Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Access to Enantioenriched 4-Phosphorylated δ-Lactones from β-Phosphorylenones and Enals via Carbene Organocatalysis

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Contents

I: General Information:

All reactions were performed under argon atmosphere in oven-dried glasswares. The enals **1b** to **1o** were prepared according to literature known methods[1]. The *β*-phosphorylenones **2a** to **2aa** were prepared by using literature known procedure[2]. Solvents were dried and distilled following the standard procedures, TLC was carried out on pre-coated plates (Merck silica gel 60, f_{254}), and the spots were visualized with UV light or by charring the plates dipped in phosphomolybdic acid (PMA) charring solution. Flash chromatography was performed using silica gel (100-200 mess) with distilled solvents. Enantiomeric excess (*ee*) of the products were determined by high performance liquid chromatography (HPLC) analysis using a chiral stationary phase. ^{1}H, ^{13}C and ^{31}P NMR for compounds were recorded at 400 MHz, 100 MHz and 162 MHz instrument respectively using $CDCl_3$ as the solvent. 98% PPh_3 as an external standard for ^{31}P NMR. Chemical shifts were recorded in parts per million (ppm, δ) relative to tetramethylsilane (δ 0.00). ^{1}H NMR splitting patterns are designated as singlet (s), doublet (d), triplet (t), quartet (q), dd (doublet of doublets) and dt (doublet of triplets); m (multiplets), etc. High-resolution mass spectral analysis (HRMS) was performed on Q-TOF Premier mass spectrometer. Optical rotation of compounds measured on an Autopol III, serial number 30700 polarimeter at wavelength 589 nm.

II: General procedure for the NHC-catalyzed synthesis of products 3

To an oven dried Schlenk tube equipped with a magnetic stir bar was added aldehyde **1** (0.2 mmol, 2.0 equiv.), *β*-phosphoryl enones **2** (0.1 mmol, 1.0 equiv.) and catalyst **C** (8.4 mg, 0.02 mmol). The Schlenk tube evacuated and backfilled with argon, after the addition of $CHCl_3$ (1.0 mL) and $CsCO_3$ (13.0 mg, 0.04 mmol) and the reaction chamber was closed. After stirring at rt for 16 h, the reaction was monitored by TLC, solvent was removed under reduced pressure and reaction mixture purified by silica gel column chromatography with EtOAc/Hexane (8:2) to obtain the desired product **3**.

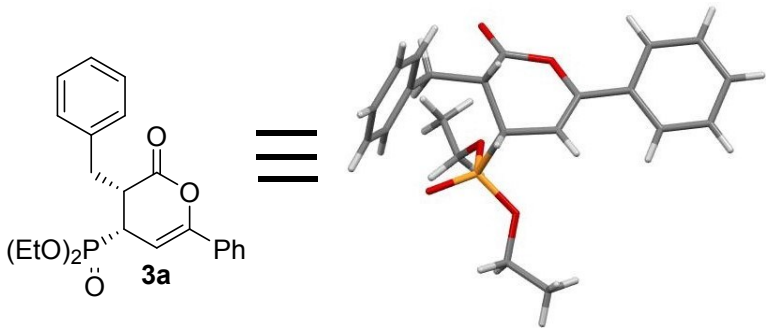
IV: Preparation of compounds 4 & 5

Compound 4: To a dry Schlenk tube equipped with magnetic stir bar lactone **3a** (40.0 mg, 0.1 mmol) was and MeOH 1.0 mL was added and reaction mixture cooled to 0 ^{o}C and DBU (3.0 µL, 0.02) was added. After stirring the reaction mixture at 0 ^{o}C for 2 h, the reaction was monitored by TLC and solvent was removed under reduced pressure through rotavapor, followed by column chromatography on silica-gel with EtOAc produced the desired product **4** in **(**43 mg) 99% yield.

Compound 5: To a dry Schlenk tube equipped with magnetic stir bar lactone **3a** (40.0 mg, 0.1 mmol), toluene 1.0 mL and benzyl amine (28.0 µL, 0.30 mmol) was added and the reaction stirred at rt for 16 h. The reaction was monitored by TLC and solvent was evaporated under reduced pressure through rotavapor, followed by column chromatography on silica-gel with EtOAc afforded the desired product **5** in (50 mg) 98% yield.

VII. X-ray crystallography of compound 3a.

The absolute stereochemistry of compound **3a** was determined by SC-XRD. Good quality crystal of **3a** (colorless crystal) was obtained by slow evaporation of a solution (few drops EtOAc in CH_2Cl_2) of compound **3a**. CCDC 1936122 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via https://www.ccdc.cam.ac.uk/.



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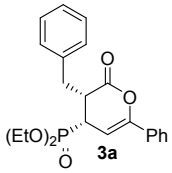
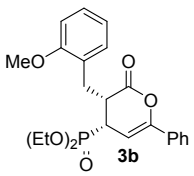
V: Characterization of the products:

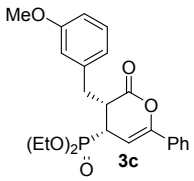
Diethyl ((3*R*,4*S*)-3-benzyl-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3a): 95% yield (38 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{28}$ = +121.60 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.20-1.30 (6H, m), 2.84-3.18 (3H, m), 3.36-3.52 (1H, m), 3.97-4.16 (4H, m), 5.72 (1H, dd, J = 6.8, 4.8 Hz), 7.11-7.19 (1H, m), 7.20-7.35 (7H, m), 7.47-7.60 (2H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, J_{C-P} = 6.0 Hz), 32.5 (d, J_{C-P} = 1.0 Hz), 33.8 (d, J_{C-P} = 140.0 Hz), 42.8 (d, J_{C-P} = 4.0 Hz), 62.7 (q, J_{C-P} = 7.0 Hz), 98.3 (d, J_{C-P} = 11.0 Hz), 124.6 (d, J_{C-P} = 1.0 Hz), 126.5, 128.4, 129.0, 129.3, 131.8 (d, J_{C-P} = 4.0 Hz), 138.9, 151.6 (d, J_{C-P} = 11.0 Hz), 168.6 (d, J_{C-P} = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.1; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{25}O_5PNa^+$ $[M+Na]^+$: 423.1332, found: 423.1327; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, UV 254 nm), Rt_1 (minor) = 34.9 min, Rt_2 (major) = 38.4 min; >99% ee.



Diethyl ((3*R*,4*S*)-3-(2-methoxybenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3b): 96% yield (42 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{25}$ = +84.80 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.28-1.40 (6H, m), 2.96-3.18 (2H, m), 3.21-3.42 (1H, m), 3.51 (1H, dd, J = 14.0, 5.6 Hz), 3.79 (3H, s), 4.09-4.24 (4H, m), 5.82 (1H, t, J = 5.6 Hz), 6.84 (1H, d, J = 8.4 Hz), 6.91 (1H, t, J = 7.6 Hz), 7.21 (1H, t, J = 8.0 Hz), 7.36 (3H, t, J = 5.6 Hz), 7.51 (1H, d, J = 7.6 Hz), 7.62 (2H, t, J = 5.6 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, J_{C-P} = 5.0 Hz), 28.3, 34.4 (d, J_{C-P} = 140.0 Hz), 40.3 (d, J_{C-P} = 5.0 Hz), 55.1, 62.6 (q, J_{C-P} = 8.0 Hz), 98.4 (d, J_{C-P} = 11.0 Hz), 110.0, 120.3, 124.6, 124.7, 127.0, 128.4, 129.2, 131.8, 131.9 (d, J_{C-P} = 4.0 Hz), 151.5 (d, J_{C-P} = 11.0 Hz), 157.4, 168.9 (d, J_{C-P} = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.6; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{27}O_6PNa^+$ $[M+Na]^+$: 453.1438, found: 453.1459; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, UV 254 nm), Rt_1 (minor) = 26.5 min, Rt_2 (major) = 33.4 min; >99% ee.

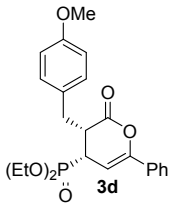


Diethyl ((3*R*,4*S*)-3-(3-methoxybenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3c): 95% yield (41 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{25}$ =



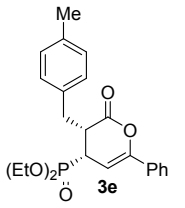
+67.40 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.20-1.30 (6H, m), 2.87-3.17 (3H, m), 3.42 (1H, dd, J = 14.0, 3.6 Hz), 3.71 (3H, s), 4.0-4.14 (4H, m), 5.73 (1H, dd, J = 6.8, 4.4 Hz), 6.66-6.73 (1H, m), 6.82-6.91 (2H, m), 7.13 (1H, t, J = 8.0 Hz), 7.25-7.33 (3H, m), 7.49-7.57 (2H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 6.0 Hz), 32.4, 33.7 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 55.0, 62.7 (q, $J_{C\text{-}P}$ = 8.0 Hz), 98.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 111.9, 114.7, 121.2, 124.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.4, 129.3, 129.4, 131.8 (d, $J_{C\text{-}P}$ = 5.0 Hz), 140.4, 151.5 (d, $J_{C\text{-}P}$ = 11.0 Hz), 159.6, 168.8 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.1; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{28}O_6P^+$ $[M+H]^+$: 431.1619, found: 431.1608; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, UV 254 nm), Rt_1 (minor) = 24.5 min, Rt_2 (major) = 26.8 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-(4-methoxybenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3d):



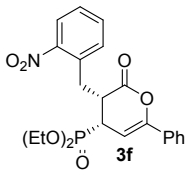
90% yield (39 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{25}$ = +72.60 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.28-1.39 (6H, m), 2.91-3.22 (3H, m), 3.46 (1H, d, J = 10.0 Hz), 3.79 (3H, s), 4.06-4.24 (4H, m), 5.80 (1H, t, J = 4.0 Hz), 6.85 (2H, d, J = 8.4 Hz), 7.27 (2H, d, J = 6.8 Hz), 7.36 (3H, d, J = 5.2 Hz), 7.61 (2H, t, J = 4.8 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 5.0 Hz), 31.6, 33.8 (d, $J_{C\text{-}P}$ = 139.0 Hz), 43.2 (d, $J_{C\text{-}P}$ = 4.0 Hz), 55.2, 62.7 (q, $J_{C\text{-}P}$ = 7.0 Hz), 98.3 (d, $J_{C\text{-}P}$ = 11.0 Hz), 113.9, 124.7 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.5, 129.3, 130.0, 130.9, 131.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 151.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 158.2, 168.7 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.3; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{28}O_6P^+$ $[M+H]^+$: 431.1619, found: 431.1602; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 25.4 min, Rt_2 (major) = 30.1 min; >98% ee.

Diethyl ((3*R*,4*S*)-3-(4-methylbenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3e):



93% yield (39 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{28}$ = +59.80 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.19-1.31 (6H, m), 2.24 (3H, s), 2.86-3.16 (3H, m), 3.38 (1H, t, J = 3.6 Hz), 3.99-4.15 (4H, m), 5.72 (1H, dd, J = 4.8 Hz), 7.04 (2H, d, J = 7.6 Hz), 7.17 (2H, d, J = 8.0 Hz), 7.24-7.33 (3H, m), 7.48-7.57 (2H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 6.0 Hz), 32.0, 33.7 (d, $J_{C\text{-}P}$ = 139.0 Hz), 42.9 (d, $J_{C\text{-}P}$ = 4.0 Hz), 55.2, 62.7 (q, $J_{C\text{-}P}$ = 8.0 Hz), 98.3 (d, $J_{C\text{-}P}$ = 10.0 Hz), 124.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.4, 128.8, 129.1, 129.3, 131.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 135.7, 136.0, 151.6 (d, $J_{C\text{-}P}$ = 12.0 Hz), 168.6 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.2; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{28}O_5P^+$ $[M+H]^+$: 415.1669, found: 415.1665; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 28.1 min, Rt_2 (major) = 42.1 min; >99% ee.

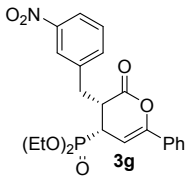
Diethyl ((3*R*,4*S*)-3-(2-nitrobenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3f):



98% yield (44 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +214.2 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.22-1.31 (6H, m), 3.13-3.31 (2H, m), 3.36 (1H, dd, J = 14.0, 4.0 Hz), 3.55 (1H, dd, J = 14.0, 8.0 Hz), 4.02-4.18 (4H, m), 5.79 (1H, dd, J = 6.8, 4.4 Hz), 7.23-7.37 (4H, m), 7.45-7.57 (3H, m), 7.85 (1H, dd, J = 7.6, 1.2 Hz), 7.92 (1H, dd, J = 8.4, 1.2 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 6.0 Hz), 31.5, 35.6 (d, $J_{C\text{-}P}$ = 139.0 Hz), 41.9 (d, $J_{C\text{-}P}$ = 5.0 Hz), 62.8 (q, $J_{C\text{-}P}$ = 7.0 Hz), 98.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 124.6 (d, $J_{C\text{-}P}$ = 1.0 Hz), 124.9, 127.9, 128.4, 129.3, 131.6 (d, $J_{C\text{-}P}$ = 4.0 Hz), 133.3, 134.2, 135.0, 148.7, 151.5 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.4 (d, $J_{C\text{-}P}$ = 4.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.0; HRMS

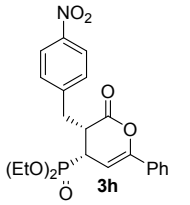
(ESI-TOF) m/z: Mass calcd. for $C_{22}H_{24}NO_7PNa^+$ $[M+Na]^+$: 468.1183, found: 468.1152; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 30:70, flow rate 0.5 mL/min, 254 nm), Rt_1 (major) = 23.9 min, Rt_2 (minor) = 27.3 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-(3-nitrobenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3g):



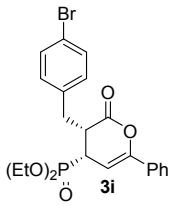
85% yield (38 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{26}$ = +79.20 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.23-1.32 (6H, m), 2.85-2.99 (1H, m), 3.03-3.28 (2H, m), 3.54 (1H, dd, J = 14.0, 5.2 Hz), 4.03-4.17 (4H, m), 5.75 (1H, dd, J = 6.8, 4.4 Hz), 7.25-7.34 (3H, m), 7.41 (1H, t, J = 7.6 Hz), 7.48-7.57 (2H, m), 7.69 (1H, d, J = 8.0 Hz), 7.98-8.05 (1H, m), 8.17 (1H, t, J = 1.6 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 5.0 Hz), 32.6, 34.2 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.4 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.9 (q, $J_{C\text{-}P}$ = 7.0 Hz), 97.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 121.7, 123.9, 124.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.5, 129.4, 129.5, 131.6 (d, $J_{C\text{-}P}$ = 5.0 Hz), 135.6, 141.0, 148.3, 151.7 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.0 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.5; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{24}NO_7PNa^+$ $[M+Na]^+$: 468.1183, found: 468.1208; HPLC analysis: (Chiralcel ODH; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 21.3 min, Rt_2 (major) = 36.9 min; >96% ee.

Diethyl ((3*R*,4*S*)-3-(4-nitrobenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3h):



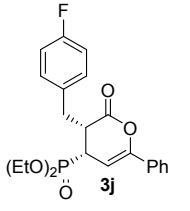
90% yield (41 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{25}$ = +68.40 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.21-1.32 (6H, m), 2.89 (1H, dt, J = 21.2, 6.8 Hz), 3.01-3.19 (1H, m), 3.23 (1H, dd, J = 14.4, 8.4), 3.53 (1H, dd, J = 14.4, 6.0 Hz), 4.01-4.17 (4H, m), 5.74 (1H, dd, J = 6.8, 4.4 Hz), 7.25-7.33 (3H, m), 7.46-7.57 (4H, m), 8.09 (2H, dd, J = 6.8, 4.0 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 6.0 Hz), 32.8, 34.2 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.3 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.9 (q, $J_{C\text{-}P}$ = 7.0 Hz), 97.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 123.6, 124.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.5, 129.5, 130.0, 131.5 (d, $J_{C\text{-}P}$ = 4.0 Hz), 146.7, 146.8, 151.7 (d, $J_{C\text{-}P}$ = 11.0 Hz), 167.9 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.4; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{24}NO_7PNa^+$ $[M+Na]^+$: 468.1183, found: 468.1180; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 30:70, flow rate 0.5 mL/min, 254 nm), Rt_1 (major) = 24.6 min, Rt_2 (minor) = 33.7 min; >94% ee.

Diethyl ((3*R*,4*S*)-3-(4-bromobenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3i):



98% yield (47 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{28}$ = +15.80 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.19-1.30 (6H, m), 2.81-3.16 (3H, m), 3.31-3.46 (1H, m), 3.97-4.16 (4H, m), 5.72 (1H, dd, J = 6.4, 4.4 Hz), 7.18 (2H, d, J = 8.4 Hz), 7.24-7.31 (3H, m), 7.34 (2H, d, J = 8.4 Hz), 7.52 (2H, d, J = 4.4 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (t, $J_{C\text{-}P}$ = 6.0 Hz), 32.0, 33.8 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.6 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.7 (q, $J_{C\text{-}P}$ = 7.0 Hz), 98.1 (d, $J_{C\text{-}P}$ = 11.0 Hz), 120.3, 124.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.4, 129.3, 130.8, 131.5, 131.7 (d, $J_{C\text{-}P}$ = 5.0 Hz), 137.9, 151.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.3 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.8; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{24}BrO_5PNa^+$ $[M+Na]^+$: 501.0437, found: 501.0426; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 14.7 min, Rt_2 (major) = 20.9 min; >99% ee.

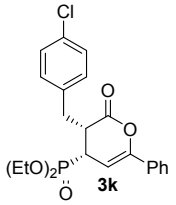
Diethyl ((3*R*,4*S*)-3-(4-fluorobenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3j):



96% yield (40 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ =

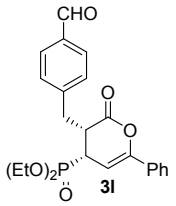
+89.00 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.20-1.30 (6H, m), 2.82-3.15 (3H, m), 3.32-3.48 (1H, m), 3.99-4.15 (4H, m), 5.73 (1H, dd, J = 6.4, 4.4 Hz), 6.86-6.96 (2H, m), 7.22-7.34 (5H, m), 7.48-7.58 (2H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (t, J_{C-P} = 6.0 Hz), 31.8, 33.8 (d, J_{C-P} = 140.0 Hz), 42.9 (d, J_{C-P} = 4.0 Hz), 62.7 (q, J_{C-P} = 7.0 Hz), 98.1 (d, J_{C-P} = 10.0 Hz), 115.2 (d, J_{C-F} = 8.4 Hz), 124.6 (d, J_{C-P} = 2.0 Hz), 128.4, 129.3, 130.6 (d, J_{C-F} = 8.0 Hz), 131.7 (d, J_{C-P} = 4.0 Hz), 134.6 (d, J_{C-P} = 4.0 Hz), 151.6 (d, J_{C-P} = 11.0 Hz), 161.6 (d, J_{C-F} = 243.0 Hz), 168.4 (d, J_{C-P} = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.9; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{24}FO_5PNa^+$ $[M+Na]^+$: 441.1238, found: 441.1232; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (major) = 19.9 min, Rt_2 (minor) = 22.9 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-(4-chlorobenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3k):



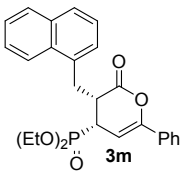
97% yield (43 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{28}$ = +18.40 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.20-1.30 (6H, m), 2.81-3.15 (3H, m), 3.32-3.47 (1H, m), 3.98-4.15 (4H, m), 5.73 (1H, dd, J = 6.8, 4.8 Hz), 7.16-7.35 (7H, m), 7.52 (2H, t, J = 4.0 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (t, J_{C-P} = 5.0 Hz), 32.0, 33.9 (d, J_{C-P} = 140.0 Hz), 42.7 (d, J_{C-P} = 4.0 Hz), 62.7 (q, J_{C-P} = 7.0 Hz), 98.1 (d, J_{C-P} = 10.0 Hz), 124.6 (d, J_{C-P} = 2.0 Hz), 128.4, 128.5, 129.4, 130.4, 131.7 (d, J_{C-P} = 4.0 Hz), 132.3, 137.4, 151.6 (d, J_{C-P} = 11.0 Hz), 168.3 (d, J_{C-P} = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.9; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{24}ClO_5PNa^+$ $[M+Na]^+$: 457.0943, found: 457.0907; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (major) = 20.2 min, Rt_2 (minor) = 22.1 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-(4-formylbenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3l):



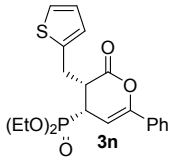
95% yield (41 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +91.80 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.30-1.40 (6H, m), 2.91-3.08 (1H, m), 3.10-3.73 (2H, m), 3.61 (1H, dd, J = 13.6, 4.8 Hz), 4.09-4.25 (4H, m), 5.82 (1H, dd, J = 6.4, 4.8 Hz), 7.38 (3H, d, J = 4.0 Hz), 7.53-7.68 (4H, m), 7.85 (2H, d, J = 8.0 Hz), 9.99 (1H, s); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (t, J_{C-P} = 5.0 Hz), 33.0, 34.2 (d, J_{C-P} = 140.0 Hz), 42.3 (d, J_{C-P} = 4.0 Hz), 62.8 (q, J_{C-P} = 8.0 Hz), 97.9 (d, J_{C-P} = 11.0 Hz), 124.6 (d, J_{C-P} = 2.0 Hz), 128.4, 128.5, 128.8, 129.4, 129.8, 129.9, 131.6 (d, J_{C-P} = 4.0 Hz), 135.0, 146.3, 151.7 (d, J_{C-P} = 11.0 Hz), 168.1 (d, J_{C-P} = 5.0 Hz), 191.7; $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.6; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{26}O_6P^+$ $[M+H]^+$: 429.1462, found: 429.1460; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 40:60, flow rate 0.5 mL/min, 254 nm), Rt_1 (major) = 36.1 min, Rt_2 (minor) = 45.2 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-(naphthalen-1-ylmethyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3m): 97% yield (44 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +125.80 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.20-1.30 (6H, m), 2.97 (1H, dt, J = 21.6, 6.8 Hz), 3.09-3.31 (1H, m), 3.55 (1H, dd, J = 14.8, 8.4 Hz), 3.93-4.22 (5H, m), 5.68 (1H, dd, J = 7.2, 4.8 Hz), 7.20-7.29 (3H, m), 7.30-7.45 (3H, m), 7.45-7.53 (2H, m), 7.63(2H, dd, J = 6.8 Hz), 7.75 (1H, d, J = 2.8 Hz), 7.88 (1H, d, J = 8.0 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.2 (d, J_{C-P} = 5.0 Hz), 29.1, 34.1 (d, J_{C-P} = 139.0 Hz), 41.5 (d, J_{C-P} = 4.0 Hz), 62.8 (q, J_{C-P} = 7.0 Hz), 98.2 (d, J_{C-P} = 11.0 Hz), 123.0, 124.6 (d, J_{C-P} = 2.0 Hz), 125.4, 125.5, 126.2, 127.3,

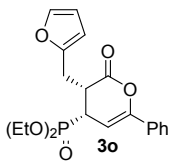


127.5, 128.4, 129.0, 129.3, 131.3, 131.7 (d, $J_{C\text{-}P}$ = 4.0 Hz), 133.9, 134.6, 151.5 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.6 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^{1}H\}$ NMR (162 MHz, $CDCl_3$): δ 23.3**;** HRMS (ESI-TOF) m/z: Mass calcd. for $C_{26}H_{28}O_5P^+$ $[M+H]^+$: 451.1669, found: 451.1666; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 25.4 min, Rt_2 (major) = 38.7 min; >99% ee.

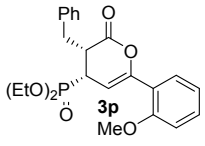
Diethyl ((3*R*,4*S*)-2-oxo-6-phenyl-3-(thiophen-2-ylmethyl)-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3n): 70% yield (29 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +129.60 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.20-1.31 (6H, m), 2.90-3.20 (2H, m), 3.35 (1H, dd, *J* = 15.2, 9.2 Hz), 3.64 (1H, dd, *J* = 15.2, 4.8 Hz), 3.98-4.17 (4H, m), 5.77 (1H, dd, *J* = 7.2, 4.8 Hz), 6.88 (1H, dd, *J* = 5.2, 3.6 Hz), 6.94 (1H, d, *J* = 7.0 Hz), 7.08 (1H, q, *J* = 0.8 Hz), 7.24-7.37 (3H, m), 7.50-7.58 (2H, m); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (t, $J_{C\text{-}P}$ = 2.0 Hz), 27.2, 33.7 (d, $J_{C\text{-}P}$ = 140.0 Hz), 43.2 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.7 (q, $J_{C\text{-}P}$ = 7.0 Hz), 98.06 (d, $J_{C\text{-}P}$ = 11.0 Hz), 123.9, 124.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.3, 126.9, 128.4, 129.4, 131.7 (d, $J_{C\text{-}P}$ = 4.0 Hz), 140.9, 151.7 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.1 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^{1}H\}$ NMR (162 MHz, $CDCl_3$): δ 22.8; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{20}H_{24}O_5PS^+$ $[M+H]^+$: 407.1077, found: 407.1076; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 37.7 min, Rt_2 (major) = 41.4 min; >98% ee.



Diethyl ((3*R*,4*S*)-3-(furan-2-ylmethyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3o): 80% yield (32 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{28}$ = +132.80 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.22 (6H, q, *J* = 7.2 Hz), 2.98 (1H, dt, *J* = 22.4, 7.2 Hz), 3.10-3.33 (2H, m), 3.37-3.52 (1H, m), 3.97-4.14 (4H, m), 5.78 (1H, dd, *J* = 6.8, 4.4 Hz), 6.14 (1H, d, *J* = 2.8 Hz), 6.23 (1H, s), 7.24 (1H, s), 7.29 (3H, d, *J* = 5.2 Hz), 7.54 (2H, q, *J* = 4.4 Hz); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 16.2 (t, $J_{C\text{-}P}$ = 5.0 Hz), 25.7, 33.8 (d, $J_{C\text{-}P}$ = 139.0 Hz), 39.6 (d, $J_{C\text{-}P}$ = 5.0 Hz), 62.6 (q, $J_{C\text{-}P}$ = 8.0 Hz), 98.0 (d, $J_{C\text{-}P}$ = 10.0 Hz), 107.2, 110.3, 124.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.4, 129.3, 131.7 (d, $J_{C\text{-}P}$ = 4.0 Hz), 141.2, 151.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 152.2, 168.1 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^{1}H\}$ NMR (162 MHz, $CDCl_3$): δ 22.9; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{20}H_{23}O_6PNa^+$ $[M+Na]^+$: 413.1125, found: 413.1117; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (major) = 37.3 min, Rt_2 (minor) = 41.5 min; >99% ee.

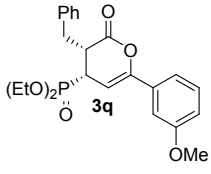


Diethyl ((3*R*,4*S*)-3-benzyl-6-(2-methoxyphenyl)-2-oxo-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3p): 95% yield (41 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{26}$ = +83.80 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.17-1.39 (6H, m), 3.01 (1H, dt, *J* = 21.6, 6.8 Hz), 3.08-3.26 (2H, m), 3.52 (1H, t, *J* = 3.6 Hz), 3.85 (3H, s), 4.09-4.23 (4H, m), 6.19 (1H, dd, *J* = 6.8, 5.2 Hz), 6.93 (1H, d, *J* = 8.0 Hz), 6.97 (1H, t, *J* = 7.6 Hz), 7.22 (1H, t, *J* = 7.2 Hz), 7.30 (3H, t, *J* = 7.6 Hz), 7.38 (2H, d, *J* = 7.6 Hz), 7.65 (1H, d, *J* = 7.6 Hz); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (q, $J_{C\text{-}P}$ = 1.0 Hz), 32.4 (d, $J_{C\text{-}P}$ = 1.0 Hz), 34.1 (d, $J_{C\text{-}P}$ = 139.0 Hz), 42.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 55.4, 62.6 (q, $J_{C\text{-}P}$ = 8.0 Hz), 103.5 (d, $J_{C\text{-}P}$ = 10.0 Hz), 111.1, 120.4, 120.7 (d, $J_{C\text{-}P}$ = 5.0 Hz), 126.4, 128.0 (d, $J_{C\text{-}P}$ = 2.0 Hz), 128.4, 129.1, 130.1, 139.1, 148.4 (d, $J_{C\text{-}P}$ = 11.0 Hz), 157.0 (d, $J_{C\text{-}P}$ = 1.0 Hz), 168.9 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^{1}H\}$ NMR (162 MHz, $CDCl_3$): δ 23.4; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{27}O_6PNa^+$ $[M+Na]^+$: 453.1438, found: 453.1450; HPLC analysis: (Chiralcel IC;



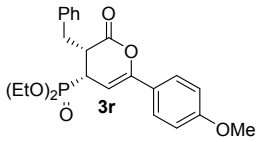
hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 38.2 min, Rt_2 (major) = 55.8 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-(3-methoxybenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3q):



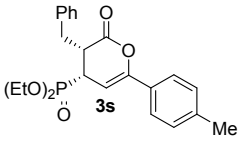
96% yield (42 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +17.40 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.29-1.38 (6H, m), 2.93-3.26 (3H, m), 3.52 (1H, dd, J = 13.6, 4.0 Hz), 3.82 (3H, s), 4.09-4.21 (4H, m), 5.82 (1H, dd, J = 6.4, 4.8 Hz), 6.91 (1H, d, J = 8.0 Hz), 7.14 (1H, s), 7.16-7.41 (7H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 5.0 Hz), 32.5, 33.8 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 55.3, 62.8 (q, $J_{C\text{-}P}$ = 7.0 Hz), 98.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 110.0 (d, $J_{C\text{-}P}$ = 1.0 Hz), 115.2, 117.1 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.5, 128.5, 129.0, 129.5, 133.2 (d, $J_{C\text{-}P}$ = 4.0 Hz), 138.9, 151.4 (d, $J_{C\text{-}P}$ = 11.0 Hz), 159.7, 168.6 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.1; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{27}O_6PNa^+$ $[M+Na]^+$: 453.1438, found: 453.1436; HPLC analysis: (Chiralcel ODH; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (major) = 15.3 min, Rt_2 (minor) = 20.3 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-(4-methoxybenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3r):



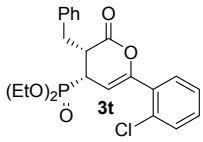
95% yield (41 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{29}$ = +68.80 (c 0.05, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.28-1.39 (6H, m), 2.91-3.25 (3H, m), 3.52 (1H, dd, J = 13.6, 3.6 Hz), 3.81 (3H, s), 4.07-4.22 (4H, m), 5.68 (1H, dd, J = 6.4, 4.8 Hz), 6.88 (2H, d, J = 8.8 Hz), 7.22 (1H, t, J = 6.8 Hz), 7.28-7.40 (4H, m), 7.54 (2H, d, J = 8.8 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 6.0 Hz), 32.5, 33.7 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.9 (d, $J_{C\text{-}P}$ = 4.0 Hz), 55.2, 62.6 (q, $J_{C\text{-}P}$ = 7.0 Hz), 96.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 113.8, 124.4 (d, $J_{C\text{-}P}$ = 4.0 Hz), 126.1 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.5, 128.4, 129.0, 139.0, 151.4 (d, $J_{C\text{-}P}$ = 11.0 Hz), 160.4, 168.7 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.5; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{27}O_6PNa^+$ $[M+Na]^+$: 453.1438, found: 453.1450; HPLC analysis: (Chiralcel ODH; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (major) = 15.0 min, Rt_2 (minor) = 20.8 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-(4-methylbenzyl)-2-oxo-6-phenyl-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3s):



90% yield (37 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +59.60 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.19-1.30 (6H, m), 2.27 (3H, s), 2.85-3.18 (3H, m), 3.43 (1H, dd, J = 13.2, 4.0 Hz), 3.98-4.14 (4H, m), 5.68 (1H, dd, J = 6.8, 4.8 Hz), 7.09 (2H, d, J = 8.0 Hz), 7.13 (1H, t, J = 7.2 Hz), 7.22 (2H, t, J = 7.6 Hz), 7.28 (2H, d, J = 7.6 Hz), 7.42 (2H, t, J = 8.0 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 5.0 Hz), 21.1, 32.4, 33.7 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.7 (q, $J_{C\text{-}P}$ = 7.0 Hz), 97.2 (d, $J_{C\text{-}P}$ = 10.0 Hz), 124.5 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.4, 128.4, 129.0, 129.1, 138.9, 139.4, 151.6, 151.7, 168.7 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.3; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{28}O_5P^+$ $[M+H]^+$: 415.1669, found: 415.1668; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 29.3 min, Rt_2 (major) = 42.2 min; >99% ee.

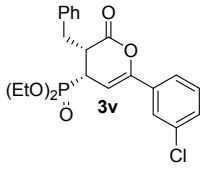
Diethyl ((3*R*,4*S*)-3-benzyl-6-(2-chlorophenyl)-2-oxo-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3t):



93% yield (40 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{28}$ =

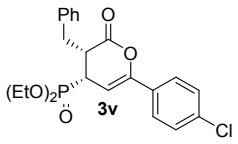
+108.00 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.31-1.40 (6H, m), 3.01 (1H, dt, J = 21.6, 6.4 Hz), 3.10-3.31 (2H, m), 3.54 (1H, dd, J = 13.2, 3.6 Hz), 4.10-4.26 (4H, m), 5.70 (1H, dd, J = 6.8, 4.4 Hz), 7.21-7.37 (5H, m), 7.40 (3H, d, J = 8.0 Hz), 7.47 (1H, t, J = 1.6 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 5.0 Hz), 32.4, 33.8 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.7 (d, $J_{C\text{-}P}$ = 5.0 Hz), 62.7 (q, $J_{C\text{-}P}$ = 7.0 Hz), 104.4 (d, $J_{C\text{-}P}$ = 10.0 Hz), 126.5, 126.7, 128.5, 129.1, 130.2, 130.3 (d, $J_{C\text{-}P}$ = 2.0 Hz), 130.4, 131.7 (d, $J_{C\text{-}P}$ = 3.0 Hz), 132.6 (d, $J_{C\text{-}P}$ = 3.0 Hz), 138.8, 150.0 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.5 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.0; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{24}ClO_5PNa^+$ $[M+Na]^+$: 457.0943, found: 457.0935; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 36.6 min, Rt_2 (major) = 39.6 min; >98% ee.

Diethyl ((3*R*,4*S*)-3-benzyl-6-(3-chlorophenyl)-2-oxo-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3u):



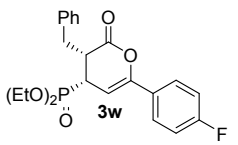
81% yield (35 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +53.20 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.29-1.39 (6H, m), 2.92-3.26 (3H, m), 3.44-3.60 (1H, m), 4.08-4.23 (4H, m), 5.84 (1H, dd, J = 6.4, 4.4 Hz), 7.20-7.40 (7H, m), 7.48 (1H, d, J = 6.8 Hz), 7.60 (1H, s); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (q, $J_{C\text{-}P}$ = 2.0 Hz), 32.4, 33.8 (d, $J_{C\text{-}P}$ = 139.0 Hz), 42.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.8 (q, $J_{C\text{-}P}$ = 6.0 Hz), 99.6 (d, $J_{C\text{-}P}$ = 10.0 Hz), 122.8 (d, $J_{C\text{-}P}$ = 2.0 Hz), 124.8 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.6, 128.5, 129.0, 129.3, 129.7, 133.6 (d, $J_{C\text{-}P}$ = 5.0 Hz), 137.7, 138.8, 151.3 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.2 (d, $J_{C\text{-}P}$ = 4.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.9; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{25}ClO_5P^+$ $[M+H]^+$: 435.1123, found: 435.1119; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 27.6 min, Rt_2 (major) = 37.6 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-benzyl-6-(4-chlorophenyl)-2-oxo-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3v):



90% yield (39 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{29}$ = +52.57 (c 0.035, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.28-1.39 (6H, m), 2.93-3.25 (3H, m), 3.46-3.58 (1H, m), 4.07-4.22 (4H, m), 5.80 (1H, dd, J = 6.4, 4.8 Hz), 7.22 (1H, t, J = 6.8 Hz), 7.28-7.40 (6H, m), 7.54 (2H, d, J = 8.4 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 5.0 Hz), 32.4, 33.8 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.8 (q, $J_{C\text{-}P}$ = 8.0 Hz), 98.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 125.9 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.5, 128.5, 128.7, 129.0, 130.3 (d, $J_{C\text{-}P}$ = 4.0 Hz), 135.3, 138.8, 150.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.3 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.0; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{24}ClO_5PNa^+$ $[M+Na]^+$: 457.0943, found: 457.0915; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 34.7 min, Rt_2 (major) = 39.5 min; >99% ee.

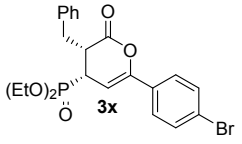
Diethyl ((3*R*,4*S*)-3-benzyl-6-(4-fluorophenyl)-2-oxo-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3w):



98% yield (42 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +105.40 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.29-1.39 (6H, m), 2.92-3.28 (3H, m), 3.52 (1H, dd, J = 13.2, 3.6 Hz), 4.07-4.22 (4H, m), 5.75 (1H, dd, J = 6.4, 4.8 Hz), 7.04 (2H, t, J = 8.4 Hz), 7.22 (1H, t, J = 7.2 Hz), 7.28-7.41 (4H, m), 7.59 (2H, dd, J = 8.4, 5.2 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.4 (d, $J_{C\text{-}P}$ = 5.0 Hz), 32.5, 33.9 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.8 (d, $J_{C\text{-}P}$ = 5.0 Hz), 62.8 (q, $J_{C\text{-}P}$ = 7.0 Hz), 98.1 (d, $J_{C\text{-}P}$ = 9.0 Hz), 115.6 (d, $J_{C\text{-}F}$ = 22.0 Hz), 126.6, 126.6 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.7 (d, $J_{C\text{-}F}$ = 1.0 Hz), 128.1 (d, $J_{C\text{-}P}$ = 7.0 Hz), 128.2 (d, $J_{C\text{-}F}$ = 27.0 Hz), 128.5, 129.0, 138.9, 150.8 (d, $J_{C\text{-}P}$ = 11.0 Hz), 163.4 (d, $J_{C\text{-}F}$ = 249.0 Hz), 168.5 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.1; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{25}FO_5P^+$ $[M+H]^+$: 419.1419, found:

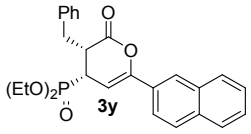
419.1417; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 34.3 min, Rt_2 (major) = 39.4 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-benzyl-6-(4-bromophenyl)-2-oxo-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3x):



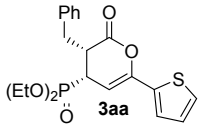
90% yield (44 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +19.00 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.27-1.38 (6H, m), 2.91-3.27 (3H, m), 3.45-3.59 (1H, m), 4.06-4.22 (4H, m), 5.82 (1H, dd, J = 6.4, 4.8 Hz), 7.22 (1H, t, J = 6.8 Hz), 7.28-7.40 (4H, m), 7.42-7.55 (4H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (t, $J_{C\text{-}P}$ = 6.0 Hz), 32.5, 33.9 (d, $J_{C\text{-}P}$ = 139.0 Hz), 42.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.8 (q, $J_{C\text{-}P}$ = 8.0 Hz), 98.9 (d, $J_{C\text{-}P}$ = 11.0 Hz), 123.6, 126.2 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.6, 128.5, 129.0, 130.7 (d, $J_{C\text{-}P}$ = 4.0 Hz), 131.7, 138.8, 150.7 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.3 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.8; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{22}H_{25}BrO_5P^+$ $[M+H]^+$: 479.0618, found: 479.0614; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 36.3 min, Rt_2 (major) = 40.4 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-benzyl-6-(naphthalen-2-yl)-2-oxo-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3y):



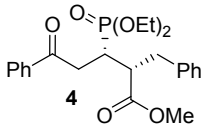
96% yield (44 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{26}$ = +61.60 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.29-1.39 (6H, m), 2.97-3.32 (3H, m), 3.56 (1H, dd, J = 13.2, 3.2 Hz), 4.08-4.24 (4H, m), 5.96 (1H, dd, J = 6.4, 4.8 Hz), 7.23 (1H, t, J = 7.2 Hz), 7.31 (2H, t, J = 7.6 Hz), 7.38 (2H, d, J = 7.6 Hz), 7.46-7.54 (2H, m), 7.63 (1H, d, J = 8.4 Hz), 7.81 (2H, d, J = 8.8 Hz), 7.86 (1H, t, J = 5.6 Hz), 8.16 (1H, s); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 6.0 Hz), 32.5, 33.9 (d, $J_{C\text{-}P}$ = 140.0 Hz), 42.9 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.7 (q, $J_{C\text{-}P}$ = 7.0 Hz), 98.8 (d, $J_{C\text{-}P}$ = 10.0 Hz), 121.8, 124.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 126.5, 126.6, 126.8, 127.5, 128.2, 128.5, 128.6, 128.8 (d, $J_{C\text{-}P}$ = 4.0 Hz), 129.0, 132.9, 133.5, 138.9, 151.5 (d, $J_{C\text{-}P}$ = 11.0 Hz), 168.7 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 23.1; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{26}H_{27}O_5PNa^+$ $[M+Na]^+$: 473.1489, found: 473.1484; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 42.8 min, Rt_2 (major) = 50.3 min; >99% ee.

Diethyl ((3*R*,4*S*)-3-benzyl-6-(furan-2-yl)-2-oxo-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3z): 97% yield (38 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{26}$ = +6.00 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.21-1.31 (6H, m), 2.82-3.19 (3H, m), 3.35-3.51 (1H, m), 3.99-4.17 (4H, m), 5.67 (1H, dd, J = 6.8, 4.8 Hz), 6.34 (1H, t, J = 3.2 Hz), 6.53 (1H, d, J = 2.0 Hz), 7.14 (1H, t, J = 6.8 Hz), 7.19-7.35 (5H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.2 (q, $J_{C\text{-}P}$ = 3.0 Hz), 32.5, 33.6 (d, $J_{C\text{-}P}$ = 140.0 Hz), 43.1 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.8 (q, $J_{C\text{-}P}$ = 3.0 Hz), 96.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 108.2 (d, $J_{C\text{-}P}$ = 3.0 Hz), 111.4, 126.5, 128.4, 129.0, 138.8, 143.3, 131.6 (d, $J_{C\text{-}P}$ = 11.0 Hz), 146.5 (d, $J_{C\text{-}P}$ = 6.0 Hz), 168.1 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.9; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{20}H_{23}O_6PNa^+$ $[M+Na]^+$: 413.1125, found: 413.1114; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 70:30, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 41.4 min, Rt_2 (major) = 57.6 min; >98% ee.

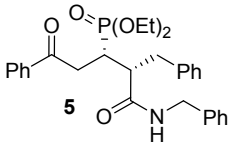


Diethyl ((3*R*,4*S*)-3-benzyl-2-oxo-6-(thiophen-2-yl)-3,4-dihydro-2*H*-pyran-4-yl)phosphonate (3aa): 95% yield (38 mg), pale yellow gummy liquid, eluent:

EtOAc/Hexane (8:2). $[\alpha]_D^{27}$ = +86.40 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.31-1.40 (6H, m), 2.98 (1H, dt, J = 21.6, 6.4 Hz), 3.08-3.30 (2H, m), 3.53 (1H, dd, J = 13.2, 3.6 Hz), 4.08-4.25 (4H, m), 5.69 (1H, dd, J = 7.2, 4.8 Hz), 7.02 (1H, t, J = 4.4 Hz), 7.20-7.43 (7H, m); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 6.0 Hz), 32.5, 33.8 (d, $J_{C\text{-}P}$ = 140.0 Hz), 43.0 (d, $J_{C\text{-}P}$ = 4.0 Hz), 62.8 (q, $J_{C\text{-}P}$ = 7.0 Hz), 97.2 (d, $J_{C\text{-}P}$ = 11.0 Hz), 125.0 (d, $J_{C\text{-}P}$ = 2.0 Hz), 126.1, 126.5, 127.5, 128.5, 129.0, 135.3 (d, $J_{C\text{-}P}$ = 5.0 Hz), 138.8, 147.5 (d, $J_{C\text{-}P}$ = 12.0 Hz), 168.1 (d, $J_{C\text{-}P}$ = 5.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 22.9; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{20}H_{24}O_5PS^+$ $[M+H]^+$: 407.1077, found: 407.1076; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, 254 nm), Rt_1 (minor) = 25.6 min, Rt_2 (major) = 27.9 min; >99% ee.



Methyl (2*R*,3*S*)-2-benzyl-3-(diethoxyphosphoryl)-5-oxo-5-phenylpentanoate (4): 99% yield (43 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{28}$ = -20.80 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.15-1.30 (6H, m), 2.87-3.18 (3H, m), 3.19-3.32 (1H, m), 3.33-3.43 (2H, m), 3.46 (3H, s), 3.97-4.15 (4H, m), 7.04-7.24 (5H, m), 7.40 (2H, t, J = 7.6 Hz), 7.50 (1H, t, J = 7.2 Hz), 7.92 (2H, d, J = 7.2 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.3 (d, $J_{C\text{-}P}$ = 4.0 Hz), 33.0 (d, $J_{C\text{-}P}$ = 142.0 Hz), 34.4 (d, $J_{C\text{-}P}$ = 3.0 Hz), 46.1 (d, $J_{C\text{-}P}$ = 1.0 Hz), 51.8, 62.1 (q, $J_{C\text{-}P}$ = 7.0 Hz), 126.3, 128.1, 128.3, 128.6, 128.9, 133.2, 136.5, 139.2, 173.5 (d, $J_{C\text{-}P}$ = 17.0 Hz), 196.7 (d, $J_{C\text{-}P}$ = 10.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 30.2; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{23}H_{29}O_6PNa^+$ $[M+Na]^+$: 455.1594, found: 455.1592; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 60:40, flow rate 0.5 mL/min, UV 254 nm), Rt_1 (major) = 30.2 min, Rt_2 (minor) = 41.3 min; >99% ee.



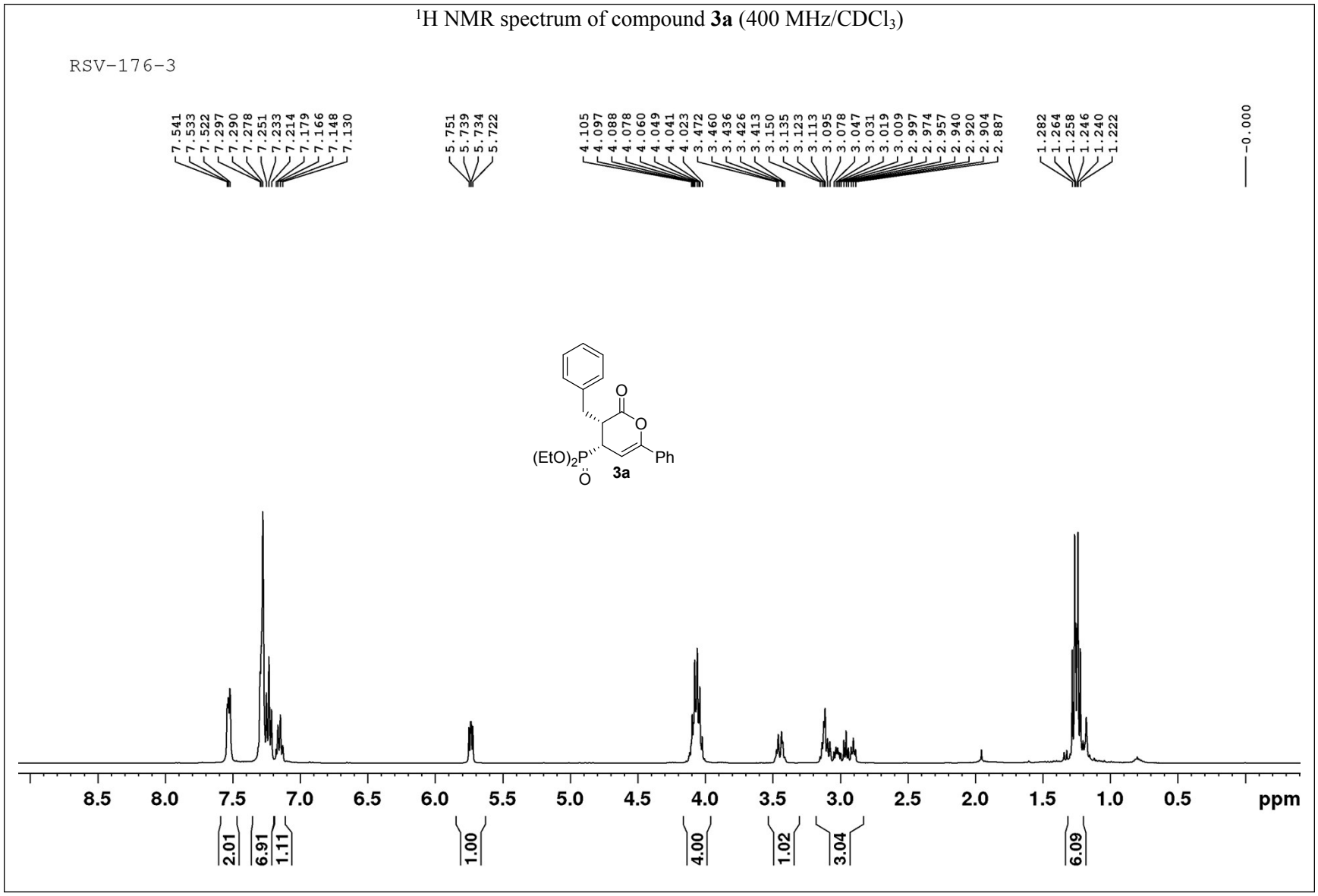
Diethyl ((2*R*,3*S*)-2-benzyl-1-(benzylamino)-1,5-dioxo-5-phenylpentan-3-yl)phosphonate (5): 98% yield (50 mg), pale yellow gummy liquid, eluent: EtOAc/Hexane (8:2). $[\alpha]_D^{28}$ = -83.00 (c 0.1, $CHCl_3$); ^1H NMR (400 MHz, $CDCl_3$): δ 1.25 (6H, t, J = 6.8 Hz), 3.02-3.19 (3H, m), 3.27-3.56 (2H, m), 3.63-3.80 (1H, m), 3.97-4.20 (5H, m), 4.37 (1H, dd, J = 6.4 Hz), 6.42 (1H, t, J = 5.6), 6.92 (2H, t, J = 3.6 Hz), 7.16-7.31 (8H, m), 7.48 (2H, t, J = 7.6 Hz), 7.57 (1H, t, J = 7.2 Hz), 8.01 (2H, d, J = 7.2 Hz); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 16.2 (t, $J_{C\text{-}P}$ = 6.0 Hz), 33.3 (d, $J_{C\text{-}P}$ = 140.0 Hz), 34.5 (d, $J_{C\text{-}P}$ = 3.0 Hz), 34.7 (d, $J_{C\text{-}P}$ = 4.0 Hz), 43.1, 47.3 (d, $J_{C\text{-}P}$ = 2.0 Hz), 61.7 (q, $J_{C\text{-}P}$ = 7.0 Hz), 126.2, 126.9, 127.3, 128.0, 128.3, 128.4, 128.5, 129.0, 133.0, 136.7, 137.9, 139.3, 172.3 (d, $J_{C\text{-}P}$ = 15.0 Hz), 196.9 (d, $J_{C\text{-}P}$ = 6.0 Hz); $^{31}P\{^1H\}$ NMR (162 MHz, $CDCl_3$): δ 30.9; HRMS (ESI-TOF) m/z: Mass calcd. for $C_{29}H_{35}NO_5P^+$ $[M+H]^+$: 508.2248, found: 508.2248; HPLC analysis: (Chiralcel IC; hexane/*i*-PrOH 40:60, flow rate 0.5 mL/min, UV 254 nm), Rt_1 (minor) = 15.8 min, Rt_2 (major) = 18.6 min; >96% ee.

References

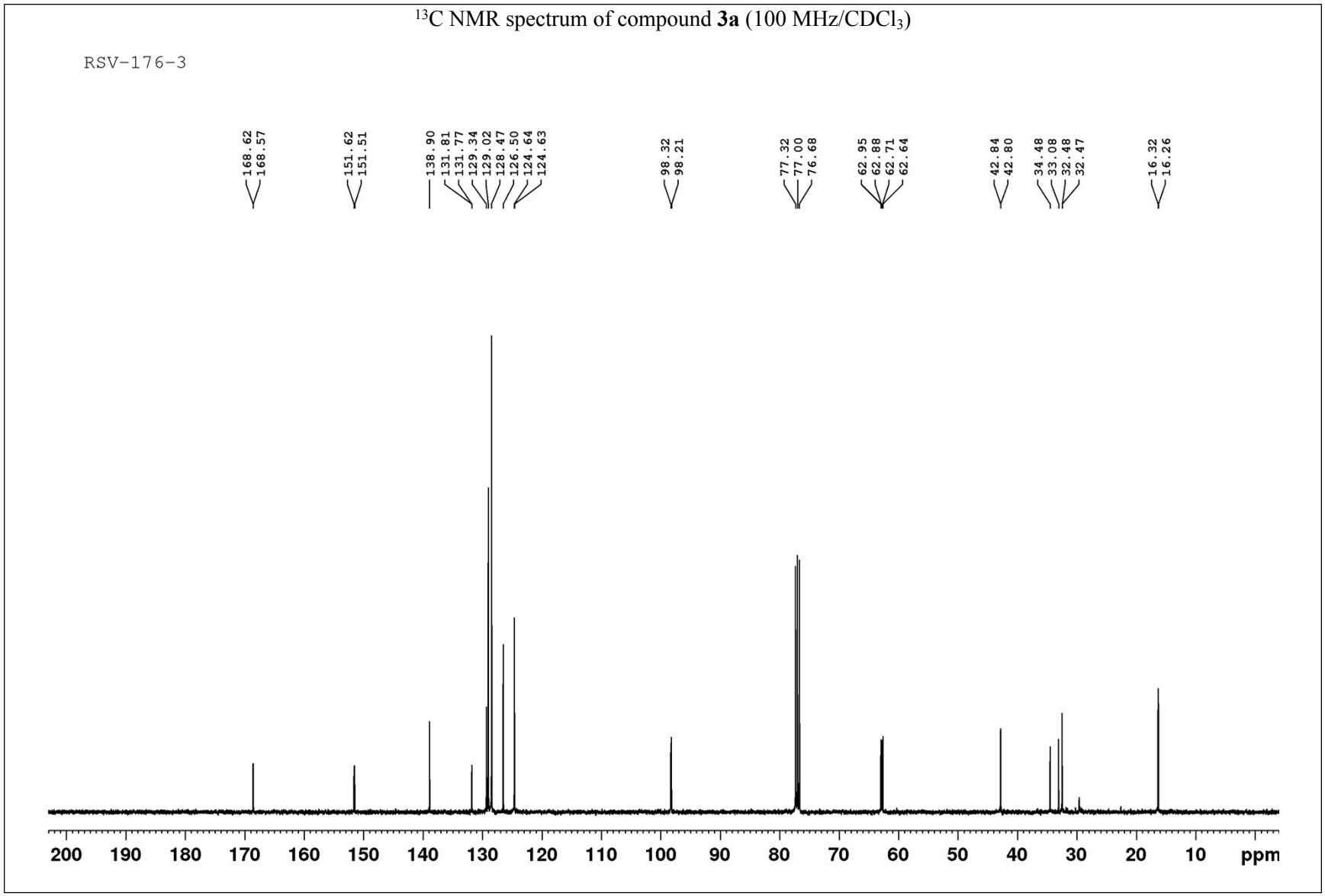
1. Chogii, I., Das, P.; Fell, J. S.; Scott, K. A.; Crawford, M. N.; Houk, K. N.; Njardarson, J. T. *J. Am. Chem. Soc*. **2017**, *139*, 13141-13146.
2. Zhou, Q. Q.; Yuan, X.; Xiao, Y. C.; Dong, L.; Chen, Y. C. *Tetrahedron*, **2013**, *69*, 10369-10374.

VI: ^1H, $^{13}C\{^1H\}$ & $^{31}P\{^1H\}$ NMR spectra of the products

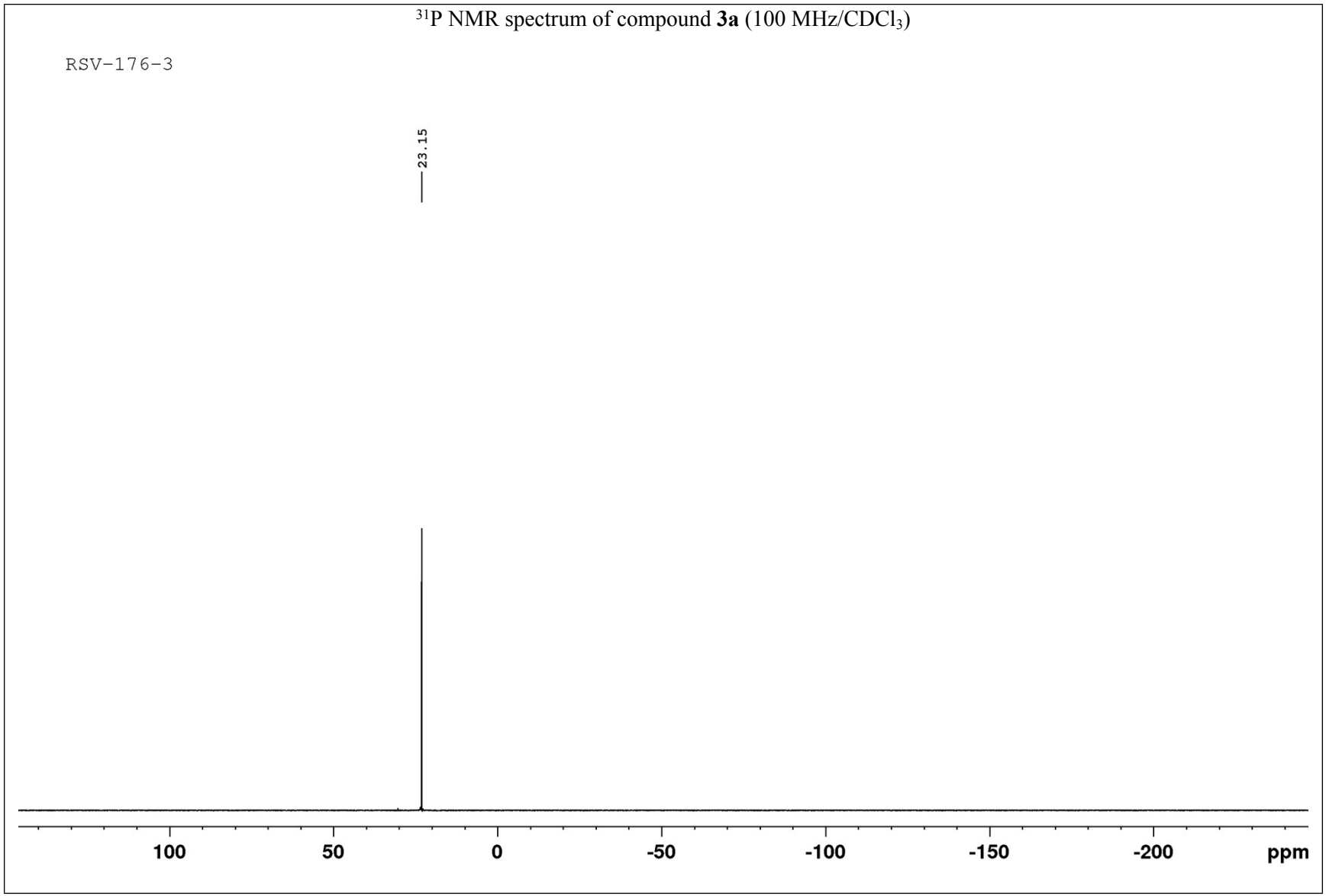
^{1}H NMR spectrum of compound **3a** (400 MHz/$CDCl_3$)



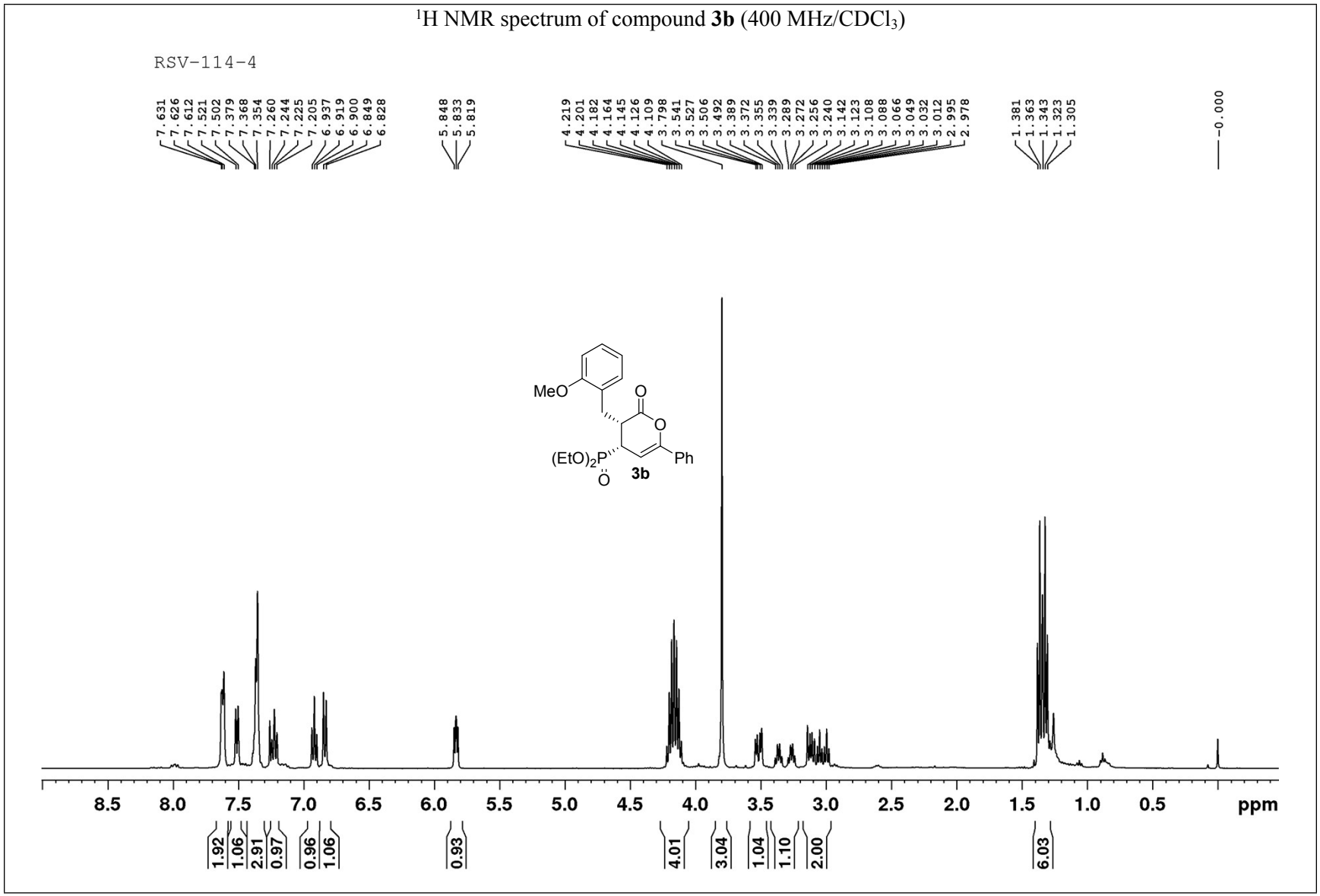
^{13}C NMR spectrum of compound **3a** (100 MHz/$CDCl_3$)



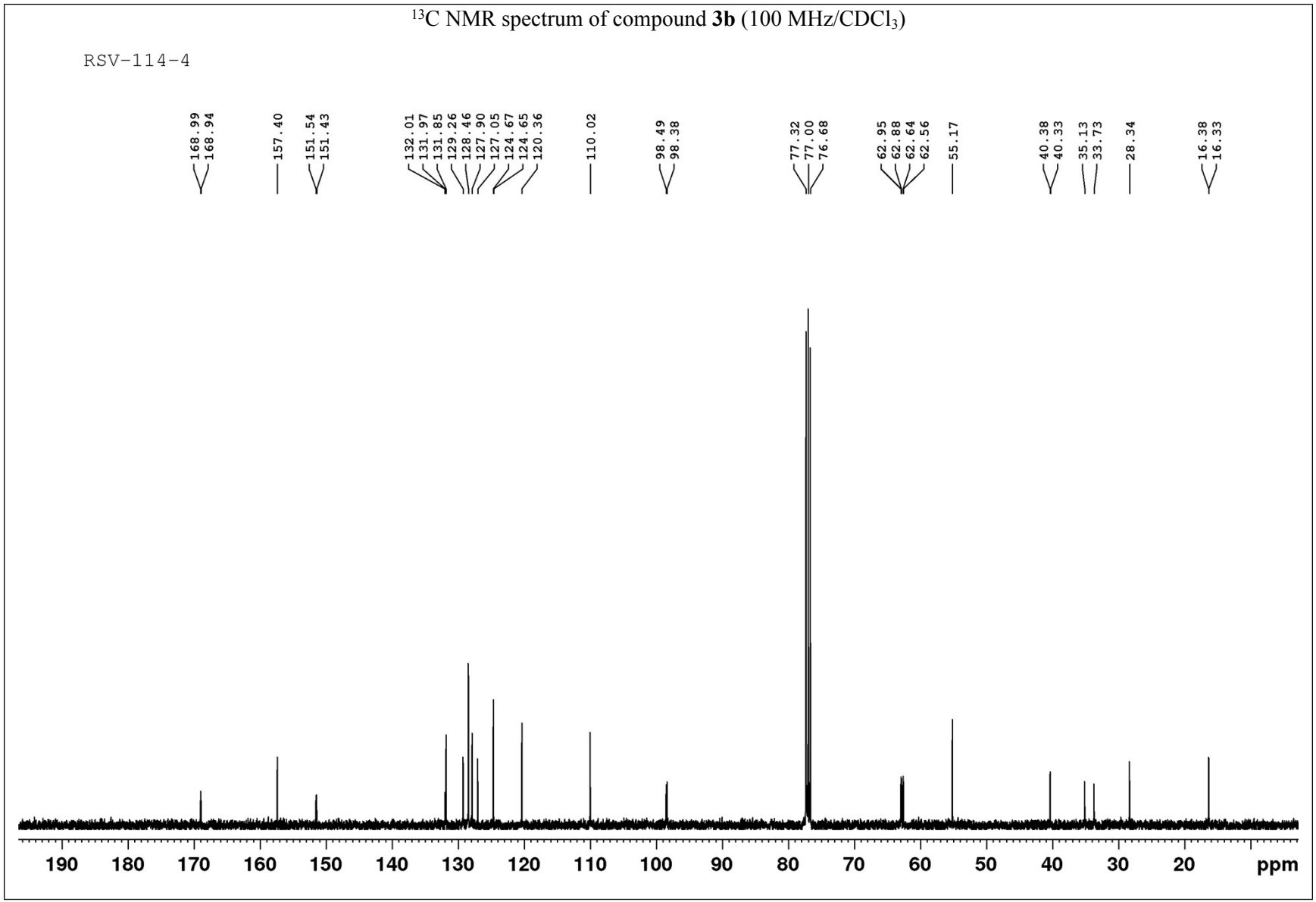
^{31}P NMR spectrum of compound **3a** (100 MHz/$CDCl_3$)



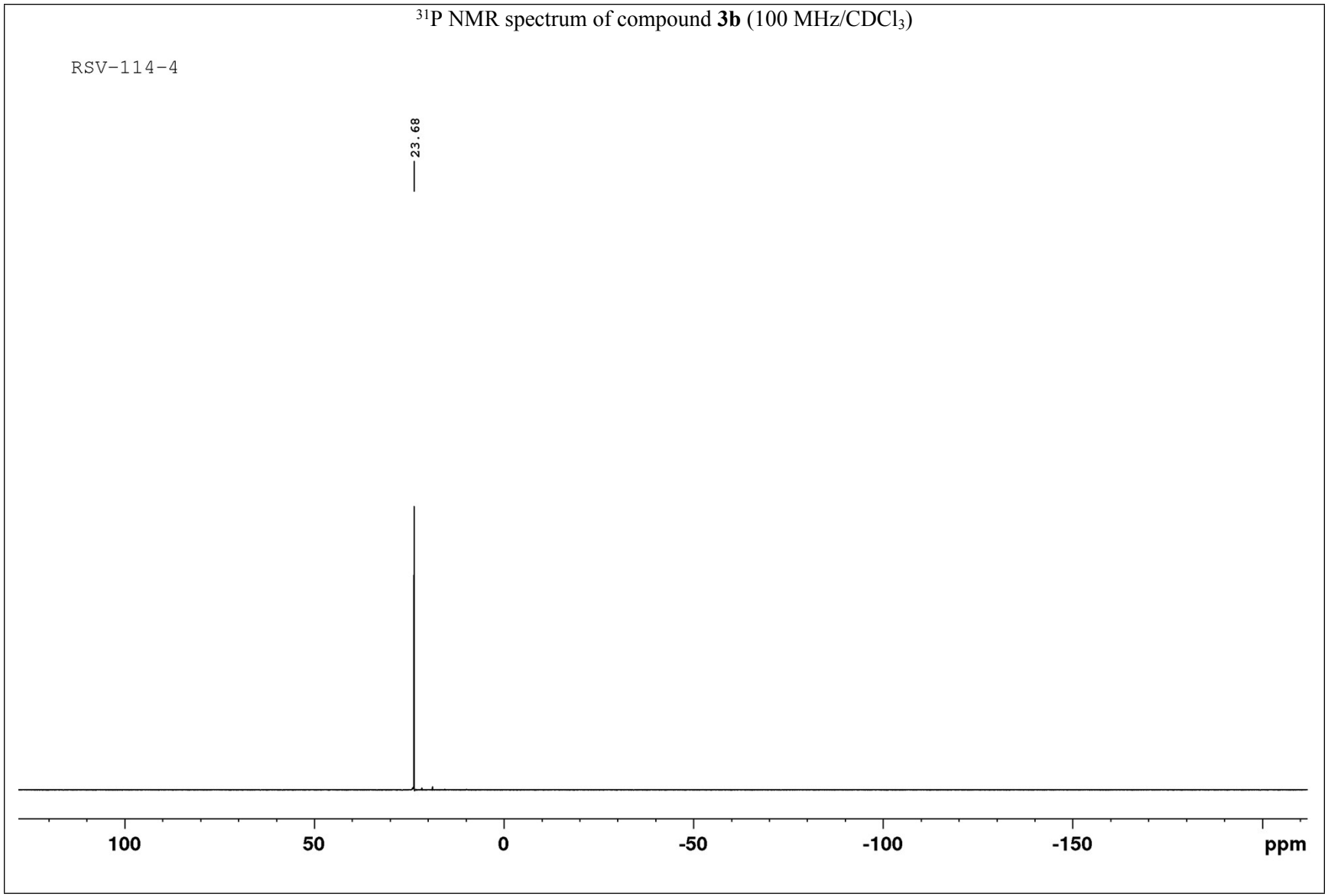
^{1}H NMR spectrum of compound **3b** (400 MHz/$CDCl_3$)



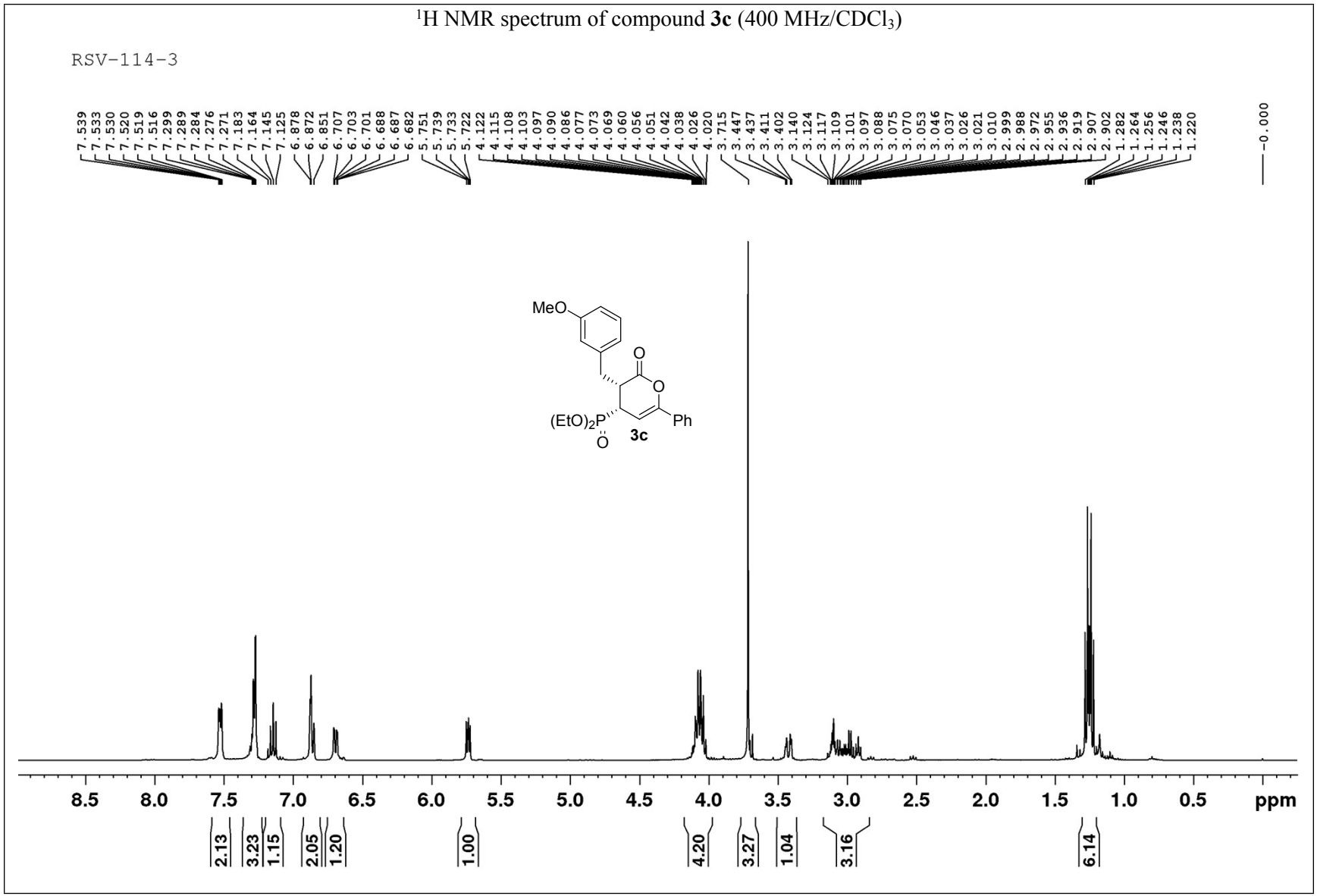
^{13}C NMR spectrum of compound **3b** (100 MHz/$CDCl_3$)



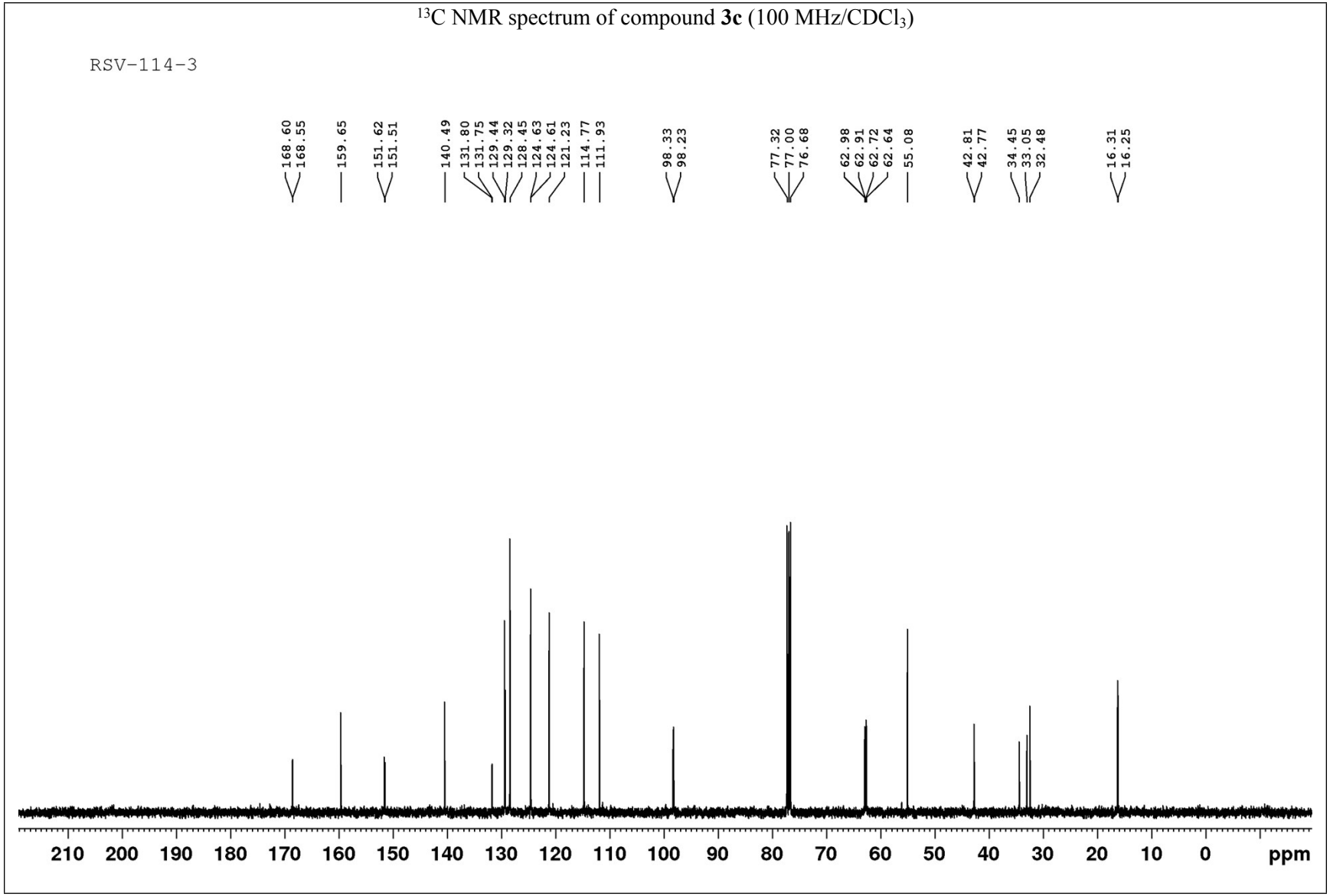
^{31}P NMR spectrum of compound **3b** (100 MHz/$CDCl_3$)



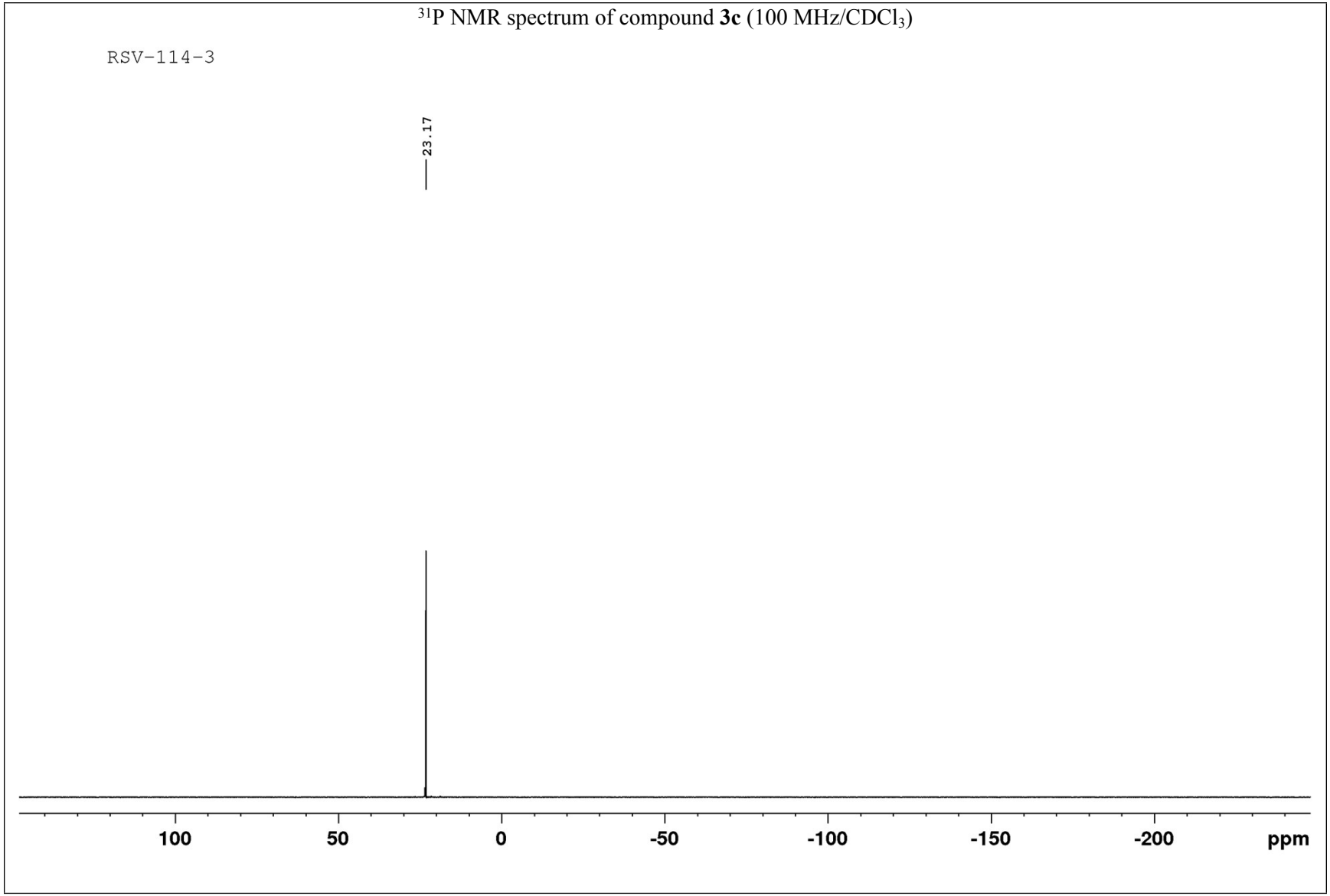
^{1}H NMR spectrum of compound **3c** (400 MHz/$CDCl_3$)



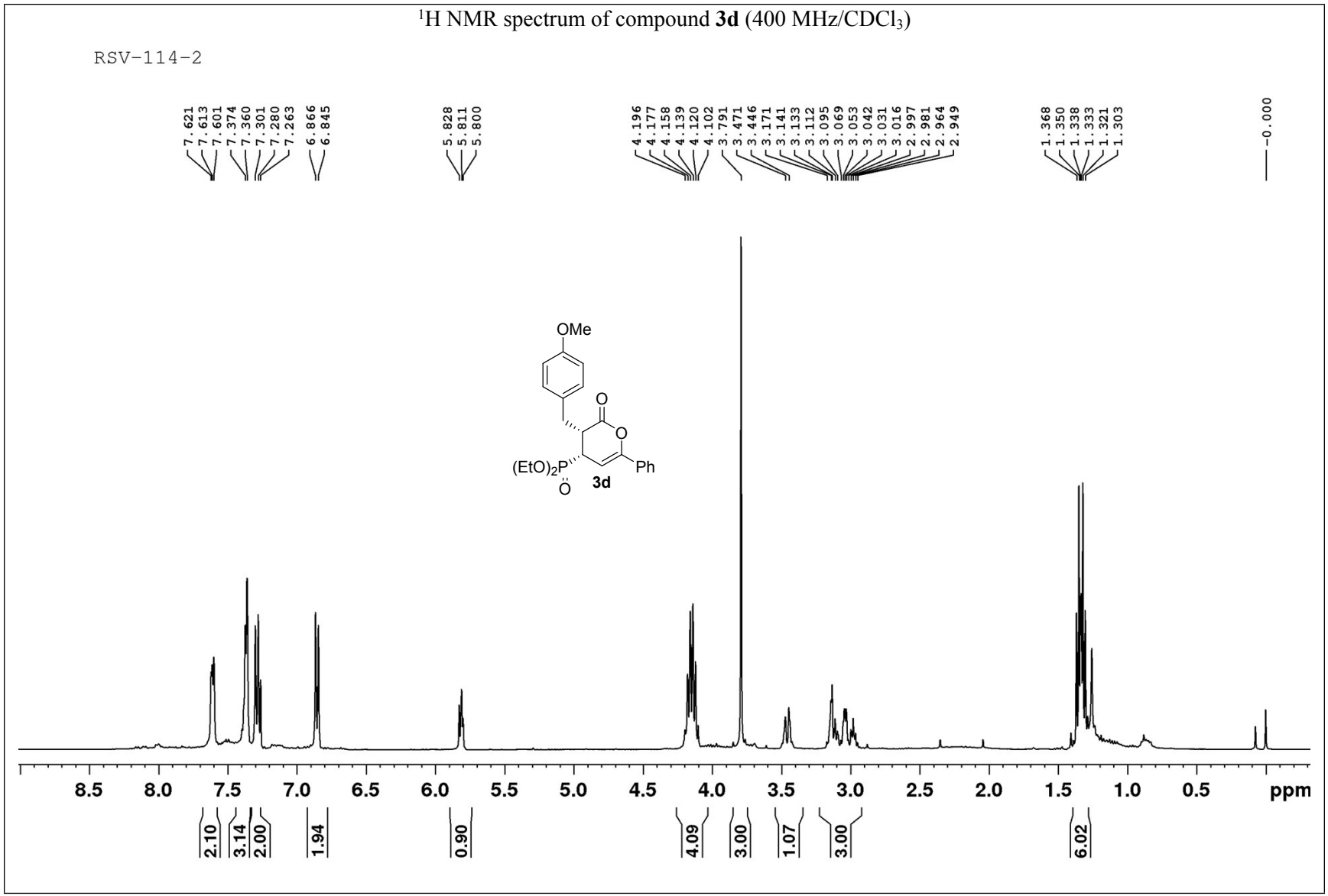
^{13}C NMR spectrum of compound **3c** (100 MHz/$CDCl_3$)



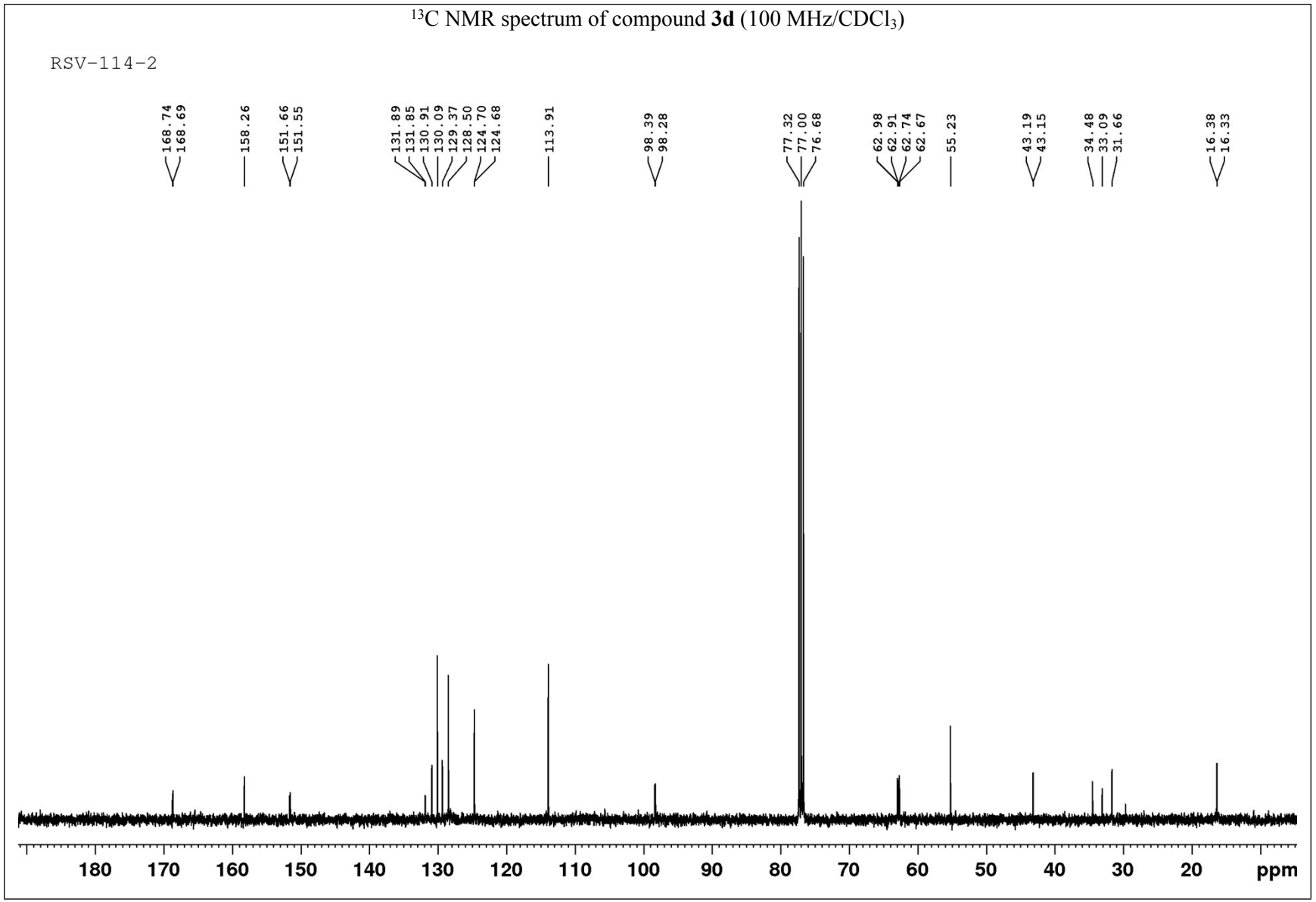
^{31}P NMR spectrum of compound **3c** (100 MHz/$CDCl_3$)



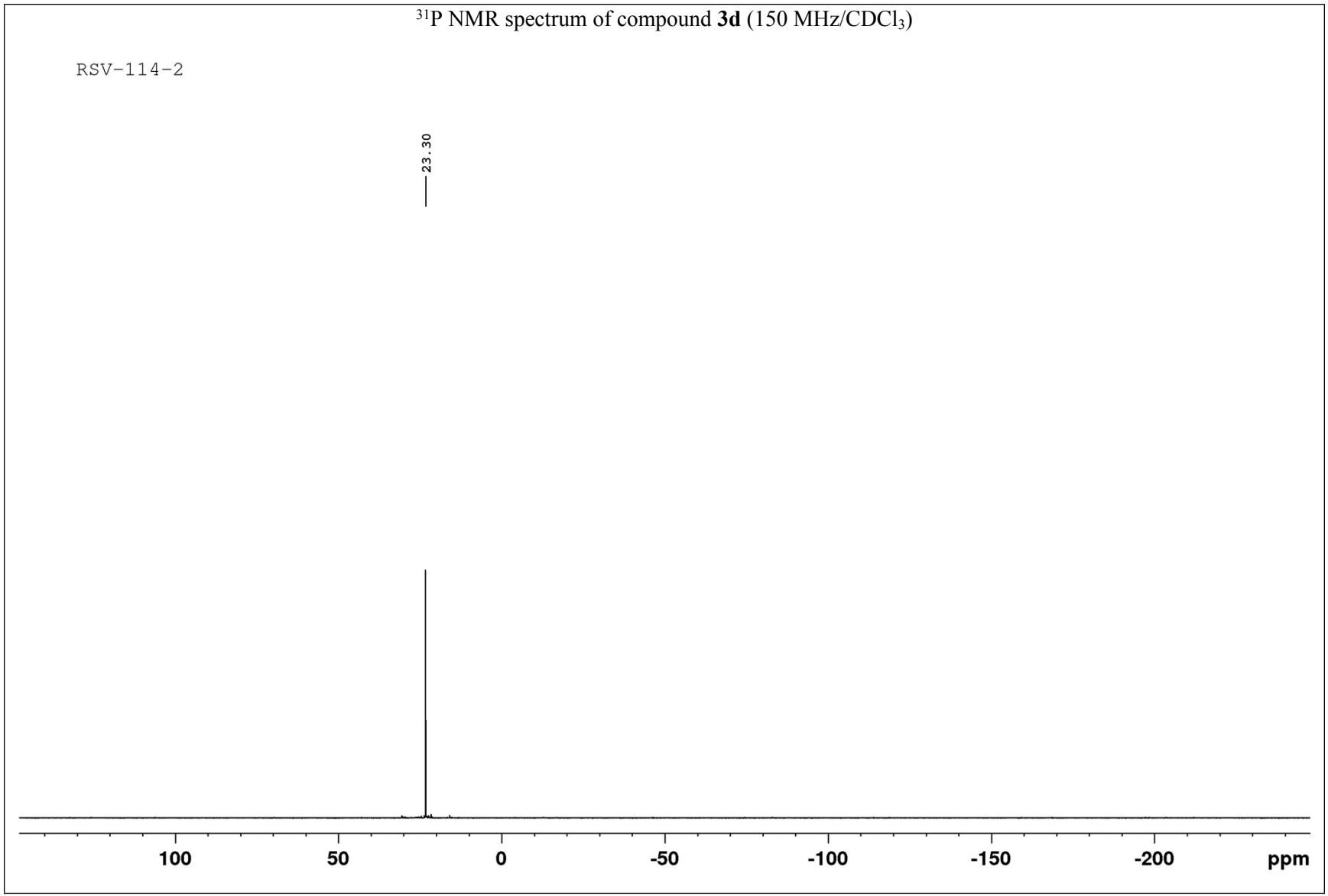
^{1}H NMR spectrum of compound **3d** (400 MHz/$CDCl_3$)



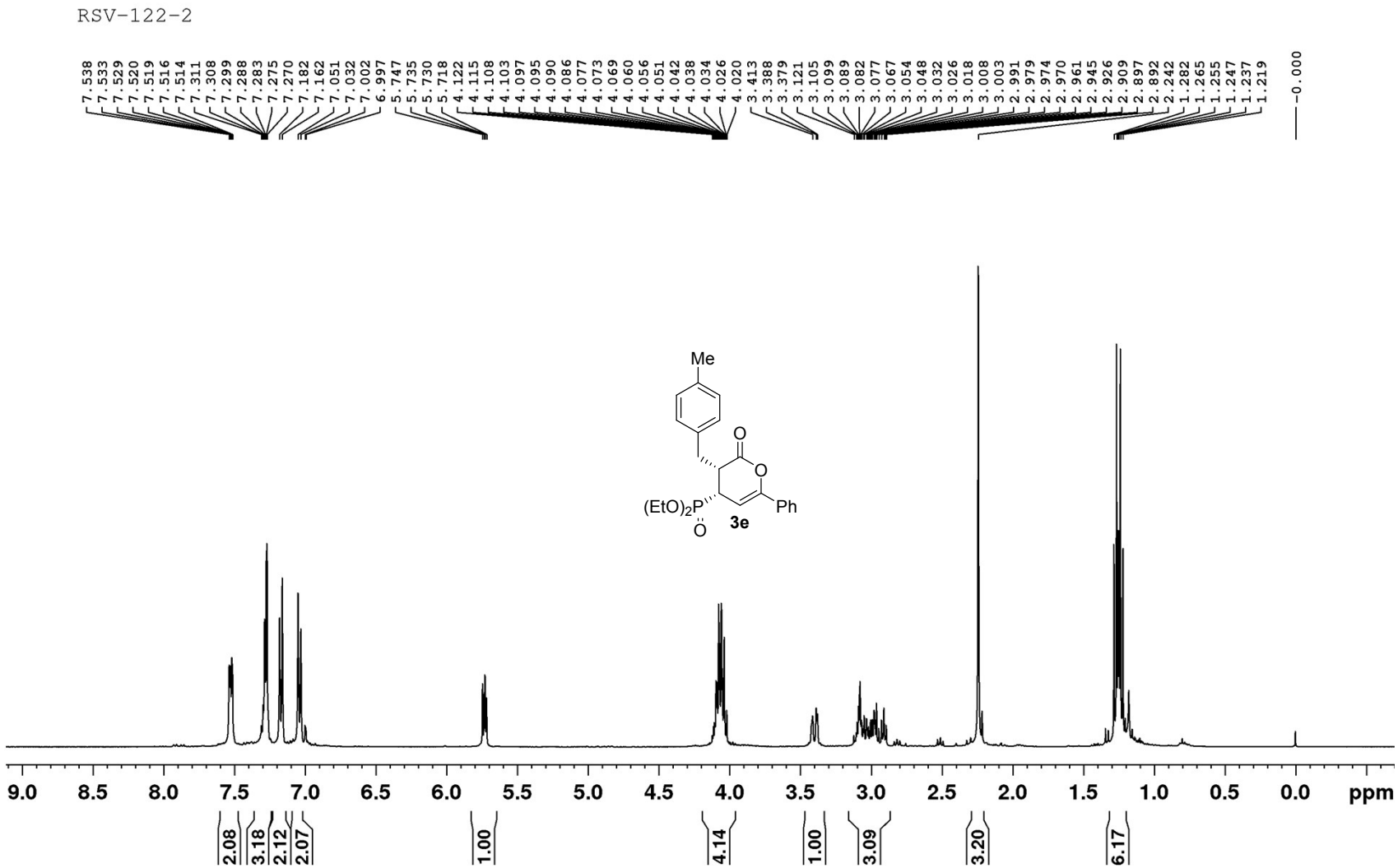
^{13}C NMR spectrum of compound **3d** (100 MHz/$CDCl_3$)



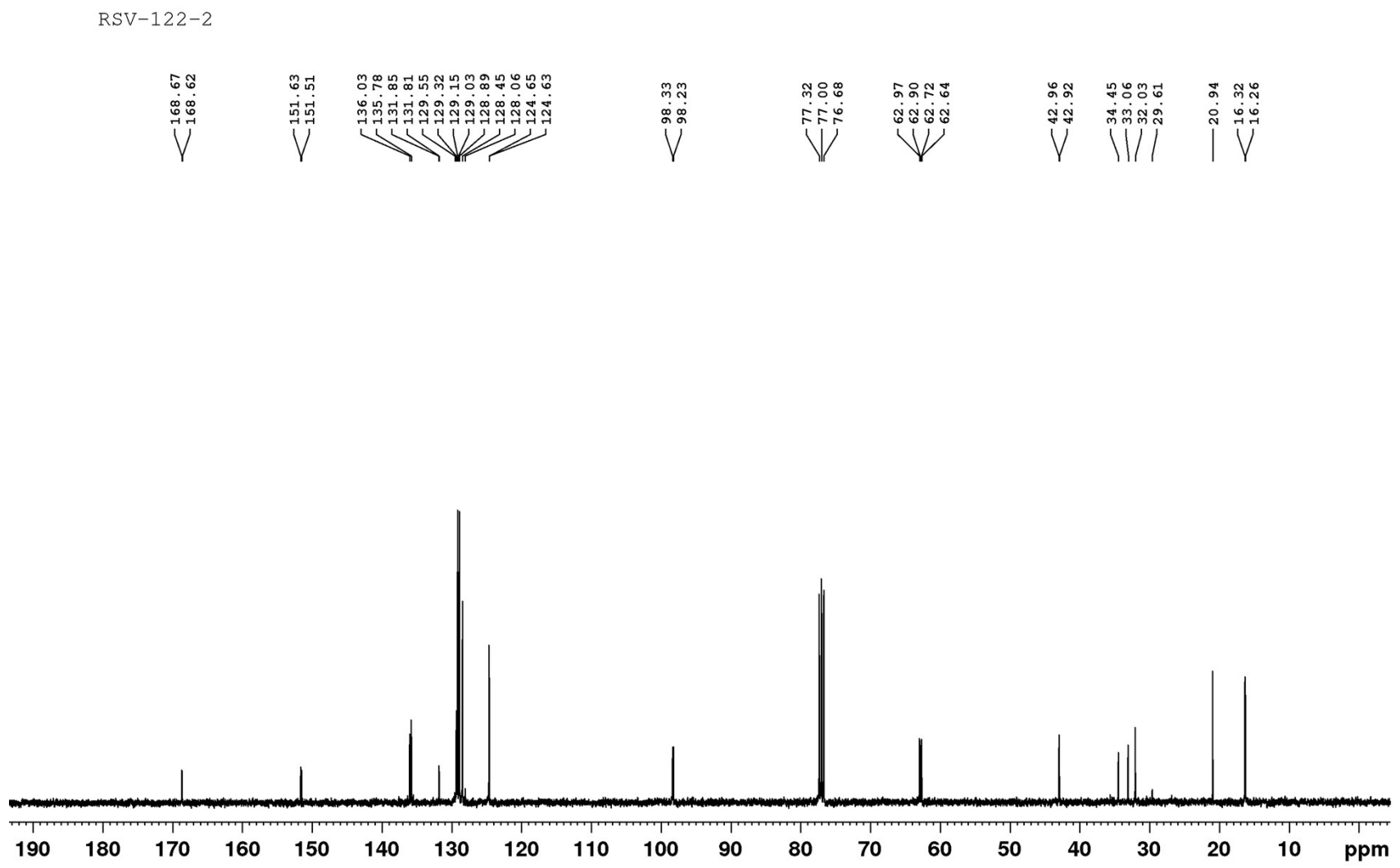
^{31}P NMR spectrum of compound **3d** (150 MHz/$CDCl_3$)



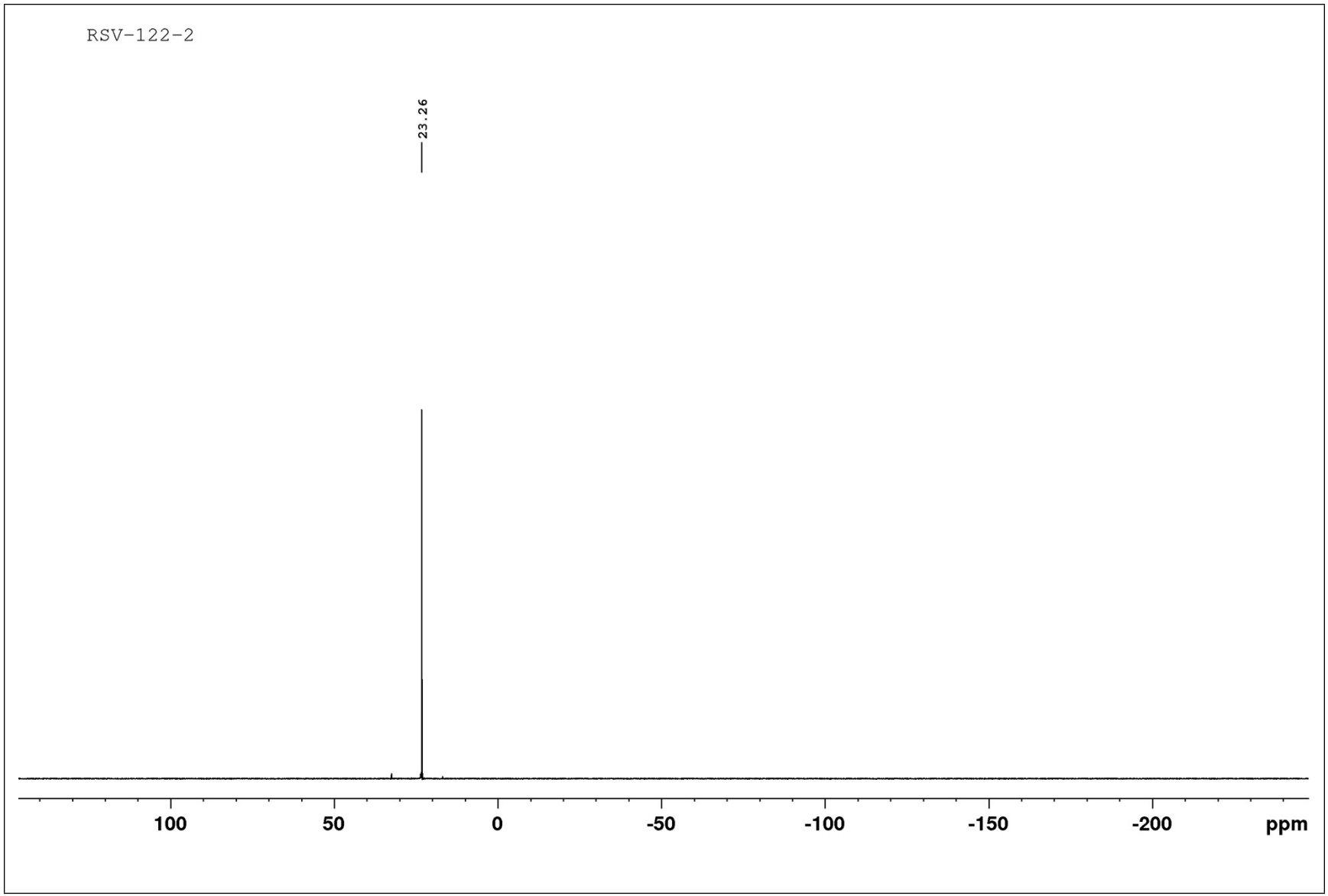
^{1}H NMR spectrum of compound **3e** (400 MHz/$CDCl_3$)



^{13}C NMR spectrum of compound **3e** (100 MHz/$CDCl_3$)



^{31}P NMR spectrum of compound **3e** (150 MHz/$CDCl_3$)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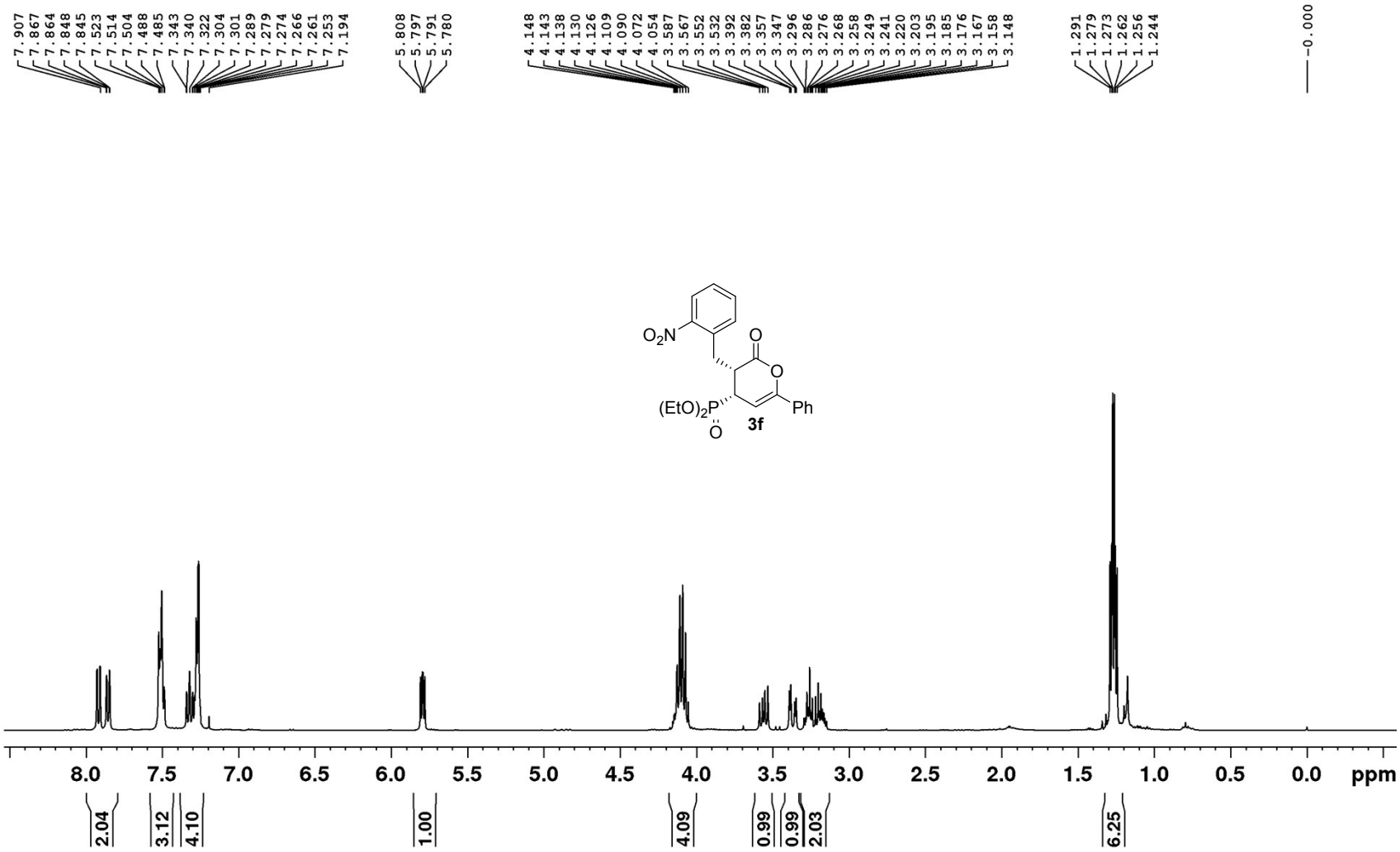
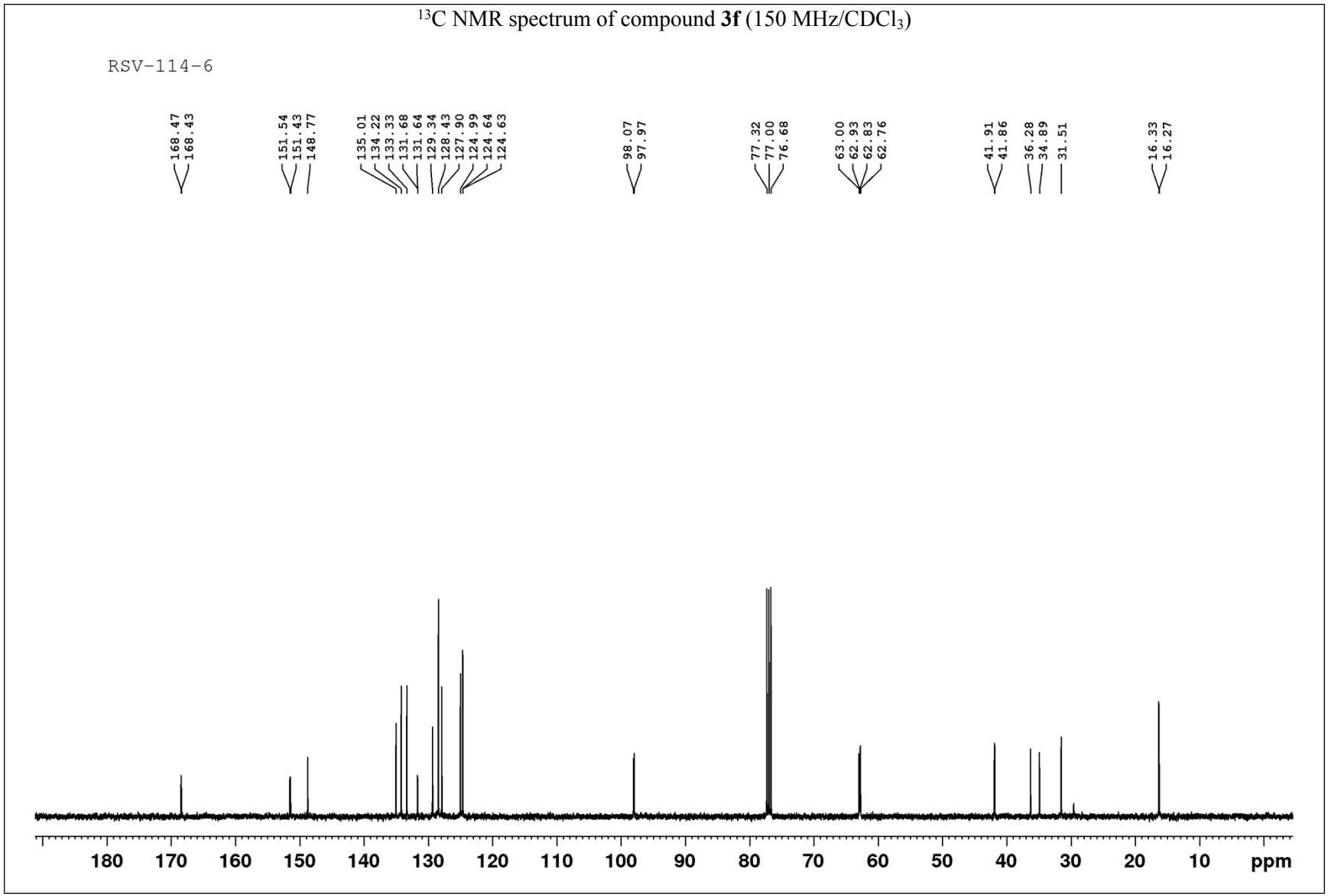
0
-50
-100
-150
-200
ppm

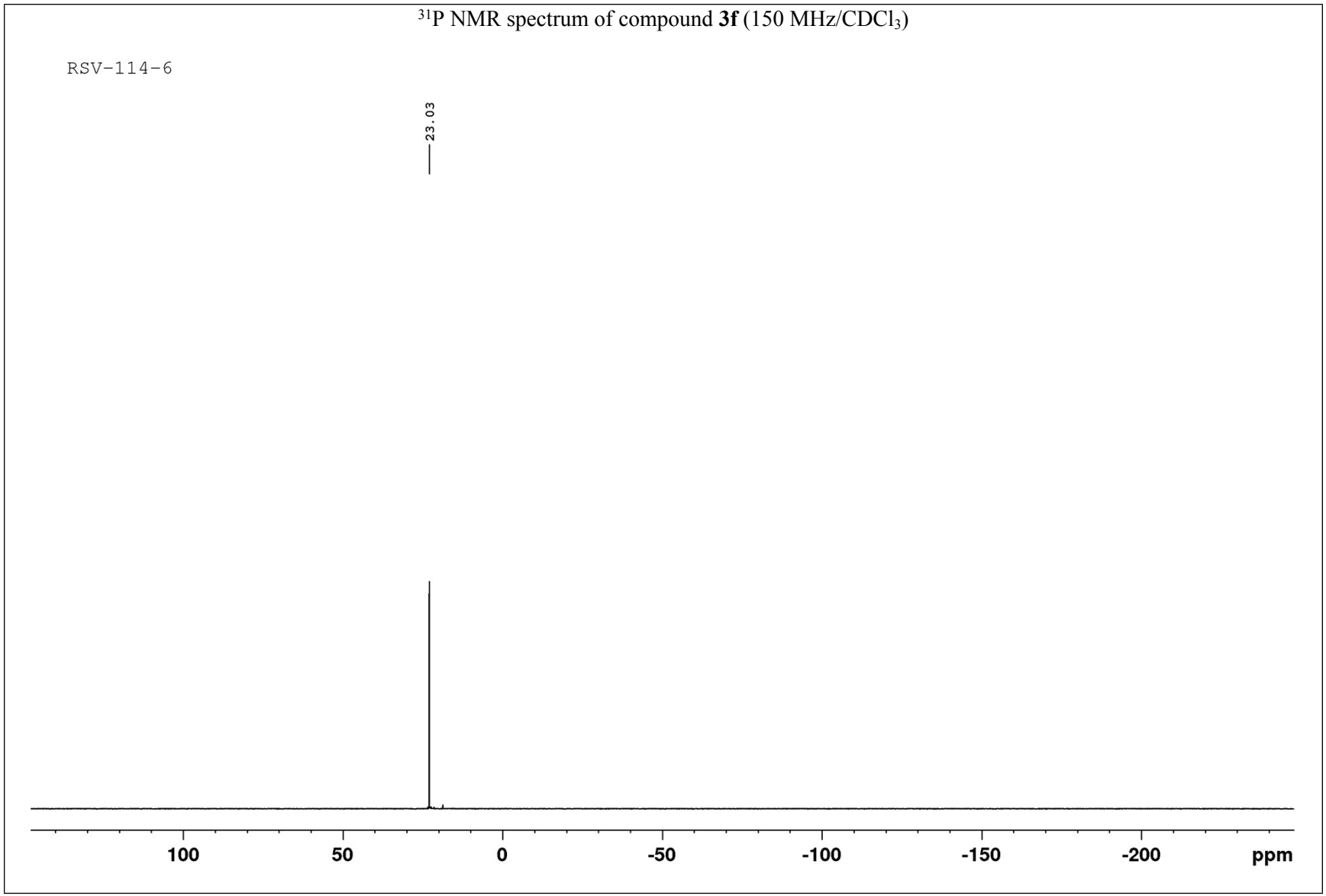
[1]H NMR spectrum of compound **3f** (400 MHz/$CDCl_3$)



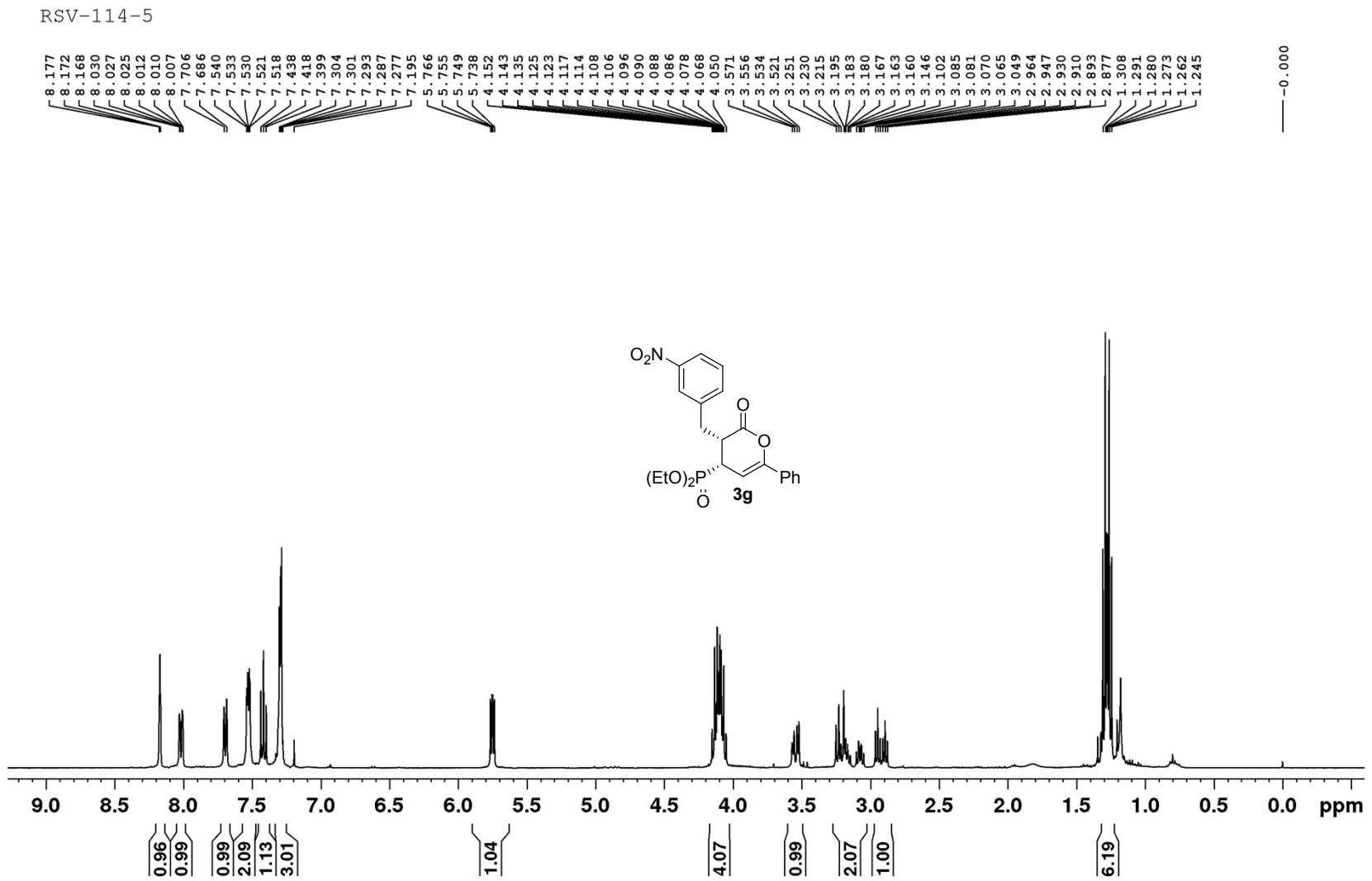
^{13}C NMR spectrum of compound **3f** (150 MHz/$CDCl_3$)



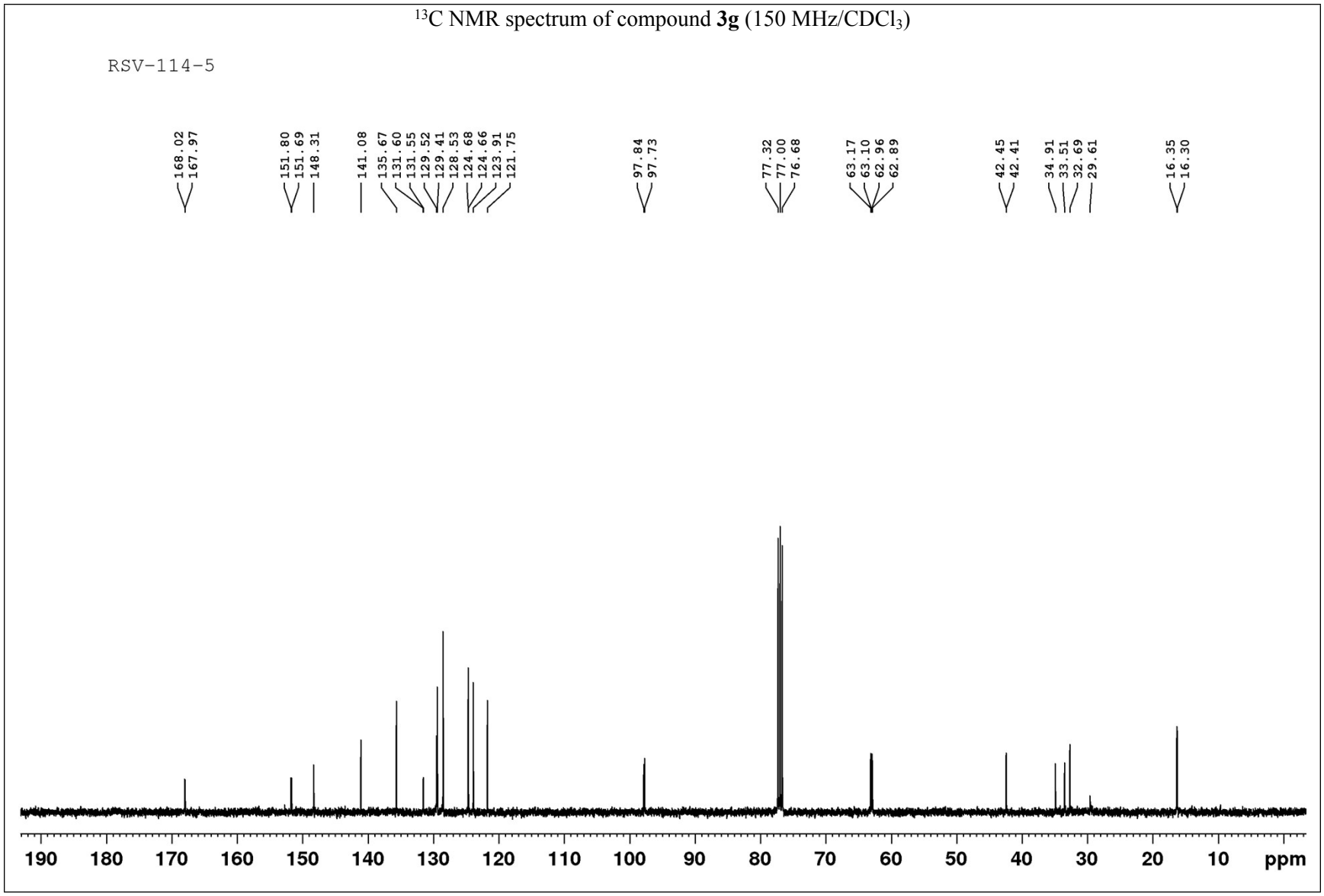
^{31}P NMR spectrum of compound **3f** (150 MHz/$CDCl_3$)



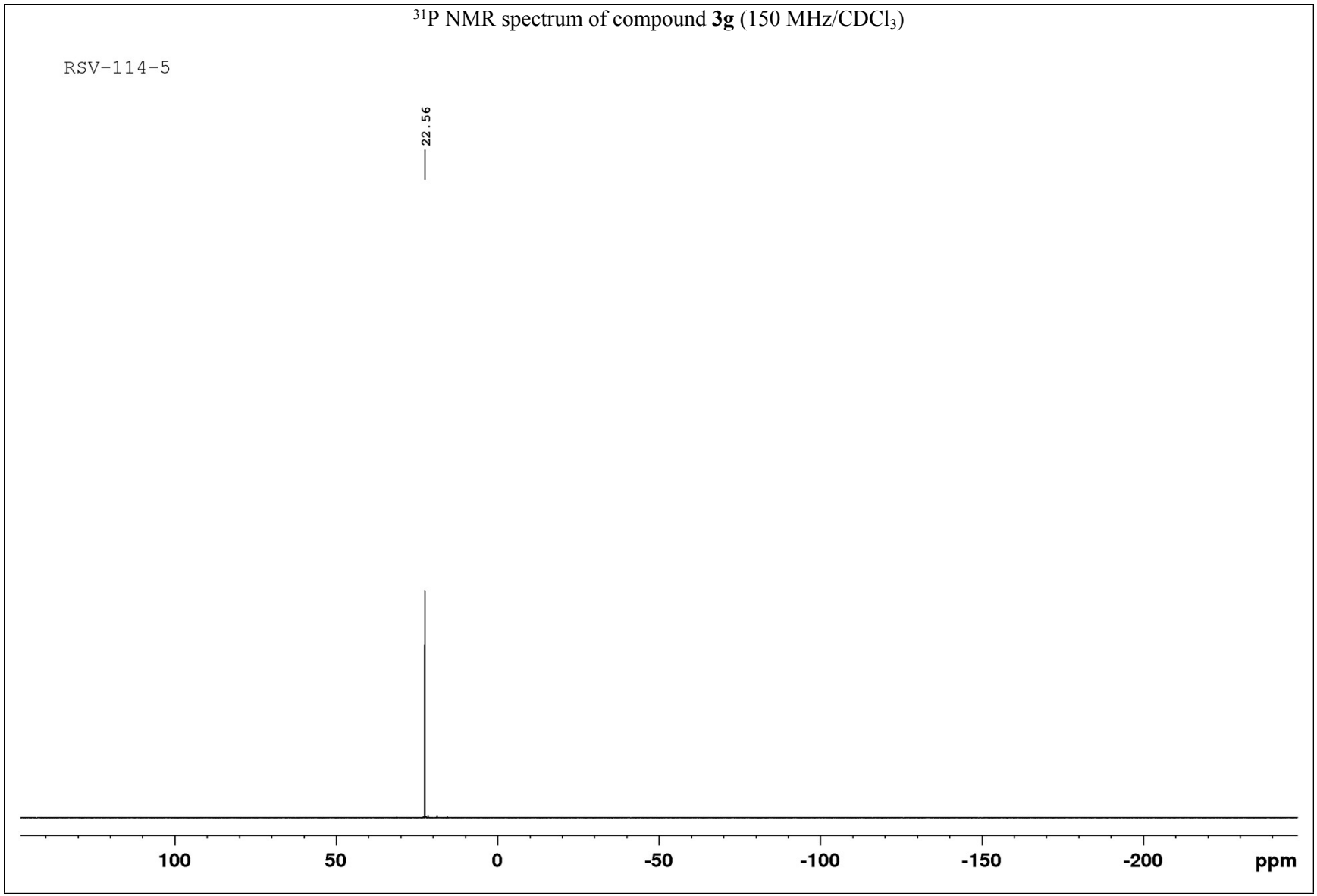
^{1}H NMR spectrum of compound **3g** (400 MHz/$CDCl_3$)



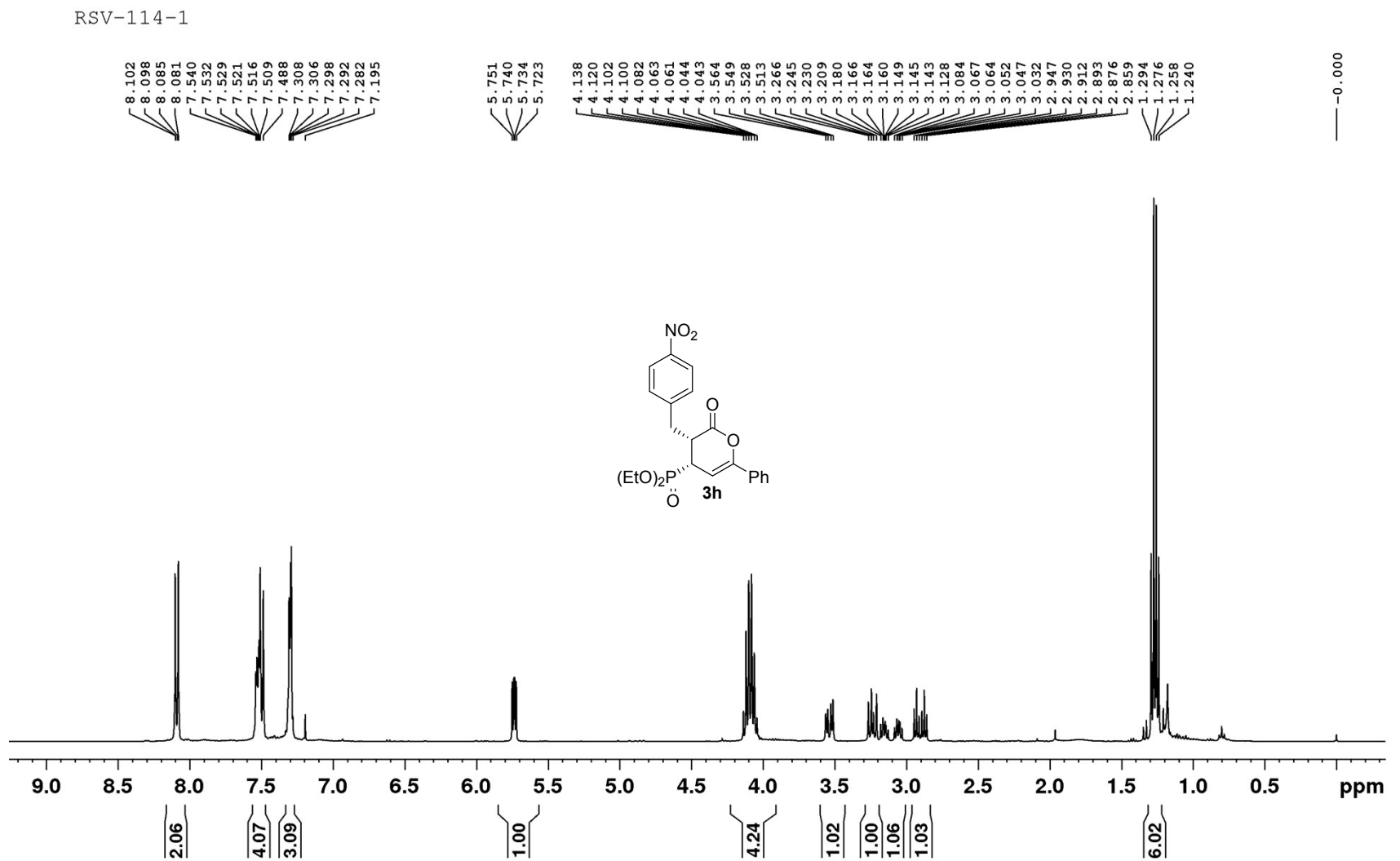
^{13}C NMR spectrum of compound **3g** (150 MHz/$CDCl_3$)



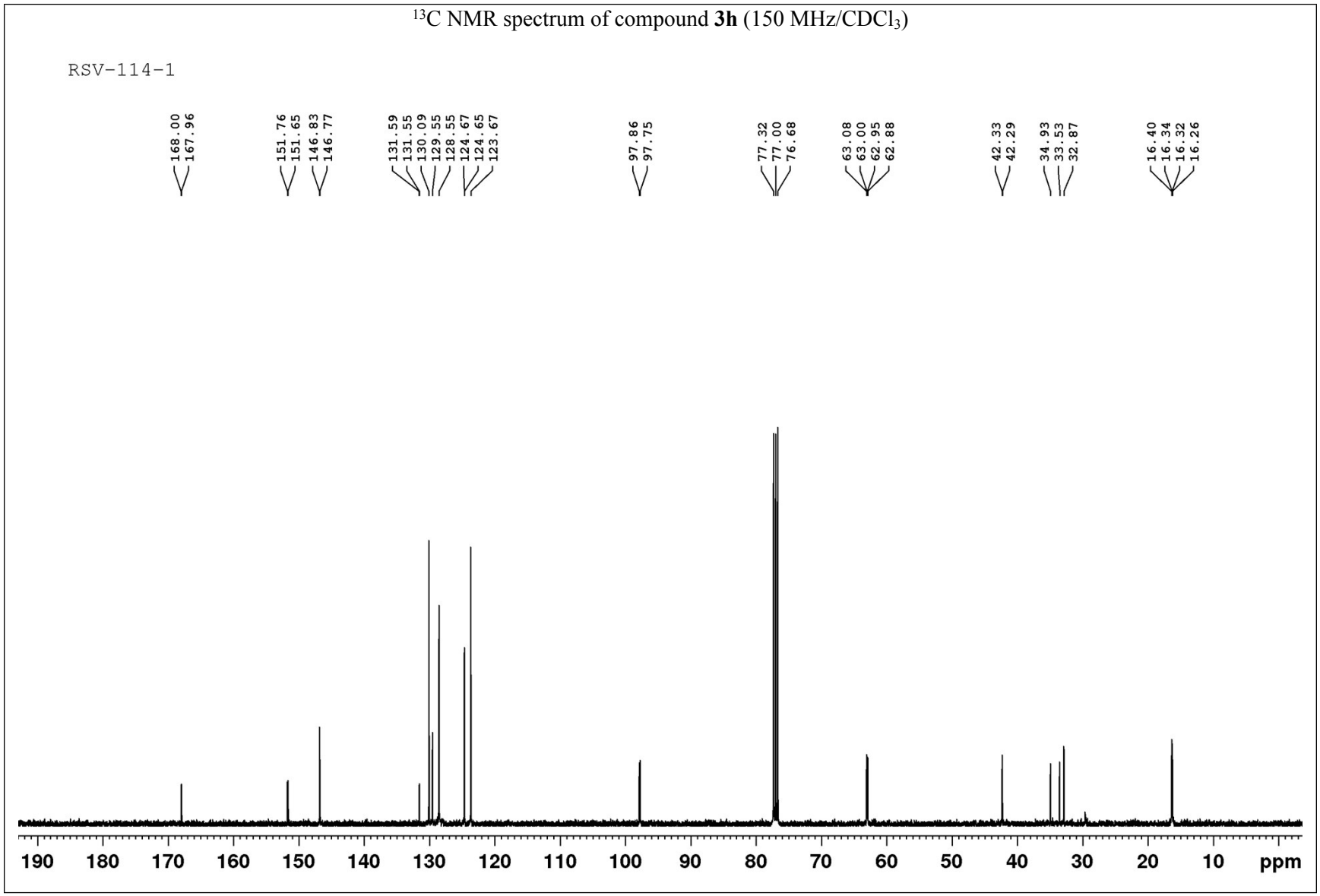
^{31}P NMR spectrum of compound **3g** (150 MHz/$CDCl_3$)



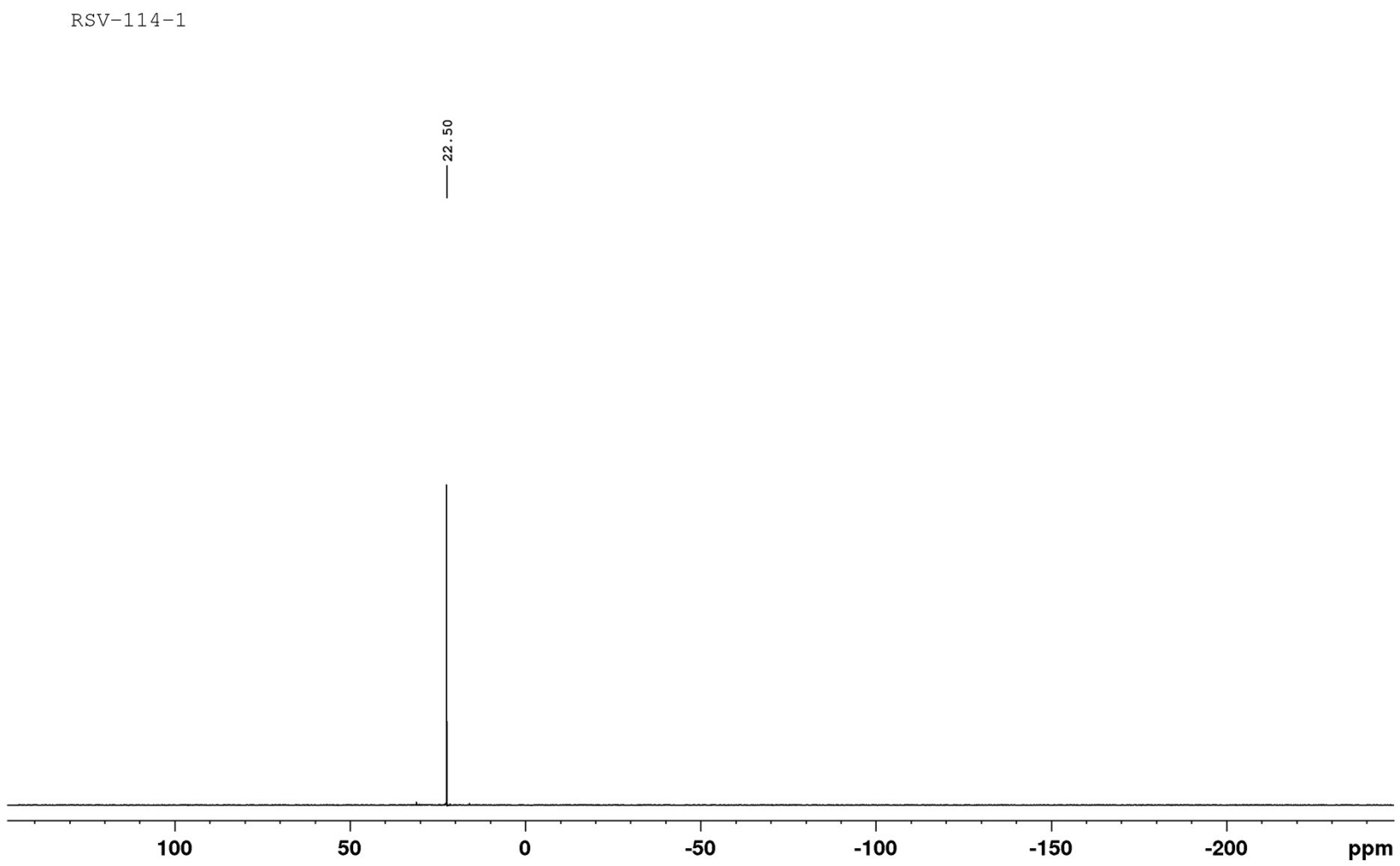
^{1}H NMR spectrum of compound **3h** (400 MHz/$CDCl_3$)



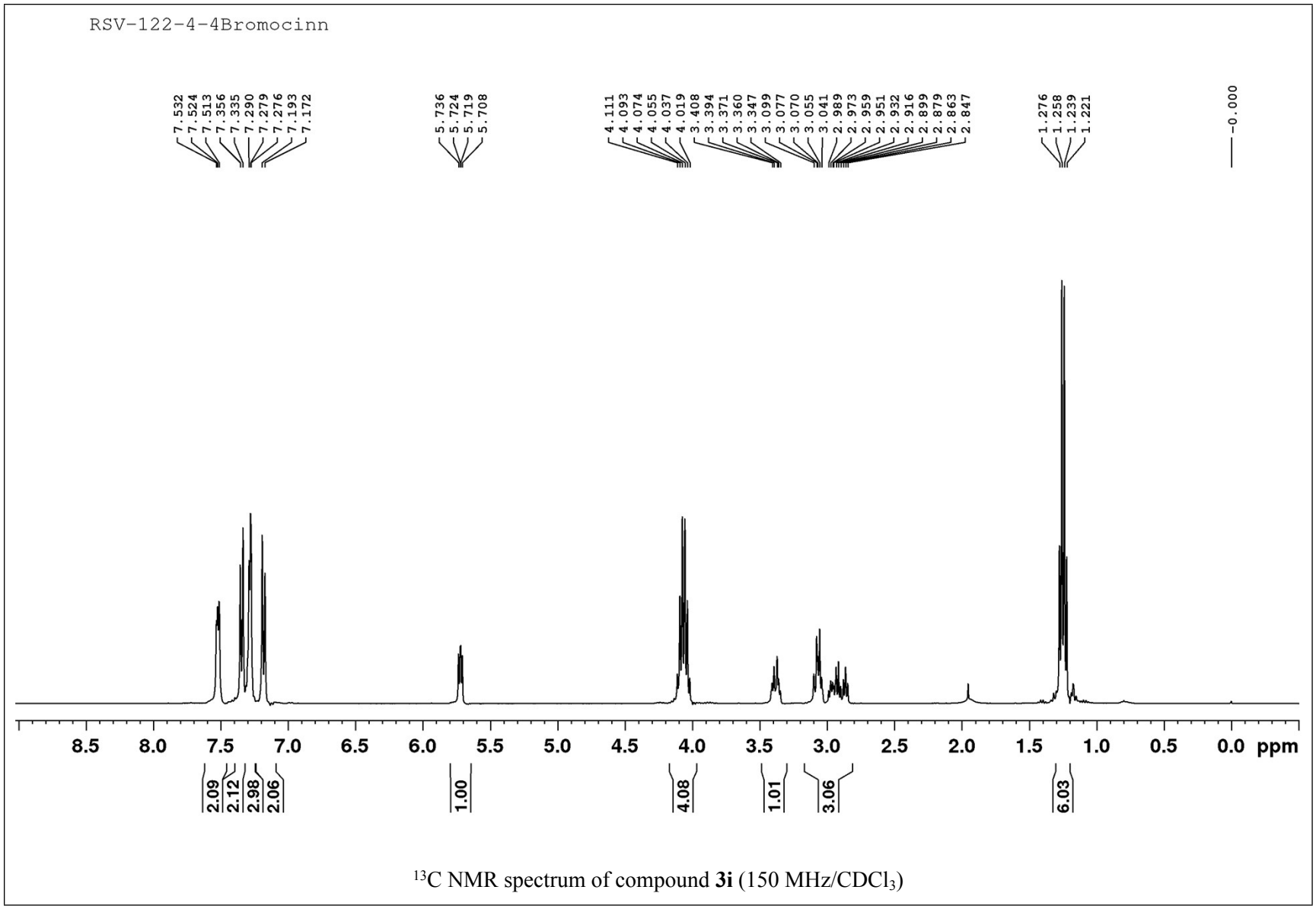
^{13}C NMR spectrum of compound **3h** (150 MHz/$CDCl_3$)



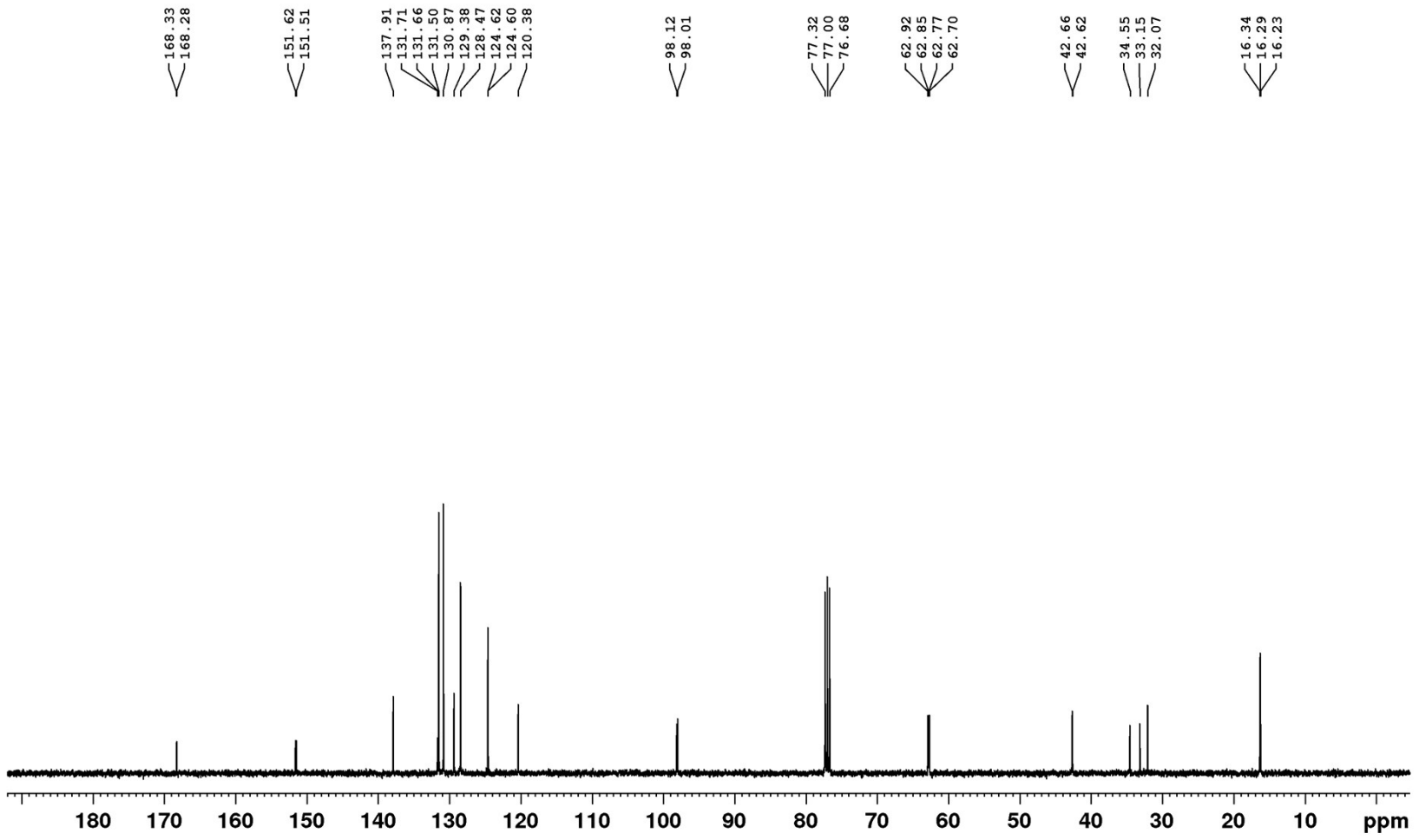
^{31}P NMR spectrum of compound **3h** (150 MHz/$CDCl_3$)



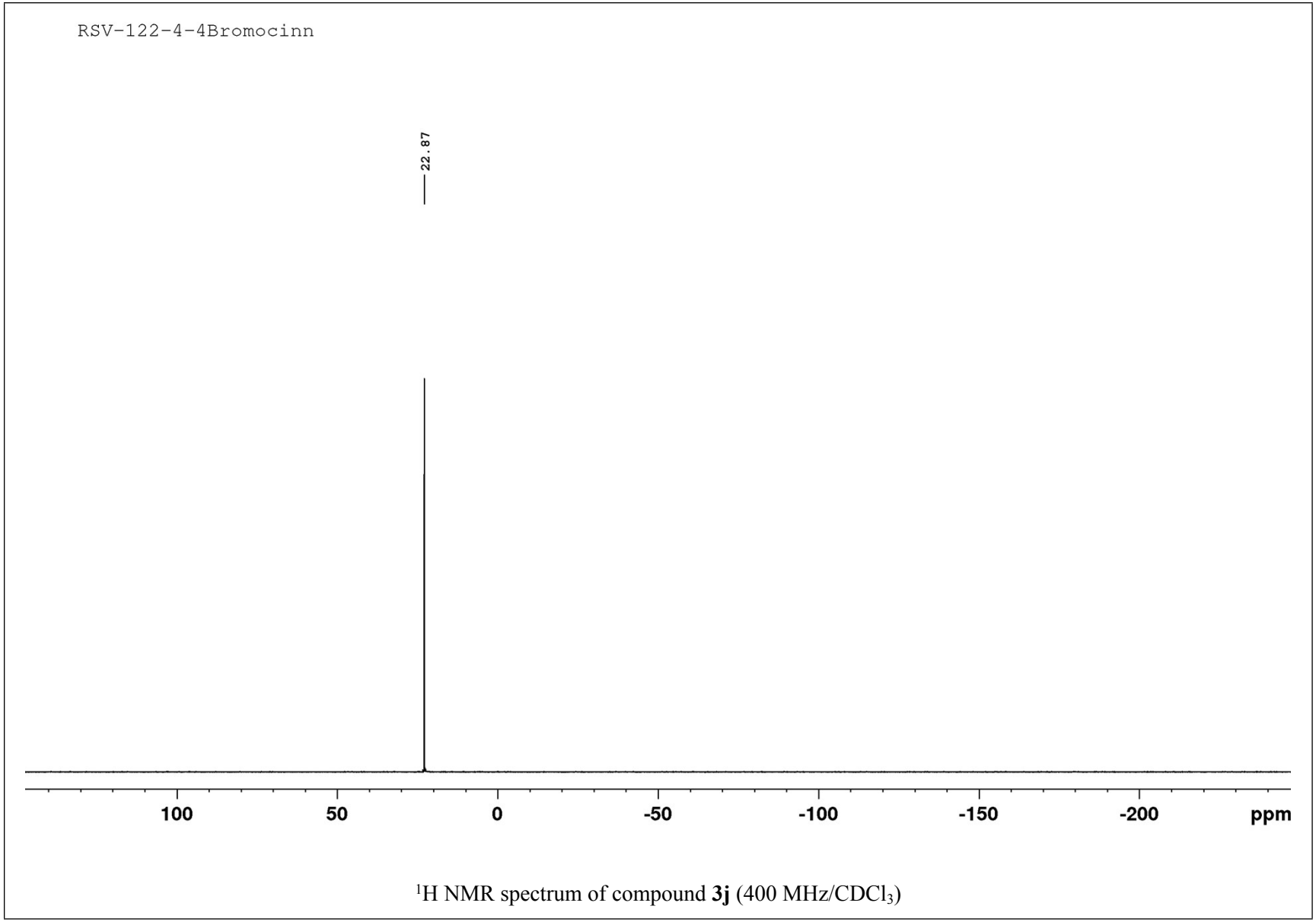
^{1}H NMR spectrum of compound **3i** (400 MHz/$CDCl_3$)



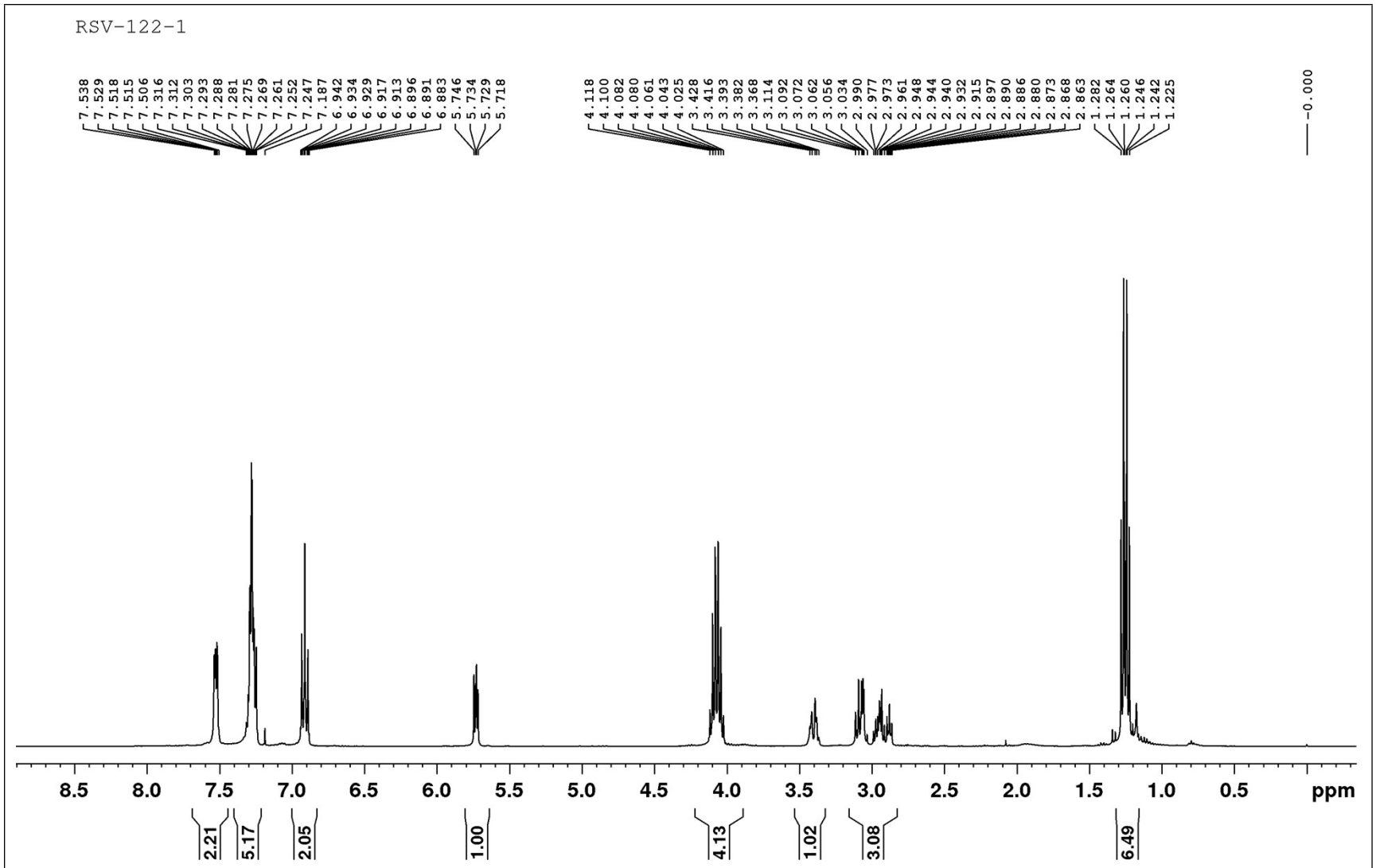
^{13}C NMR spectrum of compound **3i** (150 MHz/$CDCl_3$)



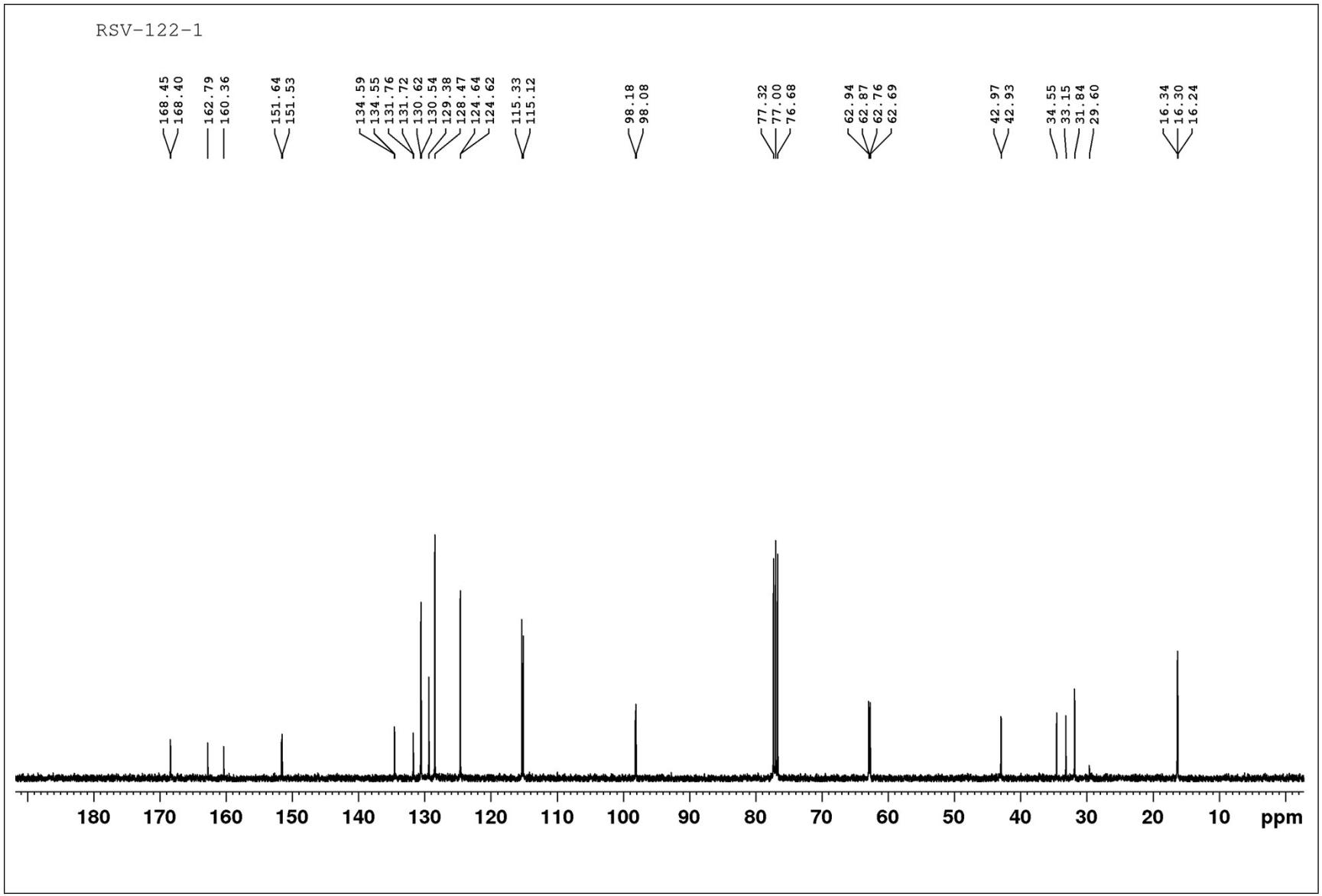
^{31}P NMR spectrum of compound **3i** (150 MHz/$CDCl_3$)



^{1}H NMR spectrum of compound **3j** (400 MHz/$CDCl_3$)



^{13}C NMR spectrum of compound **3j** (150 MHz/$CDCl_3$)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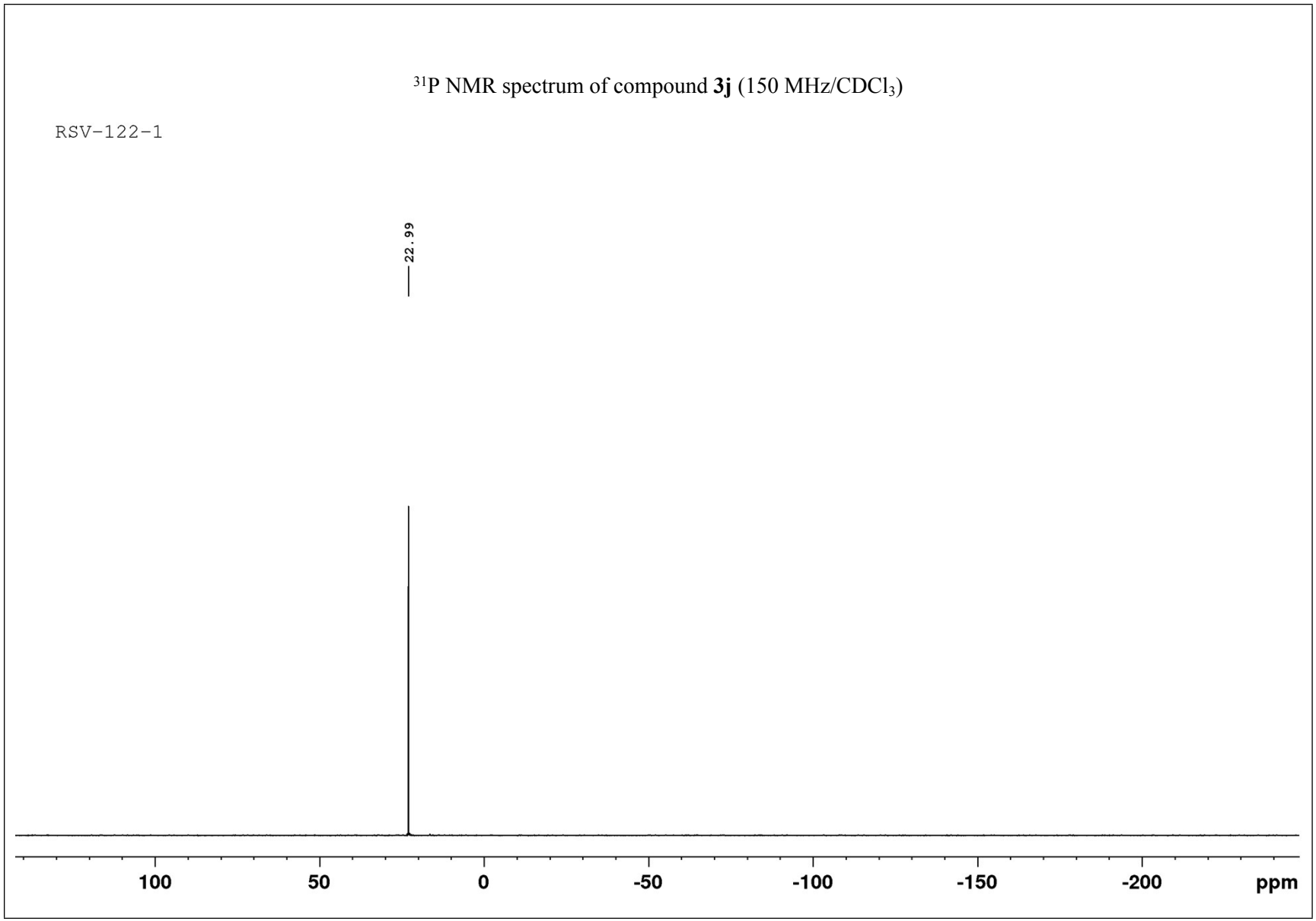
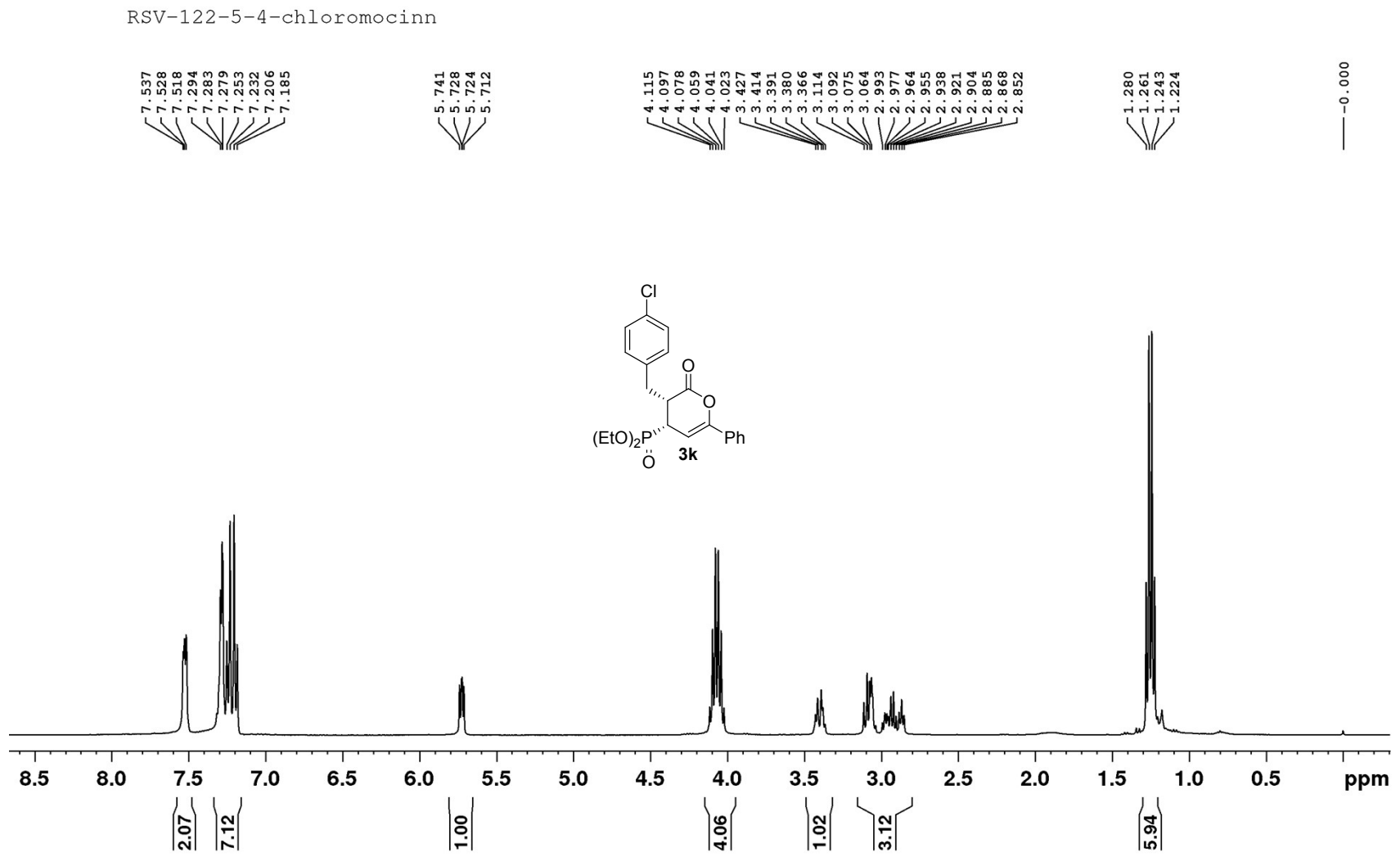
130.54
129.38
128.47
124.64
124.62
115.33
115.12
98.18
98.08
77.32
77.00
76.68
62.94
62.87
62.76
62.69
42.97
42.93
34.55
33.15
31.84
29.60
16.34
16.30
16.24
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

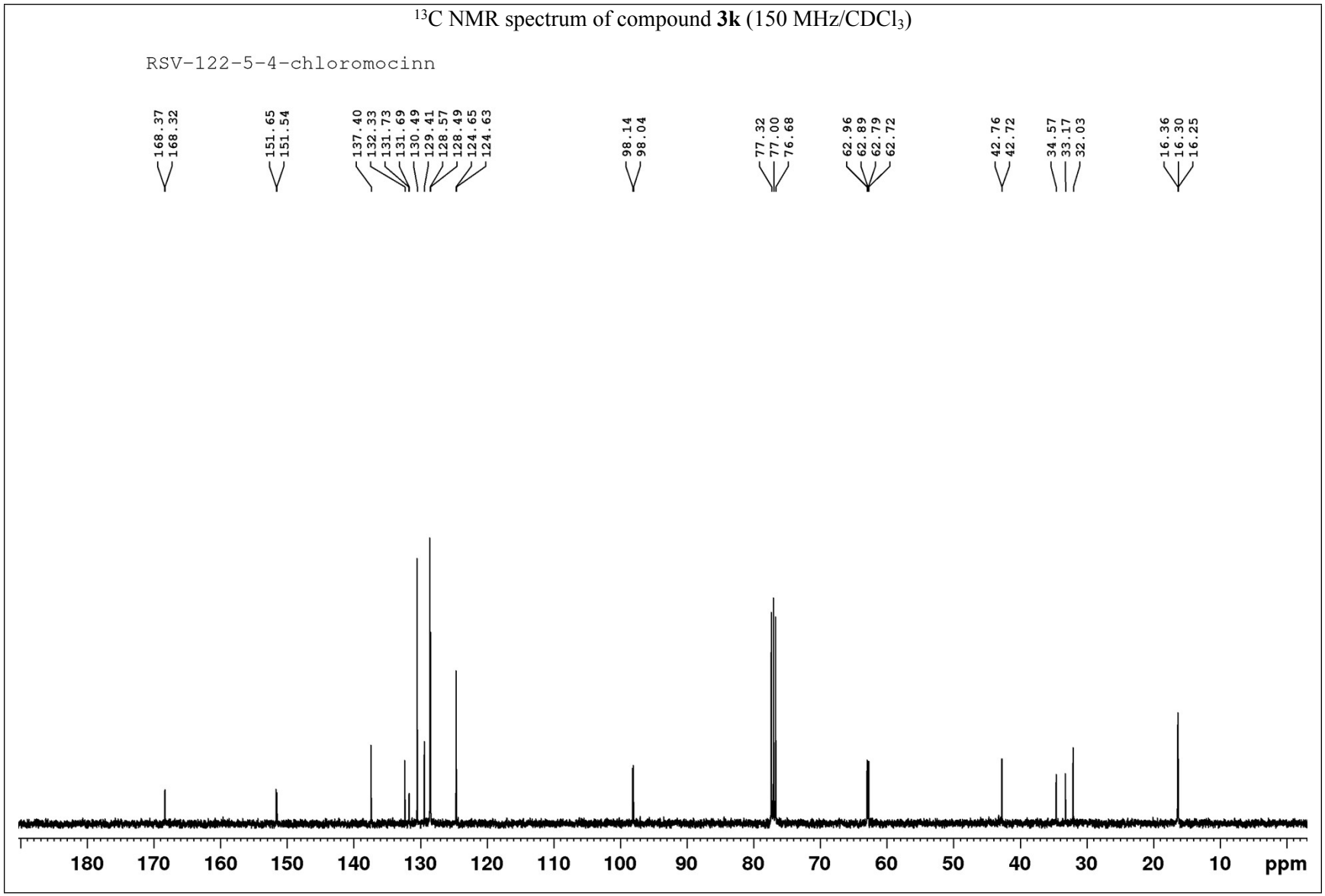
^{31}P NMR spectrum of compound **3j** (150 MHz/$CDCl_3$)



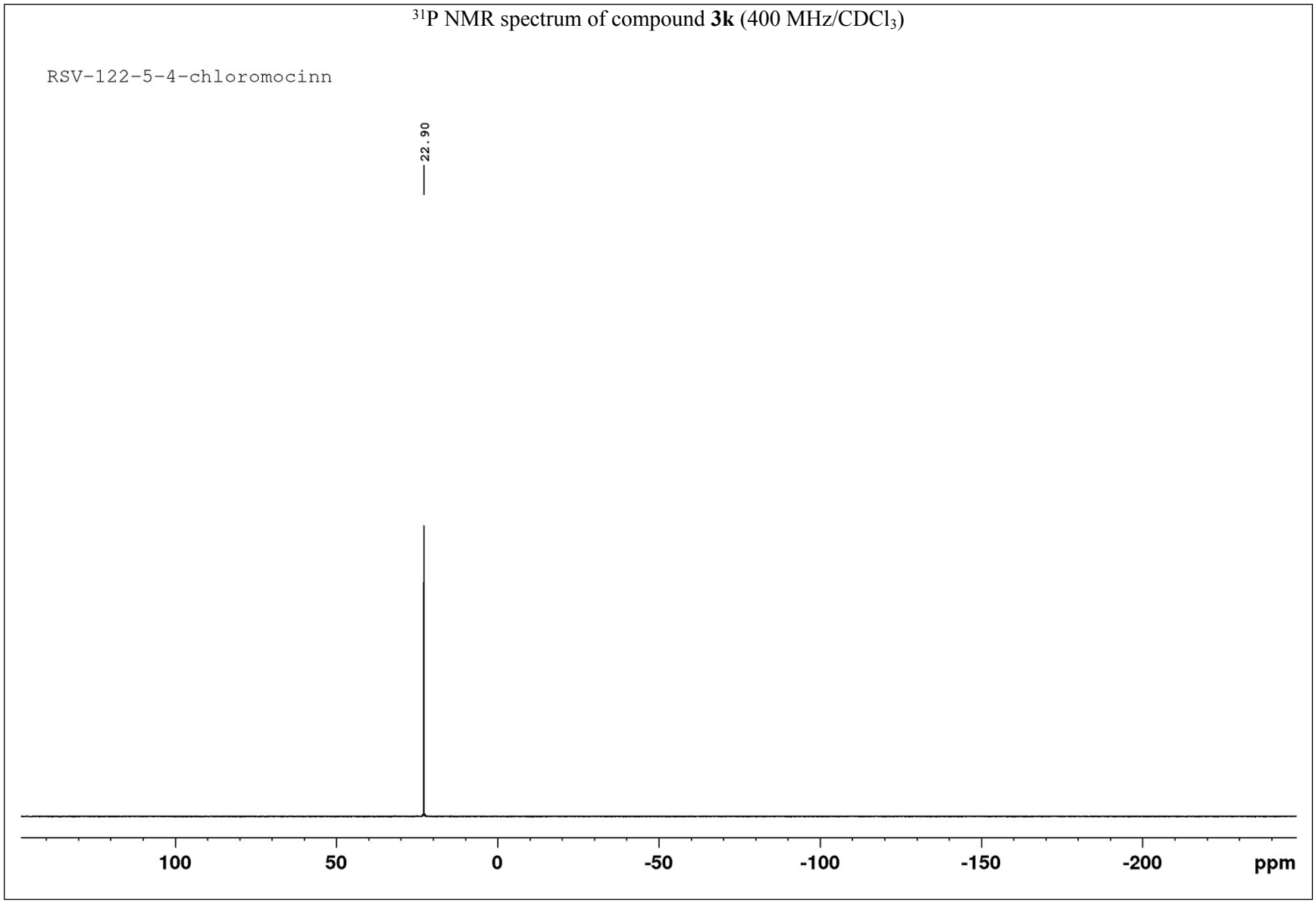
^{1}H NMR spectrum of compound **3k** (400 MHz/$CDCl_3$)



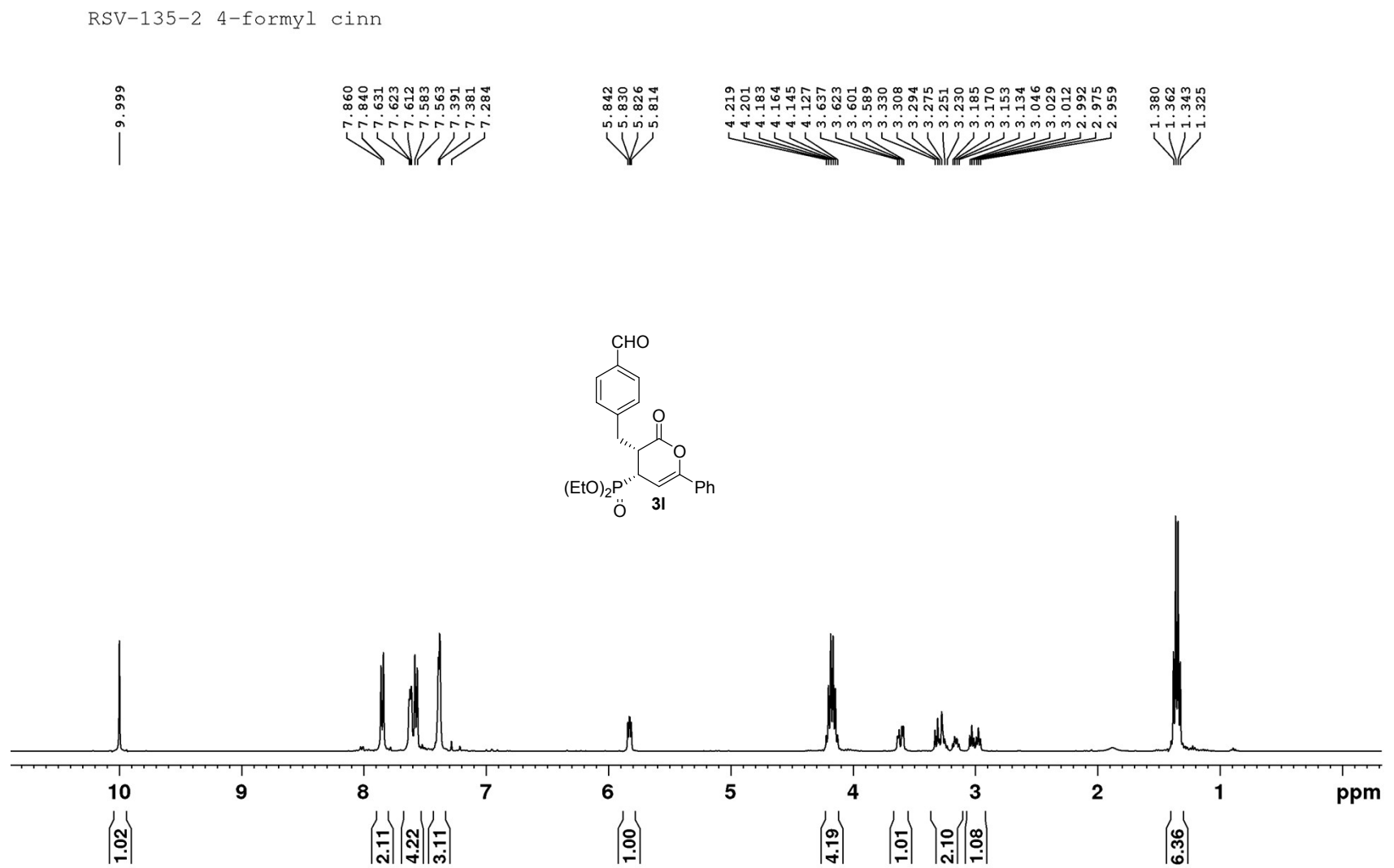
^{13}C NMR spectrum of compound **3k** (150 MHz/$CDCl_3$)



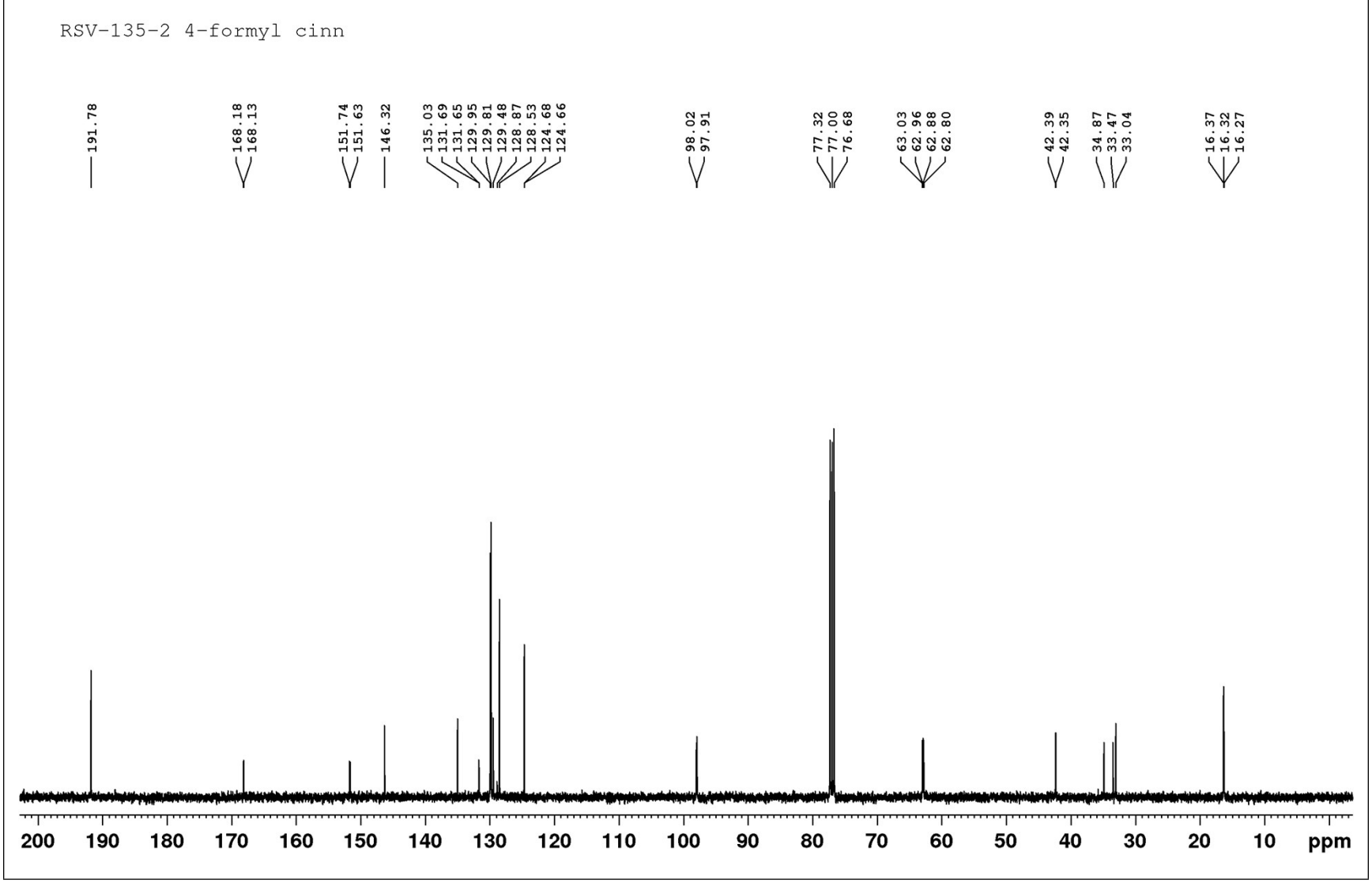
^{31}P NMR spectrum of compound **3k** (400 MHz/$CDCl_3$)



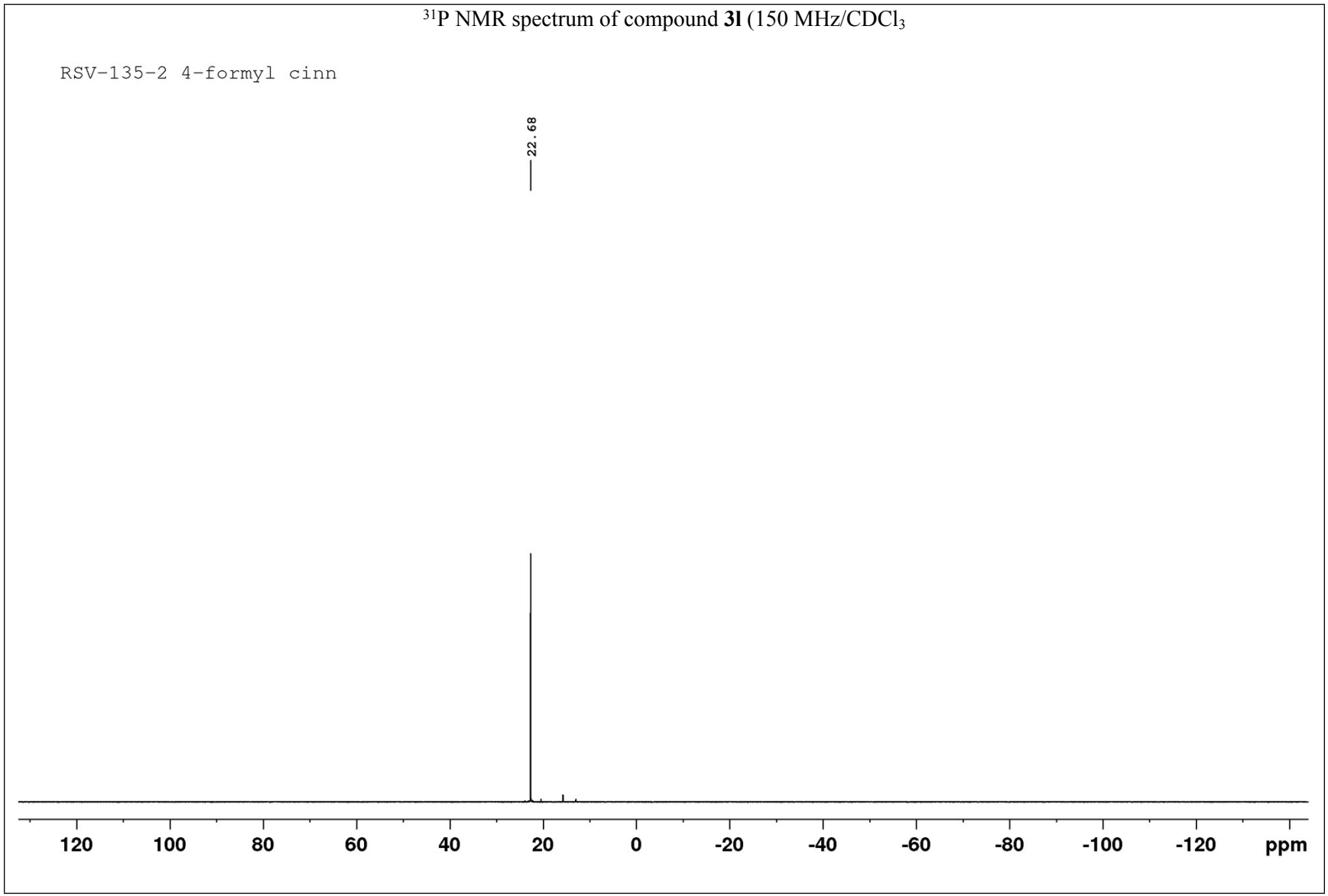
^{1}H NMR spectrum of compound **3l** (600 MHz/$CDCl_3$)



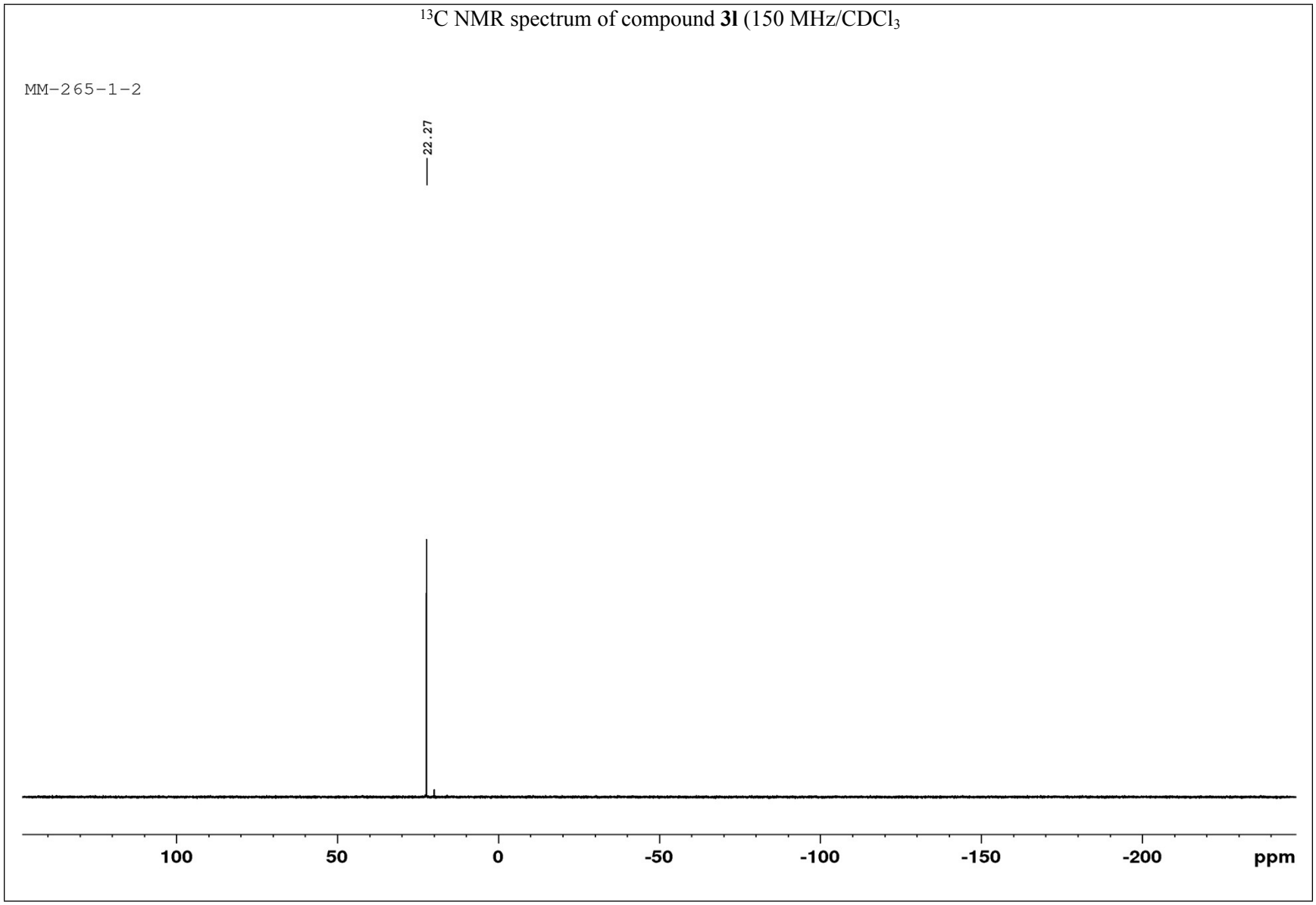
^{13}C NMR spectrum of compound **3l** (150 MHz/$CDCl_3$



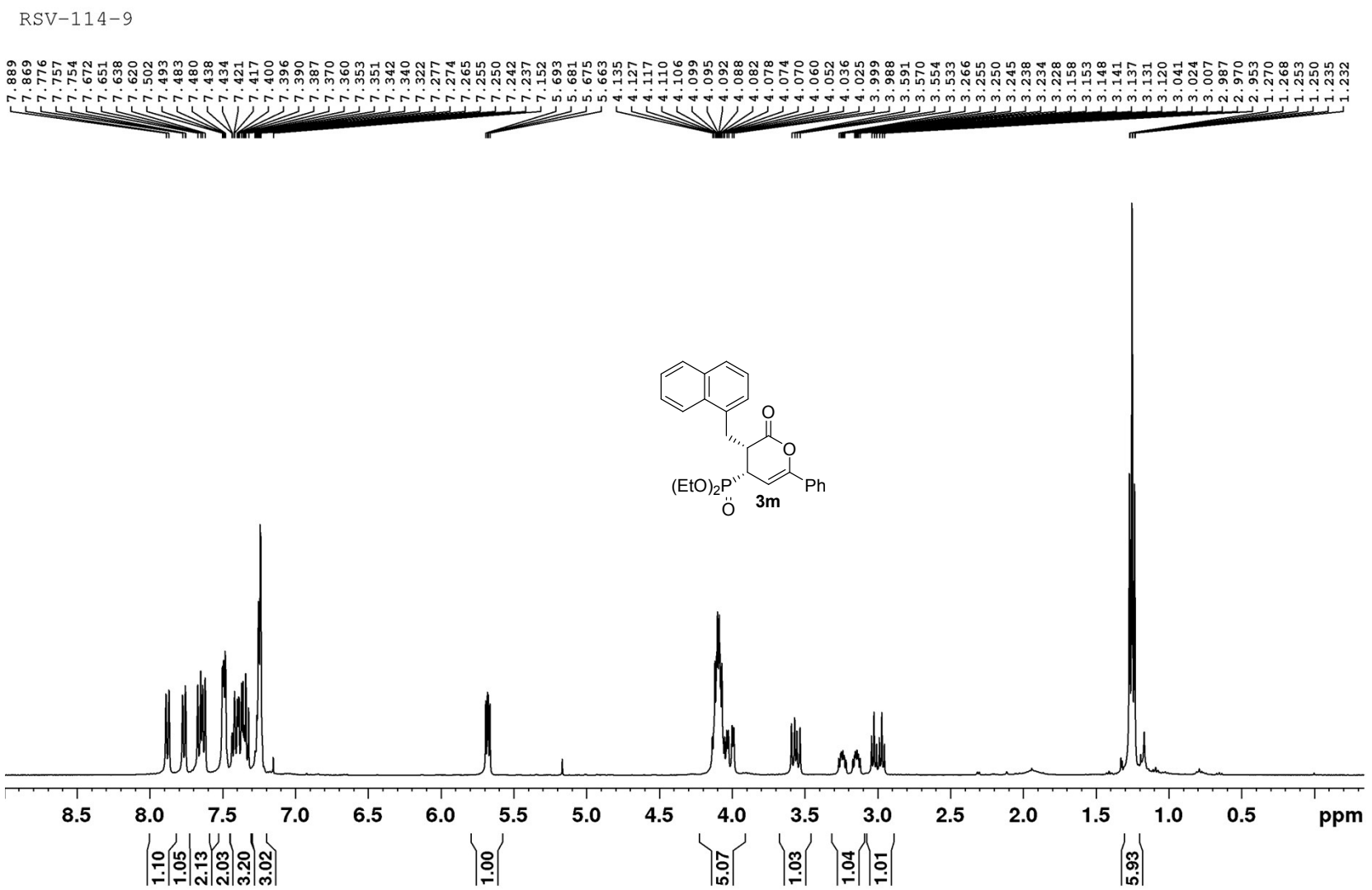
^{31}P NMR spectrum of compound **3l** (150 MHz/$CDCl_3$



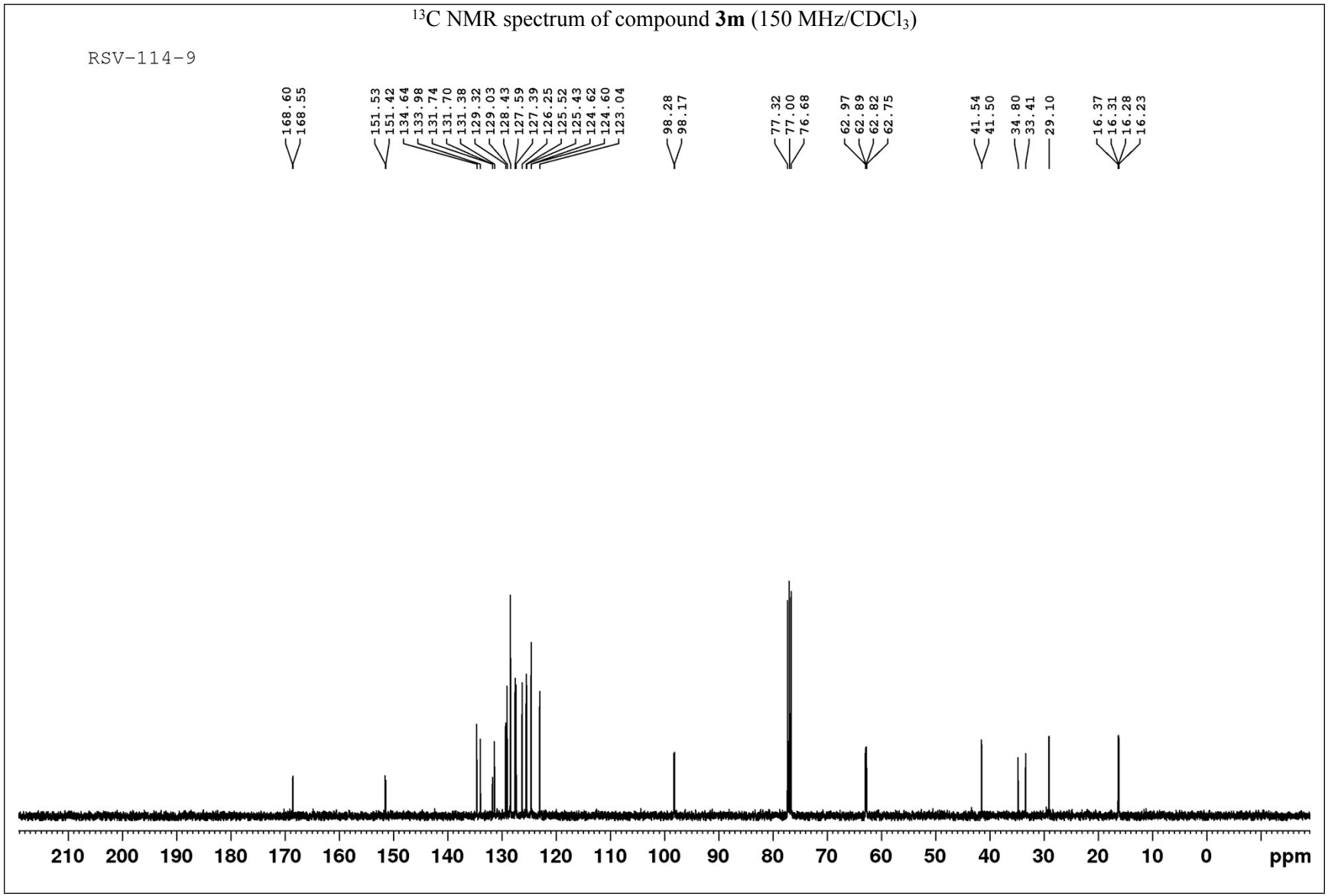
^{13}C NMR spectrum of compound **3l** (150 MHz/$CDCl_3$



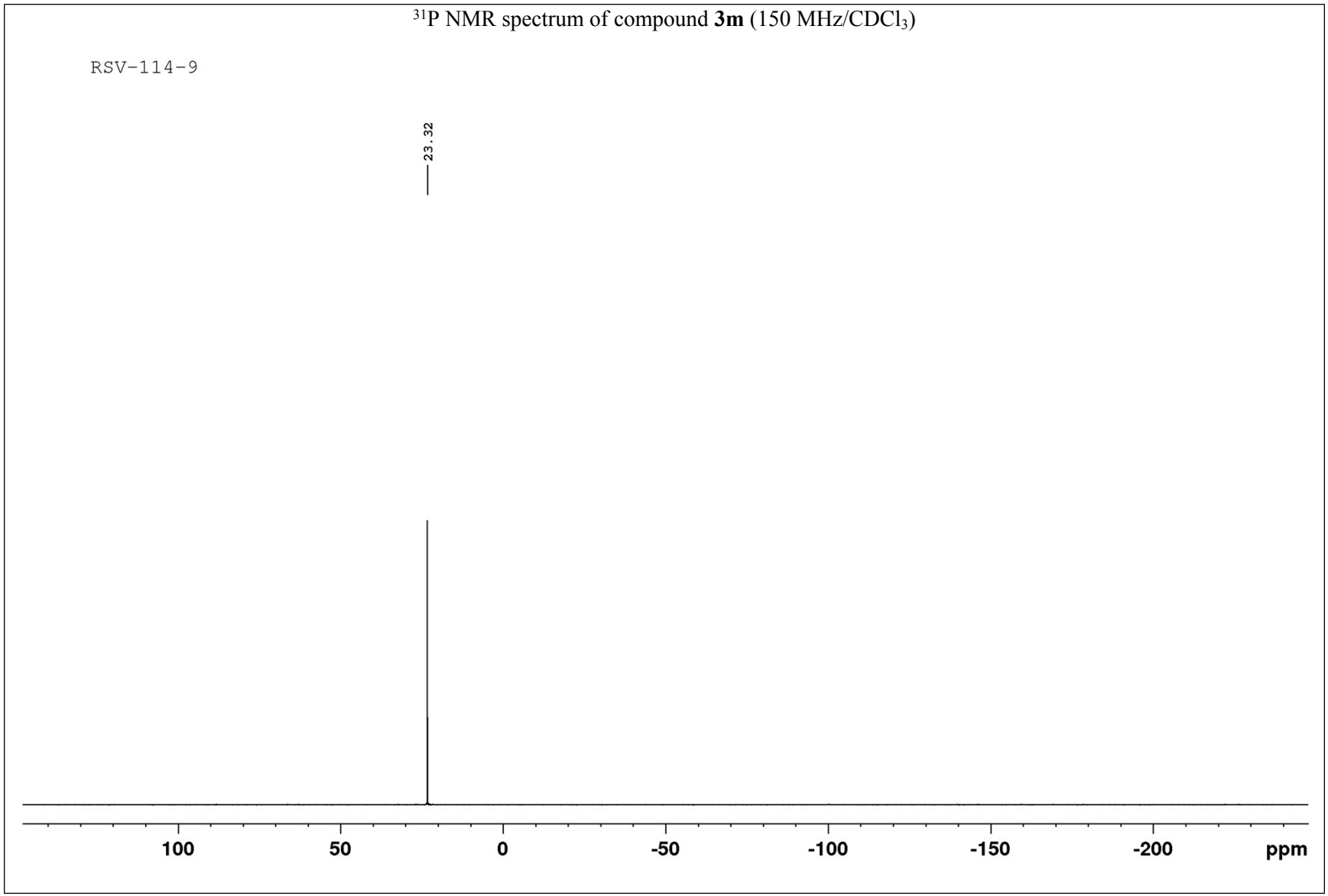
^{1}H NMR spectrum of compound **3m** (400 MHz/$CDCl_3$)



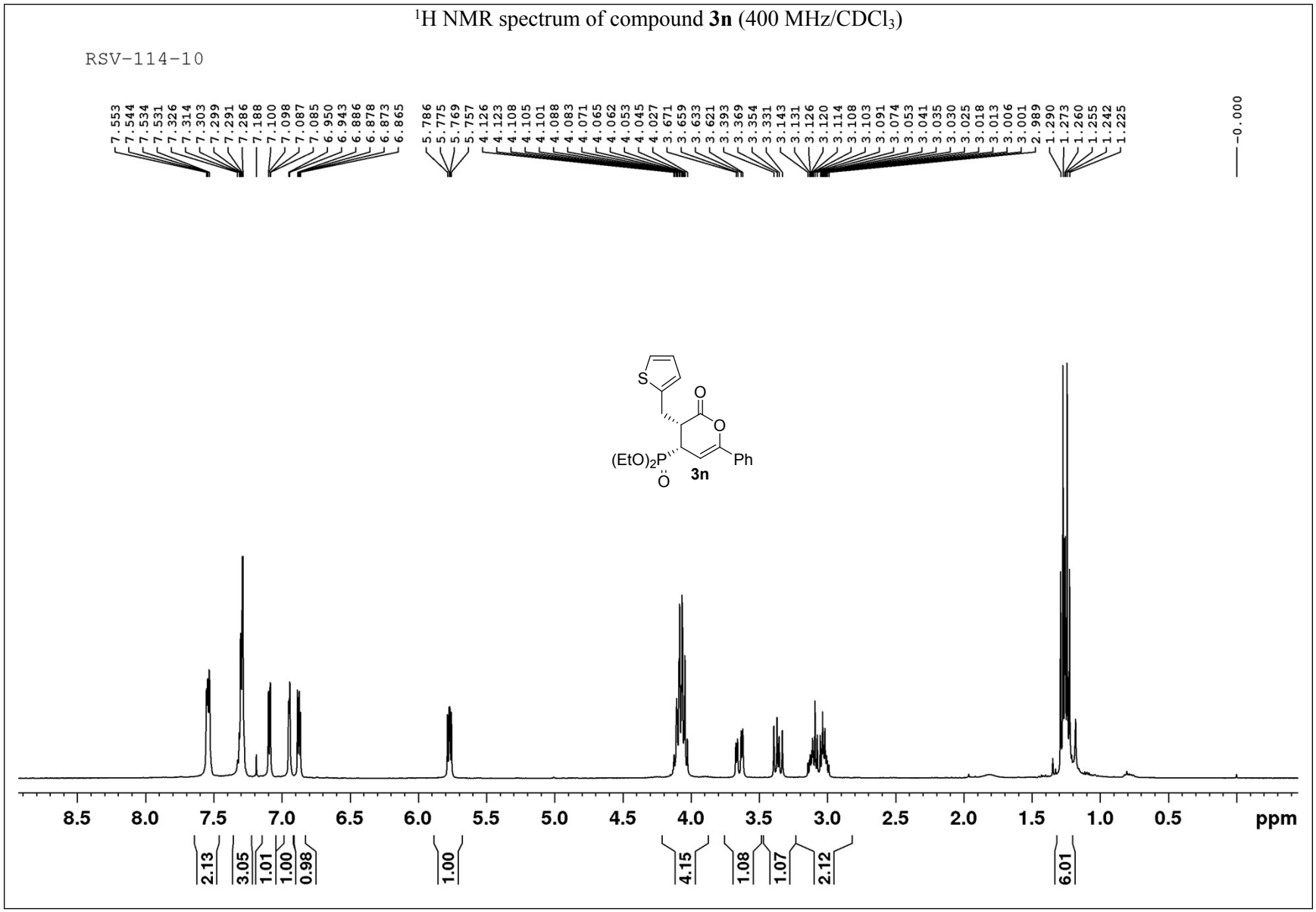
^{13}C NMR spectrum of compound **3m** (150 MHz/$CDCl_3$)



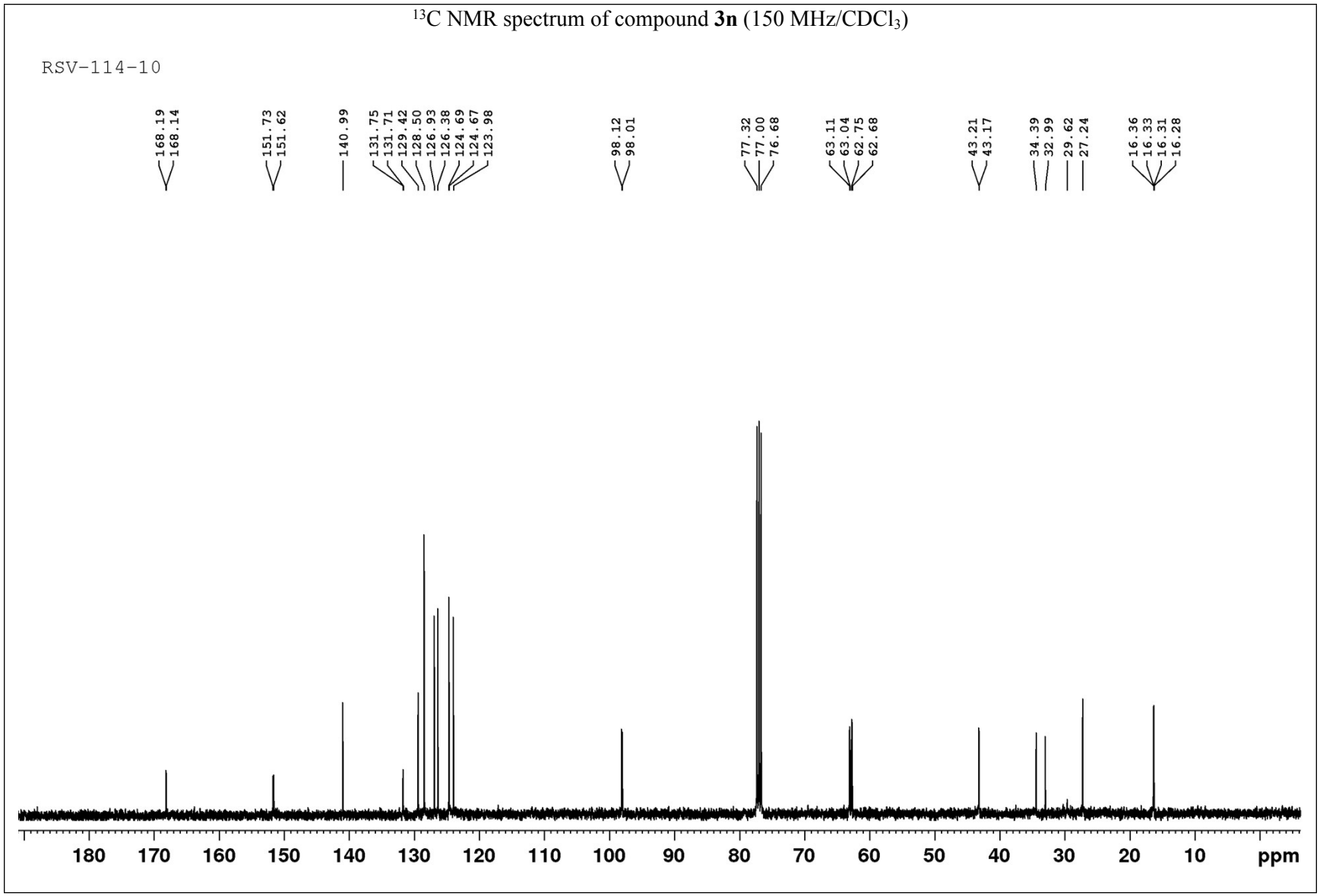
^{31}P NMR spectrum of compound **3m** (150 MHz/$CDCl_3$)



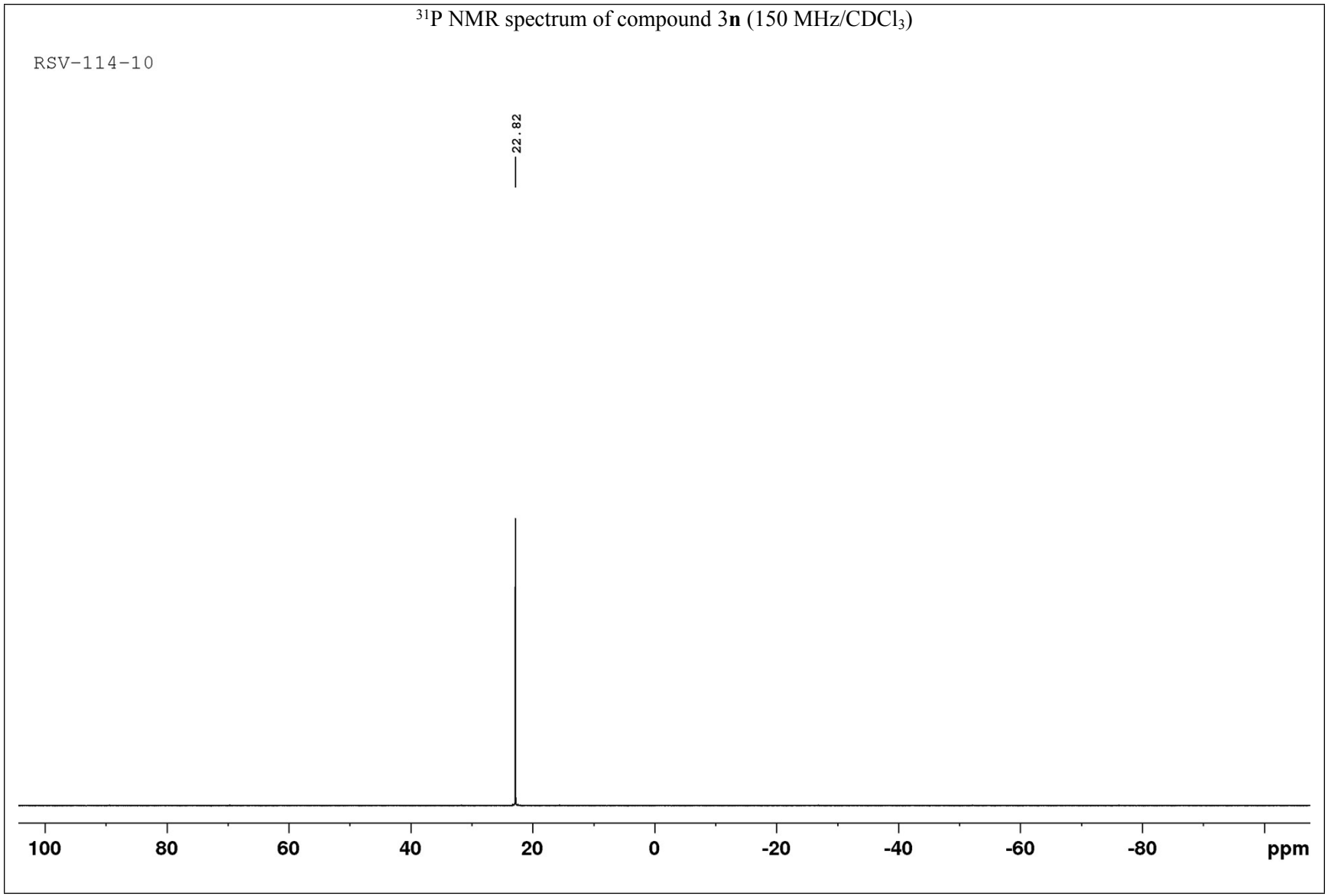
^{1}H NMR spectrum of compound **3n** (400 MHz/$CDCl_3$)



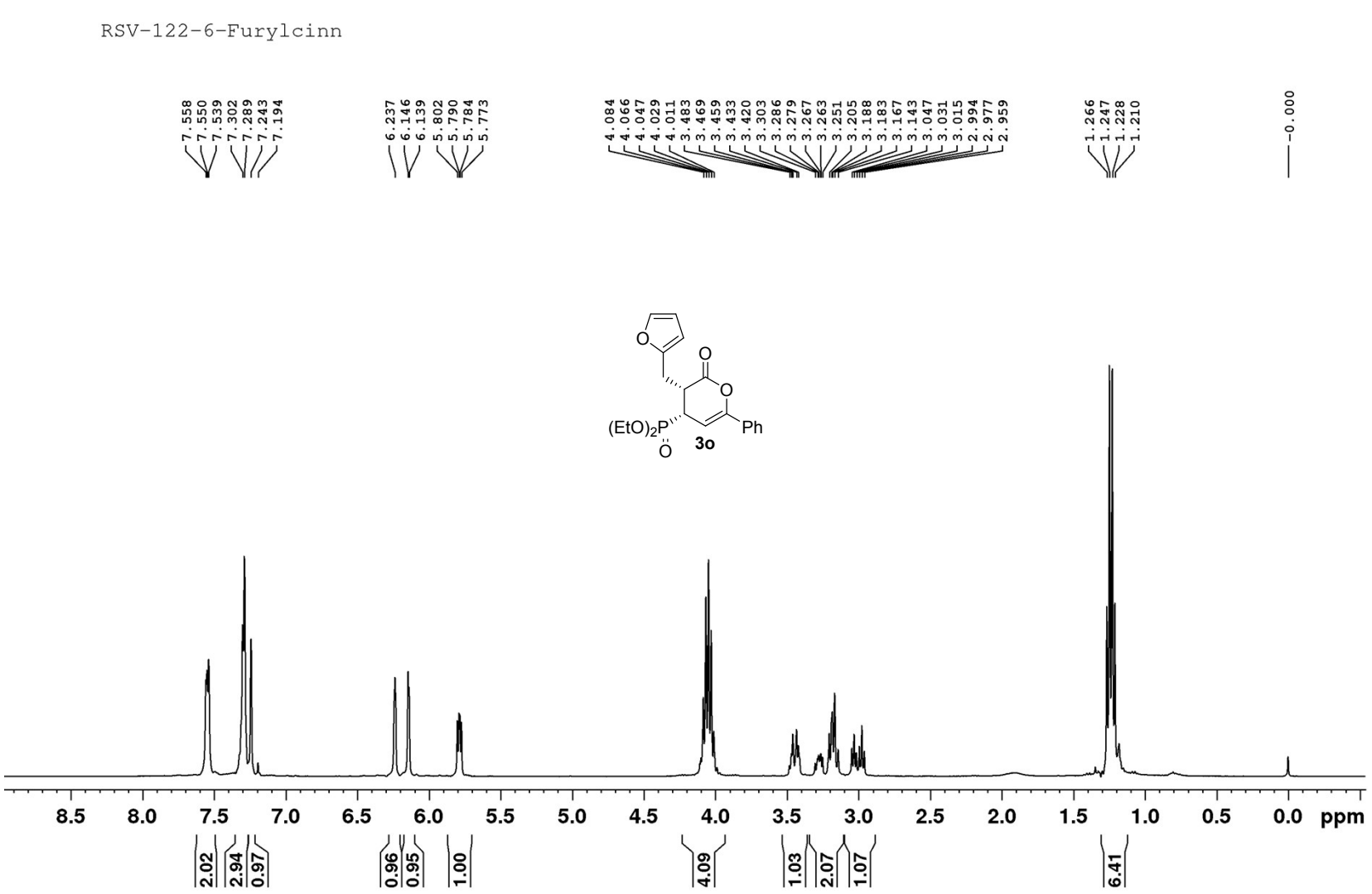
^{13}C NMR spectrum of compound **3n** (150 MHz/$CDCl_3$)



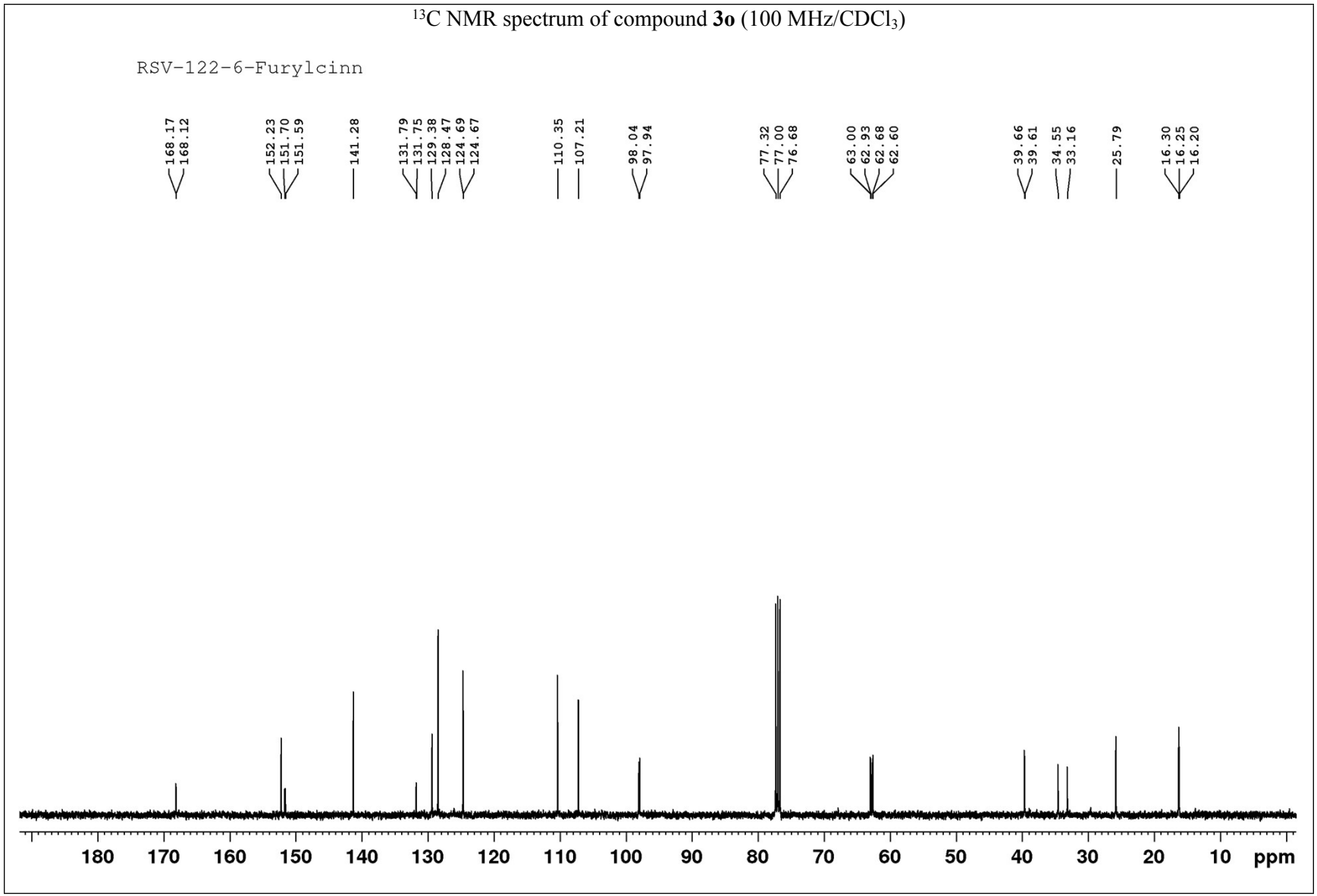
^{31}P NMR spectrum of compound 3**n** (150 MHz/$CDCl_3$)



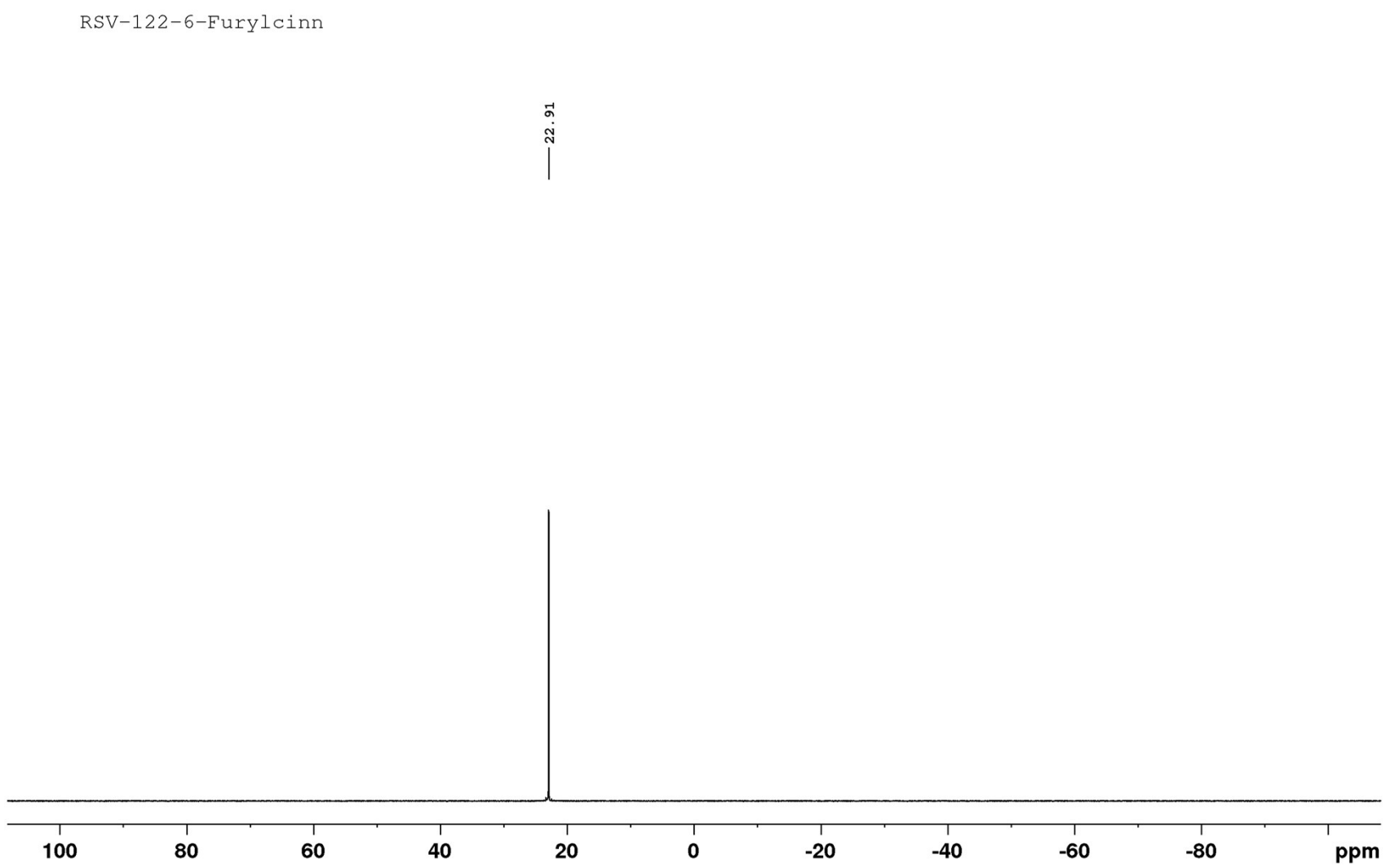
^{1}H NMR spectrum of compound **3o** (400 MHz/$CDCl_3$)



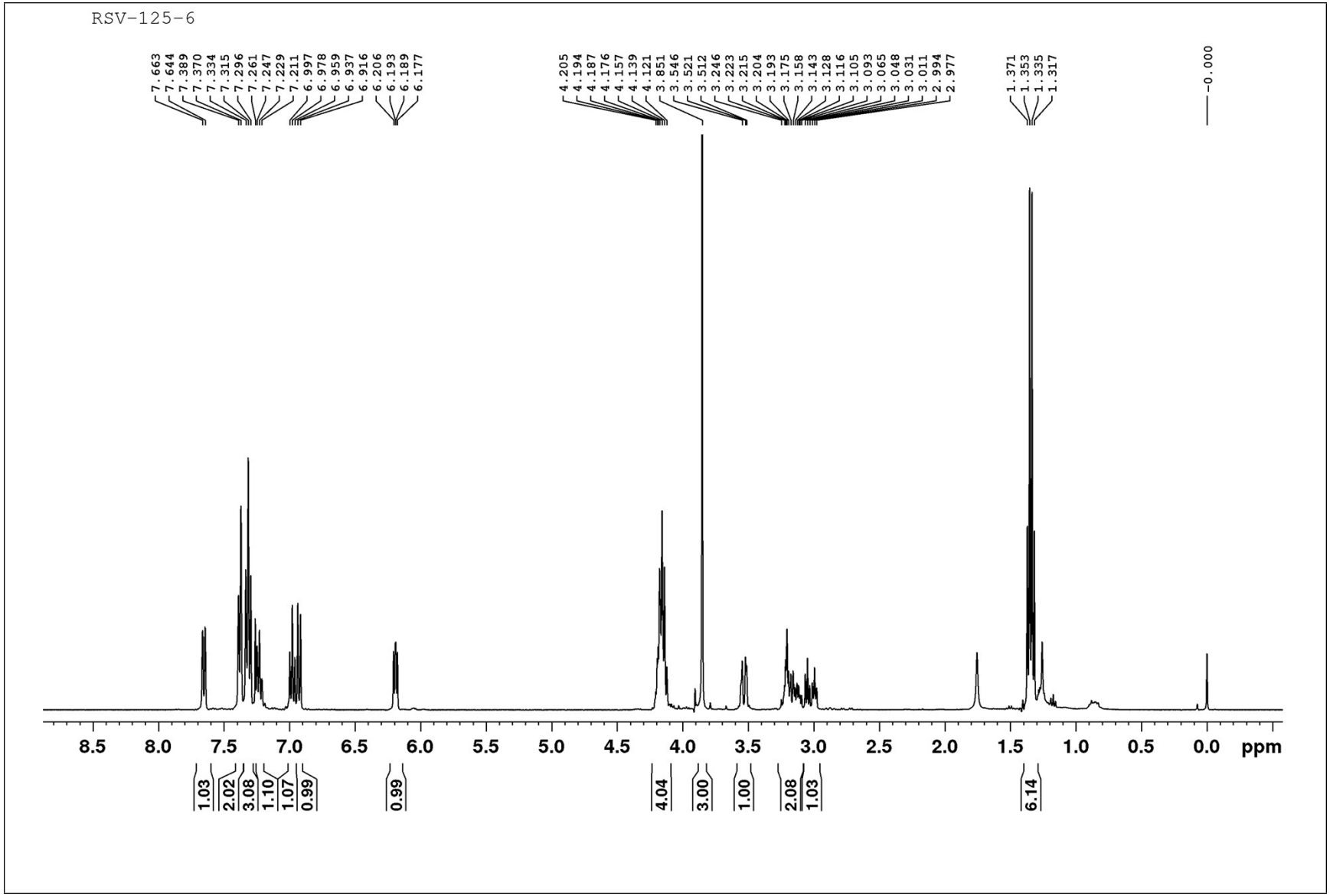
^{13}C NMR spectrum of compound **3o** (100 MHz/$CDCl_3$)



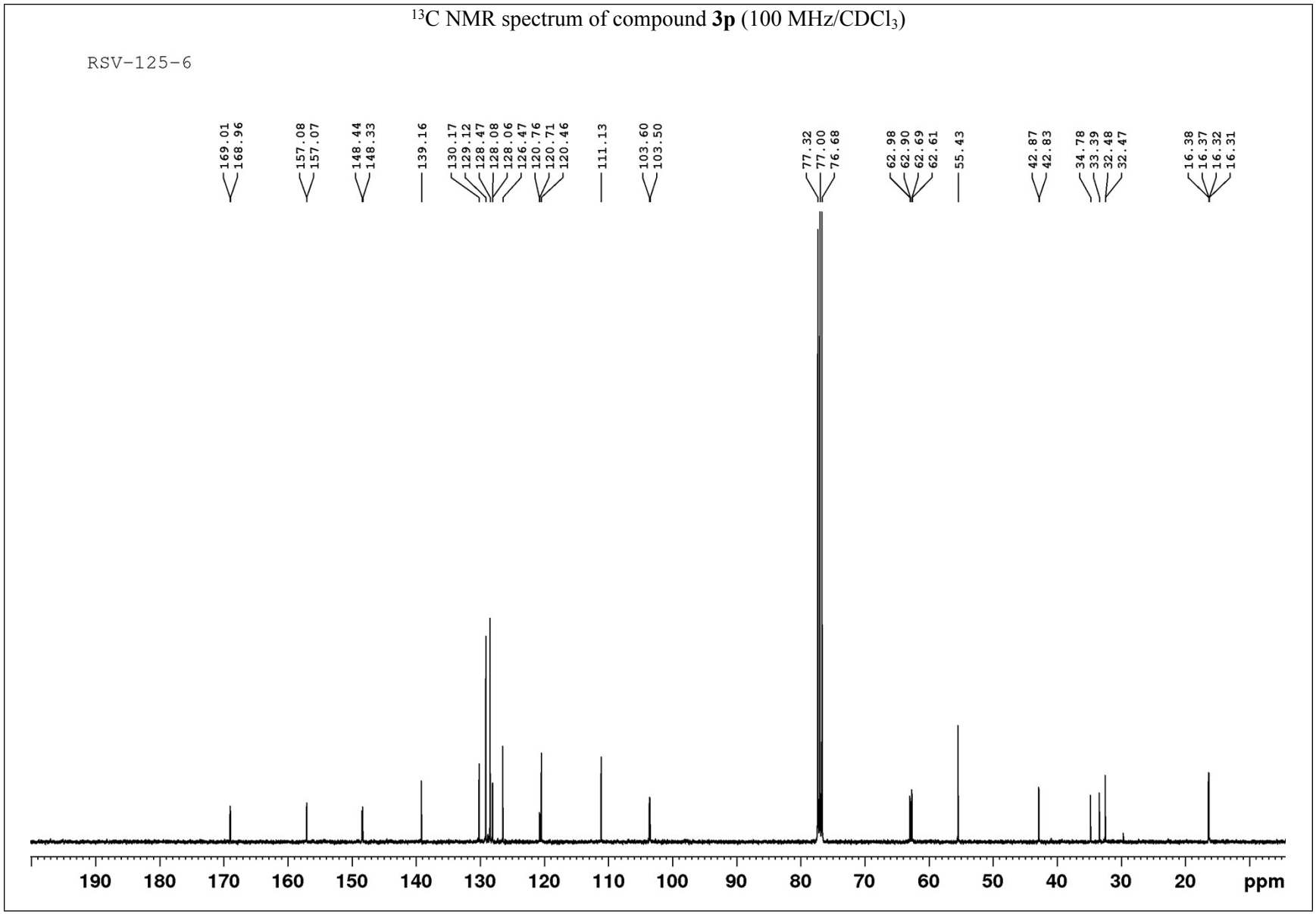
^{31}P NMR spectrum of compound **3o** (400 MHz/$CDCl_3$)



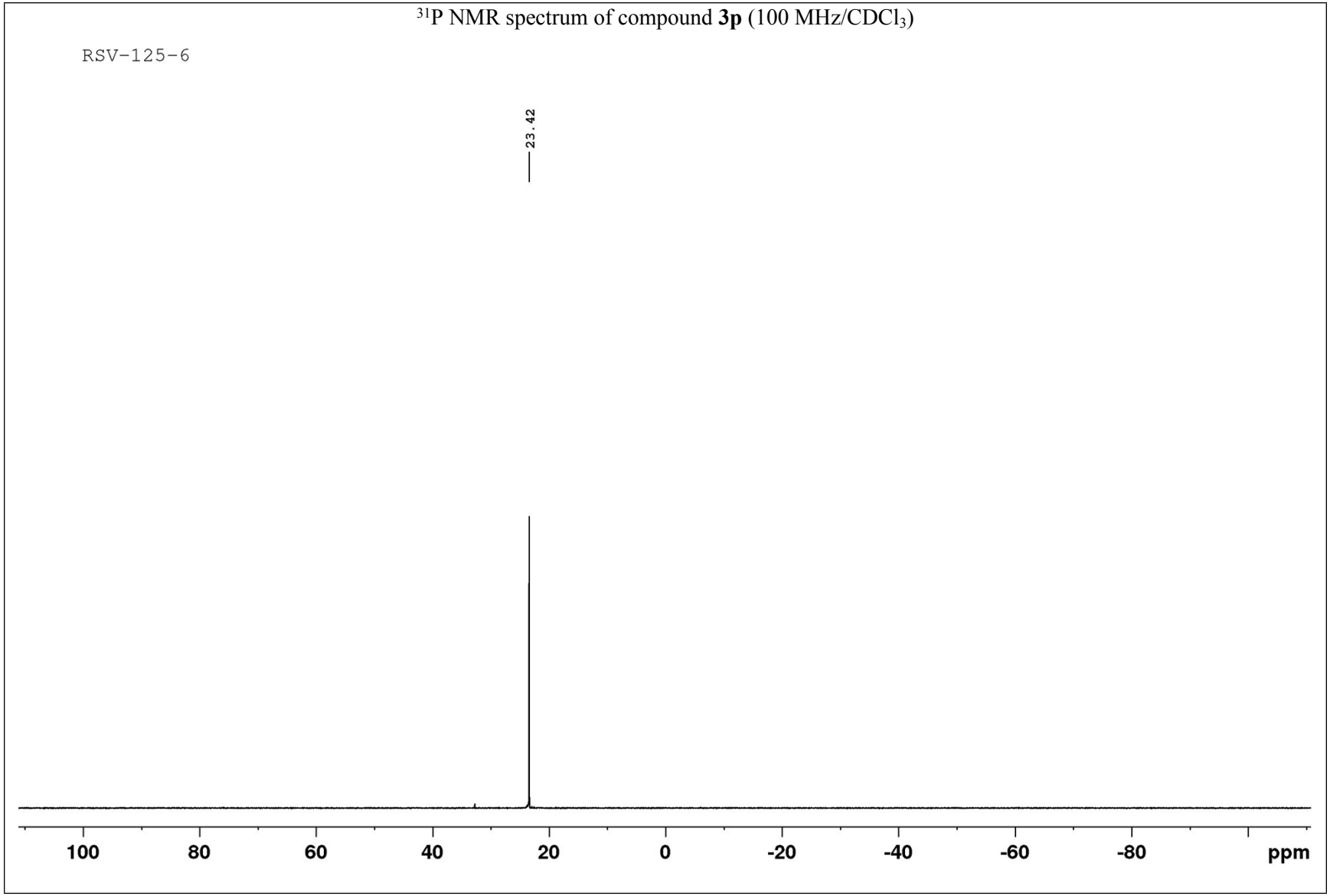
^{1}H NMR spectrum of compound **3p** (400 MHz/$CDCl_3$)


RSV-125-6
7.663
7.644
7.389
7.370
7.334
7.315
7.296
7.261
7.247
7.229
7.211
6.997
6.978
6.959
6.937
6.916
6.206
6.193
6.189
6.177
4.205
4.194
4.187
4.176
4.157
4.139
4.121
3.851
3.546
3.521
3.512
3.246
3.223
3.215
3.204
3.193
3.175
3.158
3.143
3.128
3.116
3.105
3.093
3.065
3.048
3.031
3.011
2.994
2.977
1.371
1.353
1.335
1.317
0.000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.03
2.02
3.08
1.10
1.07
0.99
0.99
4.04
3.00
1.00
2.08
1.03
6.14

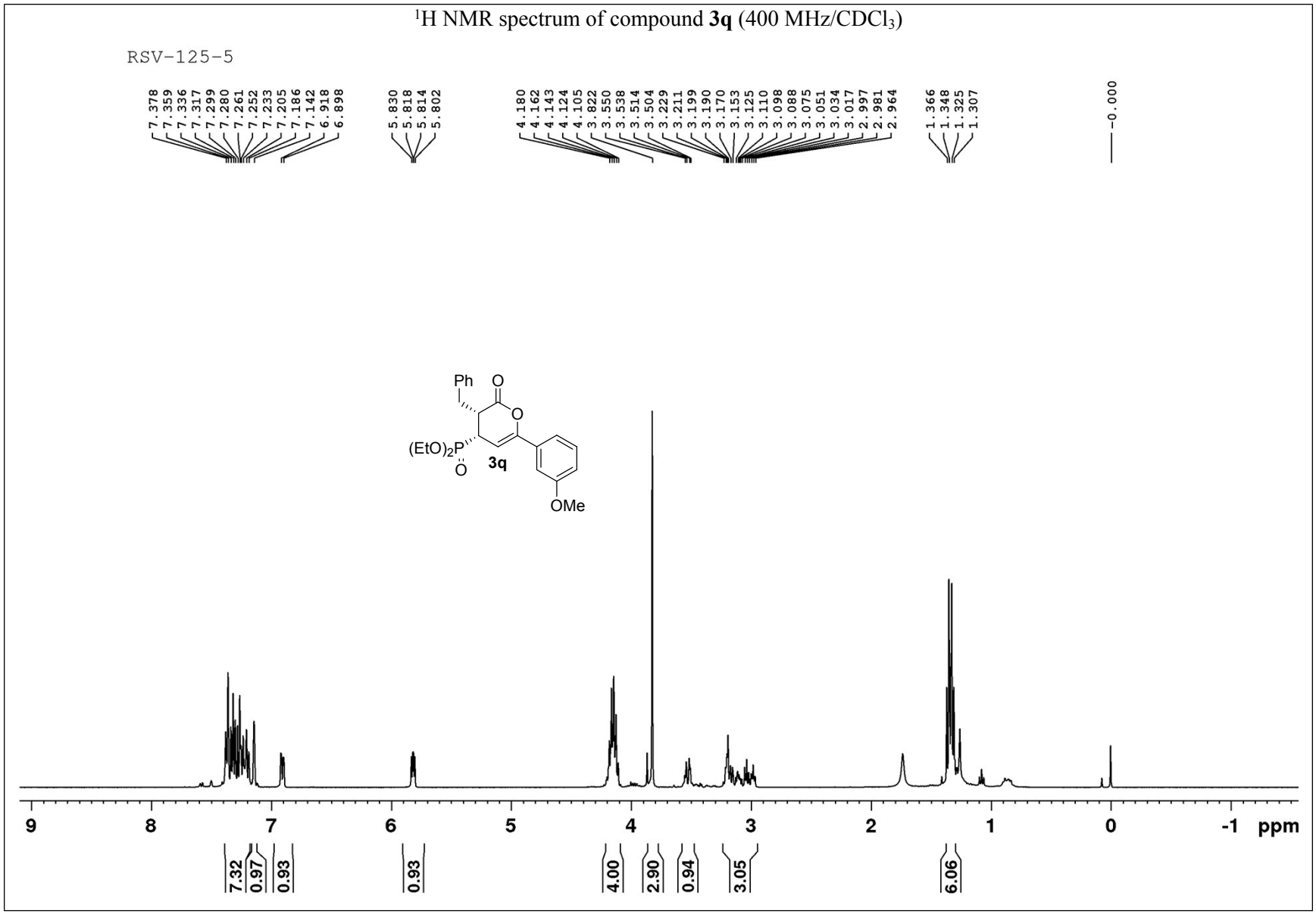
^{13}C NMR spectrum of compound **3p** (100 MHz/$CDCl_3$)



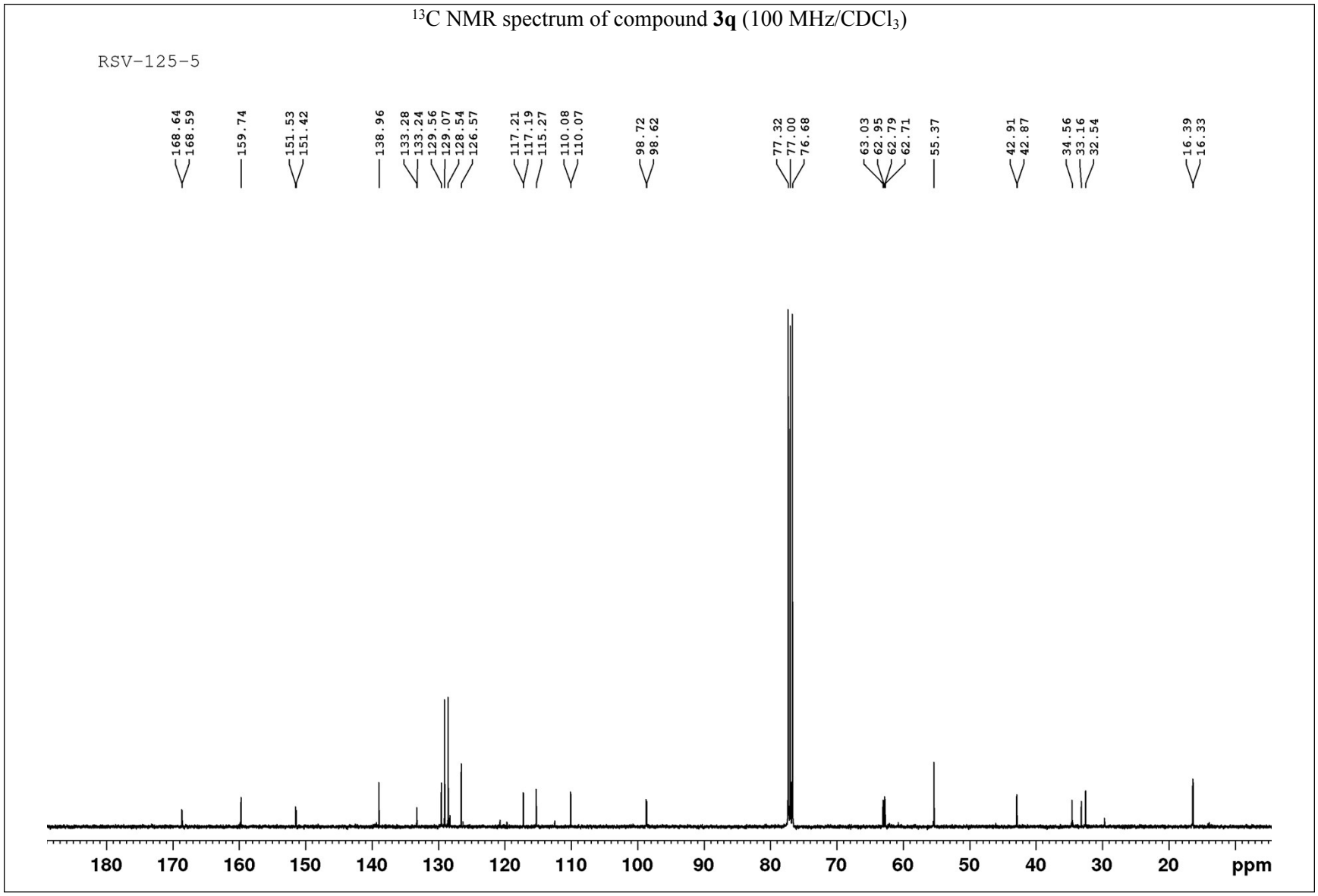
^{31}P NMR spectrum of compound **3p** (100 MHz/$CDCl_3$)



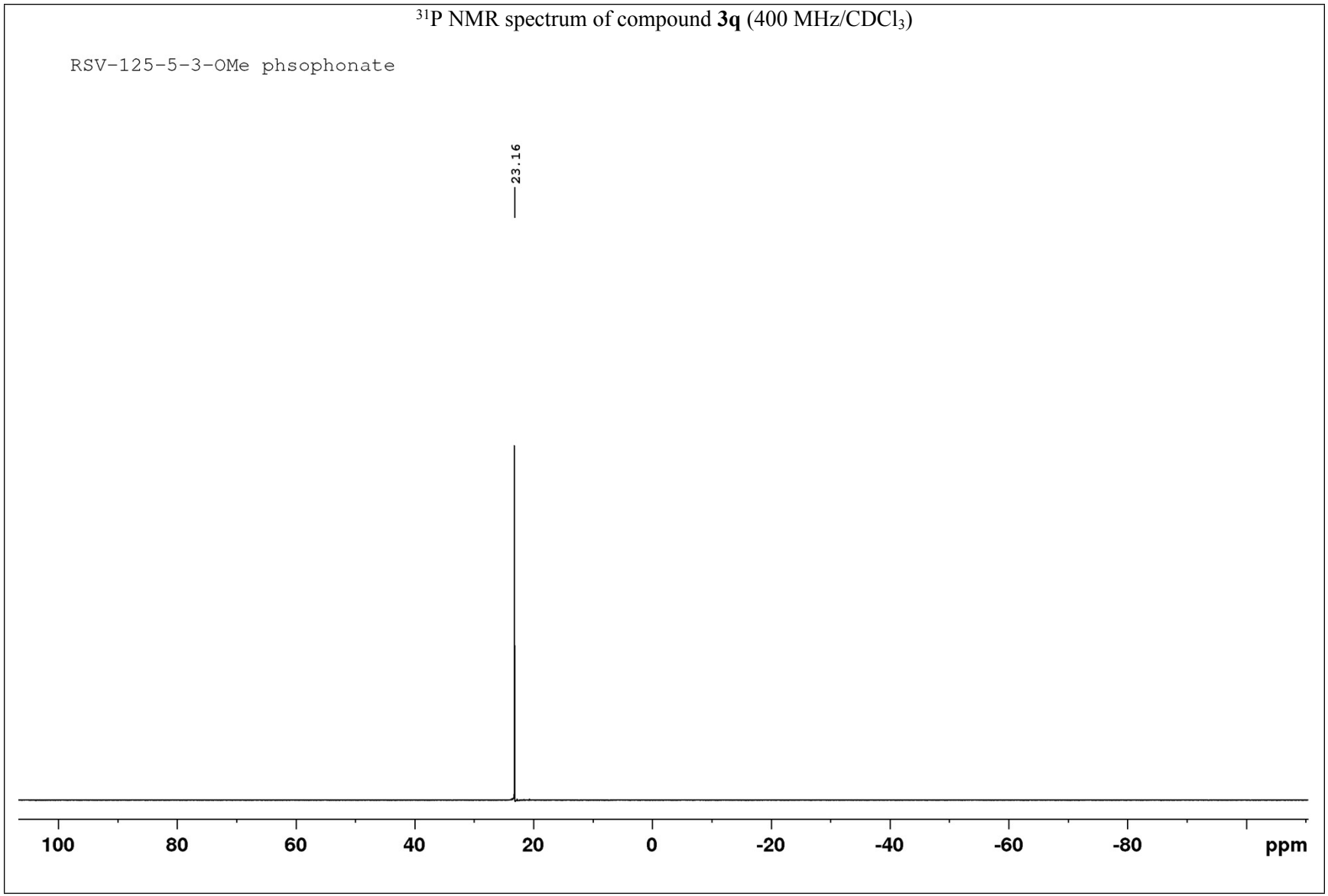
^{1}H NMR spectrum of compound **3q** (400 MHz/$CDCl_3$)



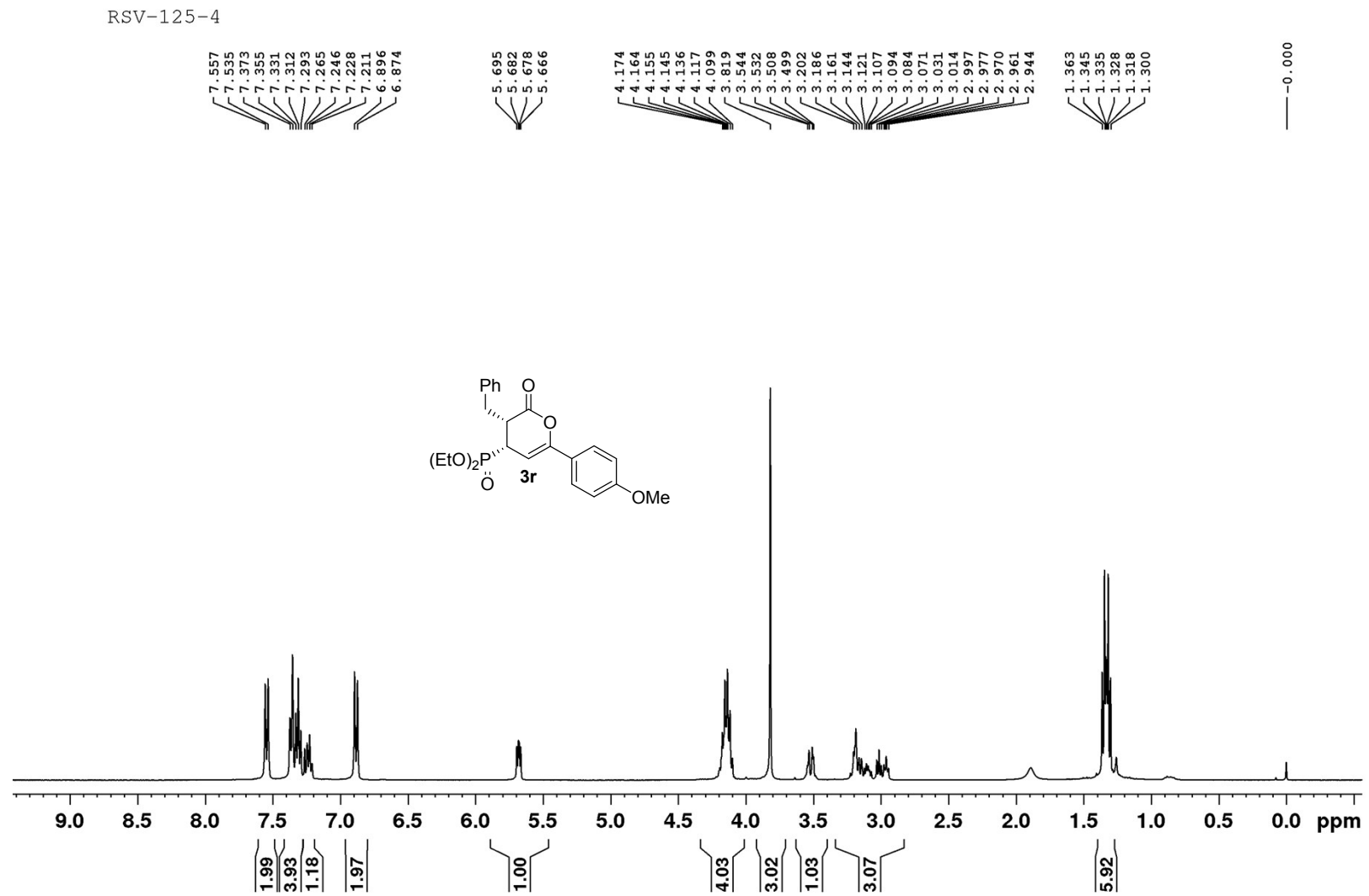
^{13}C NMR spectrum of compound **3q** (100 MHz/$CDCl_3$)



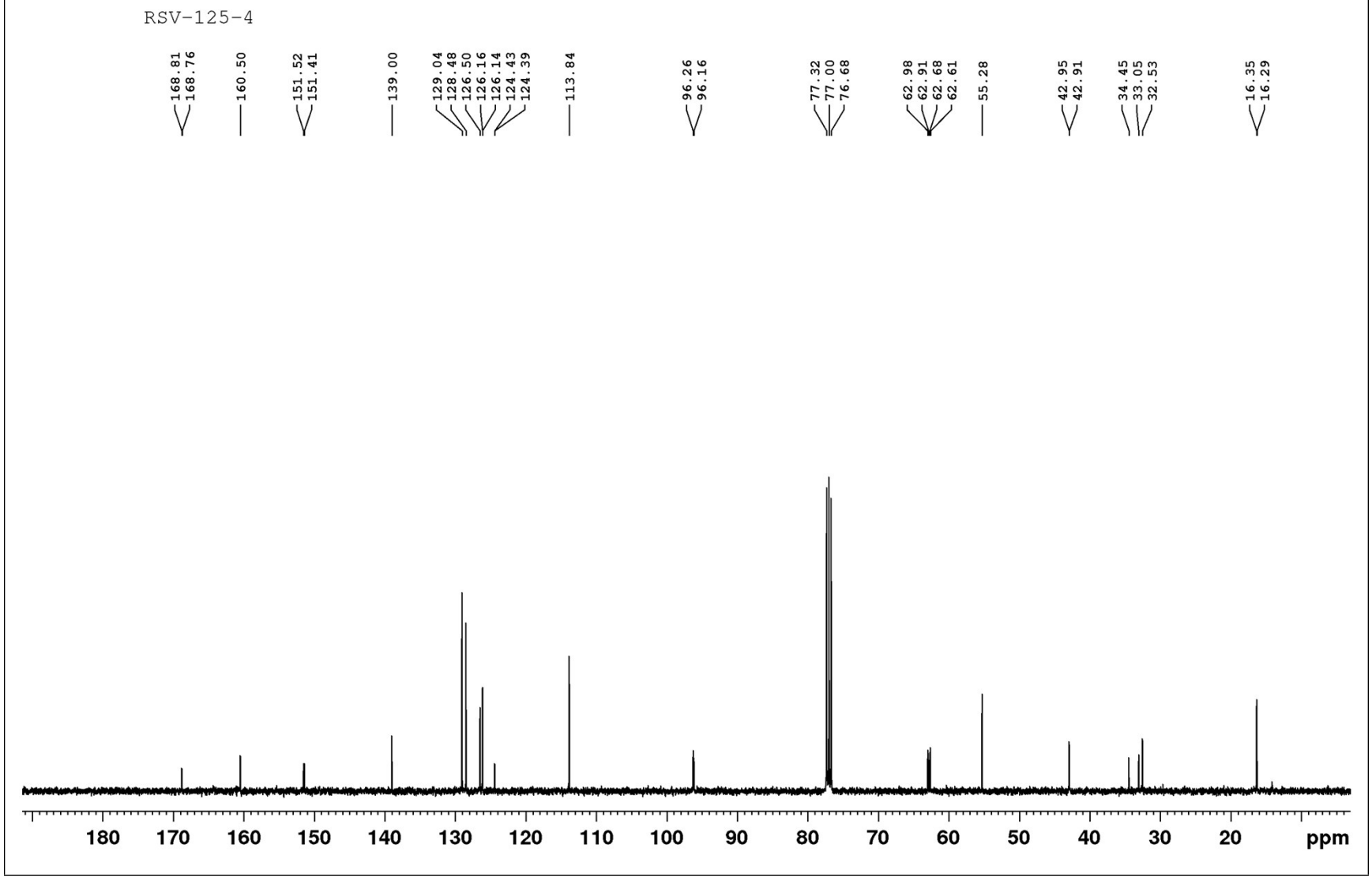
^{31}P NMR spectrum of compound **3q** (400 MHz/$CDCl_3$)



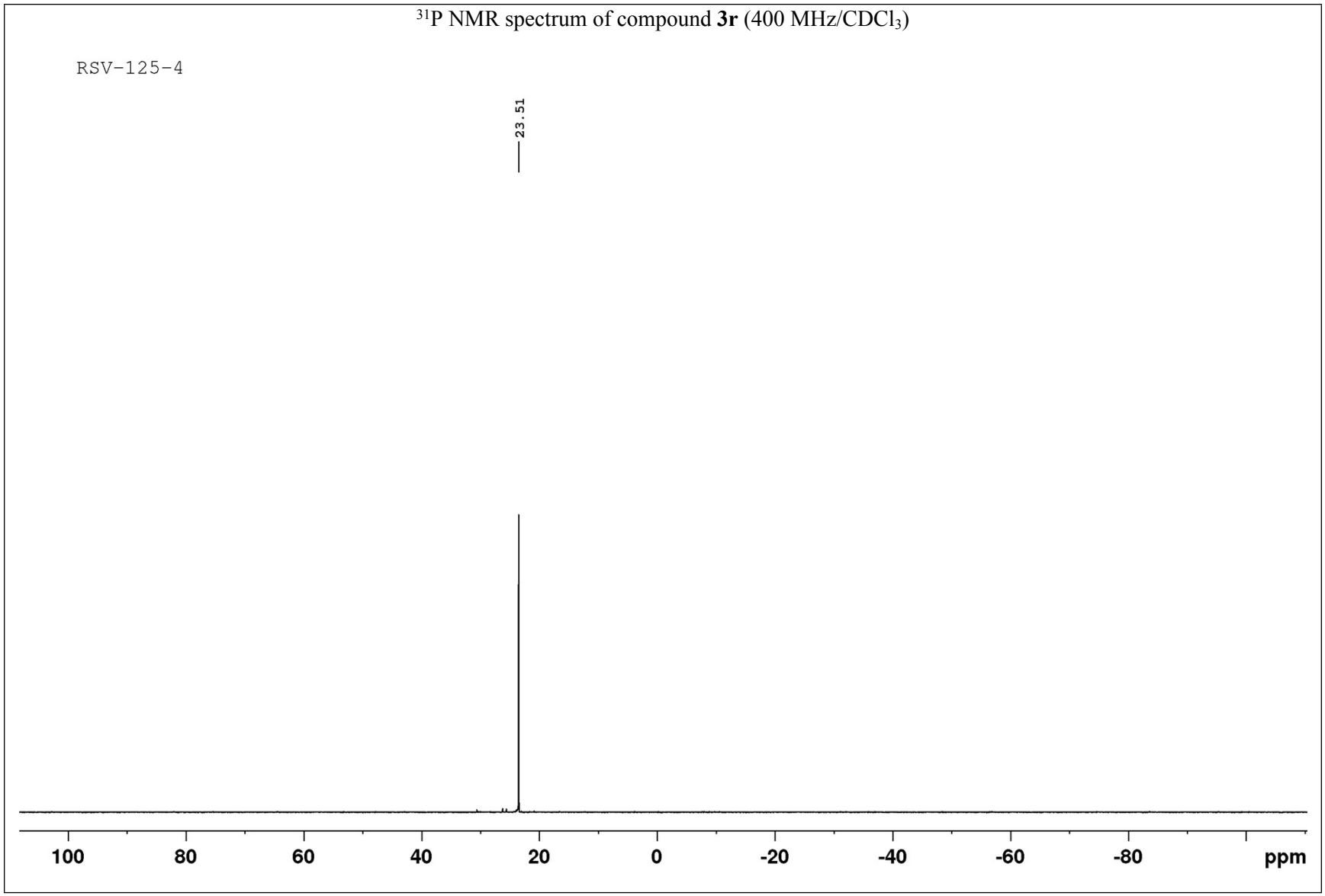
^{1}H NMR spectrum of compound **3r** (400 MHz/$CDCl_3$)



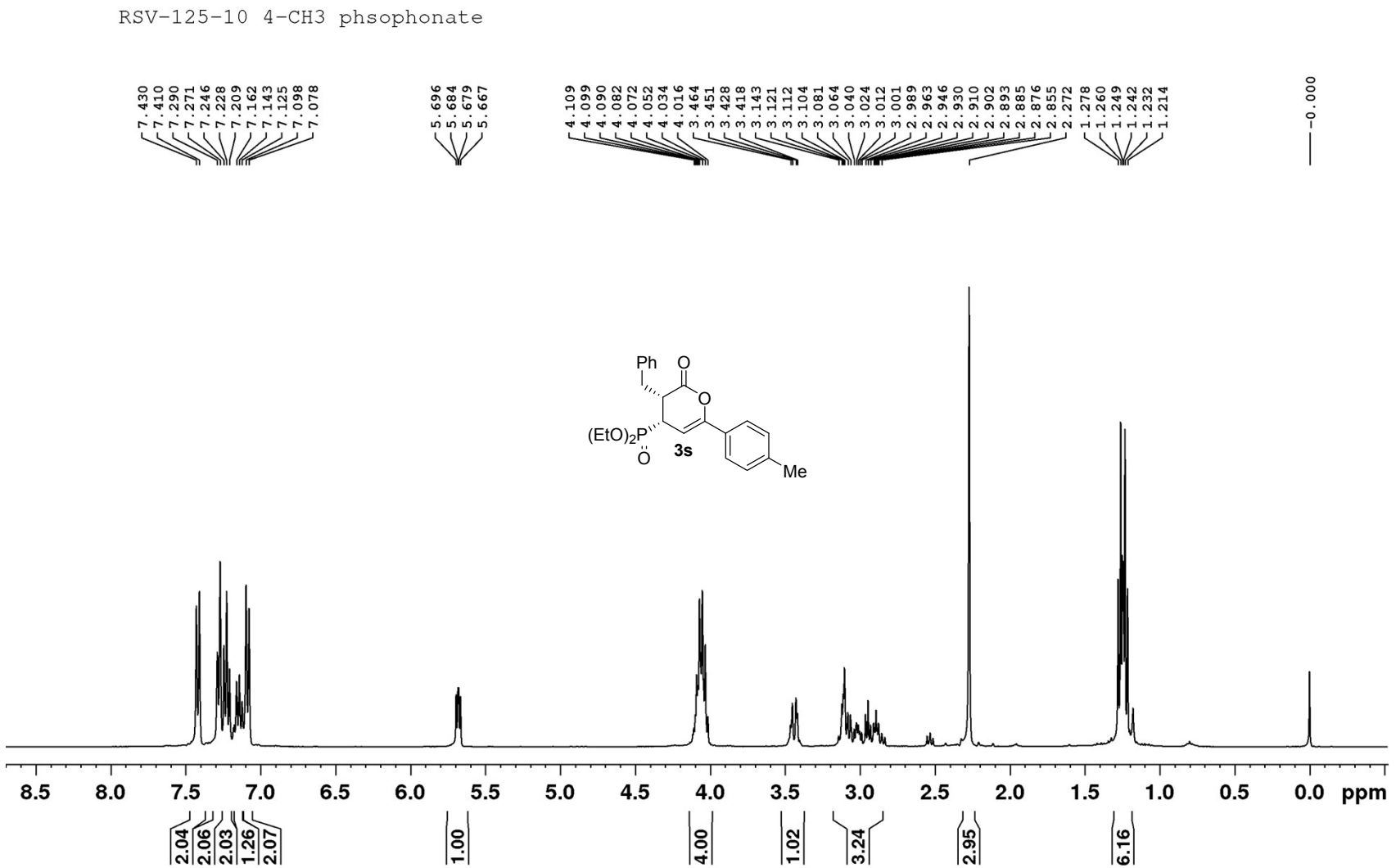
^{13}C NMR spectrum of compound **3r** (400 MHz/$CDCl_3$)



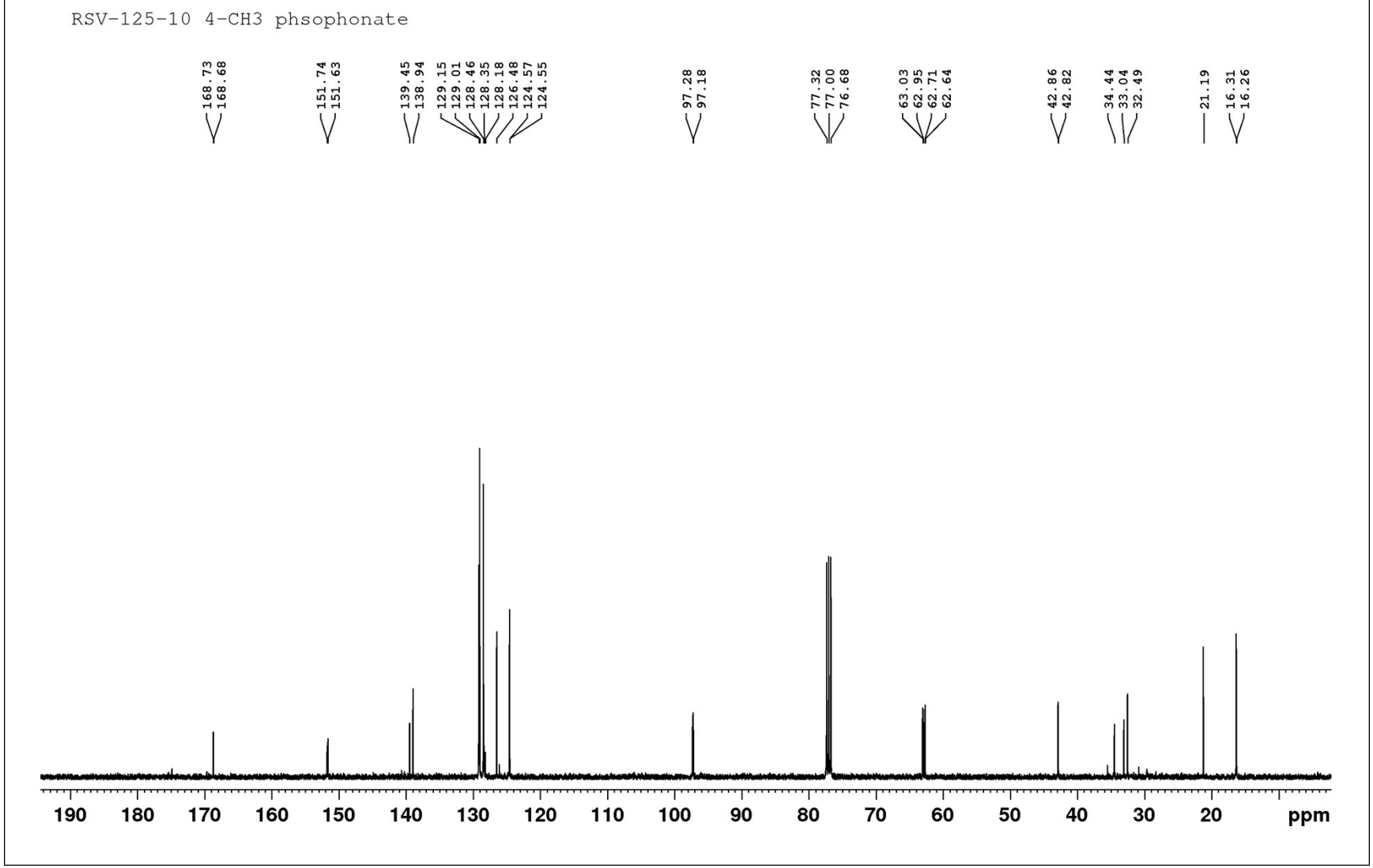
^{31}P NMR spectrum of compound **3r** (400 MHz/$CDCl_3$)



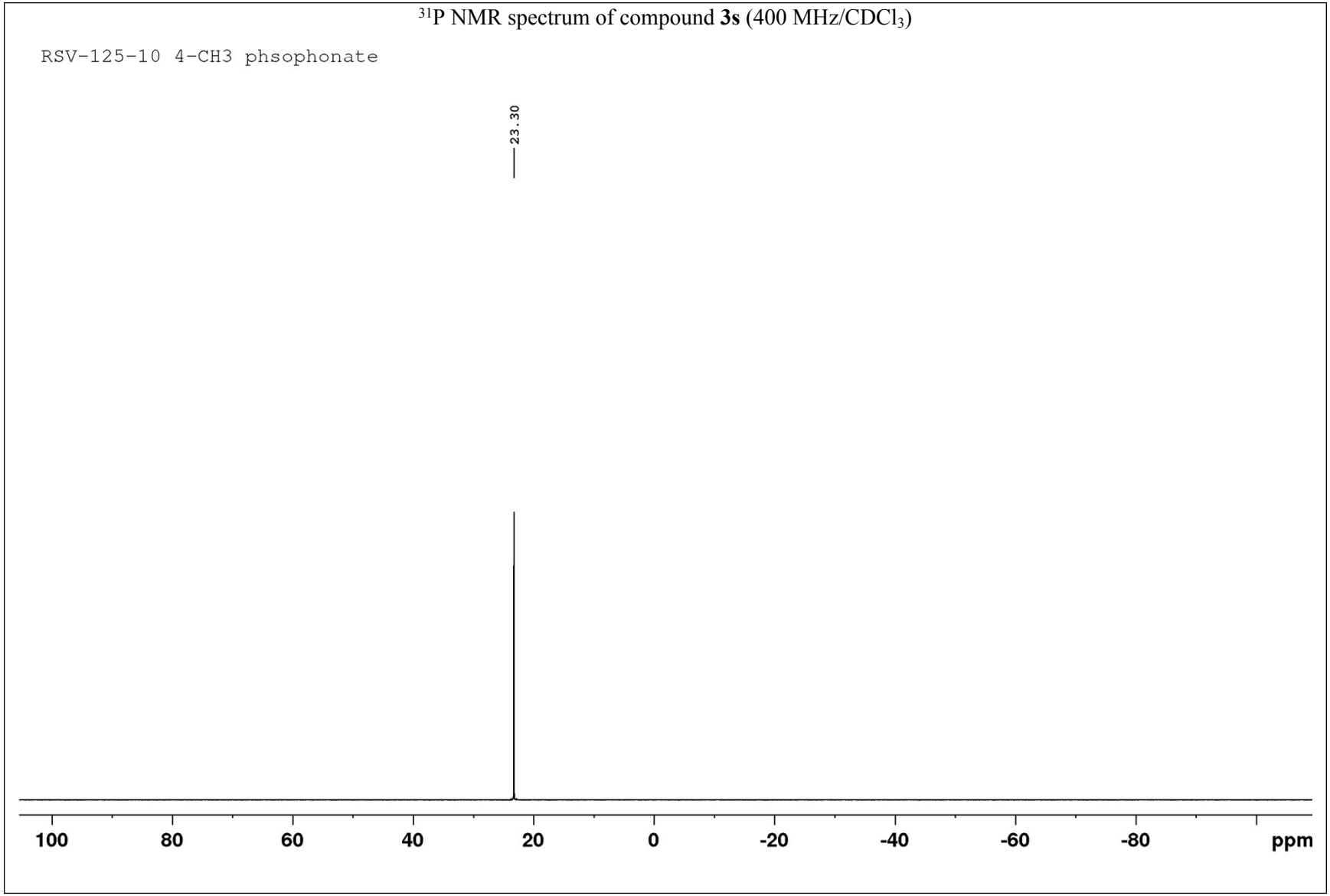
^{1}H NMR spectrum of compound **3s** (400 MHz/$CDCl_3$)



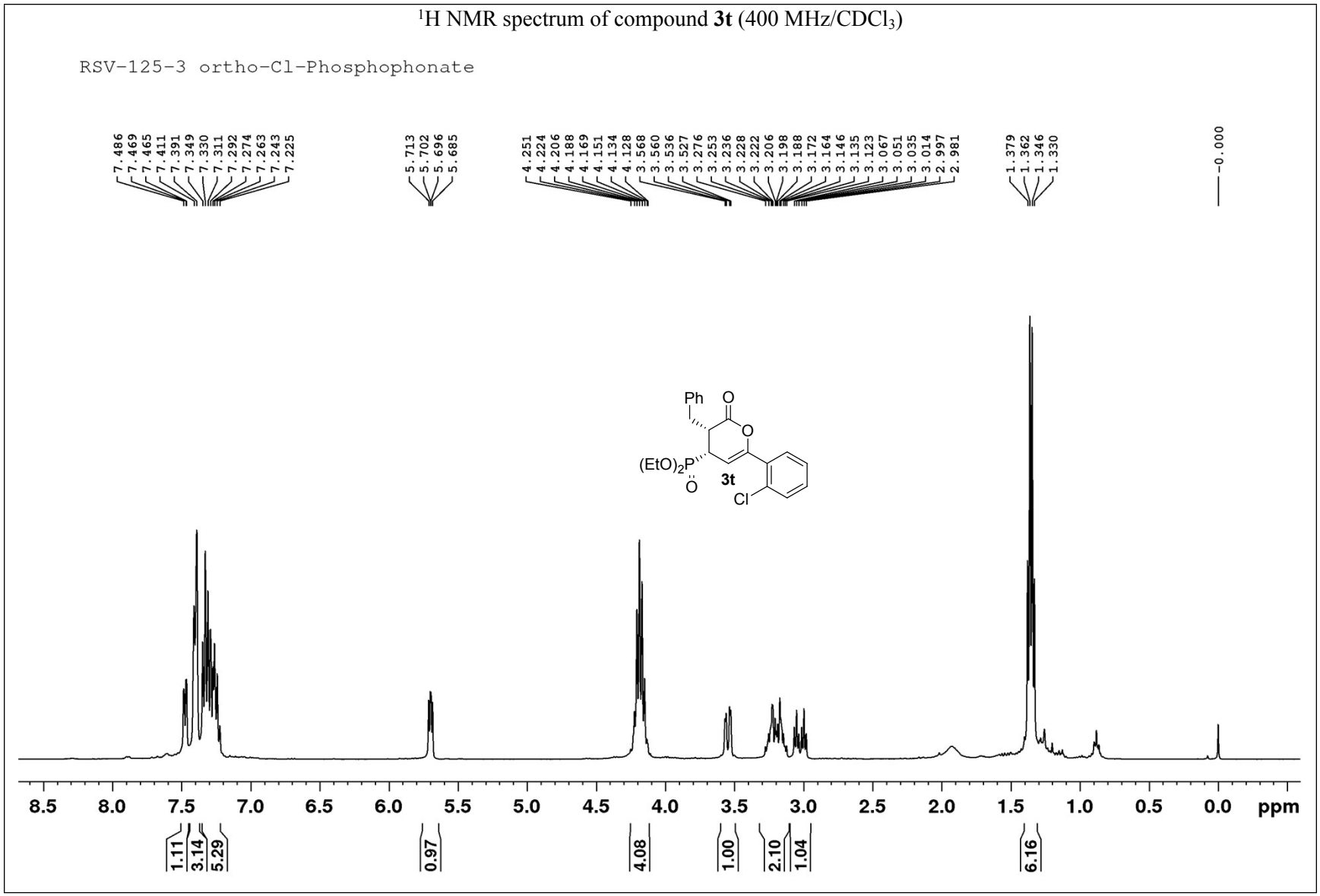
^{13}C NMR spectrum of compound **3s** (100 MHz/$CDCl_3$)



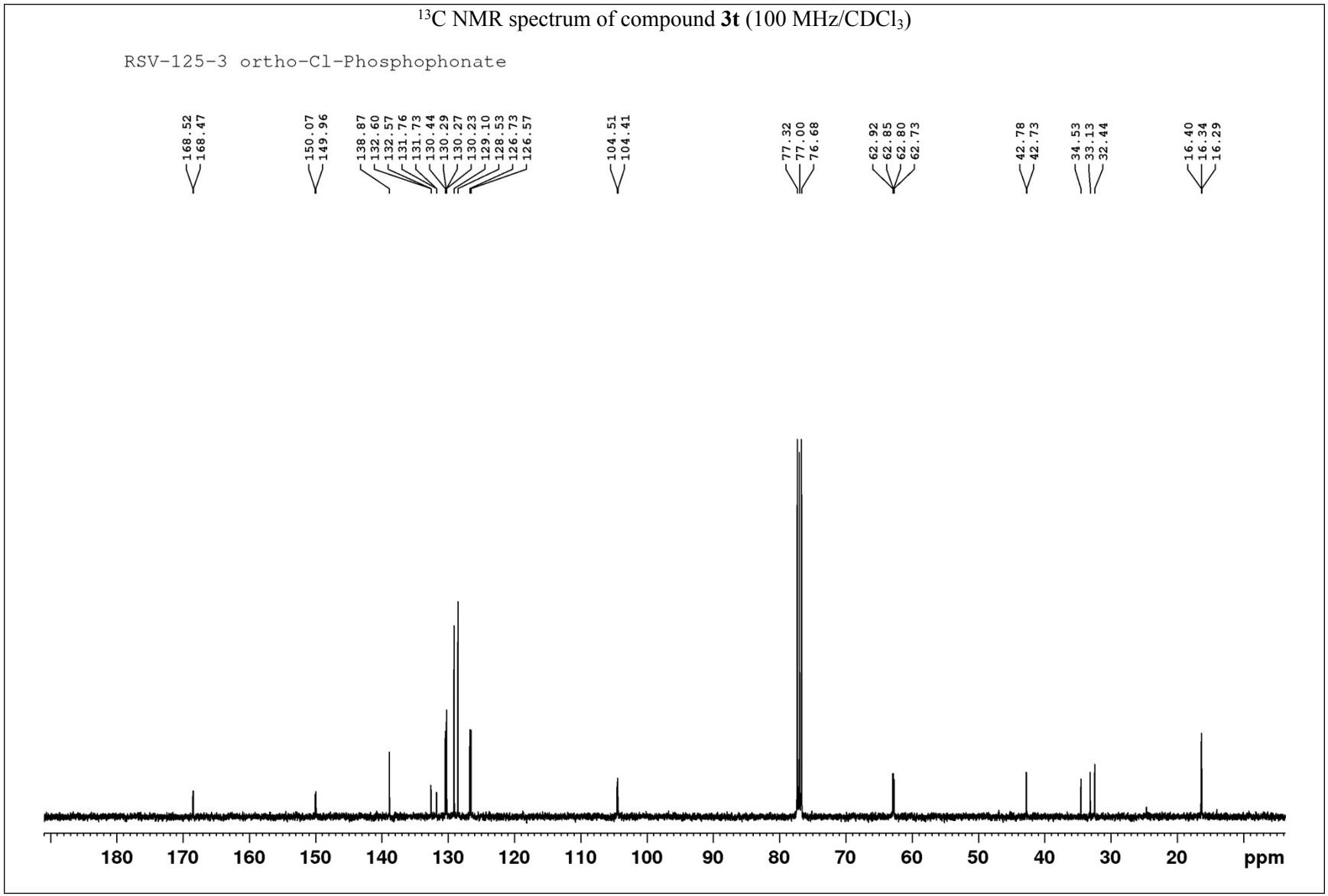
^{31}P NMR spectrum of compound **3s** (400 MHz/$CDCl_3$)



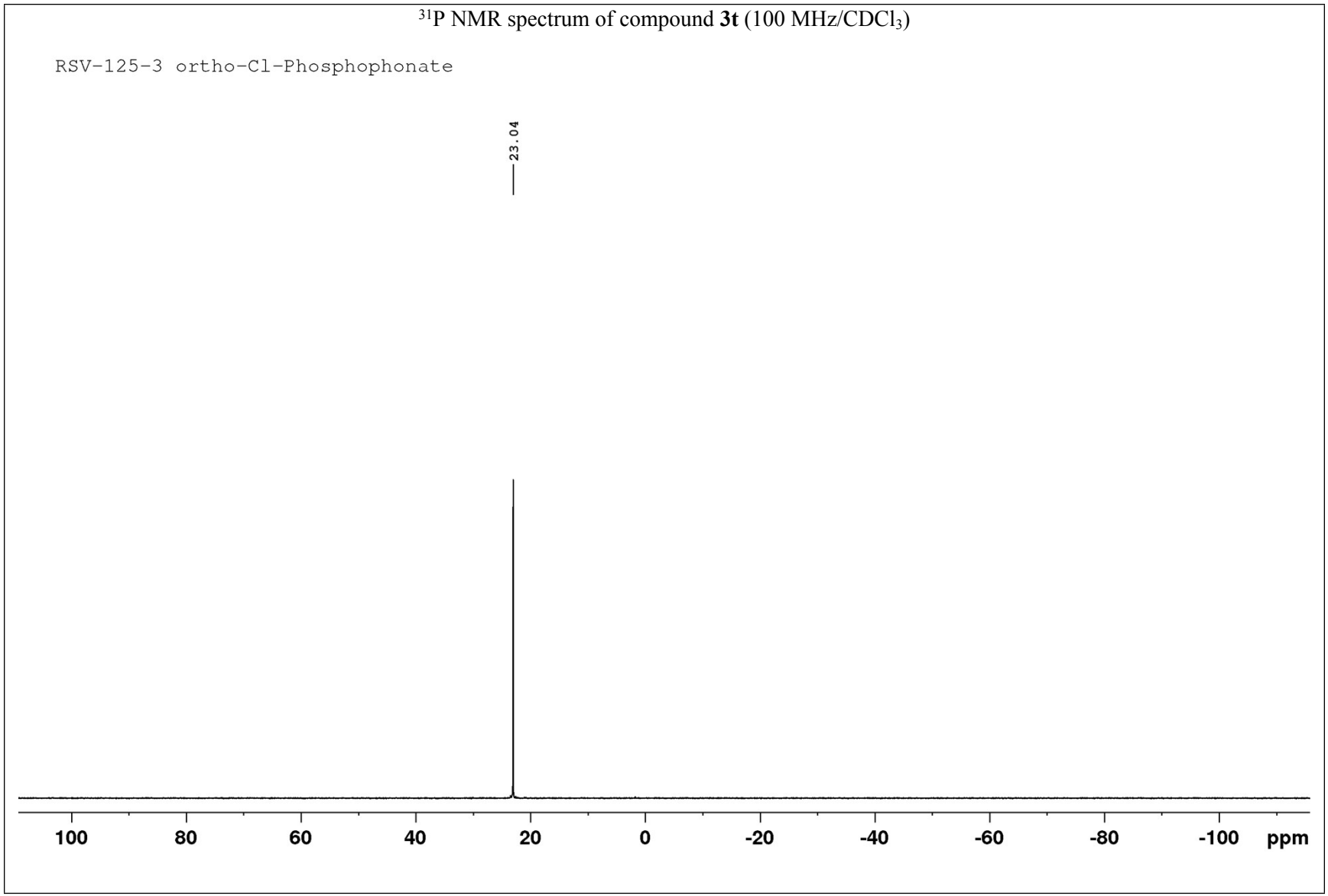
^{1}H NMR spectrum of compound **3t** (400 MHz/$CDCl_3$)



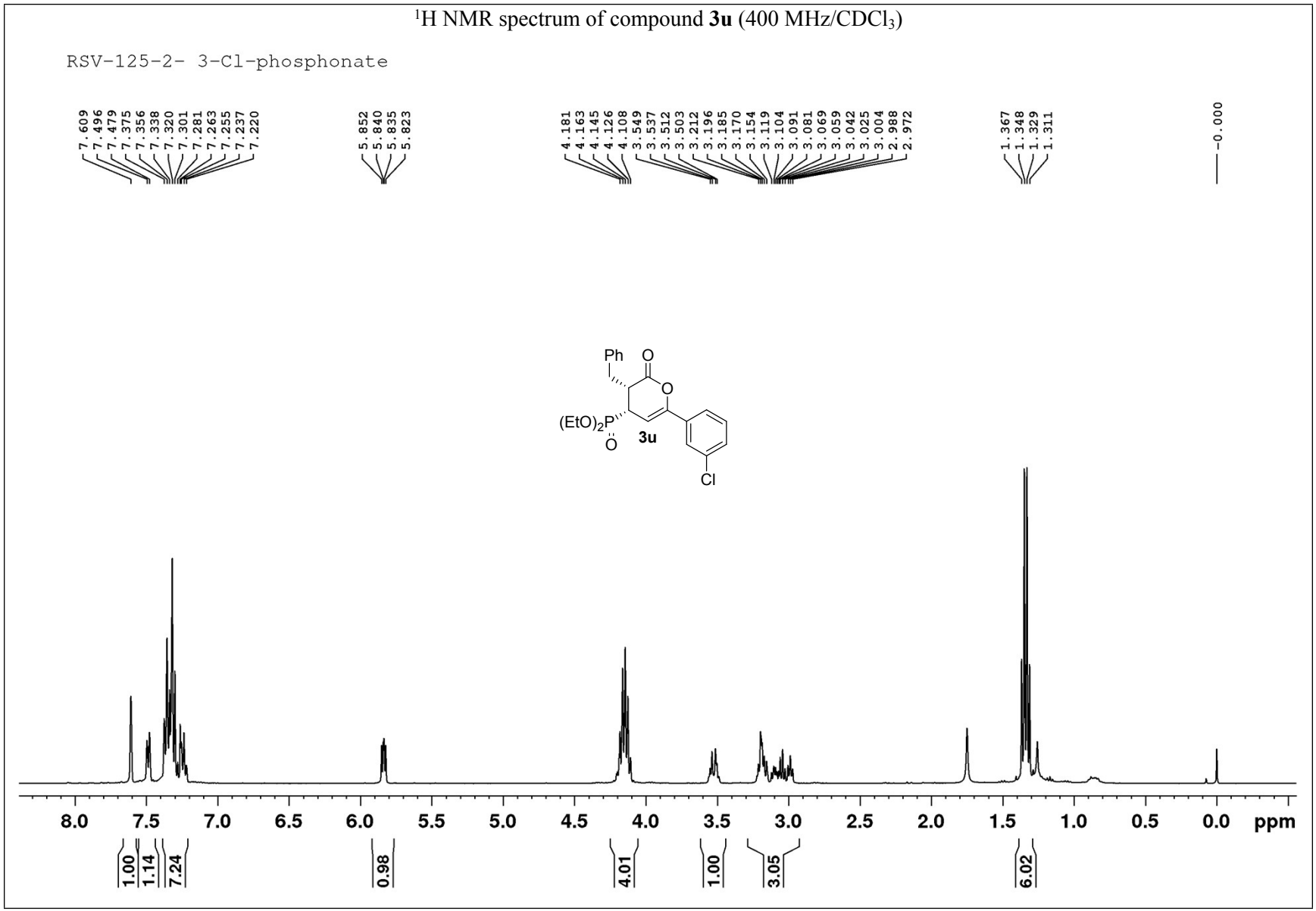
^{13}C NMR spectrum of compound **3t** (100 MHz/$CDCl_3$)



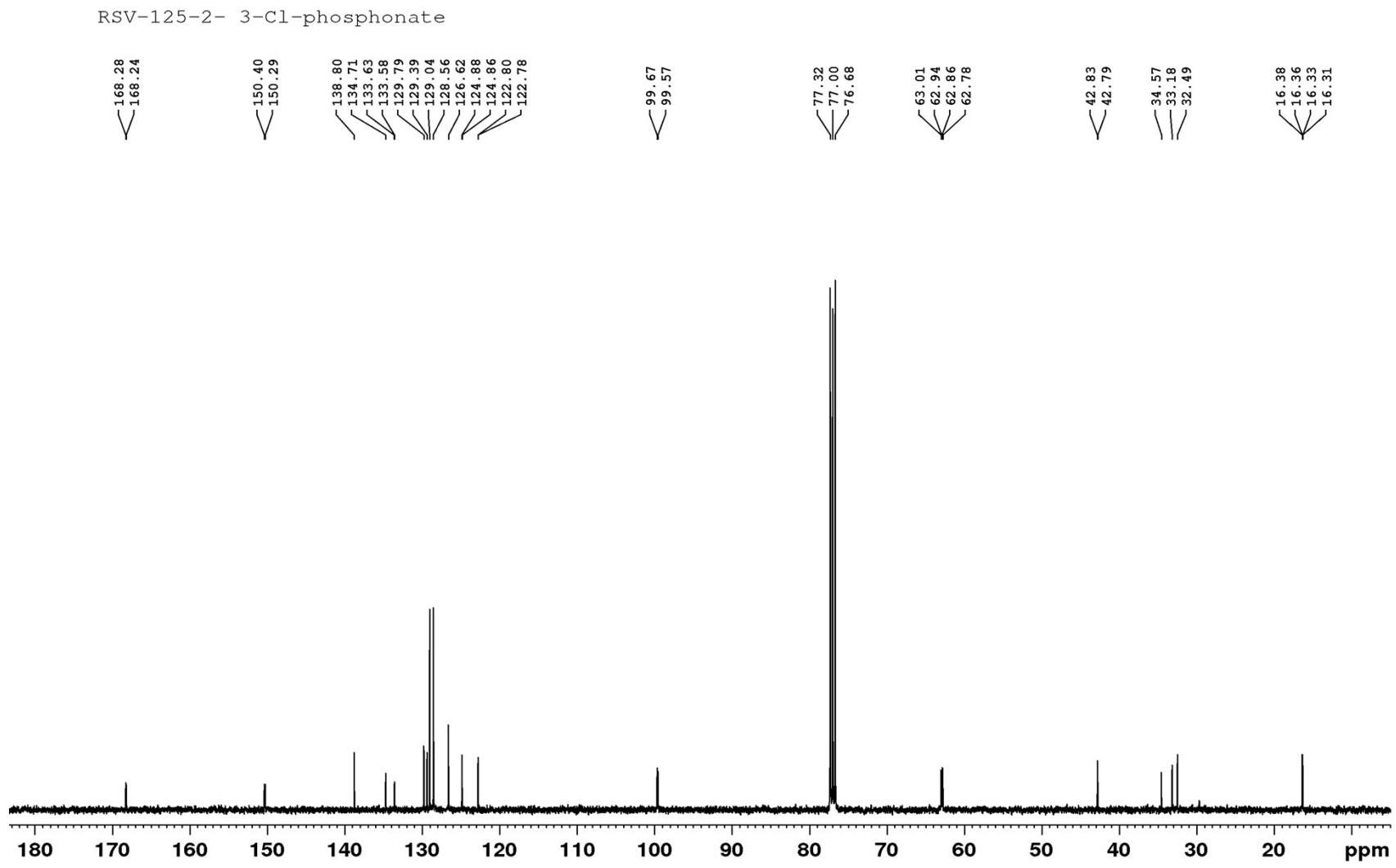
^{31}P NMR spectrum of compound **3t** (100 MHz/$CDCl_3$)



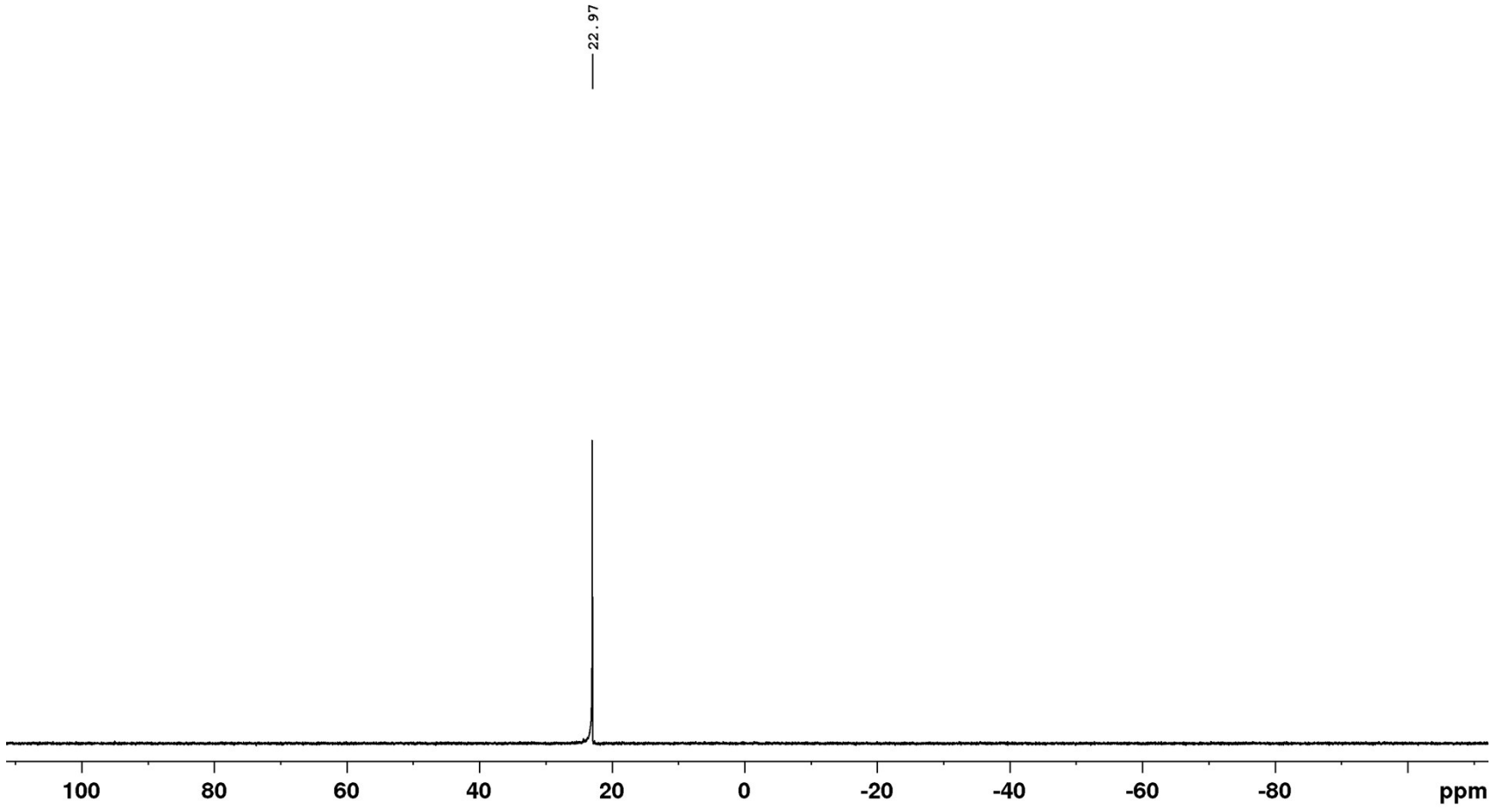
^{1}H NMR spectrum of compound **3u** (400 MHz/$CDCl_3$)



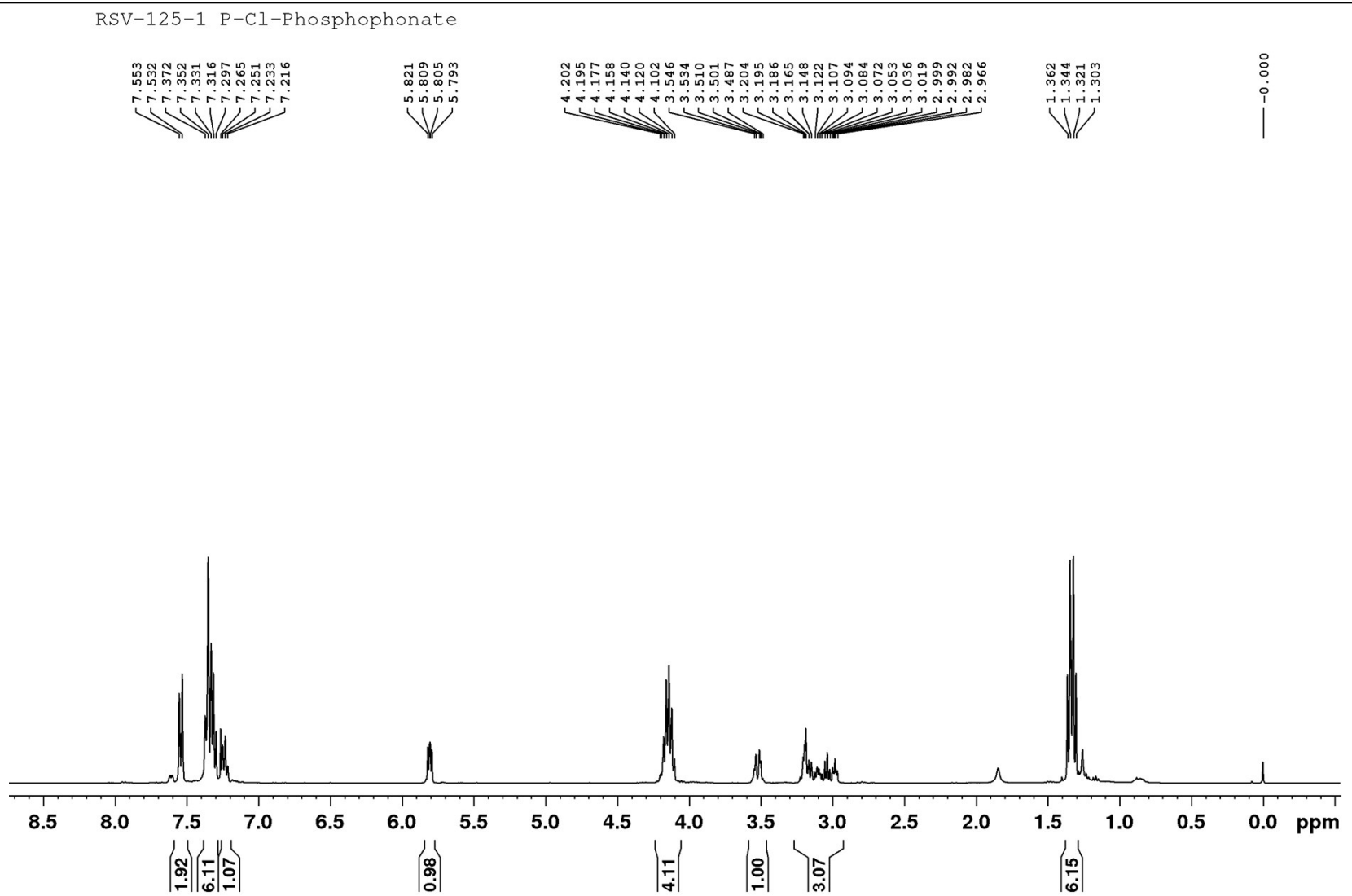
^{13}C NMR spectrum of compound **3u** (100 MHz/$CDCl_3$)



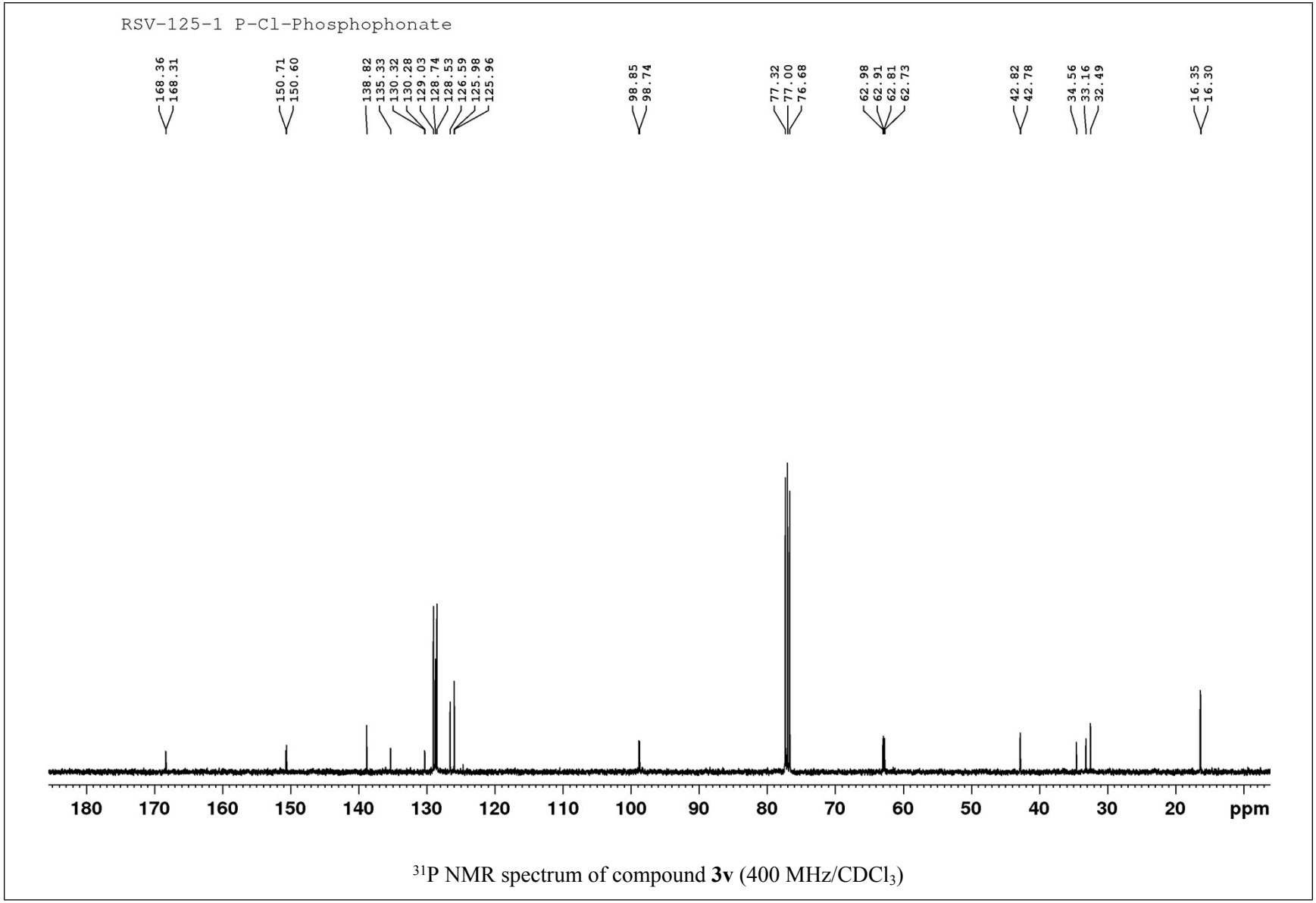
^{31}P NMR spectrum of compound **3u** (400 MHz/$CDCl_3$)



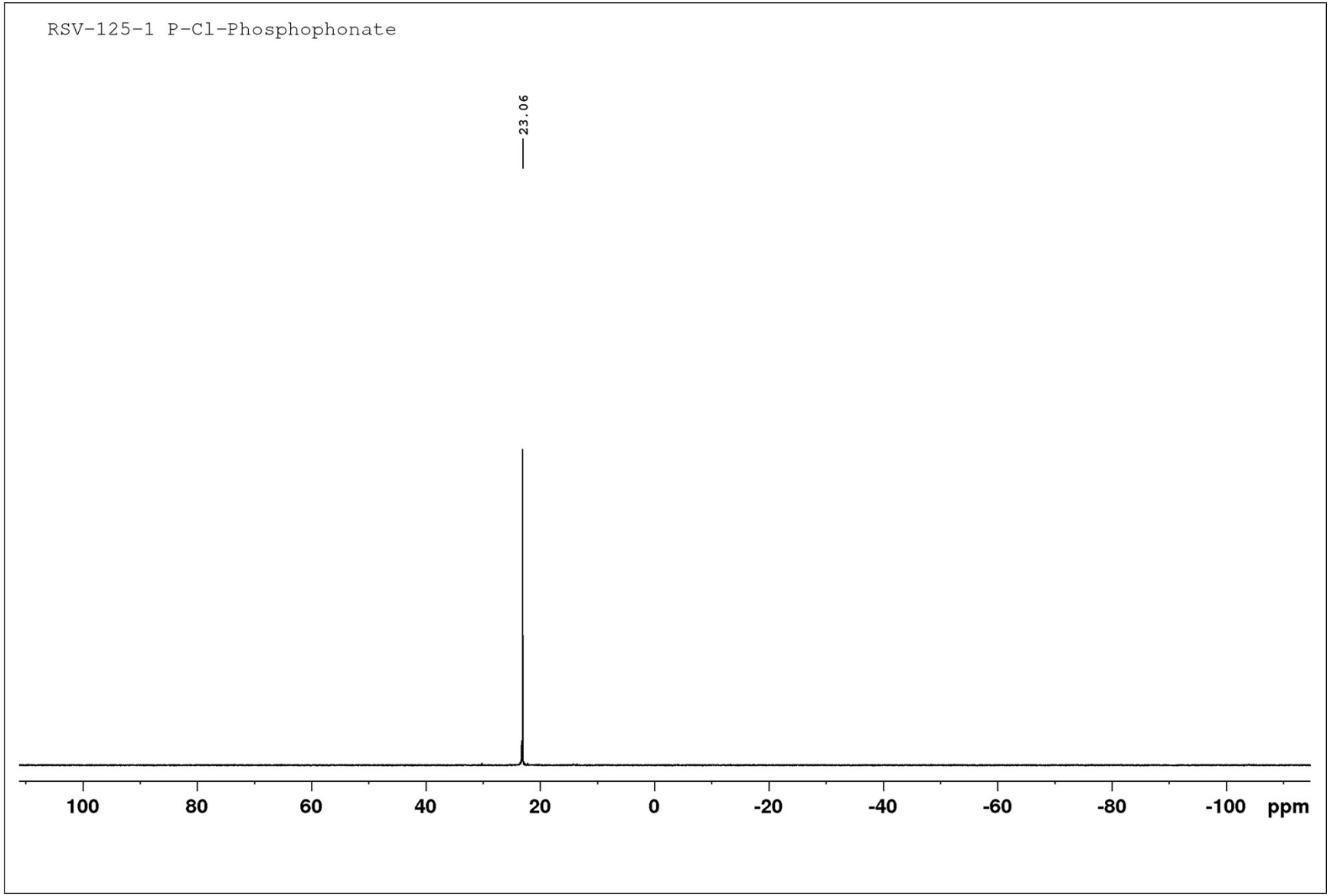
^{1}H NMR spectrum of compound **3v** (400 MHz/$CDCl_3$)



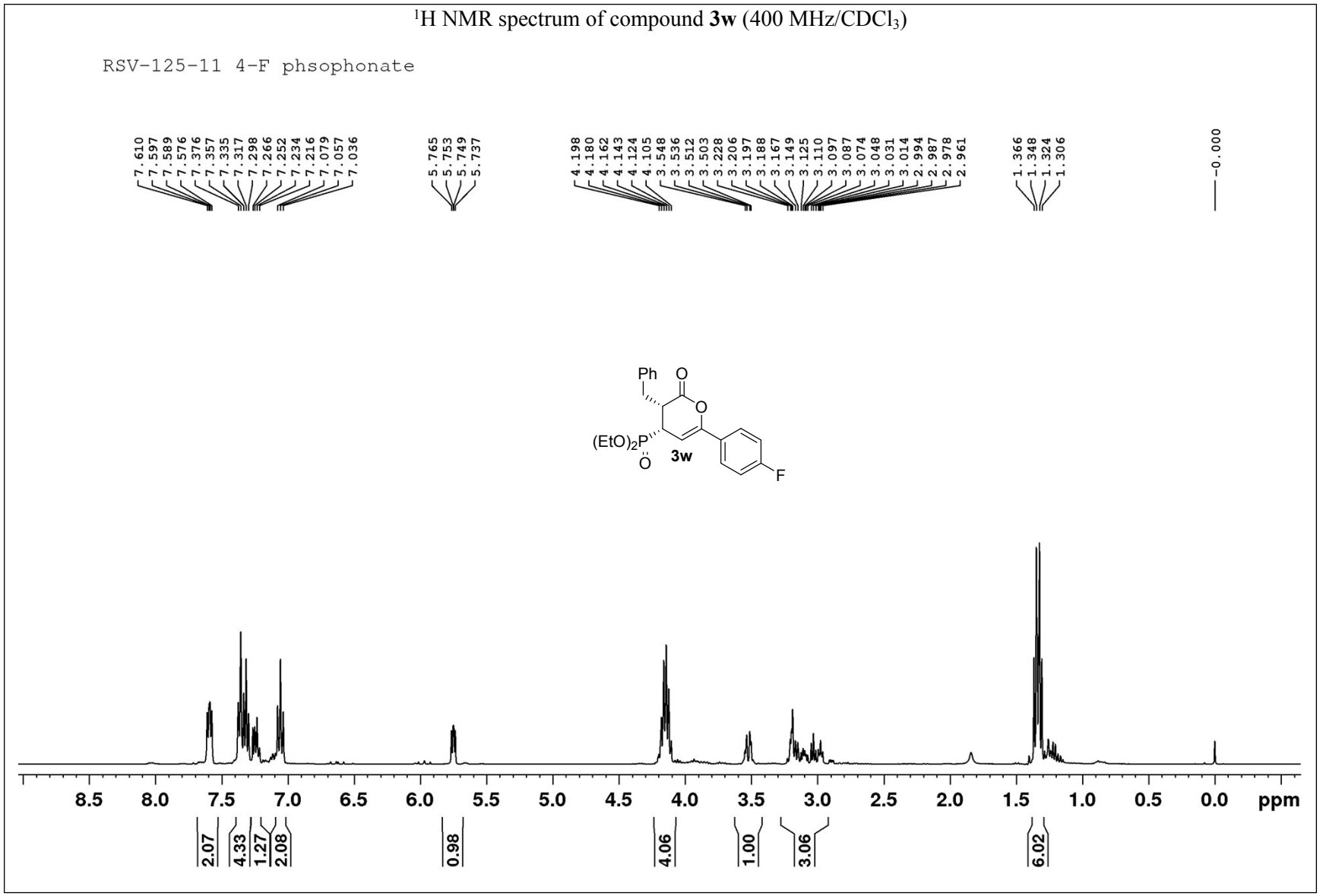
^{13}C NMR spectrum of compound **3v** (400 MHz/$CDCl_3$)



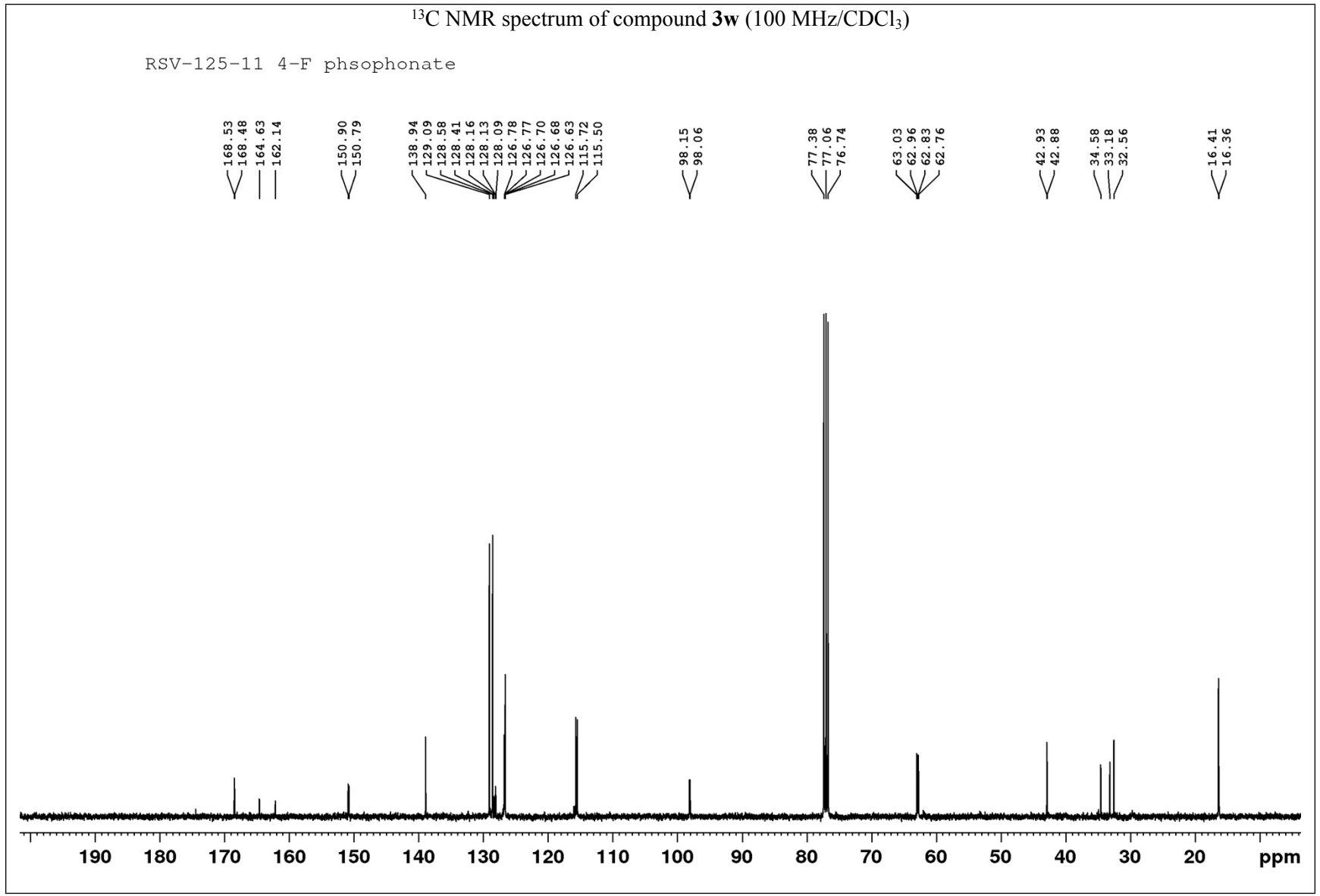
^{31}P NMR spectrum of compound **3v** (400 MHz/$CDCl_3$)


RSV-125-1 P-Cl-Phosphophonate
23.06
100
80
60
40
20
0
-20
-40
-60
-80
-100
ppm

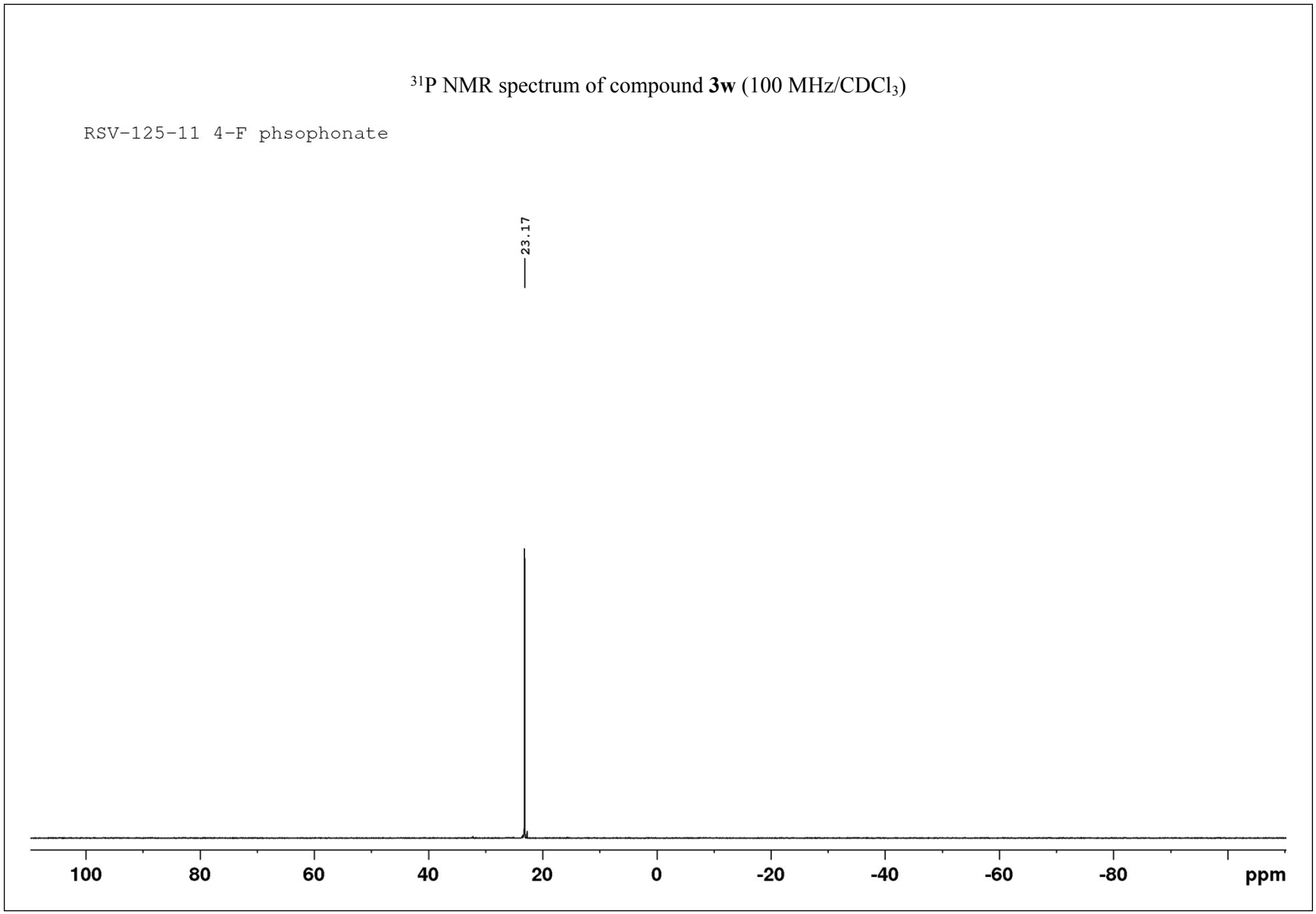
^{1}H NMR spectrum of compound **3w** (400 MHz/$CDCl_3$)



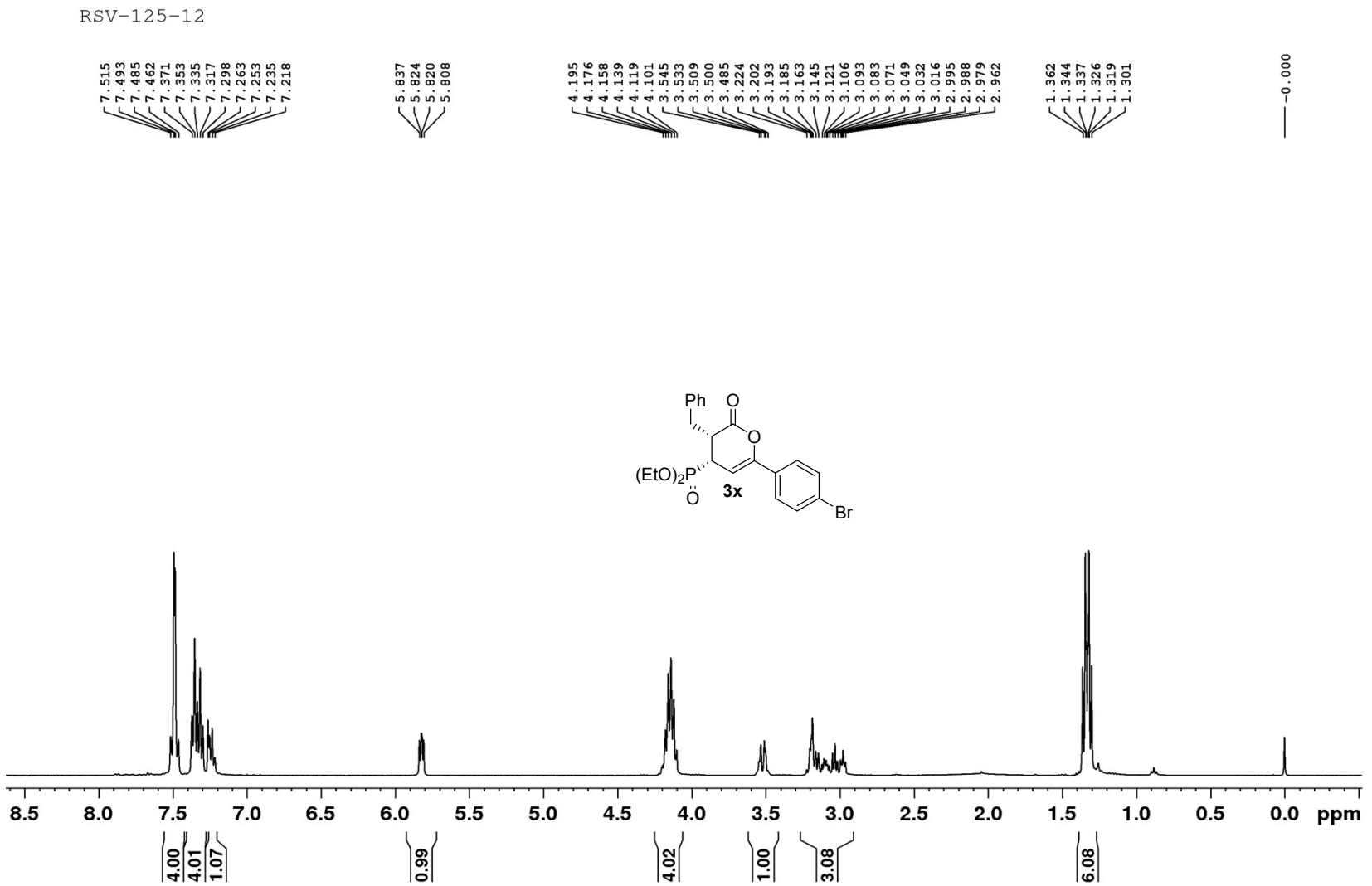
^{13}C NMR spectrum of compound **3w** (100 MHz/$CDCl_3$)



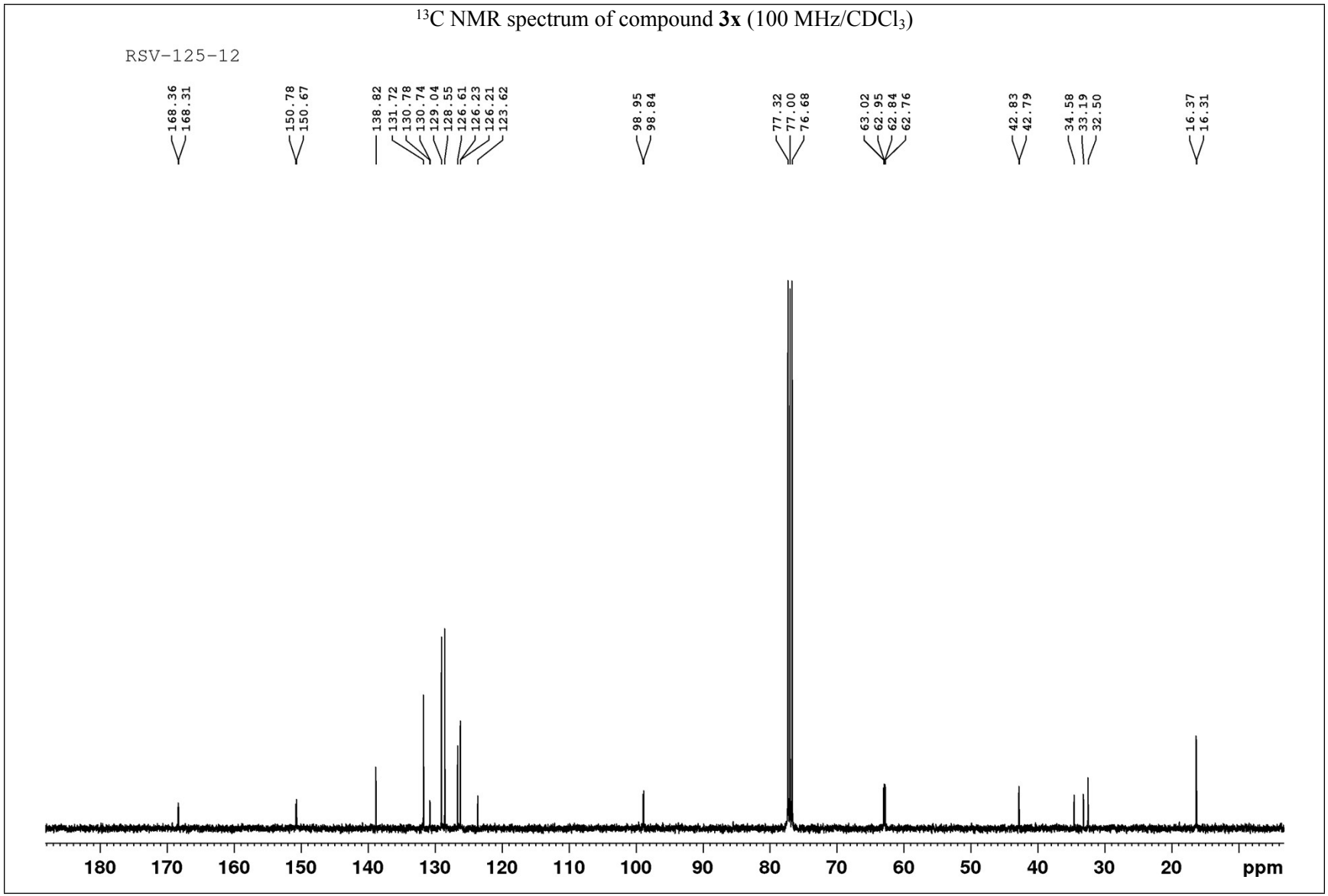
^{31}P NMR spectrum of compound **3w** (100 MHz/$CDCl_3$)



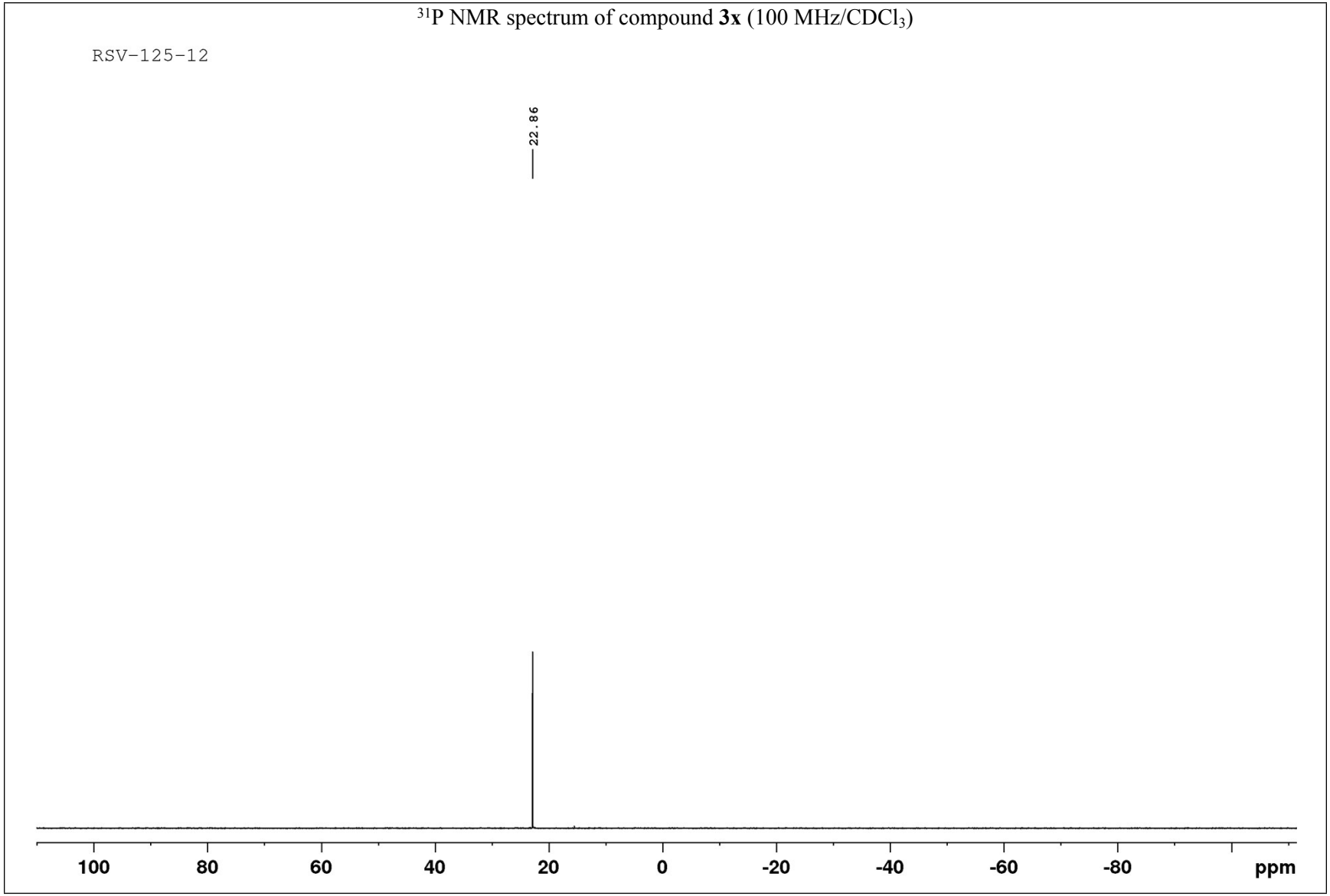
^{1}H NMR spectrum of compound **3x** (400 MHz/$CDCl_3$)



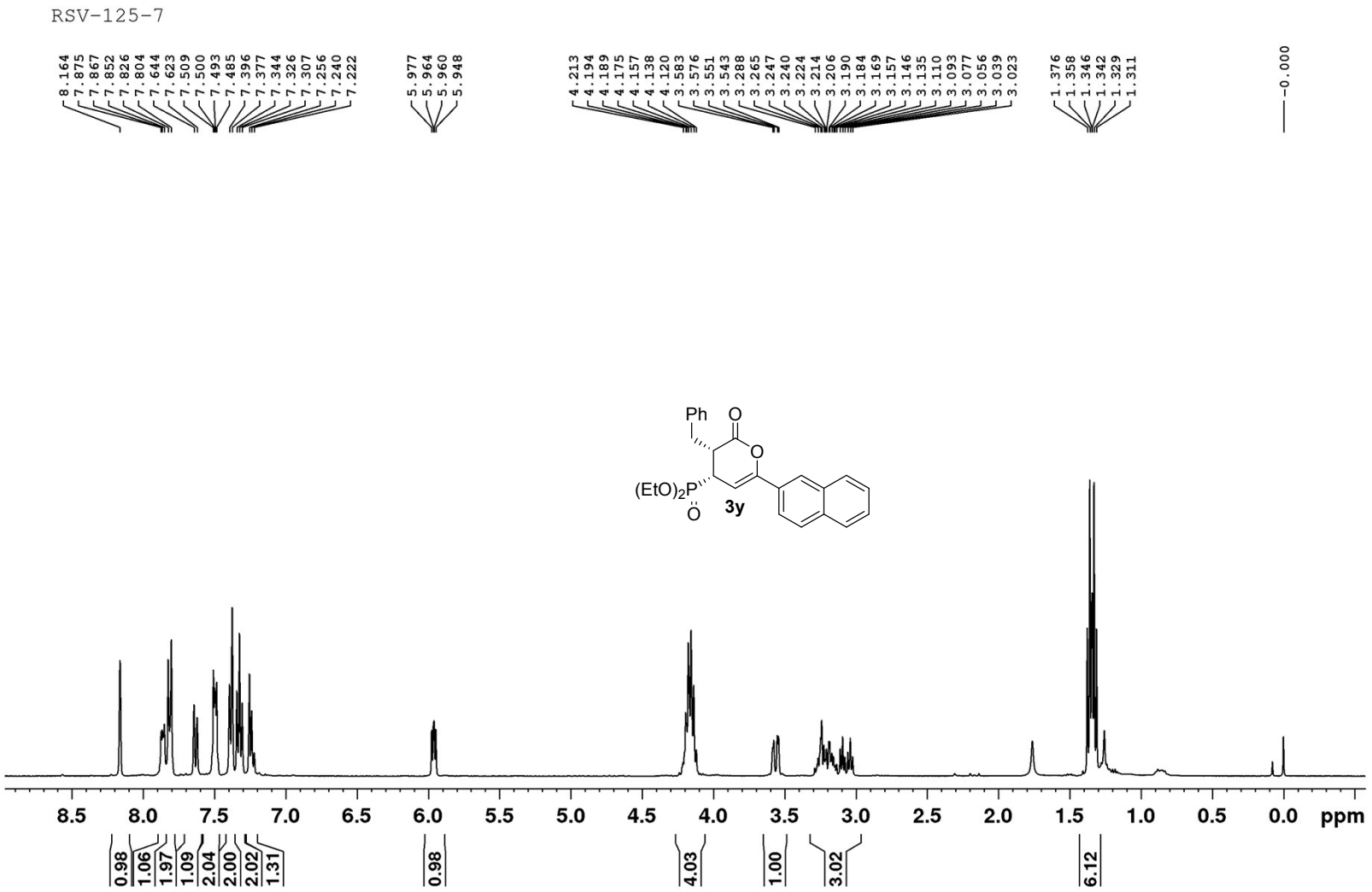
^{13}C NMR spectrum of compound **3x** (100 MHz/$CDCl_3$)



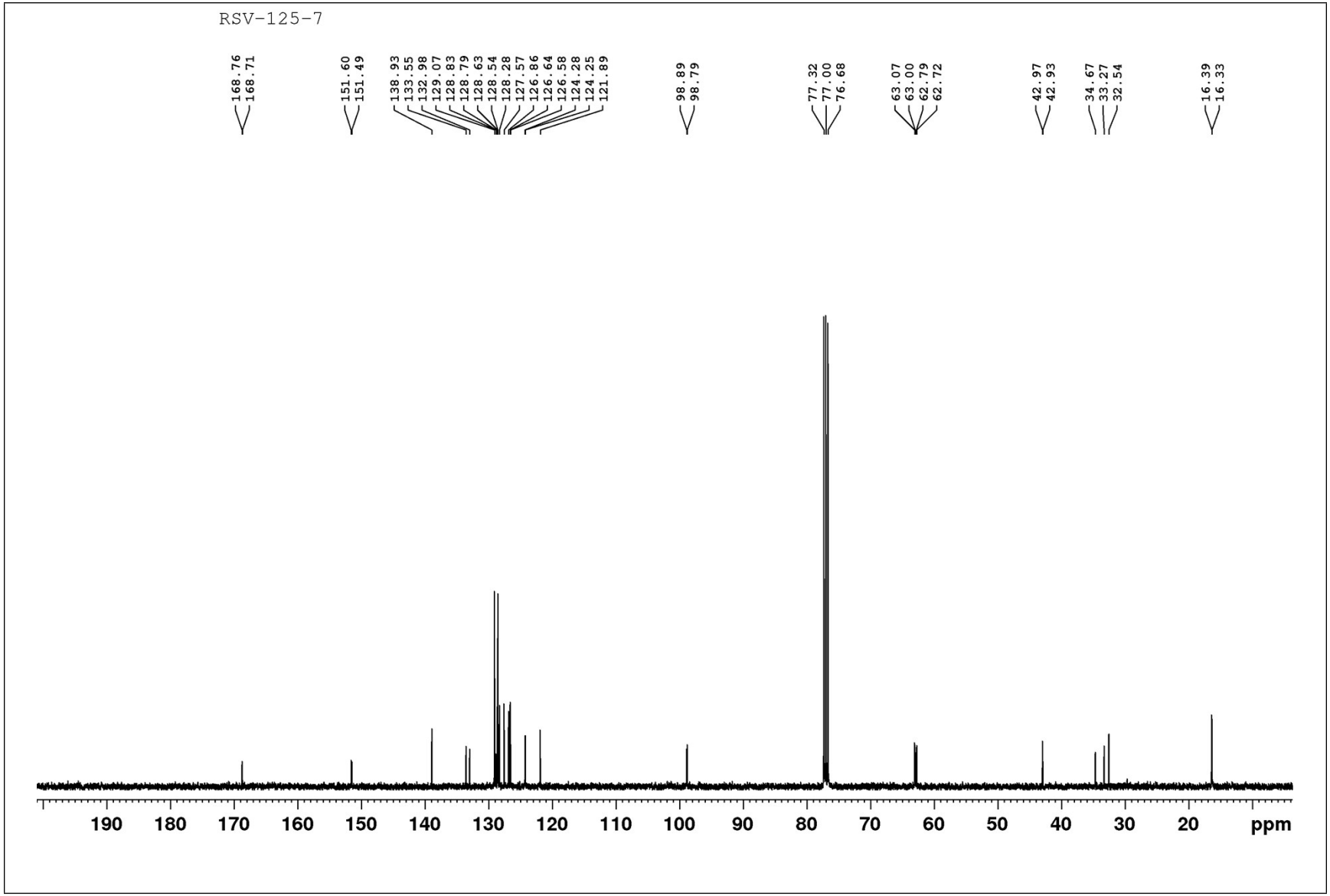
^{31}P NMR spectrum of compound **3x** (100 MHz/$CDCl_3$)



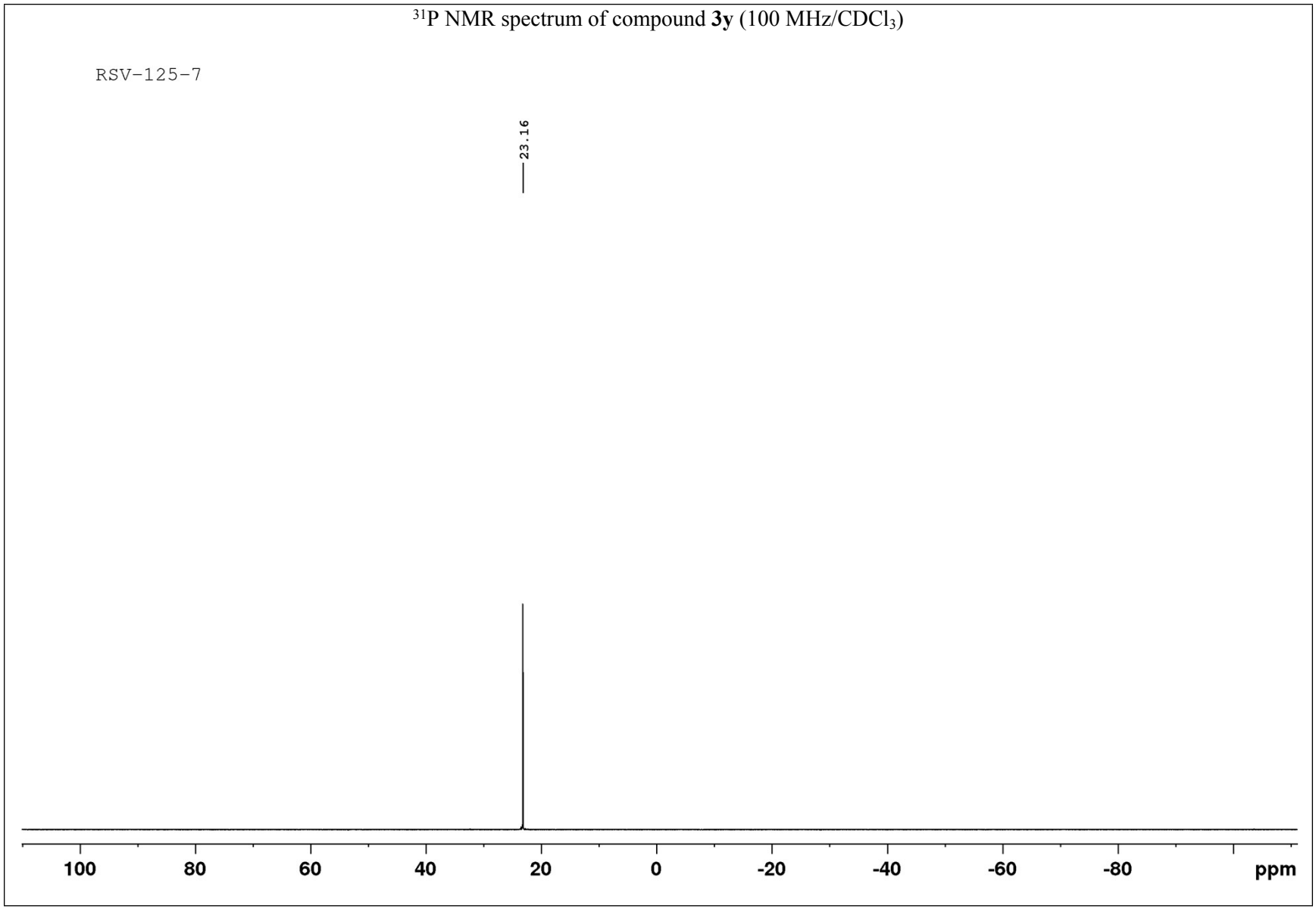
^{1}H NMR spectrum of compound **3y** (400 MHz/$CDCl_3$)



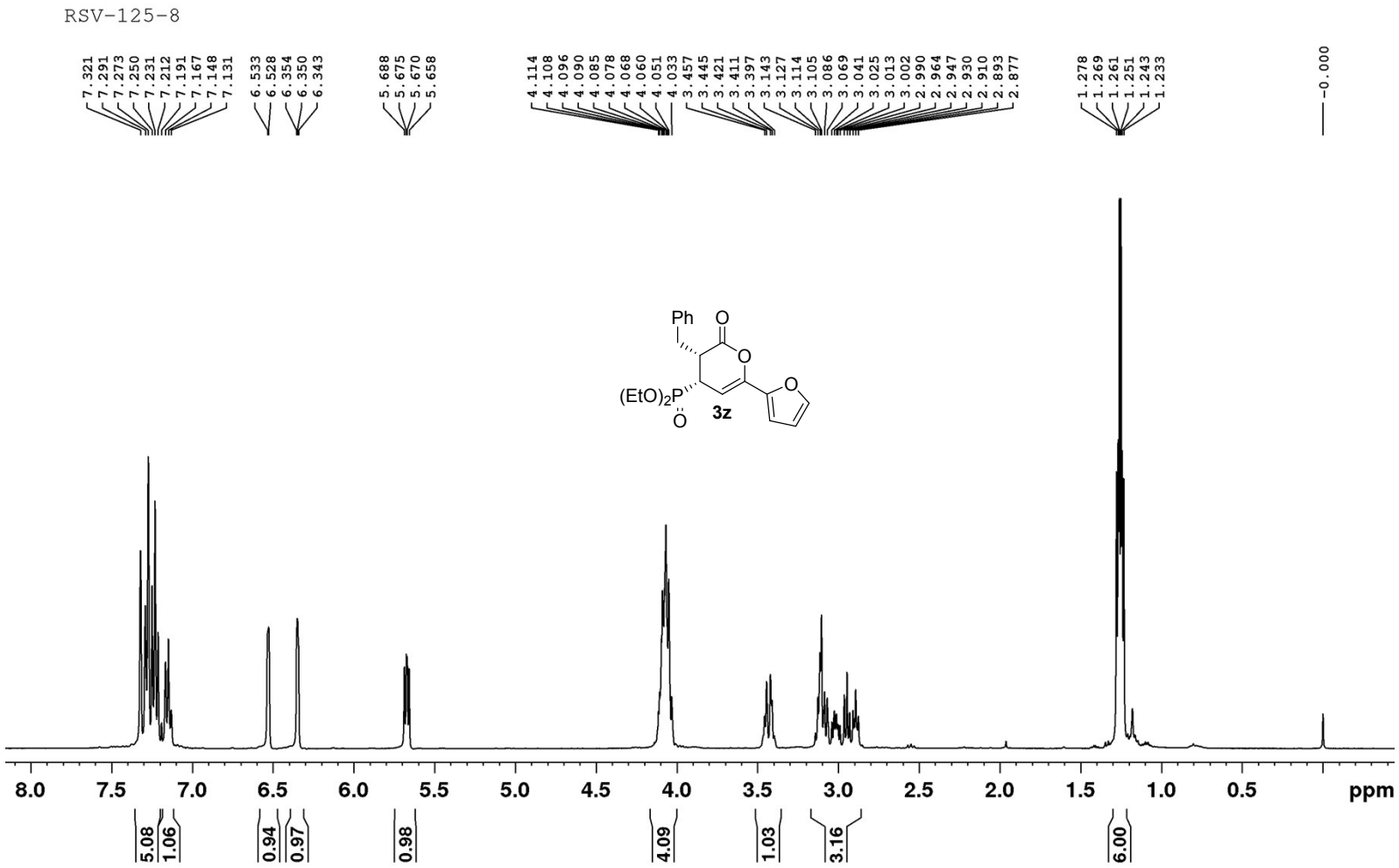
^{13}C NMR spectrum of compound **3y** (100 MHz/$CDCl_3$)


RSV-125-7
168.76
168.71
151.60
151.49
138.93
133.55
132.98
129.07
128.83
128.79
128.63
128.54
128.28
127.57
126.86
126.64
126.58
124.28
124.25
121.89
98.89
98.79
77.32
77.00
76.68
63.07
63.00
62.79
62.72
42.97
42.93
34.67
33.27
32.54
16.39
16.33
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

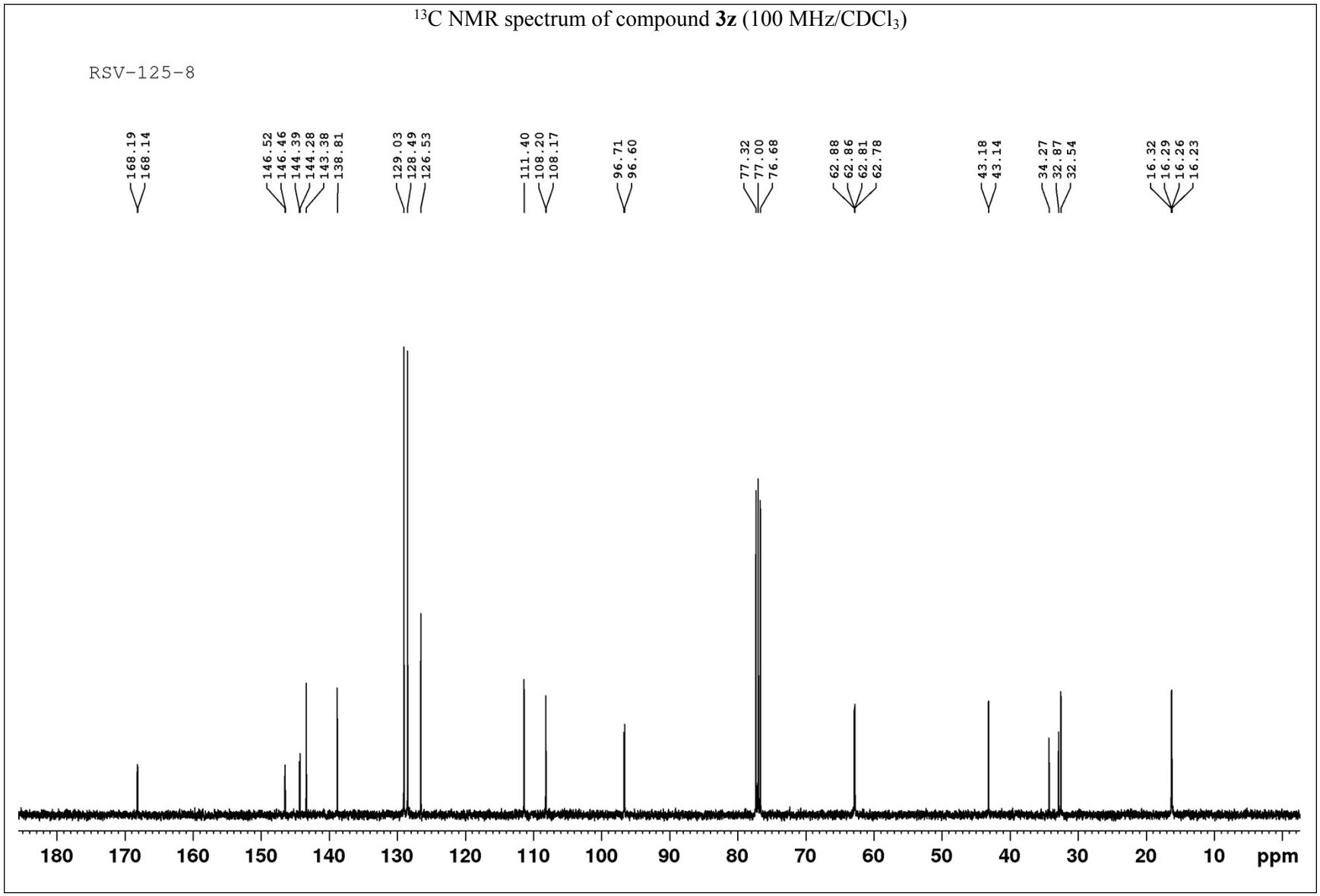
^{31}P NMR spectrum of compound **3y** (100 MHz/$CDCl_3$)



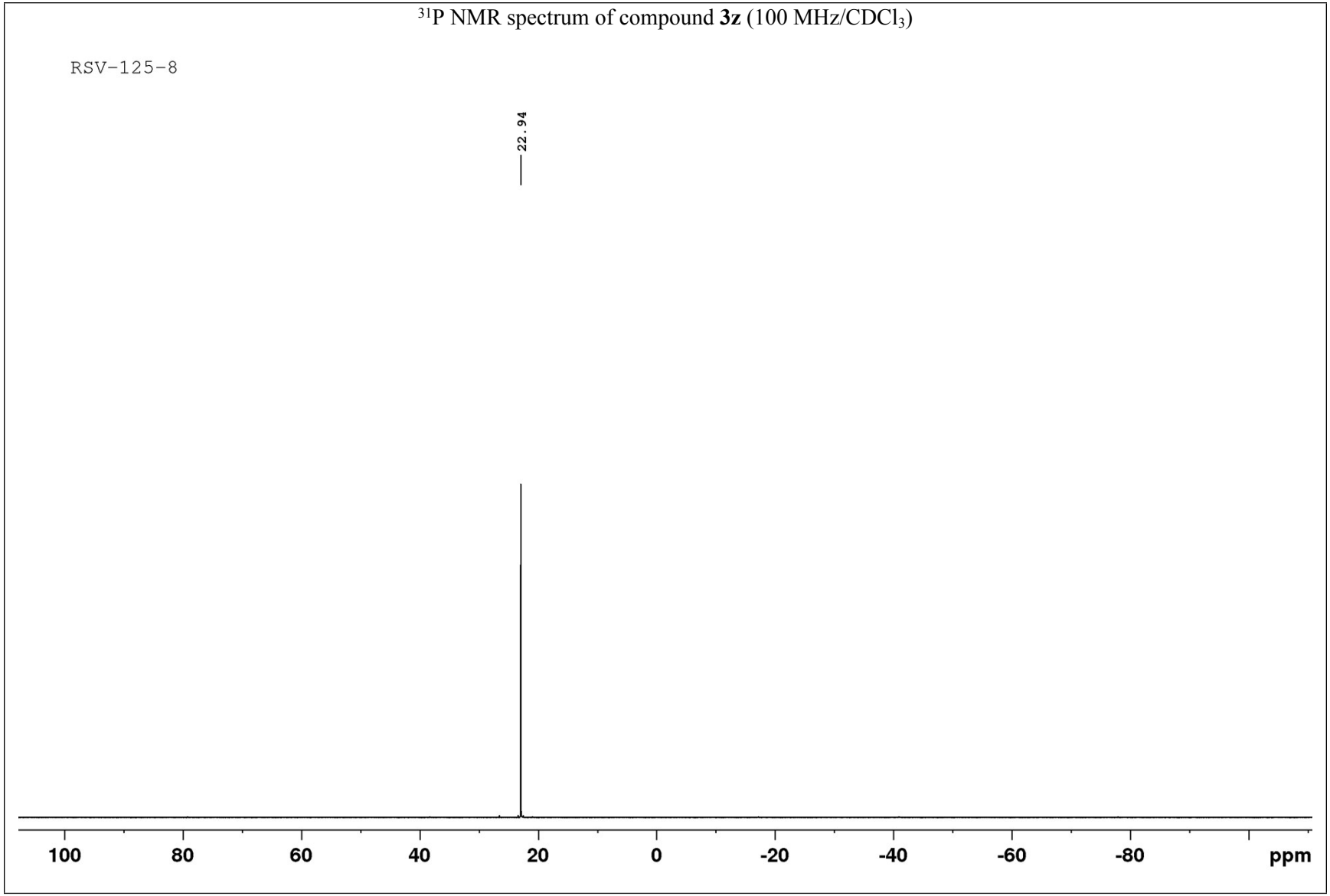
^{1}H NMR spectrum of compound **3z** (400 MHz/$CDCl_3$)



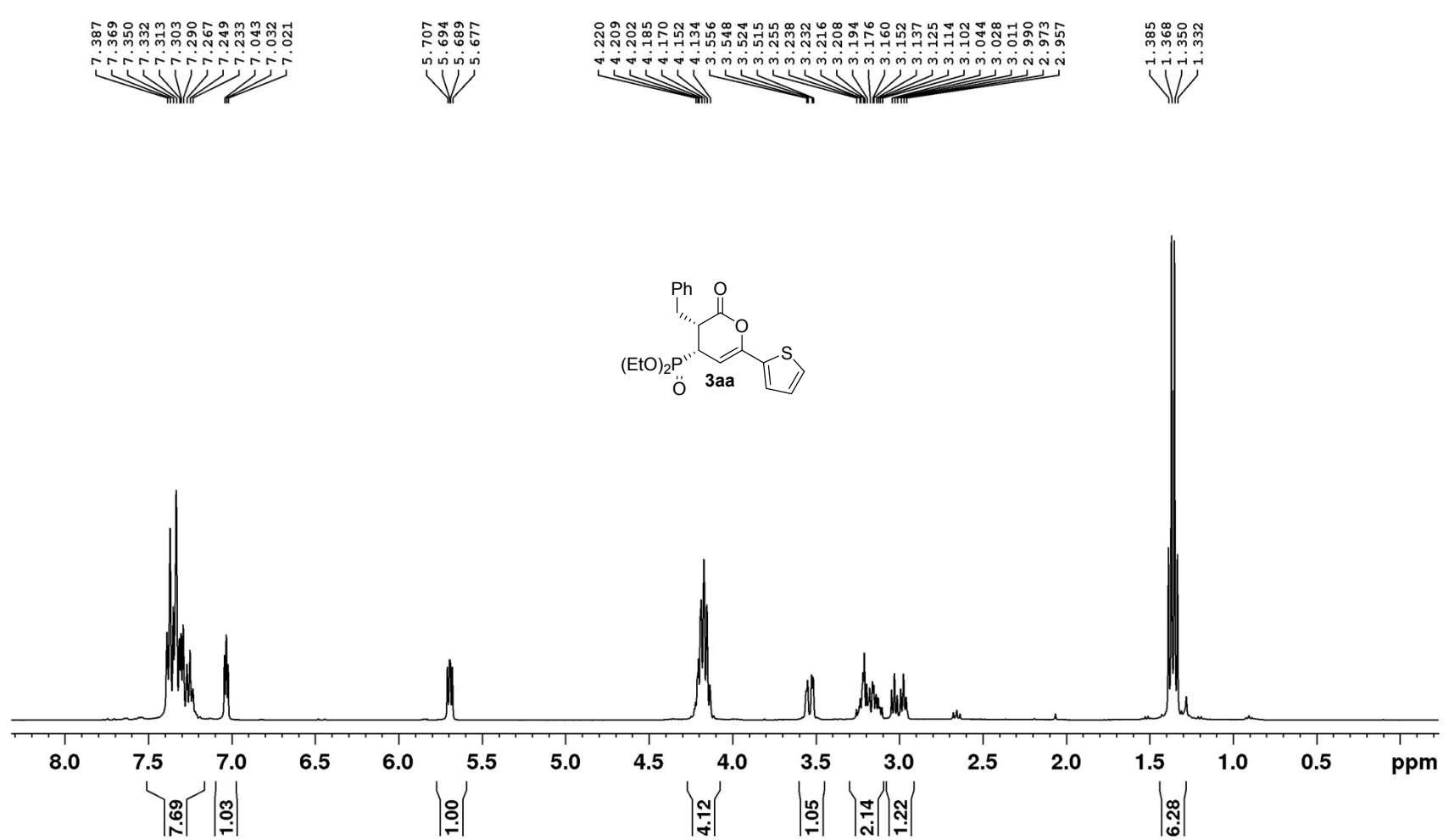
^{13}C NMR spectrum of compound **3z** (100 MHz/$CDCl_3$)



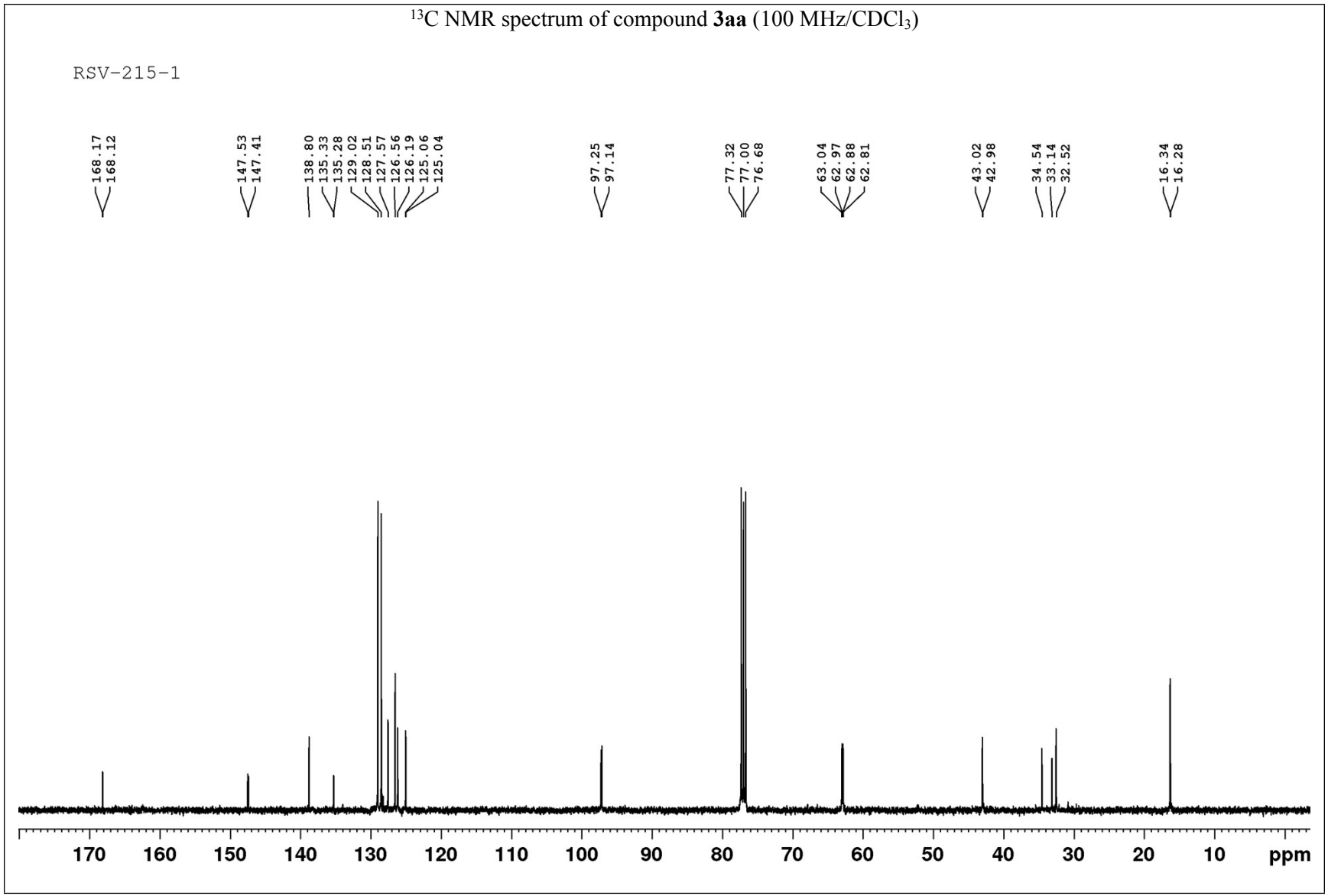
^{31}P NMR spectrum of compound **3z** (100 MHz/$CDCl_3$)



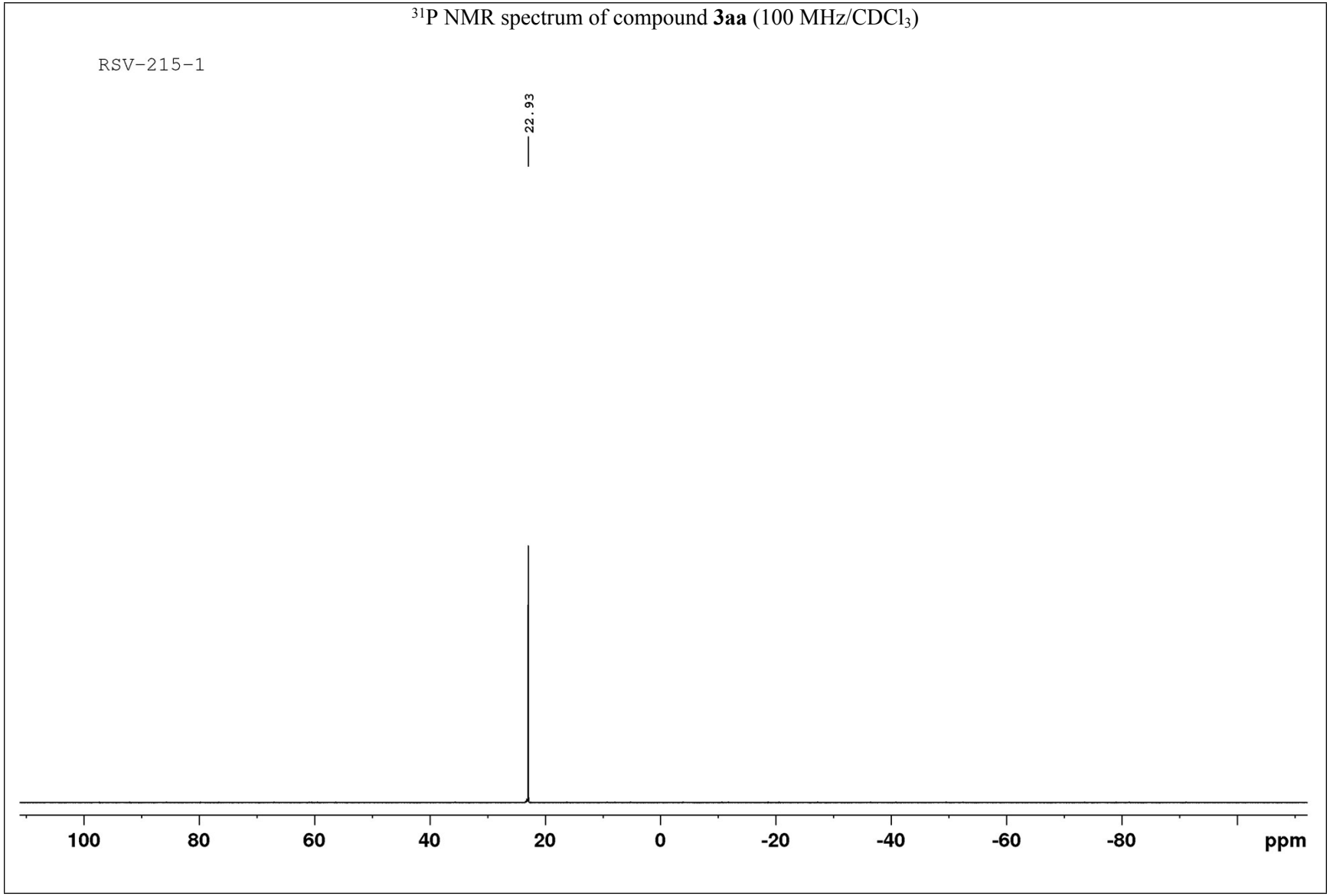
^{1}H NMR spectrum of compound **3aa** (400 MHz/$CDCl_3$)



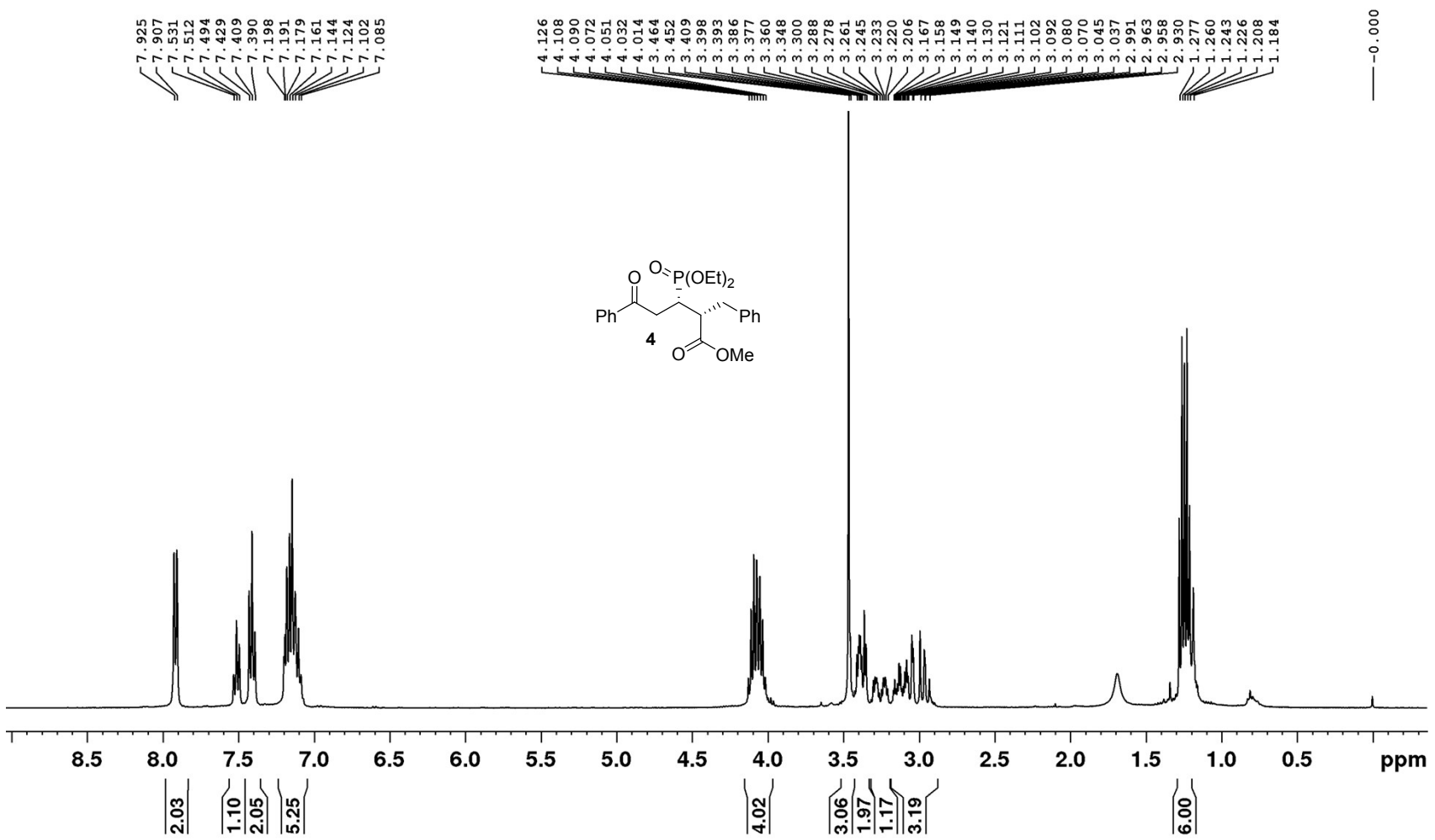
^{13}C NMR spectrum of compound **3aa** (100 MHz/$CDCl_3$)



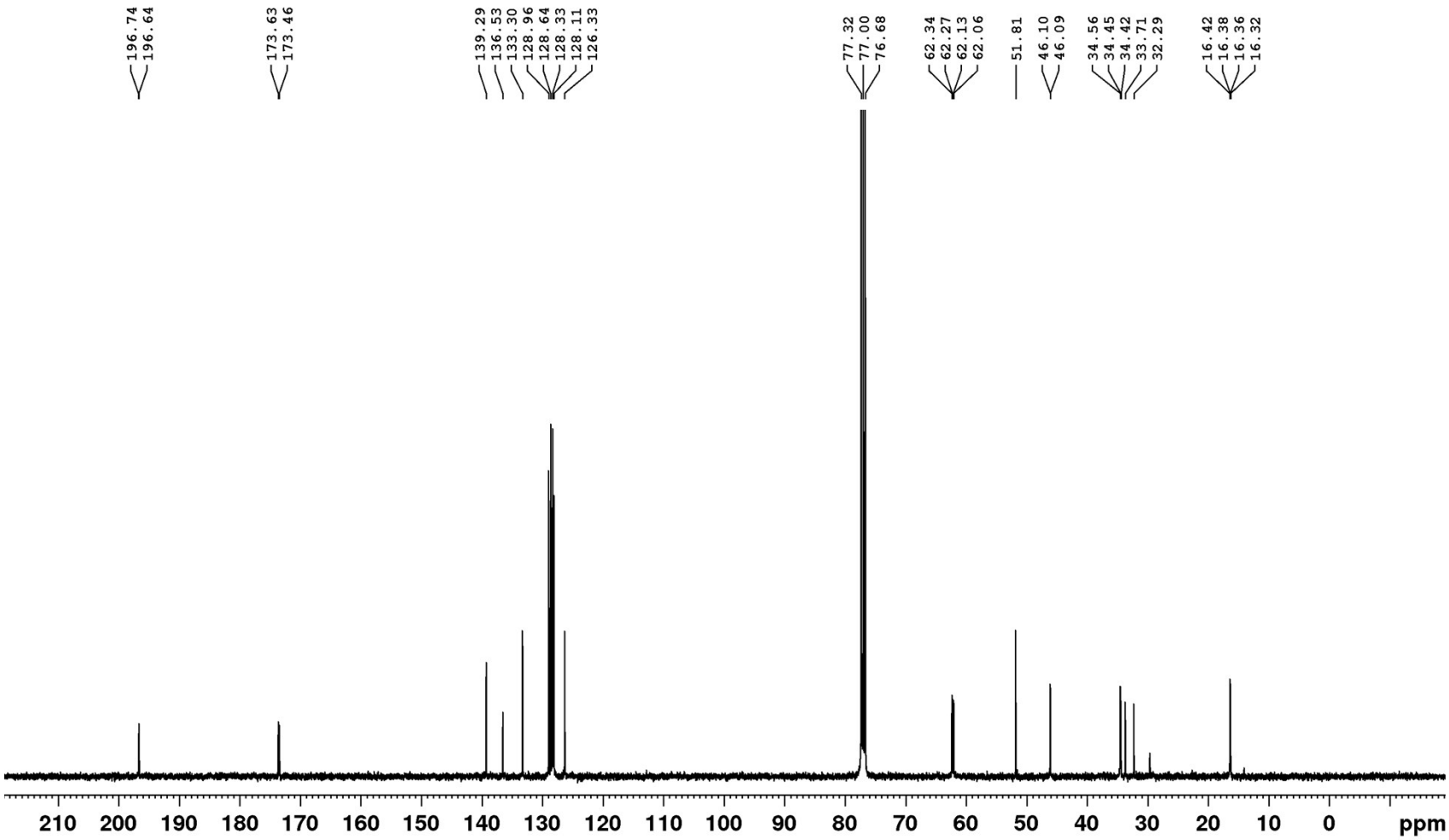
^{31}P NMR spectrum of compound **3aa** (100 MHz/$CDCl_3$)



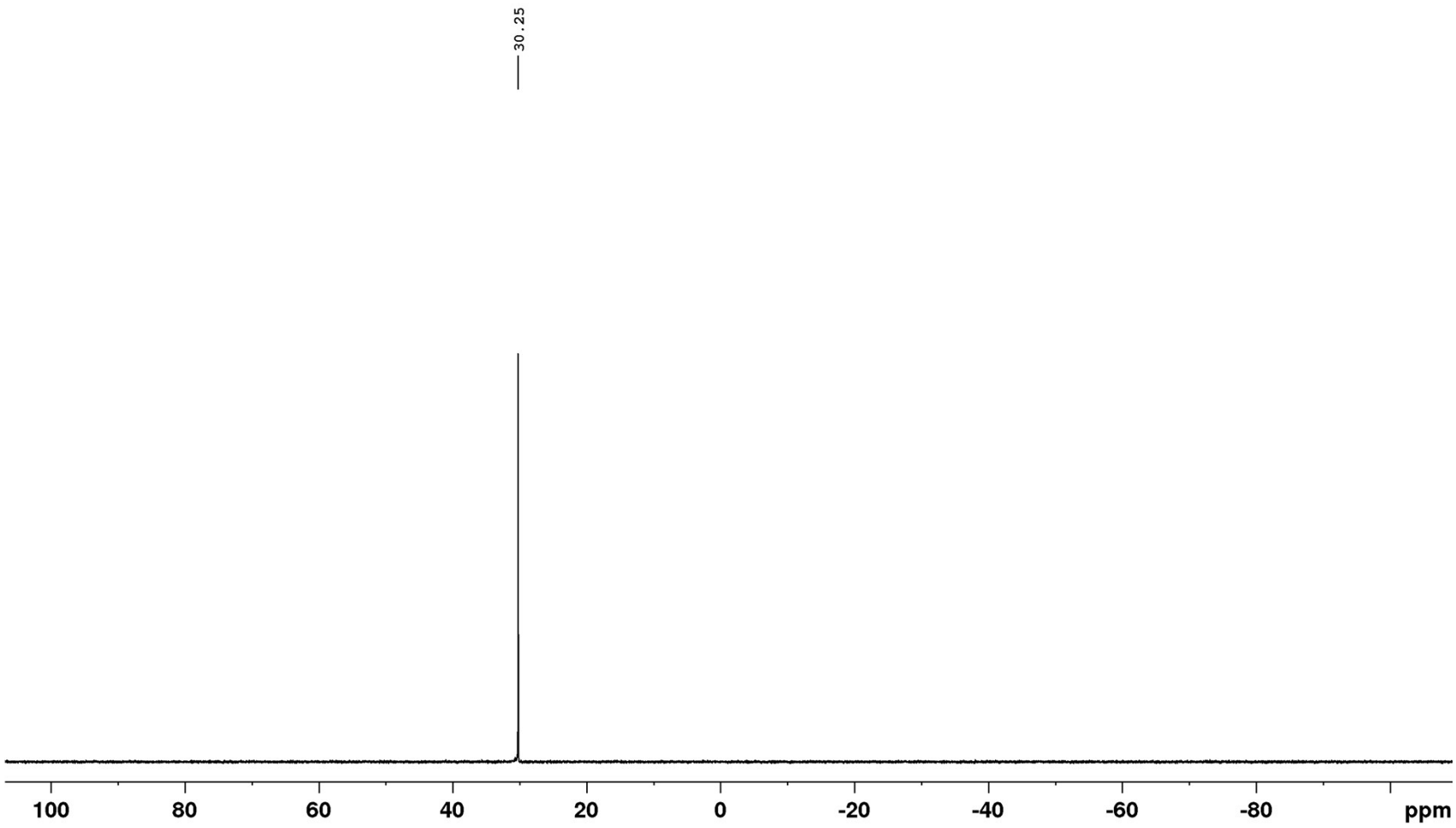
^{1}H NMR spectrum of compound **4** (400 MHz/$CDCl_3$)



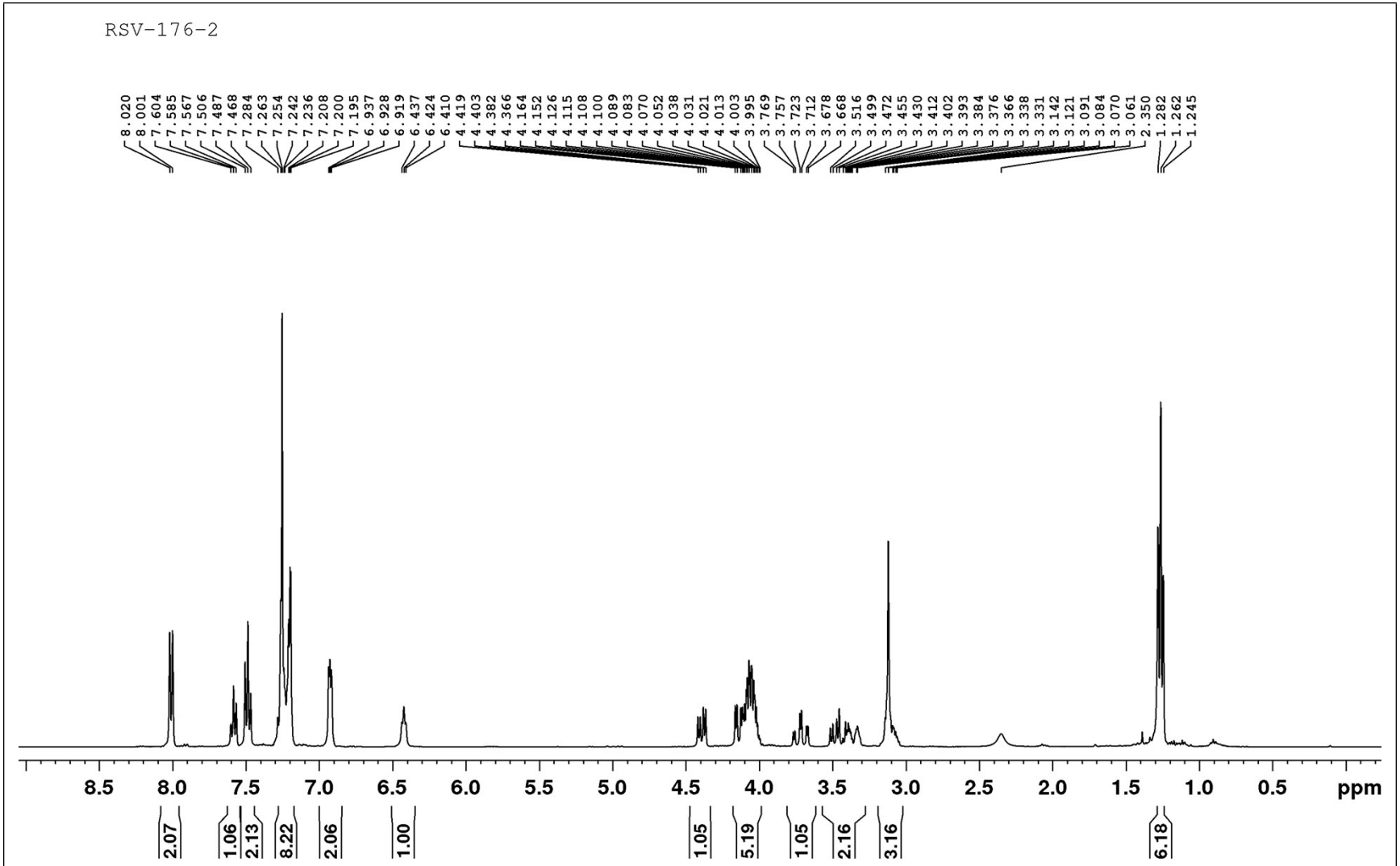
^{13}C NMR spectrum of compound **4** (100 MHz/$CDCl_3$)



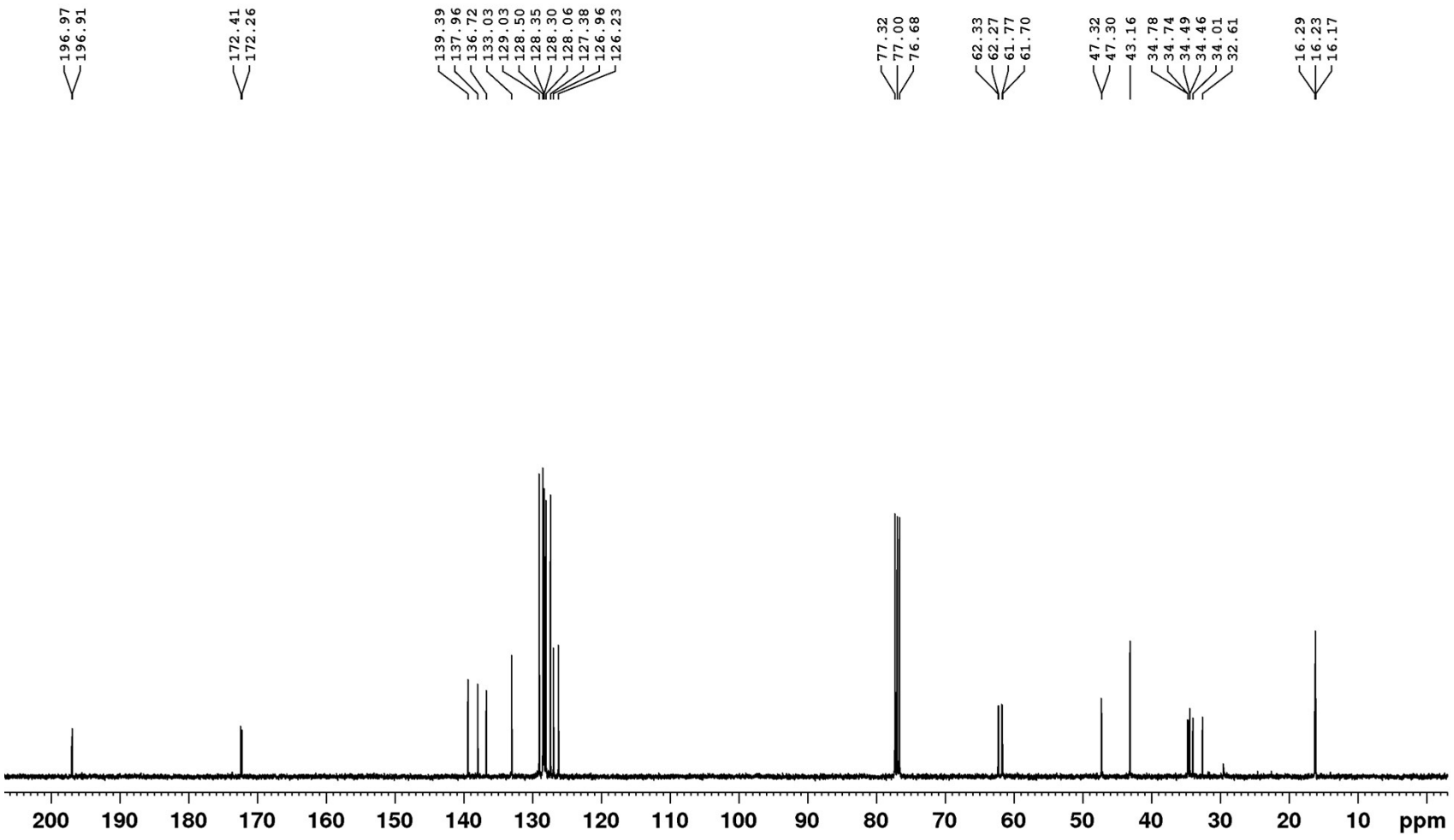
^{31}P NMR spectrum of compound **4** (100 MHz/$CDCl_3$)



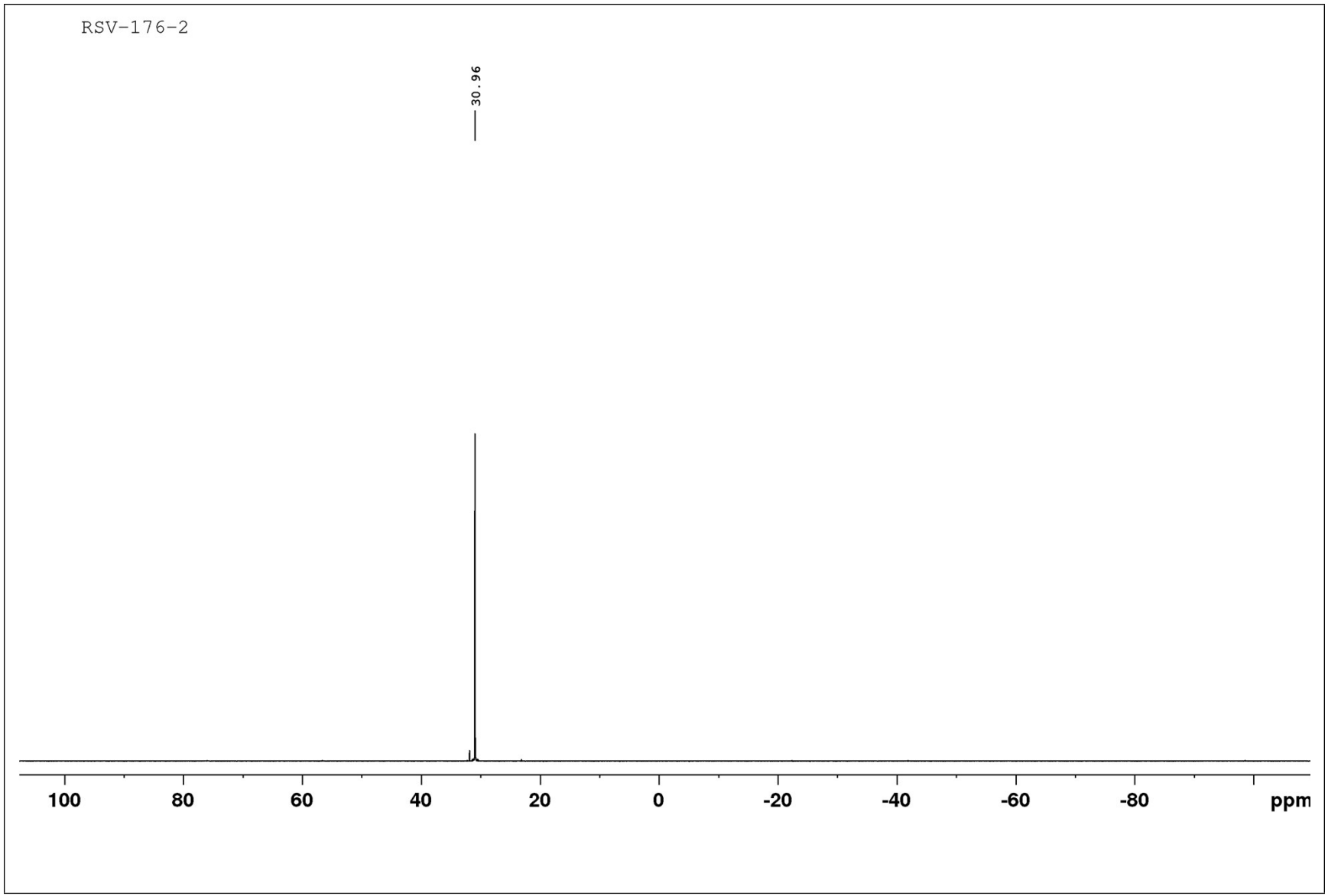
^{1}H NMR spectrum of compound **5** (400 MHz/$CDCl_3$)



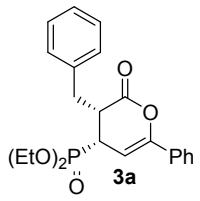
^{13}C NMR spectrum of compound **5** (100 MHz/$CDCl_3$)



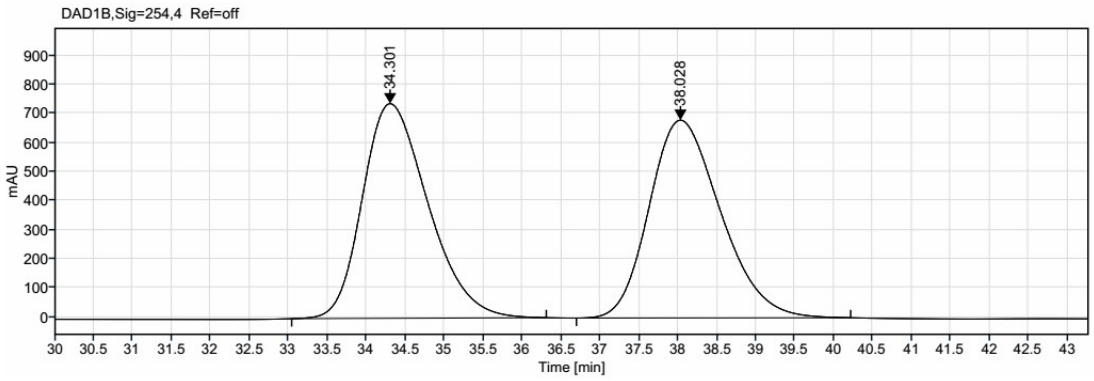
^{31}P NMR spectrum of compound **5** (100 MHz/$CDCl_3$)


RSV-176-2
30.96
100
80
60
40
20
0
-20
-40
-60
-80
ppm

HPLC Spectra:

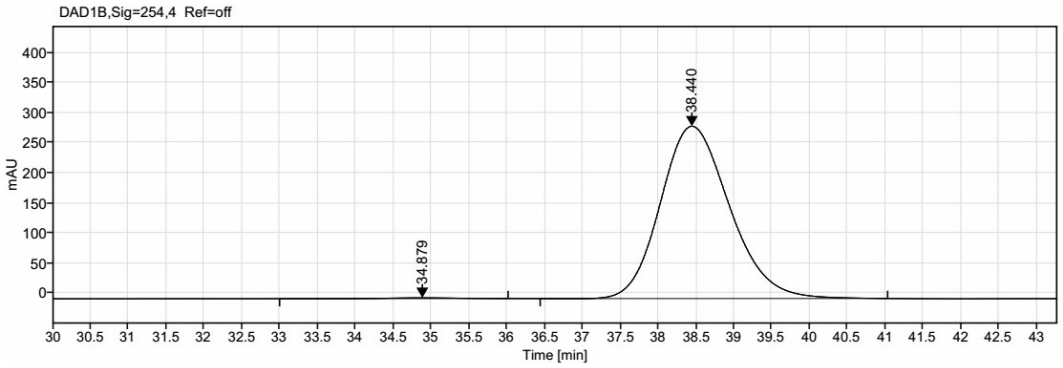


3a



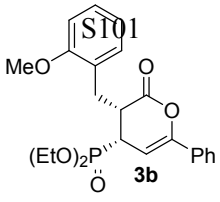
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
34.301	MM m	0.91	43209.50	738.91	49.88	
38.028	MM m	0.98	43413.15	680.96	50.12	
		Sum	86622.64			

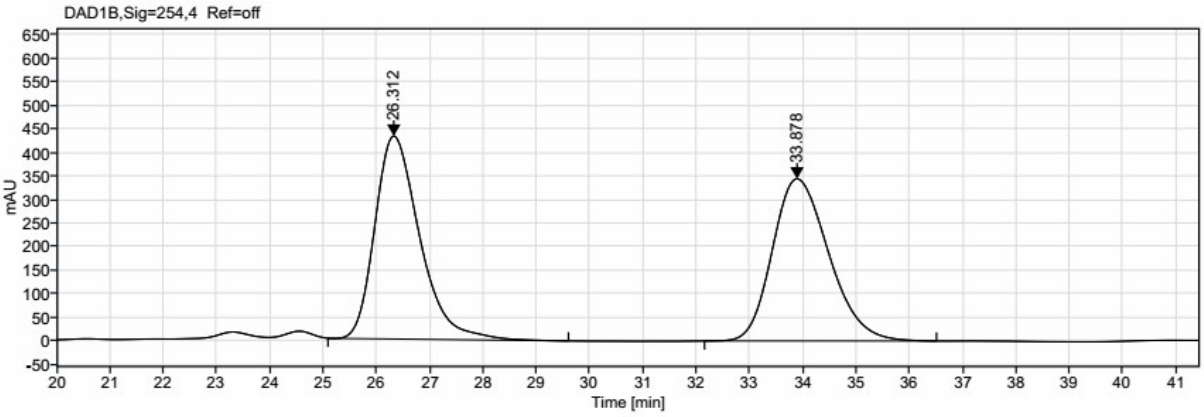


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
34.879	MM m	0.75	78.08	1.24	0.43	
38.440	MM m	0.98	18195.35	286.36	99.57	
		Sum	18273.42			

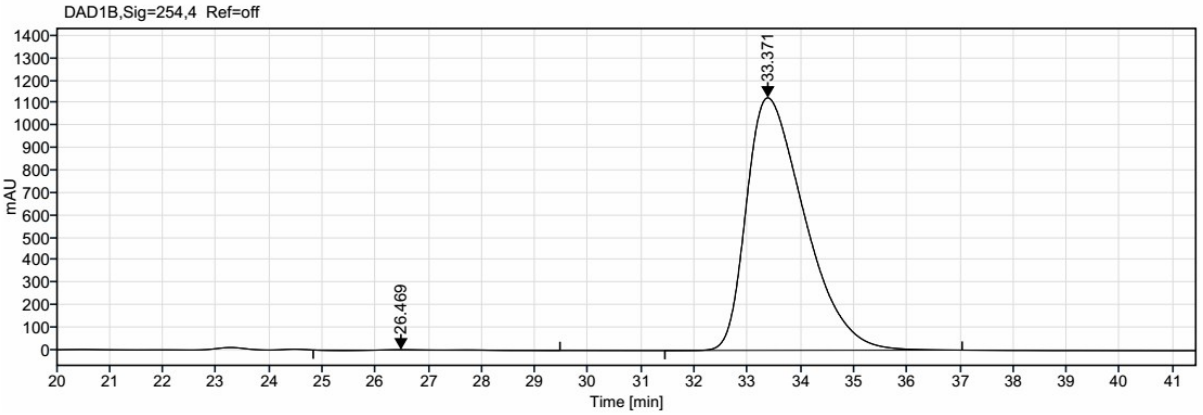


3b



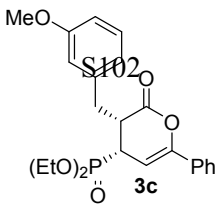
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
26.312	MM m	0.89	25121.64	430.56	49.83	
33.878	MM m	1.15	25297.07	344.03	50.17	
		Sum	50418.70			

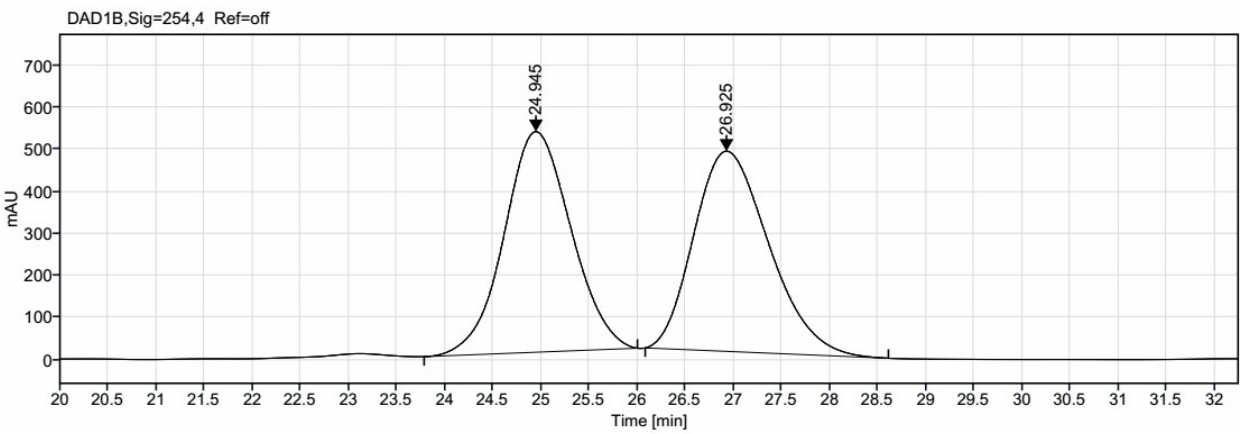


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
26.469	MM m	0.41	52.24	1.93	0.06	
33.371	MM m	1.13	85684.97	1125.35	99.94	
		Sum	85737.21			

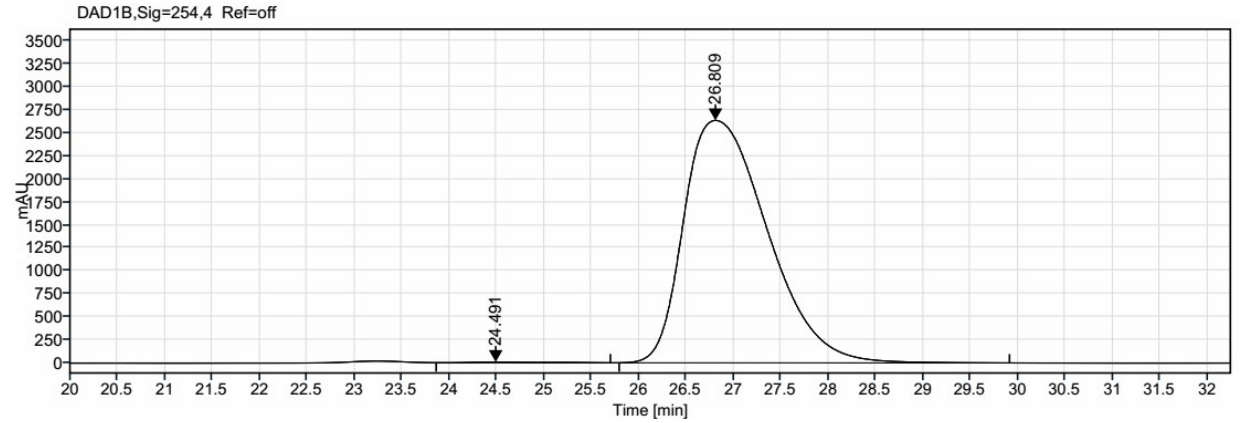


3c



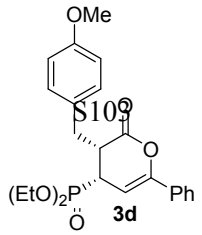
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
24.945	MM m	0.74	25319.71	524.07	49.57	
26.925	MM m	0.84	25763.75	475.61	50.43	
		Sum	51083.45			

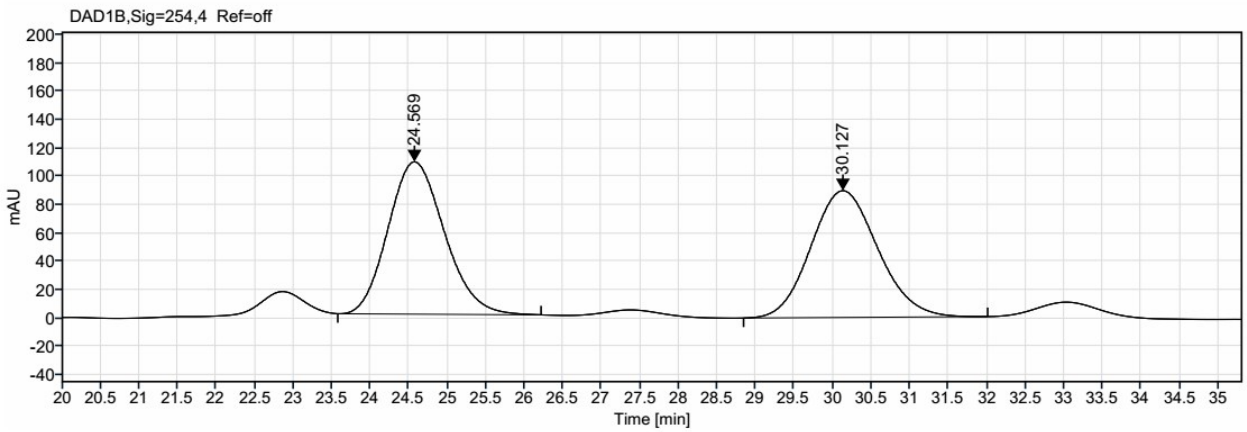


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
24.491	MM m	0.67	414.05	8.61	0.26	
26.809	MM m	0.96	161834.10	2638.56	99.74	
		Sum	162248.15			

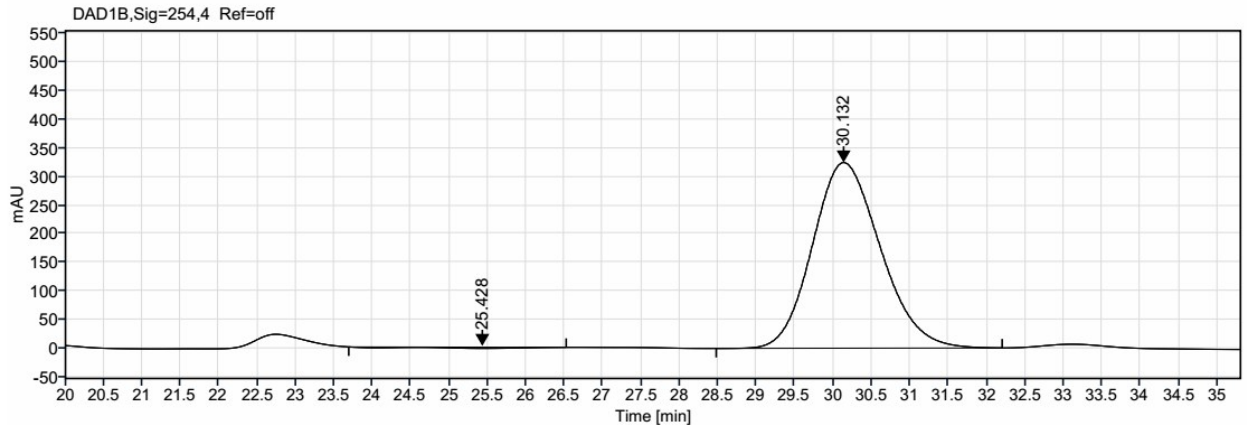


3d



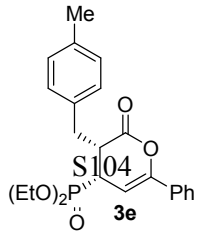
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
24.569	MM m	0.79	5457.04	107.47	50.30	
30.127	MM m	0.94	5391.83	89.41	49.70	
		Sum	10848.88			

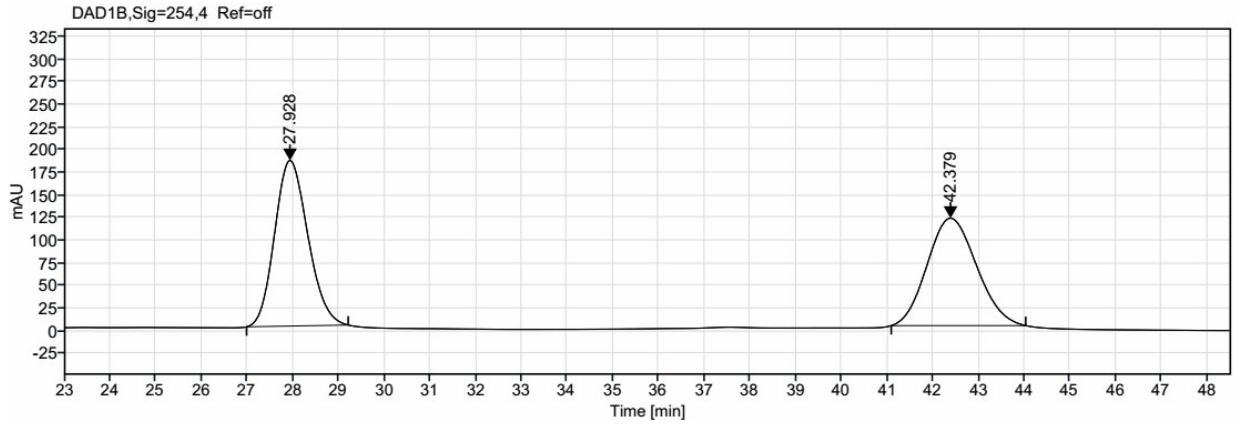


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
25.428	MM n	1.07	189.14	2.13	0.94	
30.132	MM m	0.96	19862.21	322.97	99.06	
		Sum	20051.36			

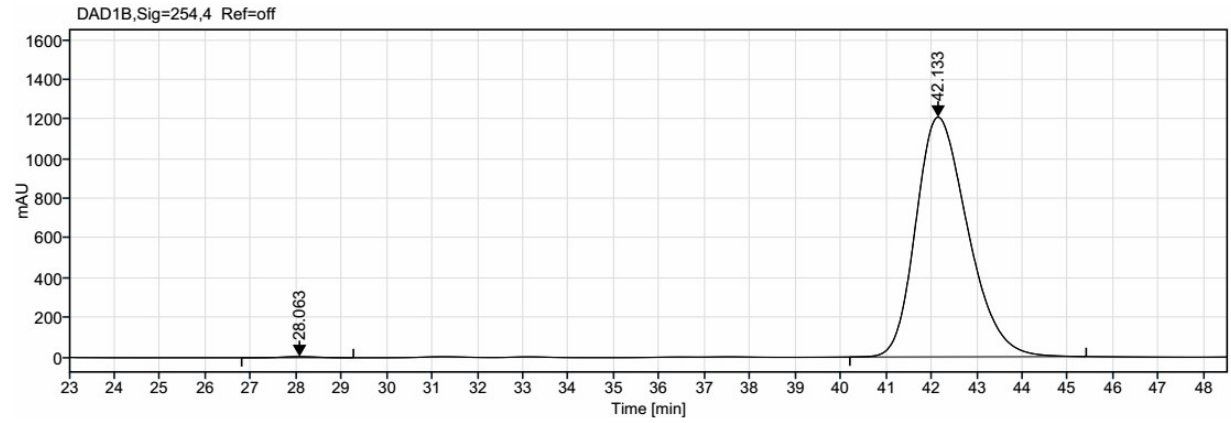


3e



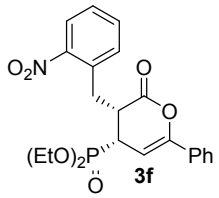
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
27.928	MM m	0.79	9239.77	182.64	50.95	
42.379	MM m	1.16	8895.07	118.47	49.05	
		Sum	18134.84			

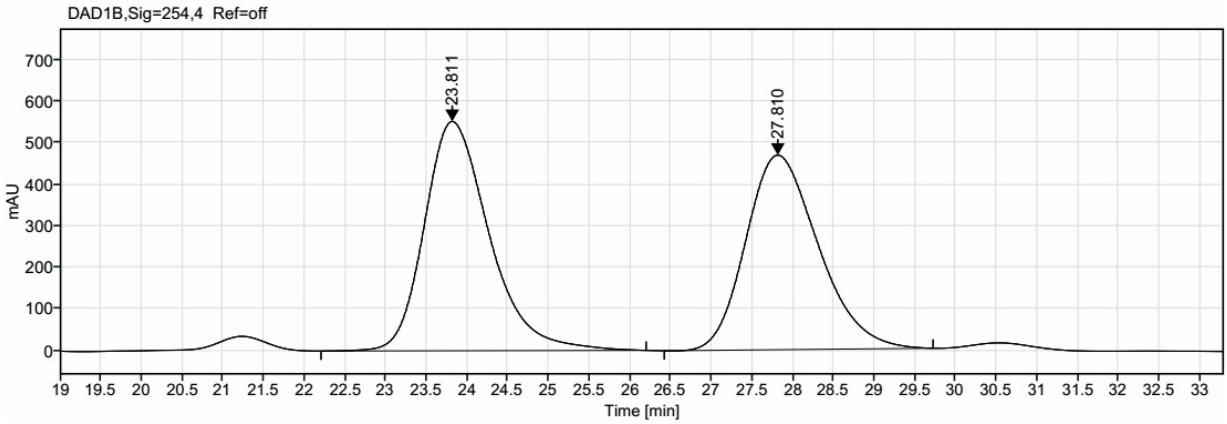


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
28.063	MM m	0.73	282.83	5.33	0.29	
42.133	MM m	1.22	95872.30	1207.48	99.71	
		Sum	96155.13			

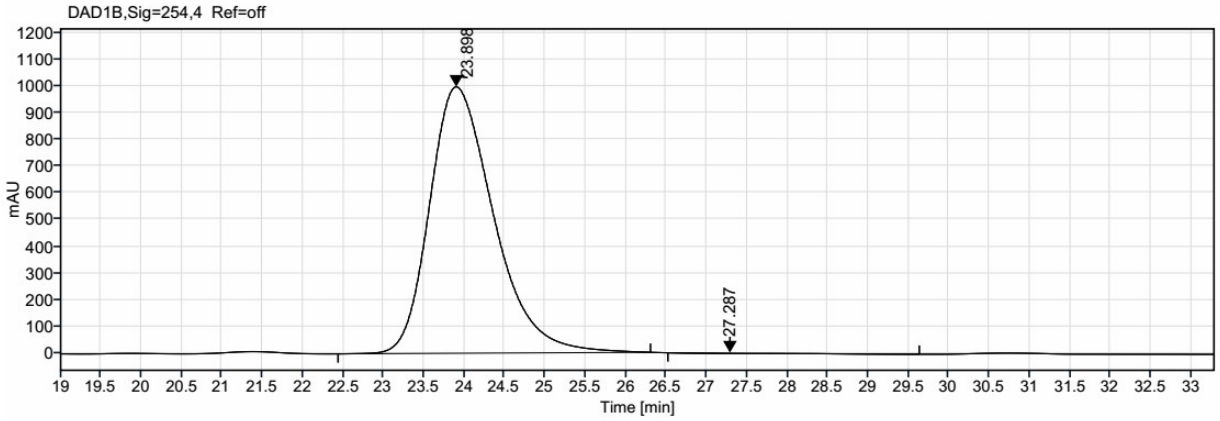


3f



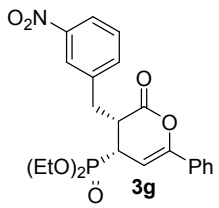
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
23.811	BM m	0.85	30537.63	551.59	51.31	
27.810	MM m	0.94	28976.78	467.89	48.69	
		Sum	59514.41			

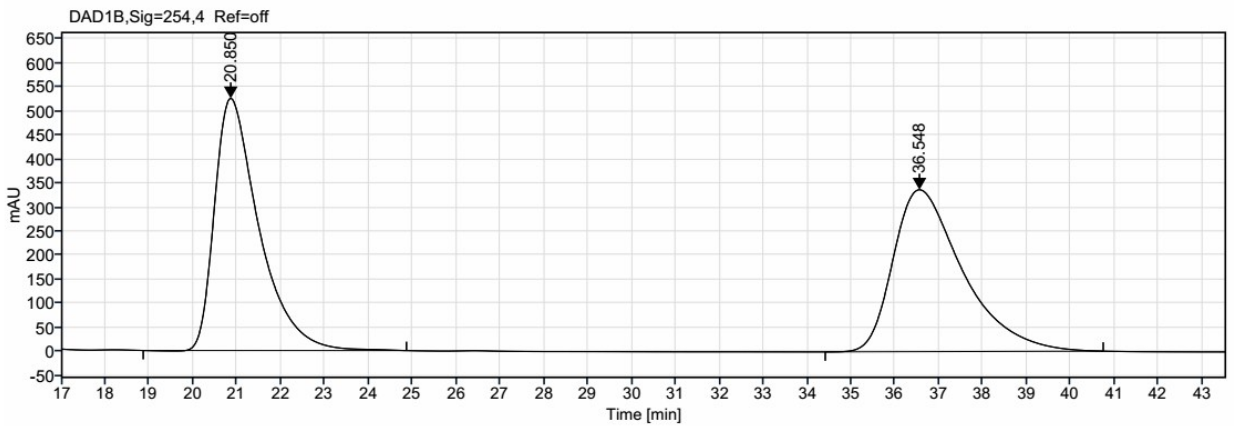


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
23.898	MM m	0.85	54910.62	996.56	99.74	
27.287	MM n	1.40	142.57	1.70	0.26	
		Sum	55053.20			

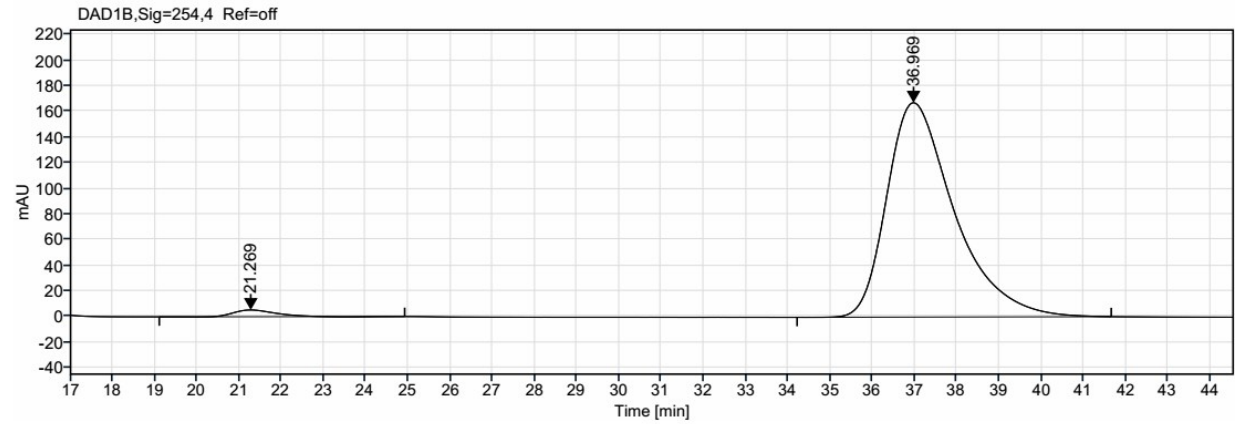


3g



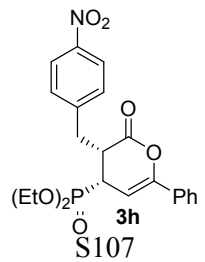
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
20.850	MM m	1.04	36762.71	523.93	49.77	
36.548	MM m	1.63	37096.34	336.55	50.23	
		Sum	73859.05			

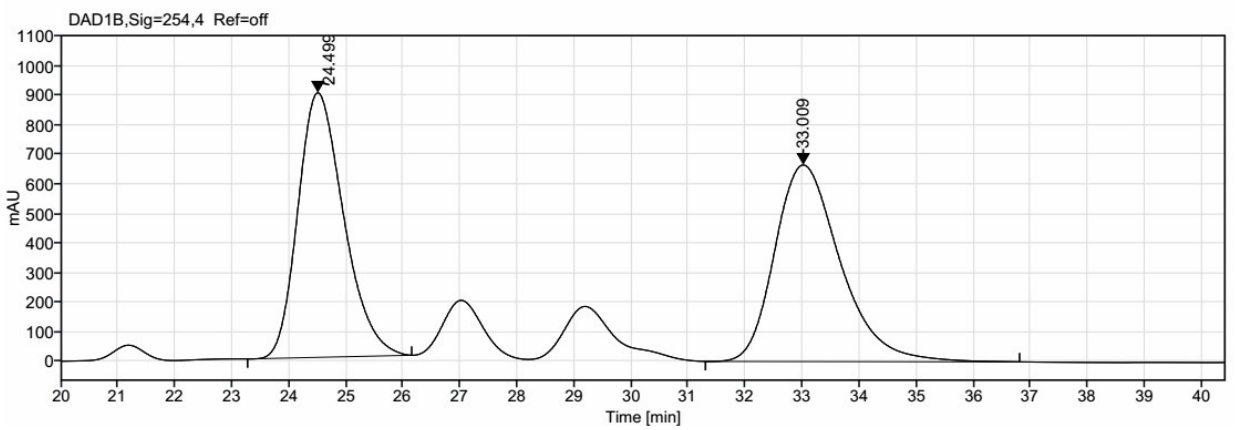


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
21.269	MM m	0.89	348.99	5.27	1.85	
36.969	MM m	1.61	18554.21	167.43	98.15	
		Sum	18903.20			

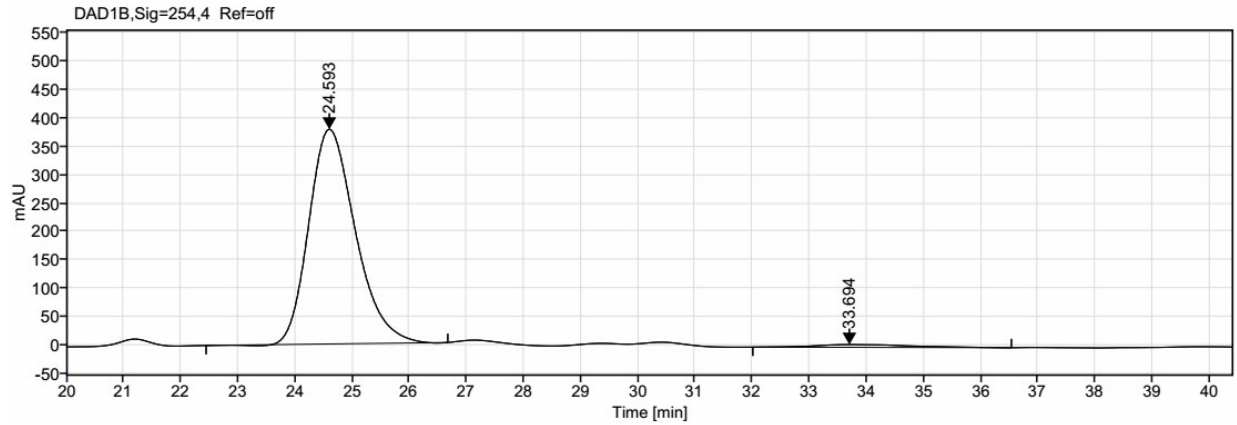


3h



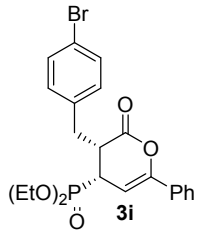
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
24.499	MM m	0.86	49877.90	894.50	48.47	
33.009	MM m	1.22	53021.73	664.65	51.53	
		Sum	102899.63			

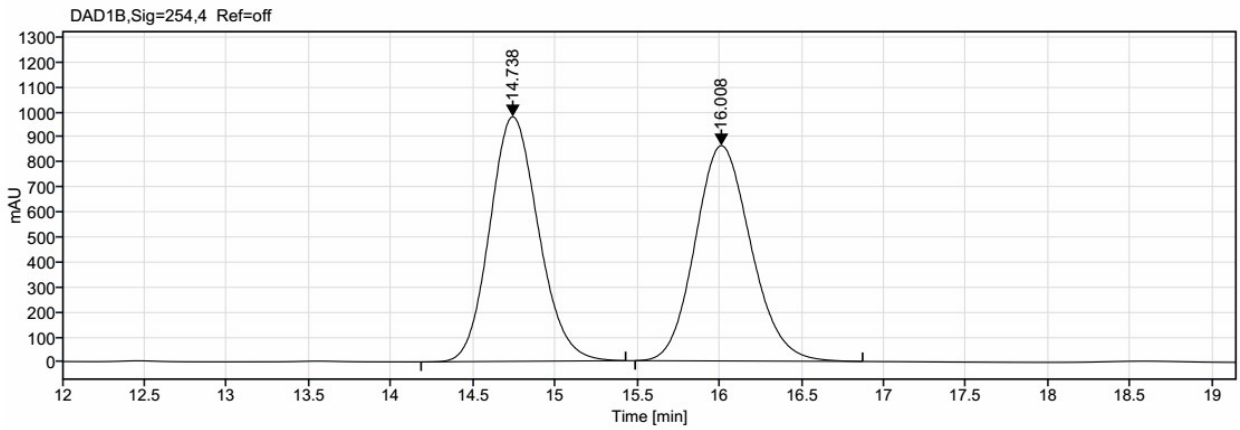


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
24.593	MM m	0.86	21266.89	376.54	97.38	
33.694	MM m	1.40	572.21	4.82	2.62	
		Sum	21839.09			

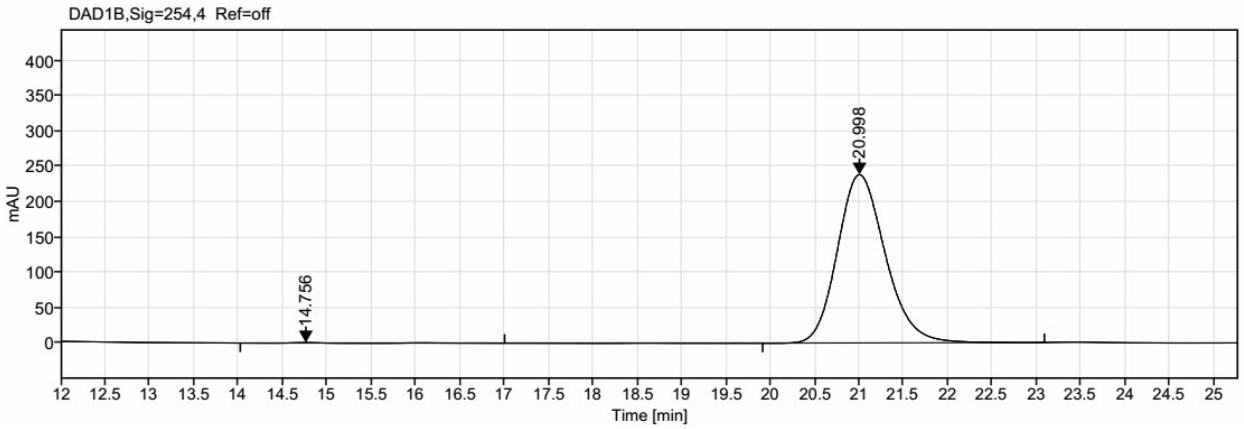


3i



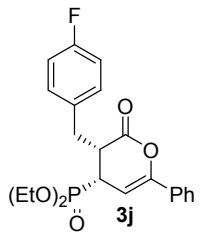
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
14.738	MM m	0.32	20158.38	978.19	49.81	
16.008	MM m	0.37	20310.35	860.74	50.19	
		Sum	40468.73			

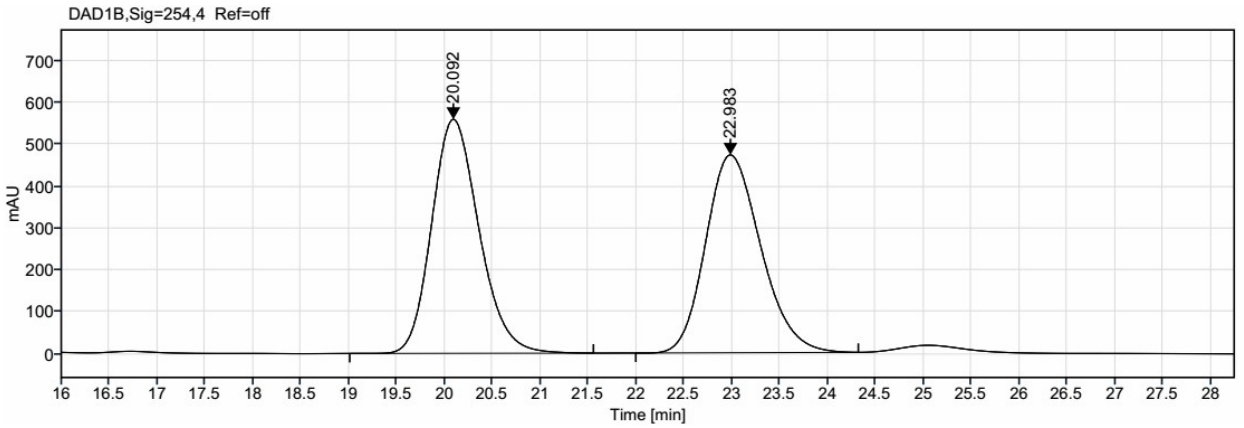


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
14.756	MM m	0.33	14.43	0.63	0.16	
20.998	MM m	0.57	8935.34	238.24	99.84	
		Sum	8949.77			

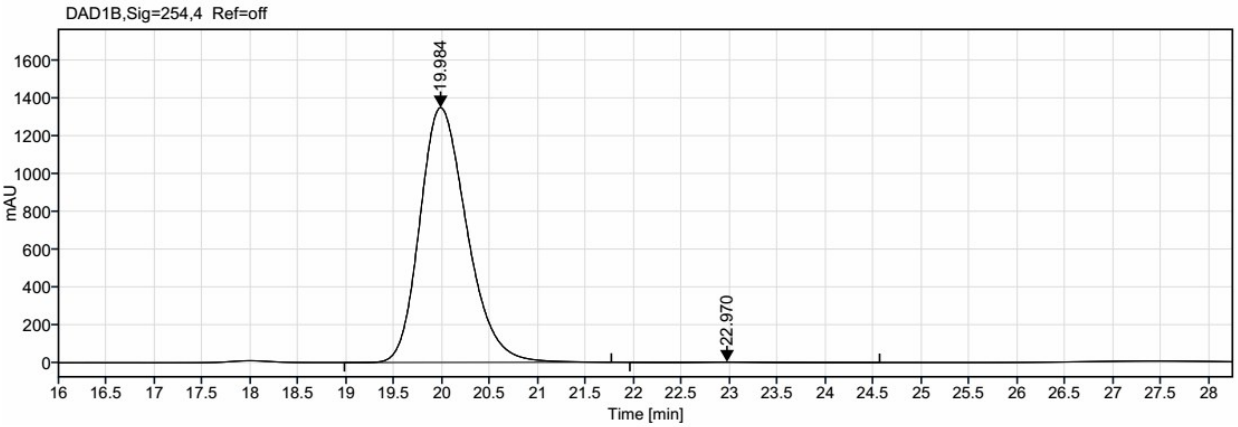


3j



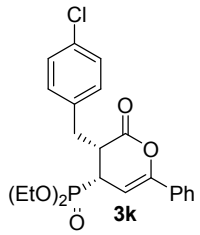
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
20.092	MM m	0.53	19139.80	558.05	50.35	
22.983	MM m	0.62	18870.71	471.82	49.65	
		Sum	38010.50			

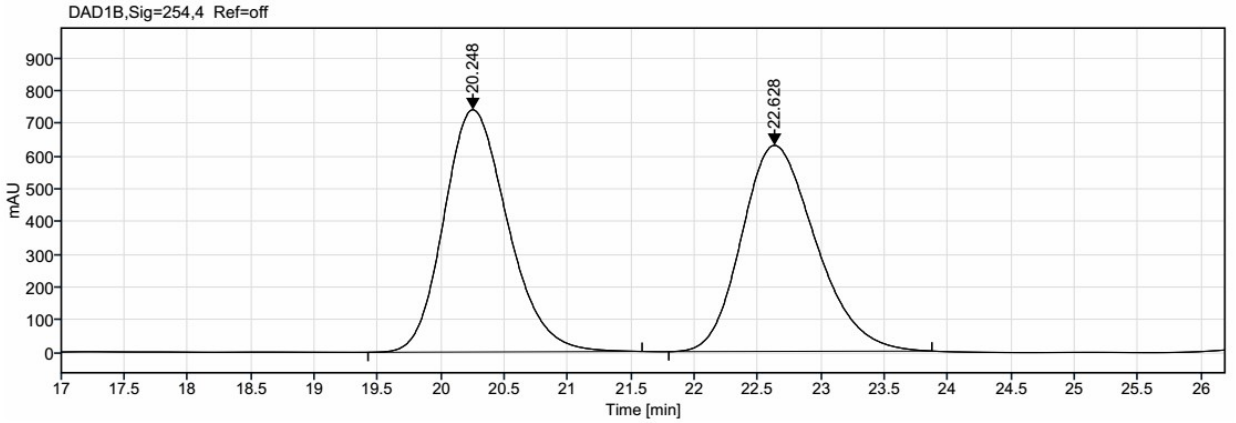


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
19.984	MM m	0.53	45910.52	1347.50	99.88	
22.970	MM m	0.35	56.63	2.32	0.12	
		Sum	45967.15			

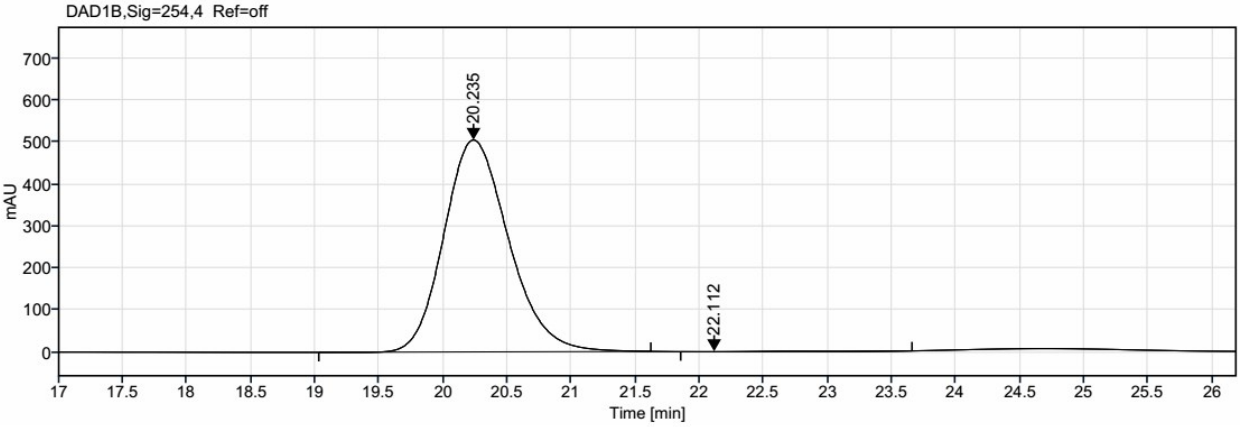


3k



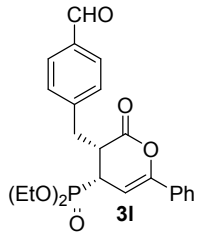
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
20.248	MM m	0.54	25883.17	739.98	50.31	
22.628	MM m	0.62	25567.21	629.27	49.69	
		Sum	51450.37			

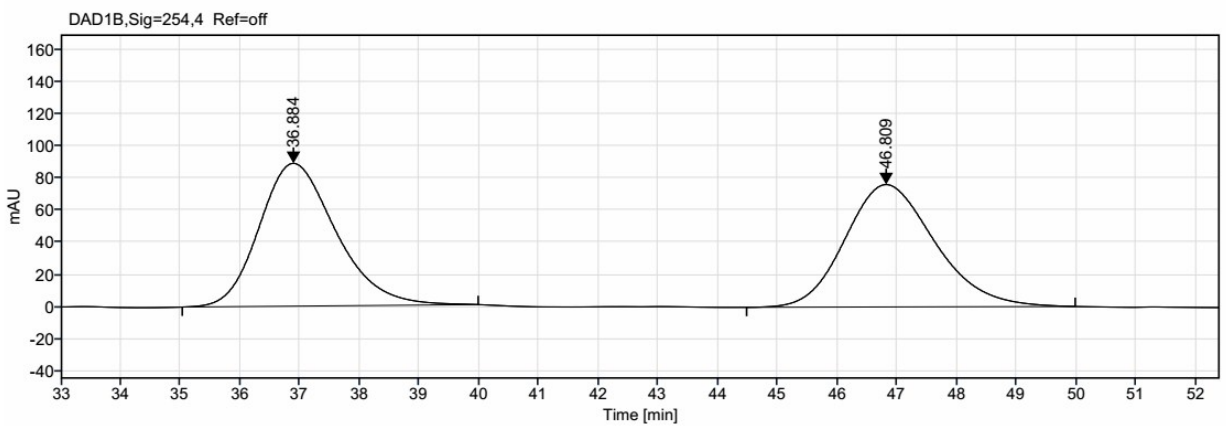


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
20.235	MM m	0.54	17682.77	504.24	99.67	
22.112	MM n	1.27	58.50	0.57	0.33	
		Sum	17741.27			

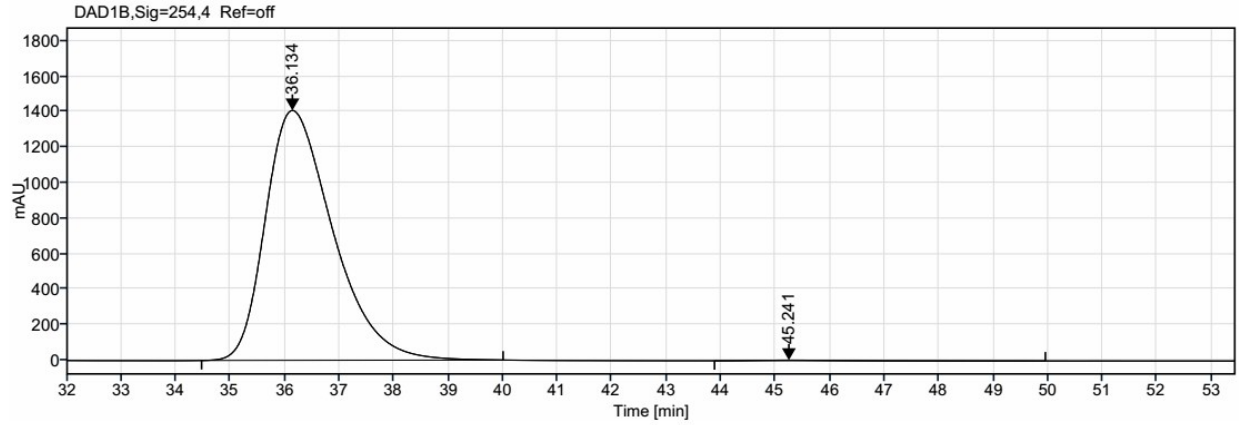


3l



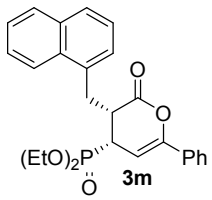
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
36.884	MM m	1.35	7902.77	88.62	49.91	
46.809	MM m	1.57	7931.83	75.96	50.09	
		Sum	15834.60			

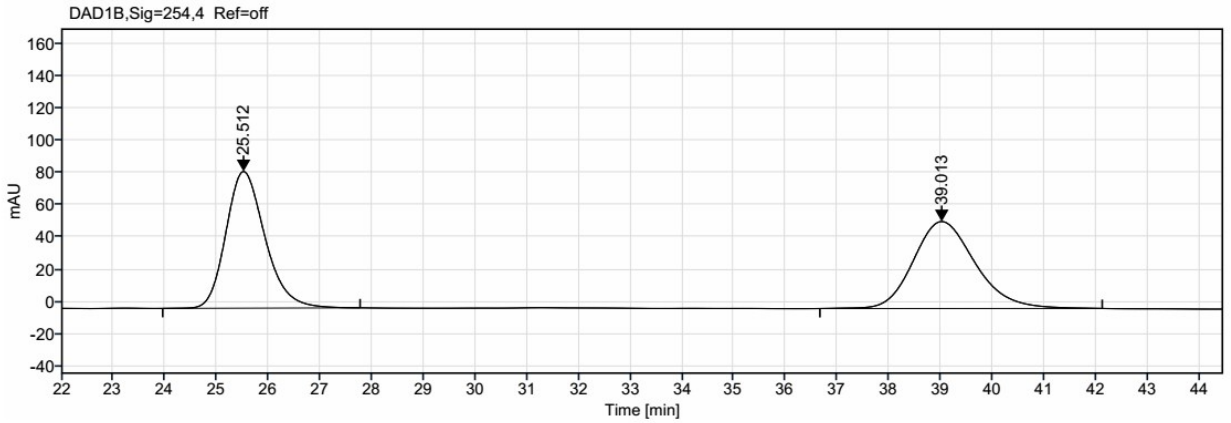


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
36.134	MM m	1.32	121114.91	1408.20	99.68	
45.241	MM m	1.35	383.53	3.36	0.32	
		Sum	121498.44			

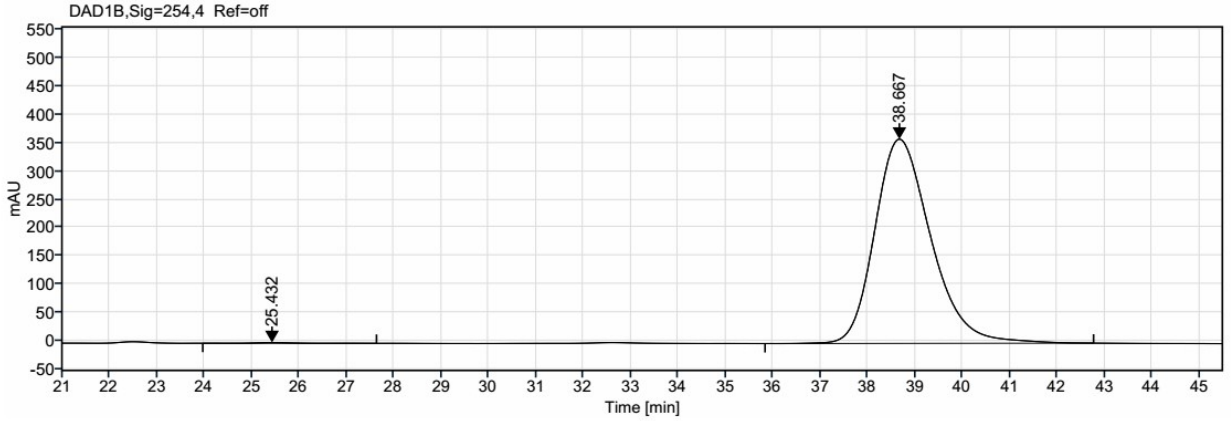


3m



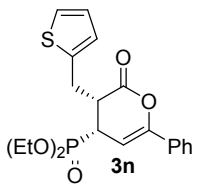
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
25.512	MM m	0.80	4442.27	84.54	49.85	
39.013	MM m	1.24	4469.09	53.79	50.15	
		Sum	8911.37			

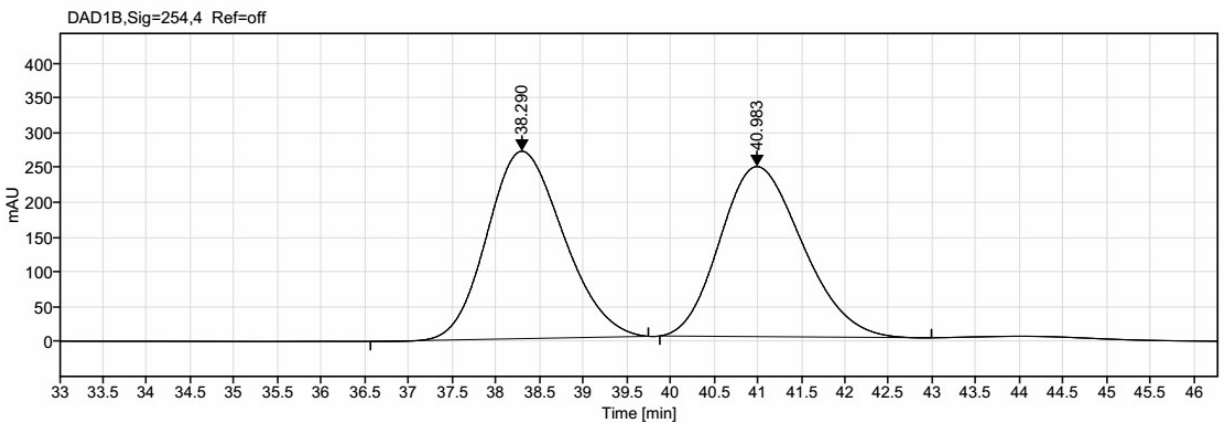


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
25.432	MM m	0.54	56.75	1.27	0.19	
38.667	MM m	1.25	29574.09	359.42	99.81	
		Sum	29630.84			

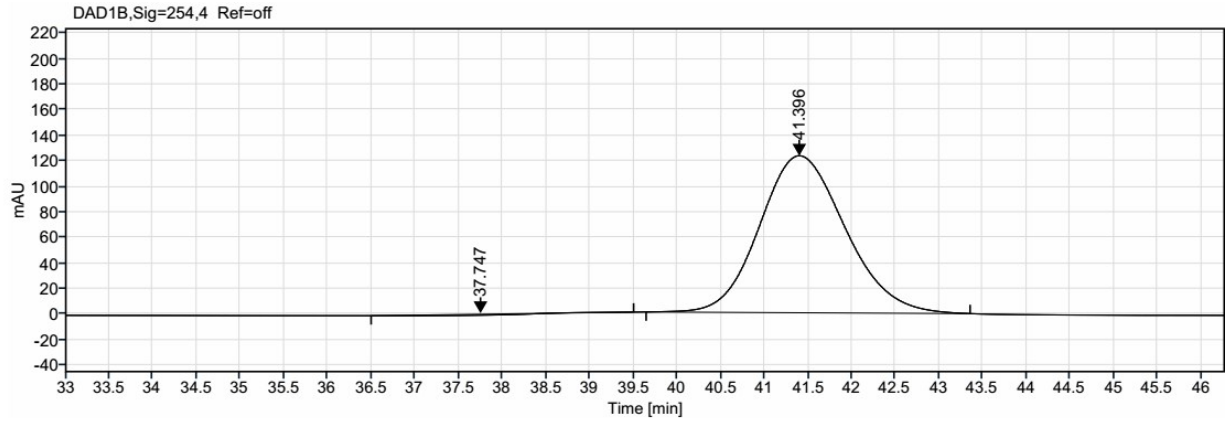


3n



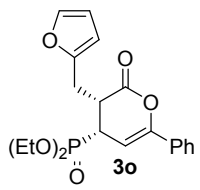
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
38.290	MM m	0.94	16336.31	269.30	50.48	
40.983	MM m	1.03	16024.64	244.31	49.52	
		Sum	32360.95			

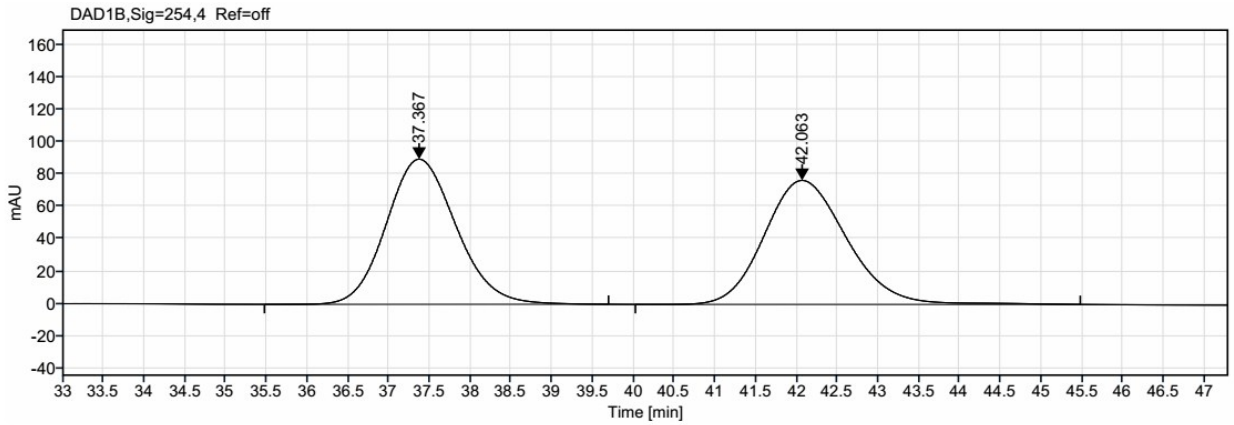


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
37.747	MM n	1.18	53.95	0.76	0.64	
41.396	MM m	1.05	8388.49	123.09	99.36	
		Sum	8442.44			

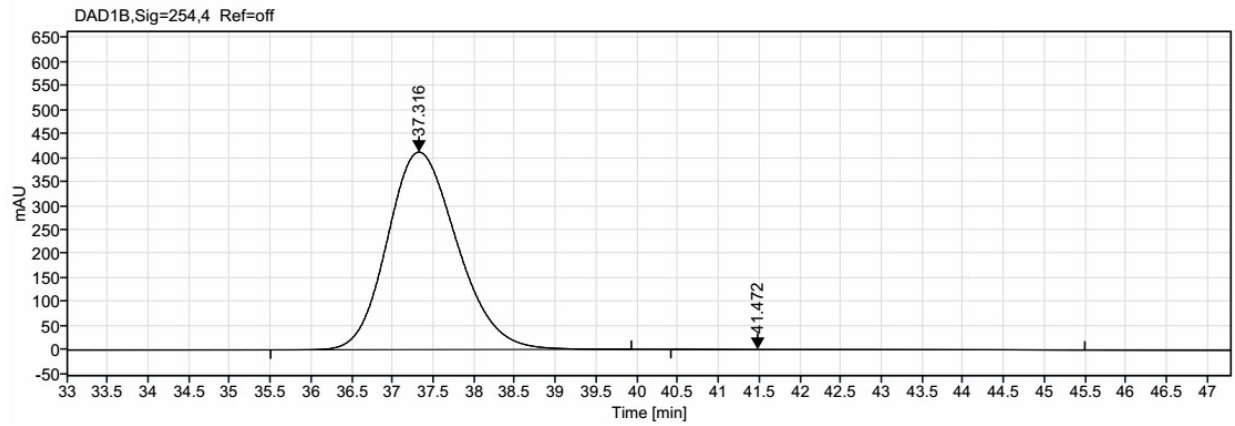


3o



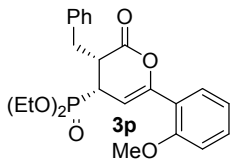
Signal: DAD1B,Sig=254,4 Ref=off

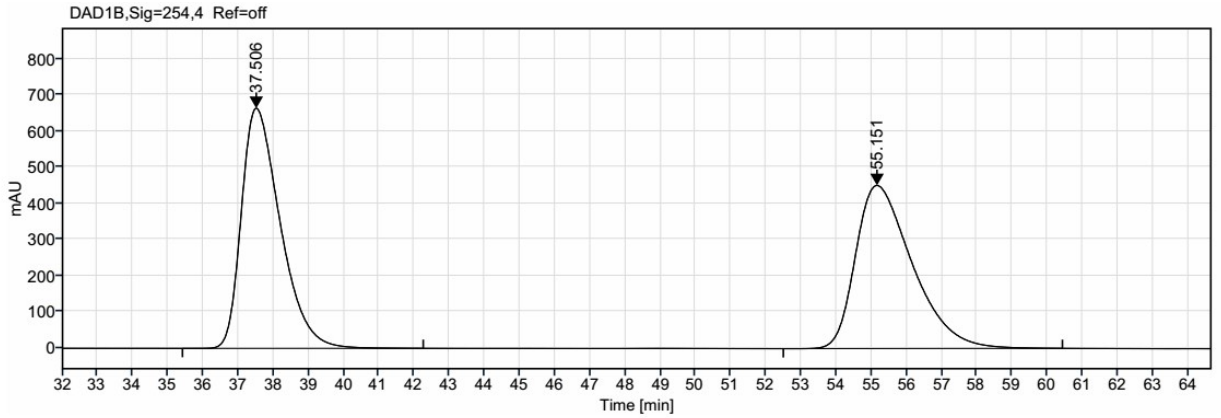
RT [min]	Type	Width [min]	Area	Height	Area%	Name
37.367	MM m	0.92	5338.18	89.53	49.76	
42.063	MM m	1.09	5389.15	76.50	50.24	
		Sum	10727.33			



Signal: DAD1B,Sig=254,4 Ref=off

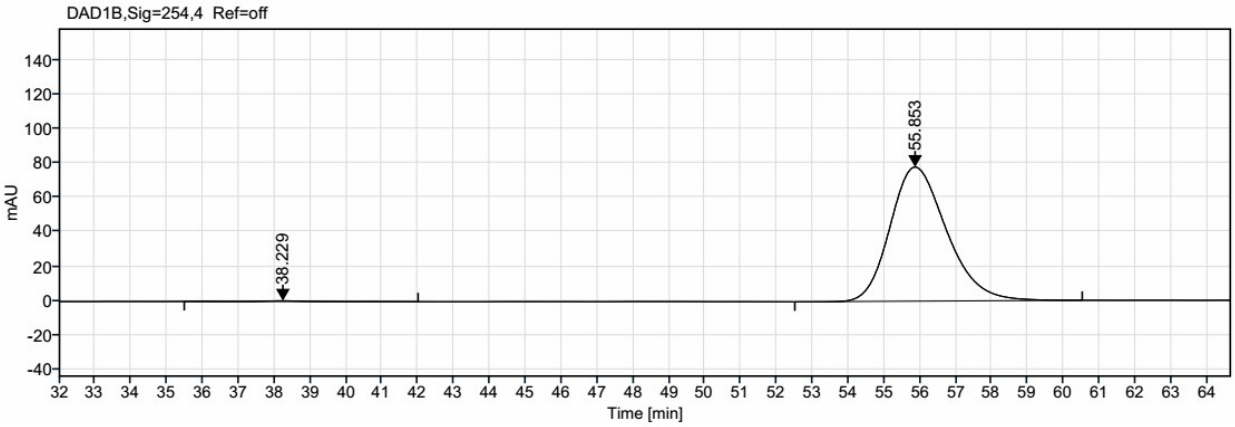
RT [min]	Type	Width [min]	Area	Height	Area%	Name
37.316	MM m	0.92	24506.69	411.38	99.98	
41.472	MM n	0.25	5.32	0.36	0.02	
		Sum	24512.01			





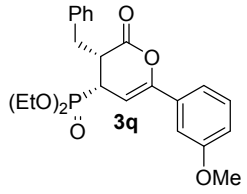
Signal: DAD1B,Sig=254,4 Ref=off

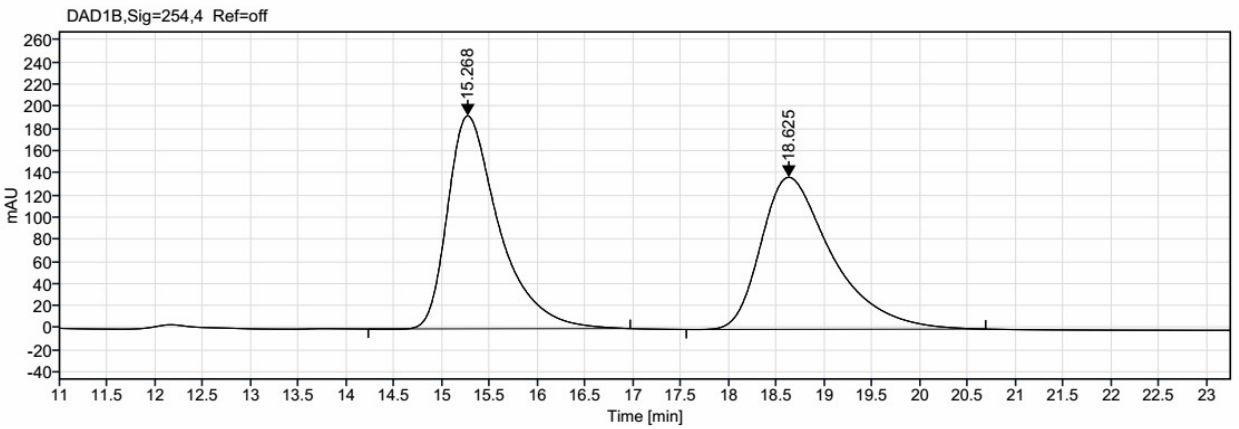
RT [min]	Type	Width [min]	Area	Height	Area%	Name
37.506	MM m	1.15	51028.24	664.35	50.20	
55.151	MM m	1.66	50629.46	451.36	49.80	
		Sum	101657.70			



Signal: DAD1B,Sig=254,4 Ref=off

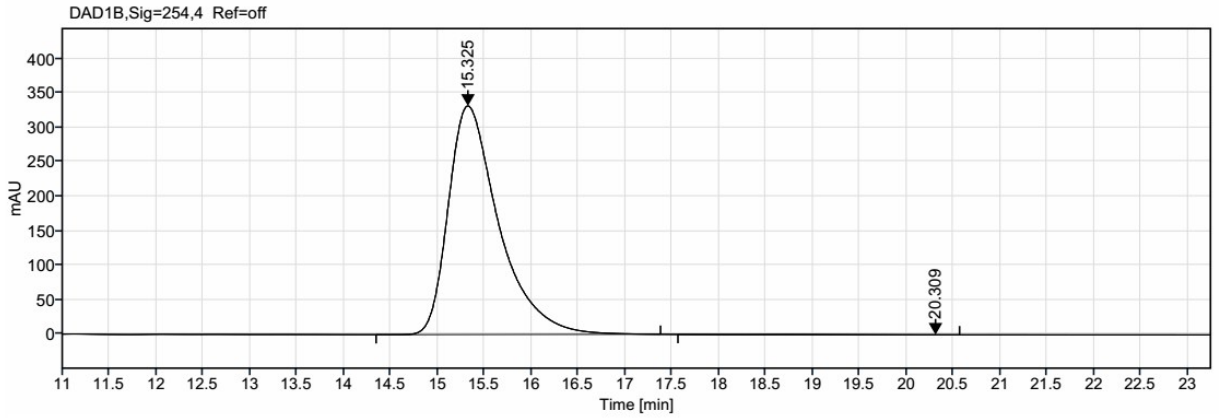
RT [min]	Type	Width [min]	Area	Height	Area%	Name
38.229	MM m	0.98	36.15	0.44	0.42	
55.853	MM m	1.65	8524.63	77.93	99.58	
		Sum	8560.78			





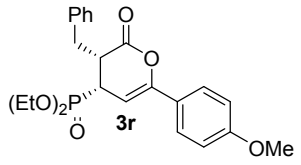
Signal: DAD1B,Sig=254,4 Ref=off

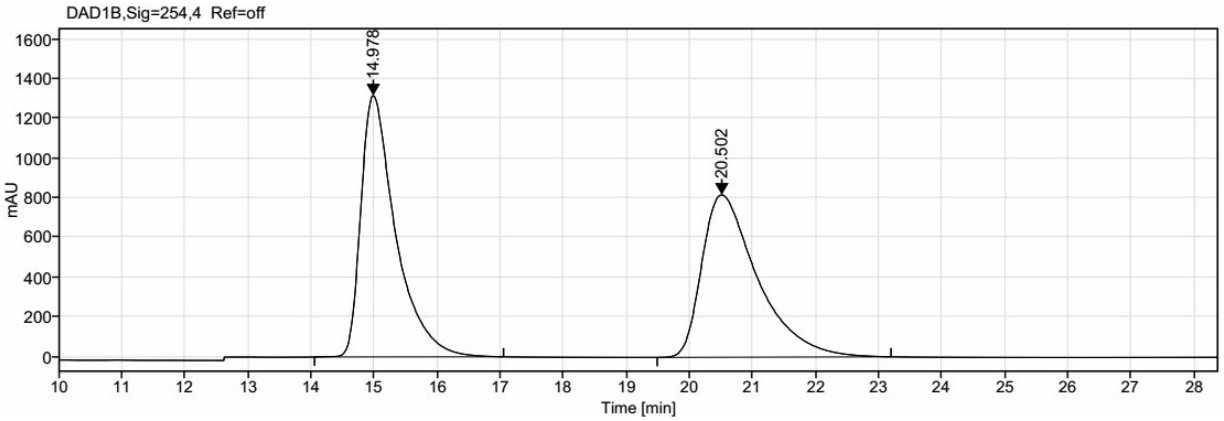
RT [min]	Type	Width [min]	Area	Height	Area%	Name
15.268	MM m	0.55	7201.34	192.37	50.38	
18.625	MM m	0.78	7091.69	137.33	49.62	
		Sum	14293.02			



Signal: DAD1B,Sig=254,4 Ref=off

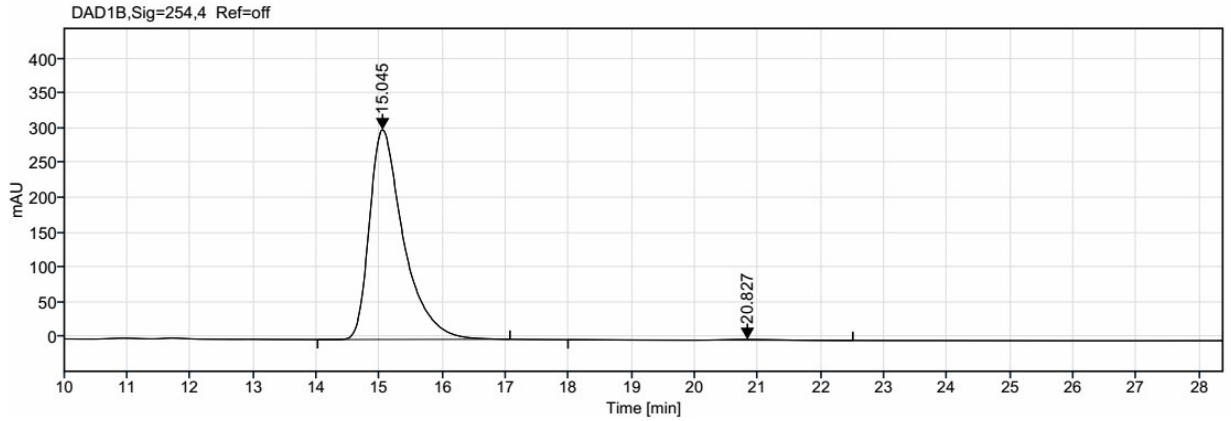
RT [min]	Type	Width [min]	Area	Height	Area%	Name
15.325	MM m	0.56	12412.03	331.73	99.91	
20.309	MM n	2.14	11.75	0.06	0.09	
		Sum	12423.78			





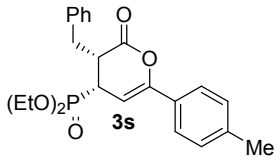
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
14.978	MM m	0.57	50457.90	1315.89	50.19	
20.502	MM m	0.91	50075.04	818.04	49.81	
		Sum	100532.94			

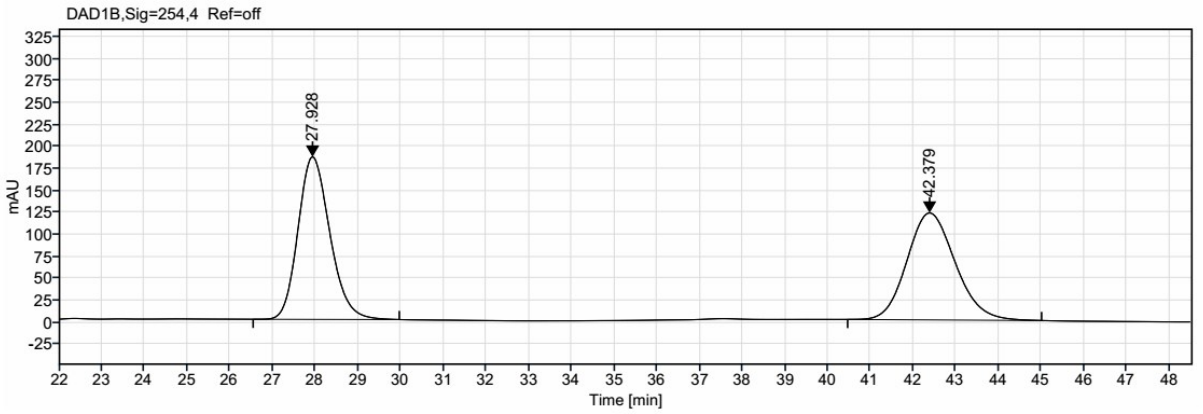


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
15.045	MM m	0.56	11291.70	301.35	99.75	
20.827	MM m	0.35	28.23	0.95	0.25	
		Sum	11319.93			

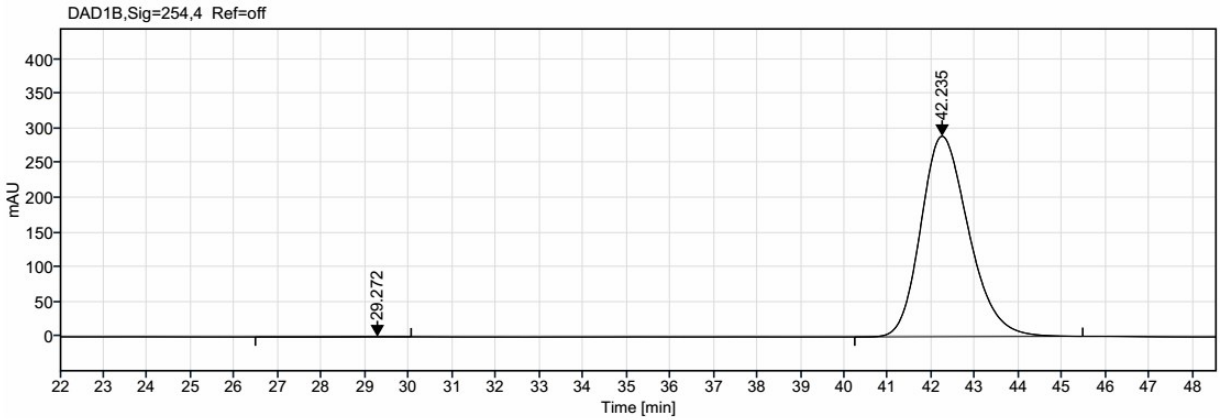


3s



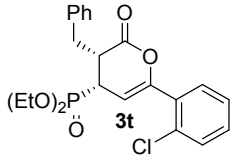
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
27.928	MM m	0.80	9591.57	184.72	50.11	
42.379	MM m	1.20	9549.49	121.62	49.89	
		Sum	19141.06			

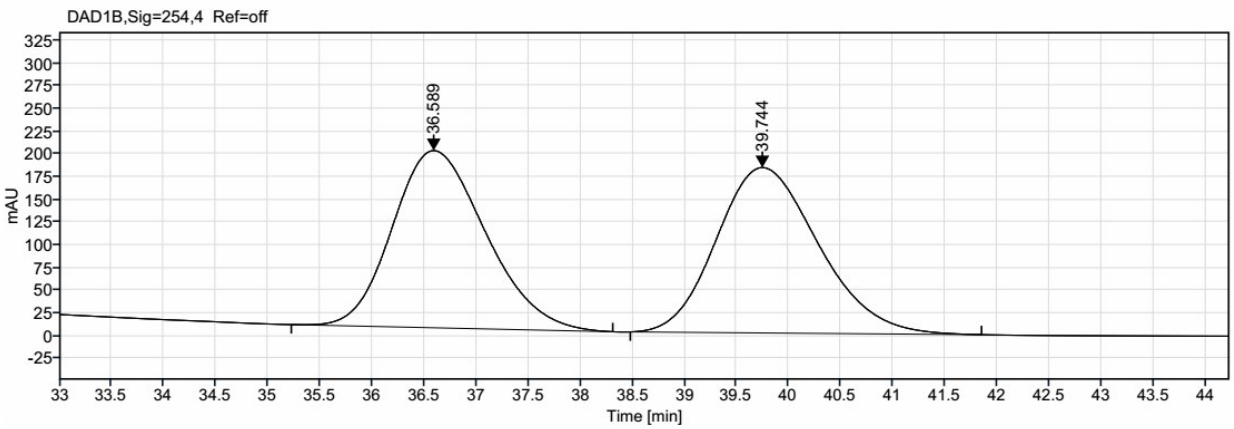


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
29.272	MM m	0.01	0.06	0.13	0.00	
42.235	MM m	1.17	22048.56	289.10	100.00	
		Sum	22048.61			

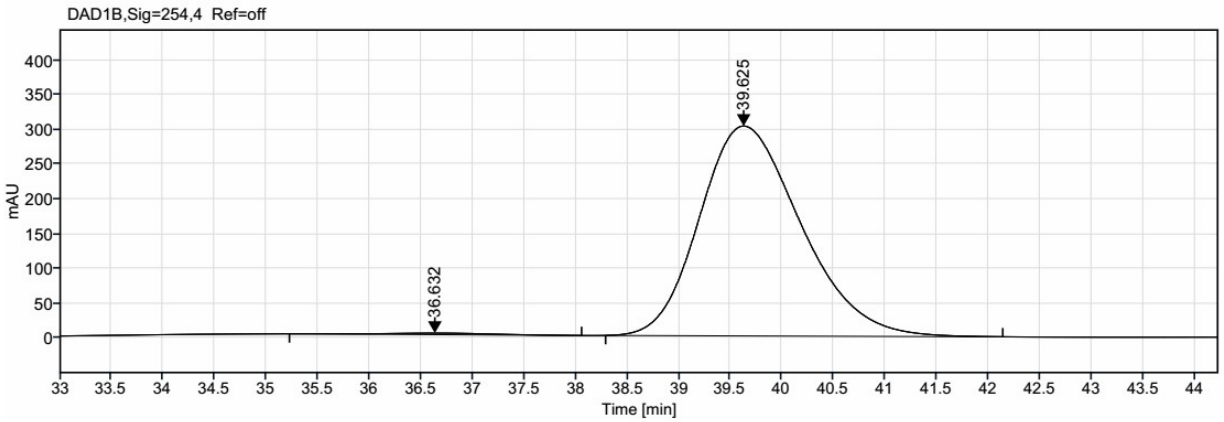


3t



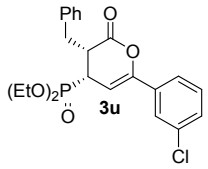
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
36.589	MM m	0.95	12124.55	194.83	49.48	
39.744	MM m	1.04	12379.84	181.85	50.52	
		Sum	24504.39			

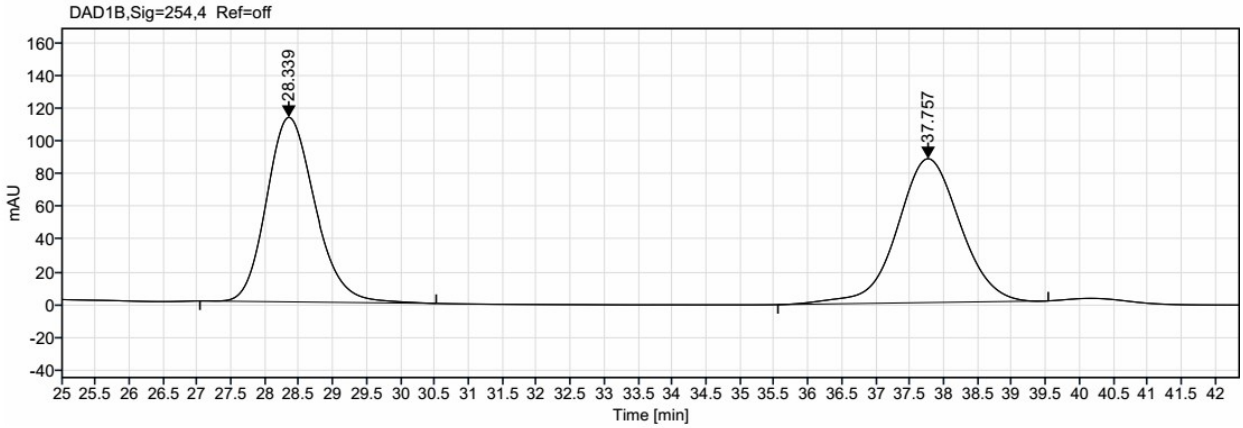


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
36.632	MM m	0.90	168.99	2.22	0.81	
39.625	MM m	1.06	20643.03	301.96	99.19	
		Sum	20812.02			

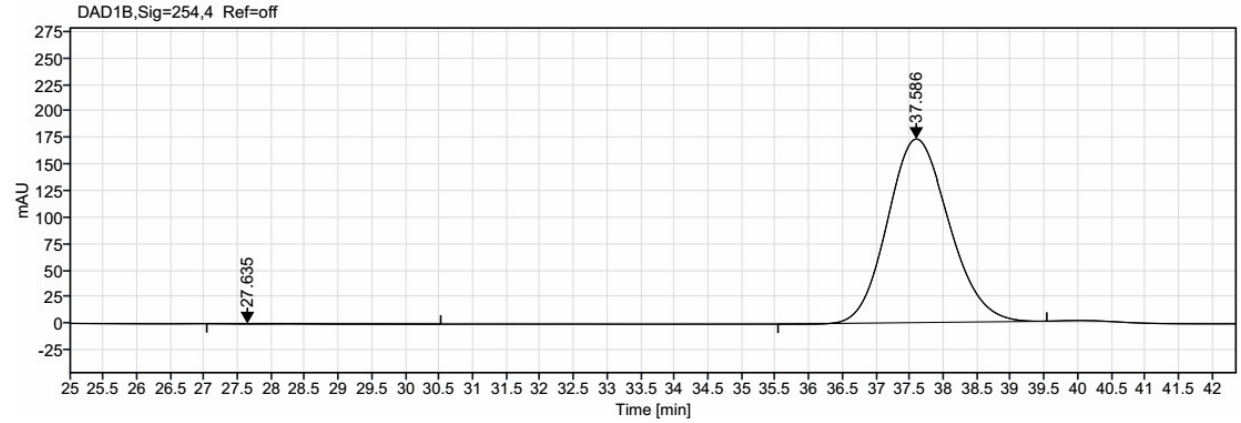


3u



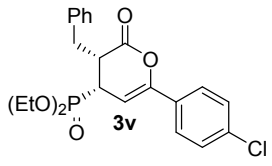
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
28.339	MM m	0.76	5591.82	112.49	49.76	
37.757	MM m	0.99	5646.27	87.72	50.24	
		Sum	11238.09			

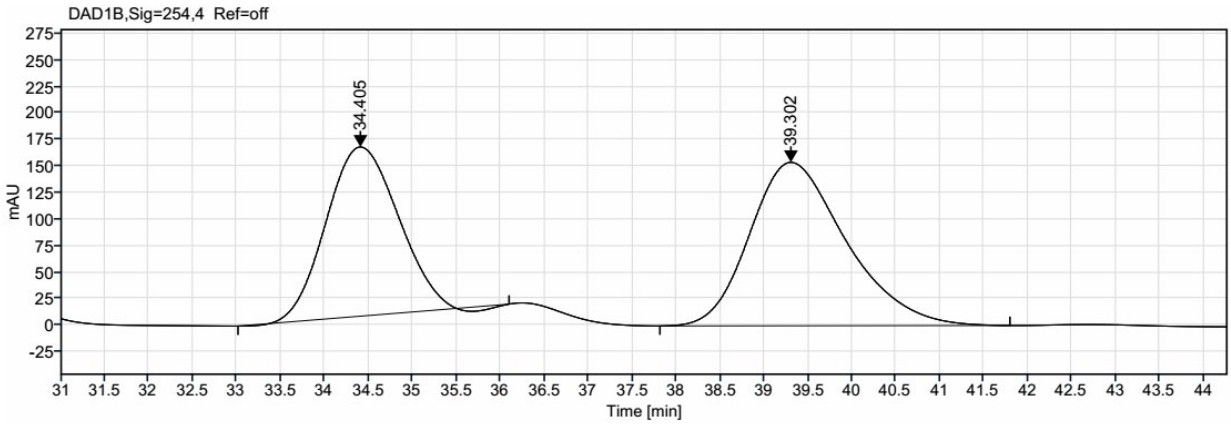


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
27.635	MM n	0.63	4.30	0.11	0.04	
37.586	MM m	0.98	11009.57	172.98	99.96	
		Sum	11013.87			

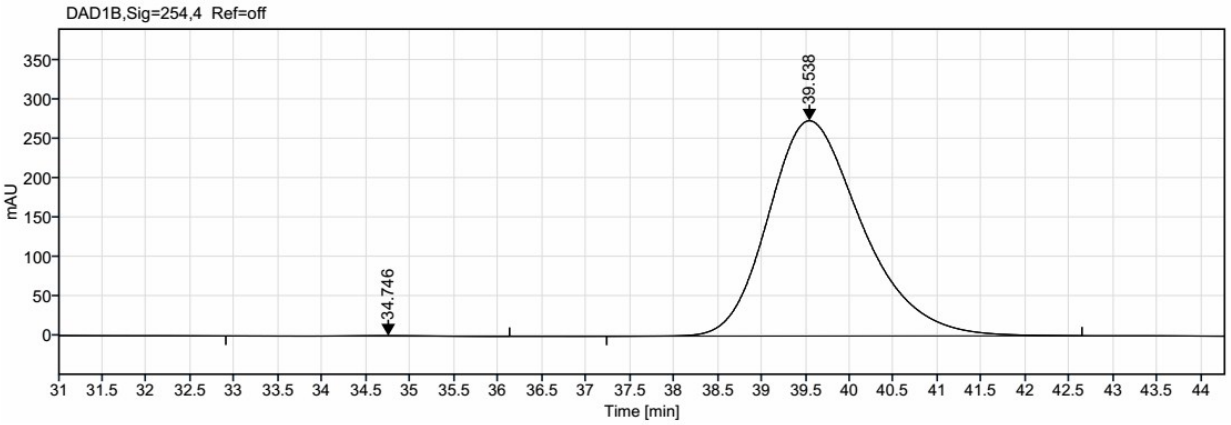


3v



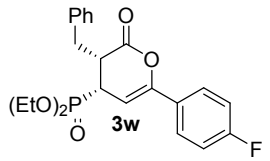
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
34.405	MM m	0.90	9032.97	159.50	43.52	
39.302	MM m	1.15	11724.73	154.05	56.48	
		Sum	20757.70			

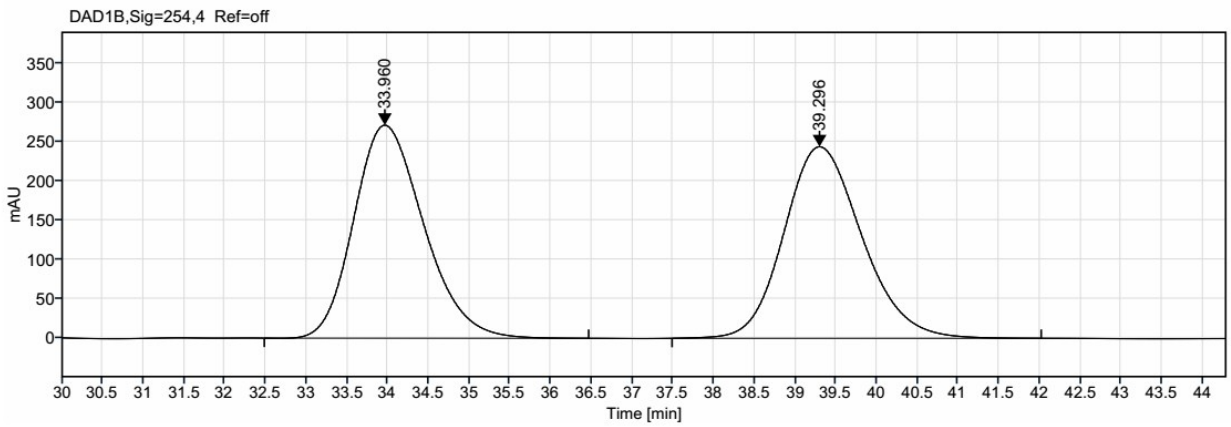


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
34.746	MM m	0.57	25.75	0.53	0.13	
39.538	MM m	1.13	20261.20	272.57	99.87	
		Sum	20286.95			

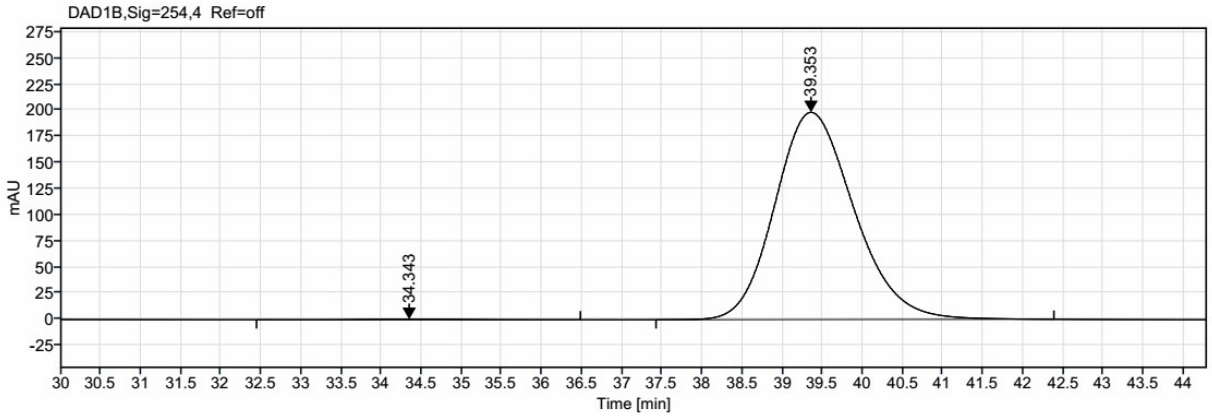


3w



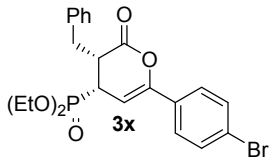
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
33.960	MM m	0.92	16131.68	270.29	49.90	
39.296	MM m	1.02	16196.98	243.09	50.10	
		Sum	32328.65			

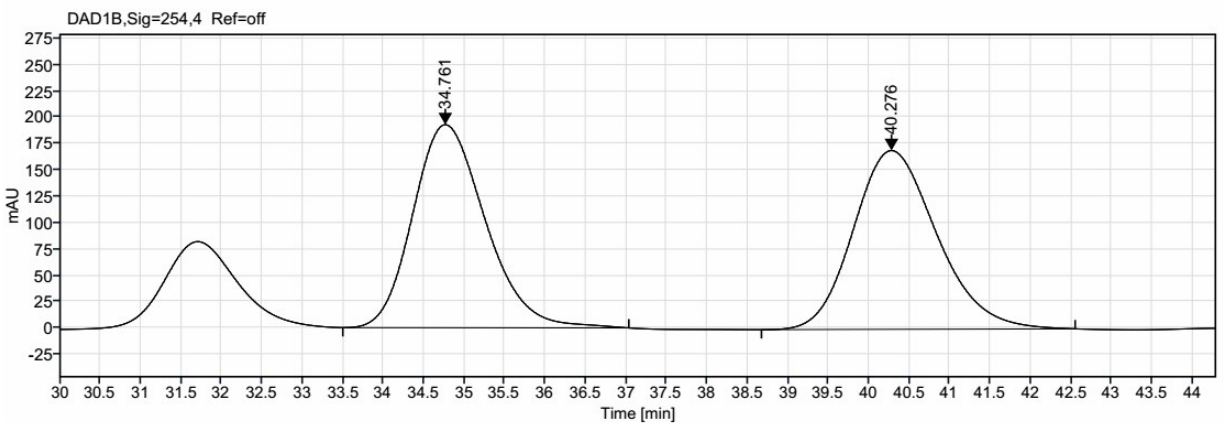


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
34.343	MM m	0.95	35.90	0.44	0.26	
39.353	MM m	1.05	13638.06	198.44	99.74	
		Sum	13673.96			

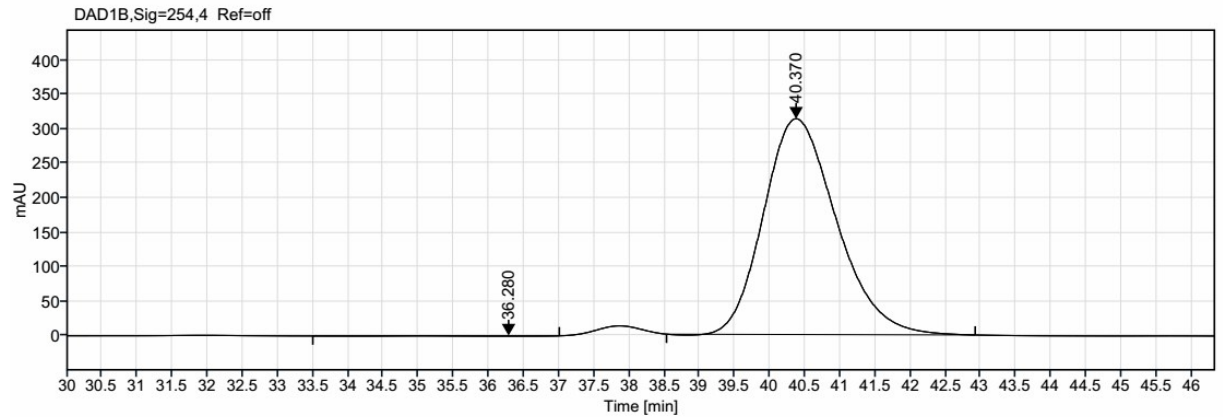


3x



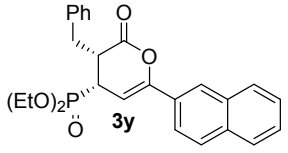
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
34.761	MM m	0.97	12021.45	192.73	49.71	
40.276	MM m	1.10	12163.07	169.67	50.29	
		Sum	24184.52			

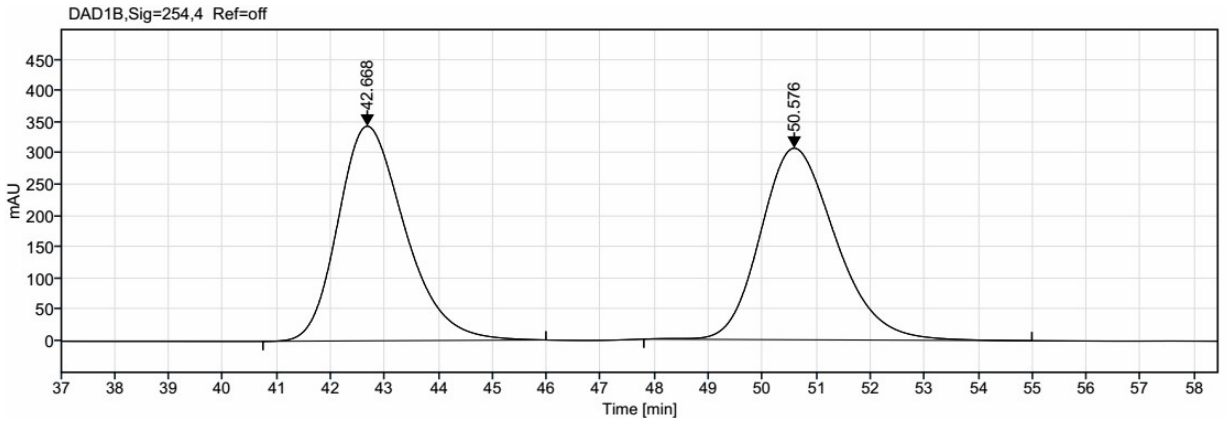


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
36.280	MM n	0.92	28.06	0.36	0.12	
40.370	MM m	1.08	22550.11	313.06	99.88	
		Sum	22578.17			

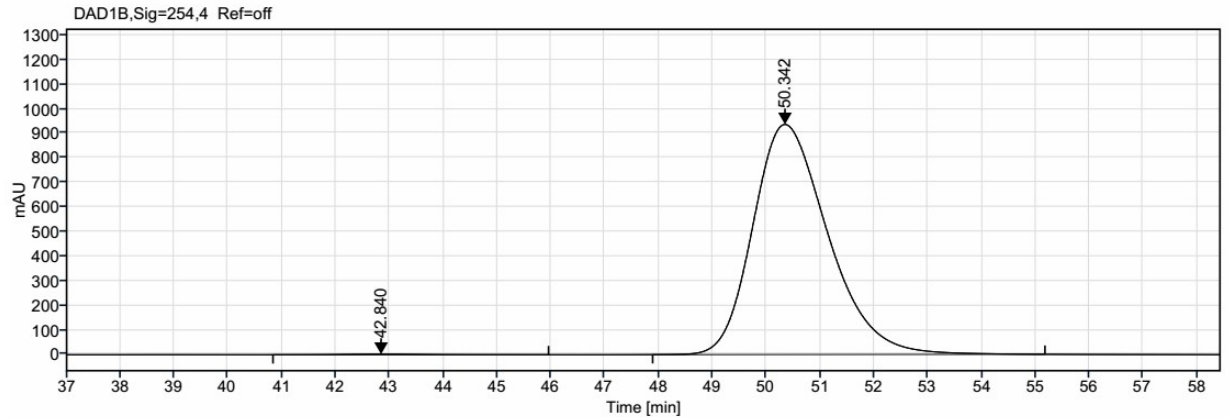


3y



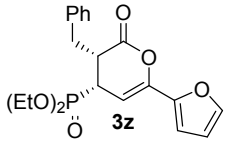
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
42.668	MM m	1.31	29633.35	343.78	49.90	
50.576	MM m	1.47	29749.32	306.74	50.10	
		Sum	59382.67			

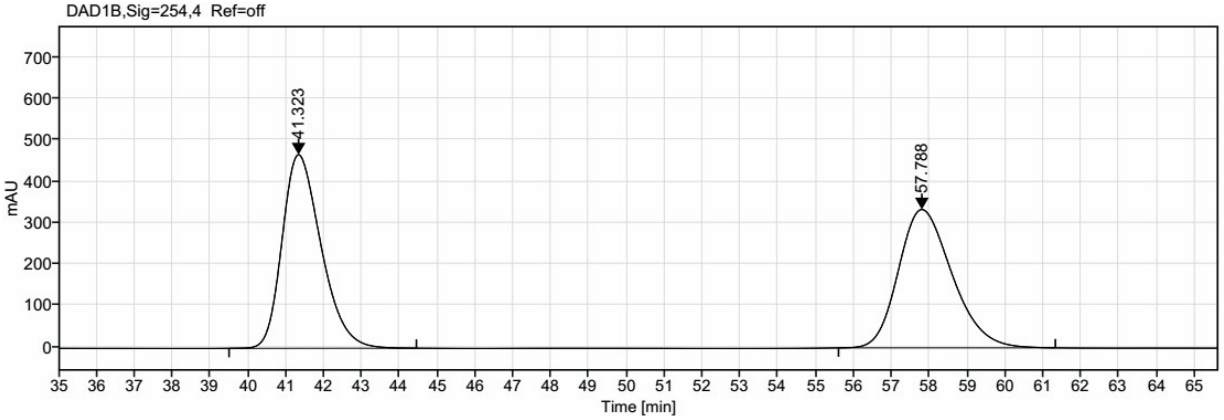


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
42.840	MM m	1.10	211.53	2.26	0.23	
50.342	MM m	1.48	90649.54	936.01	99.77	
		Sum	90861.07			

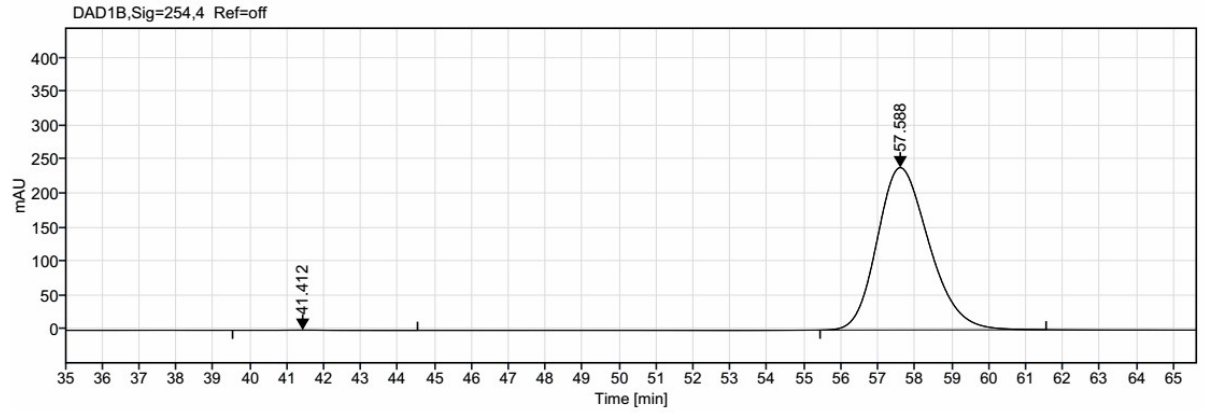


3z



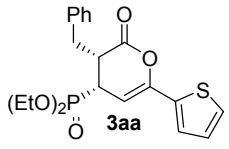
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
41.323	MM m	1.10	33460.17	467.44	50.42	
57.788	MM m	1.51	32907.40	334.05	49.58	
		Sum	66367.57			

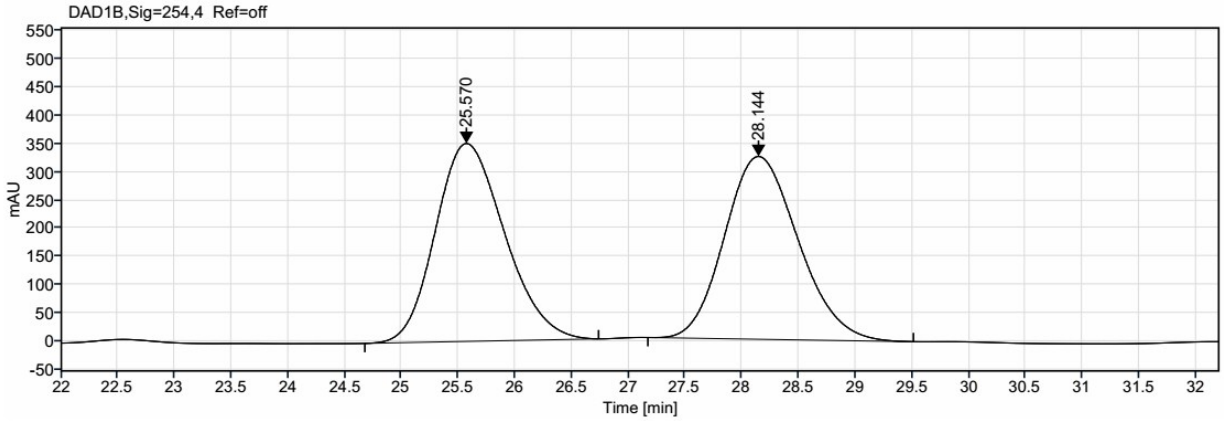


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
41.412	MM m	0.78	16.45	0.25	0.07	
57.588	MM m	1.47	23324.43	239.09	99.93	
		Sum	23340.87			

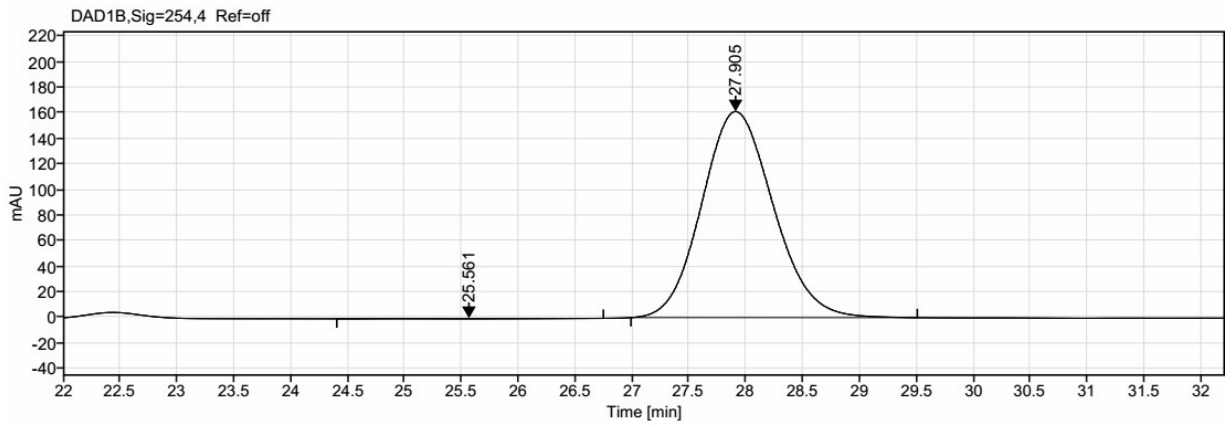


3aa



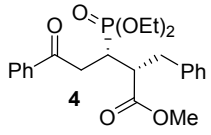
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
25.570	MM m	0.65	14786.34	348.98	50.39	
28.144	MM m	0.70	14558.21	322.30	49.61	
		Sum	29344.54			

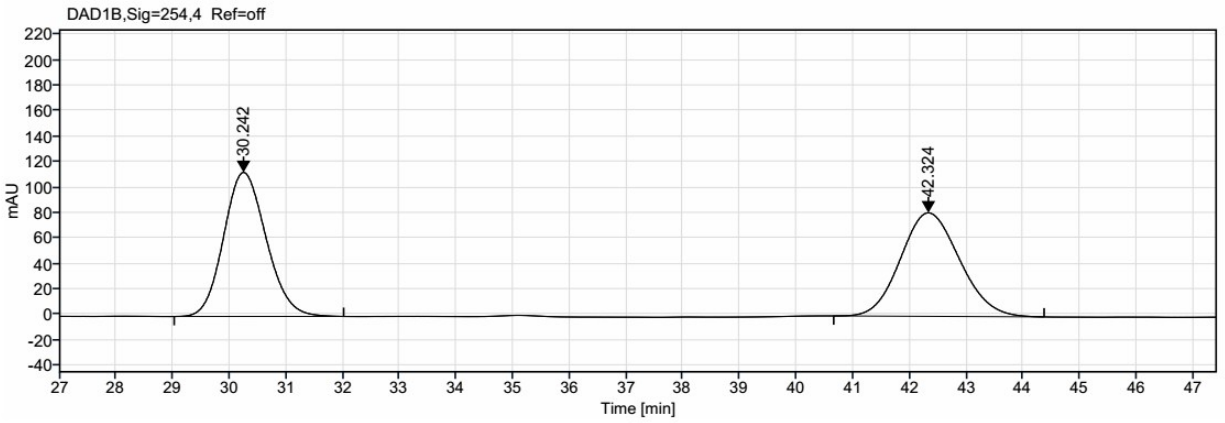


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
25.561	MM n	0.71	11.39	0.19	0.16	
27.905	MM m	0.68	7057.16	161.37	99.84	
		Sum	7068.55			

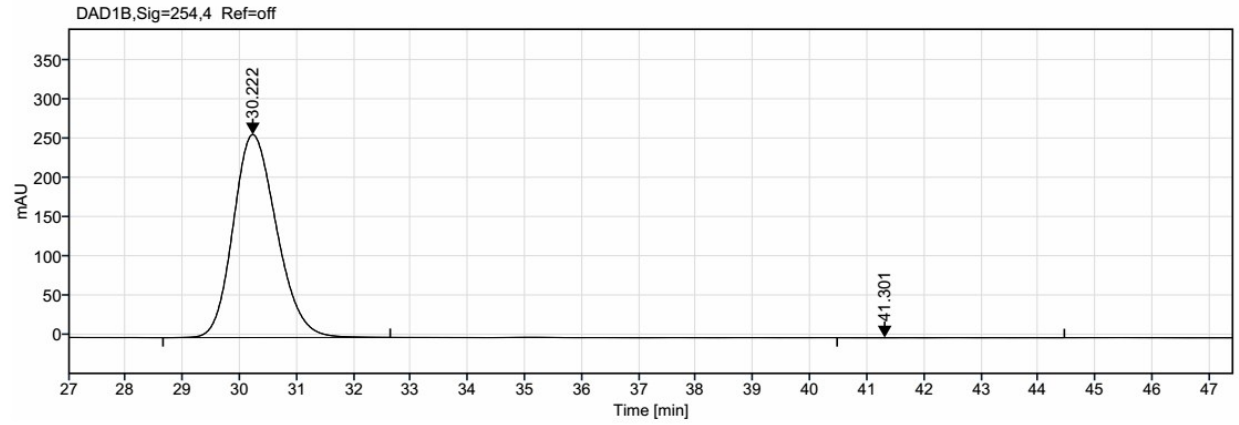


4



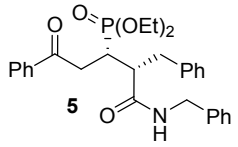
Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
30.242	MM m	0.81	5905.24	113.24	50.06	
42.324	MM m	1.10	5892.08	81.38	49.94	
		Sum	11797.32			

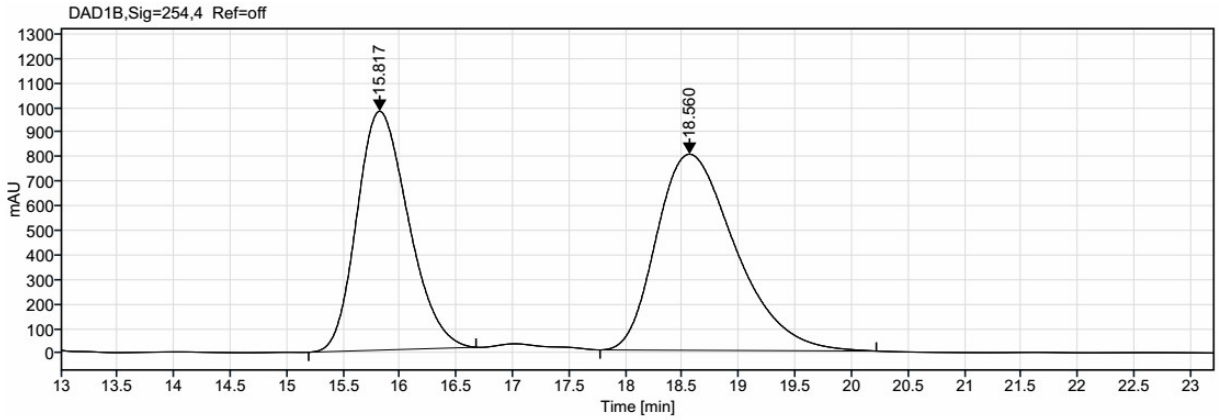


Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
30.222	MM m	0.80	13607.33	257.73	99.99	
41.301	MM n	0.16	0.96	0.07	0.01	
		Sum	13608.29			

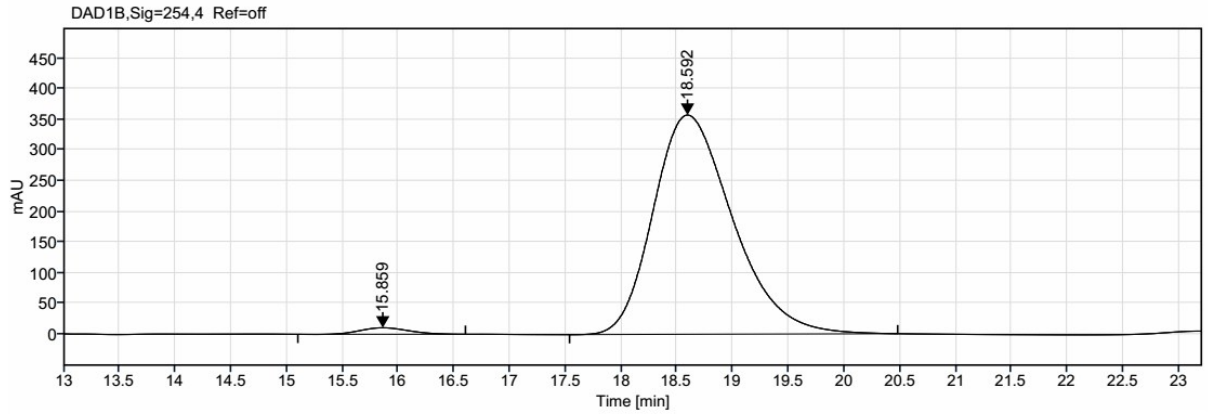


5



Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
15.817	MM m	0.49	30798.47	972.18	44.29	
18.560	MM m	0.75	38742.57	797.83	55.71	
		Sum	69541.04			



Signal: DAD1B,Sig=254,4 Ref=off

RT [min]	Type	Width [min]	Area	Height	Area%	Name
15.859	MM m	0.47	319.57	10.62	1.77	
18.592	MM m	0.76	17728.78	357.53	98.23	
		Sum	18048.34			