

Electronic Supplementary Informations

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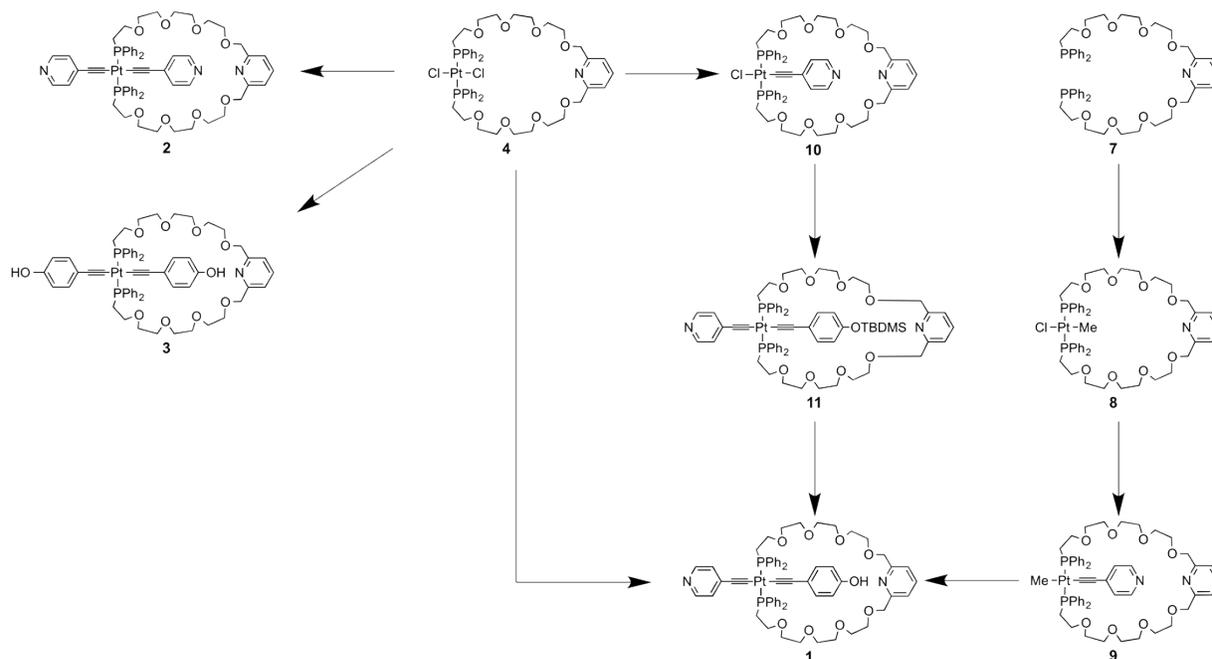
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Experimental section

General procedures

THF was distilled over Na/benzophenone. NEt₃ was distilled over KOH. CH₂Cl₂ was distilled over P₂O₅. Other analytical solvents were purchased as analytical grade and used without further purification. All commercially available products were used as received without further purification. ¹H-, ¹³C-, and ³¹P-NMR spectra were recorded at 298 K on either a Bruker AV300, AV400, AV500 or AV600 spectrometers, with the solvent peak as the internal reference. For signals assignment, the chemical shift is given first, followed in brackets by the multiplicity of the signal (br = broad, m = multiplet, s = singlet, d = doublet, t = triplet, virtual t = virtual triplet), the integration, the assignment and the coupling constants). Mass spectrometry was performed by the Service de Spectrométrie de Masse, University of Strasbourg. Elemental analyses were performed by the Service de Microanalyses, University of Strasbourg.

I–Synthesis



Compounds **2**,¹ **4**,¹ **5**,² **6**,³ **7**,¹ *cis*-Cl₂Pt(PPh₃)₂,⁴ CODPtCl₂⁵ and CODPtMeCl⁶ were synthesized according to literature procedures.

Compound 8. Diphosphine **7** (0.46 g, 0.56 mmol, 1 eq.) and CODPtMeCl (196 mg, 0.56 mmol, 1 eq.) were dissolved separately in dry, degassed CH₂Cl₂ (25 mL each) and added by syringe pump (1 mL/hour) in a dry 1 L three-necked round-bottomed flask containing dry, degassed CH₂Cl₂ (400 mL). At the end of the addition, the solution is stirred for 1 more day. After removal of the solvent under reduced pressure, the product is purified by column chromatography (SiO₂, CH₂Cl₂/MeOH 97/3 to 90/10) to yield **8** (0.55 g, 92%) as a slightly yellow solid. ¹H-NMR (CD₂Cl₂, 300 MHz): δ (ppm) = -0.07 (t, 3H, Pt-CH₃, ²J_{Pt-H} = 80.5 Hz, ³J_{P-H} = 6.5 Hz), 2.95 (m, 4H, H_i), 3.46-3.72 (m, 24H, OCH₂), 3.89 (m, 4H, H_k), 4.59 (s, 4H, H_d), 7.32 (d, 2H, H_b, ³J = 7.7 Hz), 7.36-7.46 (m, 12H, H_{o-p}), 7.62-7.75 (m, 9H, H_{a-n}). ¹³C-NMR (CD₂Cl₂, 125 MHz): δ (ppm) = -12.9 (C_{Pt}, ¹J_{Pt-C} = 662 Hz, ²J_{P-C} = 5.6 Hz), 27.0 (C_l, virtual t, J_{P-C}

= 16.9 Hz), 67.4 (C_k), 70.5, 70.6, 70.7, 70.8, 70.9, 71.0, 71.1 (OCH₂), 74.4 (C_d), 120.4 (C_b), 128.5 (C_o, virtual t, J_{P-C} = 4.9 Hz), 130.6 (C_p), 131.5 (C_m, virtual t, J_{P-C} = 26.2 Hz), 134.0 (C_n, virtual t, J_{P-C} = 6.0 Hz), 137.6 (C_a), 158.4 (C_c). ³¹P-NMR (CD₂Cl₂, 121.5 MHz): δ (ppm) = 18.7 (J_{P-Pt} = 3043 Hz). MS (ESI): m/z calcd for C₄₈H₆₂ClNO₈P₂PtNa⁺ [M + Na]⁺ 1096.318 g.mol⁻¹; found 1096.322 g.mol⁻¹; m/z calcd for C₄₈H₆₂NO₈P₂Pt⁺ [M - Cl]⁺ 1038.36 g.mol⁻¹; found 1038.37 g.mol⁻¹.

Compound 9. Compounds **8** (250 mg, 0.233 mmol, 1 eq.) and **5** (36 mg, 0.35 mmol, 1.5 eq.) were dissolved in a degassed THF/NEt₃ mixture (1:1, 30 mL) and the reaction mixture was degassed by freeze-thaw cycles. After one cycle, CuI (4.5 mg, 0.02 mmol, 0.1 eq.) was added in the cold mixture. After 5 more cycles, the reaction mixture was warmed to 50 °C and stirred overnight. After filtration and evaporation of the filtrate, the resulting solid was poured back in CH₂Cl₂ (50 mL) and washed with 3 x 50 mL of H₂O. The product was purified by column chromatography (SiO₂, CHCl₃/MeOH 97/3 to 90/10) to afford **9** as a yellow solid (0.23 g). This sample contained traces (*ca.* 5 % according to ¹H and ³¹P NMR) of the bis-coupled **2** that could not be separated at this stage. ¹H-NMR (CD₂Cl₂, 400 MHz): δ (ppm) = -0.26 (t, 3H, Pt-CH₃, ³J_{P-H} = 6.6 Hz, ²J_{Pt-H} = 53.5 Hz), 3.07 (m, 4H, H_i), 3.40-3.73 (m, 24H, OCH₂), 3.88 (m, 4H, H_k), 4.61 (s, 4H, H_d), 7.38-7.48 (m, 14H, H_{b-o-p}), 7.65-7.75 (m, 9H, H_{a-n}). ³¹P-NMR (CD₂Cl₂, 162 MHz): δ (ppm) = 13.0 (J_{P-Pt} = 2855 Hz). MS (ESI): m/z calcd for C₆₁H₆₇N₃O₈P₂PtNa⁺ [**2** + Na]⁺ 1249.395 g.mol⁻¹; found 1249.389 g.mol⁻¹; m/z calcd for C₅₅H₆₆N₂O₈P₂PtNa⁺ [M + Na]⁺ 1162.384 g.mol⁻¹; found 1162.380 g.mol⁻¹; m/z calcd for C₅₅H₆₇N₂O₈P₂Pt⁺ [M + H]⁺ 1140.402 g.mol⁻¹; found 1140.399 g.mol⁻¹.

