Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.


# Design of (β-Diazo-α,α-difluoroethyl)phosphonates and Their Application as Masked Carbene in Visible Light-Promoted Coupling Reaction with Sulfonic Acids

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## Table of Contends

## 1. General information

All the commercial reagents including solvents were used directly without further purification. Compounds **5** were synthesized according the literature.[1] All the experiments were monitored by thin layer chromatography (TLC) with UV light. The TLC employed 0.25 mm silica gel coated on glass plates. Purification of products was carried out by silica gel 60 F-254 TLC plates of 20 cm × 20 cm and column chromatography with silica gel 60 (300-400 mesh). Melting points were recorded without correction on RY-1G of Tianjin Xintianguang instrument company. NMR spectra were recorded on Bruker 400MHz and 600MHz spectrometers. High resolution mass spectra (HRMS) were measured on Agilent 6210 ESI/TOF MS instrument.

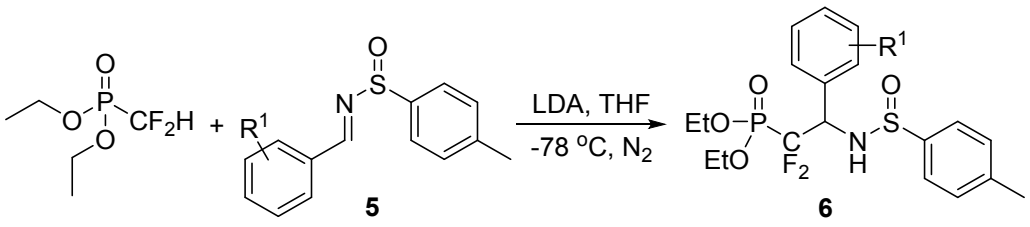
**Reference:**

(1) Morales, S.; Guijarro, F. G.; Ruano, J. L. G.; Cid, M. B. *J. Am. Chem. Soc.* **2014**, *136*, 1082-1089.

## 2. Synthesis of the starting reagents 1

Into an oven-dried reaction vial flushed with $N_2$ were taken α,α-difluoromethyl phosphonate (6 mmol) and anhydrous THF (12 mL). The reaction vial was cooled to -78 °C and LDA (2 M in THF, 3.6 mL) was added dropwise with stirring. After 0.5 h at -78 °C, imine **5** (5 mmol) dissolved in anhydrous THF (5 mL) was added dropwise. Stirring was continued at -78 °C for 1 h, then the reaction was quenched with saturated $NH_4Cl$ (5 mL), followed by $H_2O$ (10 mL) and the mixture was brought to room temperature. The organic layer was taken and the aqueous layer was extracted with $CH_2Cl_2$ (3 × 20 mL). The combined organic layers were dried with anhydrous $Na_2SO_4$, filtered and the solvent was removed to give the crude product **6**, which was purified by column chromatography using hexane/EtOAc (2:1, v/v) as eluent.

**Table S1.** Synthesis of compound **6**.



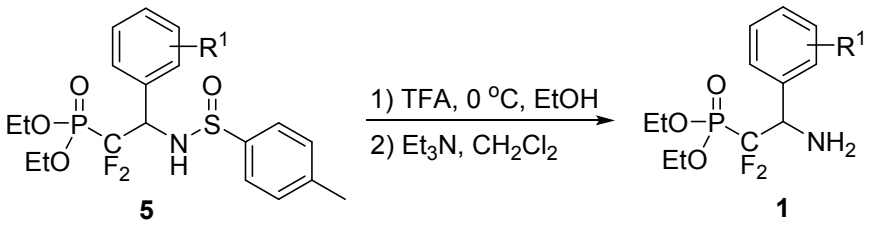

| Entry | $R^1$ | Product | Yield[a] (%) |
|---|---|---|---|

| | | | |
|---|---|---|---|
| 1 | H | **6a** | 95 |
| 2 | 4-F | **6b** | 84 |
| 3 | 3-F | **6c** | 78 |
| 4 | 4-Cl | **6d** | 58 |
| 5 | 3-Cl | **6e** | 79 |
| 6 | 2-Cl | **6f** | 73 |
| 7 | 4-Br | **6g** | 76 |
| 8 | 3-Br | **6h** | 60 |
| 9 | 4-$CF_3$ | **6i** | 64 |
| 10 | 4-CN | **6j** | 34 |
| 11 | 4-Me | **6k** | 57 |
| 12 | 3-Me | **6l** | 79 |
| 13 | 4-*i*Pr | **6m** | 81 |
| 14 | 4-*t*Bu | **6n** | 81 |
| 15 | 3-MeO | **6o** | 78 |

[a] Isolated yields.

Compound **5** (3 mmol) and ethanol (20 mL) were placed in a flask and cooled to 0 °C. $CF_3COOH$ (15 mmol) was added dropwise and stirred for 4 h. Volatiles were removed under reduced pressure. The residue was dissolved in $CH_2Cl_2$ (30 mL) followed by $Et_3N$ to neutralize the mixture. Water (30 mL) was added. The organic layer was taken and the aqueous layer was extracted with $CH_2Cl_2$ (3 × 20 mL). The combined organic layers were dried with anhydrous $Na_2SO_4$, filtered and the solvent was removed to give the crude product **1**, which was purified by column chromatography using hexane/EtOAc (2:1, v/v) as eluent.

**Table S2.** Synthesis of compound **1**.

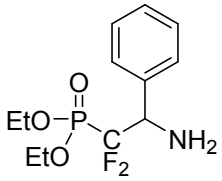



**5** **1**

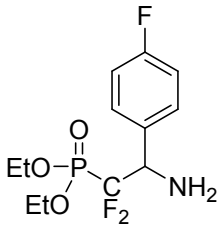
| Entry | $R^1$ | Product | Yield[a] (%) |
|---|---|---|---|
| 1 | H | **1a** | 84 |
| 2 | 4-F | **1b** | 81 |
| 3 | 3-F | **1c** | 78 |
| 4 | 4-Cl | **1d** | 72 |
| 5 | 3-Cl | **1e** | 51 |
| 6 | 2-Cl | **1f** | 77 |
| 7 | 4-Br | **1g** | 55 |
| 8 | 3-Br | **1h** | 65 |
| 9 | 4-$CF_3$ | **1i** | 30 |
| 10 | 4-CN | **1j** | 83 |
| 11 | 4-Me | **1k** | 95 |
| 12 | 3-Me | **1l** | 95 |
| 13 | 4-*i*Pr | **1m** | 63 |
| 14 | 4-*t*Bu | **1n** | 51 |
| 15 | 3-MeO | **1o** | 83 |

[a] Isolated yields.

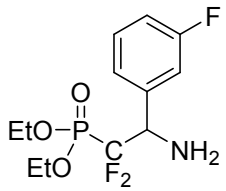
## 3. Characterization of the starting reagents 1



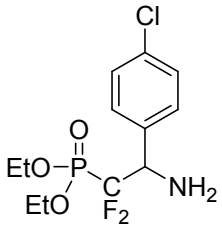

Compound **1a**: $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.44 (d, *J* = 7.38 Hz, 2H), 7.38-7.33 (m, 3H), 4.48-4.42 (m, 1H), 4.24-4.11 (m, 3H), 4.08-4.02 (m, 1H), 1.99 (s, 2H), 1.30 (t, *J* = 7.08 Hz, 3H), 1.27 (t, *J* = 7.02 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 136.5, 128.5, 128.4, 128.3, 123.3 (td, *J* = 264.9, 205.7 Hz), 64.6 (dd, *J* = 6.7, 24.5 Hz), 58.8 (td, *J* = 21.6, 16.0 Hz), 16.3 (dd, *J* = 1.9, 5.7 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$): δ = -114.6 (dd, *J* = 102.4, 298.7 Hz, 1F), -119.5 (dd, *J* = 105.1, 299.2 Hz, 1F). $^{31}$P NMR (243 MHz, $CDCl_3$): δ = 7.03 (t, *J* = 104.2 Hz, 1P).



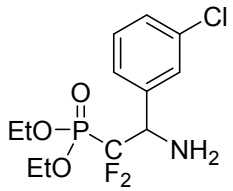
Compound **1b**: $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.39-7.37 (m, 2H), 7.03-7.00 (m, 2H), 4.43-4.38 (m, 1H), 4.21-4.02 (m, 4H), 1.94 (s, 2H), 1.27-1.22 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 163.5 (d, *J* = 245.1 Hz), 132.2 (d, *J* = 3.6 Hz), 130.3 (d, *J* = 7.9 Hz), 121.8 (td, *J* = 265.6, 206.4 Hz), 115.2 (d, *J* = 21.0 Hz), 64.6 (dd, *J* = 6.8, 35.8 Hz), 58.0 (td, *J* = 21.8, 16.0 Hz), 16.2 (dd, *J* = 2.2, 5.5 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -113.8 (s, 1F), -114.7 (dd, *J* = 102.7, 298.2 Hz, 1F), -119.6 (dd, *J* = 106.1, 298.8 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 7.05 (t, *J* = 103.0 Hz, 1P).



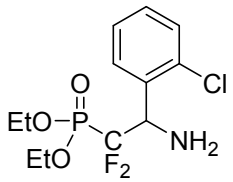
Compound **1c**: $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.32-7.27 (m, 1H), 7.19-7.12 (m, 2H), 7.02-6.97 (m, 1H), 4.45-4.37 (m, 1H), 4.22-4.05 (m, 4H), 1.97 (s, 2H), 1.28-1.23 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 163.9 (d, *J* = 244.3 Hz), 139.1, 129.8 (d, *J* = 7.9 Hz), 124.3, 123.0 (td, *J* = 265.5, 206.2 Hz), 115.6 (d, *J* = 20.9 Hz), 115.4 (d, *J* = 20.2 Hz), 64.7 (dd, *J* = 6.8, 20.8 Hz), 58.4 (td, *J* = 21.2, 16.1 Hz), 16.2 (d, *J* = 5.6 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -113.1 (s, 1F), -114.1 (dd, *J* = 102.5, 299.2 Hz, 1F), -119.9 (dd, *J* = 105.2, 299.4 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 6.95 (t, *J* = 102.9 Hz, 1P).



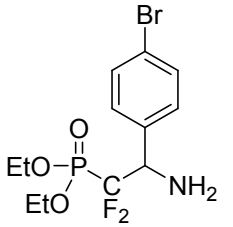
Compound **1d**: $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.36-7.31 (m, 4H), 4.44-4.38 (m, 1H), 4.21-4.07 (m, 4H), 1.96 (s, 2H), 1.28-1.25 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 135.0, 134.3, 129.9, 128.4, 121.7 (td, *J* = 264.9, 206.4 Hz), 64.7 (dd, *J* = 6.6, 32.2 Hz), 58.1 (td, *J* = 21.9, 16.1 Hz), 16.2 (d, *J* = 5.5 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$): δ = -114.5 (dd, *J* = 102.4, 298.9 Hz, 1F), -119.9 (dd, *J* = 106.2, 300.4 Hz, 1F). $^{31}$P NMR (243 MHz, $CDCl_3$): δ = 6.76 (t, *J* = 103.0 Hz, 1P).



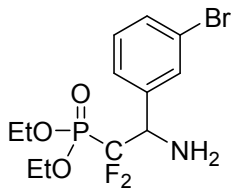

Compound **1e**: $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.46 (s, 1H), 7.35-7.30 (m, 3H), 4.47-4.41 (m, 1H), 4.27-4.10 (m, 4H), 1.94 (s, 2H), 1.32-1.29 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 138.6, 134.1, 129.5, 128.6, 128.5, 126.8, 121.7 (td, *J* = 265.2, 205.8 Hz), 64.7 (dd, *J* = 6.7, 30.9 Hz), 58.3 (td, *J* = 21.9, 15.9 Hz), 16.2 (d, *J* = 5.6 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -114.0 (dd, *J* = 102.9, 300.0 Hz, 1F), -119.8 (dd, *J* = 105.2, 299.3 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 6.93 (t, *J* = 103.0 Hz, 1P).



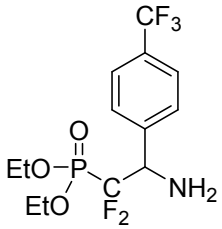

Compound **1f**: $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.54 (d, *J* = 7.72 Hz, 1H), 7.35 (d, *J* = 7.68 Hz, 1H), 7.28-7.18 (m, 2H), 5.12-5.03 (m, 1H), 4.28-4.17 (m, 4H), 1.99 (s, 2H), 1.33-1.26 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 134.9 (d, *J* = 6.8 Hz), 134.4, 129.5, 129.4, 129.2, 126.9, 123.1 (td, *J* = 266.0, 207.6 Hz), 64.8 (dd, *J* = 6.8, 11.9 Hz), 53.9-53.3 (m), 16.3 (t, *J* = 5.0 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -111.9 (dd, *J* = 102.5, 300.6 Hz, 1F), -122.6 (dd, *J* = 106.7, 300.6 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 7.23 (dd, *J* = 102.6, 107.3 Hz, 1P).



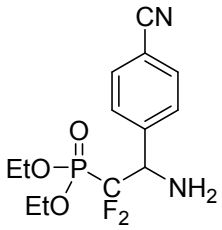

Compound **1g**: $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.52 (d, *J* = 8.46 Hz, 2H), 7.34 (d, *J* = 8.16 Hz, 2H), 4.46-4.41 (m, 1H), 4.26-4.09 (m, 4H), 1.88 (s, 2H), 1.32-1.29 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 135.6, 131.3, 130.2, 122.9 (td, *J* = 265.5, 206.0 Hz), 122.4, 64.7 (dd, *J* = 6.9, 28.8 Hz), 58.2 (td, *J* = 21.9, 15.8 Hz), 16.2 (d, *J* = 5.7 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -114.3 (dd, *J* = 102.8, 299.1 Hz, 1F), -119.8 (dd, *J* = 105.2, 299.2 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 6.94 (t, *J* = 103.2 Hz, 1P).



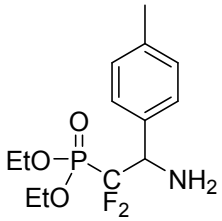

Compound **1h**: $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.52 (s, 1H), 7.40 (d, *J* = 8.04 Hz, 1H), 7.30 (d, *J* = 7.84 Hz, 1H), 7.18 (t, *J* = 7.84 Hz, 1H), 4.38-4.30 (m, 1H), 4.17-4.01 (m, 4H), 1.96 (s, 2H), 1.23-1.19 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 138.9, 131.5, 131.4, 129.8, 127.3, 122.9 (td, *J* = 265.0, 206.4 Hz), 122.2, 64.7 (dd, *J* = 6.7, 21.0 Hz), 58.3 (td, *J* = 21.6, 16.0 Hz), 16.2 (d, *J* = 5.7 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -113.9 (dd, *J* = 102.7, 299.8 Hz, 1F), -119.8 (dd, *J* = 105.1, 299.8 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 6.88 (t, *J* = 103.7 Hz, 1P).



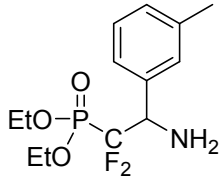

Compound **1i**: $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.65 (d, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 8.2 Hz, 2H), 4.56-4.48 (m, 1H), 4.24-4.09 (m, 4H), 2.00 (s, 2H), 1.30-1.25 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 140.5, 131.1 (q, *J* = 32.2 Hz), 129.0, 128.1 (q, *J* = 270.5 Hz), 125.2 (q, *J* = 3.6 Hz), 120.3, 64.8 (dd, *J* = 7.0, 19.3 Hz), 58.5 (td, *J* = 21.6, 15.5 Hz), 16.2 (d, *J* = 5.7 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -62.7 (s, 3F), -114.1 (dd, *J* = 102.4, 299.7 Hz, 1F), -119.7 (dd, *J* = 104.8, 300.1 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 6.72 (t, *J* = 102.5 Hz, 1P).



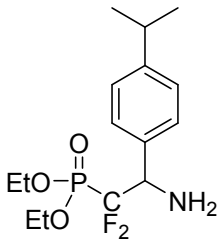

Compound **1j**: $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.65 (d, *J* = 7.96 Hz, 2H), 7.56 (d, *J* = 8.12 Hz, 2H), 4.53-4.45 (m, 1H), 4.23-4.10 (m, 4H), 2.03 (s, 2H), 1.29 (t, *J* = 7.08 Hz, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 141.8, 132.0, 129.4, 120.1 (d, *J* = 205.8 Hz), 118.5, 112.3, 64.9 (dd, *J* = 7.1, 14.5 Hz), 58.3 (td, *J* = 21.6, 15.3 Hz), 16.3 (d, *J* = 5.5 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -113.5 (dd, *J* = 102.3, 300.4 Hz, 1F), -120.1 (dd, *J* = 103.9, 300.3 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 6.54 (t, *J* = 102.6 Hz, 1P).



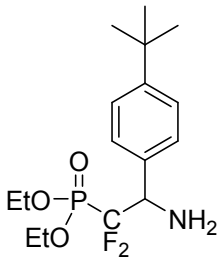
Compound **1k**: $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.31 (d, *J* = 7.88 Hz, 2H), 7.17 (d, *J* = 7.96 Hz, 2H), 4.44-4.35 (m, 1H), 4.25-4.00 (m, 4H), 2.34 (s, 3H), 1.94 (s, 2H), 1.30-1.23 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 138.2, 133.5, 129.0, 128.4, 123.3 (td, *J* = 264.9, 205.8 Hz), 64.6 (dd, *J* = 6.9, 23.7 Hz), 58.4 (td, *J* = 21.1, 15.8 Hz), 21.1, 16.3 (dd, *J* = 2.9, 5.8 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -114.1 (dd, *J* = 103.7, 298.6 Hz, 1F), -119.8 (dd, *J* = 106.4, 298.7 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 7.44 (t, *J* = 104.5 Hz, 1P).



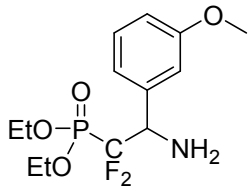
Compound **1l**: $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.27-7.23 (m, 3H), 7.17 (d, *J* = 7.44 Hz, 1H), 4.45-4.40 (m, 1H), 4.27-4.14 (m, 3H), 4.11-4.04 (m, 1H), 2.38 (s, 3H), 1.92 (s, 2H), 1.33 (t, *J* = 7.08 Hz, 3H), 1.29 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 138.0, 136.5, 129.2, 128.2, 125.6, 120.3, 118.9, 64.6 (dd, *J* = 7.2, 37.1 Hz), 58.6 (td, *J* = 22.0, 16.1 Hz), 21.4, 16.3 (t, *J* = 5.2 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -114.0 (dd, *J* = 102.9, 298.2 Hz, 1F), -119.7 (dd, *J* = 106.4, 298.7 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 7.44 (dd, *J* = 102.8, 105.9 Hz, 1P).



Compound **1m**: $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.37 (d, *J* = 7.86 Hz, 2H), 7.25 (d, *J* = 7.98 Hz, 2H), 4.46-4.40 (m, 1H), 4.24-4.10 (m, 3H), 4.06-3.99 (m, 1H), 2.96-2.89 (m, 1H), 1.98 (s, 2H), 1.30-1.23 (m, 12H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 149.2, 133.9, 128.5, 126.4, 120.4 (d, *J* = 206.1 Hz), 64.5 (dd, *J* = 6.8, 40.9 Hz), 58.4 (td, *J* = 21.1, 15.8 Hz), 33.9, 24.0 (d, *J* = 2.3 Hz), 16.3 (t, *J* = 5.0 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -114.9 (dd, *J* = 102.6, 297.8 Hz, 1F), -118.9 (dd, *J* = 106.5, 298.6 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 7.32 (t, *J* = 104.5 Hz, 1P).



Compound **1n**: $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.38-7.32 (m, 4H), 4.44-4.35 (m, 1H), 4.20-3.94 (m, 4H), 2.02 (s, 2H), 1.29 (s, 9H), 1.26-1.17 (m, 6H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 151.3, 133.5, 128.2, 125.2, 123.4 (td, *J* = 265.0, 205.0 Hz), 64.5 (dd, *J* = 6.8, 28.0 Hz), 58.4 (td, *J* = 21.2, 16.1 Hz), 34.5, 31.3, 16.2 (dd, *J* = 3.6, 6.2 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -114.9 (dd, *J* = 103.7, 298.4 Hz, 1F), -118.8 (dd, *J* = 106.2, 298.1 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 7.28 (t, *J* = 104.5 Hz, 1P).



Compound **1o**: $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.11 (t, *J* = 7.86 Hz, 1H), 6.86-6.83 (m, 2H), 6.72-6.70 (m, 1H), 4.28-4.22 (m, 1H), 4.09-3.96 (m, 3H), 3.94-3.87 (m, 1H), 3.62 (s, 3H), 1.90 (s, 2H), 1.14 (t, *J* = 7.08 Hz, 3H), 1.11 (t, *J* = 7.02 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 159.4, 138.1, 129.1, 121.9 (td, *J* = 265.1, 205.8 Hz), 120.7, 114.1, 113.7, 64.4 (dd, *J* = 6.6, 39.3 Hz), 58.5 (td, *J* = 21.7, 15.6 Hz), 55.0, 16.1 (dd, *J* = 3.5, 5.5 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$): δ = -114.1 (dd, *J* = 104.1, 299.8 Hz, 1F), -120.1 (dd, *J* = 106.4, 298.8 Hz, 1F). $^{31}$P NMR (243 MHz, $CDCl_3$): δ = 7.06 (t, *J* = 105.7 Hz, 1P).

## 4. General procedure for the reaction of amine and sulfonic acid

A vail was charged with amine **1** (0.2 mmol), sulfonic acid **2** (0.1 mmol), *t*-BuONO (0.24 mmol), $CCl_4$ (3 mL) and heated to 60 °C. The mixture was stirred in the presence of 4.5 W blue LEDs for 1 h. Then water (5 mL) was added. The organic layer was taken and the aqueous layer was extracted with $CH_2Cl_2$ (3 × 10 mL). The combined organic layers were dried with anhydrous $Na_2SO_4$, filtered and the solvent was removed to give the crude product **4**, which was purified by TLC plate using hexane/EtOAc (4:1, v/v) as eluent.

## 5. Control experiment

A vail was charged with amine **1a** (0.2 mmol), sodium sulfonate **7a** (0.1 mmol), *t*-BuONO (0.24 mmol), $CCl_4$ (2 mL), $H_2O$ (1 mL) and heated to 60 °C. The mixture was stirred in the presence of 4.5 W blue LEDs for 1 h.

A vail was charged with amine **1a** (0.2 mmol), tetrabutylammonium 4-toluenesulfonate (**7b**) (0.1 mmol), *t*-BuONO (0.24 mmol), $CCl_4$ (3 mL) and heated to 60 °C. The mixture was stirred in the presence of 4.5 W blue LEDs for 1 h.

## 6. Detection of diazo intermediate via $^{19}F$ NMR

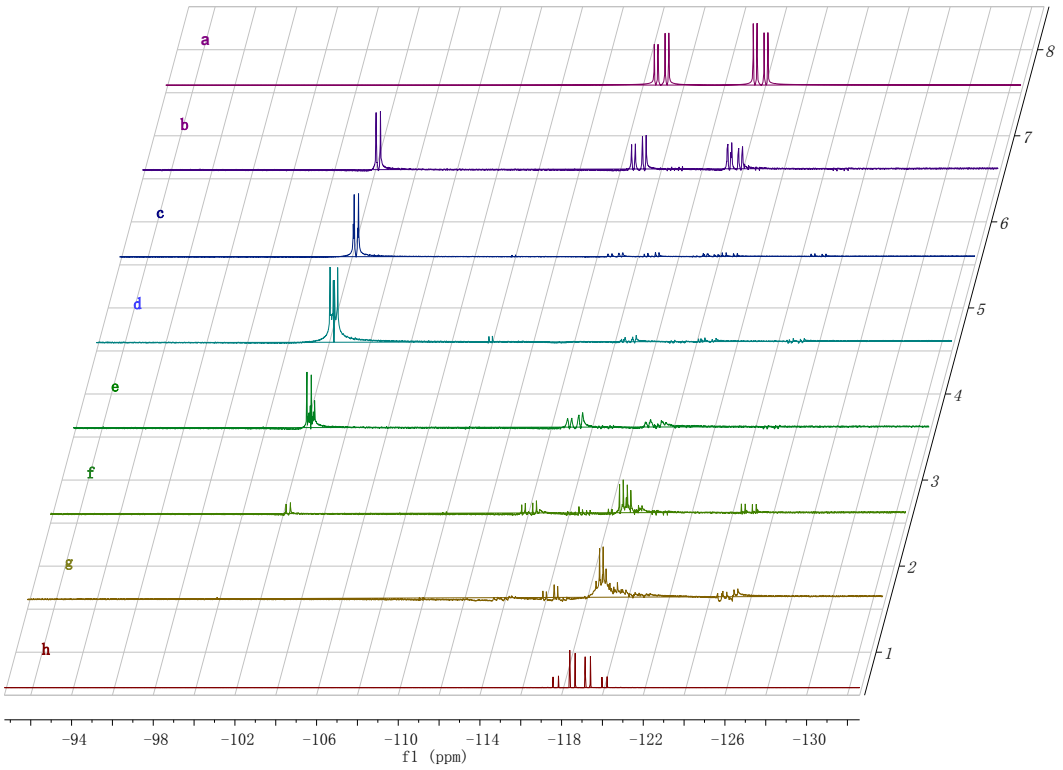



**Figure S1.** $^{19}F$ NMR detection of diazo intermediate.

**a**: pure amine **1a**

**b**: Stirring the mixture of amine **1a**, HOAc, and *t*-BuONO in $CCl_4$ for 3 min

**c**: Stirring the mixture of amine **1a**, HOAc, and *t*-BuONO in $CCl_4$ for 10 min

**d**: Stirring the mixture of amine **1a**, HOAc, and *t*-BuONO in $CCl_4$ for 20 min

**e**: Addition of *p*-toluene sulfonic acid to the above mixture and stirring for 30 second

**f**: Addition of *p*-toluene sulfonic acid to the above mixture and stirring for 3 min

**g**: Addition of *p*-toluene sulfonic acid to the above mixture and stirring for 10 min

**h**: pure product **4aa**

## 7. LEDs experimental setup

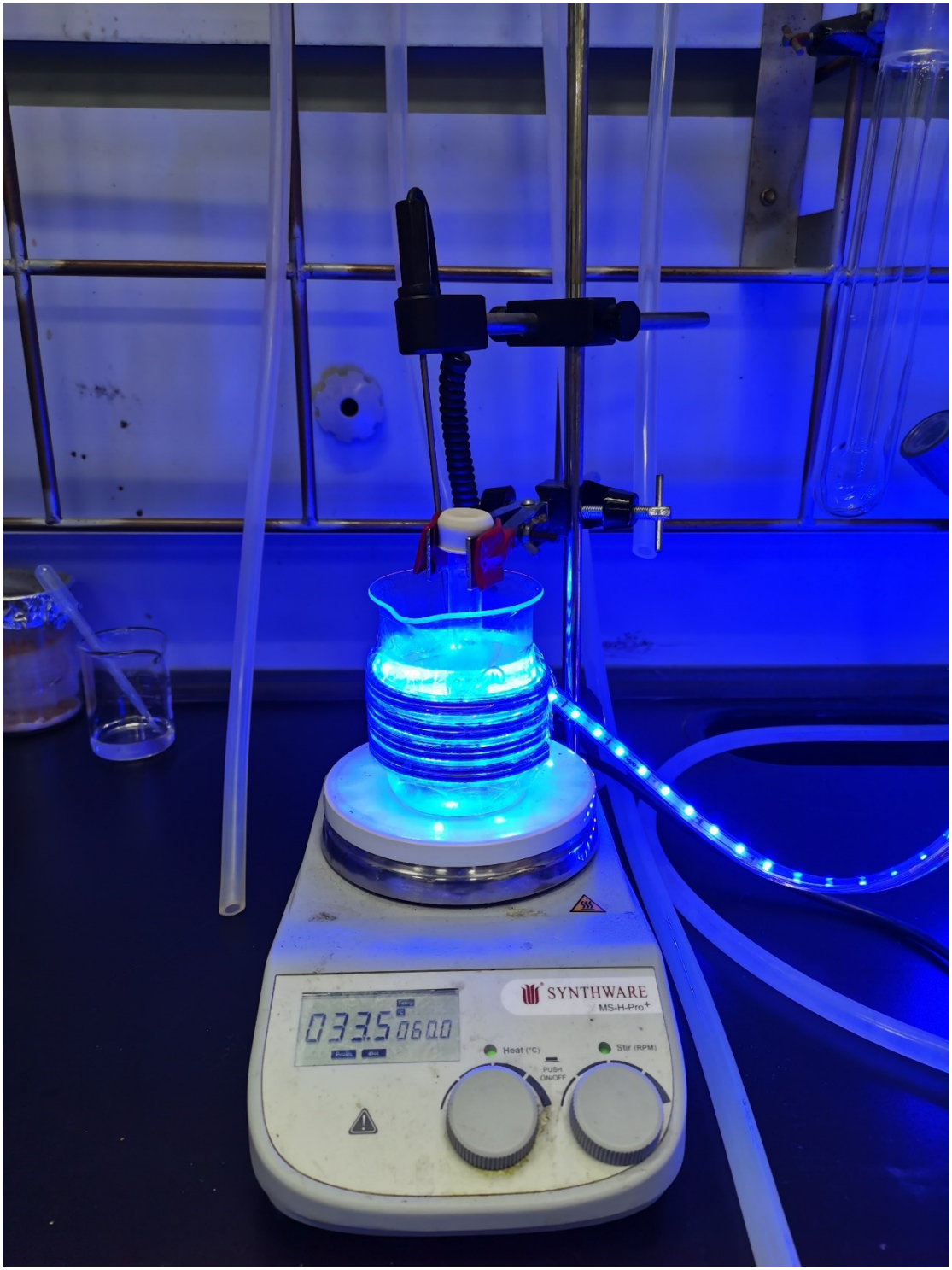
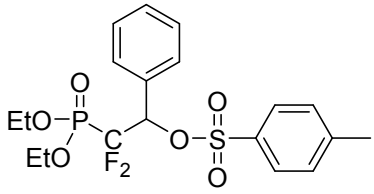
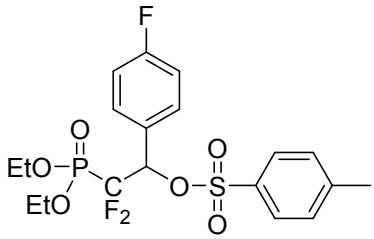



**Figure S2.** LEDs experimental setup.

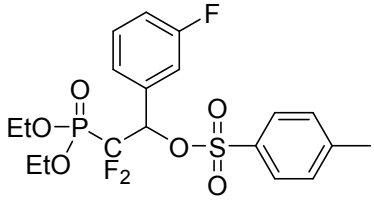
## 8. Characterization data of the product 4



Compound **4aa**: 83% yield, colorless oil. $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 8.4 Hz, 2H), 7.33-7.29 (m, 3H), 7.26-7.22 (m, 2H), 7.15 (d, *J* = 8.08 Hz, 2H), 5.87-5.80 (m, 1H), 4.32-4.16 (m, 2H), 4.11-4.02 (m, 1H), 3.99-3.89 (m, 1H), 2.37 (s, 3H), 1.34 (t, *J* = 7.08 Hz, 3H), 1.19 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 144.9, 133.5, 130.2, 129.7, 129.4, 129.1, 128.1, 127.9, 119.0 (td, *J* = 267.1, 209.1 Hz), 80.3 (td, *J* = 25.5, 20.9 Hz), 65.1 (dd, *J* = 6.6, 61.4 Hz), 21.5, 16.2 (dd, *J* = 5.5, 26.5 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -117.6 (dd, *J* = 100.0, 307.6 Hz, 1F), -119.2 (dd, *J* = 96.4, 308.4 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.59 (dd, *J* = 96.5, 101.2 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{24}F_2O_6PS^+$ $[M+H]^+$ 449.0994, found 449.0984.

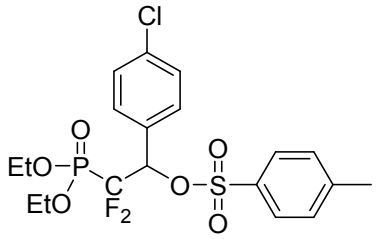


Compound **4ba**: 64% yield, white solid, mp 53-54 °C. $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 8.28 Hz, 2H), 7.32-7.30 (m, 2H), 7.18 (d, *J* = 8.1 Hz, 2H), 6.95 (t, *J* = 8.64 Hz, 2H), 5.84 (t, *J* = 12.48 Hz, 1H), 4.30-4.18 (m, 2H), 4.13-4.06 (m, 1H), 4.04-3.97 (m, 1H), 2.38 (s, 3H), 1.34 (t, *J* = 7.02 Hz, 3H), 1.21 (t, *J* = 7.02 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 164.3 (d, *J* = 248.4 Hz), 145.1, 133.5, 131.2 (d, *J* = 8.2 Hz), 129.5, 128.0, 126.3, 118.9 (td, *J* = 267.5, 209.1 Hz), 115.3 (d, *J* = 21.9 Hz), 79.5-79.1 (m), 65.2 (dd, *J* = 6.6, 58.4 Hz), 21.6, 16.3 (dd, *J* = 5.5, 24.1 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -110.9 (s, 1F), -118.0 (dd, *J* = 100.5, 308.4 Hz, 1F), -119.2 (dd, *J* = 95.8, 307.7 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.46 (t, *J* = 97.9 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{23}F_3O_6PS^+$ $[M+H]^+$ 467.0900, found 467.0900.

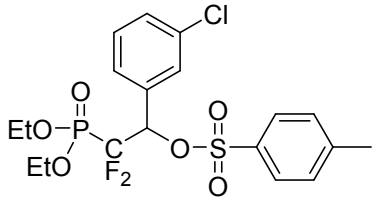


Compound **4ca**: 73% yield, white solid, mp 70-71 °C. $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.61 (d, *J* =

8.1 Hz, 2H), 7.24-7.22 (m, 1H), 7.17 (d, *J* = 8.04 Hz, 2H), 7.12 (d, *J* = 7.74 Hz, 1H), 7.02-6.99 (m, 2H), 5.83 (t, *J* = 12.24 Hz, 1H), 4.30-4.18 (m, 2H), 4.15-4.08 (m, 1H), 4.06-3.99 (m, 1H), 2.37 (s, 3H), 1.34 (t, *J* = 7.08 Hz, 3H), 1.22 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 163.1 (d, *J* = 246.1 Hz), 145.2, 133.3, 132.6, 129.8 (d, *J* = 8.6 Hz), 129.5, 128.0, 125.0 (d, *J* = 3.1 Hz), 118.7 (td, *J* = 267.7, 209.2 Hz), 116.8 (d, *J* = 20.9 Hz), 116.1 (d, *J* = 22.9 Hz), 79.4 (td, *J* = 23.9, 18.9 Hz), 65.2 (dd, *J* = 6.7, 54.9 Hz), 21.5, 16.3 (dd, *J* = 5.8, 24.6 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -112.6 (s, 1F), -118.9 (d, *J* = 97.5 Hz, 2F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.34 (t, *J* = 98.3 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{23}F_3O_6PS^+$ $[M+H]^+$ 467.0900, found, 467.0892.

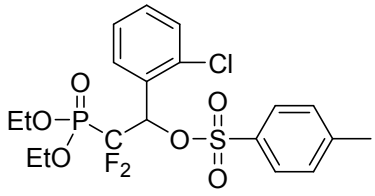



Compound **4da**: 72% yield, colorless oil. $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 8.1 Hz, 2H), 7.26 (d, *J* = 8.34 Hz, 2H), 7.23 (d, *J* = 8.58 Hz, 2H), 7.19 (d, *J* = 7.98 Hz, 2H), 5.82 (t, *J* = 12.3 Hz, 1H), 4.29-4.20 (m, 2H), 4.13-4.08 (m, 1H), 4.06-3.99 (m, 1H), 2.40 (s, 3H), 1.34 (t, *J* = 7.14 Hz, 3H), 1.22 (t, *J* = 7.14 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 145.2, 136.0, 133.4, 130.5, 129.5, 128.9, 128.4, 128.0, 117.0 (d, *J* = 209.5 Hz), 79.5 (td, *J* = 24.0, 19.9 Hz), 65.2 (dd, *J* = 6.6, 53.8 Hz), 21.6, 16.3 (dd, *J* = 6.1, 25.3 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$): δ = -118.9 (d, *J* = 99.7 Hz, 1F), -119.0 (d, *J* = 96.7 Hz, 1F). $^{31}$P NMR (243 MHz, $CDCl_3$): δ = 4.18 (t, *J* = 99.7 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{23}ClF_2O_6PS^+$ $[M+H]^+$ 483.0604, found 483.0601.

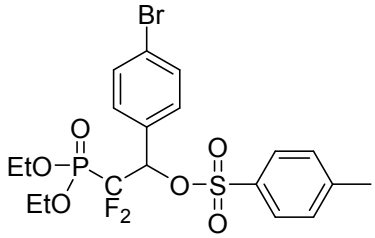



Compound **4ea**: 63% yield, white solid, 58-59 °C. $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.59 (d, *J* = 8.28 Hz, 2H), 7.27-7.25 (m, 1H), 7.23-7.16 (m, 5H), 5.80 (t, *J* = 12.48 Hz, 1H), 4.32-4.20 (m, 2H), 4.16-4.09 (m, 1H), 4.07-4.01 (m, 1H), 2.38 (s, 3H), 1.35 (t, *J* = 7.08 Hz, 3H), 1.23 (t, *J* = 7.2 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 145.2, 134.2, 133.3, 132.1, 129.8, 129.5, 129.4, 129.1, 127.9, 127.4, 118.7 (td, *J* = 268.3, 209.8 Hz), 79.4 (td, *J* = 25.1, 19.4 Hz), 65.2 (dd, *J* = 6.7, 55.1 Hz), 21.6, 16.3 (dd, *J* = 5.6, 26.1 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -118.8 (d, *J* = 97.0 Hz, 1F), -118.9 (d,

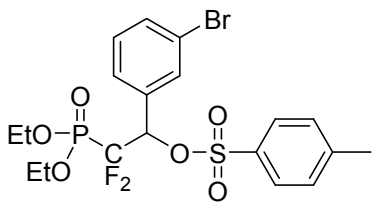
$J$ = 98.5 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.37 (t, $J$ = 98.3 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{23}ClF_2O_6PS^+$ $[M+H]^+$ 483.0604, found 483.0594.



Compound **4fa**: 83% yield, colorless oil. $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.65 (d, $J$ = 8.1 Hz, 2H), 7.41 (d, $J$ = 7.86 Hz, 1H), 7.29-7.28 (m, 1H), 7.23-7.20 (m, 1H), 7.15 (d, $J$ = 8.1 Hz, 2H), 7.12-7.09 (m, 1H), 6.45-6.41 (m, 1H), 4.32-4.21 (m, 4H), 2.35 (s, 3H), 1.37-1.33 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 145.1, 134.4, 133.0, 130.7, 130.6, 129.4, 129.2, 128.7, 128.1, 126.7, 118.6 (td, $J$ = 262.9, 211.9 Hz), 75.4-74.9 (m), 65.1 (t, $J$ = 5.9 Hz), 21.6, 16.3 (t, $J$ = 4.9 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$): δ = -115.9 (dd, $J$ = 99.8, 316.6 Hz, 1F), -120.9 (dd, $J$ = 102.4, 316.1 Hz, 1F). $^{31}$P NMR (243 MHz, $CDCl_3$): δ = 4.34 (t, $J$ = 99.9 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{23}ClF_2O_6PS^+$ $[M+H]^+$ 483.0604, found 483.0599.

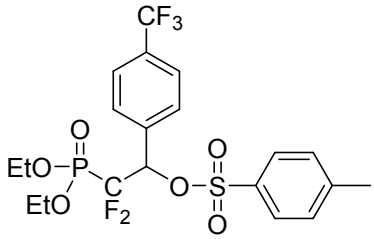


Compound **4ga**: 69% yield, white solid, 77-78 °C. $^1$H NMR (400 MHz, $CDCl_3$): δ = 7.60 (d, $J$ = 8.08 Hz, 2H), 7.38 (d, $J$ = 8.24 Hz, 2H), 7.19 (d, $J$ = 8.0 Hz, 4H), 5.81 (t, $J$ = 12.5 Hz, 1H), 4.31-4.19 (m, 2H), 4.15-4.00 (m, 2H), 2.40 (s, 3H), 1.35 (t, $J$ = 6.96 Hz, 3H), 1.23 (t, $J$ = 7.04 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 145.2, 133.4, 131.4, 130.7, 129.5, 129.4, 128.0, 124.2, 118.3-112.5 (m), 79.6 (td, $J$ = 24.4, 19.6 Hz), 65.3 (dd, $J$ = 6.6, 35.4 Hz), 21.6, 16.3 (dd, $J$ = 5.7, 16.7 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -119.0 (d, $J$ = 98.1 Hz, 2F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.37 (t, $J$ = 98.4 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{23}BrF_2O_6PS^+$ $[M+H]^+$ 527.0099, found 527.0098.

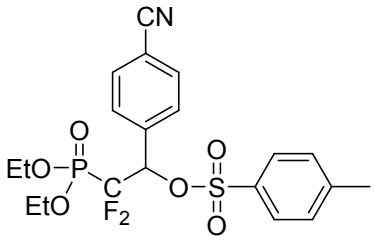


Compound **4ha**: 73% yield, white solid, 71-72 °C. $^1$H NMR (400 MHz, $CDCl_3$): δ = 7.58 (d, $J$ =

8.48 Hz, 2H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.31-7.26 (m, 2H), 7.17-7.10 (m, 3H), 5.79 (t, *J* = 12.2 Hz, 1H), 4.34-4.20 (m, 2H), 4.16-3.99 (m, 2H), 2.38 (s, 3H), 1.36 (t, *J* = 7.08 Hz, 3H), 1.24 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 145.3, 133.3, 132.7, 132.3, 131.9, 129.7, 129.5, 127.9, 122.2, 119.9 (td, *J* = 267.4, 209.7 Hz), 79.5 (td, *J* = 24.9, 19.0 Hz), 65.3 (dd, *J* = 6.9, 36.6 Hz), 21.6, 16.3 (dd, *J* = 5.7, 16.9 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -118.8 (d, *J* = 96.8 Hz, 1F), -119.0 (d, *J* = 99.7 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.37 (t, *J* = 96.6 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{23}BrF_2O_6PS^+$ $[M+H]^+$ 527.0099, found 527.0095.

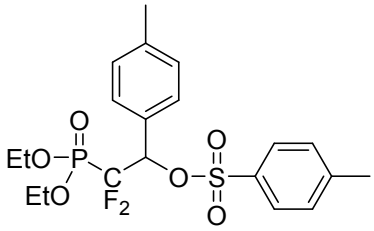


Compound **4ia**: 61% yield, colorless oil. $^1$H NMR (400 MHz, $CDCl_3$): δ = 7.58 (d, *J* = 8.08 Hz, 2H), 7.49 (d, *J* = 8.24 Hz, 2H), 7.44 (d, *J* = 8.16 Hz, 2H), 7.15 (d, *J* = 8.0 Hz, 2H), 5.91 (t, *J* = 12.12 Hz, 1H), 4.32-4.21 (m, 2H), 4.16-4.02 (m, 2H), 2.36 (s, 3H), 1.36 (t, *J* = 7.0 Hz, 3H), 1.22 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 145.3, 134.2, 133.2, 131.9, 131.6, 129.6, 129.5, 128.0, 125.1 (q, *J* = 4.2 Hz), 122.3, 79.5 (td, *J* = 24.6, 18.8 Hz), 65.3 (dd, *J* = 6.8, 38.1 Hz), 21.4, 16.3 (dd, *J* = 5.7, 22.0 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -63.0 (s, 3F), -118.8 (d, *J* = 95.7 Hz, 1F), -119.1 (d, *J* = 99.5 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.21 (t, *J* = 98.3 Hz, 1P). HRMS (ESI): calculated for $C_{20}H_{23}F_5O_6PS^+$ $[M+H]^+$ 517.0868, found 517.0863.

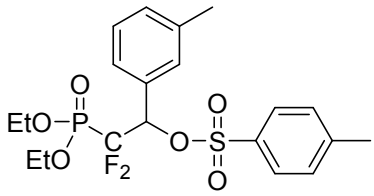


Compound **4ja**: 71% yield, colorless oil. $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.64 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 8.34 Hz, 2H), 7.48 (d, *J* = 8.22 Hz, 2H), 7.22 (d, *J* = 8.04 Hz, 2H), 5.88-5.84 (m, 1H), 4.28-4.19 (m, 2H), 4.16-4.05 (m, 2H), 2.41 (s, 3H), 1.34 (t, *J* = 7.08 Hz, 3H), 1.23 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 145.6, 135.6, 133.0, 131.8, 129.7, 129.6, 128.1, 118.0, 117.2 (d, *J* = 269.9 Hz), 113.6, 79.0-78.5 (m), 65.4 (dd, *J* = 6.7, 43.9 Hz), 21.6, 16.3 (dd, *J* = 5.5, 20.0 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$): δ = -117.9 (dd, *J* = 95.5, 310.5 Hz, 1F), -119.0 (dd, *J* = 98.8, 311.0 Hz, 1F). $^{31}$P NMR (243 MHz, $CDCl_3$): δ = 3.75 (t, *J* = 96.7 Hz, 1P). HRMS (ESI): calculated

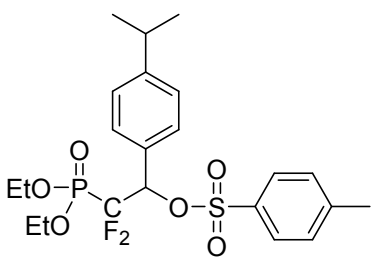
for $C_{20}H_{23}F_2NO_6PS^+$ $[M+H]^+$ 474.0946, found 474.0940.



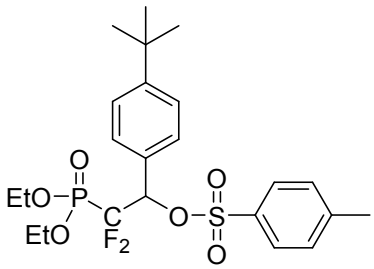
Compound **4ka**: 65% yield, white solid, 51-52 °C. $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.60 (d, *J* = 8.34 Hz, 2H), 7.21 (d, *J* = 8.1 Hz, 2H), 7.16 (d, *J* = 8.1 Hz, 2H), 7.05 (d, *J* = 7.92 Hz, 2H), 5.82 (t, *J* = 12.6 Hz, 1H), 4.29-4.17 (m, 2H), 4.11-4.05 (m, 1H), 3.99-3.93 (m, 1H), 2.37 (s, 3H), 2.31 (s, 3H), 1.33 (t, *J* = 7.14 Hz, 3H), 1.20 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 144.7, 139.8, 133.7, 129.4, 129.1, 128.8, 128.0, 127.3, 119.0 (td, *J* = 269.1, 209.7 Hz), 80.3 (td, *J* = 25.3, 21.6 Hz), 65.1 (dd, *J* = 7.0, 52.2 Hz), 21.6, 21.2, 16.3 (dd, *J* = 5.6, 26.5 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -117.6 (dd, *J* = 100.1, 307.6 Hz, 1F), -119.0 (dd, *J* = 96.9, 307.5 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.70 (t, *J* = 97.4 Hz, 1P). HRMS (ESI): calculated for $C_{20}H_{26}F_2O_6PS^+$ $[M+H]^+$ 463.1150, found 463.1146.



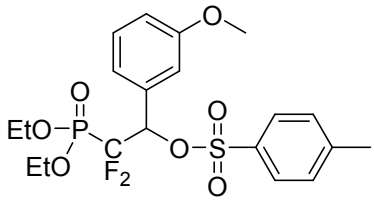
Compound **4la**: 68% yield, colorless oil. $^1$H NMR (400 MHz, $CDCl_3$): δ = 7.58 (d, *J* = 7.92 Hz, 2H), 7.15-7.08 (m, 5H), 7.05 (s, 1H), 5.82 (t, *J* = 12.84 Hz, 1H), 4.33-4.18 (m, 2H), 4.14-4.04 (m, 1H), 4.01-3.92 (m, 1H), 2.37 (s, 3H), 2.23 (s, 3H), 1.36 (t, *J* = 6.88 Hz, 3H), 1.21 (t, *J* = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 144.7, 137.8, 133.7, 130.4, 130.0, 129.7, 129.3, 128.1, 128.0, 126.4, 119.0 (td, *J* = 267.1, 209.5 Hz), 80.4 (td, *J* = 23.2, 19.7 Hz), 65.1 (dd, *J* = 6.7, 57.9 Hz), 21.5, 21.1, 16.3 (dd, *J* = 5.5, 27.5 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -118.6 (d, *J* = 99.8 Hz, 1F), -118.9 (d, *J* = 97.2 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.73 (t, *J* = 98.2 Hz, 1P). HRMS (ESI): calculated for $C_{20}H_{26}F_2O_6PS^+$ $[M+H]^+$ 463.1150, found 463.1156.



Compound **4ma**: 66% yield, colorless oil. $^{1}$H NMR (400 MHz, $CDCl_3$): δ = 7.55 (d, *J* = 8.08 Hz, 2H), 7.21 (d, *J* = 7.92 Hz, 2H), 7.11-7.04 (m, 4H), 5.84 (t, *J* = 12.8 Hz, 1H), 4.33-4.17 (m, 2H), 4.10-4.00 (m, 1H), 3.96-3.86 (m, 1H), 2.90-2.80 (m, 1H), 2.34 (s, 3H), 1.34 (t, *J* = 7.16 Hz, 3H), 1.21 (d, *J* = 6.96 Hz, 6H), 1.18 (t, *J* = 7.12 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 150.7, 144.5, 133.8, 129.3, 128.0, 127.4, 126.2, 119.1 (td, *J* = 266.2, 208.9 Hz), 80.5 (td, *J* = 25.0, 21.1 Hz), 65.1 (dd, *J* = 6.5, 70.5 Hz), 33.9, 23.9, 23.8, 21.5, 16.3 (dd, *J* = 5.6, 29.8 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -117.6 (dd, *J* = 101.1, 307.4 Hz, 1F), -119.3 (dd, *J* = 96.1, 307.1 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.73 (dd, *J* = 96.7, 102.0 Hz, 1P). HRMS (ESI): calculated for $C_{22}H_{30}F_2O_6PS^+$ $[M+H]^+$ 491.1463, found 491.1465.

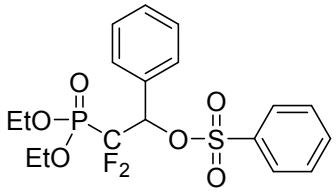


Compound **4na**: 46% yield, white solid, 56-57 °C. $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.55 (d, *J* = 8.4 Hz, 2H), 7.21 (s, 4H), 7.10 (d, *J* = 8.04 Hz, 2H), 5.84-5.80 (m, 1H), 4.31-4.21 (m, 2H), 4.09-4.02 (m, 1H), 3.95-3.88 (m, 1H), 2.35 (s, 3H), 1.35 (t, *J* = 7.08 Hz, 3H), 1.28 (s, 9H), 1.17 (t, *J* = 7.02 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 152.9, 144.4, 133.8, 129.3, 129.0, 128.0, 127.0, 125.0, 117.7 (d, *J* = 269.5 Hz), 80.5 (td, *J* = 20.7, 25.4 Hz), 65.1 (dd, *J* = 6.6, 49.4 Hz), 34.6, 31.2, 21.5, 16.3 (dd, *J* = 5.7, 20.9 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -117.6 (dd, *J* = 100.5, 307.0 Hz, 1F), -119.3 (dd, *J* = 96.2, 307.4 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.73 (dd, *J* = 96.4, 100.3 Hz, 1P). HRMS (ESI): calculated for $C_{23}H_{32}F_2O_6PS^+$ $[M+H]^+$ 505.1620, found 505.1620.

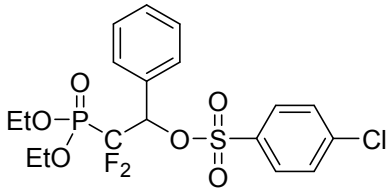


Compound **4oa**: 65% yield, colorless oil. $^{1}$H NMR (600 MHz, $CDCl_3$): δ = 7.58 (d, *J* = 7.98 Hz, 2H), 7.16-7.13 (m, 3H), 6.93-6.77 (m, 3H), 5.82 (d, *J* = 12.42 Hz, 1H), 4.30-4.20 (m, 2H), 4.13-4.07 (m, 1H), 4.02-3.96 (m, 1H), 3.70 (s, 3H), 2.36 (s, 3H), 1.34 (t, *J* = 6.9 Hz, 3H), 1.21 (t, *J* = 6.9 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 159.3, 144.8, 133.6, 131.4, 129.4, 129.2, 128.0, 121.7, 117.2 (d, *J* = 209.4 Hz), 115.8, 114.0, 80.2 (td, *J* = 24.5, 19.8 Hz), 65.1 (dd, *J* = 6.8, 53.9 Hz), 55.2,

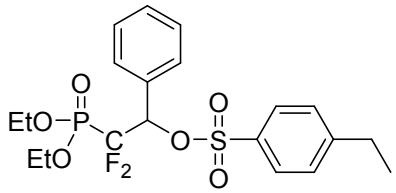
21.5, 16.3 (dd, $J$ = 6.0, 25.0 Hz). $^{19}$F NMR (565 MHz, $CDCl_3$): δ = -118.7 (d, $J$ = 99.1 Hz, 2F). $^{31}$P NMR (243 MHz, $CDCl_3$): δ = 4.46 (t, $J$ = 99.5 Hz, 1P). HRMS (ESI): calculated for $C_{20}H_{26}F_2O_7PS^+$ $[M+H]^+$ 479.1099, found 479.1096.



Compound **4ab**: 74% yield, colorless oil. $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.71 (d, $J$ = 7.56 Hz, 2H), 7.52-7.49 (m, 1H), 7.36-7.28 (m, 5H), 7.24-7.21 (m, 2H), 5.89 (t, $J$ = 12.66 Hz, 1H), 4.30-4.18 (m, 2H), 4.10-4.04 (m, 1H), 3.98-3.91 (m, 1H), 1.34 (t, $J$ = 7.02 Hz, 3H), 1.19 (t, $J$ = 7.02 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 136.6, 133.7, 130.1, 129.9, 129.2, 128.8, 128.2, 127.9, 119.0 (td, $J$ = 270.3, 209.9 Hz), 80.3 (td, $J$ =20.8, 25.9 Hz), 65.1 (dd, $J$ = 6.6, 59.6 Hz), 16.3 (dd, $J$ = 5.6, 27.4 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -117.7 (dd, $J$ = 100.8, 308.5 Hz, 1F), -119.1 (dd, $J$ = 95.5, 307.6 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.55 (dd, $J$ = 94.5, 97.8 Hz, 1P). HRMS (ESI): calculated for $C_{18}H_{22}F_2O_6PS^+$ $[M+H]^+$ 435.0837, found 435.0828.

