Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supplementary Information

Hydrofunctionalization of Alkenols Triggered by Addition of Diverse Radicals to Unactivated Alkenes and Subsequent Remote Hydrogen Atom Translocation

Na Wang,‡[a,b] Liu Ye,‡[c] Zhong-Liang Li,[c] Lei Li,[b] Zhuang Li,[b] Hong-Xia Zhang,[a] Zhen Guo,[a] Qiang-Shuai Gu*[c] and Xin-Yuan Liu*[b]

[a.] *College of Materials Science & Engineering, Key Laboratory of Interface Science and Engineering in Advanced Materials, Ministry of Education, Taiyuan University of Technology, Shanxi, 030024, China*

[b.] *Department of Chemistry and Shenzhen Grubbs Institute, Southern University of Science and Technology, Shenzhen 518055, China. liuxy3@sustc.edu.cn*

[c.] *Academy for Advanced Interdisciplinary Studies, Southern University of Science and Technology, Shenzhen, 518055, China. guqs@sustc.edu.cn*

‡ These authors contributed equally to this work.

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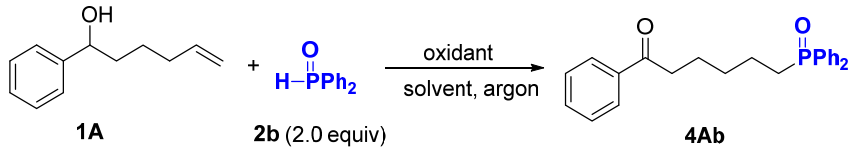
General information.

All reactions were carried out under argon using Schlenk techniques. Reagents were purchased at the commercial quality and used without further purification. Analytical thin layer chromatography (TLC) was performed on precoated silica gel 60 GF254 plates. Flash column chromatography was performed using Tsingdao silica gel (60, particle size 0.040-0.063 mm). Visualization on TLC was achieved by use of UV light (254 nm) or iodine. NMR spectra were recorded on a Bruker DPX 400 spectrometer at 400 MHz for ^{1}H NMR, 100 MHz for ^{13}C NMR and 376 MHz for ^{19}F NMR in $CDCl_3$ with tetramethylsilane (TMS) as internal standard. Microwave irradiation experiments were carried out in a dedicated Biotage Initator Robot 8 auto microwave apparatus. The chemical shifts are expressed in ppm and coupling constants are given in Hz. Data for ^{1}H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q, quarter; m, multiplet), coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). ^{19}F NMR spectra were recorded on a Bruker DPX 400 MHz spectrometer ($CFCl_3$ as an external reference (0 ppm)). Mass spectrometric data were obtained using Bruker Apex IV RTMS.

Supplementary Table S1. Screening Results of Reaction Conditions for Phosphonylation[a]

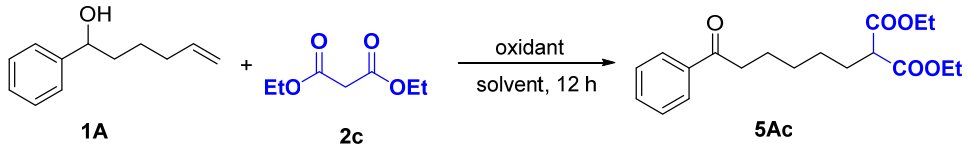


1A **2b** (2.0 equiv) **4Ab**

entry	Oxidant (X equiv)	T (°C)	solvent	yield (%)[b]
1	CuCl (2)	80	CH_3CN	No reaction
2	$K_2S_2O_8$ (2)	80	CH_3CN/H_2O (2/1)	68% (58%)[c]
3	$K_2S_2O_8$ (2)	80	CH_3CN/H_2O (2/1)	68%[d]
4	$K_2S_2O_8$ (2)	80	CH_3CN/H_2O (2/1)	60%[e]
5	AgOAc (3)	100	DMF	100% (90%)[c]
6	$AgNO_3$ (0.5)	100	EtOAc	63%
7	$AgNO_3$ (0.5)	100	CH_3CN	65%
8	$AgNO_3$ (0.5)	100	Toluene	65%
9	$AgNO_3$ (0.5)	100	DMF	60%
10	$AgNO_3$ (1)	100	CH_3CN	70%

[a] Reaction conditions: **1A** (0.2 mmol), **2b** (0.4 mmol), solvent (2.0 mL) at 80 °C for 12 h under argon. [b] Determined by NMR spectroscopy using 1,3,5-trimethylbenzene as an internal standard. [c] Isolated yield in bracket. [d] Ag_2SO_4 (0.02 mmol) was added. [e] Shortening the reaction time to 5 h.

Supplementary Table S2. Screening Results of Reaction Conditions for Addition of Diethyl Malonate to Alkene[a]



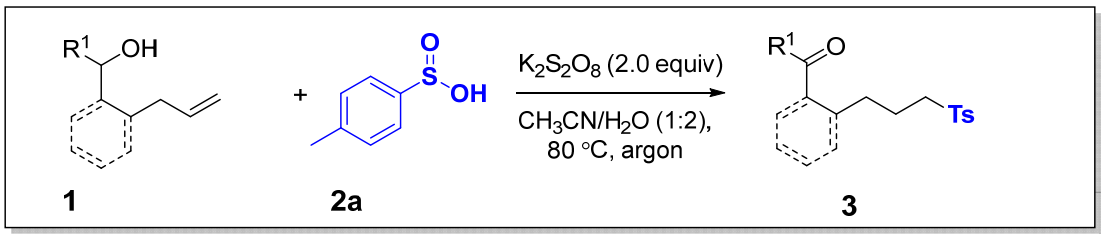
1A **2c** **5Ac**

entry	Oxidant (X equiv)	**2c** (X equiv)	Solvent/T(° C)	yield (%)[b]
1	$Cu(OAc)_2$ (2)	1.5	TFE[c] (100)	No reaction
2	Ag_2CO_3 (2)	3.0	DMF (80)	40% conversion 45% yield
3	$K_2S_2O_8$ (2)	1.5	CH_3CN/H_2O (80)	trace
4	$Mn(OAc)_3{\cdot}2H_2O$ (3)	1.5	AcOH (80)	100% conversion 14% yield
5	$Mn(OAc)_3{\cdot}2H_2O$ (3)	1.5	TFE (80)	60% conversion 34% yield
6	$Mn(OAc)_3{\cdot}2H_2O$ (3)	1.5	TFE (110)[d]	74% conversion 57% yield
7	$Mn(OAc)_3{\cdot}2H_2O$ (3.6)	1.8	TFE (110)[d]	100% conversion 50% yield

[a] Reaction conditions: **1A** (0.2 mmol), **2c**, solvent (2.0 mL) was heated for 16 h under argon. [b] Isolated yield. [c] TFE: 2,2,2-trifluoroethanol. [d] reaction time: 36 h.

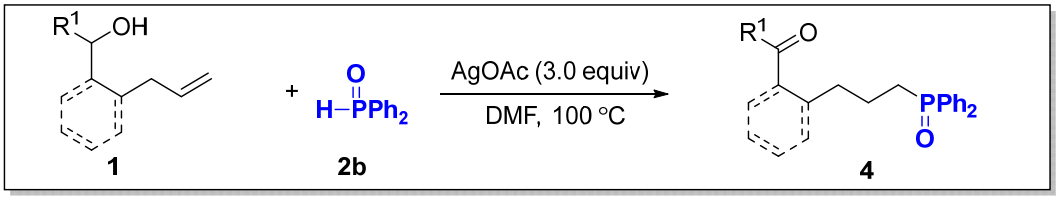
General procedures

General procedure for radical sulfonylation reaction system



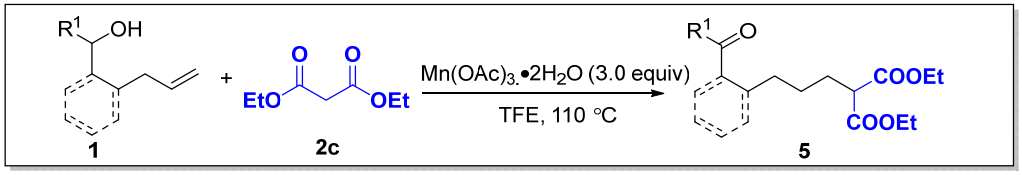
To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **1** (0.2 mmol), **2a** (0.6 mmol) and $K_2S_2O_8$ (0.4 mmol). The tube was evacuated and backfilled with argon for three times, and then CH_3CN (0.7 mL) and H_2O (1.4 mL) were added. The tube was stirred at 80 °C for 18 h and then H_2O (5 mL) was added. EtOAc was used to extract the product from the aqueous layer (3 × 20 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to afford the crude product, which was purified by flash column chromatography to afford the product **3**.

General procedures for radical phosphonylation reaction system



To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **1** (0.2 mmol), **2b** (0.4 mmol) and AgOAc (0.60 mmol). The tube was evacuated and backfilled with argon for three times, and then DMF (2 mL) were added. The tube was stirred at 80 °C for 12 h. Diethyl ether (50 mL) was added, followed by washing with water (2 × 5 mL). The organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to afford the crude product, which was purified by flash column chromatography to afford the product **4**.

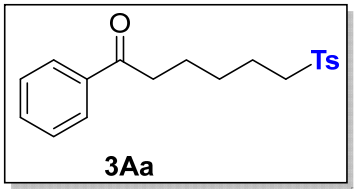
General procedure for radical reaction with diethyl malonate



To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **1** (0.2 mmol), diethyl malonate **2c** (0.3 mmol) and $Mn(OAc)_3•2H_2O$ (0.6 mmol). The tube was evacuated and backfilled with argon for three times, and then 2,2,2-trifluoroethanol (2.0 mL) were added. The tube was stirred at 110 °C for 36 h

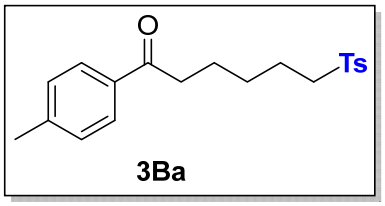
and then H_2O (5 mL) was added. EtOAc was used to extract the product from the aqueous layer (3 × 20 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to afford the crude product, which was purified by flash column chromatography to afford the product **5**.

1-phenyl-6-tosylhexan-1-one (3Aa)



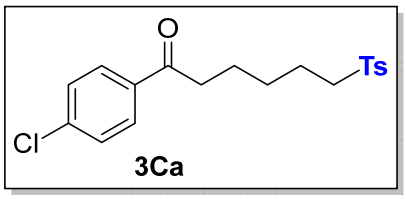
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.90 (m, 2H), 7.79 (d, *J* = 8.3 Hz, 2H), 7.66 – 7.54 (m, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 3.10 (dd, *J* = 9.1, 6.9 Hz, 2H), 2.96 (t, *J* = 7.2 Hz, 2H), 2.46 (s, 3H), 1.91 – 1.57 (m, 4H), 1.51 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.78, 144.66, 136.84, 136.15, 133.10, 129.92, 128.63, 128.08, 127.99, 56.17, 37.93, 27.90, 23.45, 22.70, 21.64. HRMS (APCI) m/z calcd. for $C_{19}H_{23}O_3S$ $[M+H]^+$ 331.1368, found 331.1363.

1-(*p*-tolyl)-6-tosylhexan-1-one (3Ba)



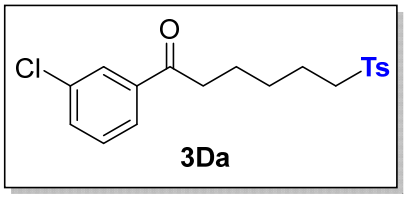
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.2 Hz, 2H), 7.79 (d, *J* = 8.2 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 7.25 (d, *J* = 8.0 Hz, 2H), 3.09 (dd, *J* = 9.1, 6.9 Hz, 2H), 2.92 (t, *J* = 7.2 Hz, 2H), 2.45 (s, 3H), 2.41 (s, 3H), 1.81 – 1.67 (m, 4H), 1.51 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.44, 144.63, 143.84, 136.19, 134.40, 129.91, 129.28, 128.11, 128.07, 56.17, 37.81, 27.91, 23.55, 22.69, 21.62. HRMS (APCI) m/z calcd. for $C_{20}H_{25}O_3S$ $[M+H]^+$ 345.1524, found 345.1521.

1-(4-chlorophenyl)-6-tosylhexan-1-one (3Ca)



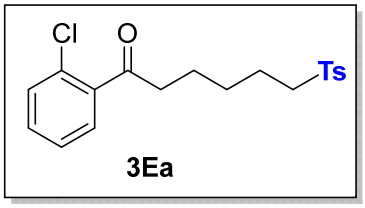
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.3 Hz, 2H), 7.78 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 8.3 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 3.13 – 3.05 (m, 2H), 2.92 (t, *J* = 7.1 Hz, 2H), 2.45 (s, 3H), 1.80 – 1.67 (m, 4H), 1.51 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.50, 144.69, 139.49, 136.14, 135.13, 129.92, 129.42, 128.96, 128.06, 56.13, 37.91, 27.83, 23.34, 22.65, 21.64. HRMS (APCI) m/z calcd. for $C_{19}H_{22}ClO_3S$ $[M+H]^+$ 365.0978, found 365.0973.

1-(3-chlorophenyl)-6-tosylhexan-1-one (3Da)



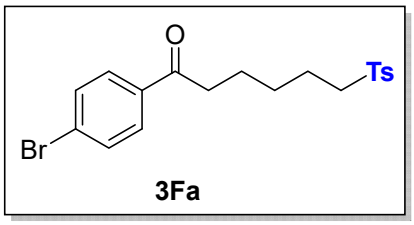
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.3 Hz, 2H), 7.42 – 7.29 (m, 6H), 3.08 (dd, *J* = 9.1, 6.9 Hz, 2H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.45 (s, 3H), 1.78 – 1.64 (m, 4H), 1.50 – 1.40 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.11, 144.69, 139.42, 136.10, 131.69, 130.68, 130.51, 129.93, 128.71, 128.07, 126.99, 56.11, 42.35, 27.71, 23.39, 22.64, 21.64. HRMS (APCI) m/z calcd. for $C_{19}H_{22}ClO_3S$ $[M+H]^+$ 365.0978, found 365.0971.

1-(2-chlorophenyl)-6-tosylhexan-1-one (3Ea):



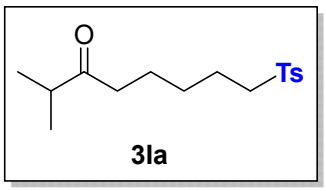
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (t, *J* = 1.8 Hz, 1H), 7.82 – 7.78 (m, 3H), 7.54 (ddd, *J* = 8.0, 2.1, 1.0 Hz, 1H), 7.41 (t, *J* = 7.9 Hz, 1H), 7.37 (d, *J* = 7.9 Hz, 2H), 3.12 – 3.07 (m, 2H), 2.93 (t, *J* = 7.1 Hz, 2H), 2.46 (s, 3H), 1.81 – 1.68 (m, 4H), 1.51 – 1.42 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.39, 144.69, 138.36, 136.16, 134.90, 133.09, 129.93, 129.90, 128.17, 128.08, 126.08, 56.13, 38.05, 27.81, 23.26, 22.66, 21.64. HRMS (APCI) m/z calcd. for $C_{19}H_{22}ClO_3S$ $[M+H]^+$ 365.0978, found 365.0970.

1-(4-bromophenyl)-6-tosylhexan-1-one (3Fa):



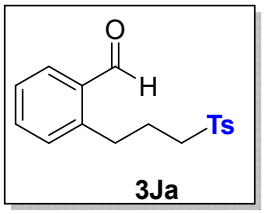
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 – 7.72 (m, 4H), 7.58 (d, *J* = 8.6 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 3.08 (t, *J* = 6.9 Hz, 2H), 2.90 (t, *J* = 7.2 Hz, 2H), 2.44 (s, 3H), 1.79 – 1.66 (m, 4H), 1.49 – 1.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.68, 144.69, 136.18, 135.55, 131.93, 129.90, 129.57, 128.16, 128.04, 56.13, 37.84, 27.81, 23.32, 22.66, 21.64. HRMS (APCI) m/z calcd. for $C_{19}H_{22}BrO_3S$ $[M+H]^+$ 409.0473, found 409.0467.

2-methyl-8-tosyloctan-3-one (3Ia):



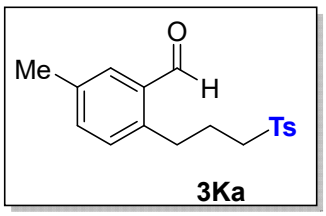
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 (d, *J* = 8.0 Hz, 2H), 7.38 (d, *J* = 7.9 Hz, 2H), 3.11 – 3.04 (m, 2H), 2.61 – 2.51 (m, 1H), 2.47 (s, 3H), 2.43 (t, *J* = 7.1 Hz, 2H), 1.78 – 1.68 (m, 2H), 1.60 – 1.50 (m, 2H), 1.41 – 1.32 (m, 2H), 1.08 (d, *J* = 6.9 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 214.33, 144.63, 136.17, 129.89, 128.06, 56.15, 40.87, 39.61, 27.84, 22.95, 22.62, 21.63, 18.23. HRMS (APCI) m/z calcd. for $C_{16}H_{25}O_3S$ $[M+H]^+$ 297.1510, found 297.1519.

2-(3-tosylpropyl)benzaldehyde (3Ja):



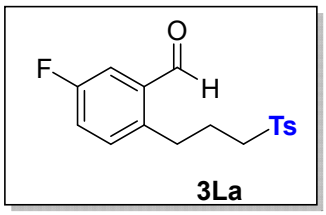
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.09 (s, 1H), 7.80 – 7.74 (m, 3H), 7.51 (td, *J* = 7.5, 1.6 Hz, 1H), 7.42 (td, *J* = 7.5, 1.3 Hz, 1H), 7.37 – 7.32 (m, 2H), 7.25 (dd, *J* = 7.6, 1.2 Hz, 1H), 3.16 – 3.10 (m, 4H), 2.44 (s, 3H), 2.06 – 1.97 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.96, 144.77, 142.62, 136.17, 134.43, 133.96, 133.80, 131.33, 130.01, 128.13, 127.27, 55.73, 31.23, 24.51, 21.73. HRMS (APCI) m/z calcd. for $C_{17}H_{19}O_3S$ $[M+H]^+$ 303.1042, found 303.1049.

5-methyl-2-(3-tosylpropyl)benzaldehyde (3Ka):



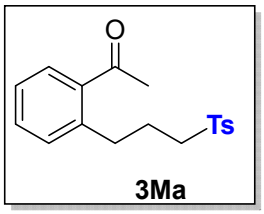
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.79 – 7.72 (m, 2H), 7.57 (d, *J* = 1.8 Hz, 1H), 7.37 – 7.28 (m, 3H), 7.13 (d, *J* = 7.7 Hz, 1H), 3.18 – 3.02 (m, 4H), 2.44 (s, 3H), 2.40 (s, 3H), 2.05 – 1.90 (m, 2H) ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.09, 144.75, 139.62, 137.06, 136.20, 134.83, 134.73, 133.65, 131.31, 130.00, 128.15, 55.74, 30.78, 24.61, 21.74, 20.87. HRMS (APCI) m/z calcd. for $C_{18}H_{21}O_3S$ $[M+H]^+$ 317.1198, found 317.1206.

5-fluoro-2-(3-tosylpropyl)benzaldehyde (3La):



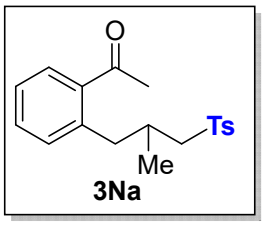
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.04 (s, 1H), 7.85 – 7.73 (m, 3H), 7.39 – 7.33 (m, 2H), 7.14 – 7.05 (m, 1H), 6.95 (dd, *J* = 9.5, 2.5 Hz, 1H), 3.20 – 3.09 (m, 4H), 2.45 (s, 3H), 2.08 – 1.95 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.09, 165.59 (d, *J* = 257.7 Hz), 146.13 (d, *J* = 8.8 Hz), 144.80, 137.02 (d, *J* = 10.1 Hz), 136.03, 129.97, 128.04, 118.20 (d, *J* = 21.7 Hz), 114.32 (d, *J* = 21.7 Hz), 55.50, 31.02, 24.15, 21.65. ^{19}F NMR (376 MHz, $CDCl_3$) δ -102.99. HRMS (APCI) m/z calcd. for $C_{17}H_{18}O_3FS$ $[M+H]^+$ 321.0948, found 321.0955.

1-(2-(3-tosylpropyl)phenyl)ethanone (3Ma):



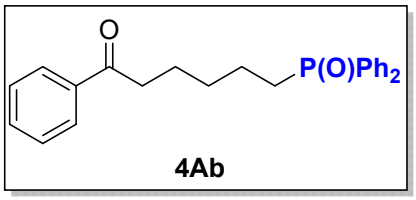
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.2 Hz, 2H), 7.73 – 7.69 (m, 1H), 7.42 (td, *J* = 7.5, 1.2 Hz, 1H), 7.37 – 7.28 (m, 3H), 7.23 (d, *J* = 7.6 Hz, 1H), 3.17 – 3.10 (m, 2H), 2.94 (t, *J* = 7.6 Hz, 2H), 2.55 (s, 3H), 2.45 (s, 3H), 2.06 – 1.96 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.47, 144.54, 140.72, 137.22, 136.21, 131.90, 131.45, 129.91, 129.87, 128.07, 127.98, 126.52, 55.82, 32.33, 29.58, 24.51, 21.63. HRMS (APCI) m/z calcd. for $C_{18}H_{21}O_3S$ $[M+H]^+$ 317.1211, found 317.1209.

1-(2-(2-methyl-3-tosylpropyl)phenyl)ethan-1-one (3Na):



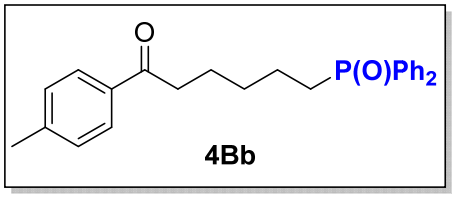
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 – 7.60 (m, 3H), 7.41 – 7.32 (m, 1H), 7.33 – 7.24 (m, 3H), 7.16 – 7.09 (m, 1H), 3.15 – 3.08 (m, 1H), 3.01 – 2.93 (m, 1H), 2.92 – 2.80 (m, 2H), 2.48 (s, 3H), 2.43 (s, 3H), 2.29 – 2.19 (m, 1H), 1.11 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.66, 144.29, 139.45, 137.74, 137.01, 132.11, 131.63, 129.88, 129.84, 127.91, 126.57, 61.49, 40.56, 30.89, 29.75, 21.70, 20.02. HRMS (APCI) m/z calcd. for $C_{19}H_{23}O_3S$ $[M+H]^+$ 331.1354, found 331.1362.

6-(diphenylphosphoryl)-1-phenylhexan-1-one (4Ab):



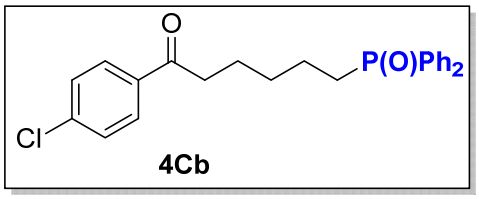
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 7.3 Hz, 2H), 7.78 – 7.70 (m, 5H), 7.54 – 7.43 (m, 7H), 7.38 (d, J = 6.7 Hz, 1H), 2.92 (t, J = 6.8 Hz, 2H), 2.28 (dd, J = 15.5, 10.4 Hz, 2H), 1.78 – 1.60 (m, 4H), 1.52 – 1.48 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.15, 136.93, 133.82 (d, $J_{C\text{-}P}$ = 97.7 Hz), 133.03, 131.74 (d, $J_{C\text{-}P}$ = 2.5 Hz), 130.86 (d, $J_{C\text{-}P}$ = 9.3 Hz), 128.76 (d, $J_{C\text{-}P}$ = 11.5 Hz), 128.52, 128.01, 38.08, 30.42 (d, $J_{C\text{-}P}$ = 14.6 Hz), 29.43 (d, $J_{C\text{-}P}$ = 71.3 Hz), 23.52, 21.21 (d, $J_{C\text{-}P}$ = 3.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.03. HRMS (APCI) m/z calcd. for $C_{24}H_{26}O_2P$ $[M+H]^+$ 377.1670, found 377.1667.

6-(diphenylphosphoryl)-1-(*p*-tolyl)hexan-1-one (4Bb):



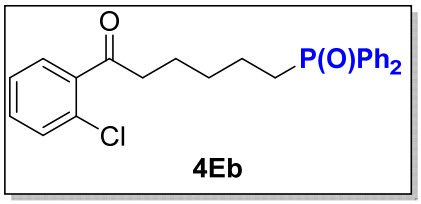
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 7.5 Hz, 2H), 7.78 – 7.68 (m, 4H), 7.50 – 7.43 (m, 6H), 7.22 (d, J = 7.3 Hz, 2H), 2.88 (t, J = 6.6 Hz, 2H), 2.38 (s, 3H), 2.32 – 2.26 (m, 2H), 1.72 – 1.65 (m, 4H), 1.51 – 1.47 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.83, 143.73, 134.44, 132.87 (d, $J_{C\text{-}P}$ = 98.0 Hz), 131.71 (d, $J_{C\text{-}P}$ = 2.4 Hz), 130.71 (d, $J_{C\text{-}P}$ = 9.7 Hz), 129.24, 128.72 (d, $J_{C\text{-}P}$ = 11.5 Hz), 128.13, 37.96, 30.40 (d, $J_{C\text{-}P}$ = 14.5 Hz), 29.42 (d, $J_{C\text{-}P}$ = 71.6 Hz), 23.62, 21.62, 21.21 (d, $J_{C\text{-}P}$ = 3.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.43. HRMS (APCI) m/z calcd. for $C_{25}H_{28}O_2P$ $[M+H]^+$ 391.1827, found 391.1823.

1-(4-chlorophenyl)-6-(diphenylphosphoryl)hexan-1-one (4Cb):



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 7.6 Hz, 2H), 7.75 – 7.71 (m, 4H), 7.55 – 7.36 (m, 8H), 2.89 (t, J = 7.1 Hz, 2H), 2.31 – 2.25 (m, 2H), 1.77 – 1.62 (m, 4H), 1.53 – 1.47 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.84, 139.33, 135.20, 132.92 (d, $J_{C\text{-}P}$ = 97.2 Hz), 131.72 (d, $J_{C\text{-}P}$ = 2.5 Hz), 130.71 (d, $J_{C\text{-}P}$ = 9.2 Hz), 129.44, 128.87, 128.75 (d, $J_{C\text{-}P}$ = 11.5 Hz), 38.02, 30.42 (d, $J_{C\text{-}P}$ = 14.3 Hz), 29.54 (d, $J_{C\text{-}P}$ = 71.7 Hz), 23.42, 21.25 ($J_{C\text{-}P}$ = 3.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.63. HRMS (APCI) m/z calcd. for $C_{24}H_{25}ClO_2P$ $[M+H]^+$ 411.1281, found 411.1274.

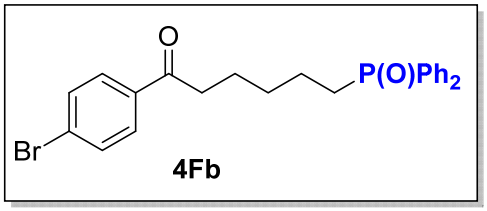
1-(2-chlorophenyl)-6-(diphenylphosphoryl)hexan-1-one (4Eb):



4Eb

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.86 (m, 1H), 7.79 – 7.69 (m, 5H), 7.51 – 7.42 (m, 7H), 7.36 (t, *J* = 7.9 Hz, 1H), 2.88 (t, *J* = 7.2 Hz, 2H), 2.33 – 2.23 (m, 2H), 1.75 – 1.61 (m, 4H), 1.52 – 1.44 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.73, 138.41, 134.88, 132.90, 132.83 (d, $J_{C\text{-}P}$ = 97.7 Hz), 131.74 (d, $J_{C\text{-}P}$ = 2.5 Hz), 130.75 (d, $J_{C\text{-}P}$ = 9.2 Hz), 129.96, 128.75 (d, $J_{C\text{-}P}$ = 11.5 Hz), 128.08, 126.12, 38.16, 30.34 (d, $J_{C\text{-}P}$ = 14.4 Hz), 29.31 (d, $J_{C\text{-}P}$ = 71.6 Hz), 23.32, 21.23 (d, $J_{C\text{-}P}$ = 3.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 33.00. HRMS (APCI) m/z calcd. for $C_{24}H_{25}ClO_2P$ $[M+H]^+$ 411.1281, found 411.1278.

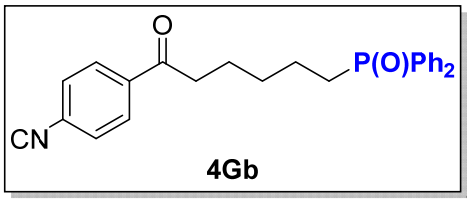
1-(4-bromophenyl)-6-(diphenylphosphoryl)hexan-1-one (4Fb):



4Fb

(47% yield, 40% starting was recovered); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 – 7.69 (m, 6H), 7.60 – 7.54 (m, 2H), 7.50 – 7.43 (m, 6H), 2.88 (t, *J* = 8.1 Hz, 2H), 2.31 – 2.24 (m, 2H), 1.76 – 1.60 (m, 4H), 1.52 – 1.47 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.00, 135.60, 133.04 (d, $J_{C\text{-}P}$ = 98.0 Hz), 131.87, 131.71 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.72 (d, $J_{C\text{-}P}$ = 9.9 Hz), 129.55, 128.72 (d, $J_{C\text{-}P}$ = 11.5 Hz), 128.16, 38.00, 30.32 (d, $J_{C\text{-}P}$ = 14.5 Hz), 29.52 (d, $J_{C\text{-}P}$ = 71.6 Hz), 23.41, 21.29 (d, $J_{C\text{-}P}$ = 3.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.49. HRMS (APCI) m/z calcd. for $C_{24}H_{25}BrO_2P$ $[M+H]^+$ 455.0776, found 455.0772.