Compound 10. In a schlenk flask, ethynylpyridyl **5** (41.2 mg, 0.40 mmol, 1 eq.) was dissolved in THF (20 mL) and the solution was degassed by freeze-pump-thaw cycles and placed at -78°C,

and *n*BuLi (1.6 M in hexanes, 0.25 mL, 0.40 mmol, 1 eq.) was added. After 30 min., SnMe₃Cl (119 mg, 0.60 mmol, 1.5 eq.) was added and the solution was slowly heated to -20°C. After 1h30, complex **4** (0.44 g, 0.40 mmol, 1 eq.) and CuI (3 mg, 16 μmol, 4 %) were added and the solution was slowly heated to 60 °C. After 18 h, the solution was allowed to reach RT and the solvents were removed. Column chromatography (SiO₂, CHCl₃/MeOH 0 to 10 %) afforded **10** as a yellow solid (0.16 g, 34 %). ¹H-NMR (CDCl₃, 500 MHz): δ (ppm) = 3.01 (m, 4H, H_l), 3.42-3.72 (m, 24H, OCH₂), 3.81 (m, 4H, H_k), 4.64 (s, 4H, H_d), 6.28 (br, 2H, H_t), 7.28-7.46 (m, 14H, H_{b-o-p}), 7.66 (t, 1H, ³J = 7.6 Hz), 7.68-7.77 (m, 8H, H_n), 8.14 (br, 2H, H_u). ¹³C-NMR HSQC/HMBC (CDCl₃, 125 MHz): δ (ppm) = 26.4 (C_l, virtual t, J_{P-C} = 17.7 Hz), 66.8 (C_k), 70.4, 70.4, 70.7, 70.8, 70.9, 71.1, 74.2 (C_d), 120.2 (H_b), 125.6 (H_t), 128.5 (H_o, virtual t, J_{P-C} = 5.2 Hz), 130.9 (H_p), 130.3 (H_m, virtual t, J_{P-C} = 27.8 Hz), 133.8 (H_n, virtual t, J_{P-C} = 5.9 Hz), 137.6 (H_a), 148.5 (H_u), 158.1 (H_c). ³¹P-NMR (CDCl₃, 121.5 MHz): δ (ppm) = 10.3 (J_{P-pt} = 2529 Hz). MS (ESI): m/z calcd for C₅₄H₆₄ClN₂O₈P₂Pt⁺ [M + H]⁺ 1161.3472 g.mol⁻¹ ; found 1161.3675 g.mol⁻¹.

Compound 11. In a schlenk flask, compounds **10** (0.16 g, 0.14 mmol, 1 eq.) and **6** (48 mg, 0.21 mmol, 1.5 eq.) were dissolved in THF (20 mL) and NEt₃ (5 mL) and the solution was degassed by freeze-pump-thaw cycles. After heating at 60 °C overnight, the solution was evaporated to dryness. Column chromatography (SiO₂, CHCl₃/MeOH 2 to 10 %) afforded **11** as a yellow solid (0.17 g, 91 %). ¹H-NMR (CD₂Cl₂, 300 MHz): δ (ppm) = 0.12 (s, 6H, H_{Si-Me}), 0.93 (s, 9H, H_{Si-tBu}), 3.15 (m, 4H, H_l), 3.39-3.71 (m, 24H, OCH₂), 3.92 (m, 4H, H_k), 4.59 (s, 4H, H_d), 6.51 (d, 2H, H_t, ³J = 8.7 Hz), 6.55 (eclipsed by H_t and H_u signals, 2H, H_T), 6.58 (d, 2H, H_u, ³J = 8.7 Hz), 7.31 (d, 2H, H_b, ³J = 7.7 Hz), 7.36-7.48 (m, 12H, H_{o-p}), 7.63 (t, 1H, H_a, ³J = 7.7 Hz), 7.76-7.88 (m, 8H, H_n), 8.18 (d, 2H, H_U, ³J = 5.2 Hz). ¹³C-NMR (CD₂Cl₂, 125 MHz): δ (ppm) = -4.4 (Si-CH₃), 18.4 (Si-C), 25.8 (Si-C-CH₃), 28.7 (C_l, virtual t, J_{P-C} = 18.7 Hz), 67.4 (C_k), 70.5,

70.6, 70.7, 70.9, 71.1, 74.3 (C_d), 108.8, 111.5, 119.8 (C_{b or t}), 120.4 (C_{b or t}), 121.7, 125.2, 127.2 (C_T), 128.5 (C_o, virtual t, J_{P-C} = 4.9 Hz), 130.9 (C_p), 131.9 (C_m, virtual t, J_{P-C} = 28.4 Hz), 133.8 (C_u), 134.0 (C_n, virtual t, J_{P-C} = 5.9 Hz), 136.4, 137.5 (C_a), 149.2 (C_U), 153.9 (C_v), 158.3 (C_c). ³¹P-NMR (CD₂Cl₂, 121.5 MHz): δ (ppm) = 6.3 (J_{P-Pt} = 2531 Hz). MS (ESI): m/z calcd for C₆₈H₈₃N₂O₉P₂PtSi⁺ [M + H]⁺ 1357.4991 g·mol⁻¹ ; found 1357.4816 g·mol⁻¹.

Compound 3. *Method A:* In a dry flask, **4** (36 mg, 33 μmol, 1 eq.), **5** (22.8 mg, 0.12 mmol, 3.6 eq.) and CuI (2 mg, 0.01 mmol, 0.3 eq.) were dissolved in CH₂Cl₂ (2 mL) and the solution was degassed by thaw-freeze cycles. After one cycle, TBAF (1M/THF, 0.23 mL, 0.23 mmol, 7 eq.) was added to the cold mixture. After 5 more cycles, the mixture was stirred at RT for 20 h. After filtration and removal of the solvents, the crude product was purified by column chromatography (SiO₂, CH₂Cl₂/MeOH, 99/1 to 95/5) and PLC (SiO₂, CH₂Cl₂/MeOH, 95/5) to afford **3** (4 mg, 10 %). *Method B:* In a dry flask, **4** (110 mg, 0.10 mmol, 1 eq.) and **6** (70 mg, 0.30 mmol, 3 eq.) were dissolved in THF/NEt₃ (1/1, 10 mL) and the reaction mixture was degassed by freeze-thaw cycles. After one cycle, CuI (2 mg, 0.01 mmol, 0.1 eq.) was added to the cold mixture. After 5 more cycles, the reaction mixture was warmed slowly to 55 °C and stirred overnight. The mixture was filtrated and the residue washed thoroughly with CH₂Cl₂. After evaporation of the solvent, the resulting solid was poured back in CH₂Cl₂ (20 mL) and washed with 4 x 25 mL of H₂O. After evaporation of the solvent, the product was purified by column chromatography (SiO₂, CHCl₃/MeOH 100/0 to 90/10) to obtain a mixture of two products (yellow solid, 140 mg). These compounds were poured back in THF/CH₂Cl₂ (1/1, 20 mL) and TBAF (1M/THF, 0.3 mL) was added. After 1 h. at RT, the solution was evaporated to dryness and the product was purified by column chromatography (SiO₂, CH₂Cl₂/MeOH, 95/5) and PLC (SiO₂, CH₂Cl₂/MeOH, 95/5) to afford **3** as a yellow solid (10 mg, 8 %). ¹H-NMR (CD₂Cl₂, 600 MHz): δ (ppm) = 3.14 (br, 4H,

H_l), 3.33-3.38 (m, 12H, H_{h-i-j}), 3.40 (m, 4H, H_g), 3.54 (m, 4H, H_f), 3.66 (m, 4H, H_e), 4.65 (s, 4H, H_d), 6.51 (d, 4H, H_u, ³J = 8.4 Hz), 6.60 (d, 4H, H_t, ³J = 8.4 Hz), 7.37 (d, 2H, H_b, ³J = 7.6 Hz), 7.39-7.47 (m, 12H, H_{o-p}), 7.76 (t, 1H, H_a, ³J = 7.6 Hz), 7.86 (m, 8H, H_n). ¹³C-NMR HSQC/HMBC (CD₂Cl₂, 150 MHz): δ (ppm) = 28.8 (C_l, virtual t, J_{P-C} = 18.8 Hz), 67.3 (C_k), 70.5, 70.7, 70.9, 71.0, 73.7 (C_d), 104.1 (H_q), 110.9 (H_r), 115.3 (H_u), 120.8 (H_s), 121.5 (H_b), 128.5 (H_o, virtual t, J_{P-C} = 5.1 Hz), 130.7 (H_p), 132.3 (H_m, virtual t, J_{P-C} = 27.8 Hz), 132.4 (H_t), 134.1 (H_n, virtual t, J_{P-C} = 6.1 Hz), 138.4 (H_a), 154.8 (H_v), 158.1 (H_c). ³¹P-NMR (CDCl₃, 162 MHz): δ (ppm) = 5.07 (J_{P-Pt} = 2568 Hz). MS (ESI): m/z calcd for C₆₅H₇₂N₂O₁₀P₂Pt⁺ [M + Na + CH₃CN]⁺ 1320.42 g.mol⁻¹; found 1320.43 g.mol⁻¹; m/z calcd for C₆₃H₆₉NO₁₀P₂PtNa⁺ [M + Na]⁺ 1279.40 g.mol⁻¹; found 1279.40 g.mol⁻¹; m/z calcd for C₆₃H₇₀NO₁₀P₂Pt⁺ [M + H]⁺ 1257.41 g.mol⁻¹; found 1257.42 g.mol⁻¹; m/z calcd for C₅₇H₆₇N₂O₉P₂Pt⁺ [M - PhOH + CH₃CN]⁺ 1180.40 g.mol⁻¹; found 1180.40 g.mol⁻¹.

Compound 1. *Method A:* In a dry flask, **4** (100 mg, 91.4 μmol, 1 eq.), **5** (14.1 mg, 0.137 mmol, 1.5 eq.) and **6** (31.9 mg, 0.137 mmol, 1.5 eq.) were dissolved in THF/NEt₃ (1/1, 20 mL) and the reaction mixture was degassed by freeze-thaw cycles. After one cycle, CuI (2 mg, 0.01 mmol, 0.11 eq.) was added to the cold mixture. After 5 more cycles, the reaction mixture was warmed slowly to 55 °C and stirred overnight. The mixture was filtrated and the residue washed thoroughly with CH₂Cl₂. After evaporation of the solvent, the resulting solid was poured back in CH₂Cl₂ (20 mL) and washed with 4 x 25 mL of H₂O. After evaporation of the solvent, the mixture of intermediates (bis-pyridine, bis-phenol and the heteroleptic complex) was purified by column chromatography (SiO₂, CHCl₃/MeOH 100/0 to 95/5). Attempts to separate this mixture (column chromatography, PLC, recrystallization) were unsuccessful and it was used as a mixture in the next step. The mixture (90 mg) was dissolved in degassed THF (3 mL) and TBAF (1M in

THF, 0.13 mL) was added. After 2 h at RT, the solvent was removed in vacuum. After column chromatography (SiO₂, CHCl₃/MeOH 98/2) and several PLC (SiO₂, CHCl₃/MeOH 90/10), the desired product was obtained (4 mg, 3.5 %). *Method B:* **9** (0.23 g, 0.20 mmol, 1 eq.) was dissolved in CH₂Cl₂ (20 mL) and HCl (0.1N in MeOH/H₂O 1000/8, 8.0 mL, 4 eq.) was added. After 20 h at RT, the solvent were removed and the product was dissolved in CH₂Cl₂ (20 mL), washed with 3 x 30 mL H₂O and the organic phases were evaporated to dryness. At this stage, several column chromatography (SiO₂, CHCl₃/MeOH) could not separate the different products obtained, and it was used as such in the following step. It is noteworthy that the characteristic signals of Pt-CH₃ from the starting material **9** were not present anymore on the ¹H-NMR spectra. In a dry flask, the mixture of products (0.21 g) and **6** (50 mg, 0.22 mmol) were dissolved in THF/NEt₃ (20 + 15 mL) and the reaction mixture was degassed by freeze-thaw cycles. After one cycle, CuI (2 mg, 0.01 mmol, 0.11 eq.) was added to the cold mixture. After 5 more cycles, the reaction mixture was warmed slowly to 60 °C and stirred overnight. The mixture was filtrated and the residue washed thoroughly with CH₂Cl₂. The silylated intermediate was partially purified by several column chromatography (SiO₂, CHCl₃/MeOH, CH₂Cl₂/MeOH, CH₂Cl₂/MeOH/NH₃) and PLC (SiO₂, CHCl₃/MeOH). The mixture (22 mg) was dissolved in degassed THF (6 mL) and TBAF (1M in THF, 40 μL) was added. After 1 h at RT, the solvent was removed. Column chromatography (SiO₂, CHCl₃/MeOH 100/0 to 90/10) and PLC (SiO₂, CHCl₃/MeOH 97/3) afforded **1** (15 mg, 6 %). *Method C:* Compound **11** (120 mg, 88 μmol, 1 eq.) was dissolved in THF (40 mL) and TBAF (1 M/THF, 0.15 mL, 0.15 mmol, 1.7 eq.) was added. After 2 h, the mixture was evaporated to dryness, the resulting solid was poured back in CH₂Cl₂ (25 mL) and washed with H₂O (3 x 20 mL). Column chromatography (SiO₂, CHCl₃/MeOH 2 to 10 %) yielded impure **1** (110 mg). Out of this mixture, 43 mg were purified by PLC (SiO₂, CHCl₃/MeOH 3 %)

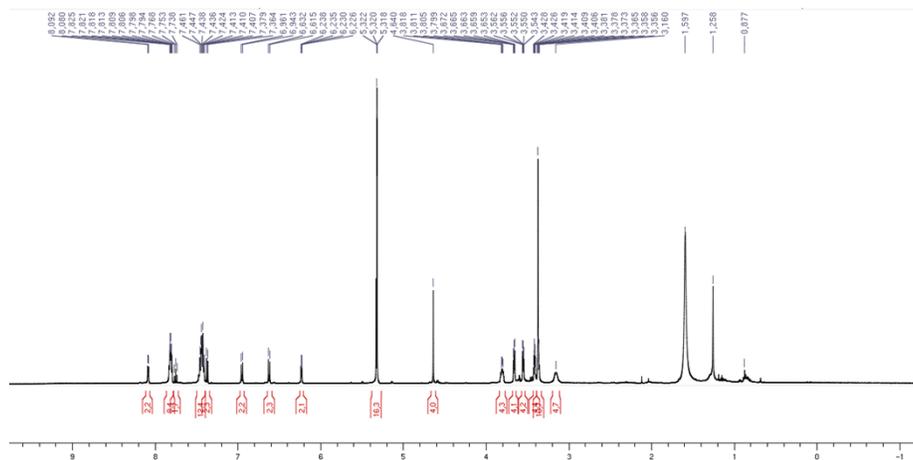
to afford **1** (18 mg, 42 %). ¹H-NMR (CD₂Cl₂, 300 MHz): δ (ppm) = 3.16 (m, 4H, H_l), 3.33-3.39 (m, 12H, H_j + OCH₂), 3.41 (OCH₂), 3.55 (H_f), 3.66 (H_e), 3.81 (m, 4H, H_k), 4.64 (s, 4H, H_d), 6.23 (d, 2H, H_T, ³J = 5.8 Hz), 6.62 (d, 2H, H_t, ³J = 8.7 Hz), 6.95 (d, 2H, H_u, ³J = 8.7 Hz), 7.37 (d, 2H, H_b, ³J = 7.8 Hz), 7.40-7.49 (m, 12H, H_{o-p}), 7.75 (t, 1H, H_a, ³J = 7.8 Hz), 7.78-7.85 (m, 8H, H_n), 8.09 (d, 2H, H_U, ³J = 5.8 Hz). ¹³C-NMR HSQC/HMBC (CD₂Cl₂, 125 MHz): δ (ppm) = 28.6 (C_l, virtual t, J_{P-C} = 18.6 Hz), 67.2 (C_k), 70.5, 70.6, 70.6, 70.8, 70.9 (OCH₂), 73.7 (C_d), 110.1 (C_{r or s}), 110.6 (C_{R or S}), 115.7 (C_t), 119.7 (C_{r or s}), 121.4 (C_b), 125.3 (C_T), 128.5 (C_o, virtual t, J_{P-C} = 5.1 Hz), 130.9 (C_p), 131.8 (C_m, virtual t, J_{P-C} = 28.4 Hz), 132.6 (C_u), 134.0 (C_n, virtual t, J_{P-C} = 6.0 Hz), 136.4 (C_{R or S}), 138.4 (C_a), 149.0 (C_U), 155.8 (C_v), 158.0 (C_c). ³¹P-NMR (CD₂Cl₂, 162 MHz): δ (ppm) = 5.37 (J_{P-Pt} = 2534 Hz). MS (ESI): m/z calcd for C₆₂H₆₉N₂O₉P₂Pt⁺ [M + H]⁺ 1242.413 g.mol⁻¹; found 1242.418 g.mol⁻¹.

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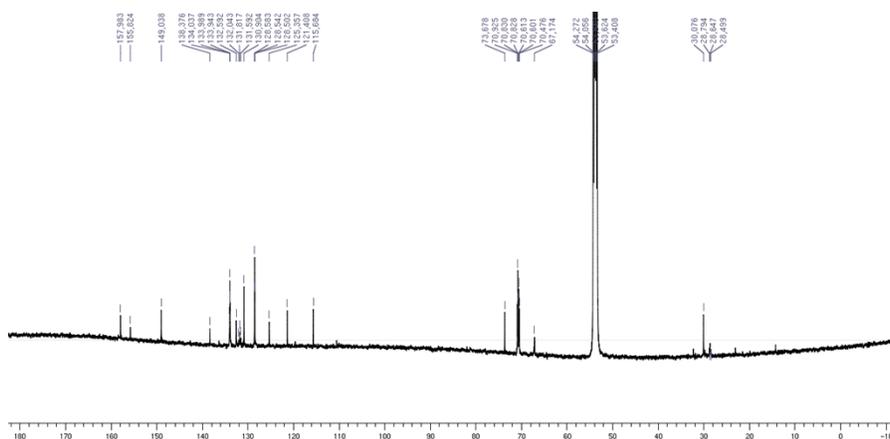
II – Compounds characterization

Compound 1

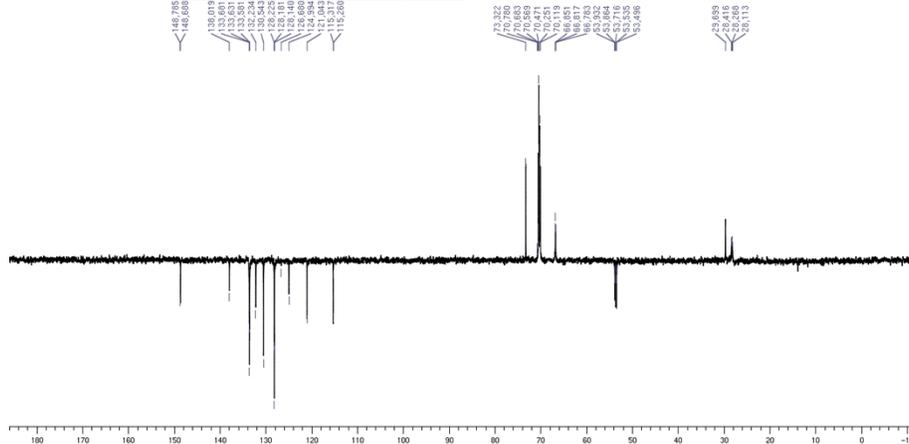
$^1\text{H-NMR}$ (CD_2Cl_2 , 298 K, 500 MHz)



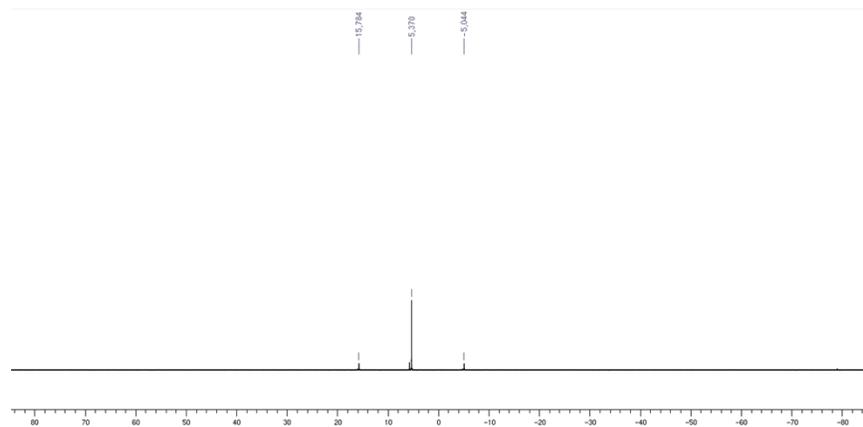
$^{13}\text{C-NMR}$ (CD_2Cl_2 , 298 K, 125 MHz)



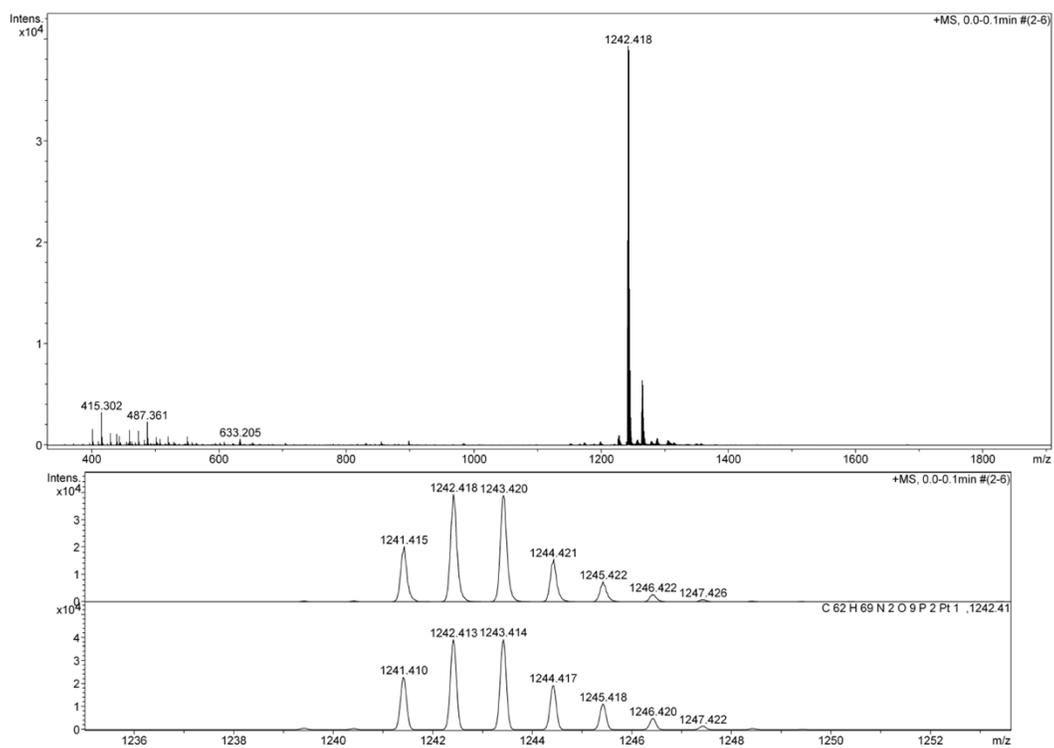
DEPT $^{13}\text{C-NMR}$ (CD_2Cl_2 , 298 K, 125 MHz)



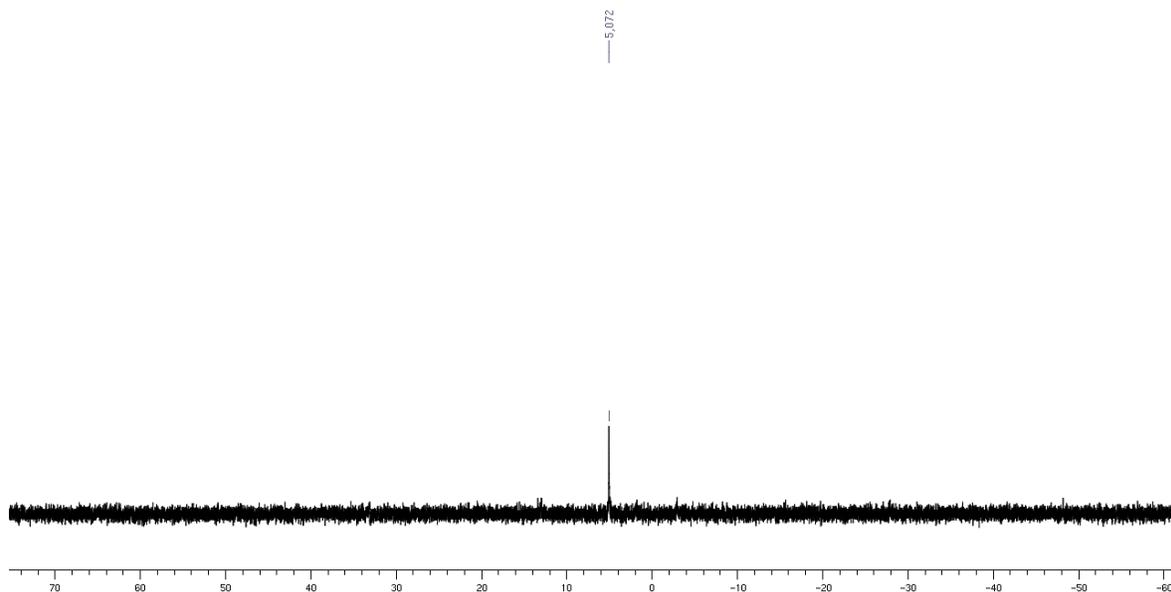
^{31}P -NMR (CD_2Cl_2 , 298 K, 162 MHz)



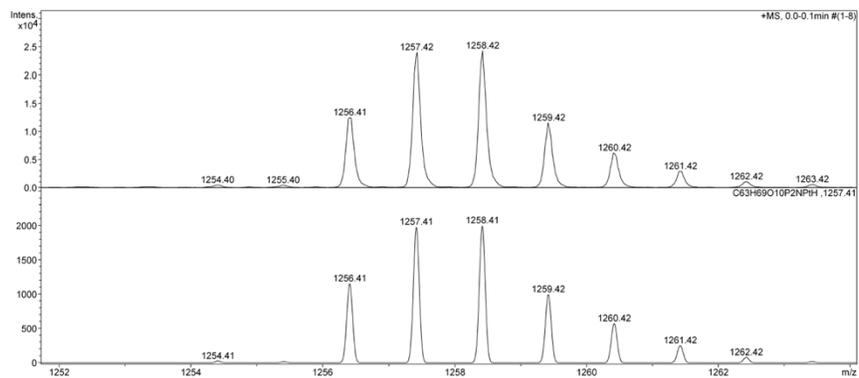
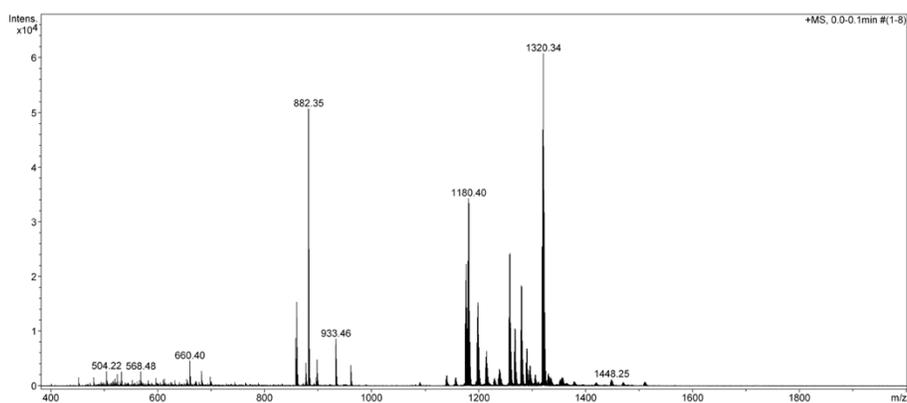
ESI MS



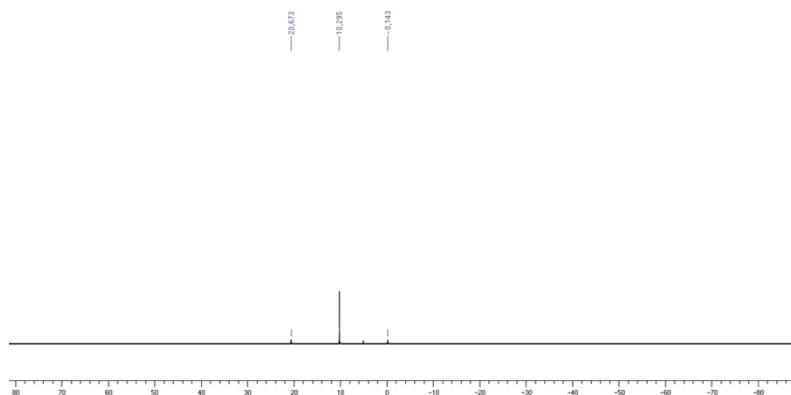
^{31}P -NMR (CDCl_3 , 298 K, 162 MHz)



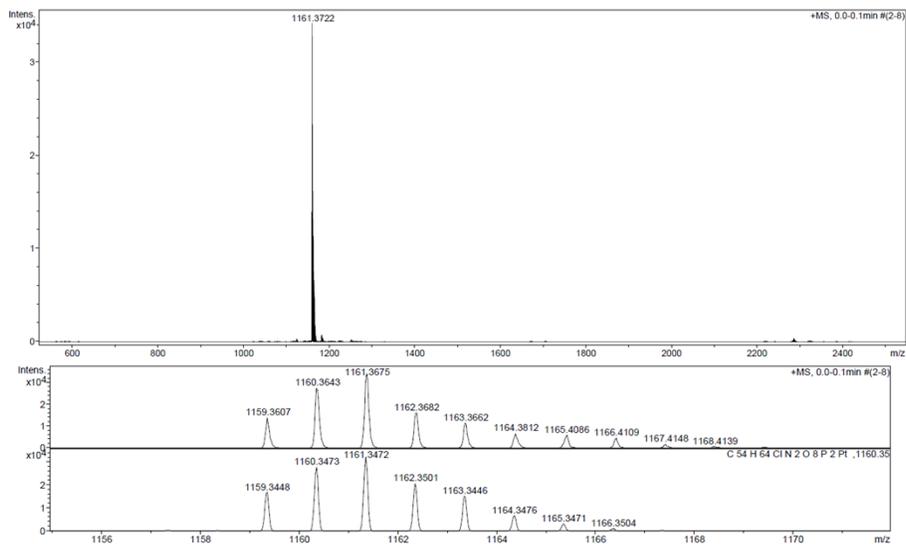
ESI MS



^{31}P -NMR (CDCl_3 , 298 K, 121.5 MHz)

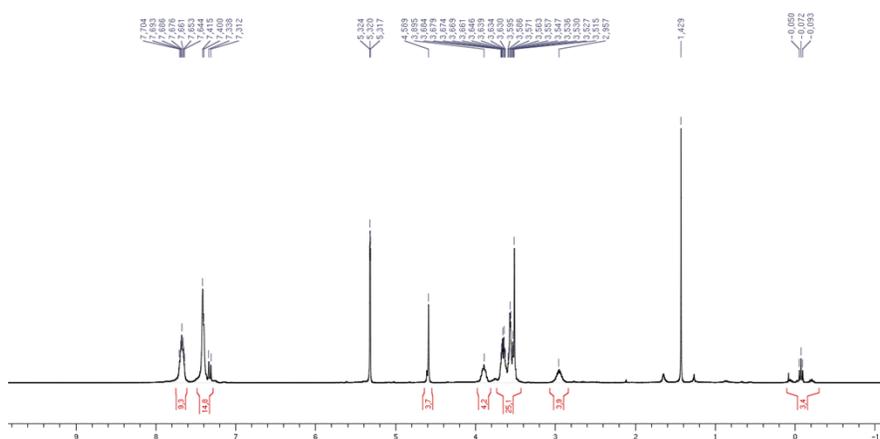


ESI MS

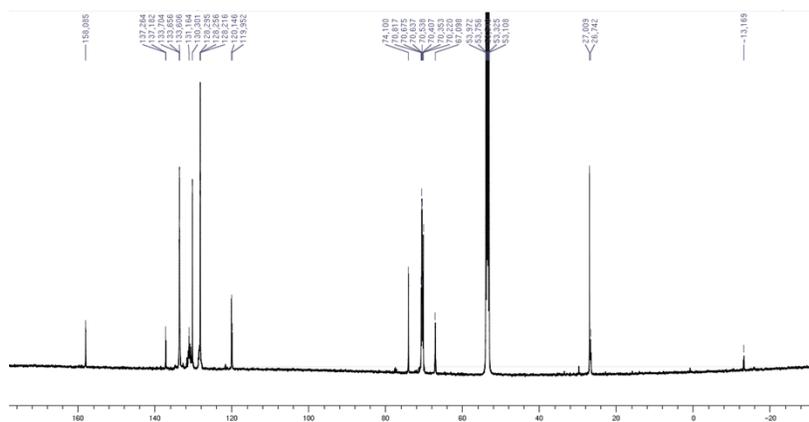


Compound 8

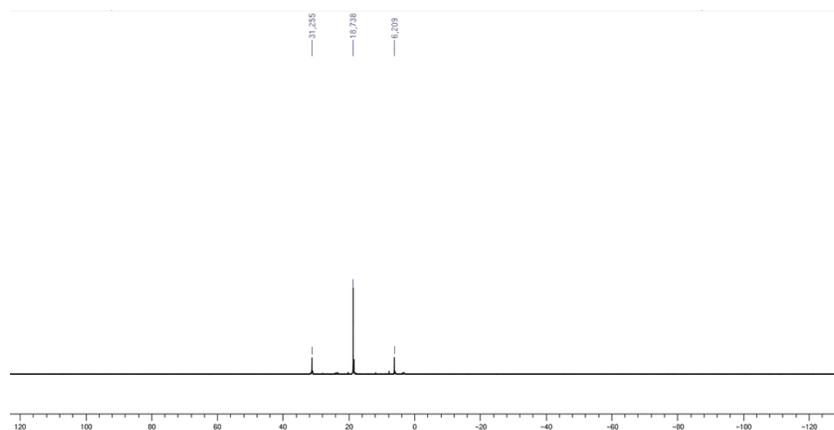
$^1\text{H-NMR}$ (CDCl_3 , 298 K, 500 MHz)



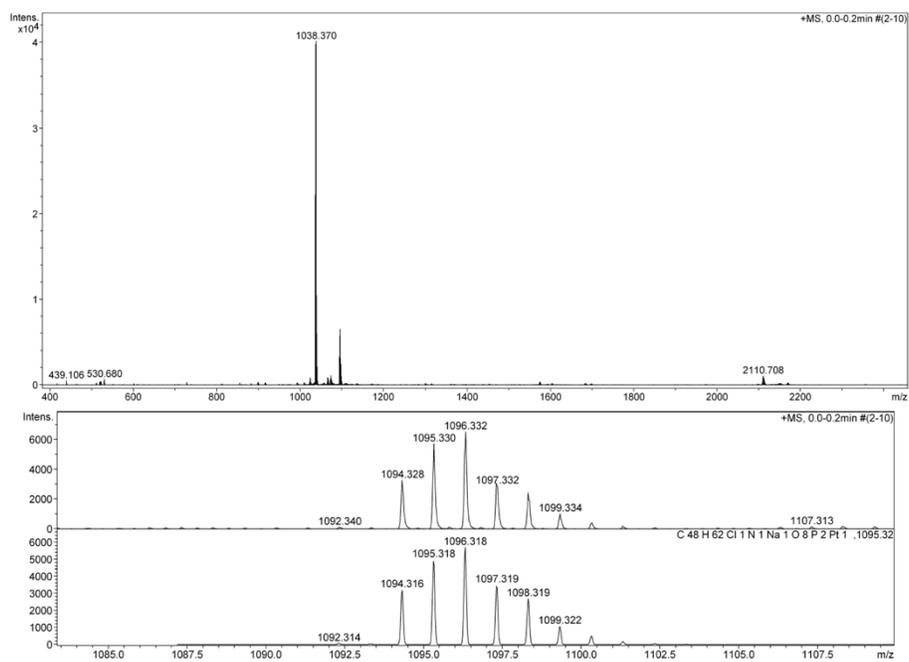
$^{13}\text{C-NMR}$ (CDCl_3 , 298 K, 125 MHz)



$^{31}\text{P-NMR}$ (CDCl_3 , 298 K, 121.5 MHz)

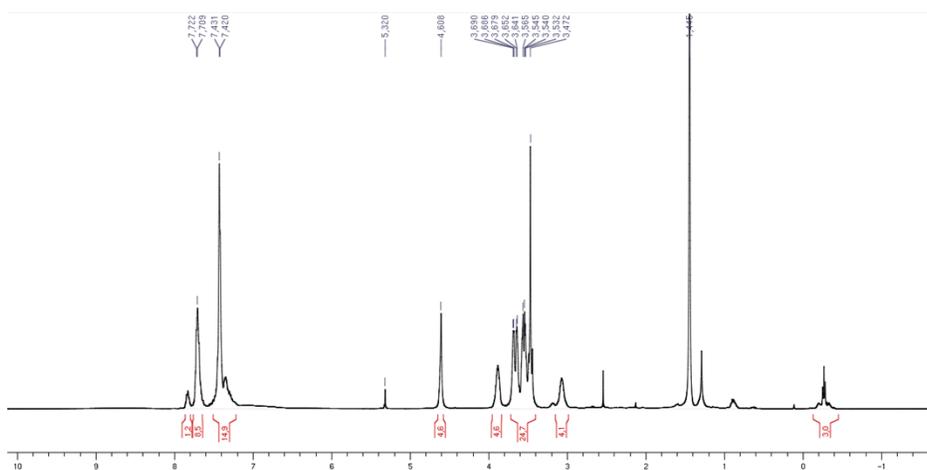


ESI MS

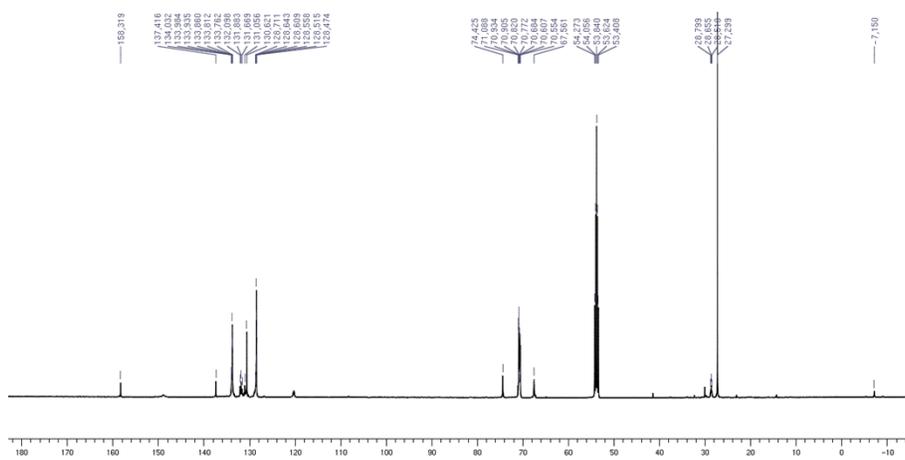


Compound 9

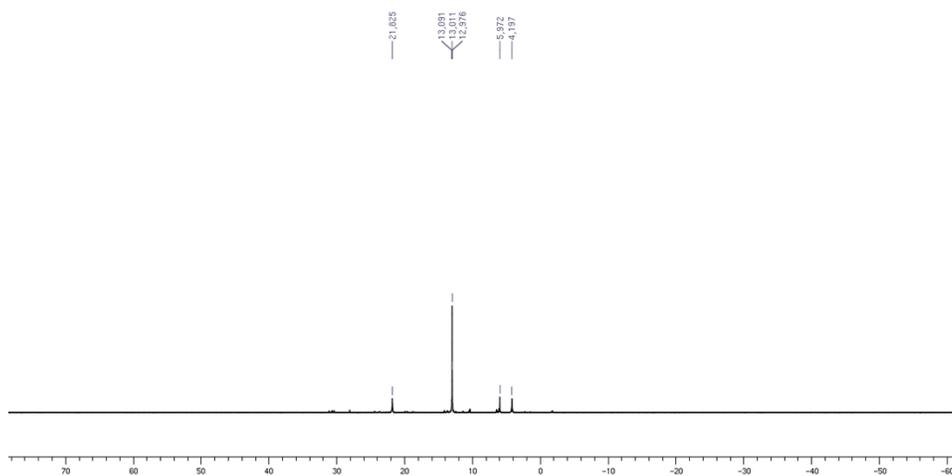
$^1\text{H-NMR}$ (CD_2Cl_2 , 298 K, 400 MHz)



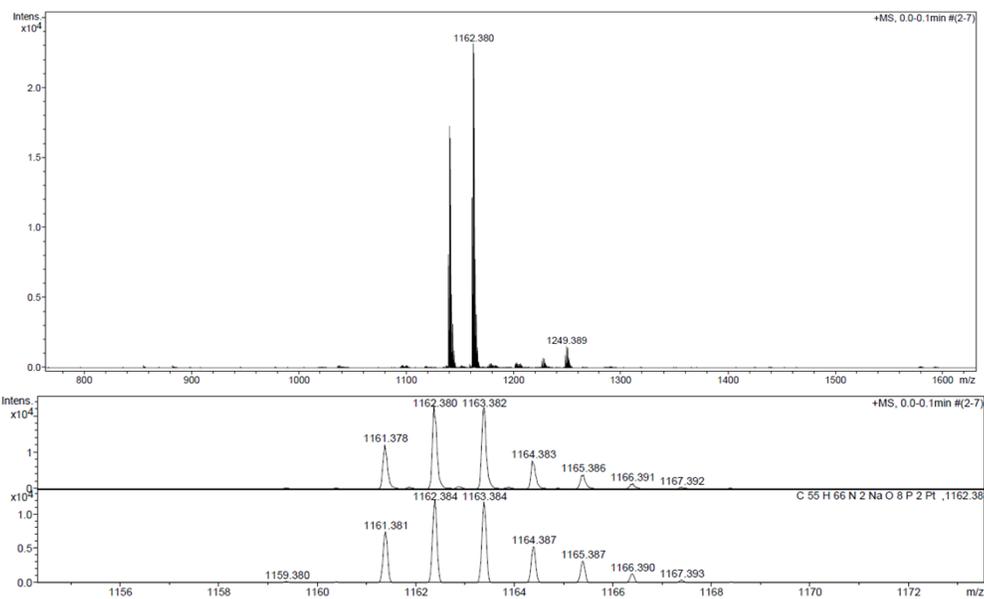
$^{13}\text{C-NMR}$ (CD_2Cl_2 , 298 K, 125 MHz)



$^{31}\text{P-NMR}$ (CD_2Cl_2 , 298 K, 162.0 MHz)

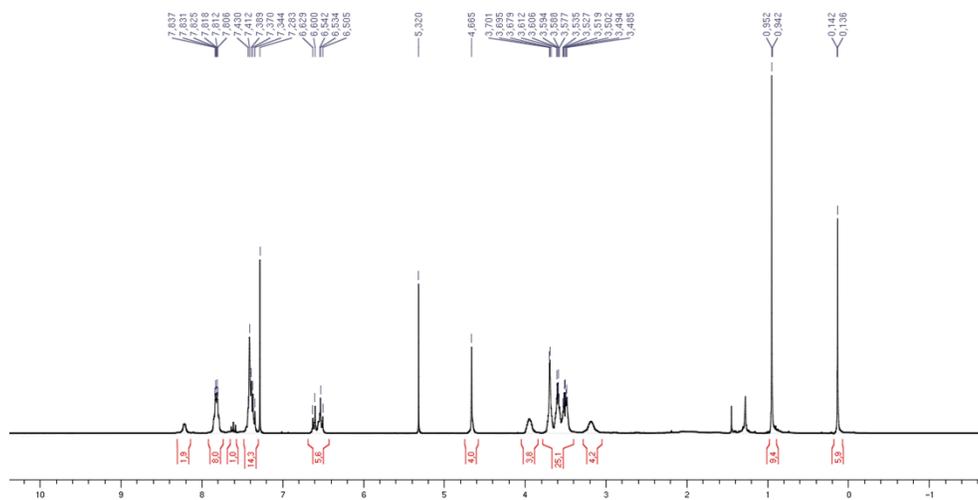


ESI MS

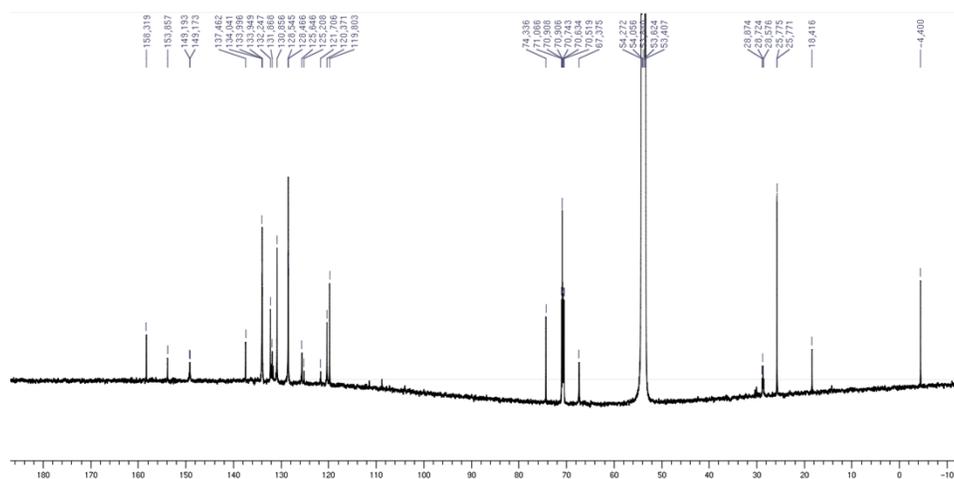


Compound 11

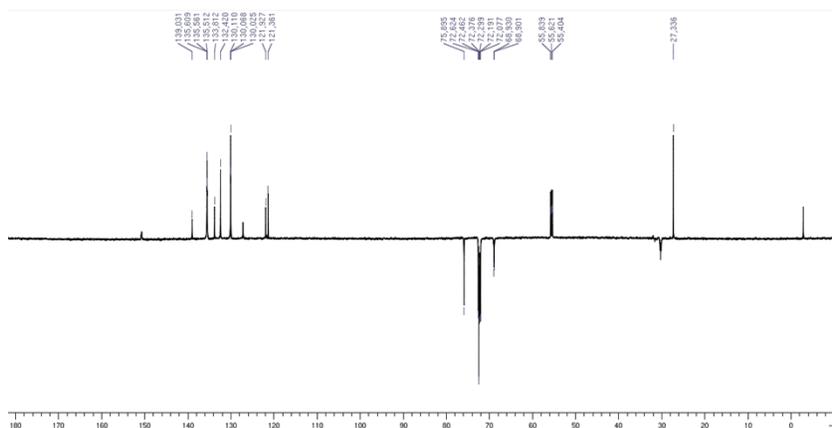
$^1\text{H-NMR}$ (CDCl_3 , 298 K, 300 MHz)



