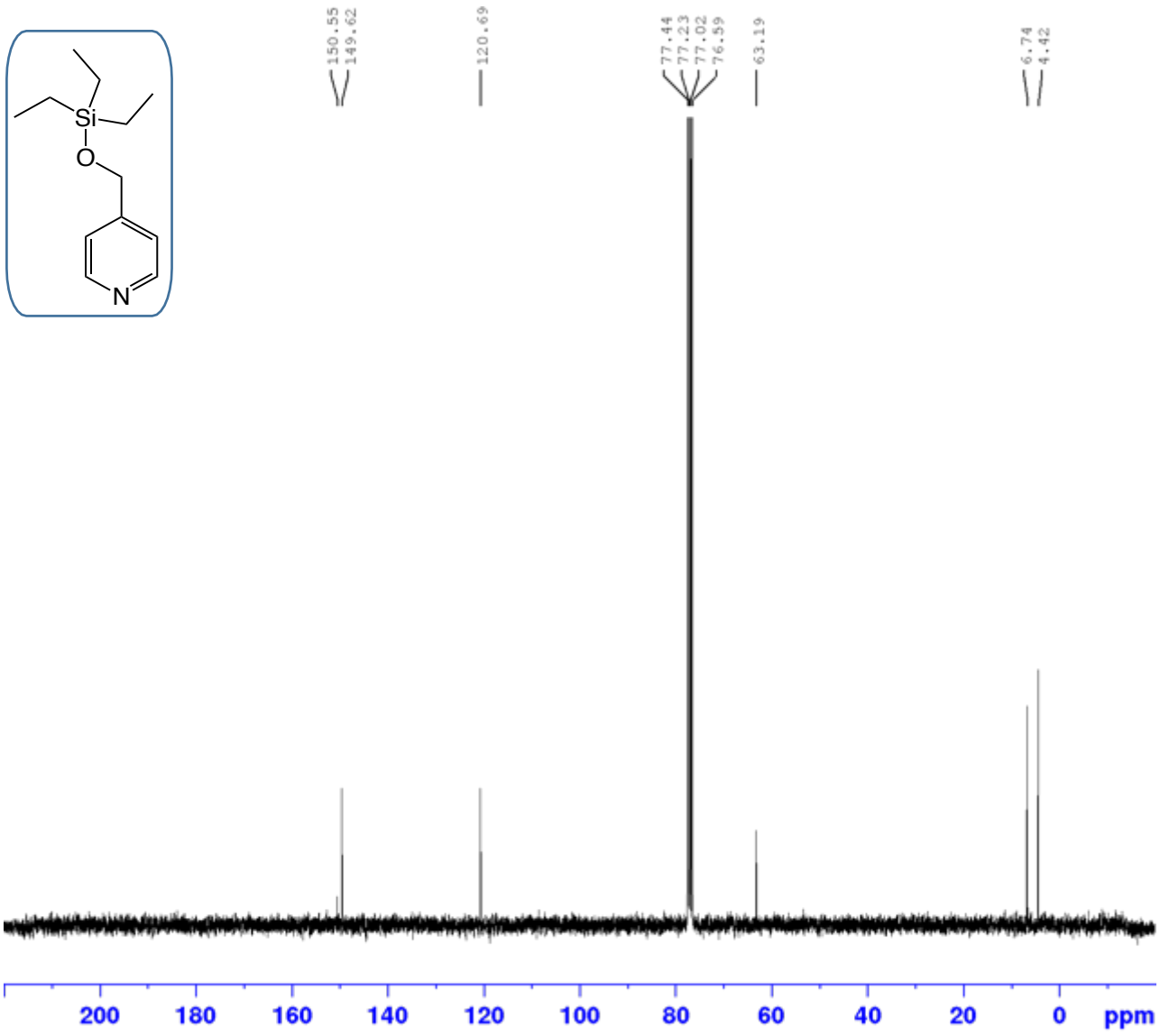

Fig. S46: ^{13}C NMR spectrum of compound **16** in $CDCl_3$

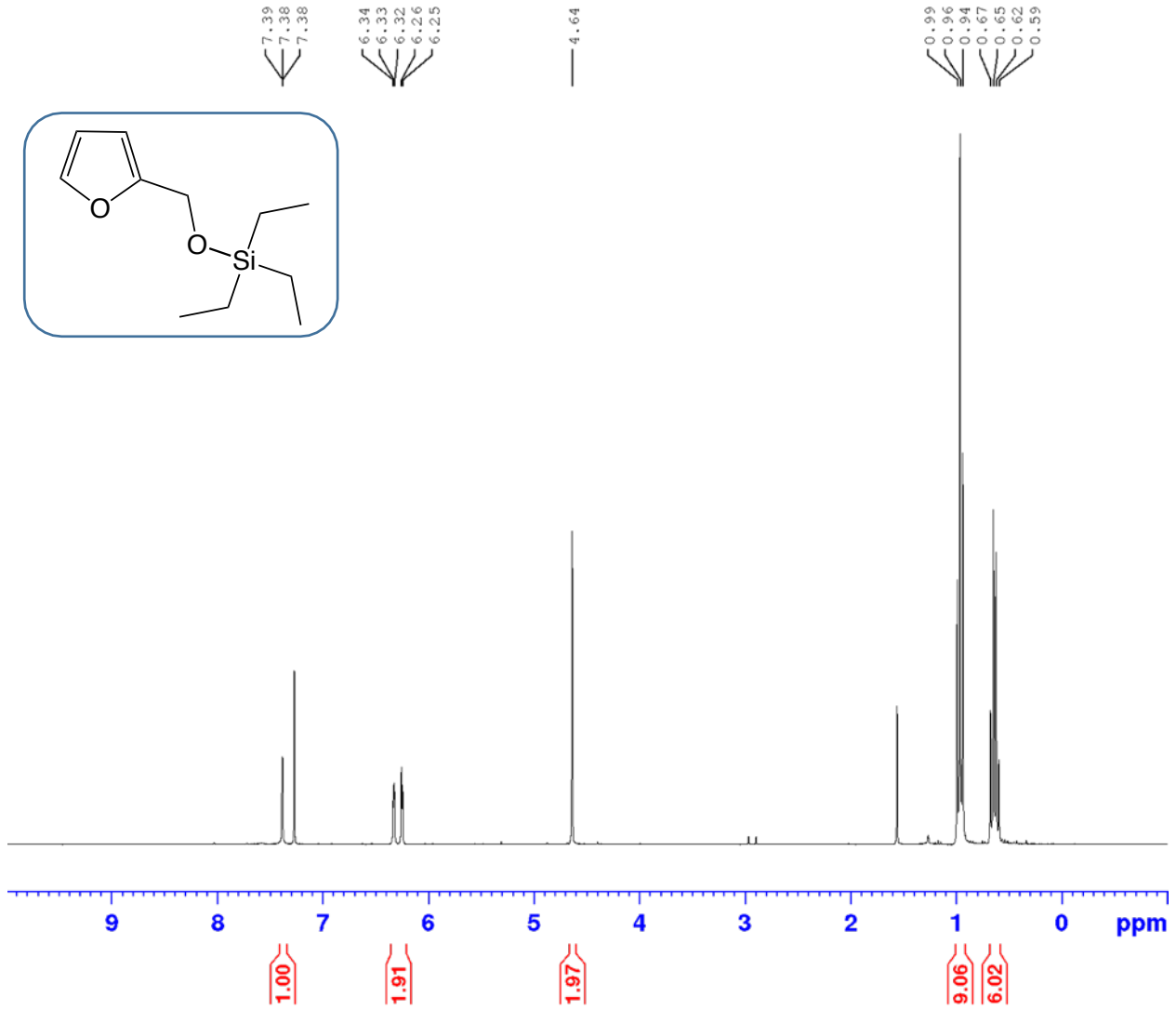

Fig. S47. ^{1}H NMR spectrum of product **17** in $CDCl_3$

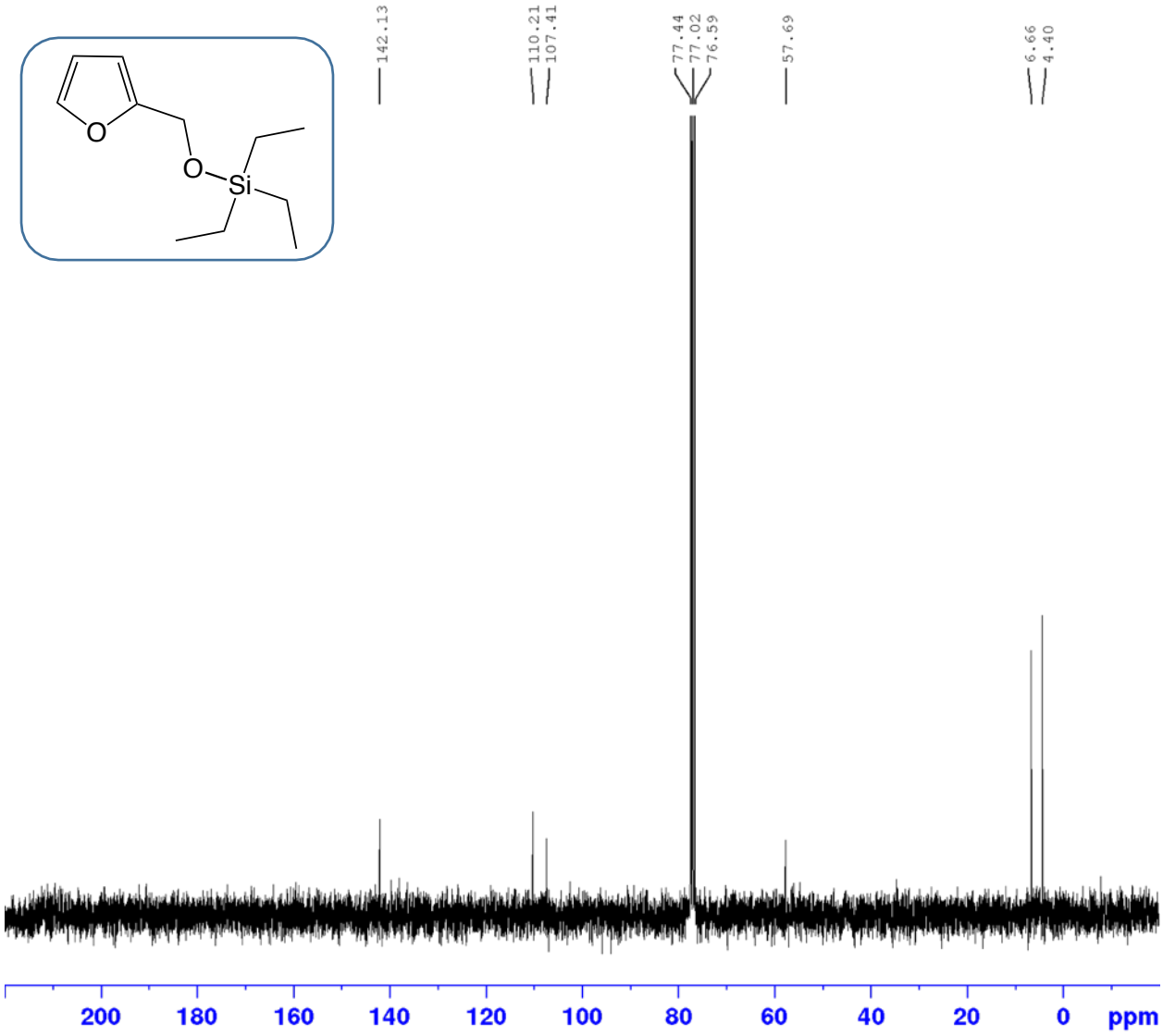

Fig. S48. ^{13}C NMR spectrum of product **17** in $CDCl_3$

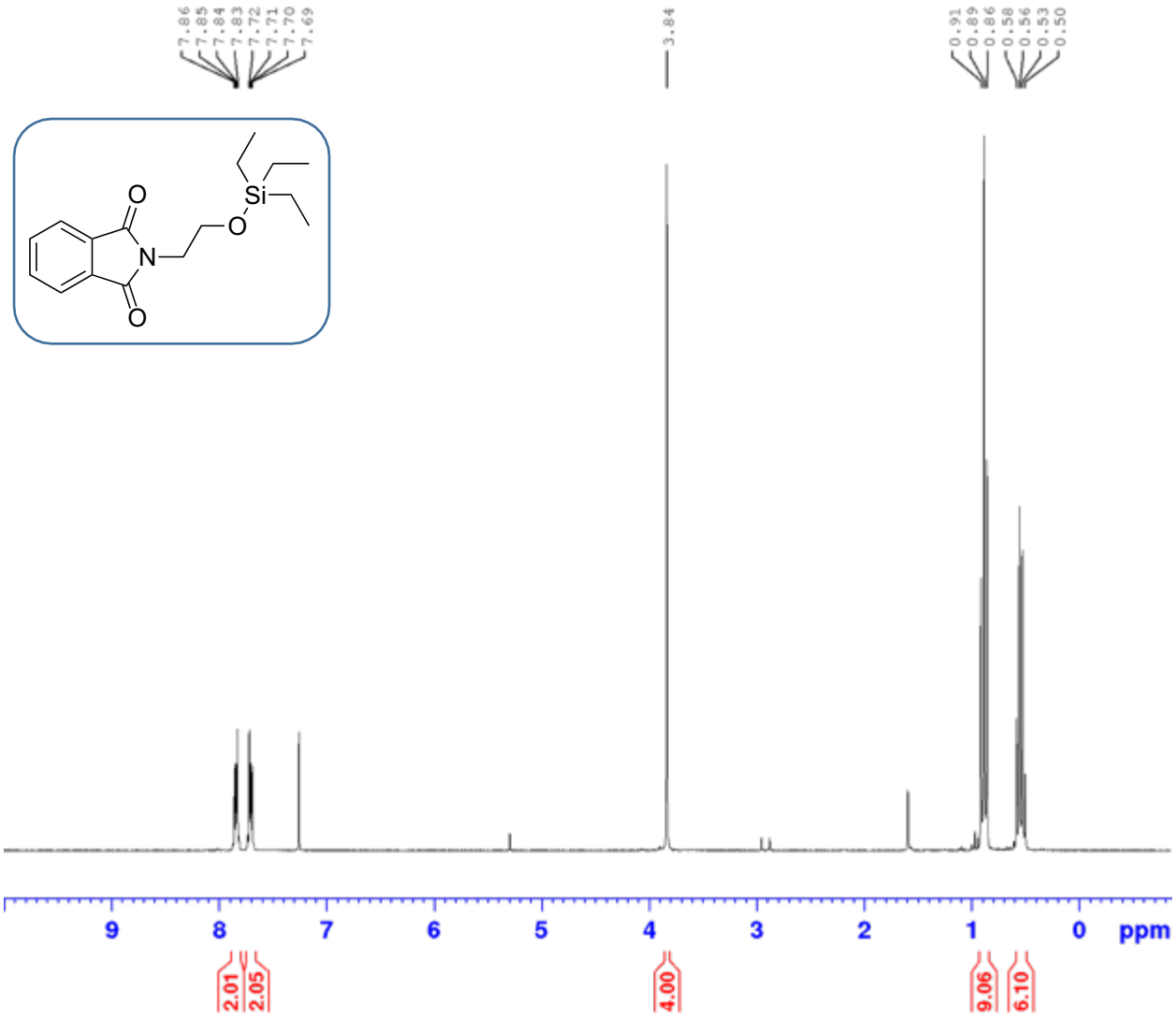

Fig. S49. ^{1}H NMR spectrum of compound **18** in $CDCl_3$

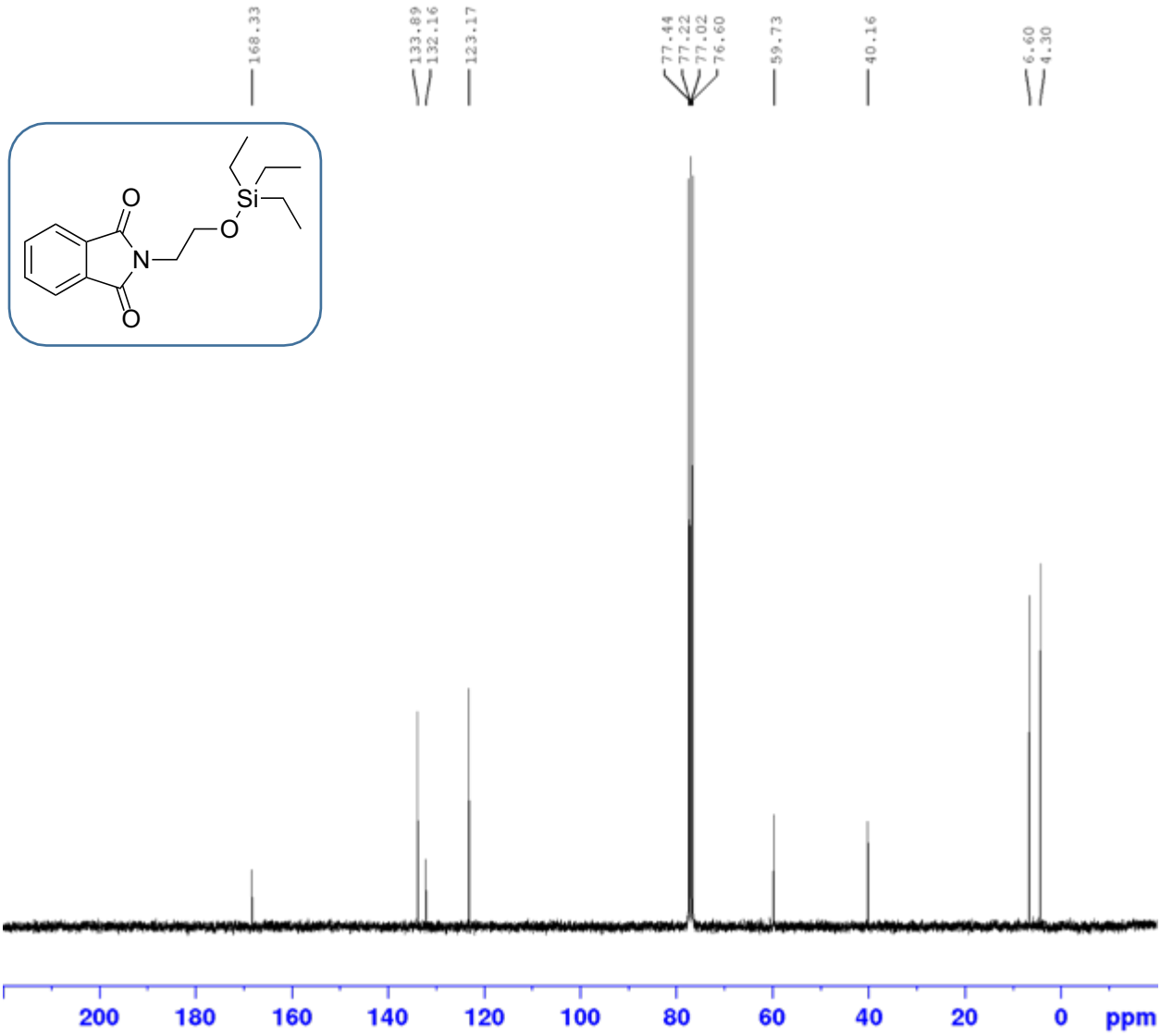

Fig. S50. ^{13}C NMR spectrum of compound **18** in $CDCl_3$

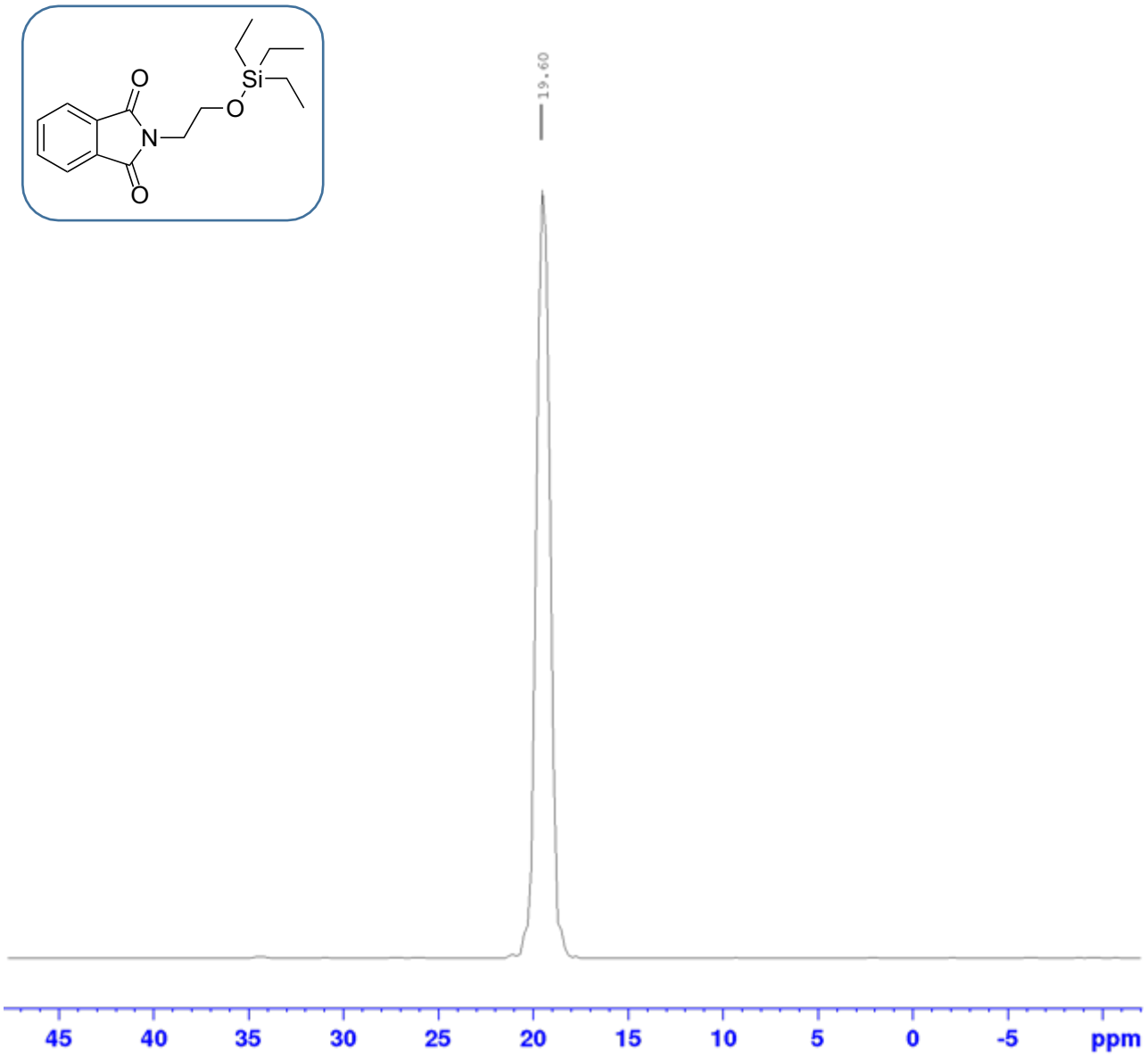

Fig. S51. ^{29}Si NMR spectrum of compound **18** in $CDCl_3$

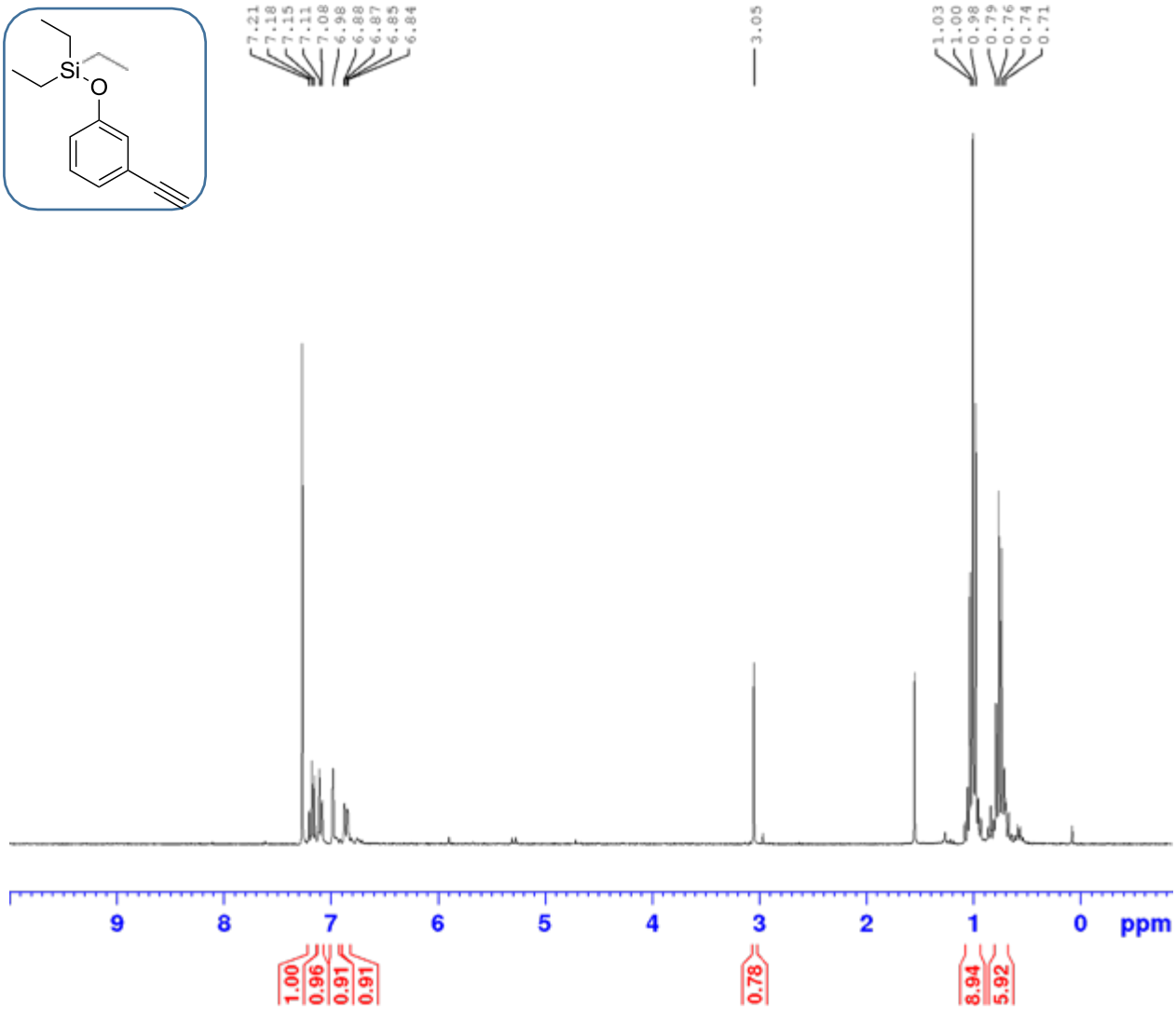

Fig. S52. ^{1}H NMR spectrum of compound **19** in $CDCl_3$

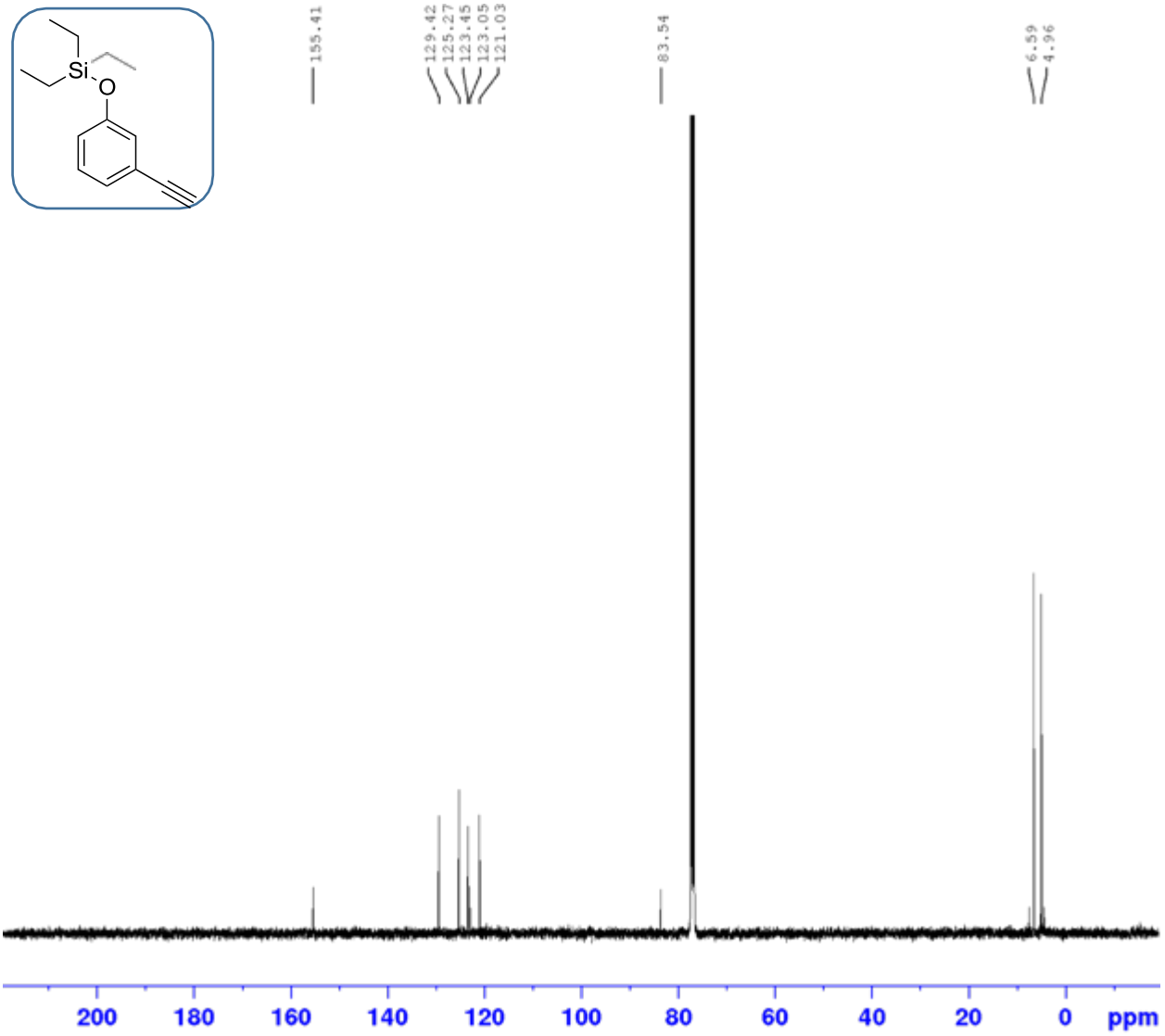

Fig. S53. ^{13}C NMR spectrum of compound **19** in $CDCl_3$

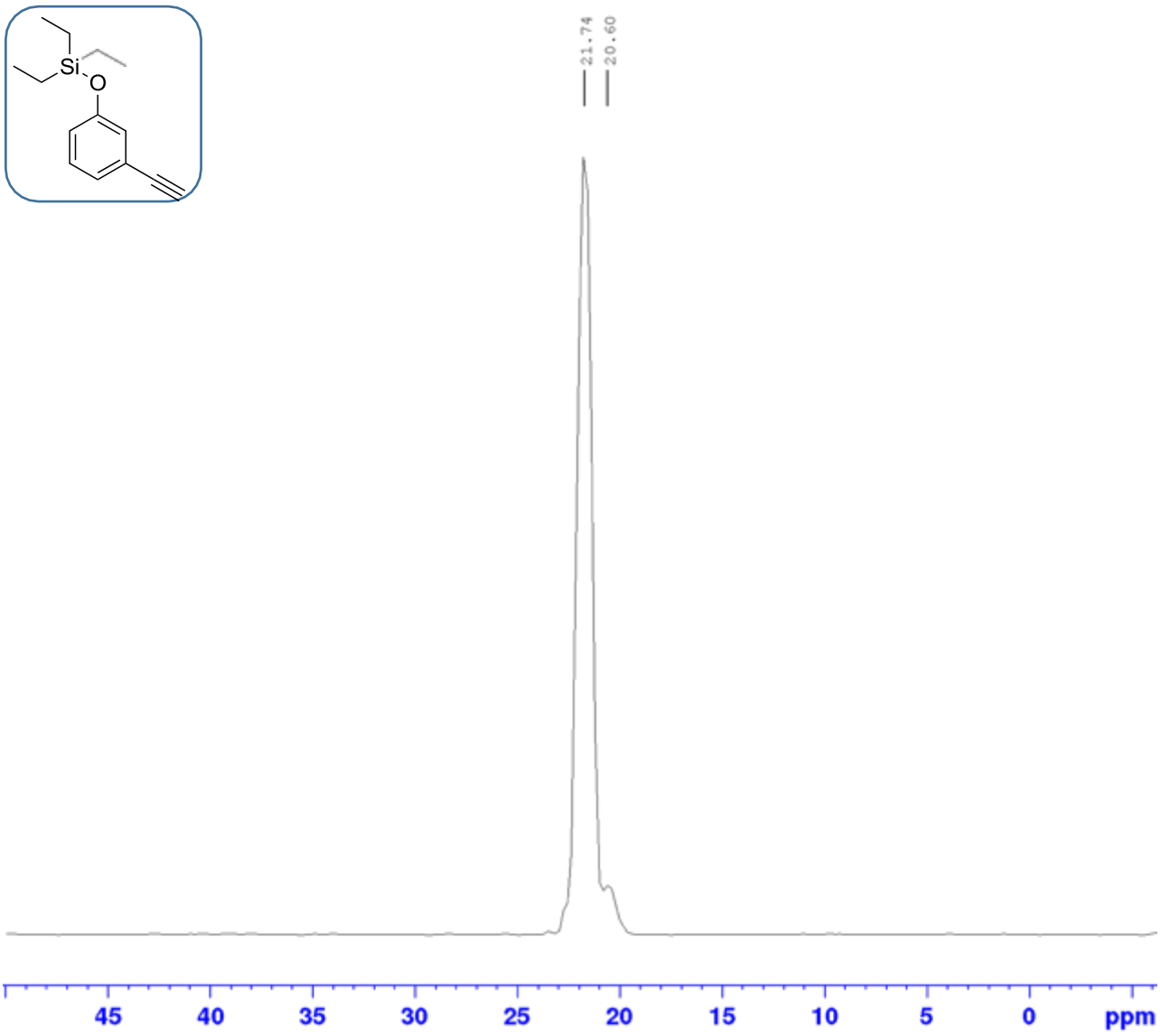

Fig. S54. ^{29}Si NMR spectrum of compound **19** in $CDCl_3$

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