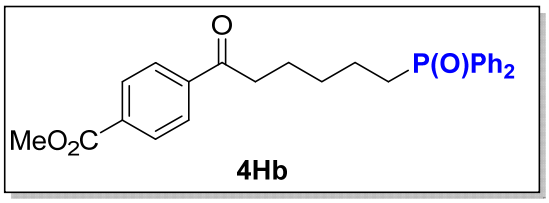
4-(6-(diphenylphosphoryl)hexanoyl)benzonitrile (4Gb):



4Gb

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.4 Hz, 2H), 7.78 – 7.70 (m, 6H), 7.58 – 7.44 (m, 6H), 2.95 (t, *J* = 7.2 Hz, 2H), 2.38 – 2.25 (m, 2H), 1.80 – 1.65 (m, 4H), 1.57 – 1.46 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.62, 139.82, 132.85 (d, *J* = 98.3 Hz), 132.50, 131.78 (d, *J* = 2.7 Hz), 130.74 (d, *J* = 9.3 Hz), 128.69 (d, *J* = 11.6 Hz), 128.42, 117.95, 116.26, 38.36, 30.26 (d, *J* = 14.4 Hz), 29.44 (d, *J* = 72.0 Hz), 23.21, 21.24 (d, *J* = 3.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.77. HRMS (APCI) m/z calcd. for $C_{25}H_{25}N_2O_2P$ $[M+H]^+$ 402.1623, found 402.1615.

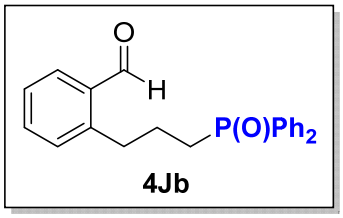
methyl 4-(6-(diphenylphosphoryl)hexanoyl)benzoate (4Hb):



4Hb

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, *J* = 8.5 Hz, 2H), 7.96 (d, *J* = 8.5 Hz, 2H), 7.77 – 7.70 (m, 4H), 7.54 – 7.43 (m, 6H), 3.95 (s, 3H), 2.96 (t, *J* = 7.2 Hz, 2H), 2.34 – 2.23 (m, 2H), 1.78 – 1.63 (m, 4H), 1.55 – 1.45 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.56, 166.23, 140.10, 133.76, 132.93 (d, *J* = 98.2 Hz), 131.73 (d, *J* = 2.8 Hz), 130.75 (d, *J* = 9.2 Hz), 129.82, 128.67 (d, *J* = 11.5 Hz), 127.90, 52.47, 38.43, 30.36 (d, *J* = 14.4 Hz), 29.48 (d, *J* = 71.9 Hz), 23.33, 21.28 (d, *J* = 3.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.65. HRMS (APCI) m/z calcd. for $C_{26}H_{28}O_4P$ $[M+H]^+$ 435.1725, found 435.1719.

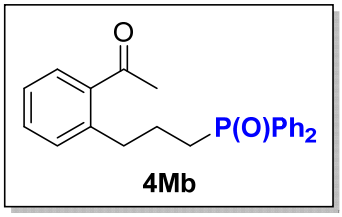
2-(3-(diphenylphosphoryl)propyl)benzaldehyde (4Jb):



4Jb

^{1}H NMR (400 MHz, $CDCl_3$) δ 10.14 (s, 1H), 7.80 – 7.69 (m, 5H), 7.55 – 7.42 (m, 7H), 7.42 – 7.36 (m, 1H), 7.29 – 7.22 (m, 1H), 3.17 (t, *J* = 7.7 Hz, 2H), 2.42 – 2.34 (m, 2H), 2.01 – 1.89 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.67, 143.59, 133.79, 133.73, 133.39, 132.84 (d, $J_{C\text{-}P}$ = 98.3 Hz), 131.75 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.12, 130.75 (d, $J_{C\text{-}P}$ = 9.3 Hz), 128.68 (d, $J_{C\text{-}P}$ = 11.5 Hz), 126.85, 33.46 (d, $J_{C\text{-}P}$ = 14.9 Hz), 29.29 (d, $J_{C\text{-}P}$ = 71.8 Hz), 23.67 (d, $J_{C\text{-}P}$ = 3.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.53. HRMS (APCI) m/z calcd. for $C_{22}H_{22}O_2P$ $[M+H]^+$ 349.1345, found 349.1352.

1-(2-(3-(diphenylphosphoryl)propyl)phenyl)ethanone (4Mb):

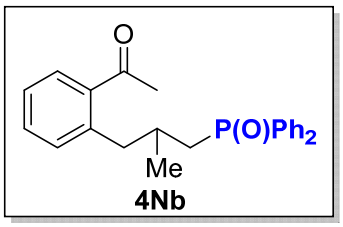


4Mb

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 – 7.65 (m, 4H), 7.56 (d, *J* = 7.7 Hz, 1H), 7.45 – 7.32 (m, 6H), 7.28 (t, *J* = 7.4 Hz, 1H), 7.19 – 7.13 (m, 2H), 2.98 – 2.86 (m, 2H), 2.42 (s, 3H), 2.33 – 2.26 (m, 2H), 1.94 – 1.80 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.71, 141.29, 137.44, 133.02 (d, $J_{C\text{-}P}$ = 97.3 Hz), 131.62 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.63, 131.20, 130.70 (d, $J_{C\text{-}P}$ = 9.2 Hz), 129.49, 128.65 (d, $J_{C\text{-}P}$ = 11.5 Hz), 126.15, 34.82 (d, $J_{C\text{-}P}$ = 15.5 Hz), 29.70, 29.31 (d, $J_{C\text{-}P}$ = 70.0 Hz), 23.52 (d, $J_{C\text{-}P}$ = 3.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.46. HRMS (APCI) m/z calcd. for $C_{23}H_{24}O_2P$ $[M+H]^+$

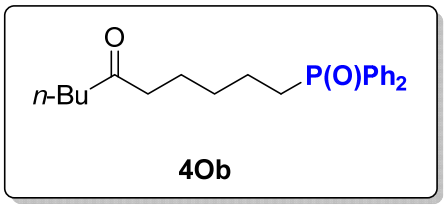
363.1514, found 363.1510.

1-(2-(3-(diphenylphosphoryl)-2-methylpropyl)phenyl)ethan-1-one (4Nb):



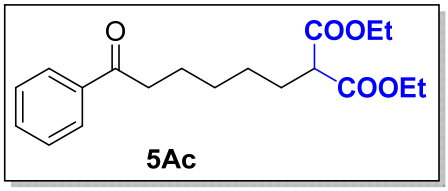
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 – 7.66 (m, 2H), 7.64 – 7.55 (m, 3H), 7.51 – 7.35 (m, 7H), 7.33 – 7.27 (m, 1H), 7.17 (d, J = 7.6 Hz, 1H), 2.98 – 2.87 (m, 2H), 2.47 – 2.39 (m, 4H), 2.19 – 2.12 (m, 2H), 1.08 (d, J = 6.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.16, 140.10, 138.25, 134.56 (d, $J_{C\text{-}P}$ = 97.8 Hz), 132.80 (d, $J_{C\text{-}P}$ = 97.5 Hz), 132.14, 131.46 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.26, 131.40 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.87 (d, $J_{C\text{-}P}$ = 9.1 Hz), 130.46 (d, $J_{C\text{-}P}$ = 9.2 Hz), 129.33, 128.61 (d, $J_{C\text{-}P}$ = 3.0 Hz), 128.50 (d, $J_{C\text{-}P}$ = 3.1 Hz), 126.21, 42.57 (d, $J_{C\text{-}P}$ = 13.9 Hz), 35.63 (d, $J_{C\text{-}P}$ = 71.3 Hz), 30.81 (d, $J_{C\text{-}P}$ = 3.5 Hz), 29.85, 21.33 (d, $J_{C\text{-}P}$ = 3.0 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.82. HRMS (APCI) m/z calcd. for $C_{24}H_{26}O_2P$ $[M+H]^+$ 377.1653, found 377.1665.

10-(diphenylphosphoryl)decan-5-one (4Ob):



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.75 – 7.67 (m, 4H), 7.52 – 7.42 (m, 6H), 2.33 (td, J = 7.4, 1.8 Hz, 4H), 2.29 – 2.18 (m, 2H), 1.63 – 1.58 (m, 2H), 1.56 – 1.46 (m, 4H), 1.42 – 1.32 (m, 2H), 1.31 – 1.21 (m, 2H), 0.87 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 211.26, 132.98 (d, $J_{C\text{-}P}$ = 98.0 Hz), 131.71 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.73 (d, $J_{C\text{-}P}$ = 9.9 Hz), 128.65 (d, $J_{C\text{-}P}$ = 11.5 Hz), 42.57, 42.20, 30.34 (d, $J_{C\text{-}P}$ = 14.5 Hz), 29.44 (d, $J_{C\text{-}P}$ = 71.6 Hz), 25.92, 23.05, 22.32, 21.21 (d, $J_{C\text{-}P}$ = 3.7 Hz), 13.86. ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.70. HRMS (APCI) m/z calcd. for $C_{22}H_{30}O_2P$ $[M+H]^+$ 357.1983, found 357.1977.

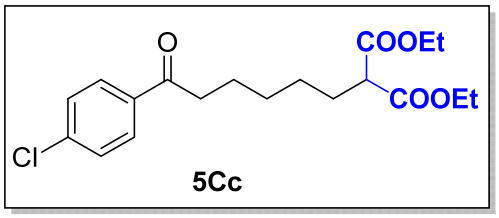
diethyl 2-(6-oxo-6-phenylhexyl)malonate (5Ac):



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (d, J = 7.6 Hz, 2H), 7.57 (t, J = 7.4 Hz, 1H), 7.47 (t, J = 7.6 Hz, 2H), 4.25 – 4.16 (m, 4H), 3.33 (t, J = 7.5 Hz, 1H), 2.97 (t, J = 7.3 Hz, 2H), 1.96 – 1.90 (m, 2H), 1.80 – 1.72 (m, 2H), 1.48 – 1.37 (m, 4H), 1.28 (t, J = 7.1 Hz,

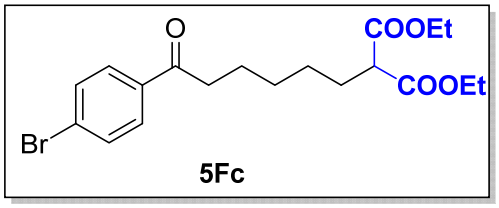
6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.29, 169.53, 137.01, 132.94, 128.58, 128.04, 61.31, 51.99, 38.37, 28.93, 28.58, 27.20, 23.98, 14.09. HRMS (APCI) m/z calcd. for $C_{19}H_{27}O_5$ $[M+H]^+$ 335.1858, found 335.1857.

diethyl 2-(6-(4-chlorophenyl)-6-oxohexyl)malonate (5Cc):



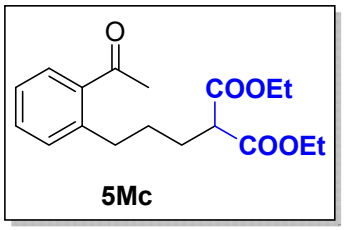
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.6 Hz, 2H), 7.45 (d, *J* = 8.6 Hz, 2H), 4.27 – 4.15 (m, 4H), 3.33 (t, *J* = 7.5 Hz, 1H), 2.94 (t, *J* = 7.3 Hz, 2H), 1.92 (dd, *J* = 14.9, 7.5 Hz, 2H), 1.80 – 1.70 (m, 2H), 1.44 – 1.40 (m, 4H), 1.28 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.99, 169.51, 139.36, 135.30, 129.47, 128.89, 61.32, 51.98, 38.34, 28.87, 28.55, 27.16, 23.87, 14.09. HRMS (APCI) m/z calcd. for $C_{19}H_{26}O_5Cl$ $[M+H]^+$ 369.1469, found 369.1466.

diethyl 2-(6-(4-bromophenyl)-6-oxohexyl)malonate (5Fc):



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 8.5 Hz, 2H), 7.59 (d, *J* = 8.5 Hz, 2H), 4.24 – 4.13 (m, 4H), 3.31 (t, *J* = 7.5 Hz, 1H), 2.92 (t, *J* = 7.3 Hz, 2H), 1.93 – 1.88 (m, 2H), 1.77 – 1.69 (m, 2H), 1.42 – 1.38 (m, 4H), 1.26 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.14, 169.49, 135.68, 131.87, 129.58, 128.05, 61.31, 51.95, 38.30, 28.84, 28.53, 27.15, 23.83, 14.09. HRMS (APCI) m/z calcd. for $C_{19}H_{26}O_5Br$ $[M+H]^+$ 413.0964, found 413.0955.

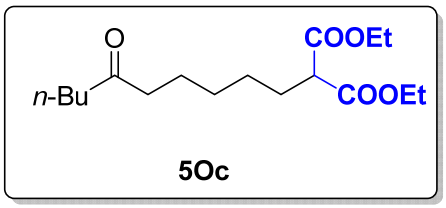
diethyl 2-(3-(2-acetylphenyl)propyl)malonate (5Mc):



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66 (dd, *J* = 7.7, 1.1 Hz, 1H), 7.38 (dt, *J* = 7.5, 3.8 Hz, 1H), 7.30 – 7.19 (m, 2H), 4.26 – 4.10 (m, 4H), 3.36 (t, *J* = 7.5 Hz, 1H), 2.93 – 2.79 (m, 2H), 2.57 (s, 3H), 1.99 – 1.93 (m, 2H), 1.66 – 1.58 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.86, 169.47, 141.96, 137.65, 131.52, 131.26, 129.36, 125.97, 61.26, 51.81, 33.69, 29.78, 29.22, 28.65, 14.07. HRMS (APCI) m/z

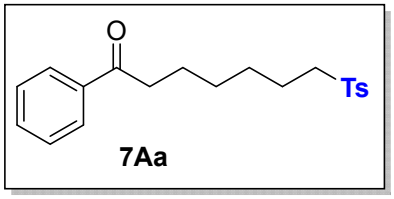
calcd. for $C_{18}H_{25}O_5$ $[M+H]^+$ 321.1702, found 321.1695.

diethyl 2-(6-oxodecyl)malonate (5Oc)



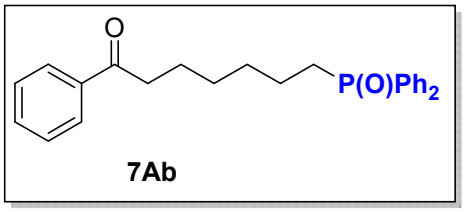
^{1}H NMR (400 MHz, $CDCl_3$) δ 4.19 (qd, *J* = 7.1, 1.0 Hz, 4H), 3.30 (t, *J* = 7.5 Hz, 1H), 2.39 (t, *J* = 7.5 Hz, 4H), 1.88 (dd, *J* = 15.0, 7.5 Hz, 2H), 1.60 – 1.50 (m, 4H), 1.36 – 1.24 (m, 12H), 0.90 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 211.41, 169.51, 61.30, 51.96, 42.55, 42.53, 28.82, 28.53, 27.12, 25.96, 23.48, 22.36, 14.08, 13.86. HRMS (ESI) m/z calcd. for $C_{17}H_{31}O_5$ $[M+H]^+$ 315.2171, found 315.2166.

1-phenyl-7-tosylheptan-1-one (7Aa)



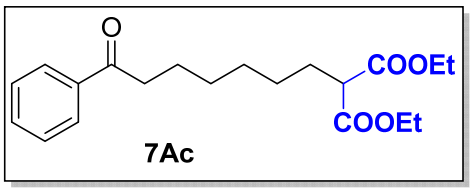
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 7.5 Hz, 2H), 7.77 (d, *J* = 8.2 Hz, 2H), 7.55 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 8.0 Hz, 2H), 3.12 – 3.03 (m, 2H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.44 (s, 3H), 1.75 – 1.66 (m, 4H), 1.43 – 1.33 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.12, 144.60, 136.96, 136.24, 133.00, 129.90, 128.60, 128.17, 128.01, 56.30, 38.24, 28.71, 28.17, 23.82, 22.65, 21.62. HRMS (APCI) m/z calcd. for $C_{20}H_{25}O_3S$ $[M+H]^+$ 345.1524, found 345.1520.

7-(diphenylphosphoryl)-1-phenylheptan-1-one (7Ab):



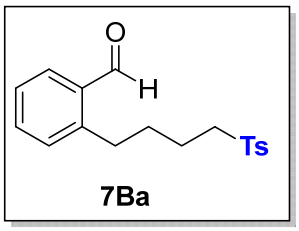
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.4 Hz, 2H), 7.78 – 7.64 (m, 4H), 7.55 – 7.34 (m, 9H), 2.89 (t, *J* = 7.3 Hz, 2H), 2.31 – 2.17 (m, 2H), 1.68 – 1.62 (m, 4H), 1.48 – 1.28 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.28, 136.95, 133.12 (d, J_{C-P} = 97.3 Hz), 132.93, 131.61 (d, J_{C-P} = 2.6 Hz), 130.74 (d, J_{C-P} = 9.2 Hz), 128.64 (d, J_{C-P} = 11.6 Hz), 128.56, 127.99, 38.34, 30.83 (d, J_{C-P} = 14.3 Hz), 29.61 (d, J_{C-P} = 71.6 Hz), 28.75, 23.96, 21.33 (d, J_{C-P} = 3.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.52. HRMS (APCI) m/z calcd. for $C_{25}H_{28}O_2P$ $[M+H]^+$ 391.1827, found 391.1824.

diethyl 2-(7-oxo-7-phenylheptyl)malonate (7Ac):



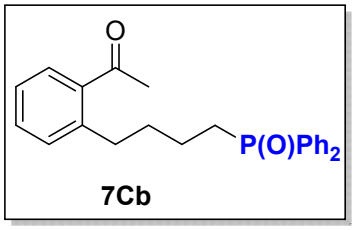
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.94 (m, 2H), 7.59 – 7.53 (m, 1H), 7.48 – 7.45 (m, 2H), 4.23 – 4.16 (m, 4H), 3.32 (t, *J* = 7.5 Hz, 1H), 2.97 (t, *J* = 7.4 Hz, 2H), 1.93 – 1.88 (m, 2H), 1.76 – 1.70 (m, 2H), 1.43 – 1.34 (m, 6H), 1.27 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.41, 169.56, 137.06, 132.90, 128.56, 128.03, 61.27, 52.03, 38.48, 29.05, 29.02, 28.67, 27.18, 24.17, 14.08. HRMS (APCI) m/z calcd. for $C_{20}H_{29}O_5$ $[M+H]^+$ 349.2015, found 349.2013.

2-(4-tosylbutyl)benzaldehyde (7Ba):



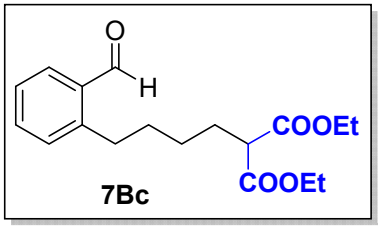
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.16 (s, 1H), 7.88 – 7.74 (m, 3H), 7.51 (t, *J* = 7.0 Hz, 1H), 7.44 – 7.35 (m, 3H), 7.23 (d, *J* = 7.6 Hz, 1H), 3.17 – 3.09 (m, 2H), 3.04 – 2.99 (m, 2H), 2.48 (s, 3H), 1.88 – 1.76 (m, 2H), 1.74 – 1.62 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.79, 144.67, 143.97, 136.16, 133.83, 133.64, 131.13, 129.93, 129.89, 128.10, 126.81, 56.09, 32.31, 30.29, 22.60, 21.65. HRMS (APCI) m/z calcd. for $C_{18}H_{21}O_3S$ $[M+H]^+$ 317.1199, found 317.1206.

2-(4-(diphenylphosphoryl)butyl)benzaldehyde (7Cb):



^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (ddd, *J* = 11.4, 8.1, 1.6 Hz, 4H), 7.67 – 7.60 (m, 1H), 7.57 – 7.41 (m, 6H), 7.36 (td, *J* = 7.5, 1.4 Hz, 1H), 7.31 – 7.20 (m, 1H), 7.19 (dd, *J* = 7.6, 1.3 Hz, 1H), 2.83 (t, *J* = 7.2 Hz, 2H), 2.55 (s, 3H), 2.37 – 2.27 (m, 2H), 1.77 – 1.66 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.01, 142.15, 137.59, 133.07 (d, $J_{C\text{-}P}$ = 98.2 Hz), 131.65 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.52, 131.27, 130.79 (d, $J_{C\text{-}P}$ = 9.3 Hz), 129.29, 128.63 (d, $J_{C\text{-}P}$ = 11.6 Hz), 125.86, 33.53, 32.88 (d, $J_{C\text{-}P}$ = 14.8 Hz), 29.82, 29.11, 21.48 (d, $J_{C\text{-}P}$ = 3.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 32.73. HRMS (APCI) m/z calcd. for $C_{24}H_{26}O_2P$ $[M+H]^+$ 377.1658, found 377.1665.

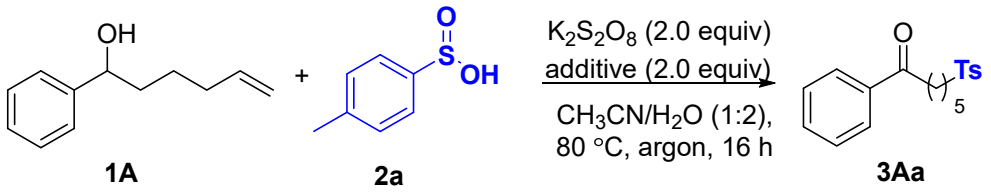
diethyl 2-(4-(2-formylphenyl)butyl)malonate (7Bc):



7Bc

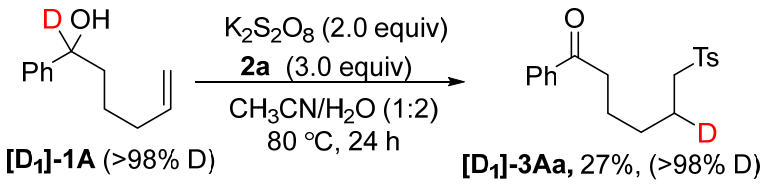
^{1}H NMR (400 MHz, $CDCl_3$) δ 10.27 (s, 1H), 7.84 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.52 (td, *J* = 7.5, 1.5 Hz, 1H), 7.39 (td, *J* = 7.5, 1.2 Hz, 1H), 7.31 – 7.24 (m, 1H), 4.21 (qd, *J* = 7.1, 1.2 Hz, 4H), 3.34 (t, *J* = 7.5 Hz, 1H), 3.08 – 3.02 (m, 2H), 2.01 – 1.89 (m, 2H), 1.72 – 1.61 (m, 2H), 1.50 – 1.38 (m, 2H), 1.28 (t, *J* = 7.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 192.40, 169.50, 145.12, 133.78, 133.66, 132.13, 131.01, 126.55, 63.24, 51.92, 32.28, 31.68, 28.51, 27.17, 14.08. HRMS (APCI) m/z calcd. for $C_{18}H_{25}O_5P$ $[M+H]^+$ 321.1689, found 321.1697.

Mechanistic Studies:



additive: BQ (2.0 equiv), **3Aa** (0%)
TEMPO (2.0 equiv), **3Aa** (22%)

Procedure: To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **1A** (0.2 mmol), **2a** (0.6 mmol) and $K_2S_2O_8$ (0.4 mmol) and additive (0.4 mmol). The tube was evacuated and backfilled with argon for three times, and then CH_3CN (0.7 mL) and H_2O (1.4 mL) were added. The tube was stirred at 80 °C for 18 h and then H_2O (5 mL) was added. EtOAc was used to extract the product from the aqueous layer (3 × 20 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to afford the crude product. When TEMPO was used as the additive, the ^{1}H NMR yield of **3Aa** is 22%. When *p*-benzoquinone was used as the additive, no **3Aa** was observed and **1A** was recovered.

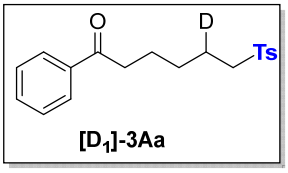


[D$_1$]-1A (>98% D)

[D$_1$]-3Aa, 27%, (>98% D)

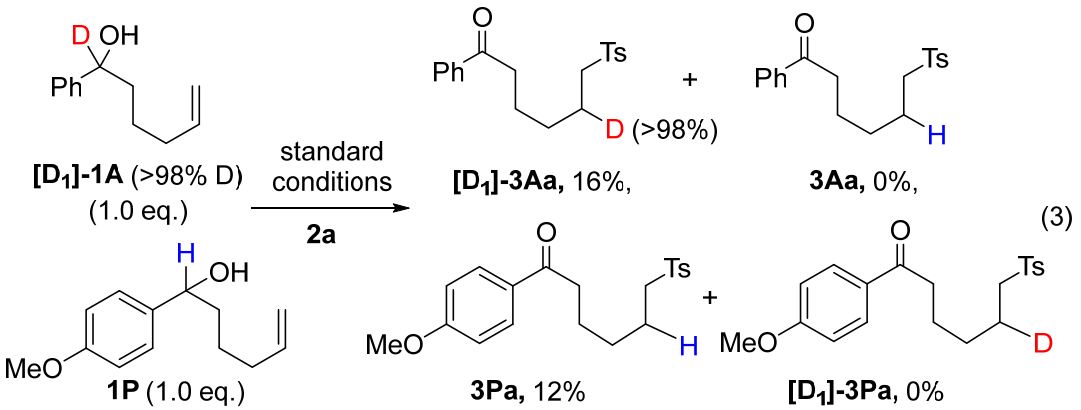
Procedure: To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **[D$_1$]-1A** (0.2 mmol), **2a** (0.6 mmol) and $K_2S_2O_8$ (0.4 mmol). The tube was evacuated and backfilled with argon for three times, and then CH_3CN (0.7 mL) and H_2O (1.4 mL) were added. The tube was stirred at 80 °C for 24 h and then H_2O (5 mL) was added. EtOAc was used to extract the product from the aqueous layer (3 × 20 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to afford the crude product, which was purified by flash column chromatography to afford the product **[D$_1$]-3Aa** (27%) and 35% starting material **[D$_1$]-1A** was recovered.

Compound [D$_1$]-3Aa:



(35% starting recovered, 27% yield) ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.89 (m, 2H), 7.80 (d, *J* = 8.2 Hz, 2H), 7.59 – 7.56 (m, 1H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.37 (d, *J* = 8.0 Hz, 2H), 3.10 (d, *J* = 8.0 Hz, 2H), 2.96 (t, *J* = 7.2 Hz, 2H), 2.46 (s, 3H), 1.78 – 1.69 (m, 3H), 1.50 – 1.44 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.77, 144.66,

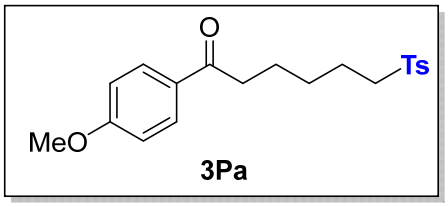
136.85, 136.18, 133.09, 129.92, 128.63, 128.08, 127.99, 56.10, 37.94, 27.81, 23.42, 22.36 (t, J = 20 Hz), 21.64. HRMS (APCI) m/z calcd. for $C_{19}H_{22}DO_3S$ $[M+H]^+$ 332.1432, found 332.1428.



(3)

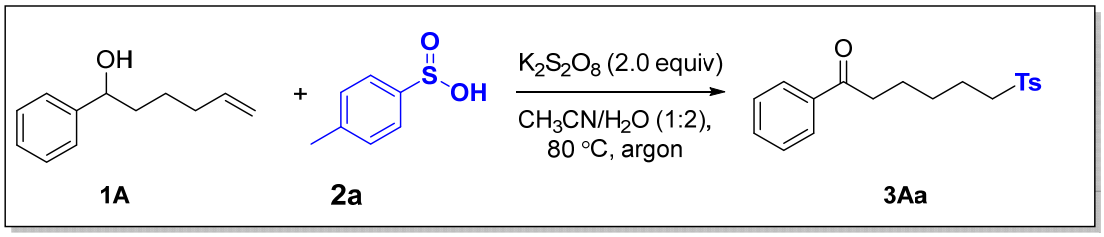
Procedure: To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **[D$_1$]-1A** (0.2 mmol), **1P** (0.2 mmol), **2a** (1.2 mmol) and $K_2S_2O_8$ (0.8 mmol). The tube was evacuated and backfilled with argon for three times, and then CH_3CN (0.7 mL) and H_2O (1.4 mL) were added. The tube was stirred at 80 °C for 24 h and then H_2O (5 mL) was added. EtOAc was used to extract the product from the aqueous layer (3 × 20 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to afford the crude product, which was purified by flash column chromatography to afford **3Pa** (12%) and **[D$_1$]-3Aa** (16%). Meanwhile, about 35% **[D$_1$]-1A** was recovered.

1-(4-methoxyphenyl)-6-tosylhexan-1-one (3Pa):



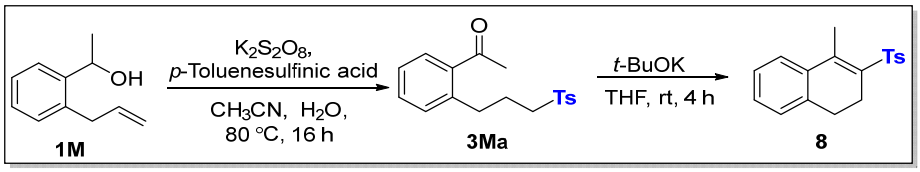
^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.9 Hz, 2H), 7.79 (d, J = 8.2 Hz, 2H), 7.37 (d, J = 8.0 Hz, 2H), 6.94 (d, J = 8.9 Hz, 2H), 3.88 (s, 3H), 3.15 – 3.06 (m, 2H), 2.91 (t, J = 7.2 Hz, 2H), 2.47 (s, 3H), 1.81 – 1.68 (m, 4H), 1.50 – 1.44 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 198.38, 163.47, 144.64, 136.18, 130.26, 129.96, 129.91, 128.08, 113.74, 56.18, 55.49, 37.57, 27.95, 23.67, 22.69, 21.62. HRMS (APCI) m/z calcd. for $C_{20}H_{25}O_4S$ $[M+H]^+$ 361.1474, found 361.1470.

Large scale preparation and transformations

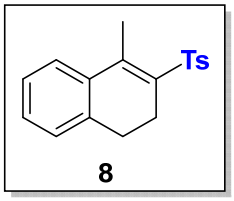


To a flame-dried Schlenk tube equipped with a magnetic stir bar were added **1A** (7.2 mmol), **2a** (21.6 mmol) and $K_2S_2O_8$ (14.4 mmol). The tube was evacuated and backfilled with argon for three times, and then CH_3CN (20 mL) and H_2O (40 mL) were added. The tube was stirred at 80 °C for 18 h and then H_2O (50 mL) was added. EtOAc was used to extract the product from the aqueous layer (3 × 40 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to afford the crude product, which was purified by flash column chromatography to afford 1.7 g product of **3Aa** (70%).

Transformation of 3Ma

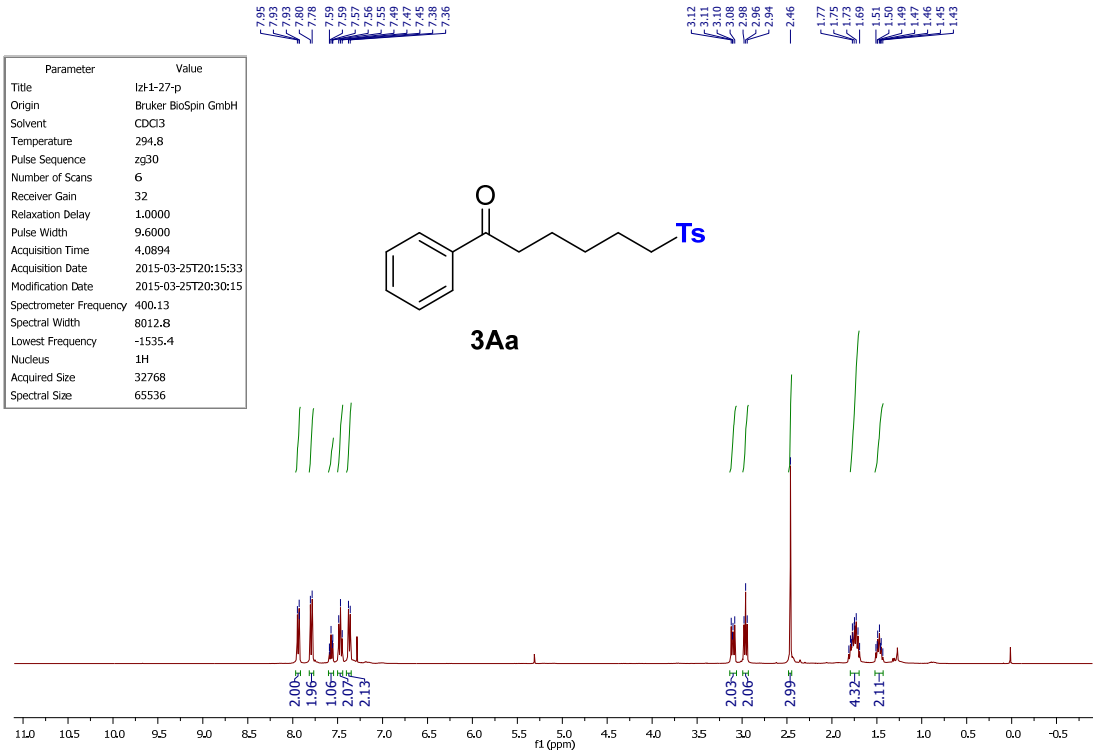


Compound **3Ma** was obtained by following the reported sulfonylation procedure.
Procedure: To a solution of *t*-BuOK (74 mg, 0.66 mmol) in anhydrous THF (8 mL) was added a solution of **3Ma** (70 mg, 0.22 mmol) in THF dropwisely. The reaction was stirred 4 h and quenched with water (5 mL). EtOAc was used to extract the product from the aqueous layer (3 × 20 mL). The combined organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to afford the crude product, which was purified by flash column chromatography to afford the product **8** in 73% yield.



4-methyl-3-tosyl-1,2-dihydronaphthalene (8): (73% isolated yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, J = 8.1 Hz, 2H), 7.49 – 7.45 (m, 1H), 7.34 (d, J = 8.0 Hz, 2H), 7.30 – 7.26 (m, 2H), 7.19 – 7.15 (m, 1H), 2.82 – 2.75 (m, 2H), 2.71 – 2.68 (m, 2H), 2.57 (s, 3H), 2.45 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.92, 142.49, 139.29, 136.77, 135.26, 135.10, 129.75, 129.66, 127.40, 127.06, 126.90, 125.26, 28.37, 24.78, 21.61, 15.70. HRMS (APCI) m/z calcd. for $C_{18}H_{19}O_2S$ $[M+H]^+$ 299.1106, found 299.1100.

NMR Spectra:



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Title	lzl-1-27-p
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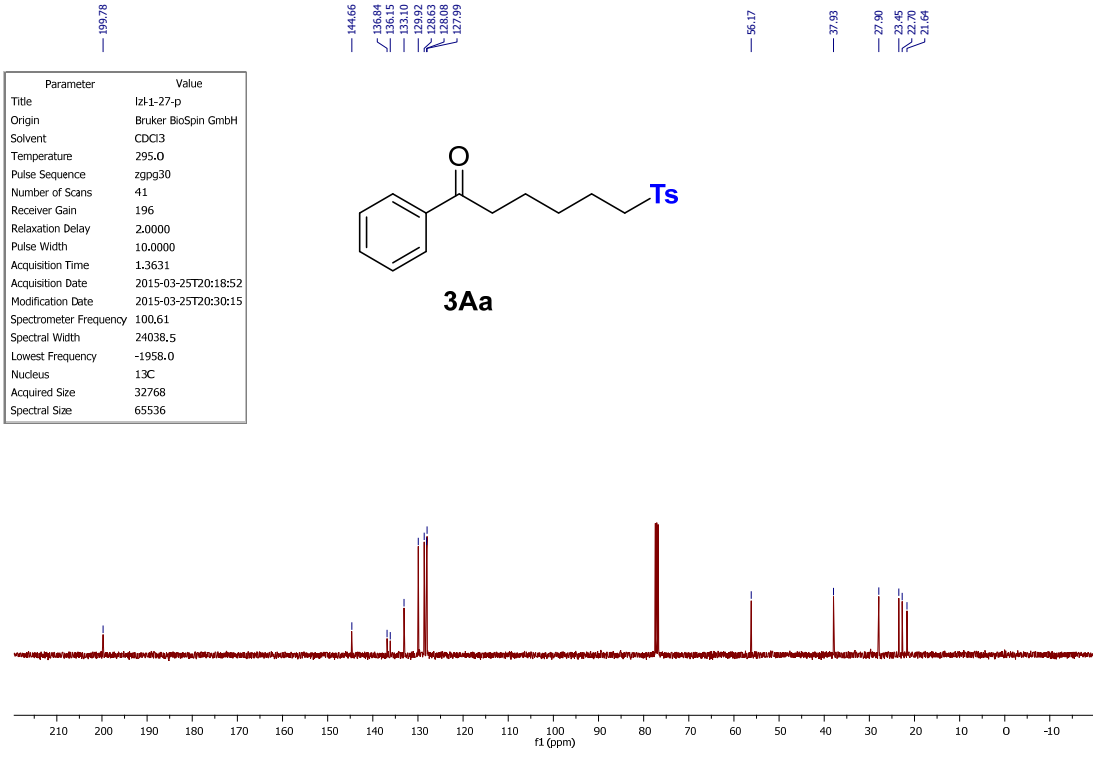
Solvent	CDCl3
Temperature	294.8
Pulse Sequence	zg30
Number of Scans	6
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	4.0894
Acquisition Date	2015-03-25T20:15:33
Modification Date	2015-03-25T20:30:15
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



Parameter	Value
Title	lzl-1-27-p
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zgpg30
Number of Scans	41
Receiver Gain	196
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Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2015-03-25T20:18:52
Modification Date	2015-03-25T20:30:15
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Spectral Width	24038.5
Lowest Frequency	-1958.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

Parameter	Value
Title	lzl-1-44
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Pulse Sequence	zg30
Number of Scans	7
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	1.9999
Acquisition Date	2015-04-02T16:01:49
Modification Date	2015-04-07T20:26:22
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
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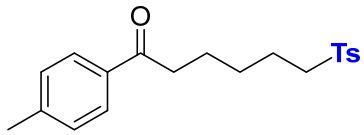
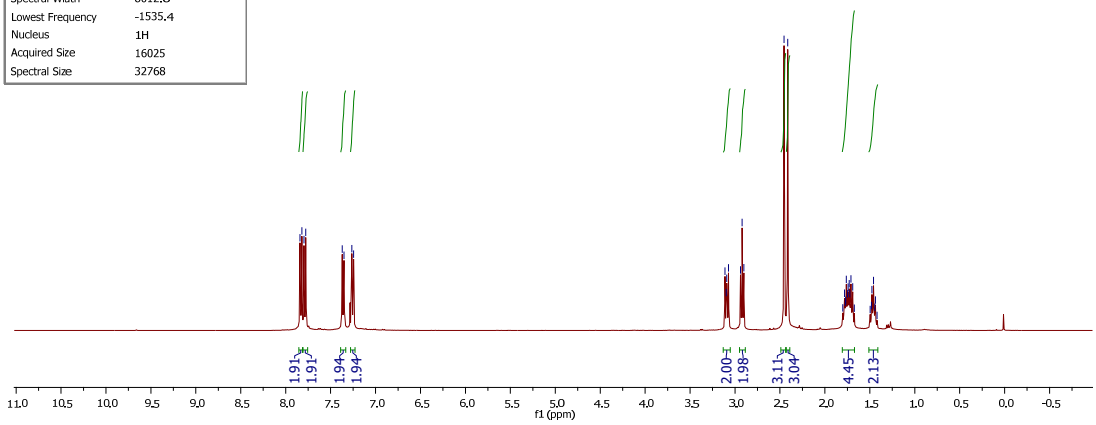


3Ba



Parameter	Value
Title	lzl-1-44
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.4
Pulse Sequence	zgpg30
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Spectrometer Frequency	100.61
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Nucleus	13C
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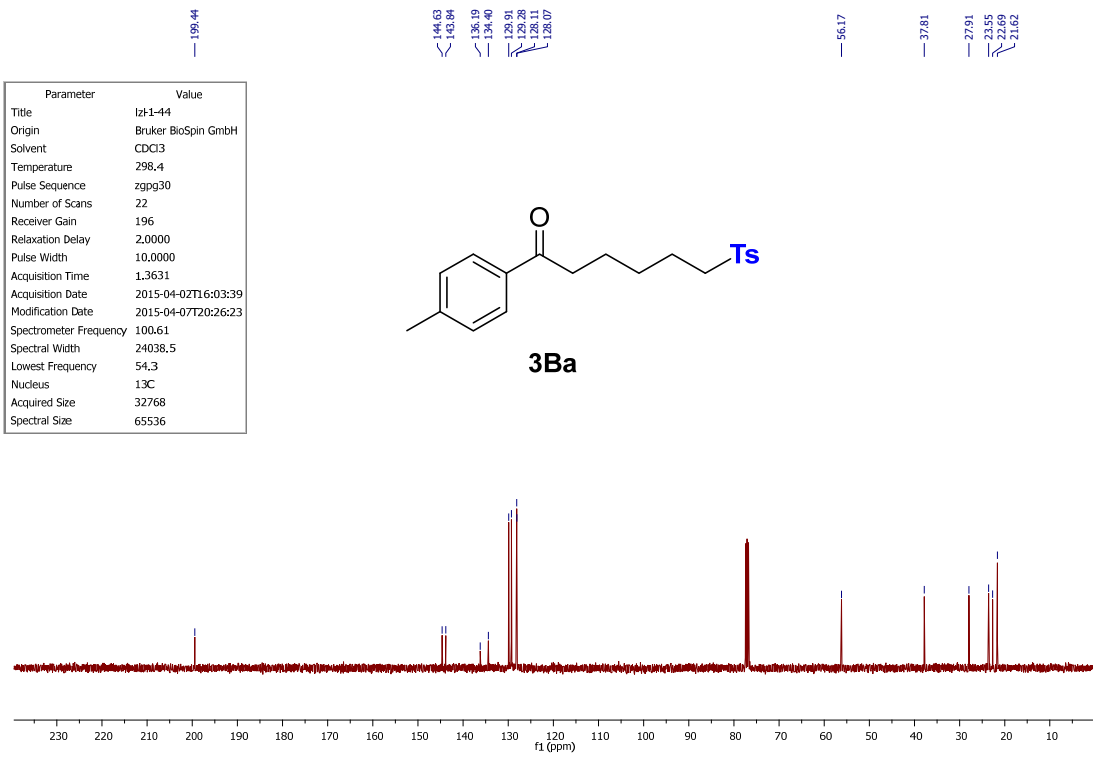
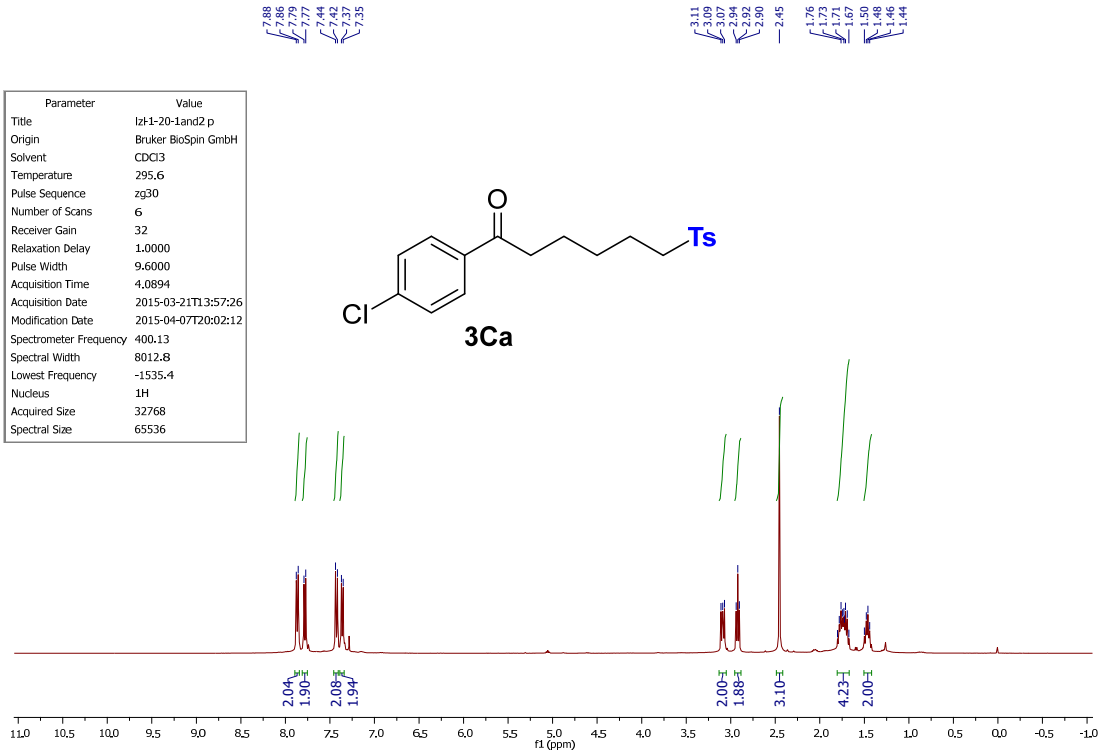
Spectral Size	65536

3Ba




7.88
7.86
7.79
7.77
7.44
7.42
7.37
7.35
3.11
3.09
3.07
2.94
2.92
2.90
2.45
1.76
1.73
1.71
1.67
1.50
1.48
1.46
1.44
Parameter Value
Title lzl-1-20-1and2 p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.6
Pulse Sequence zg30
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Receiver Gain 32
Relaxation Delay 1.0000
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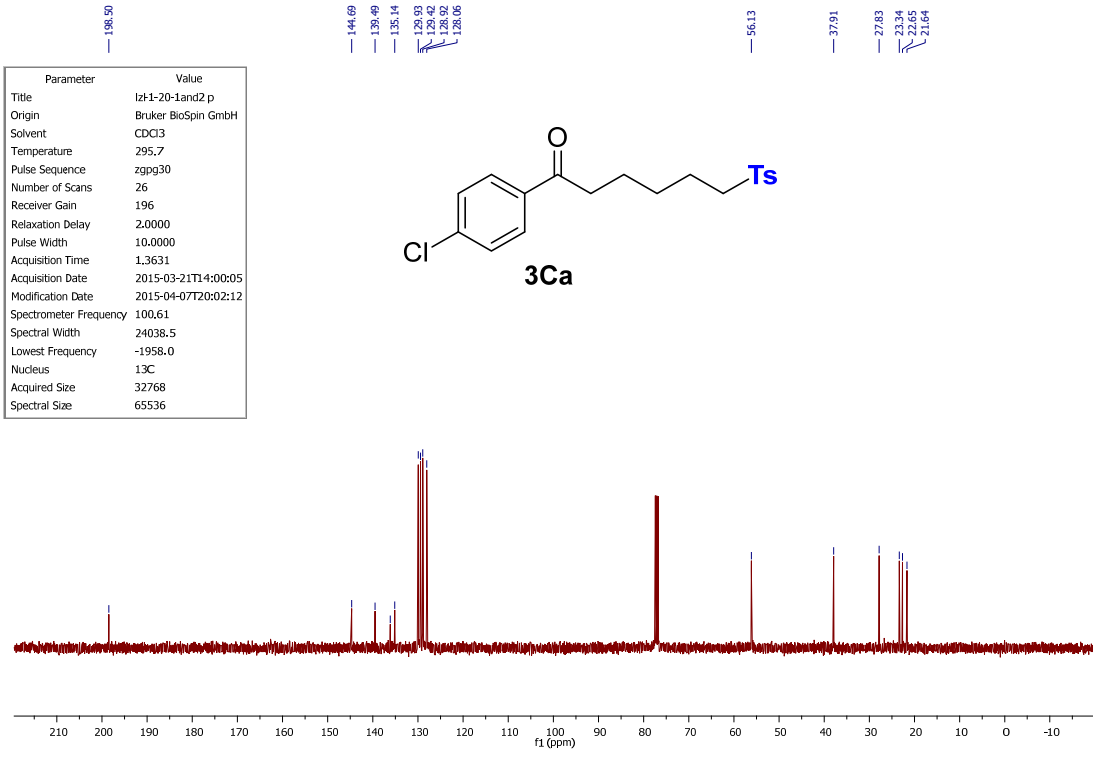
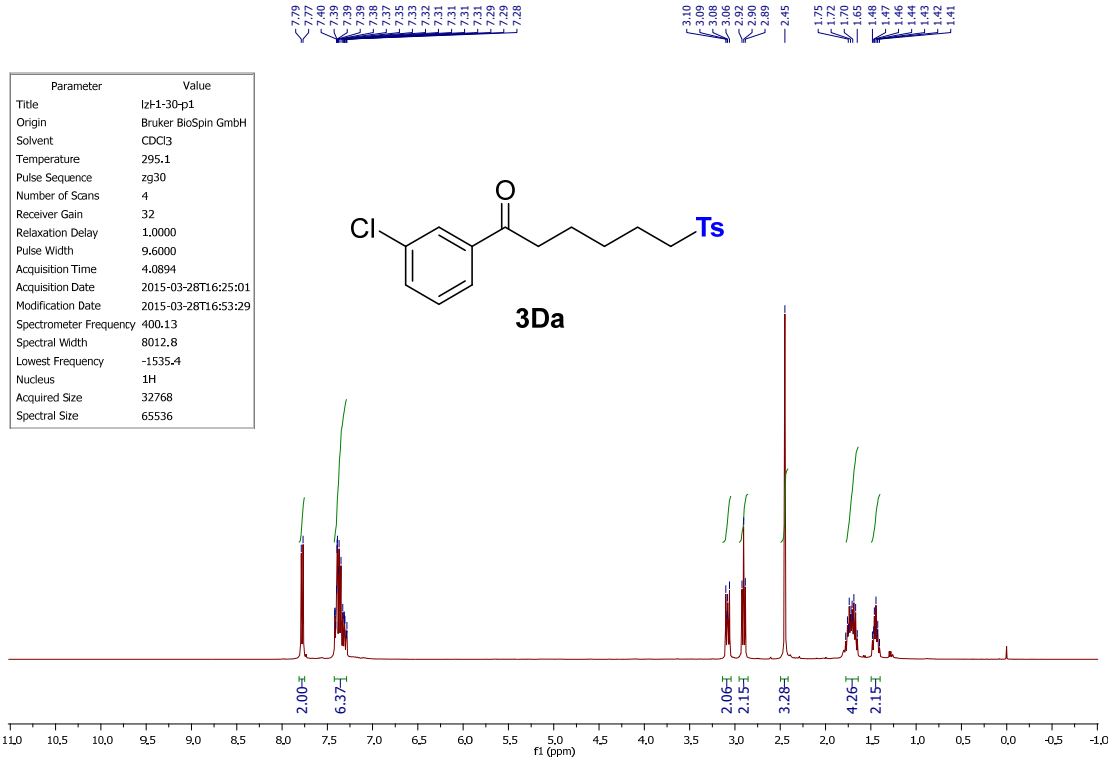
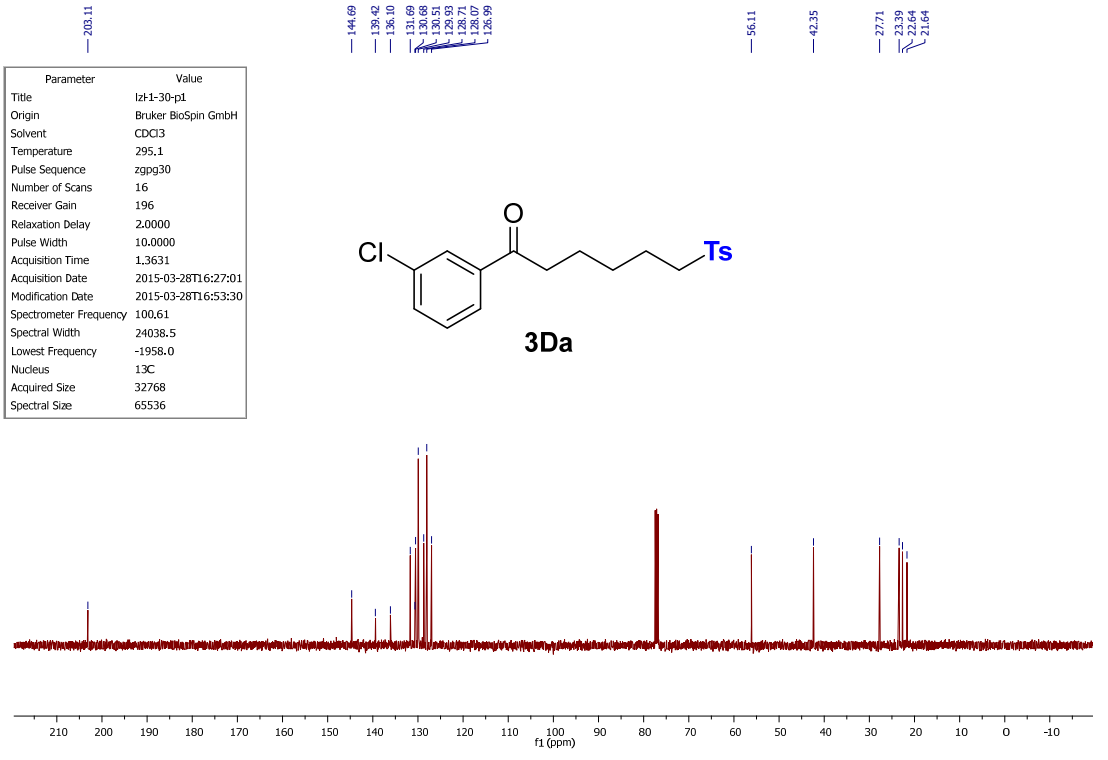
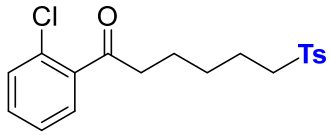
Acquisition Time 4.0894
Acquisition Date 2015-03-21T13:57:26
Modification Date 2015-04-07T20:02:12
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
O
Ts
Cl
3Ca
2.04
1.90
2.08
1.94
2.00
1.88
3.10
4.23
2.00
f1 (ppm)


198.50
144.69
139.49
135.14
129.93
129.42
128.92
128.06
56.13
37.91
27.83
23.34
22.65
21.64
Parameter Value
Title lzl-1-20-1and2 p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.7
Pulse Sequence zgpg30
Number of Scans 26
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-03-21T14:00:05
Modification Date 2015-04-07T20:02:12
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
Ts
Cl
3Ca
f1 (ppm)

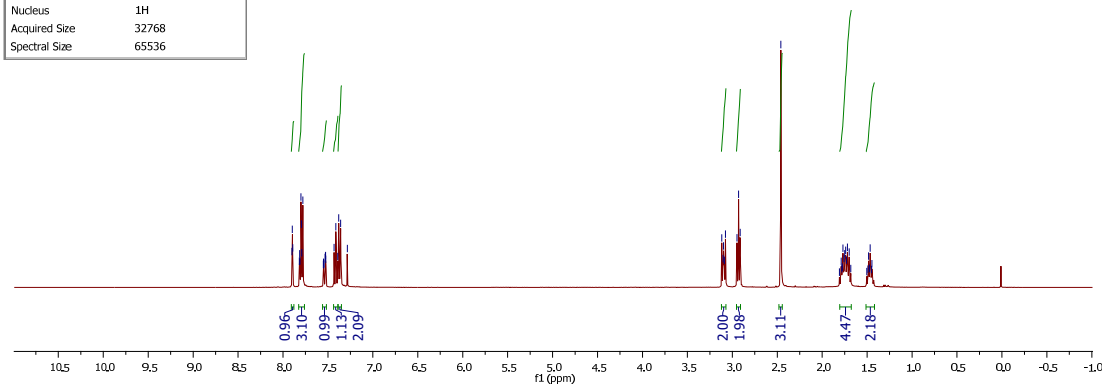

7.79
7.77
7.40
7.39
7.39
7.39
7.38
7.37
7.35
7.33
7.32
7.31
7.31
7.31
7.31
7.29
7.29
7.28
3.10
3.09
3.08
3.06
2.92
2.90
2.89
2.45
1.75
1.72
1.70
1.65
1.48
1.47
1.46
1.44
1.43
1.42
1.41
Parameter Value
Title lzl-1-30-p1
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.1
Pulse Sequence zg30
Number of Scans 4
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 4.0894
Acquisition Date 2015-03-28T16:25:01
Modification Date 2015-03-28T16:53:29
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
Cl
O
Ts
3Da
2.00
6.37
2.06
2.15
3.28
4.26
2.15
f1 (ppm)


203.11
144.69
139.42
136.10
131.69
130.68
130.51
129.93
128.71
128.07
126.99
56.11
42.35
27.71
23.39
22.64
21.64
Parameter Value
Title lzl-1-30-p1
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.1
Pulse Sequence zgpg30
Number of Scans 16
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-03-28T16:27:01
Modification Date 2015-03-28T16:53:30
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
Cl
O
Ts
3Da
f1 (ppm)

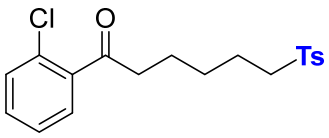
Parameter	Value
Title	lzl-1-31
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.9
Pulse Sequence	zg30
Number of Scans	4
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	4.0894
Acquisition Date	2015-03-30T16:03:20
Modification Date	2015-03-30T17:33:32
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



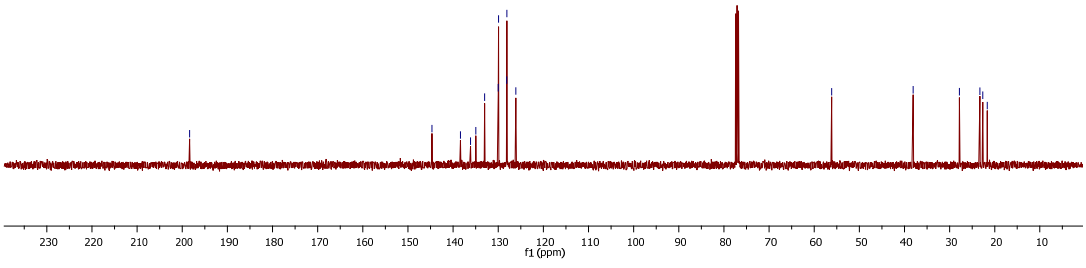
3Ea

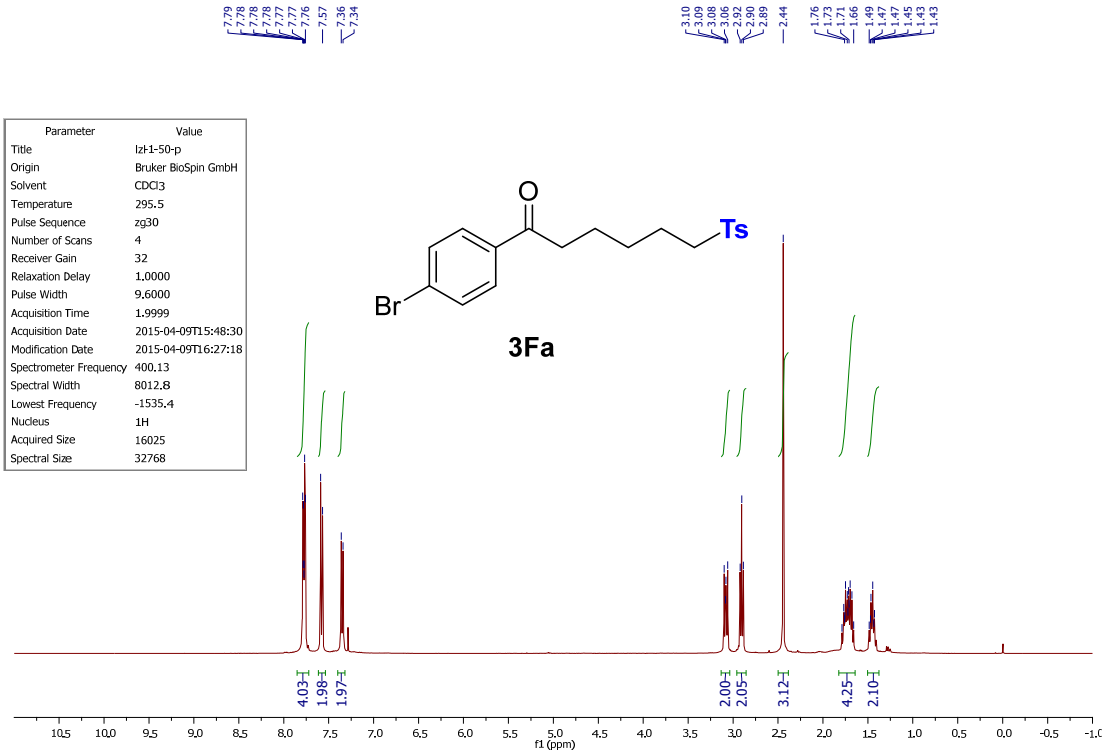


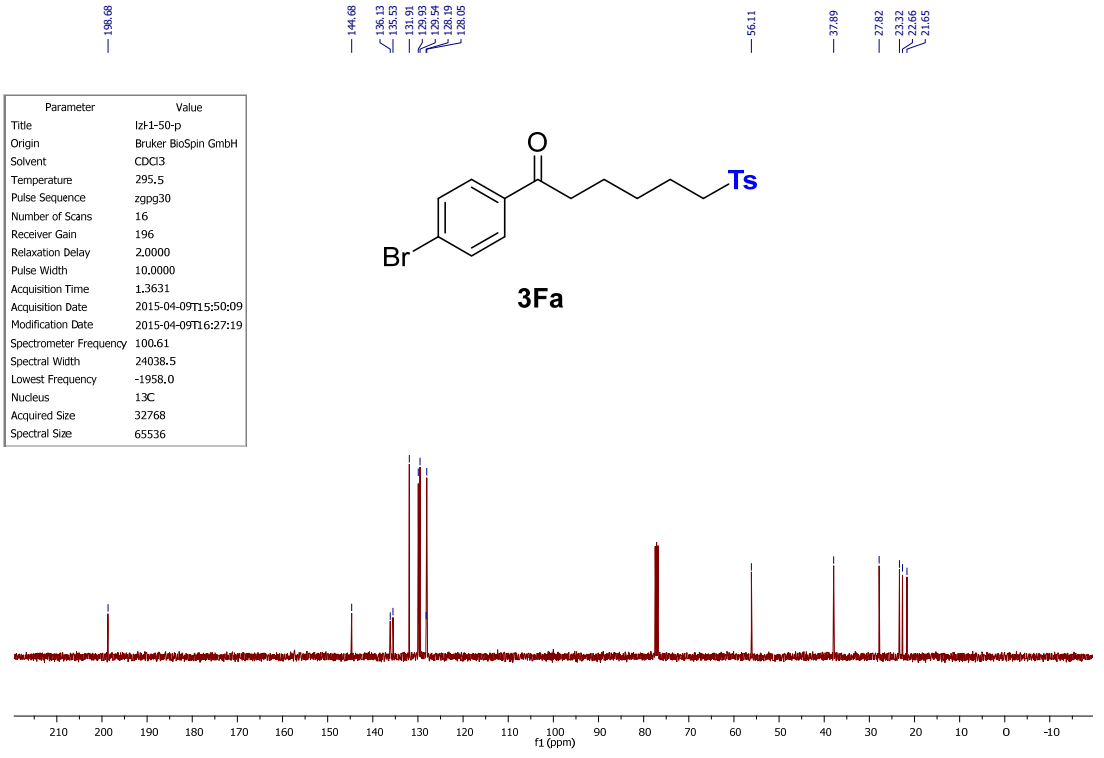
Parameter	Value
Title	lzl-1-31
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.9
Pulse Sequence	zgpg30
Number of Scans	29
Receiver Gain	196
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2015-03-30T16:05:24
Modification Date	2015-03-30T17:33:32
Spectrometer Frequency	100.61
Spectral Width	24038.5
Lowest Frequency	54.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

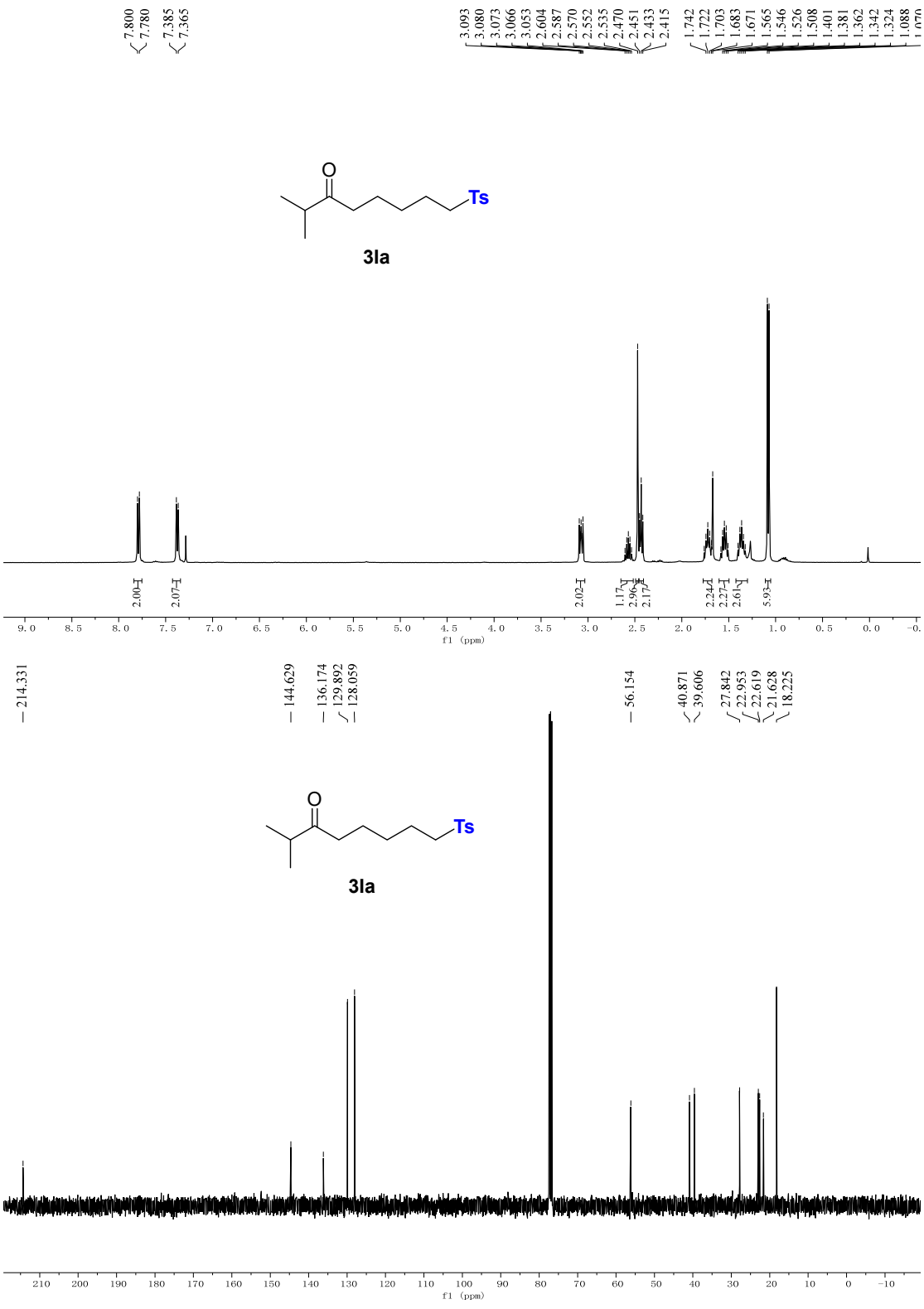


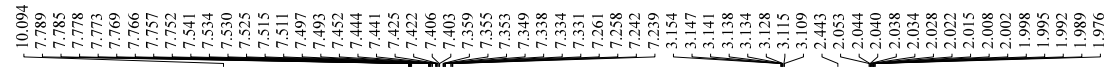
3Ea

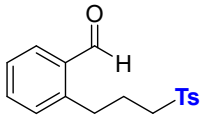



7.79
7.78
7.78
7.78
7.77
7.77
7.76
7.57
7.36
7.34
3.10
3.09
3.08
3.06
2.92
2.90
2.89
2.44
1.76
1.73
1.71
1.66
1.49
1.47
1.47
1.45
1.43
1.43
Parameter Value
Title lzl-1-50-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.5
Pulse Sequence zg30
Number of Scans 4
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-09T15:48:30
Modification Date 2015-04-09T16:27:18
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
Ts
Br
3Fa
4.03
1.98
1.97
2.00
2.05
3.12
4.25
2.10
f1 (ppm)

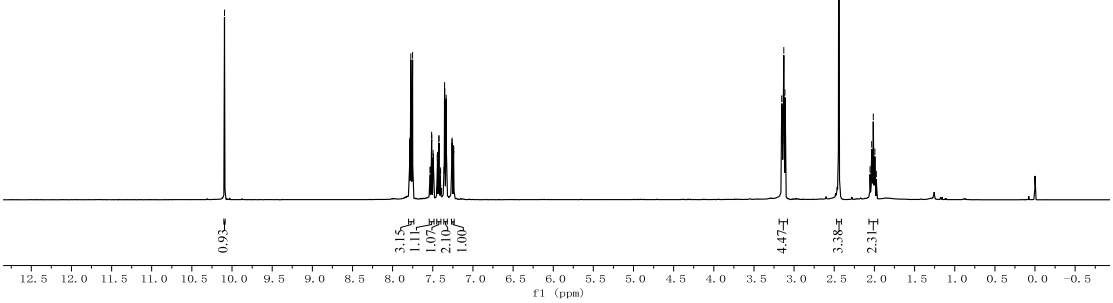

198.68
144.68
136.13
135.53
131.91
129.93
129.54
128.19
128.05
56.11
37.89
27.82
23.32
22.66
21.65
Parameter Value
Title lzl-1-50-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.5
Pulse Sequence zgpg30
Number of Scans 16
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-09T15:50:09
Modification Date 2015-04-09T16:27:19
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
Ts
Br
3Fa
f1 (ppm)


7.800
7.780
7.385
7.365
3.093
3.080
3.073
3.066
3.053
2.604
2.587
2.570
2.552
2.535
2.470
2.451
2.433
2.415
1.742
1.722
1.703
1.683
1.671
1.565
1.546
1.526
1.508
1.401
1.381
1.362
1.342
1.324
1.088
O
Ts
3la
2.00
2.07
2.02
1.17
2.96
2.17
2.24
2.27
2.61
5.93
f1 (ppm)
214.331
144.629
136.174
129.892
128.059
56.154
40.871
39.606
27.842
22.953
22.619
21.628
18.225
O
Ts
3la
f1 (ppm)

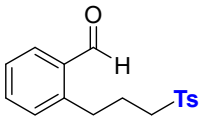

10.094
7.789
7.785
7.778
7.773
7.769
7.766
7.757
7.752
7.541
7.534
7.530
7.525
7.515
7.511
7.497
7.493
7.452
7.444
7.441
7.425
7.422
7.406
7.403
7.359
7.355
7.353
7.349
7.338
7.334
7.331
7.261
7.258
7.242
7.239
3.154
3.147
3.141
3.138
3.134
3.128
3.115
3.109
2.443
2.053
2.044
2.040
2.038
2.034
2.028
2.022
2.015
2.008
2.002
1.998
1.995
1.992
1.989
1.976


O
H
Ts

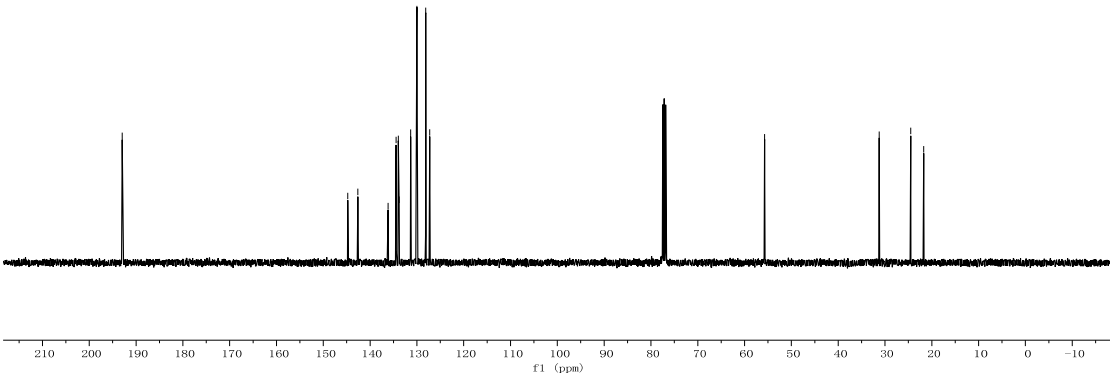
3Ja

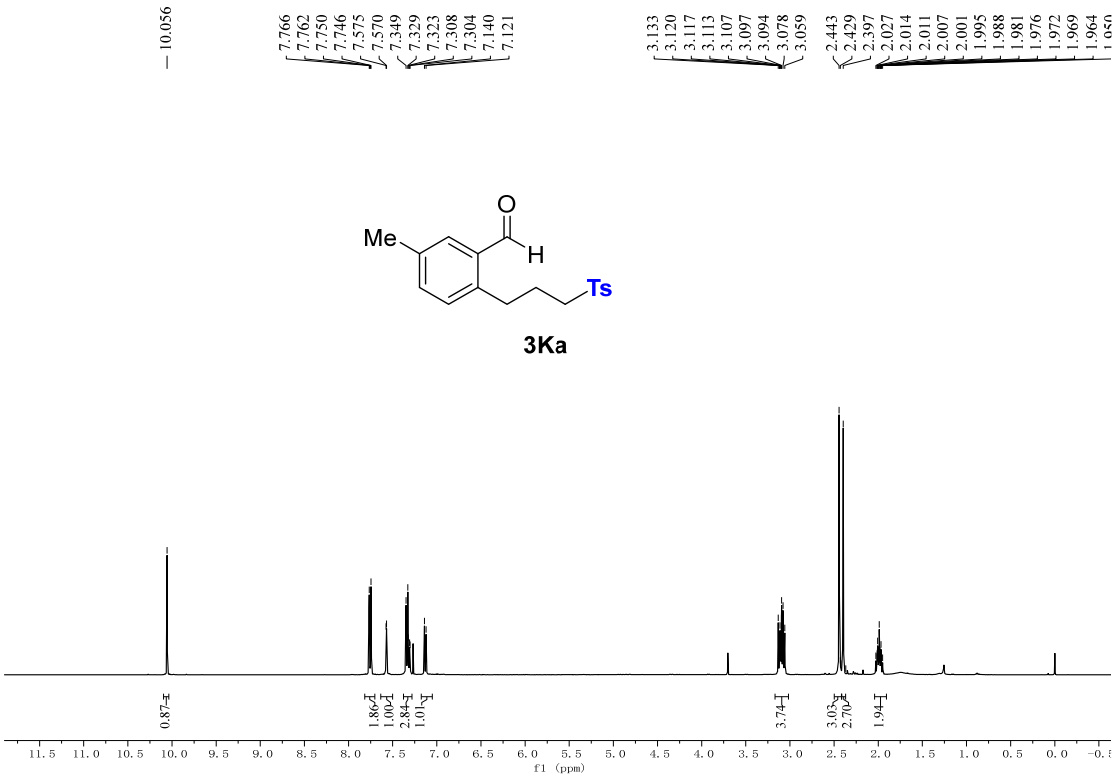

0.93
3.15
1.11
1.07
2.10
1.00
4.47
3.38
2.31
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


192.962
144.771
142.616
136.173
134.434
133.963
133.804
131.326
130.007
128.129
127.268
55.732
31.234
24.507
21.731

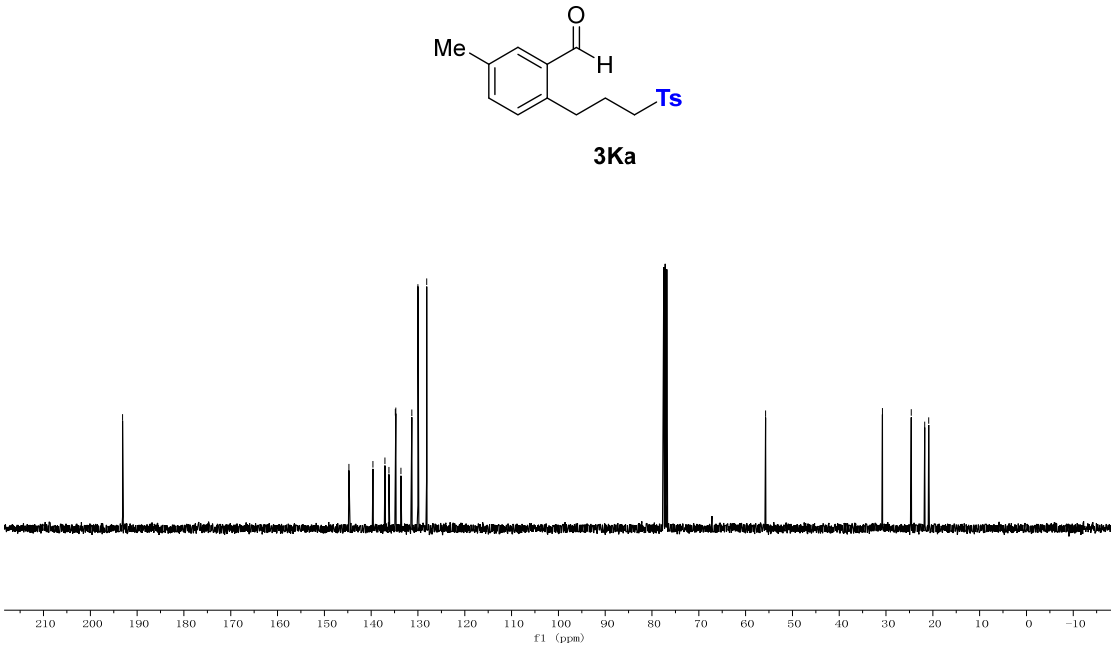

O
H
Ts

3Ja

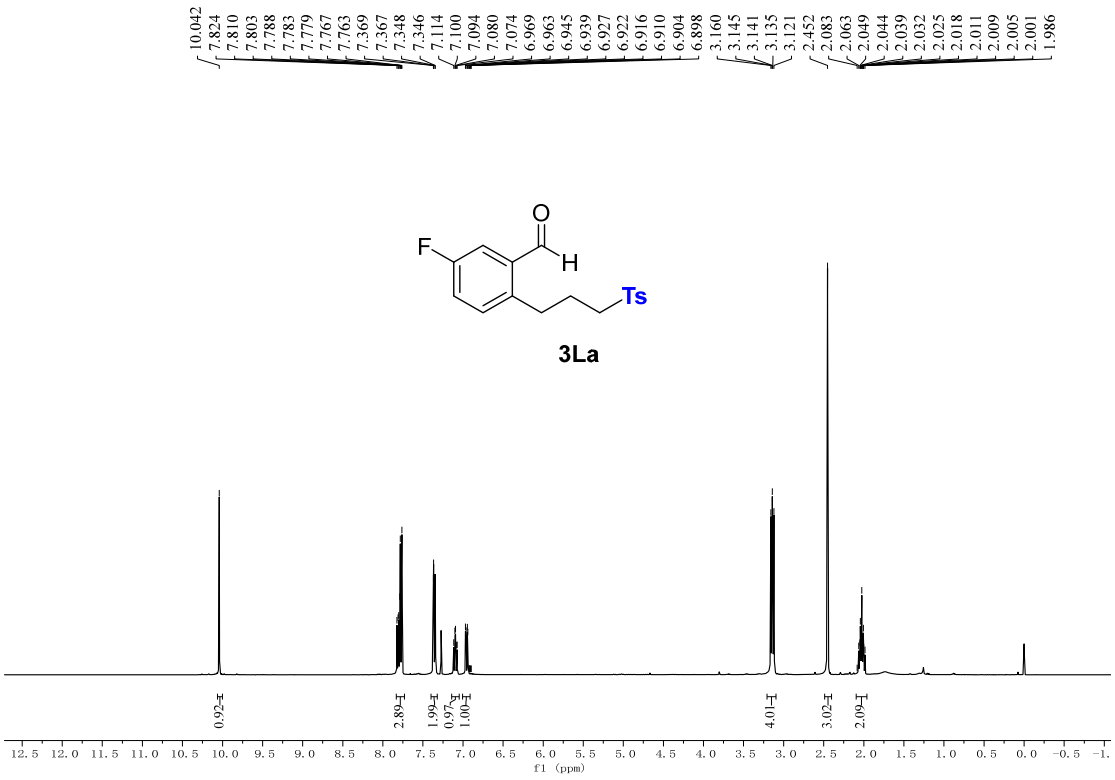

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

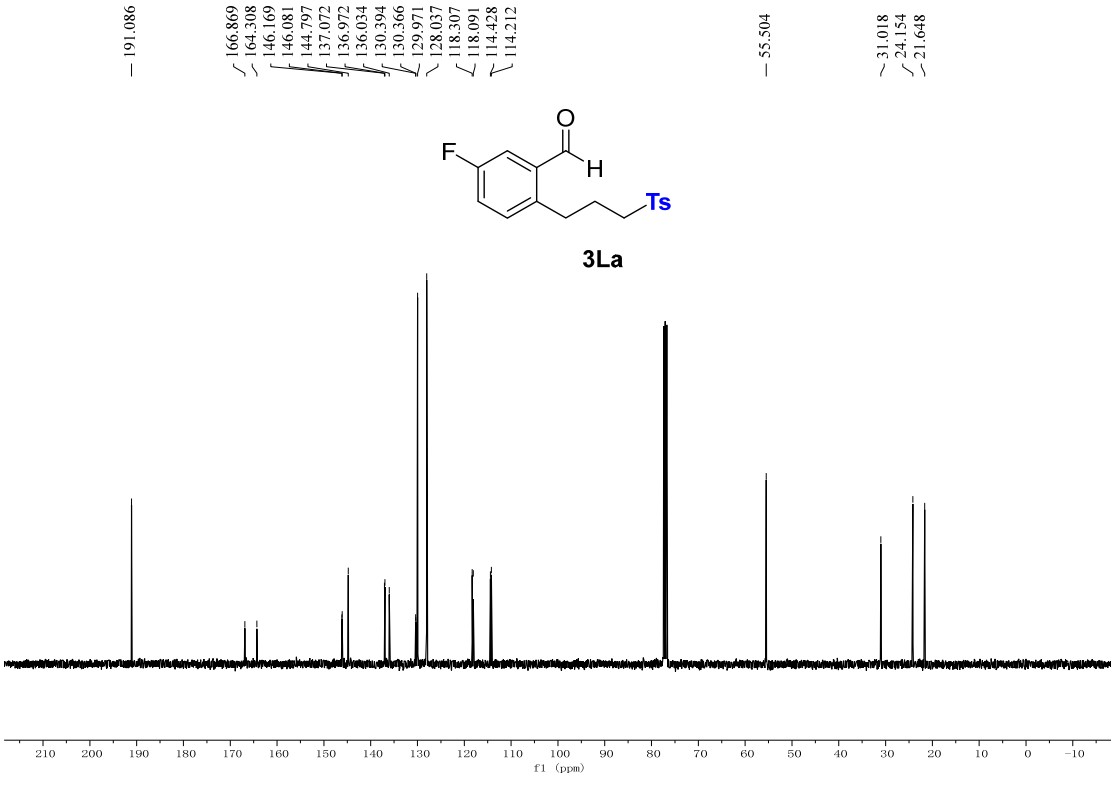

10.056
7.766
7.762
7.750
7.746
7.575
7.570
7.349
7.329
7.323
7.308
7.304
7.140
7.121
3.133
3.120
3.117
3.113
3.107
3.097
3.094
3.078
3.059
2.443
2.429
2.397
2.027
2.014
2.011
2.007
2.001
1.995
1.988
1.981
1.976
1.972
1.969
1.964
O
Me
H
Ts
0.87
1.86
1.00
2.84
1.01
3.74
3.03
2.70
1.94
f1 (ppm)

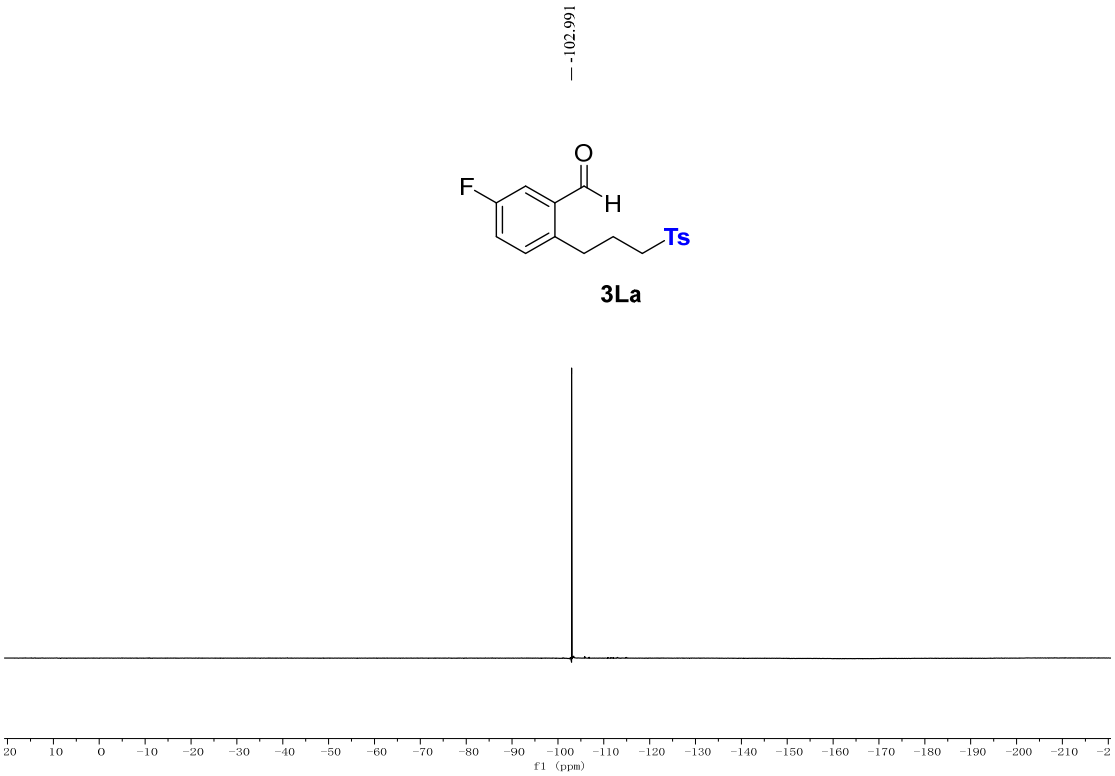
3Ka


193.094
144.747
139.622
137.059
136.200
134.835
134.733
133.646
131.306
129.998
128.149
55.737
30.783
24.611
21.743
20.868
O
Me
H
Ts
f1 (ppm)

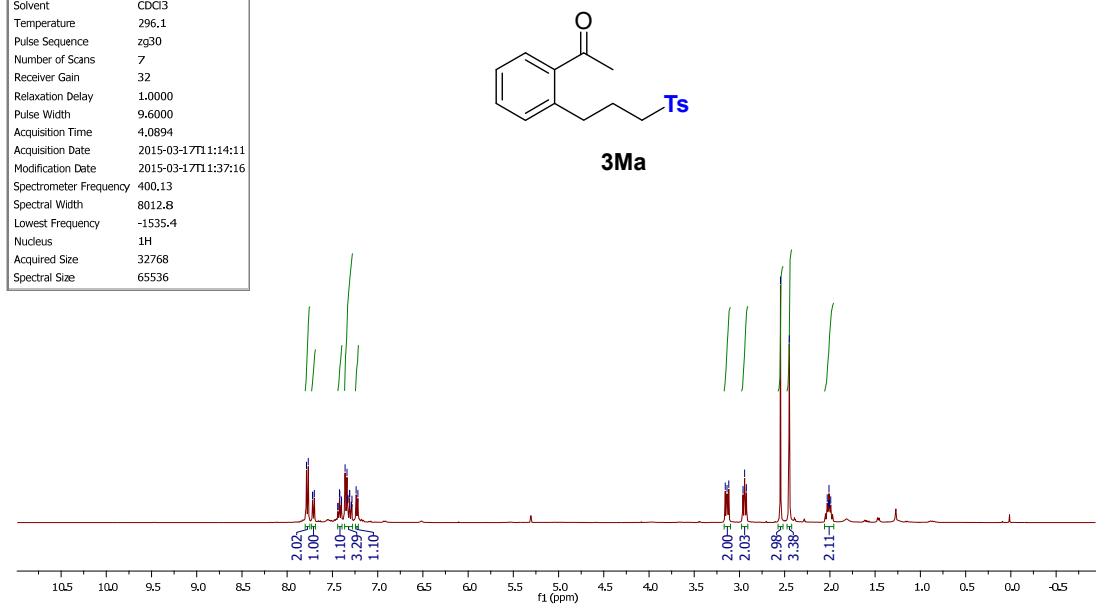
3Ka

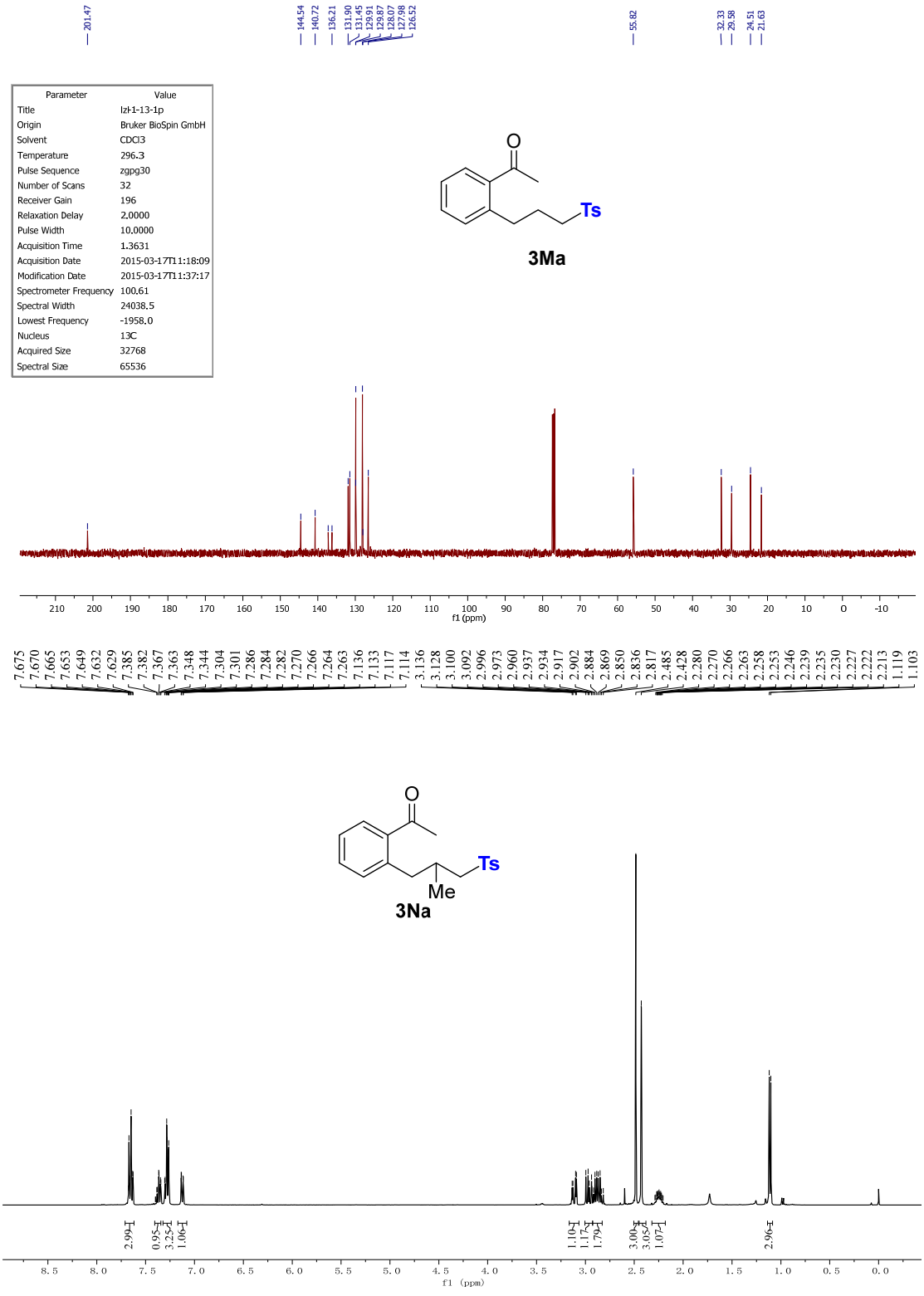

10.042
7.824
7.810
7.803
7.788
7.783
7.779
7.767
7.763
7.369
7.367
7.348
7.346
7.114
7.100
7.094
7.080
7.074
6.969
6.963
6.945
6.939
6.927
6.922
6.916
6.910
6.904
6.898
3.160
3.145
3.141
3.135
3.121
2.452
2.083
2.063
2.049
2.044
2.039
2.032
2.025
2.018
2.011
2.009
2.005
2.001
1.986
O
F
H
Ts
3La
0.92
2.89
1.99
0.97
1.00
4.01
3.02
2.09
f1 (ppm)

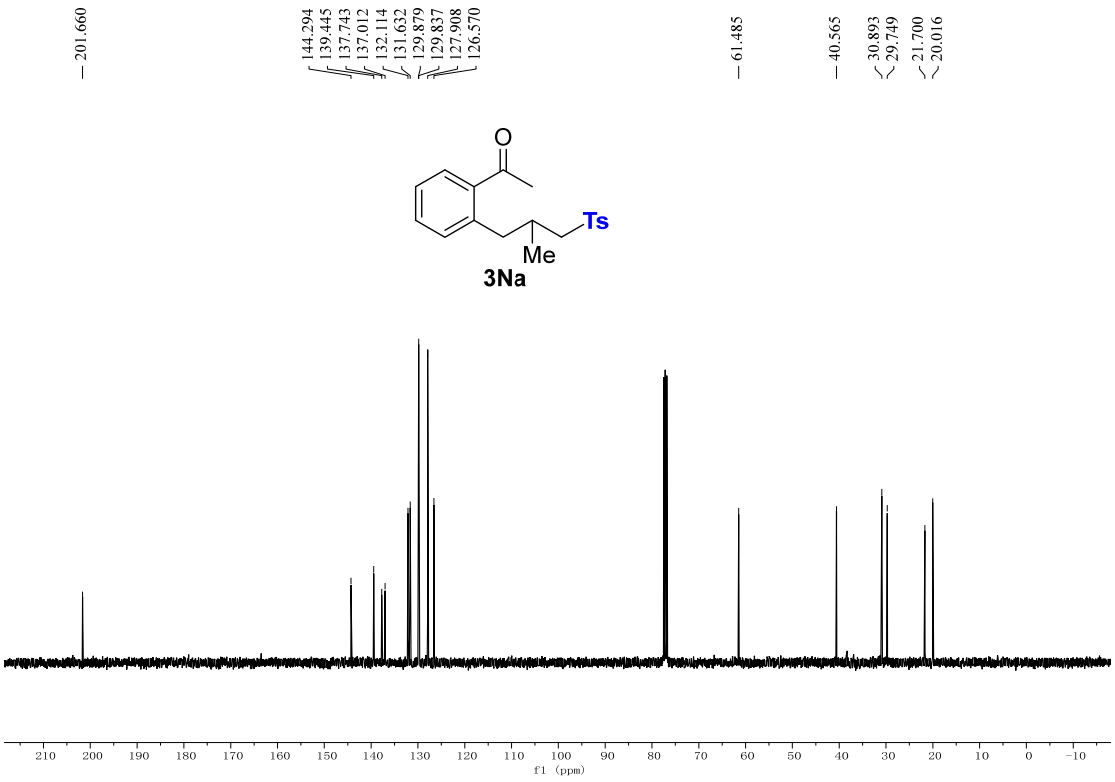

191.086
166.869
164.308
146.169
146.081
144.797
137.072
136.972
136.034
130.394
130.366
129.971
128.037
118.307
118.091
114.428
114.212
55.504
31.018
24.154
21.648
O
F
H
Ts
3La
f1 (ppm)

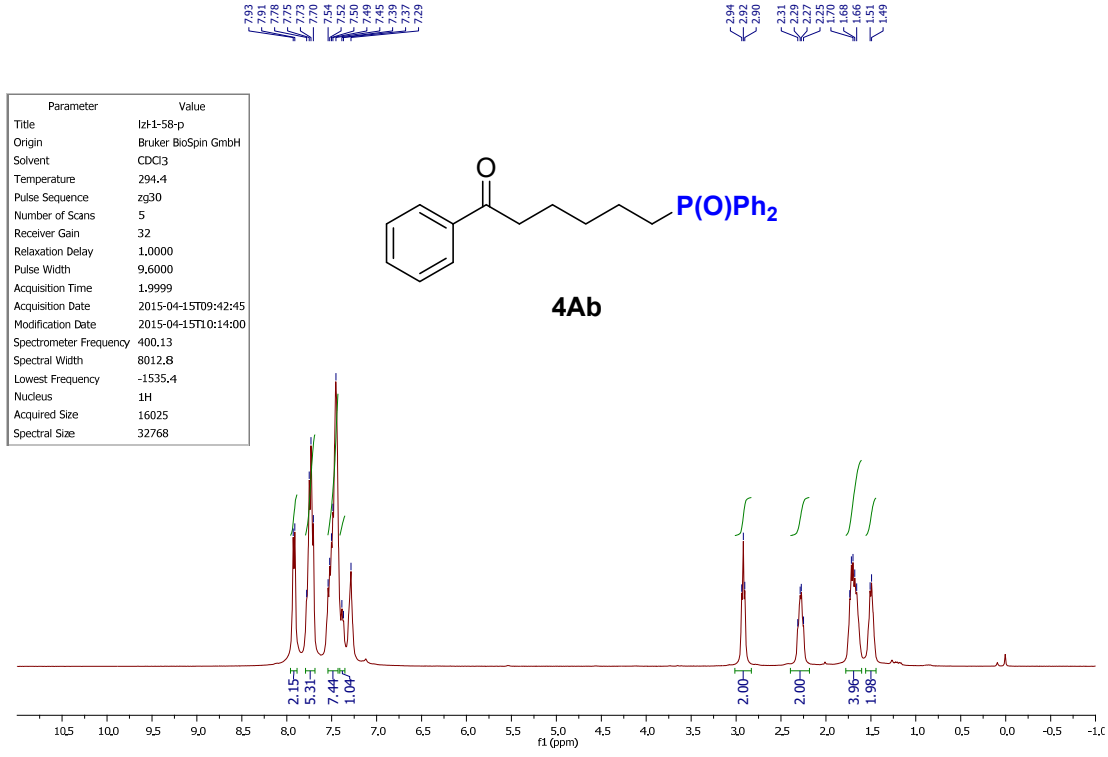


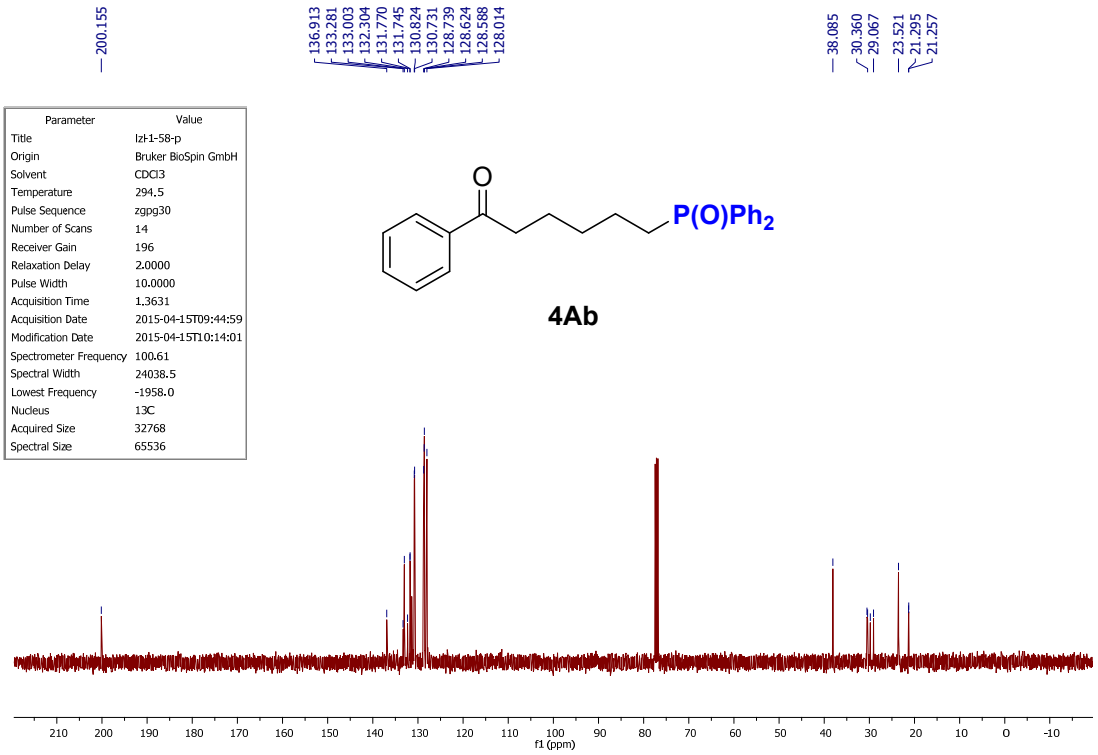
Parameter	Value
Title	lzl-1-13-1p
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	296.1
Pulse Sequence	zg30
Number of Scans	7
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	4.0894
Acquisition Date	2015-03-17T11:14:11
Modification Date	2015-03-17T11:37:16
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536

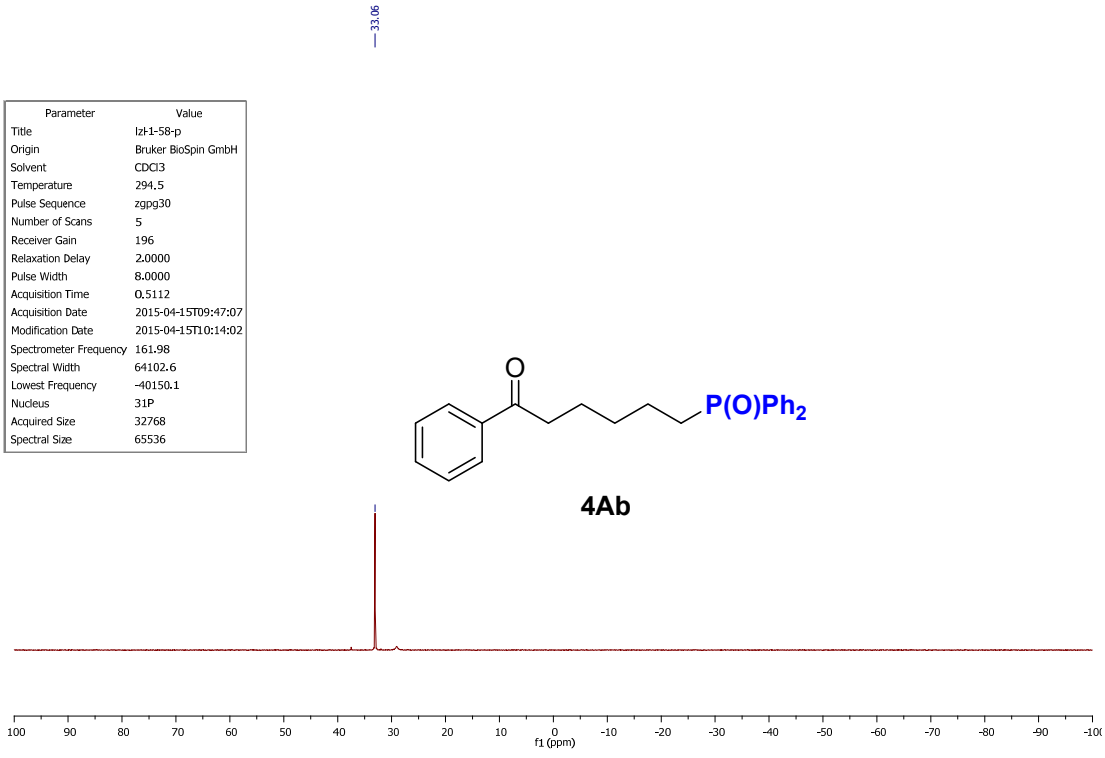


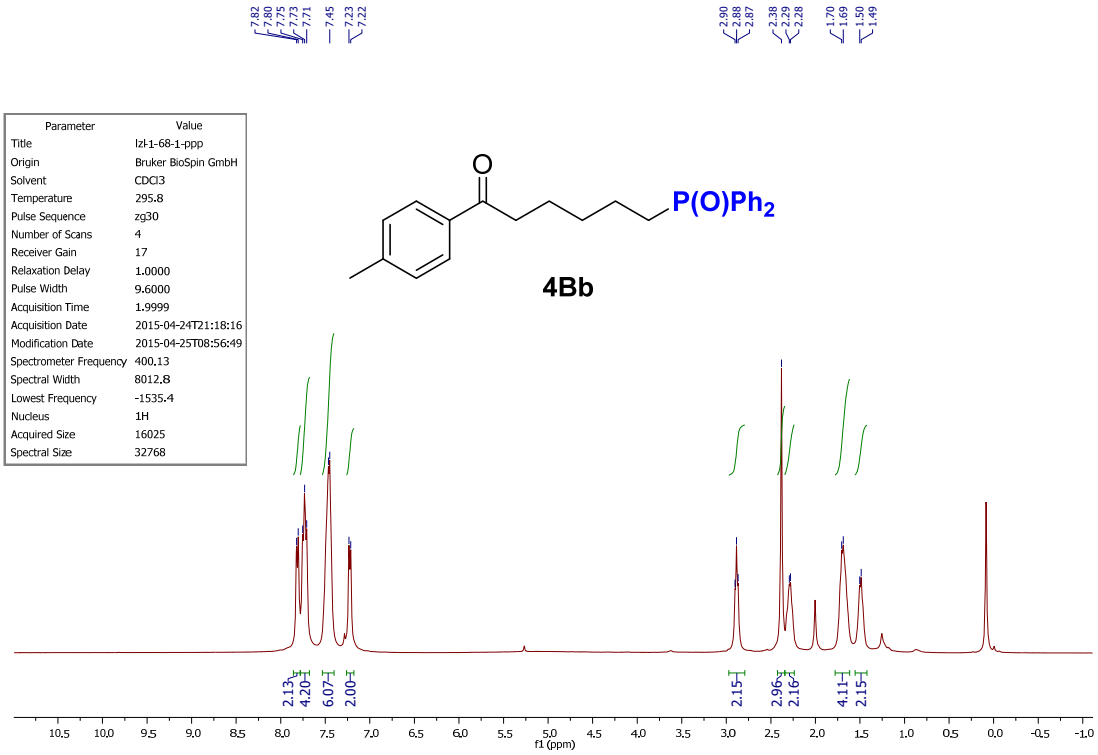

201.47
144.54
140.72
136.21
131.90
131.45
129.91
129.87
128.07
127.98
126.52
55.82
32.33
29.58
24.51
21.63
Parameter Value
Title lzl-1-13-1p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 296.3
Pulse Sequence zgpg30
Number of Scans 32
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-03-17T11:18:09
Modification Date 2015-03-17T11:37:17
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
Ts
3Ma
f1 (ppm)
7.675
7.670
7.665
7.653
7.649
7.632
7.629
7.385
7.382
7.367
7.363
7.348
7.344
7.304
7.301
7.286
7.284
7.282
7.270
7.266
7.264
7.263
7.136
7.133
7.117
7.114
3.136
3.128
3.100
3.092
2.996
2.973
2.960
2.937
2.934
2.917
2.902
2.884
2.869
2.850
2.836
2.817
2.485
2.428
2.280
2.270
2.266
2.263
2.258
2.253
2.246
2.239
2.235
2.230
2.227
2.222
2.213
1.119
1.103
O
Ts
Me
3Na
2.99
0.95
3.25
1.06
1.10
1.17
1.79
3.00
3.05
1.07
2.96
f1 (ppm)

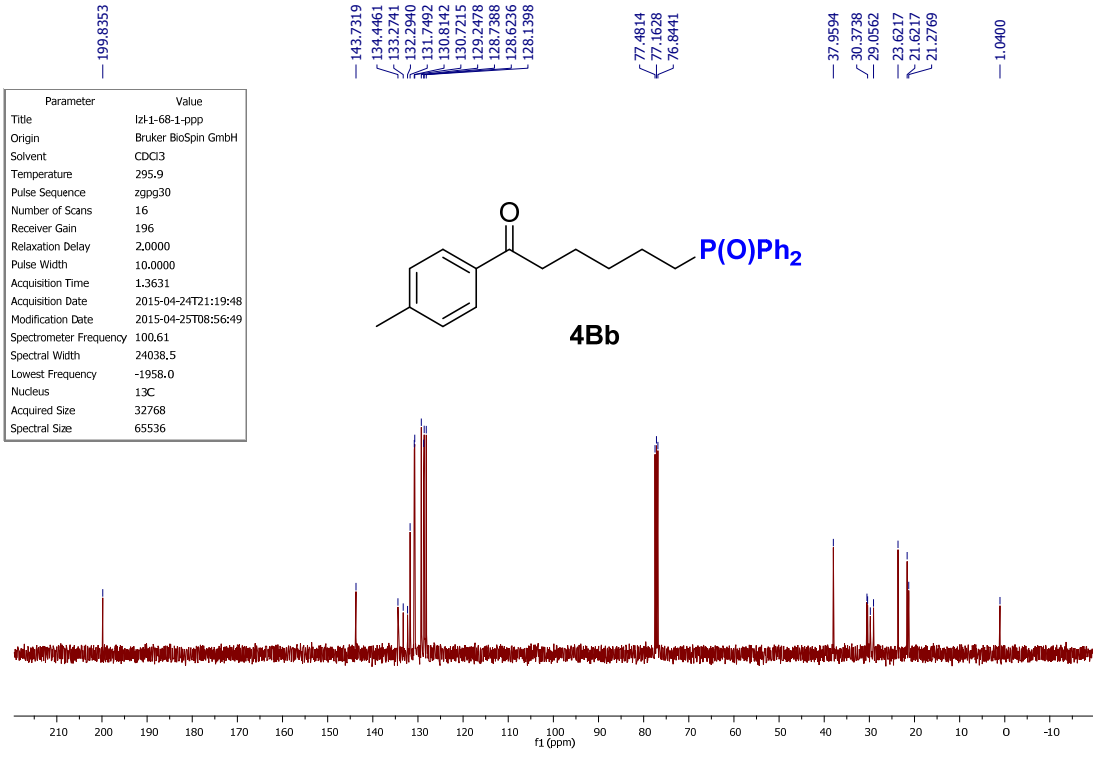

201.660
144.294
139.445
137.743
137.012
132.114
131.632
129.879
129.837
127.908
126.570
61.485
40.565
30.893
29.749
21.700
20.016
O
Ts
Me
3Na
f1 (ppm)

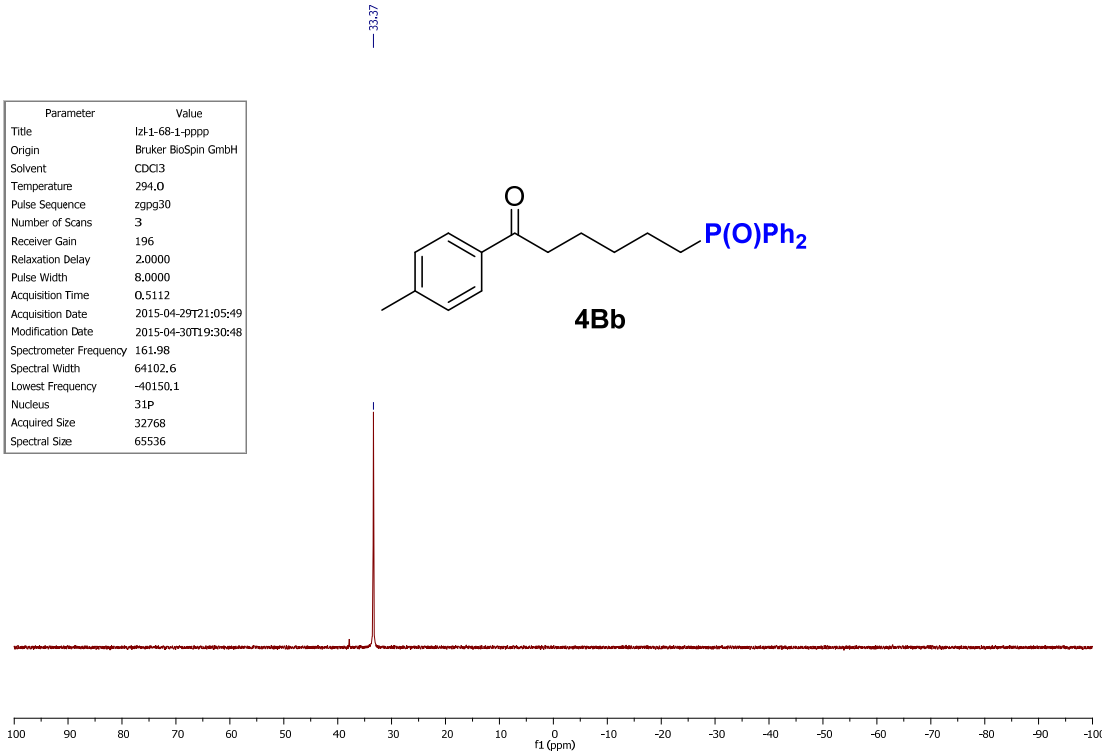

7.93
7.91
7.78
7.75
7.73
7.70
7.54
7.52
7.50
7.49
7.45
7.39
7.37
7.29
2.94
2.92
2.90
2.31
2.29
2.27
2.25
1.70
1.68
1.66
1.51
1.49
Parameter Value
Title lzl-1-58-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.4
Pulse Sequence zg30
Number of Scans 5
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-15T09:42:45
Modification Date 2015-04-15T10:14:00
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
P(O)Ph2
4Ab
2.15
5.31
7.44
1.04
2.00
2.00
3.96
1.98
f1 (ppm)

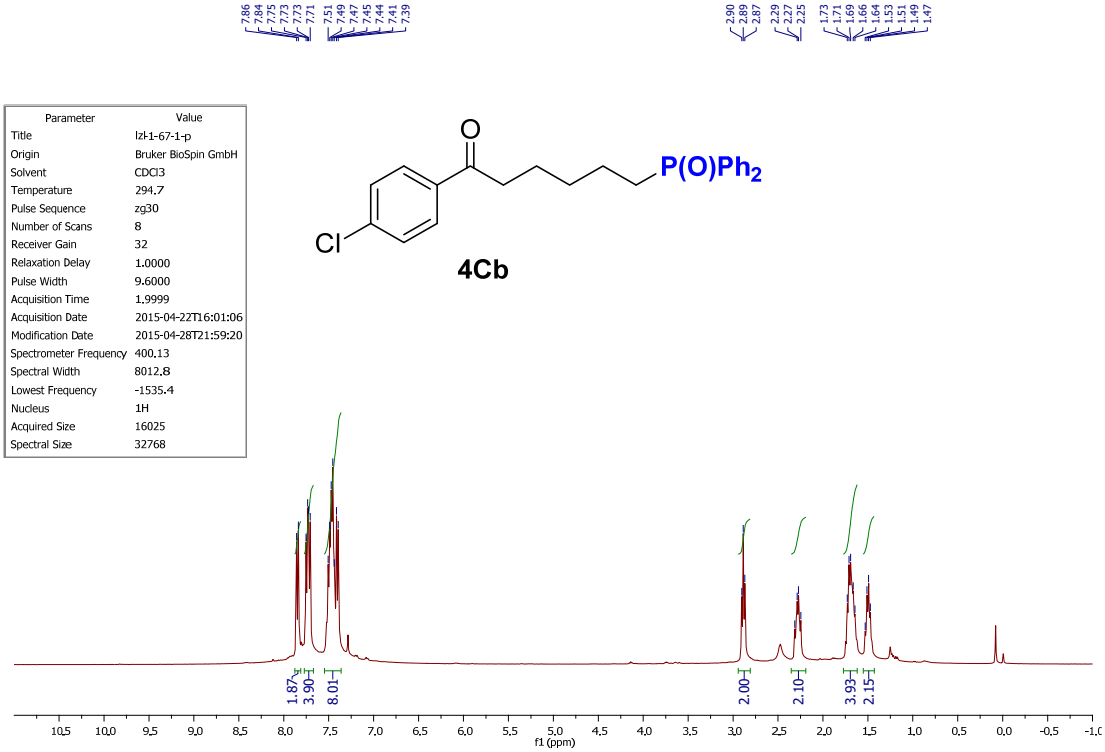

200.155
136.913
133.281
133.003
132.304
131.770
131.745
130.824
130.731
128.739
128.624
128.588
128.014
38.085
30.360
29.067
23.521
21.295
21.257
Parameter Value
Title lzl-1-58-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.5
Pulse Sequence zgpg30
Number of Scans 14
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-15T09:44:59
Modification Date 2015-04-15T10:14:01
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
4Ab
f1 (ppm)

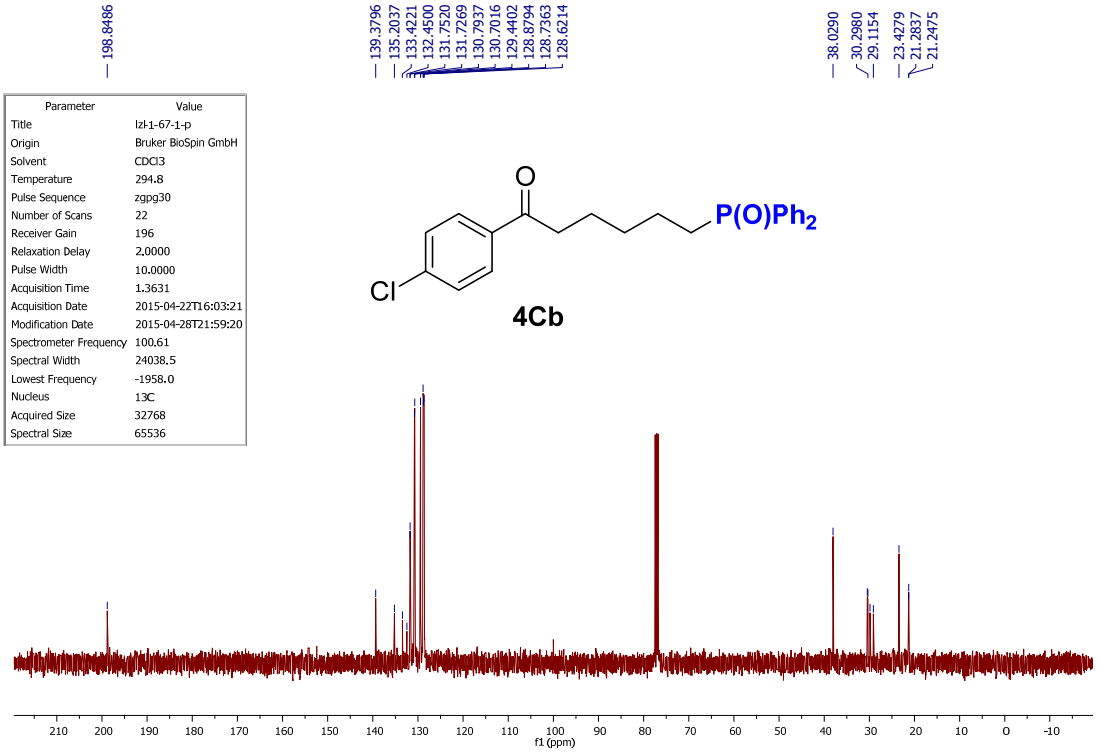

33.06
Parameter Value
Title lzl-1-58-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.5
Pulse Sequence zgpg30
Number of Scans 5
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 8.0000
Acquisition Time 0.5112
Acquisition Date 2015-04-15T09:47:07
Modification Date 2015-04-15T10:14:02
Spectrometer Frequency 161.98
Spectral Width 64102.6
Lowest Frequency -40150.1
Nucleus 31P
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
4Ab
f1 (ppm)

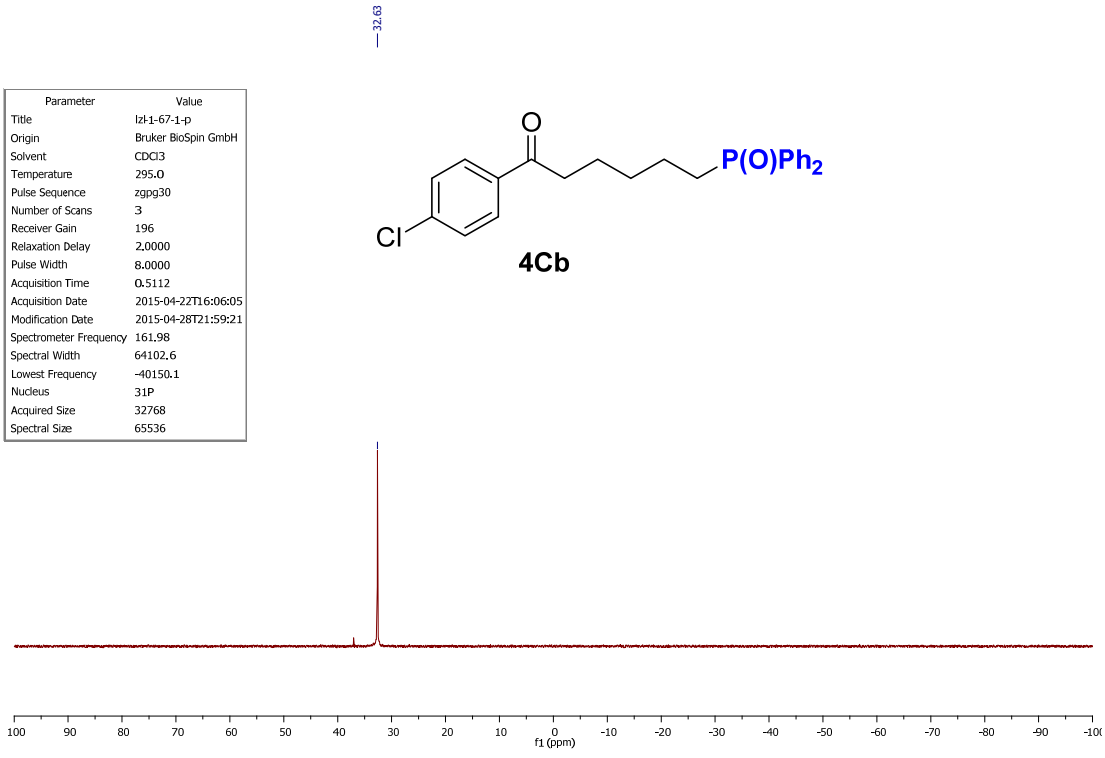

7.82
7.80
7.75
7.73
7.71
7.45
7.23
7.22
2.90
2.88
2.87
2.38
2.29
2.28
1.70
1.69
1.50
1.49
Parameter Value
Title lzl-1-68-1-ppp
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.8
Pulse Sequence zg30
Number of Scans 4
Receiver Gain 17
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-24T21:18:16
Modification Date 2015-04-25T08:56:49
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
P(O)Ph2
4Bb
2.13
4.20
6.07
2.00
2.15
2.96
2.16
4.11
2.15
f1 (ppm)


199.8353
143.7319
134.4461
133.2741
132.2940
131.7492
130.8142
130.7215
129.2478
128.7388
128.6236
128.1398
77.4814
77.1628
76.8441
37.9594
30.3738
29.0562
23.6217
21.6217
21.2769
1.0400
Parameter Value
Title lzl-1-68-1-ppp
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.9
Pulse Sequence zgpg30
Number of Scans 16
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-24T21:19:48
Modification Date 2015-04-25T08:56:49
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
4Bb
f1 (ppm)

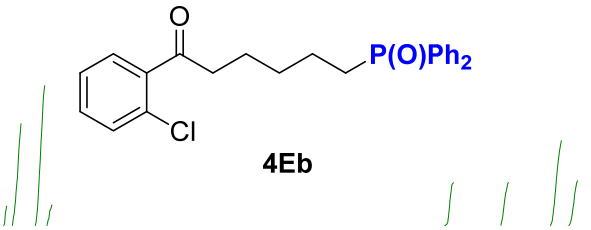

33.37
Parameter Value
Title lzl-1-68-1-pppp
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.0
Pulse Sequence zgpg30
Number of Scans 3
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 8.0000
Acquisition Time 0.5112
Acquisition Date 2015-04-29T21:05:49
Modification Date 2015-04-30T19:30:48
Spectrometer Frequency 161.98
Spectral Width 64102.6
Lowest Frequency -40150.1
Nucleus 31P
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
4Bb
f1 (ppm)


7.86
7.84
7.75
7.73
7.71
7.51
7.49
7.47
7.45
7.44
7.41
7.39
2.90
2.89
2.87
2.29
2.27
2.25
1.73
1.71
1.69
1.66
1.64
1.53
1.51
1.49
1.47
Parameter Value
Title lzl-1-67-1-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.7
Pulse Sequence zg30
Number of Scans 8
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-22T16:01:06
Modification Date 2015-04-28T21:59:20
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
P(O)Ph2
Cl
4Cb
1.87
3.90
8.01
2.00
2.10
3.93
2.15
f1 (ppm)

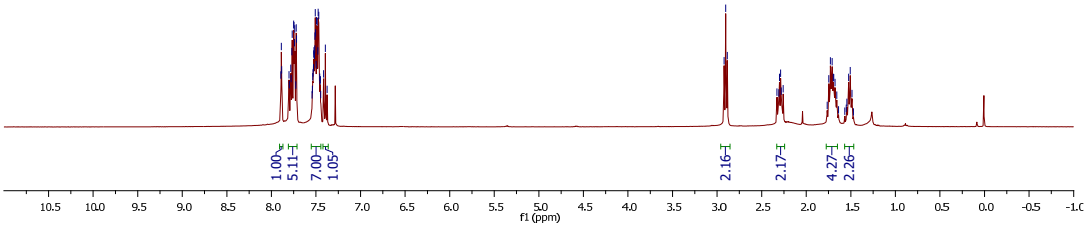

198.8486
139.3796
135.2037
133.4221
132.4500
131.7520
131.7269
130.7937
130.7016
129.4402
128.8794
128.7363
128.6214
38.0290
30.2980
29.1154
23.4279
21.2837
21.2475
Parameter Value
Title lzl-1-67-1-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.8
Pulse Sequence zgpg30
Number of Scans 22
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-22T16:03:21
Modification Date 2015-04-28T21:59:20
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
Cl
4Cb
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


32.63
Parameter Value
Title lzl-1-67-1-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Pulse Sequence zgpg30
Number of Scans 3
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 8.0000
Acquisition Time 0.5112
Acquisition Date 2015-04-22T16:06:05
Modification Date 2015-04-28T21:59:21
Spectrometer Frequency 161.98
Spectral Width 64102.6
Lowest Frequency -40150.1
Nucleus 31P
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
Cl
4Cb
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)

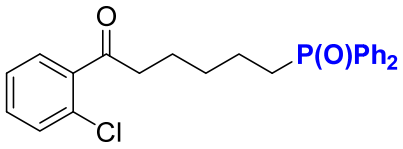
Parameter	Value
Title	lzl-3Gc2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.1
Pulse Sequence	zg30
Number of Scans	6
Receiver Gain	55
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	1.9999
Acquisition Date	2015-08-06T21:41:43
Modification Date	2015-08-18T21:57:25
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
Acquired Size	16025
Spectral Size	32768



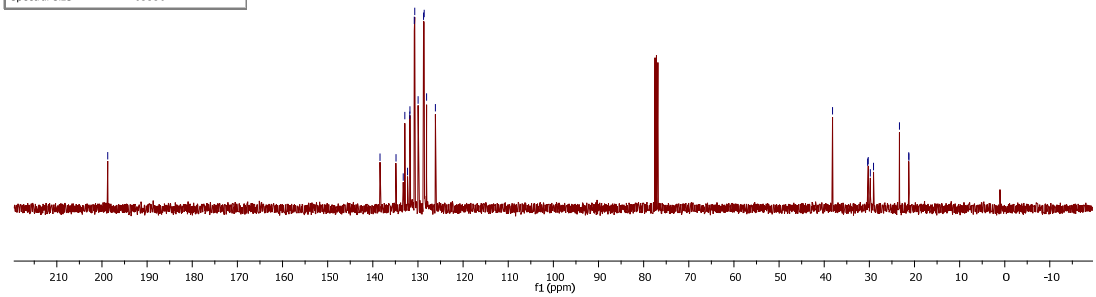
4Eb



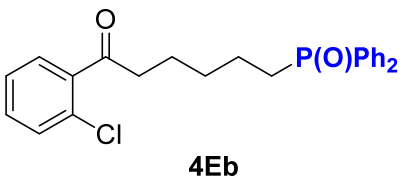
Parameter	Value
Title	lzl-1-67-2-agoac
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.0
Pulse Sequence	zgpg30
Number of Scans	14
Receiver Gain	196
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2015-04-24T10:50:41
Modification Date	2015-04-28T22:22:58
Spectrometer Frequency	100.61
Spectral Width	24038.5
Lowest Frequency	-1958.0
Nucleus	13C
Acquired Size	32768
Spectral Size	65536



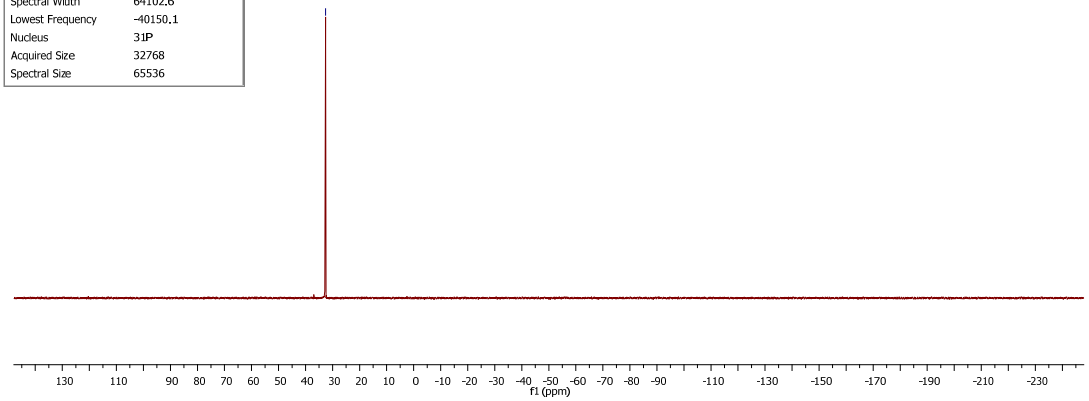
4Eb



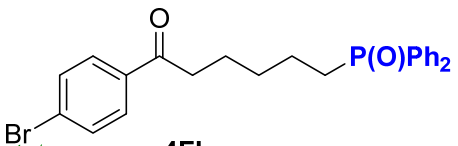
Parameter	Value
Title	lzl-3Gc2
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.2
Pulse Sequence	zgpg30
Number of Scans	4
Receiver Gain	196
Relaxation Delay	2.0000
Pulse Width	8.0000
Acquisition Time	0.5112
Acquisition Date	2015-08-06T21:43:28
Modification Date	2015-08-18T21:57:26
Spectrometer Frequency	161.98
Spectral Width	64102.6
Lowest Frequency	-40150.1
Nucleus	31P
Acquired Size	32768
Spectral Size	65536



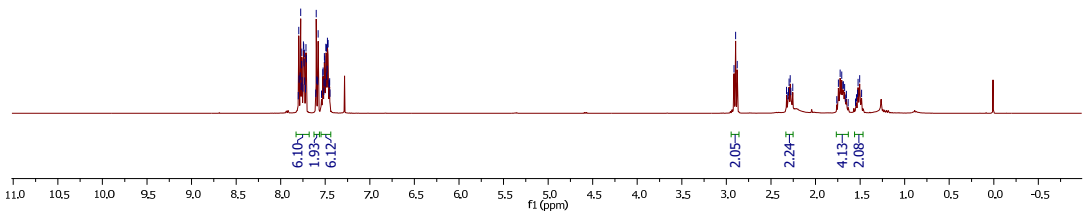
4Eb

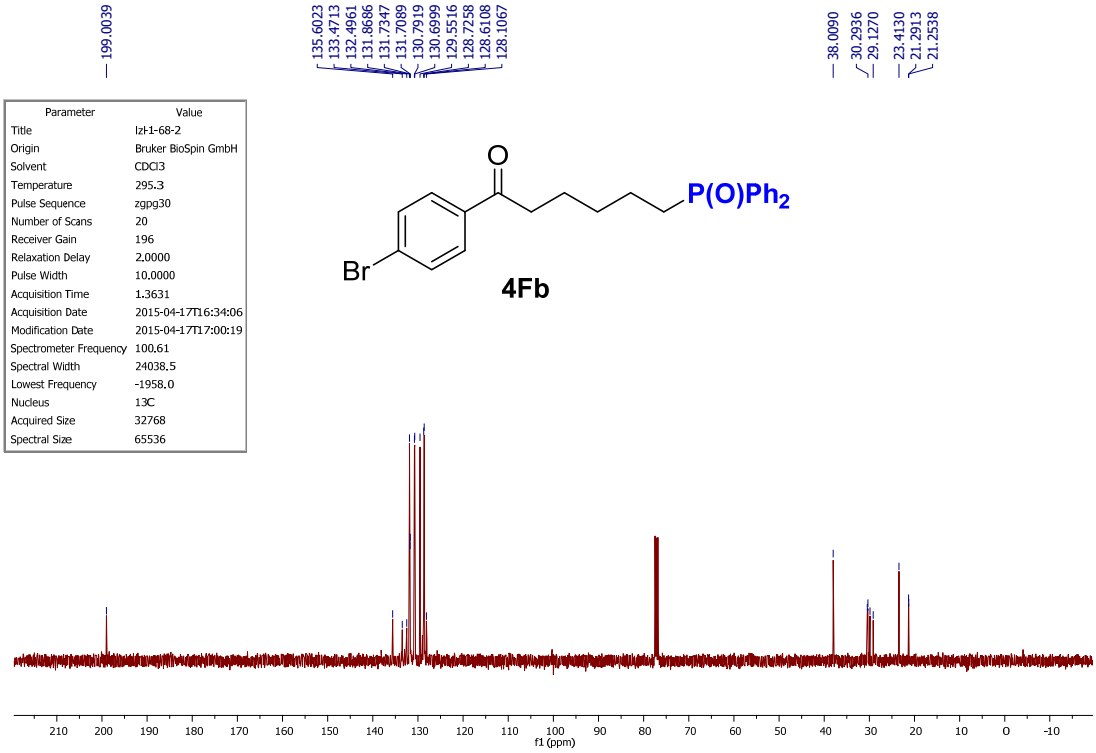


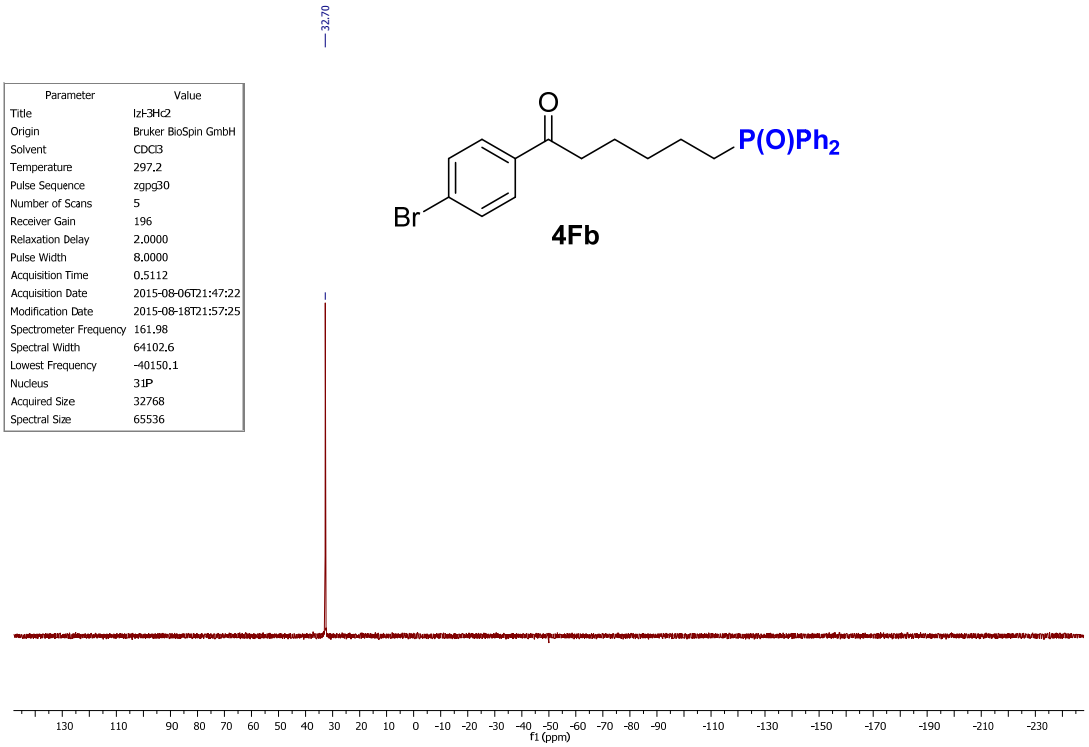
Parameter	Value
Title	lzl-3Hc-1
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.6
Pulse Sequence	zg30
Number of Scans	4
Receiver Gain	62
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	1.9999
Acquisition Date	2015-08-06T19:38:33
Modification Date	2015-08-06T19:47:22
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
Acquired Size	16025
Spectral Size	32768

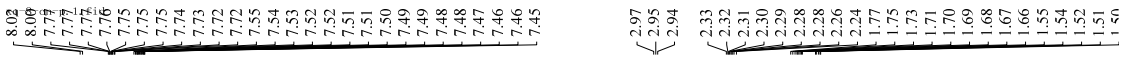


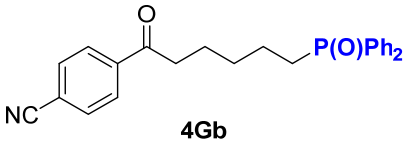
4Fb



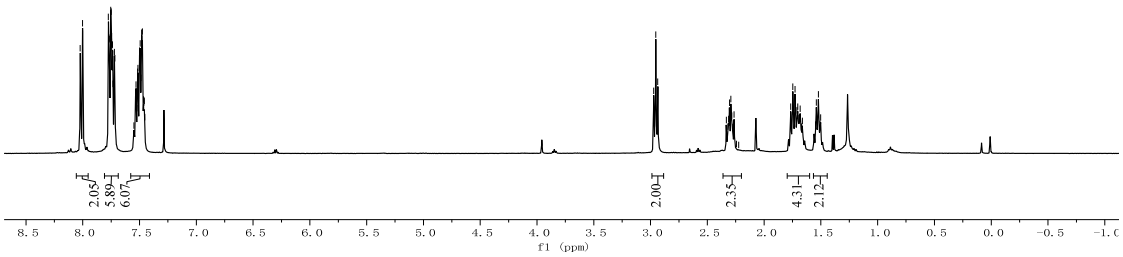

199.0039
135.6023
133.4713
132.4961
131.8686
131.7347
131.7089
130.7919
130.6999
129.5516
128.7258
128.6108
128.1067
38.0090
30.2936
29.1270
23.4130
21.2913
21.2538
Parameter Value
Title lzl-1-68-2
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.3
Pulse Sequence zgpg30
Number of Scans 20
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-17T16:34:06
Modification Date 2015-04-17T17:00:19
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
Br
4Fb
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

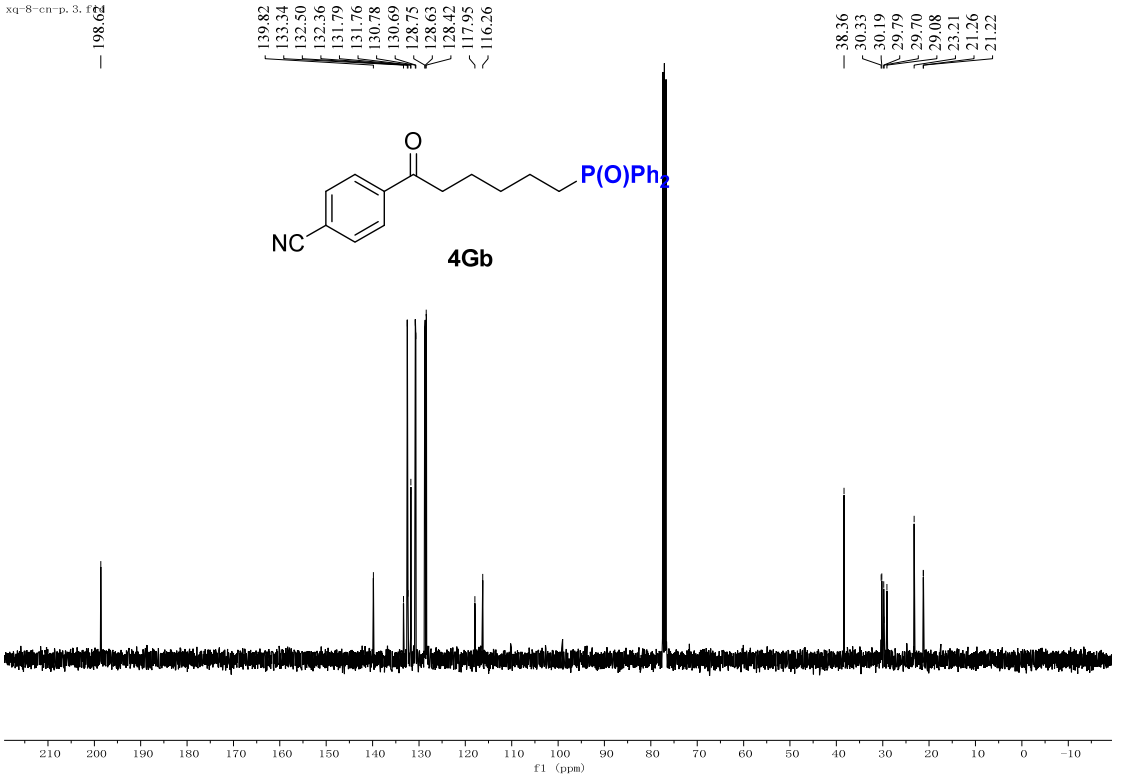

32.70
Parameter Value
Title lzl-3Hc2
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 297.2
Pulse Sequence zgpg30
Number of Scans 5
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 8.0000
Acquisition Time 0.5112
Acquisition Date 2015-08-06T21:47:22
Modification Date 2015-08-18T21:57:25
Spectrometer Frequency 161.98
Spectral Width 64102.6
Lowest Frequency -40150.1
Nucleus 31P
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
Br
4Fb
130 110 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)