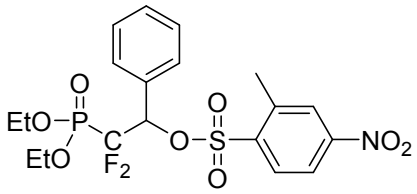


Compound **4ac**: 45% yield, white solid, 65-66 °C. $^1$H NMR (400 MHz, $CDCl_3$): δ = 7.63 (d, $J$ = 8.6 Hz, 2H), 7.36-7.24 (m, 7H), 5.89 (t, $J$ = 12.68 Hz, 1H), 4.33-4.17 (m, 2H), 4.12-4.02 (m, 1H), 3.99-3.89 (m, 1H), 1.35 (t, $J$ = 6.92 Hz, 3H), 1.19 (t, $J$ = 7.08 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 140.4, 135.1, 130.0, 129.9, 129.3, 129.2, 129.1, 128.3, 118.9 (td, $J$ = 266.4, 209.2 Hz), 80.8 (td, $J$ =20.9, 25.2 Hz), 65.2 (dd, $J$ = 7.1, 64.8 Hz), 16.3 (dd, $J$ = 5.6, 27.6 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -117.7 (dd, $J$ = 99.9, 307.6 Hz, 1F), -119.4 (dd, $J$ = 94.6, 307.6 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.42 (dd, $J$ = 96.0, 100.3 Hz, 1P). HRMS (ESI): calculated for $C_{18}H_{21}ClF_2O_6PS^+$ $[M+H]^+$ 469.0448, found 469.0442.

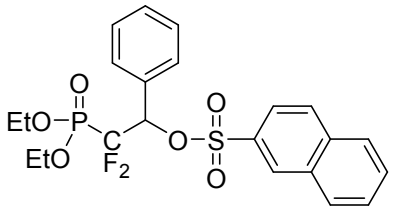


Compound **4ad**: 65% yield, colorless oil. $^1$H NMR (600 MHz, $CDCl_3$): δ = 7.61 (d, $J$ = 8.4 Hz, 2H),

7.31-7.27 (m, 3H), 7.23-7.20 (m, 2H), 7.15 (d, $J$ = 8.34 Hz, 2H), 5.86 (t, $J$ = 12.6 Hz, 1H), 4.31-4.18 (m, 2H), 4.10-4.04 (m, 1H), 3.99-3.92 (m, 1H), 2.66 (q, $J$ = 7.68 Hz, 2H), 1.34 (t, $J$ = 7.08 Hz, 3H), 1.22-1.17 (m, 6H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 150.9, 133.7, 130.2, 129.7, 129.2, 128.3, 128.2, 128.1, 119.0 (td, $J$ = 265.9, 209.0 Hz), 80.3 (td, $J$ =20.3, 25.2 Hz), 65.1 (dd, $J$ = 6.6, 59.2 Hz), 28.9, 16.3 (dd, $J$ = 5.5, 27.4 Hz), 15.2. $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -117.8 (dd, $J$ = 100.9, 308.7 Hz, 1F), -119.0 (dd, $J$ = 96.5, 308.4 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.64 (t, $J$ = 99.4 Hz, 1P). HRMS (ESI): calculated for $C_{20}H_{26}F_2O_6PS^+$ $[M+H]^+$ 463.1150, found 463.1145.

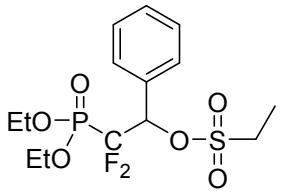


Compound **4ae**: 42% yield, yellow oil. $^1$H NMR (400 MHz, $CDCl_3$): δ = 8.42 (d, $J$ = 2.4 Hz, 1H), 8.19-8.16 (m, 1H), 7.40-7.29 (m, 3H), 7.24-7.16 (m, 3H), 5.90 (t, $J$ = 12.76 Hz, 1H), 4.30-4.18 (m, 2H), 4.09-4.00 (m, 1H), 3.95-3.85 (m, 1H), 2.76 (s, 3H), 1.34 (t, $J$ = 7.2 Hz, 3H), 1.19 (t, $J$ = 7.08 Hz, 3H). $^{13}$C NMR (100 MHz, $CDCl_3$): δ = 145.7, 145.6, 137.3, 133.3, 130.3, 129.3, 129.2, 128.4, 127.6, 124.7, 118.2-112.8 (m), 82.0 (td, $J$ =21.6, 24.5 Hz), 65.3 (dd, $J$ = 6.6, 49.9 Hz), 20.7, 16.3 (dd, $J$ = 5.5, 18.0 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -116.5 (dd, $J$ = 100.5, 306.3 Hz, 1F), -120.7 (dd, $J$ = 94.1, 306.9 Hz, 1F). $^{31}$P NMR (162 MHz, $CDCl_3$): δ = 4.19 (dd, $J$ = 93.9, 99.8 Hz, 1P). HRMS (ESI): calculated for $C_{19}H_{23}F_2NO_8PS^+$ $[M+H]^+$ 494.0845, found 494.0841.



Compound **4af**: 30% yield, colorless oil. $^1$H NMR (600 MHz, $CDCl_3$): δ = 8.24 (d, $J$ = 1.74 Hz, 1H), 7.86-7.79 (m, 3H), 7.69-7.64 (m, 2H), 7.61-7.58 (m, 1H), 7.32 (d, $J$ = 6.84 Hz, 2H), 7.16-7.10 (m, 3H), 5.95 (t, $J$ = 12.54 Hz, 1H), 4.30-4.18 (m, 2H), 4.09-4.03 (m, 1H), 3.97-3.90 (m, 1H), 1.32 (t, $J$ = 7.08 Hz, 3H), 1.18 (t, $J$ = 7.02 Hz, 3H). $^{13}$C NMR (150 MHz, $CDCl_3$): δ = 135.1, 133.4, 131.6, 130.0, 129.9, 129.8, 129.4, 129.3, 129.2, 128.1, 127.8, 127.5, 122.5, 119.0 (td, $J$ = 267.2, 209.0 Hz), 80.6 (td, $J$ =20.8, 25.0 Hz), 65.1 (dd, $J$ = 6.6, 63.8 Hz), 16.3 (dd, $J$ = 5.5, 26.4 Hz). $^{19}$F NMR (376 MHz, $CDCl_3$): δ = -117.6 (dd, $J$ = 100.1, 307.7 Hz, 1F), -119.2 (dd, $J$ = 95.6, 307.7 Hz, 1F). $^{31}$P

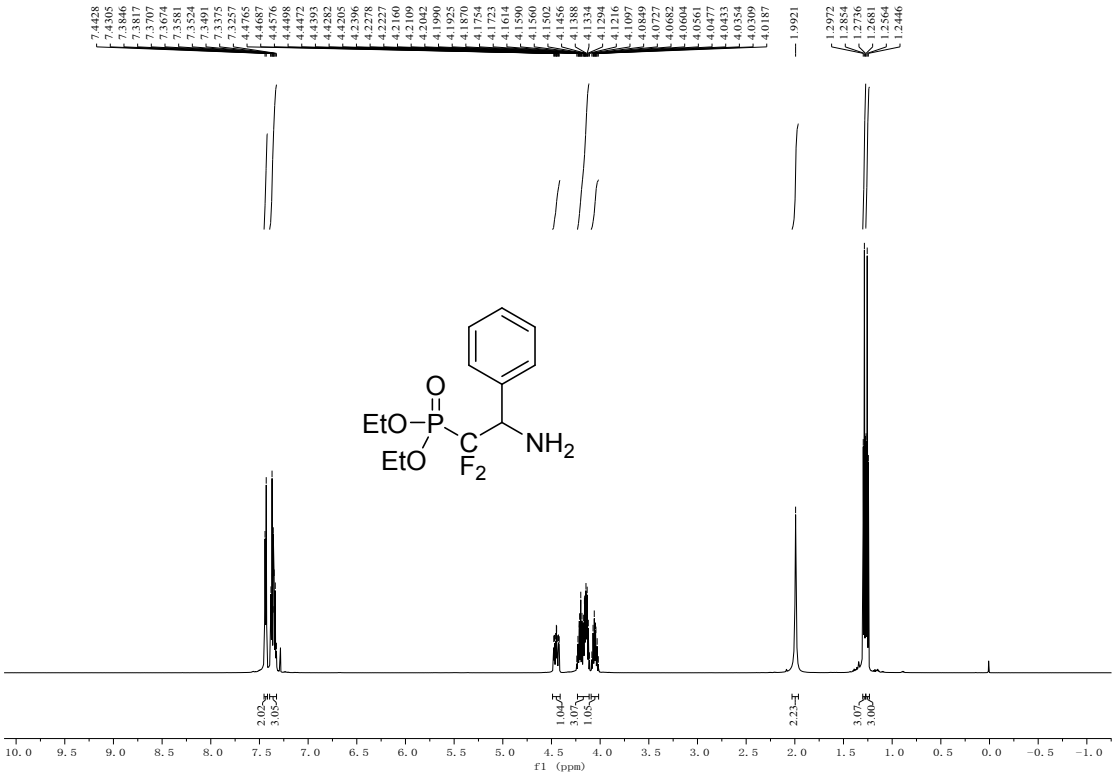
NMR (162 MHz, $CDCl_3$): δ = 4.59 (dd, *J* = 96.4, 102.3 Hz, 1P). HRMS (ESI): calculated for $C_{22}H_{24}F_2O_6PS^+$ $[M+H]^+$ 485.0994, found 485.0994.



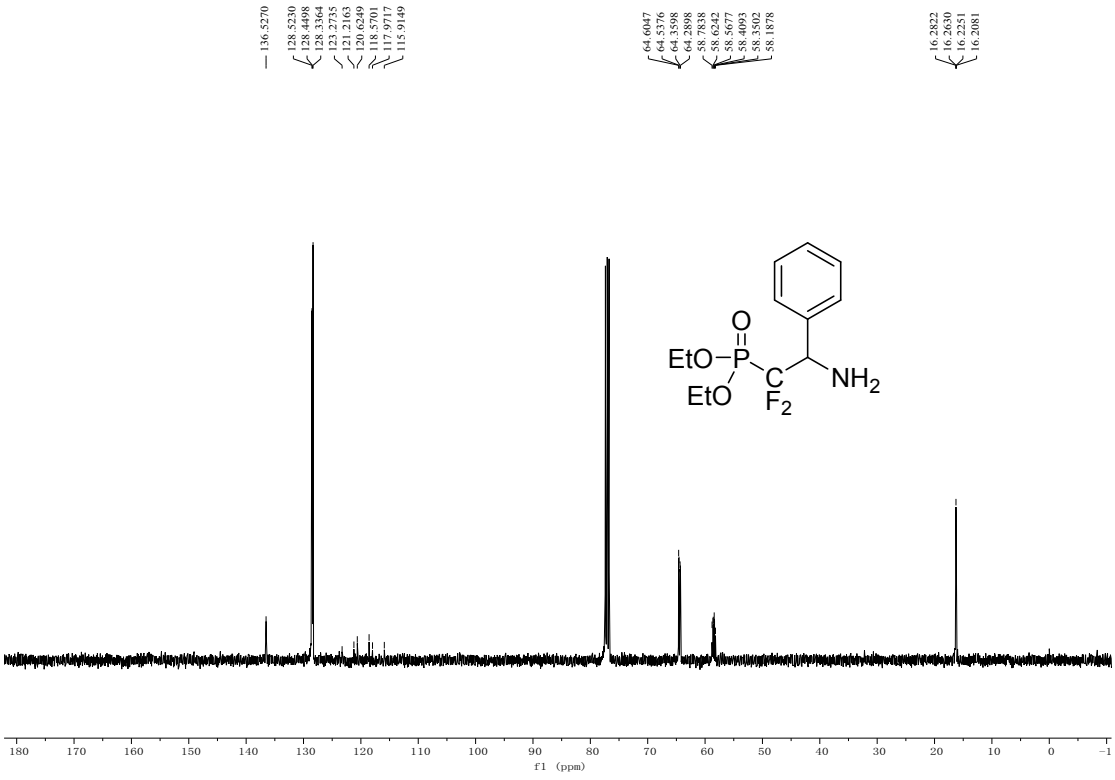
Compound **4ah**: 85% yield, colorless oil. $^1H$ NMR (400 MHz, $CDCl_3$): δ = 7.56-7.54 (m, 2H), 7.46-7.43 (m, 3H), 5.92 (t, *J* = 12.8 Hz, 1H), 4.35-4.19 (m, 2H), 4.15-4.06 (m, 1H), 4.04-3.94 (m, 1H), 1.37 (t, *J* = 7.28 Hz, 6H), 1.23 (t, *J* = 7.08 Hz, 3H). $^{13}C$ NMR (150 MHz, $CDCl_3$): δ = 130.9, 130.3, 129.1, 128.6, 128.1, 119.2-116.0 (m), 80.0 (td, *J* =21.4, 24.9 Hz), 65.3 (dd, *J* = 6.9, 71.4 Hz), 46.9, 16.3 (dd, *J* = 5.5, 23.2 Hz), 7.9. $^{19}F$ NMR (376 MHz, $CDCl_3$): δ = -117.1 (dd, *J* = 101.3, 306.5 Hz, 1F), -119.3 (dd, *J* = 94.8, 306.9 Hz, 1F). $^{31}P$ NMR (162 MHz, $CDCl_3$): δ = 4.48 (dd, *J* = 96.2, 102.2 Hz, 1P). HRMS (ESI): calculated for $C_{14}H_{22}F_2O_6PS^+$ $[M+H]^+$ 387.0837, found 387.0832.

## 9. $^{1}H$, $^{13}C$, $^{19}F$ and $^{31}P$ NMR spectra of compounds 1 and 4

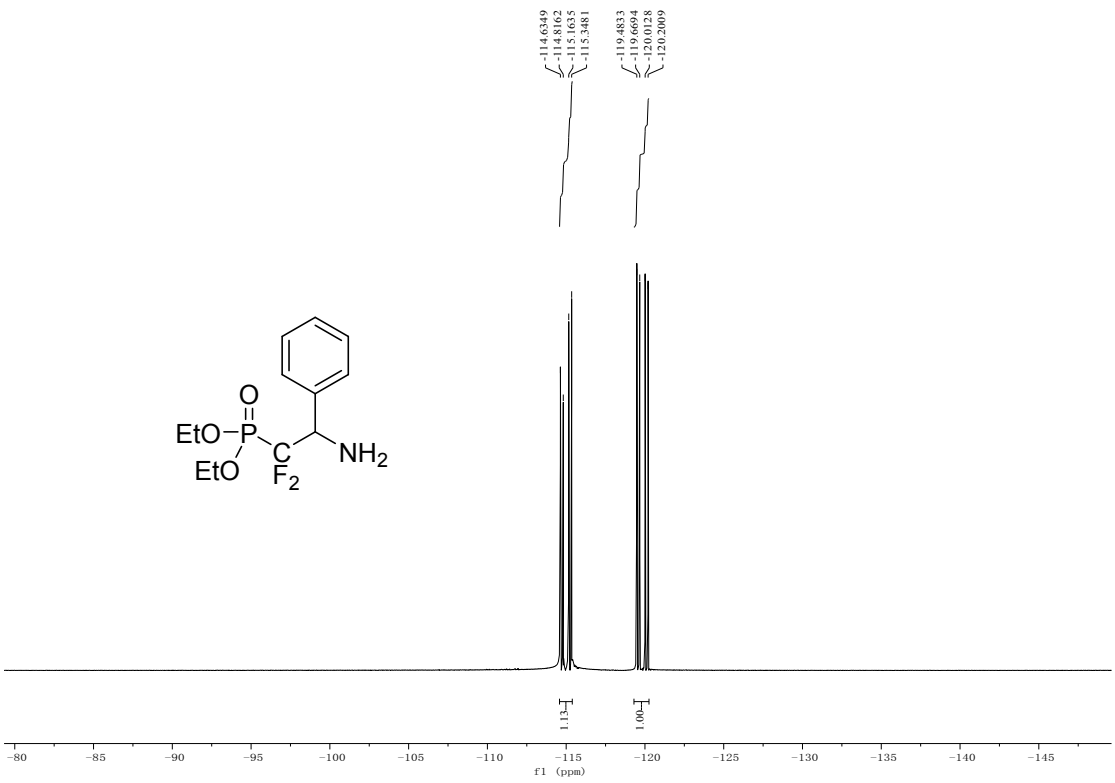
$^{1}H$ NMR (600 MHz, $CDCl_3$) of **1a**:



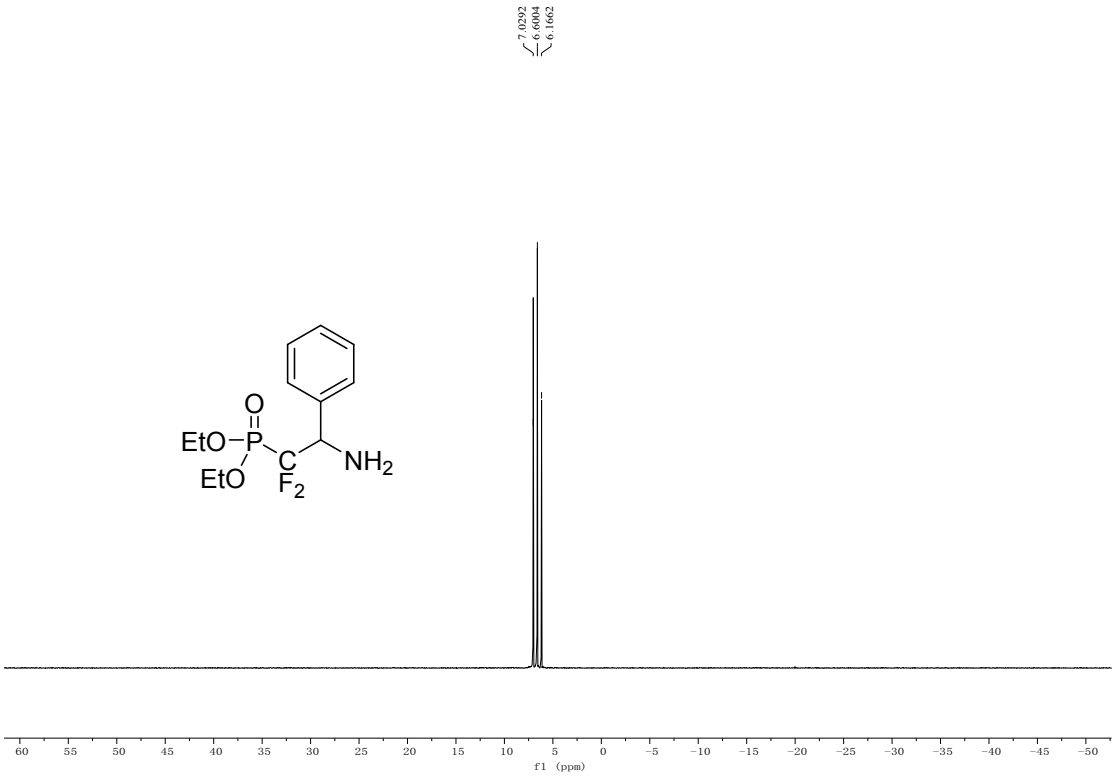

$^{13}C$ NMR (100 MHz, $CDCl_3$) of **1a**:



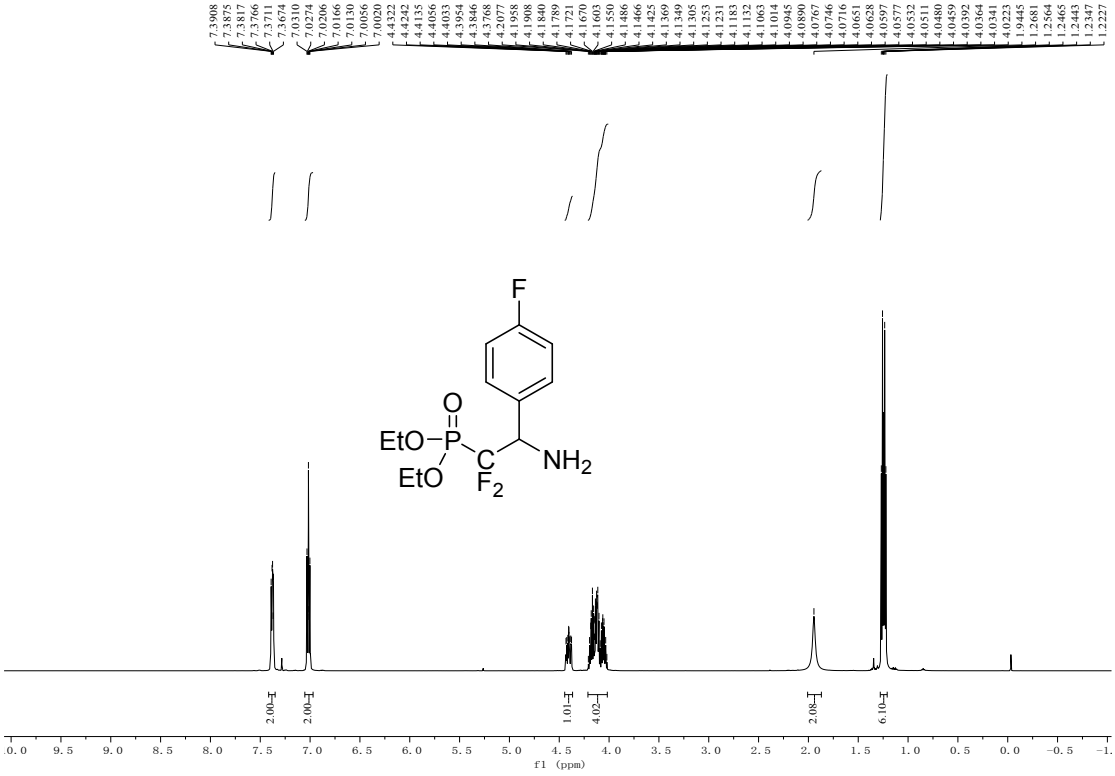
$^{19}F$ NMR (565 MHz, $CDCl_3$) of **1a**:



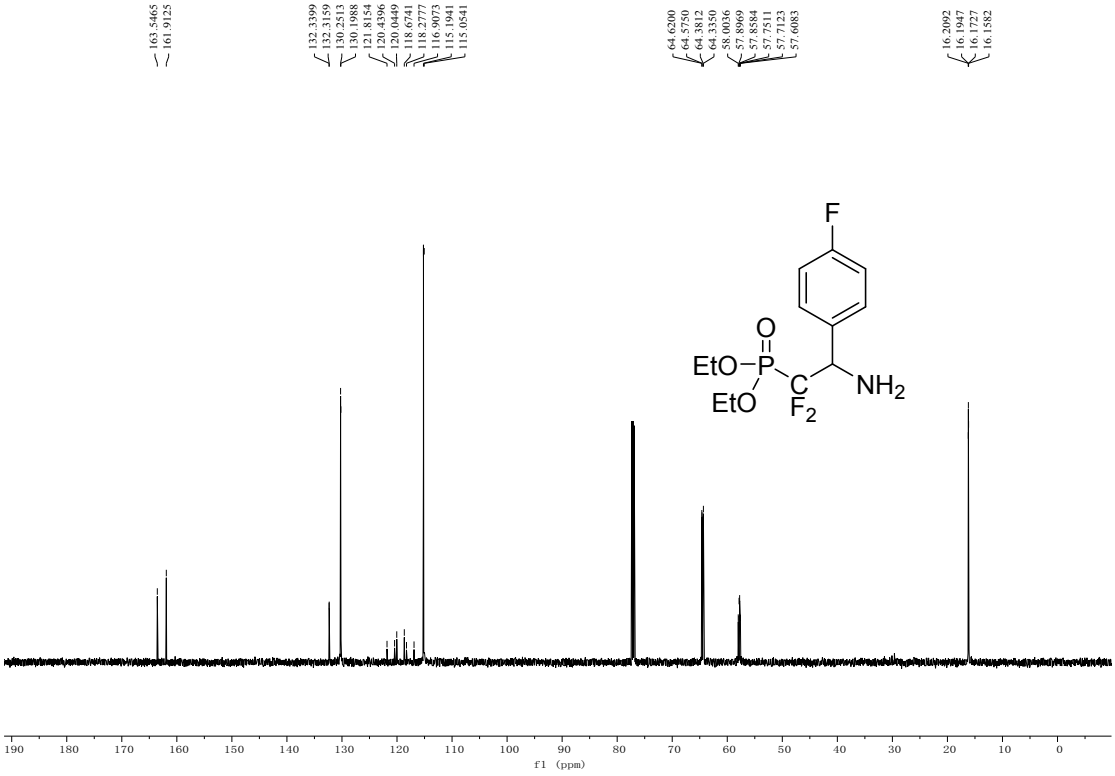

$^{31}P$ NMR (243 MHz, $CDCl_3$) of **1a**:



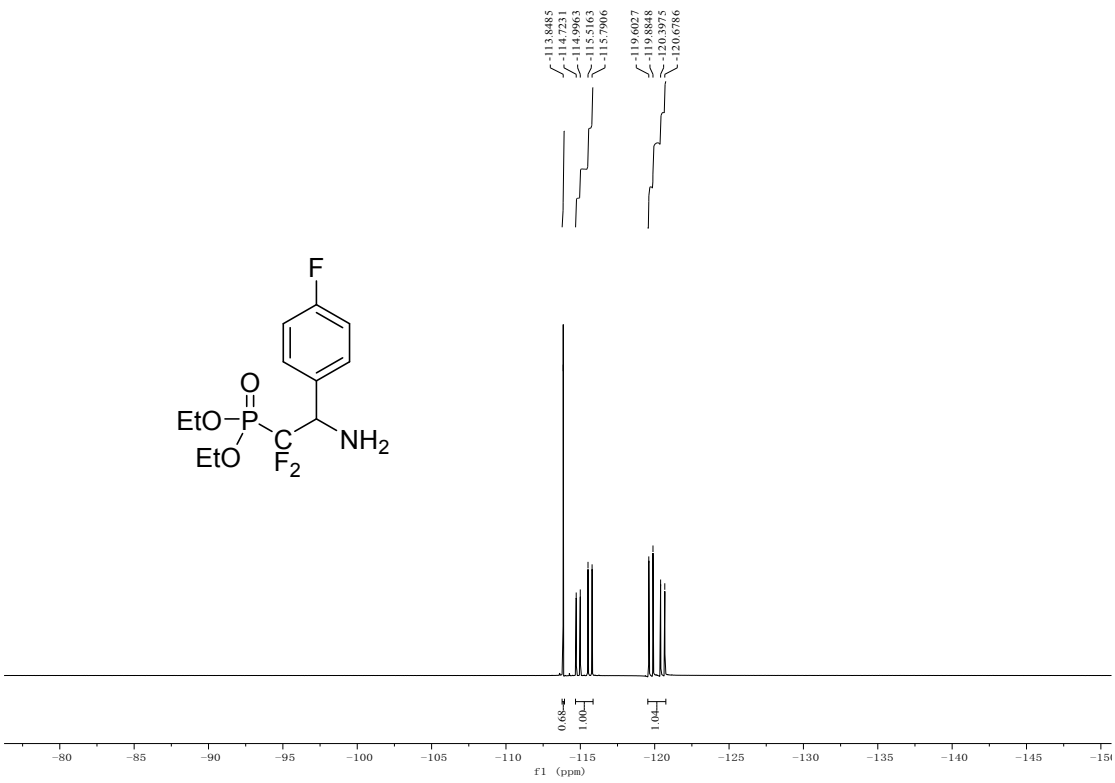
$^{1}$H NMR (600 MHz, $CDCl_3$) of **1b**:



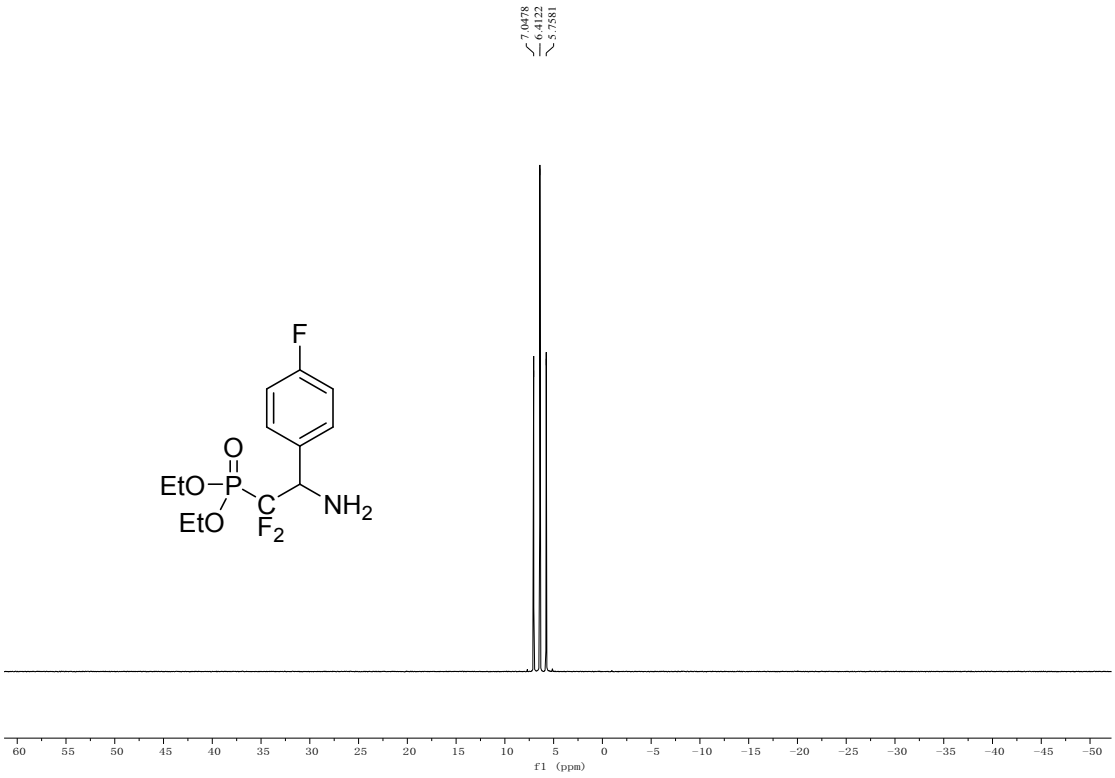

$^{13}$C NMR (150 MHz, $CDCl_3$) of **1b**:



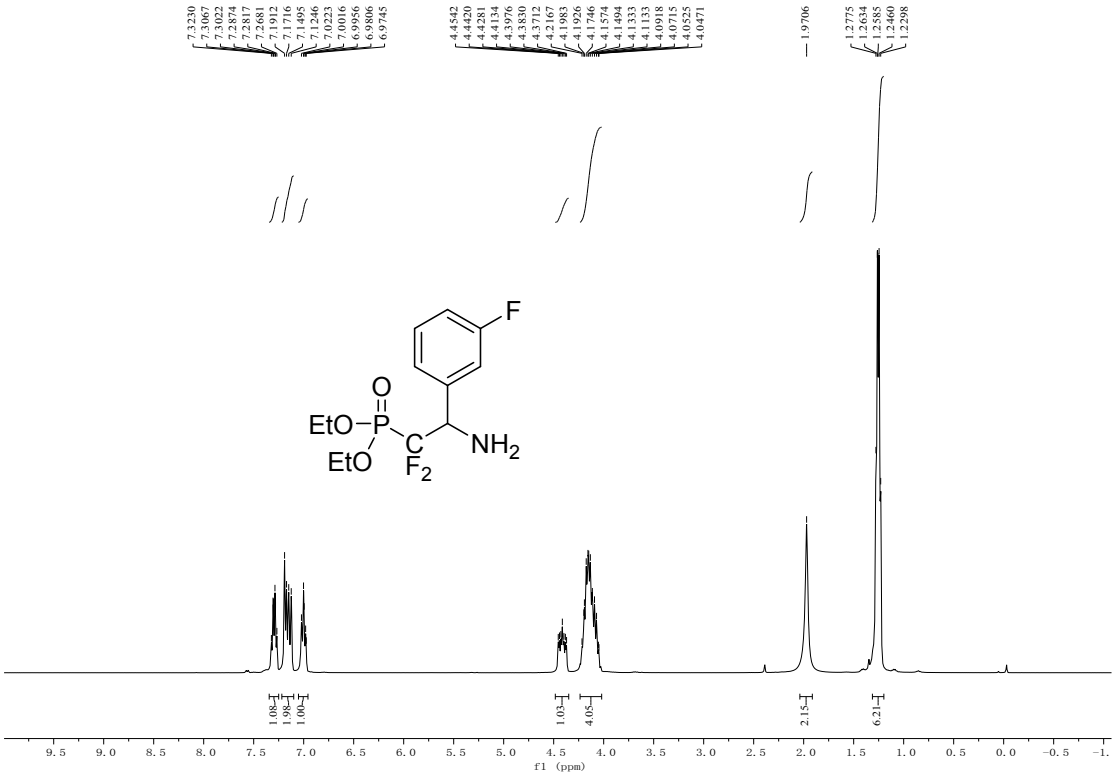
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **1b**:



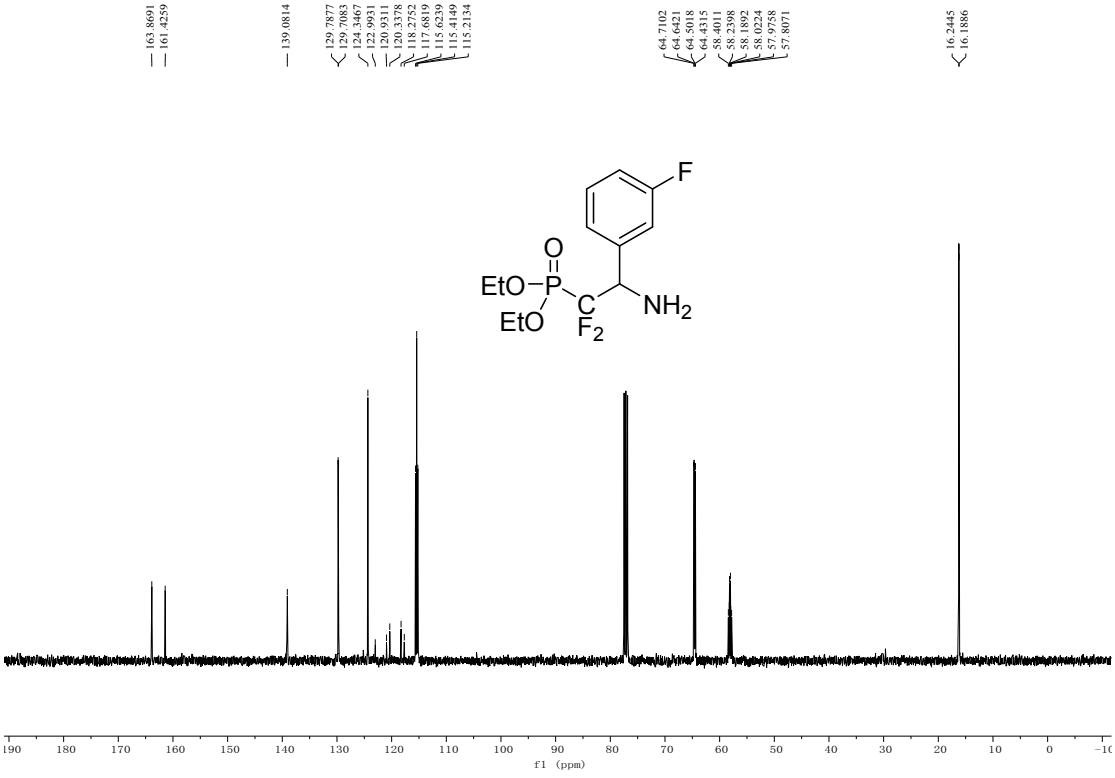

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **1b**:



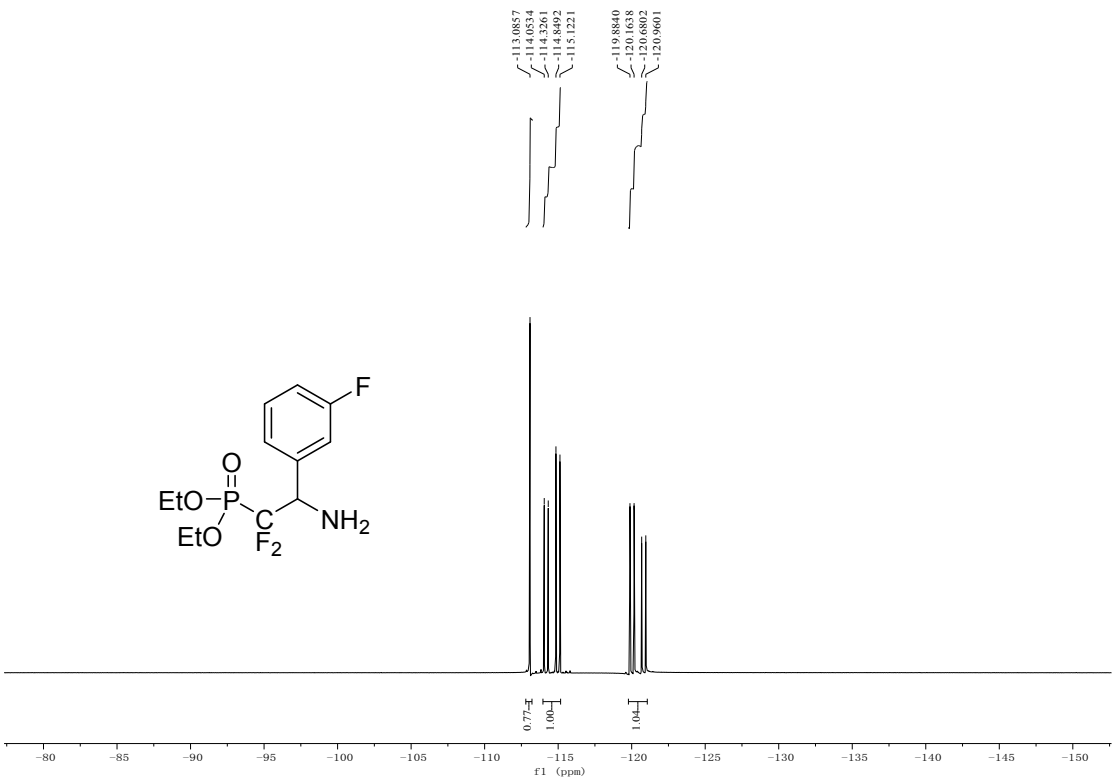
$^{1}$H NMR (400 MHz, $CDCl_3$) of **1c**:



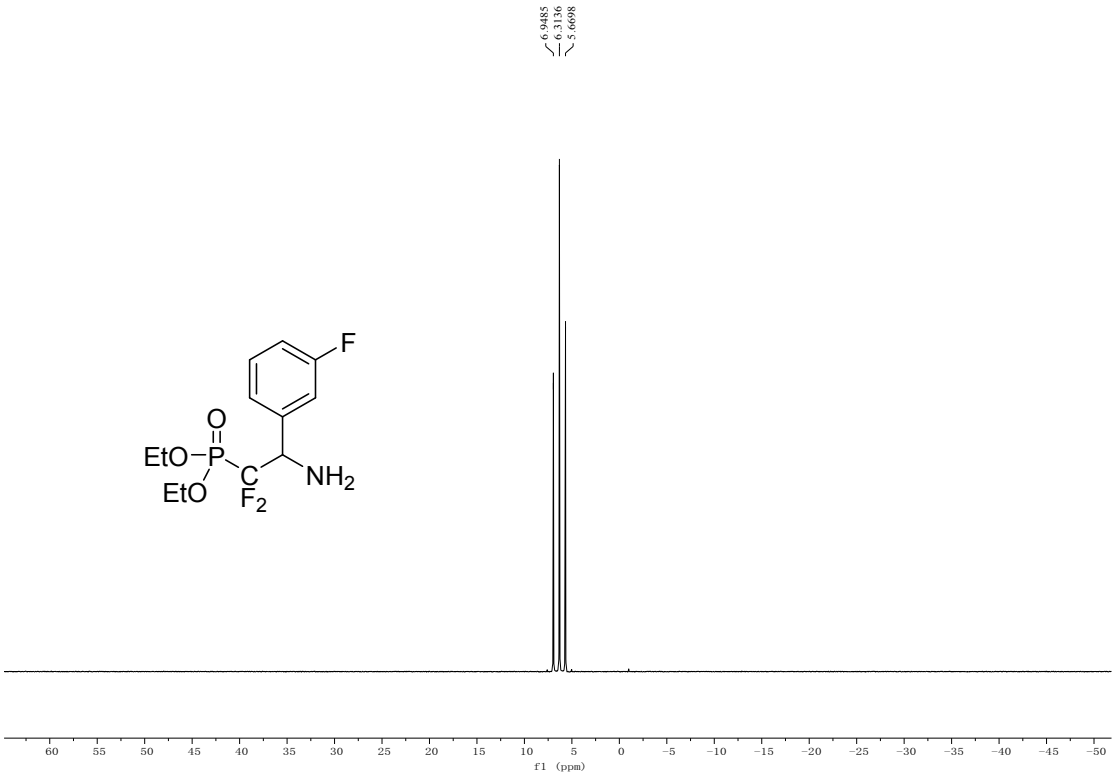

$^{13}$C NMR (100 MHz, $CDCl_3$) of **1c**:



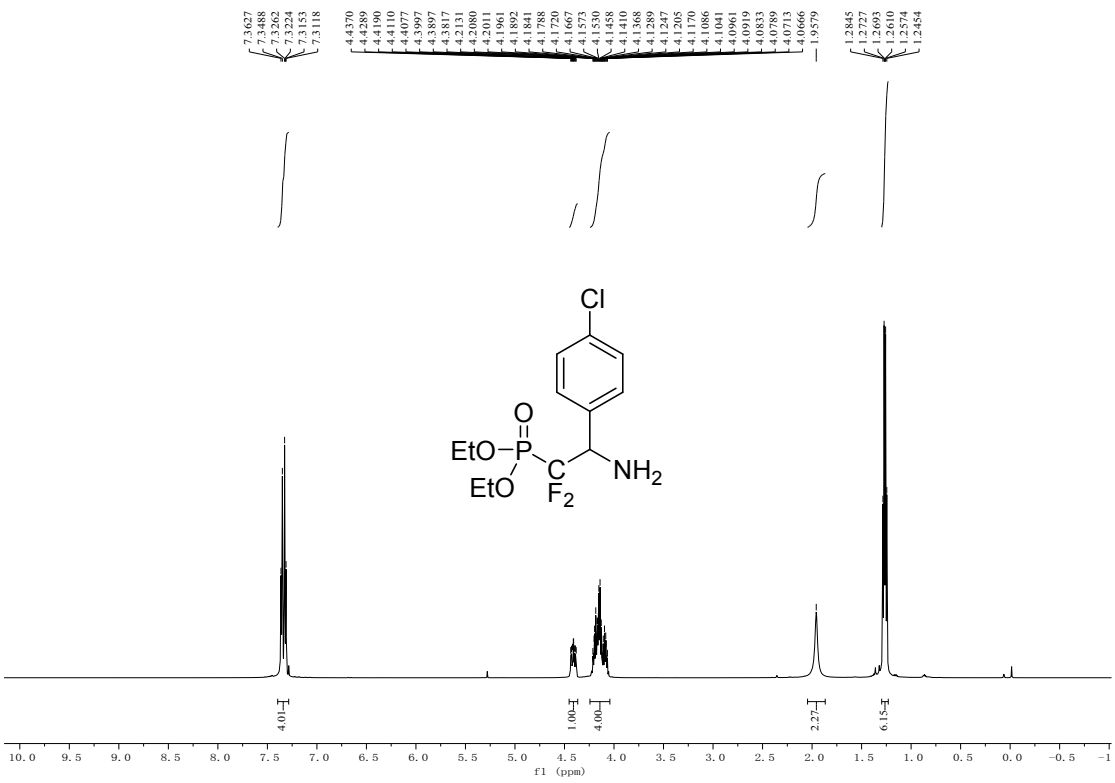
$^{19}$F NMR (376 MHz, $CDCl_3$) of **1c**:



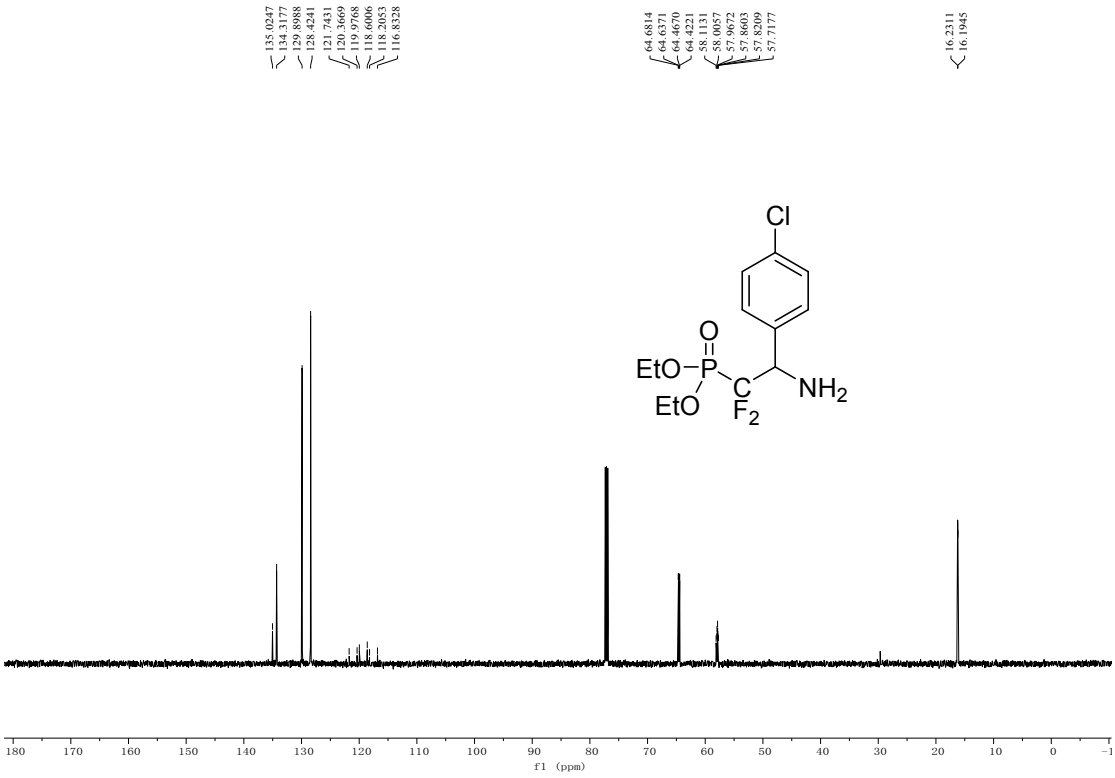

$^{31}$P NMR (162 MHz, $CDCl_3$) of **1c**:



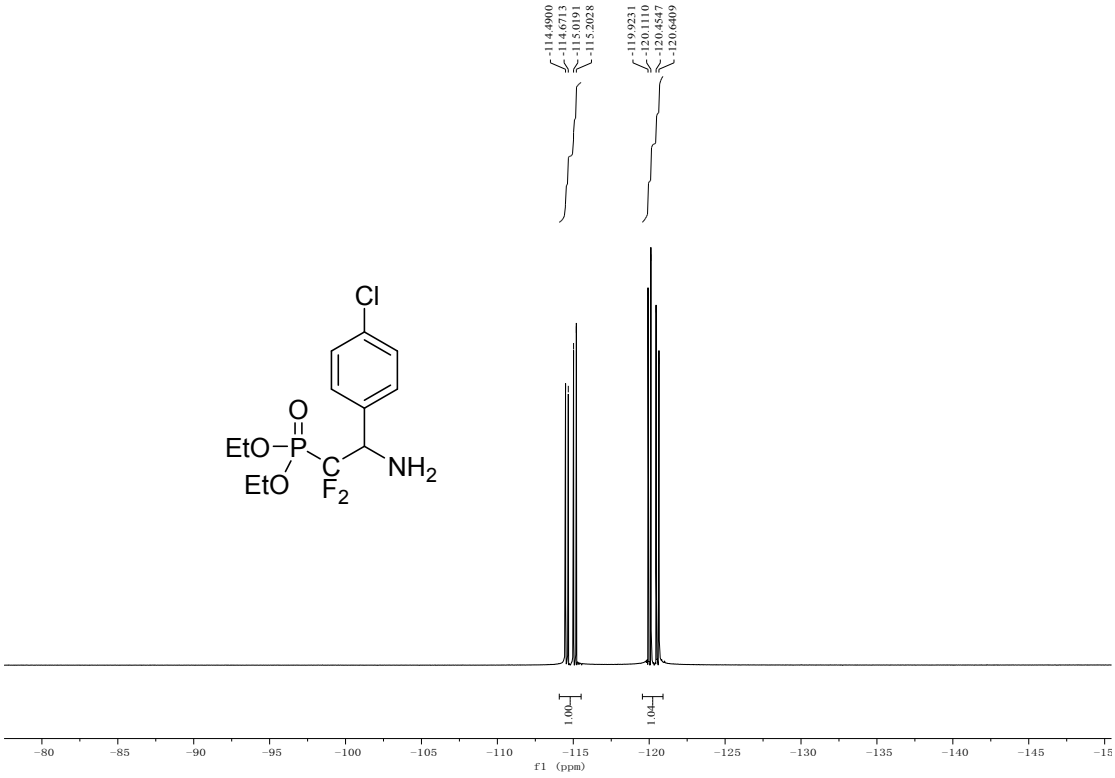
$^{1}$H NMR (600 MHz, $CDCl_3$) of **1d**:



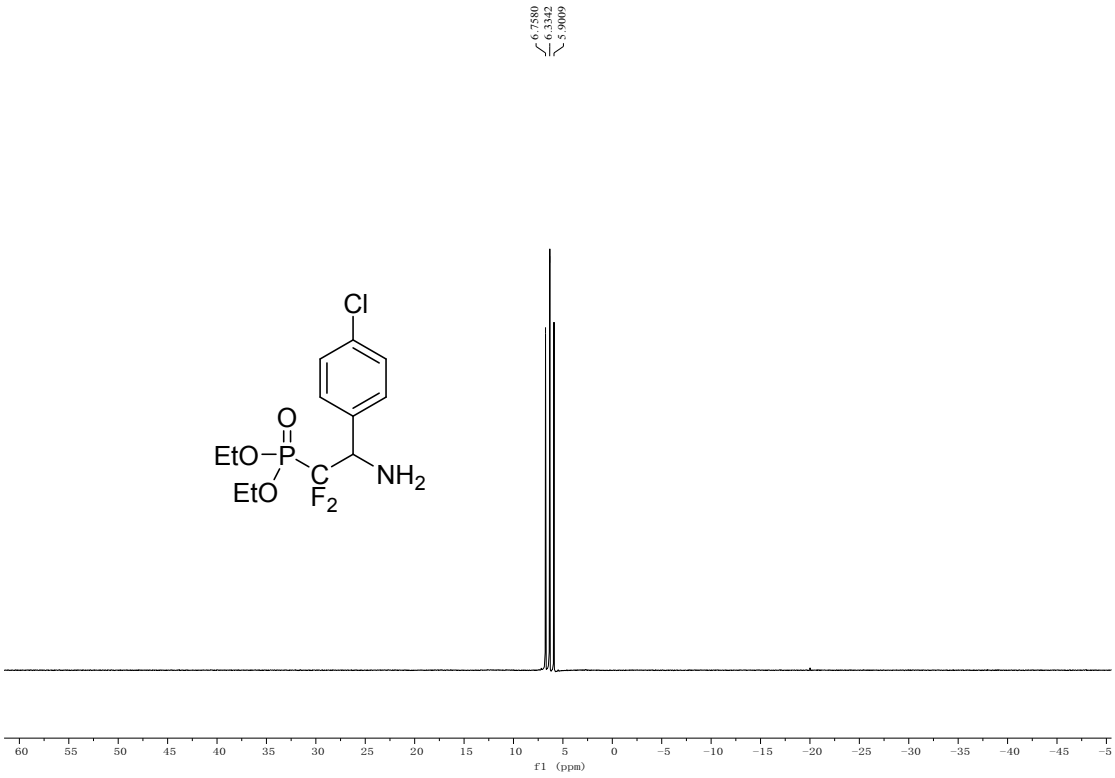

$^{13}$C NMR (150 MHz, $CDCl_3$) of **1d**:



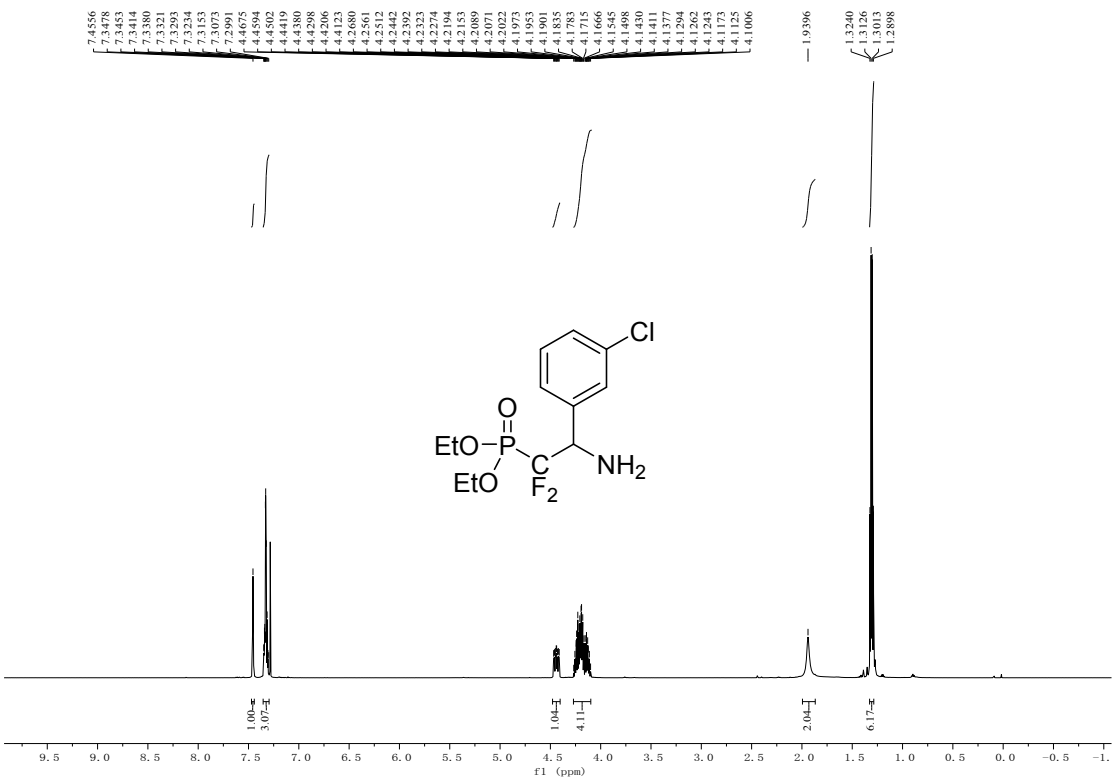
$^{19}F$ NMR (565 MHz, $CDCl_3$) of **1d**:



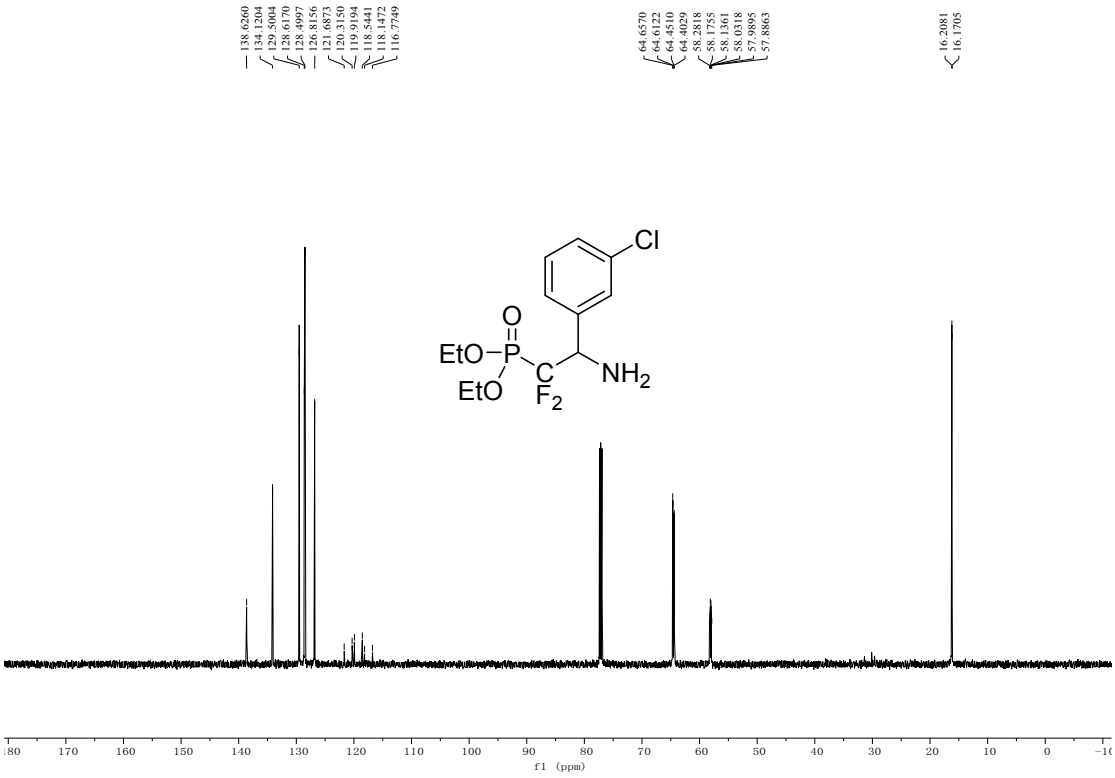

$^{31}P$ NMR (243 MHz, $CDCl_3$) of **1d**:



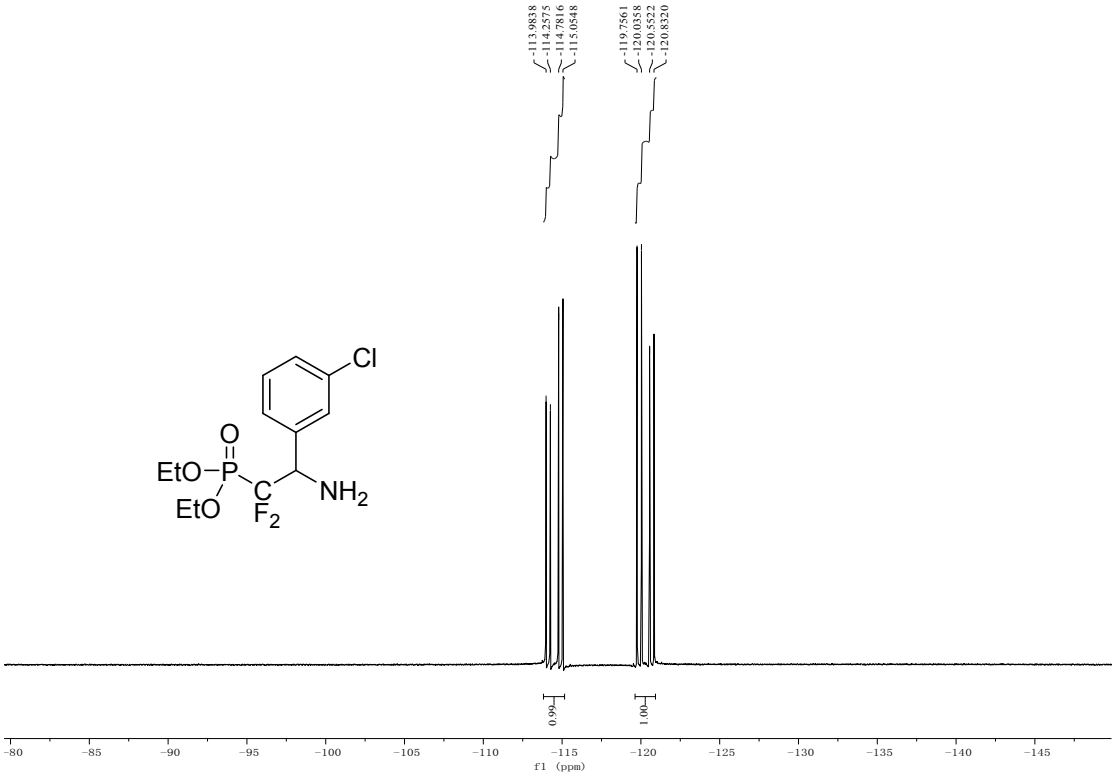
$^{1}$H NMR (600 MHz, $CDCl_3$) of **1e**:



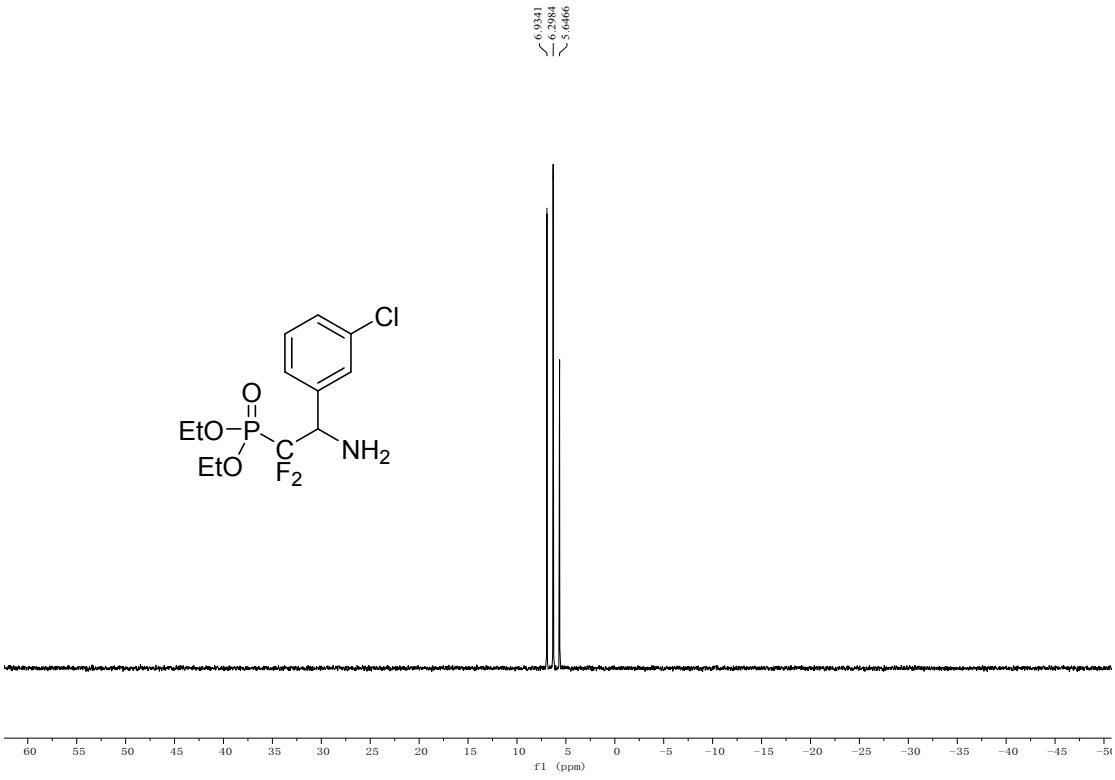

$^{13}$C NMR (150 MHz, $CDCl_3$) of **1e**:



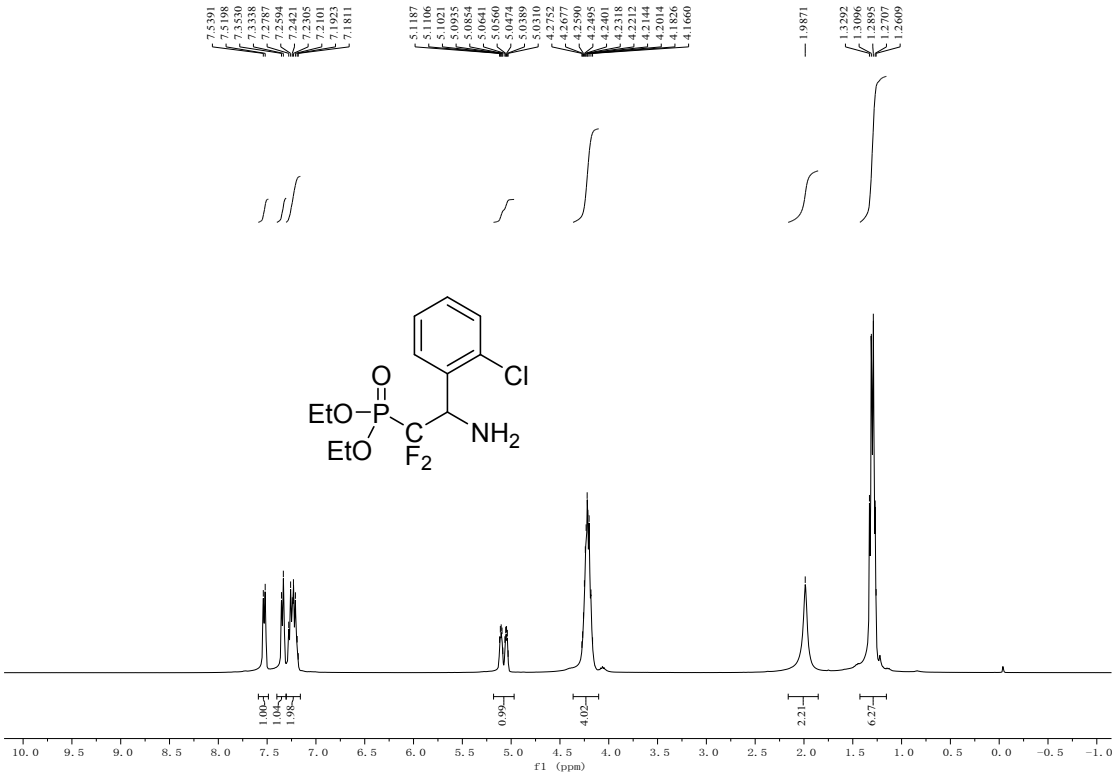
$^{19}$F NMR (376 MHz, $CDCl_3$) of **1e**:



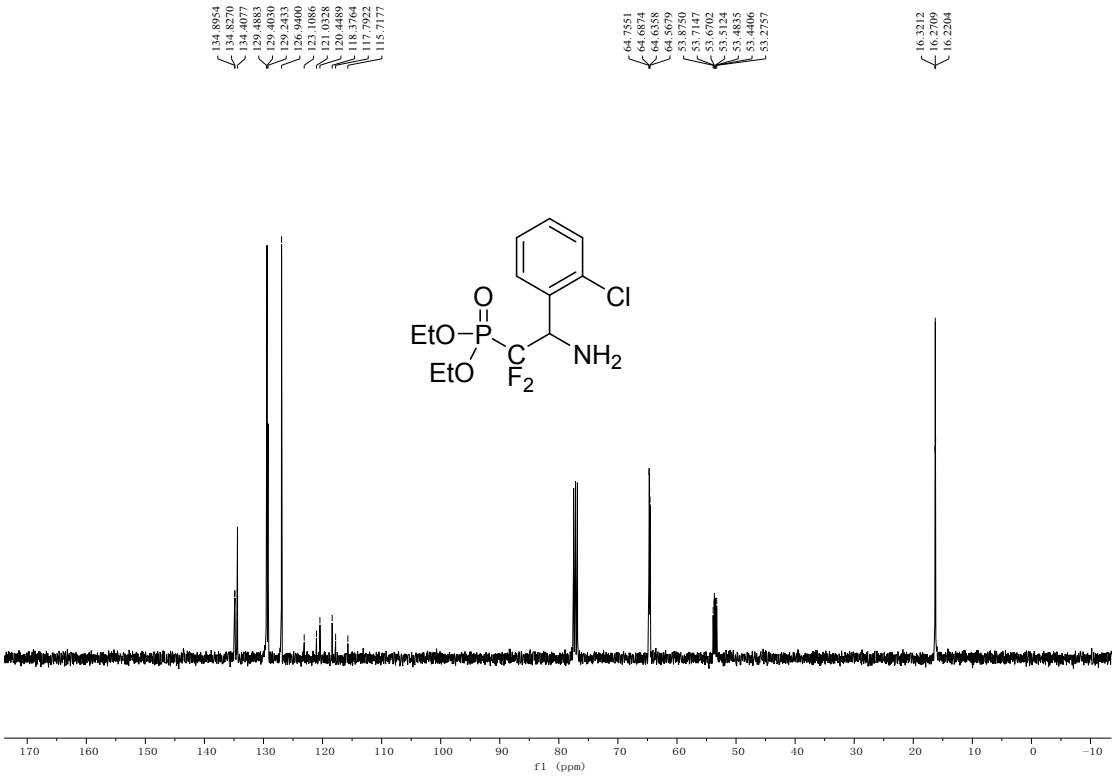

$^{31}$P NMR (162 MHz, $CDCl_3$) of **1e**:



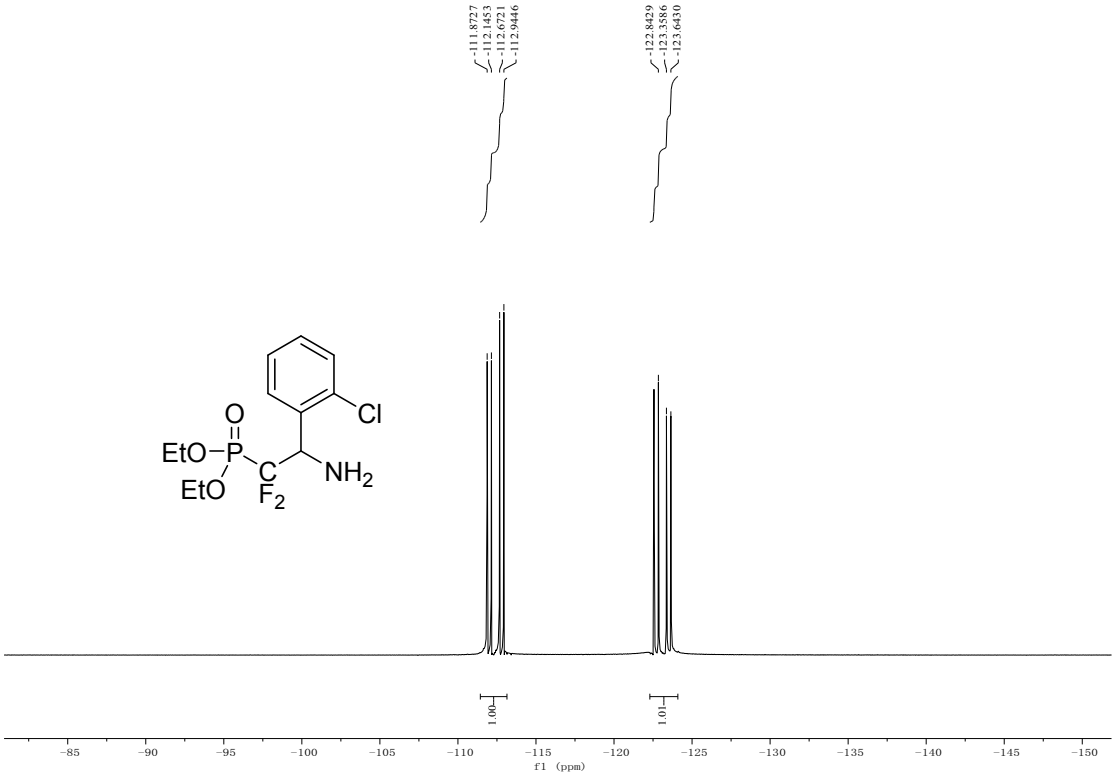
$^{1}$H NMR (400 MHz, $CDCl_3$) of **1f**:



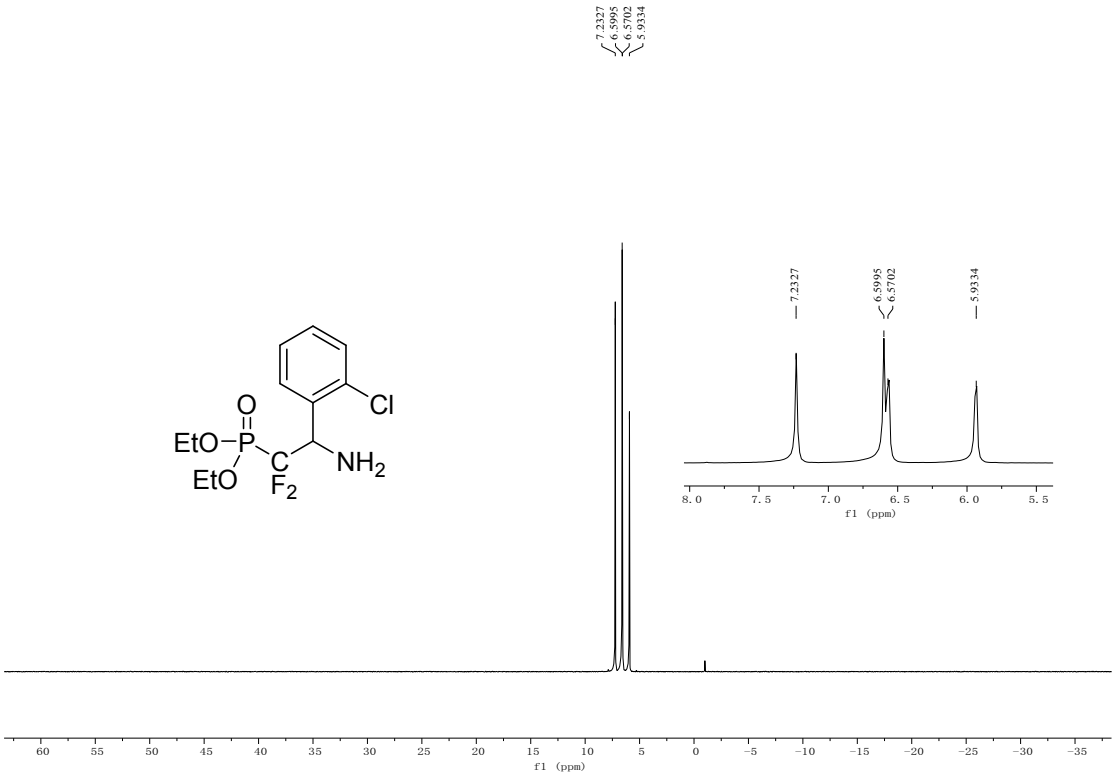

$^{13}$C NMR (100 MHz, $CDCl_3$) of **1f**:



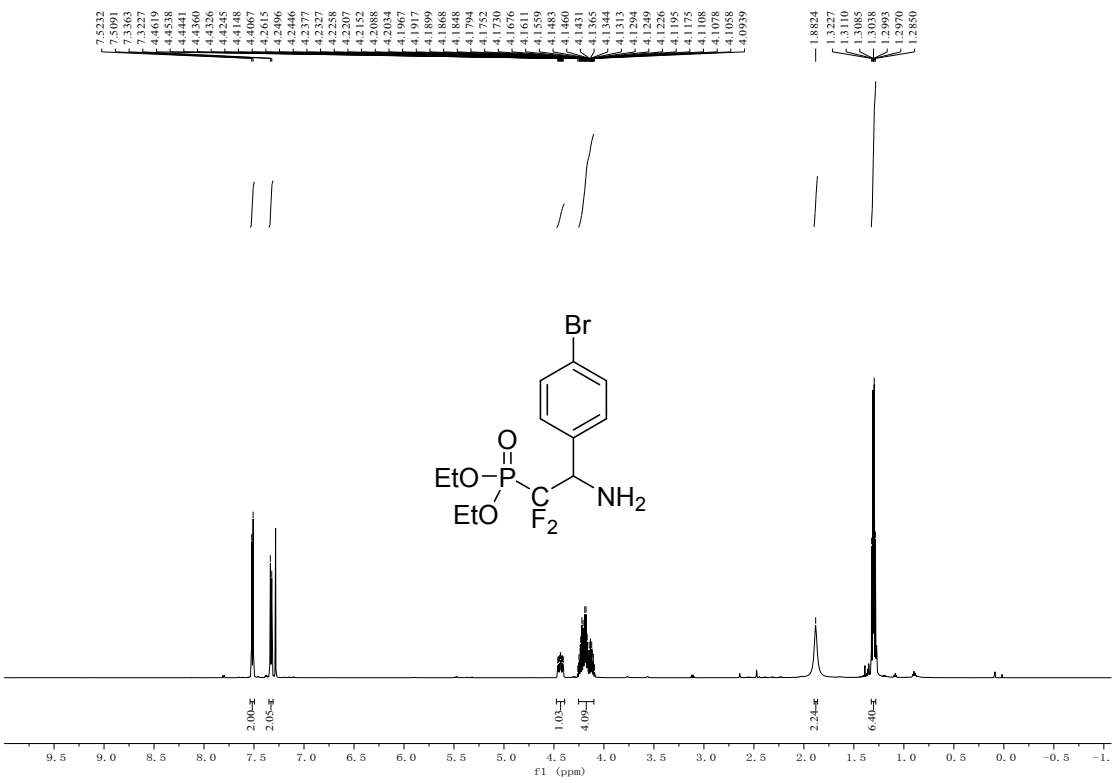
$^{19}$F NMR (376 MHz, $CDCl_3$) of **1f**:



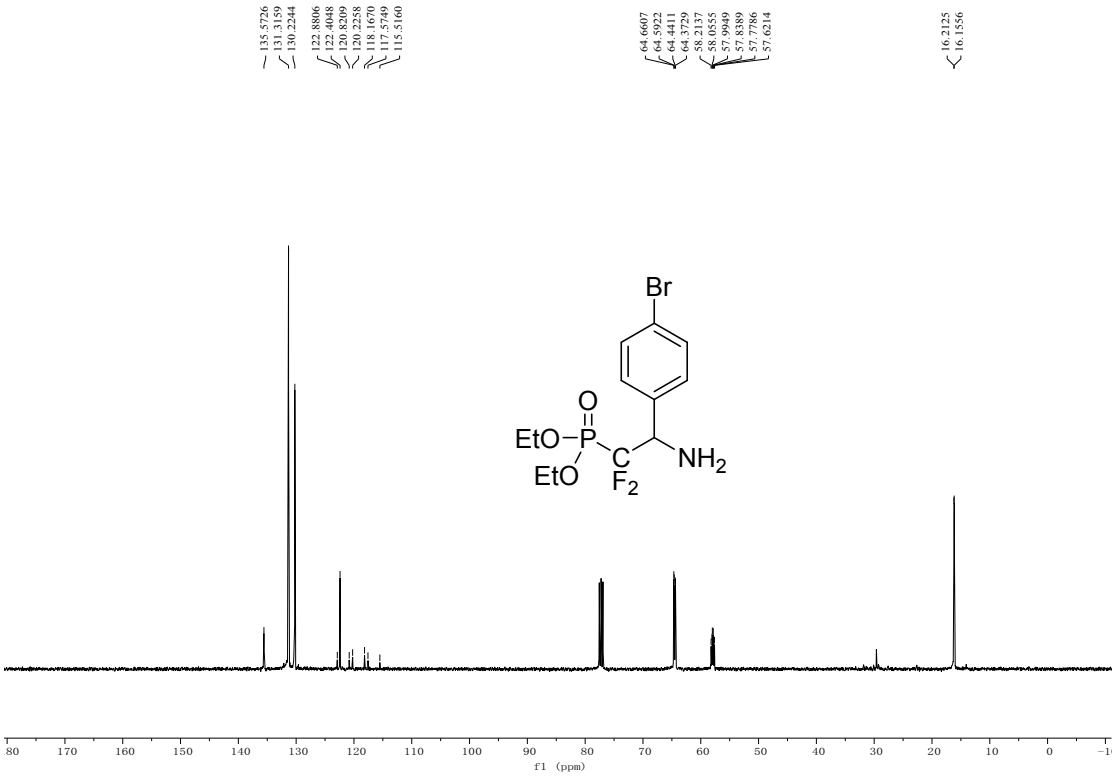

$^{31}$P NMR (162 MHz, $CDCl_3$) of **1f**:



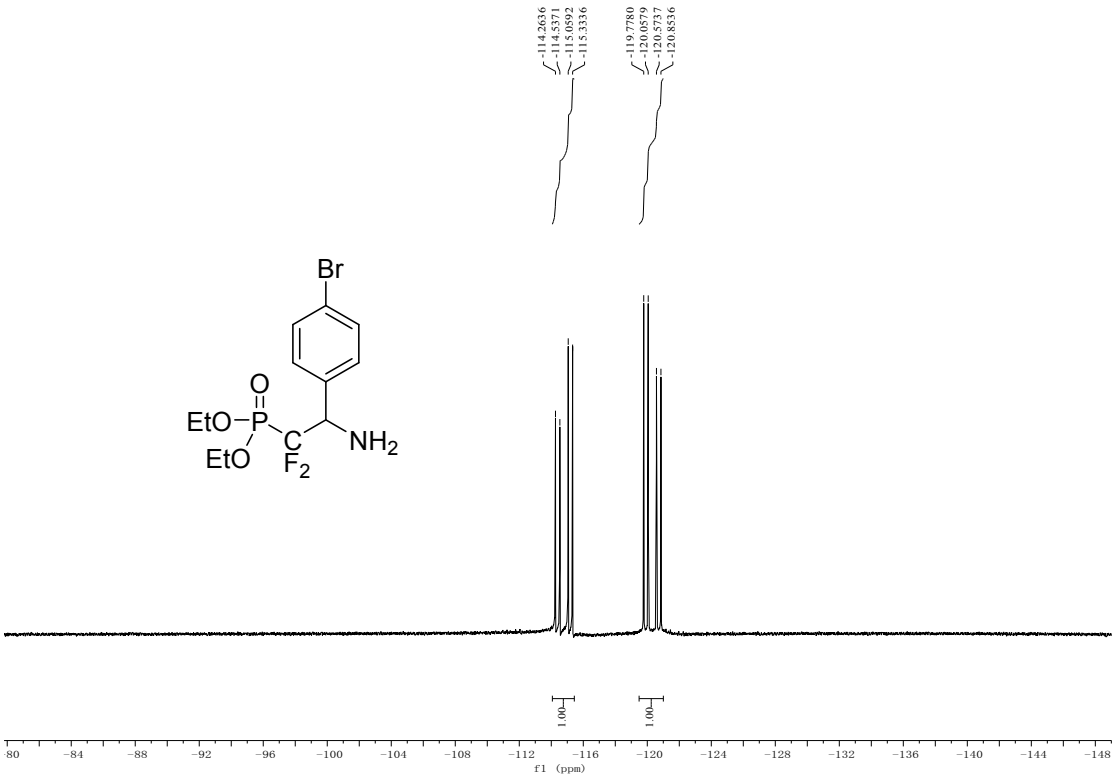
$^{1}$H NMR (600 MHz, $CDCl_3$) of **1g**:



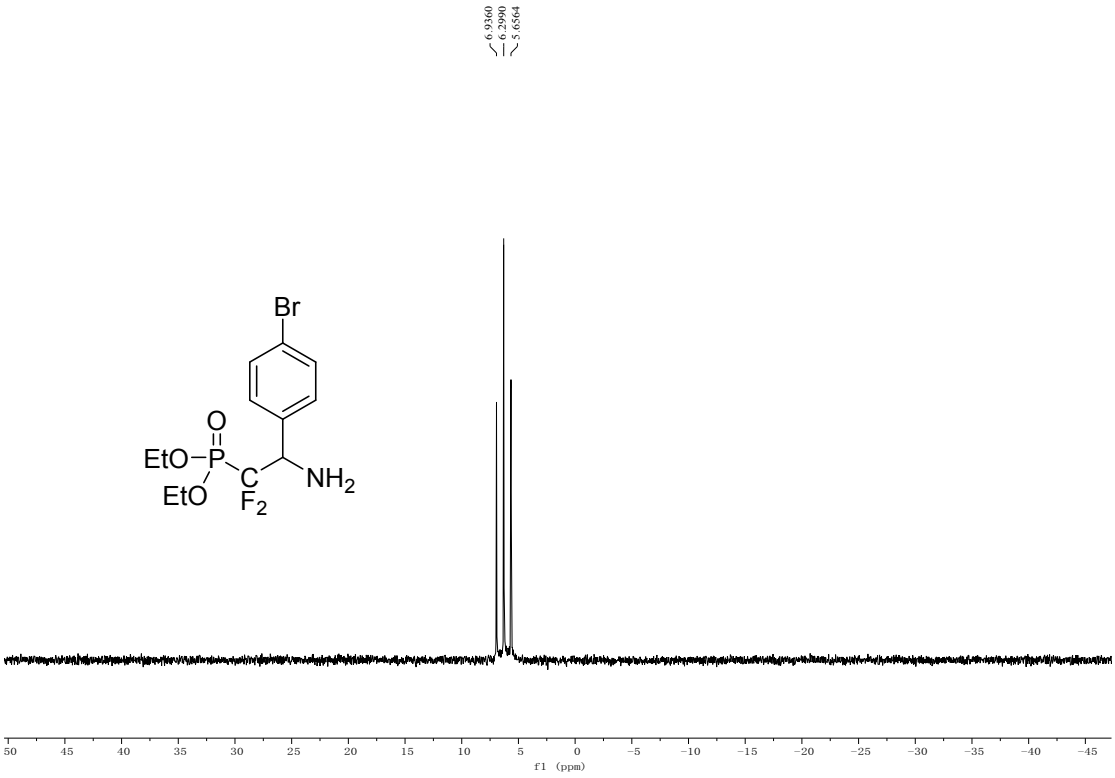

$^{13}$C NMR (100 MHz, $CDCl_3$) of **1g**:



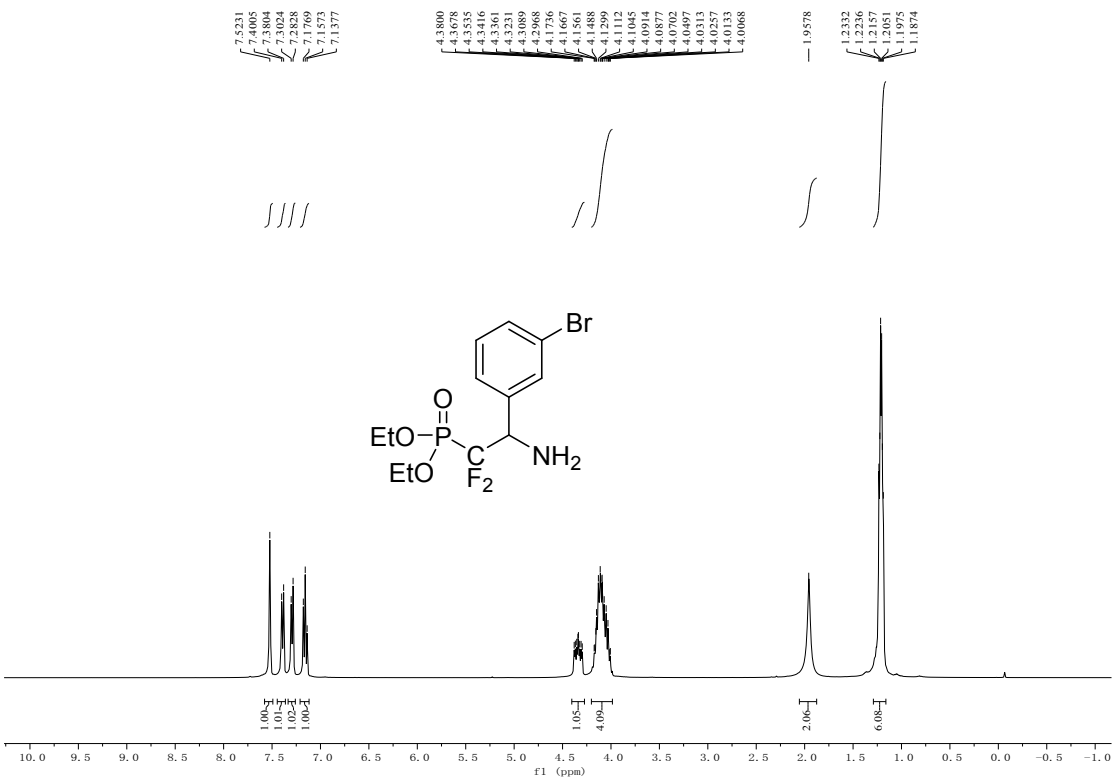
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **1g**:



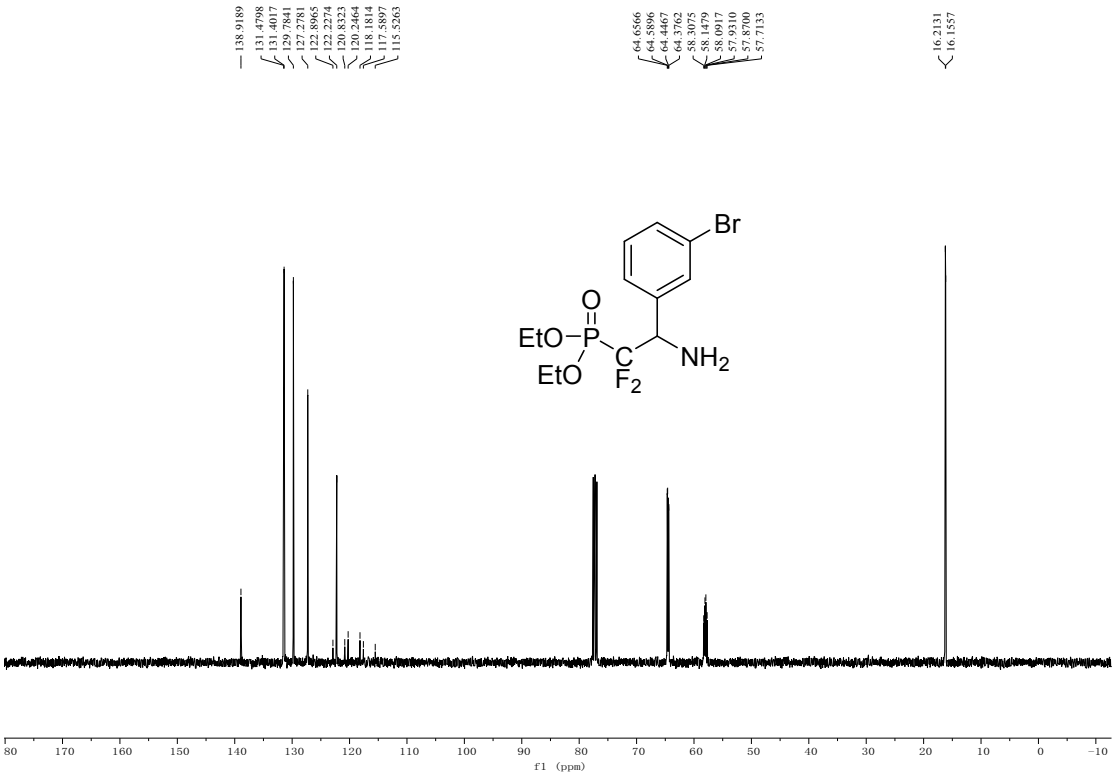

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **1g**:



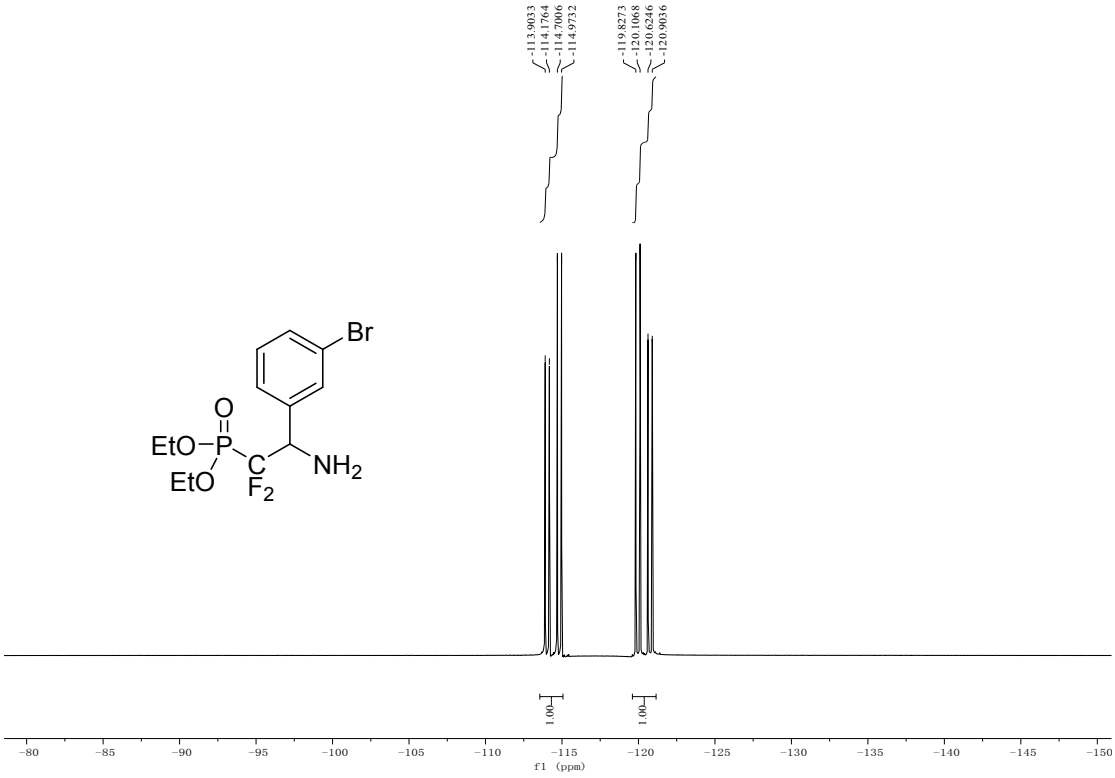
$^{1}$H NMR (400 MHz, $CDCl_3$) of **1h**:



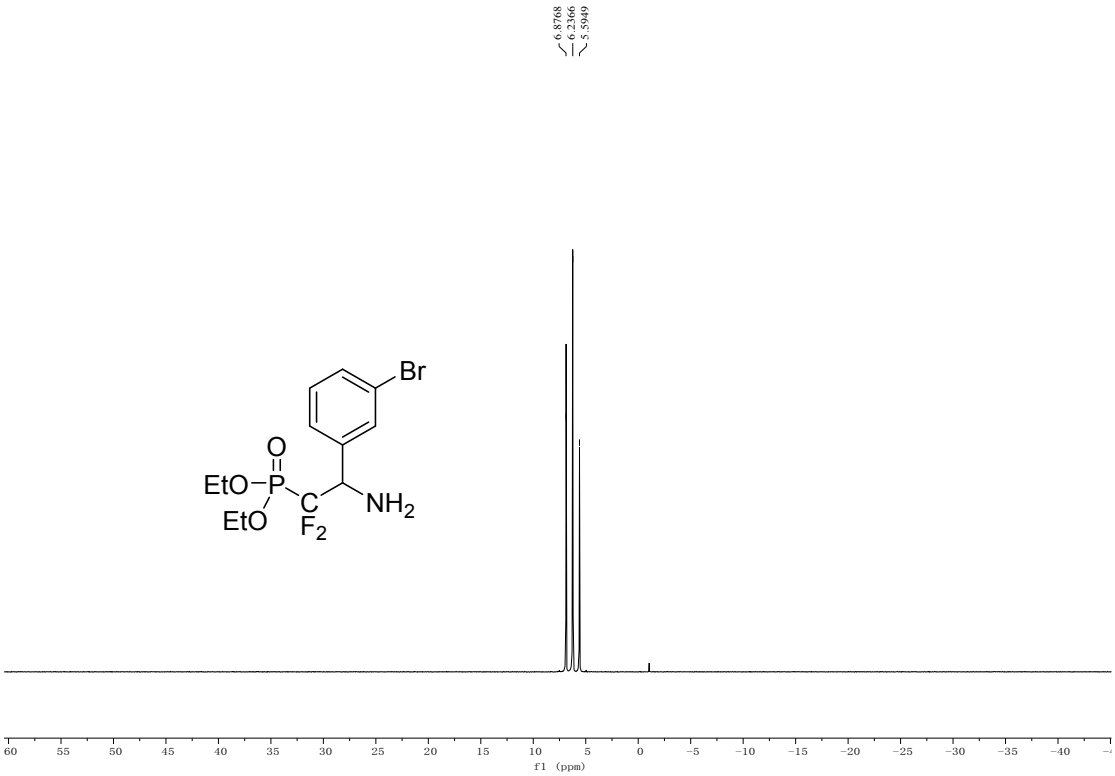

$^{13}$C NMR (100 MHz, $CDCl_3$) of **1h**:



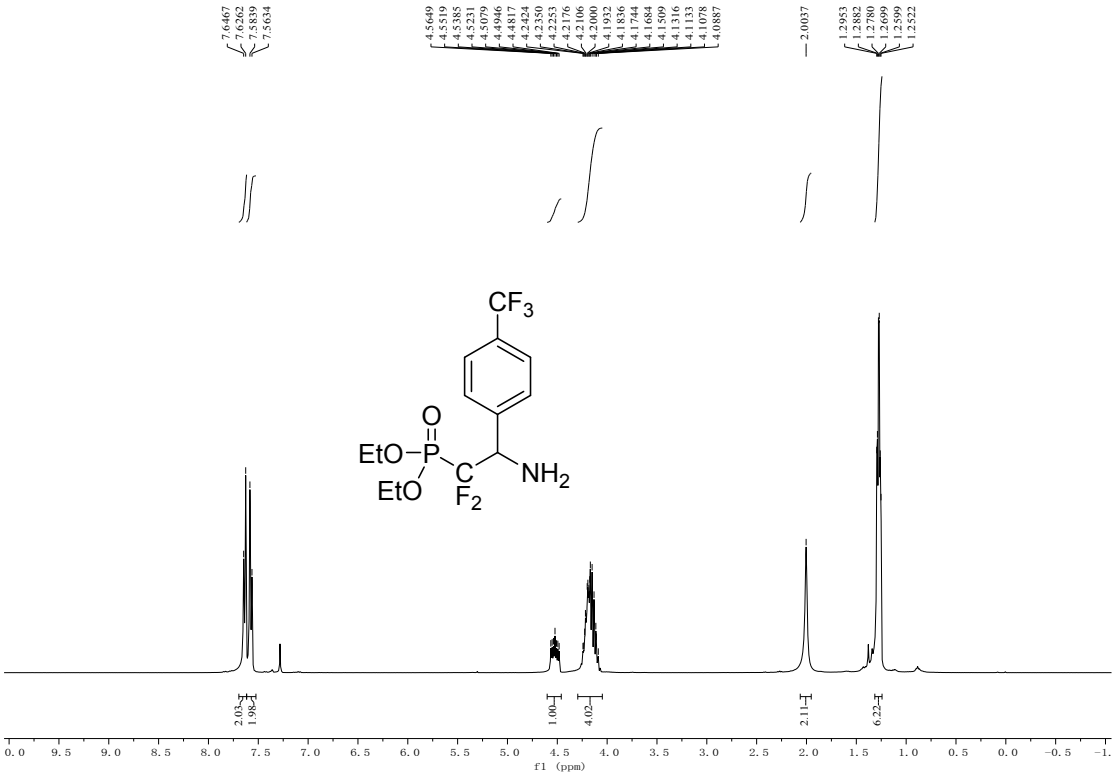
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **1h**:



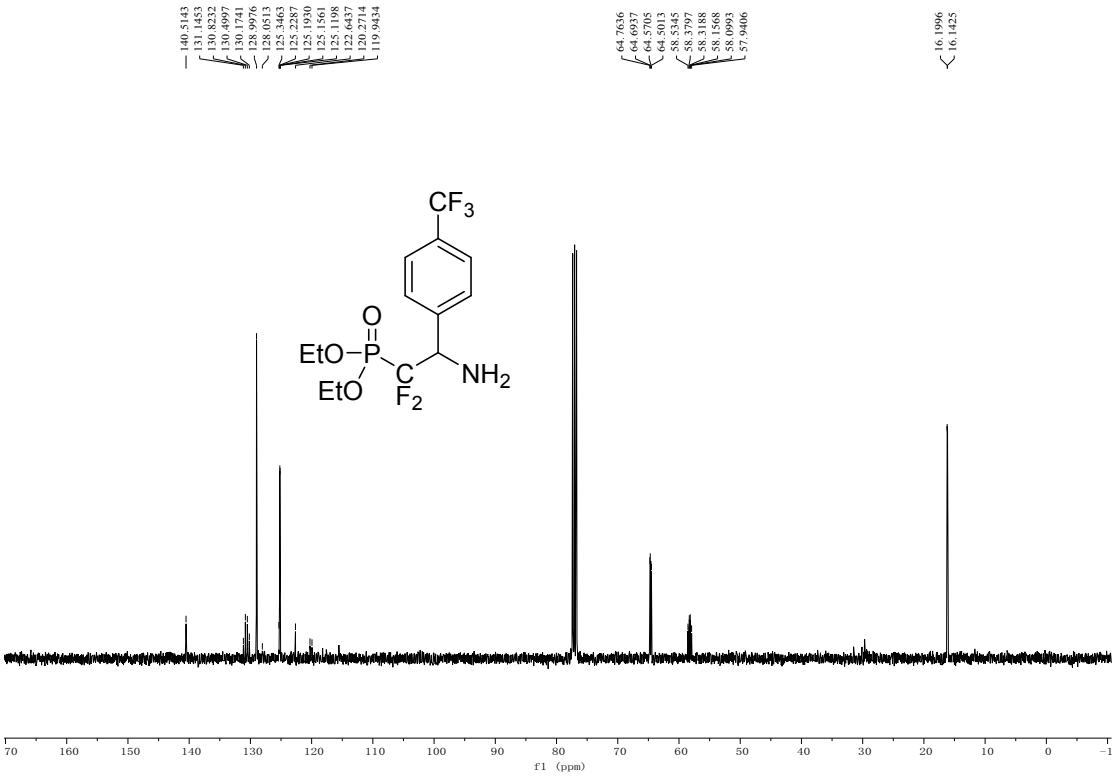

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **1h**:



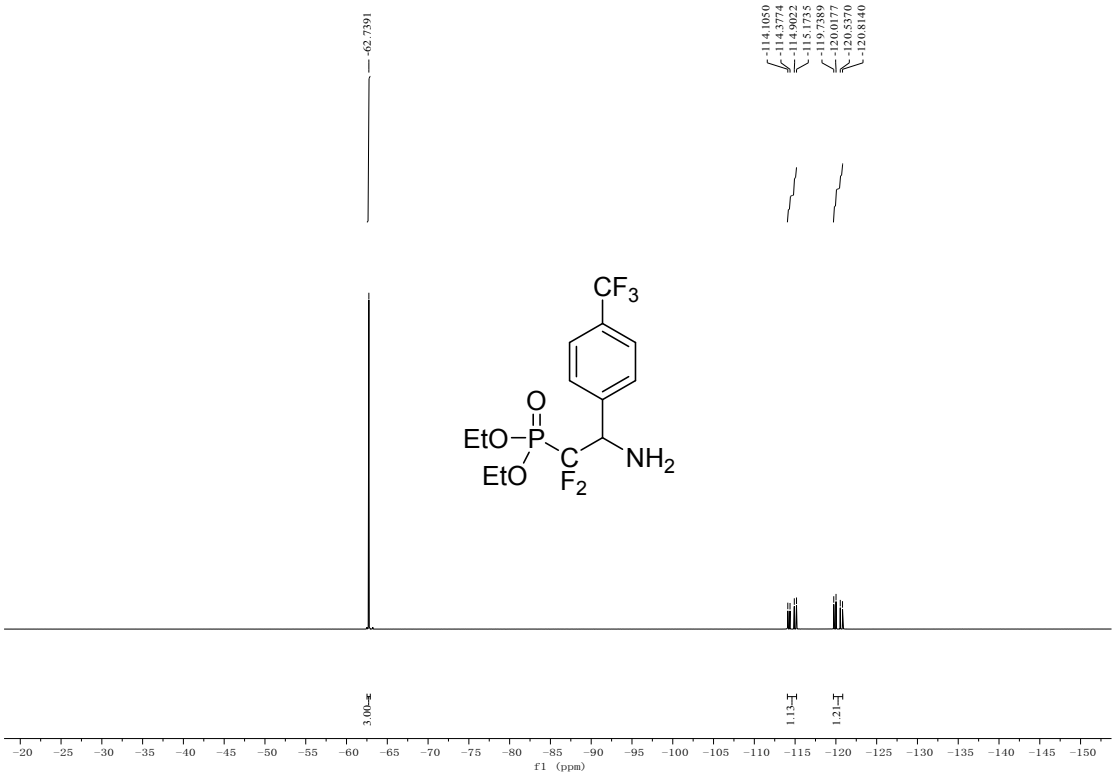
$^{1}$H NMR (400 MHz, $CDCl_3$) of **1i**:



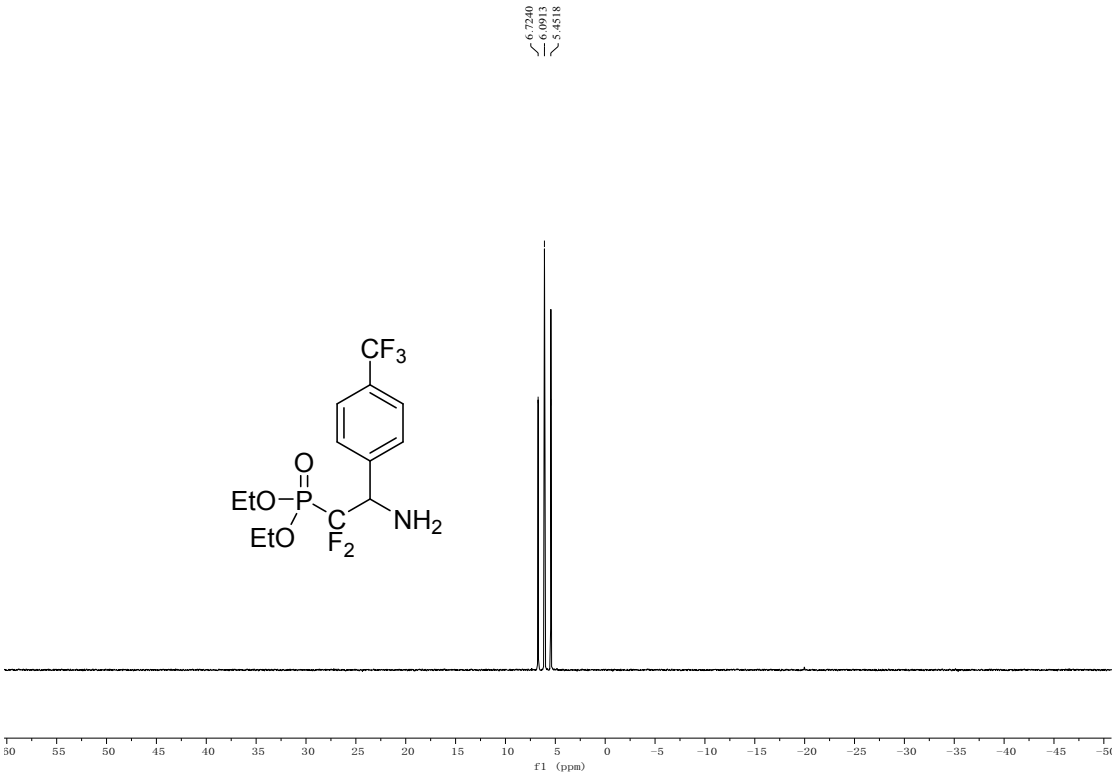

$^{13}$C NMR (100 MHz, $CDCl_3$) of **1i**:



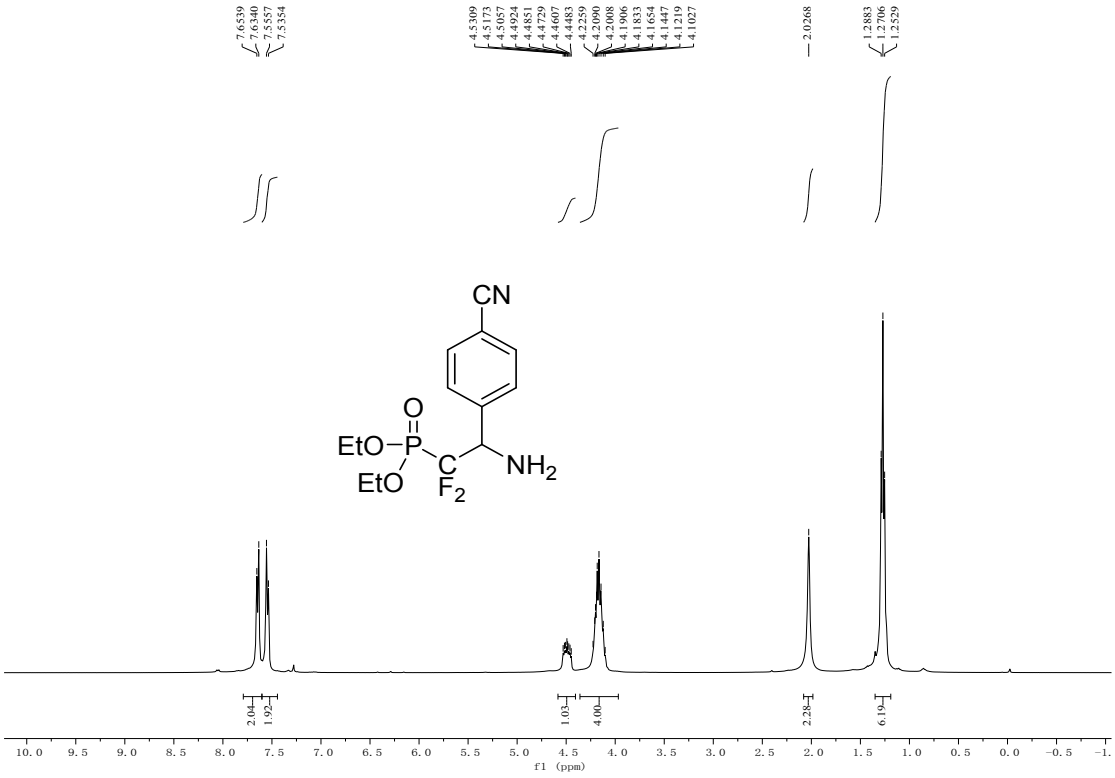
$^{19}$F NMR (376 MHz, $CDCl_3$) of **1i**:



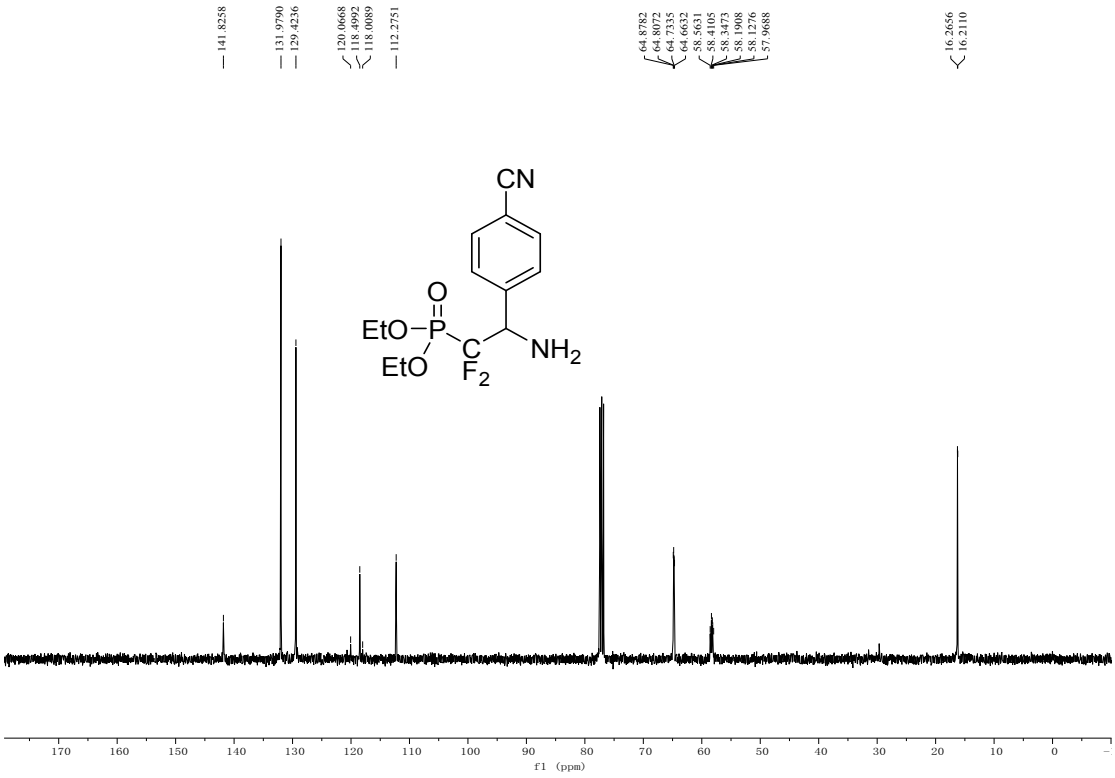

$^{31}$P NMR (162 MHz, $CDCl_3$) of **1i**:



$^{1}$H NMR (400 MHz, $CDCl_3$) of **1j**:




$^{13}$C NMR (100 MHz, $CDCl_3$) of **1j**:



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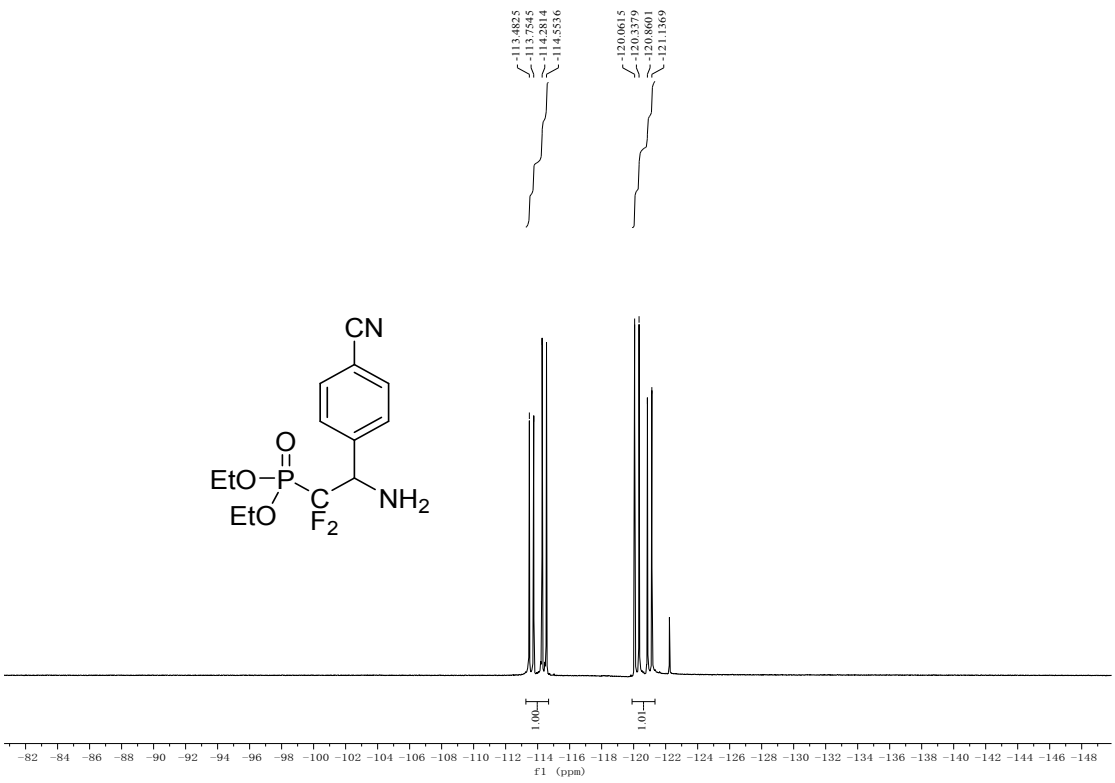
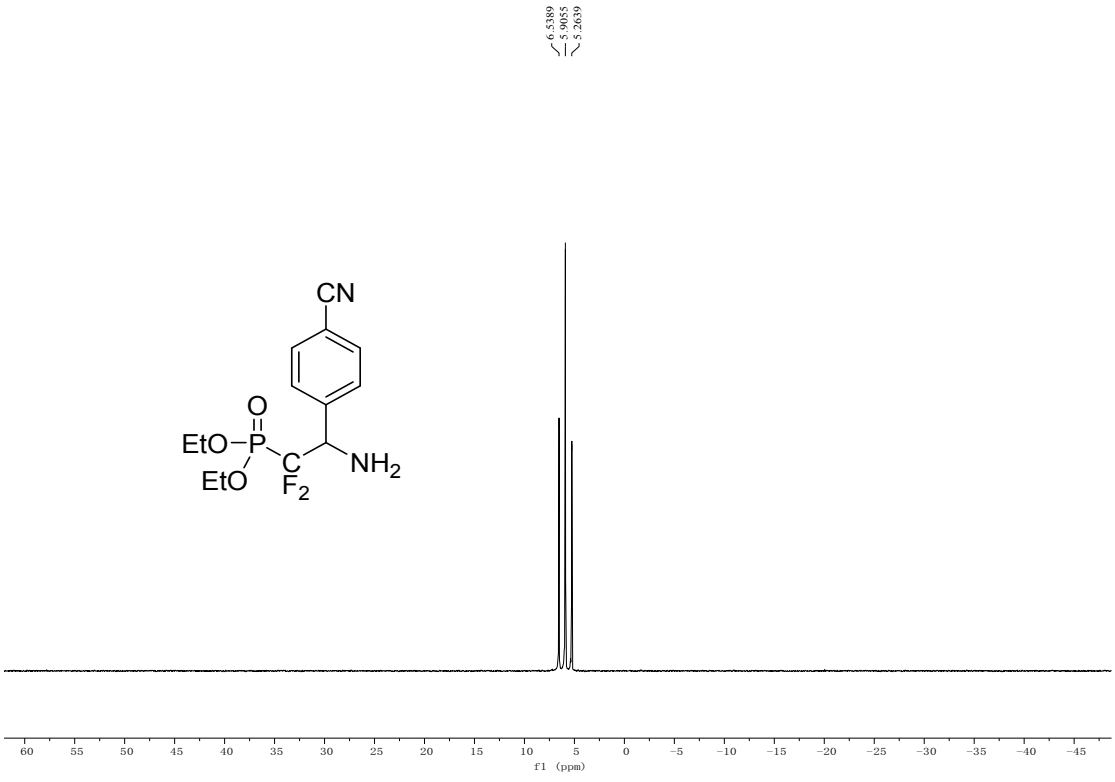
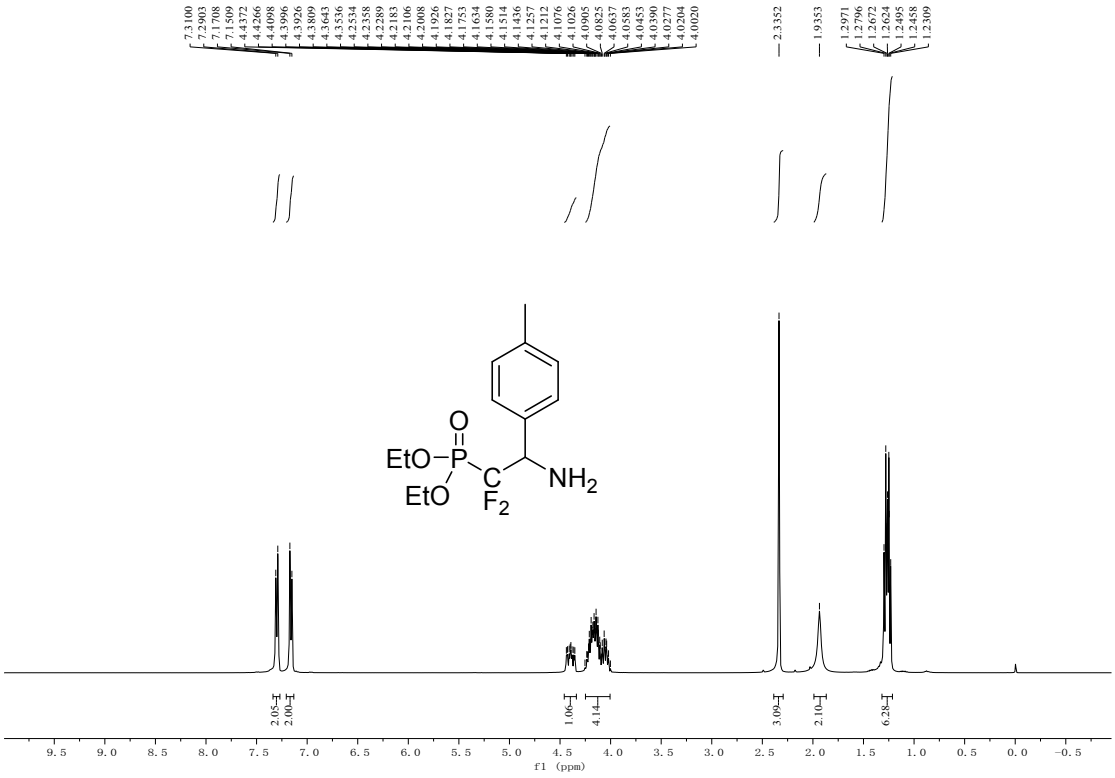


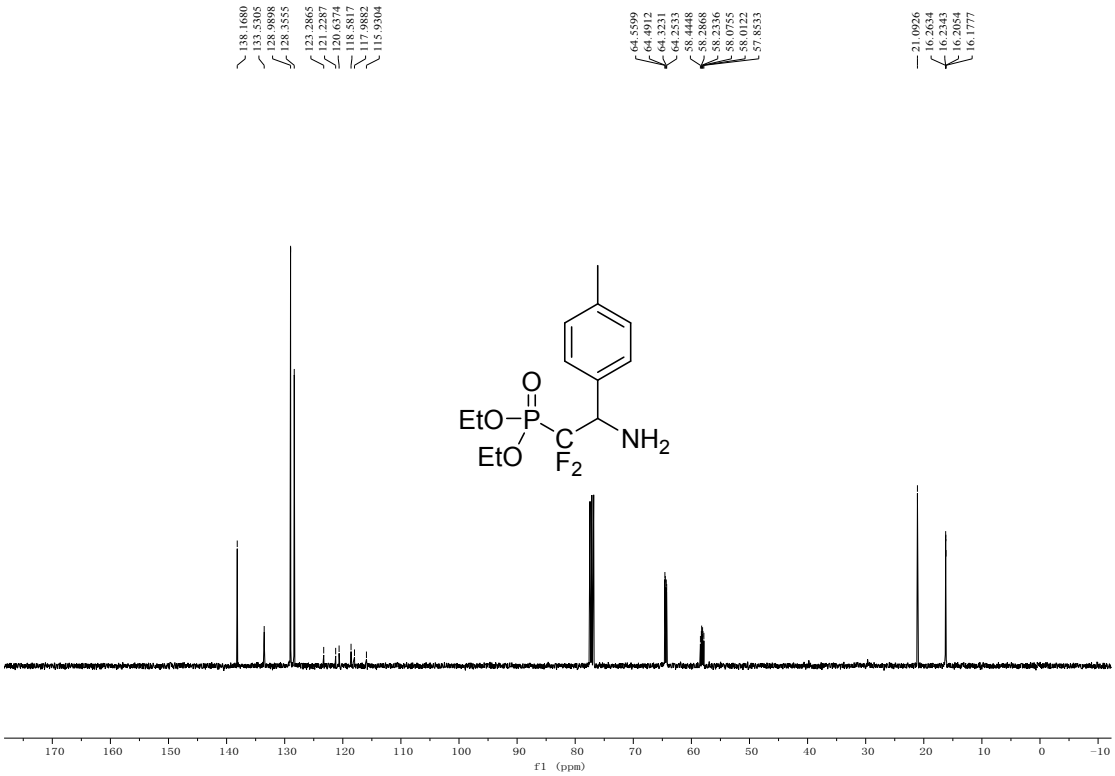

$^{31}$P NMR (162 MHz, $CDCl_3$) of **1j**:



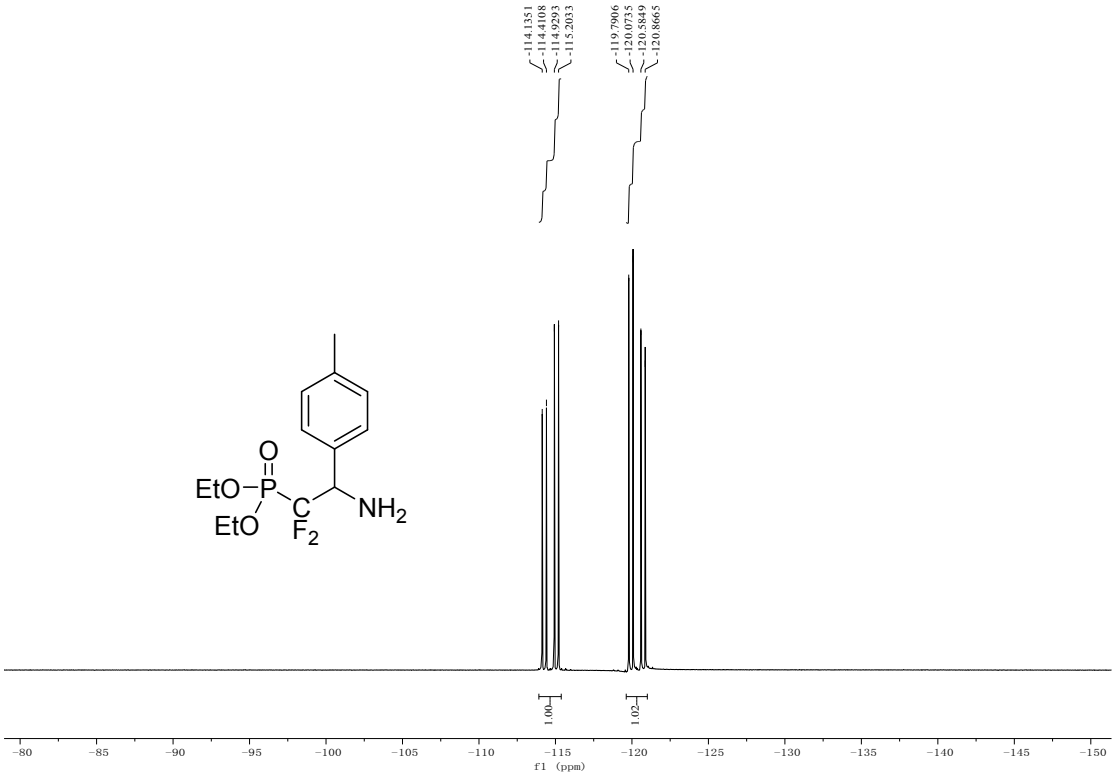
$^{1}$H NMR (400 MHz, $CDCl_3$) of **1k**:



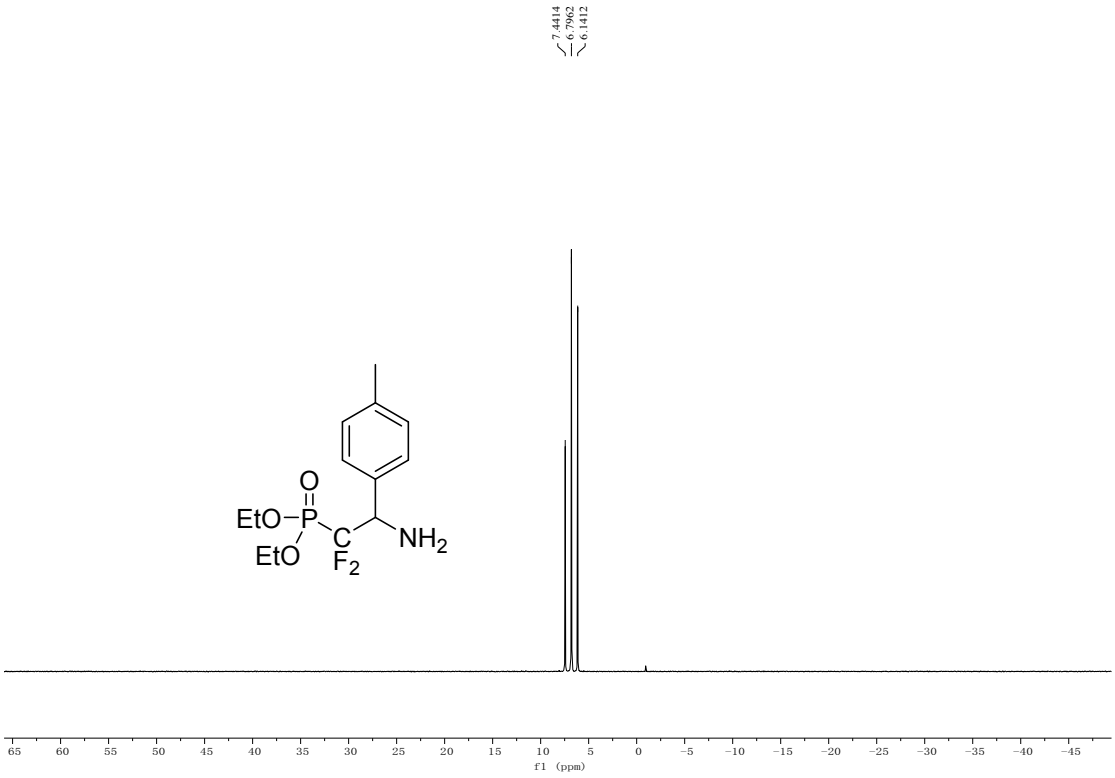

$^{13}$C NMR (100 MHz, $CDCl_3$) of **1k**:



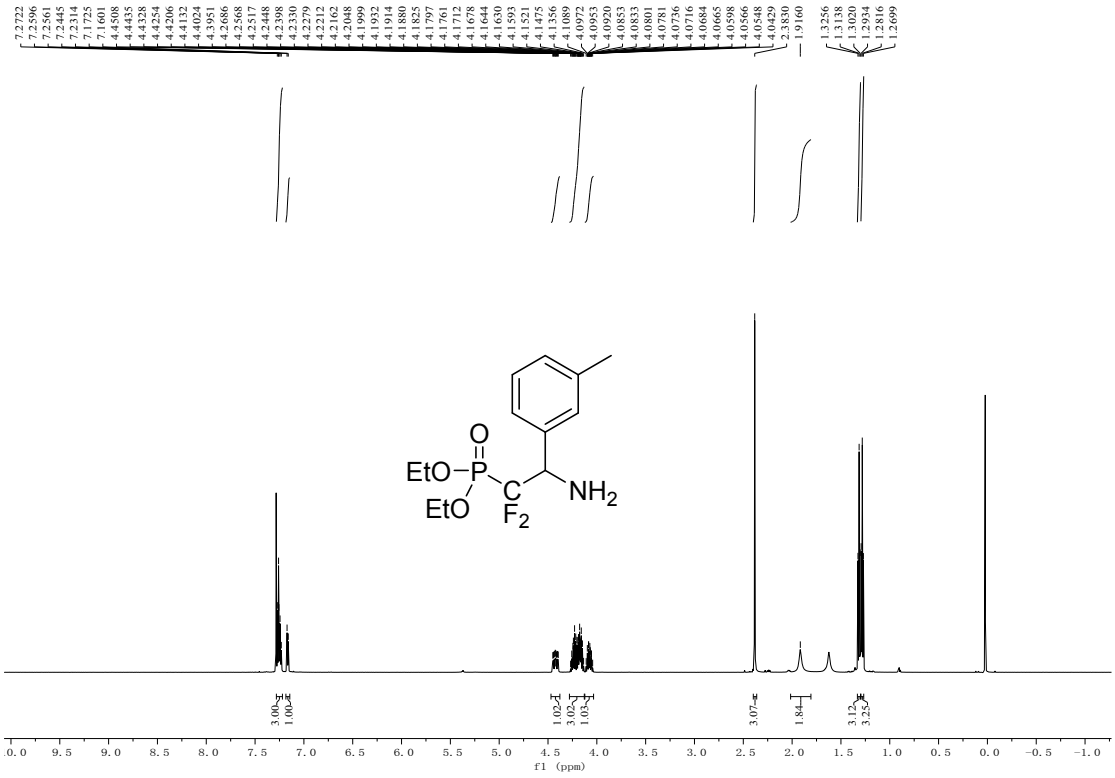
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **1k**:



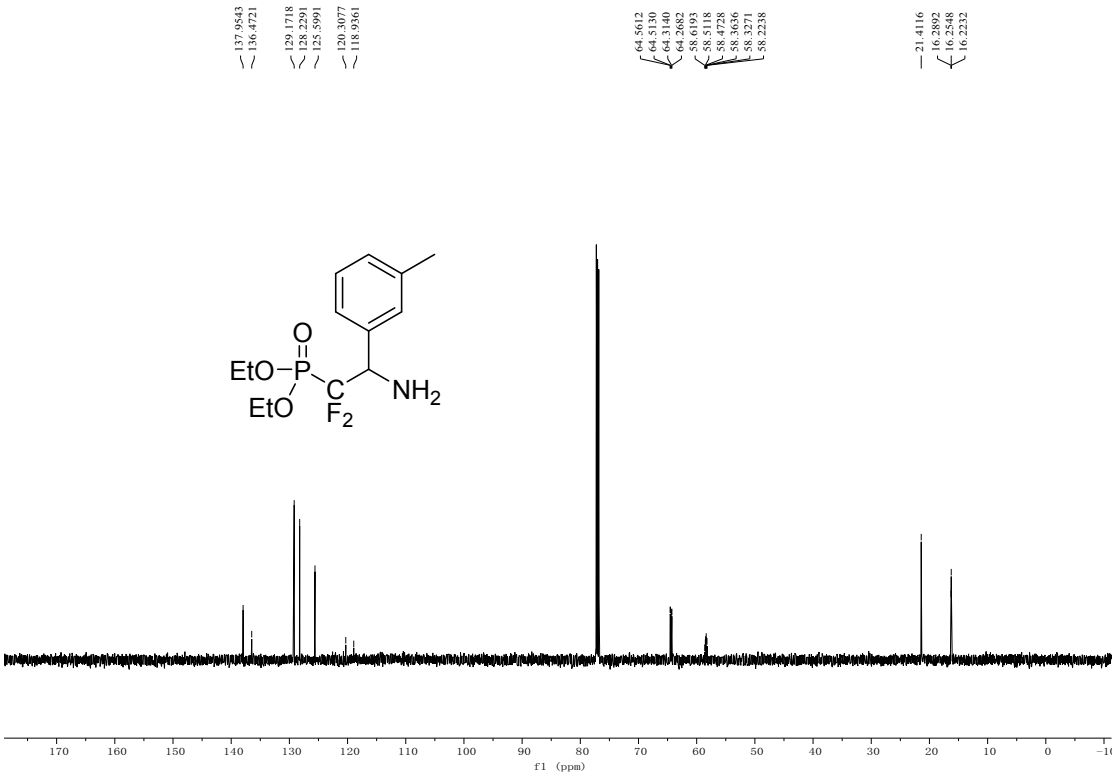

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **1k**:



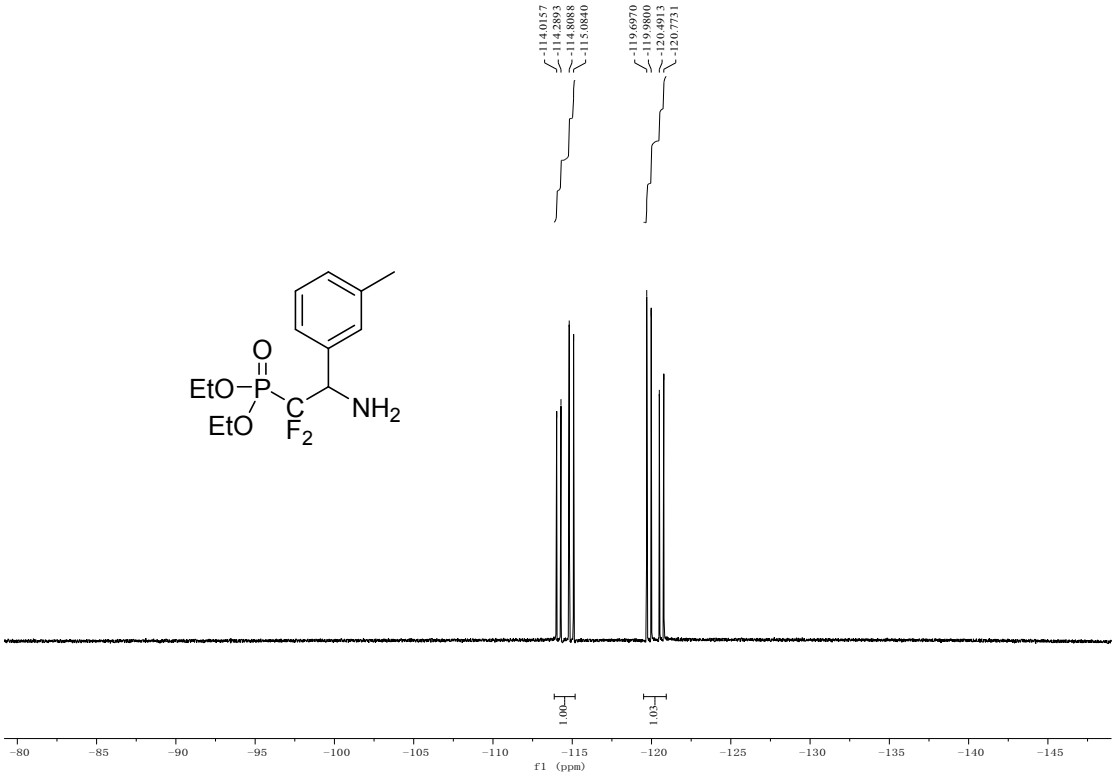
$^{1}$H NMR (600 MHz, $CDCl_3$) of **1l**:



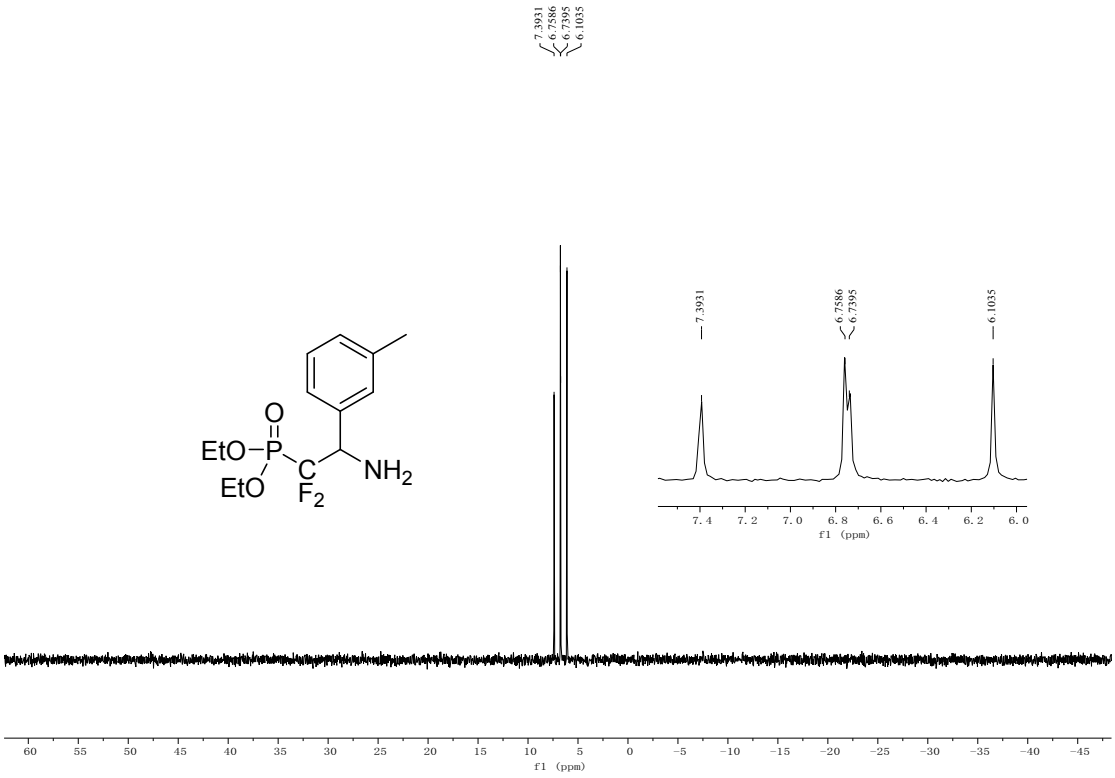

$^{13}$C NMR (150 MHz, $CDCl_3$) of **1l**:



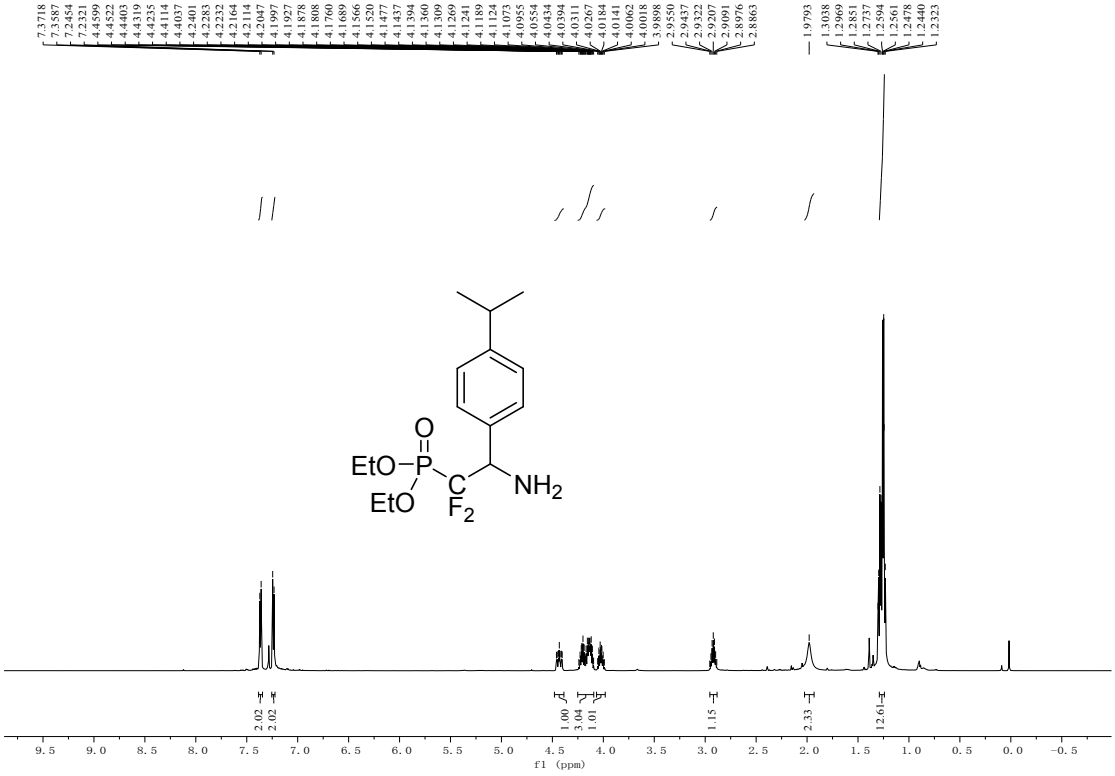
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **1l**:



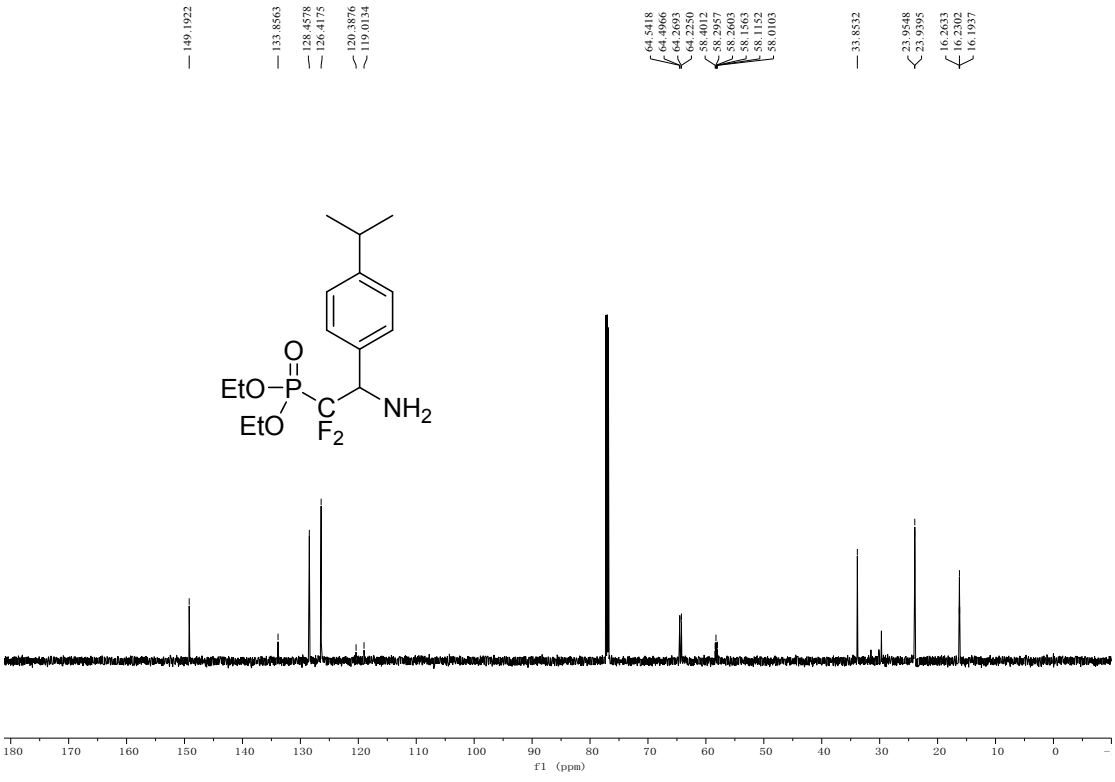

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **1l**:



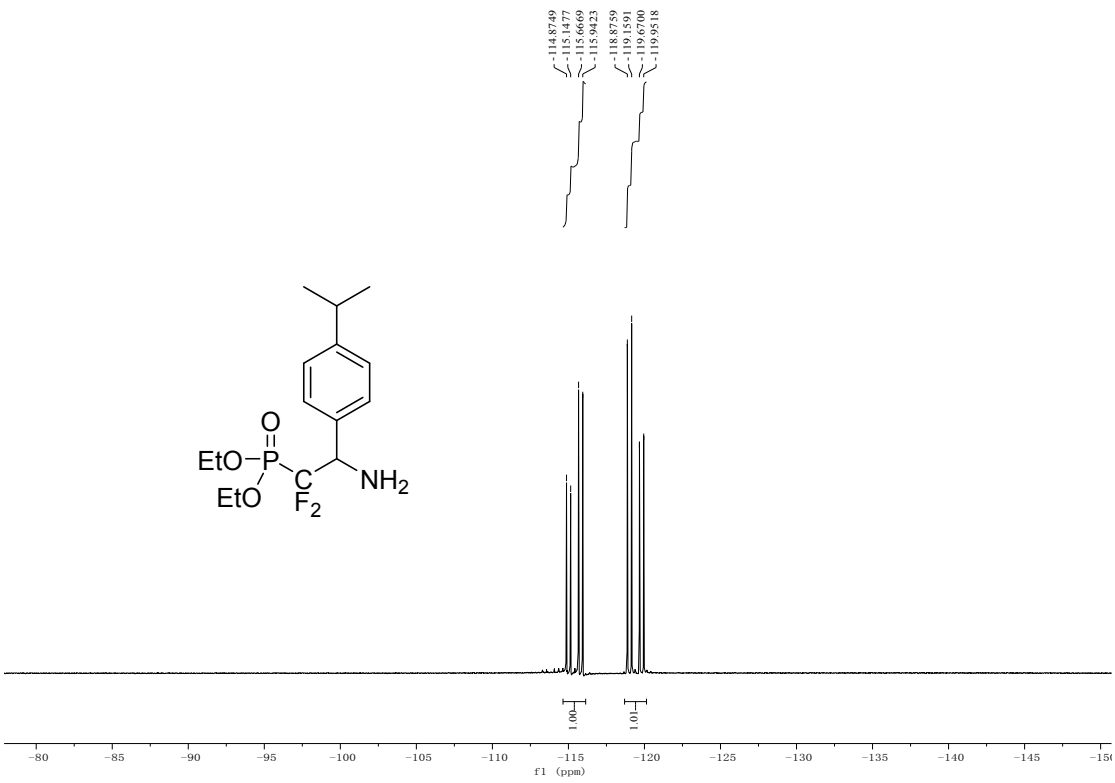
$^{1}$H NMR (600 MHz, $CDCl_3$) of **1m**:



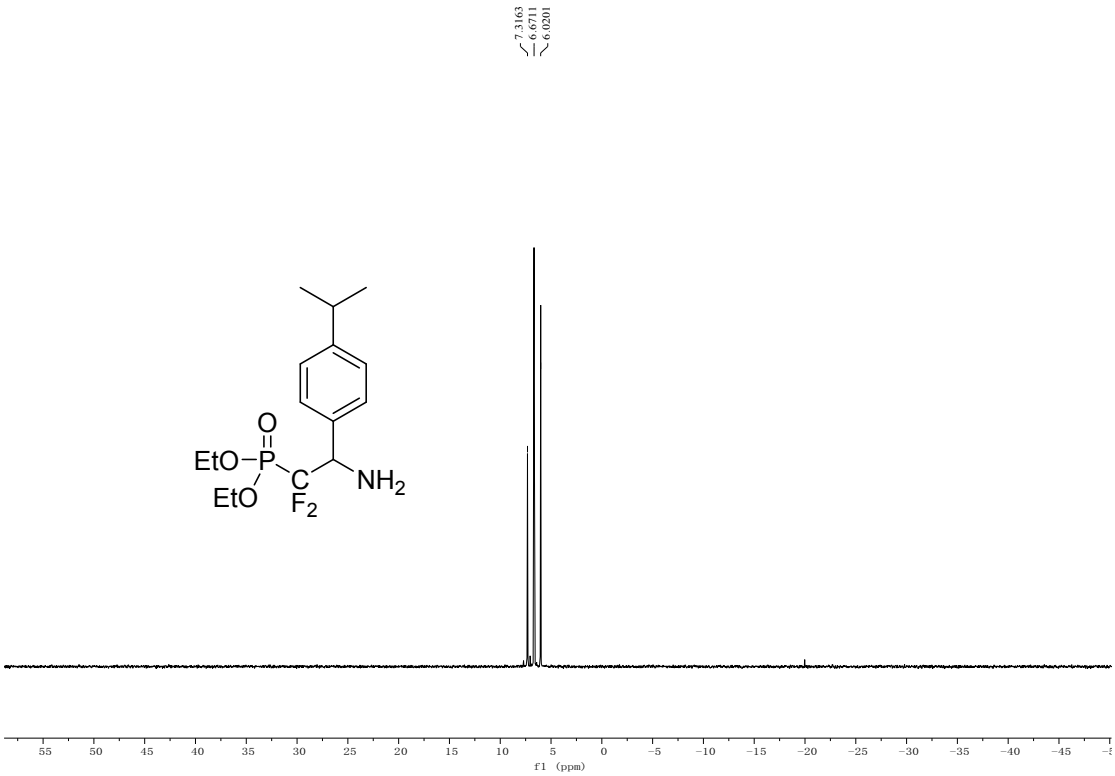

$^{13}$C NMR (150 MHz, $CDCl_3$) of **1m**:



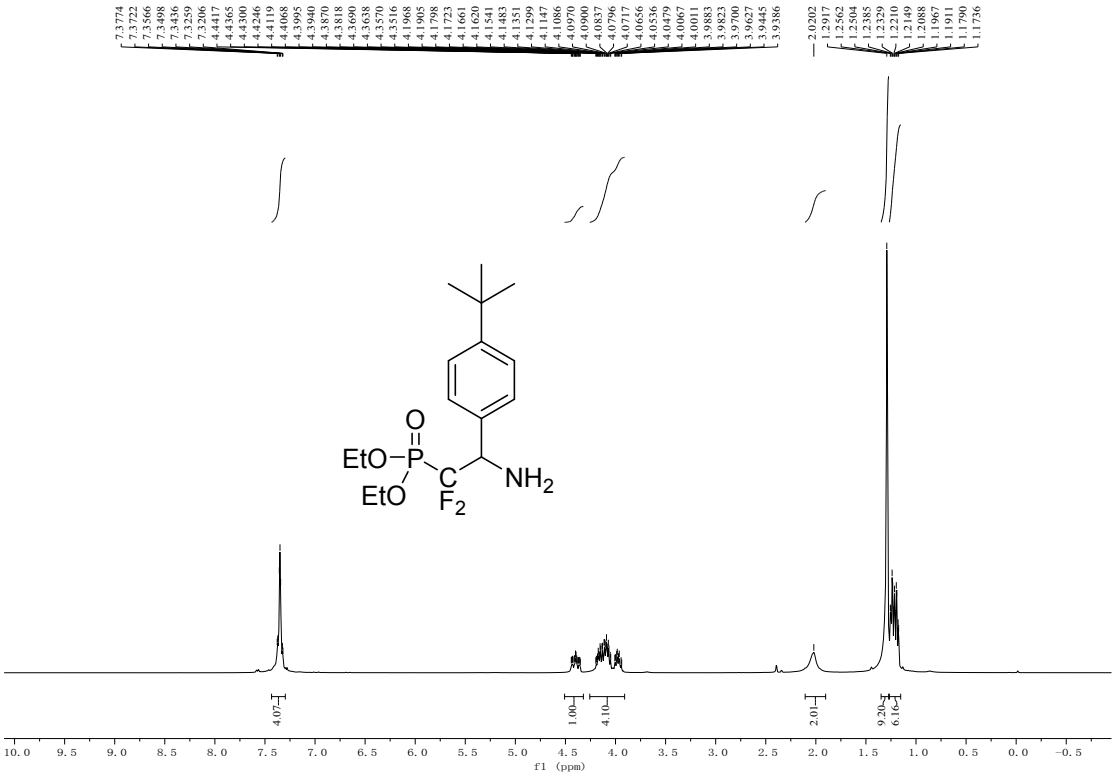
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **1m**:



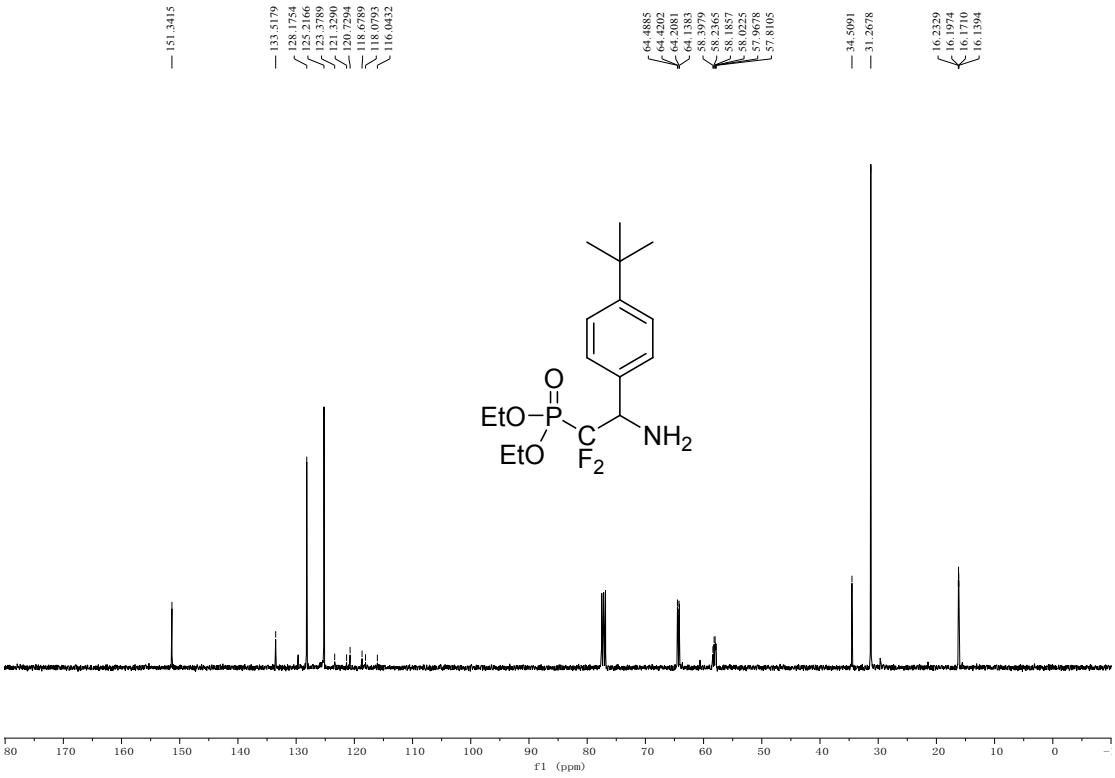

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **1m**:



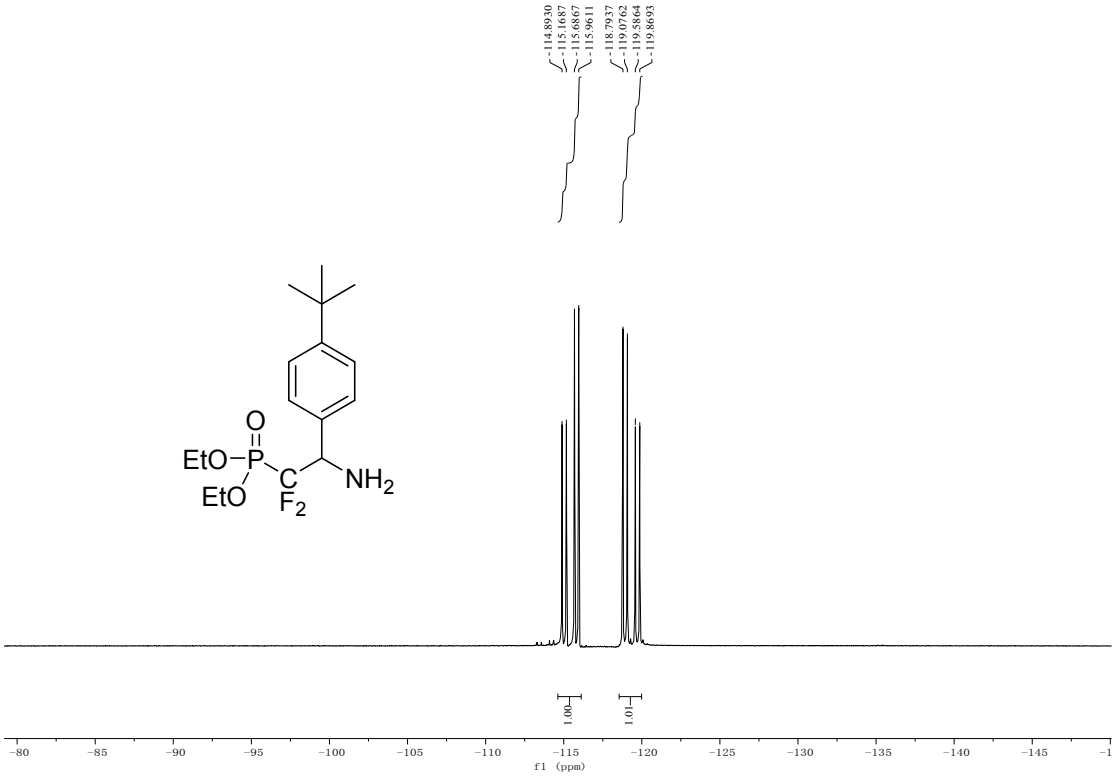
$^{1}$H NMR (400 MHz, $CDCl_3$) of **1n**:



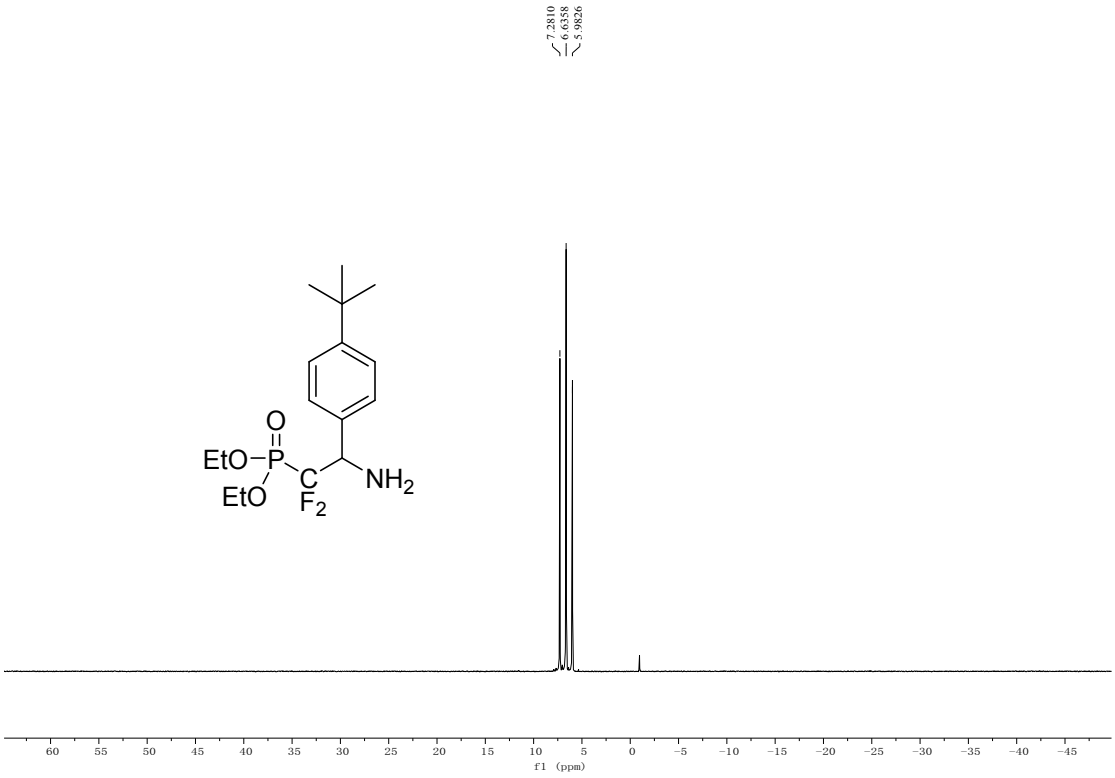

$^{13}$C NMR (100 MHz, $CDCl_3$) of **1n**:



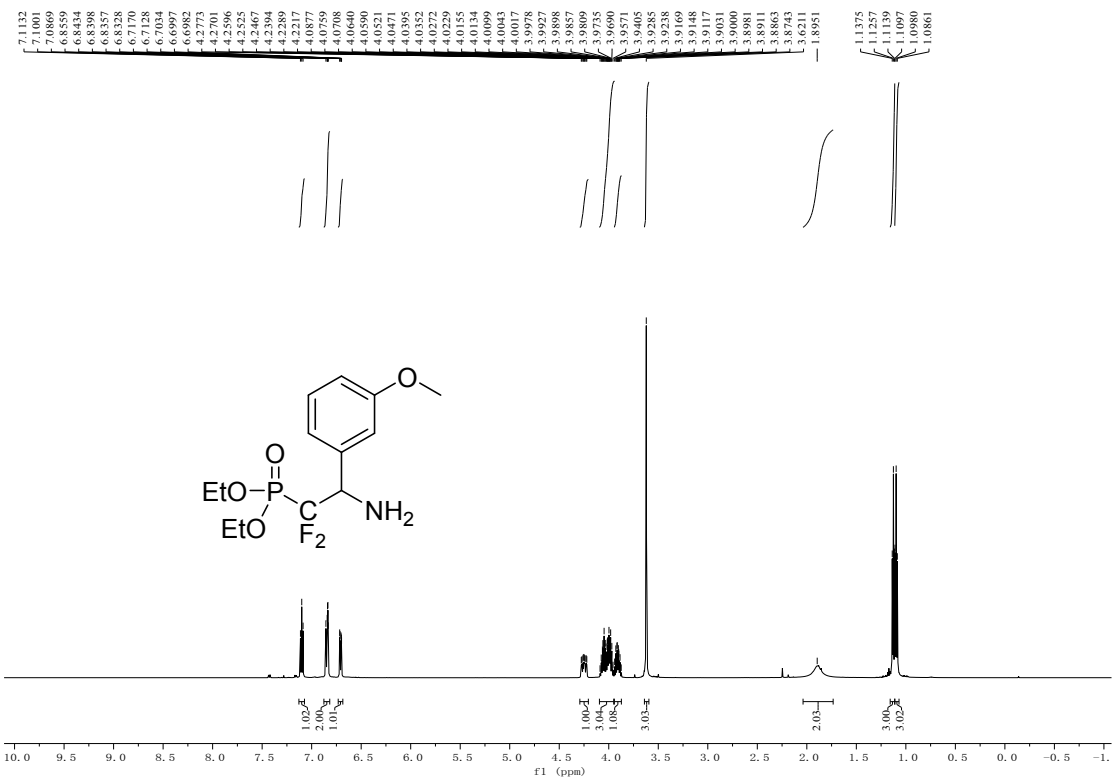
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **1n**:



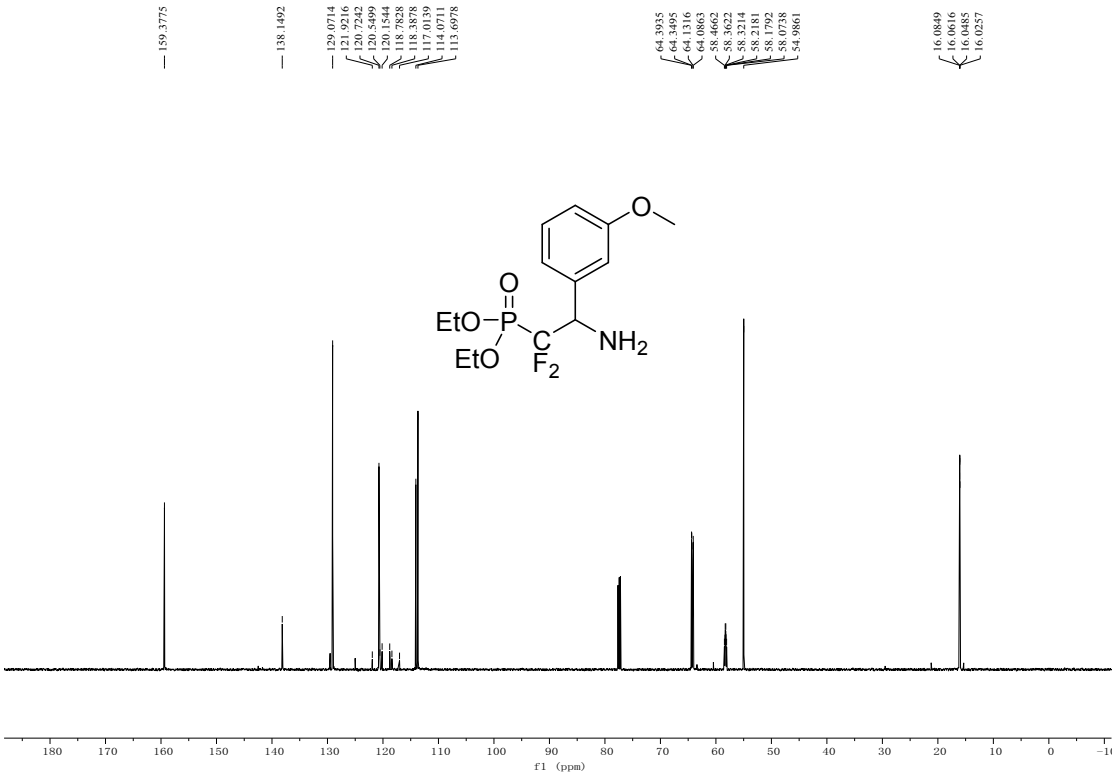

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **1n**:



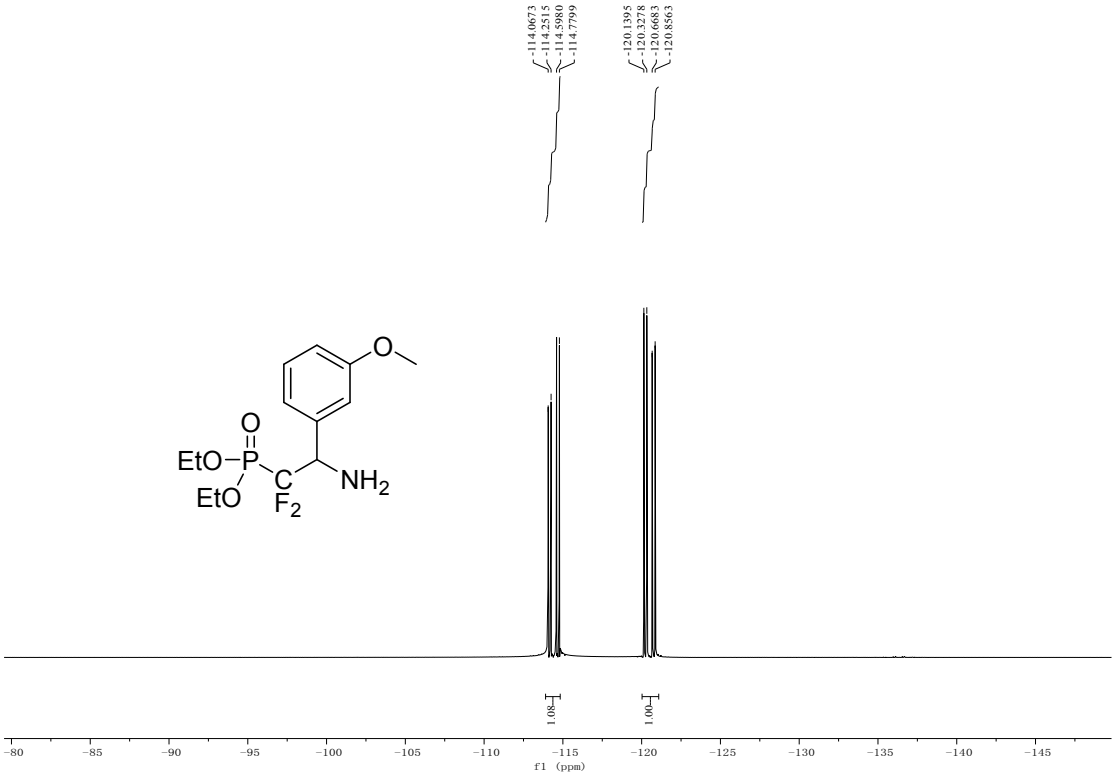
$^{1}$H NMR (600 MHz, $CDCl_3$) of **1o**:



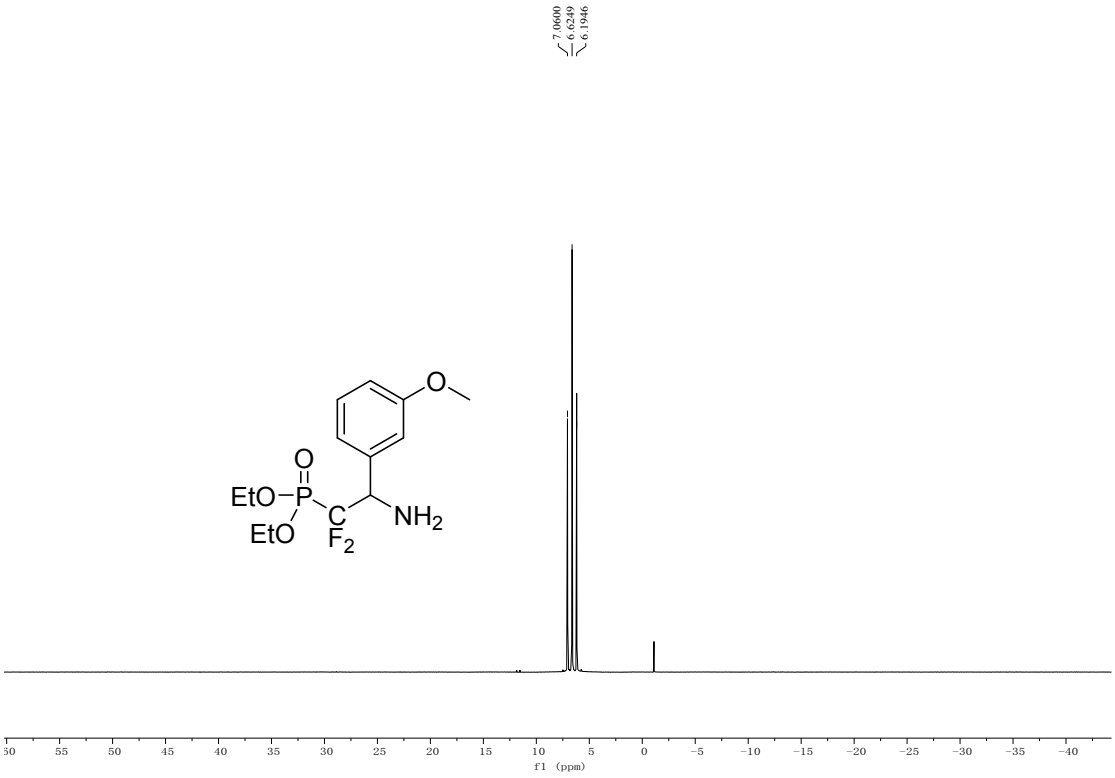

$^{13}$C NMR (150 MHz, $CDCl_3$) of **1o**:



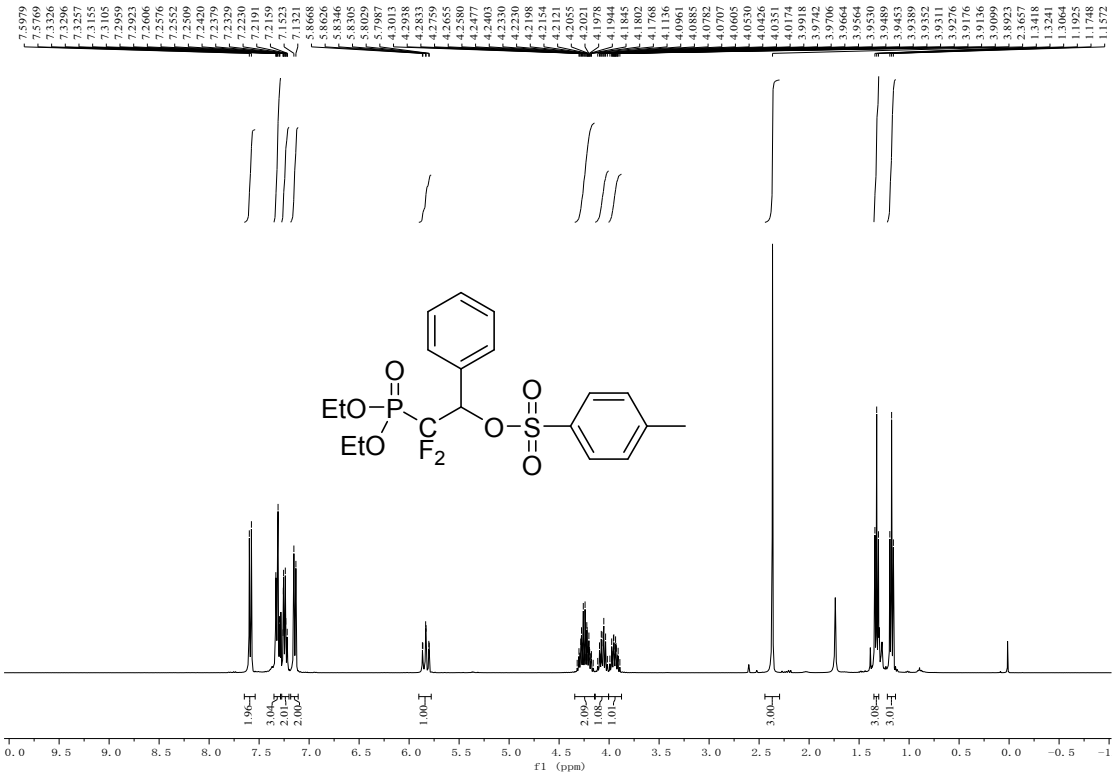
$^{19}F$ NMR (565 MHz, $CDCl_3$) of **1o**:



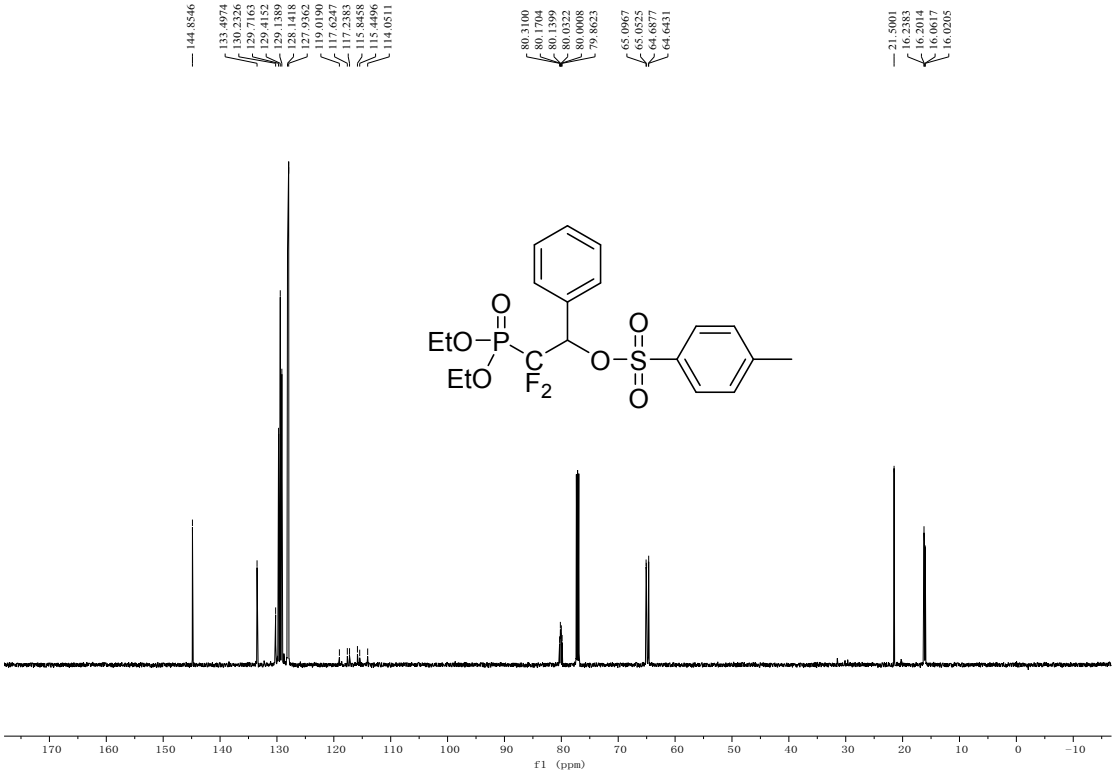

$^{31}P$ NMR (243 MHz, $CDCl_3$) of **1o**:



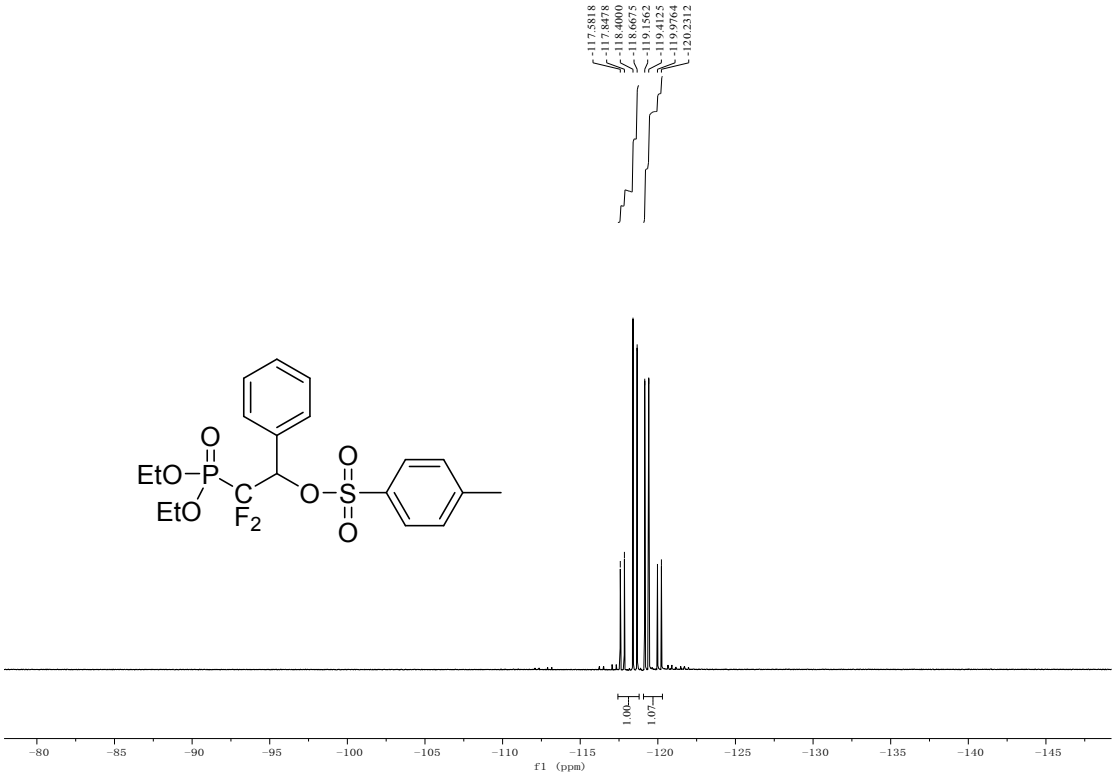
$^{1}$H NMR (400 MHz, $CDCl_3$) of **4aa**:



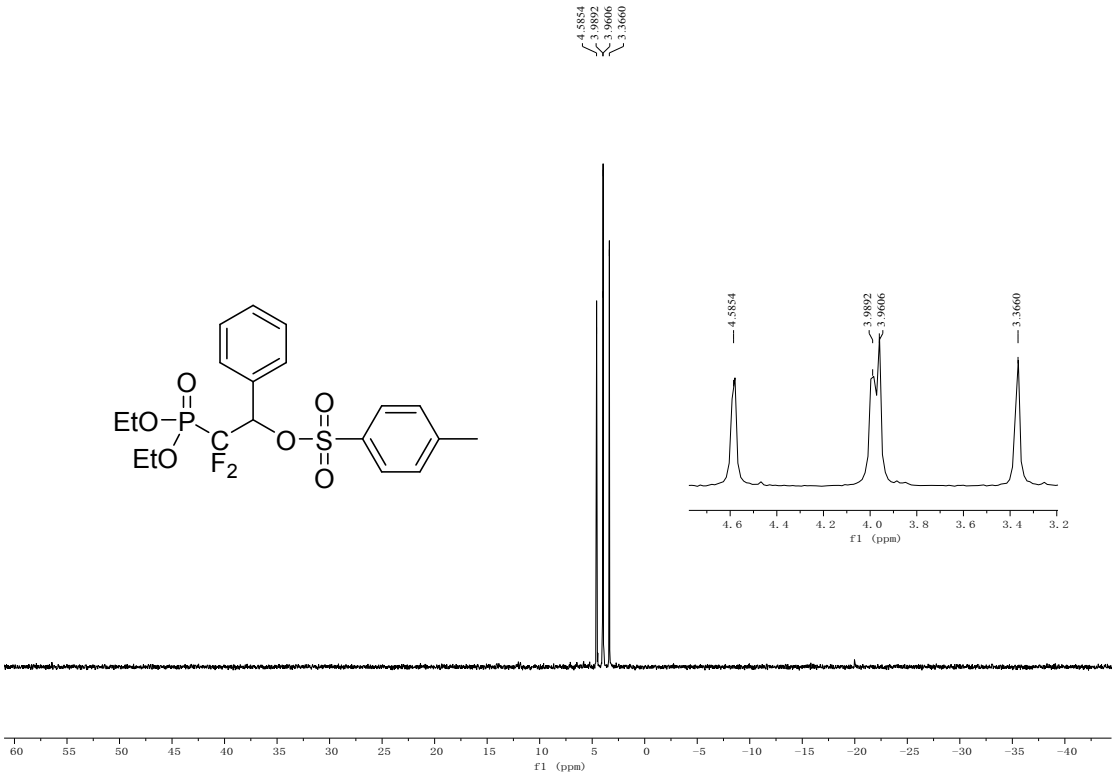

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4aa**:



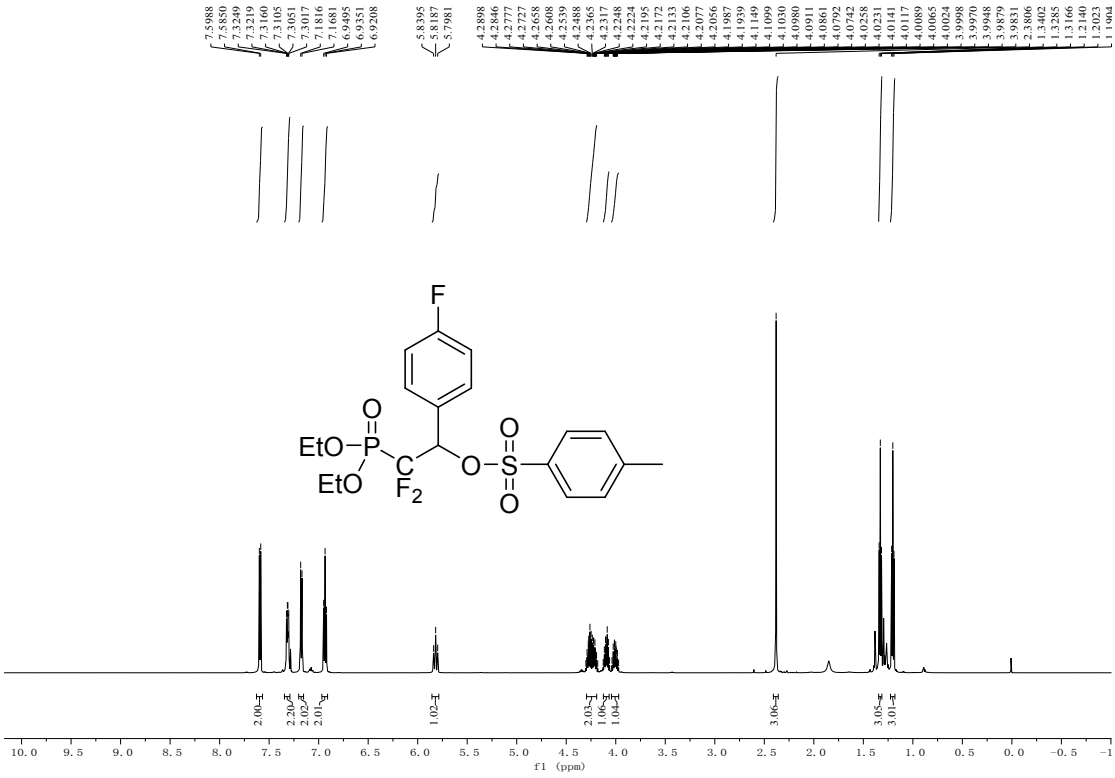
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4aa**:



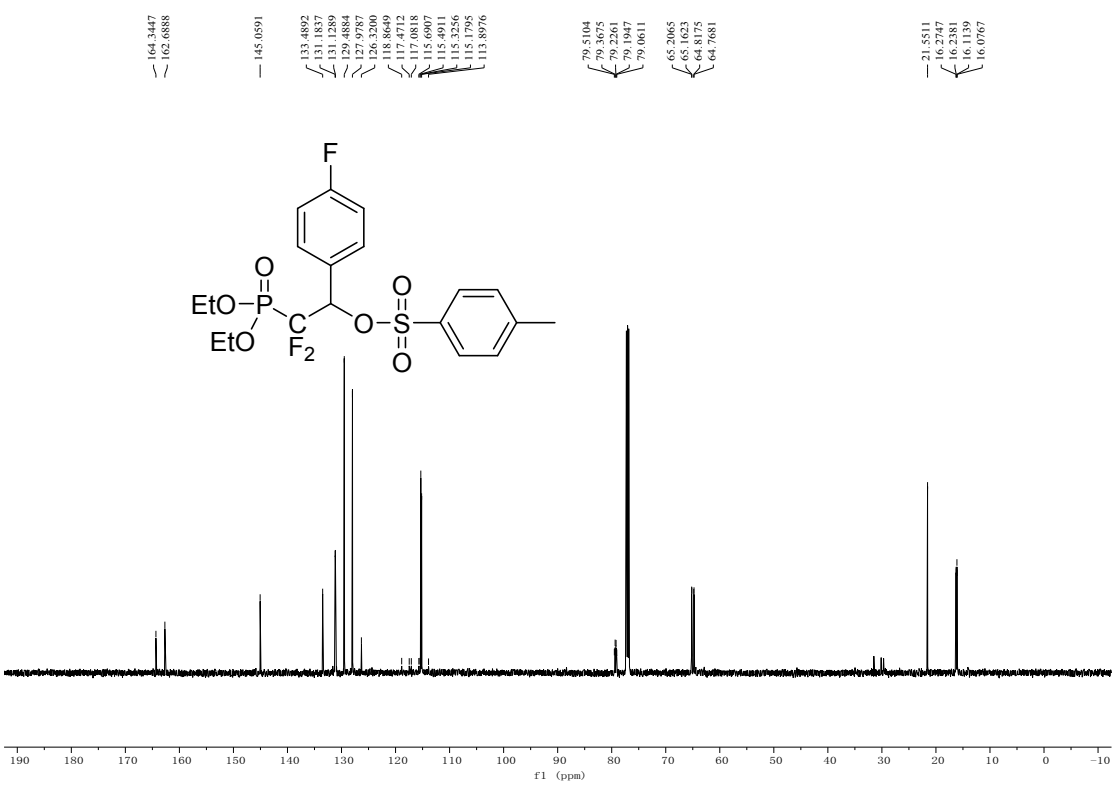

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4aa**:



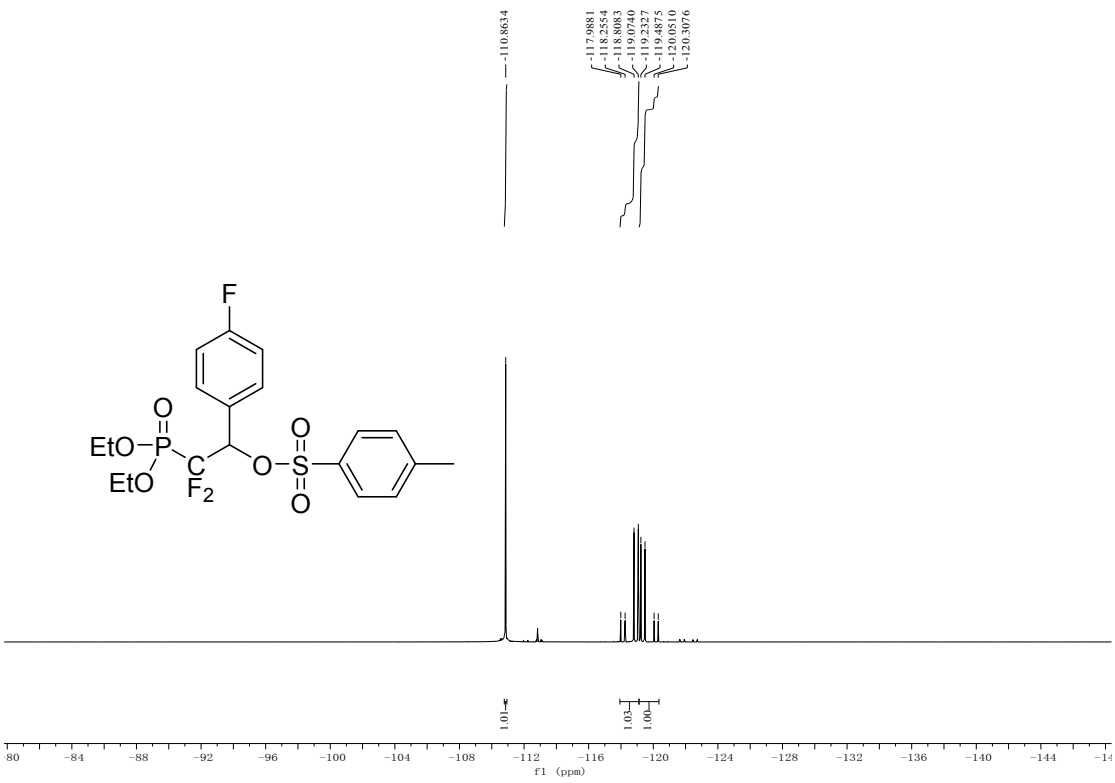
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4ba**:



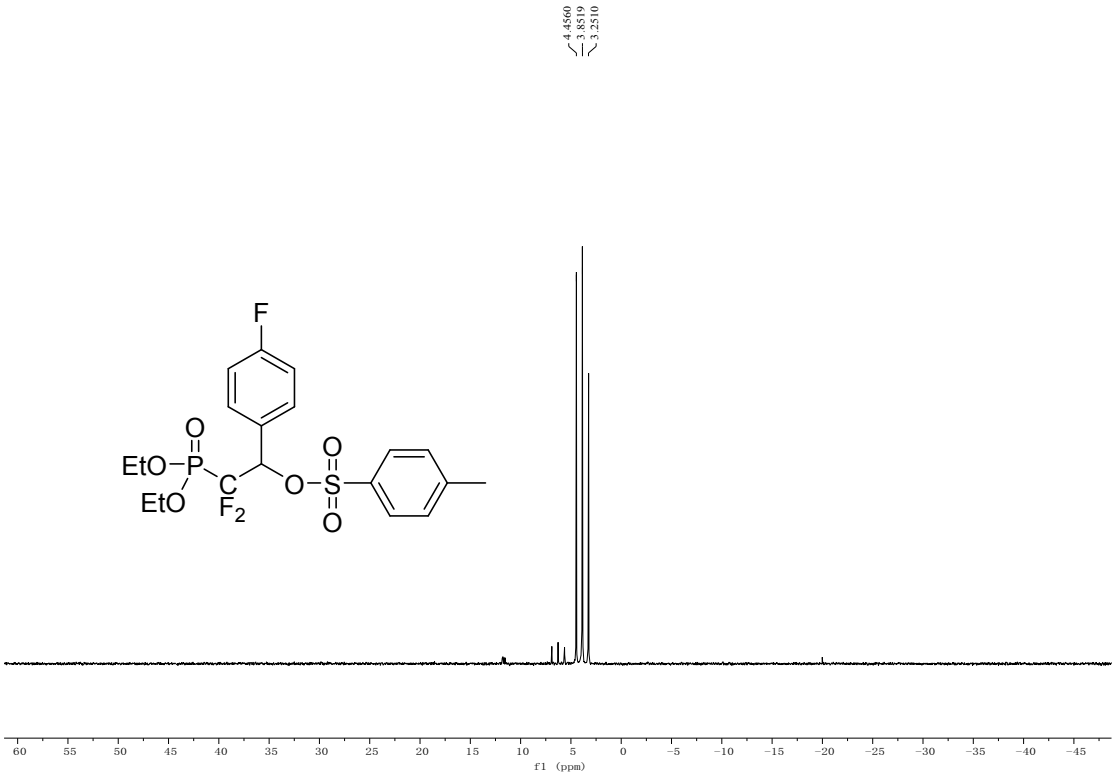

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ba**:



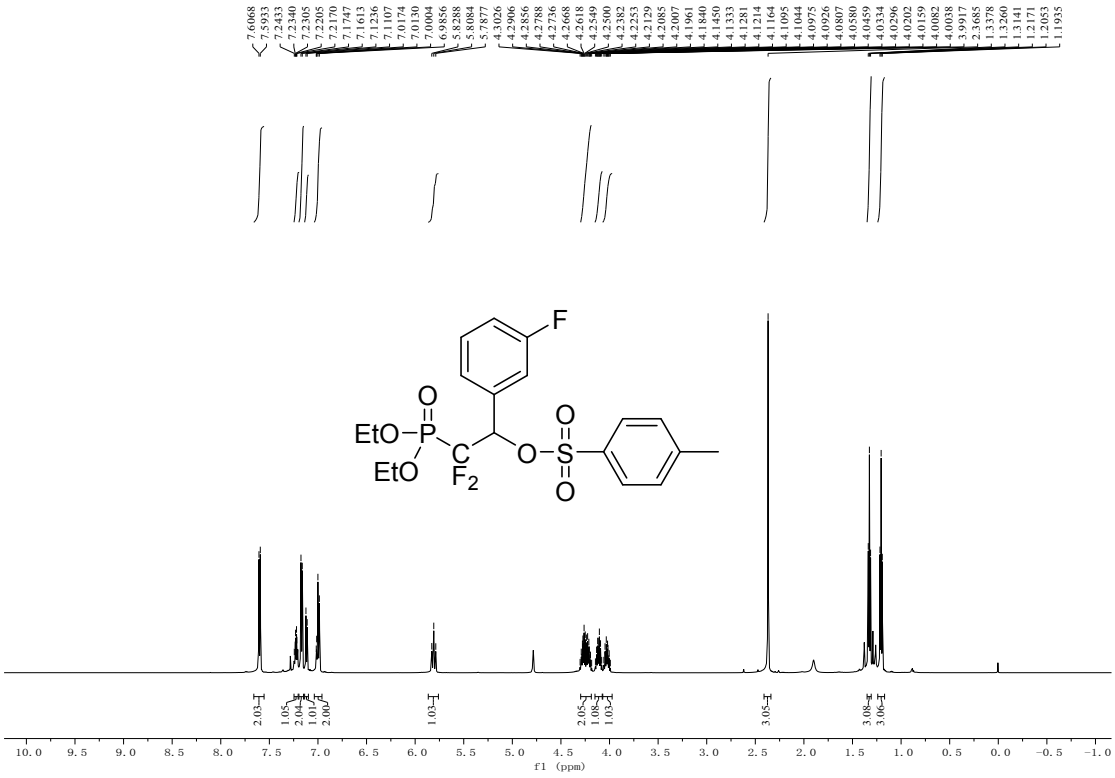
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ba**:



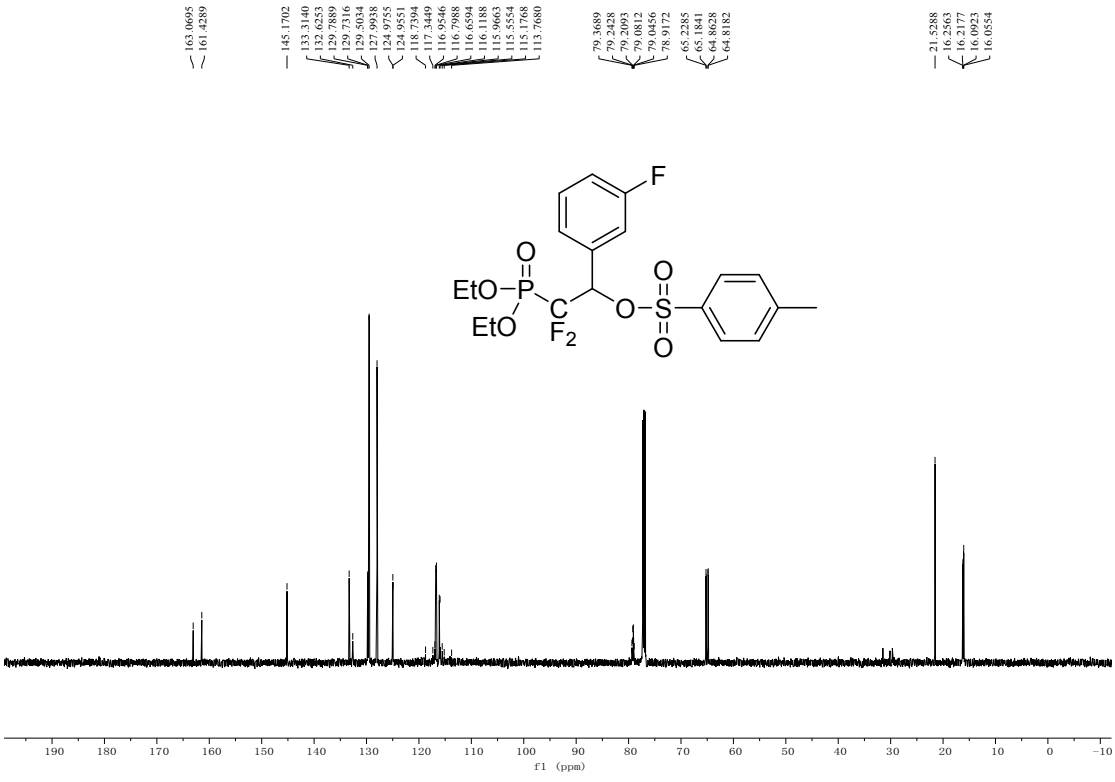

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ba**:



$^{1}$H NMR (600 MHz, $CDCl_3$) of **4ca**:

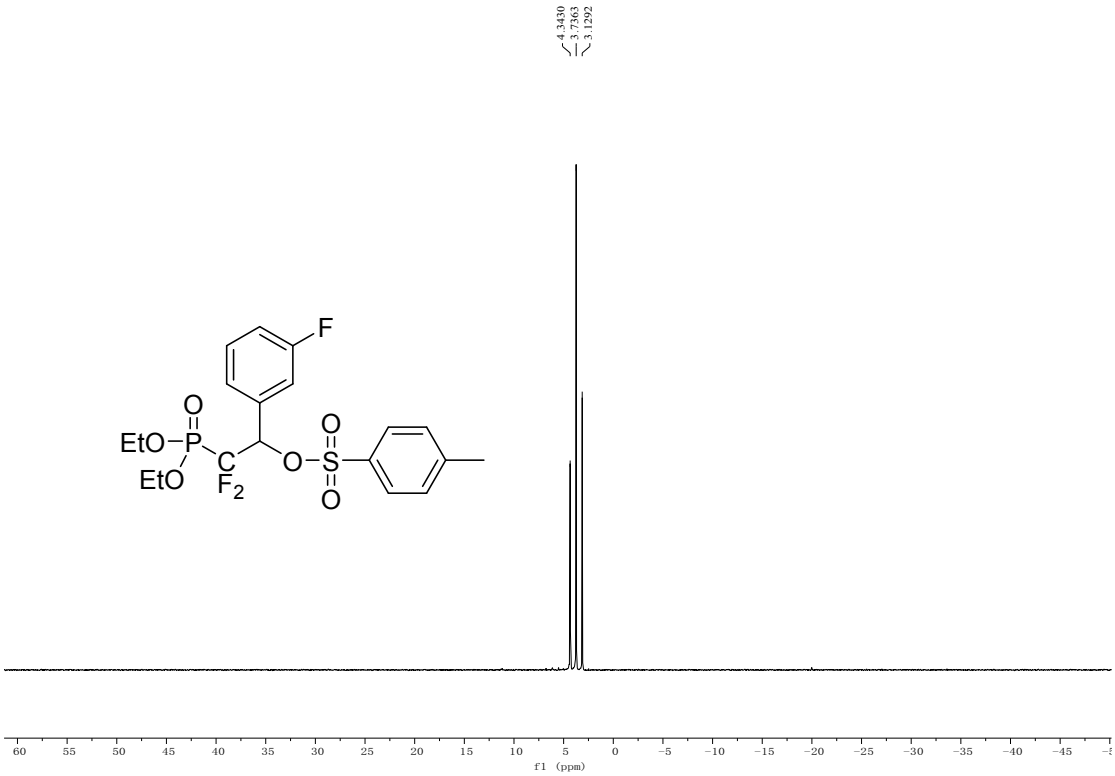


$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ca**:

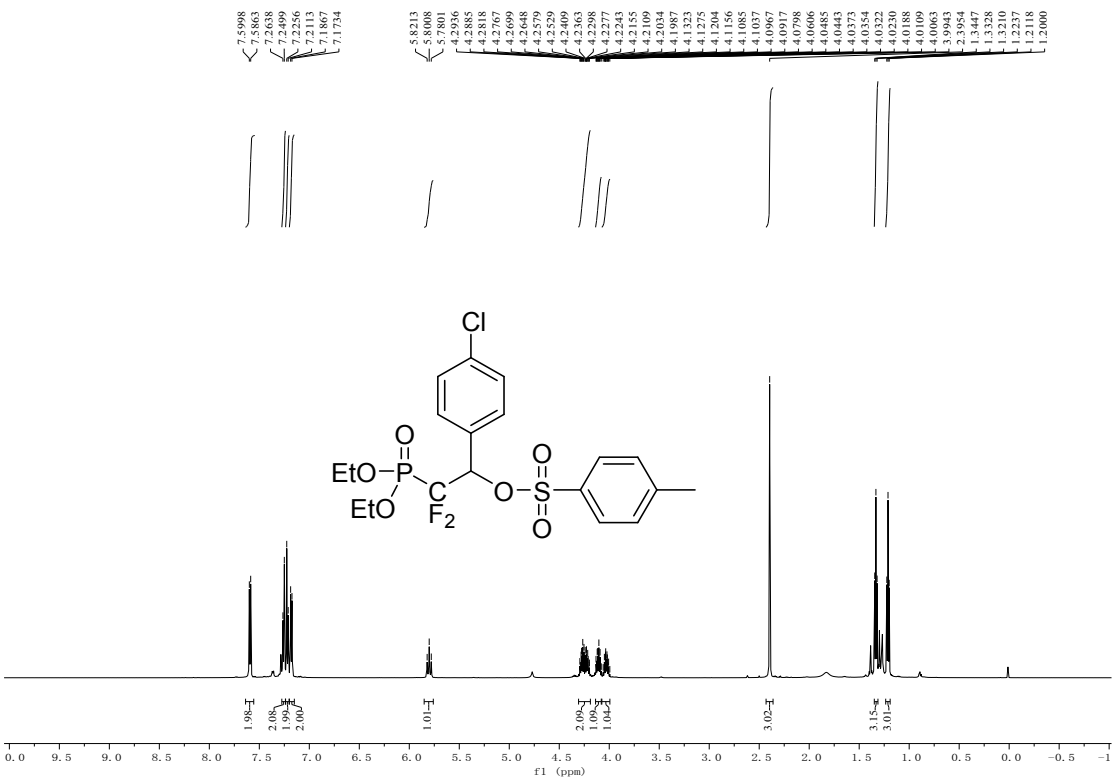


$^{19}$F NMR (376 MHz, $CDCl_3$) of **4ca**:

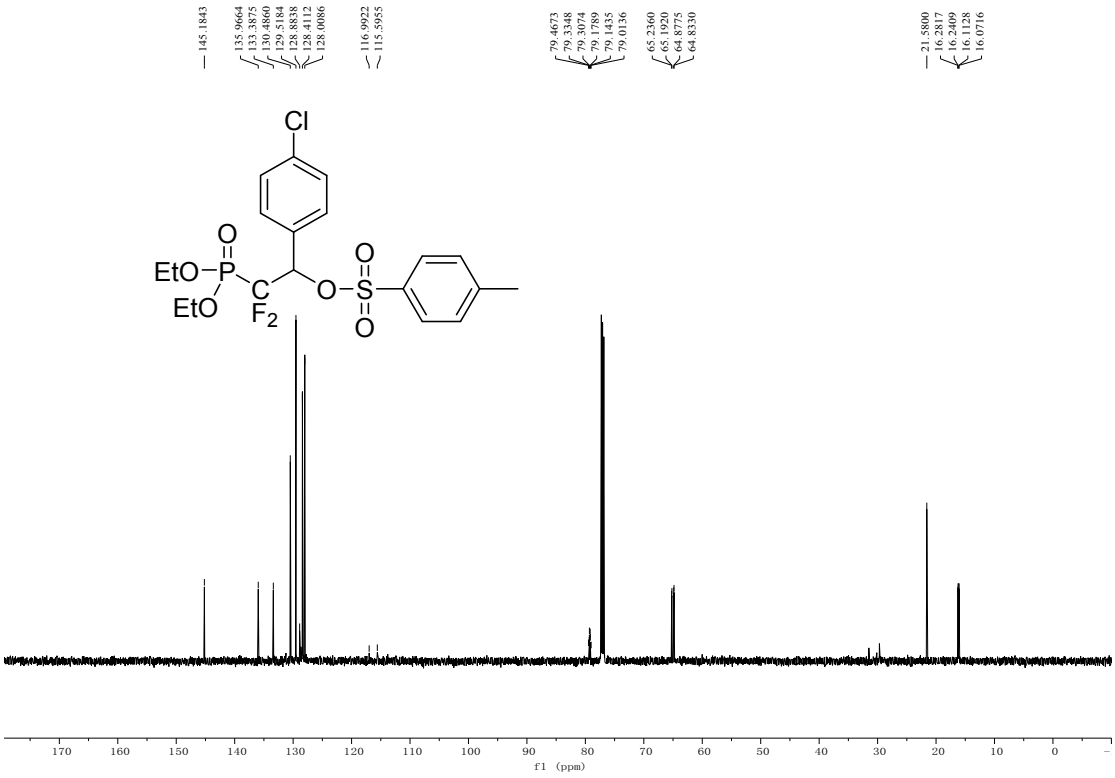

$^{31}$P NMR (162 MHz, $CDCl_3$) of **4ca**:



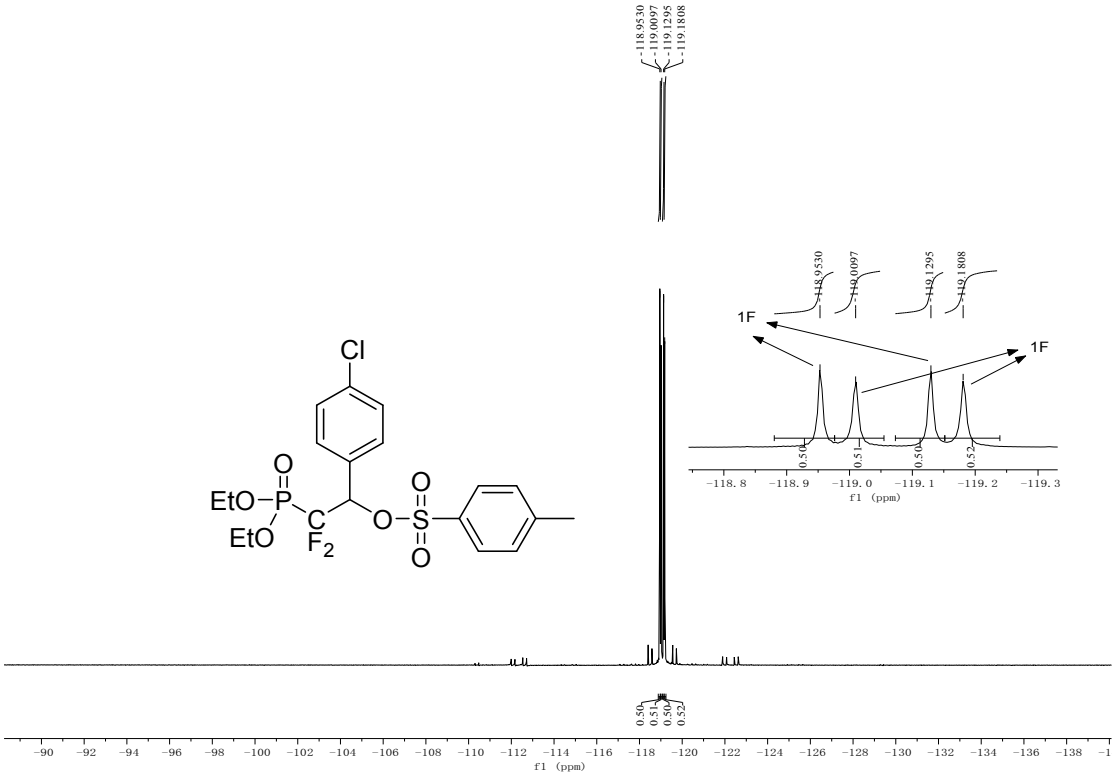
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4da**:



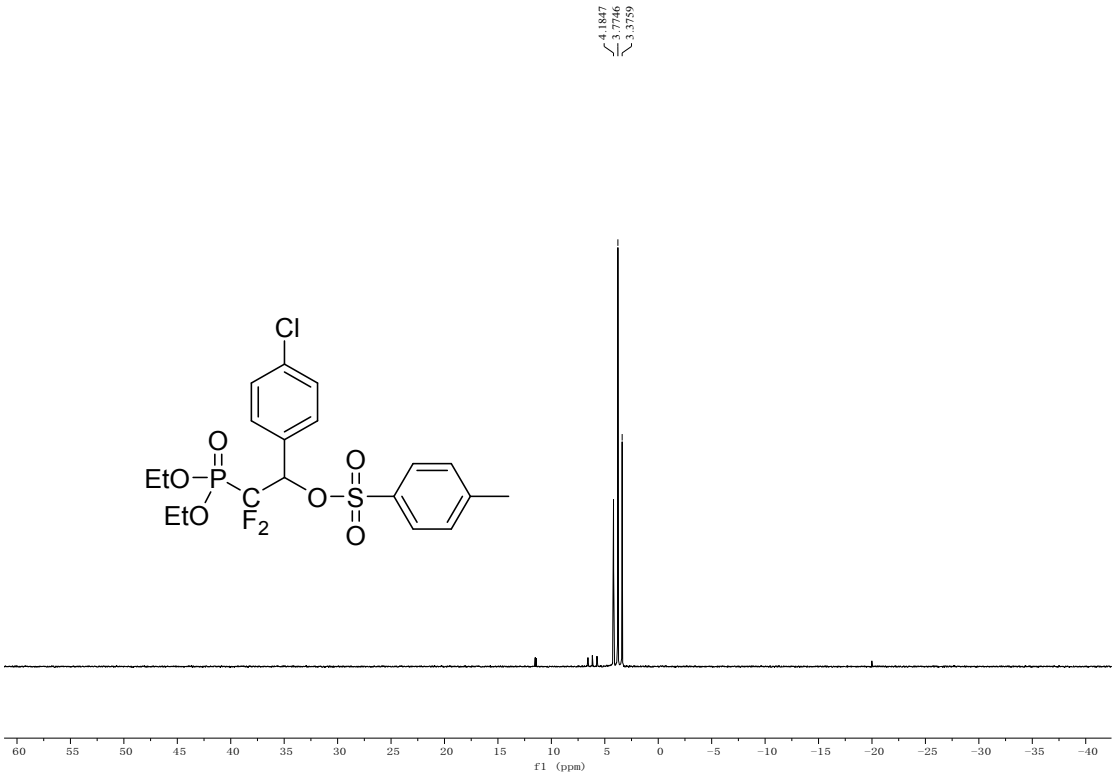

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4da**:



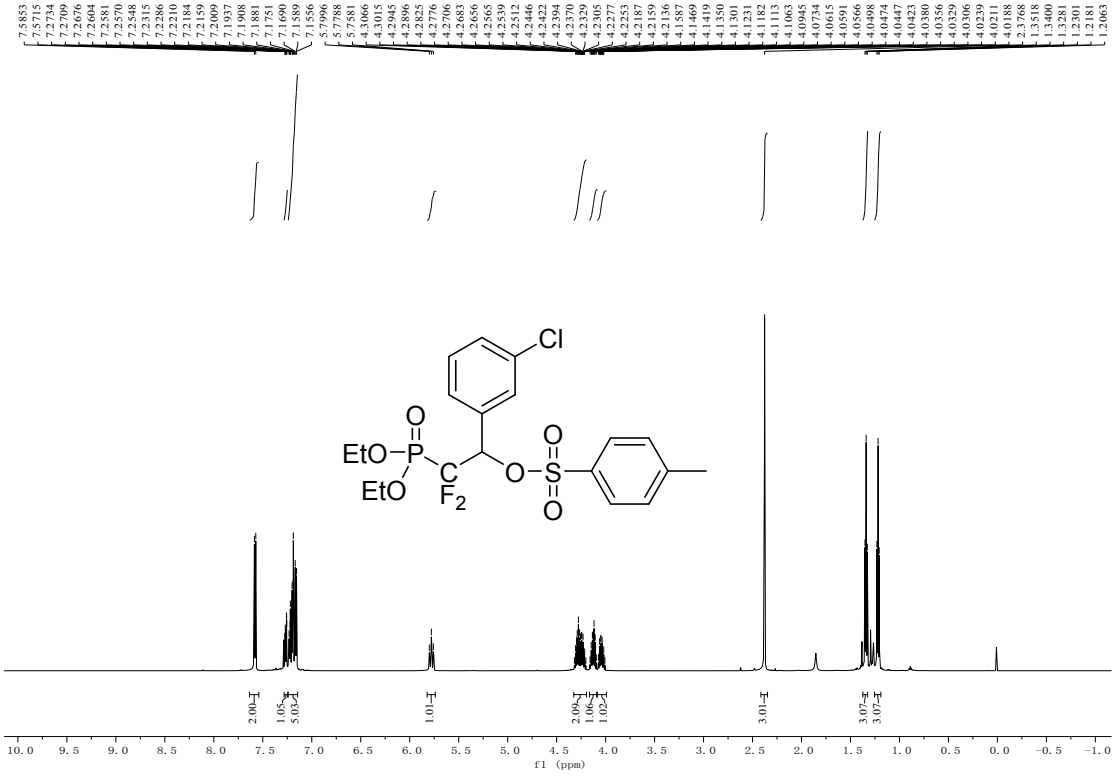
$^{19}F$ NMR (565 MHz, $CDCl_3$) of **4da**:



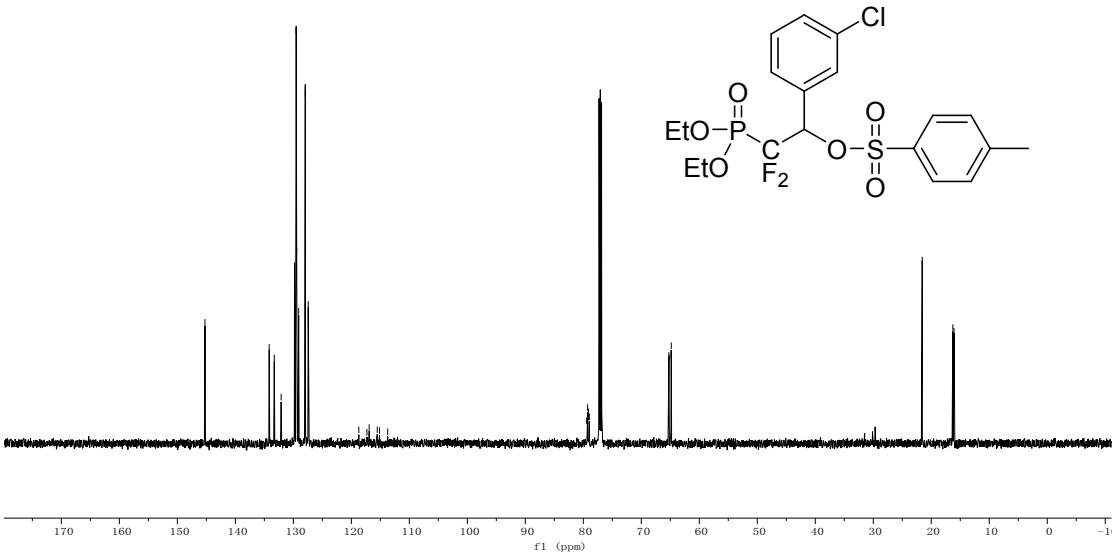

$^{31}P$ NMR (243 MHz, $CDCl_3$) of **4da**:



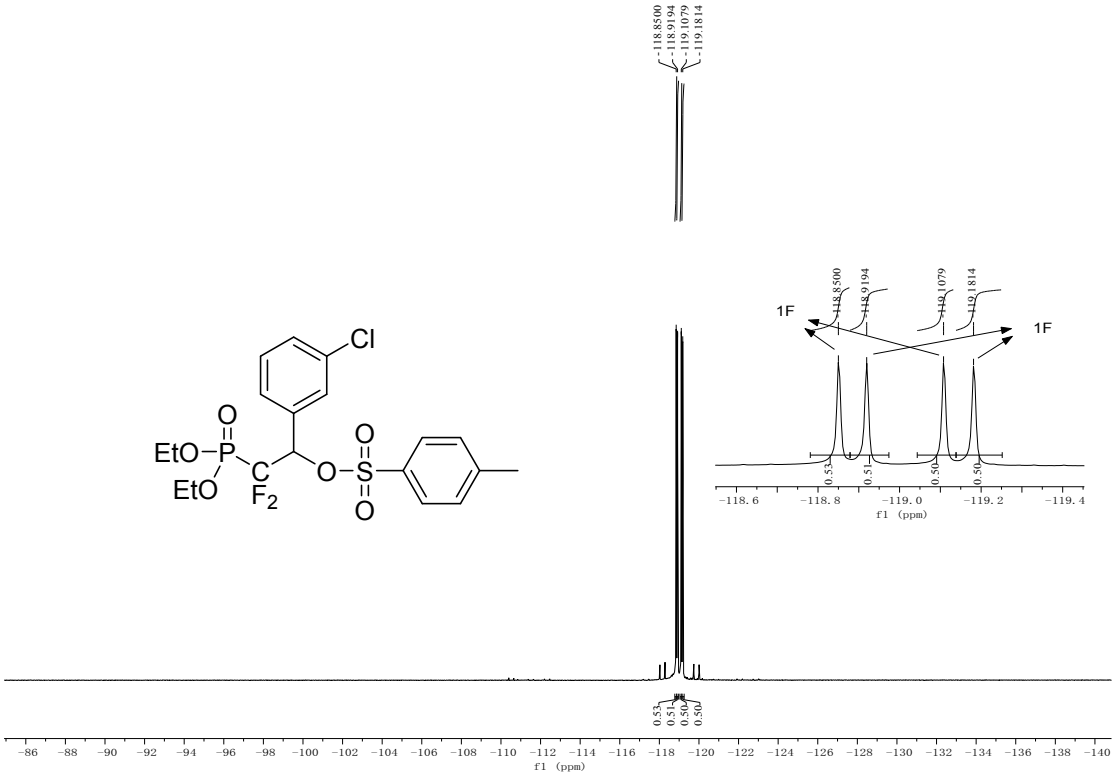
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4ea**:




$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ea**:

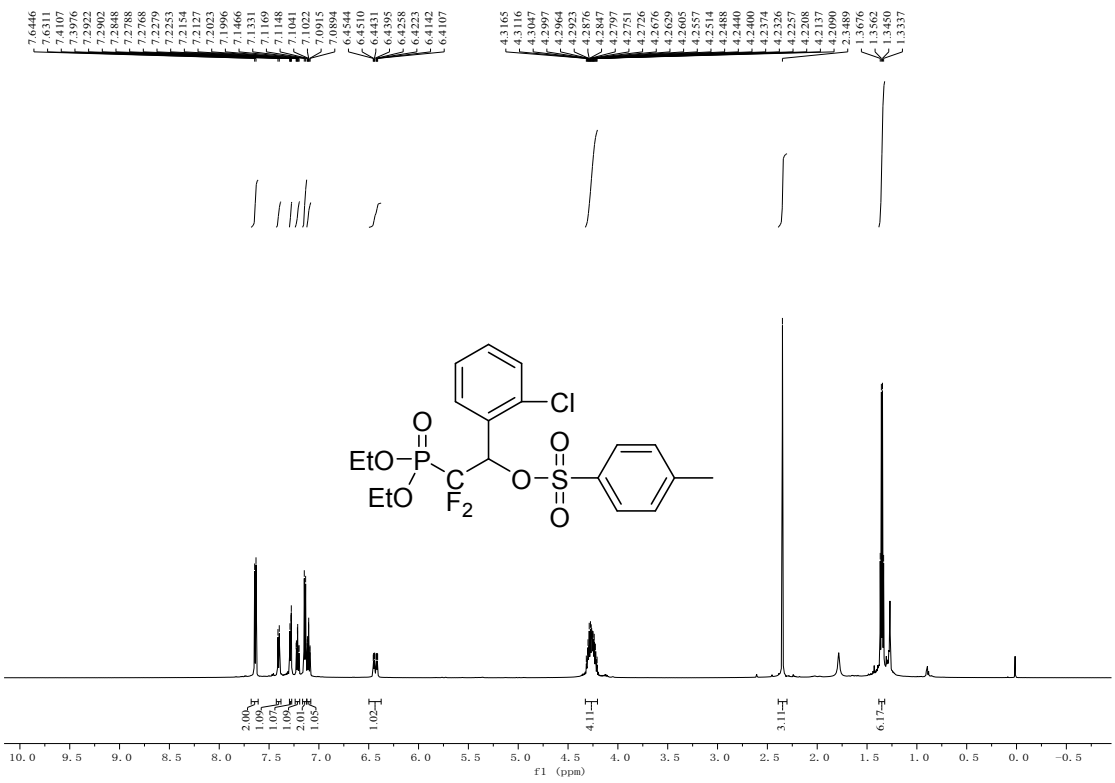


$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ea**:

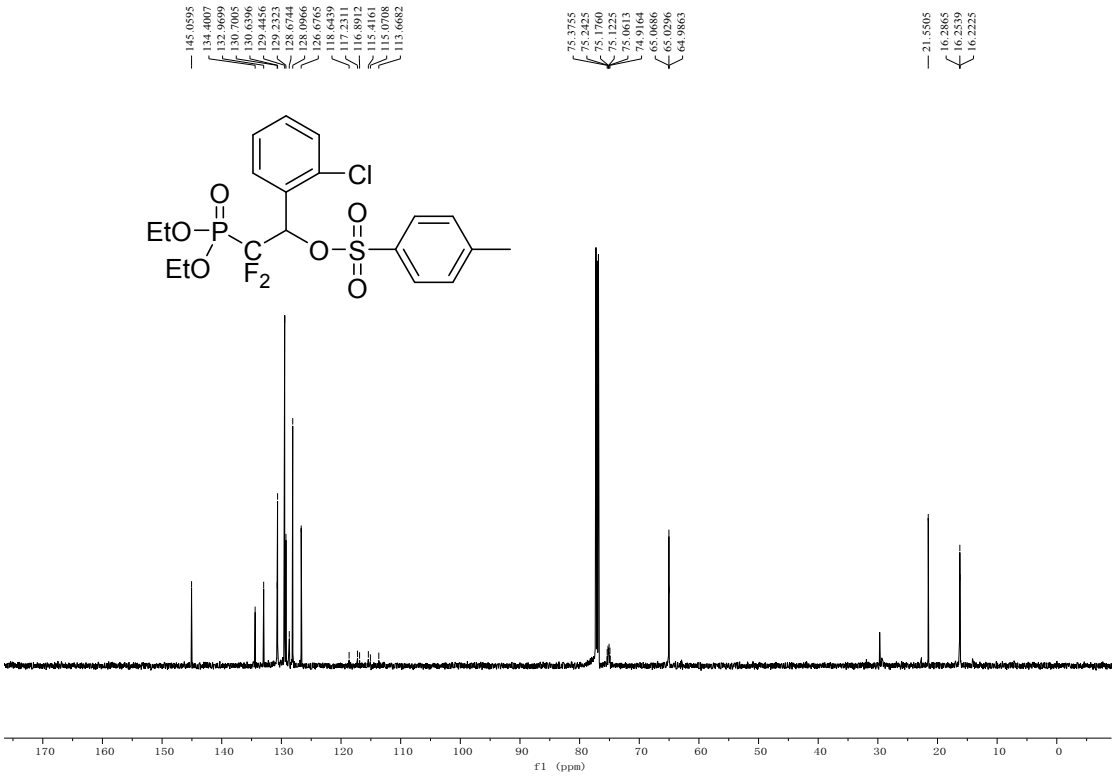



$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ea**:

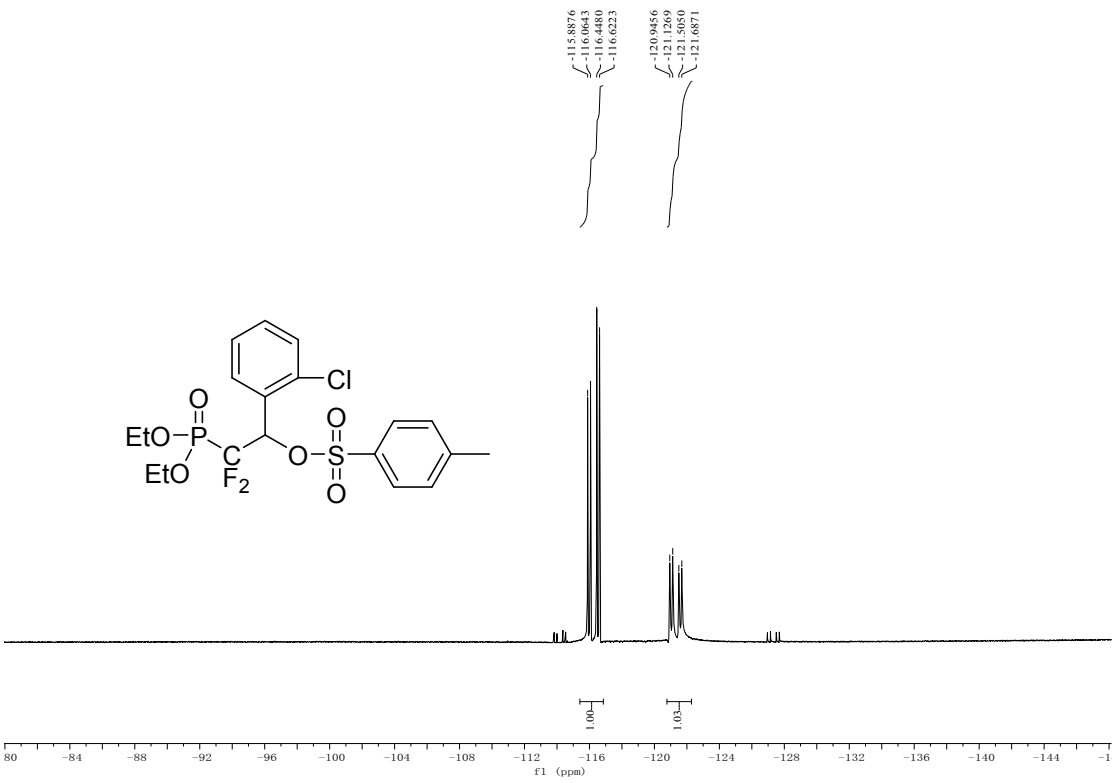
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4fa**:




$^{13}$C NMR (150 MHz, $CDCl_3$) of **4fa**:

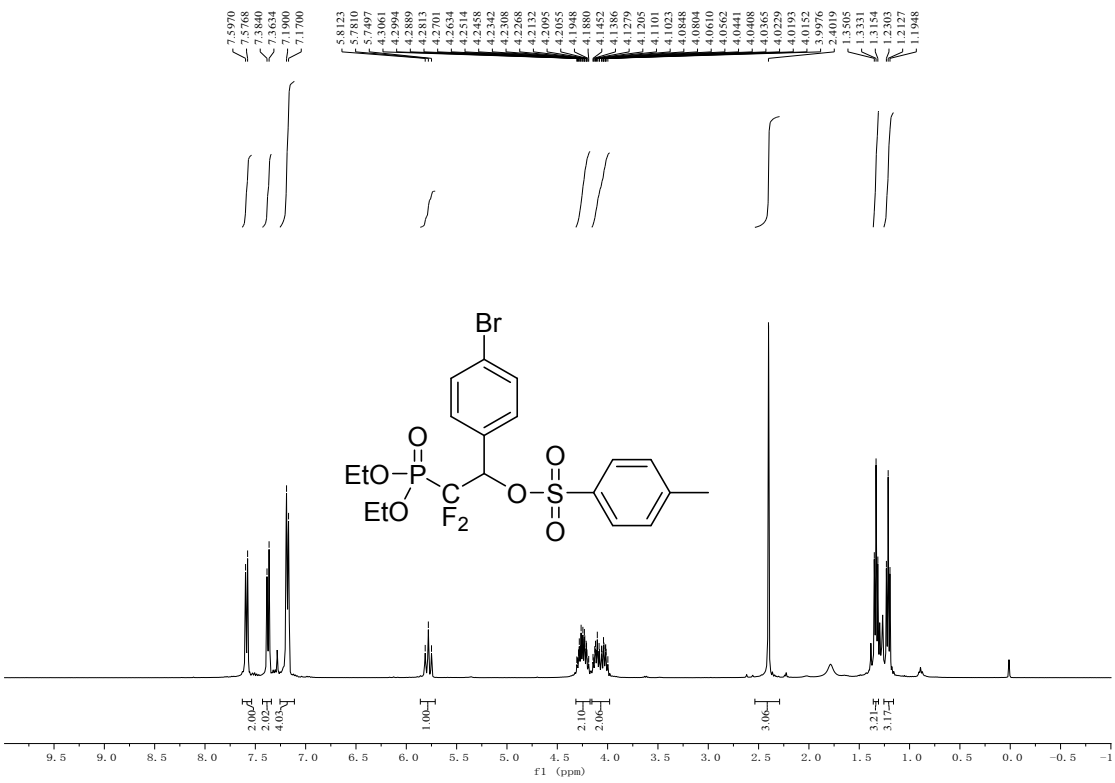


$^{19}$F NMR (565 MHz, $CDCl_3$) of **4fa**:

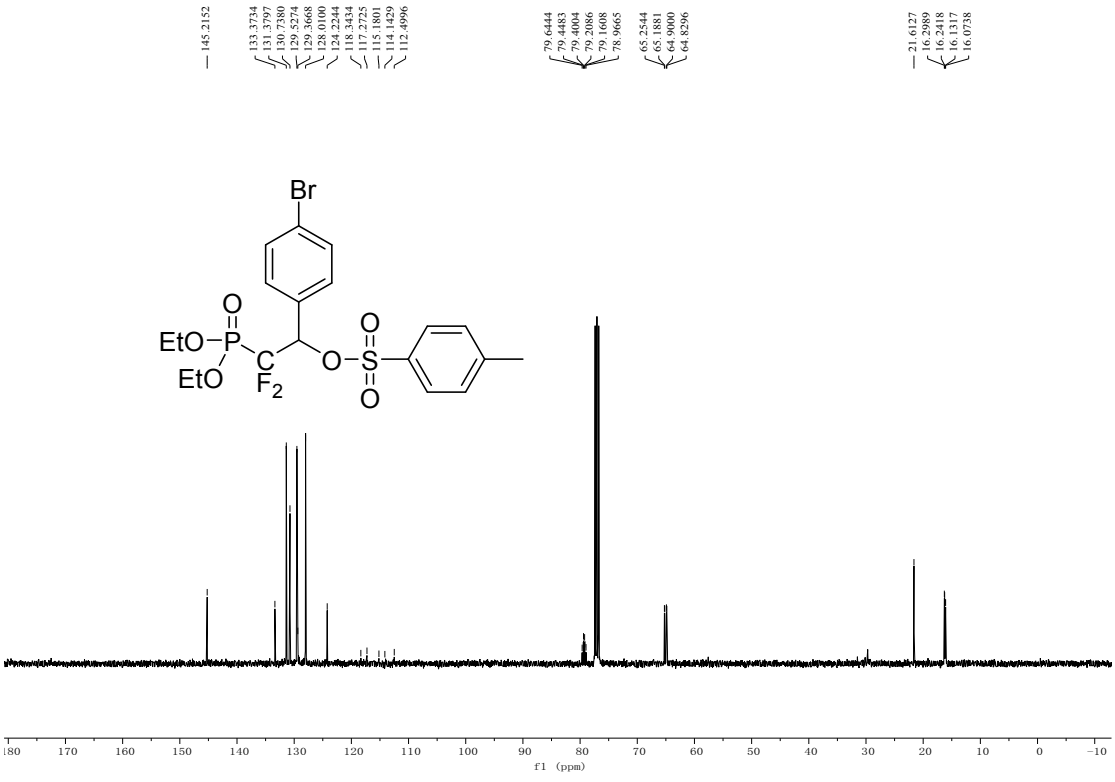



$^{31}$P NMR (243 MHz, $CDCl_3$) of **4fa**:

$^{1}$H NMR (400 MHz, $CDCl_3$) of **4ga**:

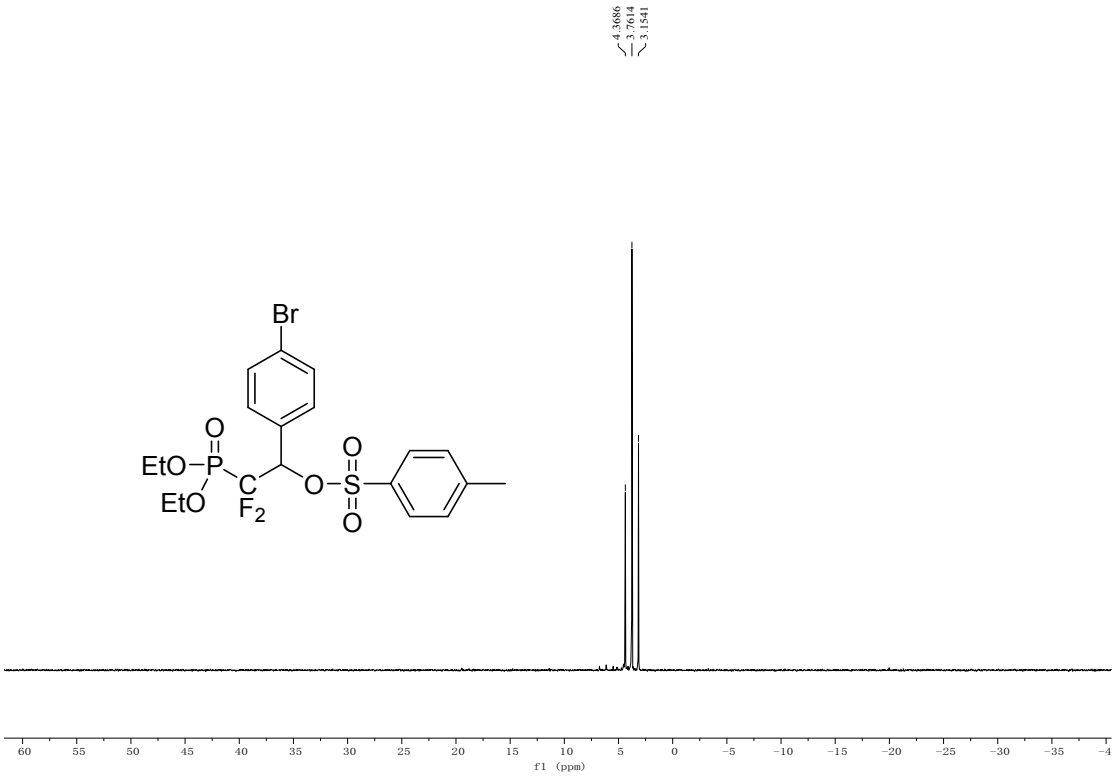



$^{13}$C NMR (100 MHz, $CDCl_3$) of **4ga**:

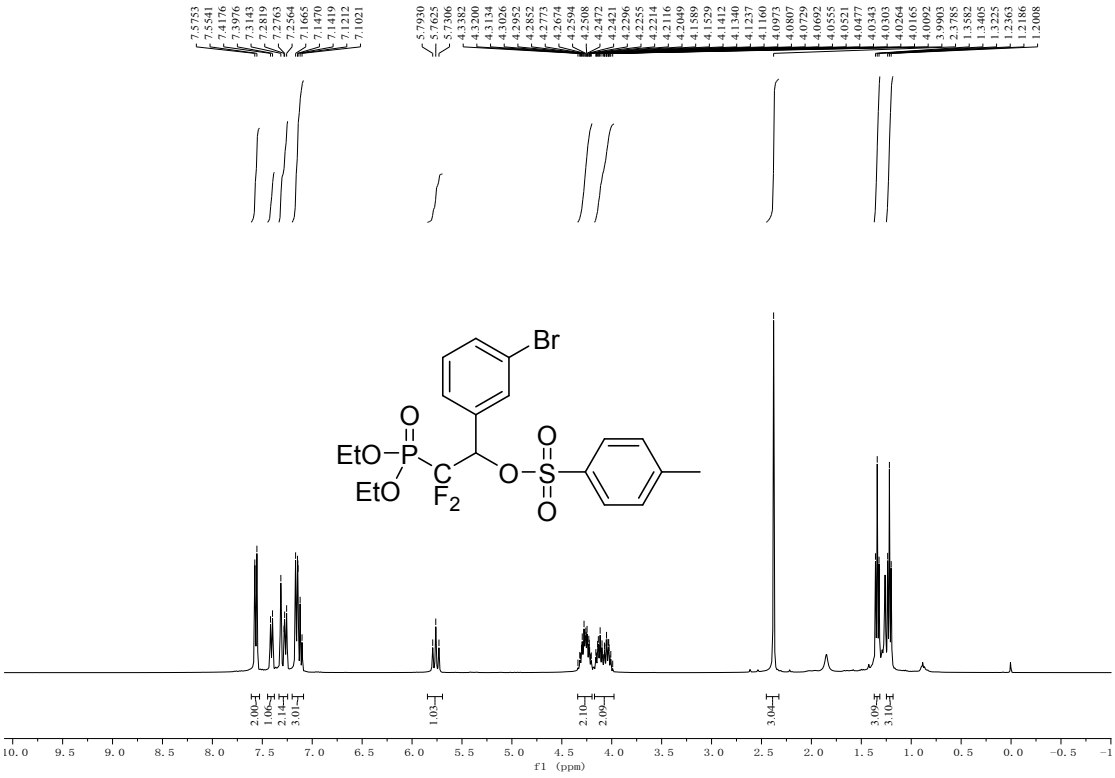


$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ga**:

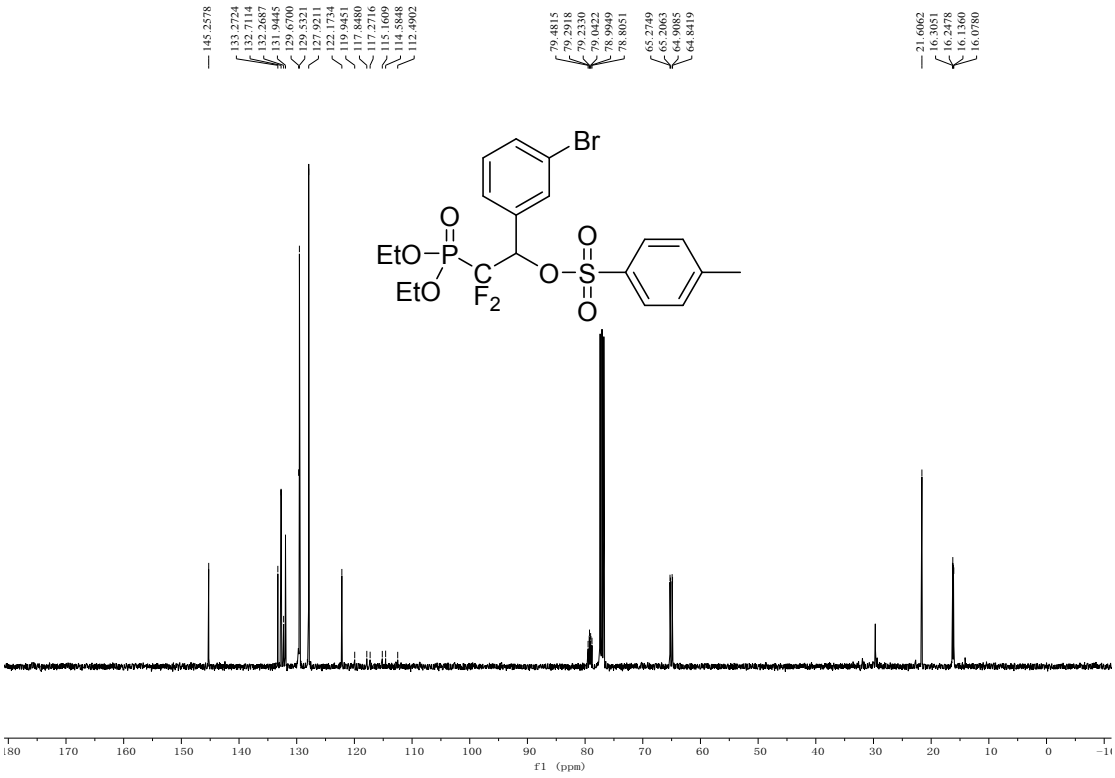

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ga**:



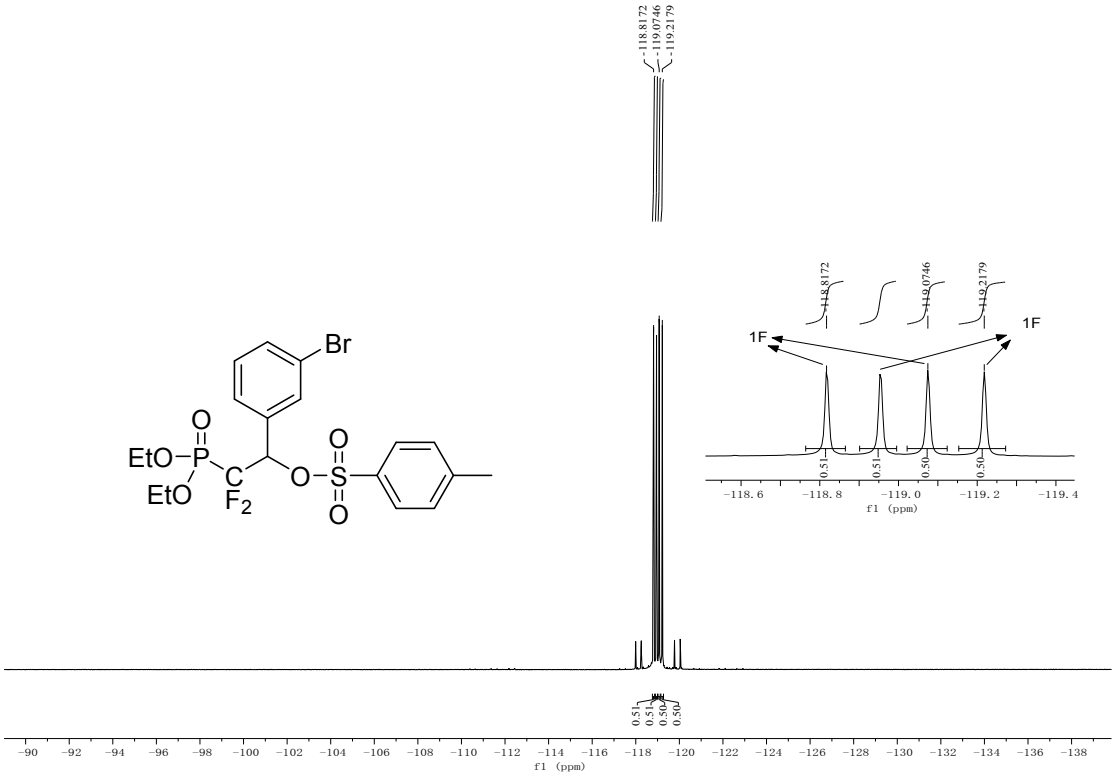
$^{1}$H NMR (400 MHz, $CDCl_3$) of **4ha**:




$^{13}$C NMR (100 MHz, $CDCl_3$) of **4ha**:

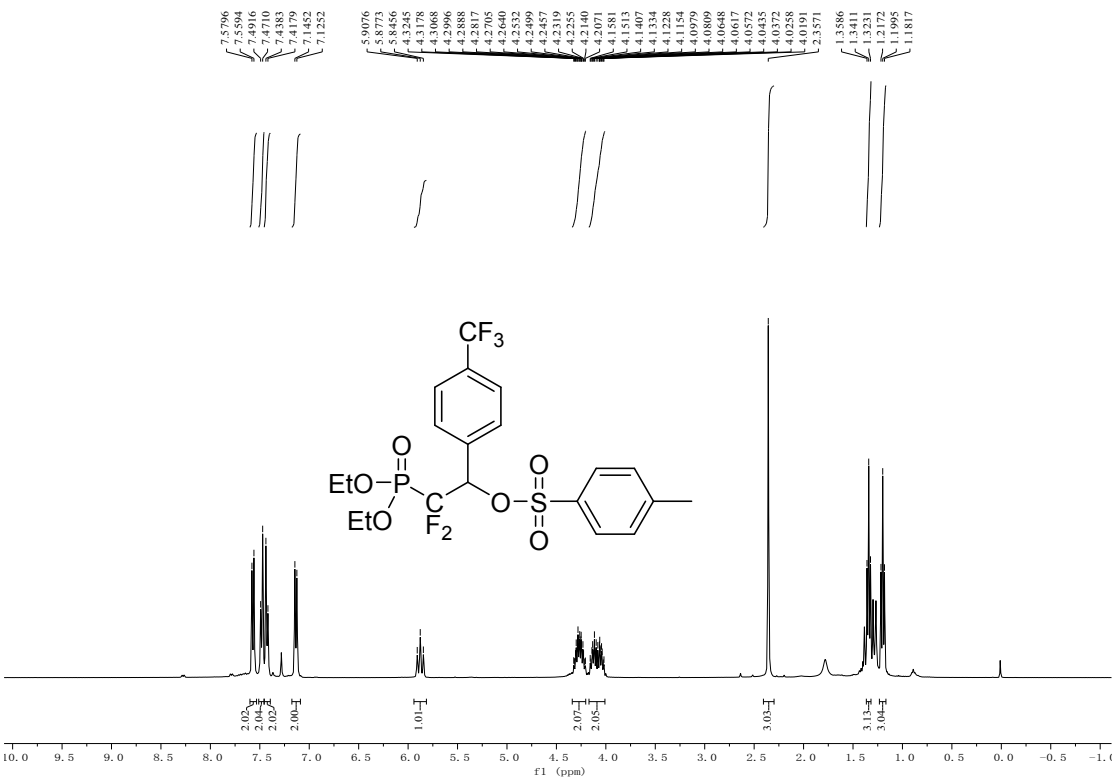


$^{19}$F NMR (376 MHz, $CDCl_3$) of **4ha**:

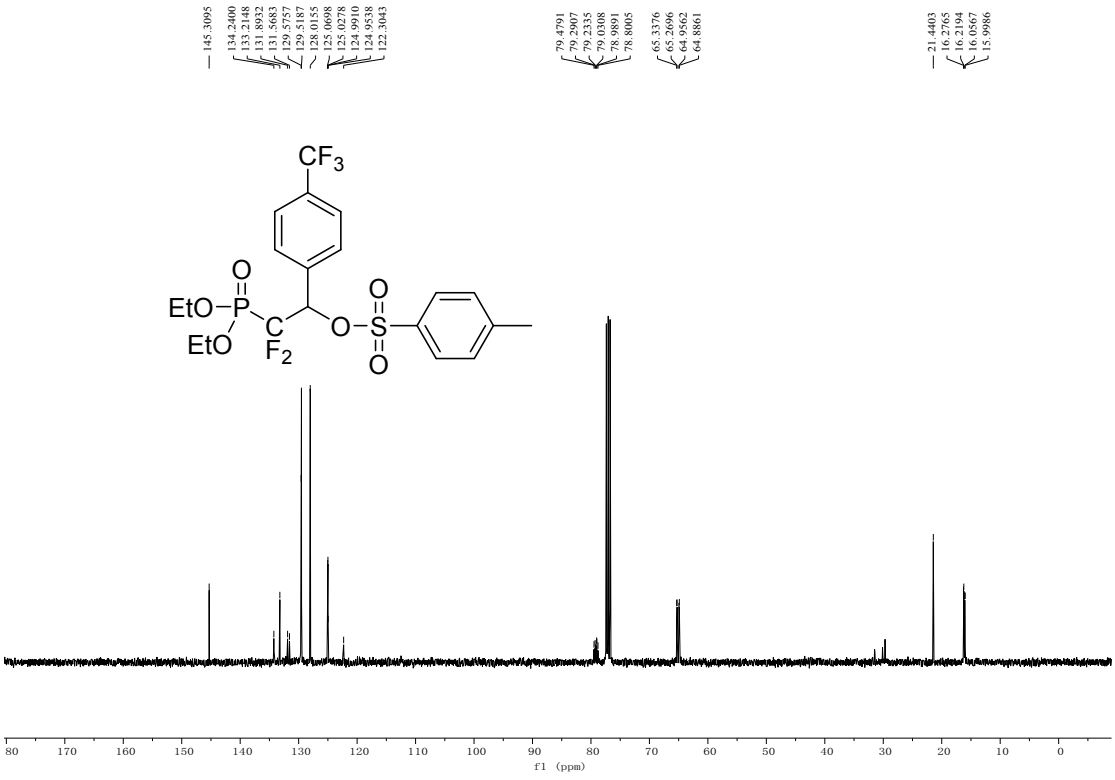



$^{31}$P NMR (162 MHz, $CDCl_3$) of **4ha**:

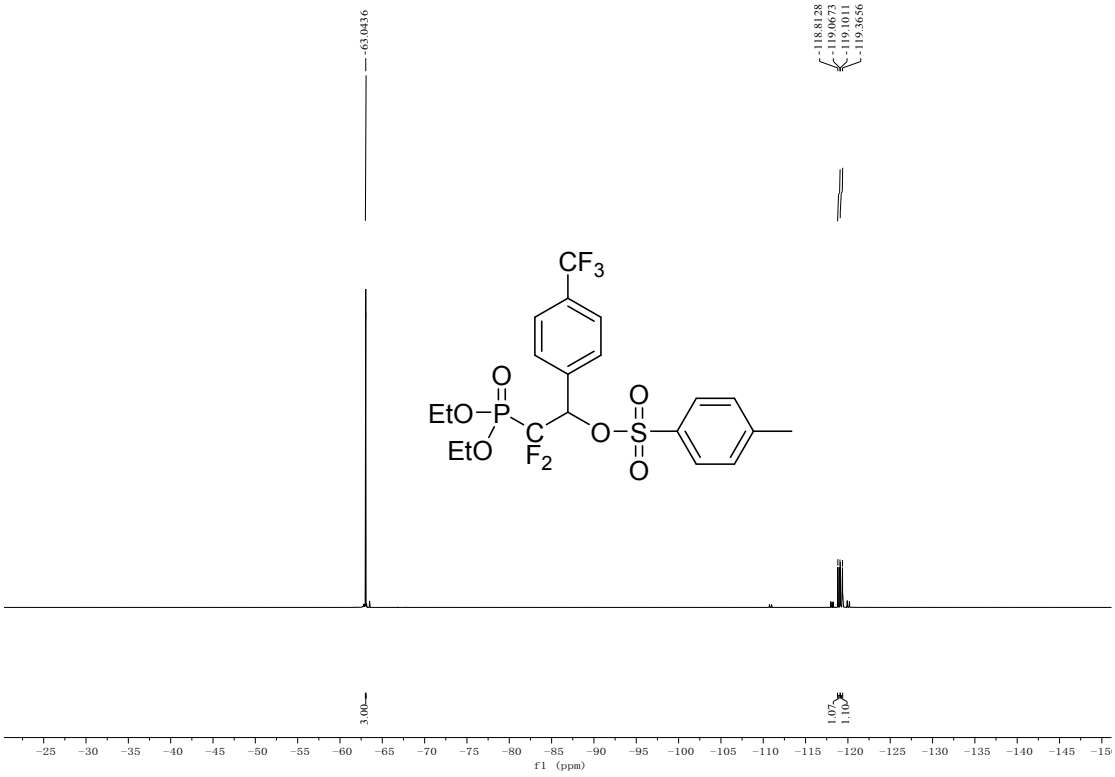
$^{1}$H NMR (400 MHz, $CDCl_3$) of **4ia**:



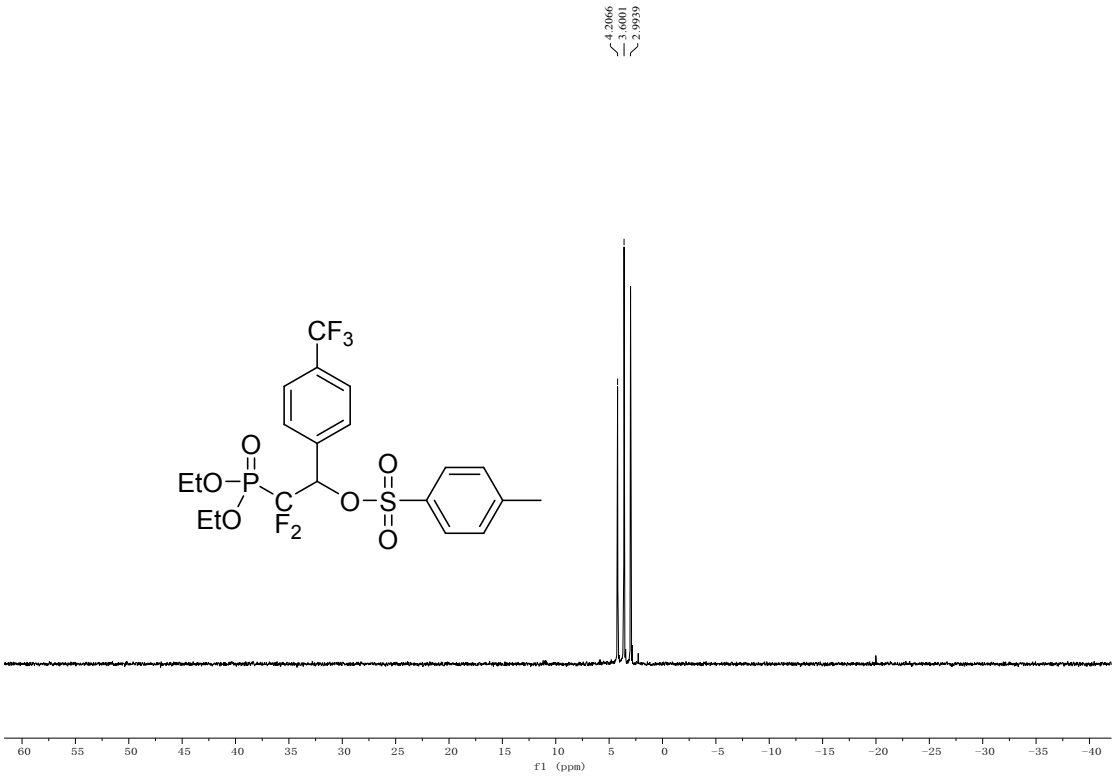

$^{13}$C NMR (100 MHz, $CDCl_3$) of **4ia**:



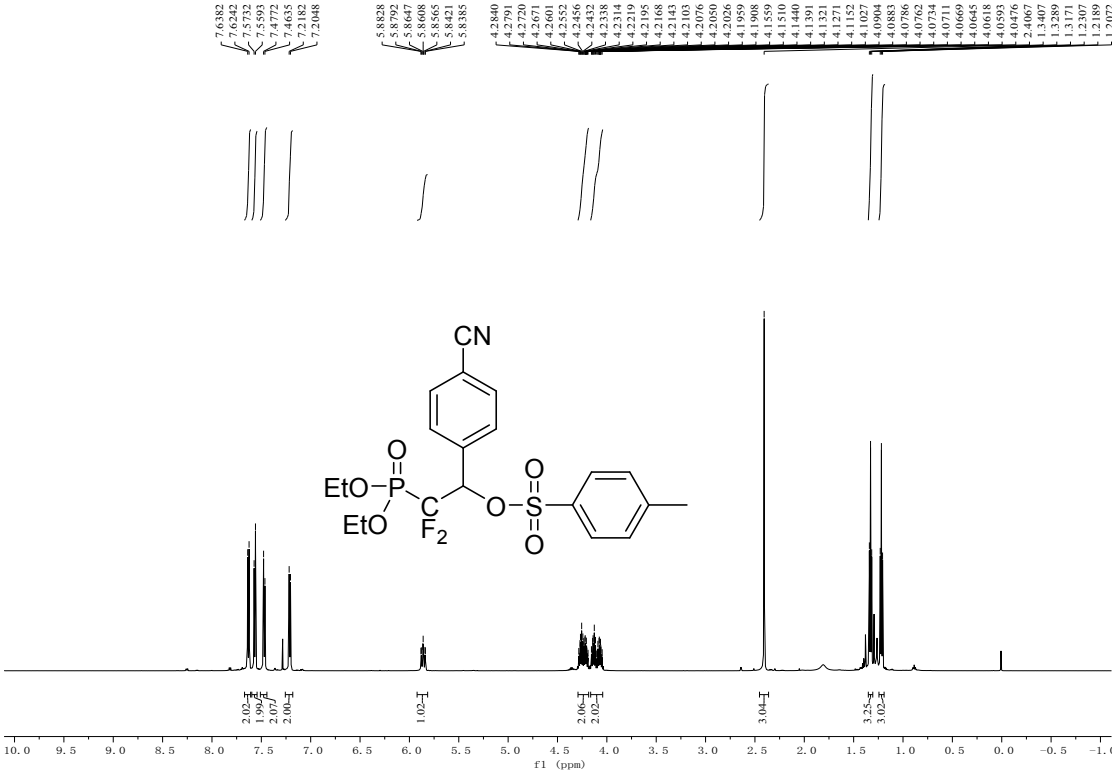
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ia**:



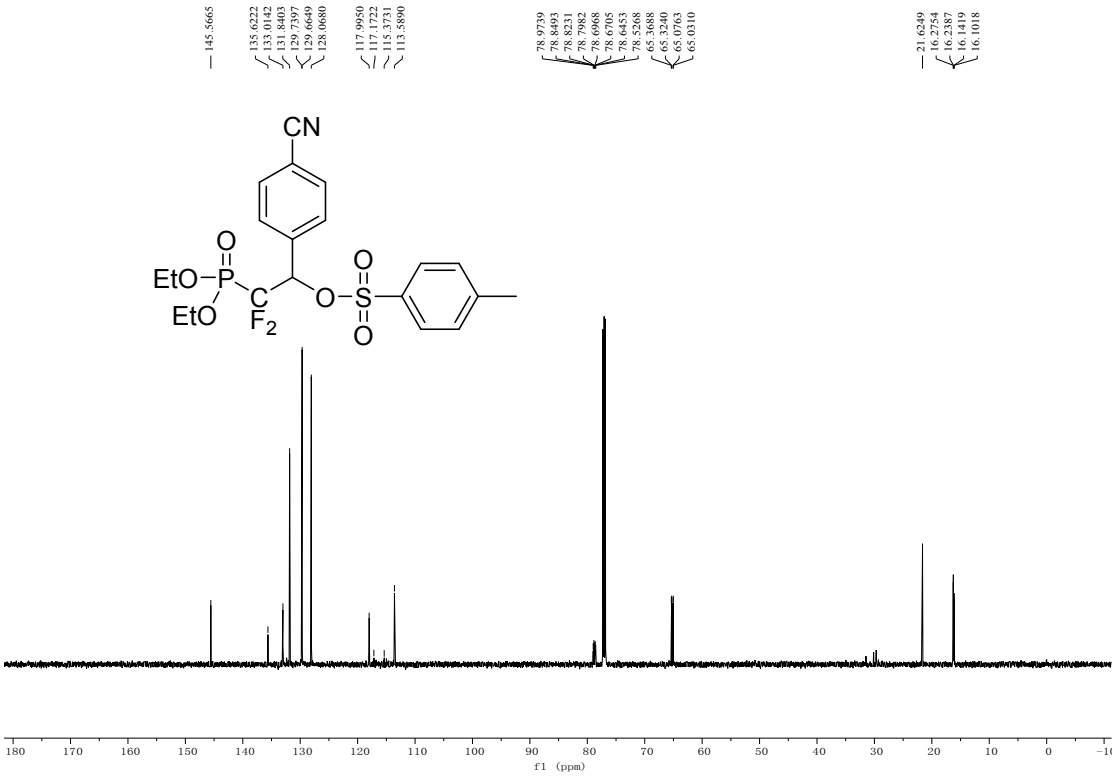

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ia**:



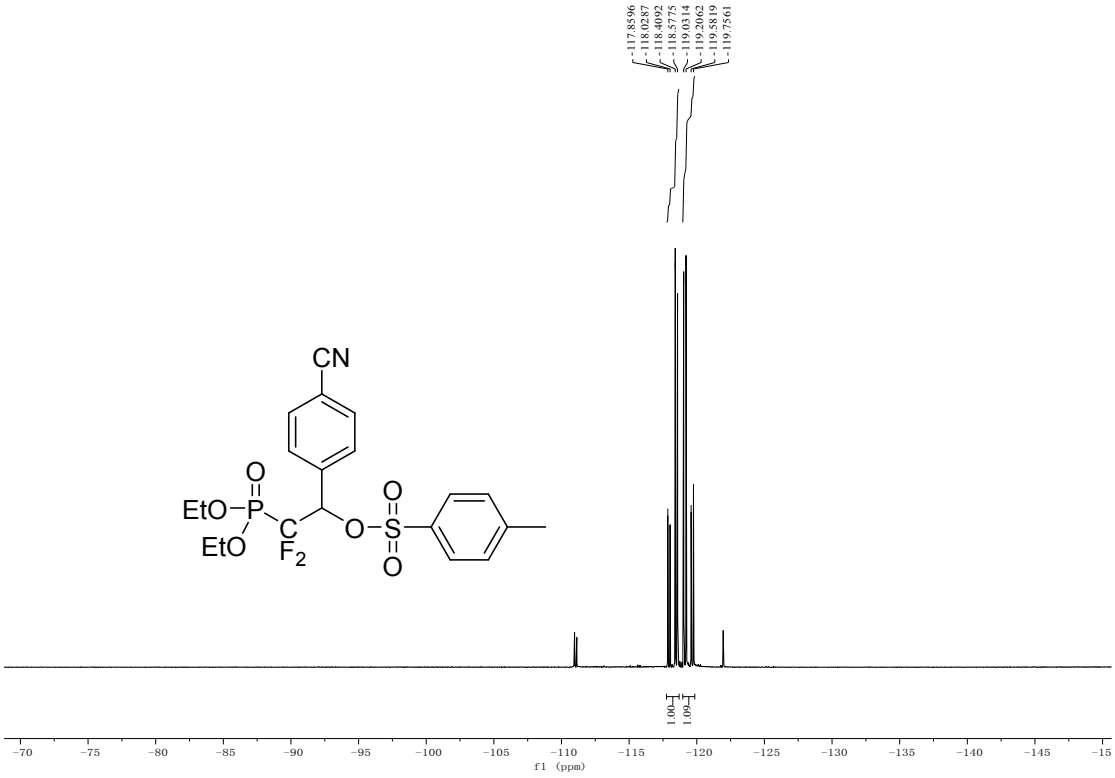
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4ja**:



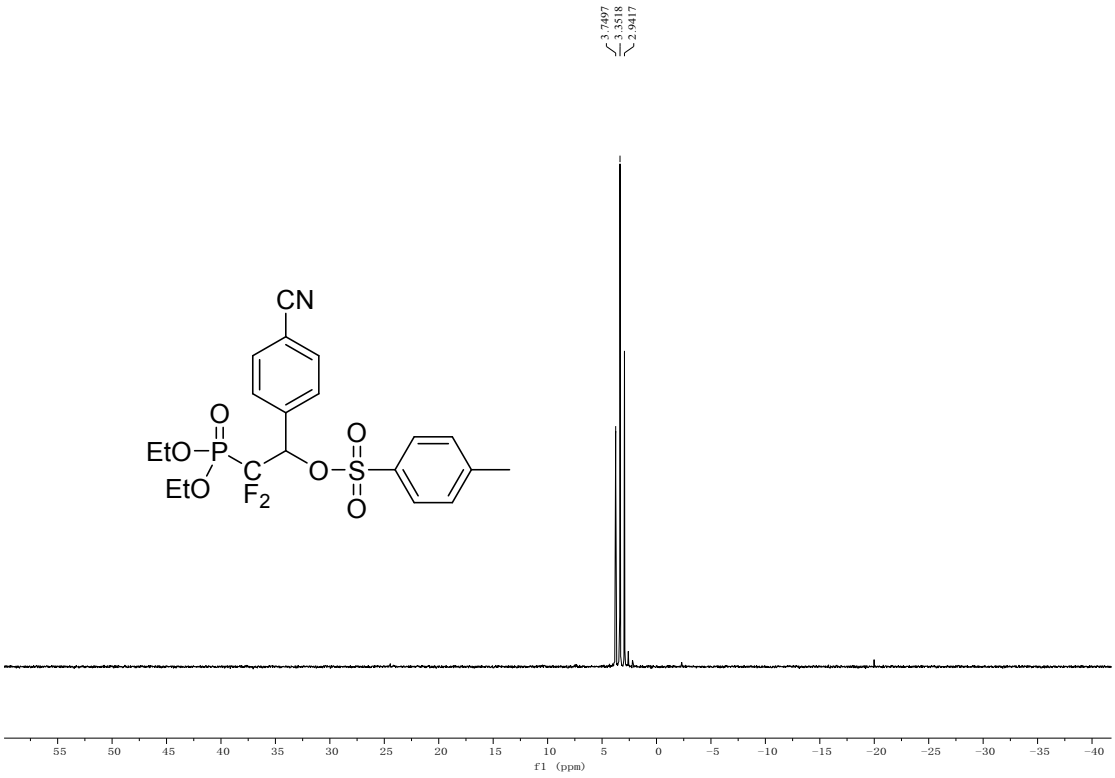

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ja**:



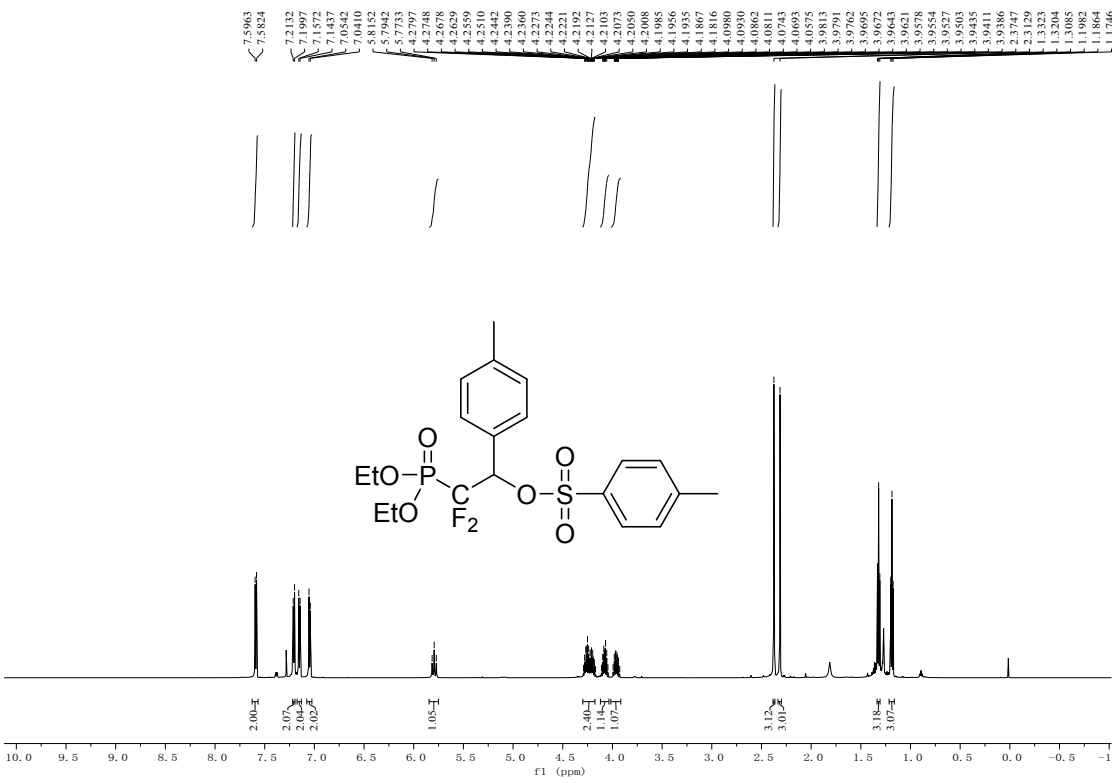
$^{19}F$ NMR (565 MHz, $CDCl_3$) of **4ja**:



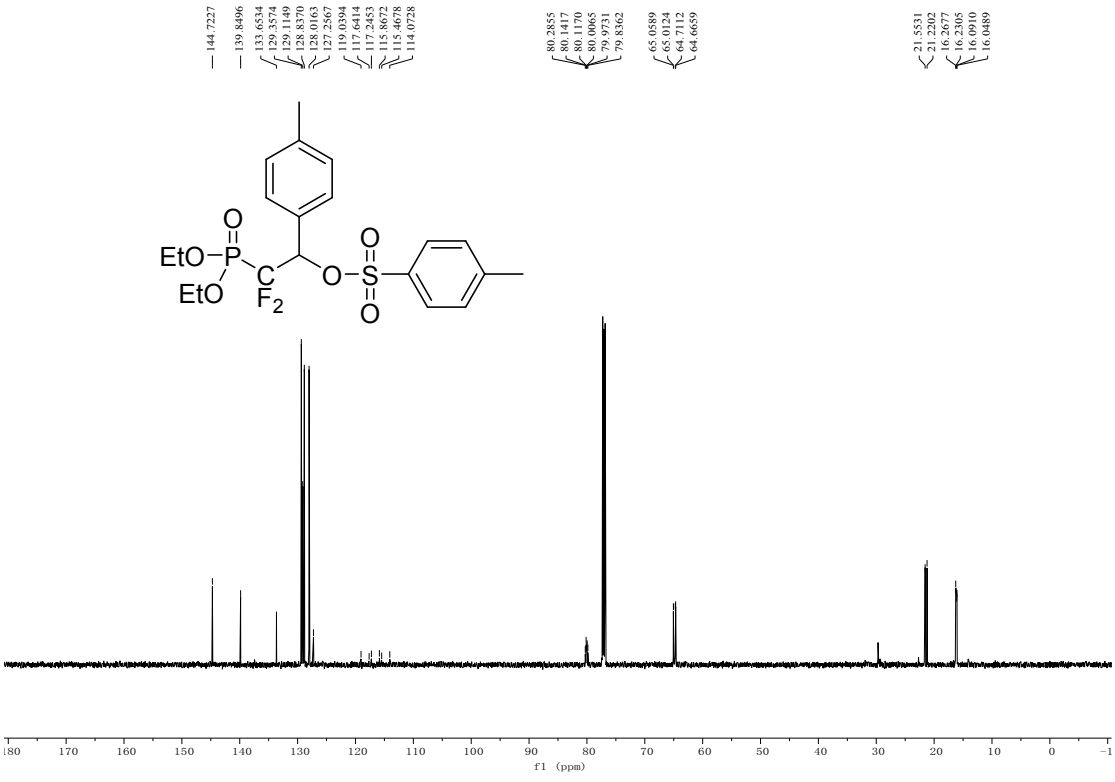

$^{31}P$ NMR (243 MHz, $CDCl_3$) of **4ja**:



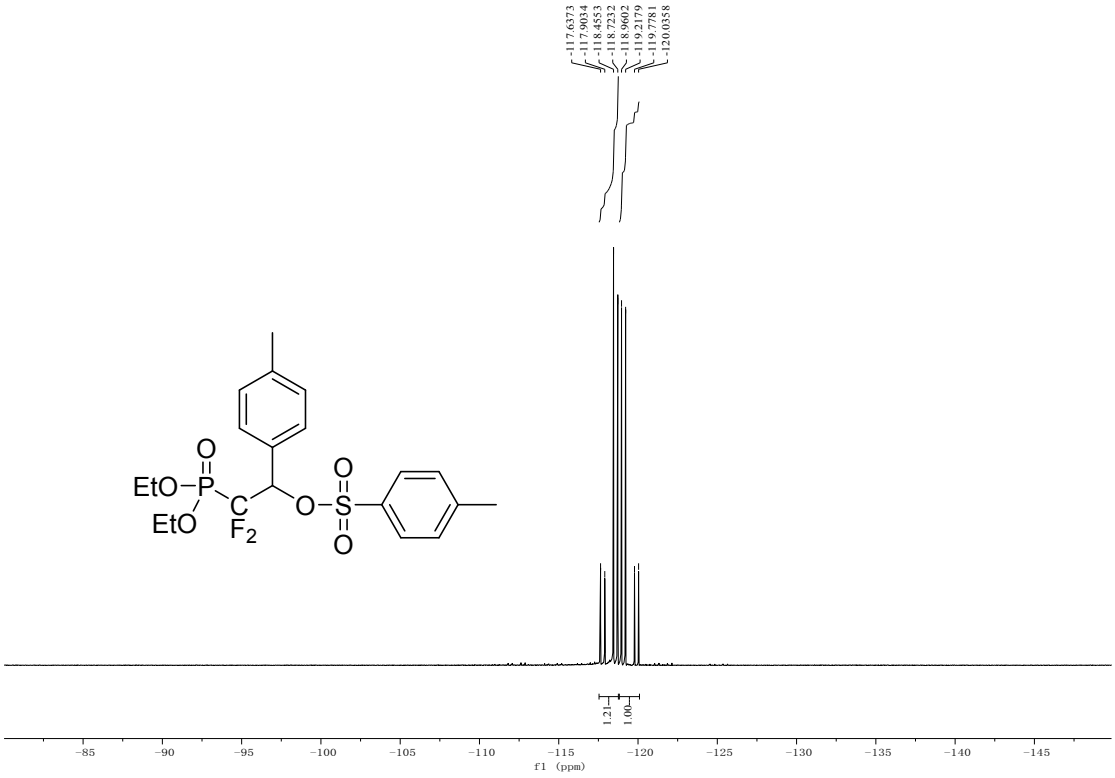
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4ka**:



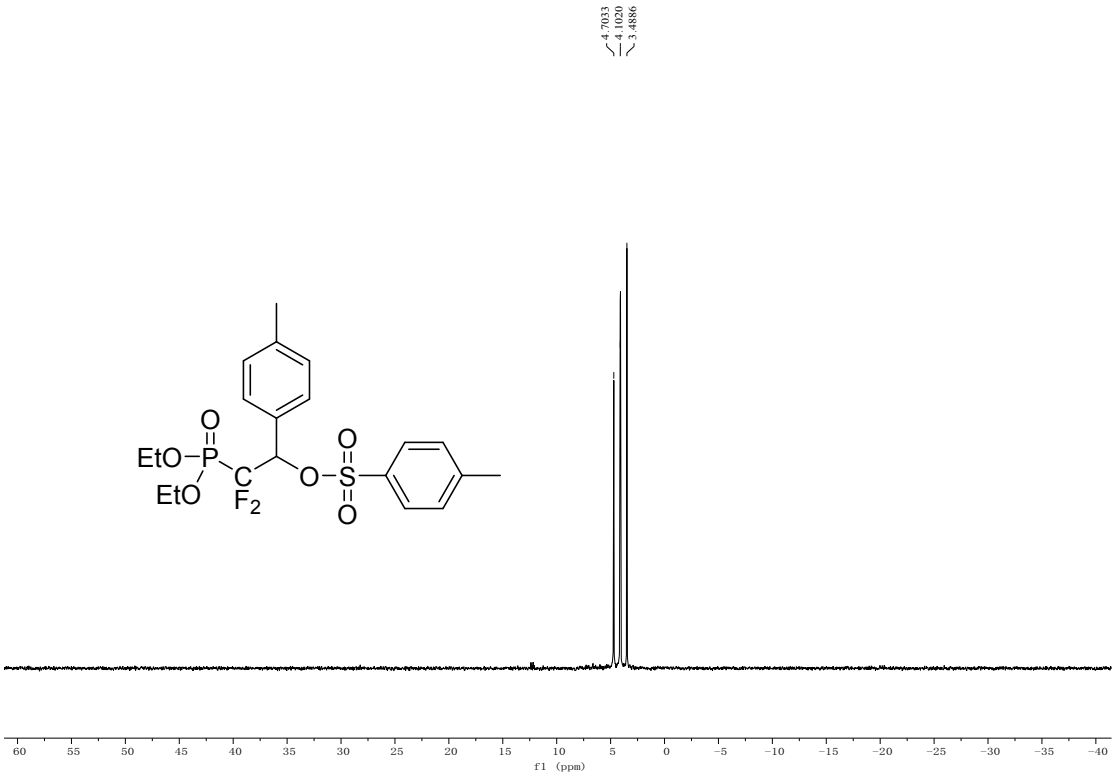
$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ka**:



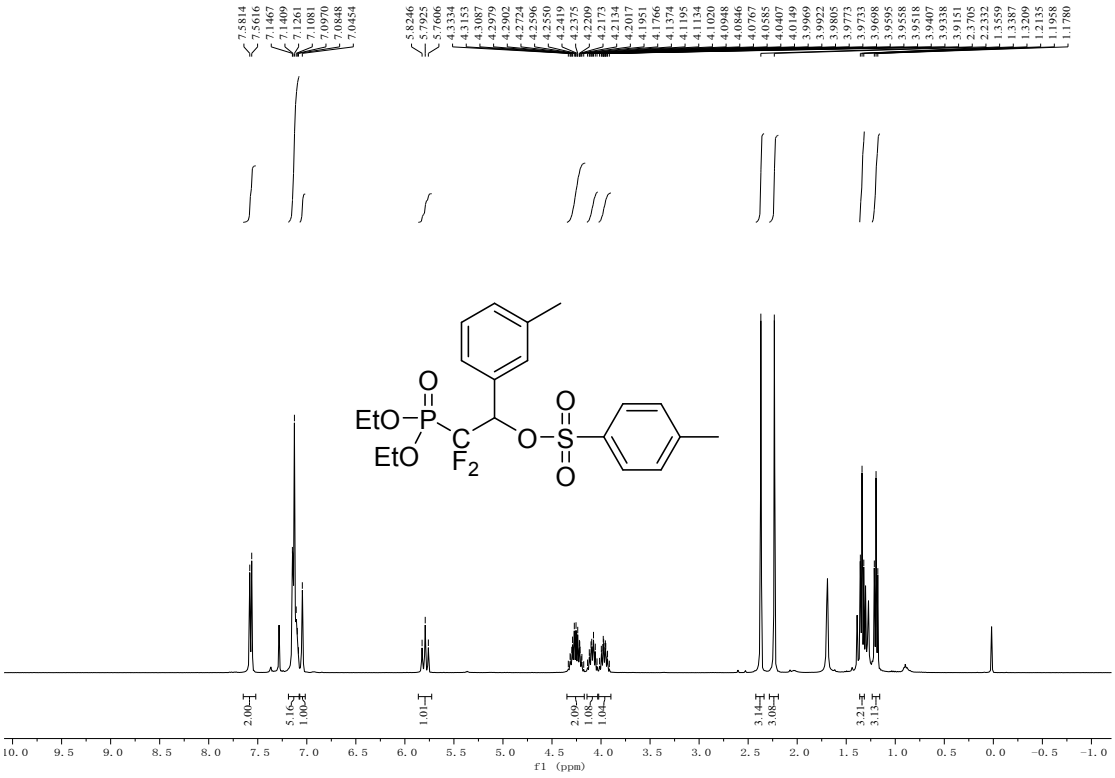
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ka**:



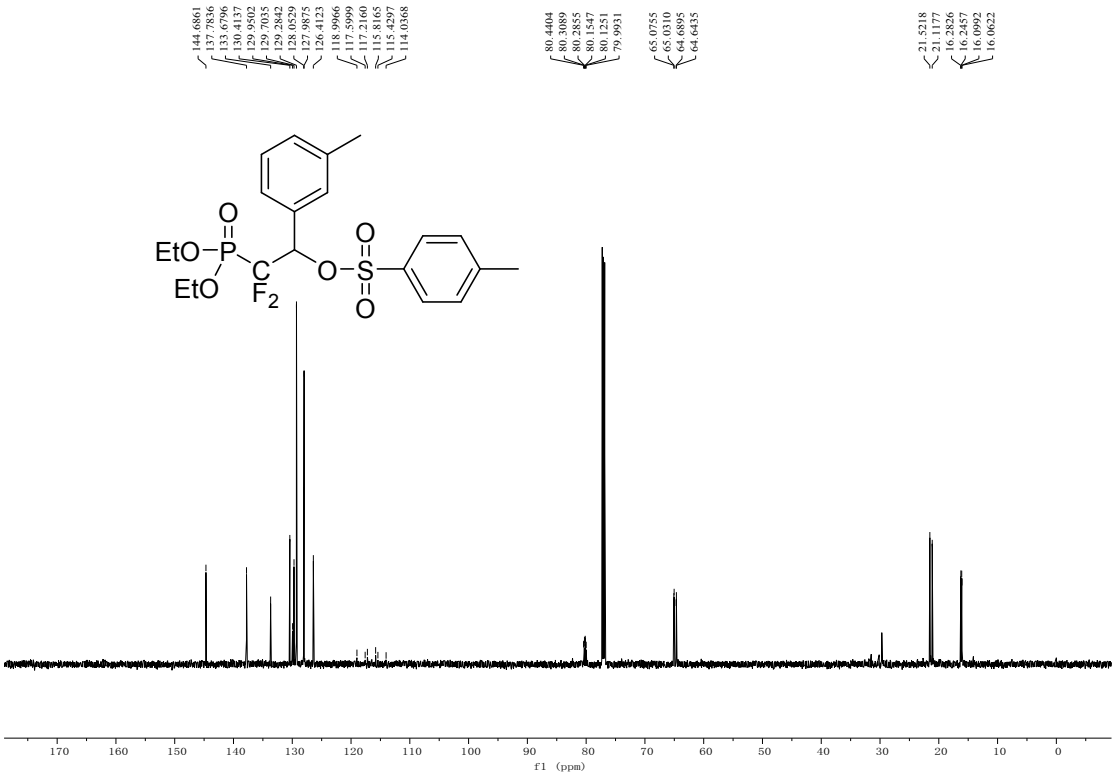

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ka**:



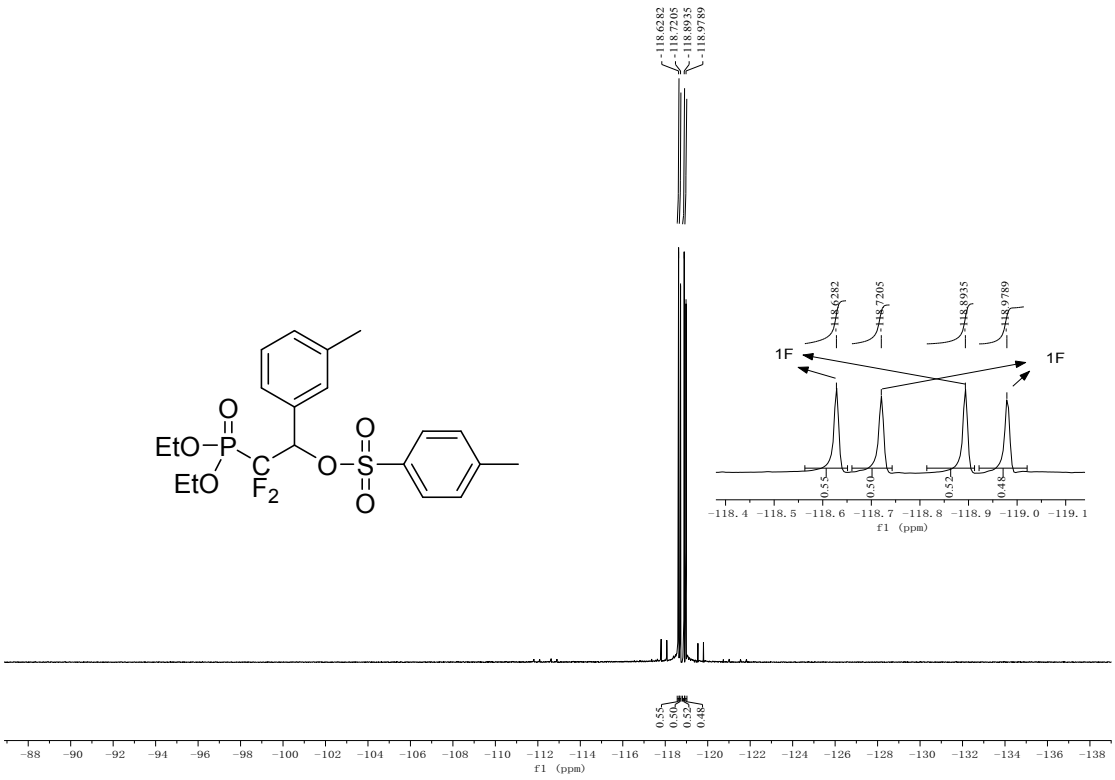
$^{1}$H NMR (400 MHz, $CDCl_3$) of **4la**:



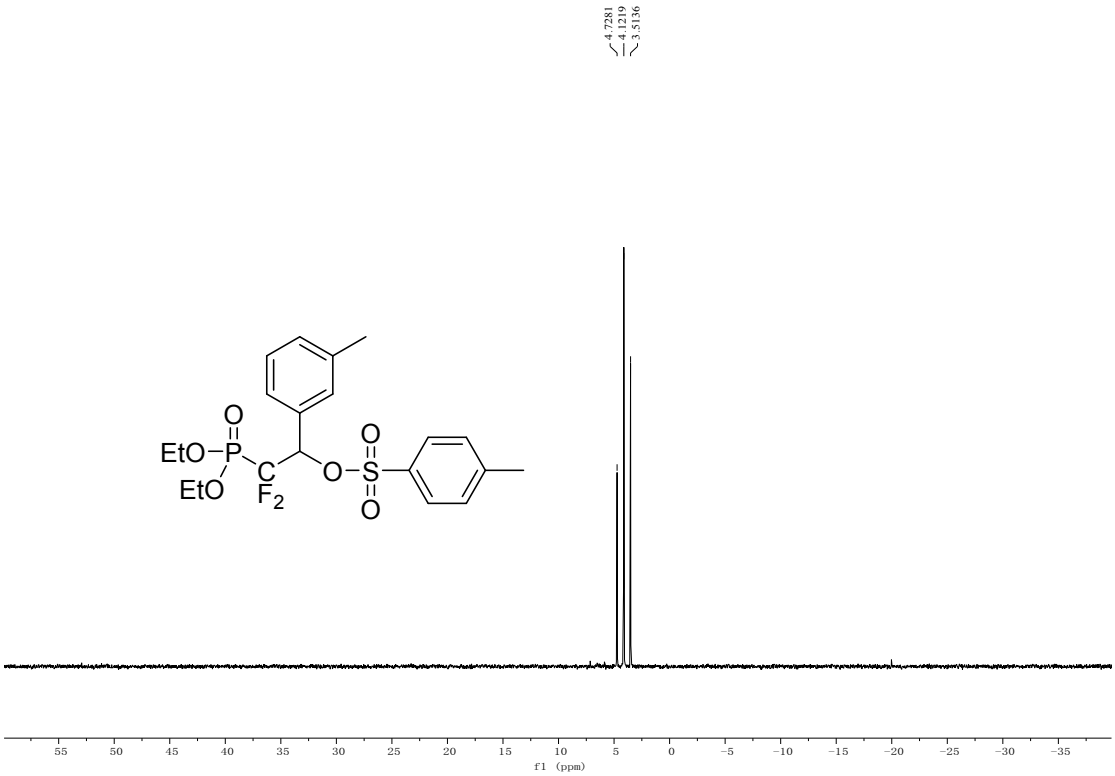

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4la**:



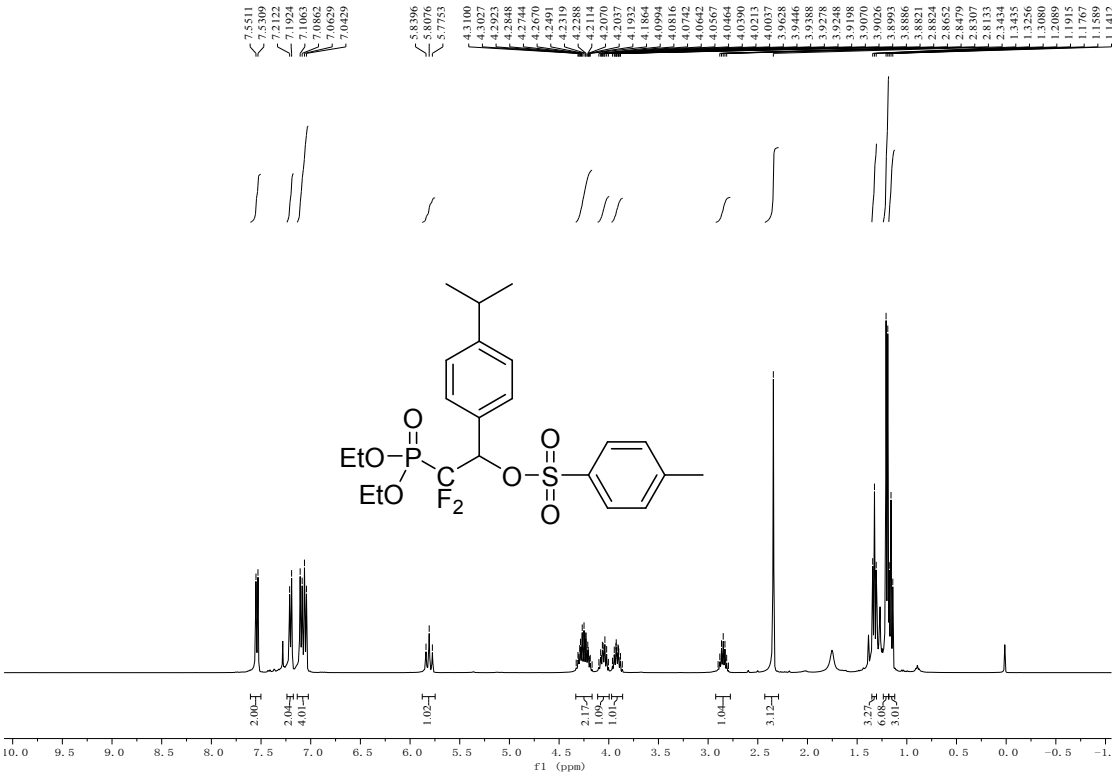
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4la**:



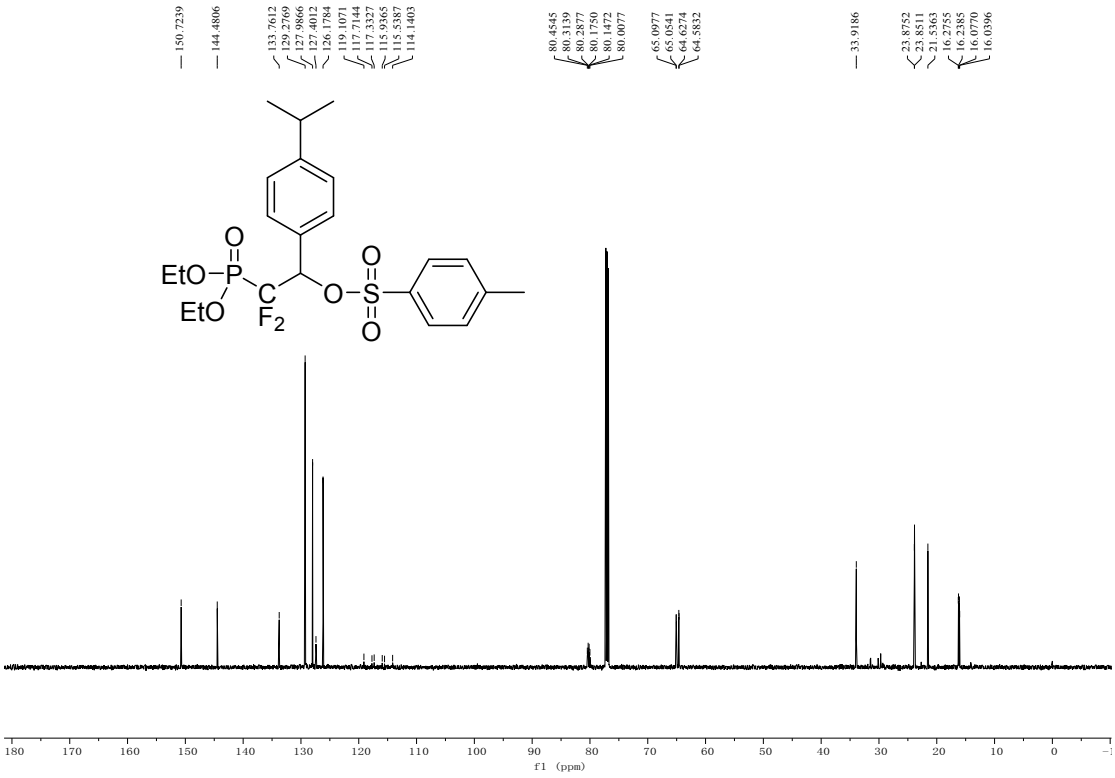

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4la**:



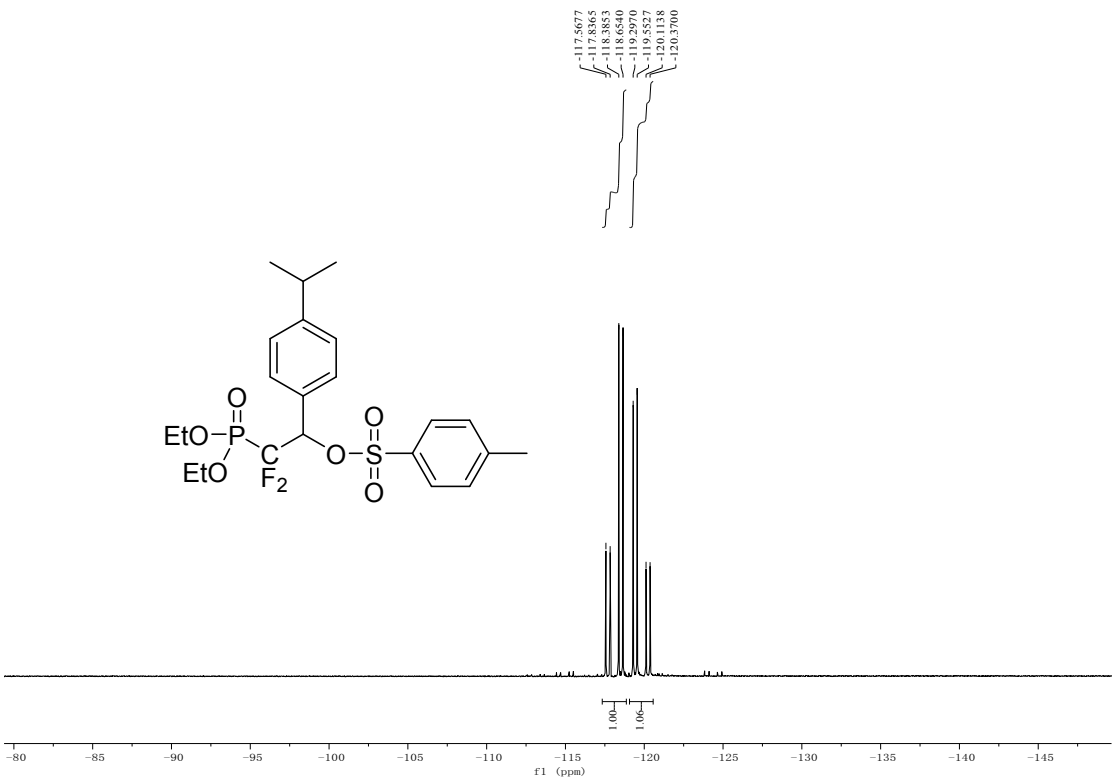
$^{1}$H NMR (400 MHz, $CDCl_3$) of **4ma**:



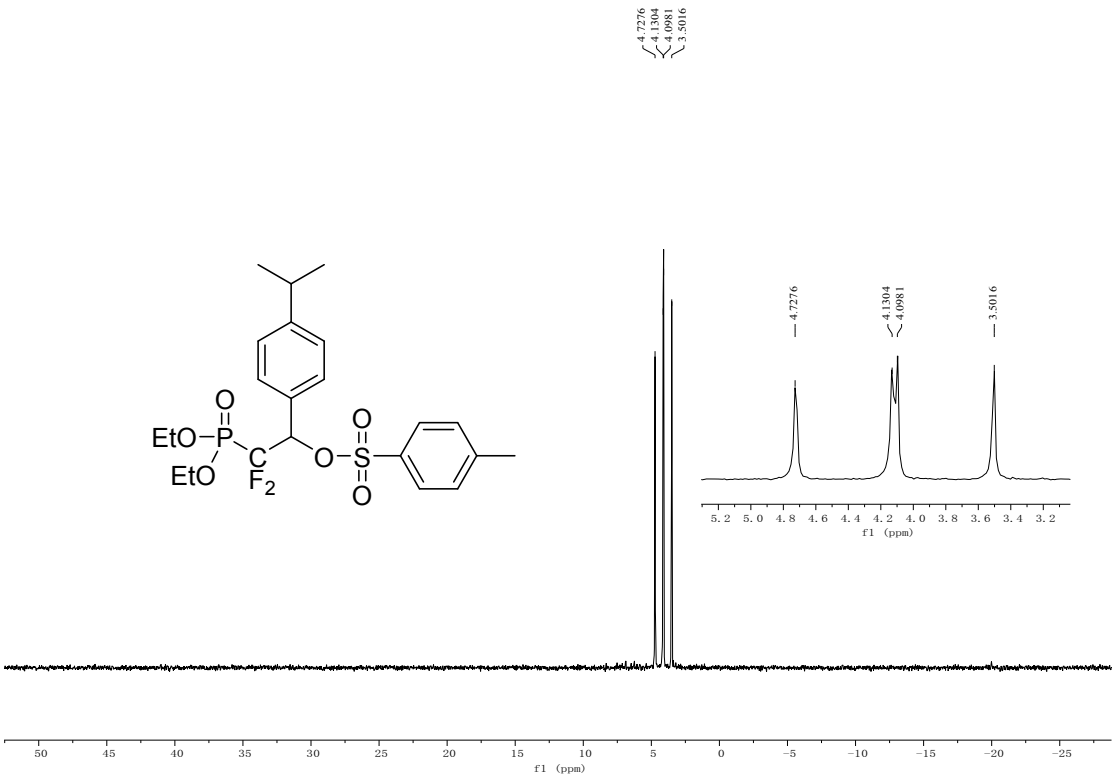

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ma**:



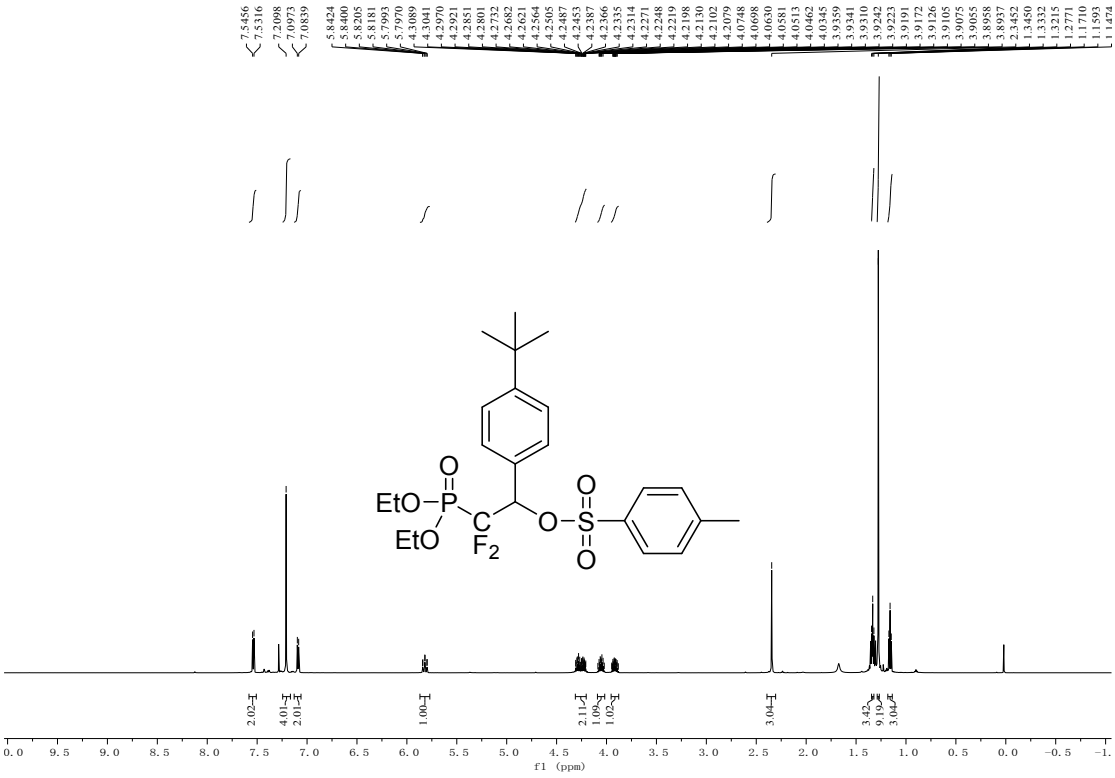
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ma**:



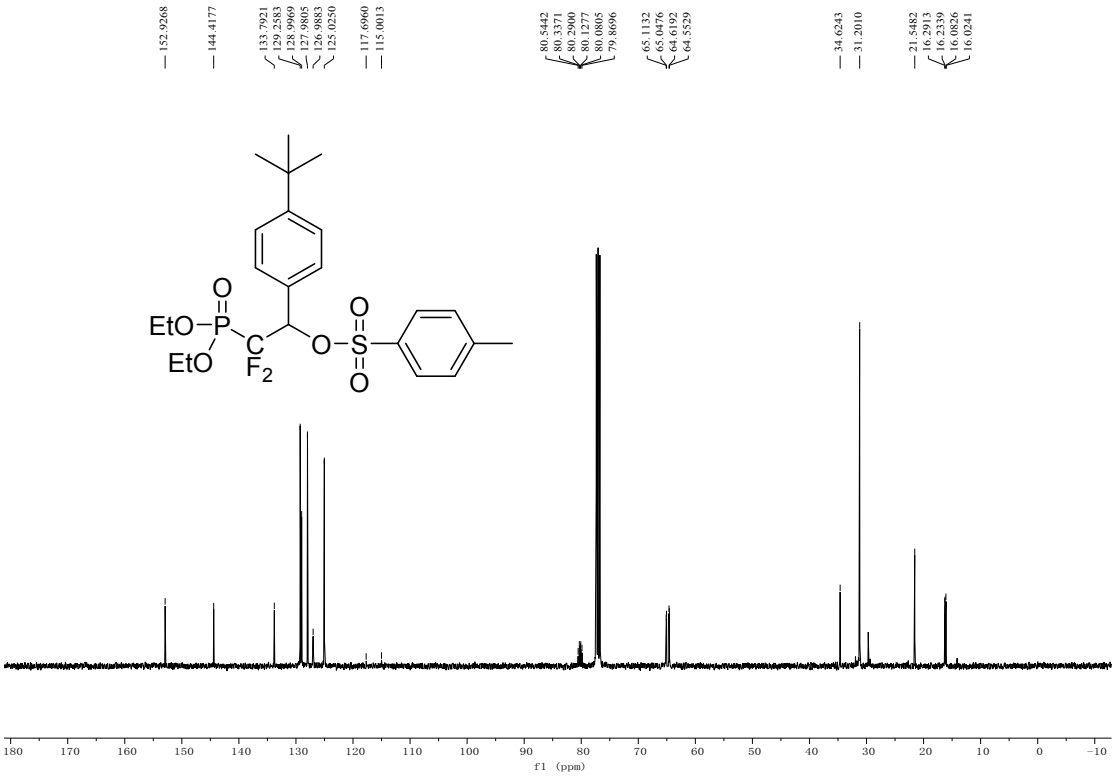

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ma**:



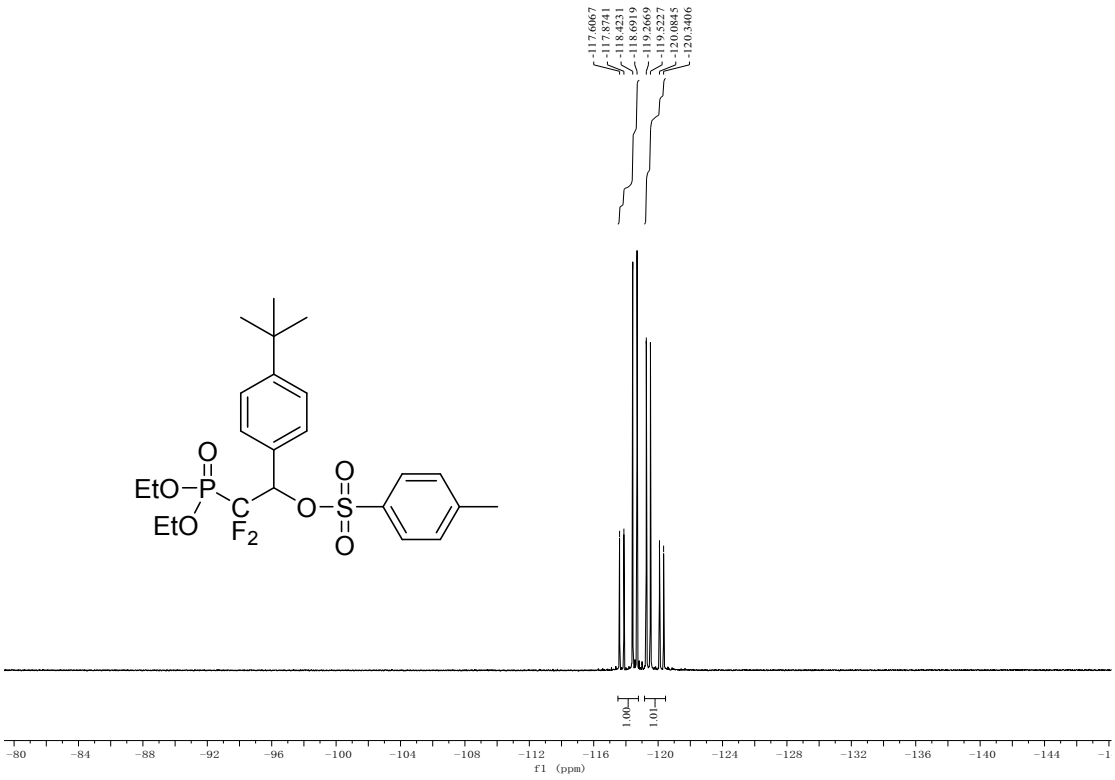
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4na**:



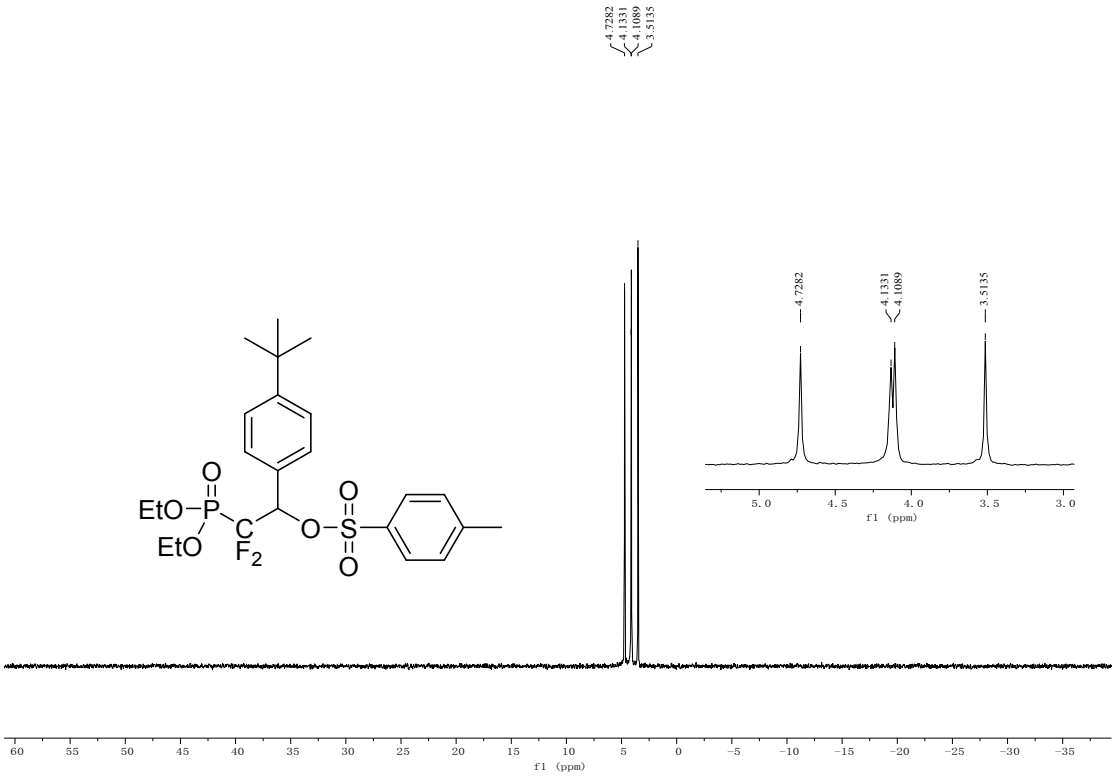

$^{13}$C NMR (100 MHz, $CDCl_3$) of **4na**:



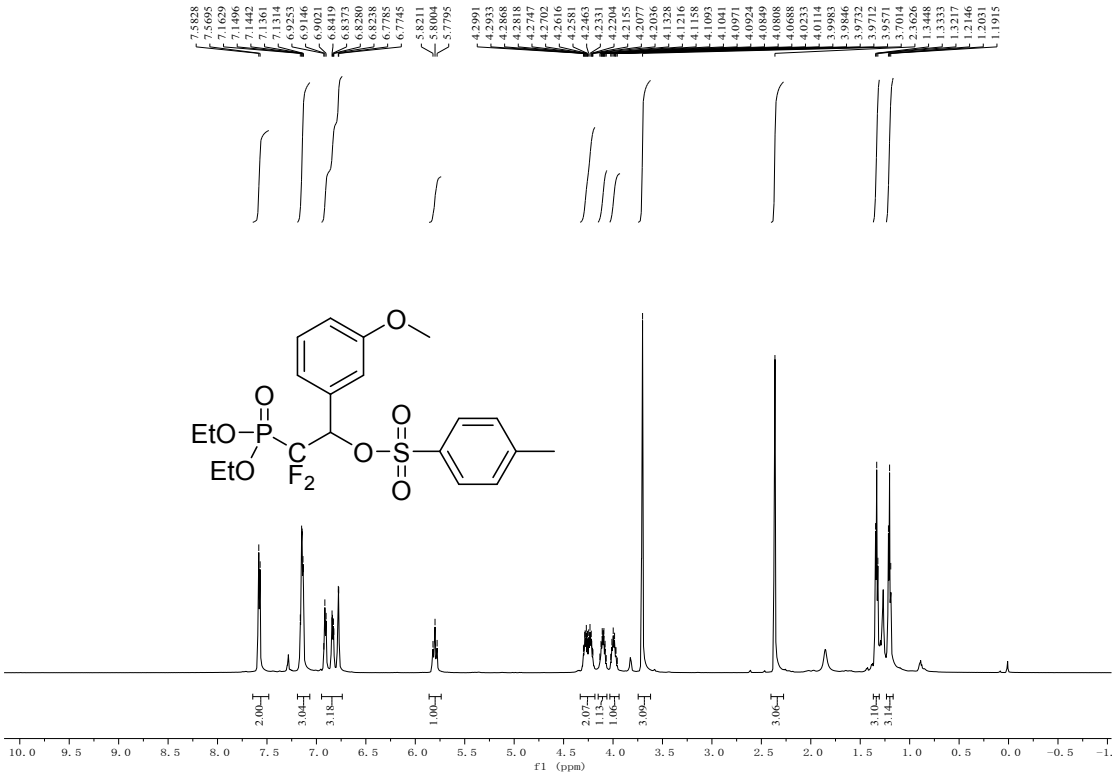
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4na**:



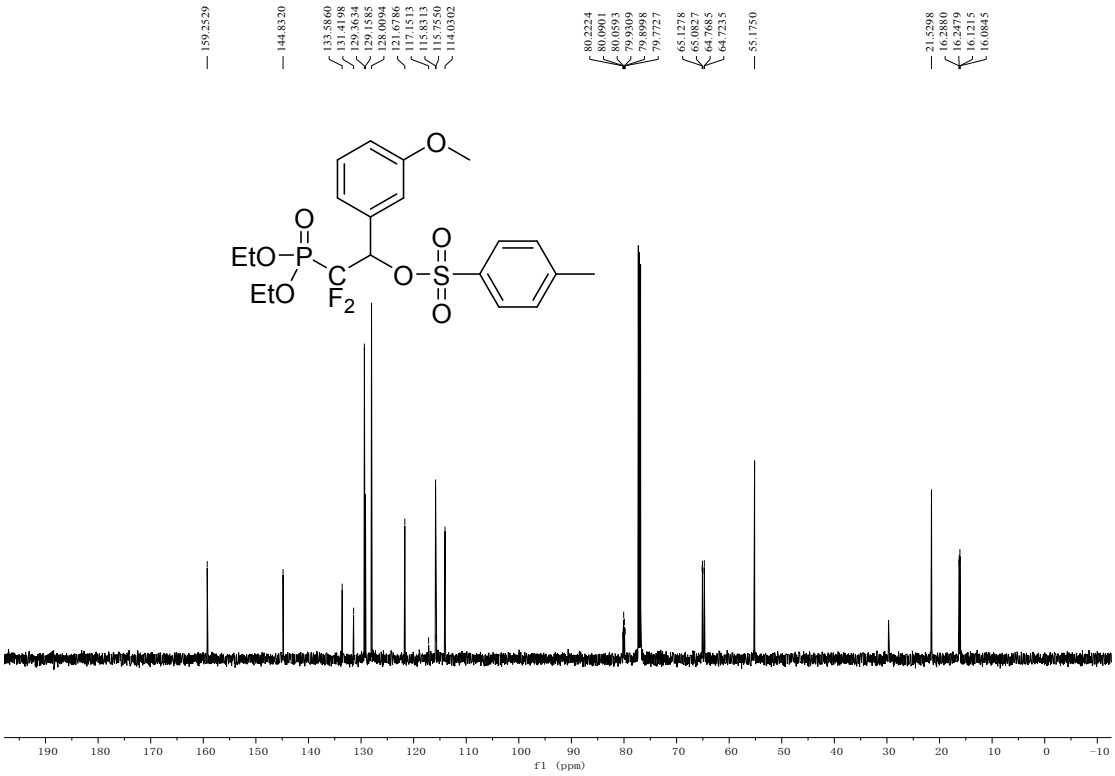

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4na**:



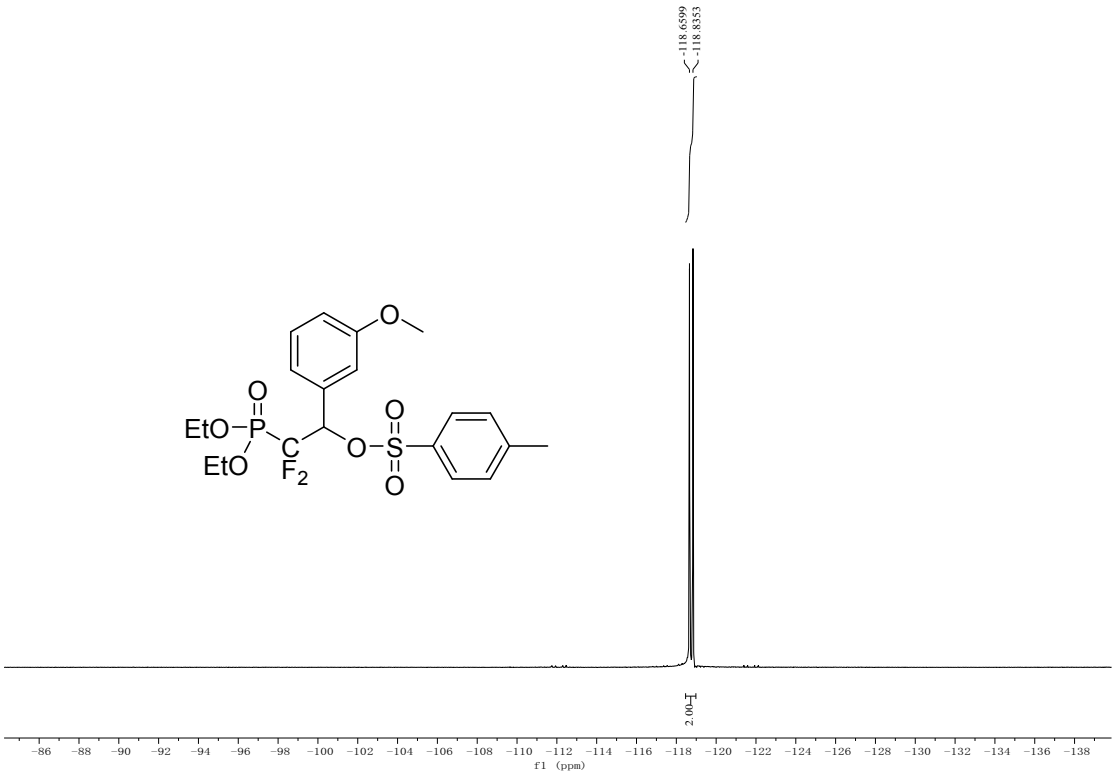
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4oa**:



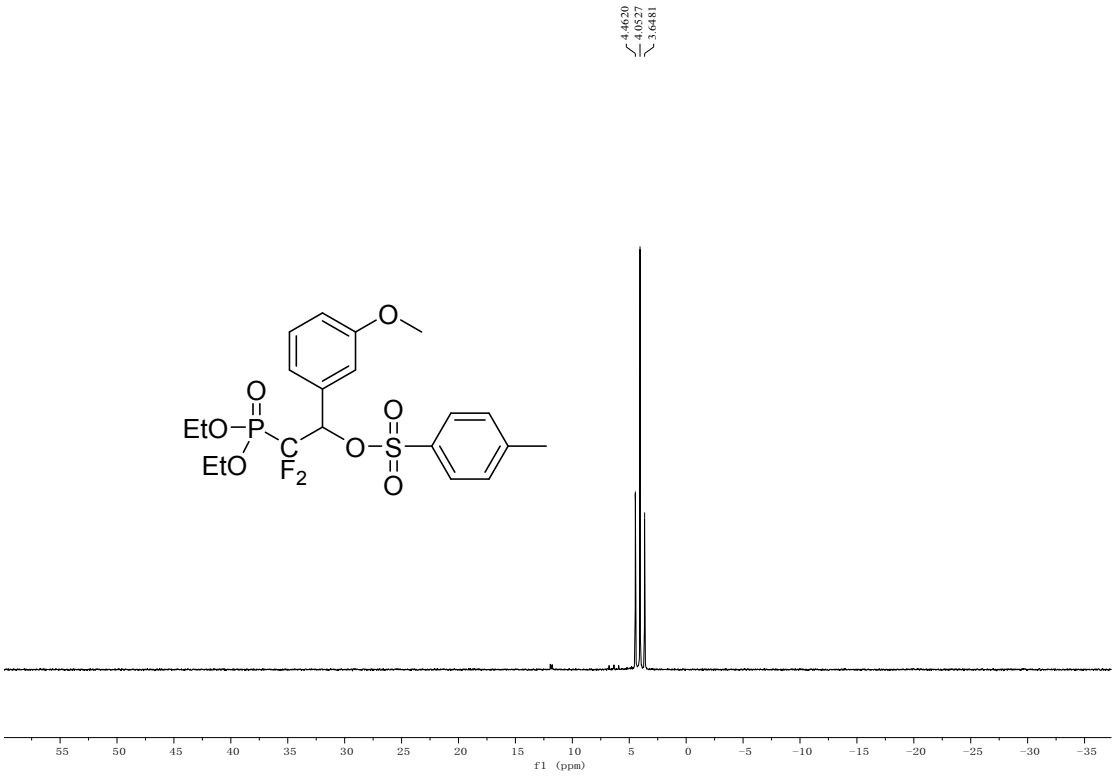

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4oa**:



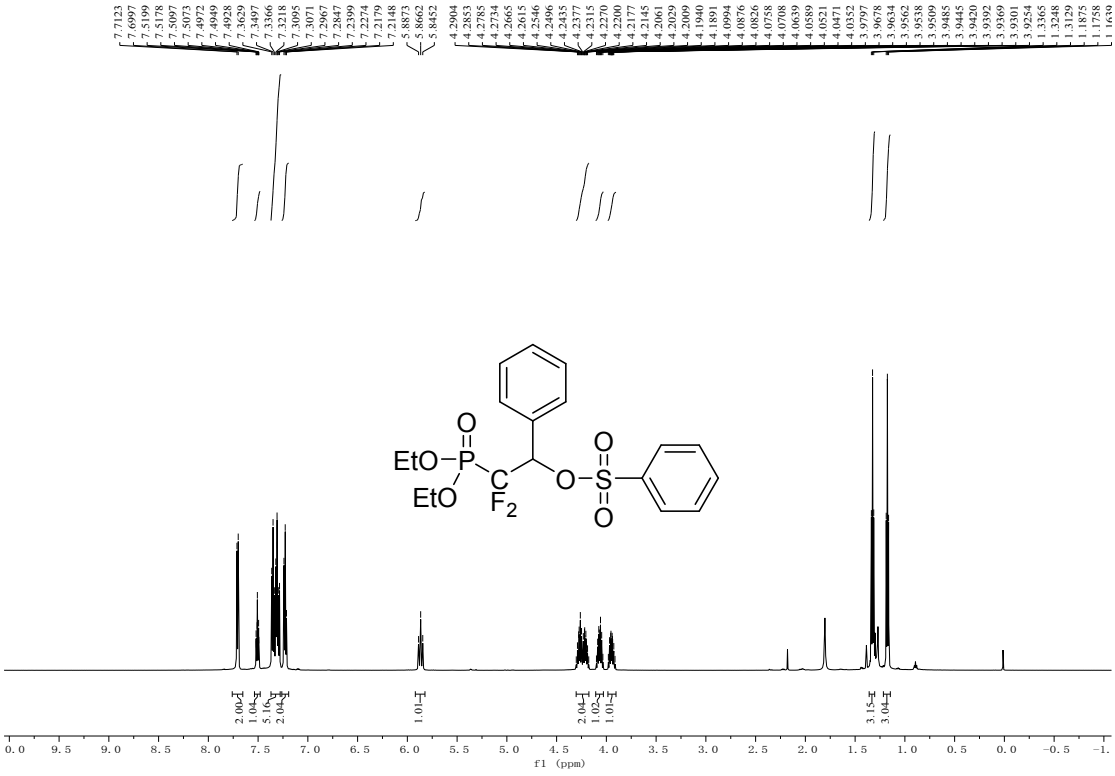
$^{19}F$ NMR (565 MHz, $CDCl_3$) of **4oa**:



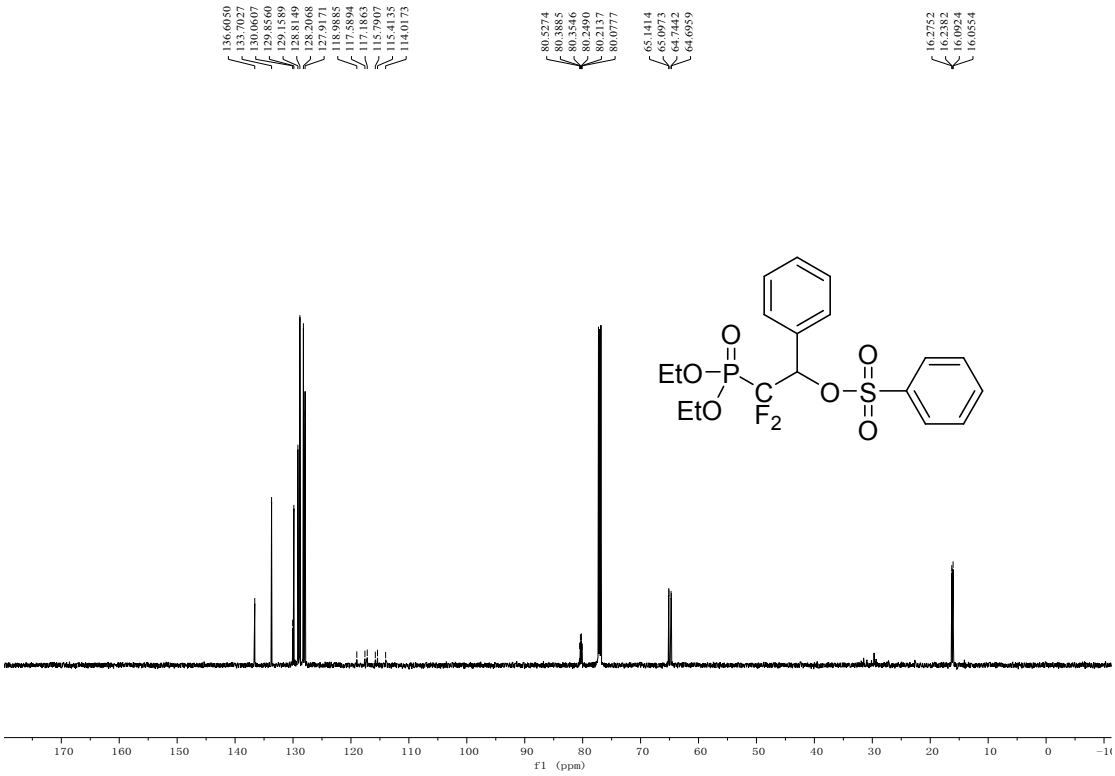

$^{31}P$ NMR (243 MHz, $CDCl_3$) of **4oa**:



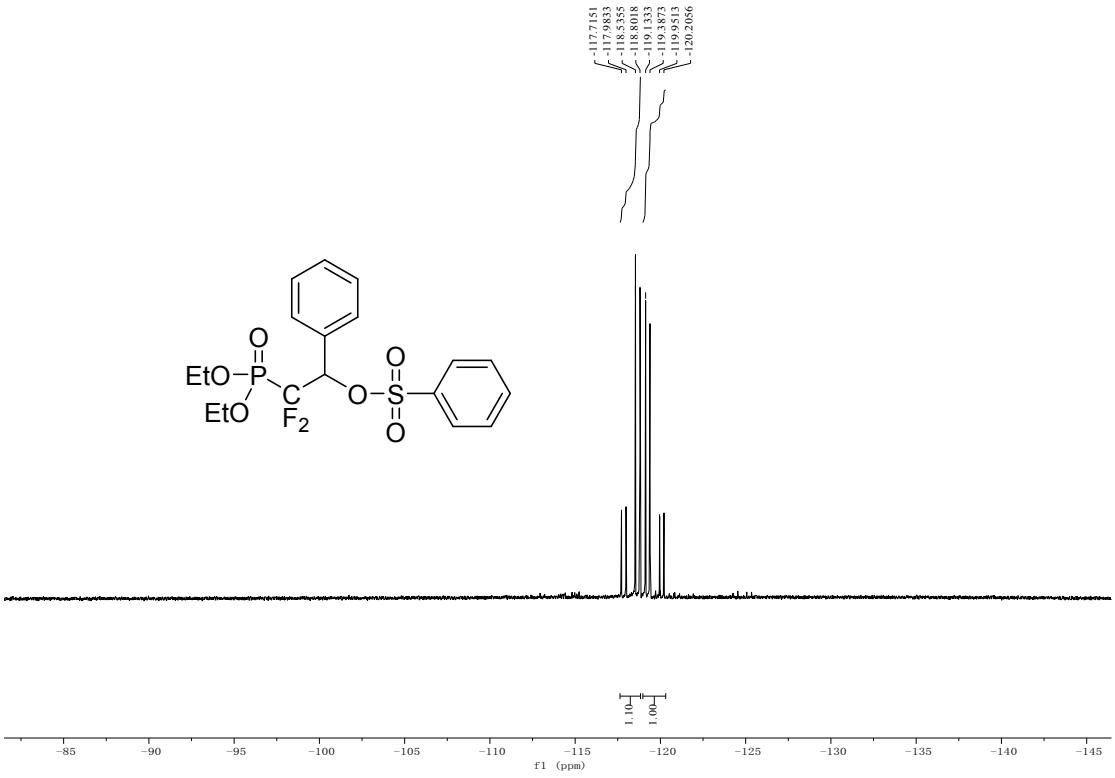
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4ab**:



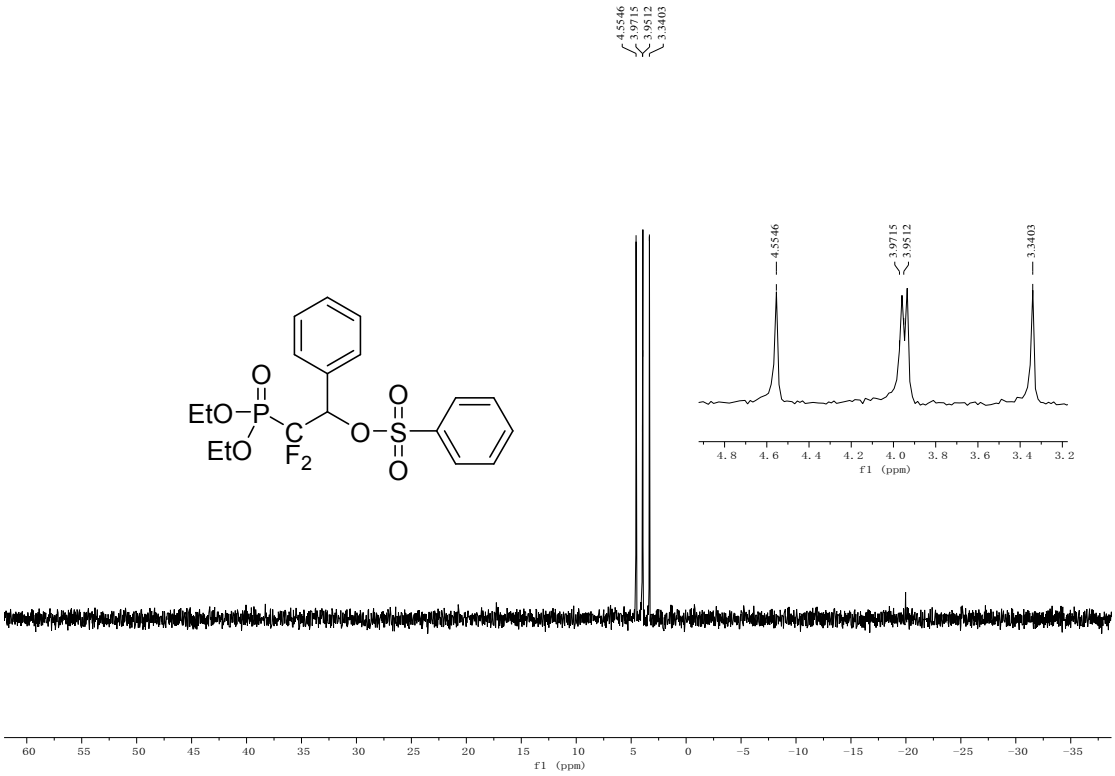

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ab**:



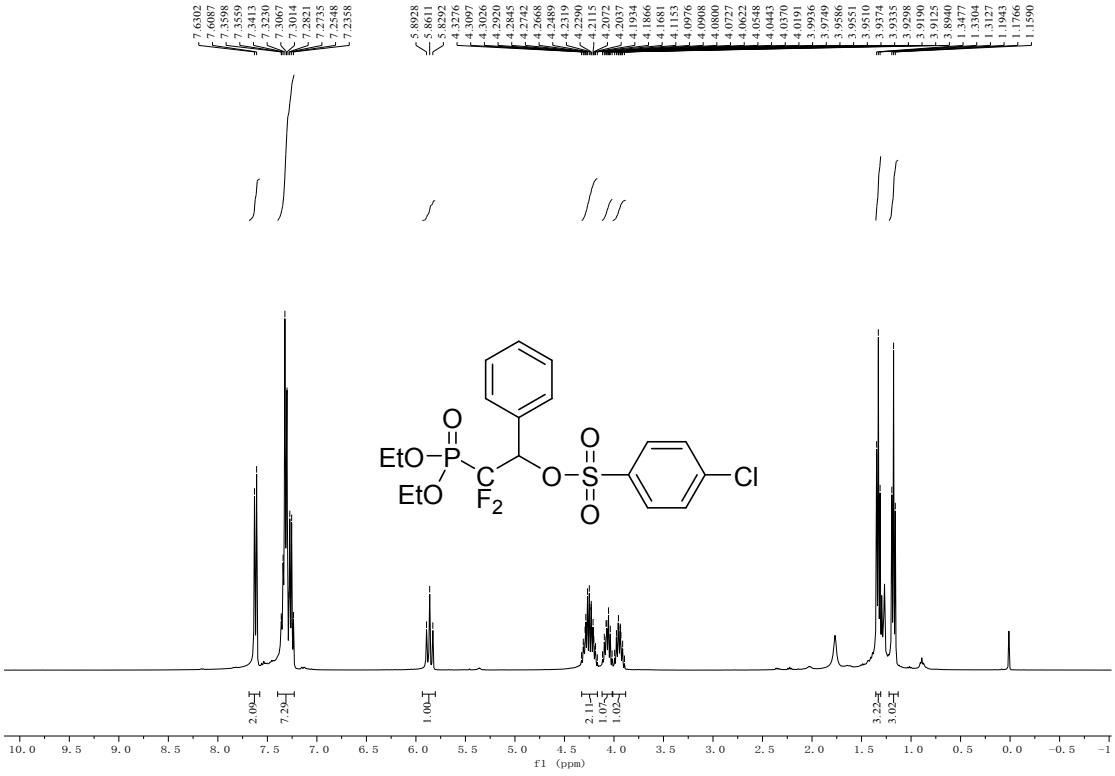
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ab**:



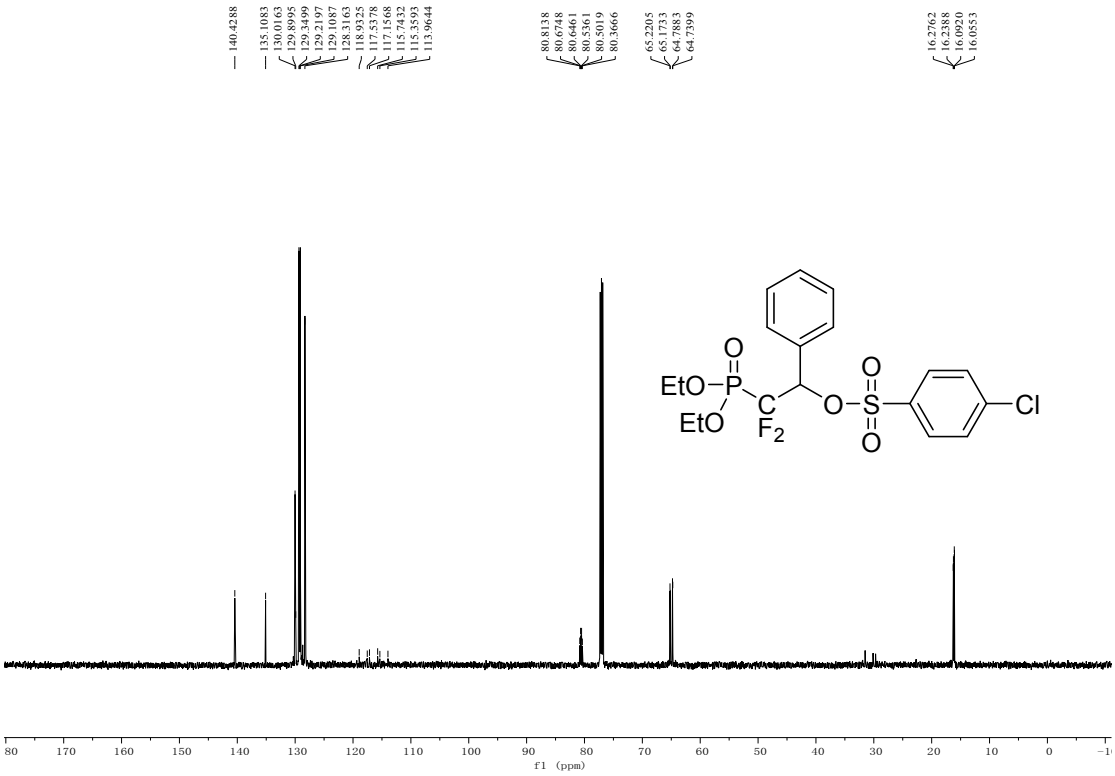

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ab**:



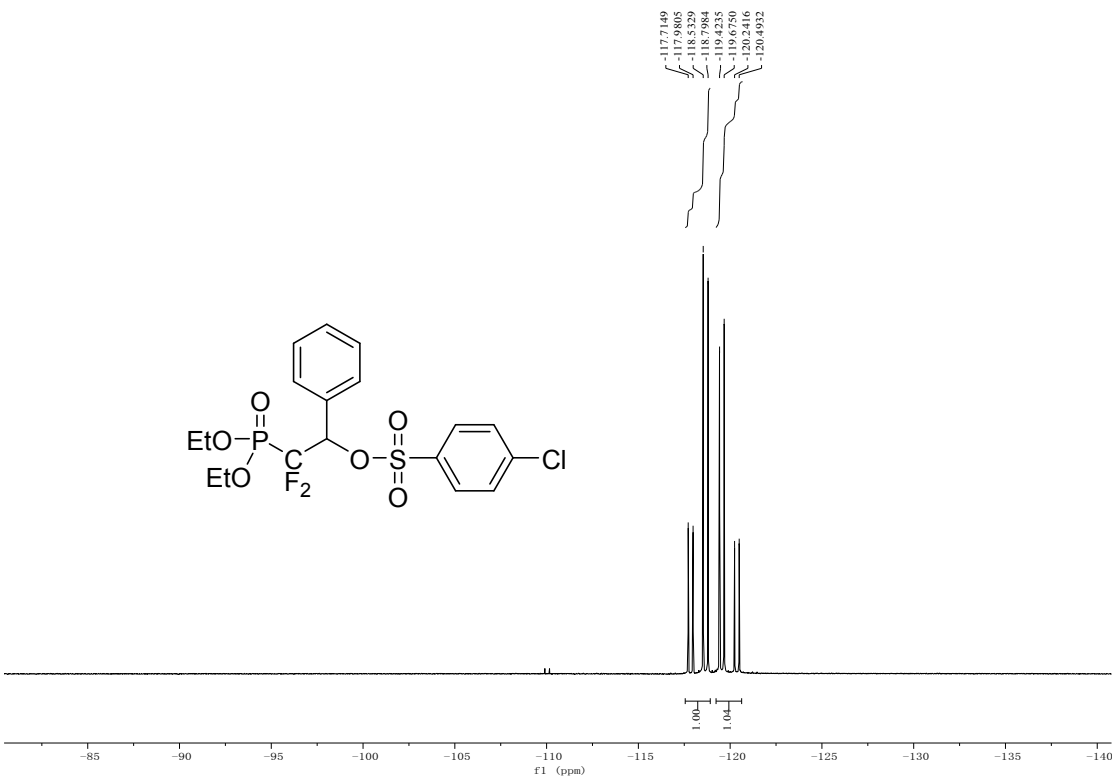
$^{1}$H NMR (400 MHz, $CDCl_3$) of **4ac**:



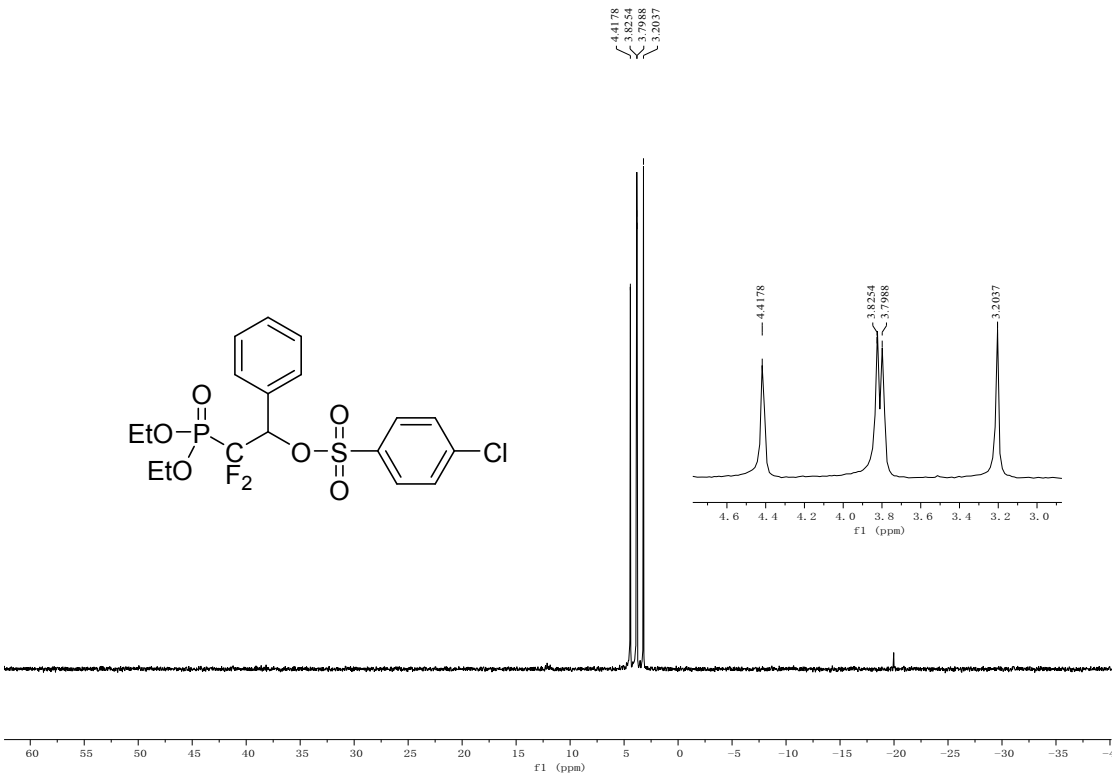

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ac**:



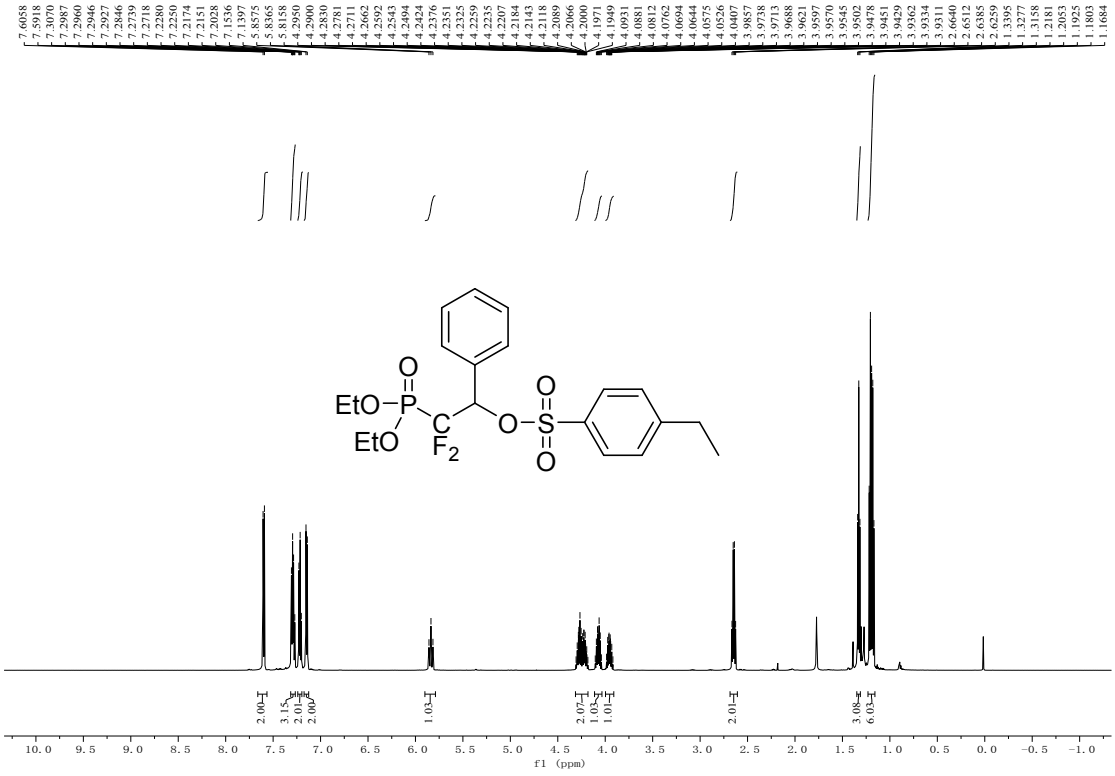
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ac**:



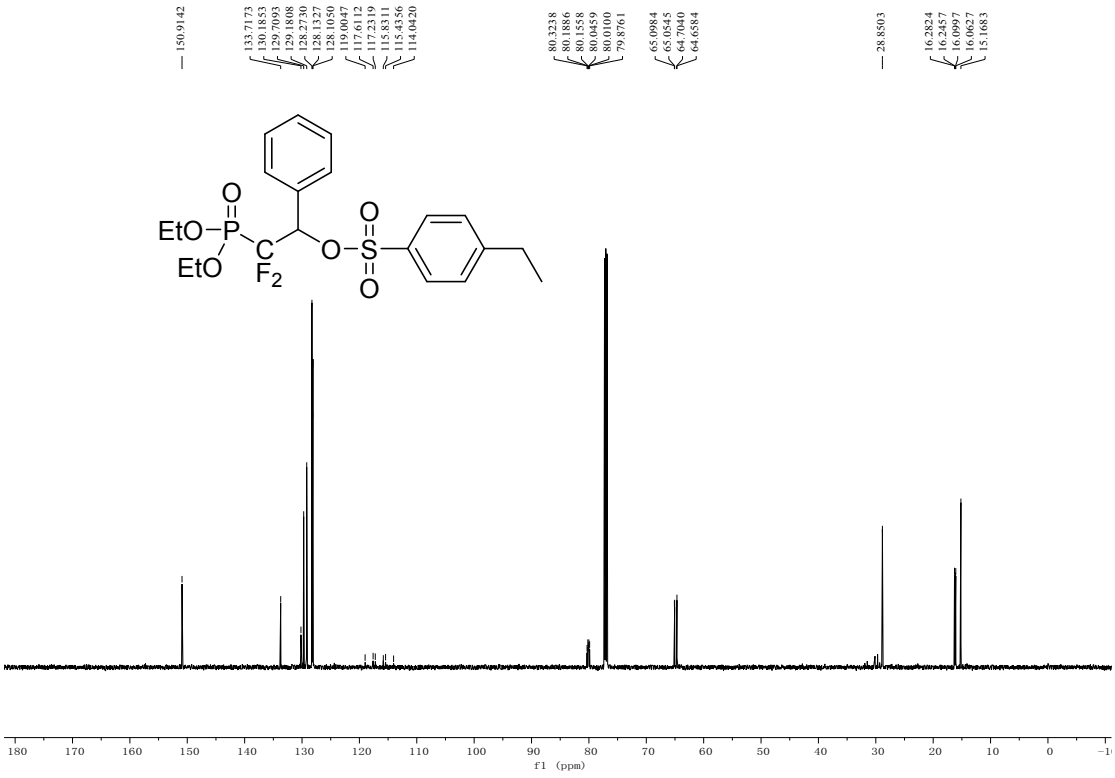

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ac**:



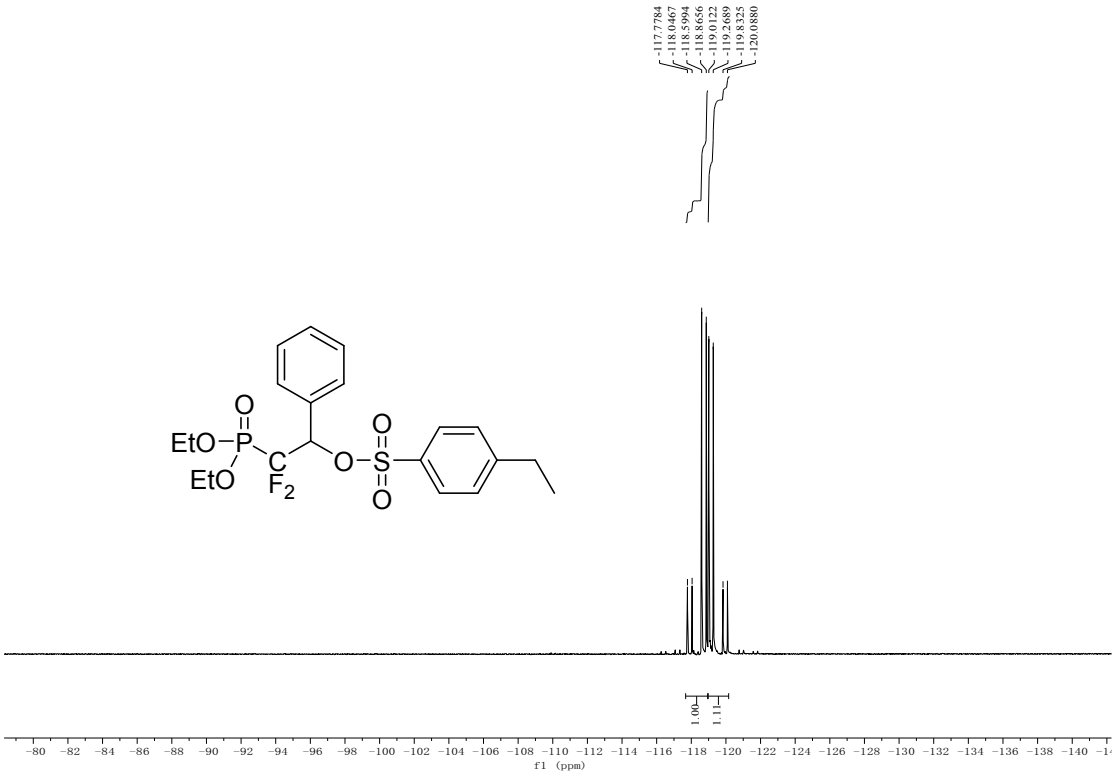
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4ad**:



$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ad**:

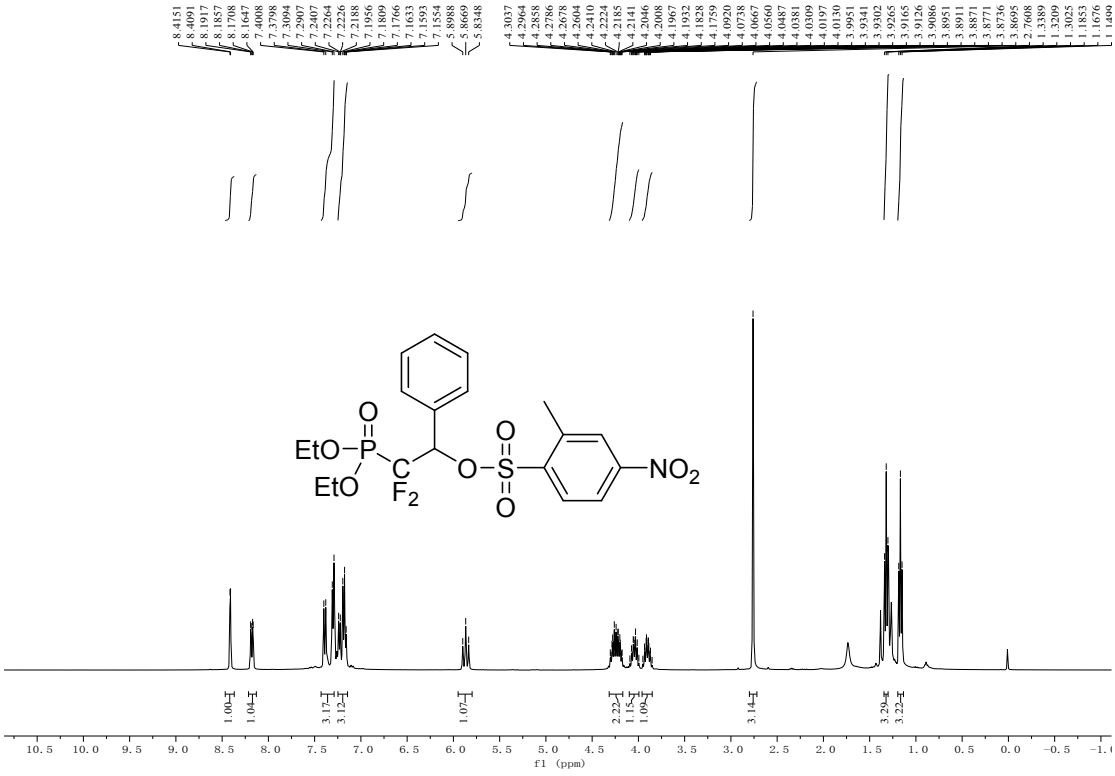


$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ad**:

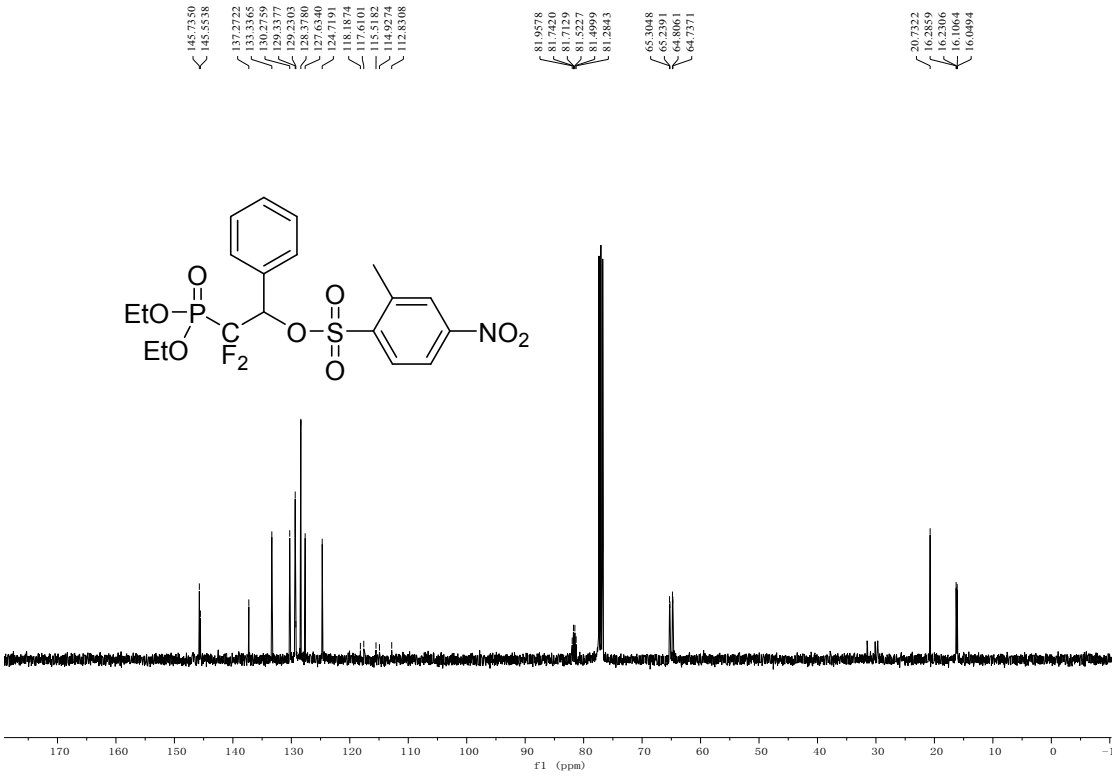



$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ad**:

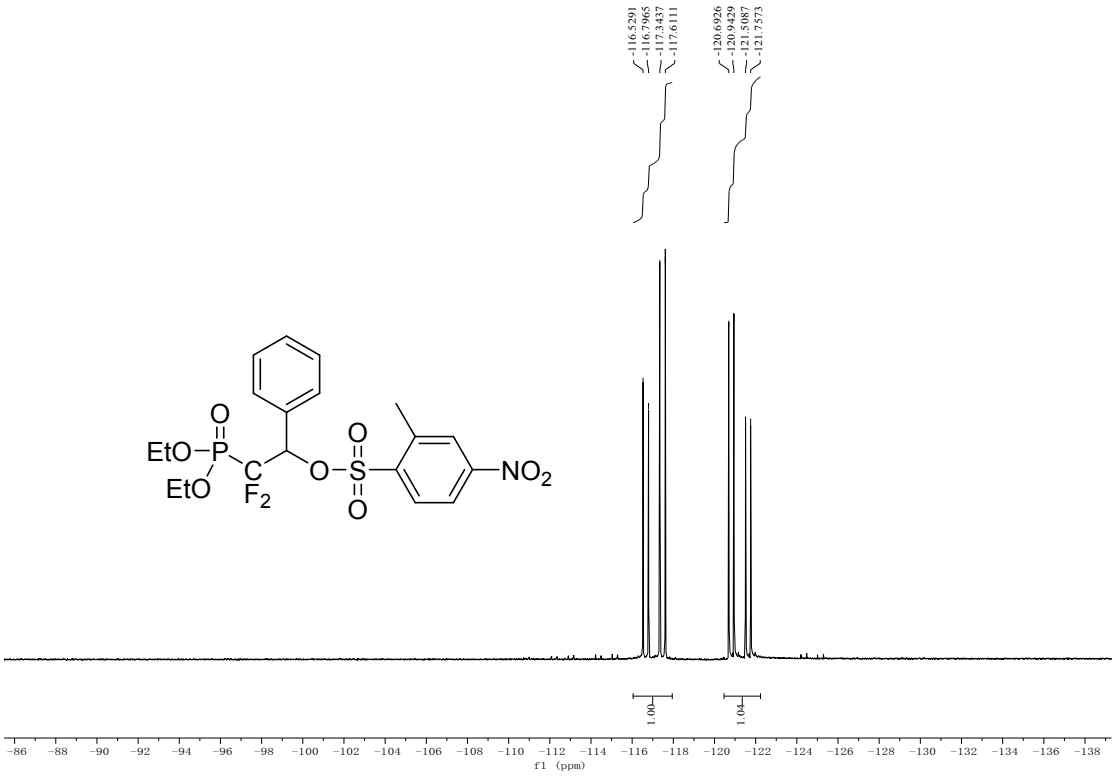
$^{1}$H NMR (400 MHz, $CDCl_3$) of **4ae**:



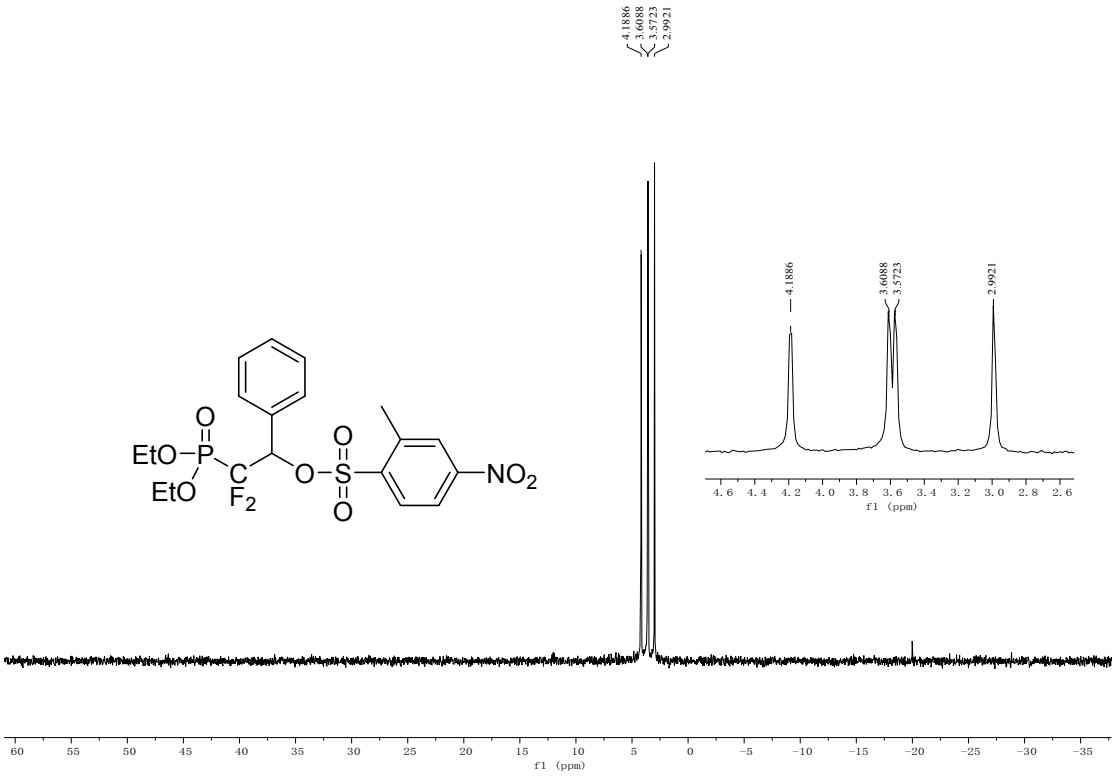

$^{13}$C NMR (100 MHz, $CDCl_3$) of **4ae**:



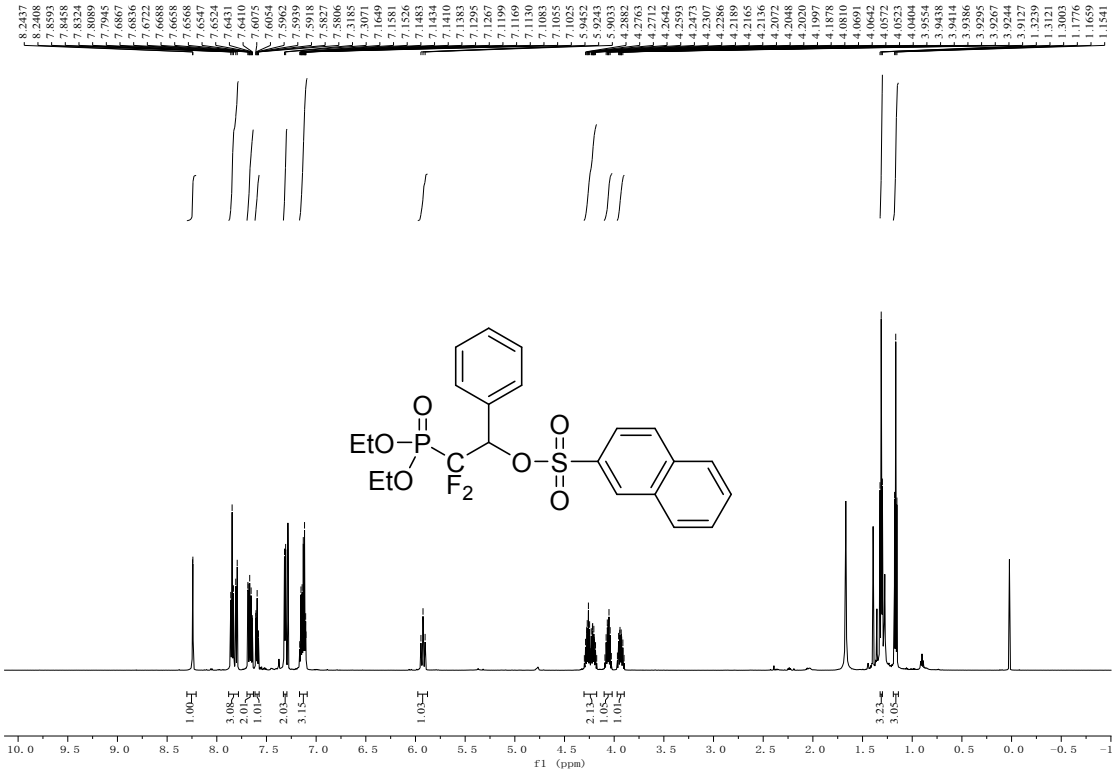
$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ae**:



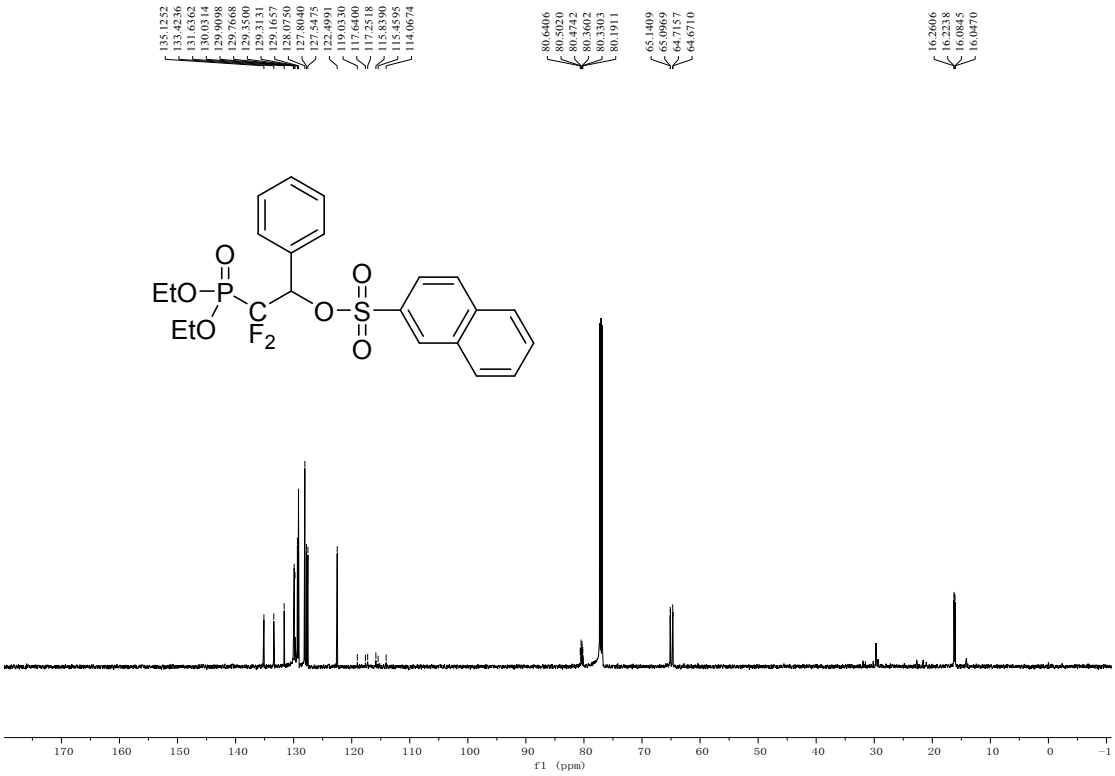

$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ae**:



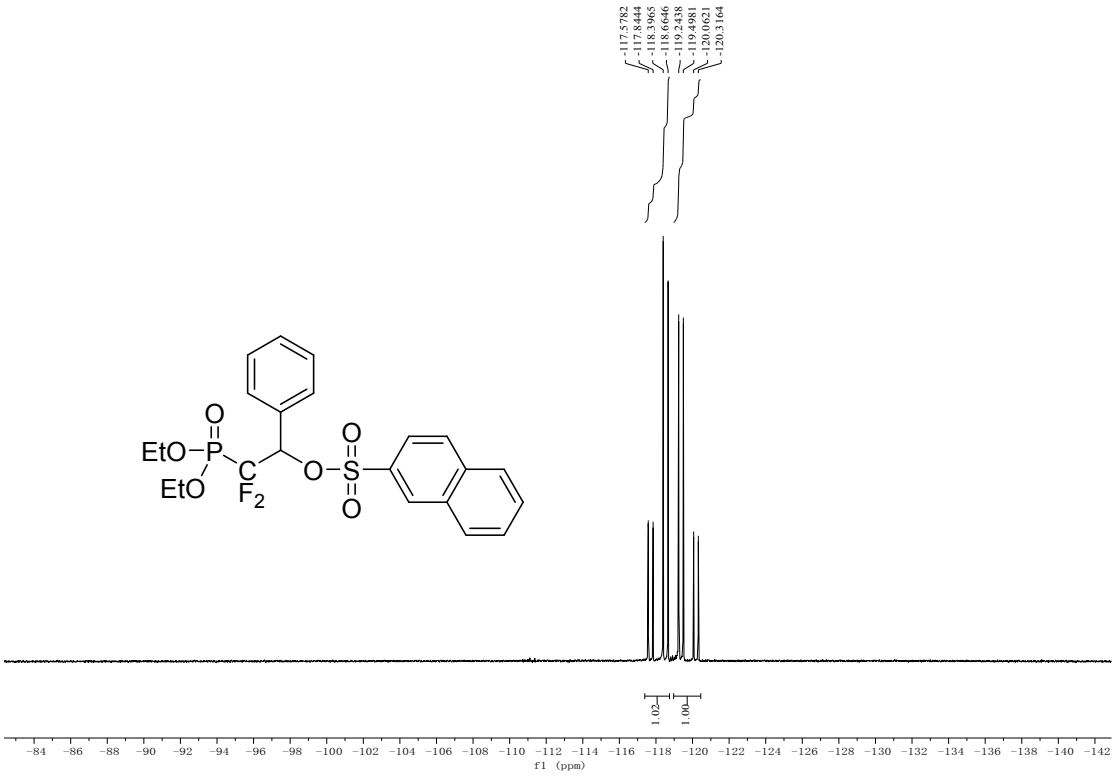
$^{1}$H NMR (600 MHz, $CDCl_3$) of **4af**:



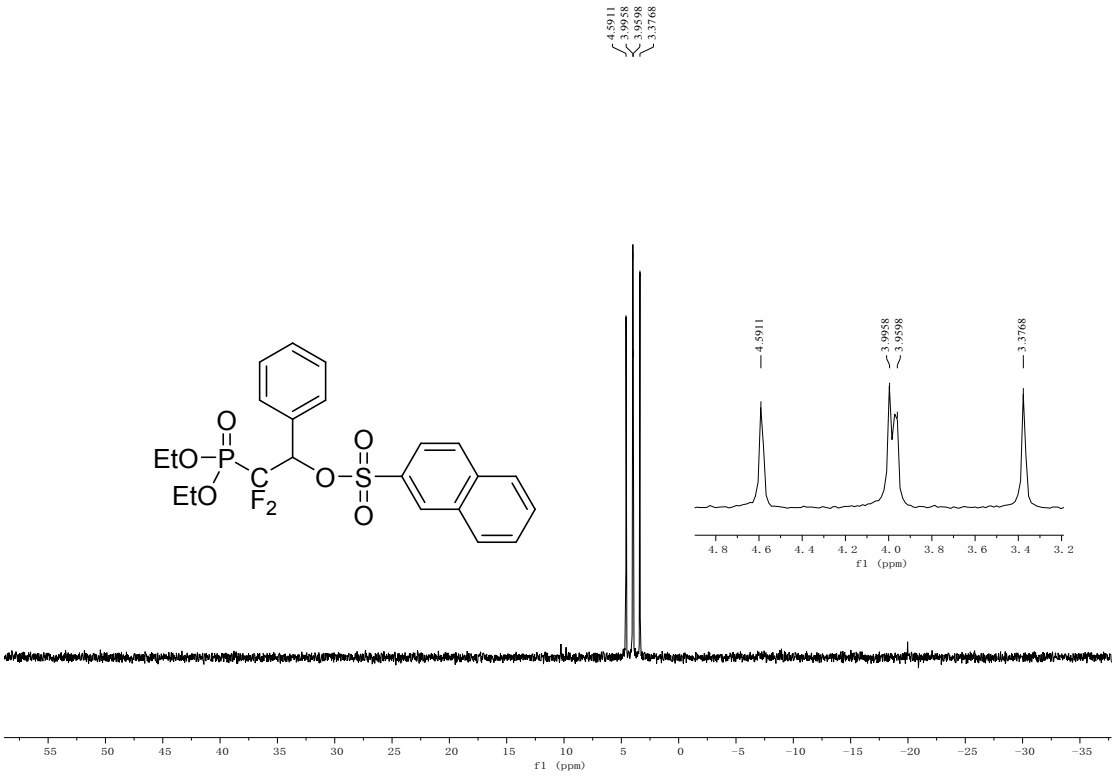

$^{13}$C NMR (150 MHz, $CDCl_3$) of **4af**:



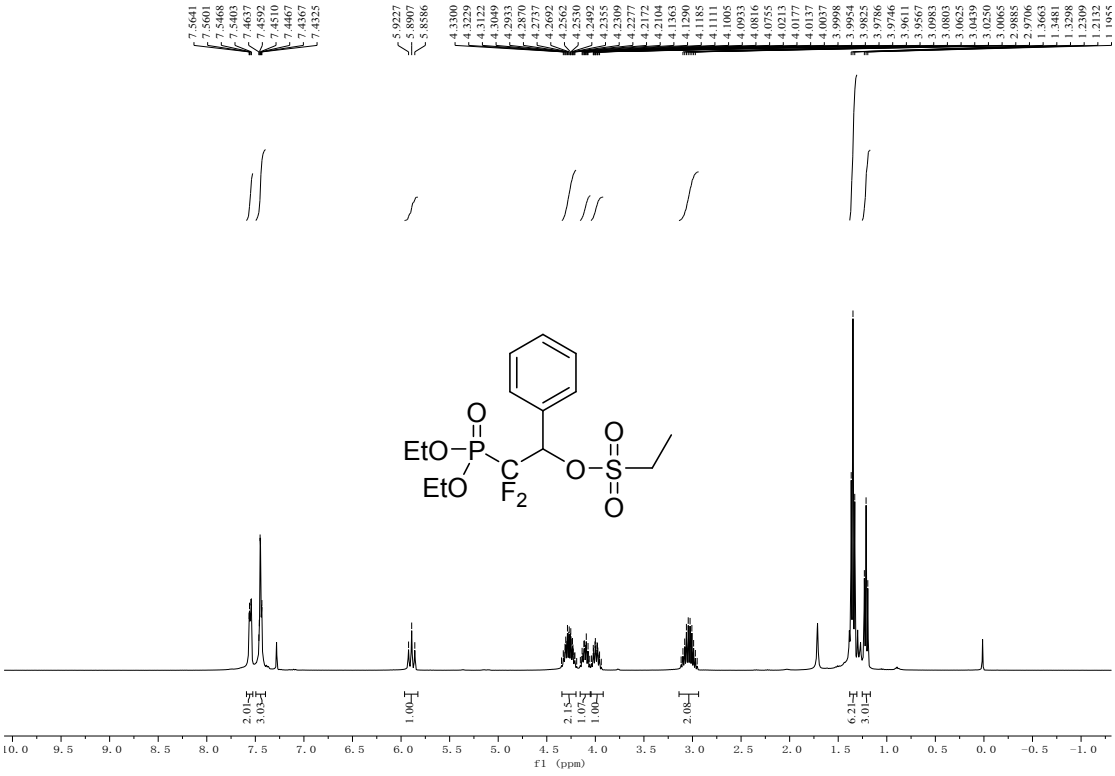
$^{19}$F NMR (376 MHz, $CDCl_3$) of **4af**:



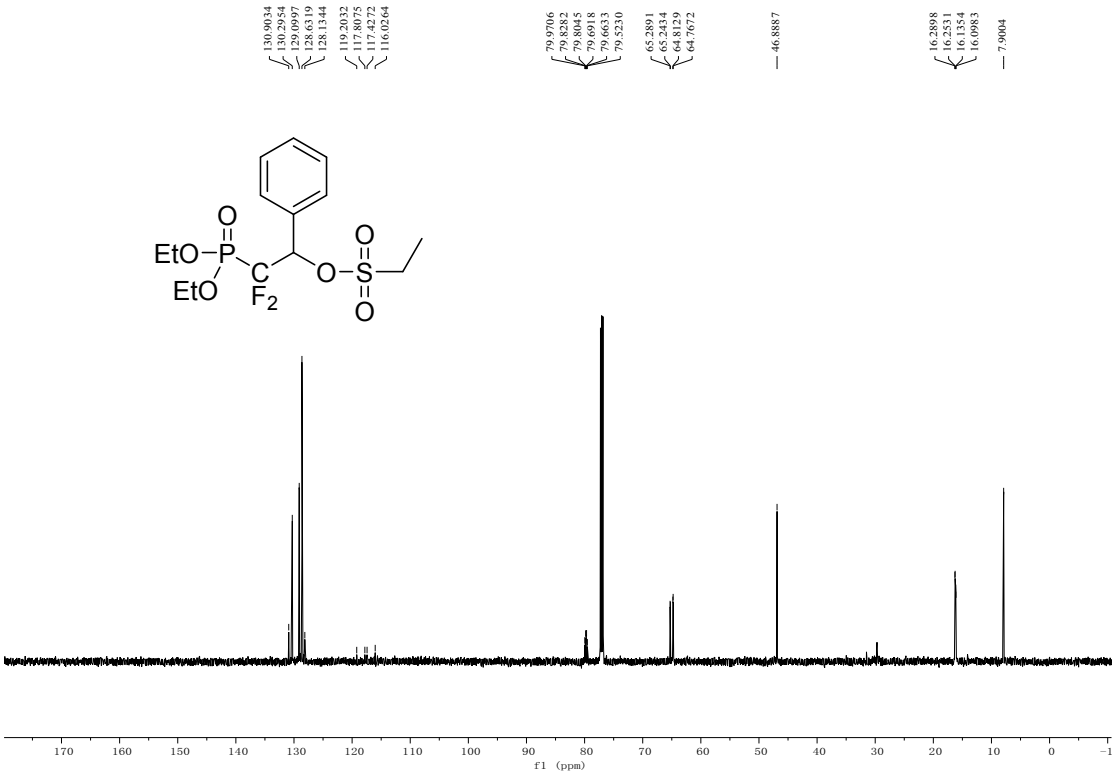

$^{31}$P NMR (162 MHz, $CDCl_3$) of **4af**:



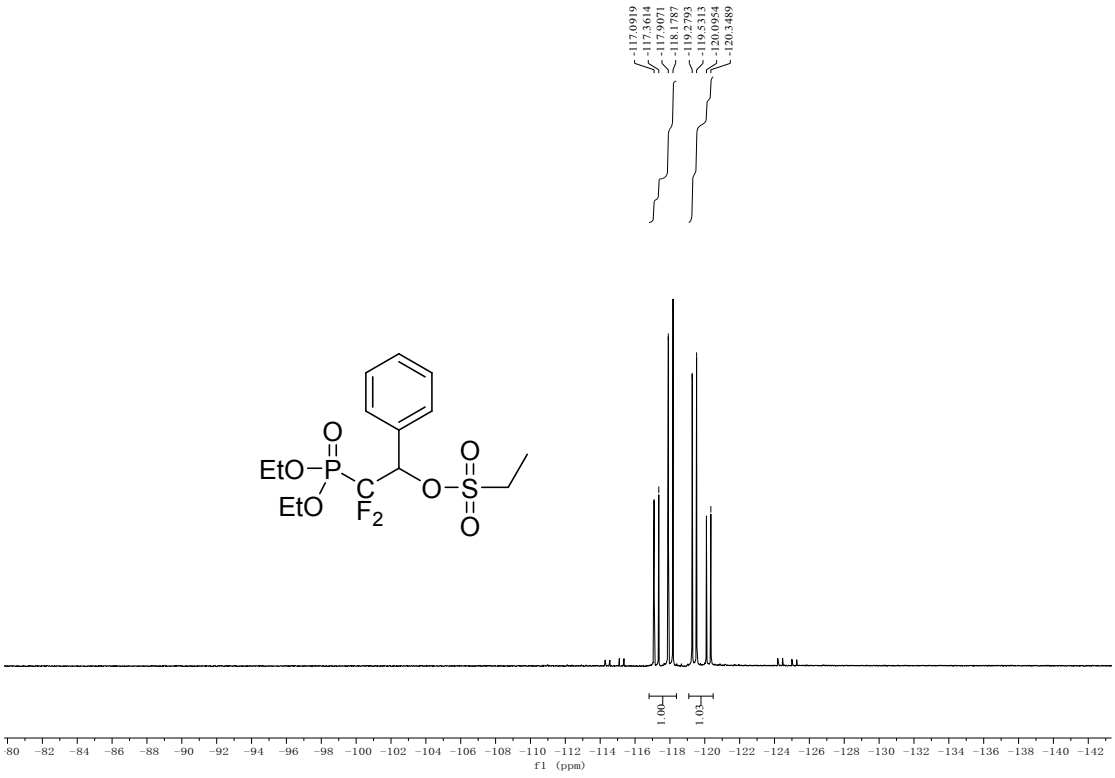
$^{1}$H NMR (400 MHz, $CDCl_3$) of **4ah**:



$^{13}$C NMR (150 MHz, $CDCl_3$) of **4ah**:



$^{19}F$ NMR (376 MHz, $CDCl_3$) of **4ah**:




$^{31}P$ NMR (162 MHz, $CDCl_3$) of **4ah**: