

Supporting Information

**Cross Hetero Dehydrogenative Coupling Reaction of Phosphites: A
Catalytic Metal-Free Phosphorylation of amines and Alcohols**

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General experimental

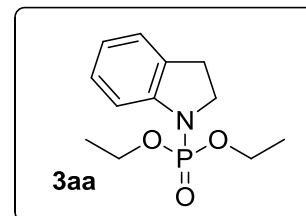
NMR spectra were recorded on a JEOL LA-300, BRUKER-AV400 spectrometer in CDCl₃. Tetramethylsilane (TMS; δ = 0.00 ppm) served as an internal standard for ¹H NMR. The corresponding residual non-deuterated solvent signal (CDCl₃; δ = 77.00 ppm) was used as internal standard for ¹³C NMR. IR spectra were measured using a JASCO FT/IR-410 spectrometer and Perkin-Elmer FT/IR Spectrum BX, GX. Mass spectra were measured with Micromass Q-Tof (ESI-HRMS). Column chromatography was carried out on Silica gel 100-200 mesh (commercial suppliers) and thin-layer chromatography was carried out using SILICA GEL GF-254.

Typical experimental procedure

- A. Amine with dialkyl *H*- phosphite:** Diethyl *H*-phosphite (100 mg, 0.72 mmol), indoline (1 equiv. 0.72 mmol) and molecular iodine (0.1 equiv. 0.072 mmol) and H₂O₂ (50% solution in water, 1 equiv, 0.72 mmol, 0.04 mL) in CH₂Cl₂ (2 mL) were stirred at room temperature (24-36 h). After the completion of the reaction (monitored by TLC) the solvent was removed under vacuum, added water (3 mL) and extracted with CH₂Cl₂ (3x10mL). The combined organic layer was dried over Na₂SO₄ and concentrated under reduced pressure. The crude product was purified on a silica gel column using hexane/ EtOAc to get the pure product.
- B. Alcohol with dialkyl *H*- phosphite:** Diethyl *H*-phosphite (100 mg, 0.72 mmol), alcohol (3equiv, 2.17 mmol)* and molecular iodine (0.1 equiv. 0.072 mmol) and H₂O₂ (50% solution in water, 1 equiv, 0.72 mmol, 0.04 mL) in neat condition were stirred at room temperature (12-24 h). After the completion of the reaction (monitored by TLC) the solvent was removed under vacuum, added water (3 mL) and extracted with EtOAc (3x10mL). The combined organic layer was dried over Na₂SO₄ and concentrated under reduced pressure. The crude product was purified on a silica gel column using hexane/ EtOAc to get the pure product.
- * 0.5 mL of following alcohols was needed to complete the reaction: MeOH (**5aa**), EtOH (**5ab**), propanol (**5ac**), butanol (**5ad**), pentanol (**5ae**), 2-methylpropanol (**5af**), allyl alcohol (**5ah**), propargyl alcohol (**5ai**), and 2-propanol (**5an**).
- C. Sulfoximine with dialkyl *H*- phosphite:** Diethyl *H*-phosphite (100 mg, 0.72 mmol), sulfoximine (1equiv, 0.72 mmol) and molecular iodine (0.1 equiv. 0.072mmol) and H₂O₂ (50% solution in water, 1 equiv, 0.72 mmol, 0.04 mL) in DCM (2 mL) were stirred at room temperature (12-24h). After the completion of the reaction (monitored by TLC) the solvent was removed under vacuum, added water (3 mL) and extracted with EtOAc (3x10mL). The combined organic layer was dried over Na₂SO₄ and concentrated under reduced pressure. The crude product was purified on a silica gel column using MeOH/EtOAc to get the pure product.

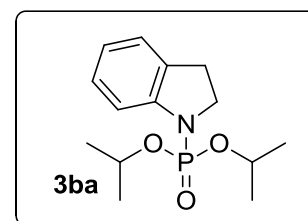
Characterization data for Phosphoramidates

Diethyl indolin-1-ylphosphonate: Dark brown liquid; Yield - 95%; R_f (30% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 2982, 2932, 1484, 1265, 1050, 1023, 970; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ



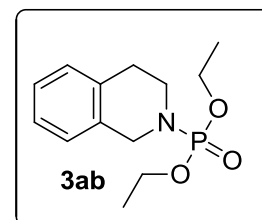
7.16-7.08 (m, 3H), 6.86 (t, $J = 7.2$ Hz, 1H), 4.20-4.01 (m, 4H), 3.91 (t, $J = 8.8$ Hz, 2H), 3.11 (t, $J = 8.8$ Hz, 2H), 1.32 (t, $J = 7.2$ Hz, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 144.7 (d, $J = 6.4$ Hz), 131.0 (d, $J = 12.6$ Hz), 127.4, 124.7, 121.0, 112.2, 62.6 (d, $J = 5.1$ Hz), 49.3 (d, $J = 4.5$ Hz), 28.9 (d, $J = 6.7$ Hz), 16.1 (d, $J = 7.1$ Hz); $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 1.640; **HRESI-MS** (m/z): Calculated for $\text{C}_{12}\text{H}_{18}\text{NO}_3\text{P}$ ($M + \text{Na}$): 278.0922, found ($M + \text{Na}$): 278.0923.

Diisopropyl indolin-1-ylphosphonate: Dark brown liquid; Yield - 82%; R_f (30% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3404, 2923, 1590, 1265, 1041, 1019, 990; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.17-7.06 (m,



3H), 6.83 (t, $J = 7.2$ Hz, 1H), 4.70-4.60 (m, 2H), 3.90 (t, $J = 8.8$ Hz, 2H), 3.08 (t, $J = 8.4$ Hz, 2H), 1.37 (d, $J = 6.4$ Hz, 6H), 1.24 (d, $J = 6.0$ Hz, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 145.0 (d, $J = 6.5$ Hz), 130.9 (d, $J = 12.3$ Hz), 127.3, 124.6, 120.7, 112.5, 71.3 (d, $J = 5.3$ Hz), 49.3 (d, $J = 4.7$ Hz), 28.8 (d, $J = 6.9$ Hz), 23.8 (d, $J = 4.6$ Hz), 23.6 (d, $J = 5.0$ Hz); $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 8.099; **HRESI-MS** (m/z): Calculated for $\text{C}_{14}\text{H}_{22}\text{NO}_3\text{P}$ ($M + \text{H}$): 284.1416, found ($M + \text{H}$): 284.1420.

Diethyl (3,4-dihydroisoquinolin-2(1H)-yl)phosphonate: Orange liquid; Yield - 82%; R_f (50% EtOAc/Hexane) 0.2; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3449, 2983, 2855, 1637, 1256, 1058, 1027, 966; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.16-7.09 (m, 3H), 7.06-7.02 (m, 1H), 4.31 (d, $J = 6$ Hz,



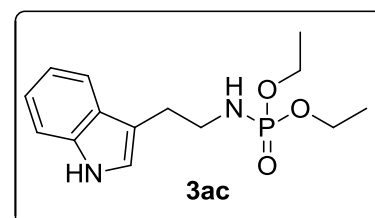
2H), 4.11-3.93 (m, 4H), 3.51-3.40 (m, 2H), 2.84 (t, $J = 5.6$ Hz, 6H), 1.29 (t, $J = 7.2$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 134.1, 133.6 (d, $J = 6.6$ Hz), 129.2, 126.3, 126.0 (d, $J = 2.9$ Hz), 62.2 (d, $J = 5.5$ Hz), 46.2 (d, $J = 4.0$ Hz), 42.1 (d, $J = 2.6$ Hz), 28.9 (d, $J = 3.7$ Hz), 16.1 (d, $J = 7.0$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.547; **HRESI-MS** (m/z): Calculated for $\text{C}_{13}\text{H}_{20}\text{NO}_3\text{P}$ ($M + \text{Na}$): 292.1079, found ($M + \text{Na}$): 292.1076.

Diethyl (2-(1H-indol-3-yl)ethyl)phosphoramidate: Dark brown

liquid; Yield - 64%; R_f (50% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3400, 3281, 2927,

1618, 1441, 1221, 1054, 1028, 968; ^1H NMR (400 MHz, CDCl_3): δ 8.55 (s, 1H), 7.58 (d, $J = 7.6$ Hz, 1H), 7.37 (d, $J = 8.4$ Hz, 1H), 7.18 (t,

$J = 7.2$ Hz, 1H), 7.10 (t, $J = 7.6$ Hz, 1H), 7.01 (s, 1H), 4.12-3.93 (m, 4H), 3.26-3.20 (m, 2H), 2.95 (t, $J = 6.4$ Hz, 2H), 2.73 (brs, 1H), 1.27 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3): δ 136.5, 127.1, 122.5, 122.1, 119.3, 118.5, 112.3 11.3, 62.2 (d, $J = 5.4$ Hz), 41.4, 27.5 (d, $J = 6.3$ Hz), 16.2 (d, $J = 7.0$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.173; **HRESI-MS** (m/z): Calculated for $\text{C}_{14}\text{H}_{21}\text{N}_2\text{O}_3\text{P}$ ($M + \text{Na}$): 319.1188, found ($M + \text{Na}$): 319.1184.

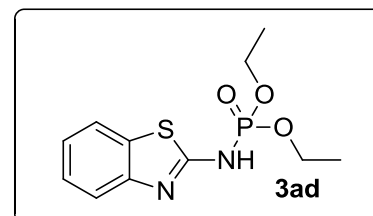


Diethyl benzo[d]thiazol-2-ylphosphoramidate: Brown oil;

Yield - 40%; R_f (50% EtOAc/Hexane) 0.6; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3449,

2931, 1597, 1581, 1470, 1204, 1031, 979; ^1H NMR (400 MHz, CDCl_3): δ 7.54-7.51 (m, 2H), 7.33 (t, $J = 7.2$ Hz, 1H),

7.19 (t, $J = 8.0$ Hz, 1H), 4.25-4.10 (m, 4H), 1.36 (t, $J = 7.2$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 167.6 (d, $J = 3.7$ Hz), 141.1, 126.8, 126.5, 123.4, 121.6, 115.0, 63.1 (d, $J = 5.6$ Hz), 16.2 (d, $J = 6.8$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 4.274; **HRESI-MS** (m/z): Calculated for $\text{C}_{11}\text{H}_{15}\text{N}_2\text{O}_3\text{PS}$ ($M + \text{Na}$): 309.0439, found ($M + \text{Na}$): 309.0439.

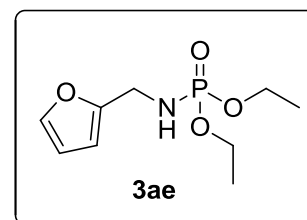


Diethyl (furan-2-ylmethyl)phosphoramidate: Yellow liquid;

Yield - 85%; R_f (100% EtOAc) 0.7; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3398, 3229, 2983, 1443,

1233, 1055, 1029, 968; **^1H NMR** (400 MHz, CDCl_3): δ 7.35 (s, 1H), 6.31 (d, J = 2.8 Hz, 1H), 6.21 (d, J = 2.8 Hz, 1H), 4.10-3.98

(m, 6H), 3.21 (s, 1H), 1.30 (t, J = 6.8 Hz, 6H); **^{13}C NMR** (100 MHz, CDCl_3): δ 152.9 (d, J = 6.4 Hz), 142.0, 110.3, 106.6, 62.3 (d, J = 5.2 Hz), 38.3, 16.1 (d, J = 7.1 Hz); **^{31}P NMR** (162 MHz, CDCl_3): δ 8.099; **HRESI-MS** (m/z): Calculated for $\text{C}_9\text{H}_{16}\text{NO}_4\text{P}$ ($M + \text{Na}$): 256.0715, found ($M + \text{Na}$): 256.0711.



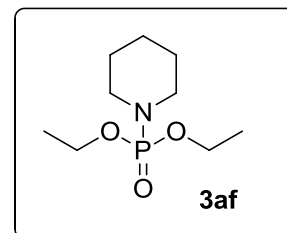
Diethyl piperidin-1-ylphosphonate¹: Yellow liquid; Yield - 82%; R_f

(100% MeOH/EtOAc) 0.4; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3468, 2981, 2935, 2854,

1384, 1244, 1167, 1057, 1028, 964; **^1H NMR** (400 MHz, CDCl_3): δ 4.09-3.95 (m, 4H), 3.11-3.07 (m, 4H), 1.6-1.57 (m, 3H), 1.56-1.51 (m,

3H), 1.31 (t, J = 7.2 Hz, 6H); **^{13}C NMR** (100 MHz, CDCl_3): δ 61.9

(d, J = 5.5 Hz), 45.2 (d, J = 2.0 Hz), 25.9 (d, J = 4.9 Hz), 24.3, 16.0 (d, J = 7.2 Hz); **^{31}P NMR** (162 MHz, CDCl_3): δ 8.864; **HRESI-MS** (m/z): Calculated for $\text{C}_9\text{H}_{20}\text{NO}_3\text{P}$ ($M + \text{Na}$): 244.1079, found ($M + \text{Na}$): 244.1077.

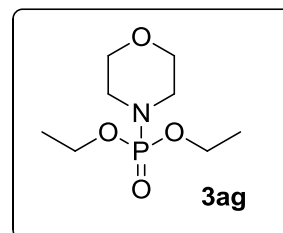


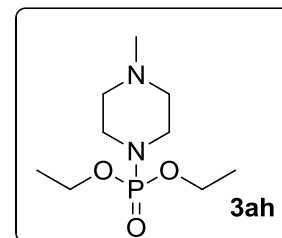
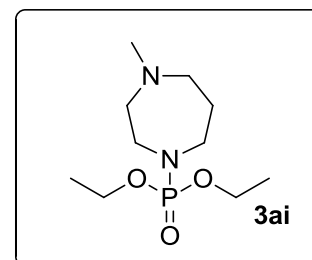
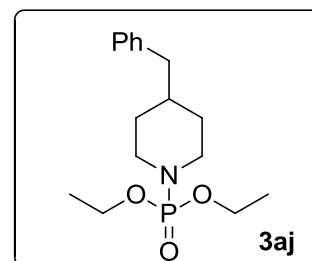
Diethyl morpholinophosphonate¹: Pale yellow liquid; Yield -

78%; R_f (100% MeOH/EtOAc) 0.7; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3448, 2981, 1647, 1254,

1022, 974; **^1H NMR** (400 MHz, CDCl_3): δ 4.08-4.03 (m, 4H), 3.66-3.64 (m, 4H), 3.16-3.12 (m, 4H), 1.33 (t, J = 5.6 Hz, 6H); **^{13}C**

NMR (100 MHz, CDCl_3): δ 66.9 (d, J = 5.8 Hz), 62.3 (d, J = 5.7 Hz), 44.5, 16.1 (d, J = 6.8 Hz); **^{31}P NMR** (162 MHz, CDCl_3): δ 7.749; **HRESI-MS** (m/z): Calculated for $\text{C}_8\text{H}_{18}\text{NO}_4\text{P}$ ($M + \text{Na}$): 246.0871, found ($M + \text{Na}$): 246.0872.

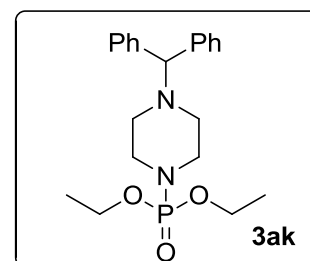


Diethyl (4-methylpiperazin-1-yl)phosphonate: Yellow liquid;Yield - 78%; R_f (100% MeOH/EtOAc) 0.6; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3436, 2981,1242, 1052, 1021, 978; **^1H NMR** (400 MHz, CDCl_3): δ 4.11-3.97 (m, 4H), 3.19-3.17 (m, 4H), 2.37-2.35 (m, 4H), 2.29 (s, 3H), 1.32(t, $J = 6.4$ Hz, 6H); **^{13}C NMR** (100 MHz, CDCl_3): δ 62.1 (d, $J = 5.5$ Hz), 55.0 (d, $J = 5.7$ Hz), 46.2, 44.2, (d, $J = 1.4$ Hz), 16.0 (d, $J = 7$ Hz); **^{31}P NMR** (162 MHz, CDCl_3): δ 8.315;**HRESI-MS** (m/z): Calculated for $\text{C}_9\text{H}_{21}\text{N}_2\text{O}_3\text{P}$ ($M + H$): 237.1368, found ($M + H$): 237.1366.**Diethyl (4-methyl-1,4-diazepan-1-yl)phosphonate:** Yellowliquid; Yield - 73%; R_f (100% MeOH/EtOAc) 0.5; Prepared asshown in general experimental procedure. **IR** (Neat, cm^{-1}): 3449,2936, 1655, 1239, 1058, 1025, 963; **^1H NMR** (400 MHz, CDCl_3): δ 4.10-3.96 (m, 4H), 3.33-3.24 (m, 4H), 2.64-2.60 (m, 4H), 2.37(s, 3H), 1.90-1.84 (m, 2H), 1.32 (t, $J = 7.2$ Hz, 6H); **^{13}C NMR**(100 MHz, CDCl_3): δ 62.0 (d, $J = 5.3$ Hz), 60.3, 56.8, 46.8, 45.9 (d, $J = 3.5$ Hz), 28.6 (d, $J =$ 5.8 Hz), 16.2 (d, $J = 7.1$ Hz); **^{31}P NMR** (162 MHz, CDCl_3): δ 9.575; **HRESI-MS** (m/z):Calculated for $\text{C}_{10}\text{H}_{23}\text{N}_2\text{O}_3\text{P}$ ($M + H$): 251.1525, found ($M + H$): 251.1520.**Diethyl (4-benzylpiperidin-1-yl)phosphonate:** Yellow liquid;Yield - 83%; R_f (50% EtOAc/Hexane) 0.4; Prepared as shown ingeneral experimental procedure. **IR** (Neat, cm^{-1}): 3439, 2924,1604, 1244, 1046, 1027, 959; **^1H NMR** (400 MHz, CDCl_3): δ 7.27 (t, $J = 7.2$ Hz, 2H), 7.18 (t, $J = 7.2$ Hz, 1H), 7.13 (d, $J = 7.2$ Hz, 2H), 4.08-3.93 (m, 4H), 3.54-3.50 (m, 2H), 2.64 (m, 2H), 2.53 (d, $J = 6.8$ Hz, 2H), 1.61(m, 3H), 1.30 (t, $J = 7.2$ Hz, 6H), 1.20-1.11 (m, 2H); **^{13}C NMR** (100 MHz, CDCl_3): δ 140.0,129.0, 128.1, 125.8, 62.0 (d, $J = 5.6$ Hz), 44.7 (d, $J = 2.3$ Hz), 43.2, 37.9, 32.2 (d, $J = 5.2$ 

Hz), 16.1 (d, $J = 7.0$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.816; **HRESI-MS** (m/z): Calculated for $\text{C}_{16}\text{H}_{26}\text{NO}_3\text{P}$ ($\text{M} + \text{Na}$): 334.1548, found ($\text{M} + \text{Na}$): 334.1549.

Diethyl (4-benzhydrylpiperazin-1-yl)phosphonate: Yellow liquid;

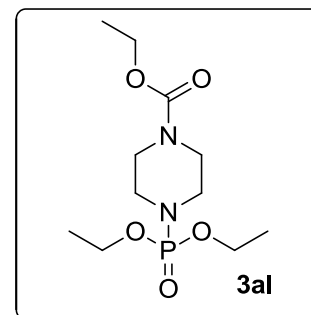
Yield - 76%; R_f (50% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3401, 1654, 1450, 1253, 1199, 1138, 1110, 1059, 1026, 979; ^1H NMR (400 MHz, CDCl_3): δ 7.40 (d, $J = 7.2$ Hz, 4H), 7.28-7.18 (m, 4H), 7.16 (t, $J = 4.0$ Hz, 2H), 4.23 (s, 1H), 4.08-3.94 (m, 4H), 3.18-3.14 (m, 4H),



2.35-2.32(m, 4H), 1.29 (t, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 142.3, 128.5, 127.9, 127.0, 76.3, 62.1 (d, $J = 5.6$ Hz), 52.1 (d, $J = 6.2$ Hz), 44.6, 16.2 (d, $J = 6.9$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.363; **HRESI-MS** (m/z): Calculated for $\text{C}_{21}\text{H}_{29}\text{N}_2\text{O}_3\text{P}$ ($\text{M} + \text{Na}$): 411.1814, found ($\text{M} + \text{Na}$): 411.1808.

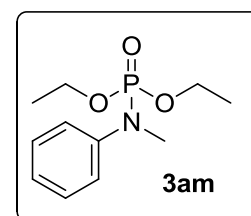
Ethyl 4-(diethoxyphosphoryl)piperazine-1-carboxylate: Pale

yellow liquid; Yield - 96%; R_f (100% EtOAc) 0.6; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3479, 2984, 2909, 1703, 1697, 1434, 1246, 1055, 1024, 968; ^1H NMR (400 MHz, CDCl_3): δ 4.15 (q, $J = 7.2$ Hz, 2H), 4.10-3.99 (m, 4H), 3.45-3.44 (m, 4H), 3.14-3.09 (m, 4H), 1.74 (s, 1H), 1.32 (t, $J = 6.8$ Hz, 6H), 1.26 (t, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ



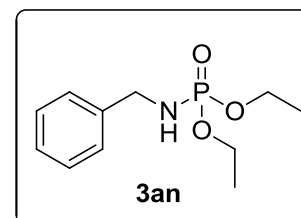
155.4, 62.4 (d, $J = 5.6$ Hz), 61.5, 44.2, 16.1 (d, $J = 6.8$ Hz), 14.6; ^{31}P NMR (162 MHz, CDCl_3): δ 8.022; **HRESI-MS** (m/z): Calculated for $\text{C}_{11}\text{H}_{23}\text{N}_2\text{O}_5\text{P}$ ($\text{M} + \text{Na}$): 317.1242, found ($\text{M} + \text{Na}$): 317.1242.

Diethyl methyl(phenyl)phosphoramidate: Brown liquid; Yield - 60%; R_f (30% EtOAc/Hexane) 0.2; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3474, 2983, 2932, 2907, 1600, 1497, 1276, 1047, 1024, 967; ^1H NMR (400 MHz, CDCl_3): δ

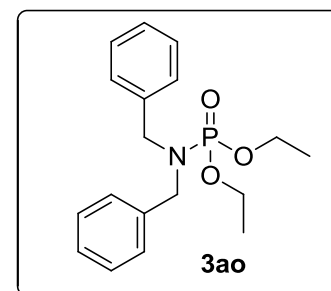


7.32-7.28(m, 3H), 7.08 (t, $J = 6.8$ Hz, 1H), 4.17-3.98 (m, 4H), 3.21 (d, $J = 8.8$ Hz, 3H), 1.28 (t, $J = 7.2$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 144.0 (d, $J = 5.0$ Hz), 128.9, 123.5, 121.8 (d, $J = 4$ Hz), 62.5 (d, $J = 6$ Hz), 36.8 (d, $J = 5$ Hz), 15.9 (d, $J = 7$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 5.857; **HRESI-MS** (m/z): Calculated for $\text{C}_{11}\text{H}_{18}\text{NO}_3\text{P}$ ($\text{M} + \text{Na}$): 266.0922, found ($\text{M} + \text{Na}$): 266.0925.

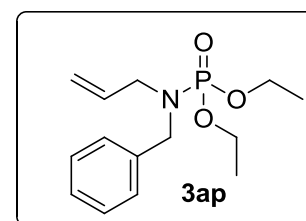
Diethyl benzylphosphoramidate¹: Orange liquid; Yield - 70%; R_f (50% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3422, 2982, 2929, 1646, 1457, 1261, 1054, 1027, 966; ^1H NMR (400 MHz, CDCl_3): δ 7.33-7.25(m, 5H), 4.11-3.98 (m, 6H), 3.22 (brs, 1H), 1.29 (t, $J = 7.2$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 139.6 (d, $J = 6.2$ Hz), 128.5, 128.3, 127.2, 62.3 (d, $J = 5.3$ Hz), 45.2, 16.9 (d, $J = 7$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 8.501; **HRESI-MS** (m/z): Calculated for $\text{C}_{11}\text{H}_{18}\text{NO}_3\text{P}$ ($\text{M} + \text{Na}$): 266.0922, found ($\text{M} + \text{Na}$): 266.0925.



Diethyl dibenzylphosphoramidate: Yellow liquid; Yield - 72%; R_f (30% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3449, 2982, 2930, 1637, 1253, 1056, 1027, 953; ^1H NMR (400 MHz, CDCl_3): δ 7.35-7.27(m, 10H), 4.18-4.00 (m, 8H), 1.32 (t, $J = 7.2$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 137.4 (d, $J = 2.5$ Hz), 128.6, 128.4, 127.3, 62.4 (d, $J = 4.9$ Hz), 48.3 (d, $J = 4.9$ Hz), 16.2 (d, $J = 7.3$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ 9.758; **HRESI-MS** (m/z): Calculated for $\text{C}_{18}\text{H}_{24}\text{NO}_3\text{P}$ ($\text{M} + \text{Na}$): 356.1392, found ($\text{M} + \text{Na}$): 356.1392.

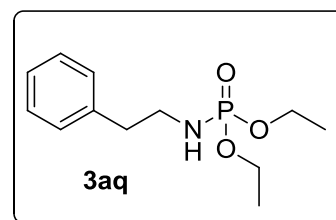


Diethyl allyl(benzyl)phosphoramidate: Yellow oil; Yield - 78%; R_f (30% EtOAc/Hexane) 0.4; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3460, 2981,



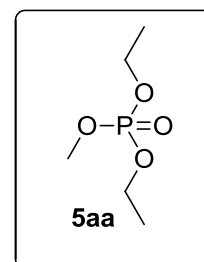
1638, 1369, 1252, 1054, 1027, 957; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.35-7.24 (m, 5H), 5.80-5.70 (m, 1H), 5.12 (dd, $J = 14$ Hz, 10 Hz, 2H), 4.17 (d, $J = 9.6$ Hz, 2H), 4.15- 3.98(m, 4H), 3.49 (dd, 6.4 Hz, 4.8 Hz, 2H), 1.32 (t, $J = 7.2$ Hz, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 137.8 (d, $J = 3.6$ Hz), 134.2, 128.5, 128.3, 127.2, 118.1, 62.3 (d, $J = 5.6$ Hz), 48.5 (d, $J = 4.9$ Hz), 16.1 (d, $J = 7.2$ Hz); $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 9.771; **HRESI-MS** (m/z): Calculated for $\text{C}_{14}\text{H}_{22}\text{NO}_3\text{P}$ ($\text{M} + \text{Na}$): 306.1235, found ($\text{M} + \text{Na}$): 306.1234.

Diethyl phenethylphosphoramidate: Pale green liquid; Yield - 71%; R_f (50% EtOAc/Hexane) 0.3; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3420, 3236, 2981, 1455, 1233, 1057, 1030, 965; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.32-7.24 (m, 2H), 7.22-7.18 (m, 3H), 4.09-3.93 (m, 4H), 3.21-3.14 (m, 2H), 2.79 (t, $J = 6.8$ Hz, 2H), 2.64 (d, $J = 8$ Hz, 1H), 1.30 (t, $J = 7.2$ Hz, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 138.6, 128.8, 128.6, 126.5, 62.1 (d, $J = 5.3$ Hz), 42.6, 37.9 (d, $J = 6.2$ Hz), 16.1 (d, $J = 7.0$ Hz); $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 8.970; **HRESI-MS** (m/z): Calculated for $\text{C}_{12}\text{H}_{20}\text{NO}_3\text{P}$ ($\text{M} + \text{Na}$): 280.1079, found ($\text{M} + \text{Na}$): 280.1077.

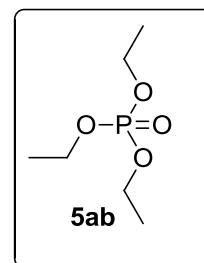


Characterization data for phosphate triester.

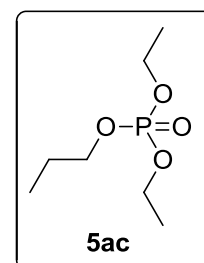
Diethyl methyl phosphate: Dark orange liquid; Yield - 97%; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3439, 2920, 1578, 1411, 1020, 848; $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 4.12 (q, $J = 7.2$ Hz, 4H), 3.76 (d, $J = 11.2$ Hz, 3H), 1.35 (t, $J = 7.2$ Hz, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 63.7 (d, $J = 6\text{Hz}$), 54.0 (d, $J = 6.1$ Hz), 16.0 (d, $J = 6$ Hz); $^{31}\text{P NMR}$ (162 MHz, CDCl_3): δ 0.116; **HRESI-MS** (m/z): Calculated for $\text{C}_5\text{H}_{13}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 191.0449, found ($\text{M} + \text{Na}$): 191.0455.



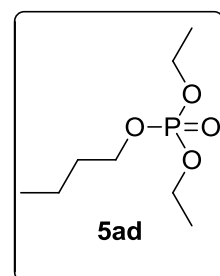
Triethyl phosphate: Pale yellow liquid; Yield - 96%; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3411, 2923, 1619, 1425, 1019; **^1H NMR** (400 MHz, CDCl_3): δ 4.11 (q, $J = 7.2$ Hz, 6H), 1.34 (t, $J = 7.2$ Hz, 9H); **^{13}C NMR** (100 MHz, CDCl_3): δ 63.5 (d, $J = 5.8$ Hz), 16.0 (d, $J = 6.7$ Hz); **^{31}P NMR** (162 MHz, CDCl_3): δ -1.004; **HRESI-MS** (m/z): Calculated for $\text{C}_6\text{H}_{15}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 205.0606, found ($\text{M} + \text{Na}$): 205.0607.



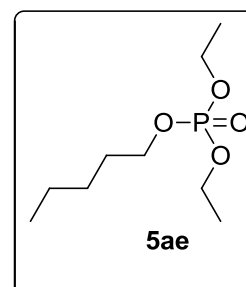
Diethyl propyl phosphate: Pale yellow liquid; Yield - 95%; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3489, 2979, 1266, 1033, 980; **^1H NMR** (400 MHz, CDCl_3): δ 4.11 (quin, $J = 7.2$ Hz, 4H), 4.0 (q, $J = 6.8$ Hz, 2H), 1.71 (sex, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 6.8$ Hz, 6H), 0.97 (t, $J = 7.6$ Hz, 3H); **^{13}C NMR** (100 MHz, CDCl_3): δ 69.1 (d, $J = 6.2$ Hz), 63.6 (d, $J = 5.7$ Hz), 23.6 (d, $J = 6.0$ Hz), 16.1 (d, 5.7 Hz), 9.9 ; **^{31}P NMR** (162 MHz, CDCl_3): δ -0.916; **HRESI-MS** (m/z): Calculated for $\text{C}_7\text{H}_{17}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 219.0762, found ($\text{M} + \text{Na}$): 219.0768.



Butyl diethyl phosphate: Orange liquid; Yield - 97%; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3483, 2963, 1265, 1031, 980; **^1H NMR** (400 MHz, CDCl_3): δ 4.11 (quin, $J = 8.0$ Hz, 4H), 4.0 (q, $J = 7.6$ Hz, 2H), 1.67 (quin, $J = 7.8$ Hz, 2H), 1.41 (sex, $J = 8.0$ Hz, 2H), 1.34 (t, $J = 8.1$ Hz, 6H), 0.94 (t, $J = 8.0$ Hz, 3H); **^{13}C NMR** (100 MHz, CDCl_3): δ 67.3 (d, $J = 5.8$ Hz), 63.6 (d, $J = 5.9$ Hz), 32.2 (d, $J = 6.7$ Hz), 18.6, 16.1 (d, $J = 6.8$ Hz), 13.5 ; **^{31}P NMR** (162 MHz, CDCl_3): δ -0.868; **HRESI-MS** (m/z): Calculated for $\text{C}_8\text{H}_{18}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 233.0919, found ($\text{M} + \text{Na}$): 233.0914.



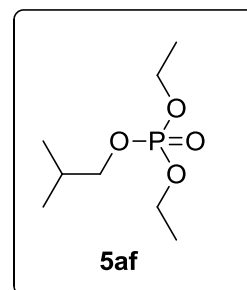
Diethyl pentyl phosphate: Pale green liquid; Yield - 97%; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3482, 2960, 1264, 1033, 980; **^1H NMR** (400 MHz, CDCl_3): δ 4.11 (quin, $J = 8.0$ Hz,



4H), 4.0 (q, $J = 8.0$ Hz, 2H), 1.68 (t, $J = 4$ Hz, 2H), 1.36-1.32 (m, 10H), 0.91 (t, $J = 4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 67.6 (d, $J = 6$ Hz), 63.6 (d, $J = 6$ Hz), 29.9 (d, $J = 6.2$ Hz), 27.5, 22.1, 16.1 (d, $J = 5.8$ Hz), 13.9; ^{31}P NMR (162 MHz, CDCl_3): δ -0.915; **HRESI-MS** (m/z): Calculated for $\text{C}_9\text{H}_{21}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 247.1075, found ($\text{M} + \text{Na}$): 247.1074.

Diethyl isobutyl phosphate:² Pale yellow liquid; Yield - 96%; Prepared as

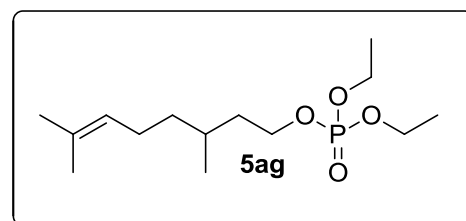
shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3487, 2966, 1263, 1026, 975, 866; ^1H NMR (400 MHz, CDCl_3): δ 4.11 (m, 4H), 3.80 (t, $J = 8.0$ Hz, 2H), 1.96 (m, 1H), 1.34 (t, 7.2 Hz, 6H), 0.95 (d, $J = 6.8$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 73.5 (d, $J = 6.3$ Hz), 63.6 (d, $J = 5.8$ Hz), 29.0 (d, $J = 7.2$ Hz), 18.6, 16.1 (d, $J = 6.6$ Hz); ^{31}P NMR (162 MHz,



CDCl_3): δ -0.870; **HRESI-MS** (m/z): Calculated for $\text{C}_8\text{H}_{19}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 233.0919, found ($\text{M} + \text{Na}$): 233.0916.

3,7-dimethyloct-6-en-1-yl diethyl phosphate:² Orange

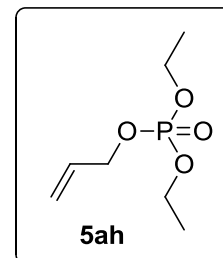
liquid; Yield - 74%; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3736, 2965, 2927, 1525, 1263, 1032; ^1H NMR (400 MHz, CDCl_3): δ 5.08 (t, $J = 6.7$ Hz, 1H), 4.10-4.14 (m, 6H), 2.02-1.92 (m, 2H), 1.78-1.71



(m, 1H), 1.68 (s, 3H), 1.64-1.52 (m, 4H), 1.52-1.41 (m, 1H), 1.39-1.28 (m, 7H), 1.22-1.12 (m, 2H), 0.91 (d, $J = 6.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 131.3, 124.5, 77.3, 66.0 (d, $J = 6$ Hz), 37.1 (d, $J = 7$ Hz), 36.9, 28.9, 25.7, 25.3, 19.2, 17.6, 16.1 (d, $J = 6.7$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ -0.831; **HRESI-MS** (m/z): Calculated for $\text{C}_{14}\text{H}_{29}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 315.1701, found ($\text{M} + \text{Na}$): 315.1704.

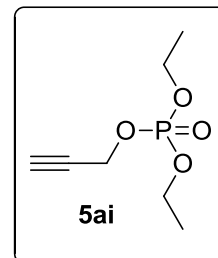
Allyl diethyl phosphate:² Dark orange liquid; Yield - 95%; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3854, 3736,

1683, 1526, 1260, 1020; ^1H NMR (400 MHz, CDCl_3): δ 6.02-5.9 (m, 1H), 5.37 (d, $J = 17$ Hz, 1H), 5.25 (d, $J = 10.4$ Hz, 1H), 4.53 (t, $J = 6.8$ Hz, 2H), 4.12 (m, 4H), 1.34 (t, 7.2 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 132.6

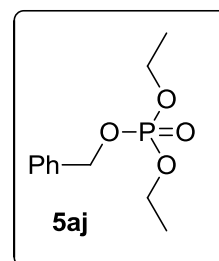


(d, $J = 6.8$ Hz), 118.0, 67.9 (d, $J = 5.4$ Hz), 63.8 (d, $J = 5.6$ Hz), 16.0 (d, $J = 6.6$ Hz); ^{31}P NMR (162 MHz, CDCl_3); δ -0.958; **HRESI-MS** (m/z): Calculated for $\text{C}_7\text{H}_{15}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 217.0606, found ($\text{M} + \text{Na}$): 217.0606.

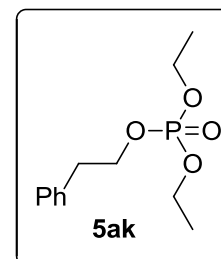
Diethyl prop-2-yn-1-yl phosphate:² Pale green liquid; Yield - 89%; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3220, 2986, 2125, 1652, 1523, 1260, 1023; ^1H NMR (400 MHz, CDCl_3): δ 4.67 (dd, $J = 8$ Hz, $J_2 = 2.4$ Hz, 2H), 4.15 (m, 4H), 2.57 (t, $J = 2.4$ Hz, 1H), 1.36 (t, 7.2 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 77.7 (d, $J = 7.5$ Hz), 75.7, 64.1 (d, $J = 5.7$ Hz), 54.8 (d, $J = 4.6$ Hz), 16.0 (d, $J = 6.8$ Hz); ^{31}P NMR (162 MHz, CDCl_3); δ -1.089; **HRESI-MS** (m/z): Calculated for $\text{C}_7\text{H}_{13}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 215.0449, found ($\text{M} + \text{Na}$): 215.0447.



Benzyl diethyl phosphate:² Pale yellow liquid; Yield - 82%; R_f (30% EtOAc/Hexane) 0.3. Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 2985, 1265, 1025, 983; ^1H NMR (400 MHz, CDCl_3): δ 7.41-7.31 (m, 5H), 5.06 (d, $J = 8.0$ Hz, 2H), 4.15-4.02 (m, 4H), 1.30 (t, 8 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 136.0 (d, $J = 7.0$ Hz), 128.5, 128.4, 127.8, 69.0 (d, $J = 5.2$ Hz), 63.8 (d, $J = 5.4$ Hz), 16.0 (d, $J = 6.0$ Hz); ^{31}P NMR (162 MHz, CDCl_3); δ -0.961; **HRESI-MS** (m/z): Calculated for $\text{C}_{11}\text{H}_{17}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 267.0762, found ($\text{M} + \text{Na}$): 267.0763.



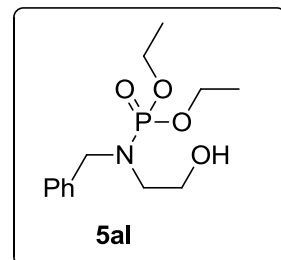
Diethyl phenethyl phosphate:² Pale green liquid; Yield - 71%; R_f (30% EtOAc/Hexane) 0.2. Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3448, 2983, 1266, 1029, 975; ^1H NMR (400 MHz, CDCl_3): δ 7.32-7.24 (m, 2H), 7.23-7.22 (m, 3H), 4.23 (q, $J = 7.8$ Hz, 2H), 4.03 (m, 4H), 2.99 (t, $J = 6.0$ Hz, 2H), 1.28 (t, 6.8 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 137.1, 128.9, 128.4, 126.6, 67.8 (d, $J = 6.1$ Hz), 63.6 (d, J



= 6 Hz), 36.7 (d, J = 6 Hz), 16.0 (d, J = 6.1 Hz); ^{31}P NMR (162 MHz, CDCl_3); δ -1.141; **HRESI-MS** (m/z): Calculated for $\text{C}_{12}\text{H}_{19}\text{O}_4\text{P}$ ($\text{M} + \text{Na}$): 281.0919, found ($\text{M} + \text{Na}$): 281.0921.

Diethyl (2-hydroxyethyl)(phenyl)phosphoramidate: Liquid; Yield - 44%;

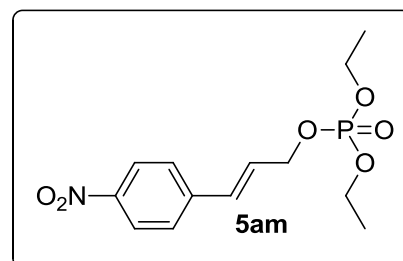
R_f (100% EtOAc) 0.3. Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3510(br), 2892, 1266, 1029, 975; ^1H NMR (400 MHz, CDCl_3): δ 7.34-7.27 (m, 5H), 4.23 (d, J = 9.5 Hz, 2H), 4.19-4.04 (m, 4H), 3.61 (t, J = 5.0 Hz, 2H), 3.16-3.11 (m, 2H), 1.34 (t, 7.3 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 137.6, 129.0, 128.5, 128.2, 127.5, 126.3, 77.2,



62.8 (d, J = 6.3 Hz), 60.6, 50.2 (d, J = 4.5 Hz), 48.2 (d, J = 4.8 Hz), 16.1 (d, J = 7.1 Hz); ^{31}P NMR (162 MHz, CDCl_3); δ 11.36; **HRESI-MS** (m/z): Calculated for $\text{C}_{13}\text{H}_{22}\text{NO}_4\text{P}$ ($\text{M} + \text{Na}$): 310.1184, found ($\text{M} + \text{Na}$): 310.1186.

(E)-diethyl (3-(4-nitrophenyl)allyl) phosphate: Yellow

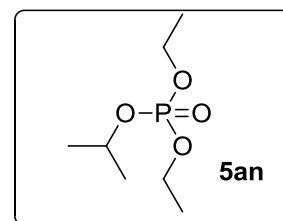
liquid; Yield - 50%; R_f (50% EtOAc/Hexane) 0.4. Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3430, 1636, 1518, 1344, 1261, 1027, 979; ^1H NMR (400 MHz, CDCl_3): δ 8.20 (d, J = 8.8 Hz, 2H), 7.53 (d, J = 8.8 Hz, 2H), 6.76 (d, J = 16 Hz, 1H), 6.47 (dt, J = 8 Hz, 5.6 Hz,



1H), 4.80-4.73 (m, 2H), 4.15 (m, 4H), 1.36 (t, 8 Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 147.3, 142.4, 130.7, 128.5 (d, J = 7 Hz), 127.2, 124.0, 123.7, 67.0 (d, J = 5.3 Hz), 64.0 (d, J = 5.3 Hz), 16.1 (d, J = 6.7 Hz); ^{31}P NMR (162 MHz, CDCl_3); δ -0.0827; **HRESI-MS** (m/z): Calculated for $\text{C}_{13}\text{H}_{18}\text{NO}_6\text{P}$ ($\text{M} + \text{Na}$): 338.0769, found ($\text{M} + \text{Na}$): 338.0769.

Diethyl isopropyl phosphate: ² Colorless liquid; Yield - 97%;

Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3421, 2982, 1683, 1522, 1260, 1016, 814; ^1H NMR (400



MHz, CDCl_3): δ 4.65 (sep, J = 6.4 Hz, 1H), 4.01 (m, 4H), 1.34 (m, 12H); ^{13}C NMR (100 MHz, CDCl_3): δ 72.4 (d, J = 5.9 Hz), 63.4 (d, J = 5.9 Hz), 23.6 (d, J = 5.1 Hz), 16.1 (d, J = 6.3 Hz); ^{31}P NMR (162 MHz, CDCl_3): δ -1.850; HRESI-MS (m/z): Calculated for $\text{C}_7\text{H}_{17}\text{O}_4\text{P}$ ($M + \text{Na}$): 219.0762, found ($M + \text{Na}$): 219.0762.

Diethyl ((1R,2S,5R)-2-isopropyl-5-methylcyclohexyl)

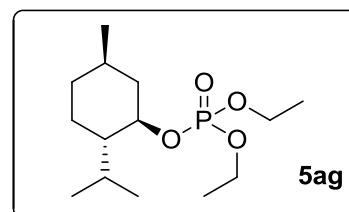
phosphate:² Liquid; Yield - 32%; Prepared as shown in general experimental procedure. IR (Neat, cm^{-1}): 3435, 2956,

2928, 2871, 1644, 1261, 1015; ^1H NMR (400 MHz, CDCl_3):

δ 4.19-4.05 (m, 5H), 2.25-2.13 (m, 2H), 1.67-1.59(m, 3H),

1.36-1.25 (m, 10H), 0.92-0.86 (m, 5H), 0.81 (d, J = 7 Hz, 4H); ^{13}C NMR (100 MHz, CDCl_3): δ 79.1 (d, J = 6.6 Hz), 77.2, 63.42 (d, J = 6.3 Hz), 48.47 (d, J = 7.27Hz), 42.5, 34.0,

31.5, 25.5, 22.8, 21.9, 20.9, 16.1 (d, J = 6.8 Hz), 15.6; ^{31}P NMR (162 MHz, CDCl_3): δ -1.634; HRESI-MS (m/z): Calculated for $\text{C}_{14}\text{H}_{29}\text{O}_4\text{P}$ ($M + \text{Na}$): 315.1701, found ($M + \text{Na}$): 315.1706.



(3S,8S,9S,10R,13R,14S,17R)-10,13-dimethyl-17-((R)-6-methylheptan-2-yl)-

2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl

diethyl phosphate:² Liquid; Yield - 23%;

Prepared as shown in general experimental procedure. IR (Neat, cm^{-1}):

3445, 2986, 2929, 2865, 1620, 1258,

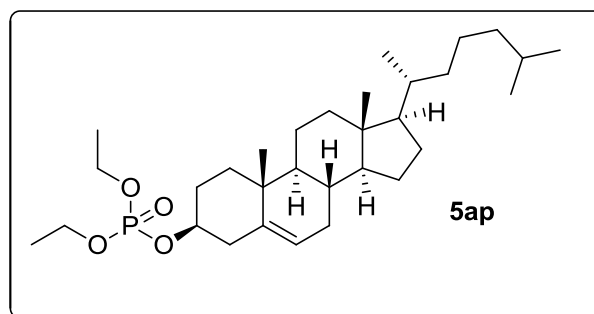
1112; ^1H NMR (400 MHz, CDCl_3): δ

5.38-5.37 (m, 1H), 4.25-4.06 (m, 4H),

2.44-2.42 (m, 1H), 2.02-1.94 (m, 3H),

1.87-1.80 (m, 2H), 1.70-1.25 (m, 25H), 1.22-1.0 (m, 6H), 0.94-0.85 (m, 10H), 0.68 (s, 3H);

^{13}C NMR (100 MHz, CDCl_3): δ 139.4, 122.9, 63.5 (d, J = 6.8 Hz), 56.7, 56.1, 50.0, 42.31, 39.7, 39.5, 36.9, 36.4, 36.1, 35.8, 31.9, 31.0, 29.6, 28.2, 28.0, 24.3, 23.8, 22.8, 22.5, 21.0.

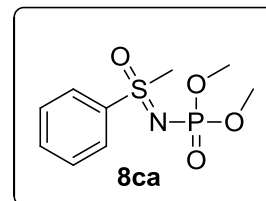


19.2, 18.7, 16.1 (d, $J = 7.12$ Hz), 11.84; ^{31}P NMR (162 MHz, CDCl_3); δ -1.860; **HRESI-MS** (m/z): Calculated for $\text{C}_{31}\text{H}_{55}\text{O}_4\text{P}$ ($M + \text{Na}$): 545.3736, found ($M + \text{Na}$): 545.3737.

Characterization data for CHDC reaction of sulfoximines with dialkyl *H*- phosphites.

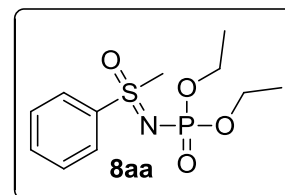
Dimethyl (S-methylsulfonimidoyl)benzene phosphoroamidate: Yellow oil; Yield - 50%;

R_f (100% EtOAc) 0.2; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3433, 2954, 2924, 1254, 1168, 1034, 834; ^1H NMR (400 MHz, CDCl_3): δ 8.04 (d, $J = 8.0$ Hz, 2H), 7.69 (t, $J = 7.2$ Hz, 1H), 7.60 (t, $J = 7.6$ Hz, 2H), 3.75 (dd, $J = 12$ Hz, 12 Hz, 6H), 3.37 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 140.3 (d, $J = 9.3$ Hz), 133.9, 129.5, 127.2, 53.4 (d, $J = 5.9$ Hz), 46.8 (d, $J = 3.0$ Hz); ^{31}P NMR (162 MHz, CDCl_3); δ 0.698; **HRESI-MS** (m/z): Calculated for $\text{C}_9\text{H}_{14}\text{NO}_4\text{PS}$ ($M + \text{Na}$): 286.0279, found ($M + \text{Na}$): 286.0275.



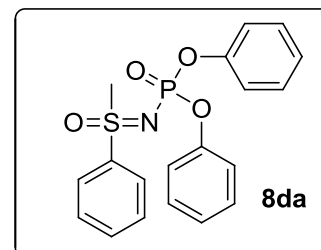
Diethyl (S-methylsulfonimidoyl)benzene phosphoroamidate:³

Dark brown oil; Yield - 31%; R_f (100% EtOAc) 0.2; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3399, 2981, 1259, 1163, 1022, 748; ^1H NMR (400 MHz, CDCl_3): δ 8.04 (d, $J = 8.0$ Hz, 2H), 7.69 (t, $J = 7.6$ Hz, 1H), 7.60 (t, $J = 7.6$ Hz, 2H), 4.15-4.05 (m, 4H), 3.38 (s, 3H), 1.30 (m, 6H); ^{13}C NMR (100 MHz, CDCl_3): δ 140.3 (d, $J = 8.8$ Hz), 133.8, 129.4, 127.2, 62.7 (d, $J = 6.1$ Hz), 46.8 (d, $J = 3.5$ Hz), 16.1 (d, $J = 7.9$ Hz); ^{31}P NMR (162 MHz, CDCl_3); δ -2.020; **HRESI-MS** (m/z): Calculated for $\text{C}_{11}\text{H}_{18}\text{NO}_4\text{PS}$ ($M + \text{Na}$): 314.0592, found ($M + \text{Na}$): 314.0593.



Diphenyl (S-methylsulfonimidoyl)benzene phosphoroamidate: Yellow oil; Yield - 35%;

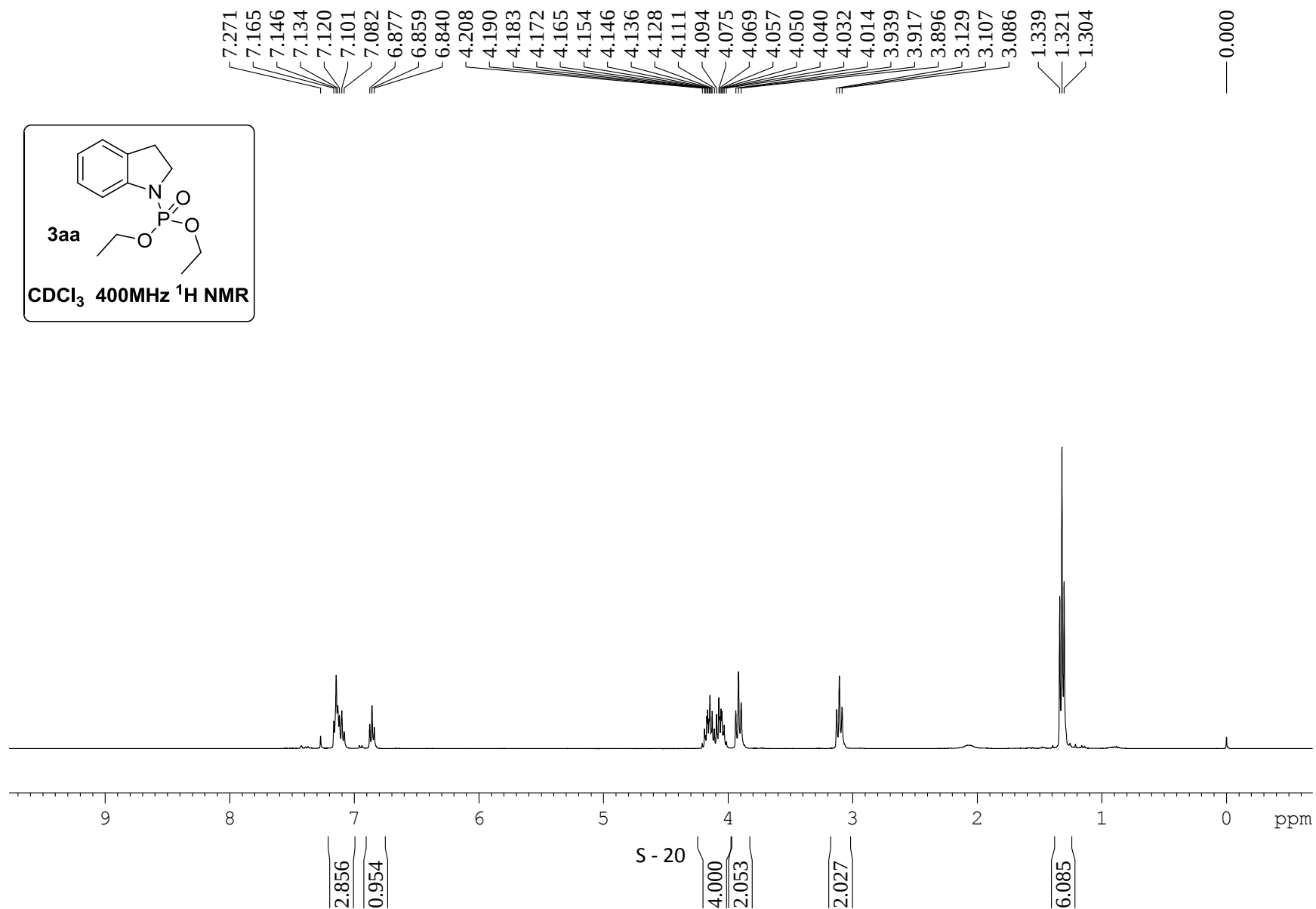
R_f (100% EtOAc) 0.2; Prepared as shown in general experimental procedure. **IR** (Neat, cm^{-1}): 3468, 2926, 1489, 1216, 1193, 1159, 929; ^1H NMR (400 MHz, CDCl_3): δ 7.84 (d, $J = 7.6$ Hz, 2H), 7.66

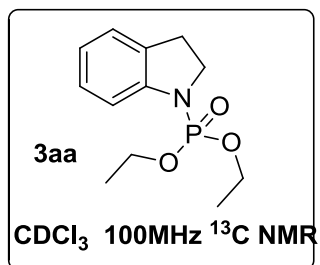


(t, $J = 4.0$ Hz, 1H), 7.51 (t, $J = 7.6$ Hz, 2H), 7.33-7.23 (m, 8H), 7.15 (t, $J = 6.8$ Hz, 2H), 3.32 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 151.2 (d, $J = 8.6$ Hz), 151.0, 139.9 (d, $J = 9.3$ Hz), 133.9, 129.5 (d, $J = 10.9$ Hz), 129.4 (d, $J = 2.7$ Hz), 127.1 (d, $J = 9.1$ Hz), 124.8, 120.7 (d, $J = 4.9$ Hz), 120.6 (d, $J = 5.1$ Hz), 46.8 (d, $J = 3.2$ Hz); ^{31}P NMR (162 MHz, CDCl_3): δ -12.172; **HRESI-MS** (m/z): Calculated for $\text{C}_{19}\text{H}_{18}\text{NO}_4\text{PS}$ (M + Na): 410.0592, found (M + Na): 410.0590.

References:

1. Fraser, J.; Wilson, L. J.; Blundell, R. K.; Hayes, C. J. *Chem. Commun.* **2013**, 49, 8919.
2. (a) Jones, S.; Selitsianos, D. *Org. Lett.* **2002**, 4, 3671-3673. (b) Jones, S.; Selitsianos, D.; Thompson, K. J.; Toms, S. M. *J. Org. Chem.* **2003**, 68, 5211-5216. (c) Jones, S.; Smanmoo, C. *Org. Lett.* **2005**, 7, 3271. (d) Liu, C.-Y.; Pawar, V. D.; Kao, J.-Q.; Chen, C.-T. *Adv. Synth. Catal.* **2010**, 352, 188–194.
3. Reinhard, B. M.; Licastro, S. A. *Molecules* **2005**, 10, 1369.





144.707
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124.696
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112.220

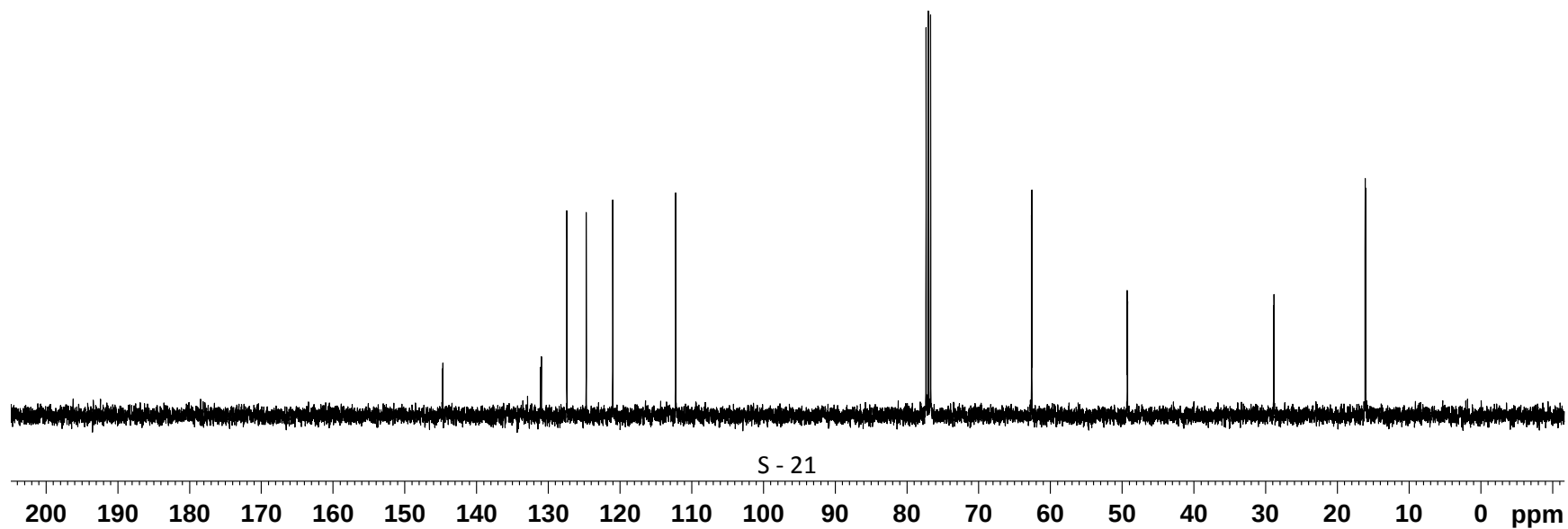
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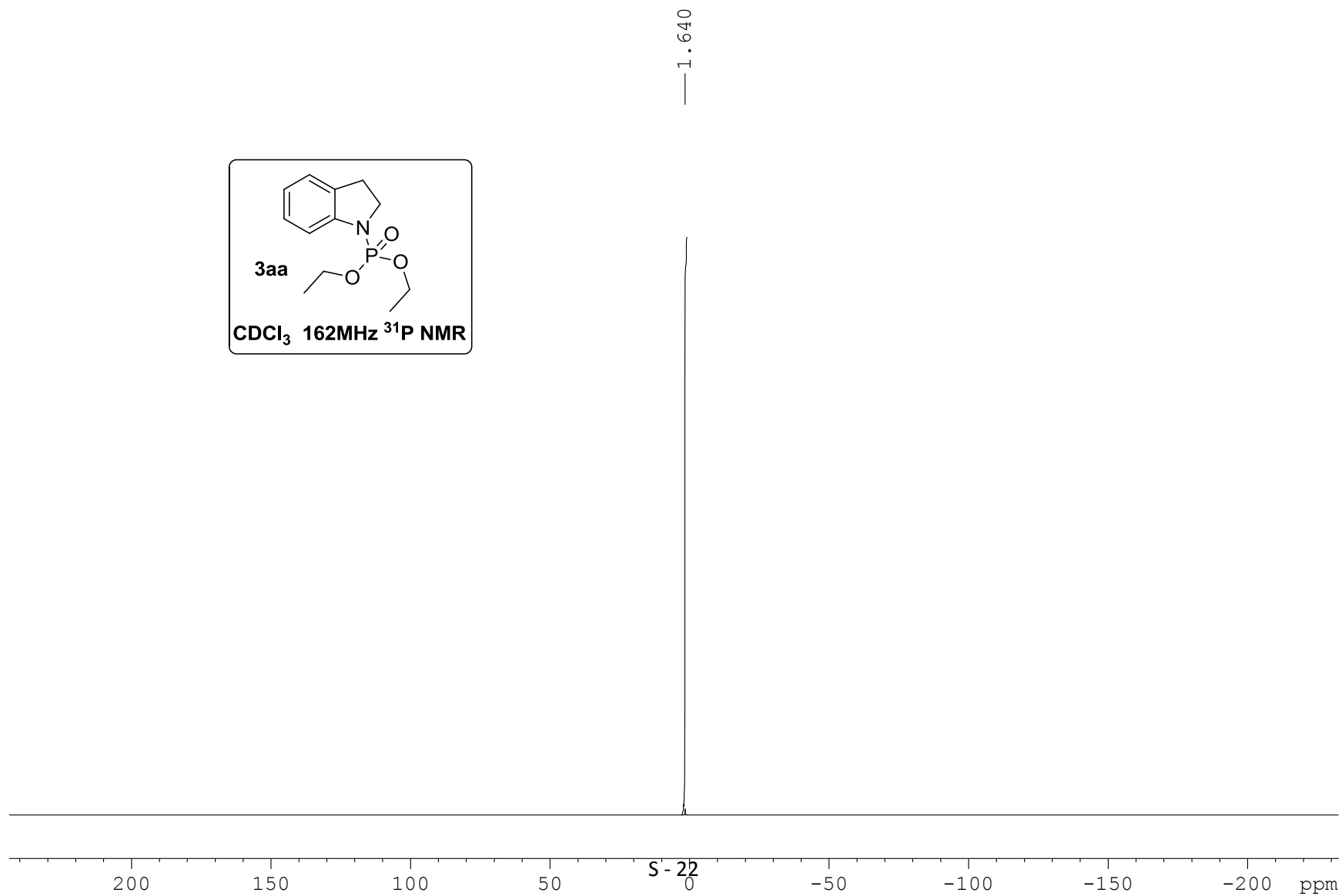
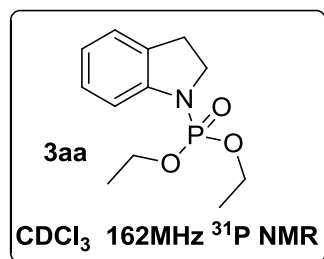
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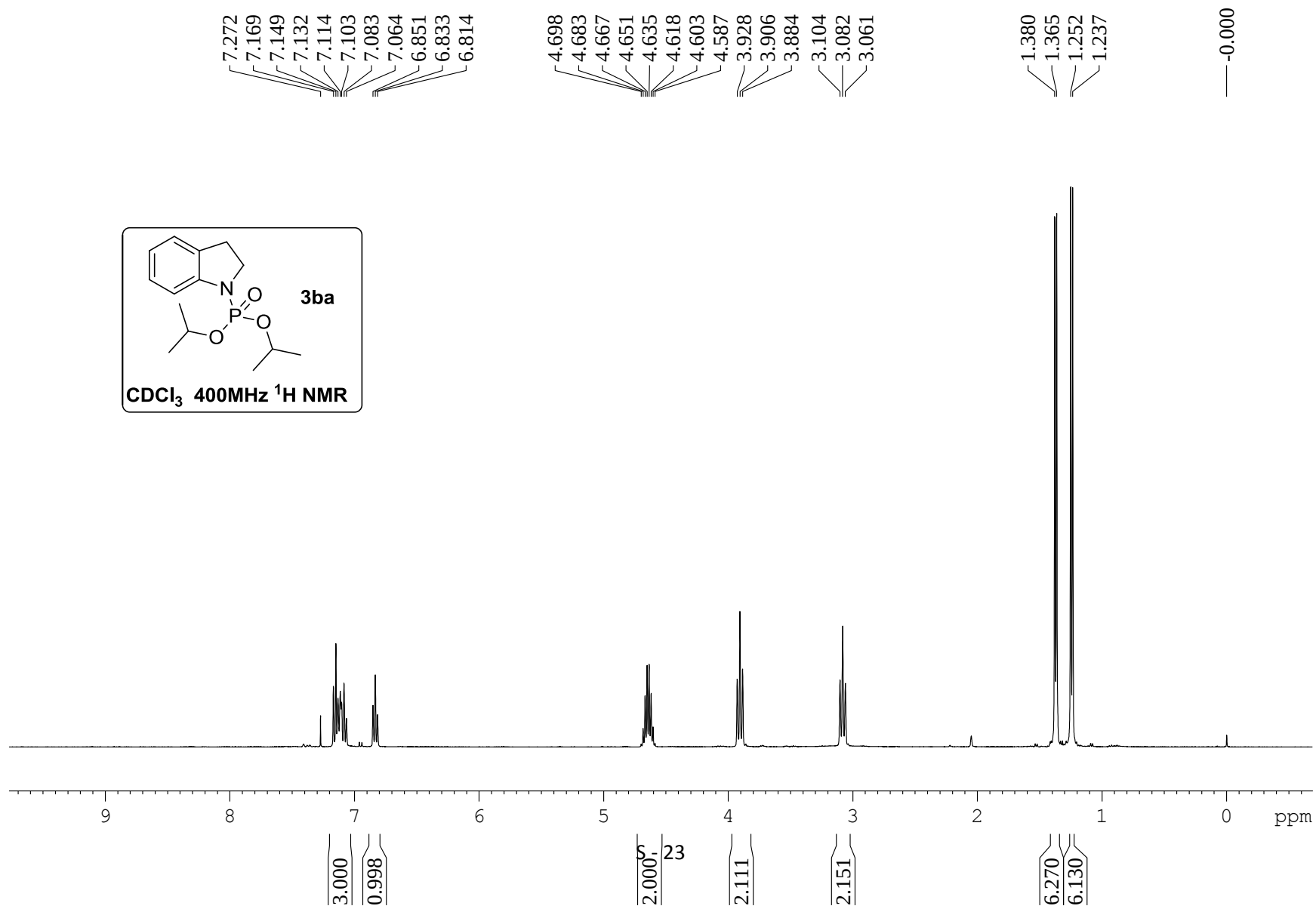
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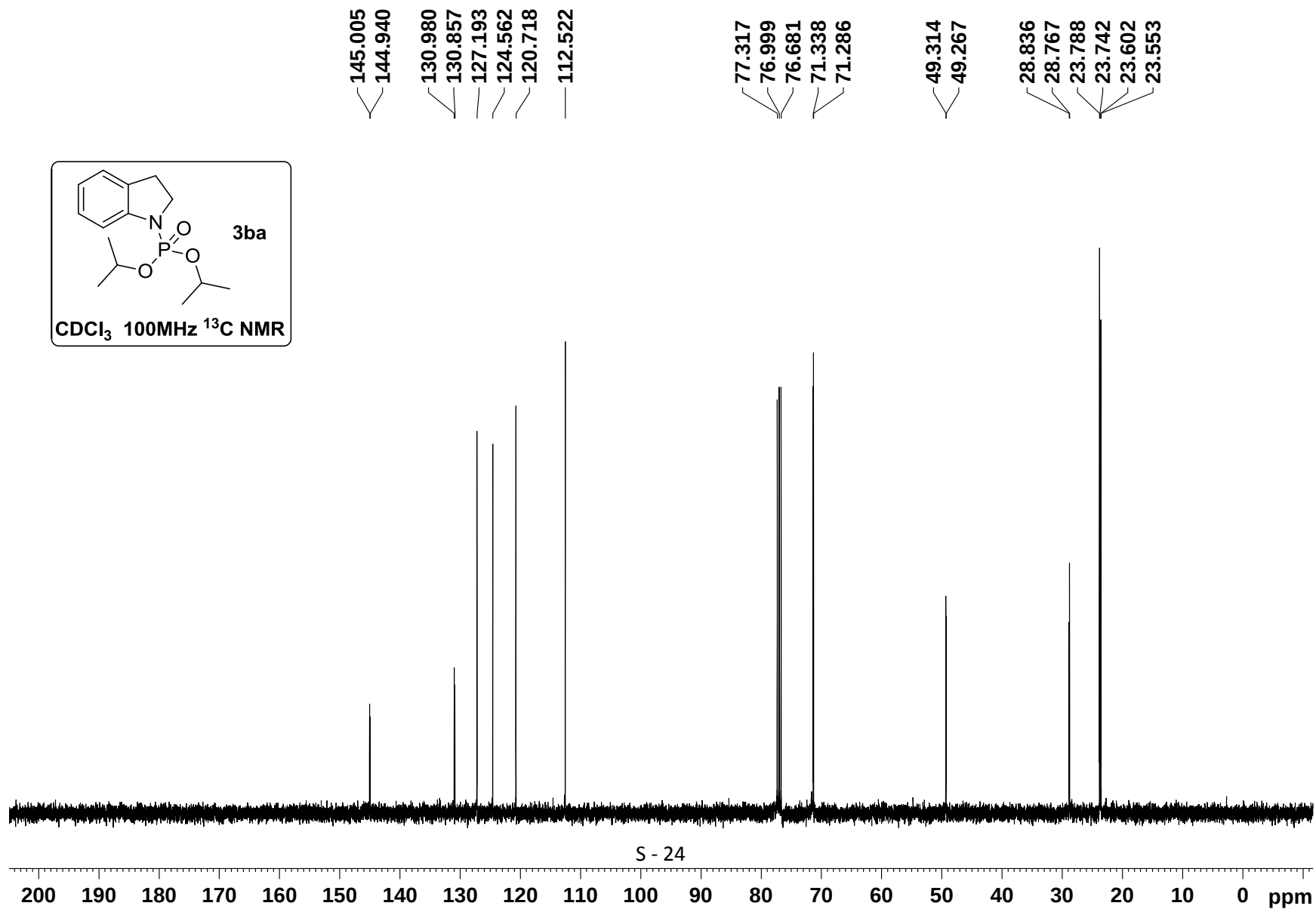
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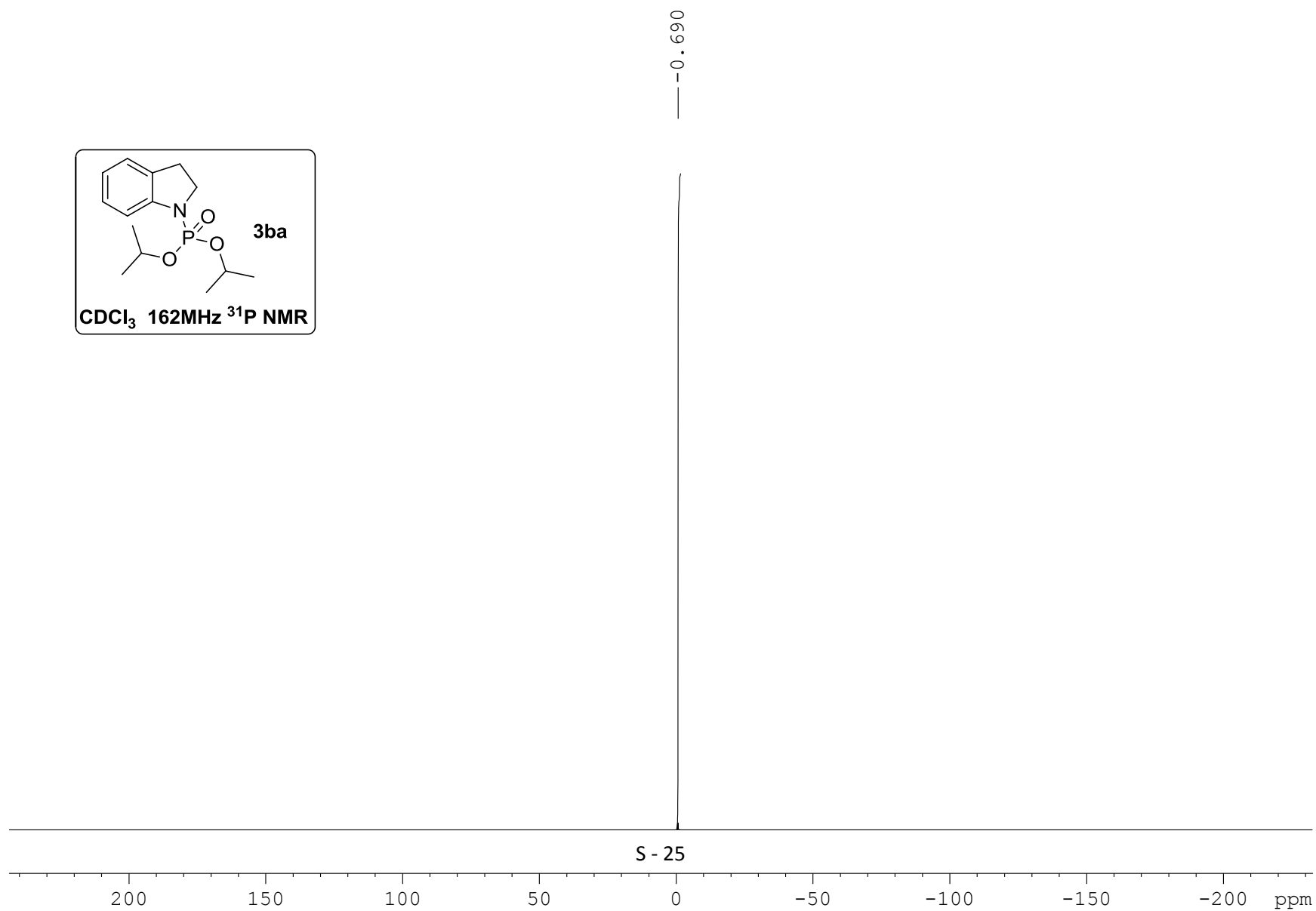
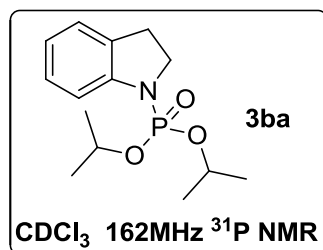
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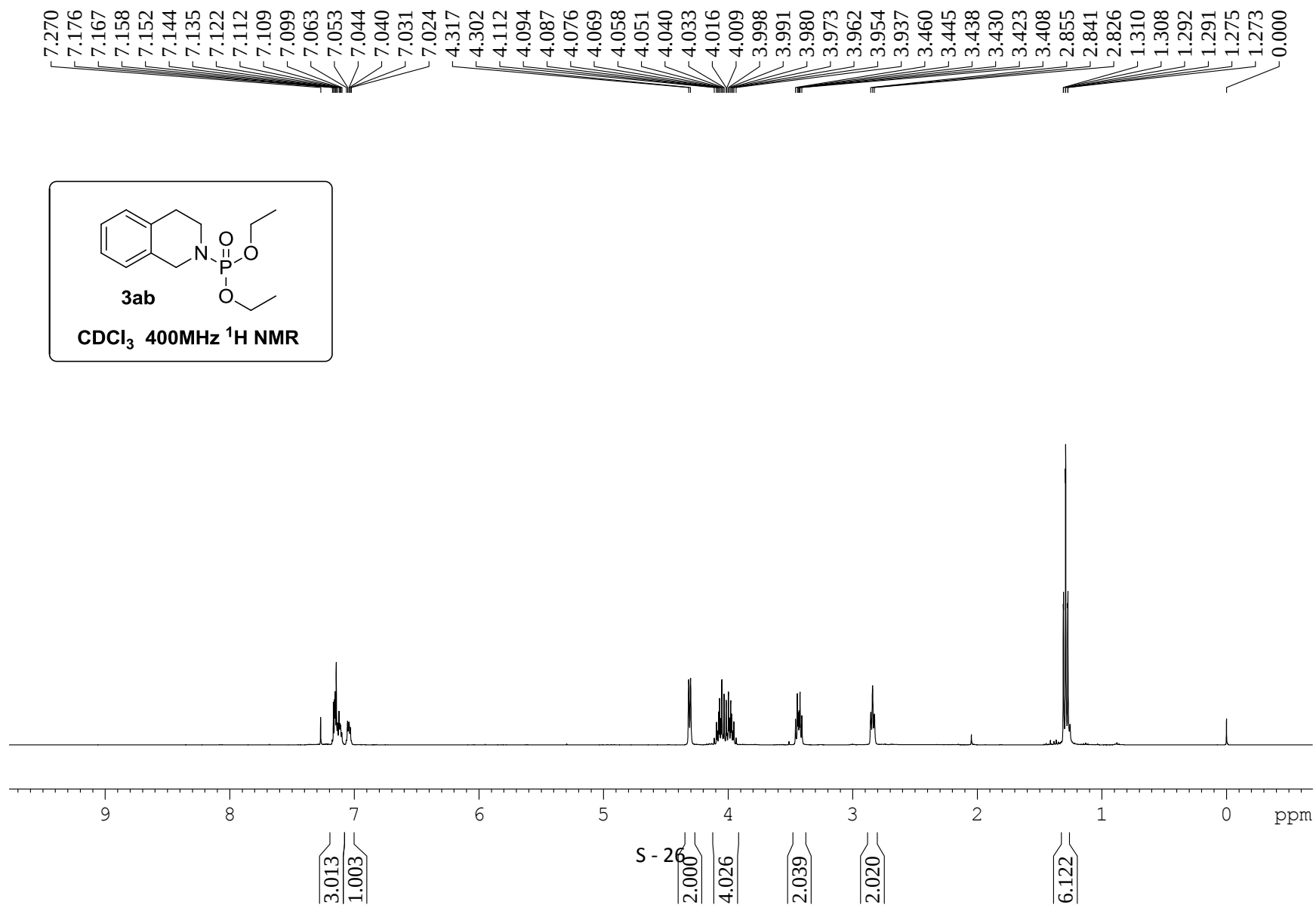


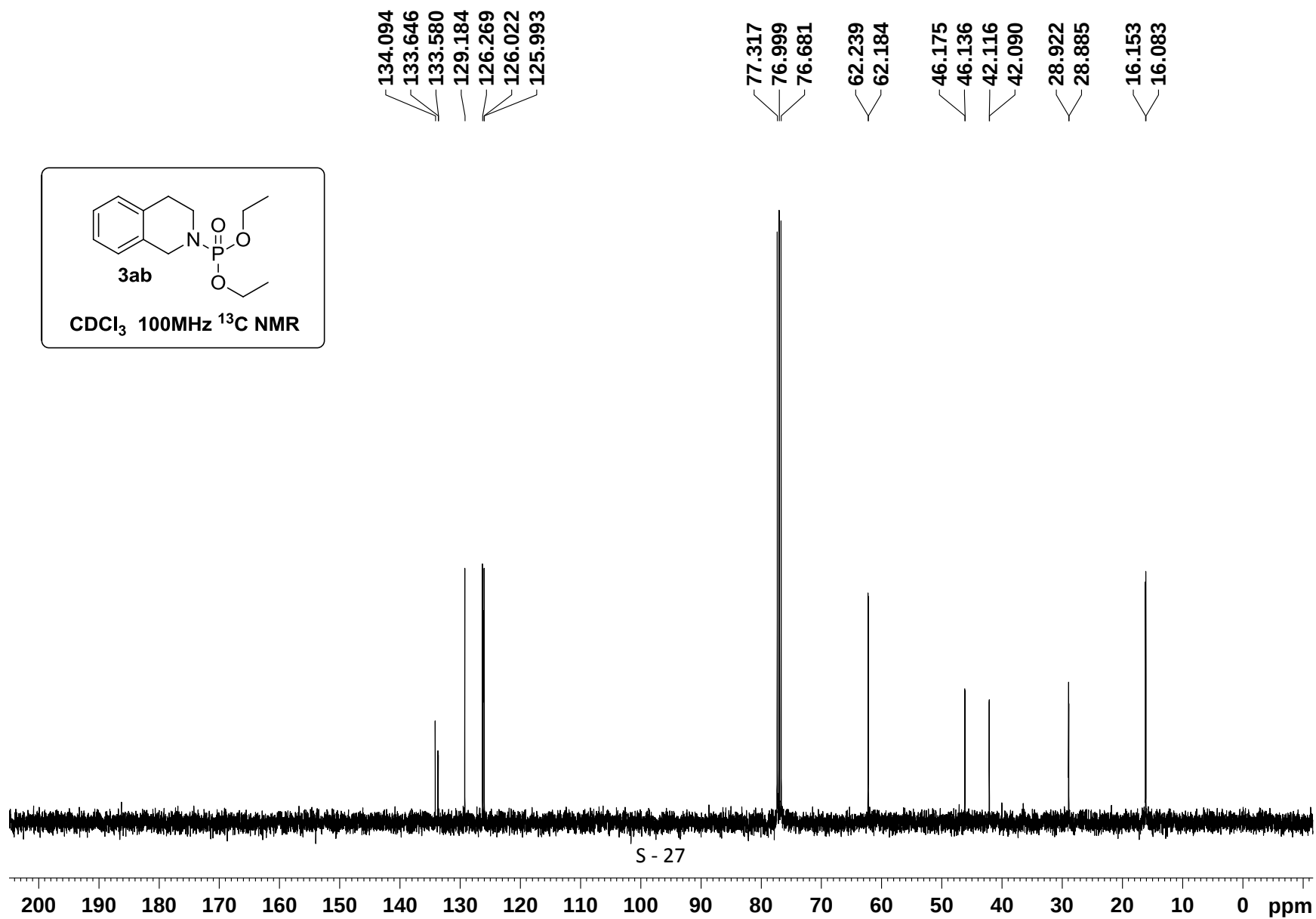


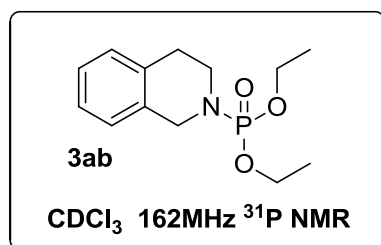




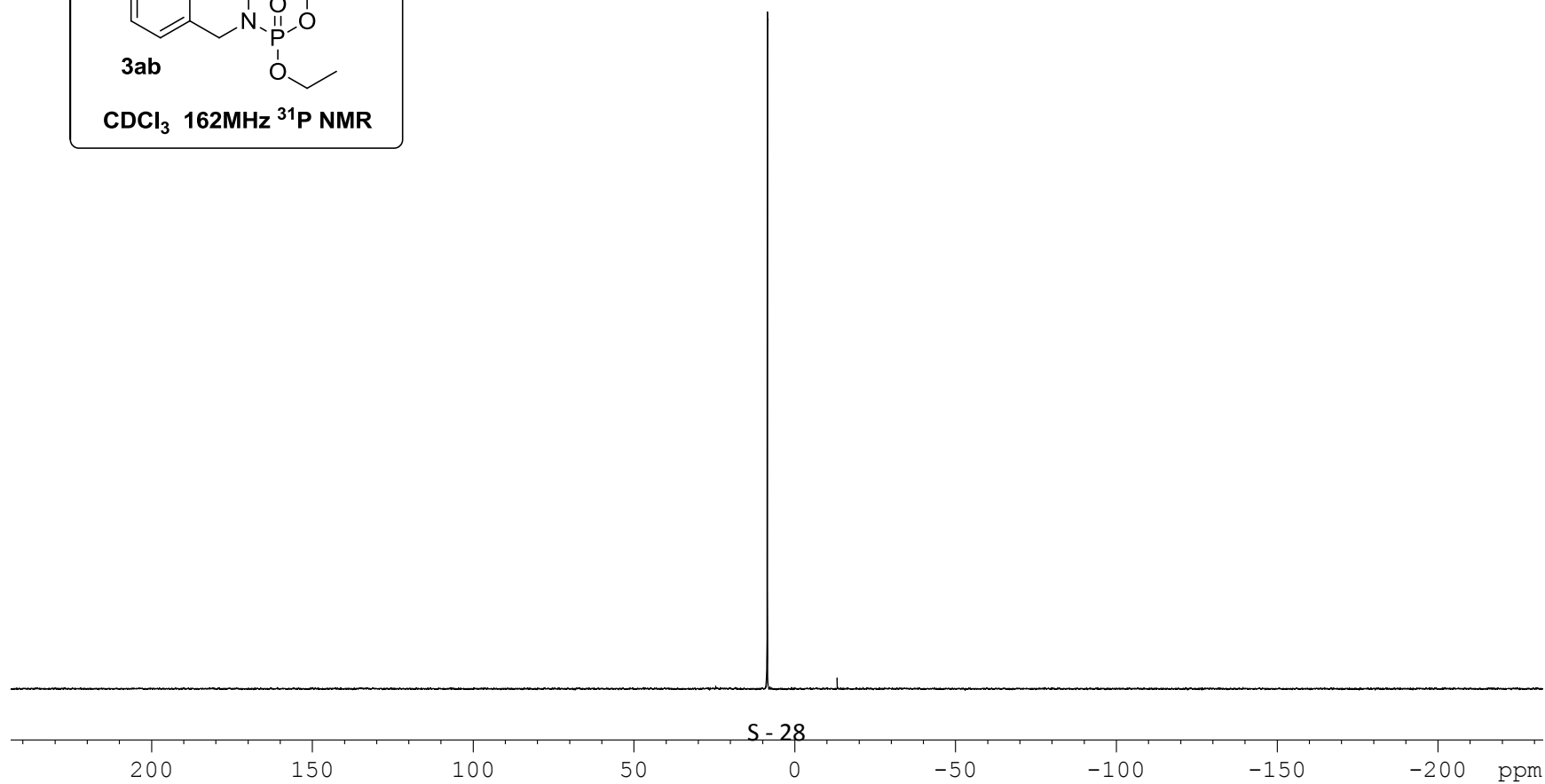


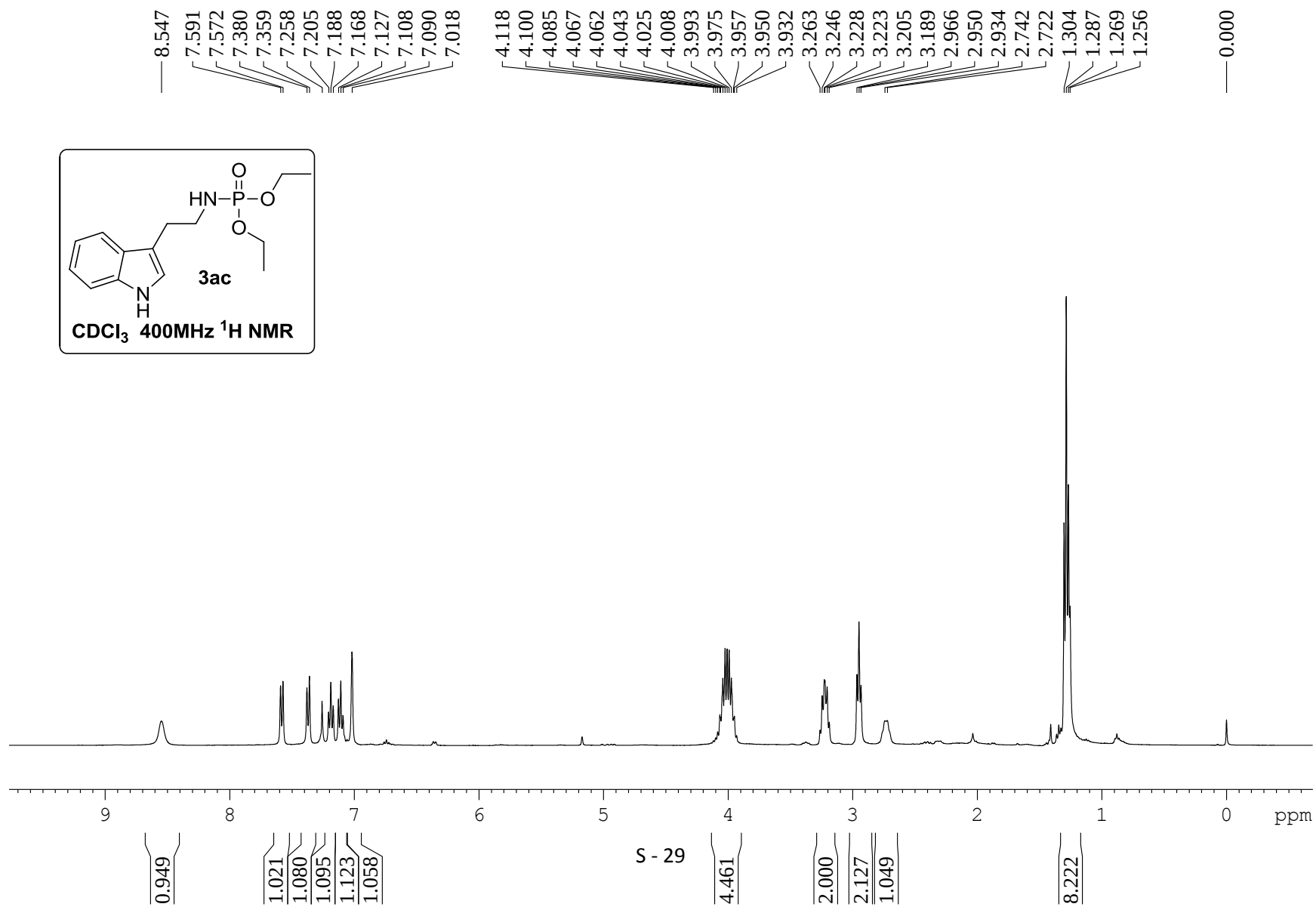


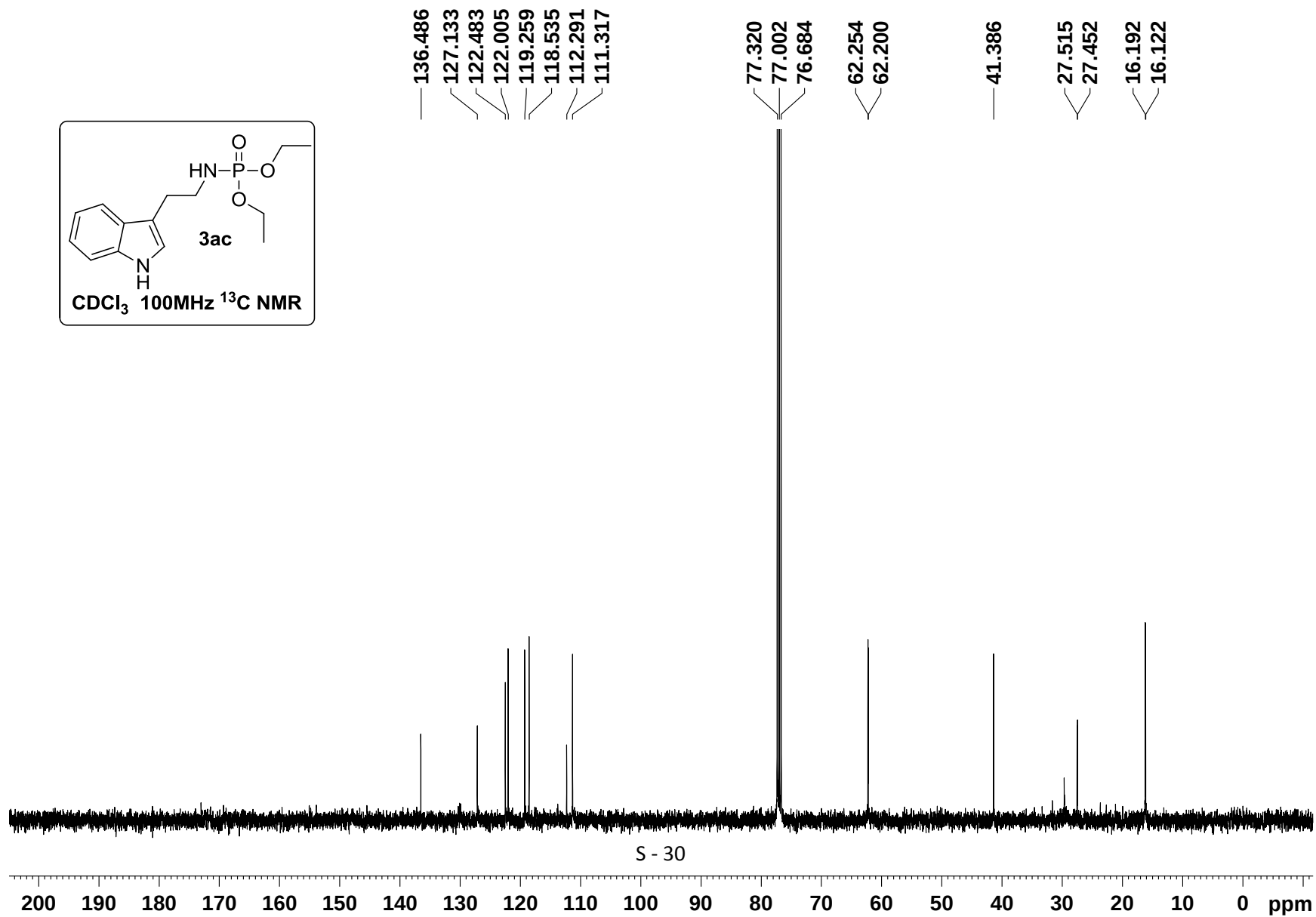
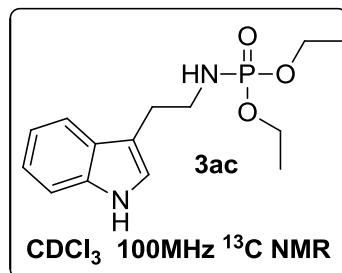


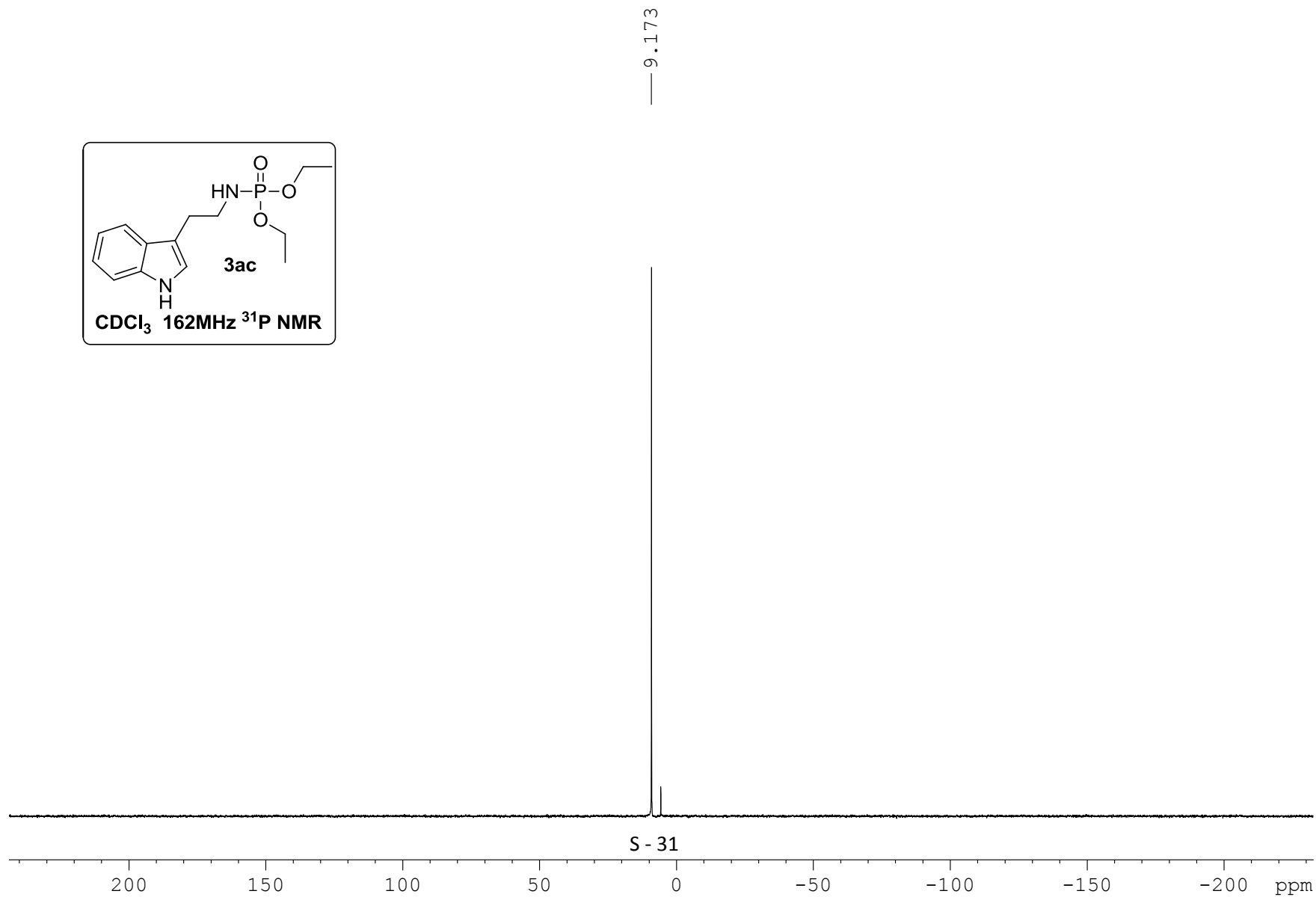
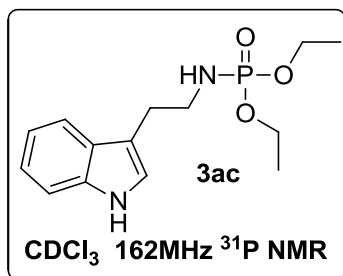


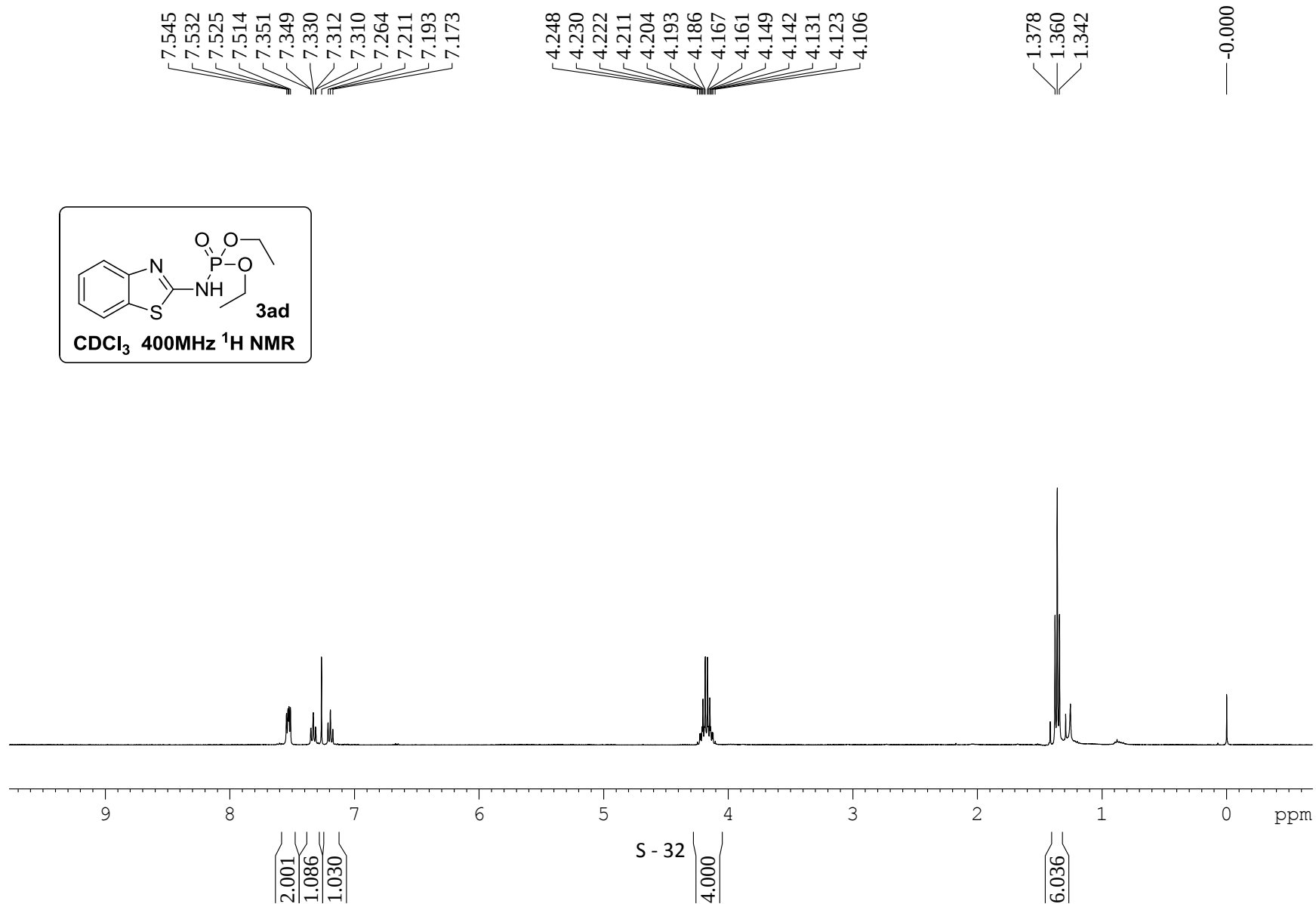
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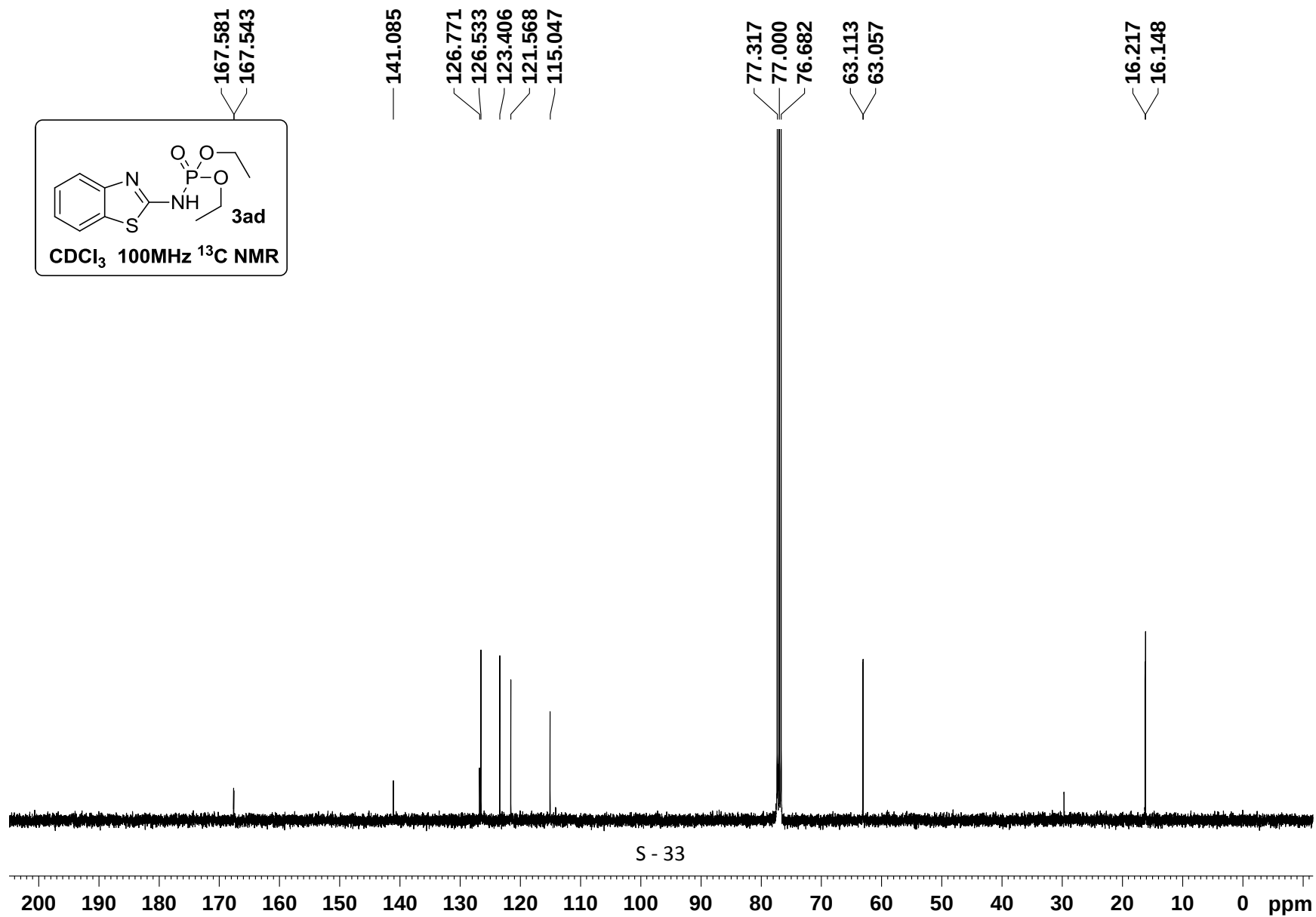


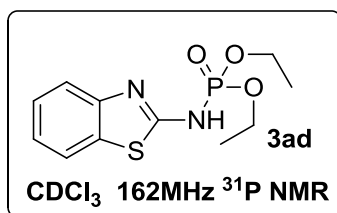








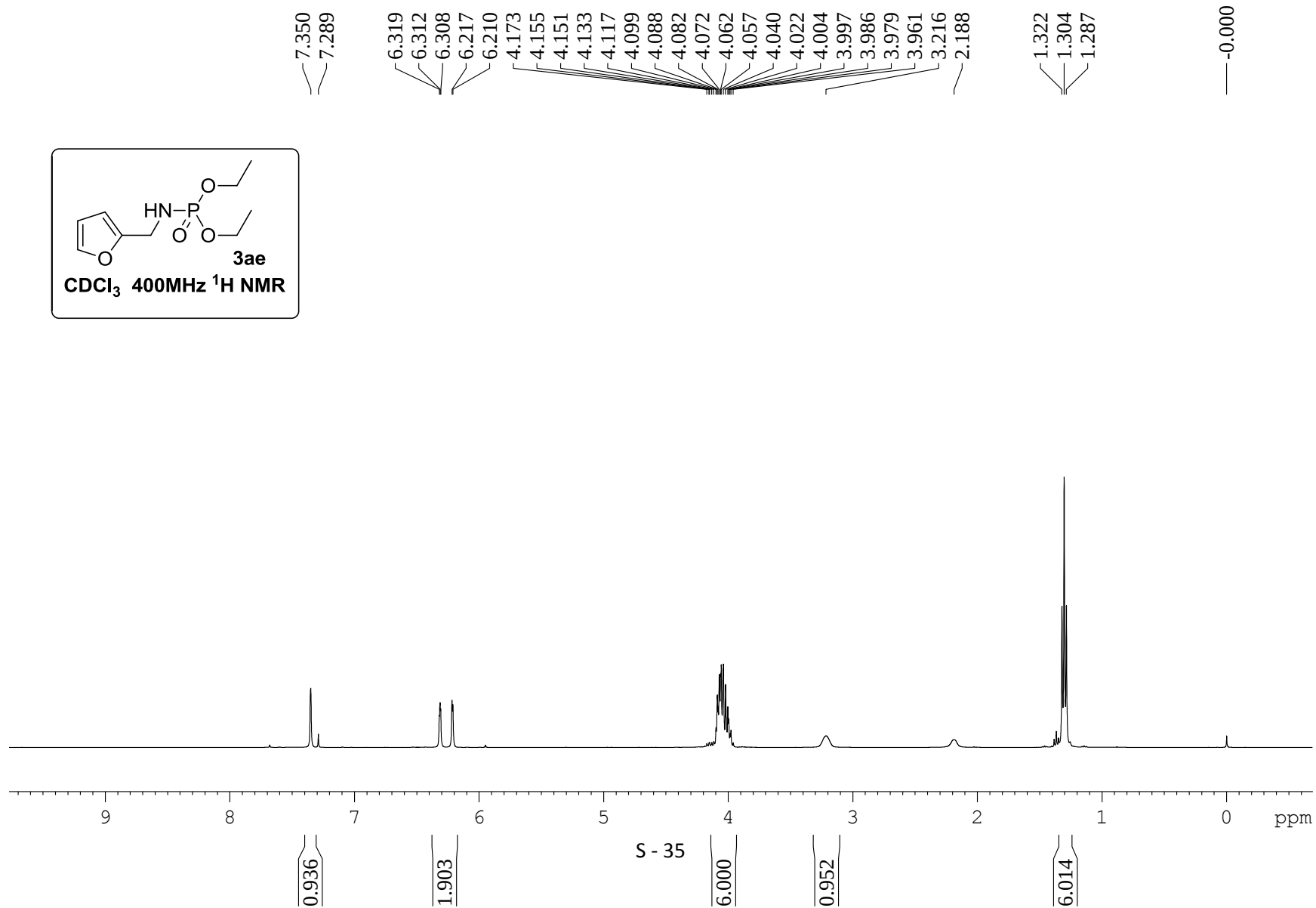
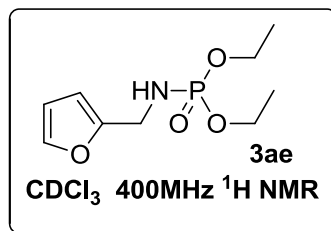


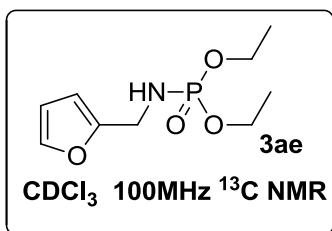


4.274

S - 34

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152.843

141.969

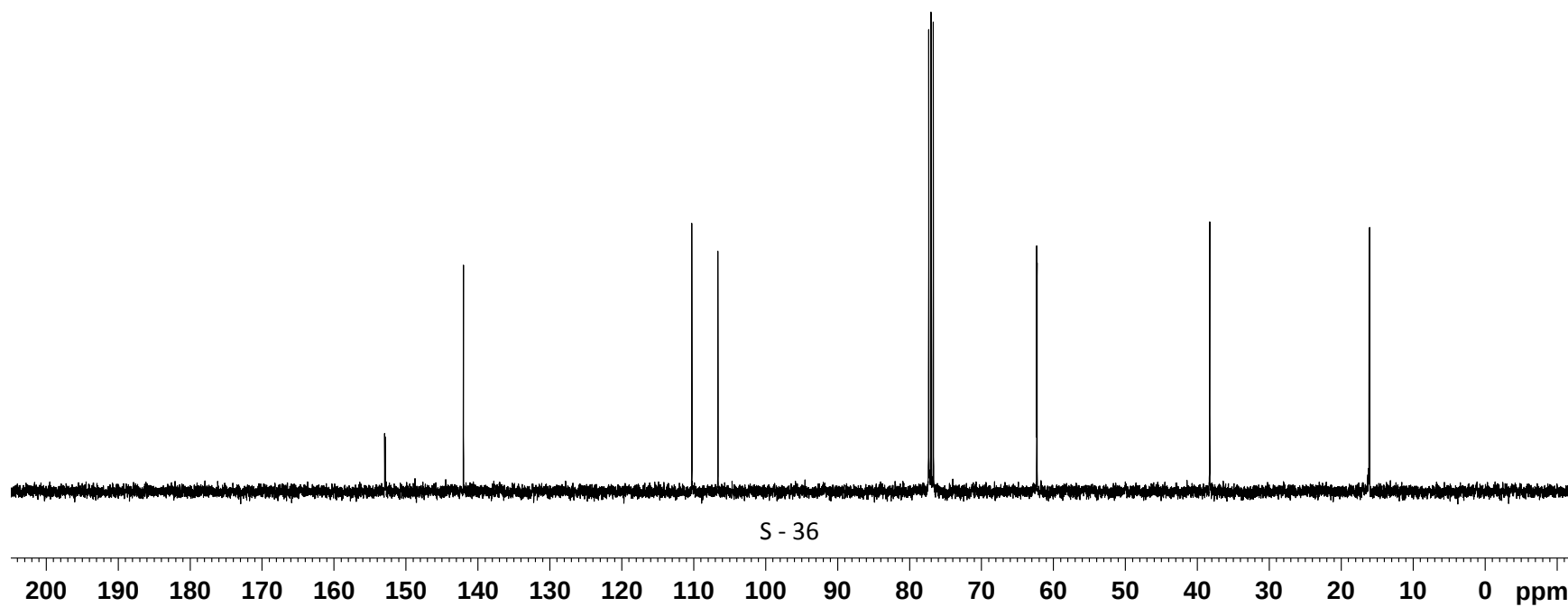
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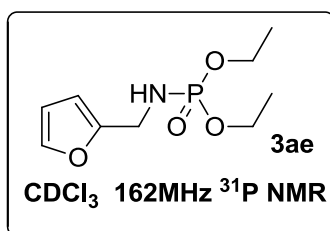
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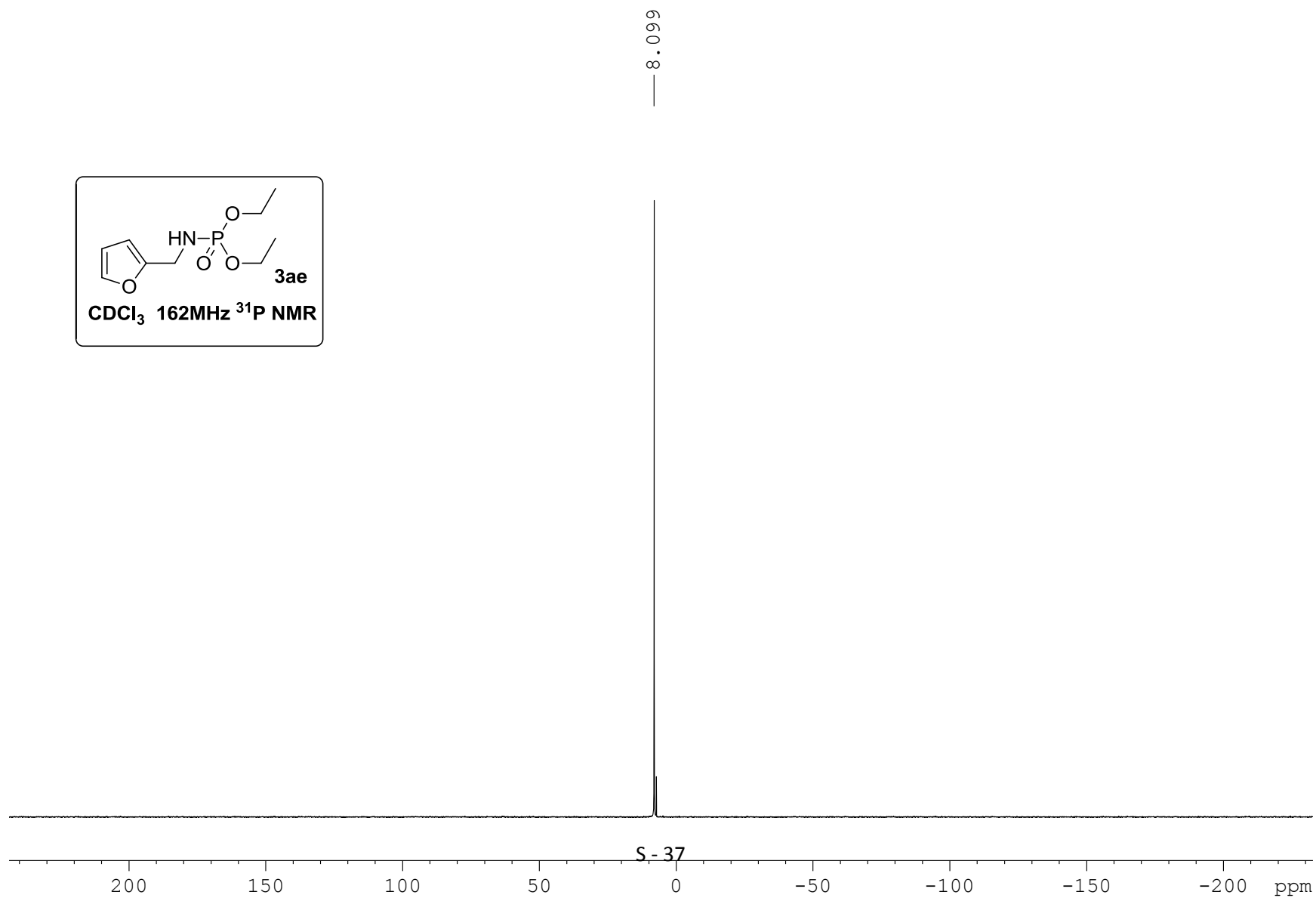
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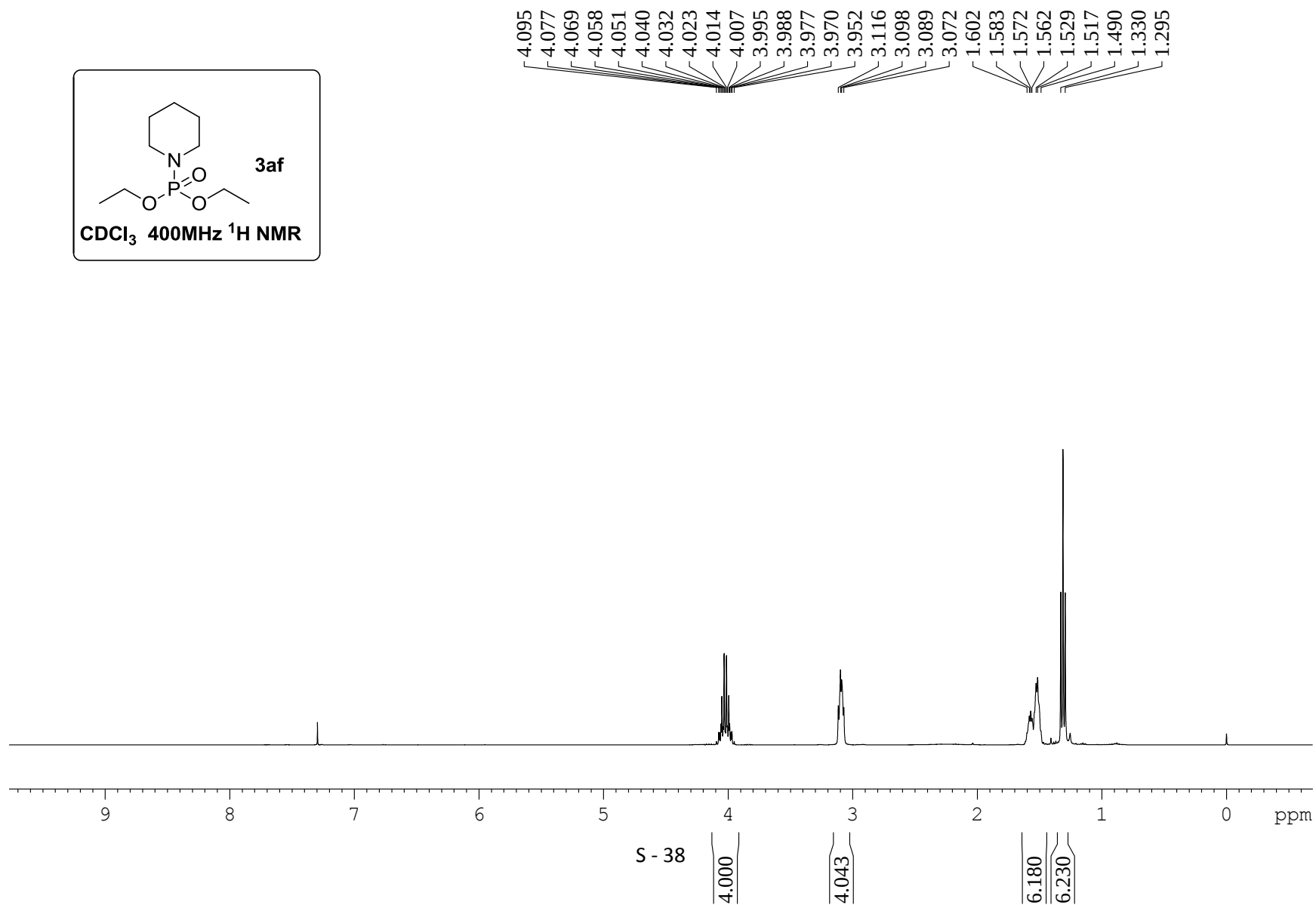
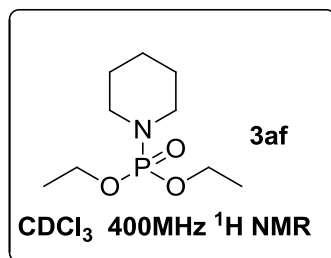
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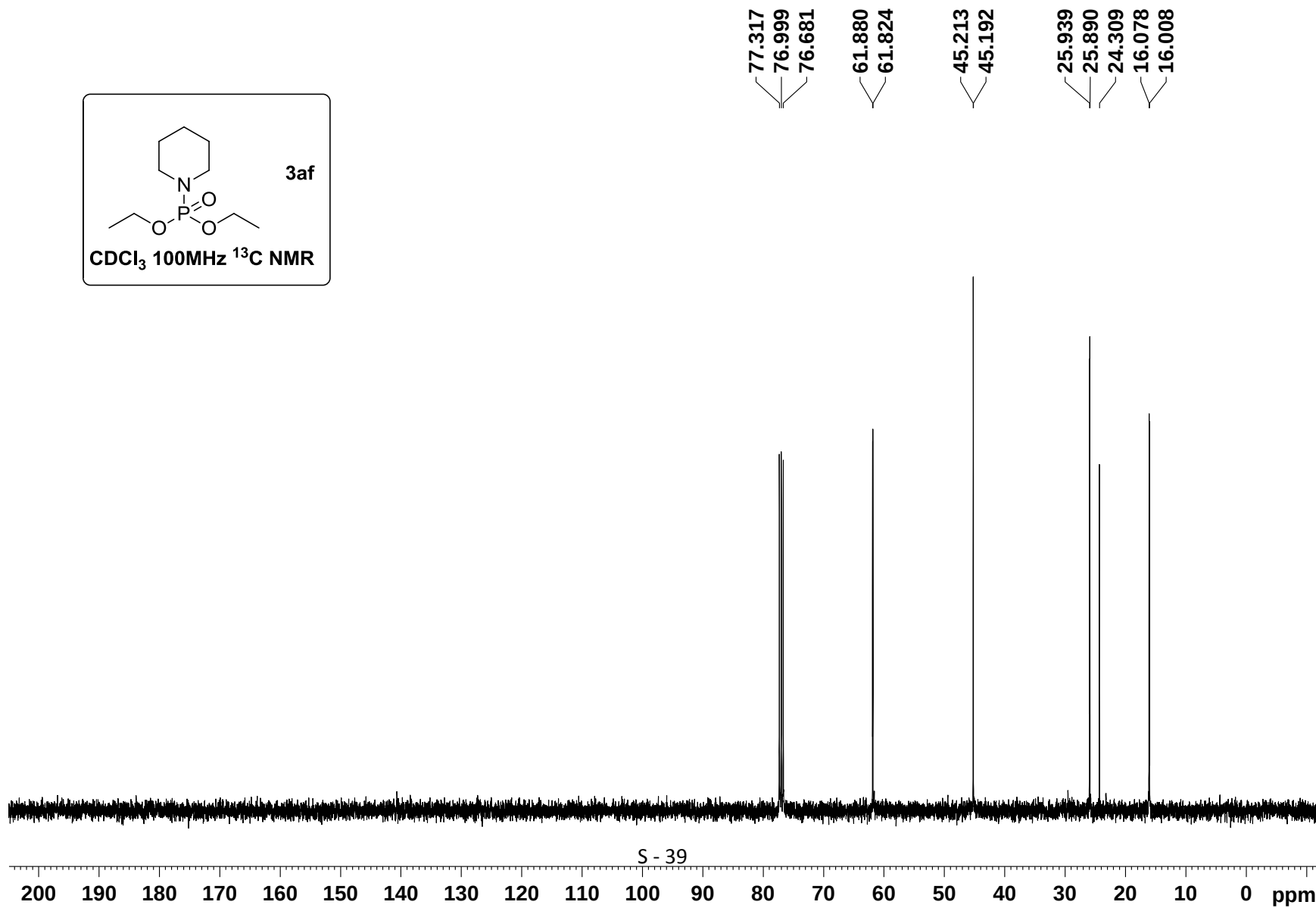
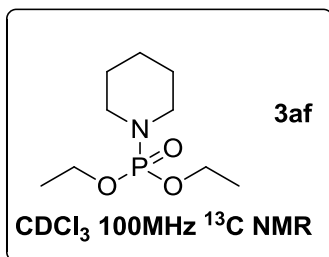


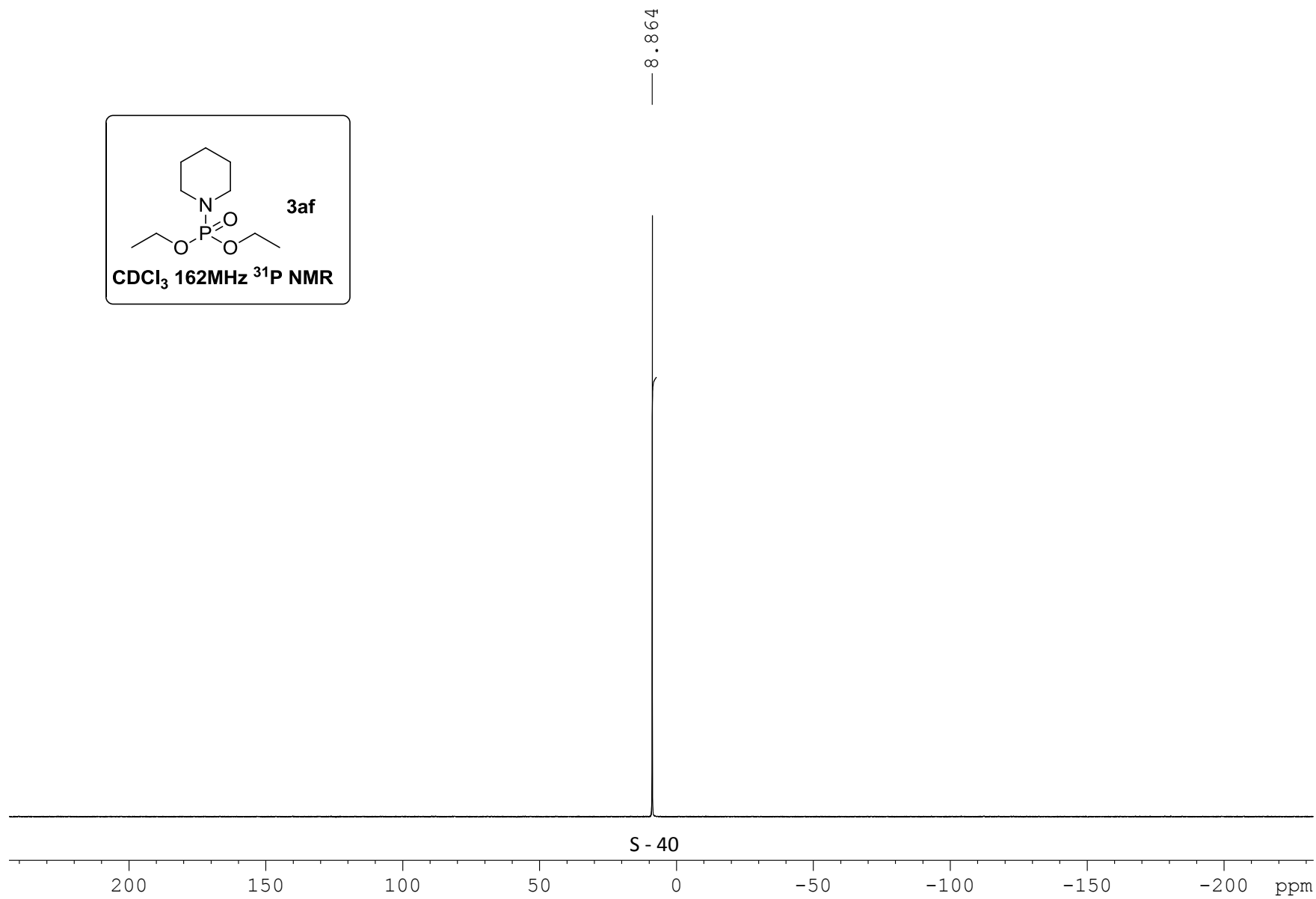
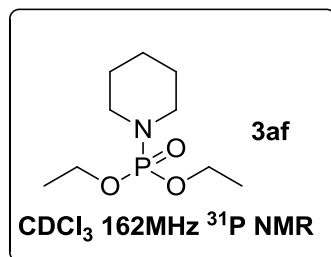


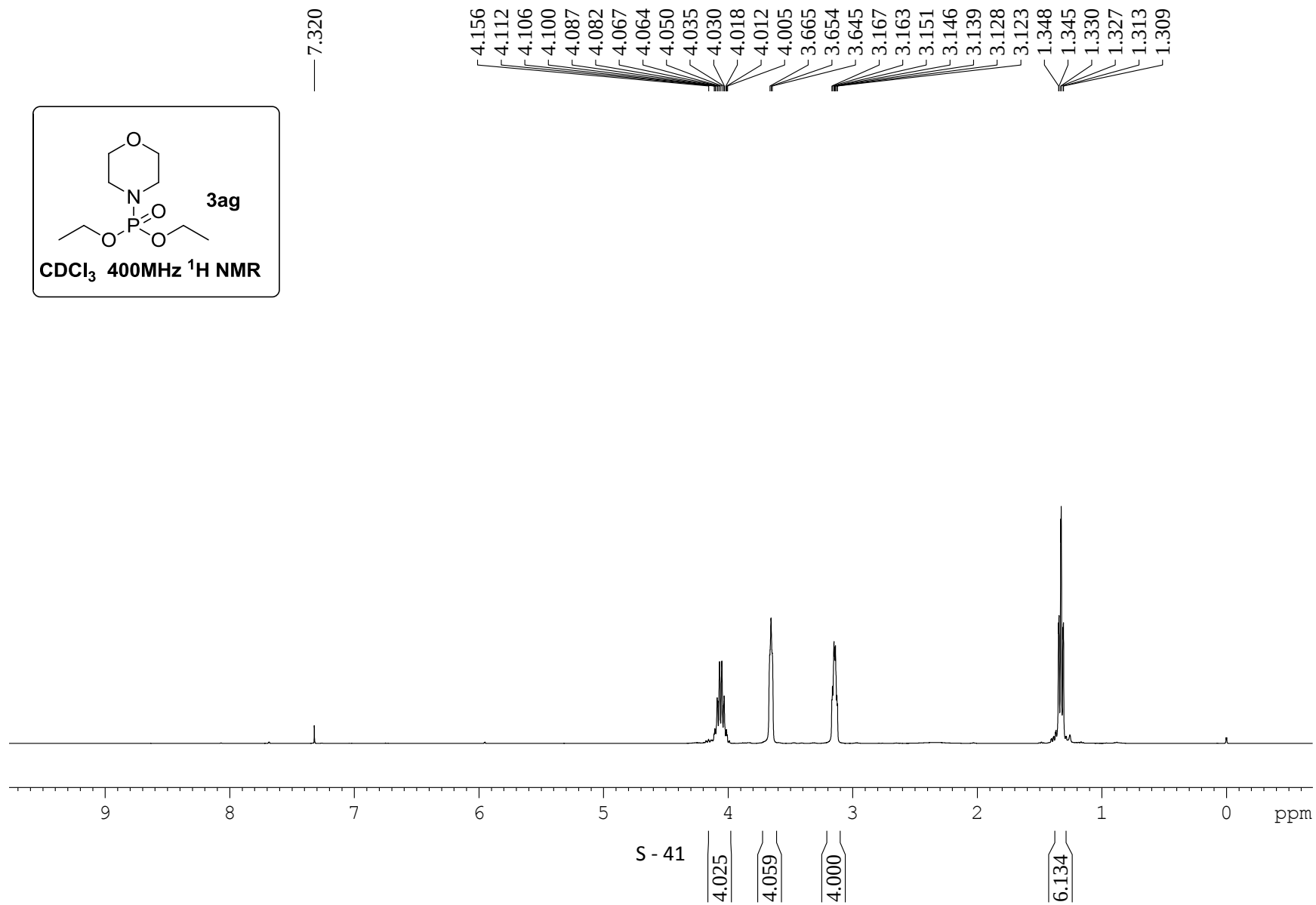
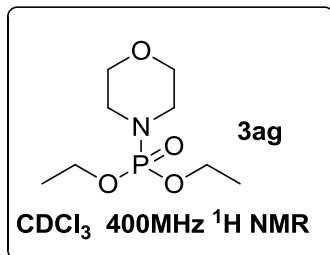
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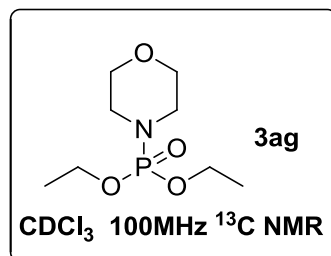




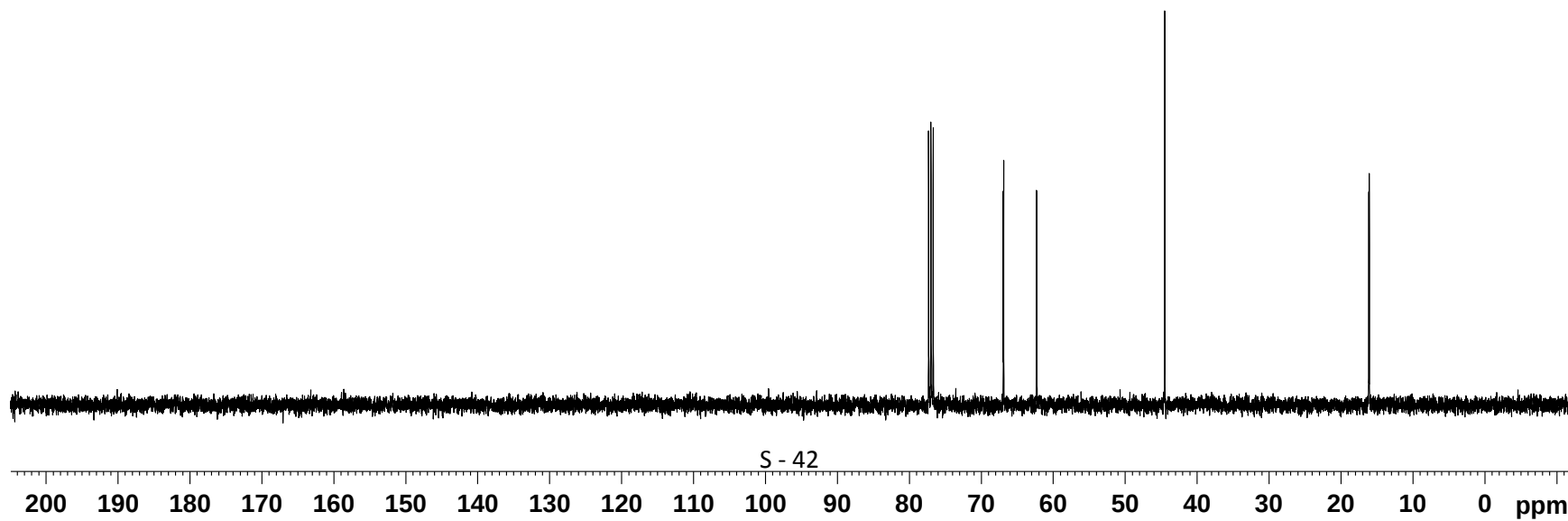


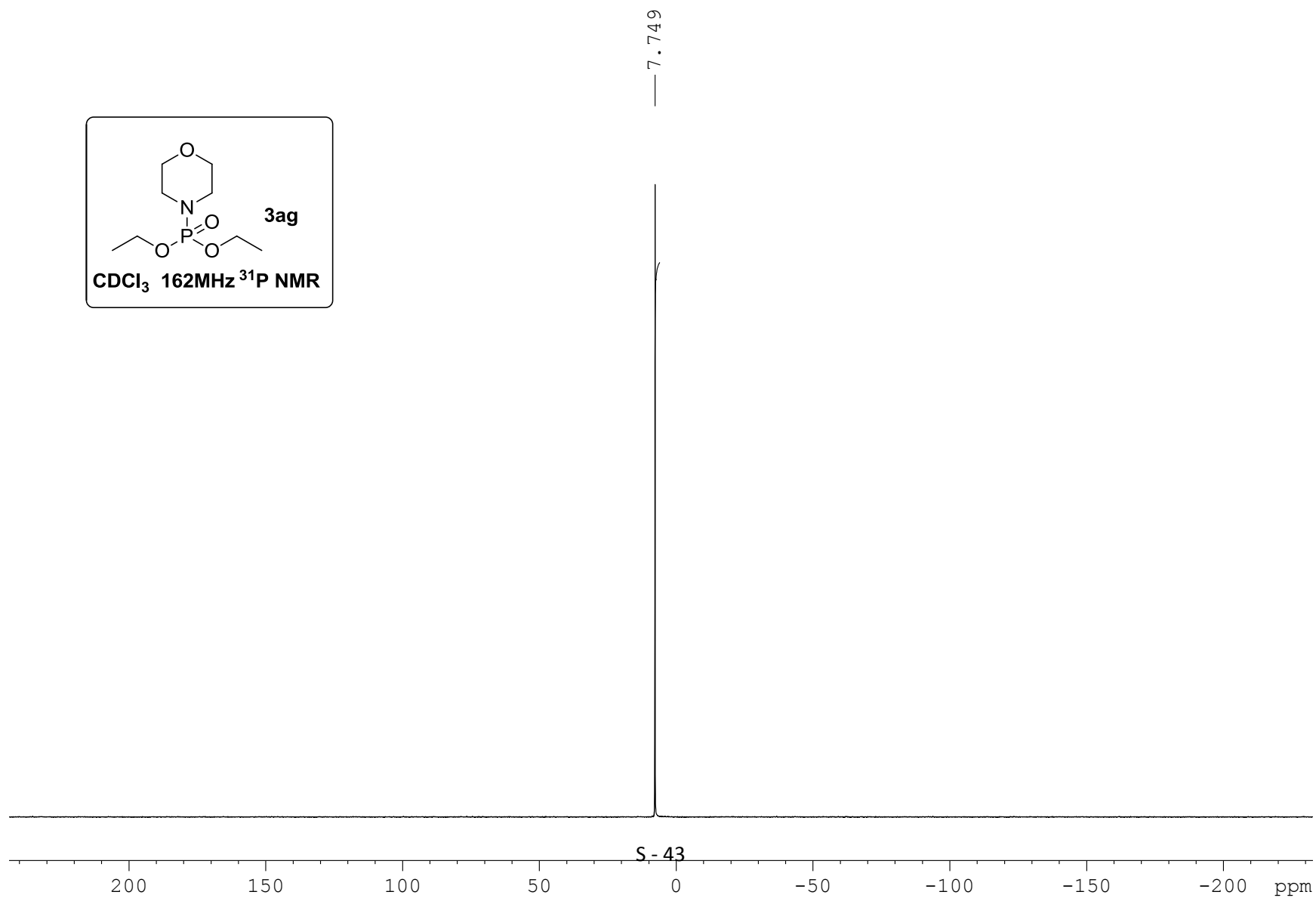
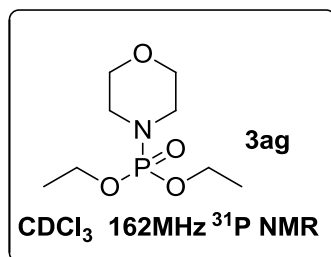


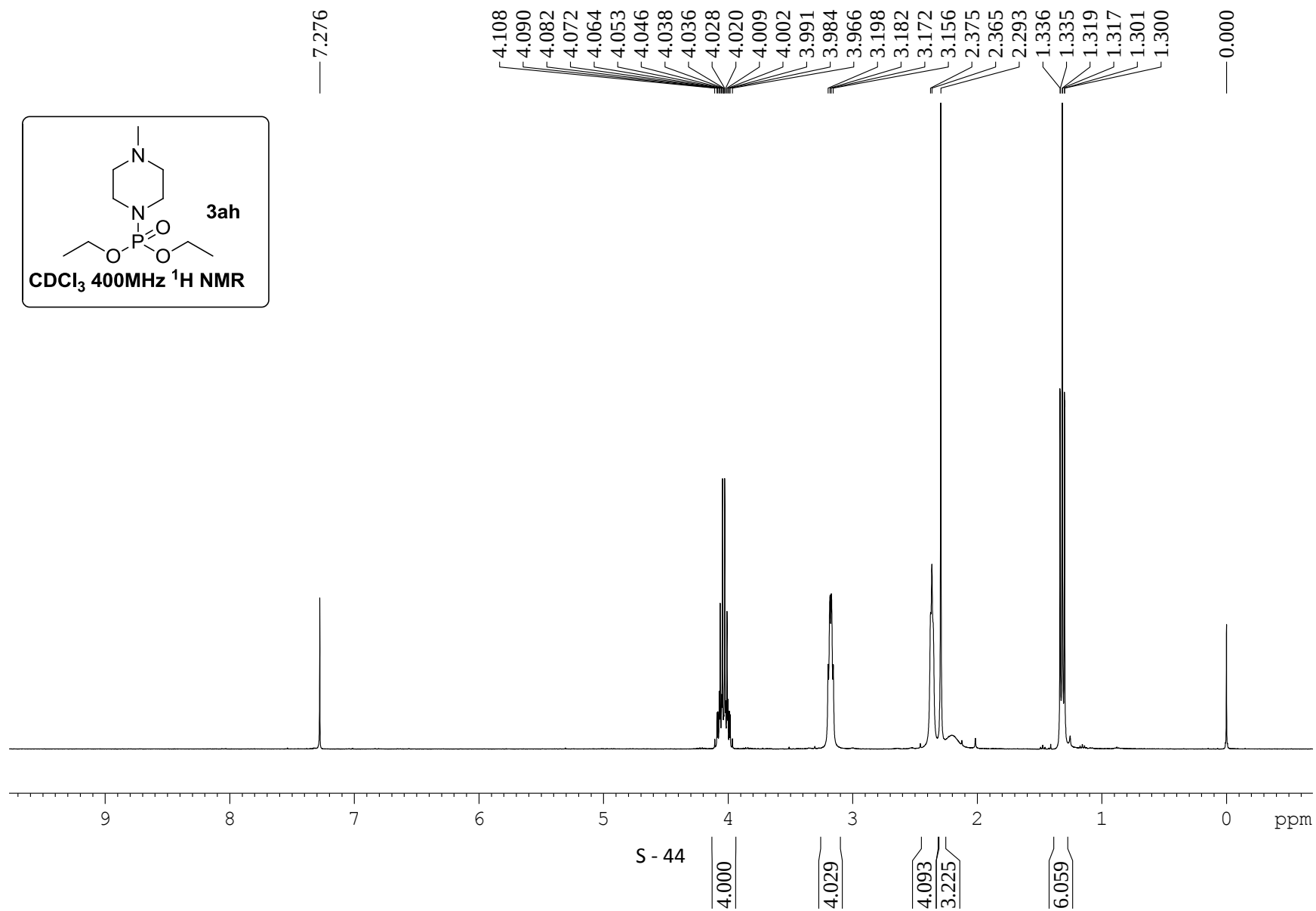


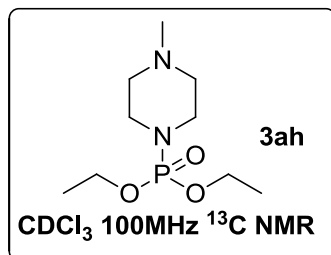


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66.879
62.326
62.270
— 44.484
16.118
16.050

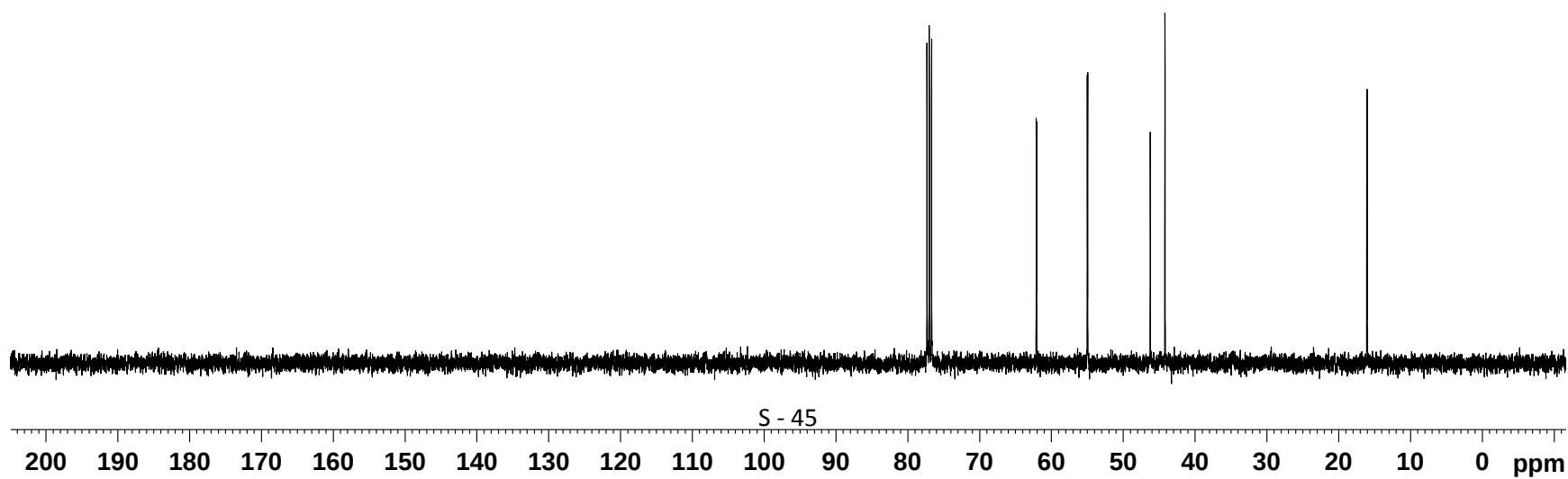


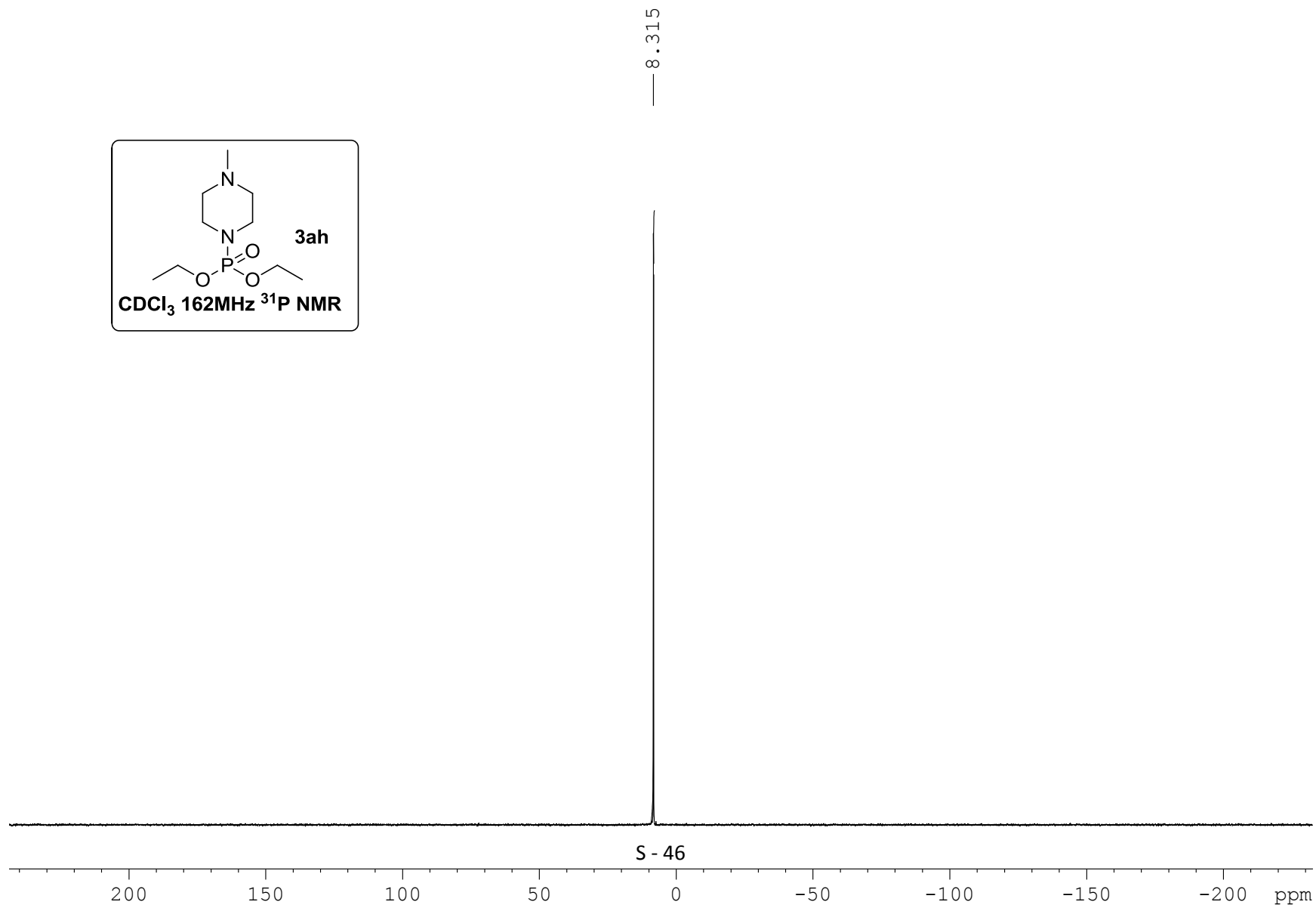


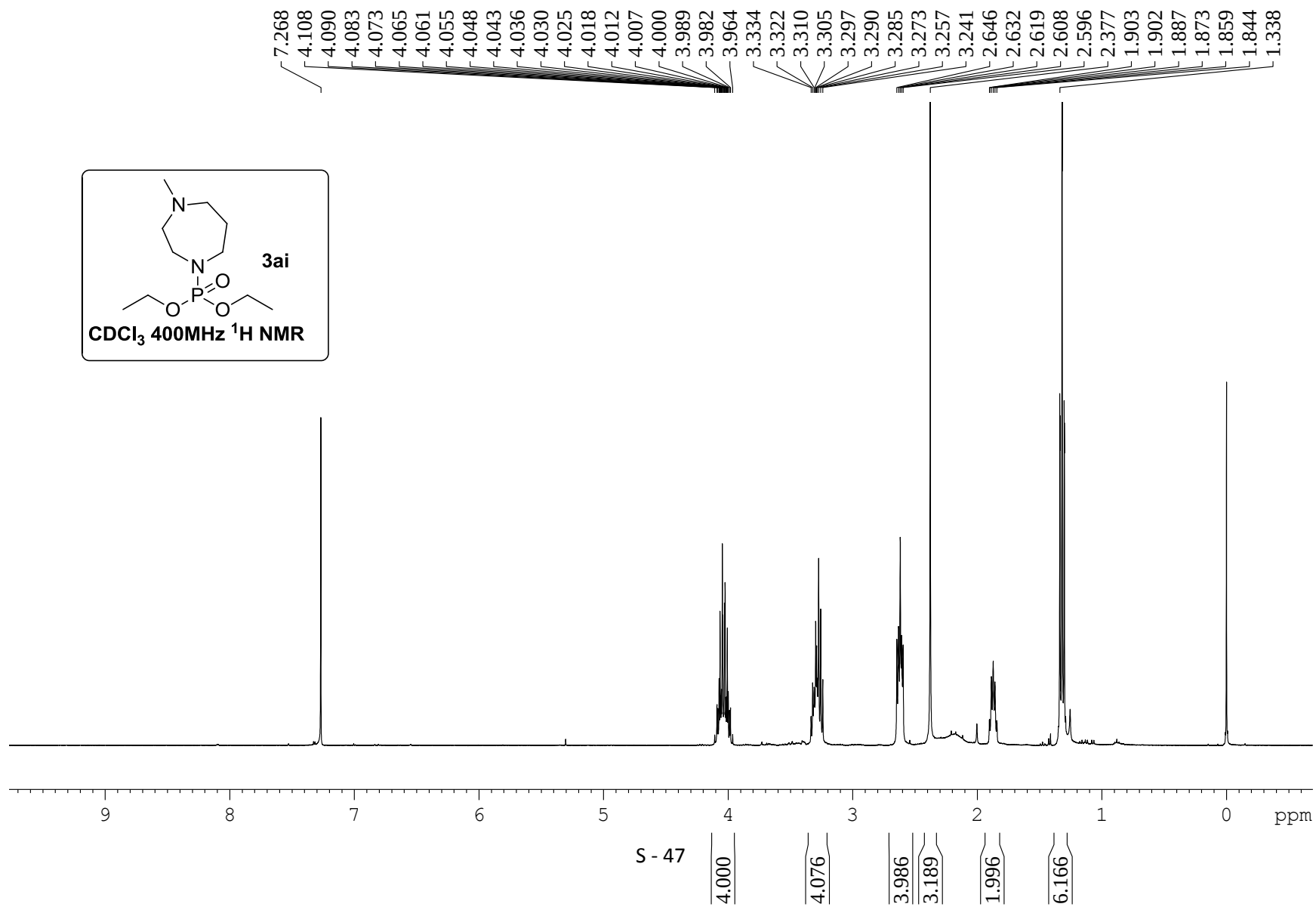


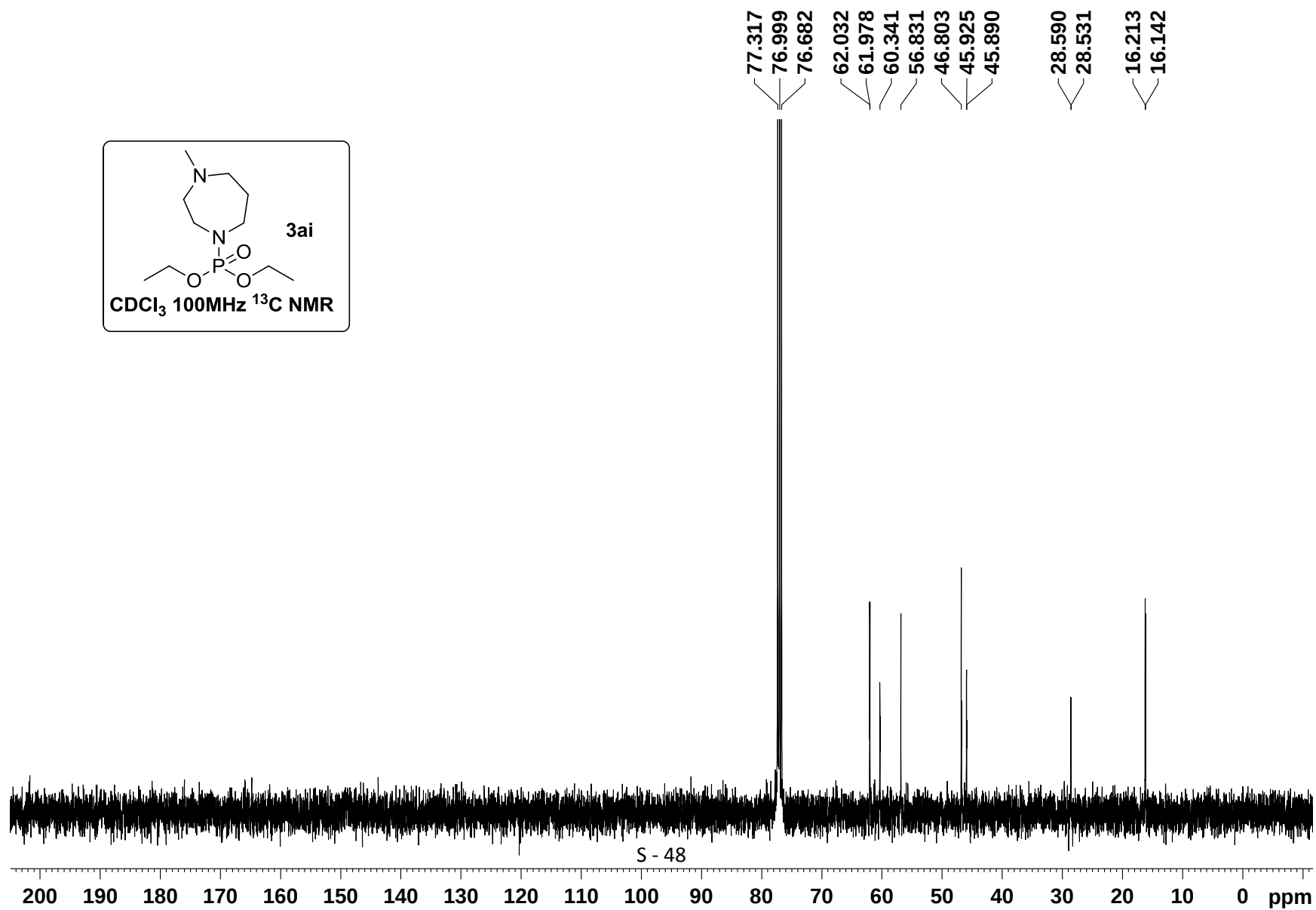
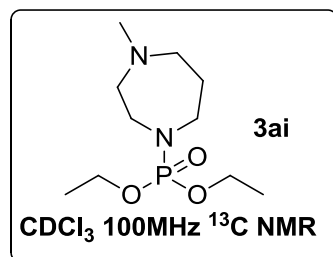


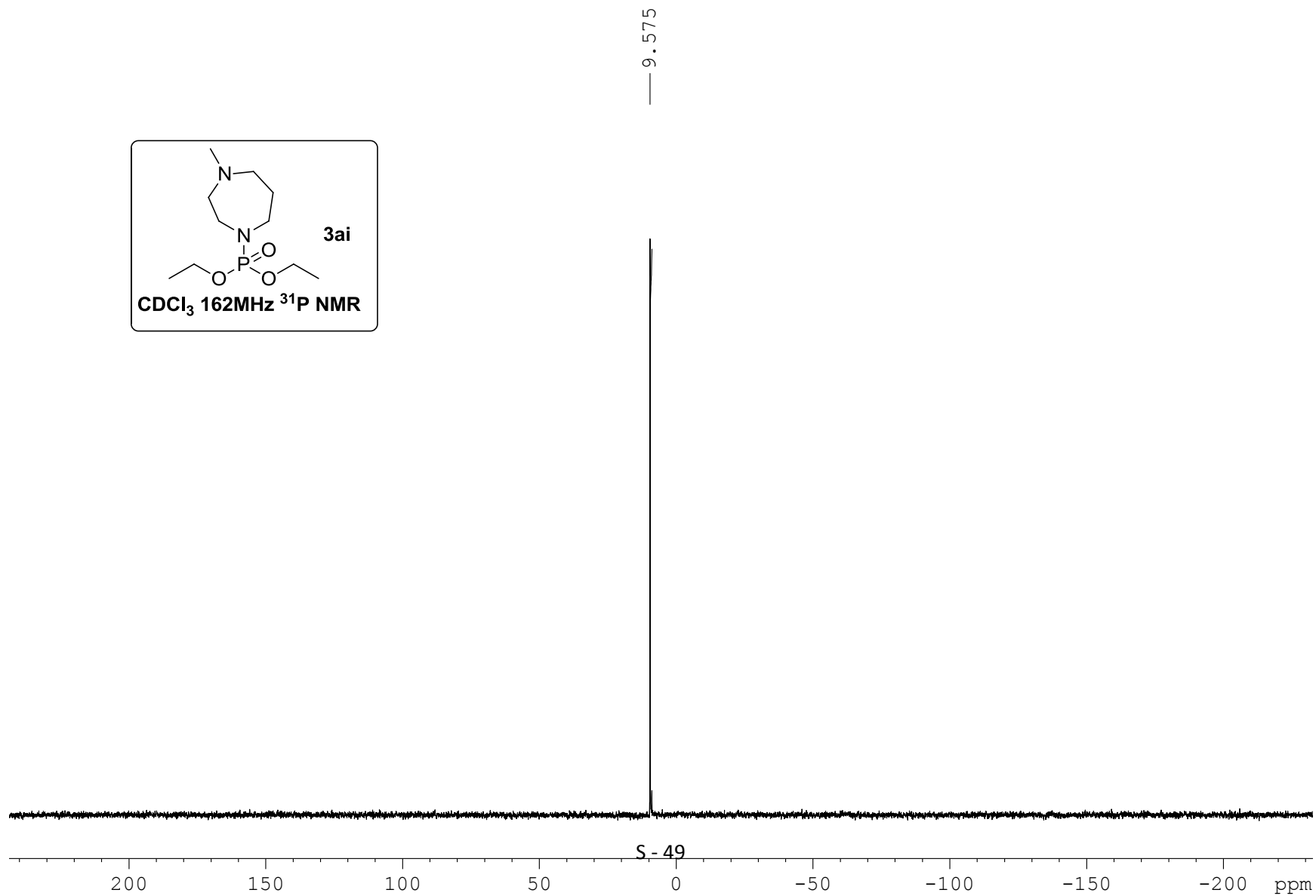
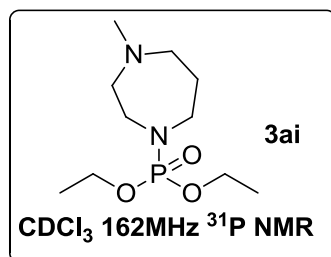
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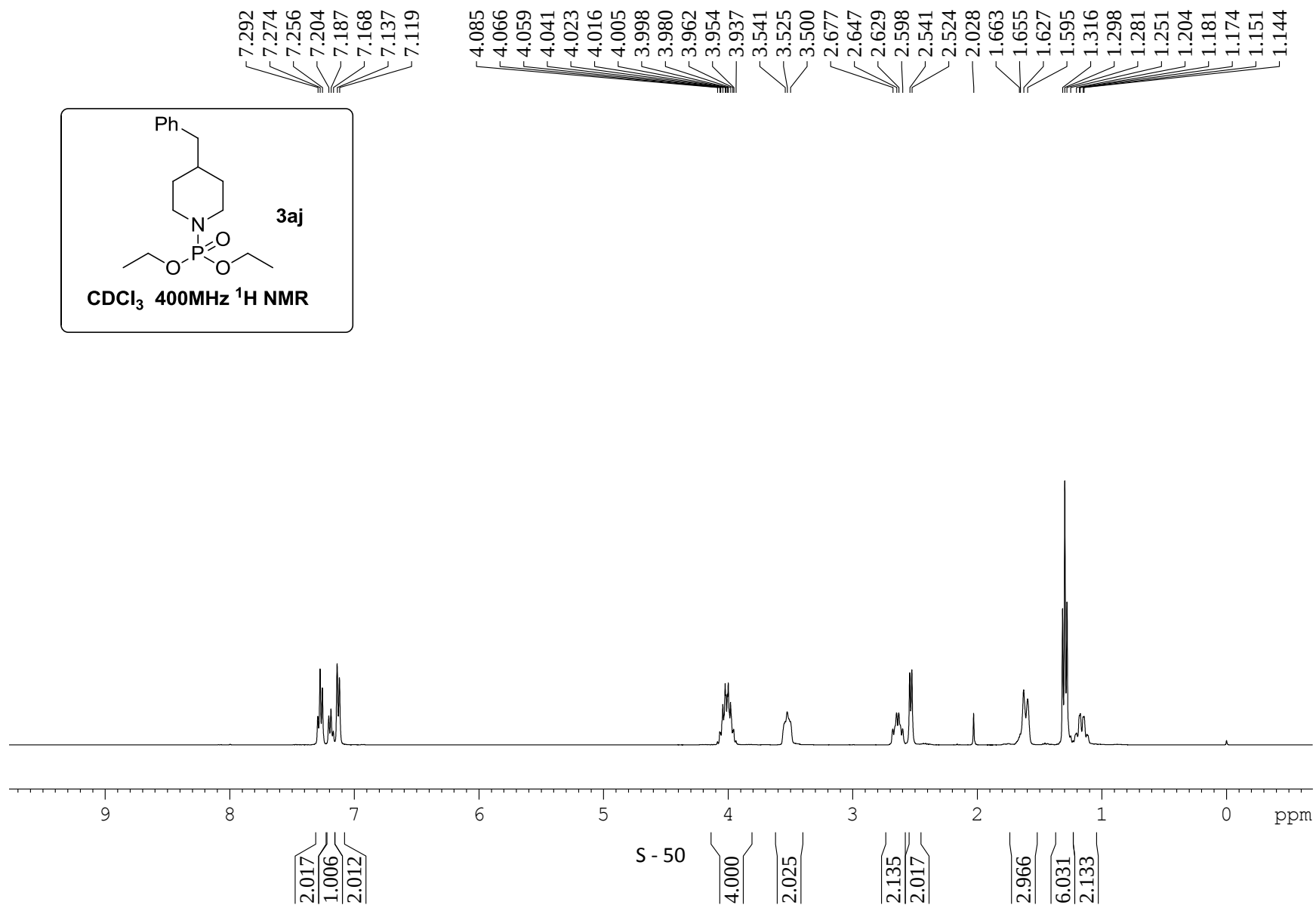
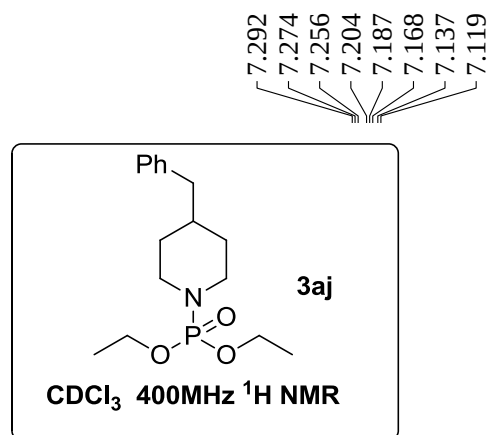


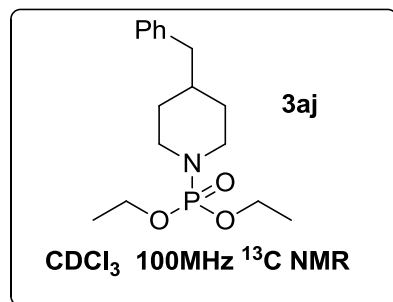












— 139.958

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128.084

125.793

77.318

76.999

76.681

62.021

61.965

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43.230

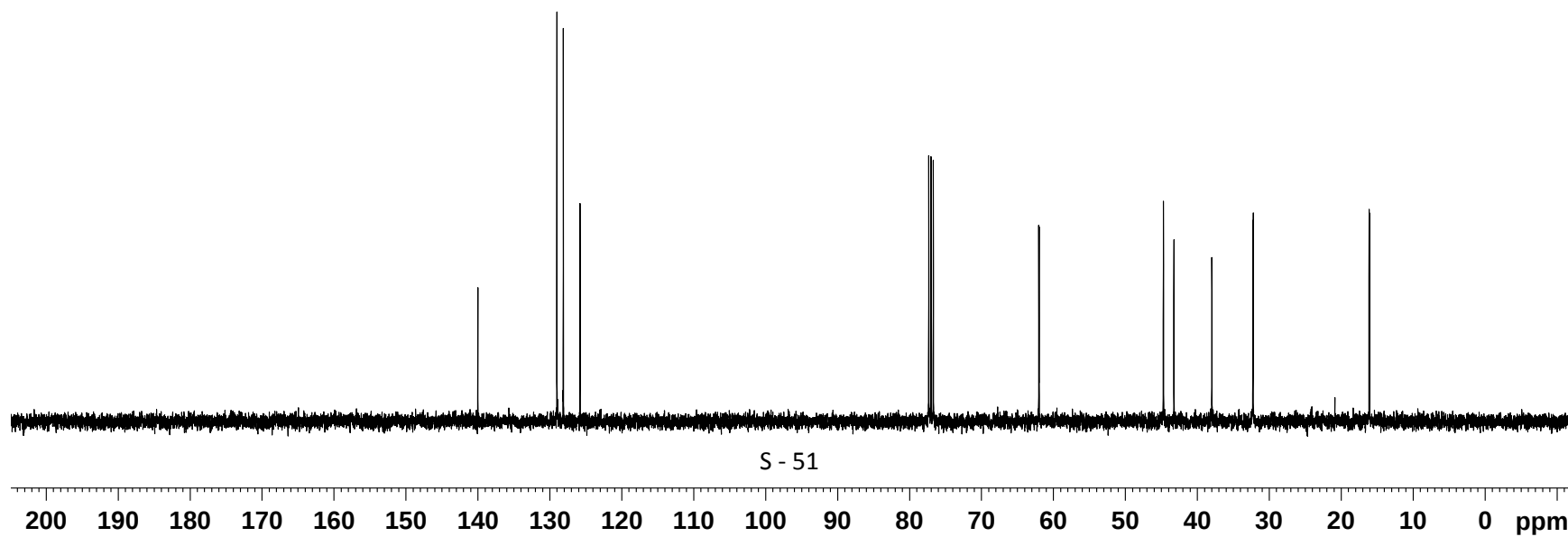
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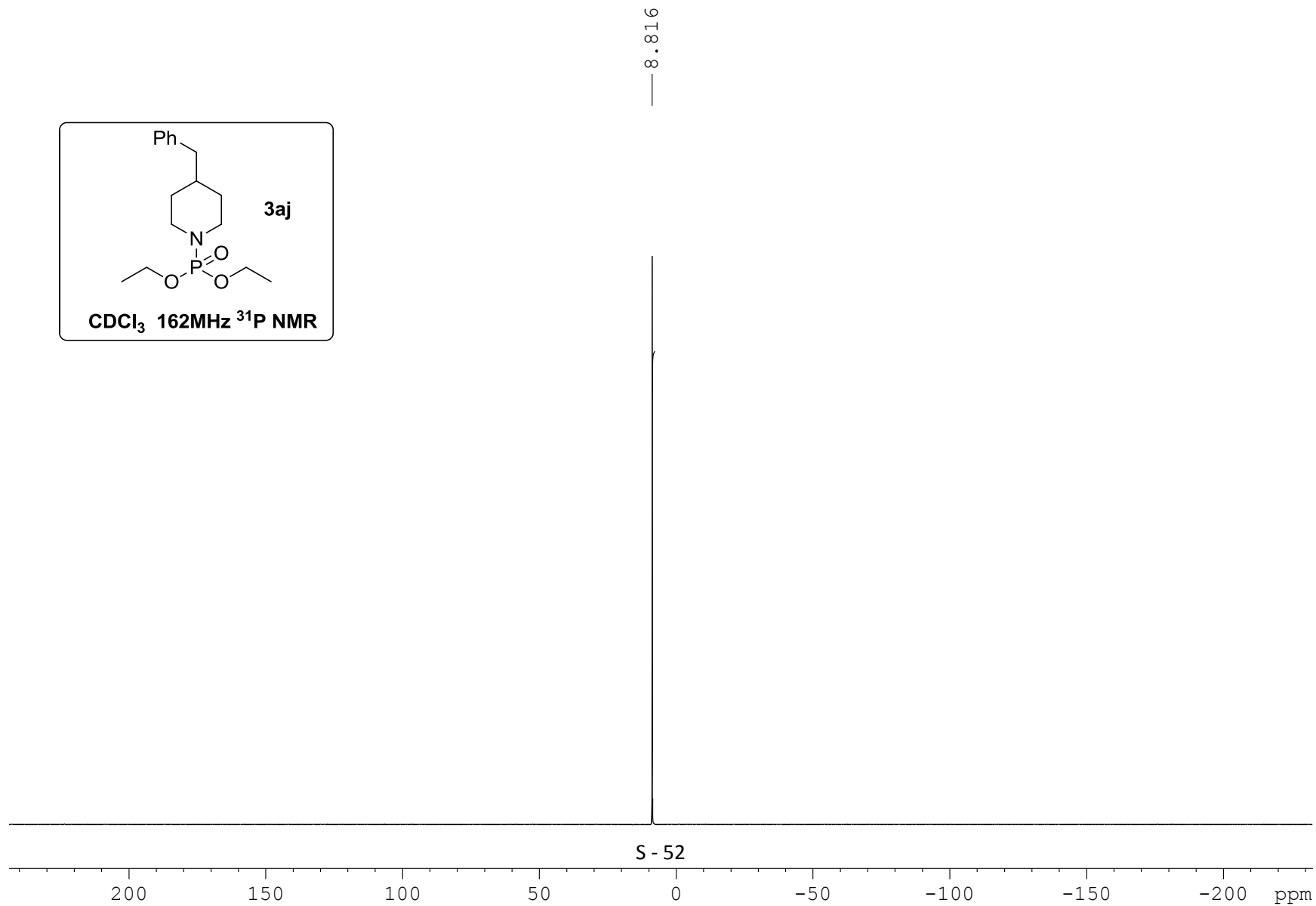
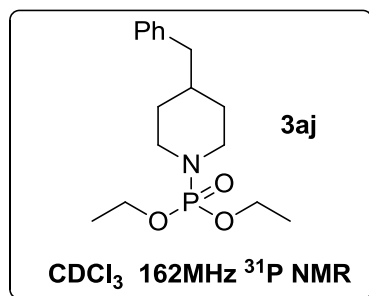
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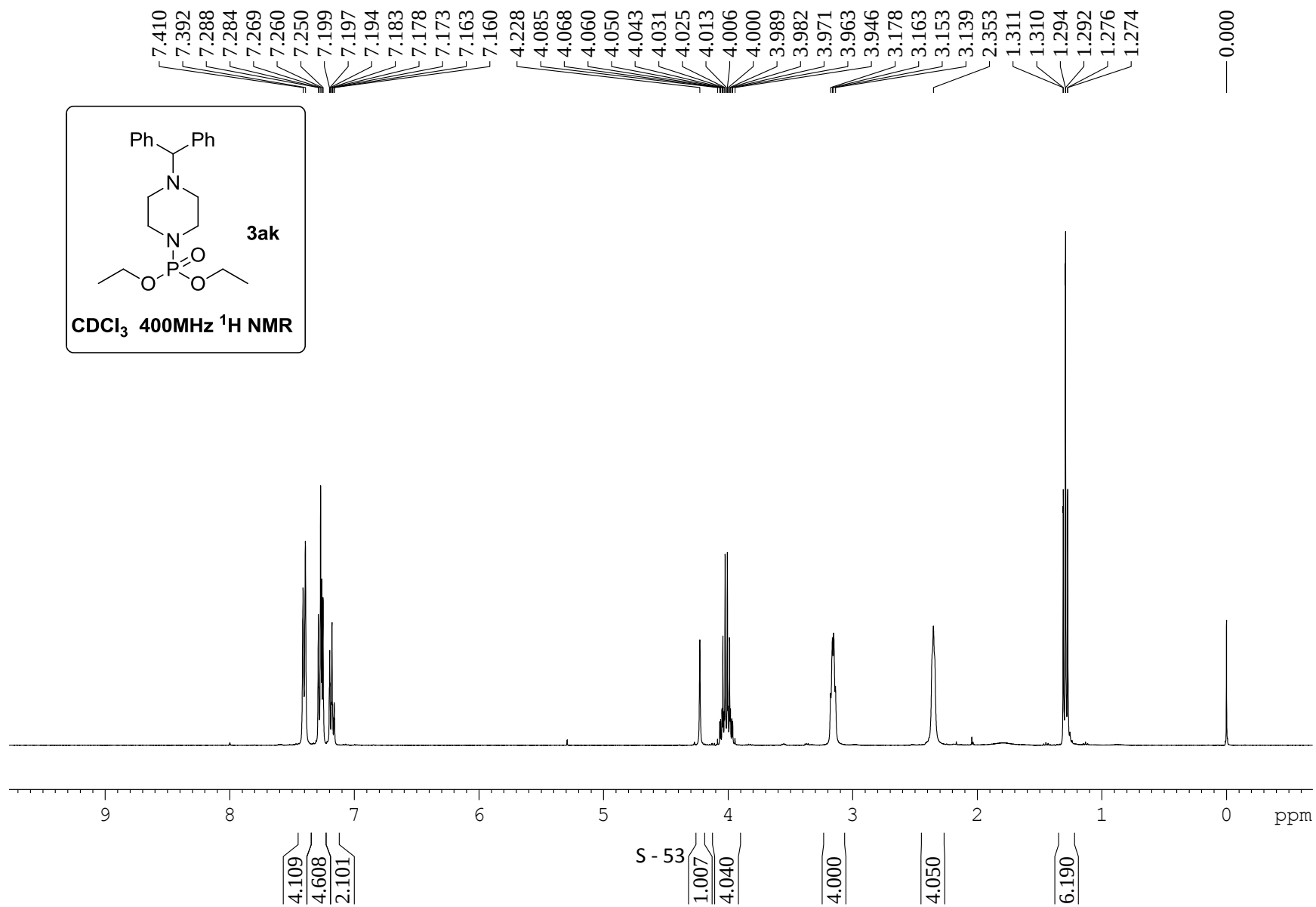
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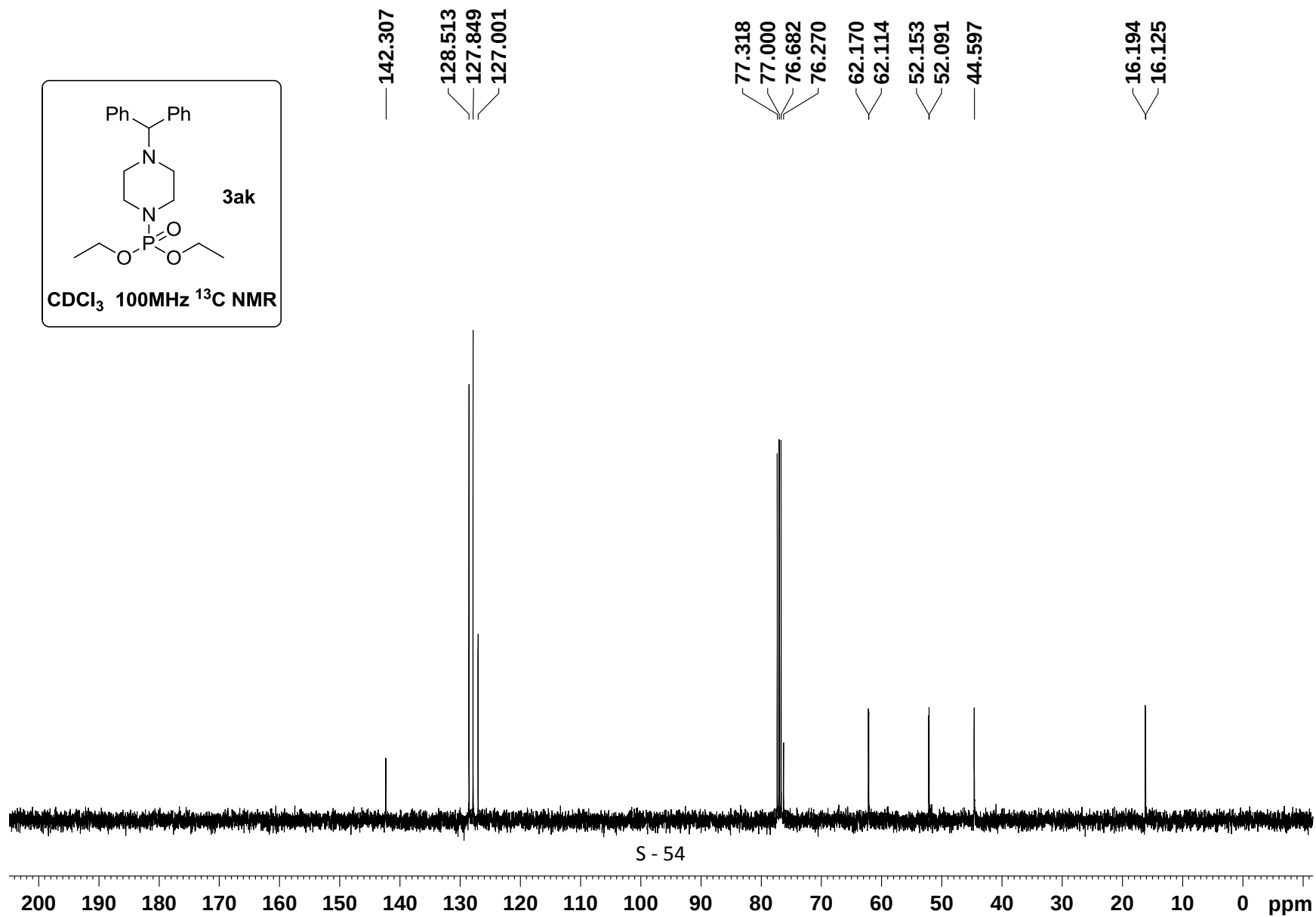
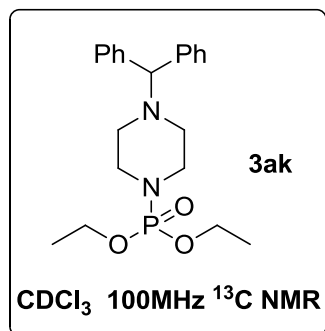
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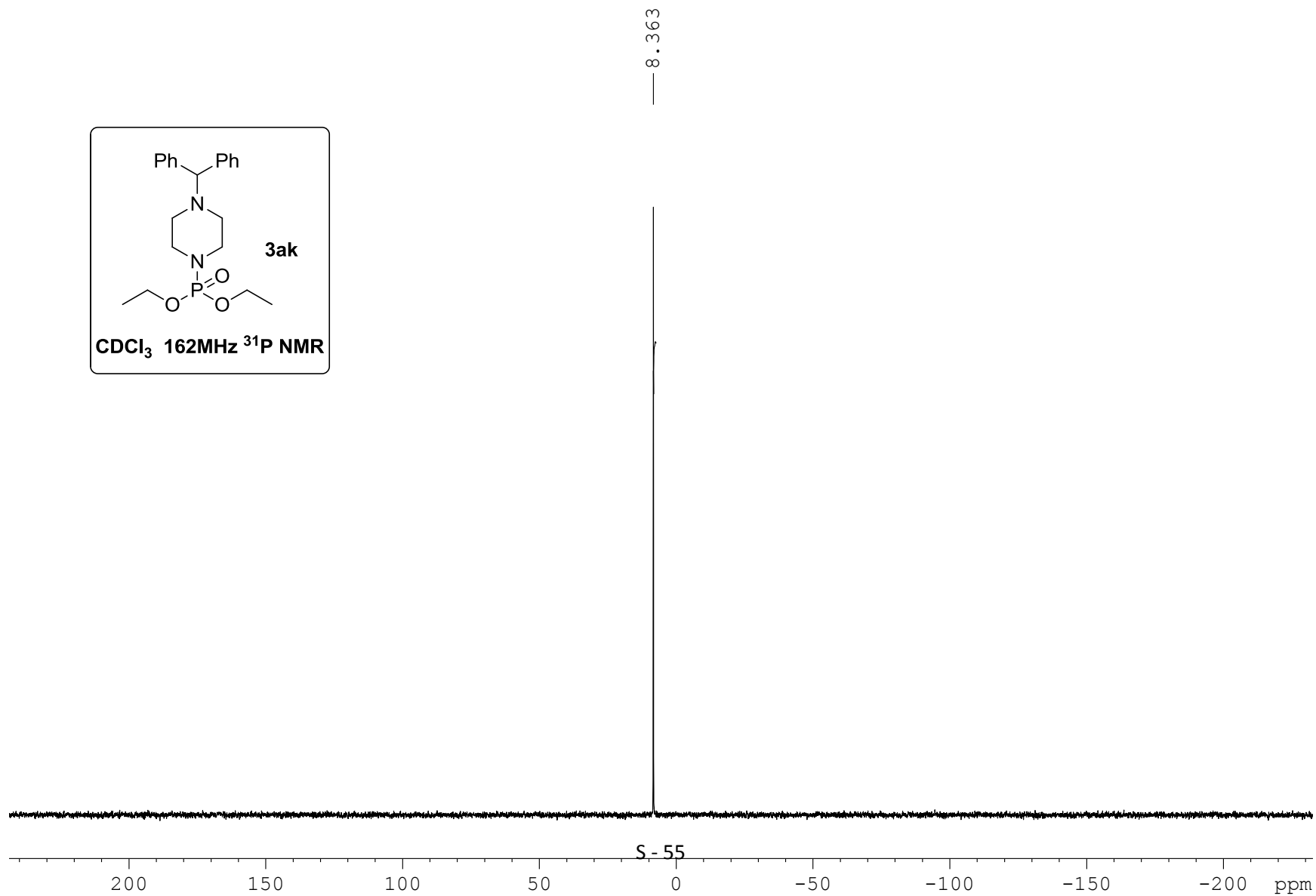
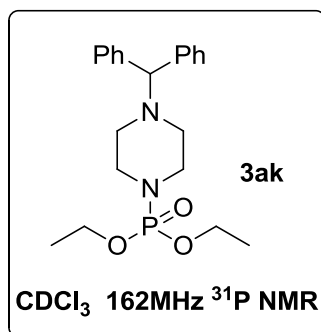
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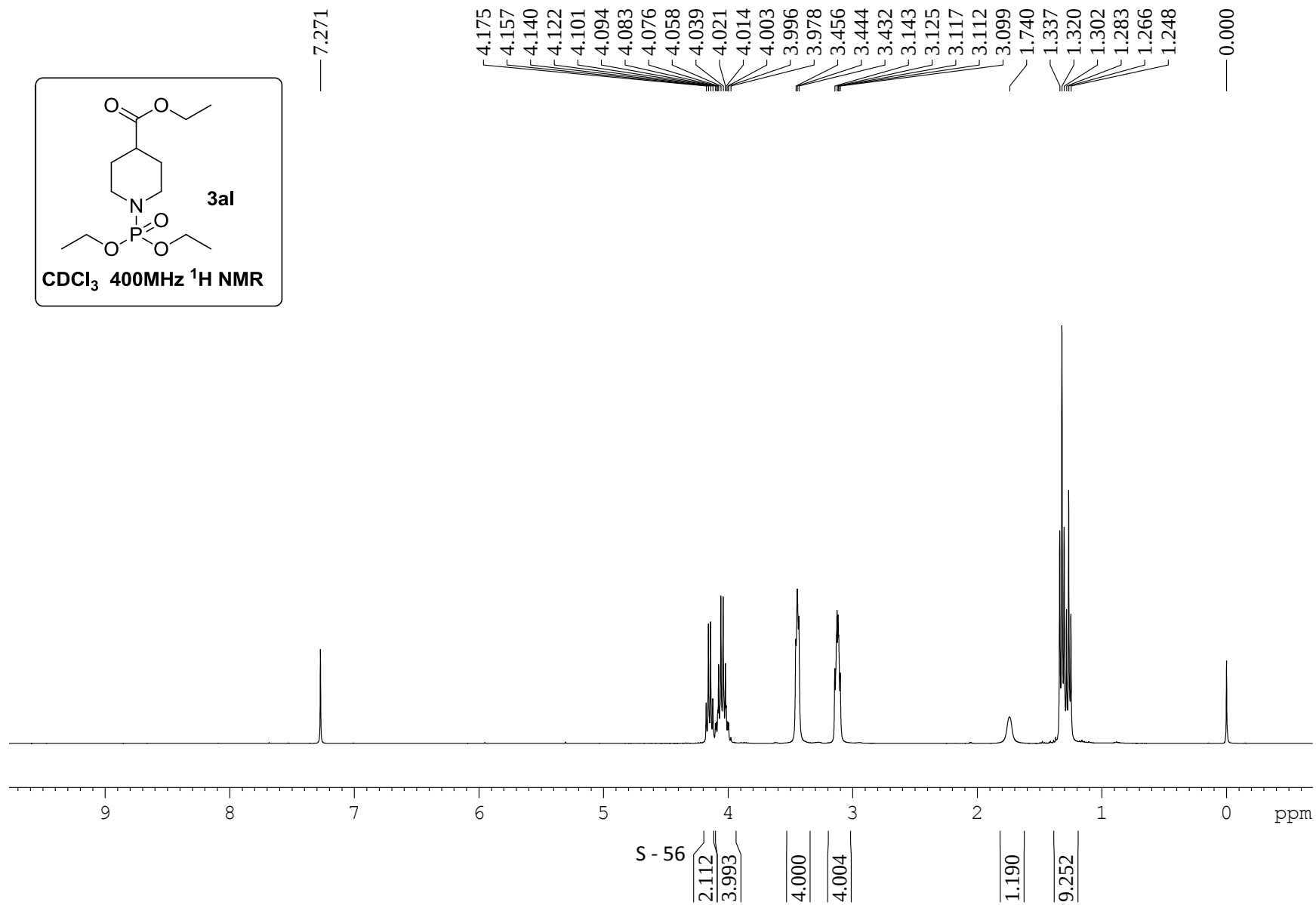
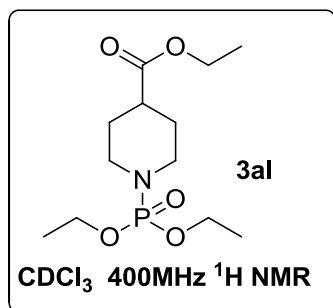


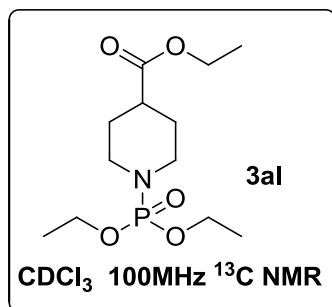








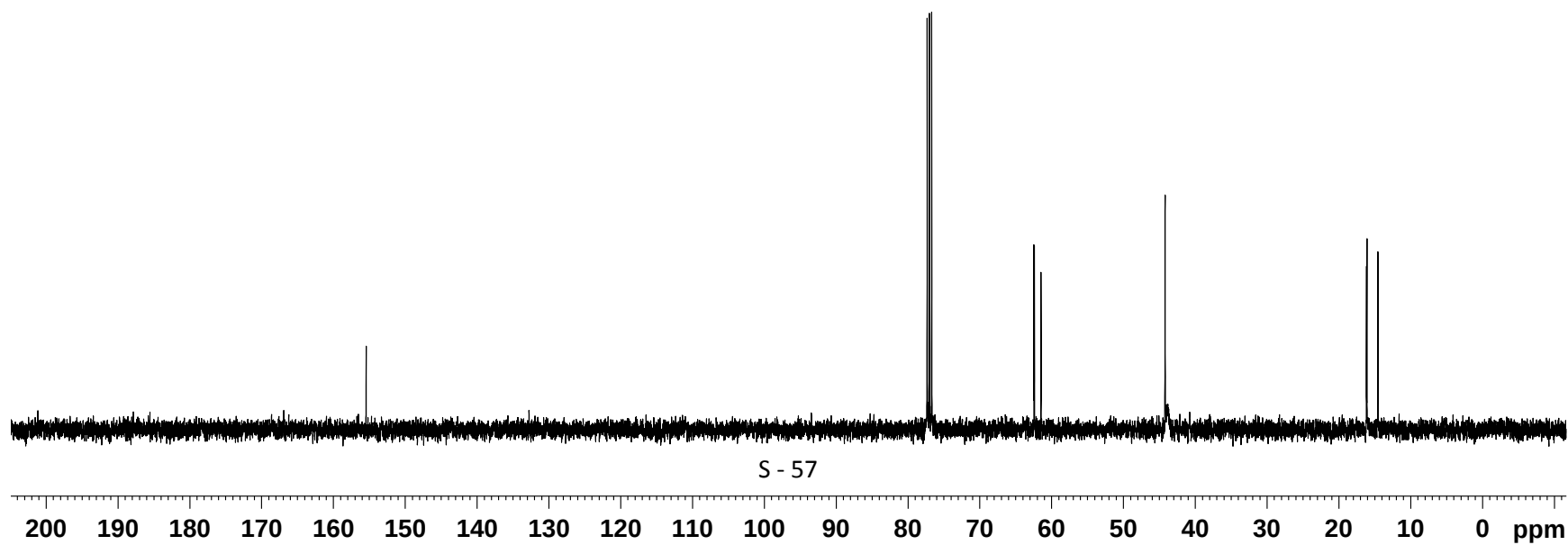


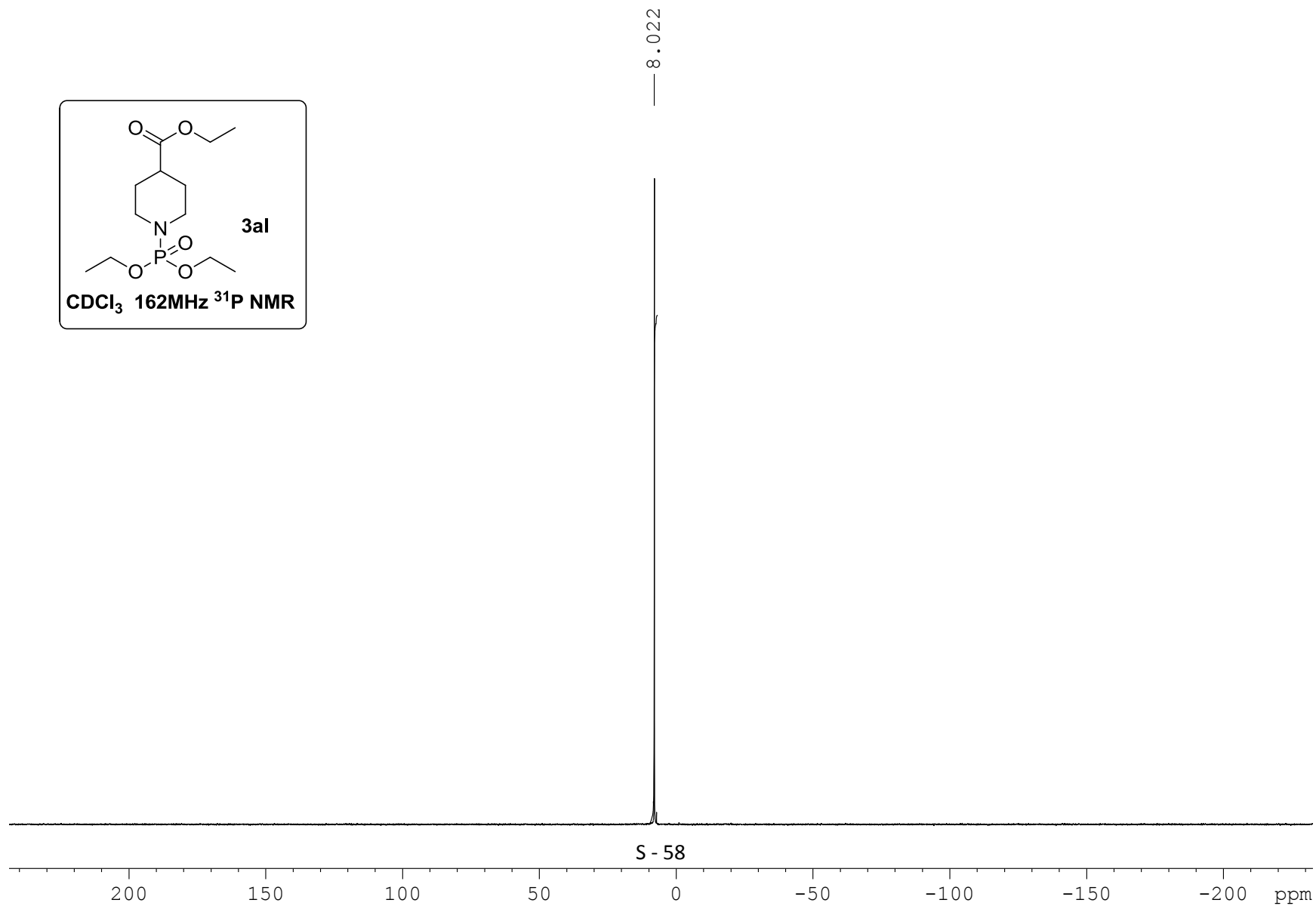
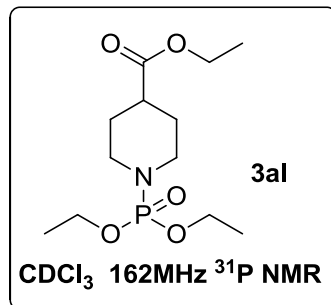


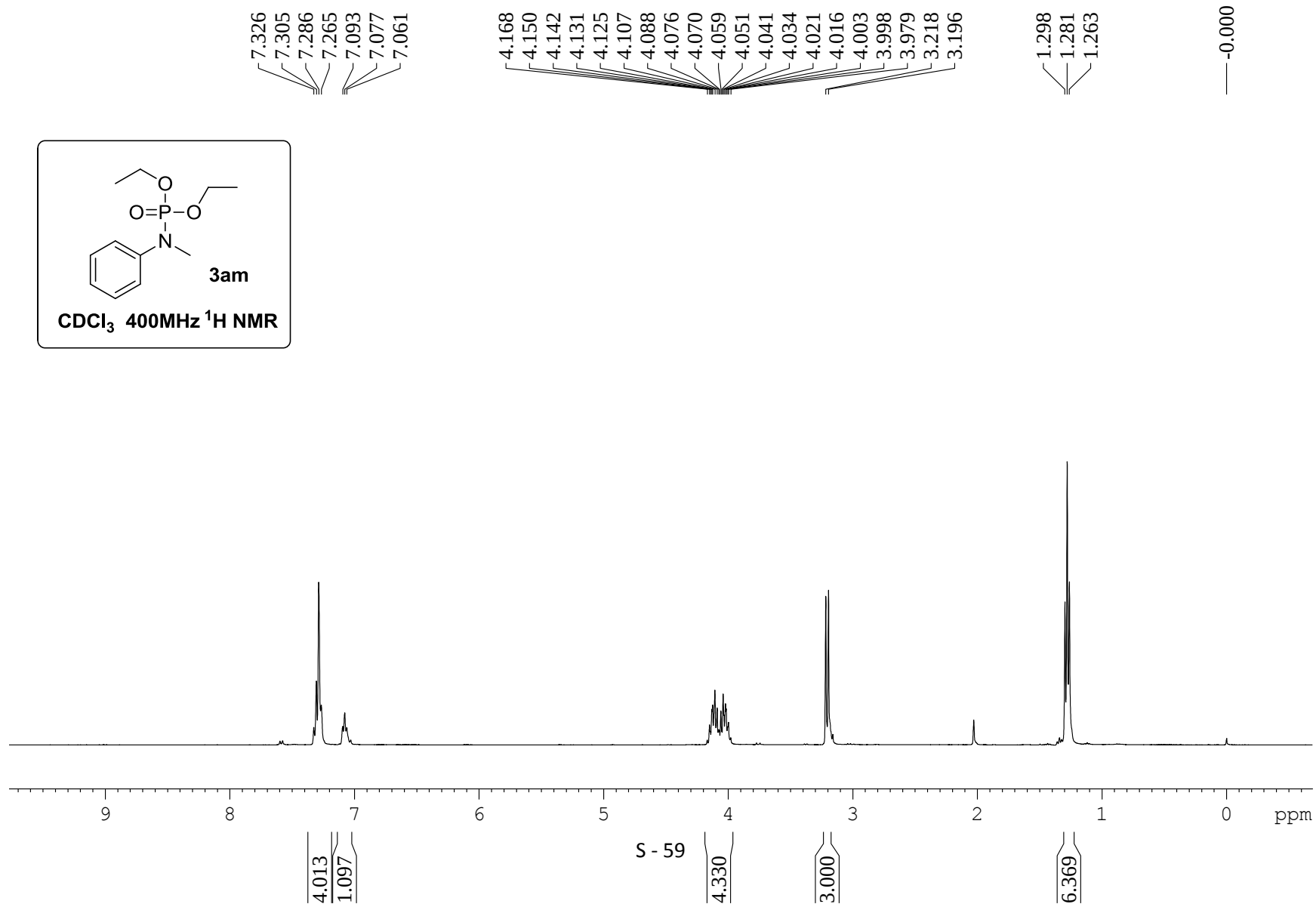
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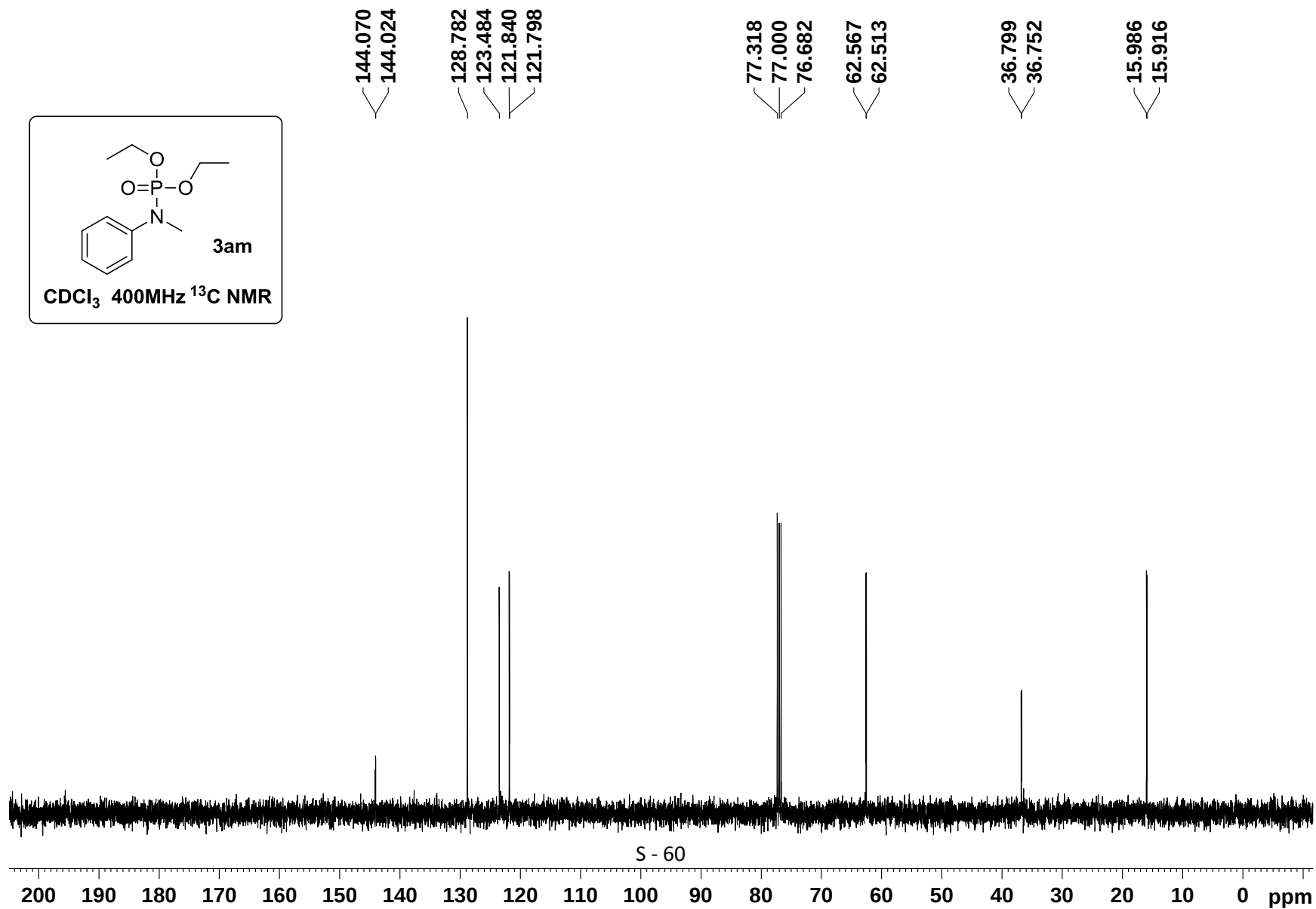
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61.461

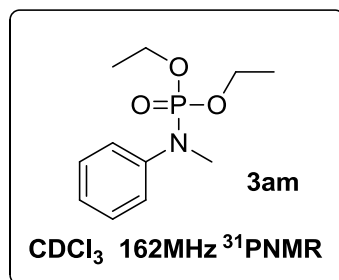
— 44.187

16.145
16.077
14.552

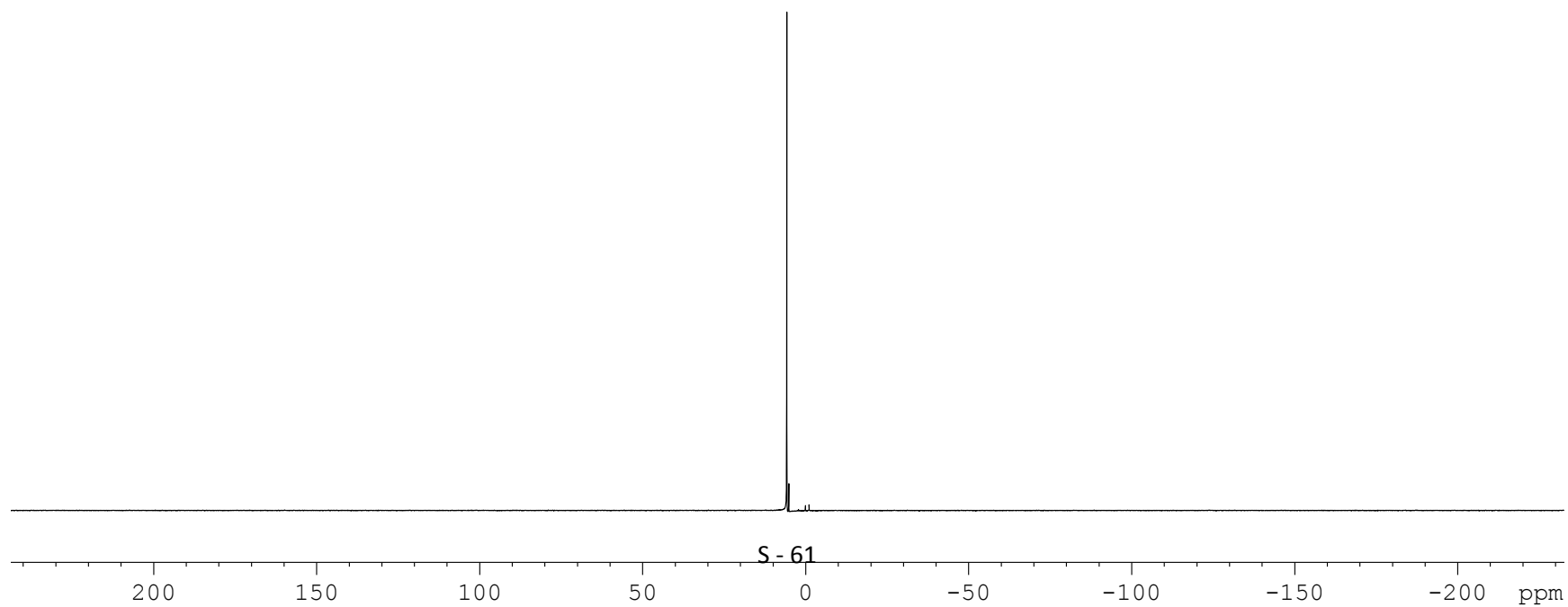


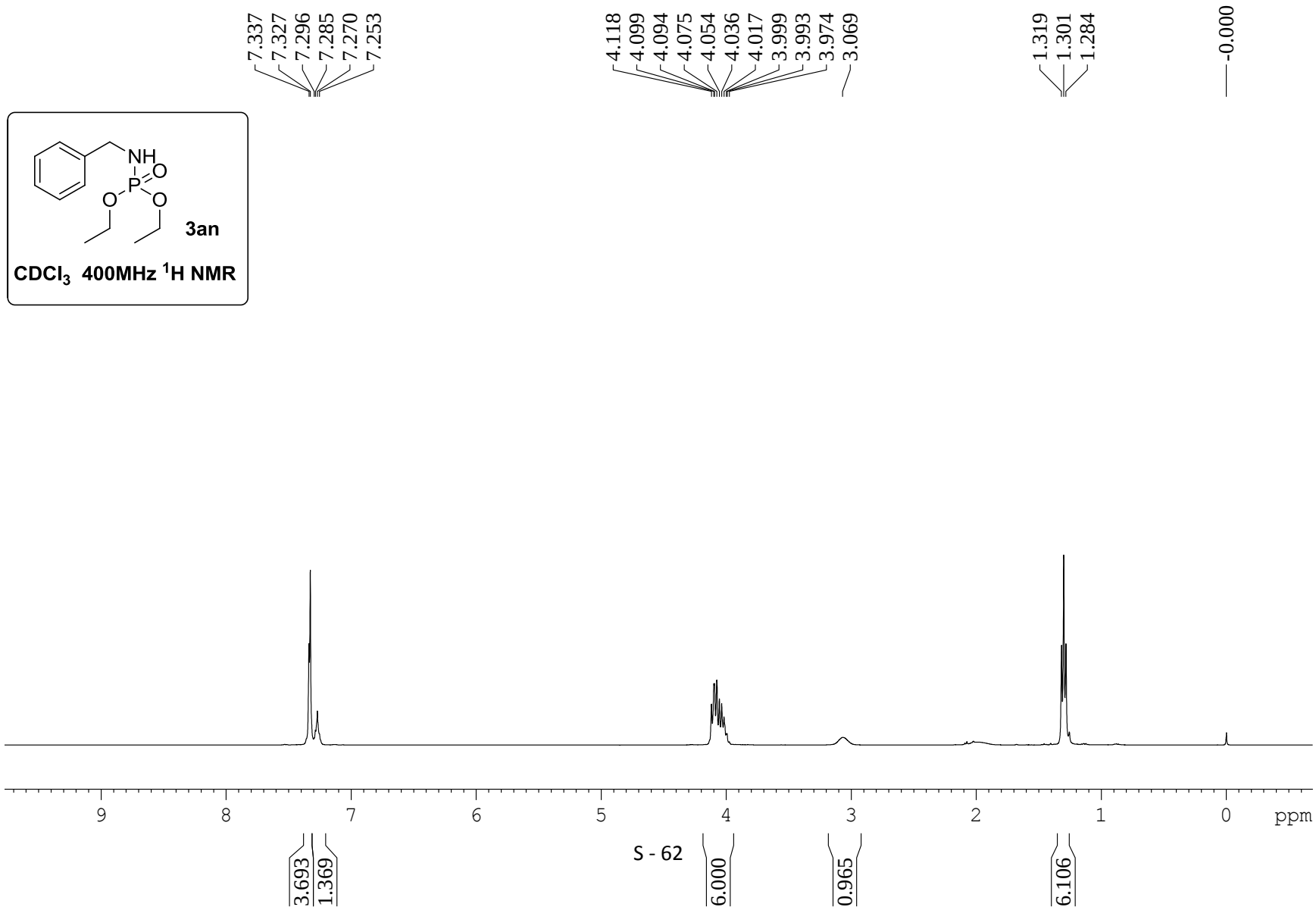


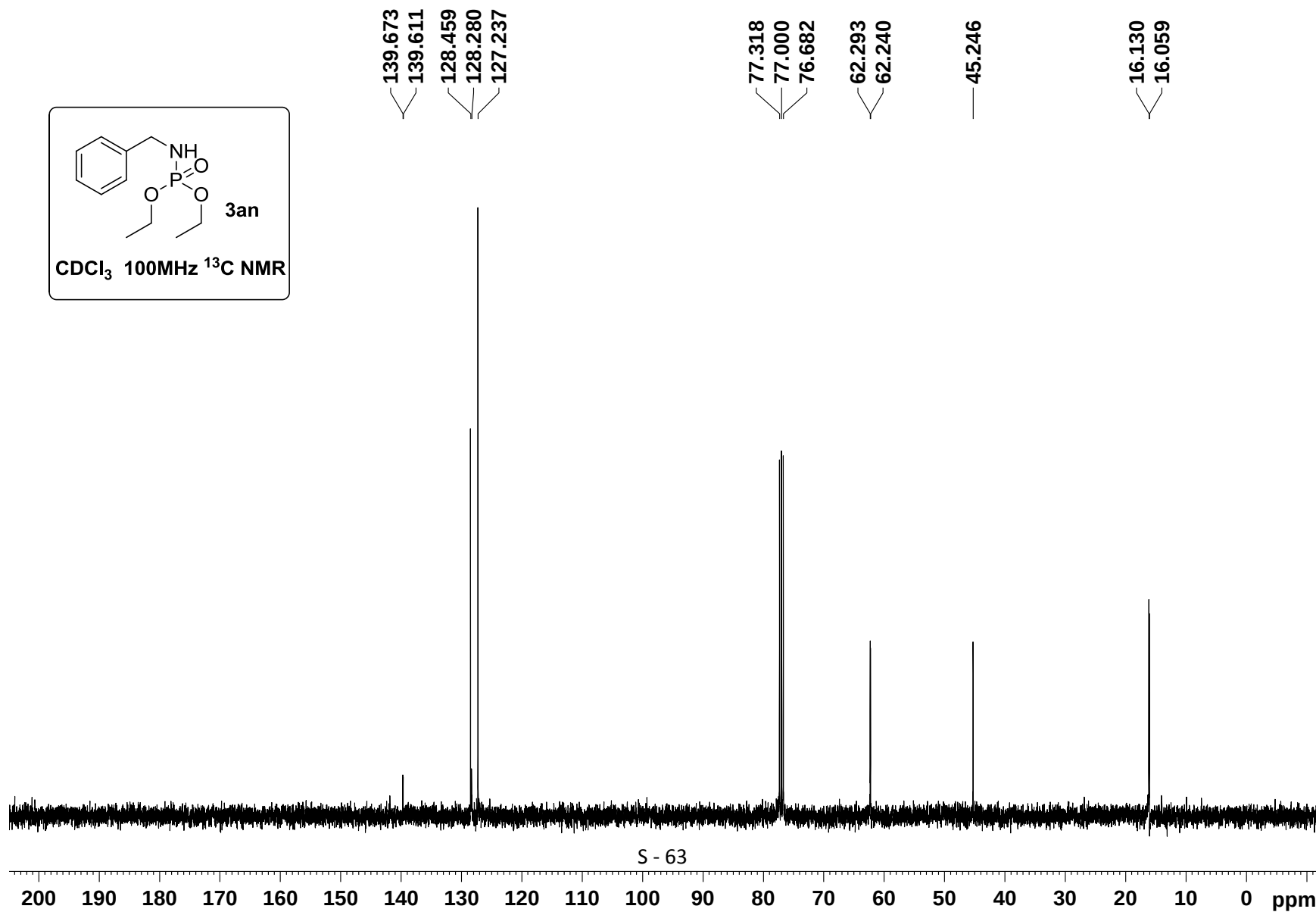
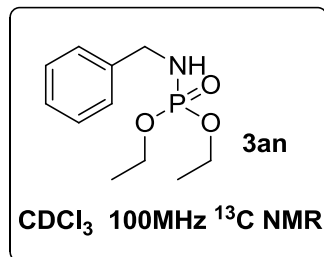


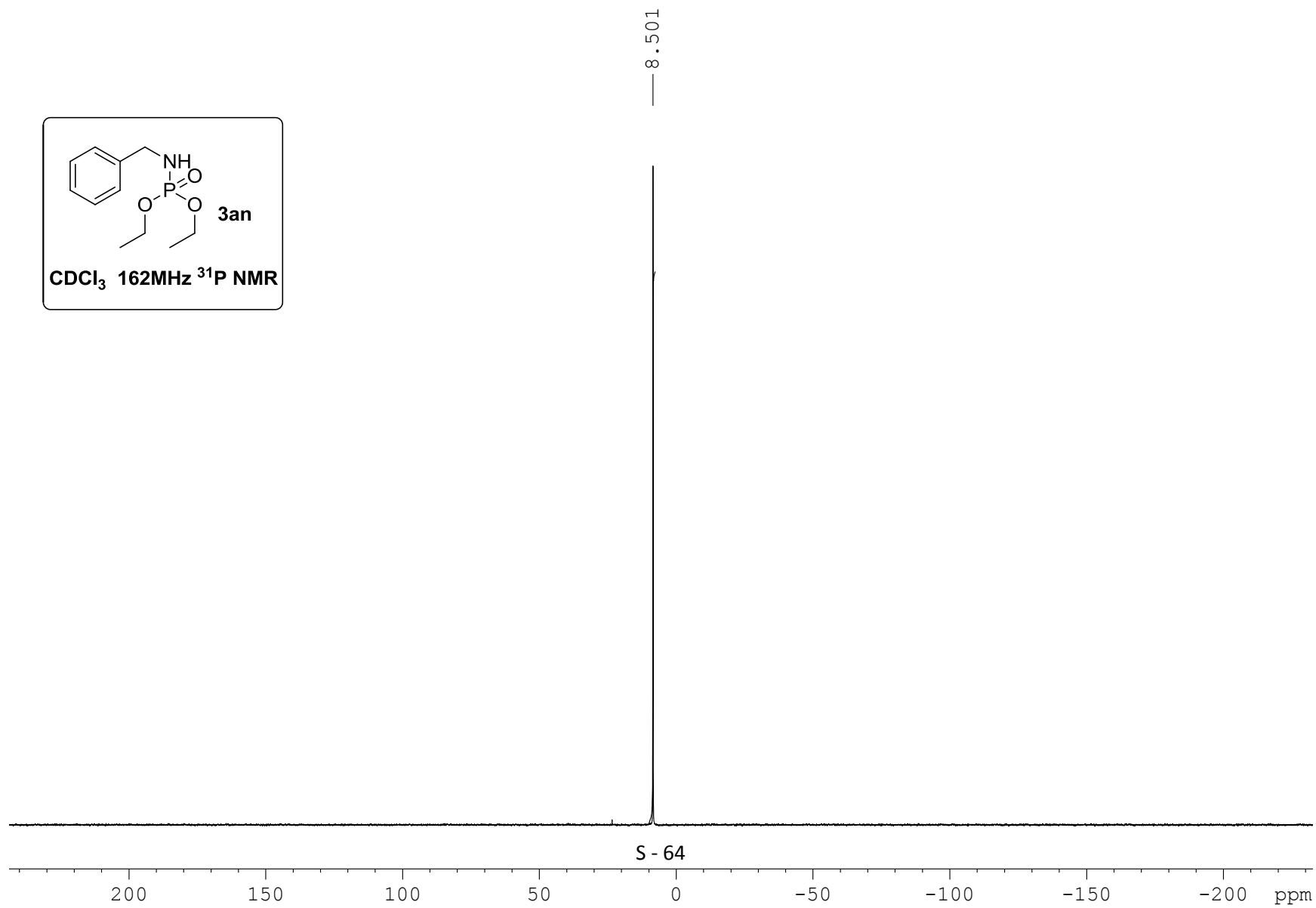
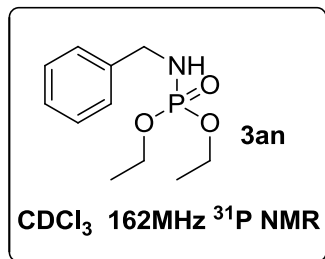


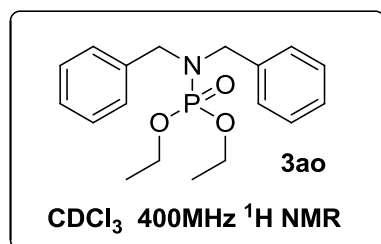
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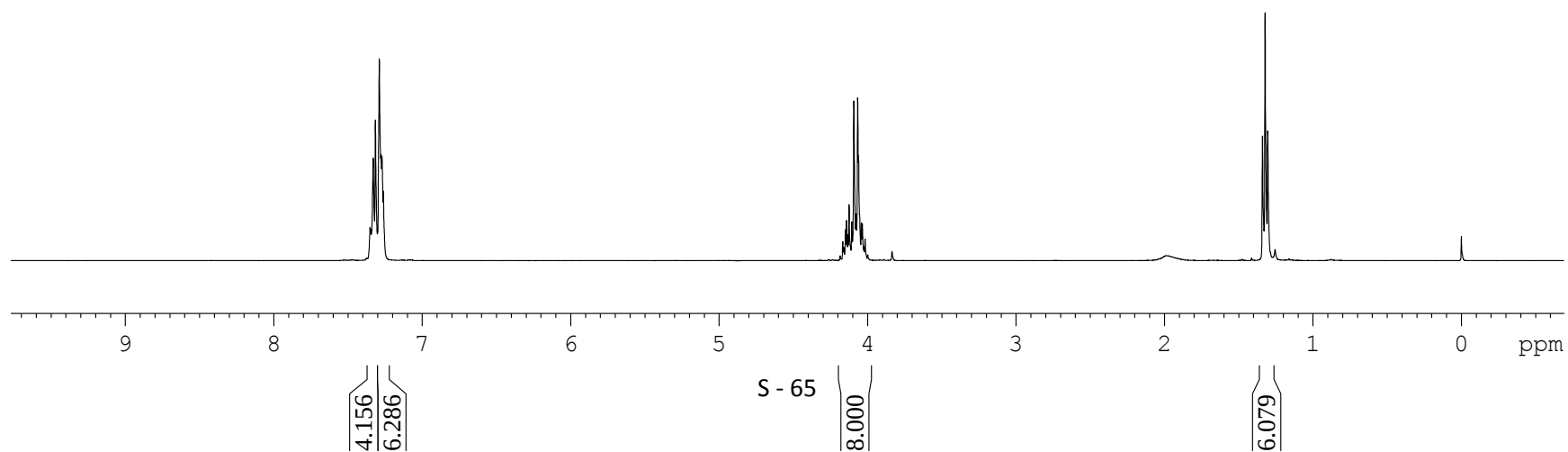


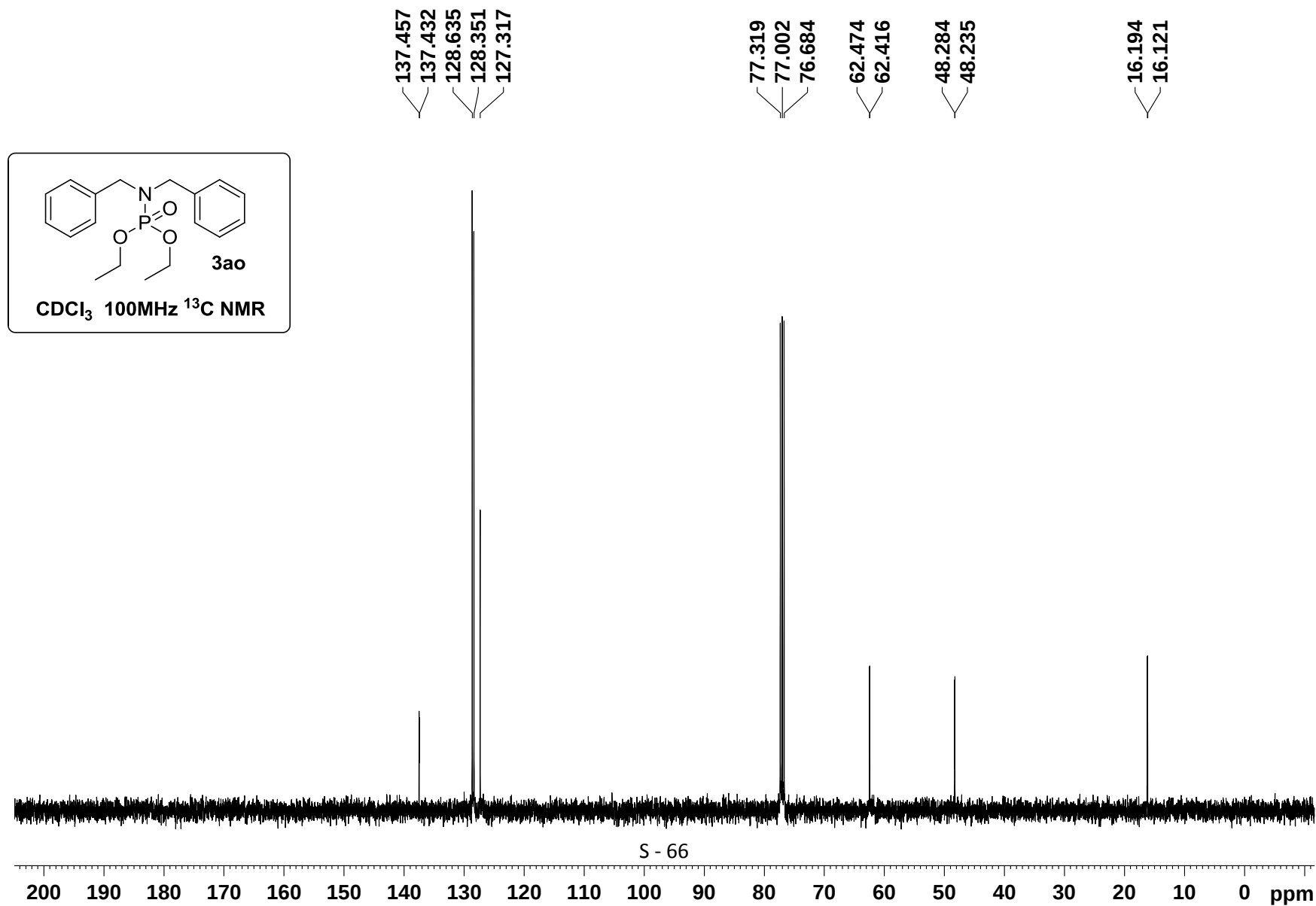
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7.346
7.329
7.314
7.288
7.276
7.270
7.261

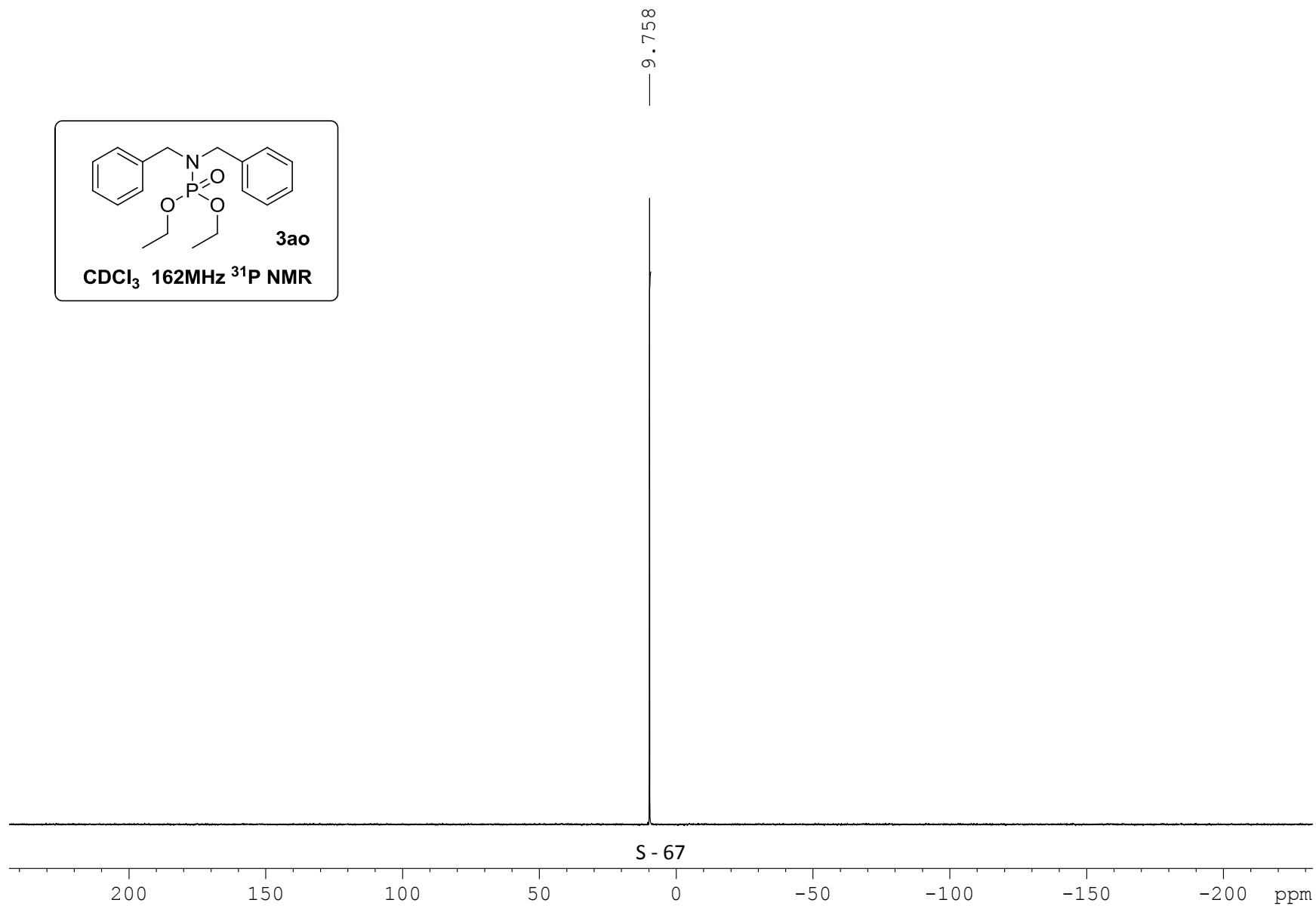
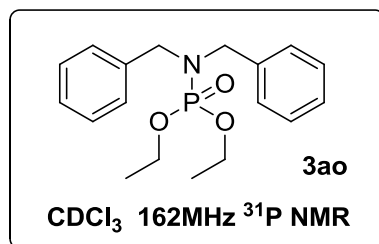
4.184
4.166
4.159
4.149
4.141
4.131
4.123
4.114
4.106
4.091
4.077
4.065
4.059
4.053
4.041
4.034
4.023
4.016
3.998

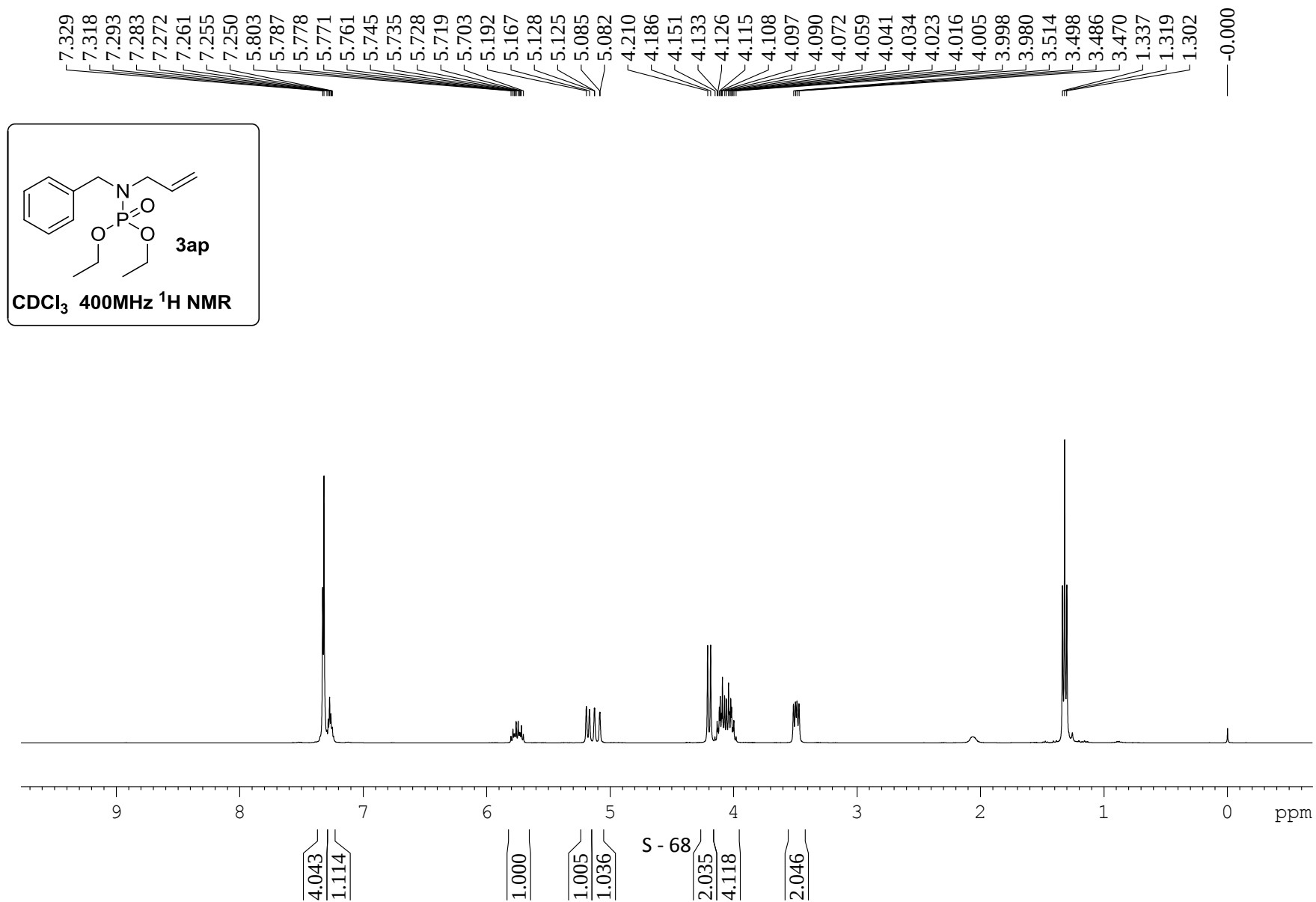
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1.322
1.305

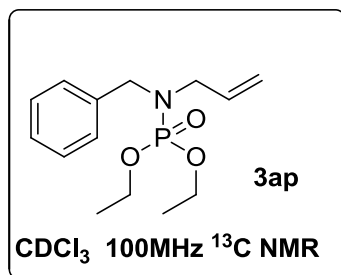
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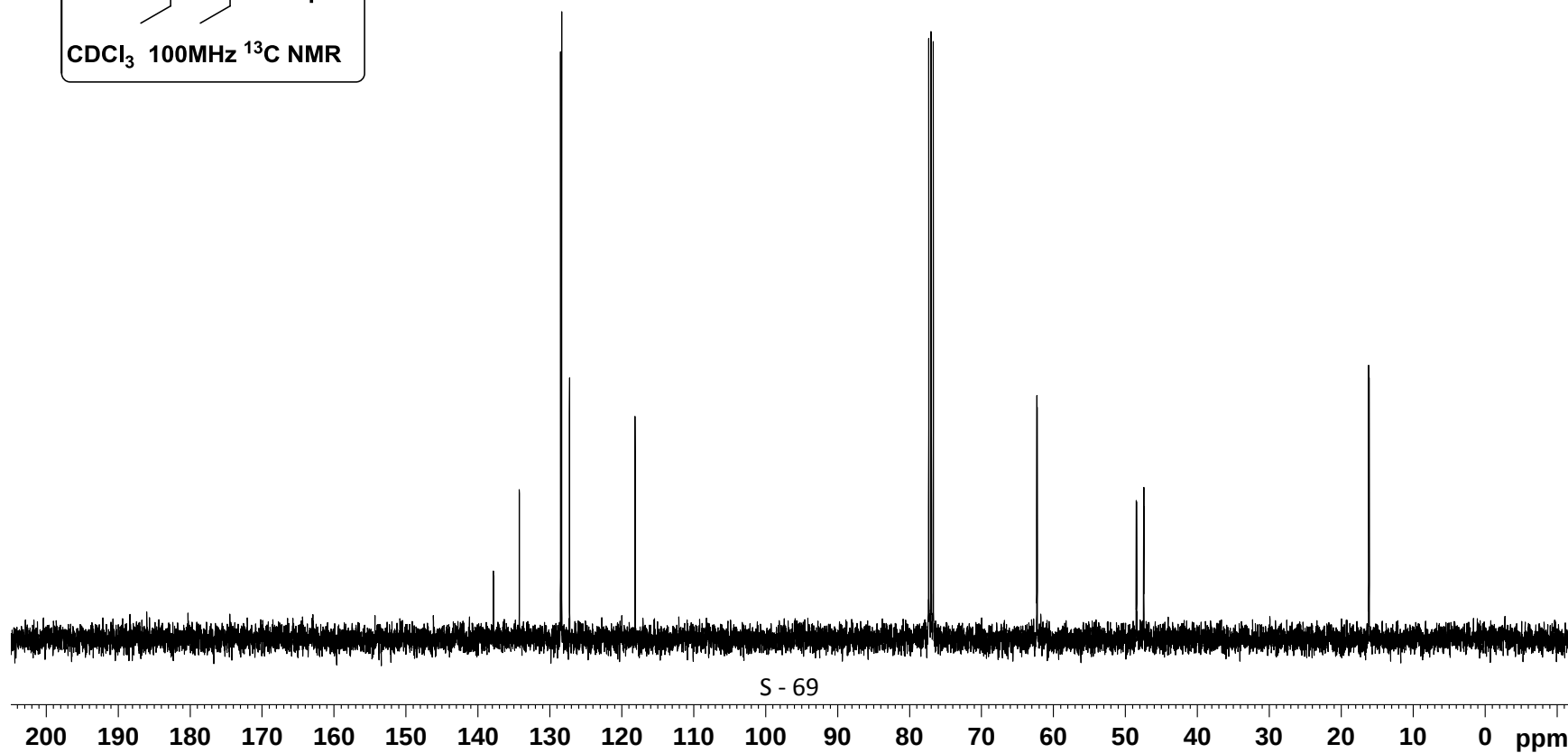
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128.462
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127.217
118.130

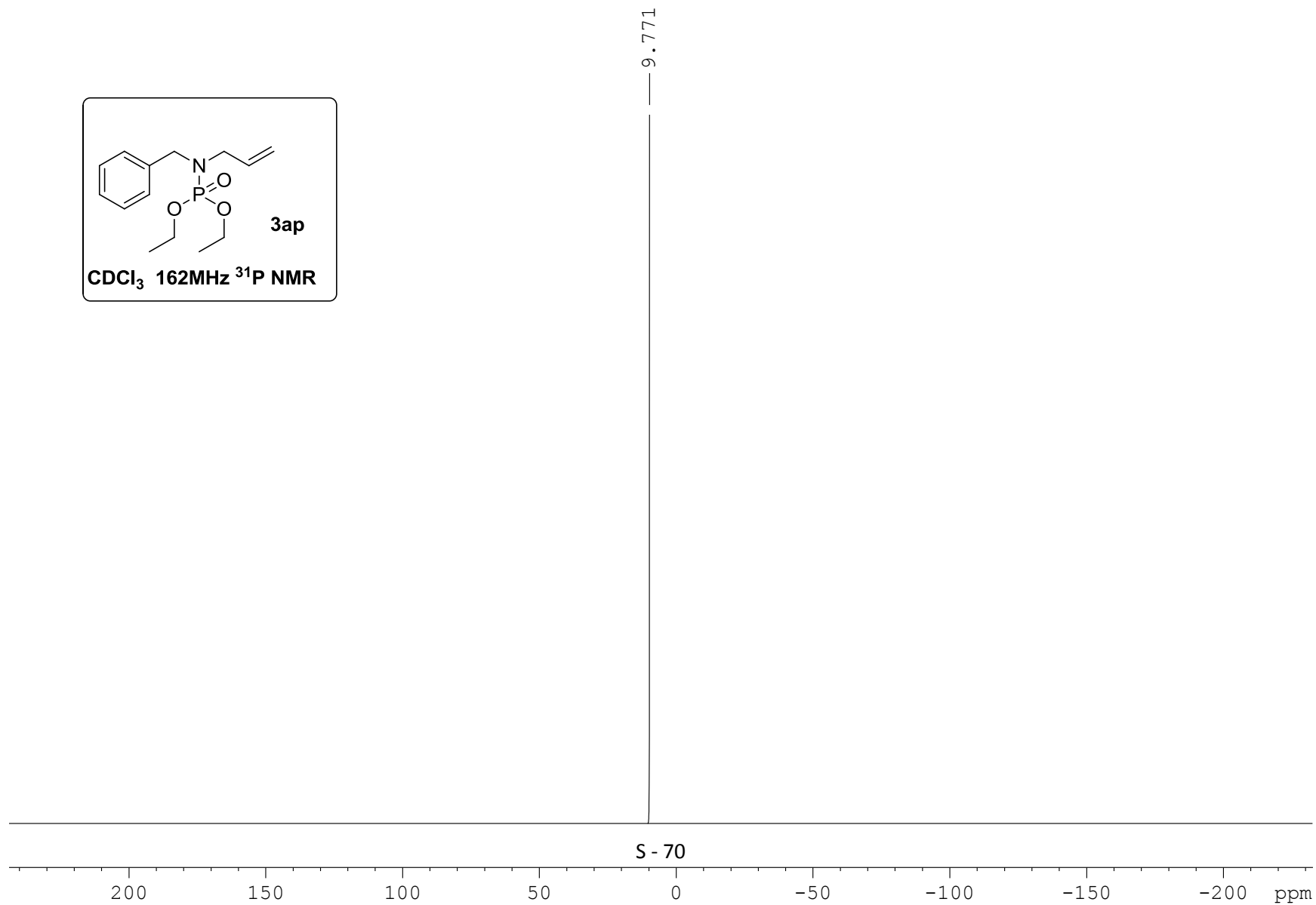
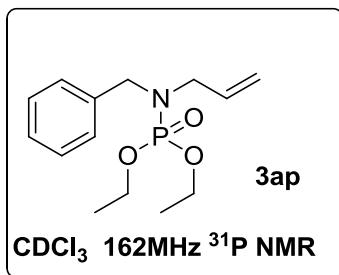
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76.999
76.681

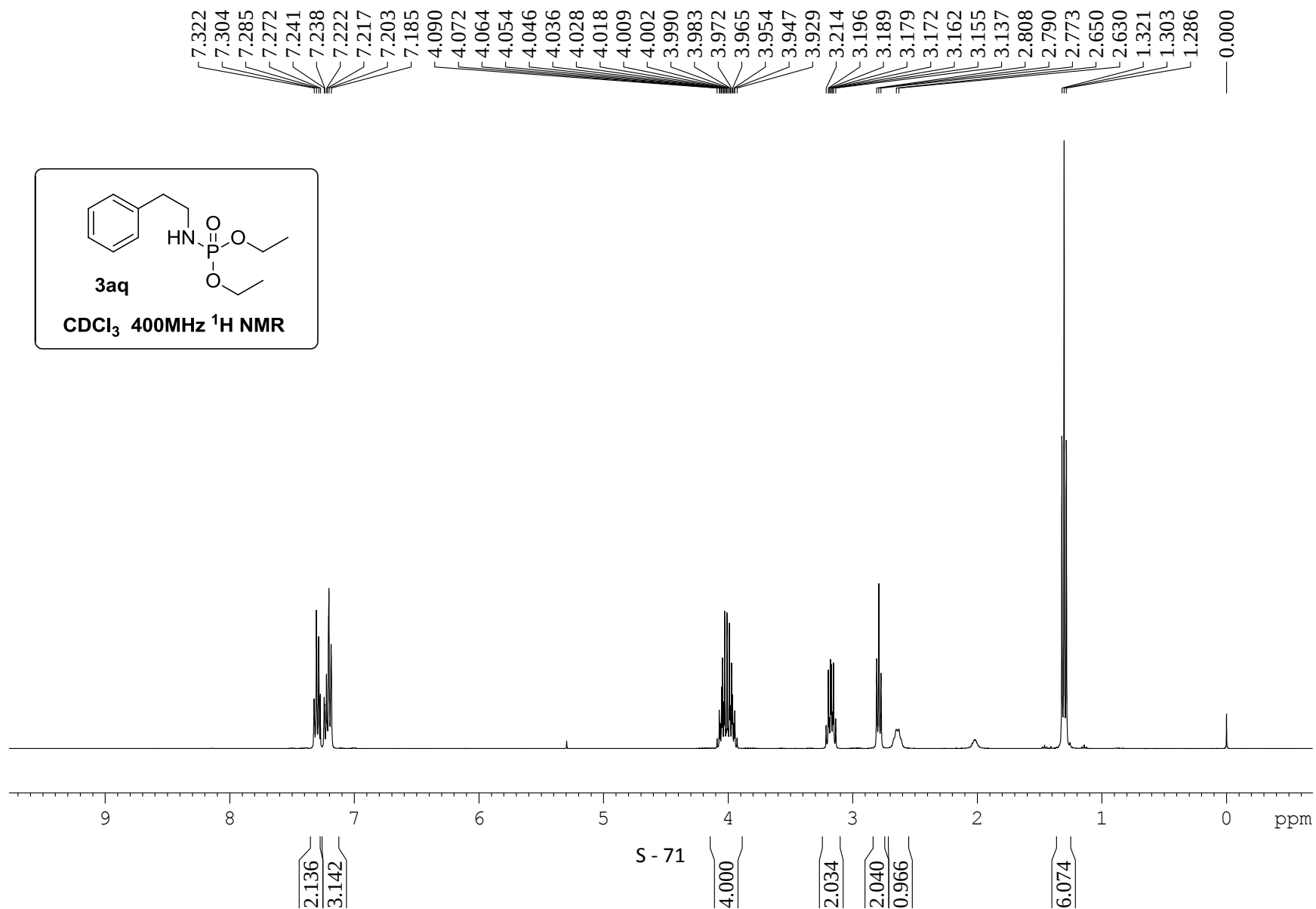
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62.237

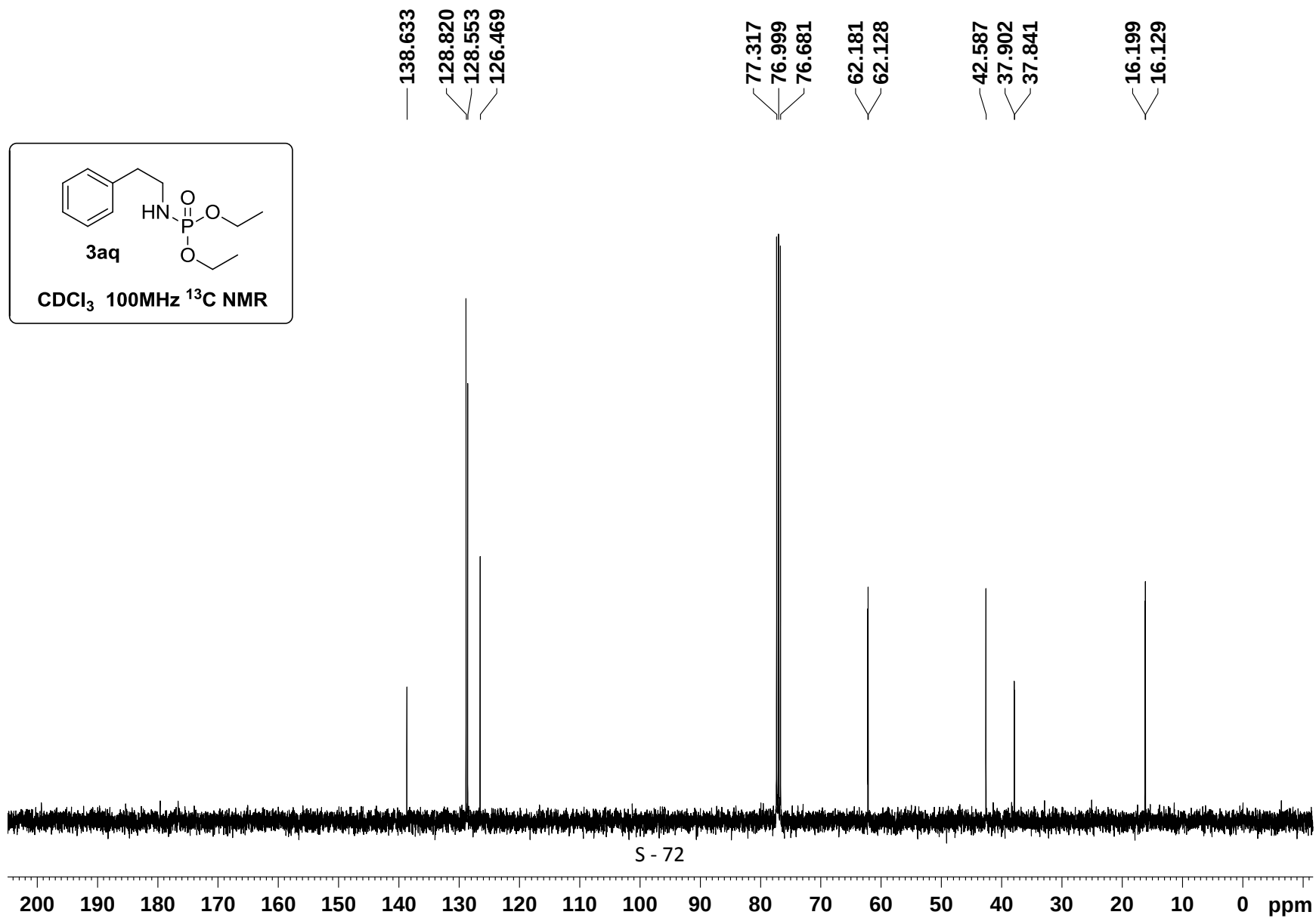
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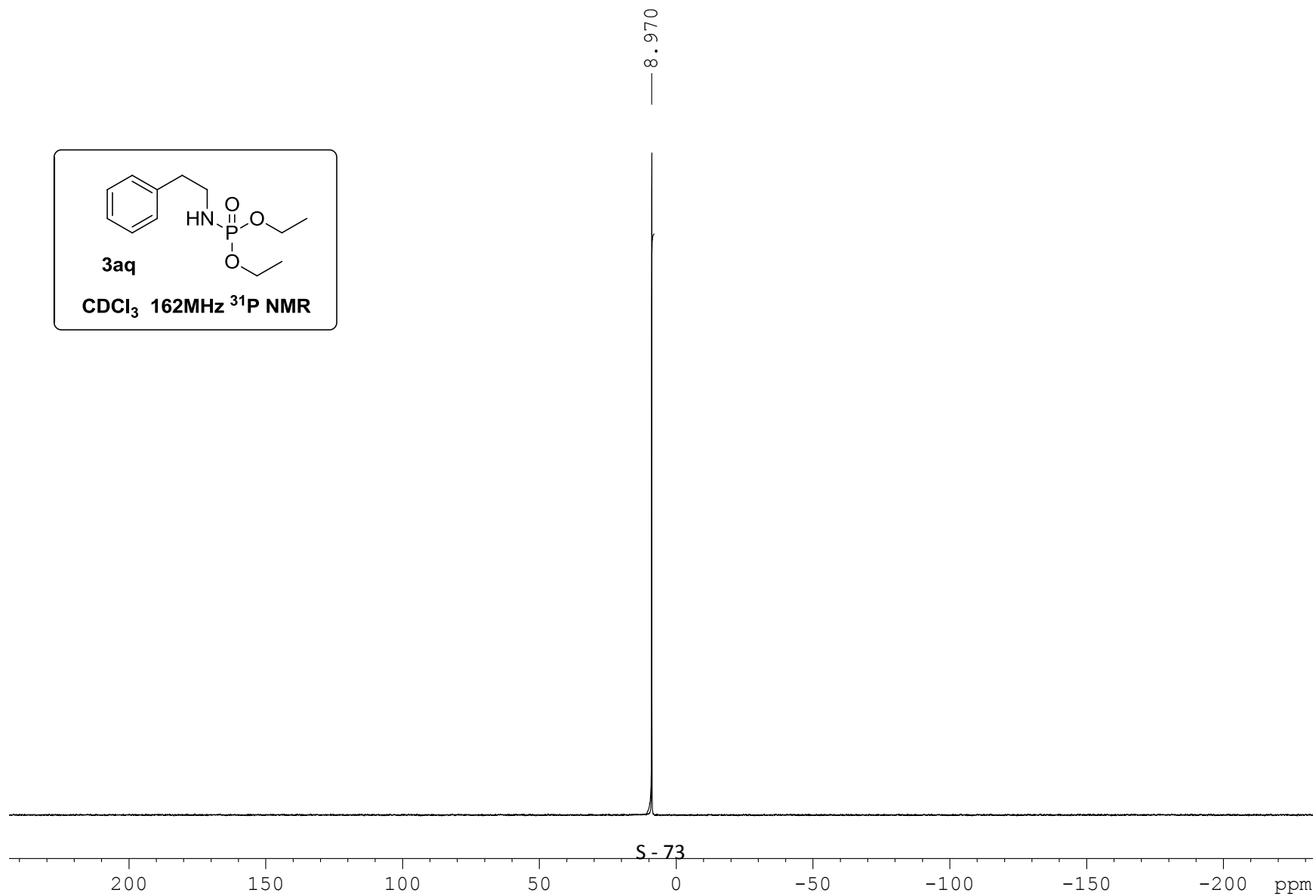
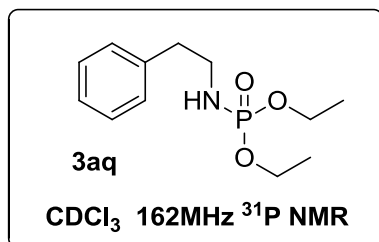
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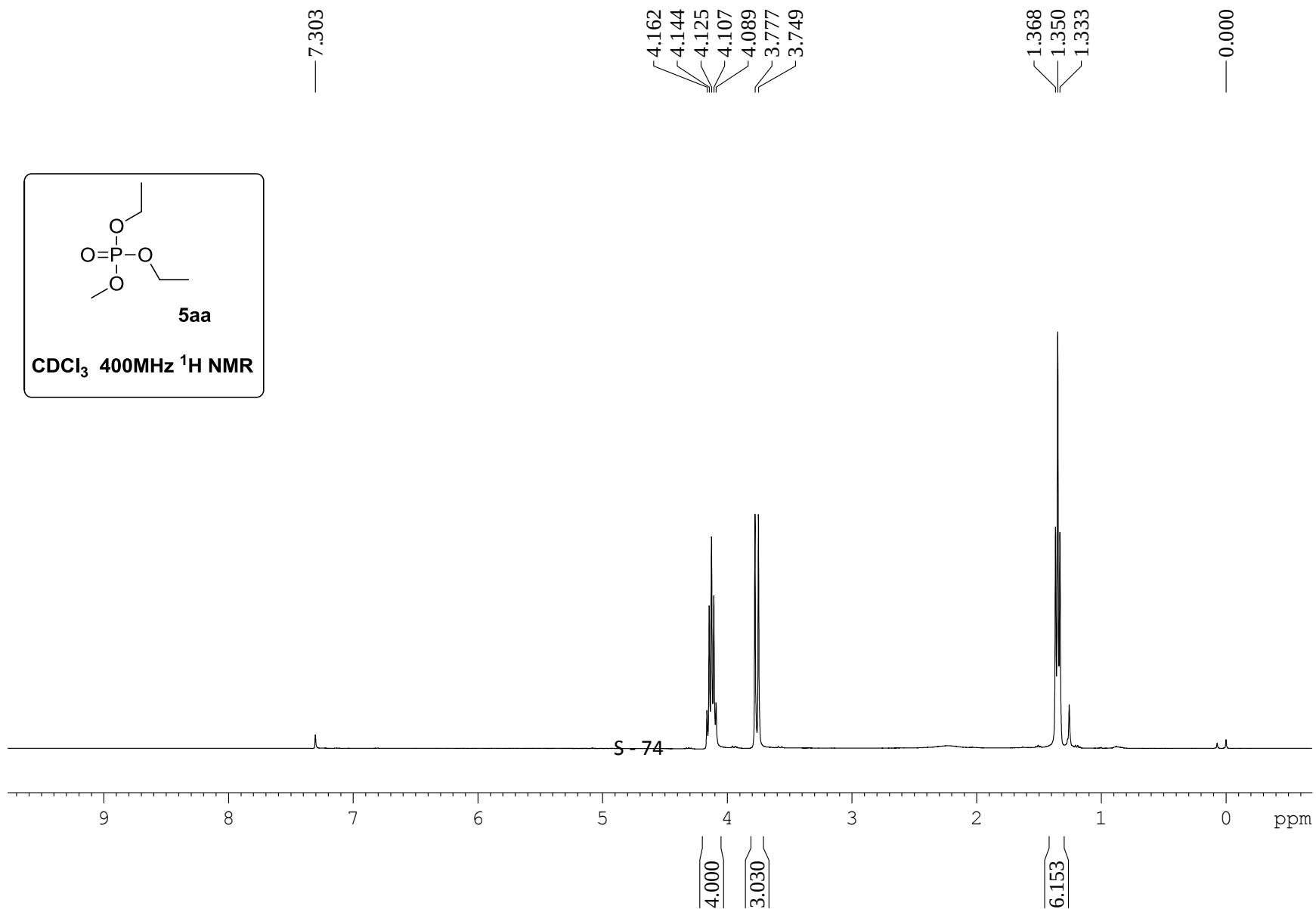


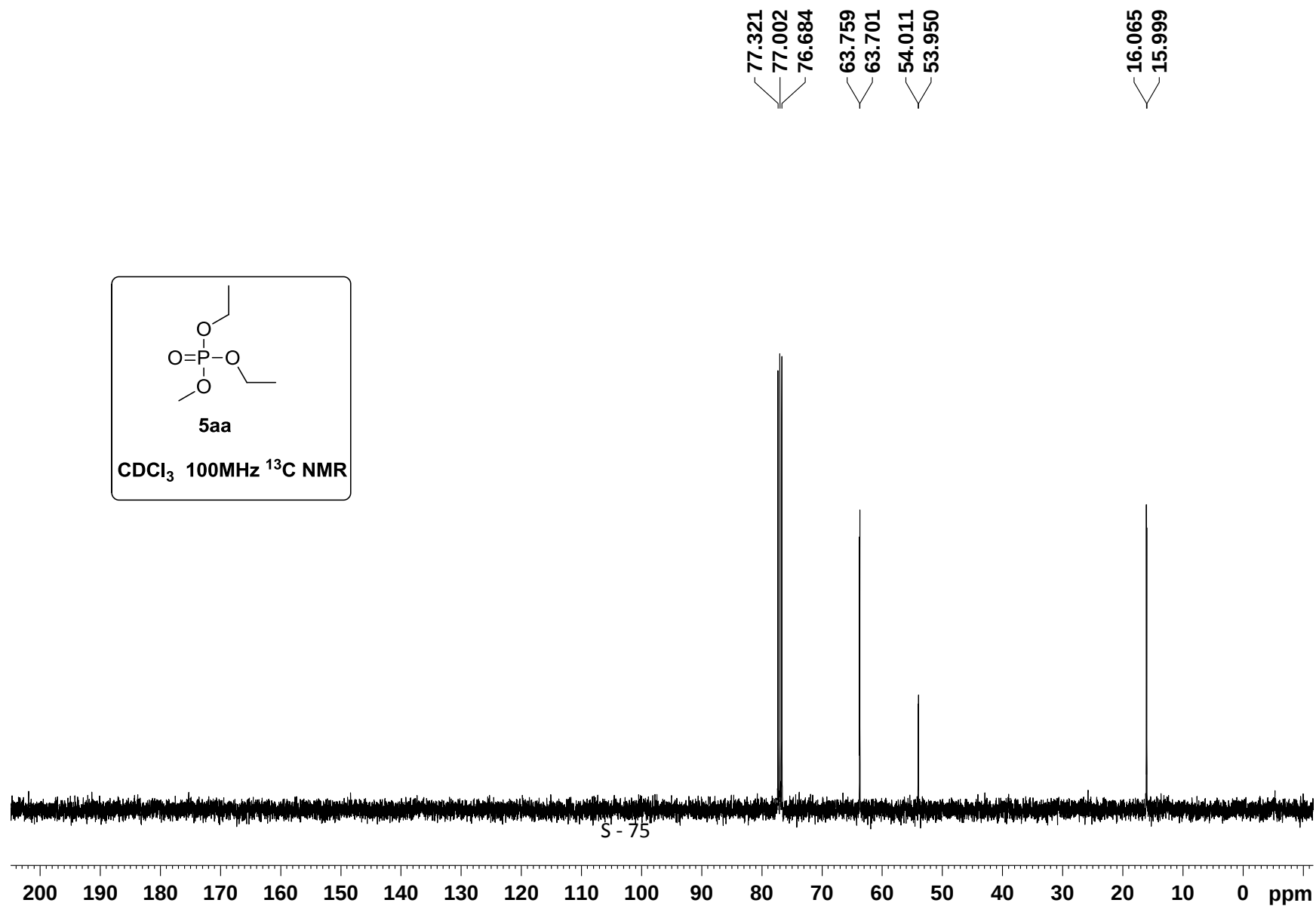
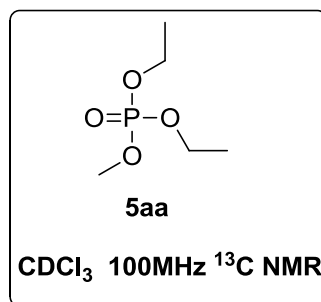


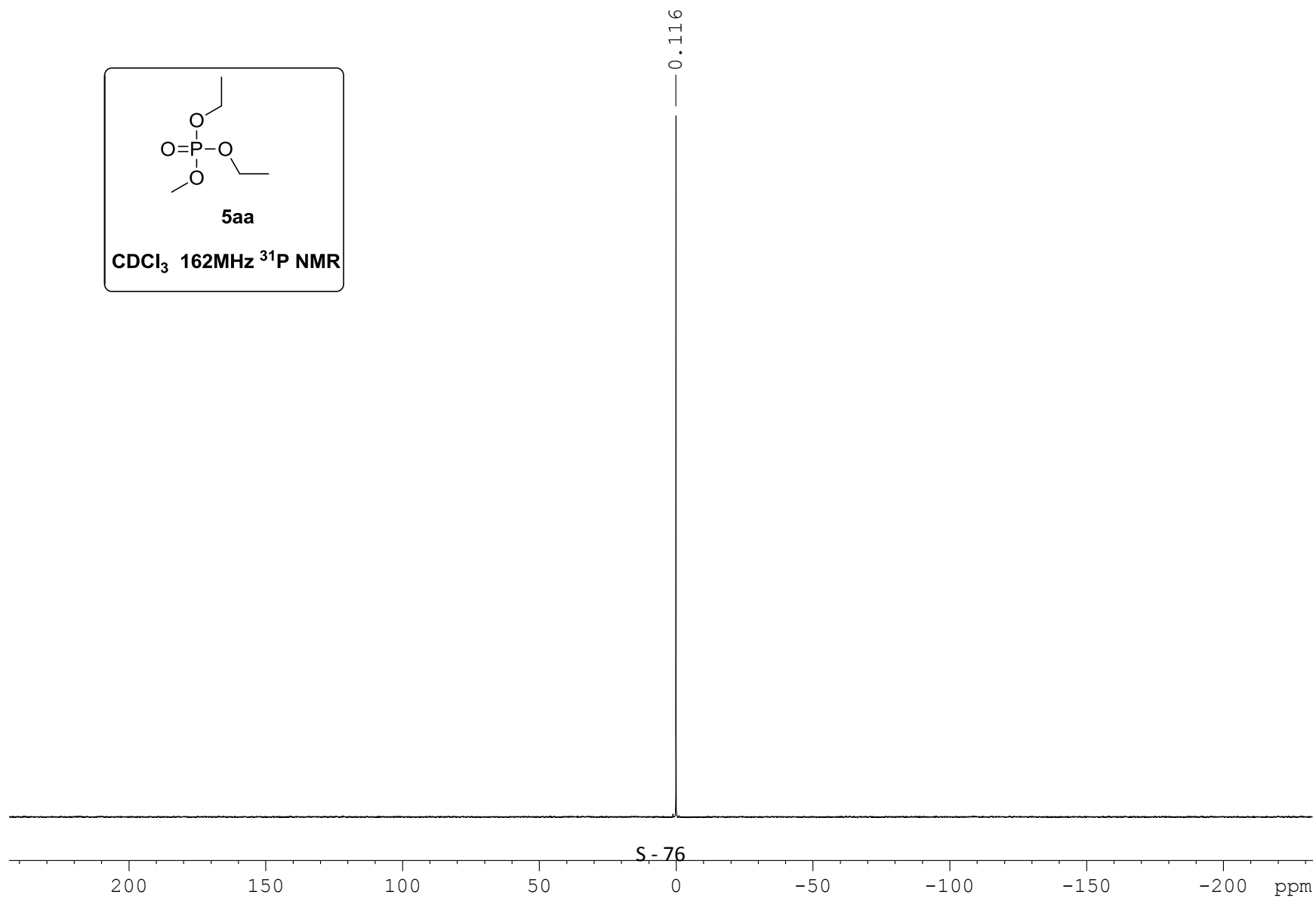
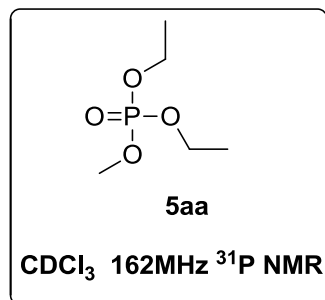


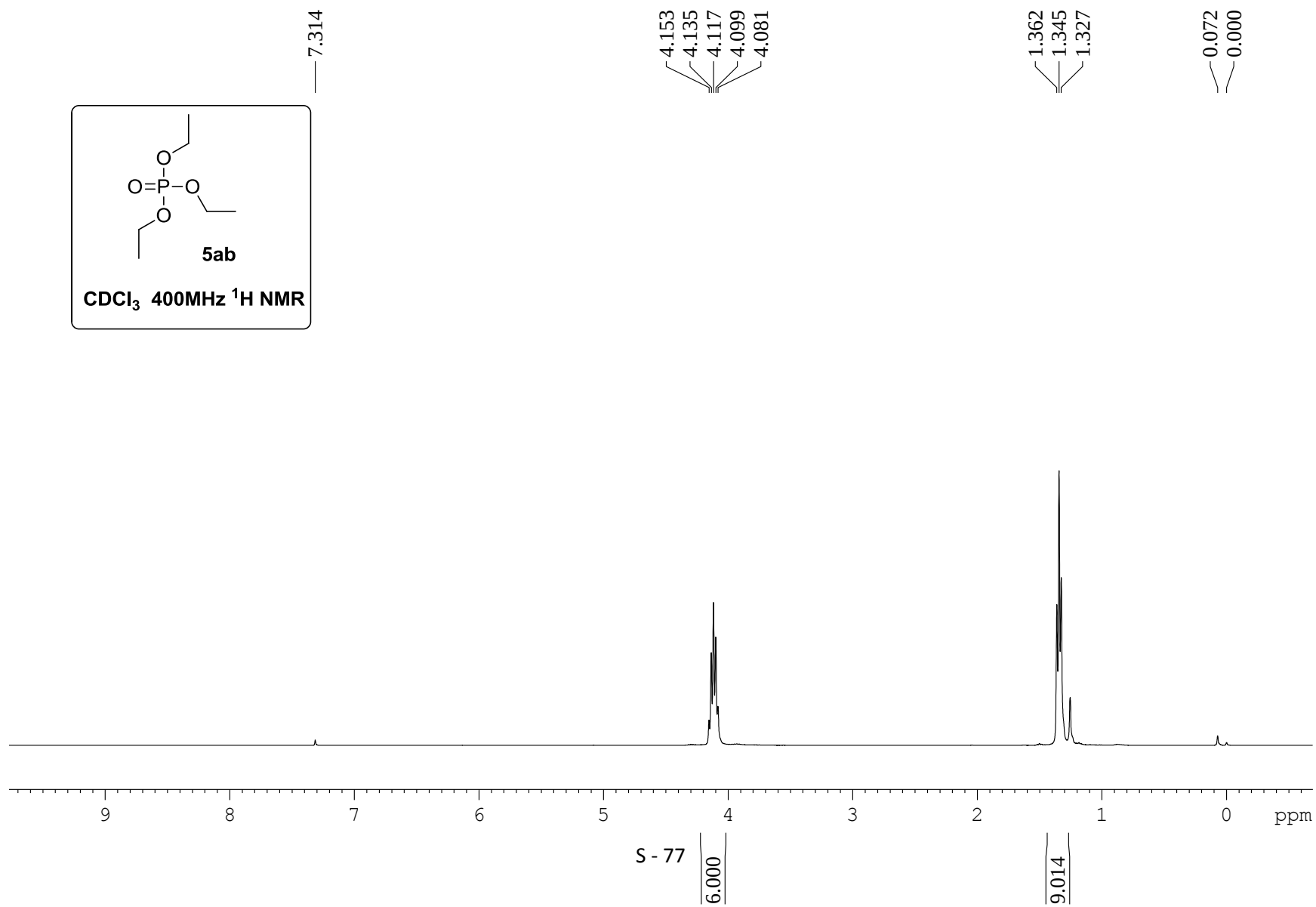
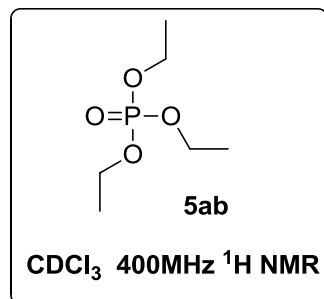


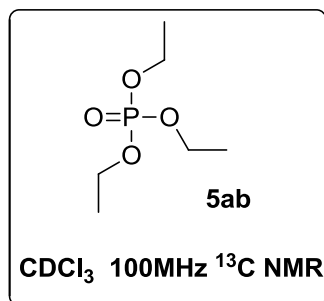






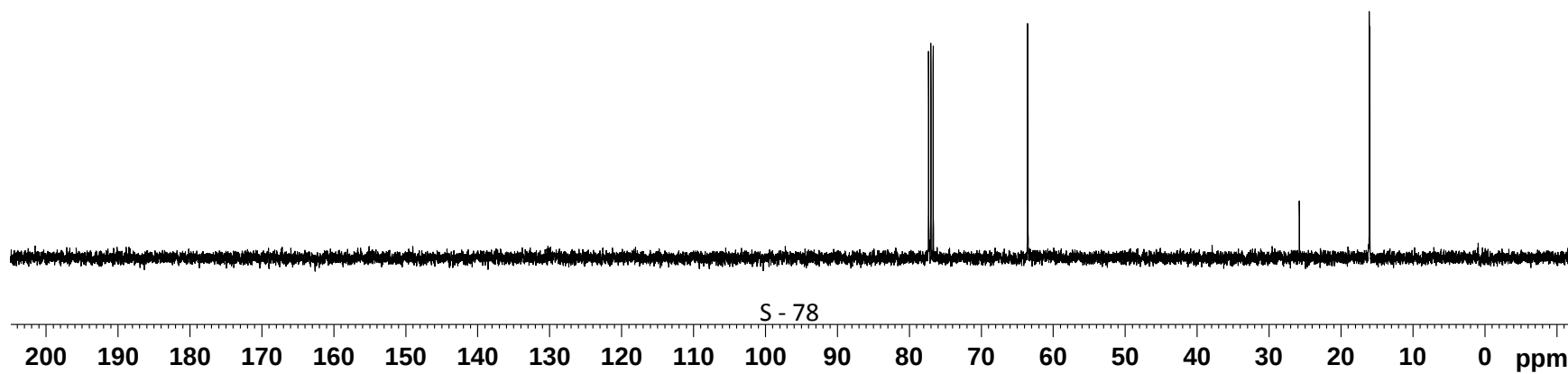


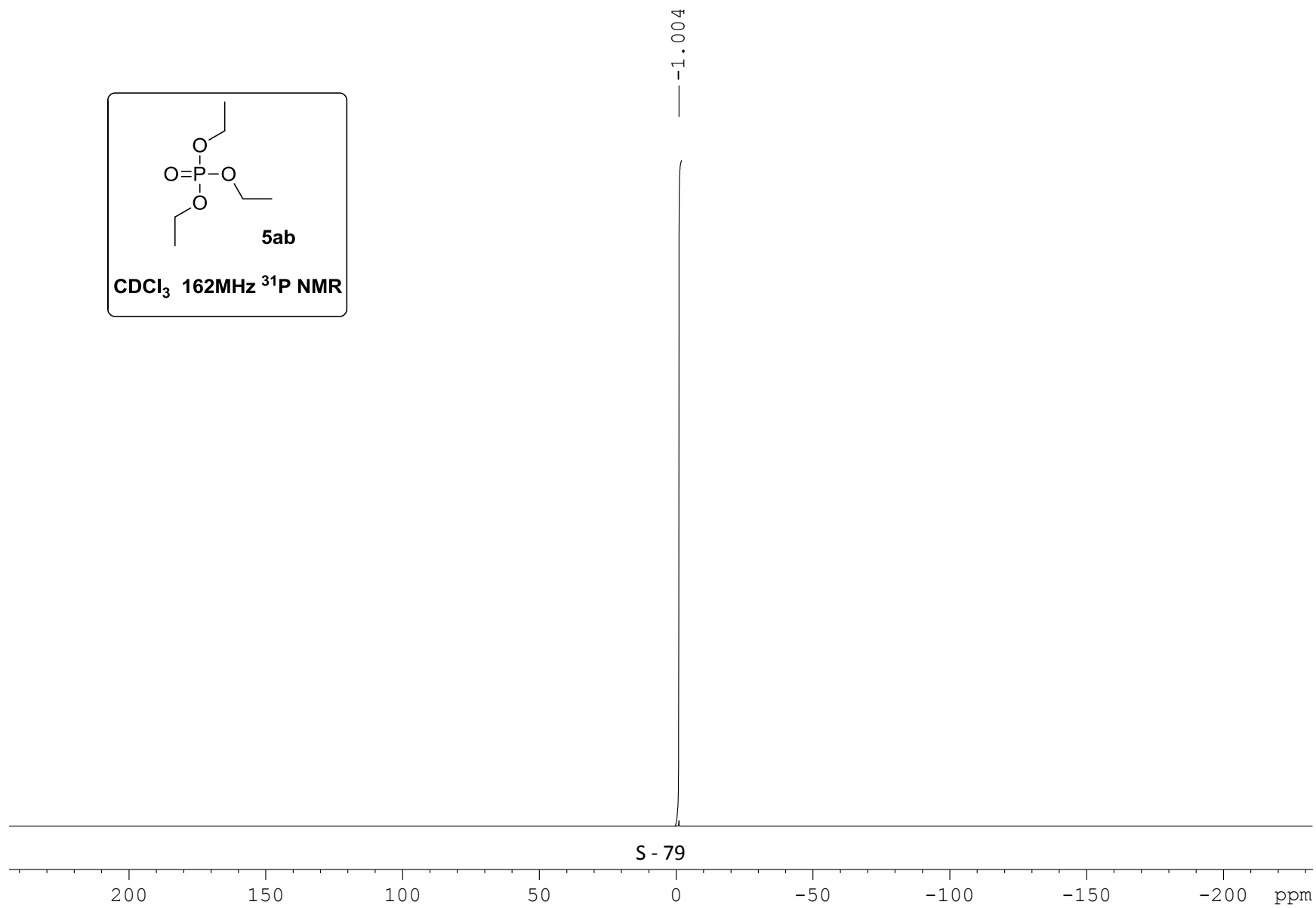
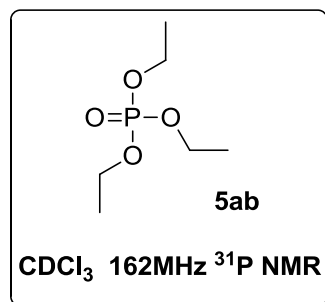


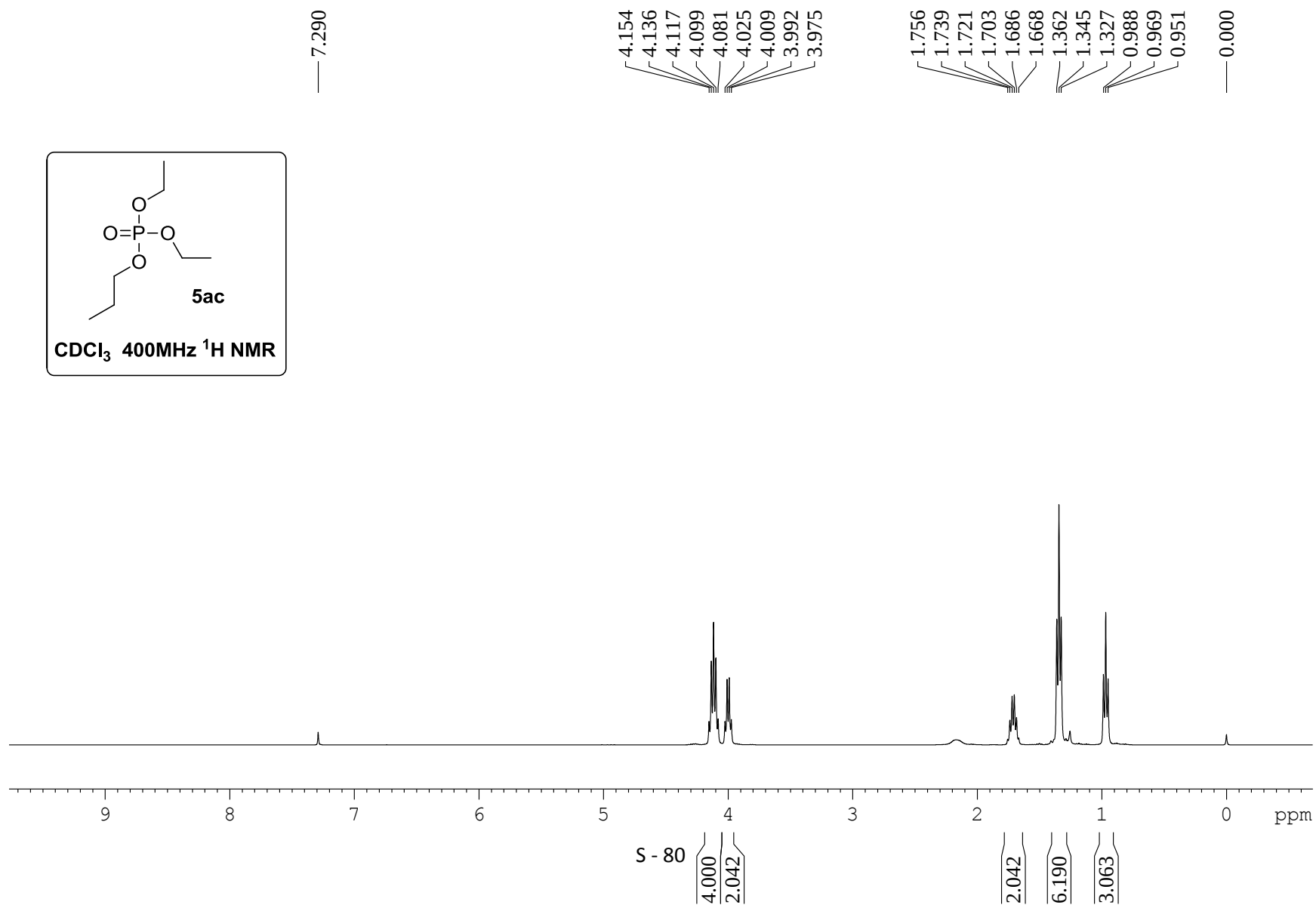
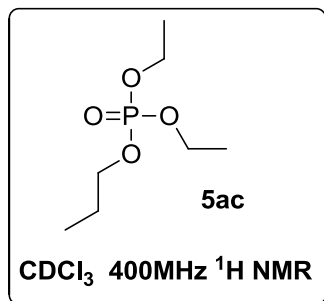


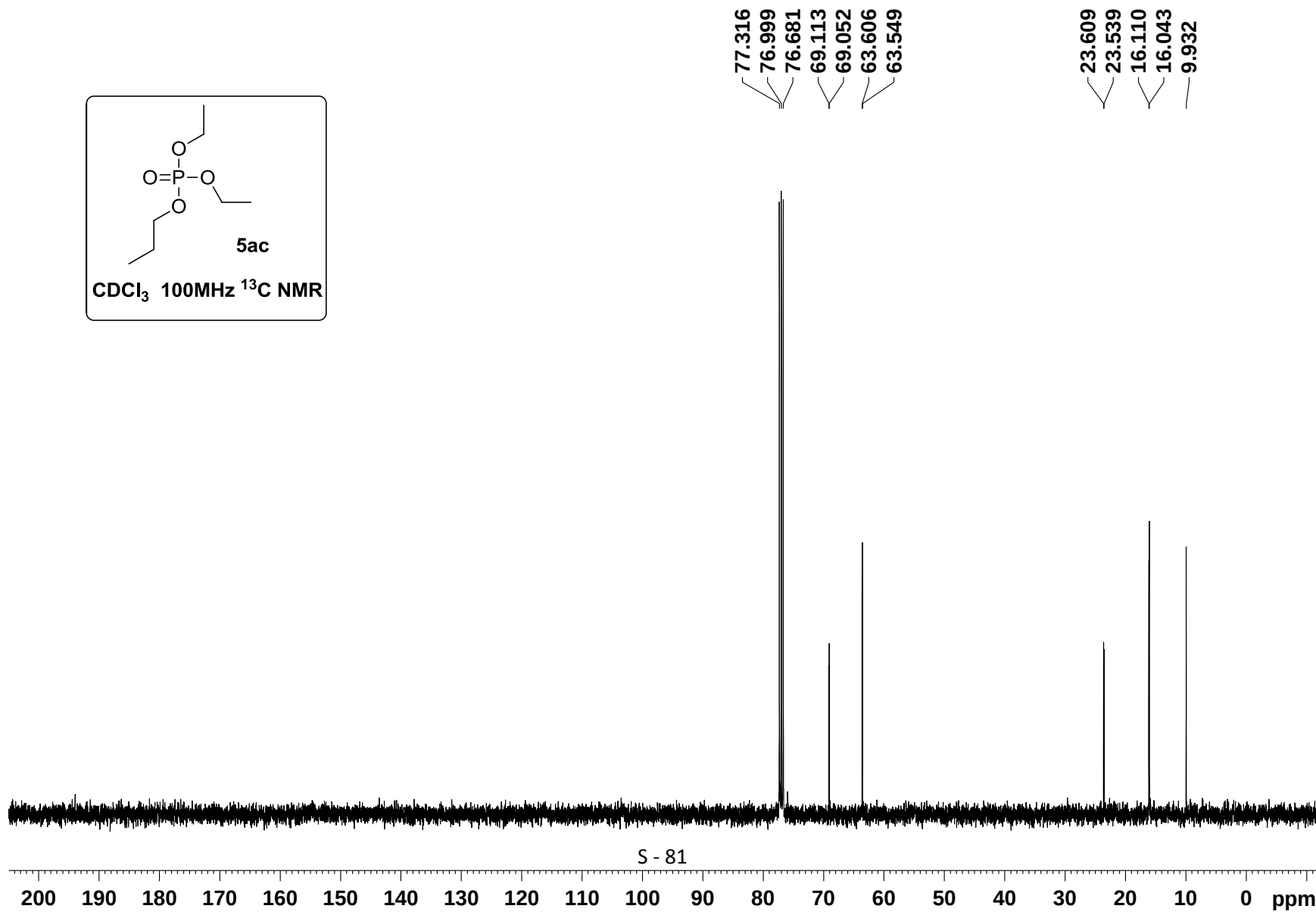
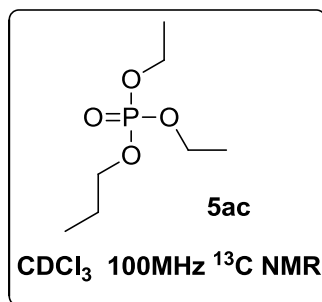
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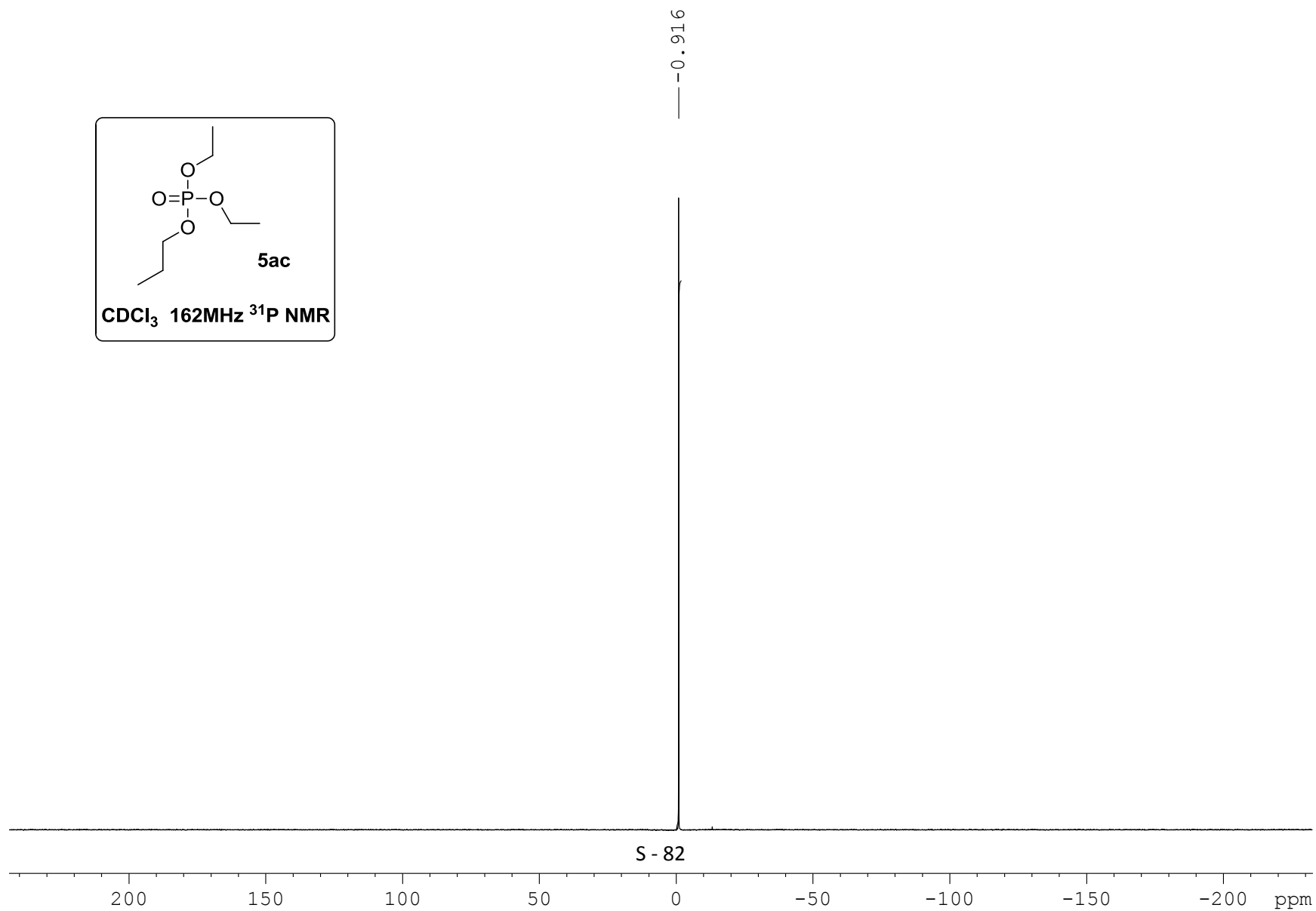
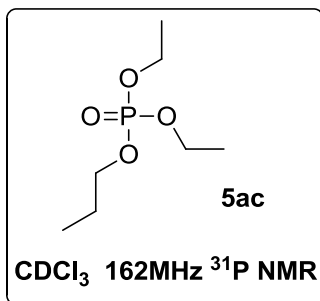
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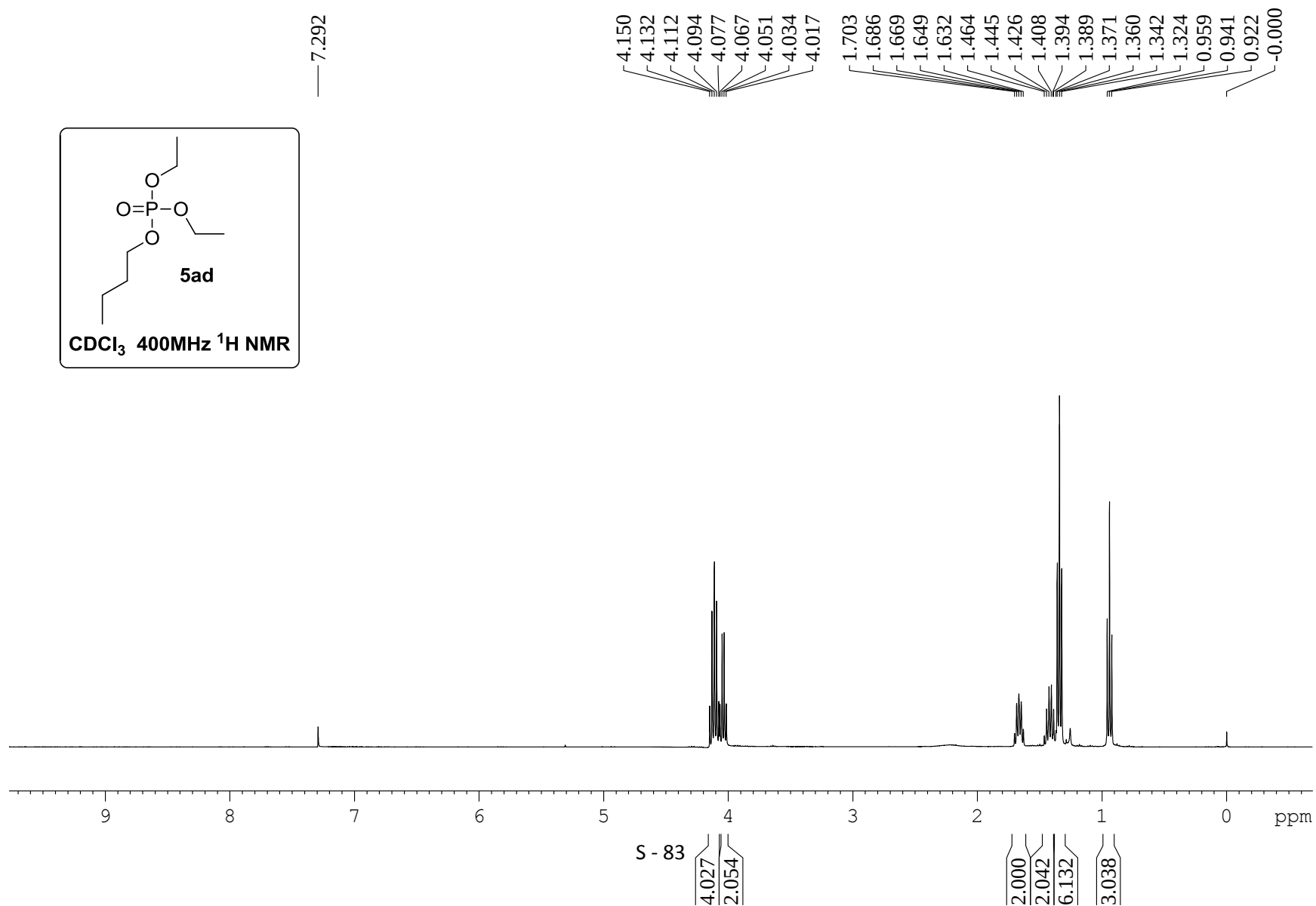