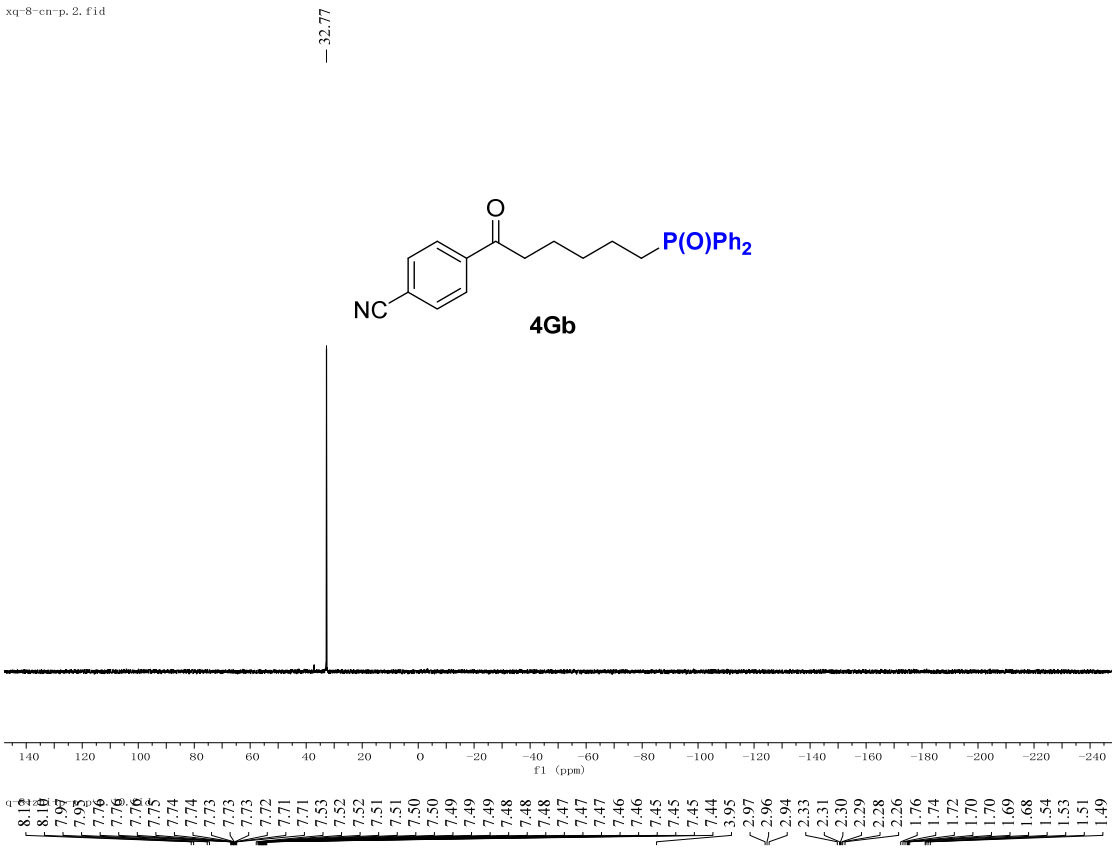

O
P(O)Ph2
NC

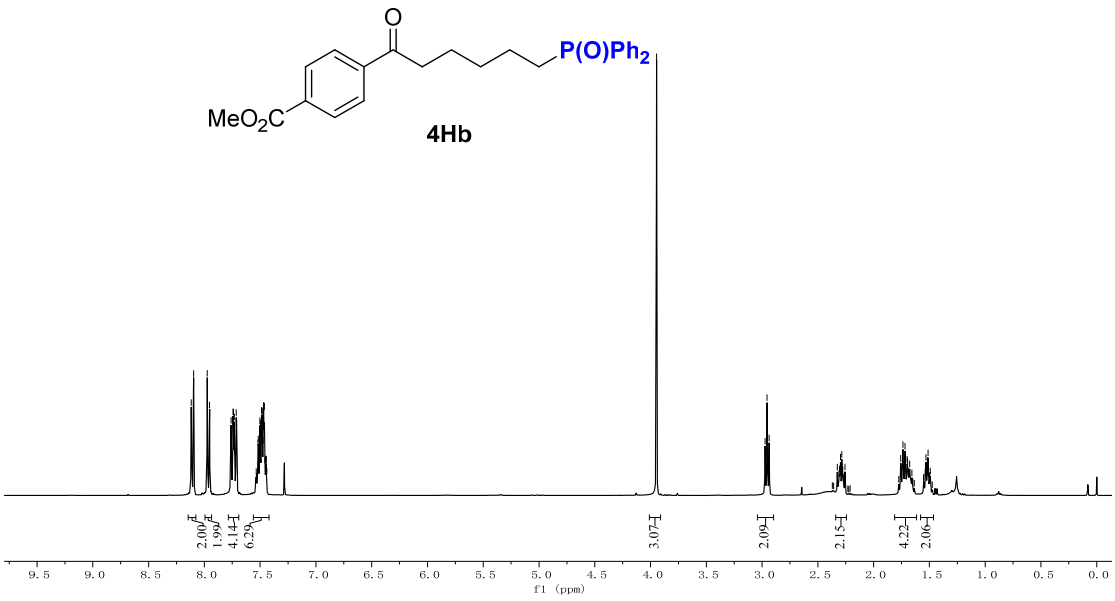
4Gb

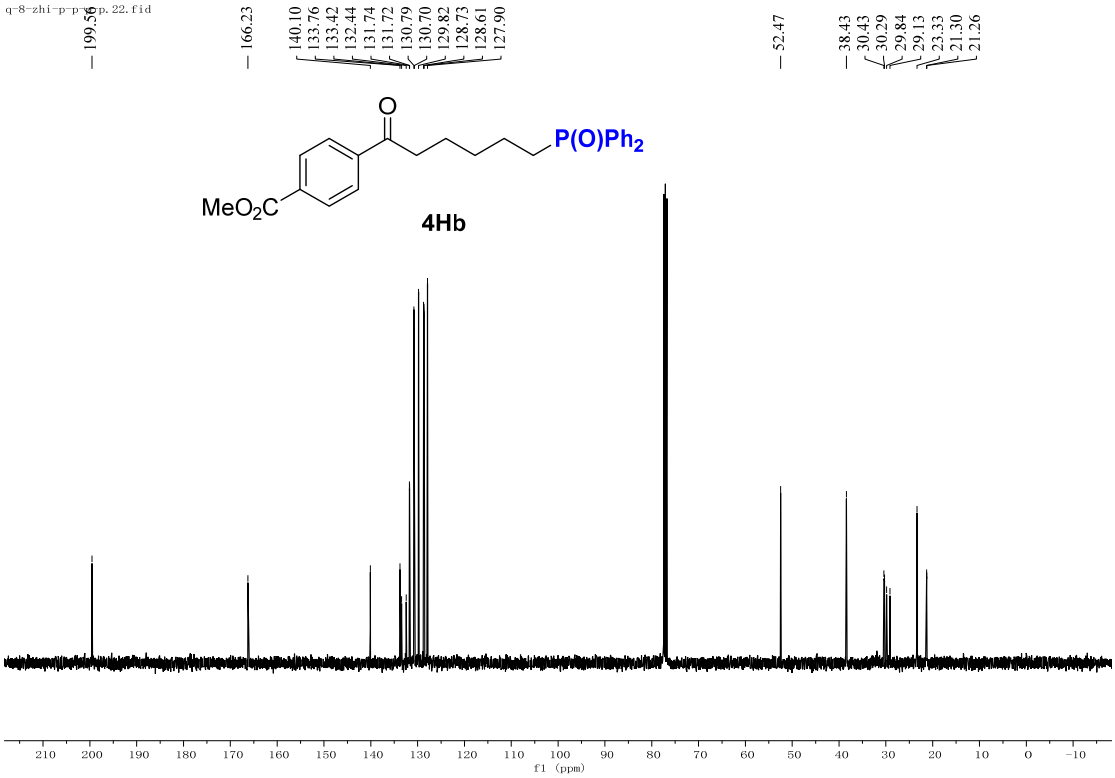

2.05
5.89
6.07
2.00
2.35
4.31
2.12
f1 (ppm)

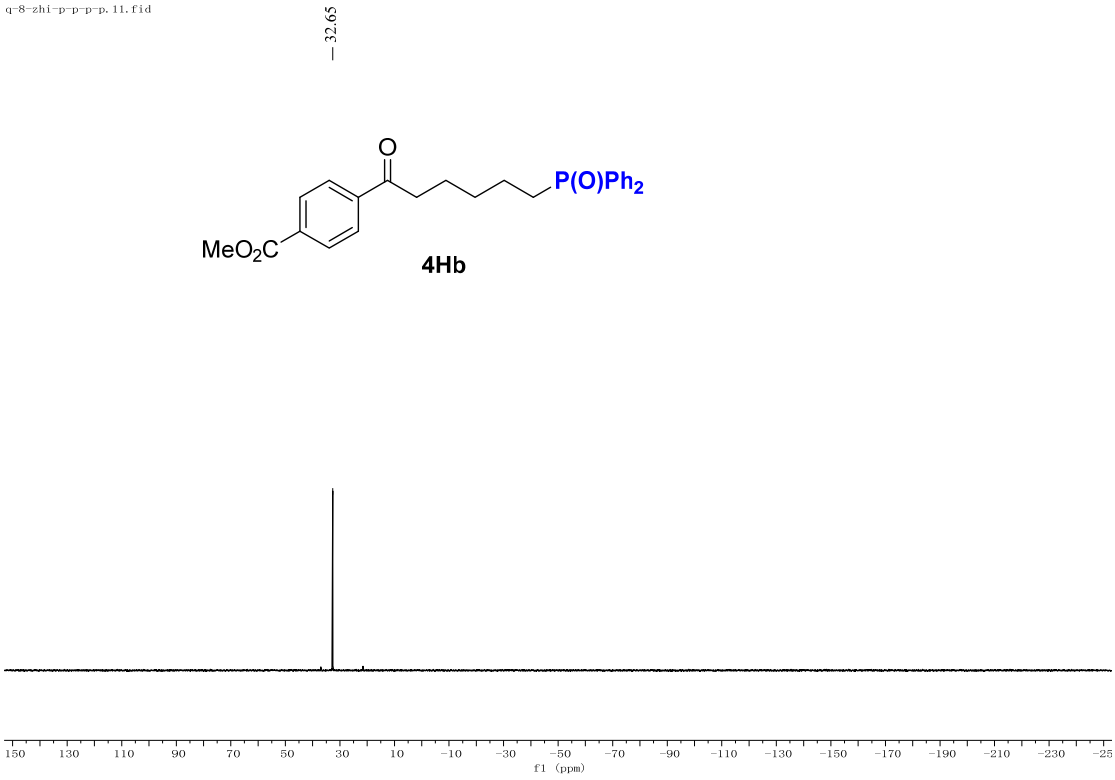

198.62
139.82
133.34
132.50
132.36
131.79
131.76
130.78
130.69
128.75
128.63
128.42
117.95
116.26
38.36
30.33
30.19
29.79
29.70
29.08
23.21
21.26
21.22
O
P(O)Ph2
NC
f1 (ppm)

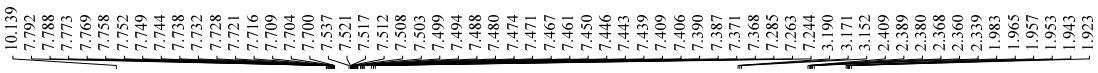
4Gb

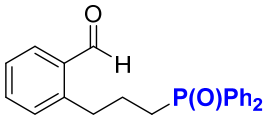

xq-8-cn-p.2.fid
32.77
O
P(O)Ph2
NC
4Gb
f1 (ppm)


O
P(O)Ph2
MeO2C
4Hb
2.00
1.99
4.14
6.29
3.07
2.09
2.15
4.22
2.06
f1 (ppm)

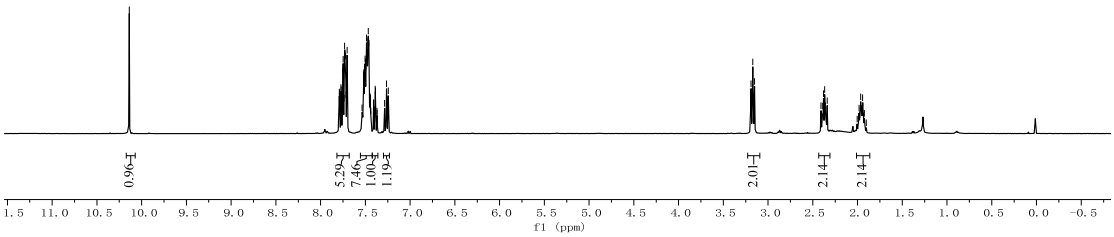

q-8-zhi-p-p-p-p.22.fid
199.56
166.23
140.10
133.76
133.42
132.44
131.74
131.72
130.79
130.70
129.82
128.73
128.61
127.90
52.47
38.43
30.43
30.29
29.84
29.13
23.33
21.30
21.26
O
P(O)Ph2
MeO2C
4Hb
f1 (ppm)

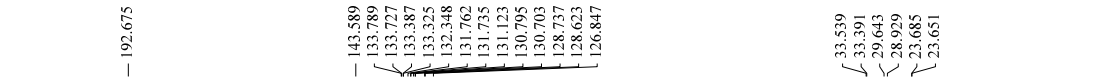

q-8-zhi-p-p-p-p.11.fid
32.65
O
P(O)Ph2
MeO2C
4Hb
f1 (ppm)


10.139
7.792
7.788
7.773
7.769
7.758
7.752
7.749
7.744
7.738
7.732
7.728
7.721
7.716
7.709
7.704
7.700
7.537
7.521
7.517
7.512
7.508
7.503
7.499
7.494
7.488
7.480
7.474
7.471
7.467
7.461
7.450
7.446
7.443
7.439
7.409
7.406
7.390
7.387
7.371
7.368
7.285
7.263
7.244
3.190
3.171
3.152
2.409
2.389
2.380
2.368
2.360
2.339
1.983
1.965
1.957
1.953
1.943
1.923


O
H
P(O)Ph2

4Jb


0.96
5.29
7.46
1.00
1.19
2.01
2.14
2.14
f1 (ppm)


192.675
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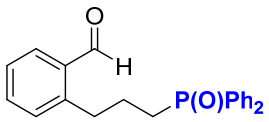
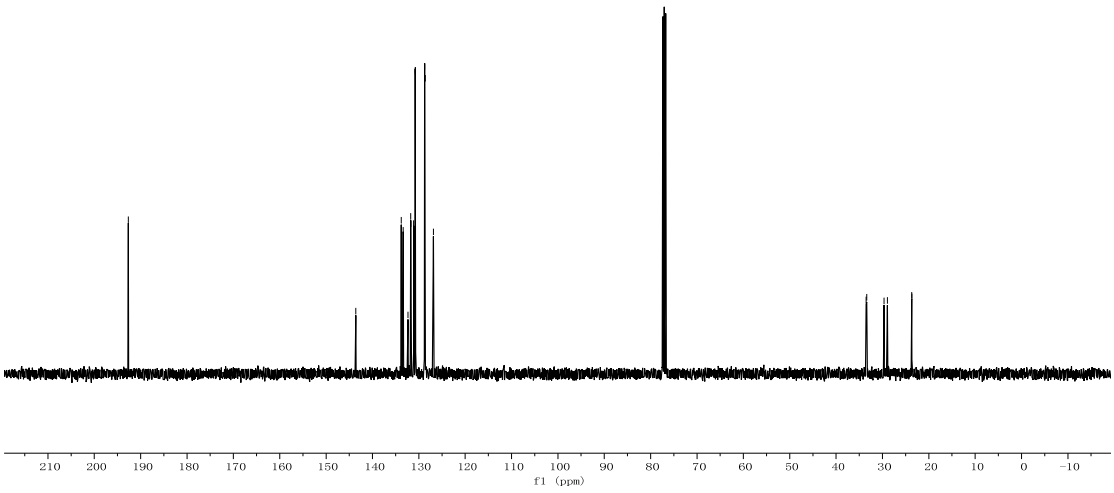
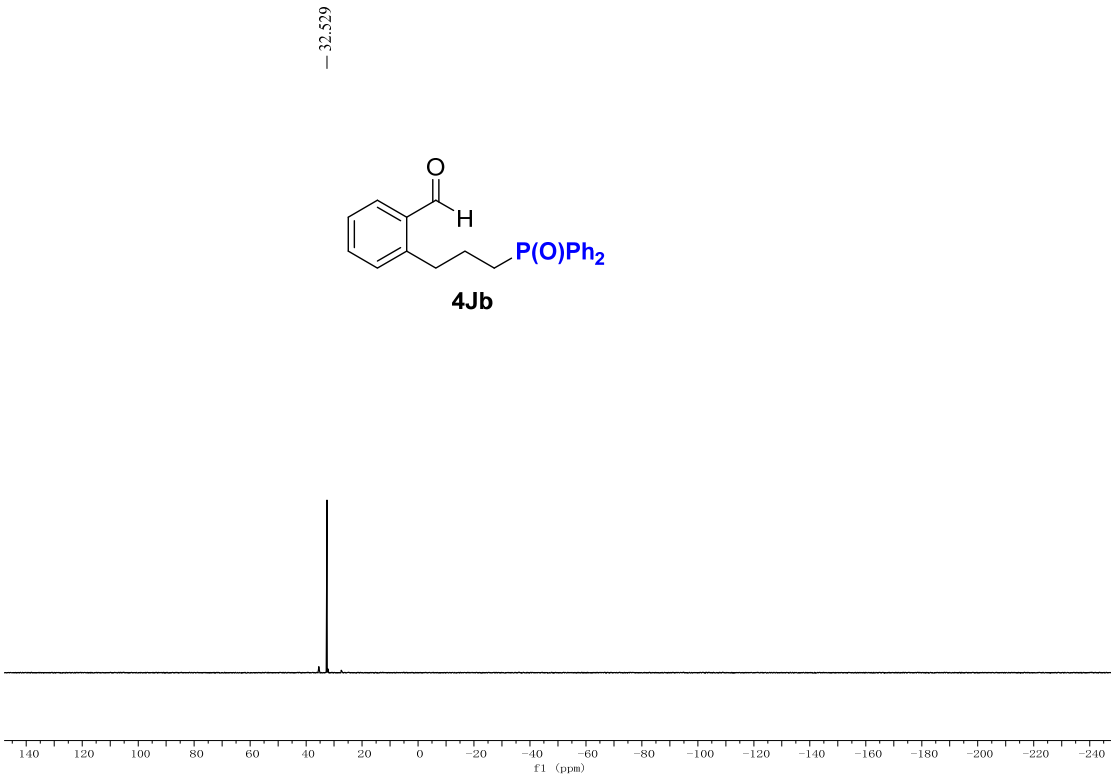
133.789
133.727
133.387
133.325
132.348
131.762
131.735
131.123
130.795
130.703
128.737
128.623
126.847
33.539
33.391
29.643
28.929
23.685
23.651

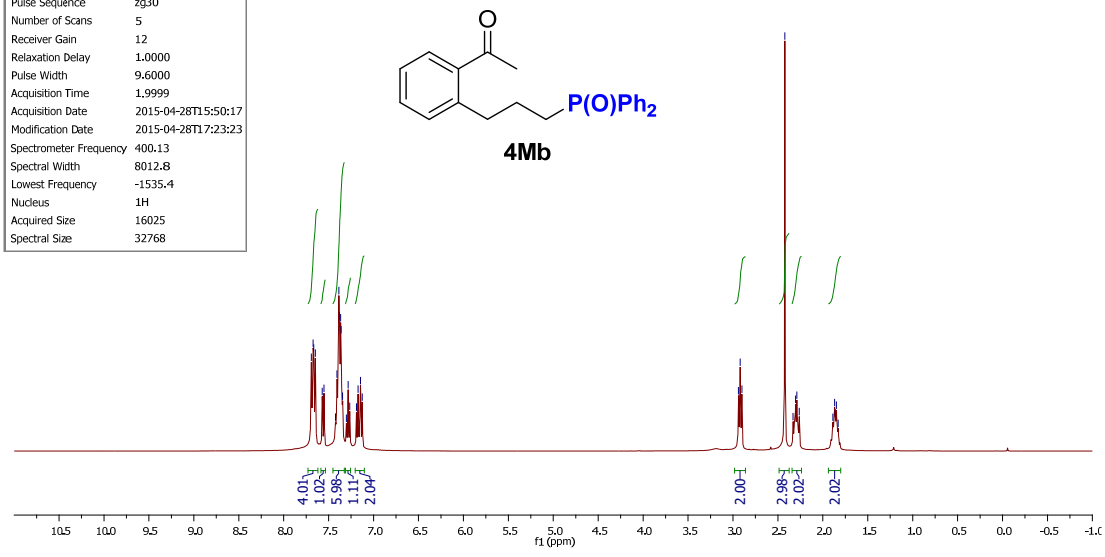

O
H
P(O)Ph2

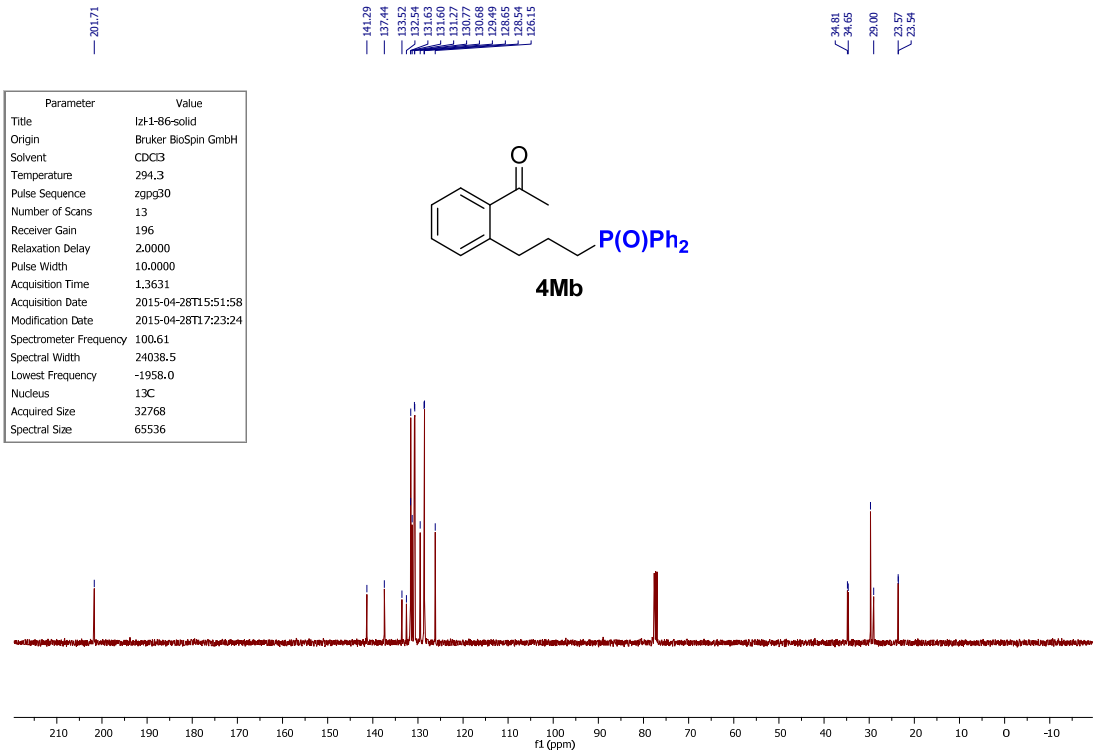
4Jb

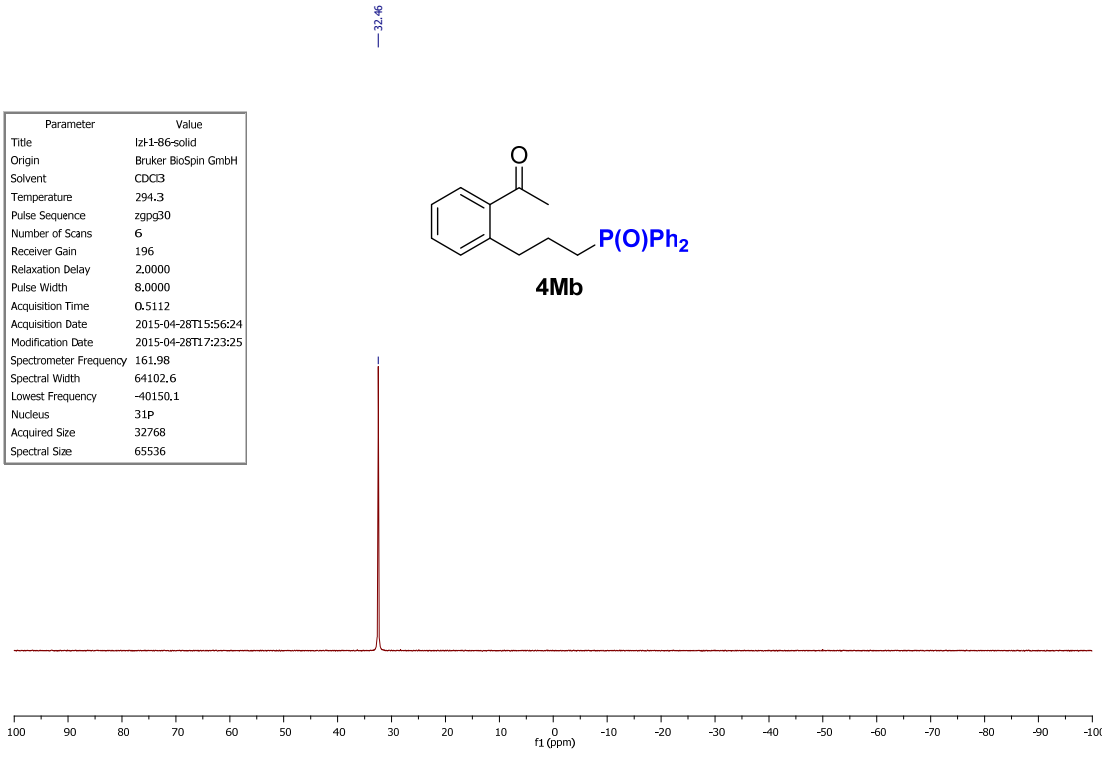

f1 (ppm)

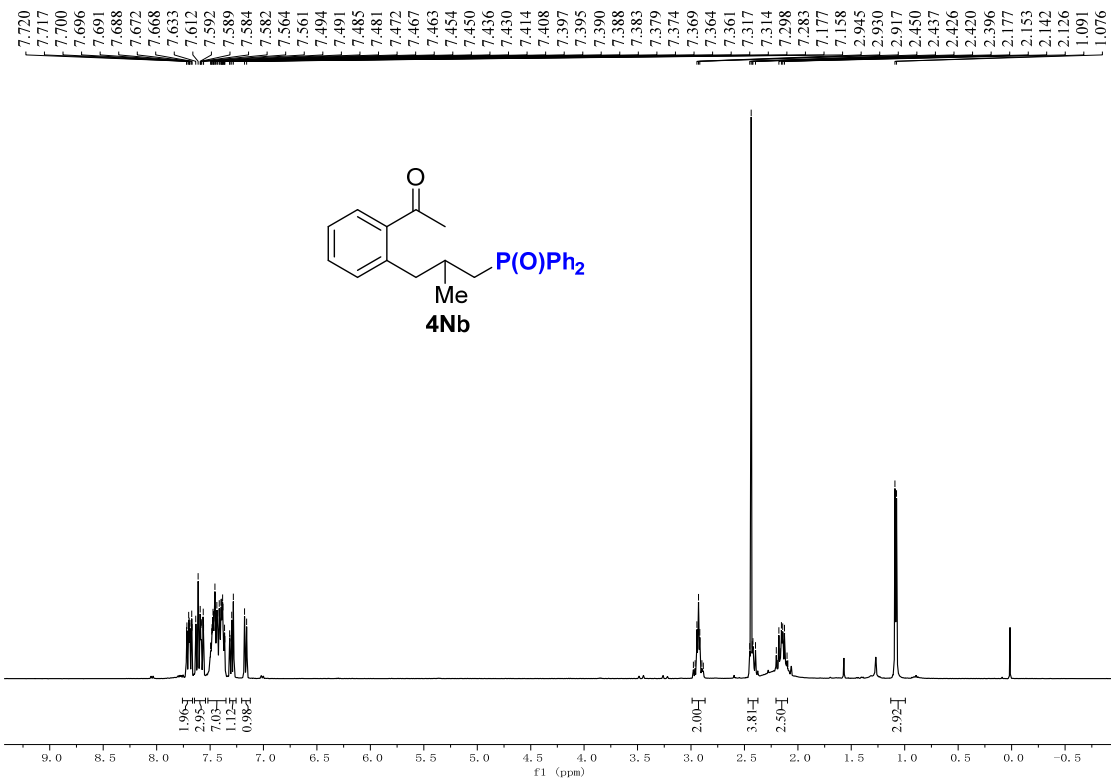


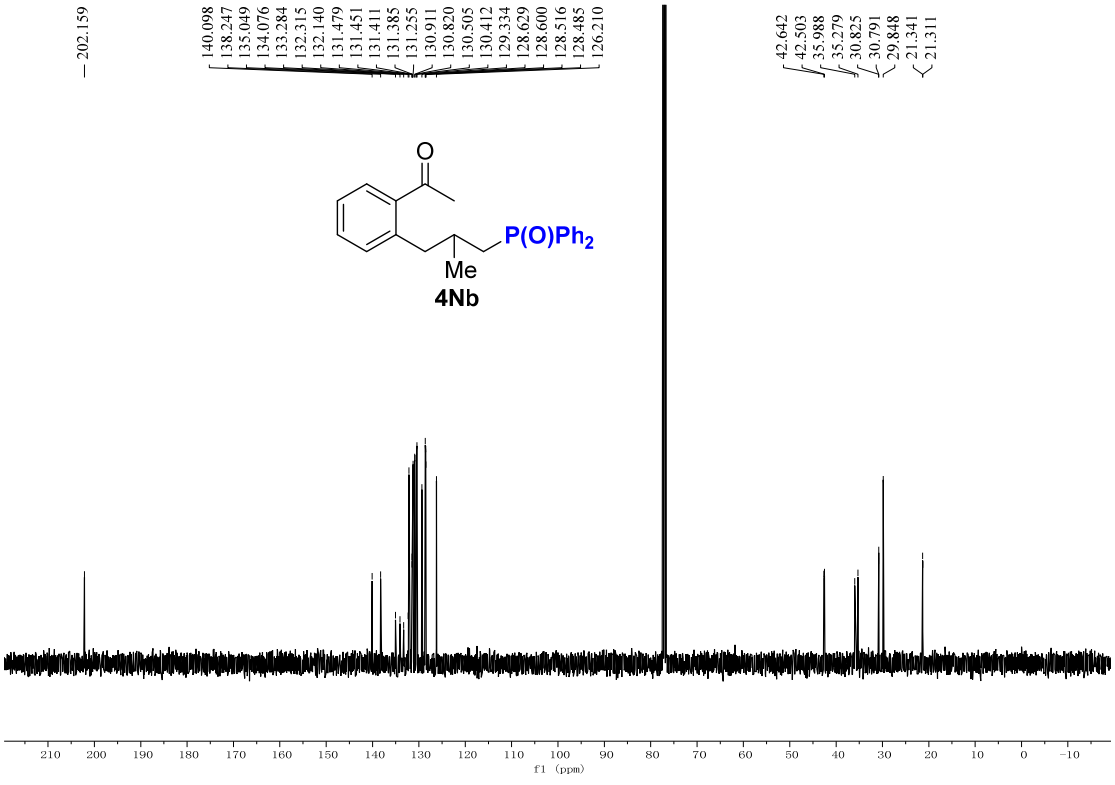
Parameter	Value
Title	lzl-1-86-solid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.3
Pulse Sequence	zg30
Number of Scans	5
Receiver Gain	12
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	1.9999
Acquisition Date	2015-04-28T15:50:17
Modification Date	2015-04-28T17:23:23
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
Acquired Size	16025
Spectral Size	32768

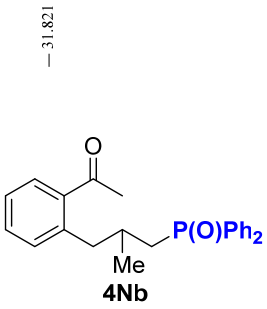



201.71
141.29
137.44
133.52
132.54
131.63
131.60
131.27
131.17
130.68
129.49
128.65
128.54
126.15
34.81
34.65
29.00
23.57
23.54
Parameter Value
Title lzl-1-86-solid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.3
Pulse Sequence zgpg30
Number of Scans 13
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-28T15:51:58
Modification Date 2015-04-28T17:23:24
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
4Mb
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