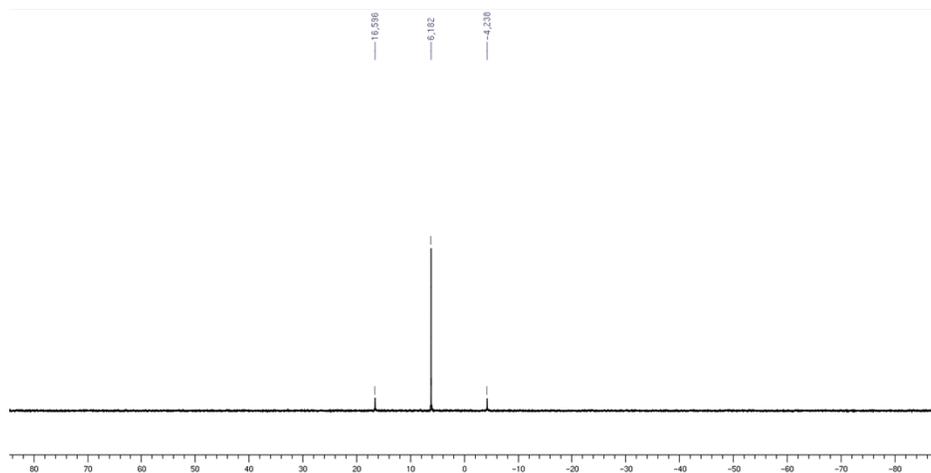
$^{13}\text{C-NMR}$ (CD_2Cl_2 , 298 K, 125 MHz)



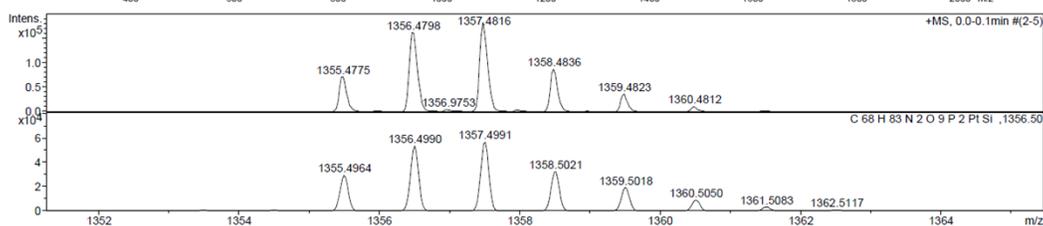
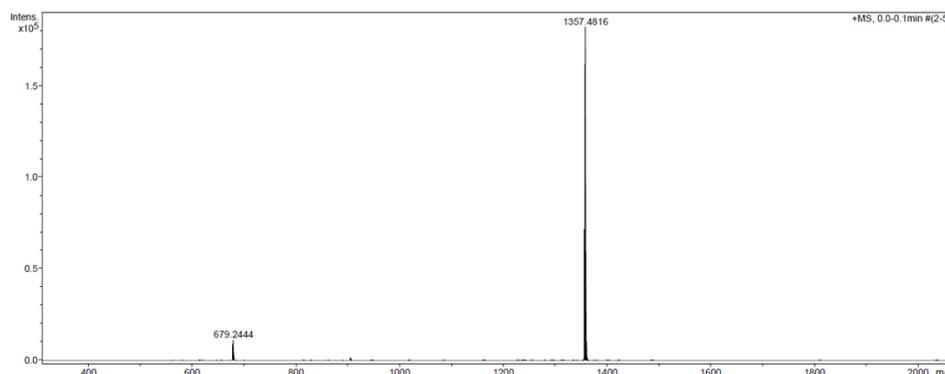
DEPT $^{13}\text{C-NMR}$ (CD_2Cl_2 , 298 K, 125 MHz)



^{31}P -NMR (CDCl₃, 298 K, 121.5 MHz)

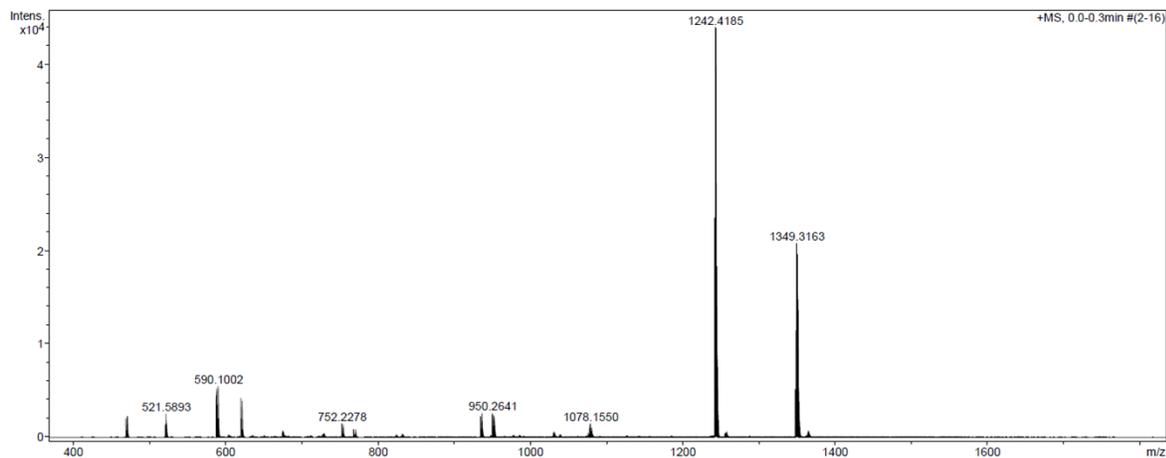


ESI MS

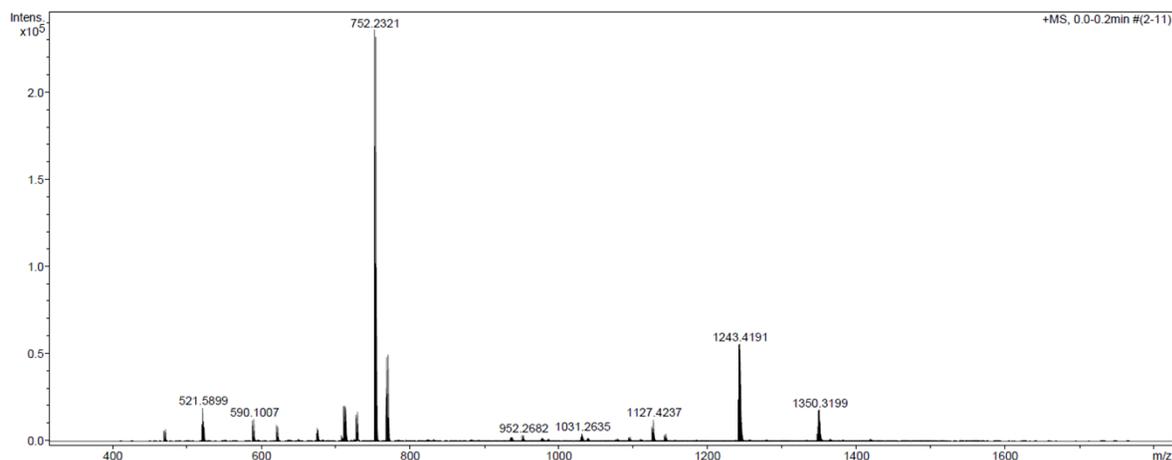


III Titration – ESI-MS spectra of 1 + n eq. of Ag(I)

1 + 1 eq. Ag⁺



1 + 2 eq. Ag⁺



1 + 3 eq. Ag⁺