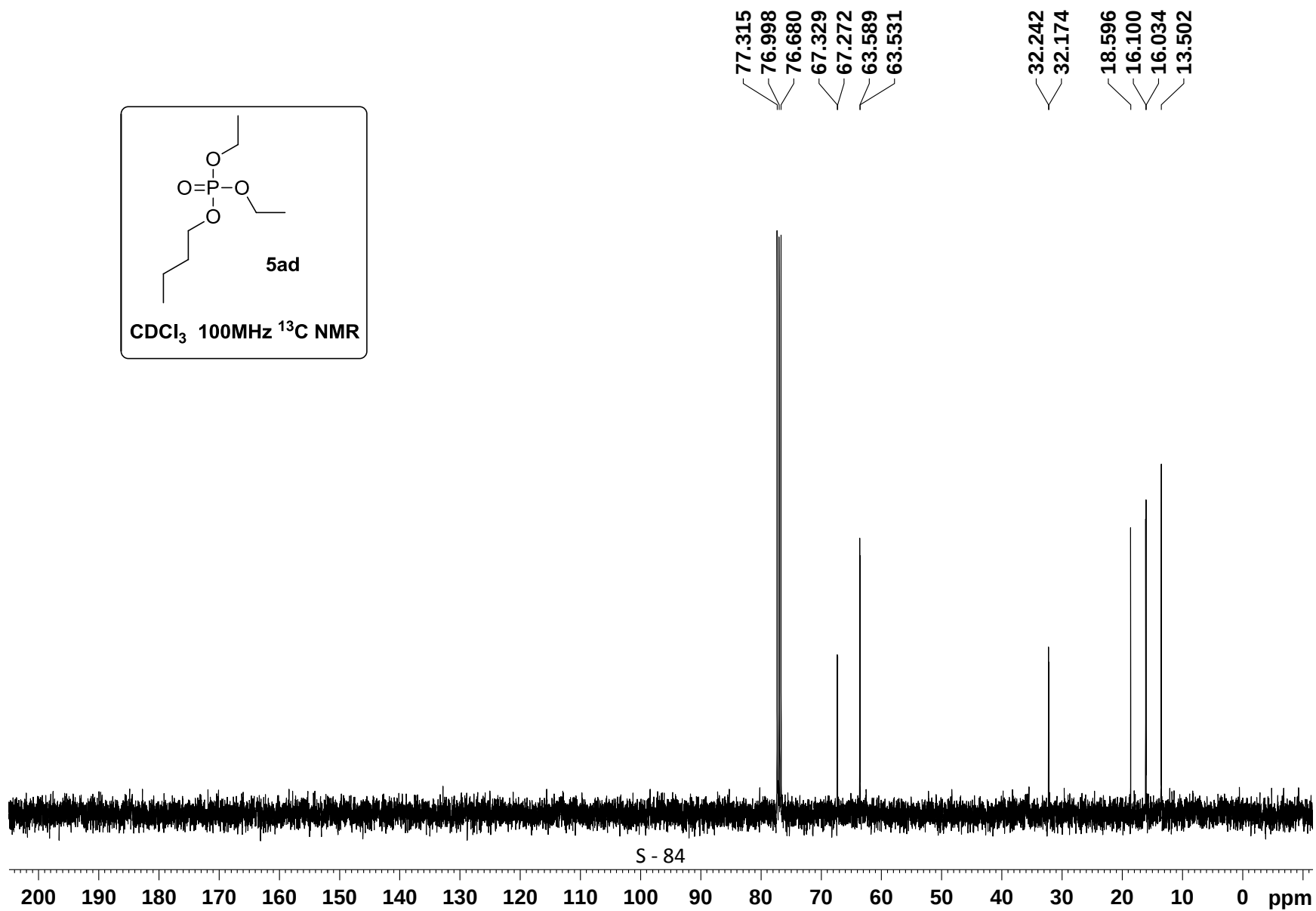
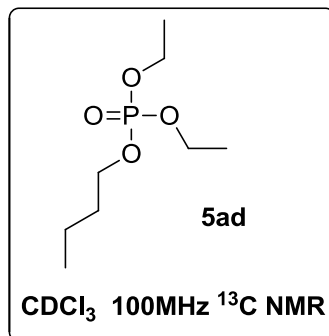


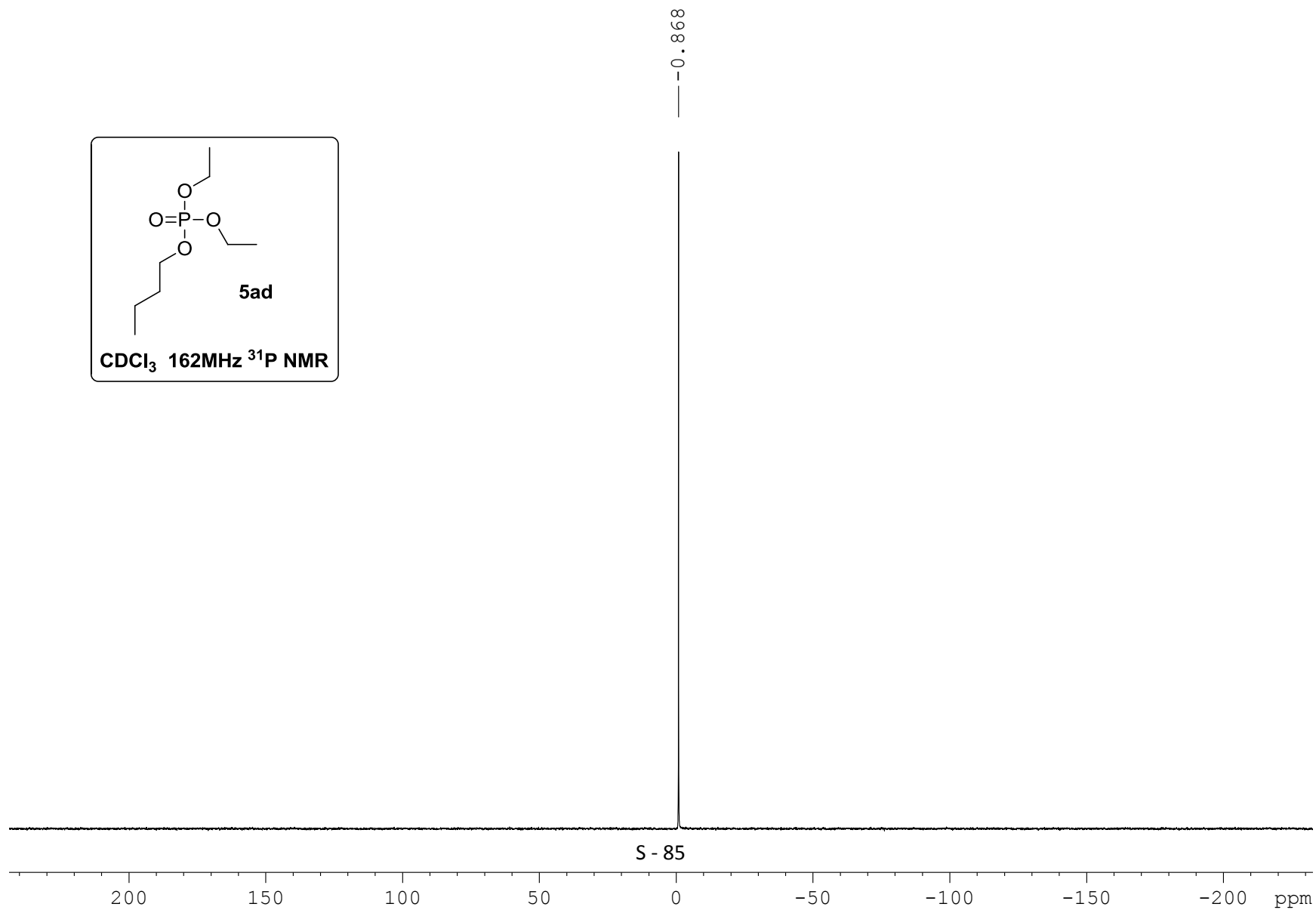
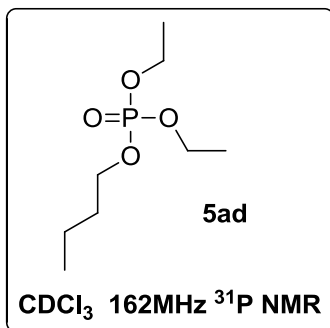


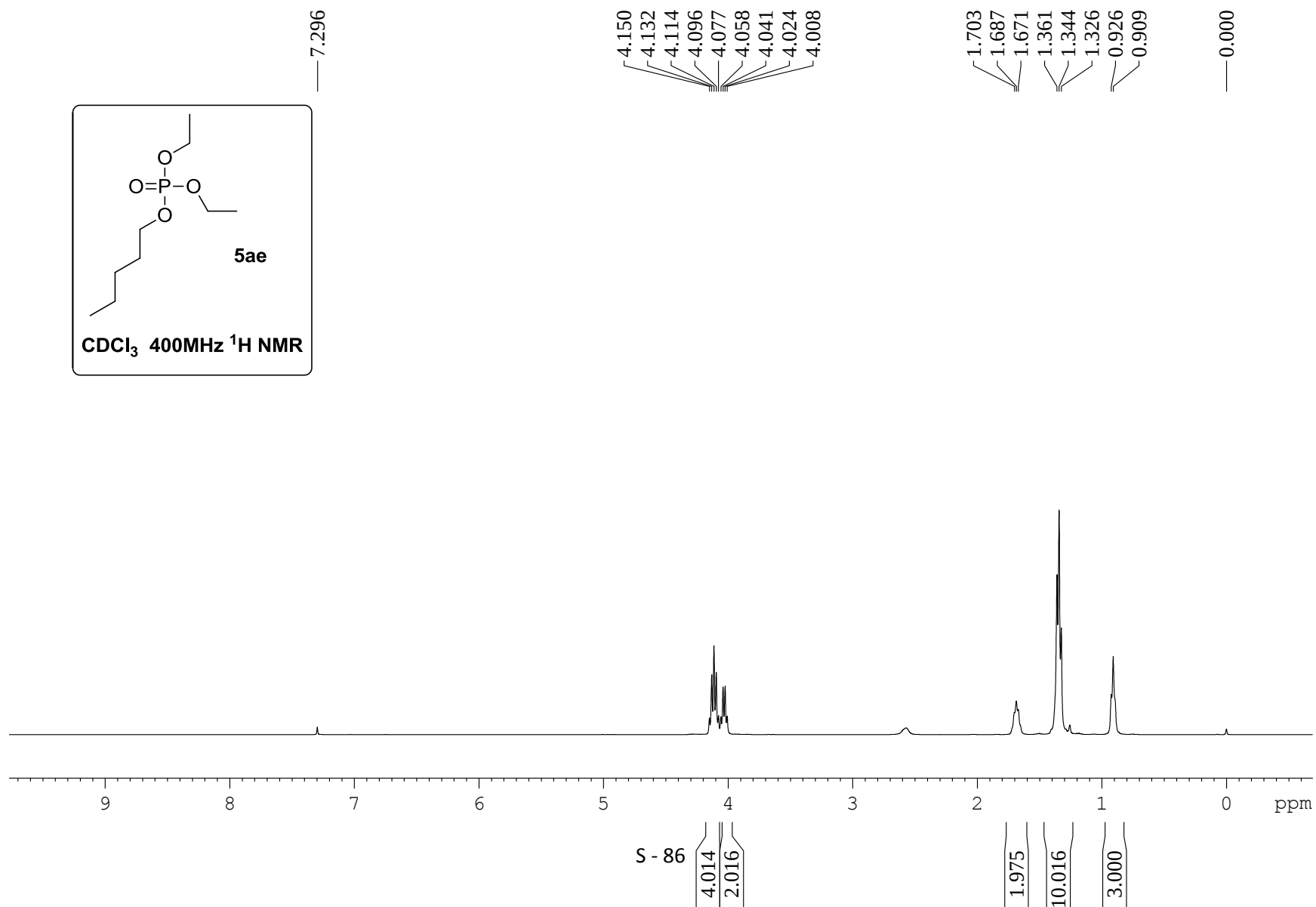


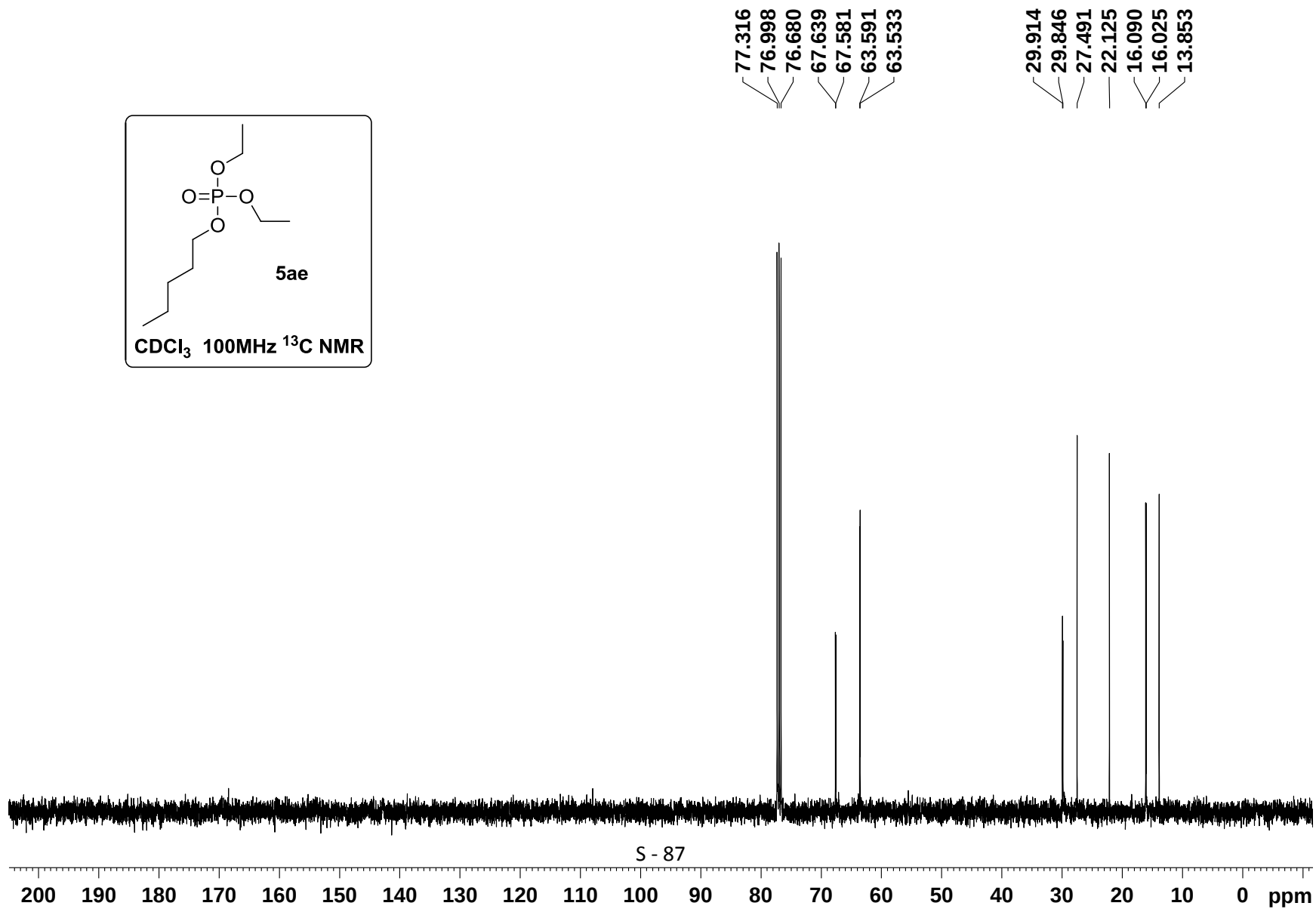
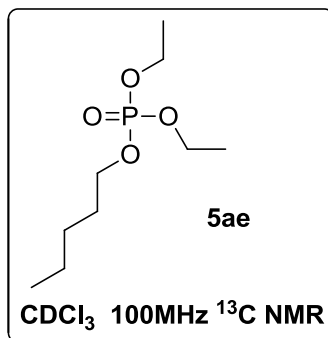


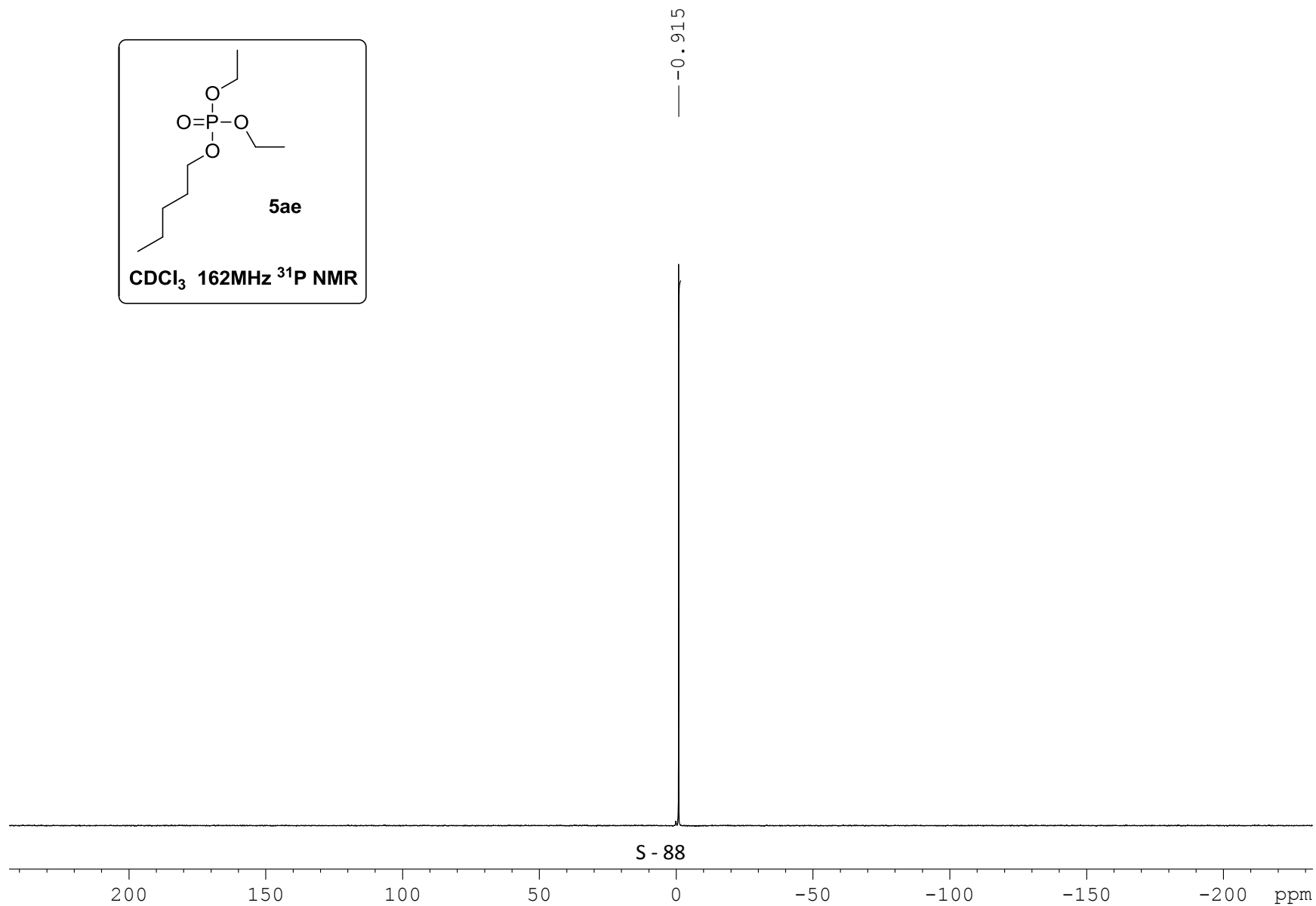
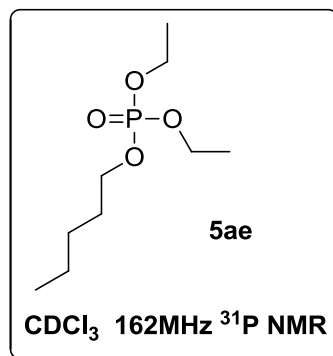


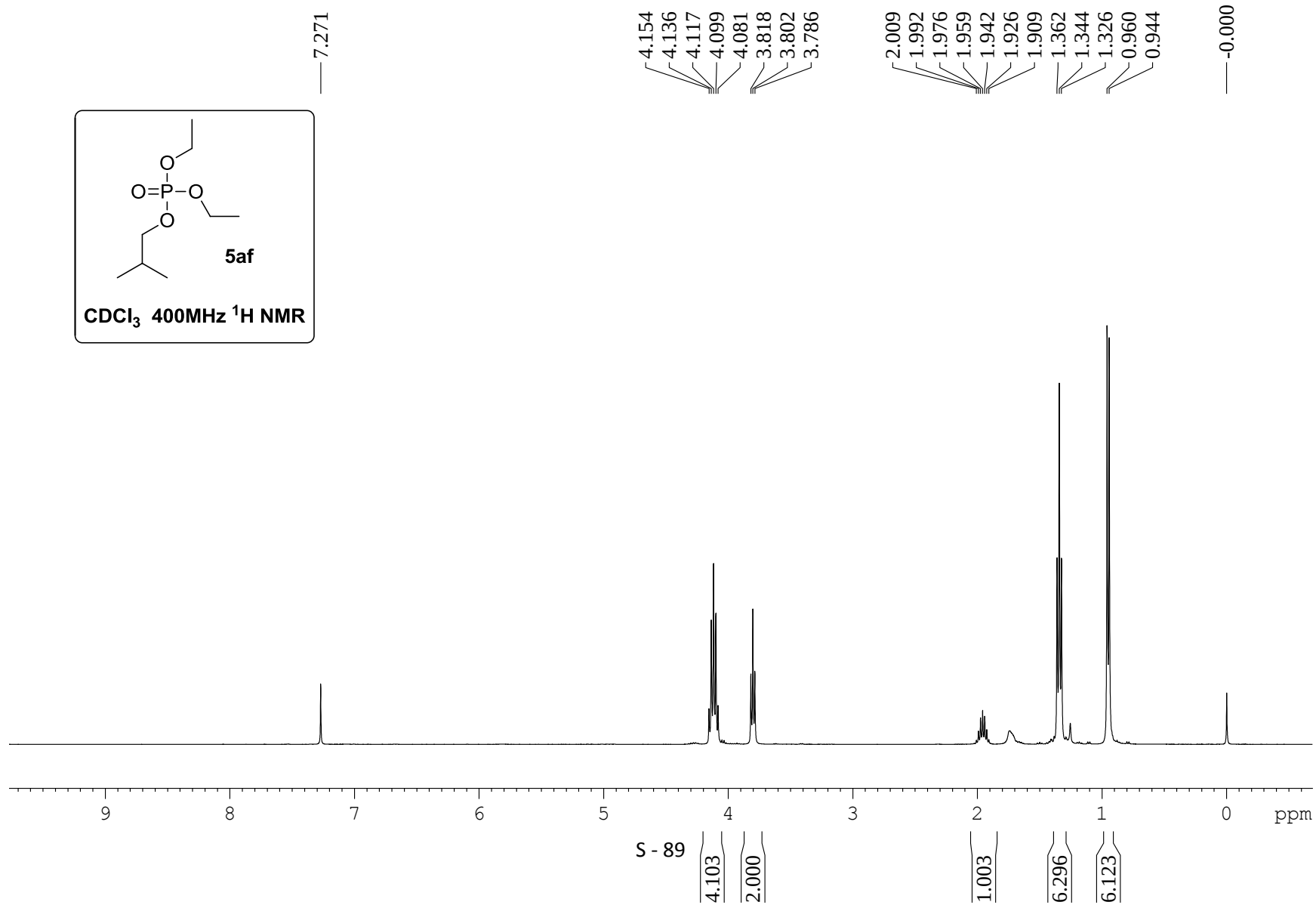


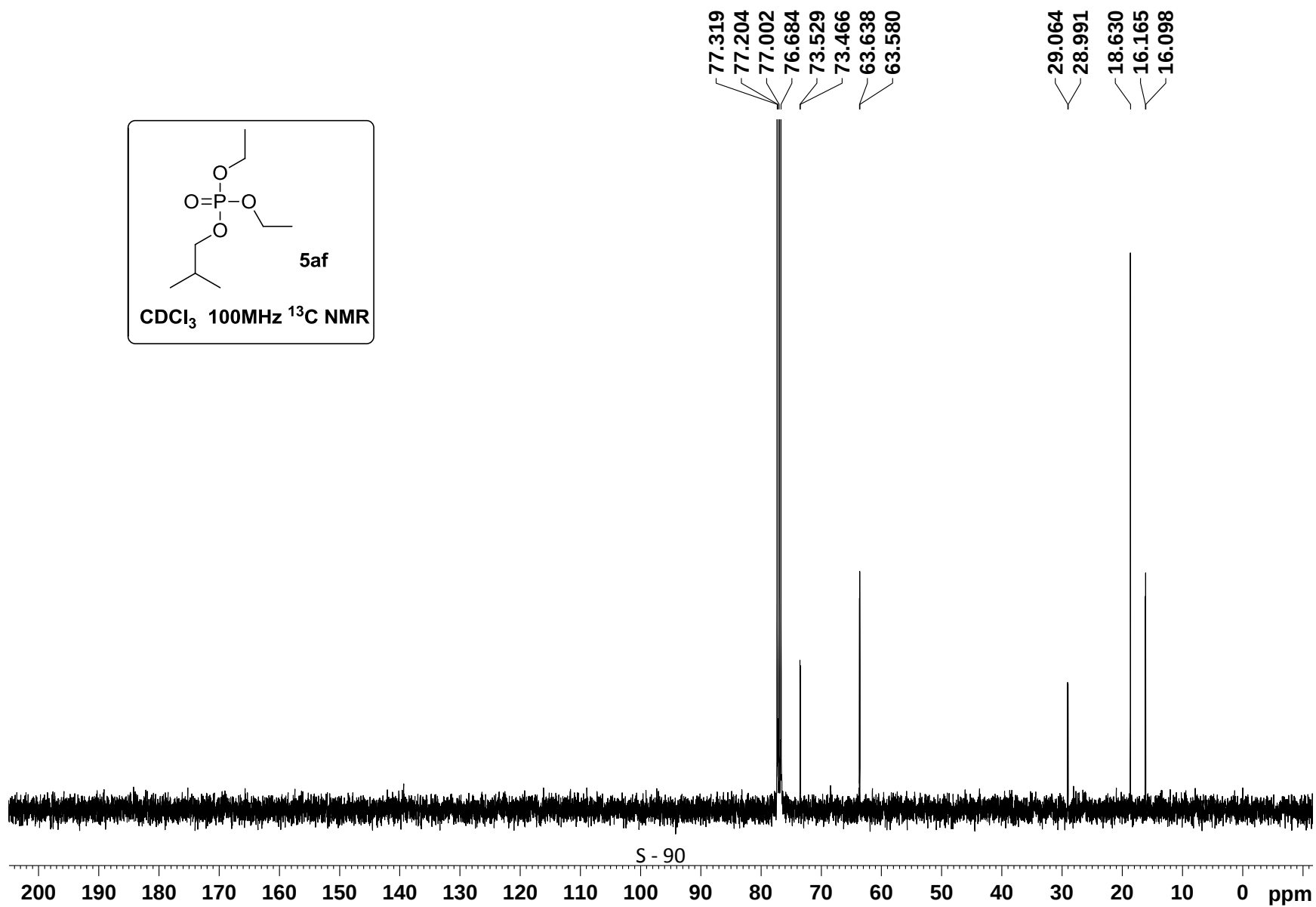
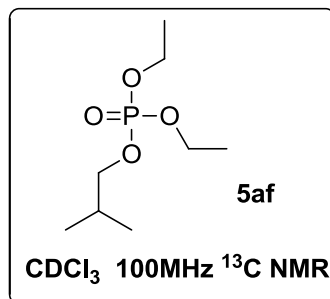


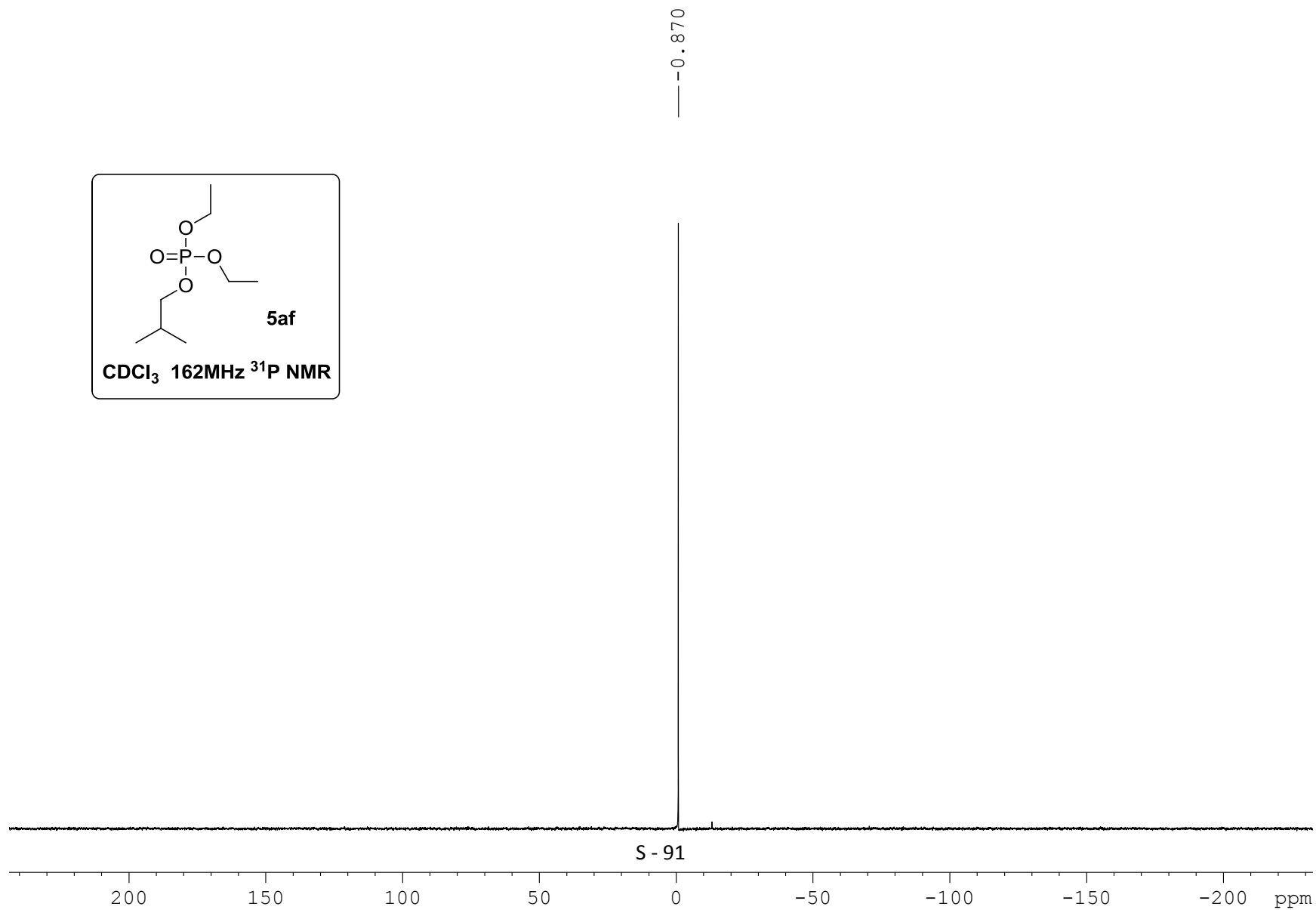
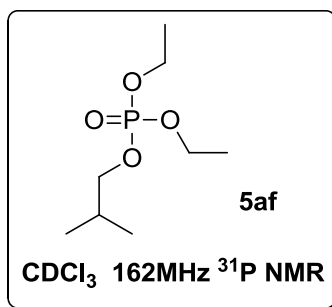


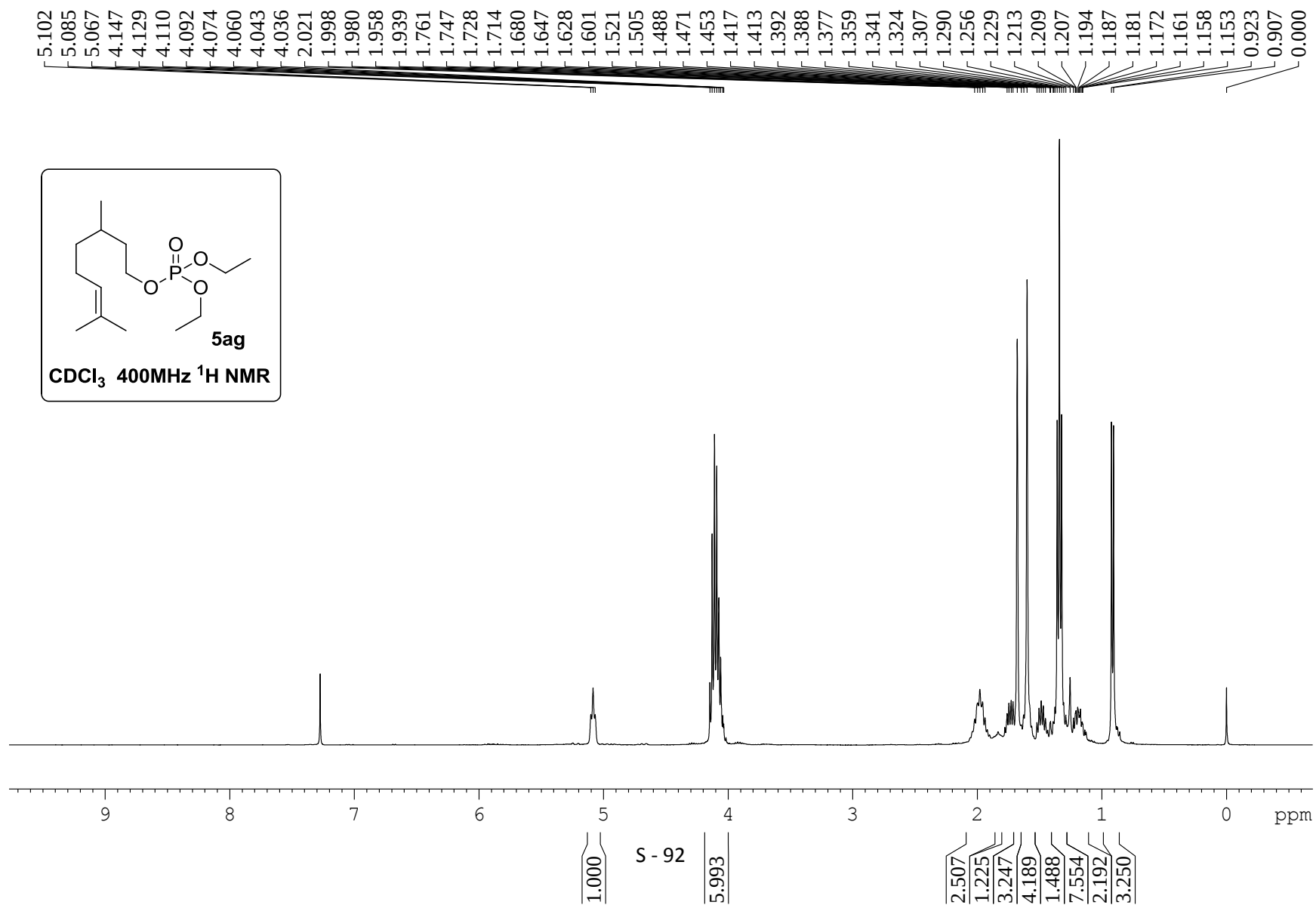


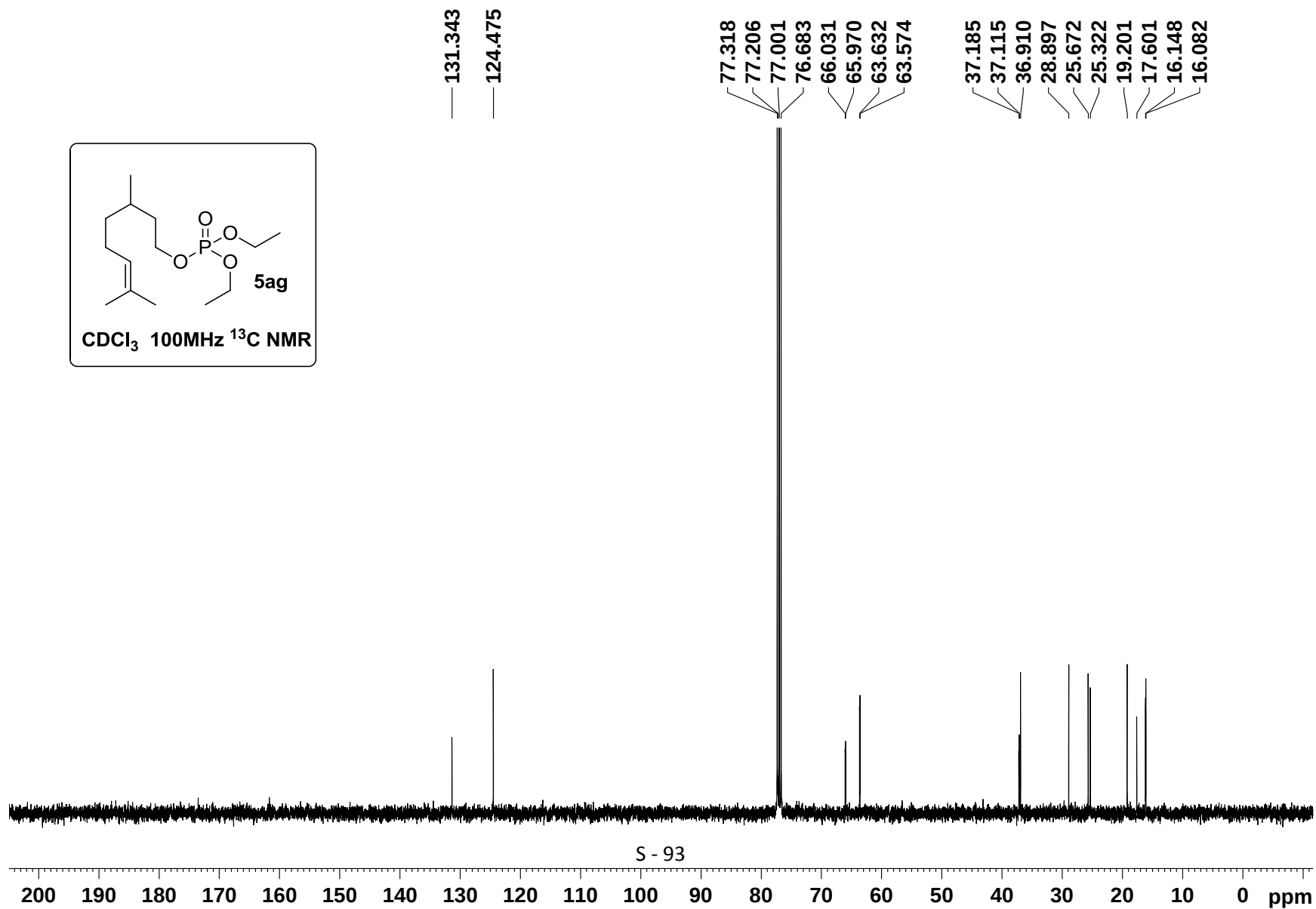
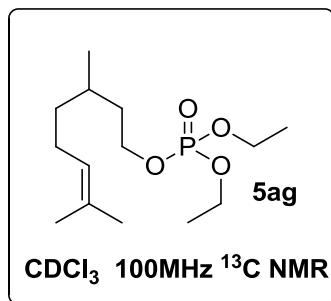


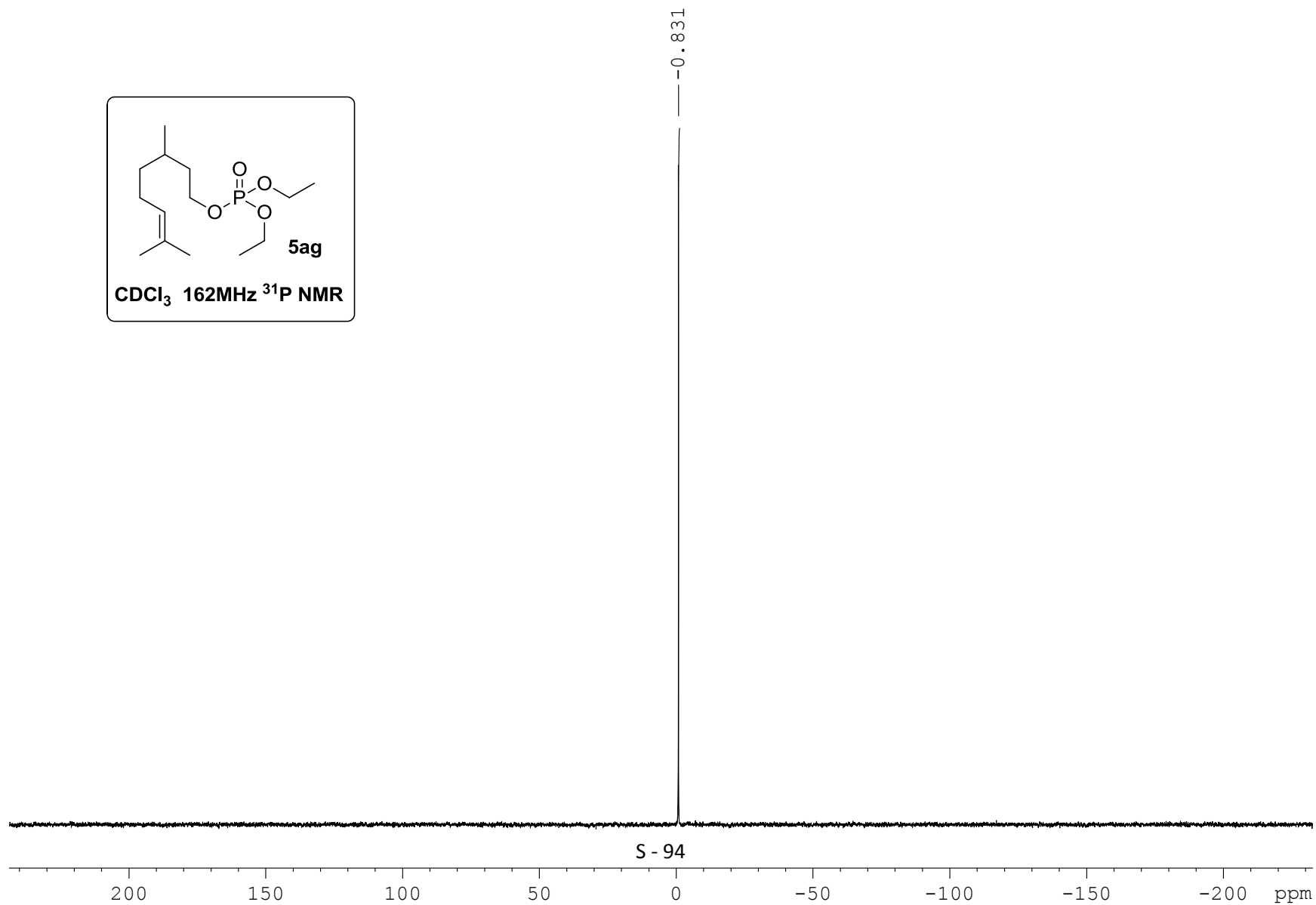
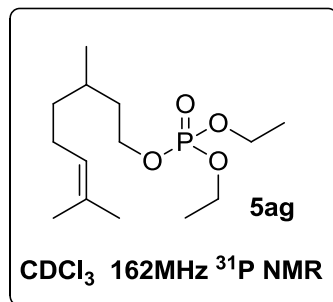


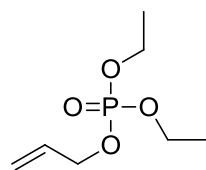




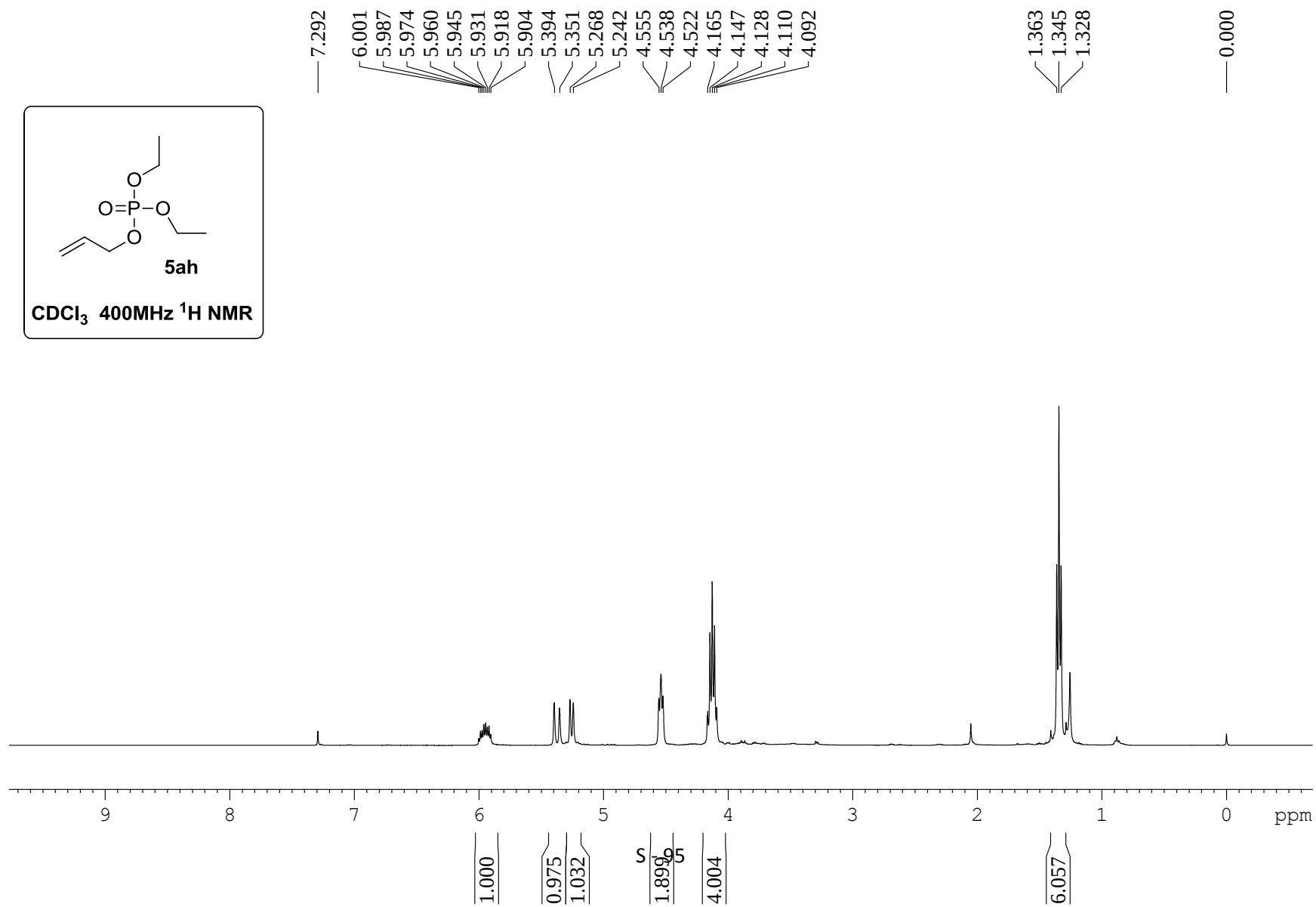


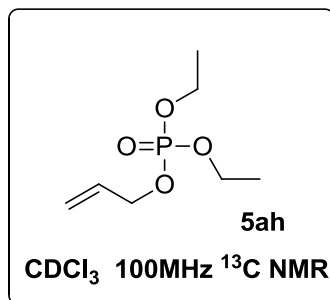






5ah

 CDCl_3 400MHz ^1H NMR

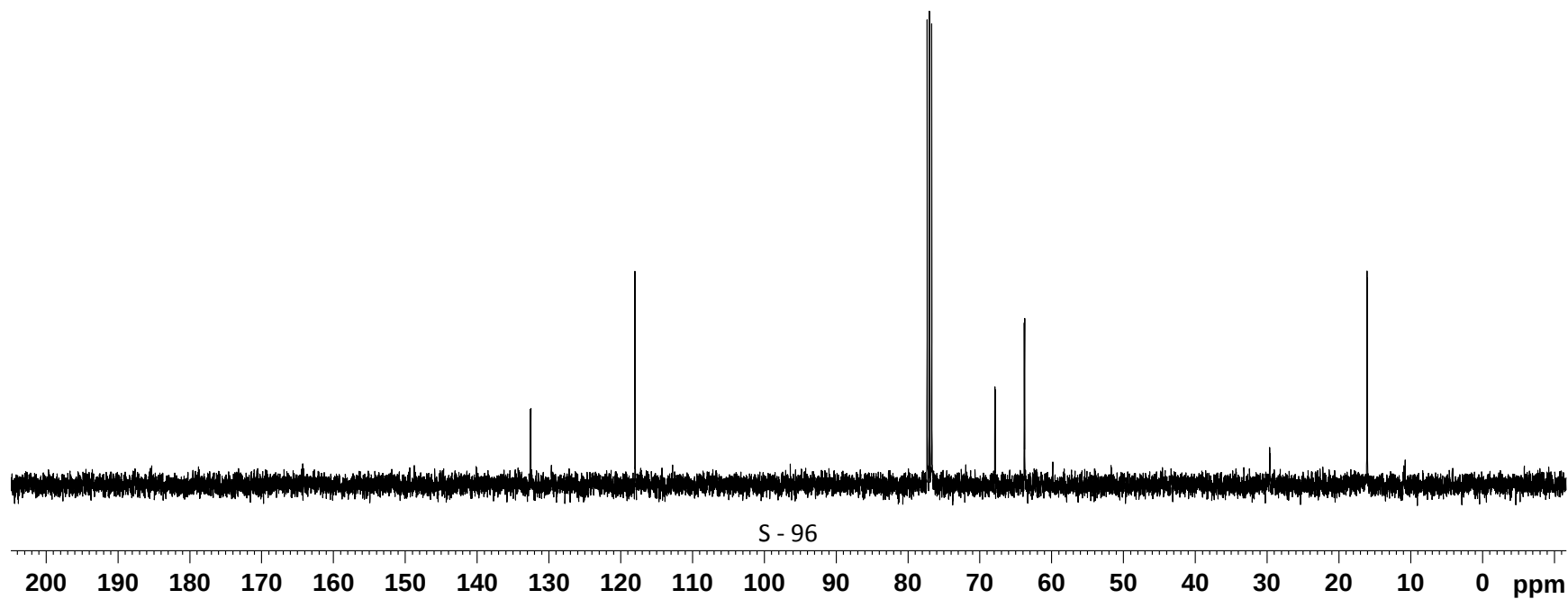


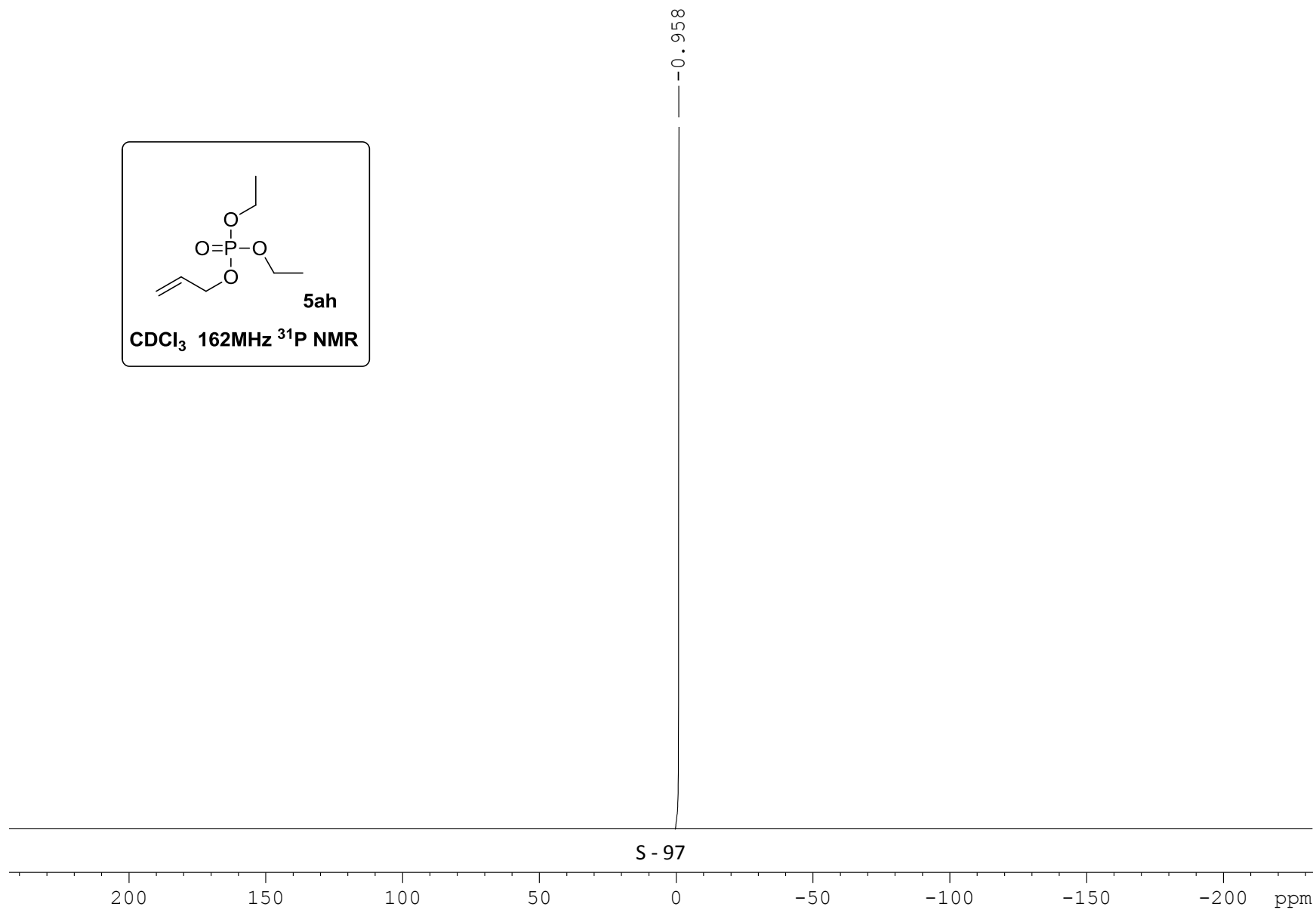
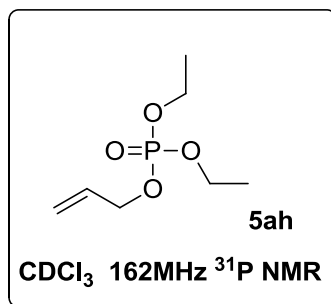
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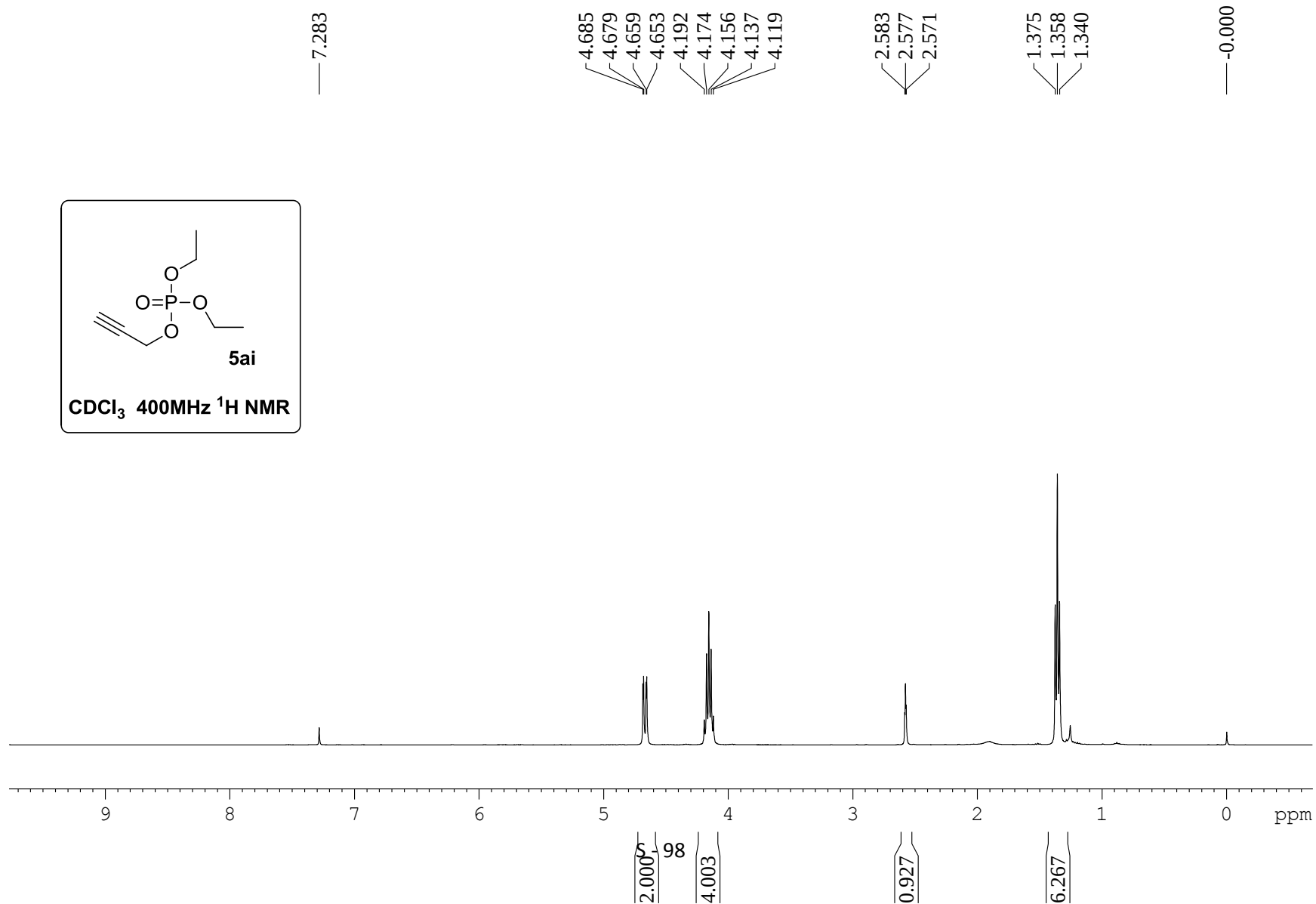
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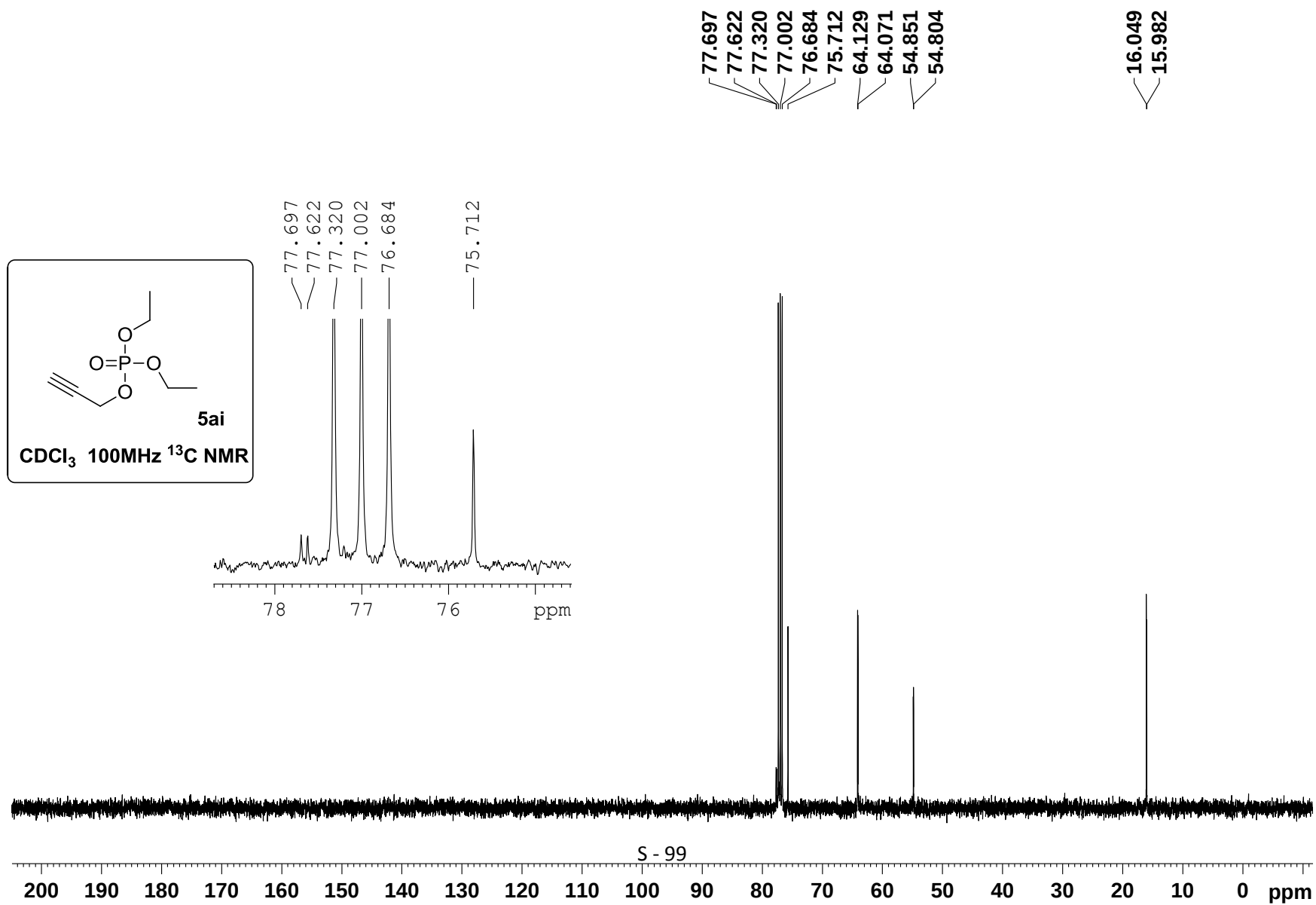
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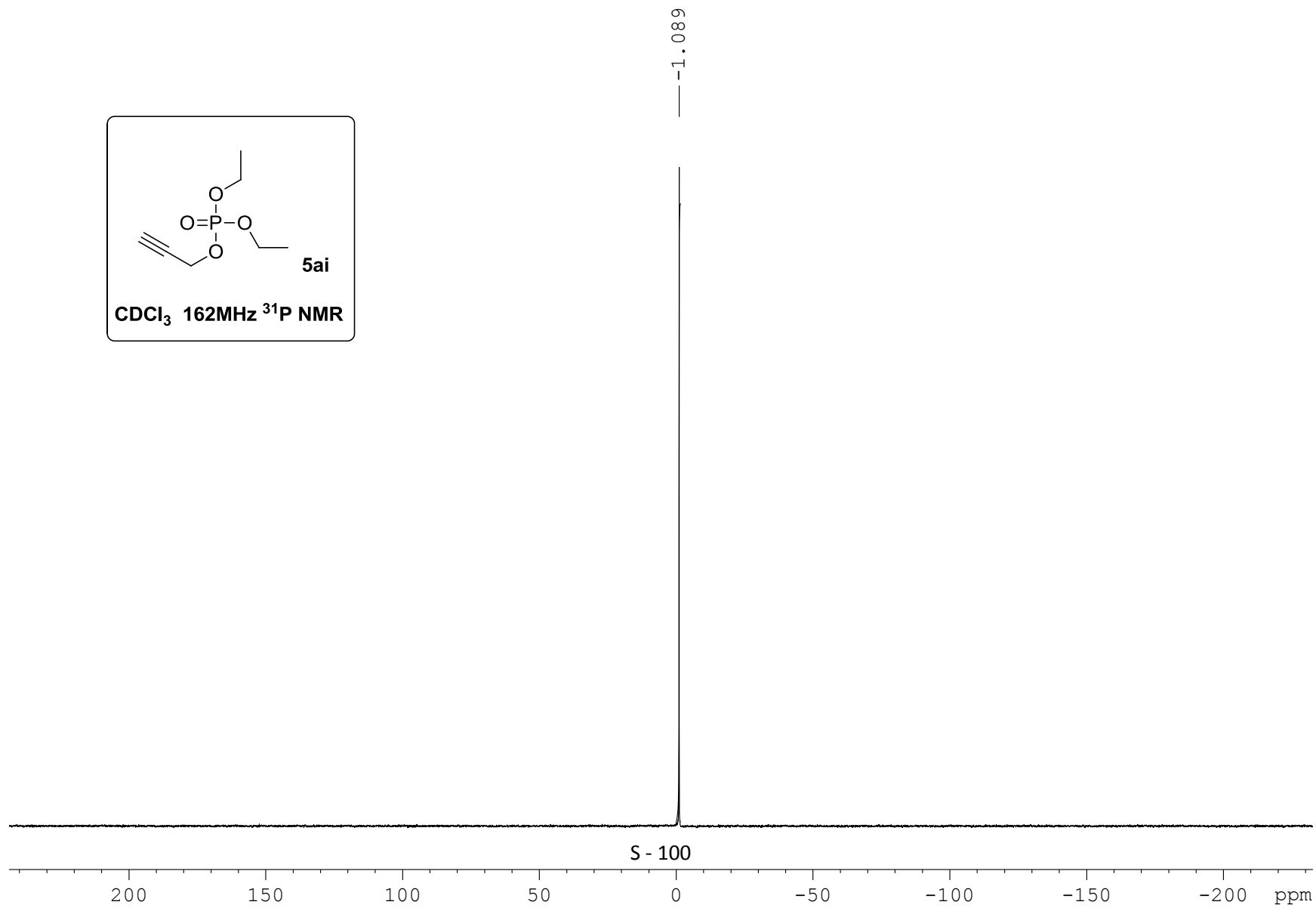
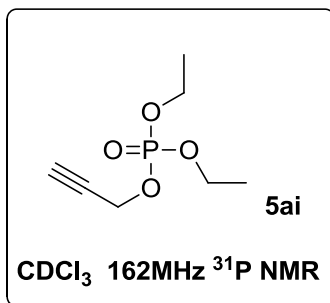
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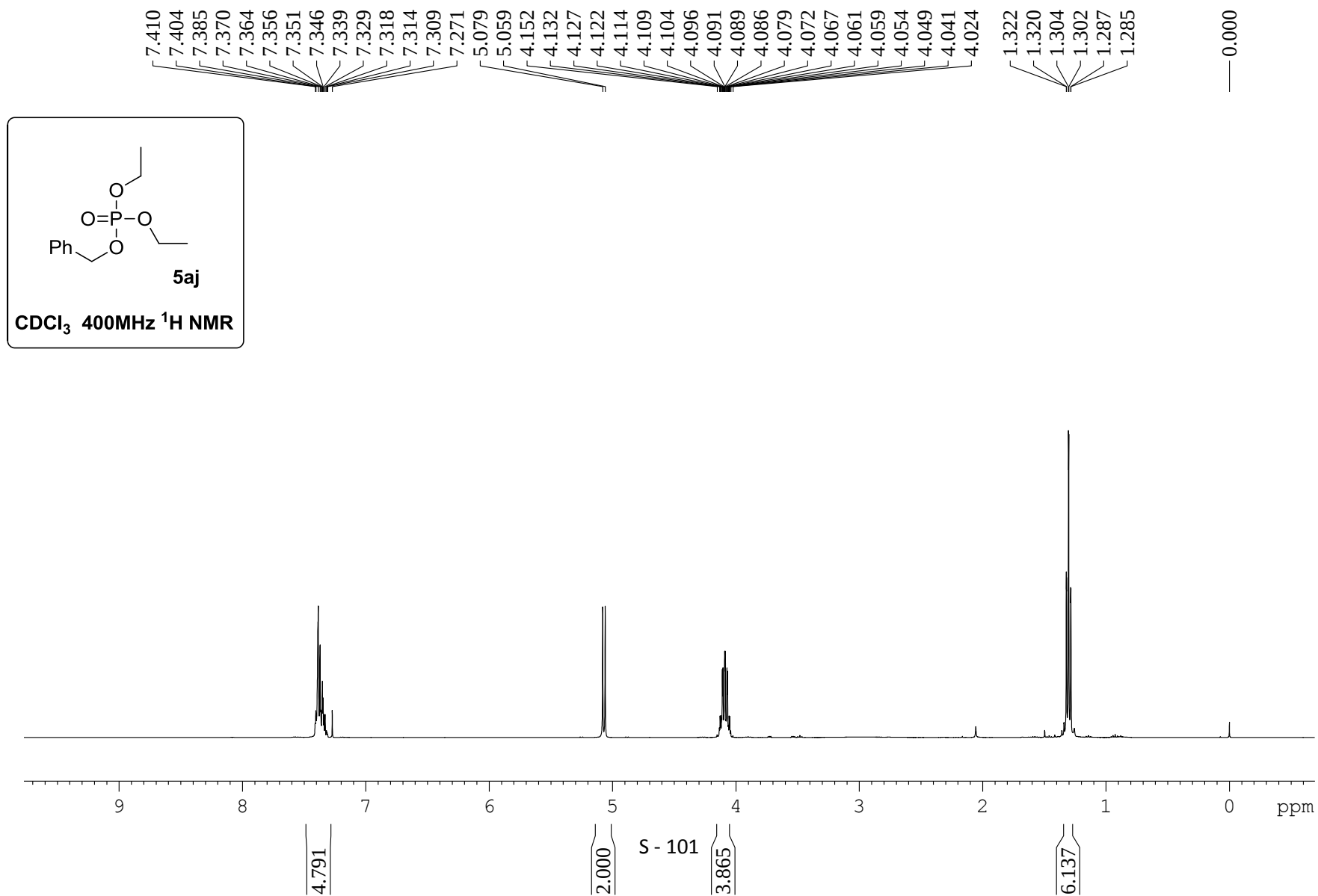


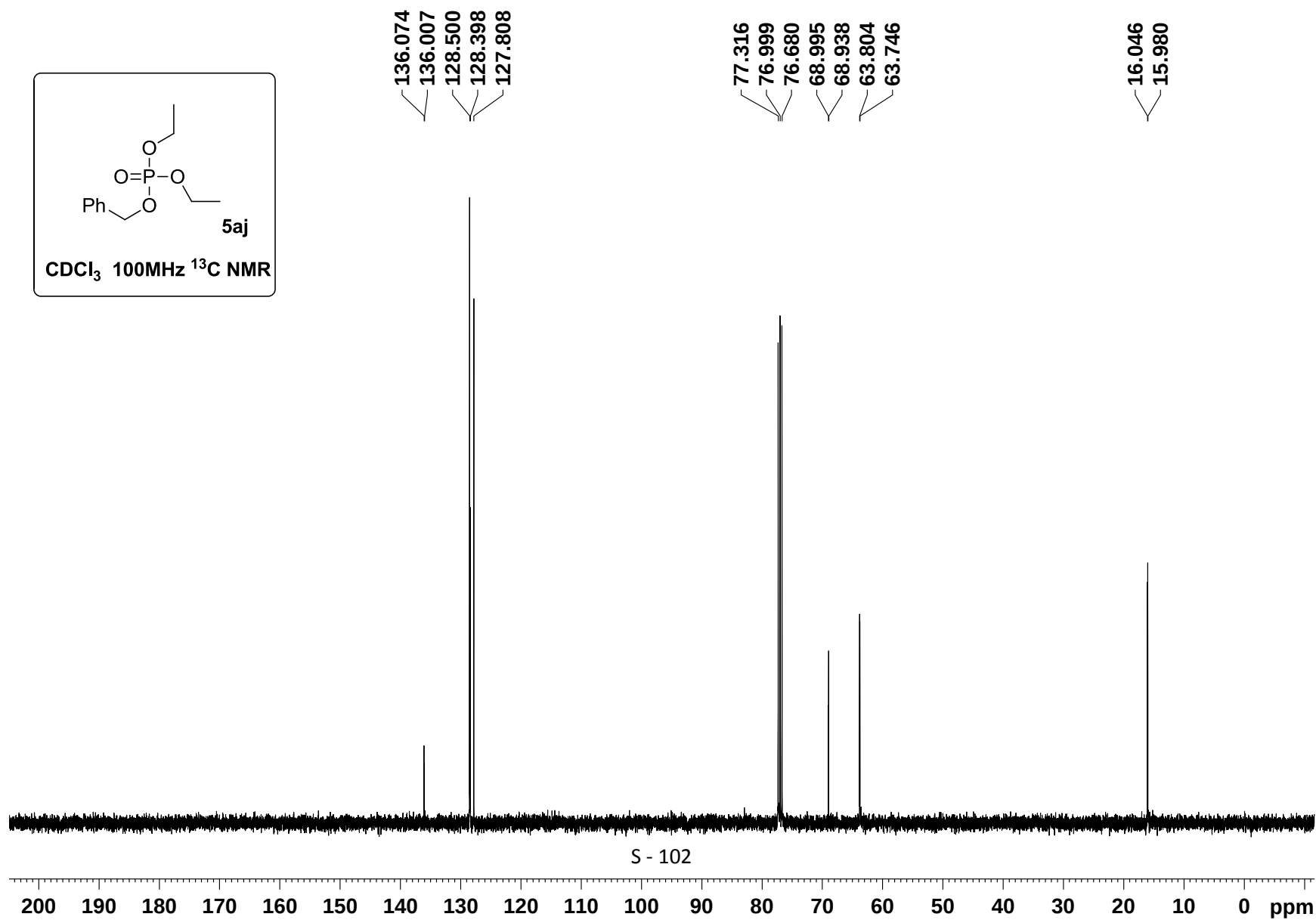
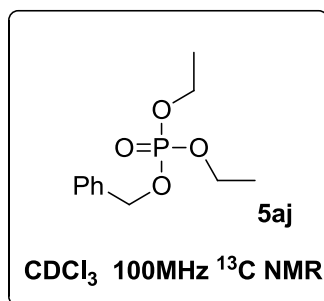


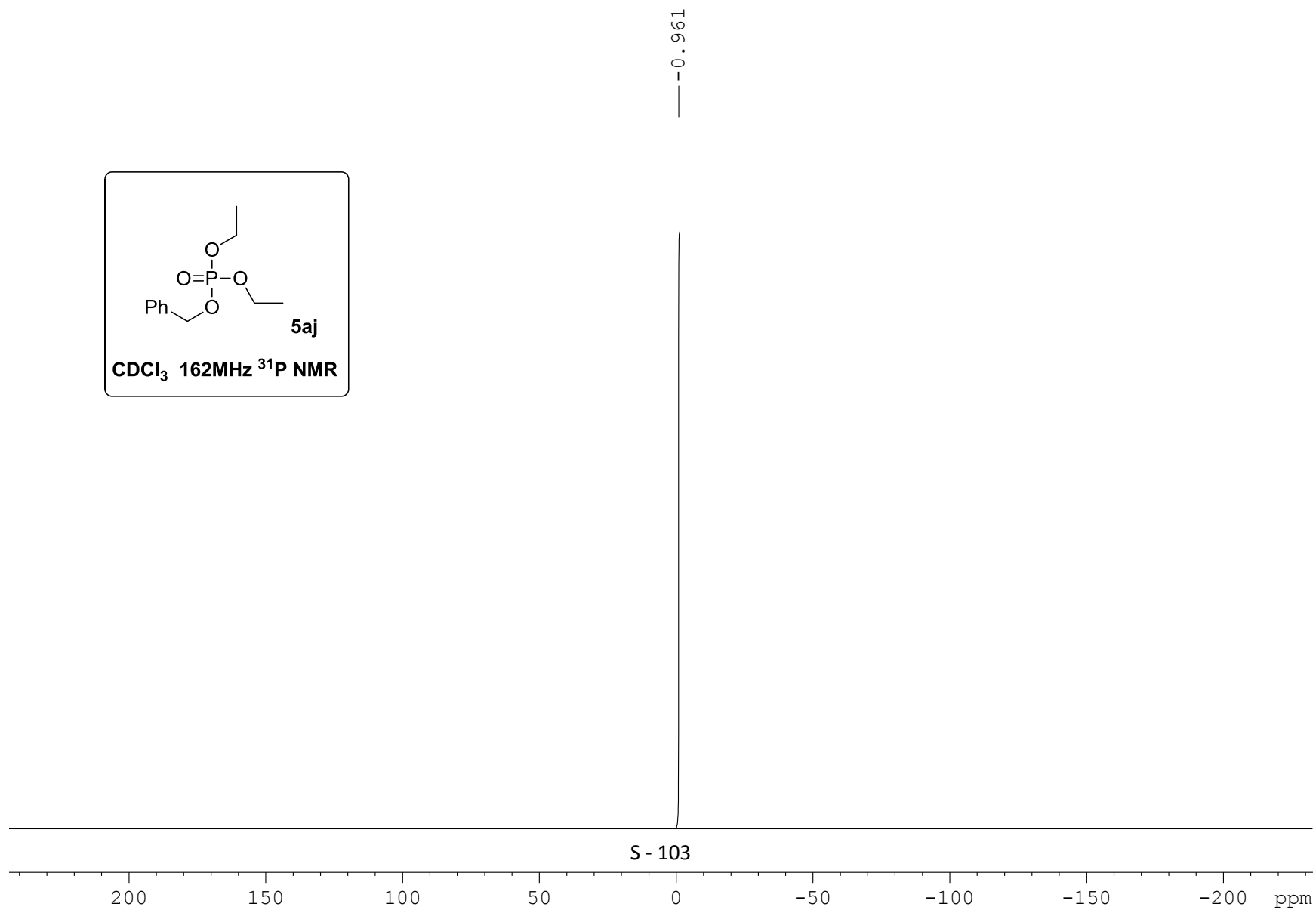
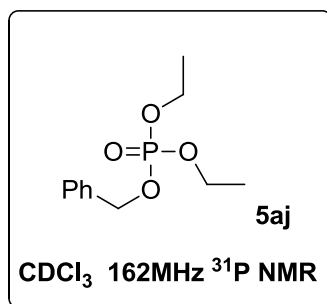


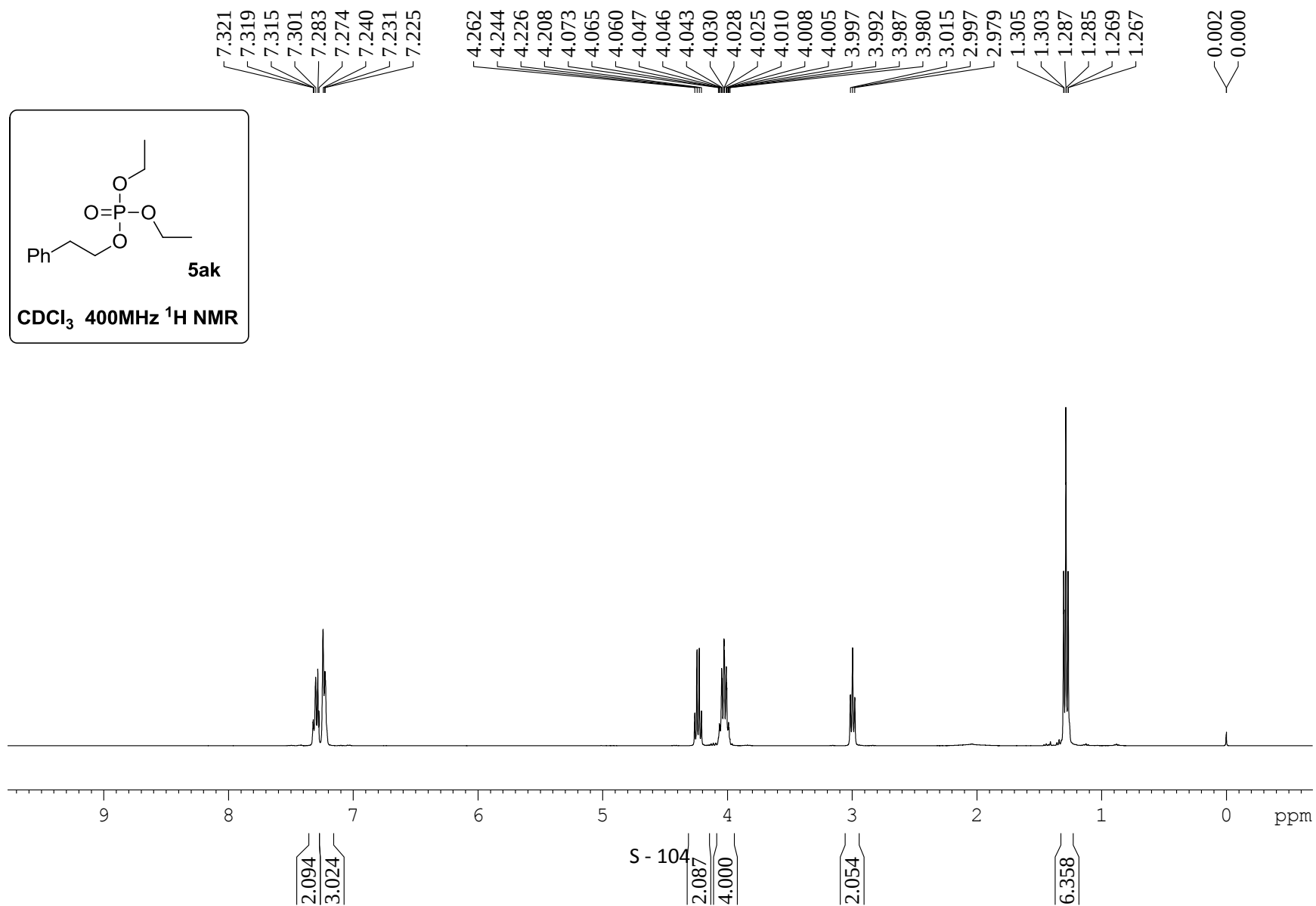


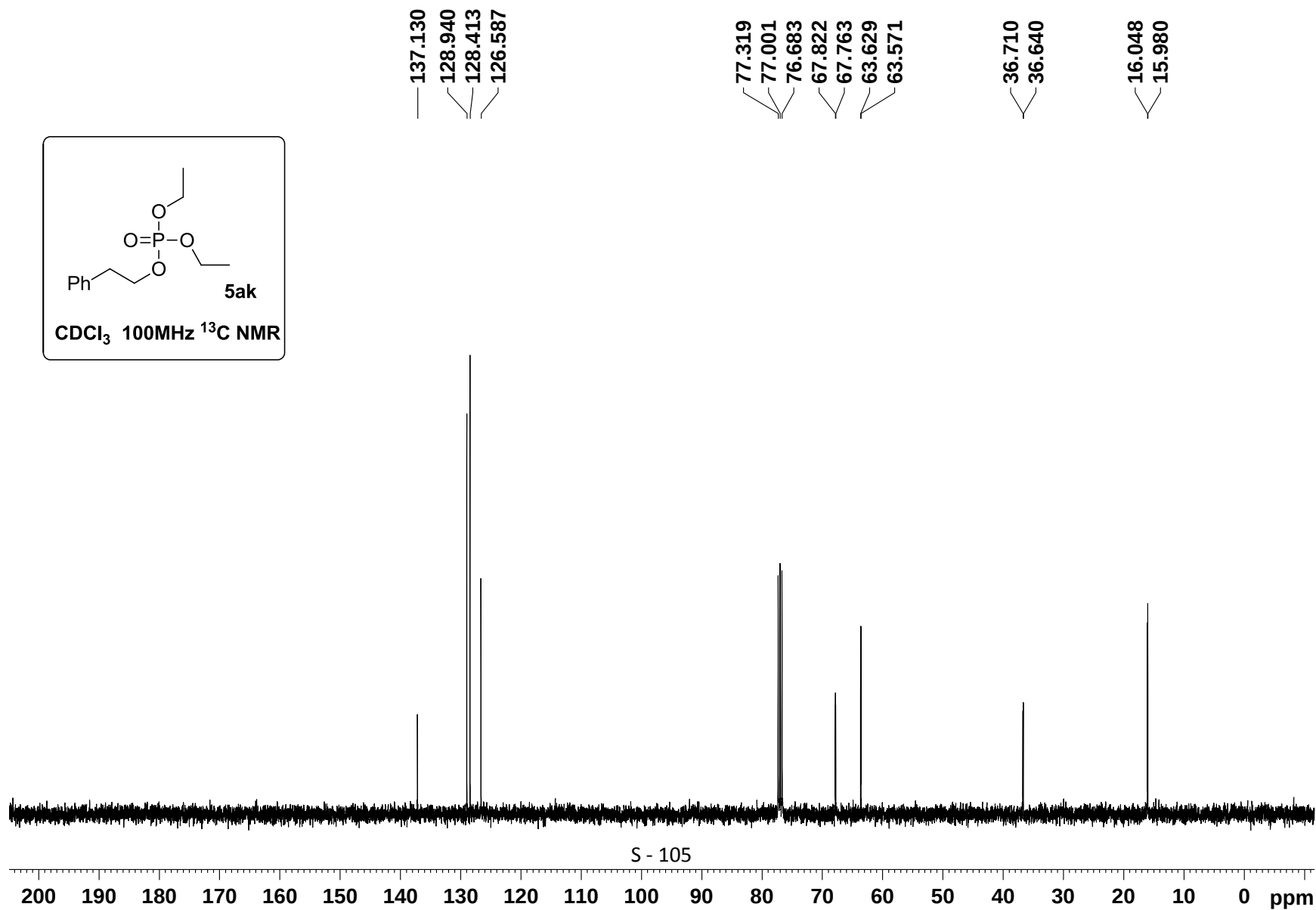
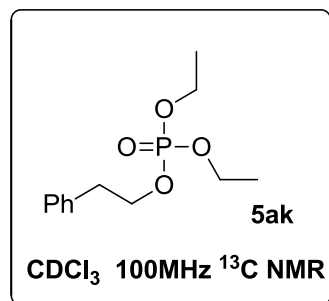


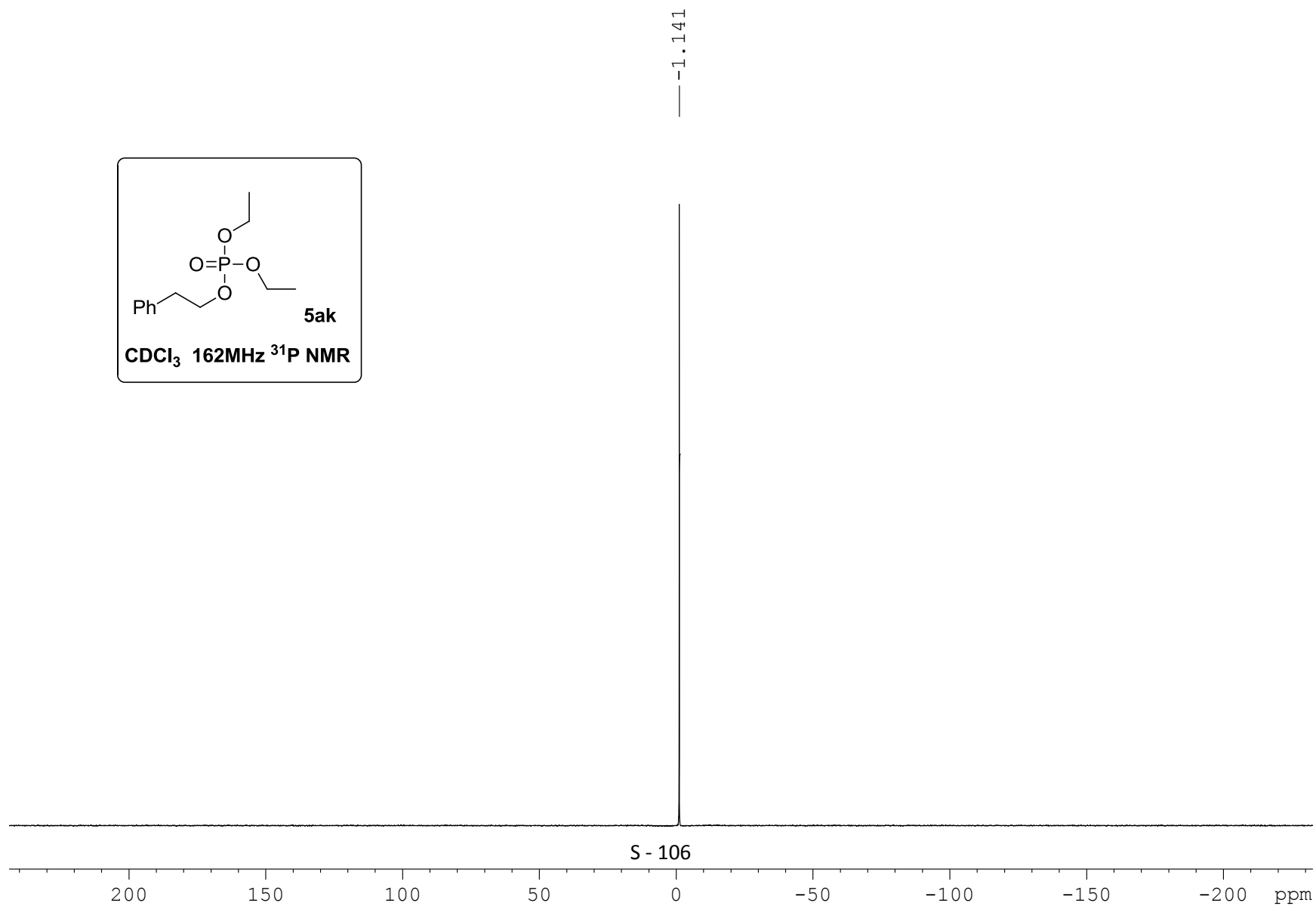
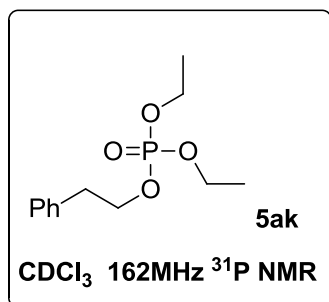


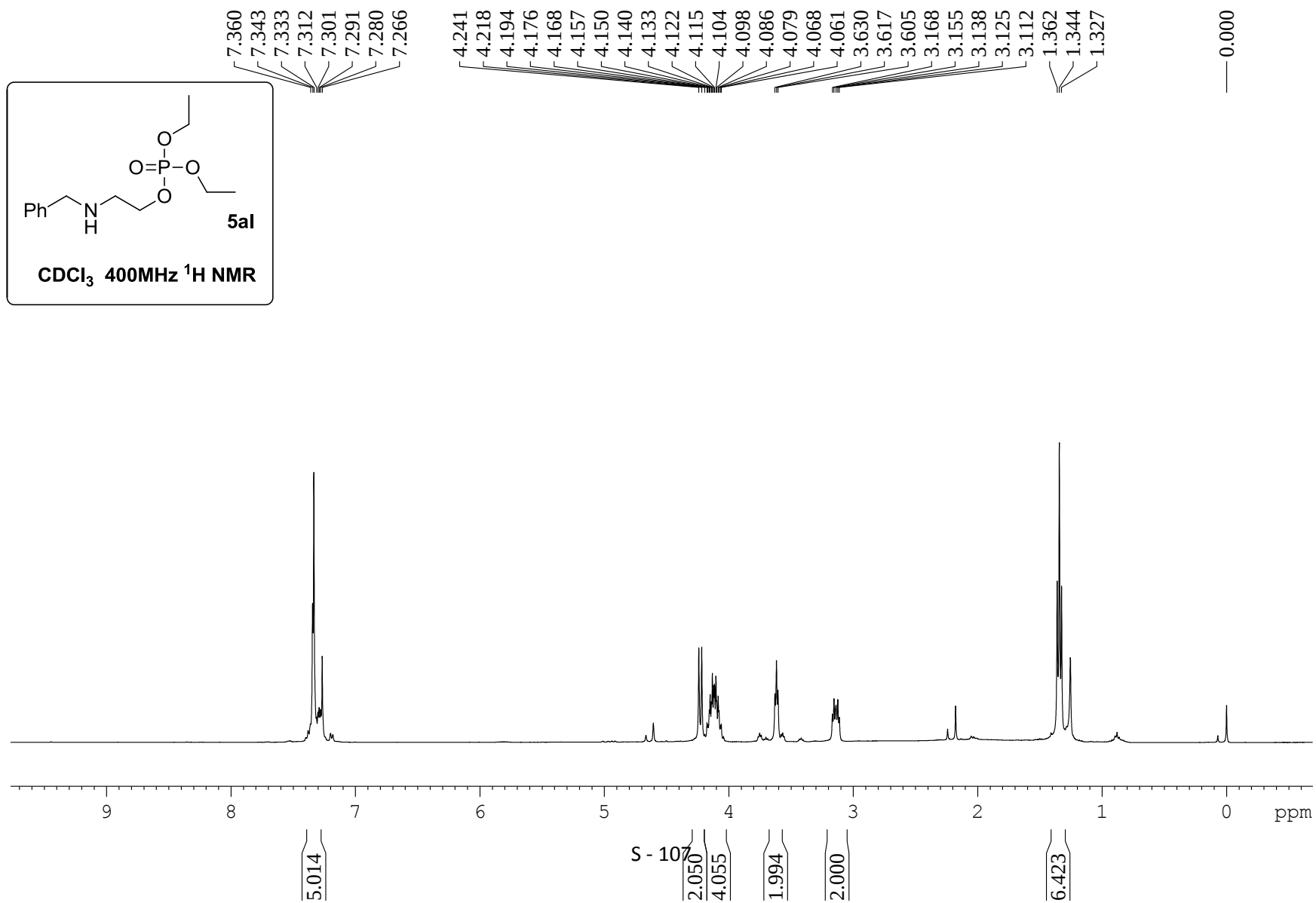


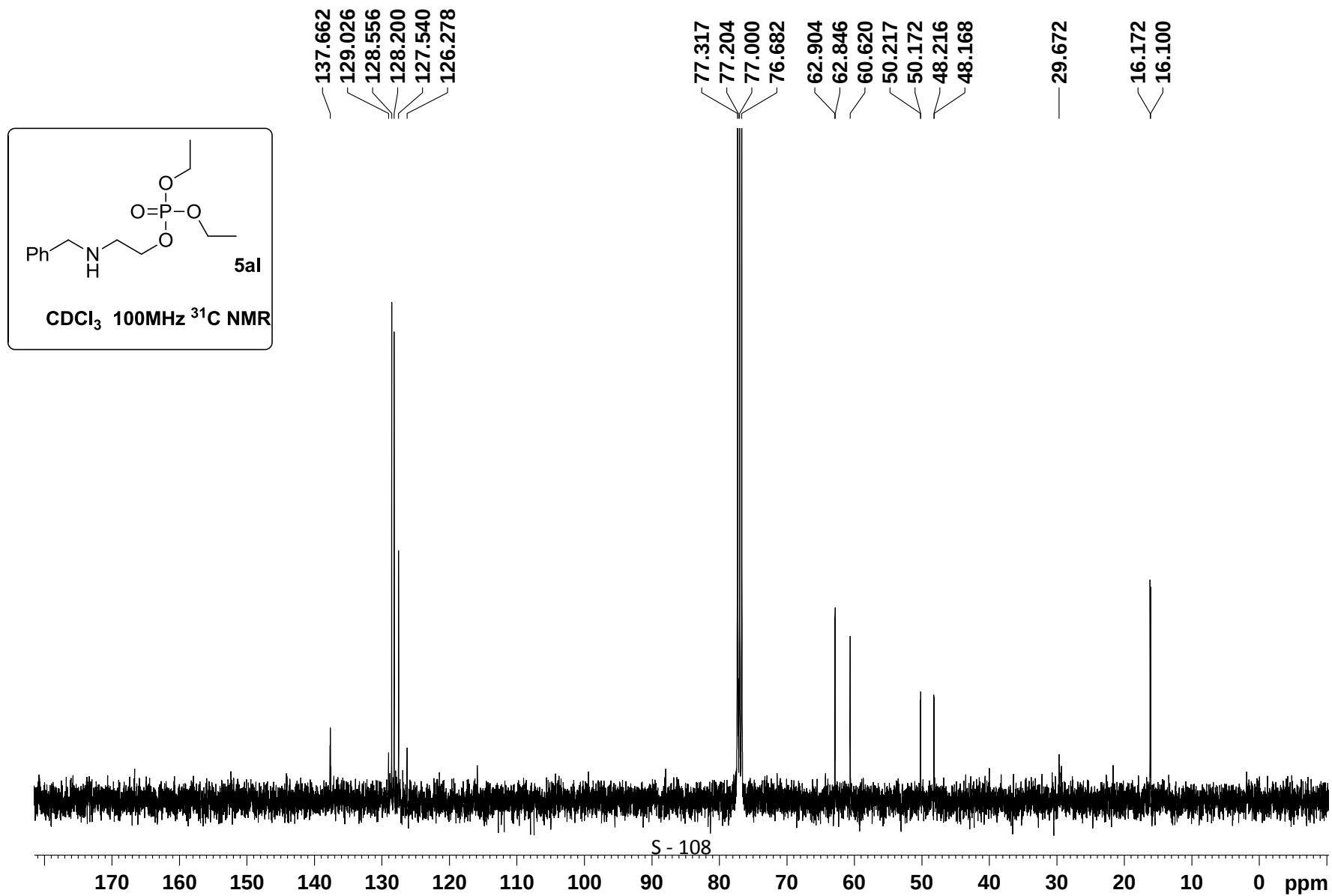


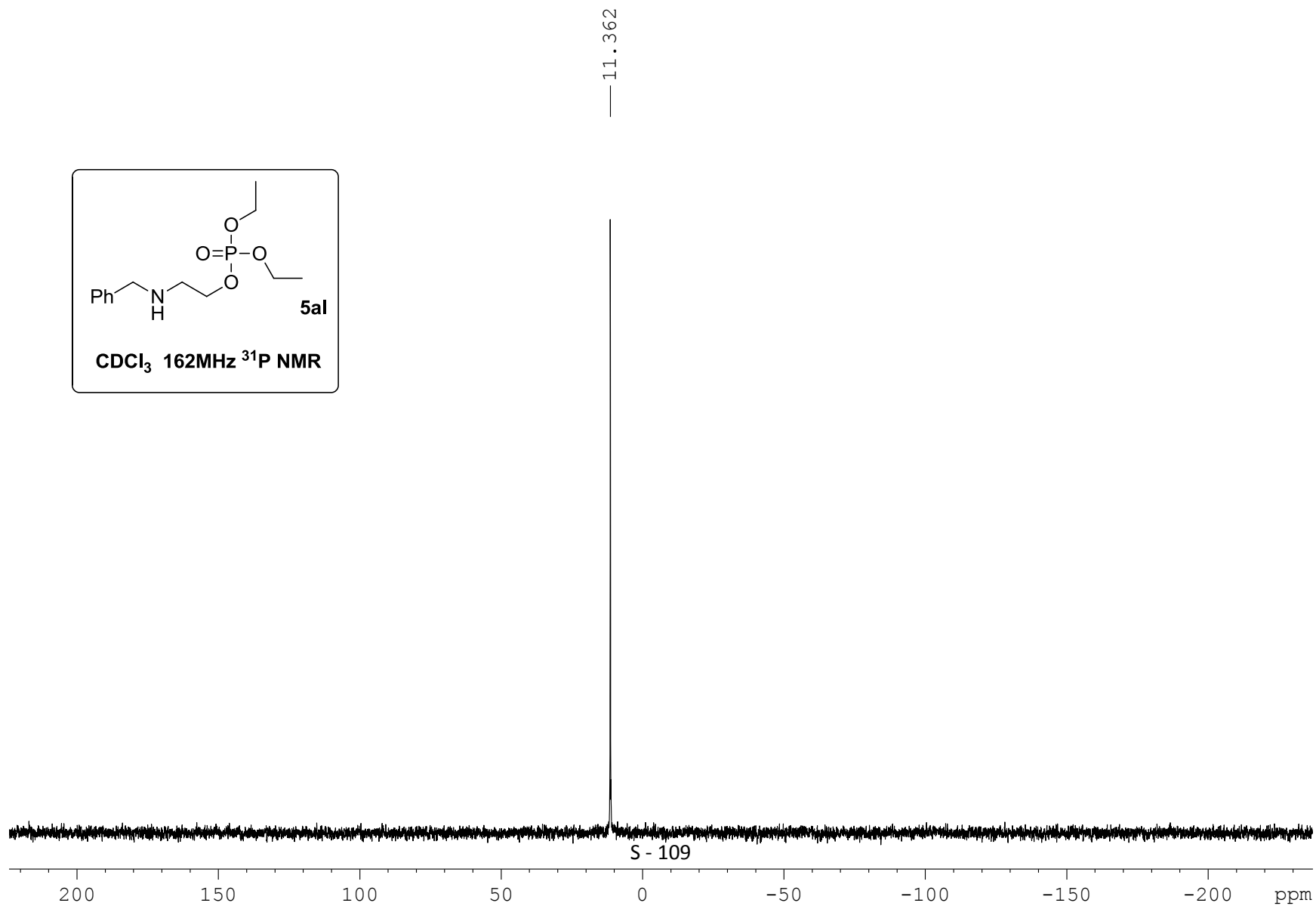
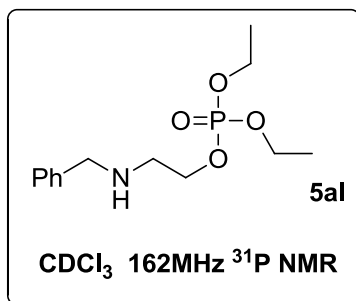


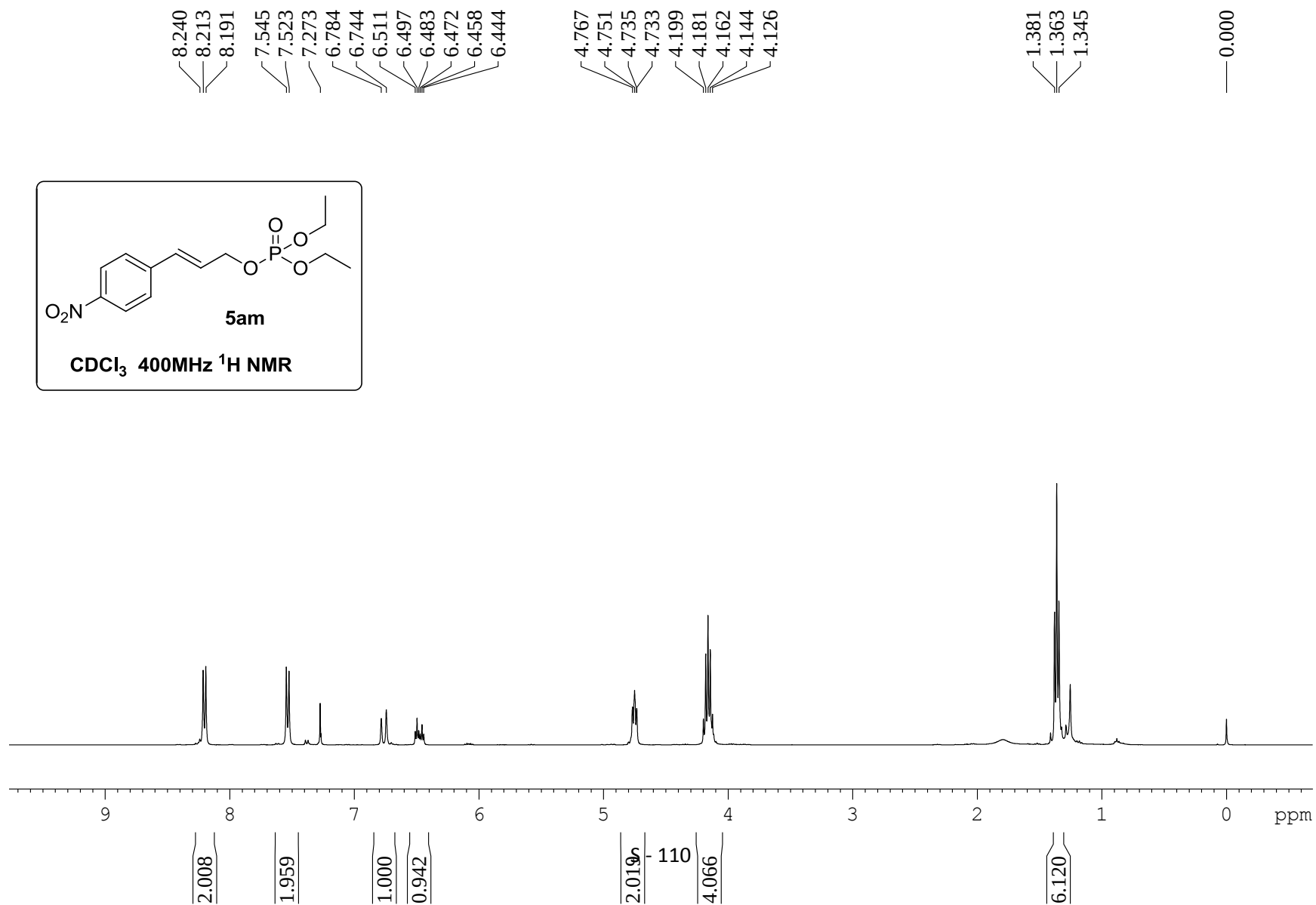
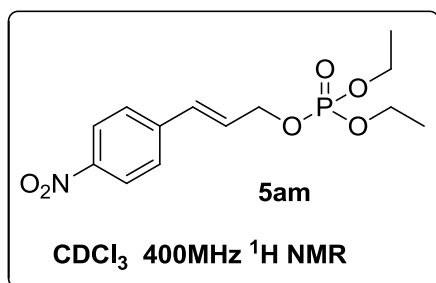


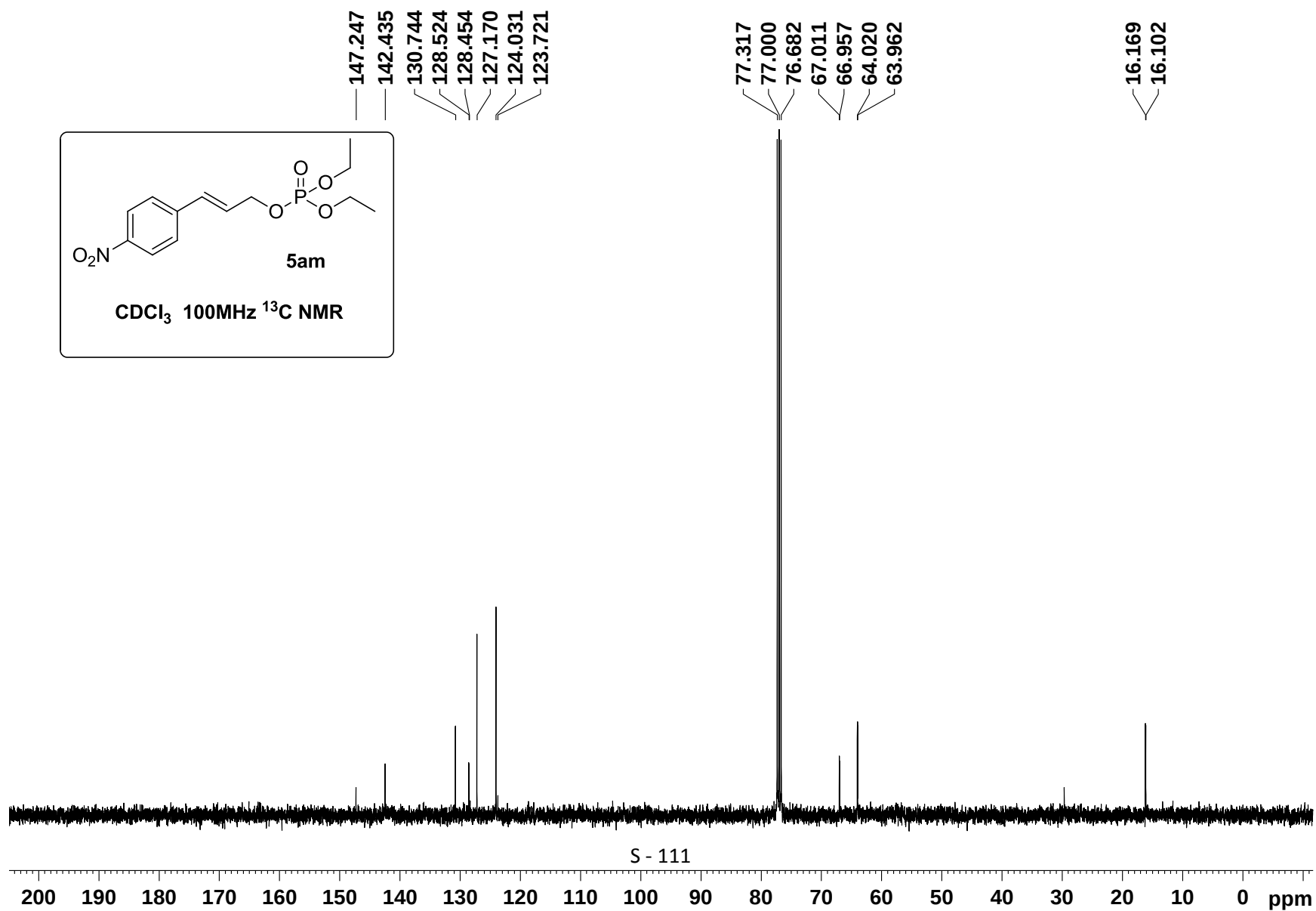


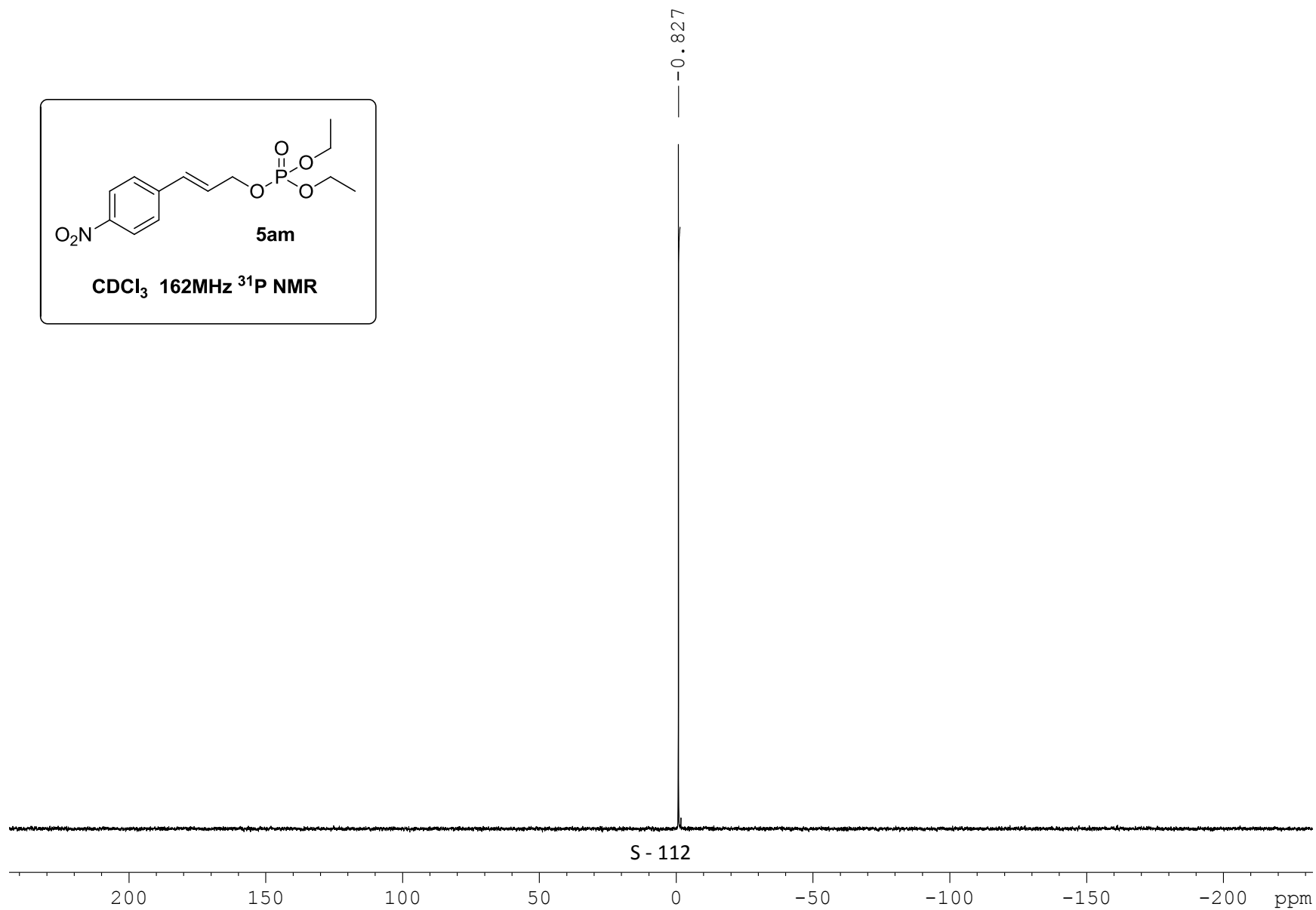
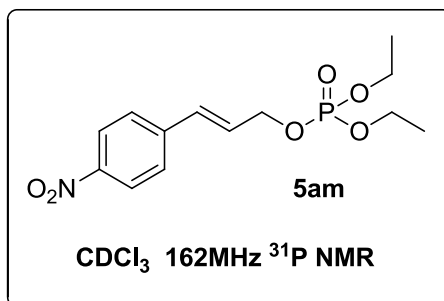


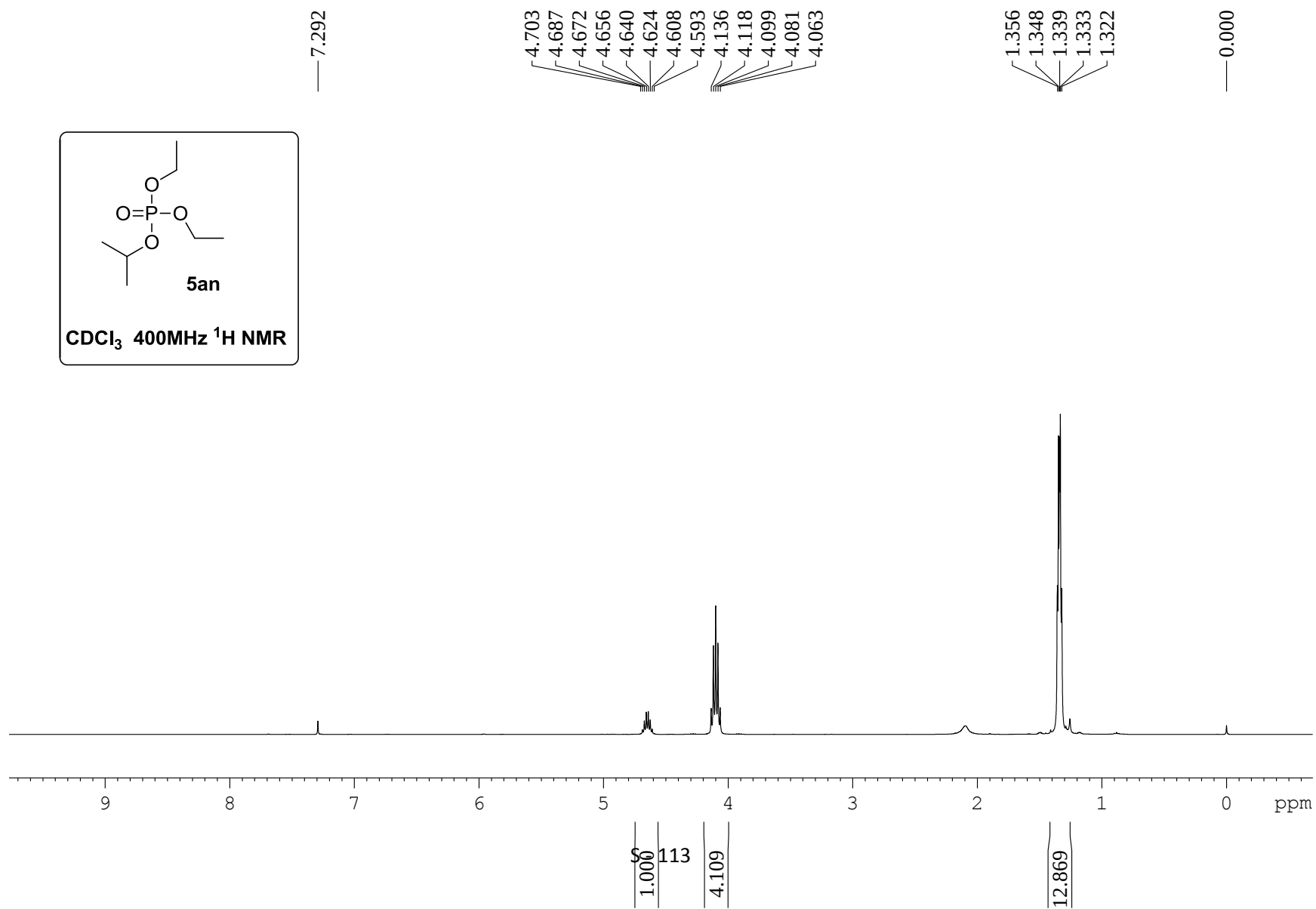


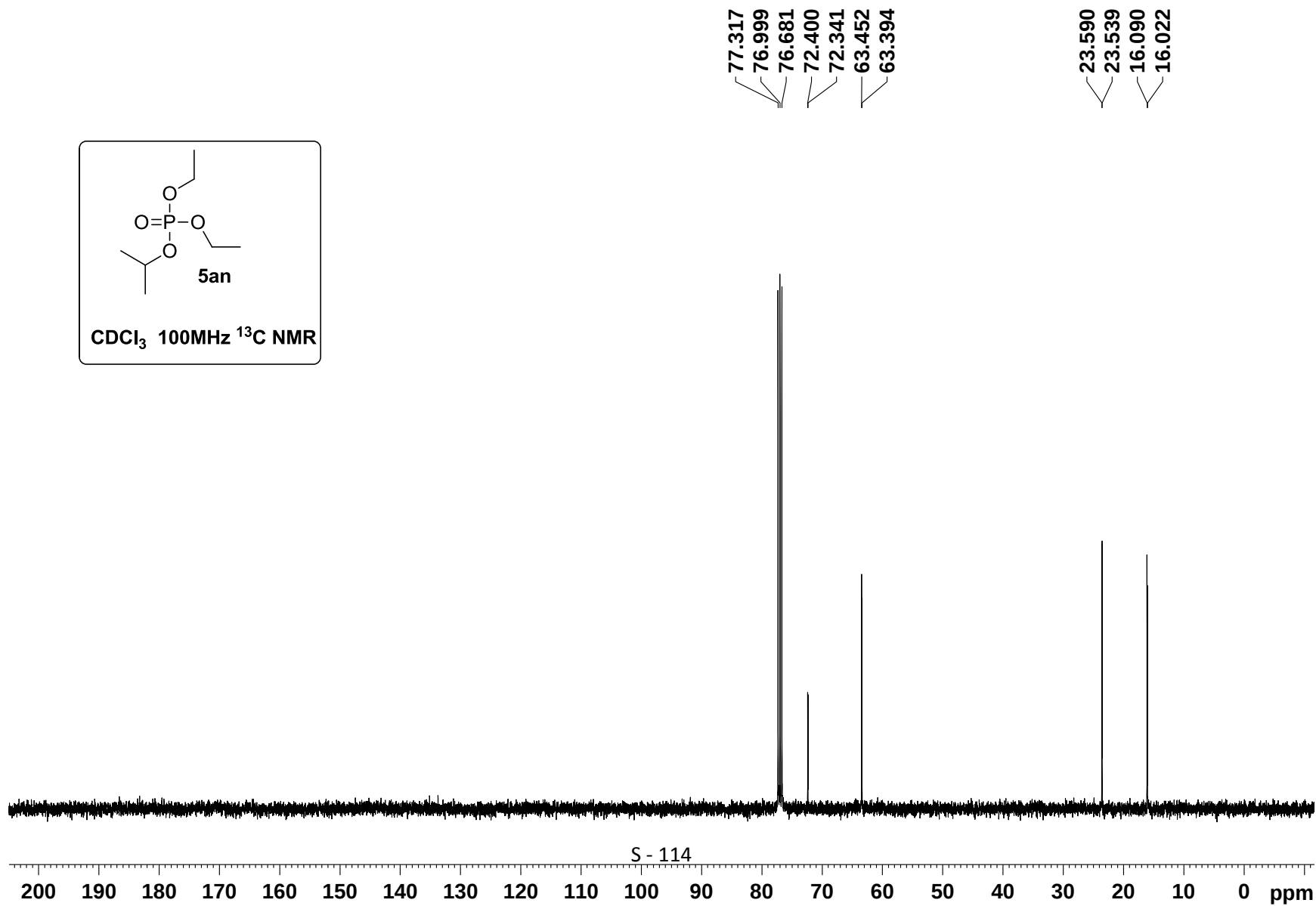
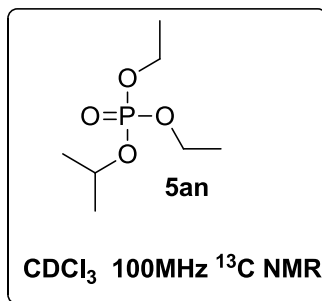


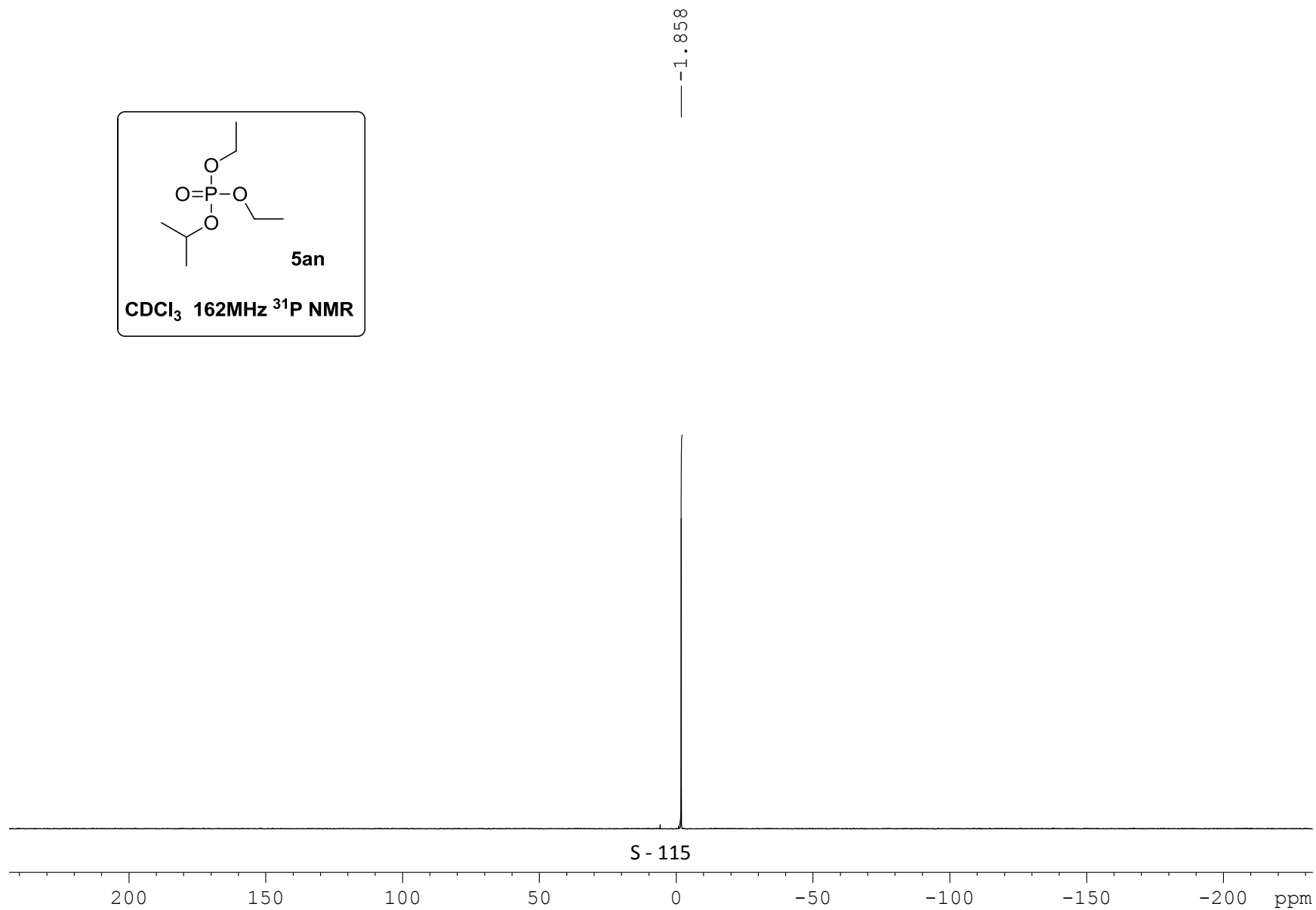
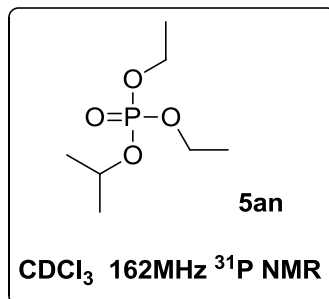


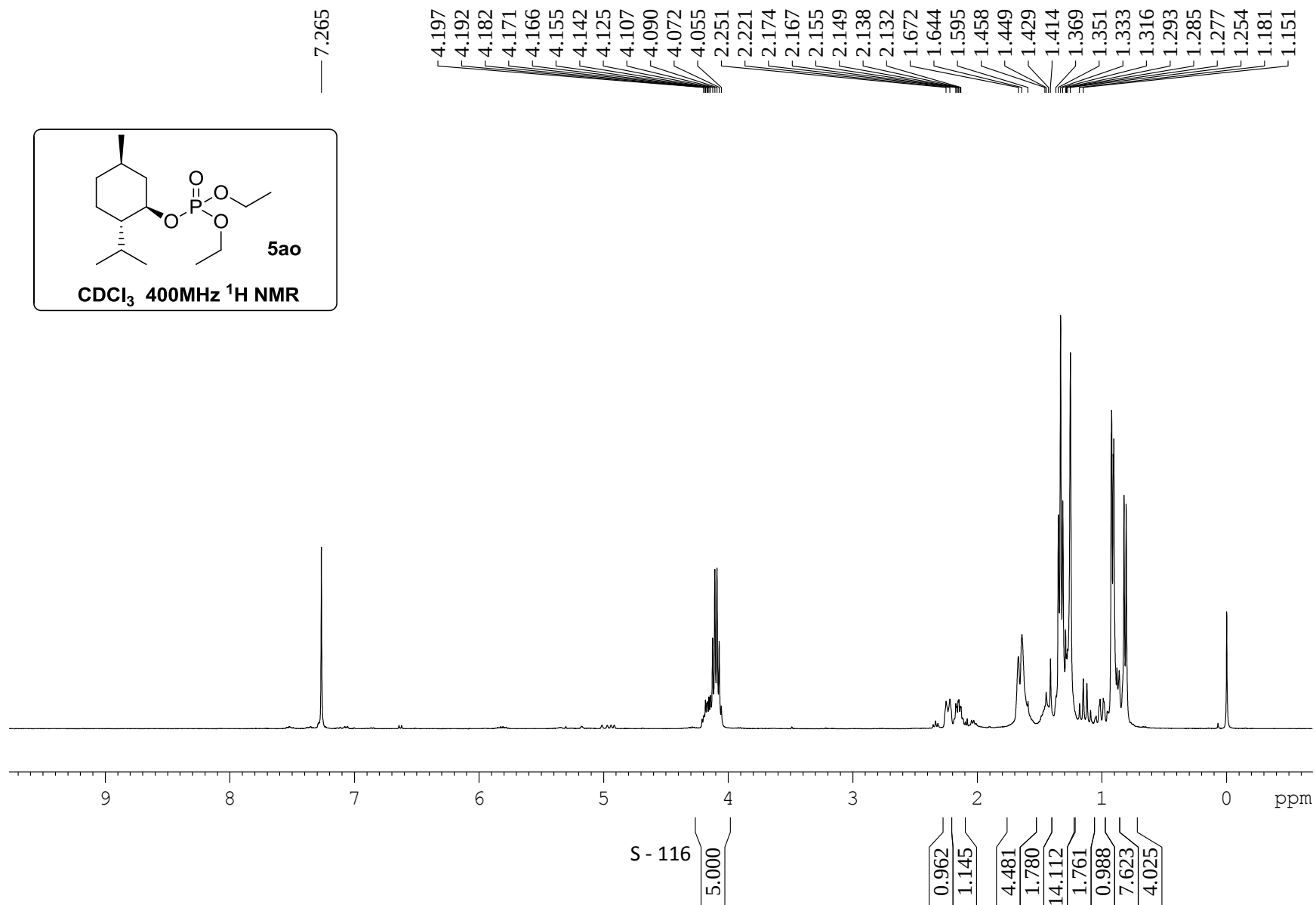


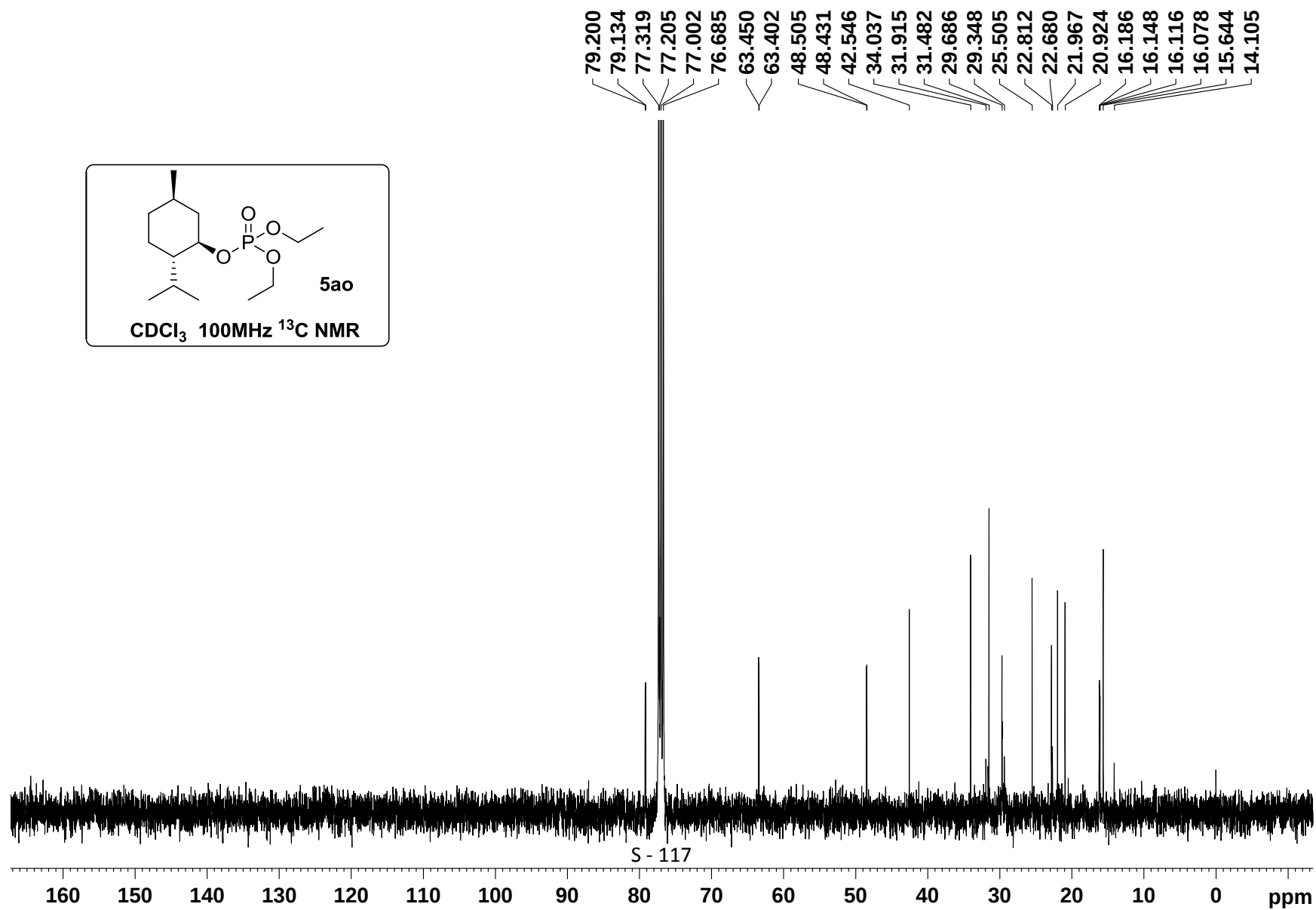
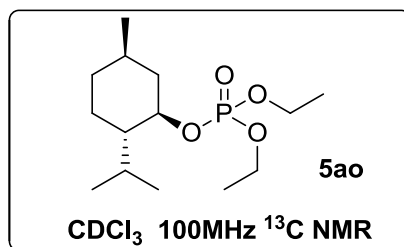


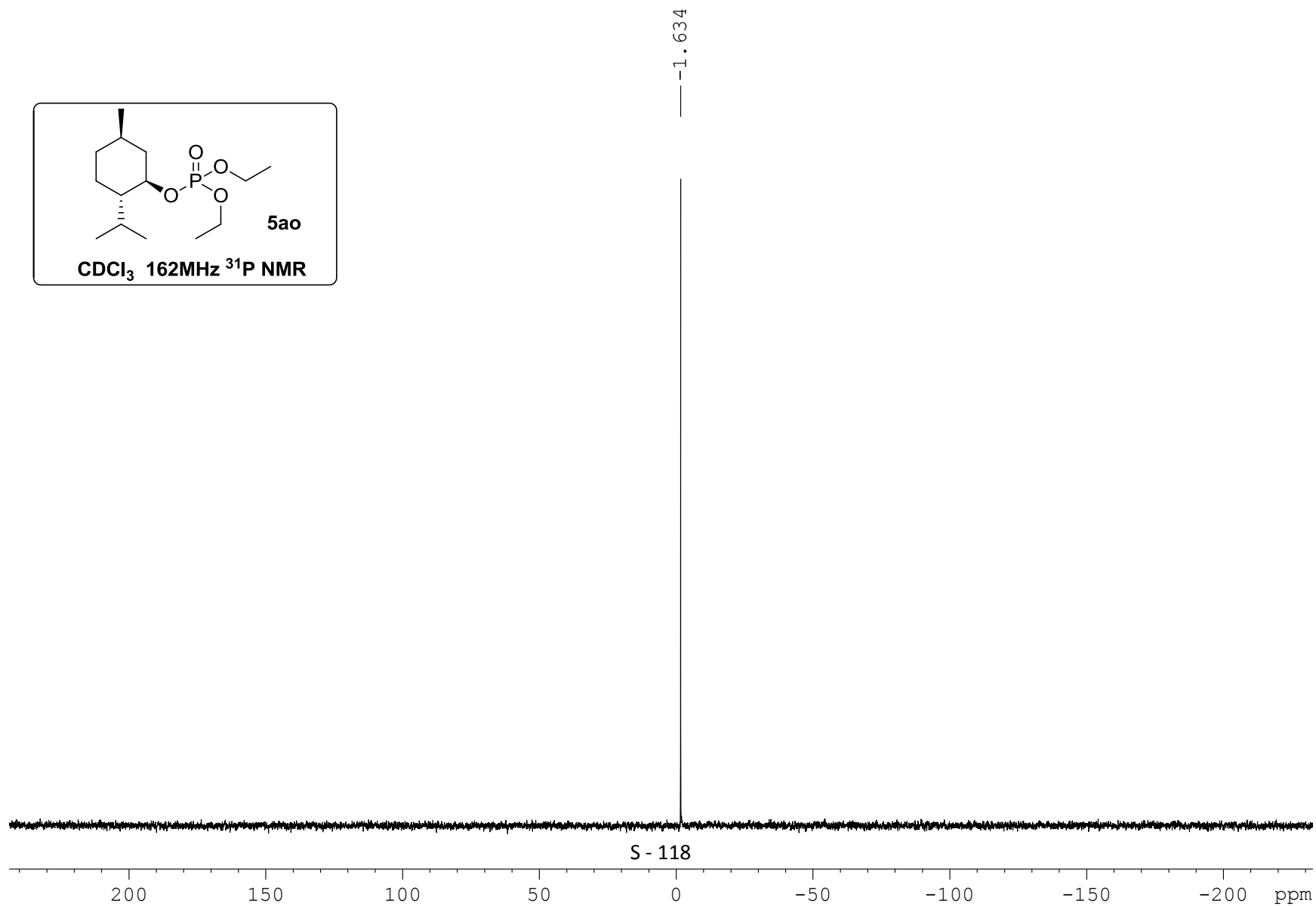
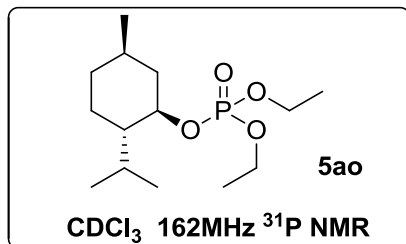


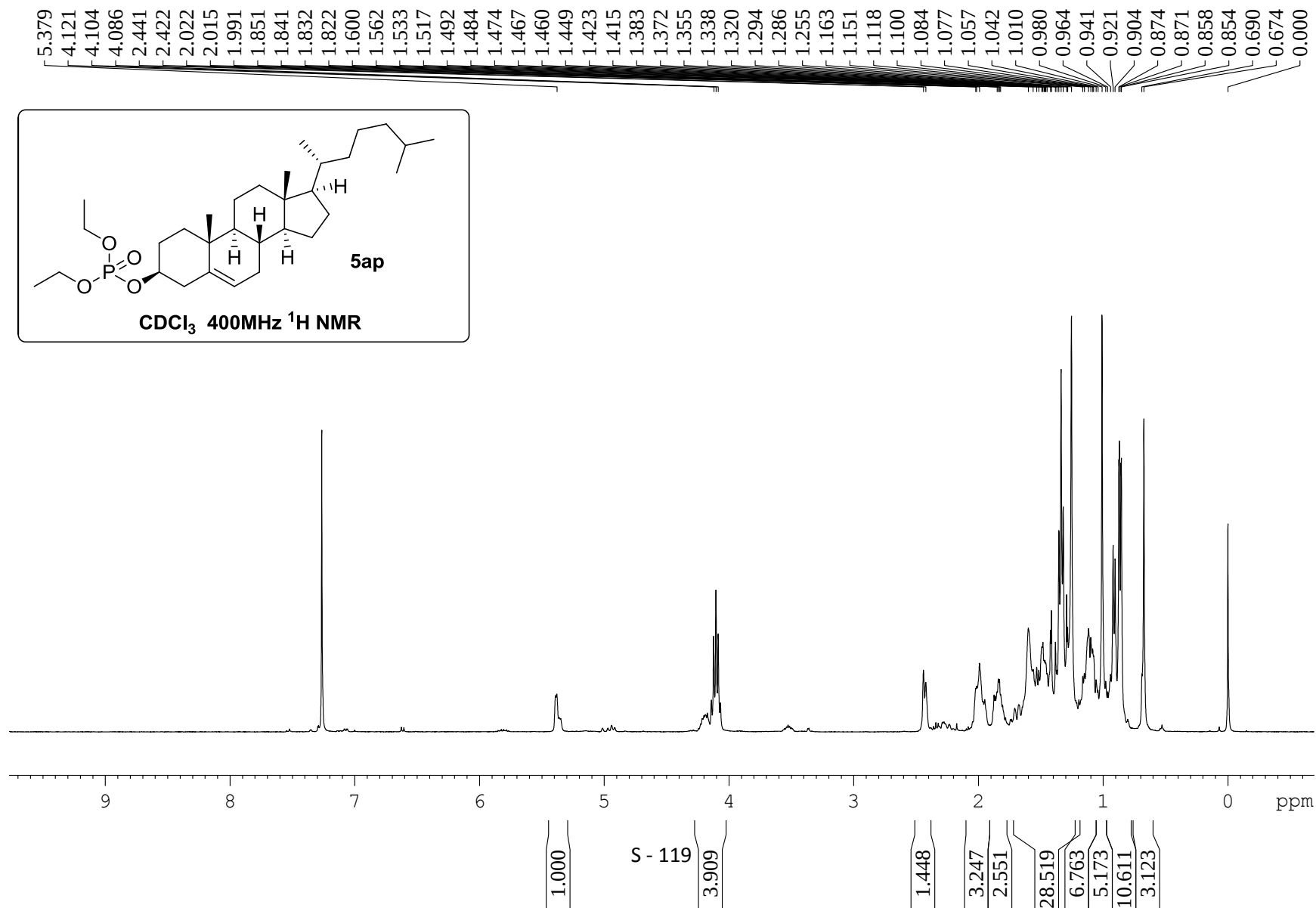


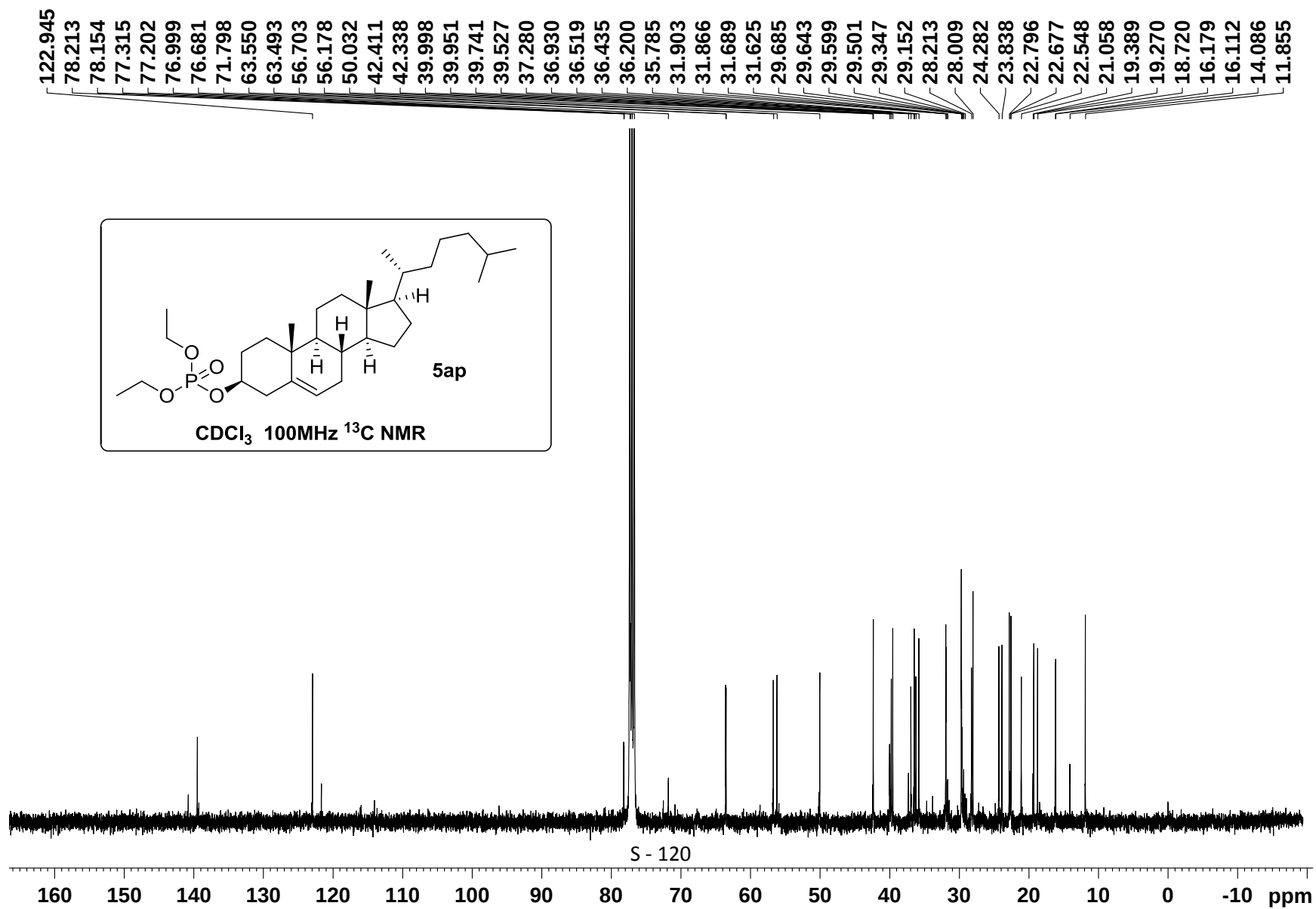


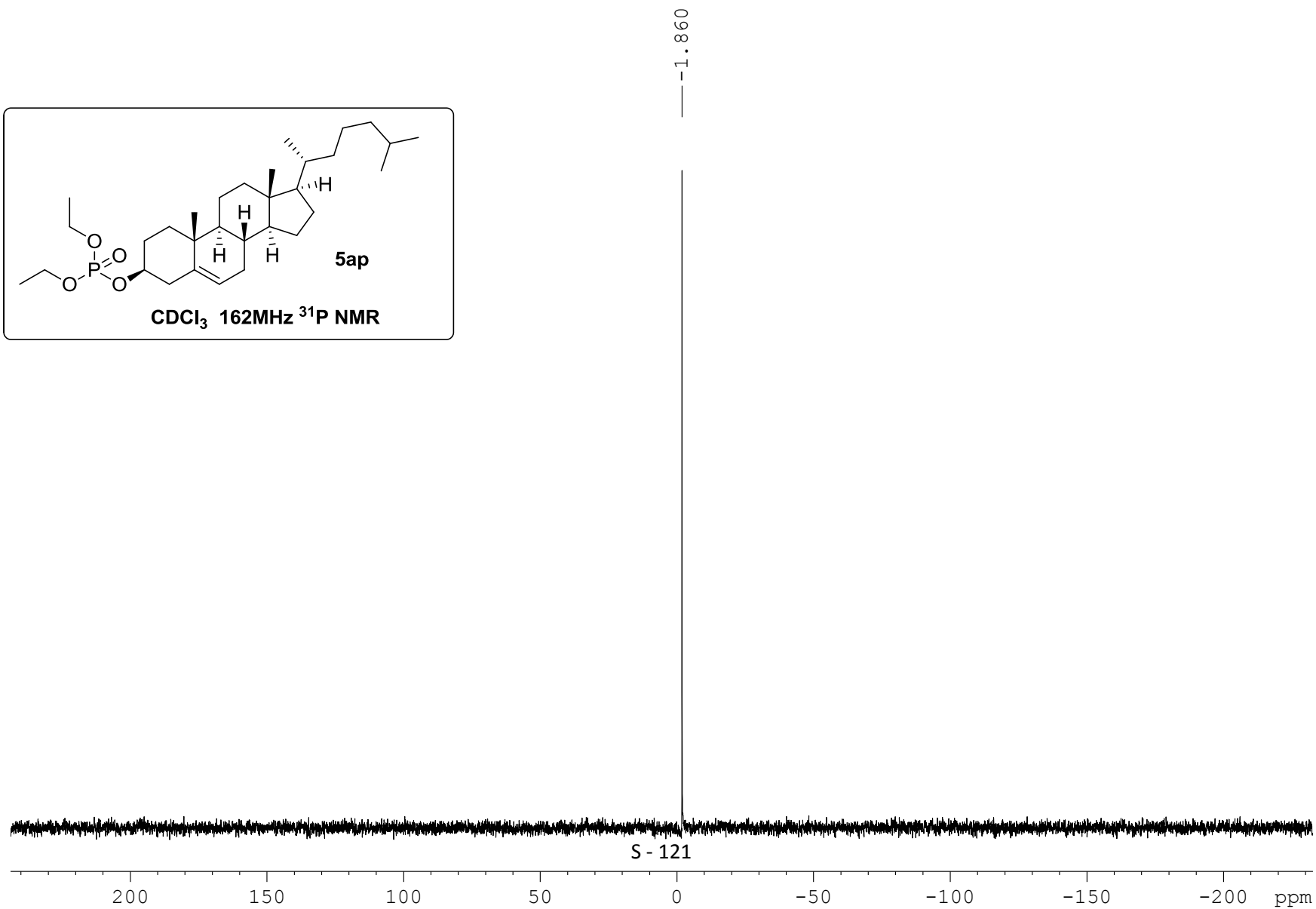
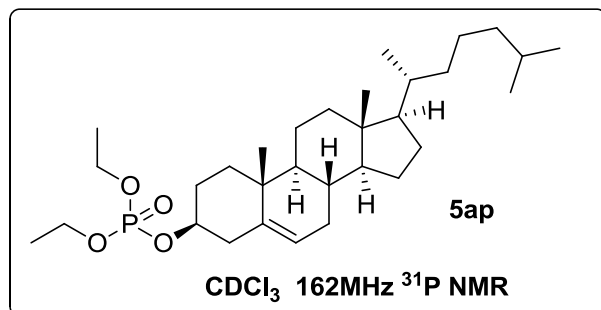


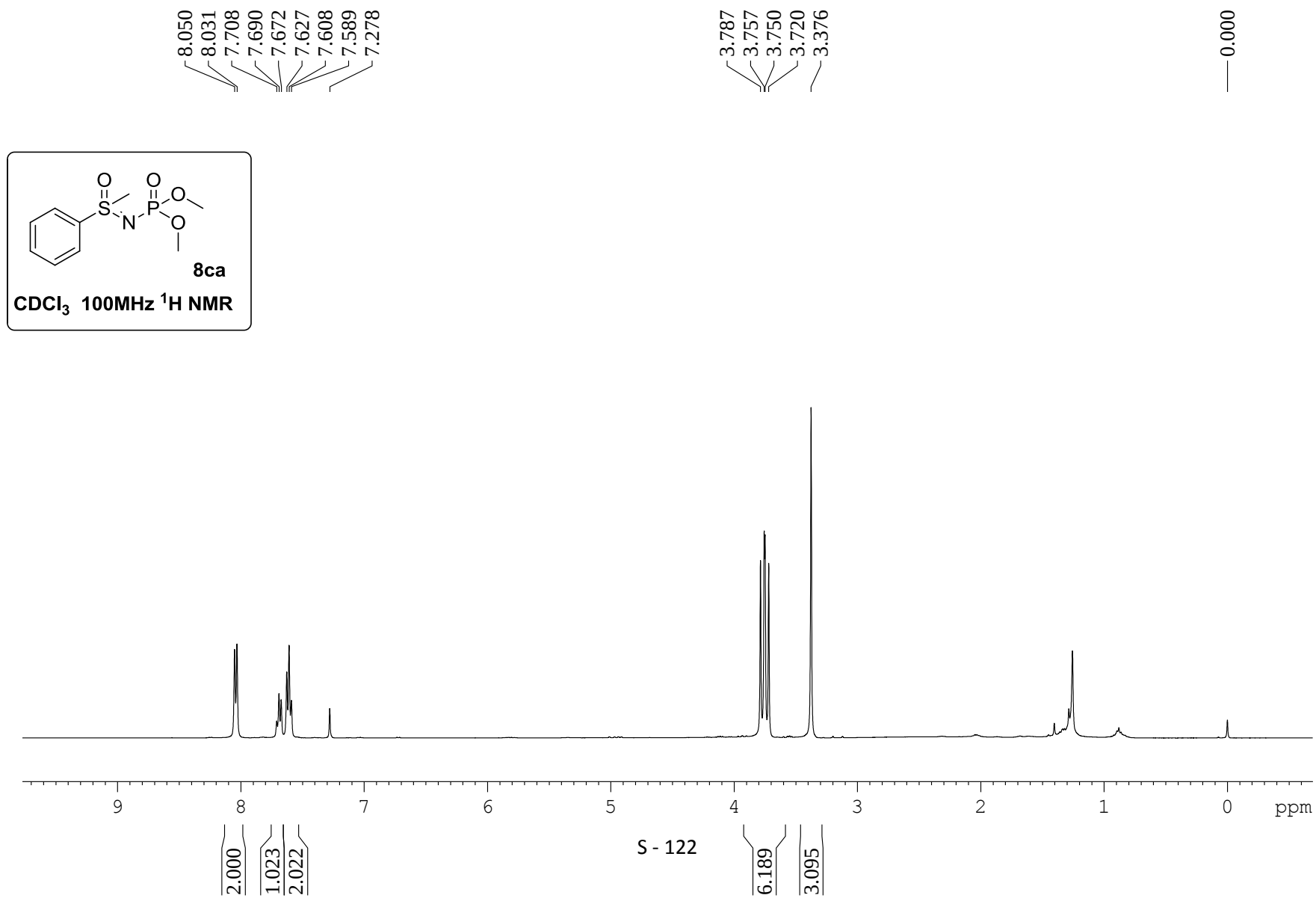


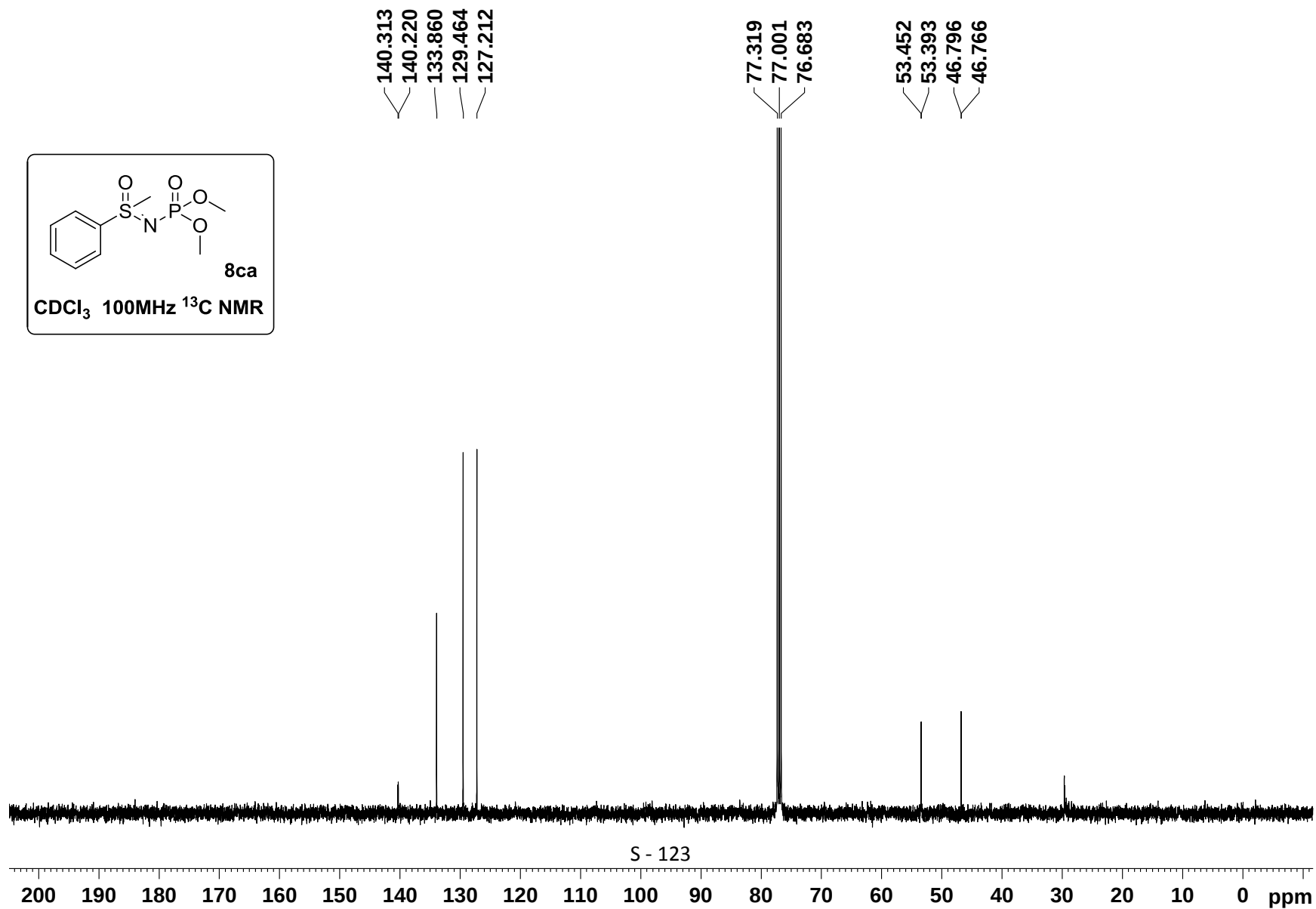


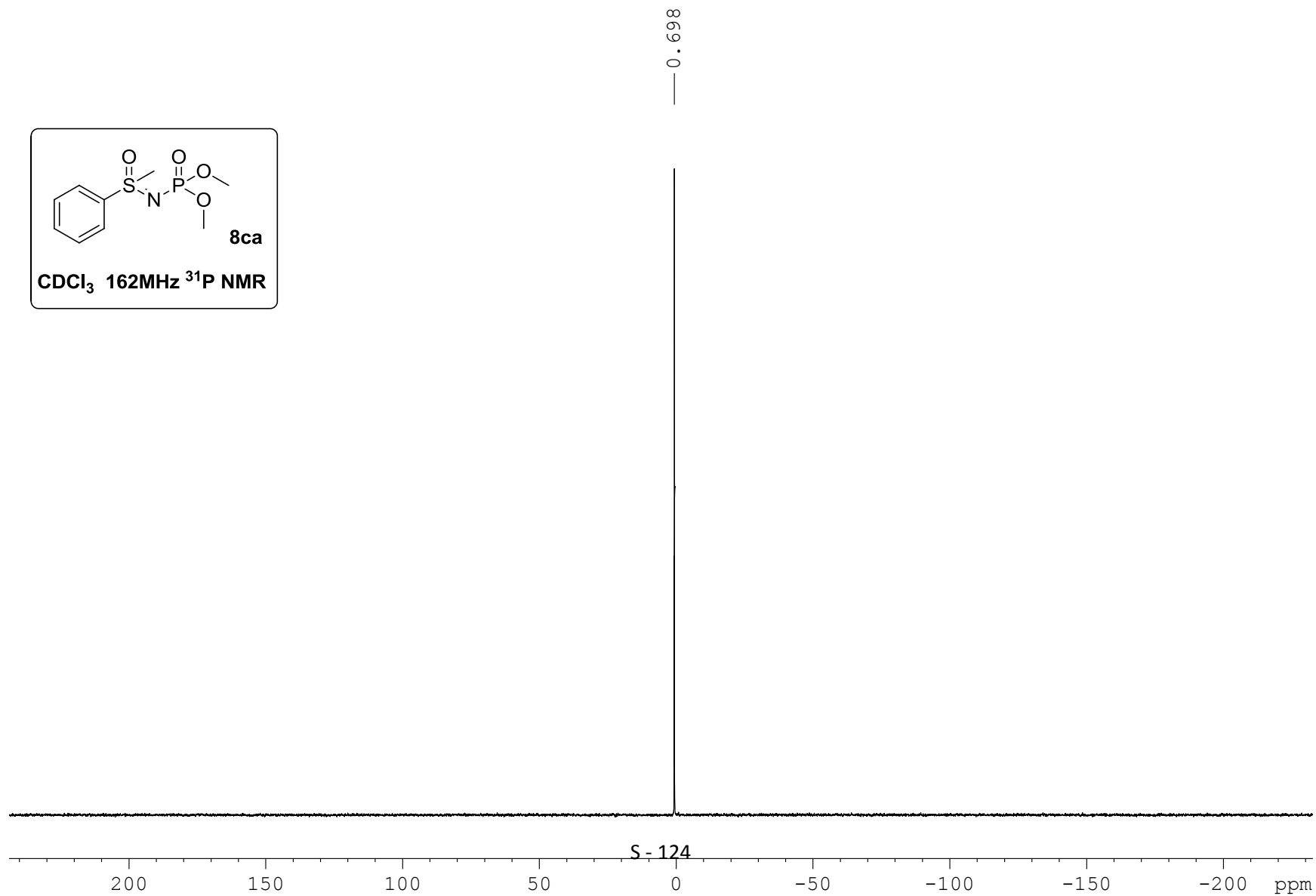
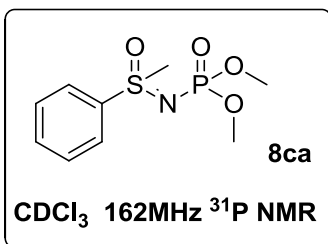


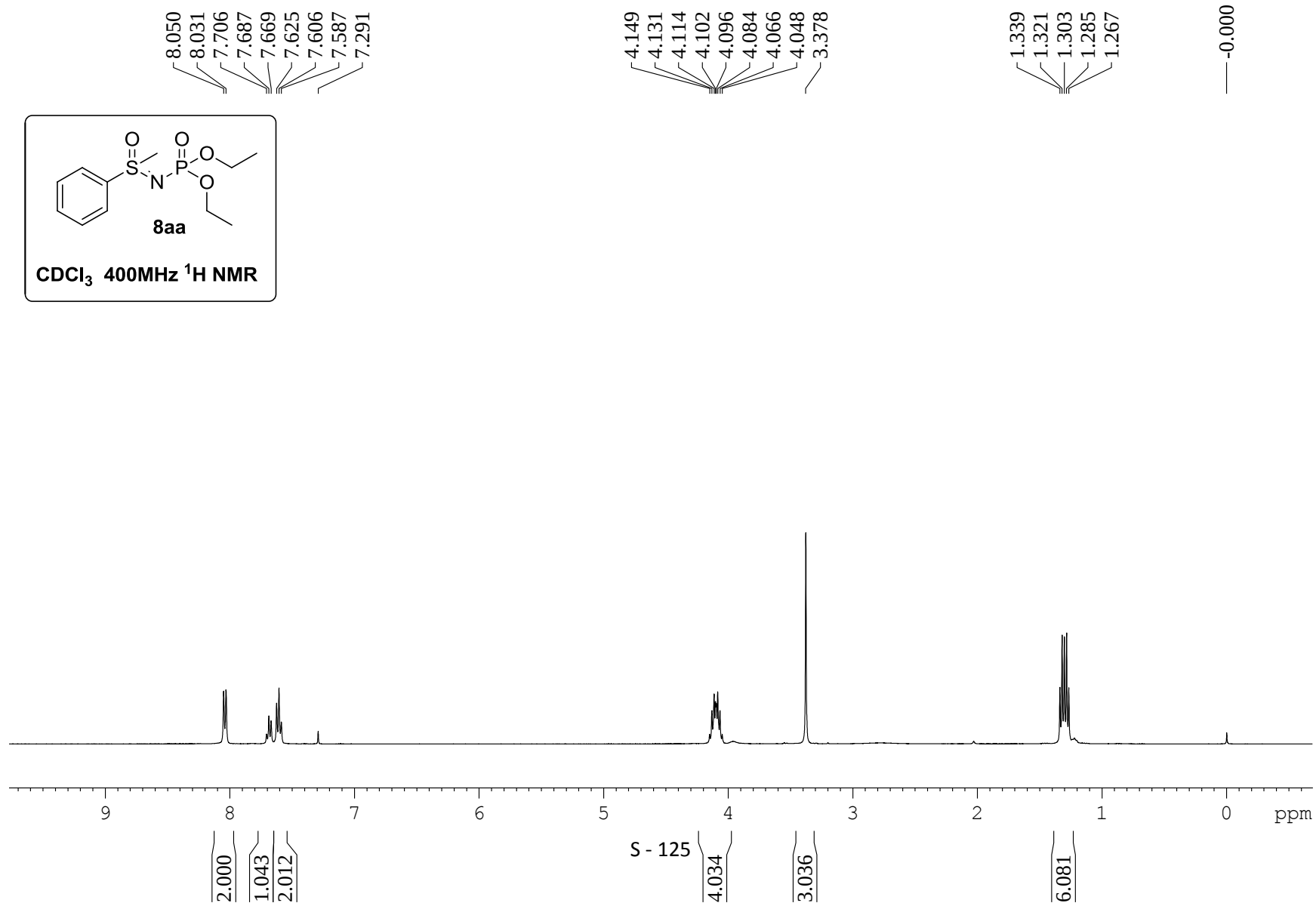


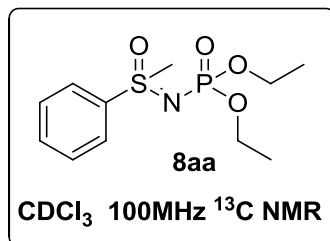












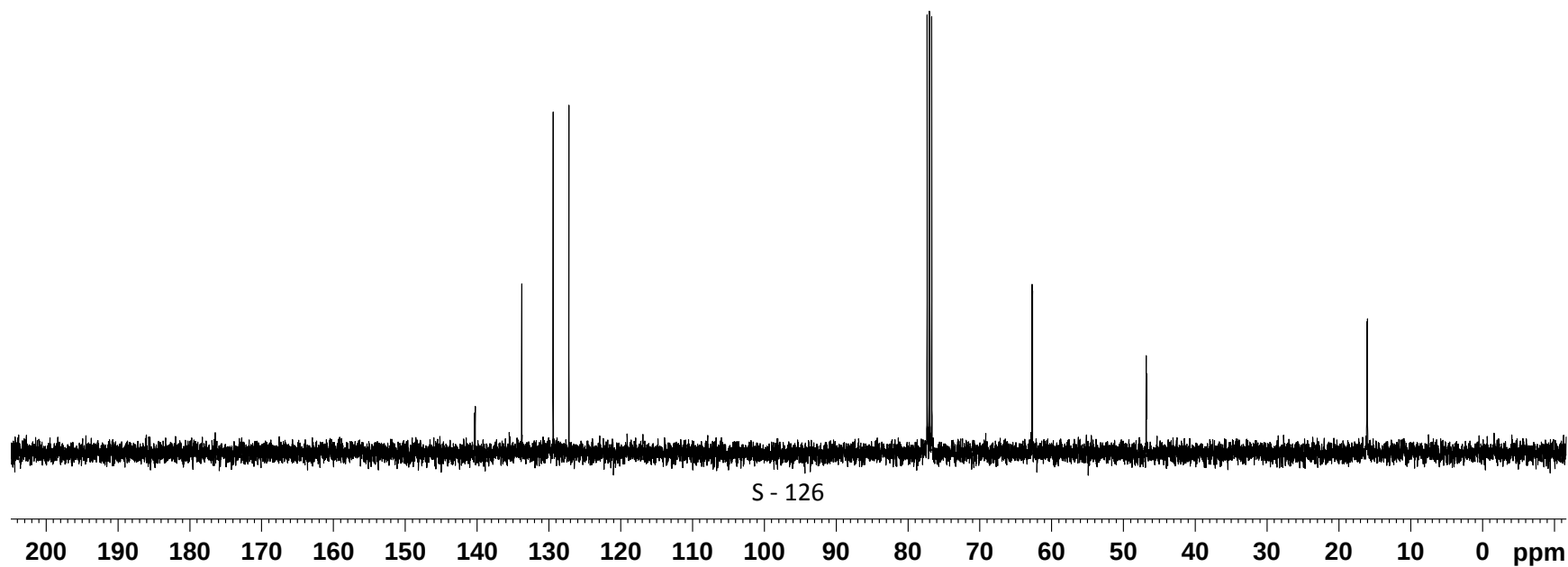
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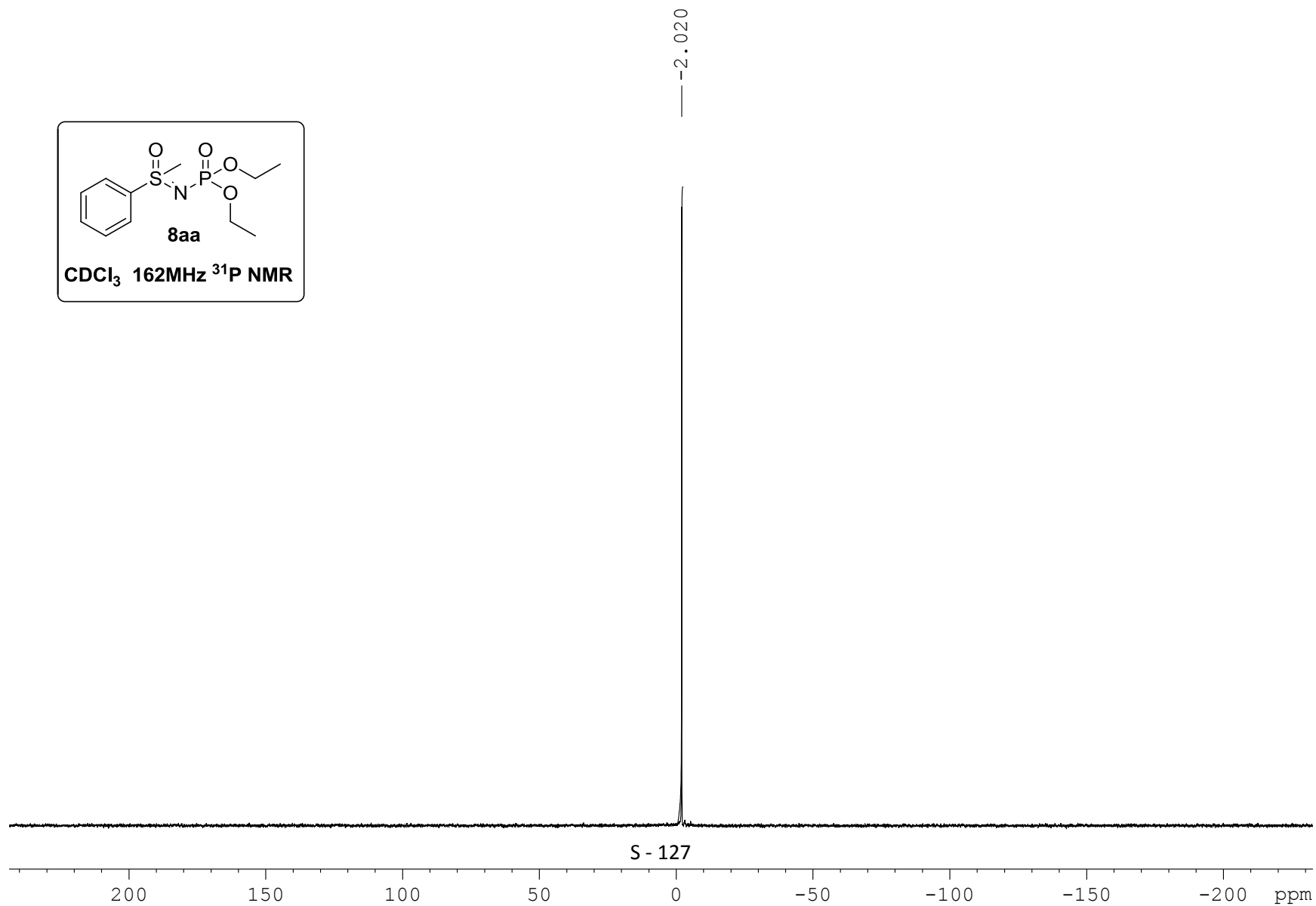
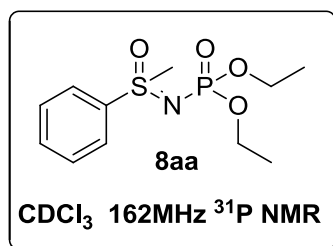
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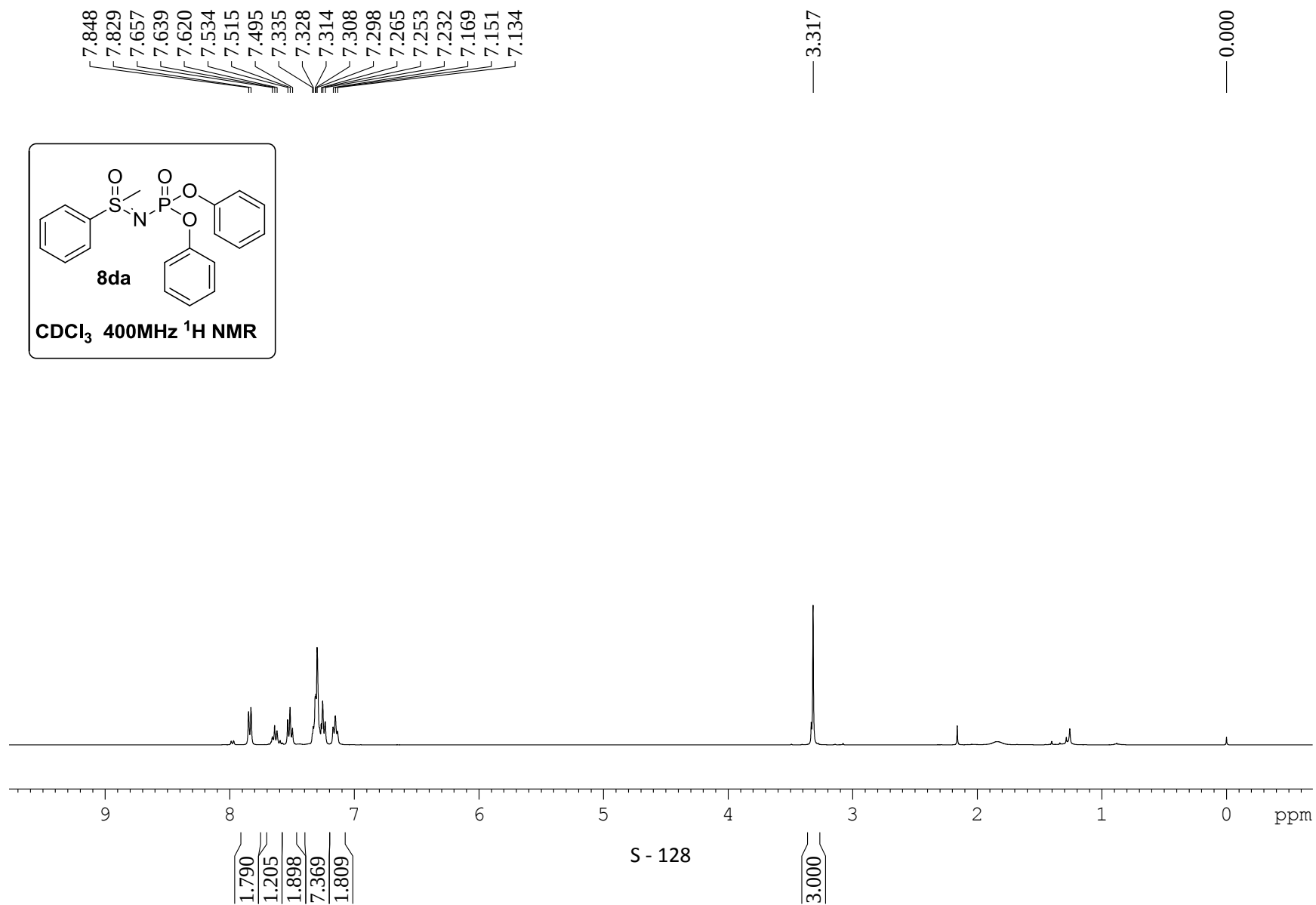
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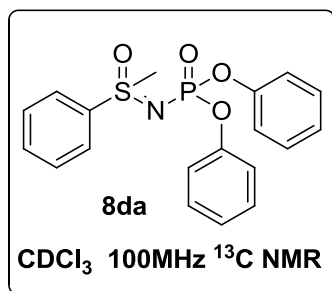
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