

Synthesis of alkynyltellurides mediated by K_3PO_4 and DMSO

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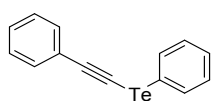
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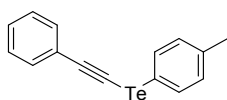
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General Information: The reactions were monitored by TLC carried out on Merck silica gel (60 F254) by using UV light as visualizing agent and 5% vanillin in 10% H₂SO₄ and heat as developing agents. Baker silica gel (particle size 0.040–0.063mm) was used for flash chromatography. Hydrogen nuclear magnetic resonance spectra (¹H NMR) were obtained at 400 MHz on Bruker Avance III HD spectrometer. Spectra were recorded in CDCl₃ solutions. Chemical shifts are reported in ppm, referenced to tetramethylsilane (TMS) as the external reference. Coupling constants (*J*) are reported in Hertz. Abbreviations to denote the multiplicity of a particular signal are s (singlet), d (doublet), t (triplet), q (quartet), quin (quintet), sex (sextet) and m (multiplet). Carbon-13 nuclear magnetic resonance spectra (¹³C NMR) were obtained at 100 MHz on Bruker Avance HD III spectrometer. Chemical shifts are reported in ppm, referenced to the solvent peak of CDCl₃. Low-resolution mass spectra were obtained with a Shimadzu GCMS-QP 2010 Plus mass spectrometer. High-resolution mass spectra (HRMS) were recorded on a Bruker Micro TOF-QII spectrometer 10416.

General procedure for the synthesis of alkynyltellurides 3a-s: To a 5 mL Schlenk tube equipped with a small magnetic stirring bar, containing a solution of an appropriated alkyne (**1a-m**; 0.5 mmol) in DMSO (1.5 mL), K₃PO₄ (5 mol%) and the corresponding diorganyl ditellurides (**2a-f**; 0.25 mmol) were added. The mixture was stirred at 25 °C for the time indicated in Table 2. The reaction mixture was then diluted with ethyl acetate (30 mL) and washed with distilled H₂O (3 x 30 mL). The organic phase was dried with MgSO₄ and concentrated in vacuum and the crude product was purified by column chromatography on silica gel using a hexane or a mixture of hexane/ethyl acetate as the eluent to afford the desired products.

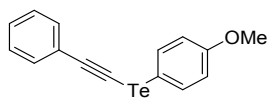


Phenyl(phenylethynyl)tellane (3a): Yield: 0.143 g (93%); yellow oil. ¹H NMR (CDCl₃, 400 MHz) δ 7.73 – 7.68 (m, 2H), 7.45 – 7.43 (m, 2H), 7.31 – 7.21 (m, 6H). ¹³C NMR (CDCl₃, 100 MHz) 135.0 (2C), 131.8 (2C), 129.6 (2C), 128.5, 128.2 (2C), 127.8, 123.2, 114.1, 113.1, 47.6. MS (relative intensity) *m/z*: 308 (M⁺, 6), 178 (100), 101 (14), 77 (15), 44 (18). HRMS calculated for C₁₄H₁₁Te [M + H]⁺: 308.9923. Found: 308.9915.

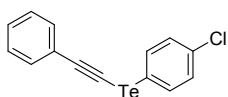


(Phenylethynyl)(*p*-tolyl)tellane (3b): Yield: 0.150 g (93%); yellow solid; mp 61 – 64 °C. ¹H NMR (CDCl₃, 400 MHz) δ 7.63 (d, *J* = 8.2 Hz, 2H), 7.44 – 7.42 (m, 2H), 7.32 – 7.26 (m, 3H), 7.08 – 7.06 (m, 2H), 2.33 (s, 3H). ¹³C NMR (CDCl₃, 100 MHz): δ 138.1, 135.7 (2C), 131.8 (2C),

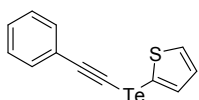
130.6 (2C), 128.4, 128.2 (2C), 123.4, 113.6, 108.6, 47.6, 21.1. MS (relative intensity) m/z : 322 (M^+ , 13), 192 (100), 189 (12), 101 (13), 65 (10). HRMS calculated for $C_{15}H_{12}Te$ [M] $^+$: 322.0001. Found: 322.0016.



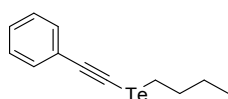
(4-Methoxyphenyl)(phenylethynyl)tellane (3c): Yield: 0.146 g (86%); yellow solid; mp 67 - 68 °C. 1H NMR ($CDCl_3$, 400 MHz): δ 7.70 (d, J = 8.8 Hz, 2H), 7.43 - 7.40 (m, 2H), 7.30 - 7.26 (m, 3H), 6.82 (d, J = 8.8 Hz, 2H), 3.77 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 160.0, 138.2 (2C), 131.8 (2C), 128.4, 128.1 (2C), 123.4, 115.6 (2C), 113.0, 101.3, 55.1, 47.9. MS (relative intensity) m/z : 338 (M^+ , 9), 208 (100), 193 (37), 165 (24), 101 (10), 75 (6). HRMS calculated for $C_{15}H_{13}OTe$ [$M + H$] $^+$: 339.0029. Found: 339.0011.



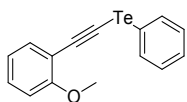
(4-Chlorophenyl)(phenylethynyl)tellane (3d): Yield: 0.152 g (89%); white solid; mp 107 - 110 °C. 1H NMR ($CDCl_3$, 400 MHz): δ 7.63 (d, J = 8.1 Hz, 2H), 7.46 - 7.21 (m, 7H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 136.3 (2C), 134.4, 131.9 (2C), 129.9 (2C), 128.7, 128.2 (2C), 123.1, 114.6, 110.7, 47.1. MS (relative intensity) m/z : 342 (M^+ , 12), 212 (100), 176 (20), 151 (6), 101 (22), 75 (19). HRMS calculated for $C_{14}H_9ClTe$ [M] $^+$: 341.9533. Found: 341.9461.



2-((phenylethynyl)tellanyl)thiophene (3e): Yield: 0.089 g (62%); yellow solid; mp 54 - 57 °C. 1H NMR ($CDCl_3$, 400 MHz) δ (ppm) = 7.52 - 7.50 (m, 2H), 7.41 - 7.38 (m, 2H), 7.28 - 7.26 (m, 3H), 7.0 - 6.98 (m, 1H). ^{13}C NMR ($CDCl_3$, 100 MHz) δ 140.9, 134.8, 131.9 (2C), 128.9, 128.6, 128.1 (2C), 123.1, 111.9, 98.2, 47.9. MS (relative intensity) m/z : 314 (M^+ , 9), 185 (14), 184 (100), 152 (11), 139 (13), 101 (16). HRMS calculated for $C_{12}H_9STe$ [$M + H$] $^+$: 314.9487. Found: 314.9490.

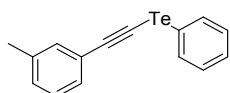


Butyl(phenylethynyl)tellane (3f): Yield: 0.058 g (40%); orange oil. 1H NMR ($CDCl_3$, 400 MHz) δ 7.42 - 7.39 (m, 2H), 7.31 - 7.26 (m, 3H), 2.88 (t, J = 7.4, Hz, 2H), 1.91 (quint. J = 7.4 Hz, 2H), 1.45 (sex. J = 7.4 Hz, 2H), 0.95 (t, J = 7.4 Hz, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 131.6 (2C), 128.1 (2C), 123.7, 111.3, 44.5, 33.6, 24.6, 13.3, 9.9. MS (relative intensity) m/z : 288 (M^+ , 22), 286 (21), 232 (20), 143 (9), 129 (11), 102 (100), 75 (19), 57 (26), 41 (31). HRMS calculated for $C_{12}H_{14}Te$ [M] $^+$: 288.0158. Found: 288.0175.



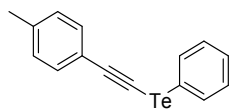
((2-Methoxyphenyl)ethynyl)(phenyl)tellane (3g): Yield: 0.150 g (89%); yellow oil.

^1H NMR (CDCl_3 , 400 MHz) δ 7.76 – 7.71 (m, 2H); 7.42 – 7.39 (m, 1H); 7.25 – 7.21 (m, 4H); 6.88 (td, $J = 7.5$ Hz; 1H); 6.86 – 6.81 (m, 1H); 3.84 (s, 3H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 160.2, 134.5 (2C), 133.5, 129.9, 129.5 (2C), 127.5, 120.2, 113.6, 112.4, 110.6, 110.4, 55.6, 51.2. MS (relative intensity) m/z : 338 (M^+ , 19), 208 (100), 178 (11), 165 (27), 131 (51), 88 (15), 77 (37), 44 (22). HRMS calculated for $\text{C}_{15}\text{H}_{13}\text{OTe}$ [$\text{M}+\text{H}$] $^+$: 339.0029. Found: 339.0018.



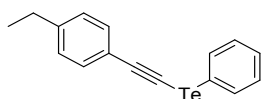
Phenyl(m-tolyethynyl)tellane (3h): Yield: 0.130 g (81%); Yellow oil. ^1H NMR

(CDCl_3 , 400 MHz) δ 7.82 – 7.79 (m, 2H), 7.37 – 7.32 (m, 5H), 7.30 – 7.26 (m, 1H), 7.20 – 7.17 (m, 1H), 2.38 (s, 3H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 137.9, 135.0 (2C), 132.4, 129.7 (2C), 129.5, 128.9, 128.1, 127.8, 123.1, 114.4, 113.2, 46.9, 21.2. MS (relative intensity) m/z : 322 (M^+ , 12), 192 (100), 165 (8), 115 (20), 77 (10), 51 (11). HRMS calculated for $\text{C}_{15}\text{H}_{13}\text{Te}$ [$\text{M}+\text{H}$] $^+$: 323.0079. Found: 323.0065.



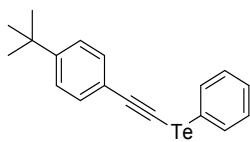
Phenyl(p-tolyethynyl)tellane (3i): Yield: 0.145 g (90%); yellow solid; mp 59 -

62 °C. ^1H NMR (CDCl_3 , 400 MHz) δ 7.72 – 7.69 (m, 2H), 7.35 (d, $J = 7.9$ Hz, 2H), 7.24 – 7.22 (m, 3H), 7.10 (d, $J = 7.9$ Hz, 2H), 2.33 (s, 3H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 138.8, 134.9 (2C), 131.8 (2C), 129.6 (2C), 128.9 (2C), 127.7, 120.3, 114.3, 113.3, 46.3, 21.4. MS (relative intensity) m/z : 322 (M^+ , 11), 207 (178), 192 (100), 115 (26), 77(13), 44 (22). HRMS calculated for $\text{C}_{15}\text{H}_{13}\text{Te}$ [$\text{M} + \text{H}$] $^+$: 323,0079. Found: 323.0079.

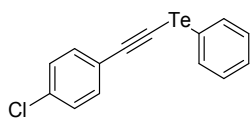


((4-Ethylphenyl)ethynyl)(phenyl)tellane (3j): Yield: 0.144 g (86%); yellow

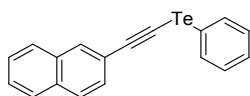
solid; mp 46 – 48 °C. ^1H NMR (CDCl_3 , 400 MHz) δ 7.72 – 7.69 (m, 2H), 7.40 – 7.37 (m, 2H), 7.25 – 7.21 (m, 3H), 7.14 – 7.11 (m, 2H), 2.62 (q, $J = 7.6$, Hz, 2H), 1.20 (t, $J = 7.6$ Hz, 3H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 145.1, 134.8 (2C), 131.9 (2C), 129.6 (2C), 127.7 (2C), 127.7, 120.5, 114.4, 113.3, 46.3, 28.7, 15.3. MS (relative intensity) m/z : 336 (M^+ , 16), 206 (90), 191 (100), 114 (11), 77 (11), 51 (10). HRMS calculated for $\text{C}_{16}\text{H}_{15}\text{Te}$ [$\text{M} + \text{H}$] $^+$: 337.0236, Found: 337.0229.



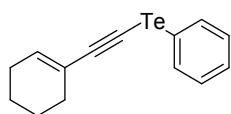
(4-*tert*-Butylphenyl)ethynyl(phenyl)tellane (3k): Yield: 0.133 g (73%); yellow solid; mp 67 – 69 °C. ¹H NMR (CDCl₃, 400 MHz) δ 7.72 – 7.69 (m, 2H), 7.42 – 7.39 (m, 2H), 7.35 – 7.32 (m, 2H), 7.26 – 7.22 (m, 3H), 1.29 (s, 9H). ¹³C NMR (CDCl₃, 100 MHz): δ 151.9, 134.8 (2C), 131.7 (2C), 129.7 (2C), 127.7, 125.2 (2C), 120.3, 114.4, 113.3, 46.3, 34.7, 31.1(3). MS (relative intensity) *m/z*: 364 (M⁺, 14), 291 (13), 234 (34), 219 (100), 141 (9), 77 (9). HRMS calculated for C₁₈H₁₈Te [M]⁺: 364.0471, Found: 364.0464.



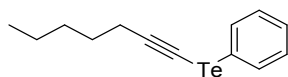
(4-Chlorophenyl)ethynyl(phenyl)tellane (3l): Yield: 0.147 g (86%); yellow solid. mp 93 – 96 °C. ¹H NMR (CDCl₃, 400 MHz) δ 7.73 (d, *J* = 7.0 Hz, 2H), 7.44 – 7.11 (m, 7H). ¹³C NMR (CDCl₃, 100 MHz): 135.3 (2C), 134.6, 133.0 (2C), 129.8 (2C), 128.5 (2C), 128.0, 121.8, 112.9, 112.8, 49.0. MS (relative intensity) *m/z*: 342 (M⁺, 11), 212 (100), 176 (21), 100 (8), 77 (12), 51 (17). HRMS calculated for C₁₄H₉ClTe [M]⁺: 341.9455. Found: 341.9450.



(Naphthalen-2-ylethynyl)(phenyl)tellane (3m): Yield: 0.159 g (89%); Yellow solid. mp 59 – 62 °C. ¹H NMR (CDCl₃, 400 MHz) δ 7.95 (s, 1H), 7.77 – 7.73 (m, 5H), 7.50 – 7.41 (m, 3H), 7.27 – 7.23 (m, 3H). ¹³C NMR (CDCl₃, 100 MHz): δ 135.1 (2C), 132.8, 132.7, 131.8, 129.7 (2C), 128.4, 127.8, 127.6, 126.8, 126.5, 120.5, 114.6, 113.1, 48.0. MS (relative intensity) *m/z*: 358 (M⁺, 6), 228 (100), 151 (22), 77 (21), 51 (15). HRMS calculated for C₁₈H₁₃Te [M + H]⁺: 359.0079, Found: 359.0065.

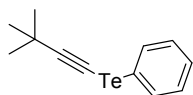


(Cyclohex-1-en-1-ylethynyl)(phenyl)tellane (3n): Yield: 0.098 g (63%); yellow oil. ¹H NMR (CDCl₃, 400 MHz) δ 7.75 – 7.68 (m, 2H), 7.30 – 7.27 (m, 3H), 6.20 (m, 1H), 2.23 – 2.18 (m, 4H), 1.72 – 1.59 (m, 4H). ¹³C NMR (CDCl₃, 100 MHz) δ 136.3, 134.6 (2C), 129.6 (2C), 127.6, 121.1, 116.5, 113.5, 43.4, 29.2, 25.5, 22.2, 21.3. MS (relative intensity) *m/z*: 312 (M⁺, 35), 308 (19), 165 (86), 153 (49), 103 (21), 77 (100), 51 (51). HRMS calculated for C₁₄H₁₄Te [M]⁺: 312.0158. Found: 312.0154.



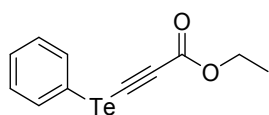
Hept-1-yn-1-yl(phenyl)tellane (3o): Yield: 0.107 g (71%); orange oil. ¹H NMR (CDCl₃, 400 MHz) δ 7.69 – 7.64 (m, 2H), 7.25 – 7.22 (m, 3H), 2.56 (t, *J* = 7.1 Hz, 2H), 1.57

(quint. $J = 6.9$ Hz, 2H), 1.43 – 1.28 (m, 4H), 0.90 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (CDCl_3 , 100 MHz): δ 134.6 (2C), 129.5 (2C), 127.5, 116.1, 113.2, 34.7, 31.0, 28.6, 22.1, 21.0, 13.9. MS (relative intensity) m/z : 302 (M^+ , 23), 143 (42), 129 (23), 115 (100), 95 (30), 77 (41), 55 (19), 41 (17). HRMS calculated for $\text{C}_{13}\text{H}_{16}\text{Te}$ [M] $^+$: 302.0314, Found: 302.0314.



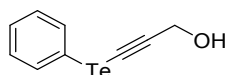
(3,3-Dimethylbut-1-yn-1-yl)(phenyl)tellane (3p): Yield: 0.043 g (30%); yellow oil.

^1H NMR (CDCl_3 , 400 MHz) δ 7.70 – 7.64 (m, 2H), 7.31 – 7.23 (m, 3H), 1.34 (s, 9H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 134.0 (2C), 129.5 (2C), 127.4, 124.4, 113.5, 33.5, 31.1 (3C), 29.7. MS (relative intensity) m/z : 288 (M^+ , 19), 207 (13), 143 (100), 128 (30), 77 (28), 53 (11), 41 (19). HRMS calculated for $\text{C}_{12}\text{H}_{14}\text{Te}$ [M] $^+$: 288.0158. Found: 288.0173.



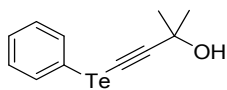
Ethyl 3-(phenyltellanyl)propiolate (3q): Yield: 0.122 g (80%); orange oil. ^1H

NMR (CDCl_3 , 400 MHz) δ 7.77 – 7.74 (m, 2H), 7.38 – 7.28 (m, 3H), 4.25 (q, $J = 7.1$ Hz, 2H), 1.32 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 152.0, 136.3 (2C), 129.9 (2C), 128.7, 110.8, 107.2, 61.8, 55.4, 13.9. MS (relative intensity) m/z : 304 (M^+ , 21), 232 (12), 129 (91), 102 (100), 77 (62), 51 (74), 44 (8). HRMS calculated for $\text{C}_{11}\text{H}_{11}\text{O}_2\text{Te}$ [$\text{M} + \text{H}$] $^+$: 304.9821. Found: 304.9811.



3-(phenyltellanyl)prop-2-yn-1-ol (3r): Yield: 0.084 g (64%); yellow oil. ^1H NMR

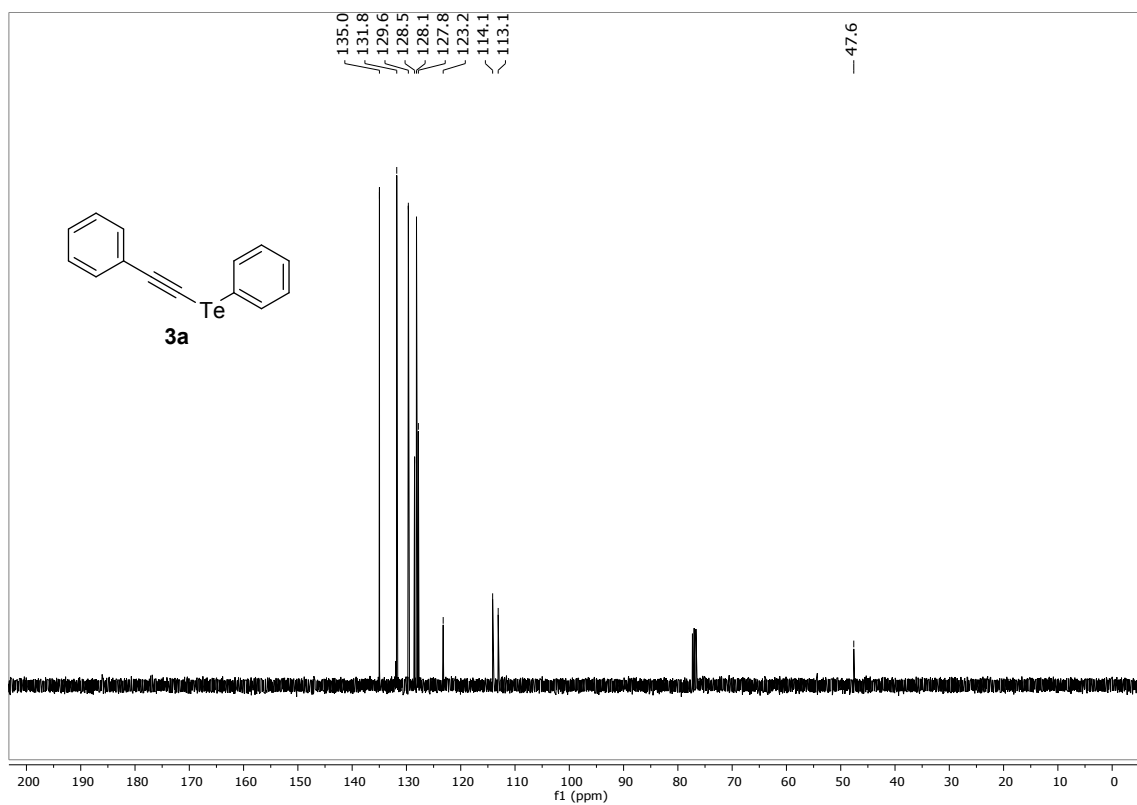
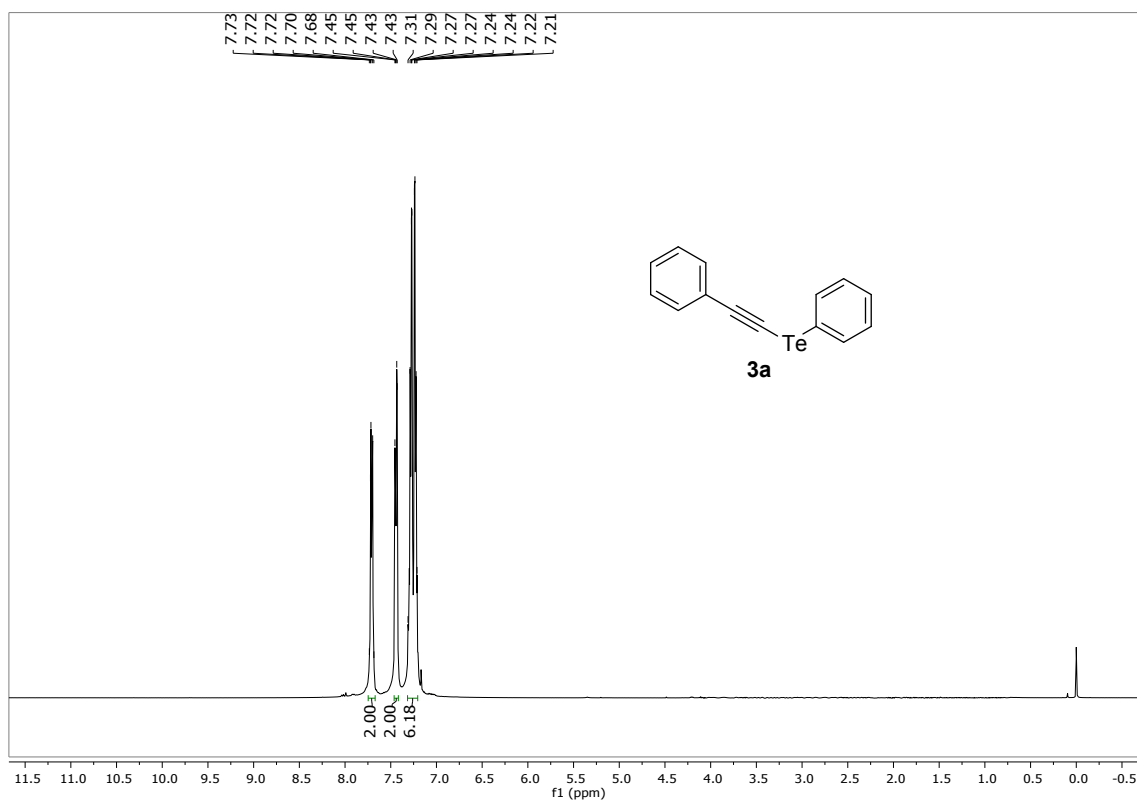
(CDCl_3 , 400 MHz) δ 7.71 – 7.67 (m, 2H), 7.29 – 7.21 (m, 3H), 4.48 (s, 2H), 2.74 (s, 1H). ^{13}C NMR (CDCl_3 , 100 MHz) δ 135.4 (2C), 129.6 (2C), 128.0, 113.2, 112.3, 52.0, 44.5. MS (relative intensity) m/z : 262 (M^+ , 11), 115 (72), 77 (86), 55 (23), 44 (100). HRMS calculated for $\text{C}_9\text{H}_8\text{OTe}$ [M] $^+$: 261.9637. Found: 261.9633.



2-Methyl-4-(phenyltellanyl)but-3-yn-2-ol (3s): Yield: 0.078 g (54%); yellow oil.

^1H NMR (CDCl_3 , 400 MHz) δ (ppm) = 7.67-7.64 (m, 2H); 7.28-7.22 (m, 3H); 2.34 (s, 1H); 1.57 (s, 6H). ^{13}C NMR (CDCl_3 , 100 MHz) δ (ppm) = 134.8 (2C), 129.7 (2C), 127.8, 119.7, 112.7, 66.3, 40.0, 31.4. MS (relative intensity) m/z : 290 (10), 272 (11), 141 (100), 102 (26). HRMS calcd. for $\text{C}_{11}\text{H}_{12}\text{OTe}$ [M] $^+$: 289.9945. Found: 289.9945.

SELECTED SPECTRA



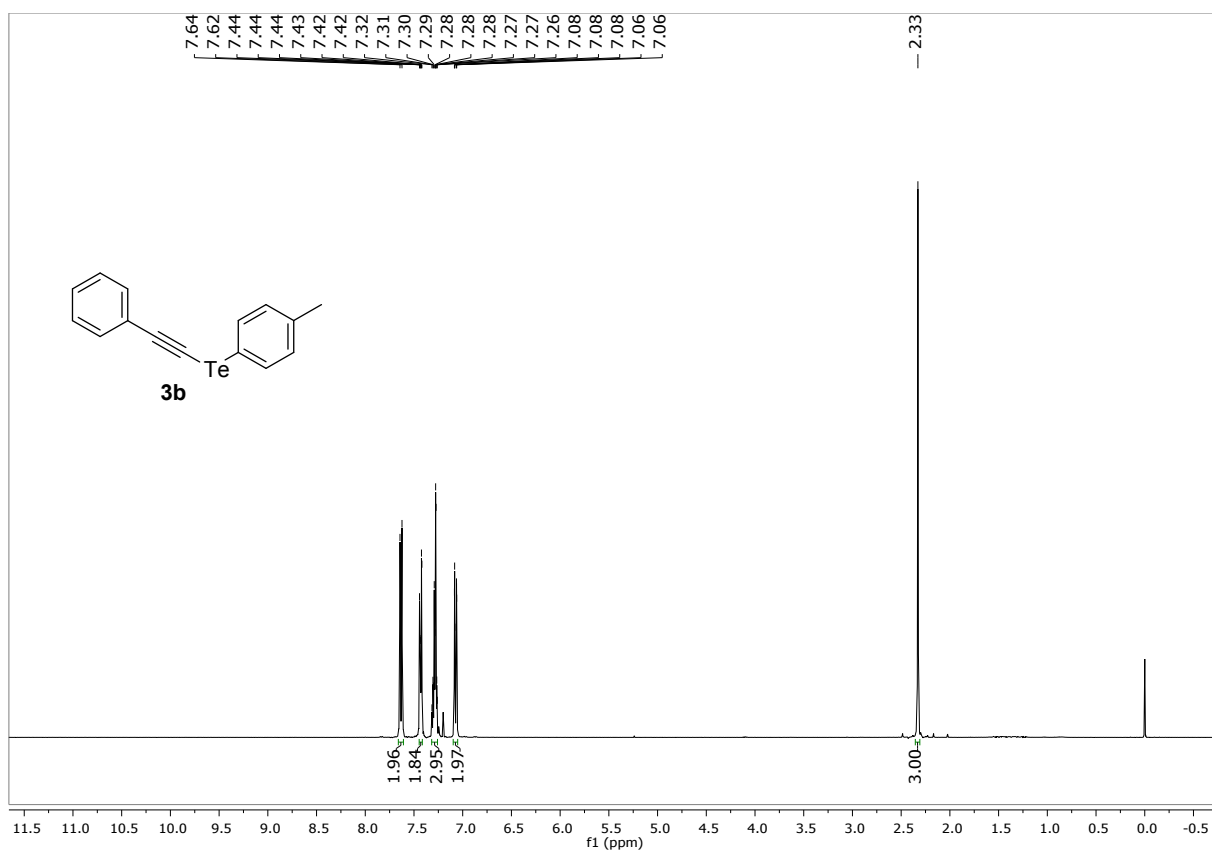


Figure 3. ^1H NMR (400 MHz) spectrum for compound **3b** in CDCl_3 .

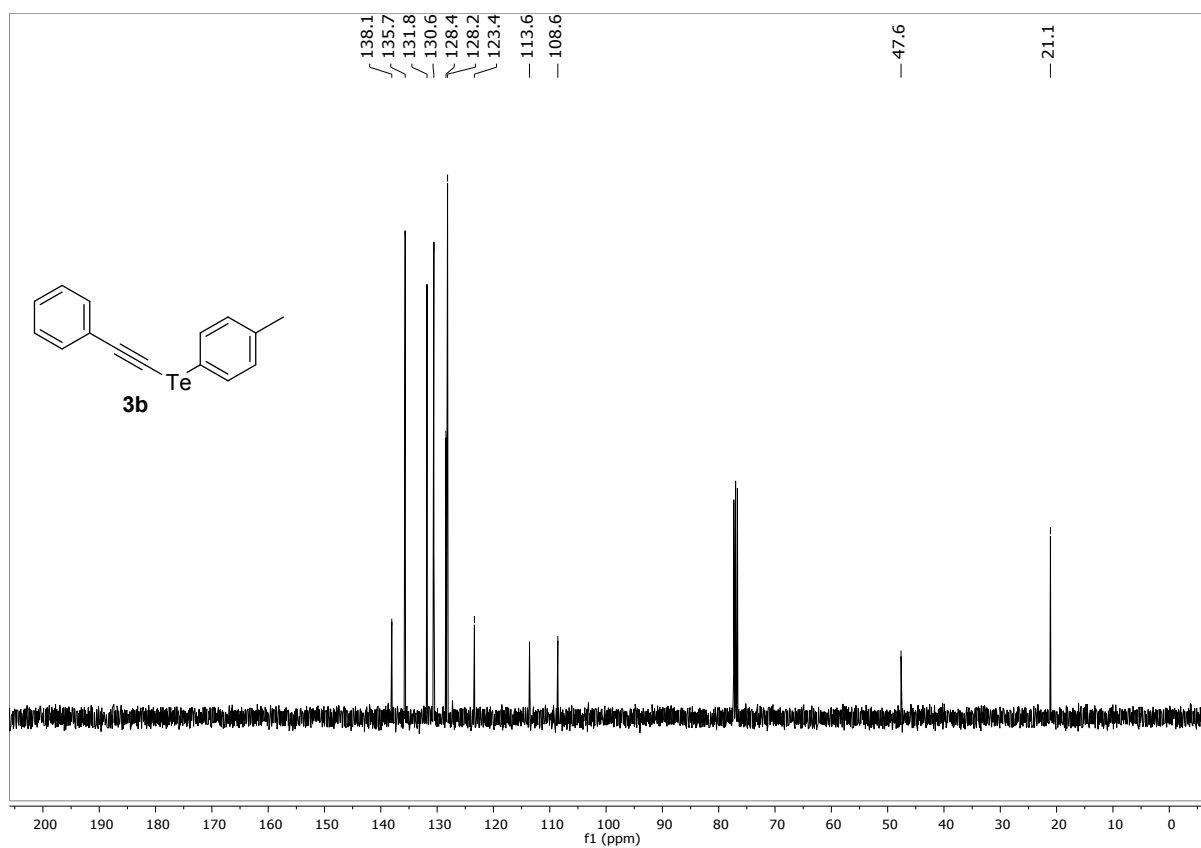


Figure 4. ^{13}C NMR (100 MHz) spectrum for compound **3b** in CDCl_3 .

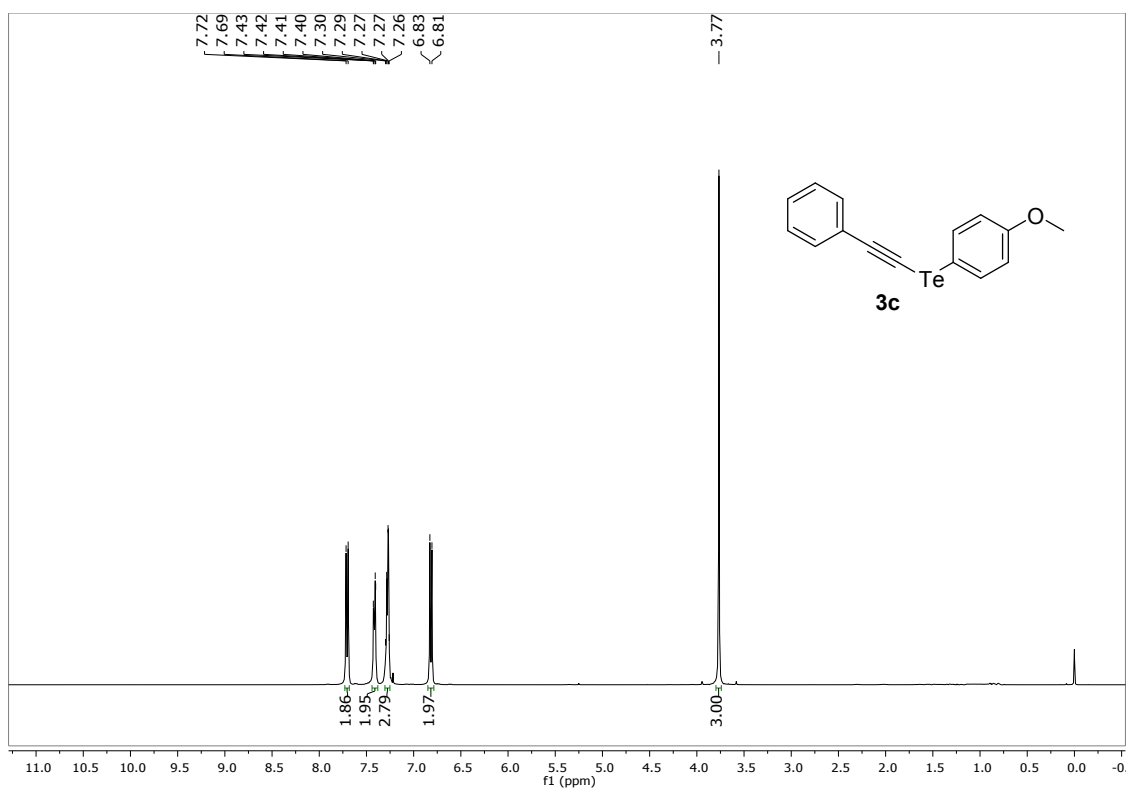


Figure 5. ¹H NMR (400 MHz) spectrum for compound **3c** in CDCl₃.

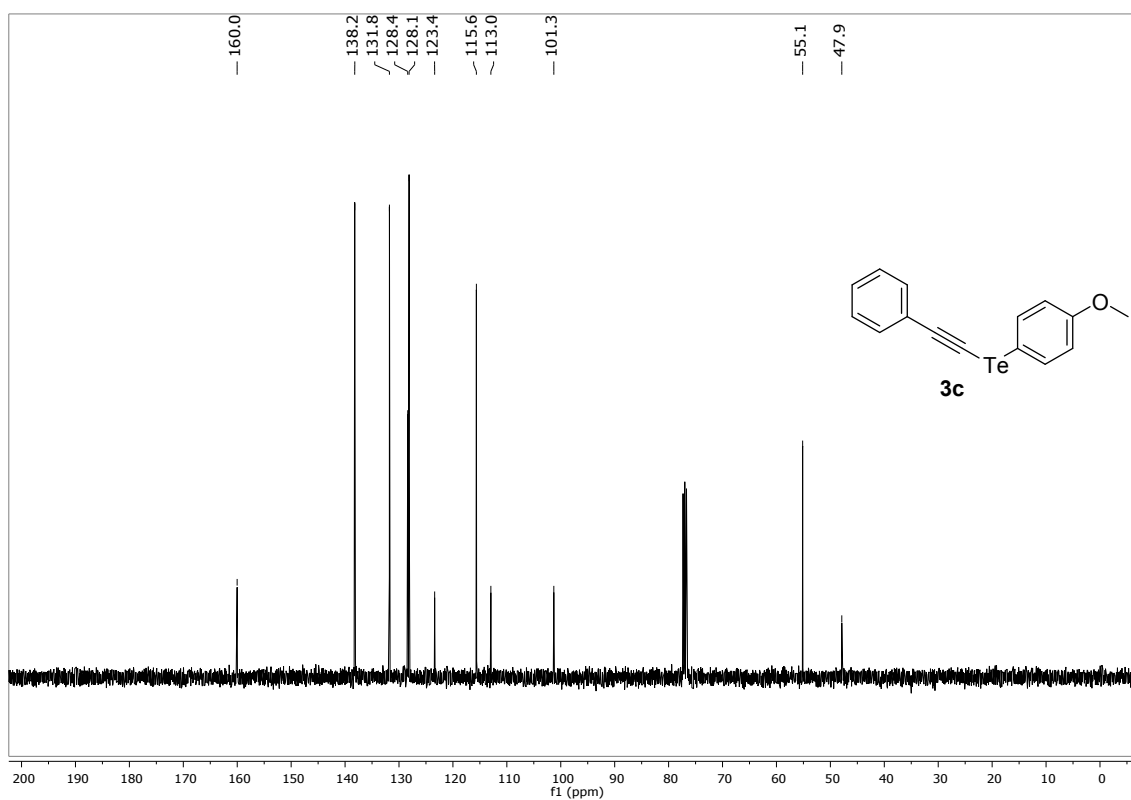


Figure 6. ¹³C NMR (100 MHz) spectrum for compound **3c** in CDCl₃.

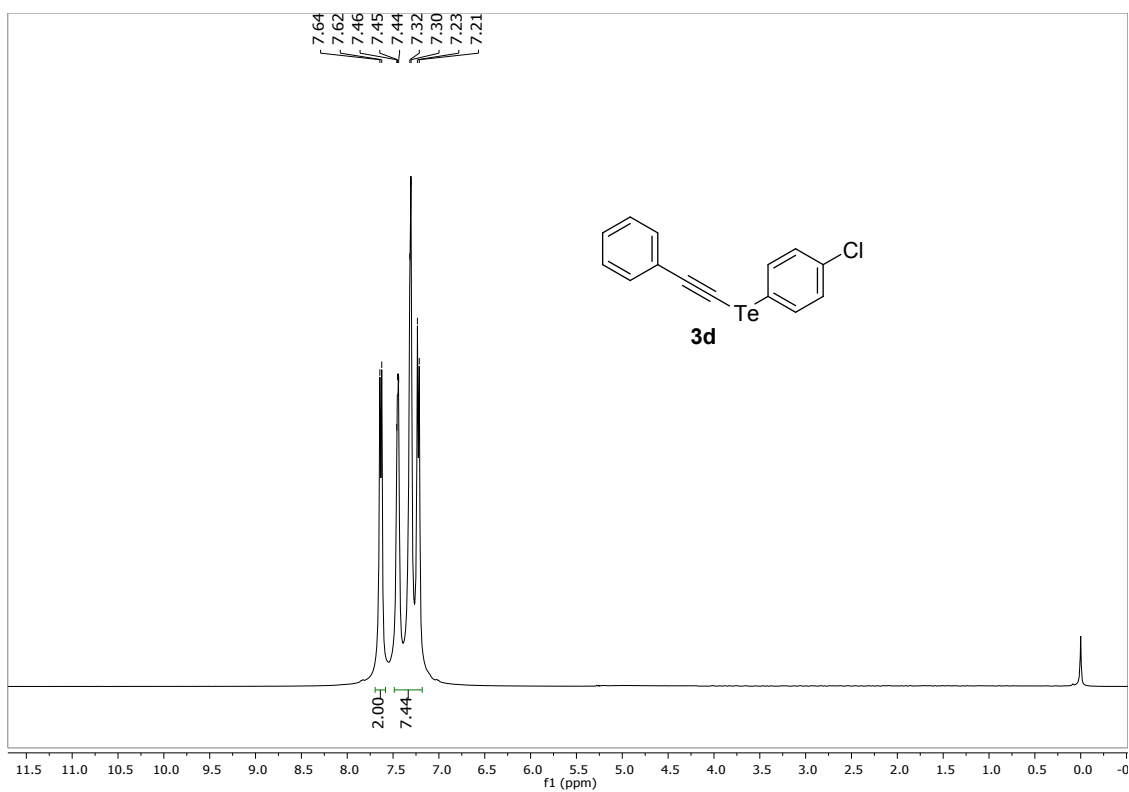


Figure 7. ^1H NMR (400 MHz) spectrum for compound **3d** in CDCl_3 .

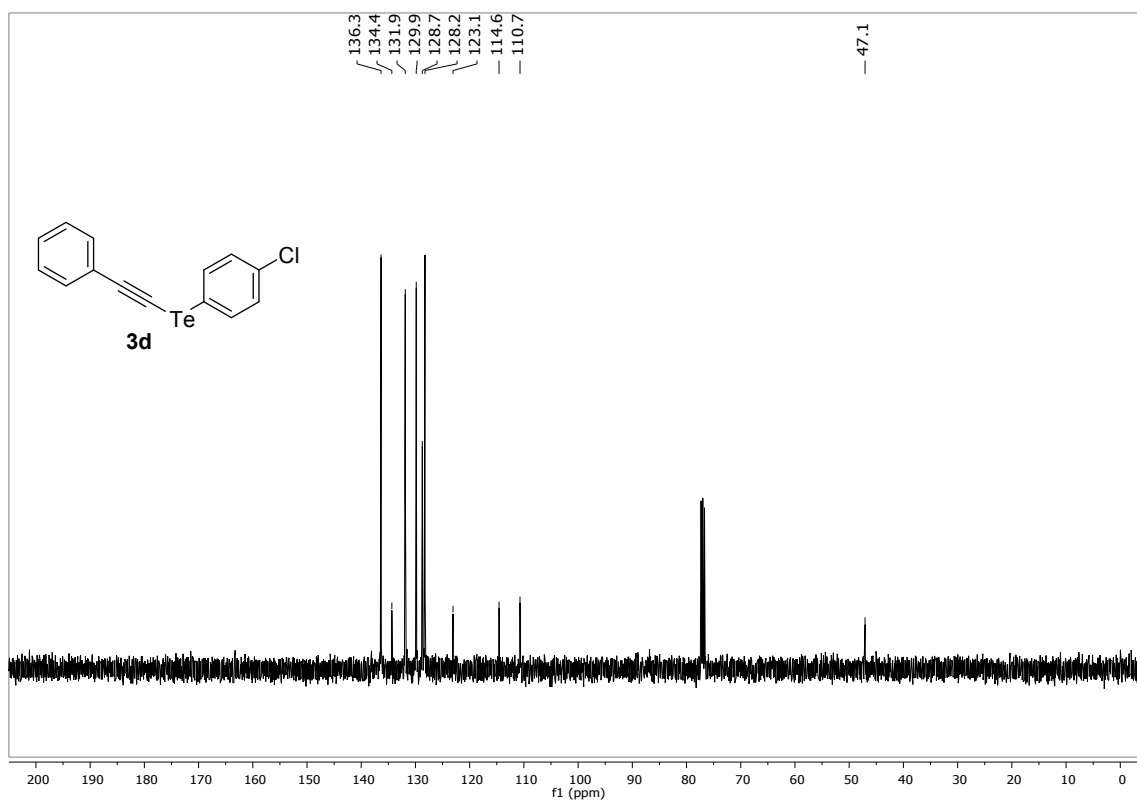


Figure 8. ^{13}C NMR (100 MHz) spectrum for compound **3d** in CDCl_3 .

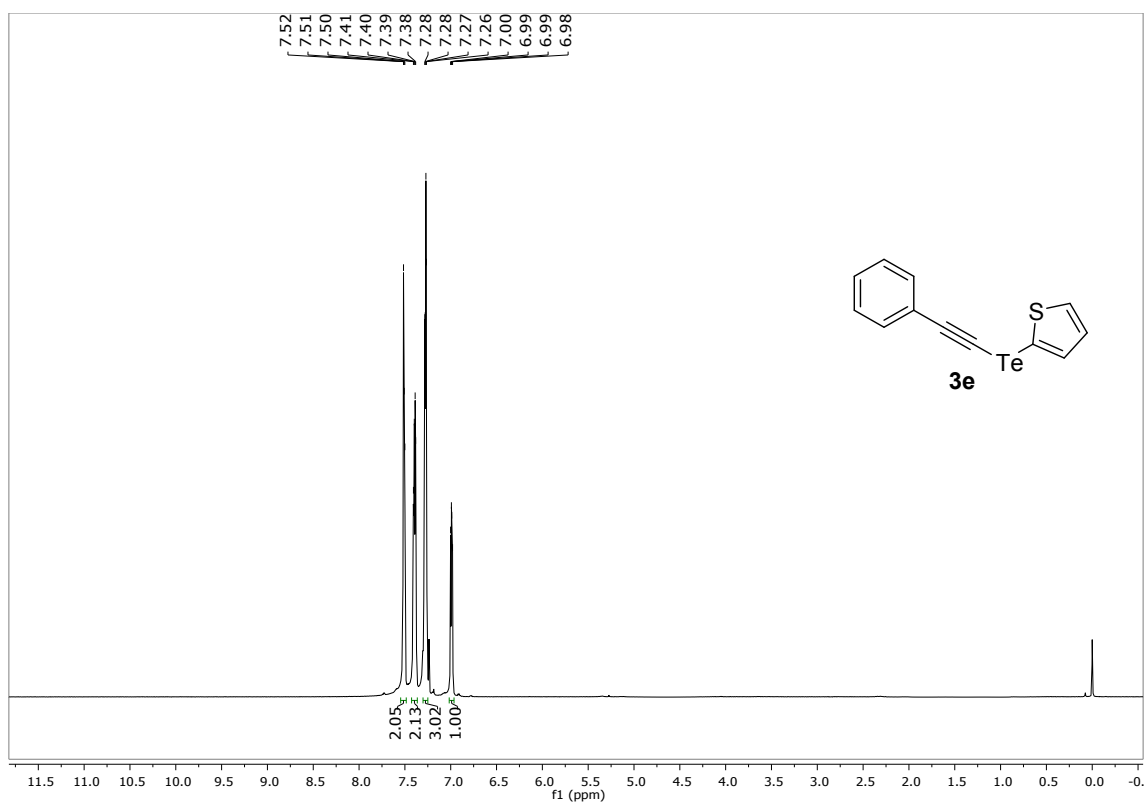


Figure 9. ^1H NMR (400 MHz) spectrum for compound **3e** in CDCl_3 .

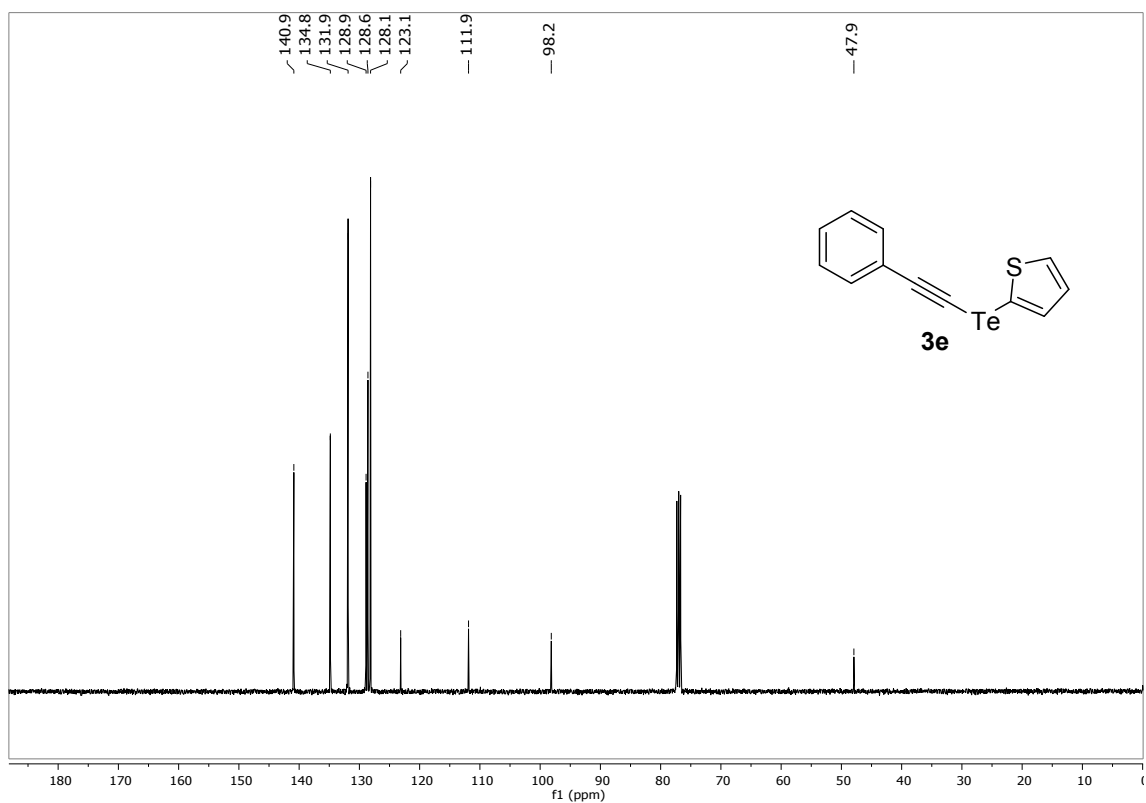


Figure 10. ^{13}C NMR (100 MHz) spectrum for compound **3e** in CDCl_3 .

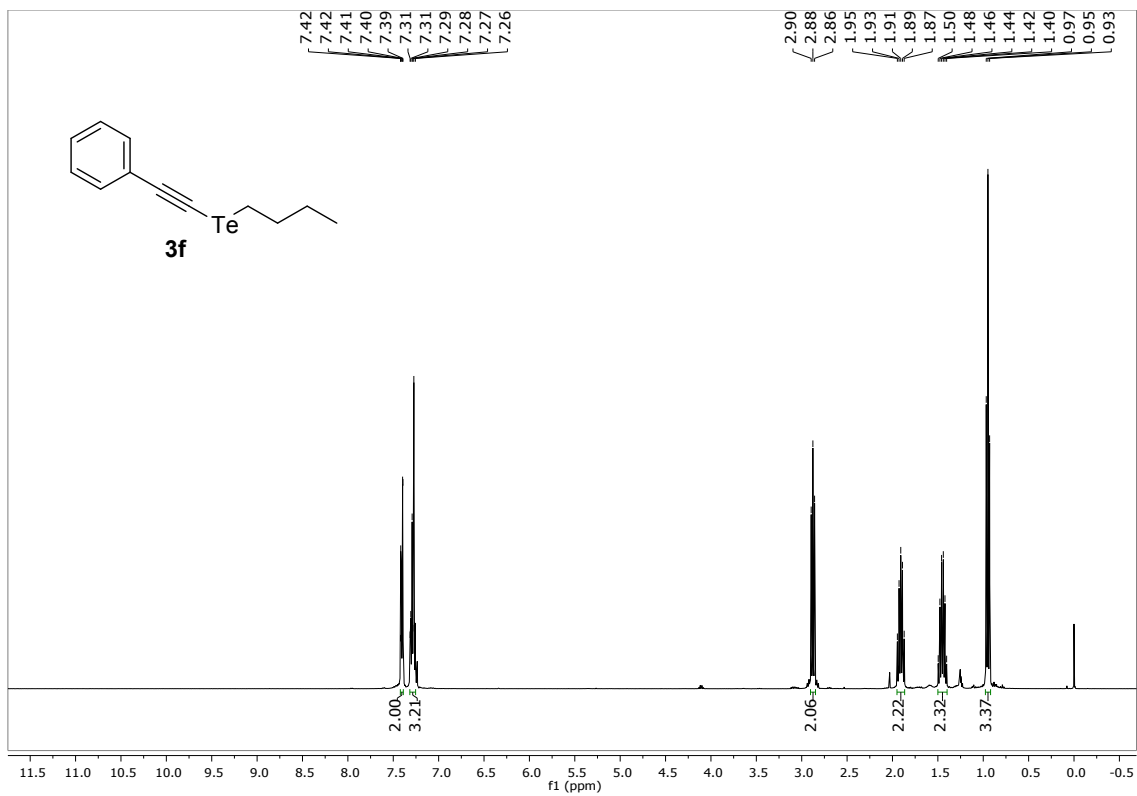


Figure 11. ¹H NMR (400 MHz) spectrum for compound **3f** in CDCl₃.

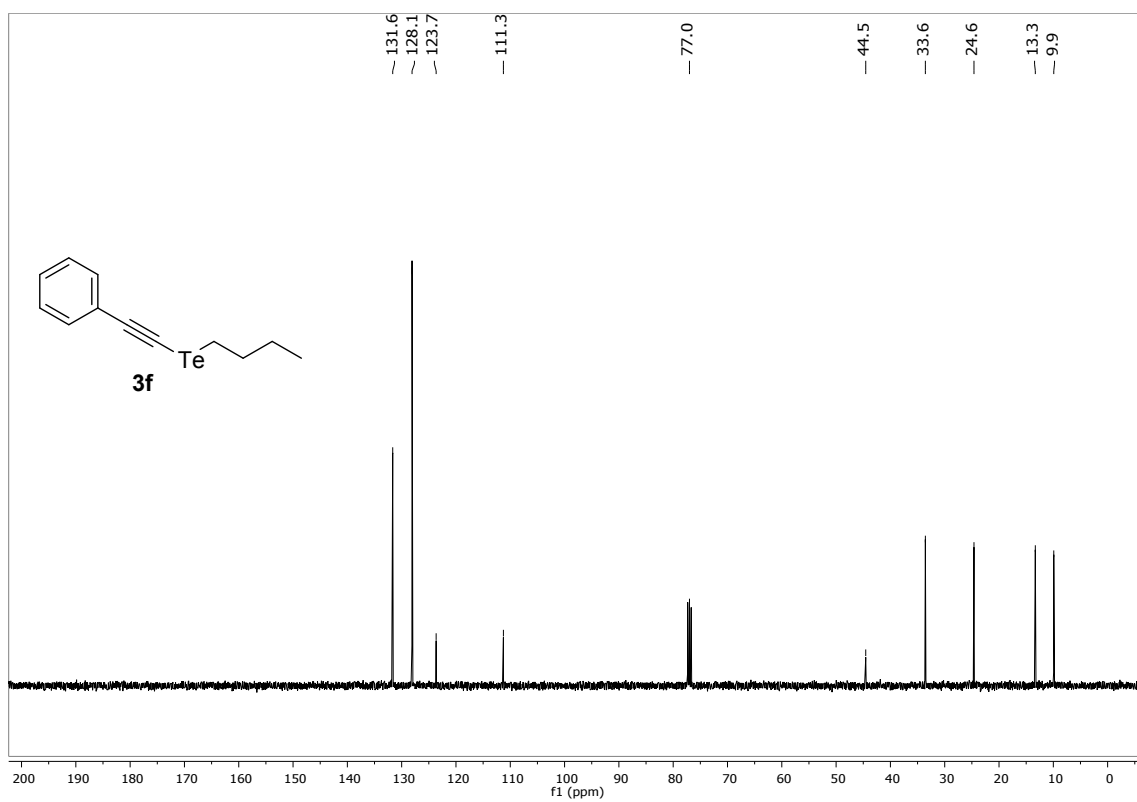


Figure 12. ¹³C NMR (100 MHz) spectrum for compound **3f** in CDCl₃.

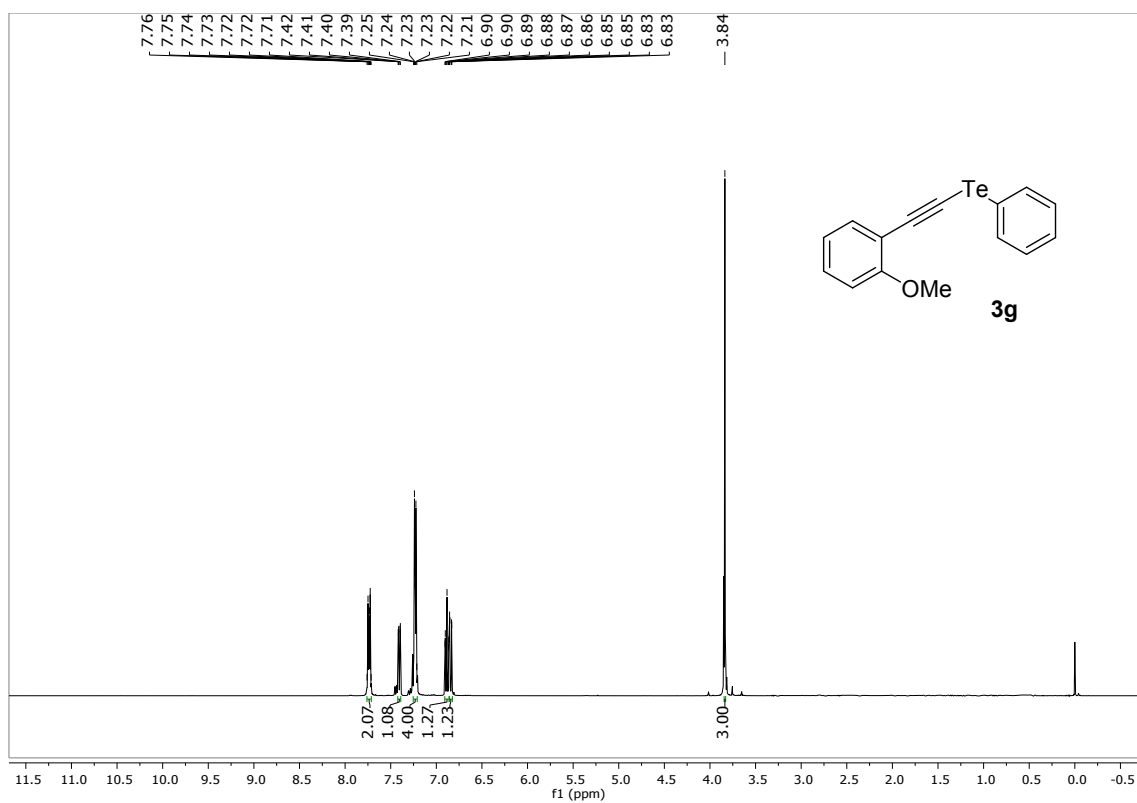


Figure 13. ^1H NMR (400 MHz) spectrum for compound **3g** in CDCl_3 .

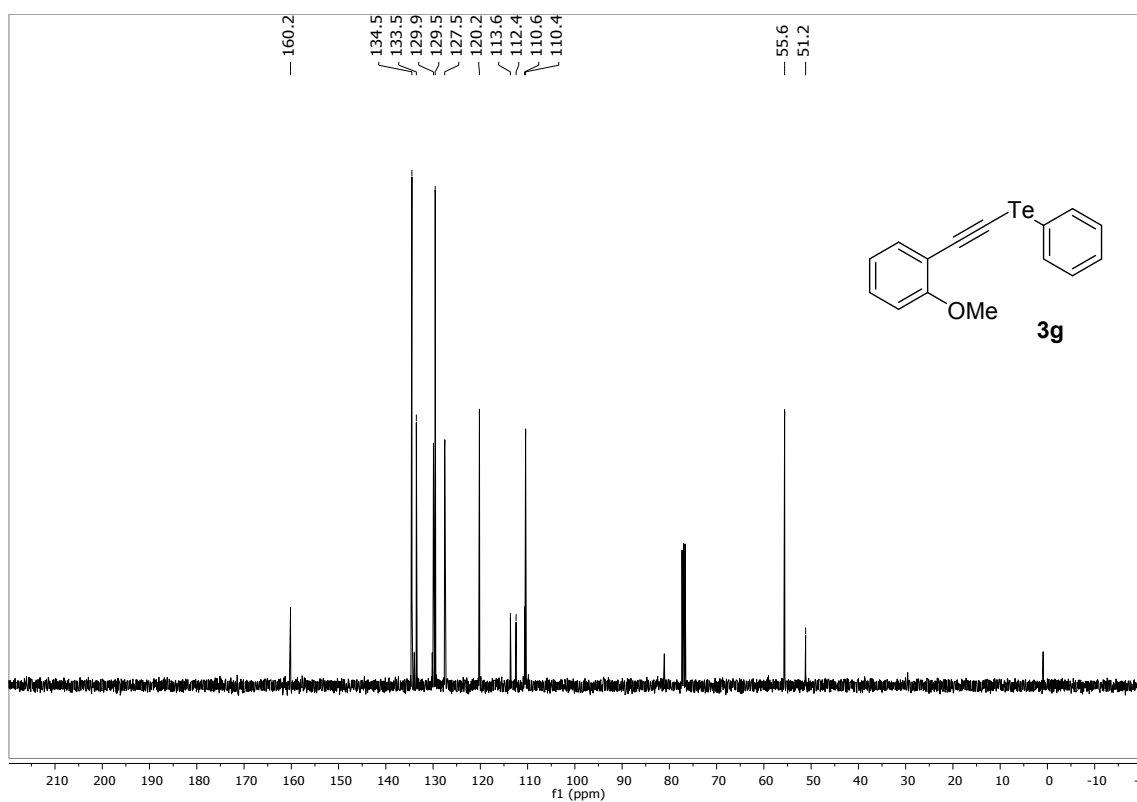


Figure 14. ^{13}C NMR (100 MHz) spectrum for compound **3g** in CDCl_3 .

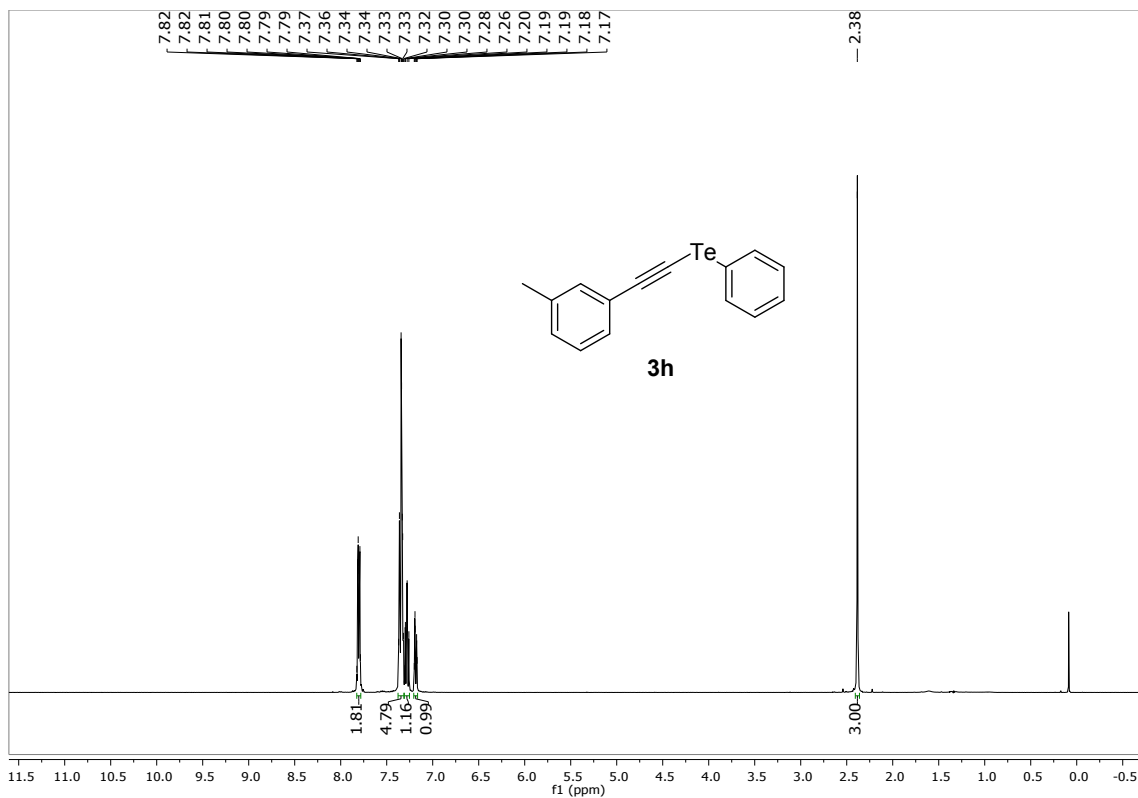


Figure 15. ¹H NMR (400 MHz) spectrum for compound **3h** in CDCl₃.

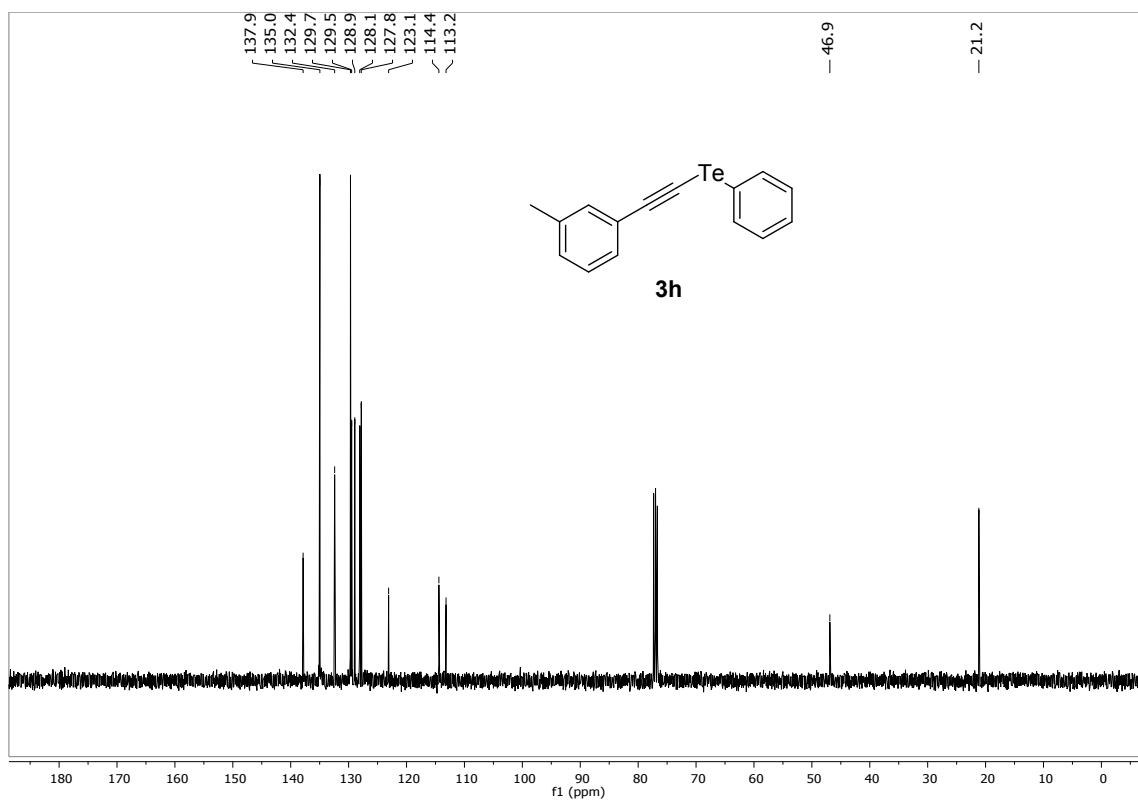


Figure 16. ¹³C NMR (100 MHz) spectrum for compound **3h** in CDCl₃.

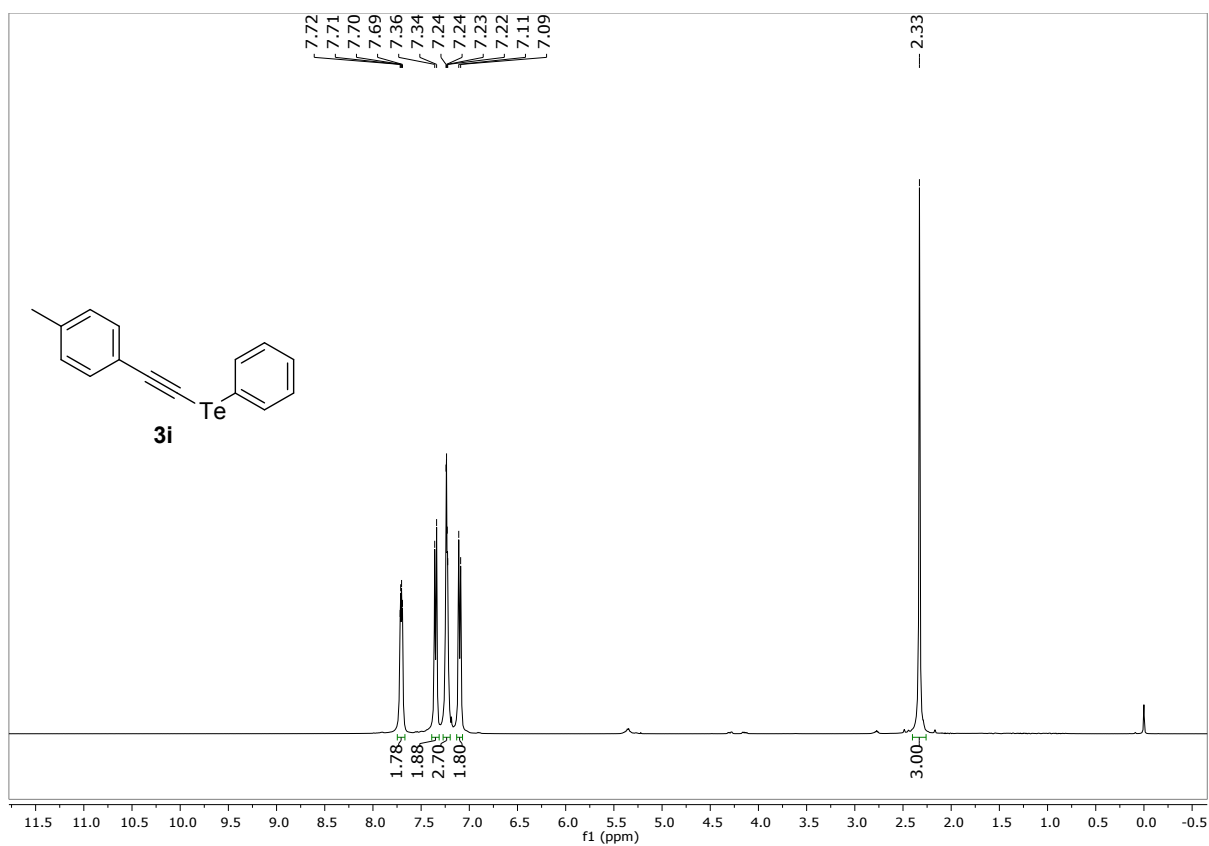


Figure 17. ¹H NMR (400 MHz) spectrum for compound **3i** in CDCl₃.

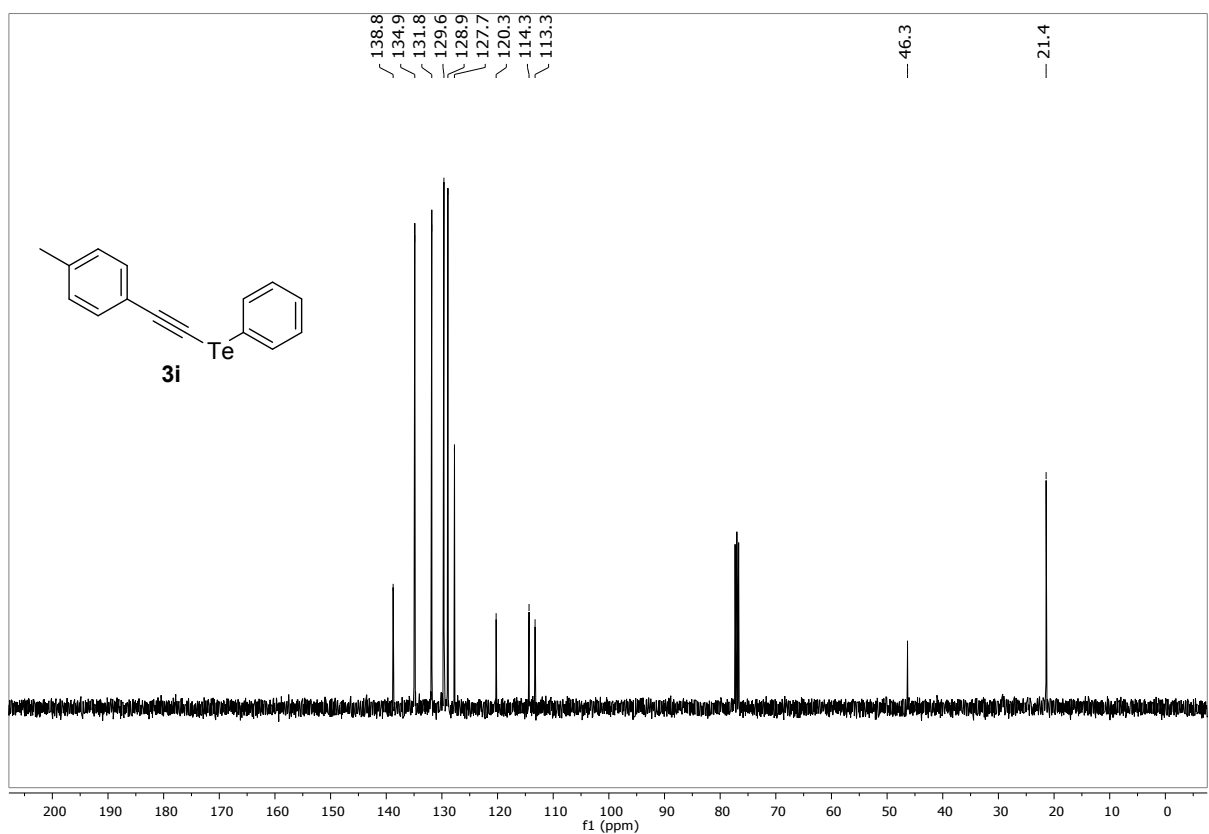


Figure 18. ¹³C NMR (100 MHz) spectrum for compound **3i** in CDCl₃.

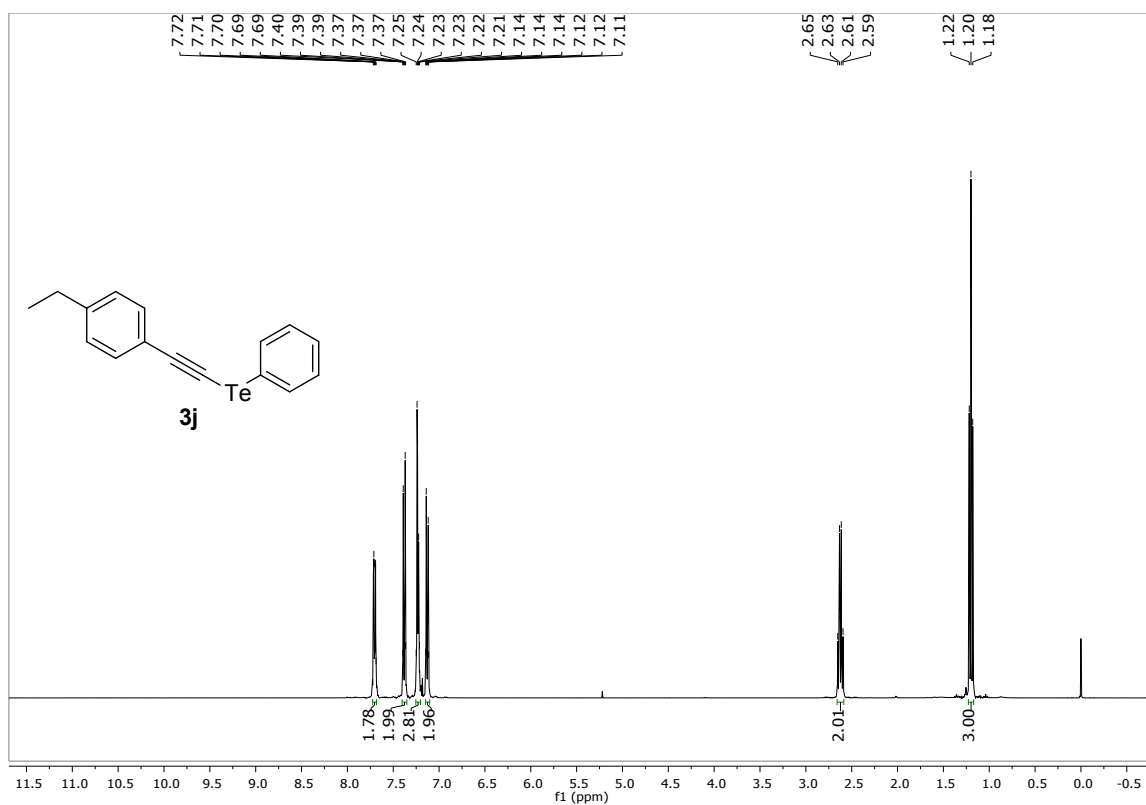


Figure 19. ¹H NMR (400 MHz) spectrum for compound **3j** in CDCl₃.

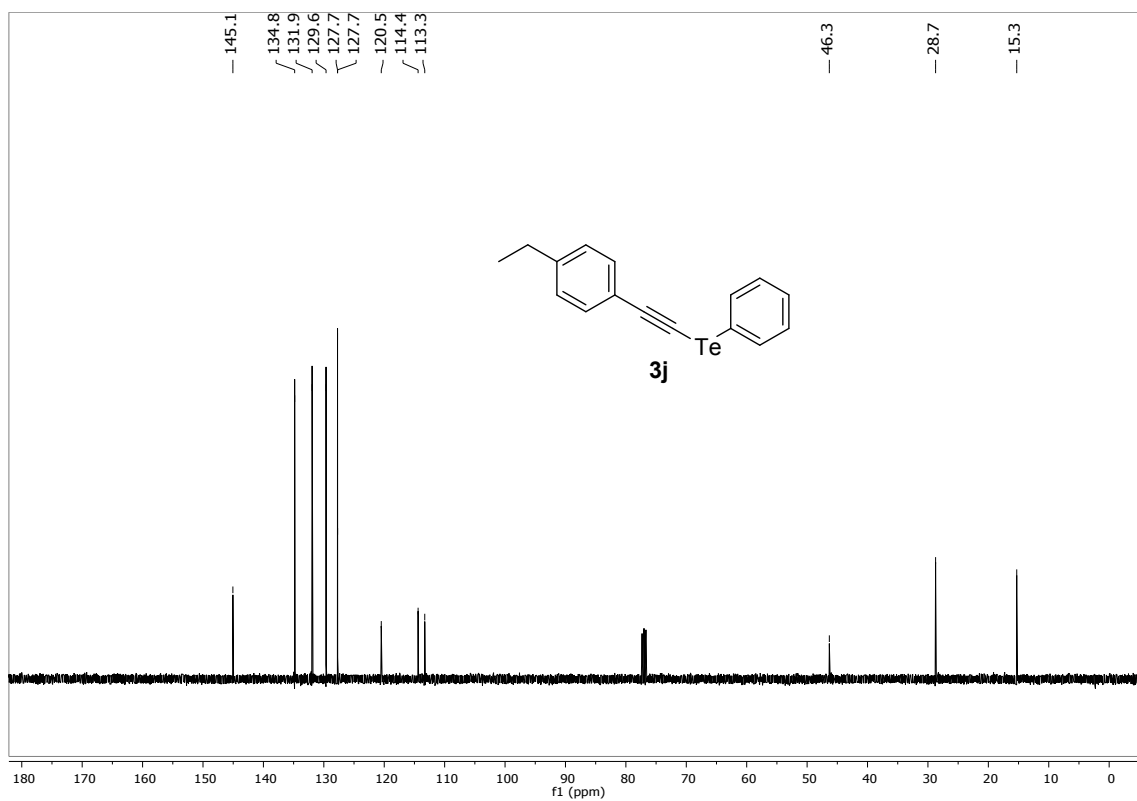


Figure 20. ¹³C NMR (100 MHz) spectrum for compound **3j** in CDCl₃.

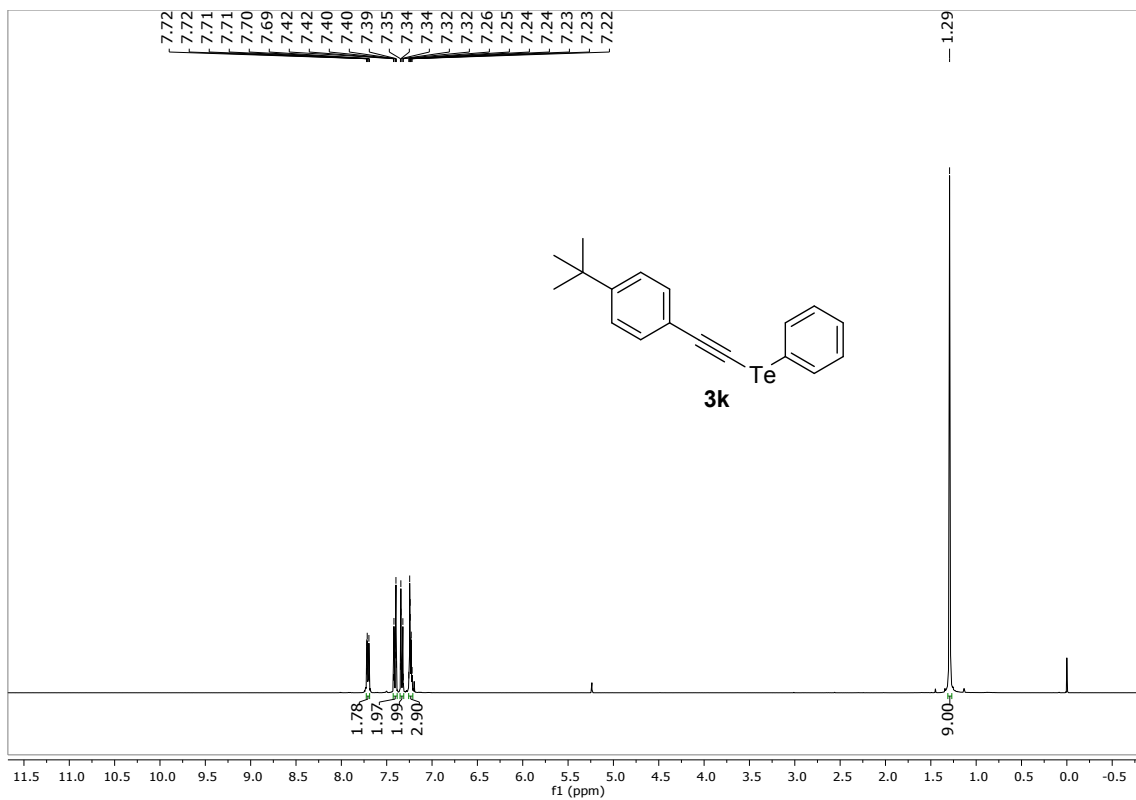


Figure 21. ^1H NMR (400 MHz) spectrum for compound **3k** in CDCl_3 .

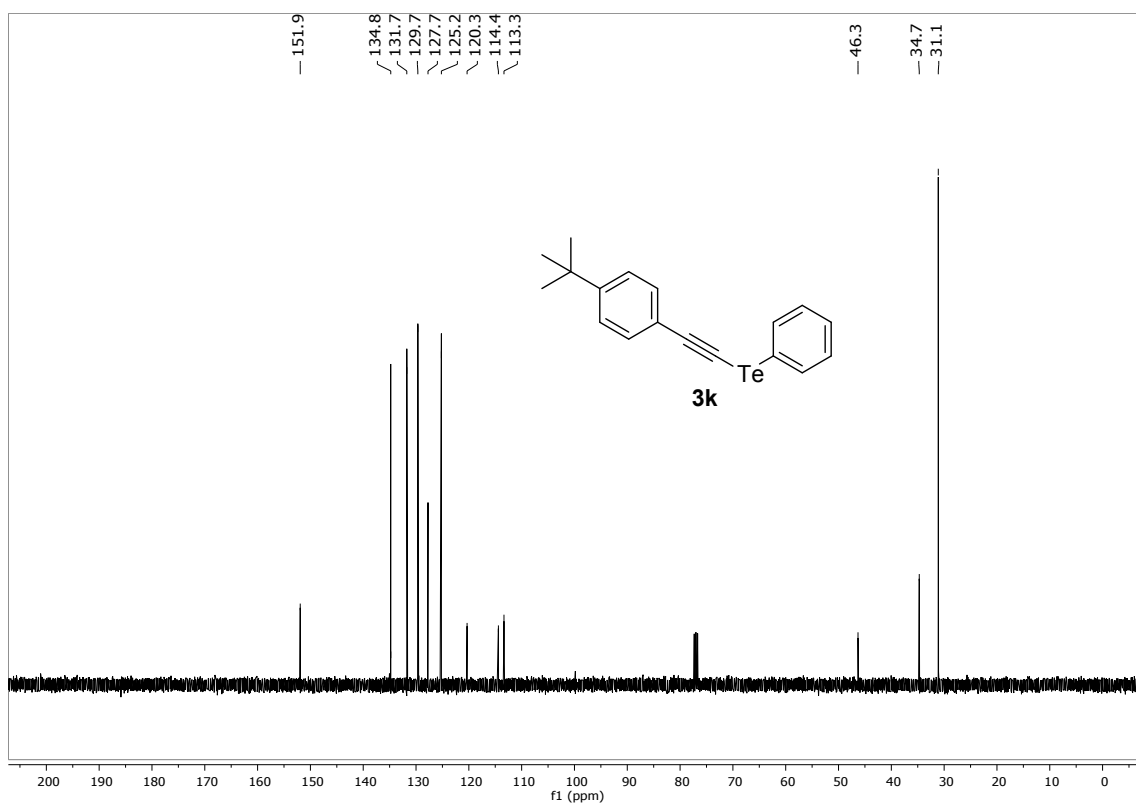


Figure 22. ^{13}C NMR (100 MHz) spectrum for compound **3k** in CDCl_3 .

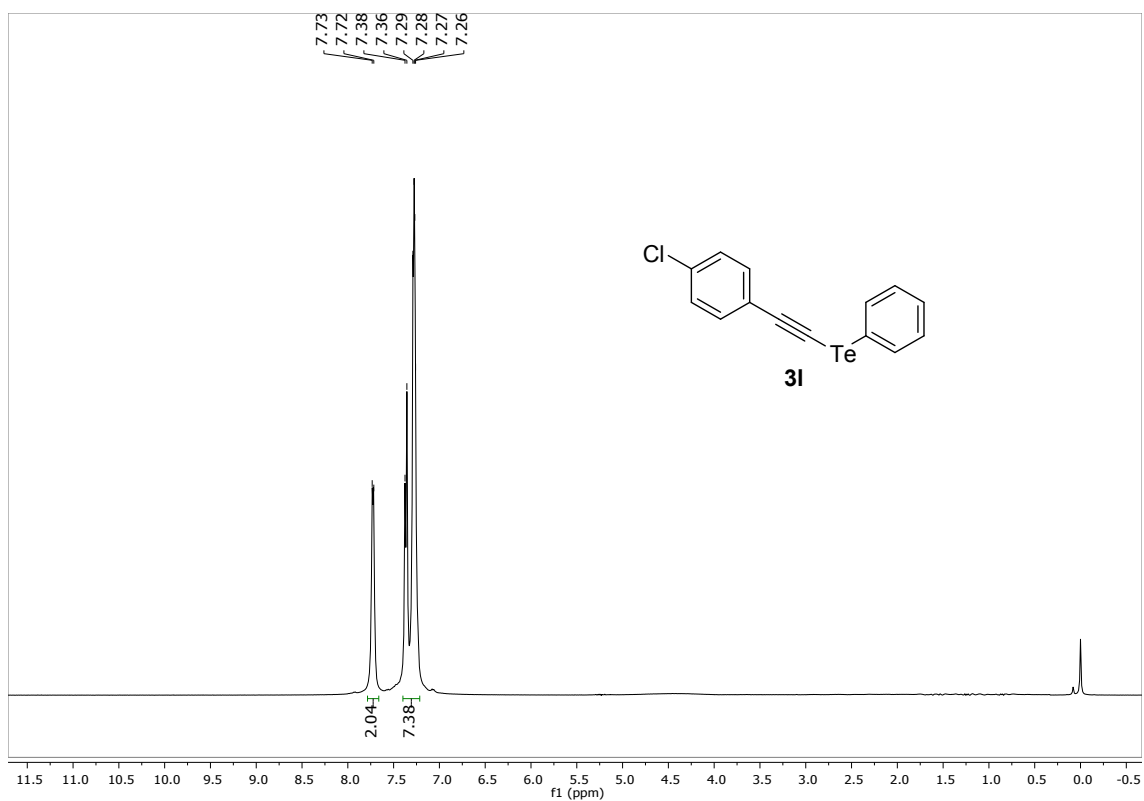


Figure 23. ¹H NMR (400 MHz) spectrum for compound **3I** in CDCl₃.

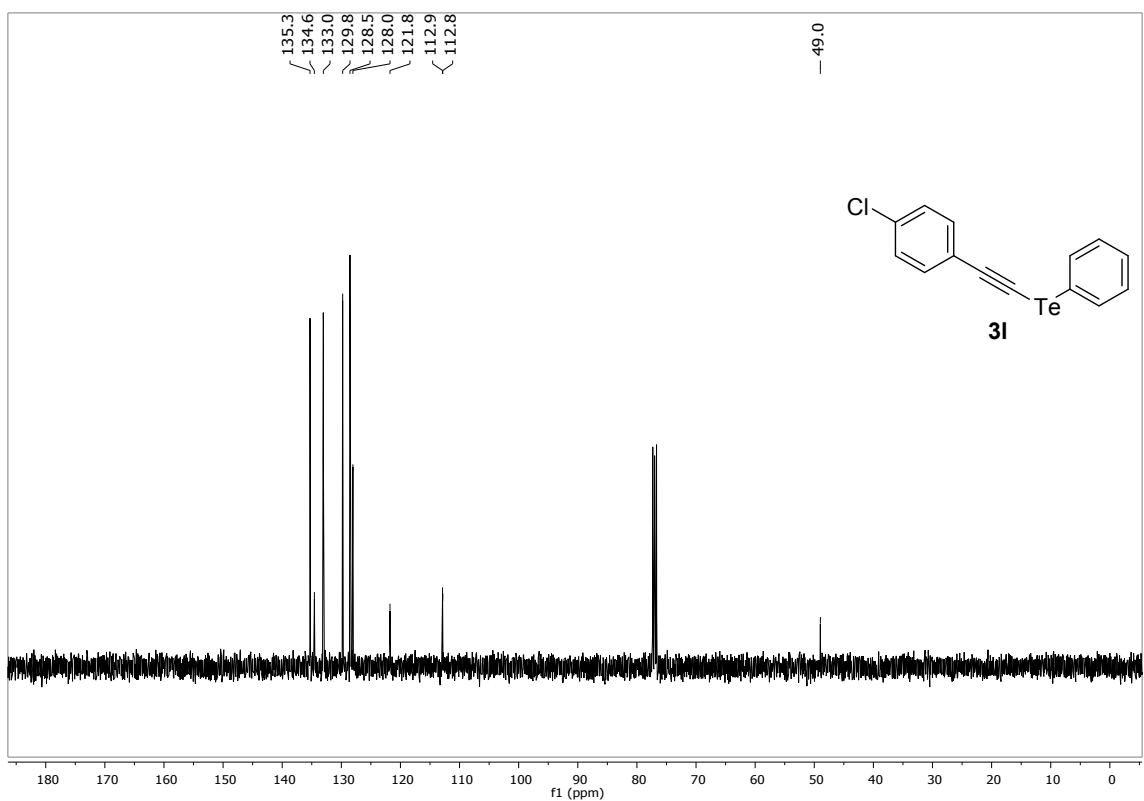


Figure 24. ¹³C NMR (100 MHz) spectrum for compound **3I** in CDCl₃.

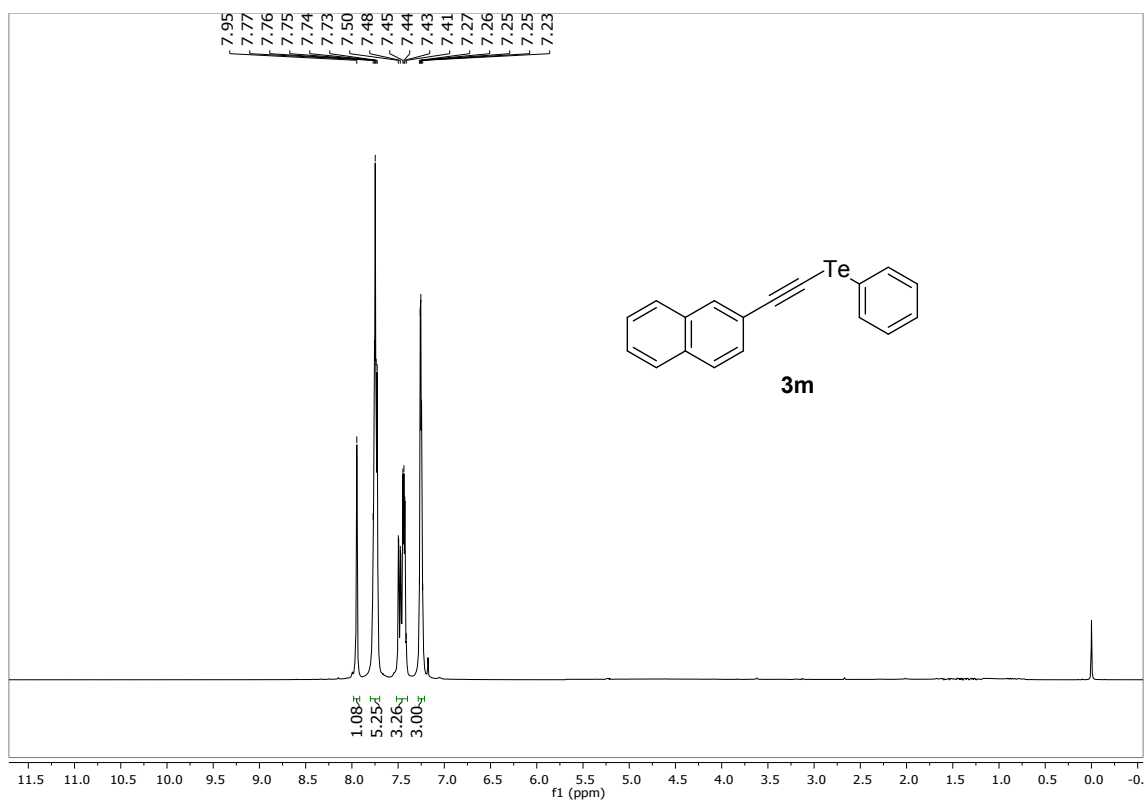


Figure 25. ^1H NMR (400 MHz) spectrum for compound **3m** in CDCl_3 .

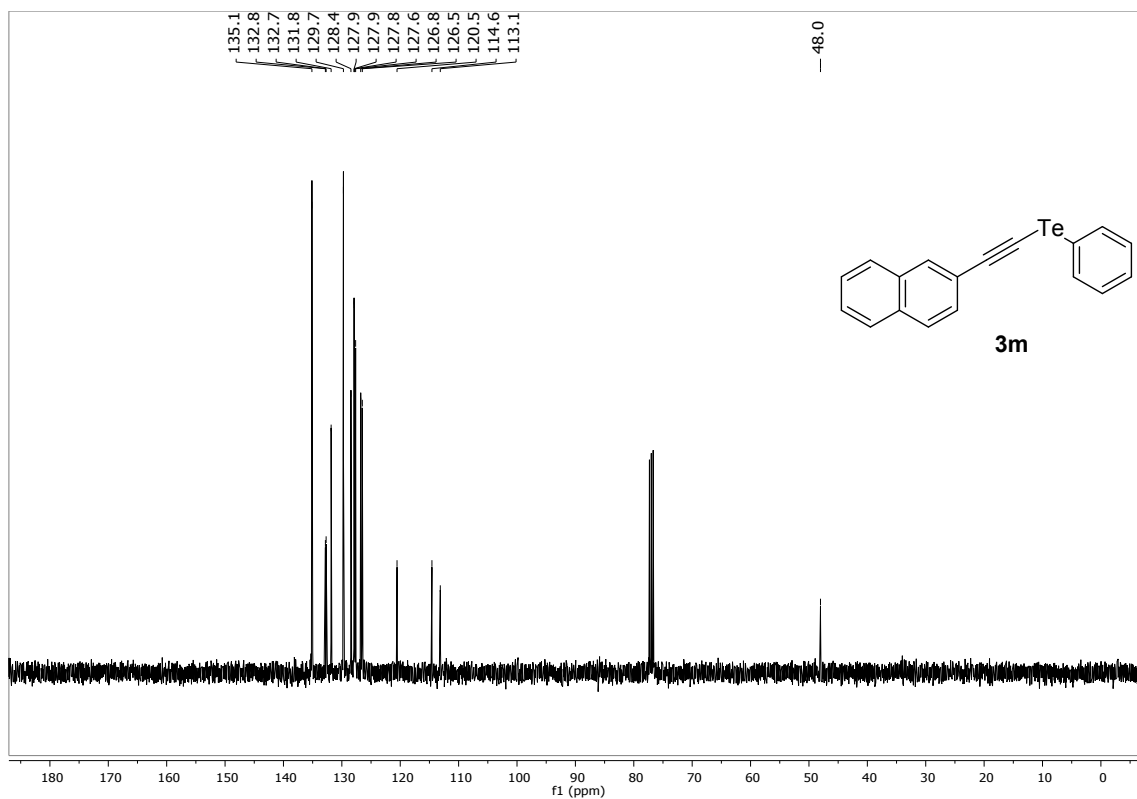


Figure 26. ^{13}C NMR (100 MHz) spectrum for compound **3m** in CDCl_3 .

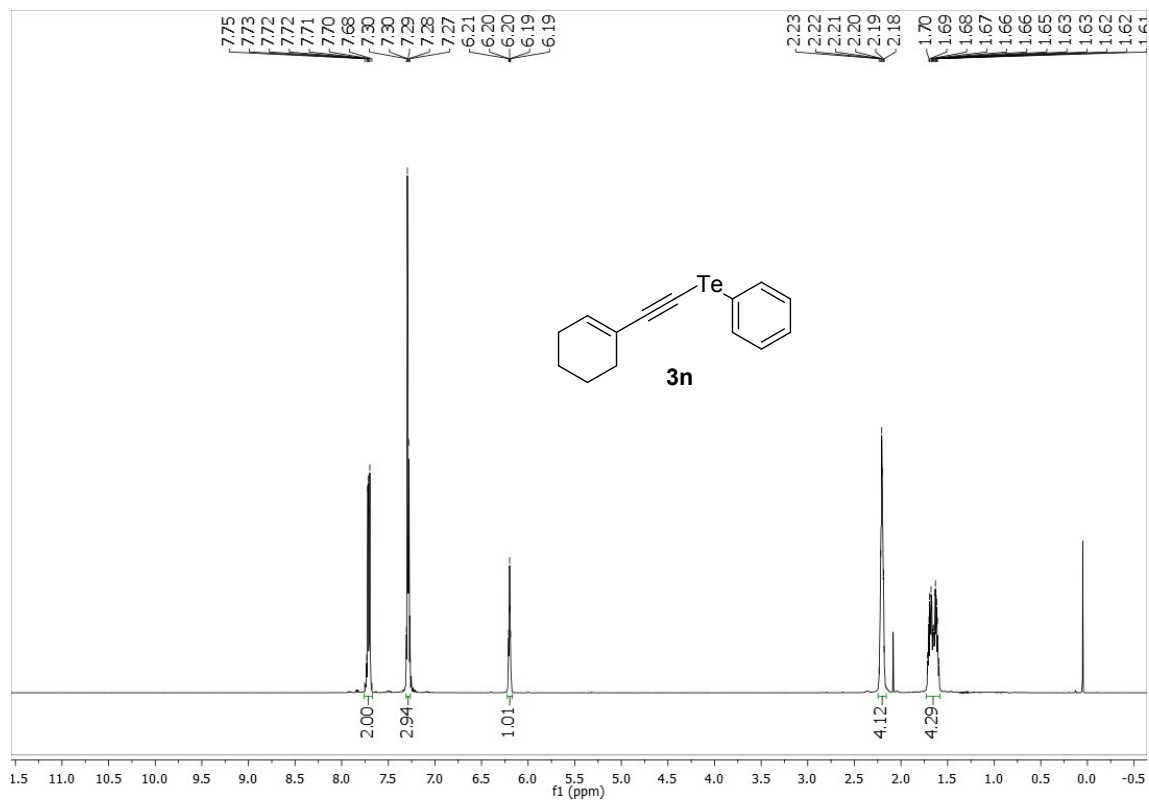


Figure 27. ¹H NMR (400 MHz) spectrum for compound **3n** in CDCl₃.

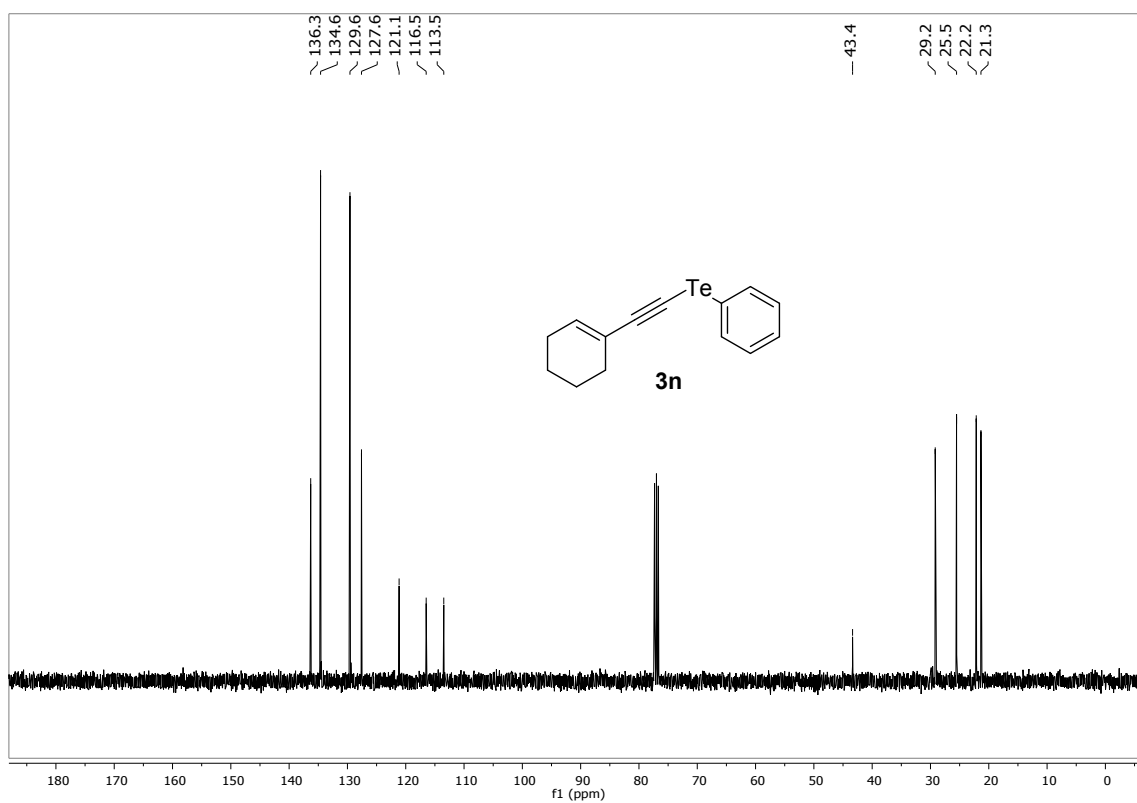


Figure 28. ¹³C NMR (100 MHz) spectrum for compound **3n** in CDCl₃.

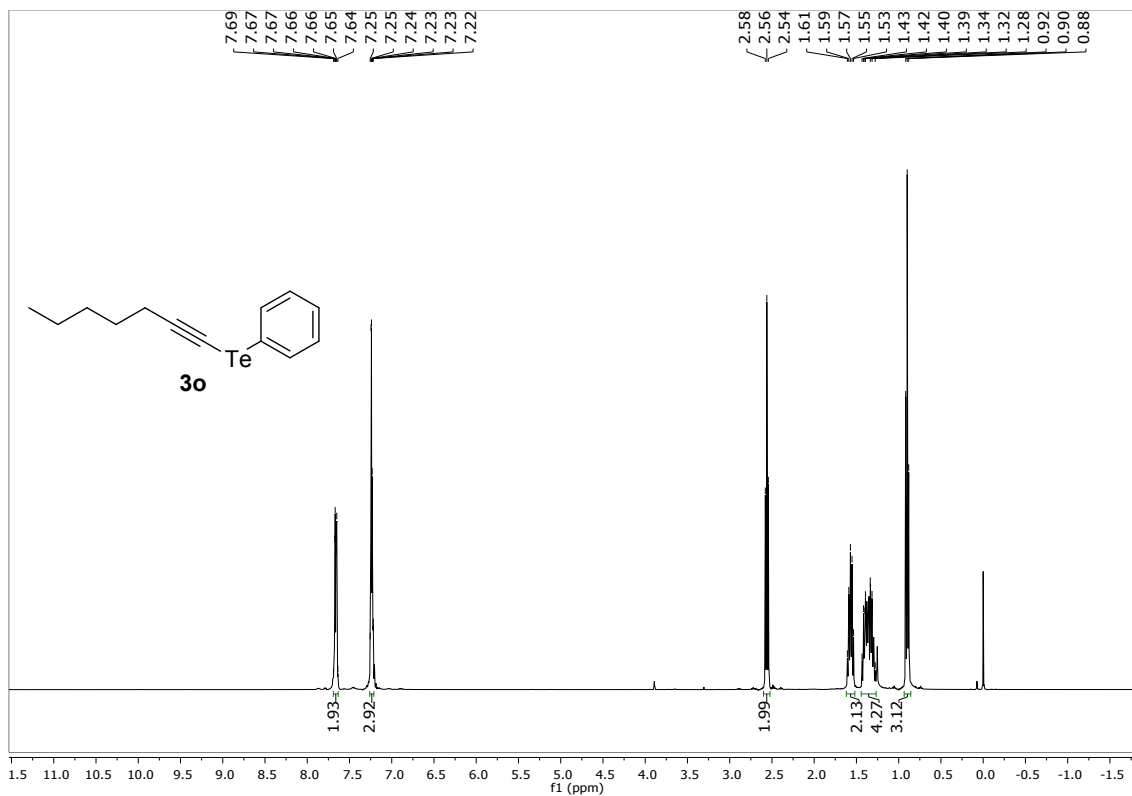


Figure 29. ¹H NMR (400 MHz) spectrum for compound **3o** in CDCl₃.

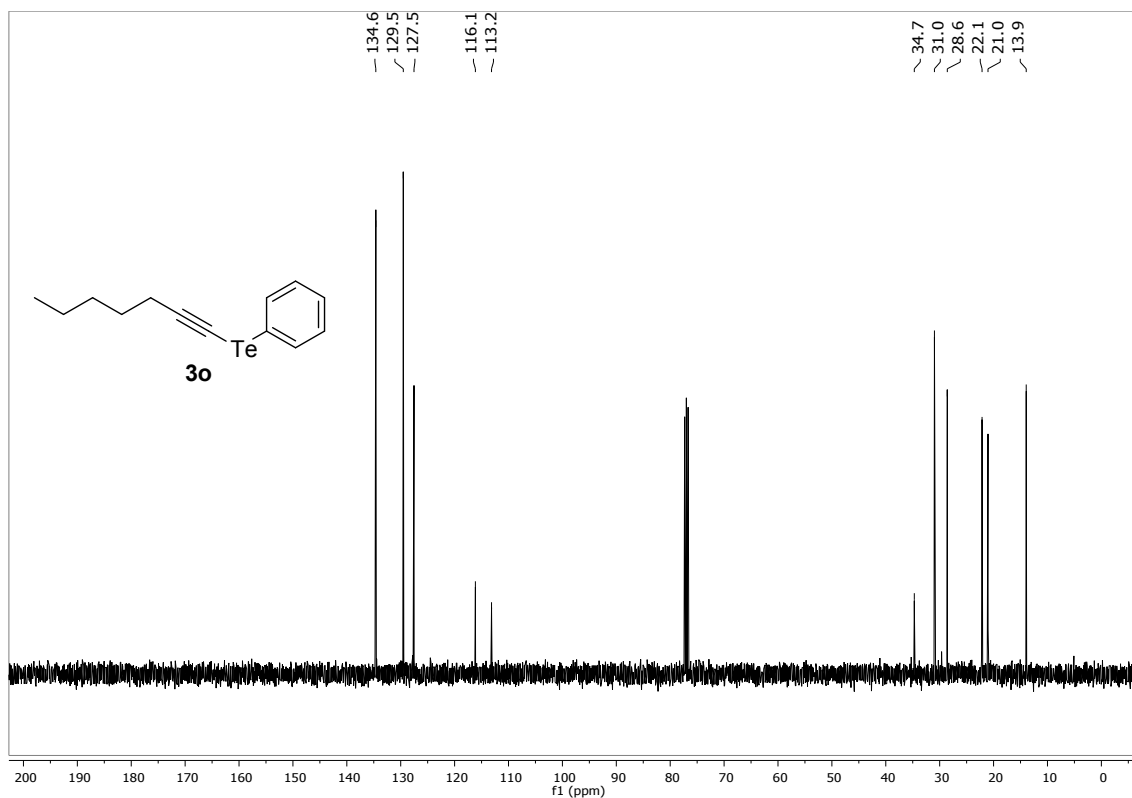


Figure 30. ¹³C NMR (100 MHz) spectrum for compound **3o** in CDCl₃.

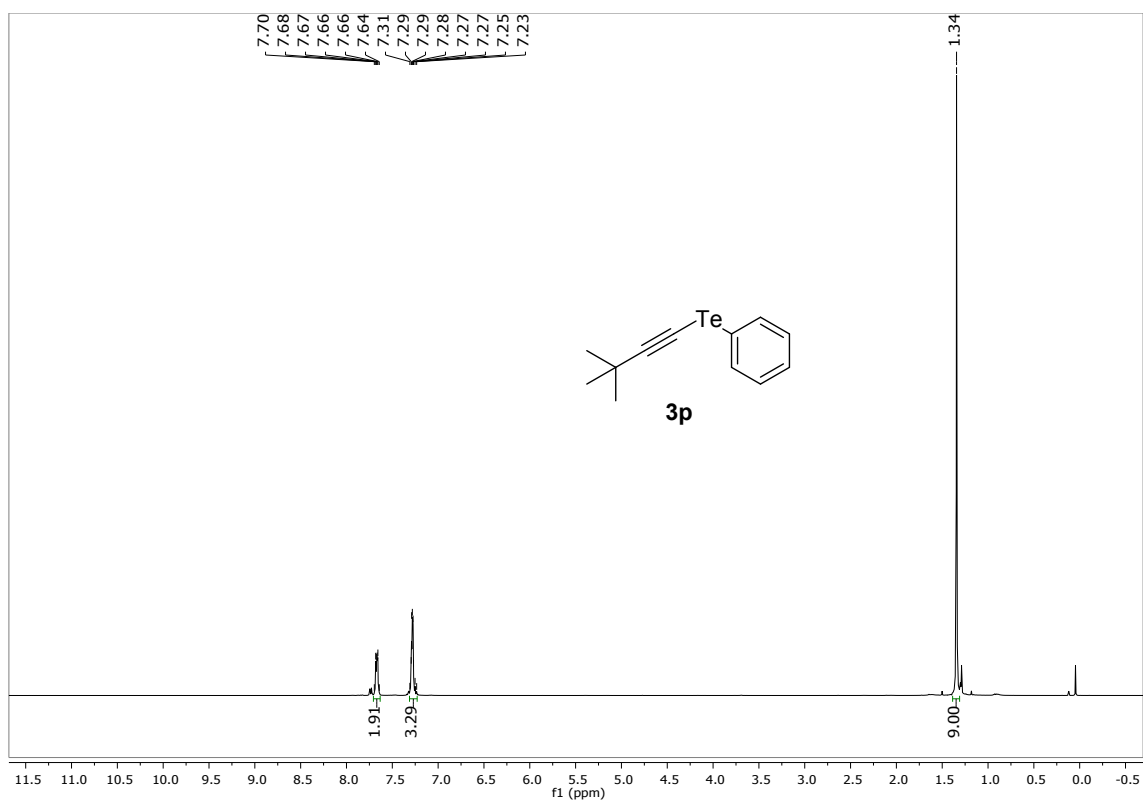


Figure 31. ^1H NMR (400 MHz) spectrum for compound **3p** in CDCl_3 .

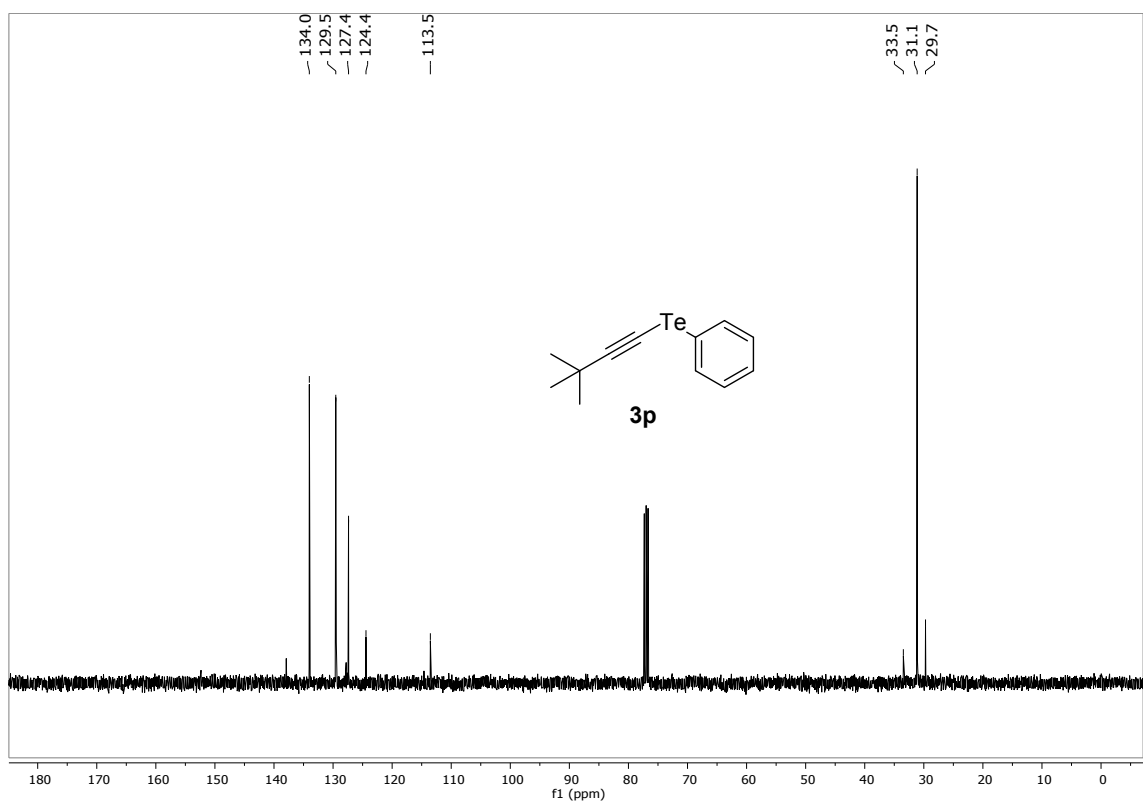
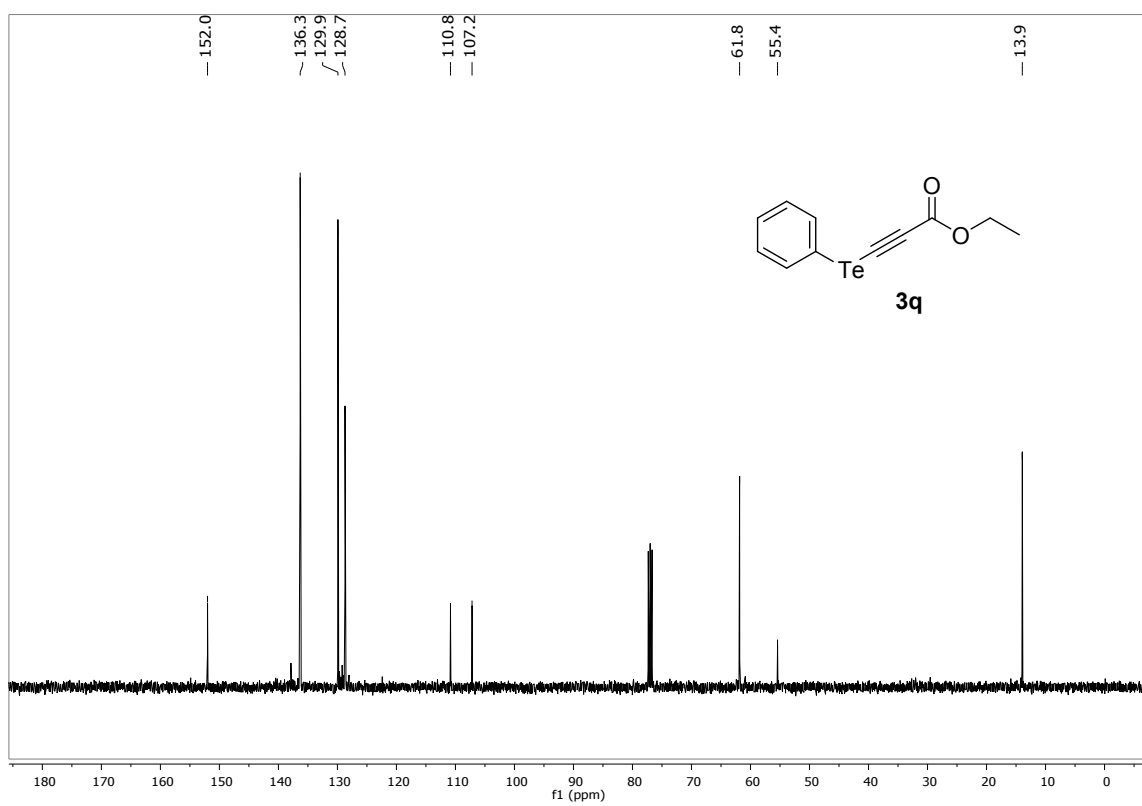
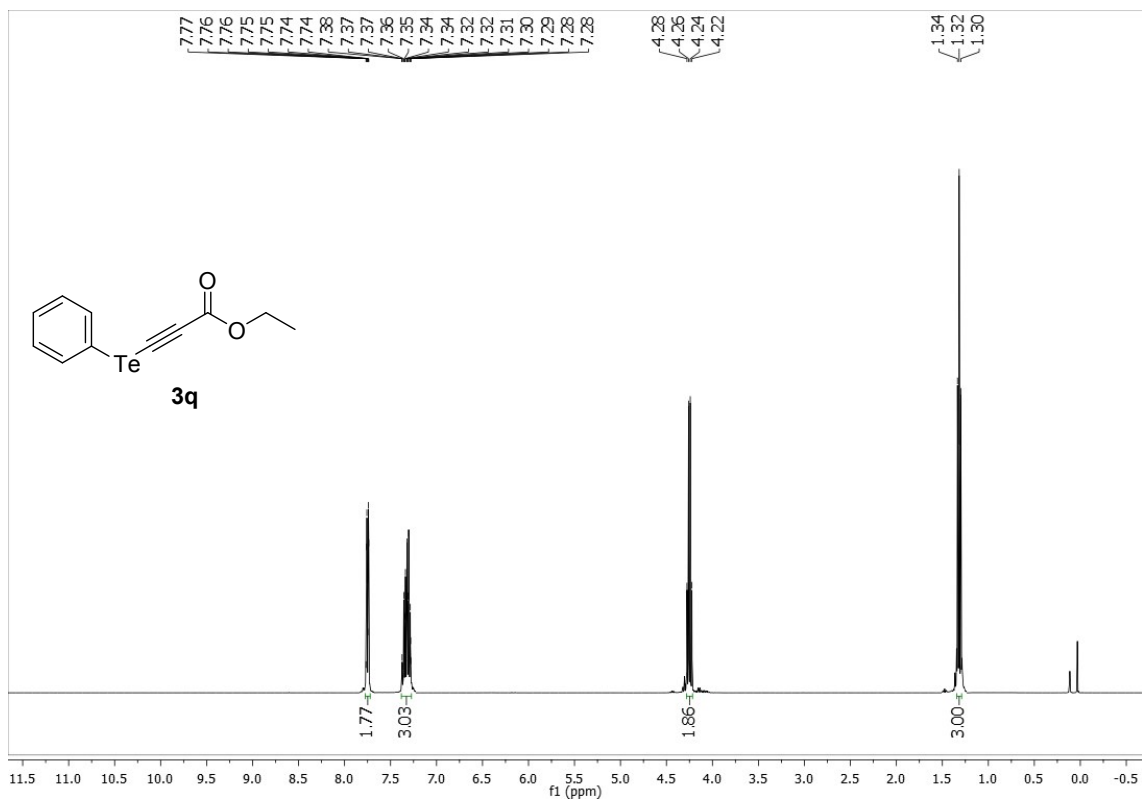


Figure 32. ^{13}C NMR (100 MHz) spectrum for compound **3p** in CDCl_3 .



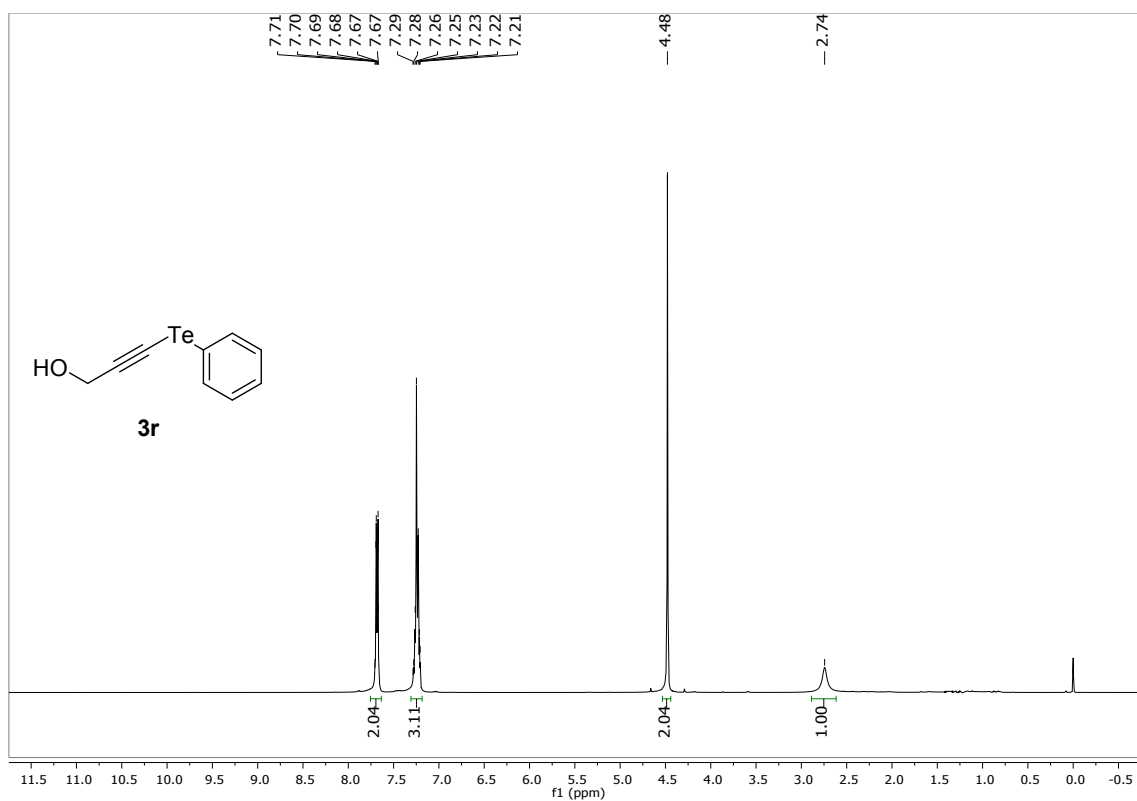


Figure 35. ¹H NMR (400 MHz) spectrum for compound **3r** in CDCl₃.

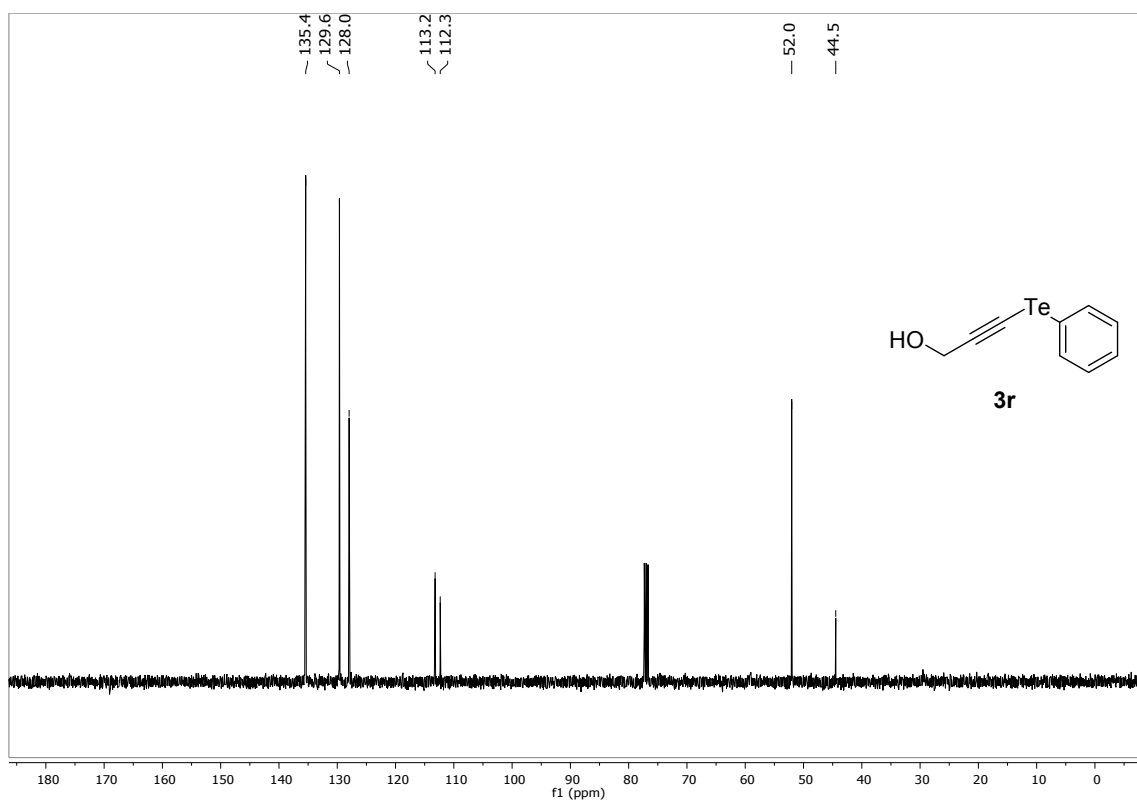


Figure 36. ¹³C NMR (100 MHz) spectrum for compound **3r** in CDCl₃.

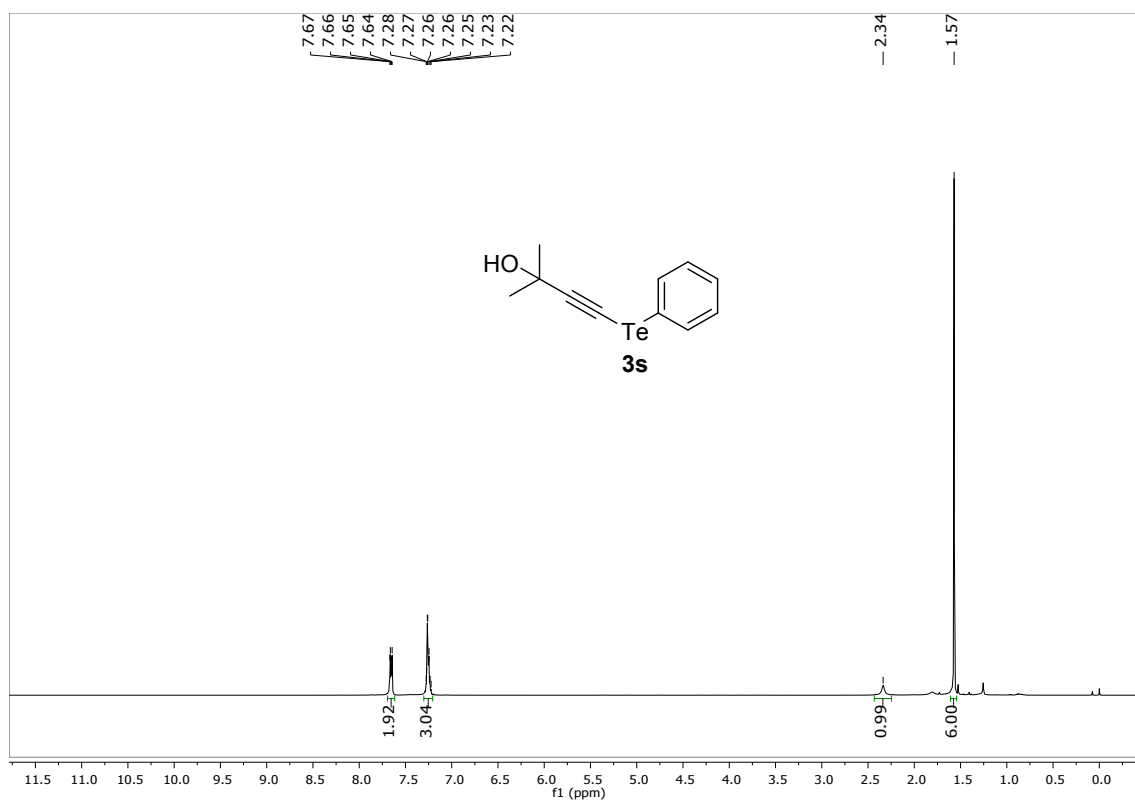


Figure 37. ^1H NMR (400 MHz) spectrum for compound **3s** in CDCl_3

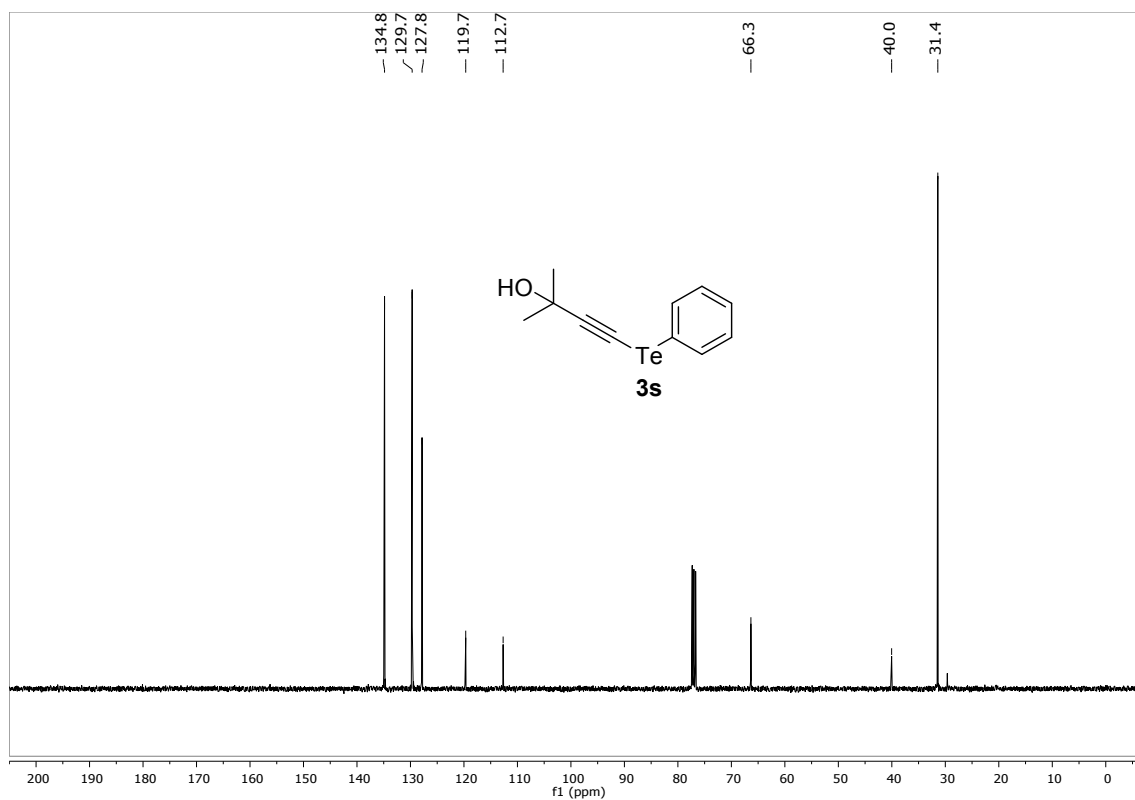


Figure 38. ^{13}C NMR (100 MHz) spectrum for compound **3s** in CDCl_3 .