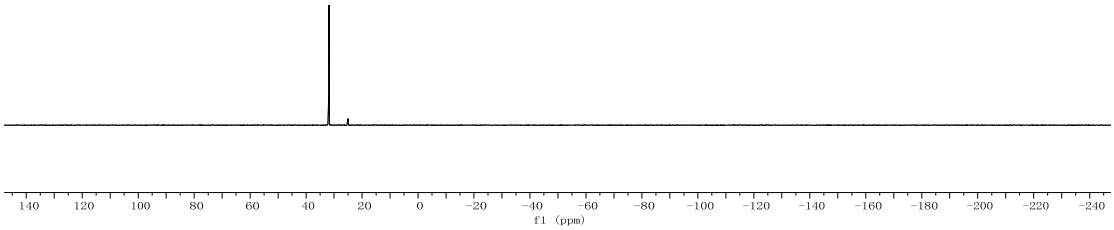

32.46
Parameter Value
Title lzl-1-86-solid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.3
Pulse Sequence zgpg30
Number of Scans 6
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 8.0000
Acquisition Time 0.5112
Acquisition Date 2015-04-28T15:56:24
Modification Date 2015-04-28T17:23:25
Spectrometer Frequency 161.98
Spectral Width 64102.6
Lowest Frequency -40150.1
Nucleus 31P
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
4Mb
100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90
f1 (ppm)


7.720
7.717
7.700
7.696
7.691
7.688
7.672
7.668
7.633
7.612
7.592
7.589
7.584
7.582
7.564
7.561
7.494
7.491
7.485
7.481
7.472
7.467
7.463
7.454
7.450
7.436
7.430
7.414
7.408
7.397
7.395
7.390
7.388
7.383
7.379
7.374
7.369
7.364
7.361
7.317
7.314
7.298
7.283
7.177
7.158
2.945
2.930
2.917
2.450
2.437
2.426
2.420
2.396
2.177
2.153
2.142
2.126
1.091
1.076
O
P(O)Ph2
Me
4Nb
1.96
2.95
7.03
1.12
0.98
2.00
3.81
2.50
2.92
f1 (ppm)

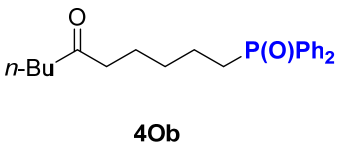

202.159
140.098
138.247
135.049
134.076
133.284
132.315
132.140
131.479
131.451
131.411
131.385
131.255
130.911
130.820
130.505
130.412
129.334
128.629
128.600
128.516
128.485
126.210
42.642
42.503
35.988
35.279
30.825
30.791
29.848
21.341
21.311
O
P(O)Ph2
Me
4Nb
f1 (ppm)



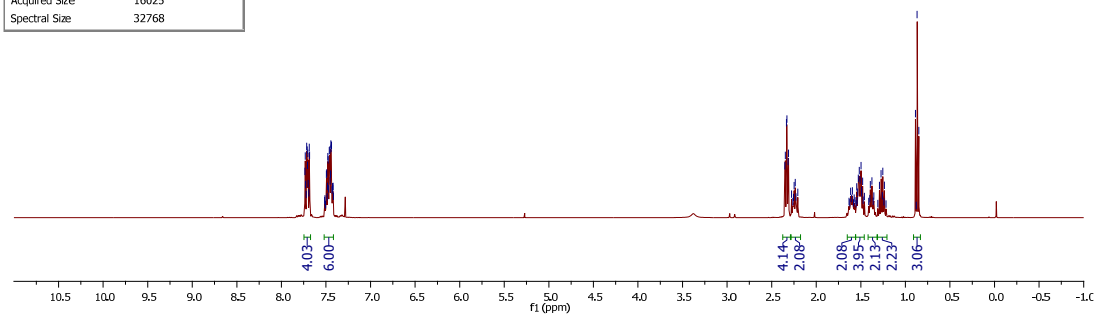
4Nb

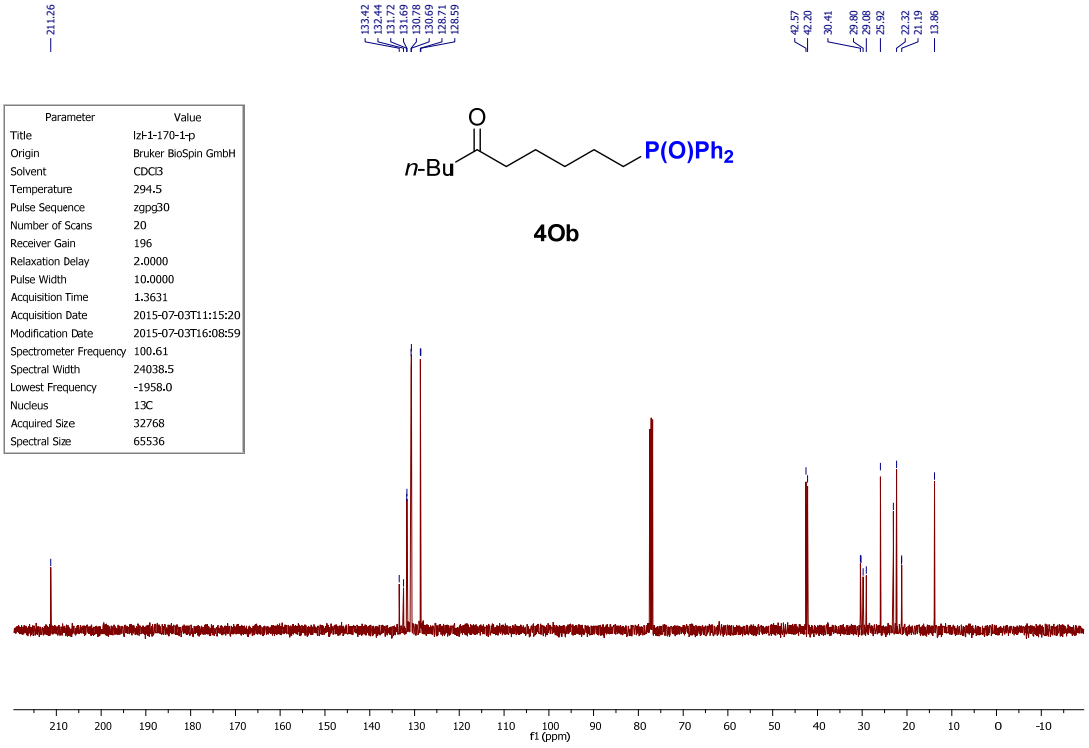


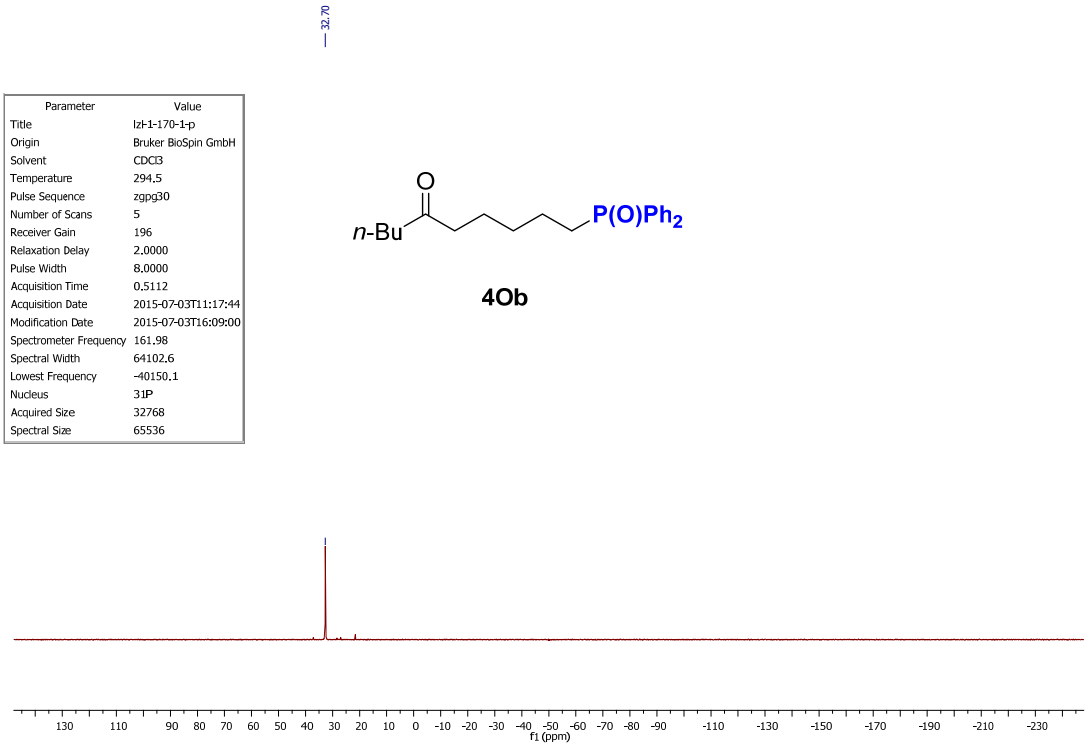
Parameter	Value
Title	lzl-1-170-1-p
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	294.4
Pulse Sequence	zg30
Number of Scans	4
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	1.9999
Acquisition Date	2015-07-03T11:13:27
Modification Date	2015-07-03T16:08:59
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
Acquired Size	16025
Spectral Size	32768



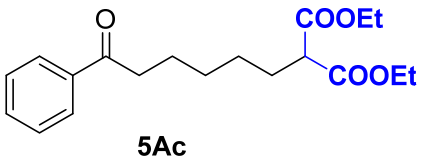
4Ob



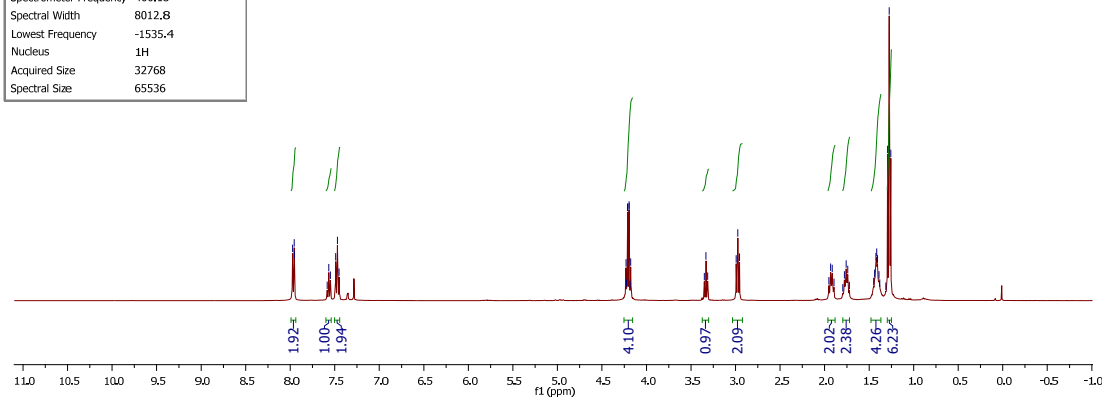

211.26
133.42
132.44
131.72
131.69
130.78
130.69
128.71
128.59
42.57
42.20
30.41
29.80
29.08
25.92
22.32
21.19
13.86
Parameter Value
Title lzl-1-170-1-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.5
Pulse Sequence zgpg30
Number of Scans 20
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-07-03T11:15:20
Modification Date 2015-07-03T16:08:59
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
n-Bu
P(O)Ph2
4Ob
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


32.70
Parameter Value
Title lzl-1-170-1-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.5
Pulse Sequence zgpg30
Number of Scans 5
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 8.0000
Acquisition Time 0.5112
Acquisition Date 2015-07-03T11:17:44
Modification Date 2015-07-03T16:09:00
Spectrometer Frequency 161.98
Spectral Width 64102.6
Lowest Frequency -40150.1
Nucleus 31P
Acquired Size 32768
Spectral Size 65536
O
n-Bu
P(O)Ph2
4Ob
130 110 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -130 -150 -170 -190 -210 -230
f1 (ppm)

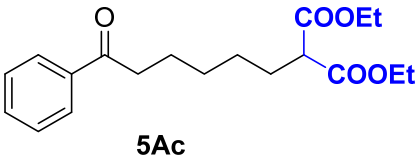
Parameter	Value
Title	lzl-1-36-2-p
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.0
Pulse Sequence	zg30
Number of Scans	5
Receiver Gain	32
Relaxation Delay	1.0000
Pulse Width	9.6000
Acquisition Time	4.0894
Acquisition Date	2015-03-27T21:06:01
Modification Date	2015-03-27T22:51:16
Spectrometer Frequency	400.13
Spectral Width	8012.8
Lowest Frequency	-1535.4
Nucleus	1H
Acquired Size	32768
Spectral Size	65536



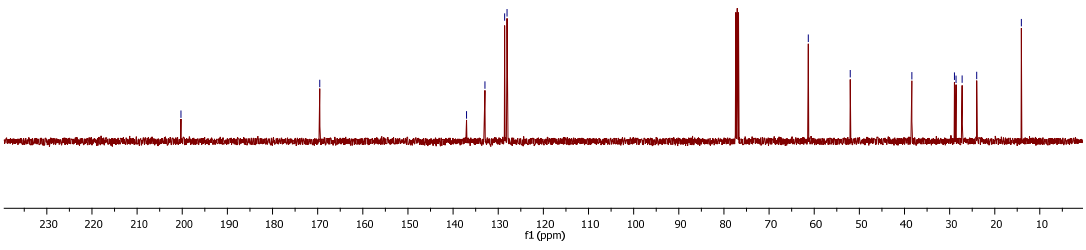
5Ac

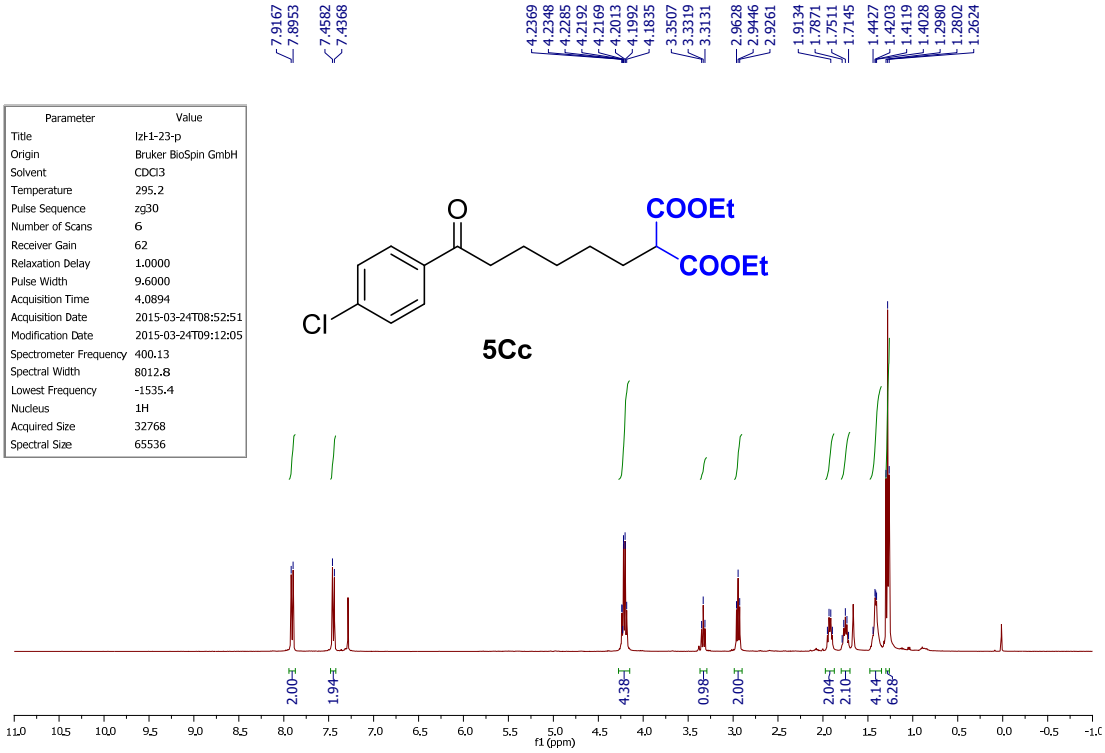


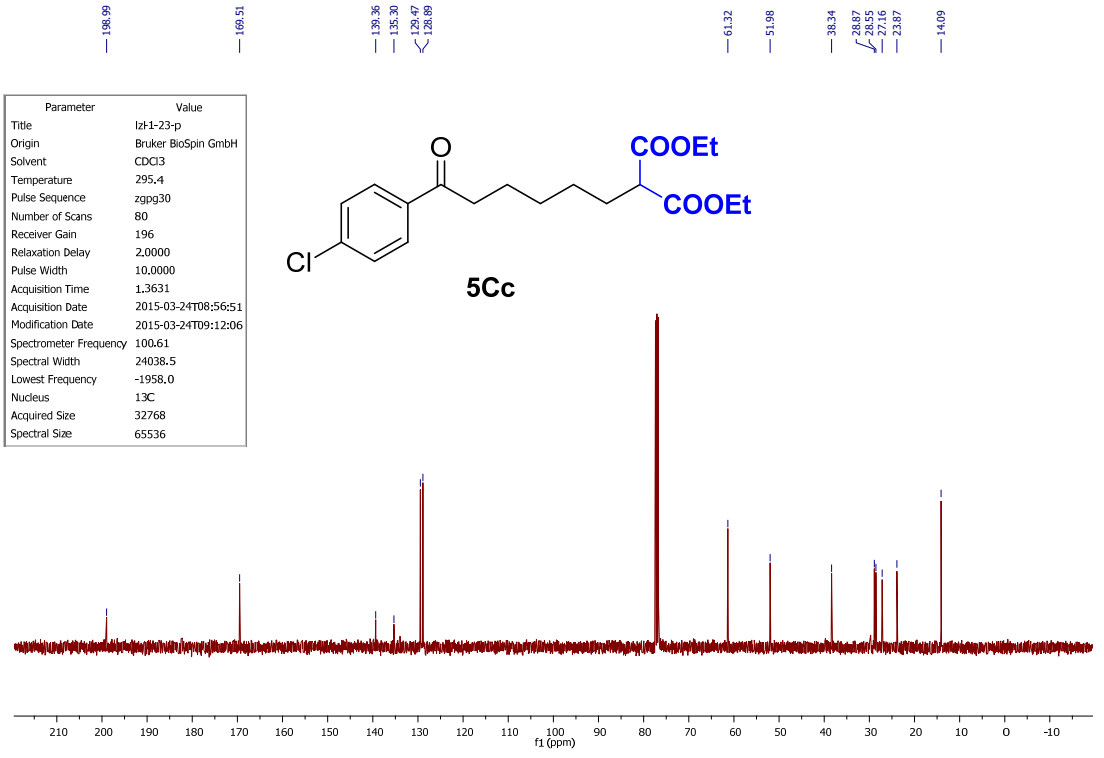
Parameter	Value
Title	lzl-1-36-2-p
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.1
Pulse Sequence	zgpg30
Number of Scans	35
Receiver Gain	196
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2015-03-27T21:08:02
Modification Date	2015-03-27T22:51:17
Spectrometer Frequency	100.61
Spectral Width	24038.5
Lowest Frequency	54.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

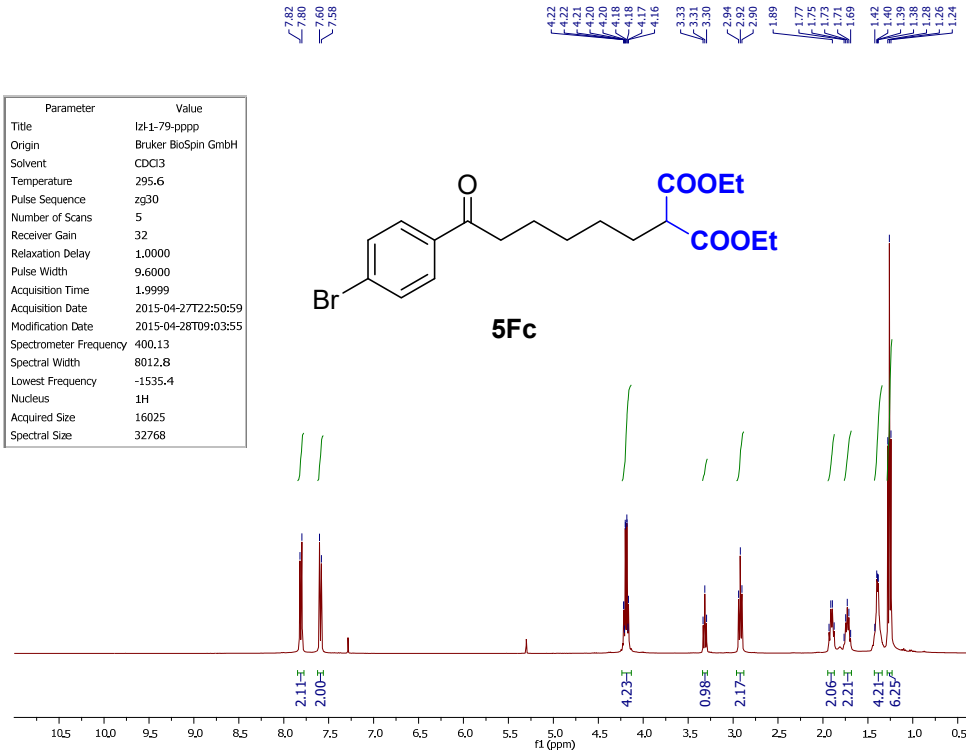


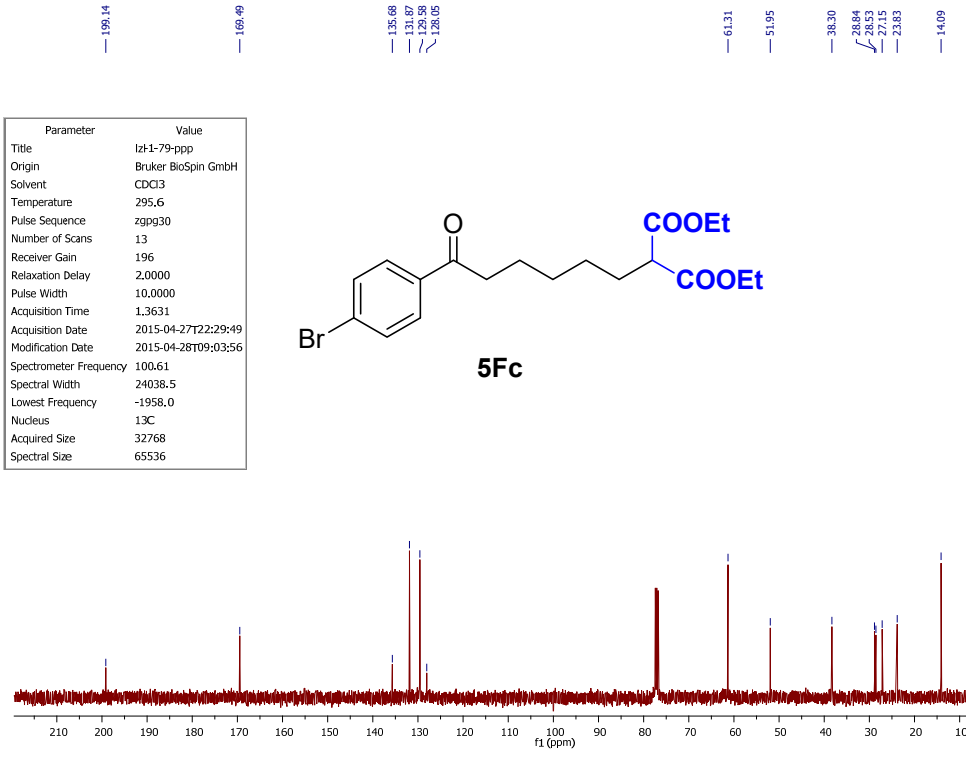
5Ac

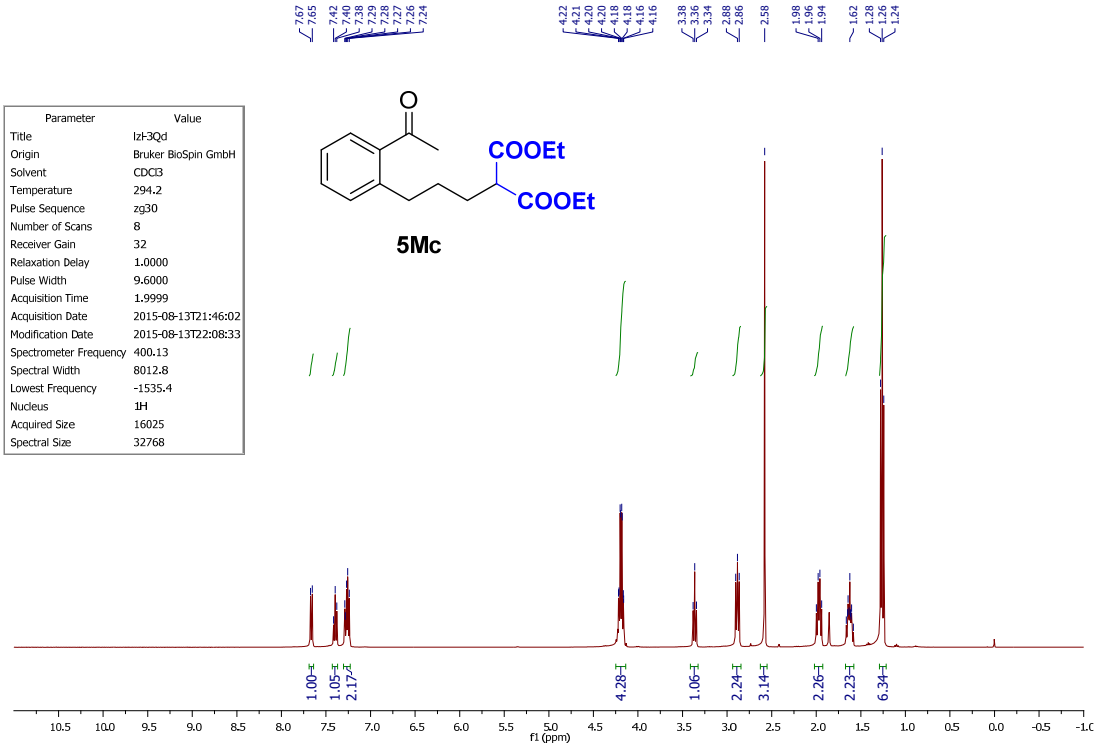


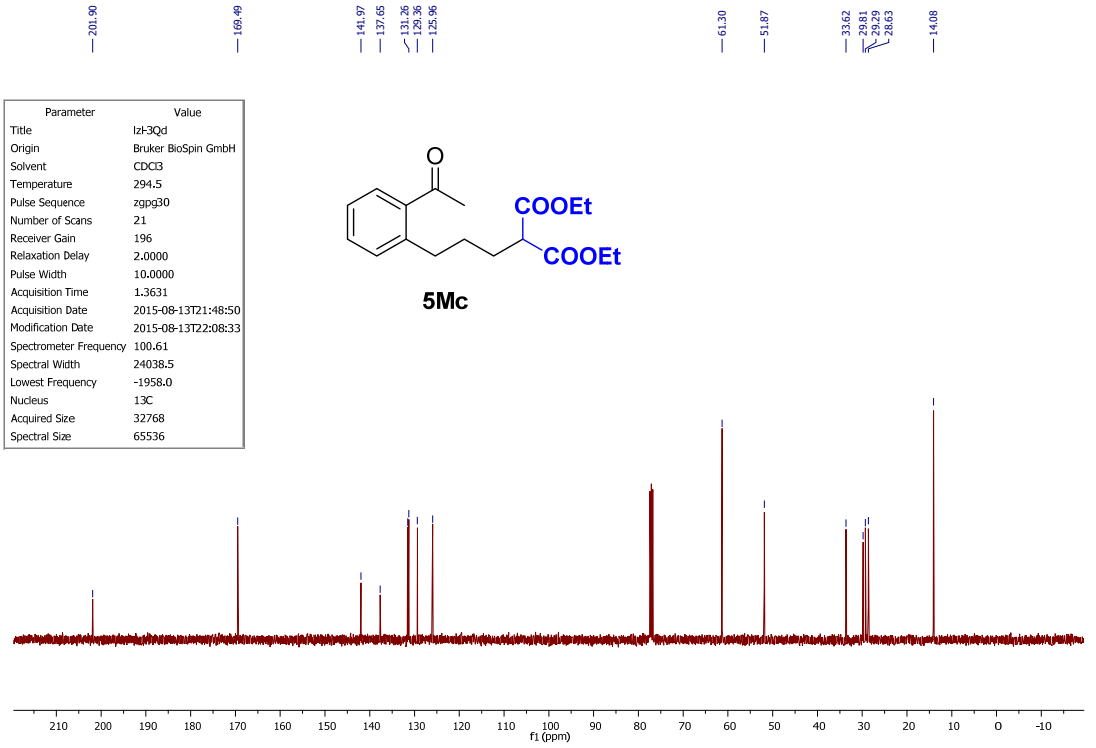

7.9167
7.8953
7.4582
7.4368
4.2369
4.2348
4.2285
4.2192
4.2169
4.2013
4.1992
4.1835
3.3507
3.3319
3.3131
2.9628
2.9446
2.9261
1.9134
1.7871
1.7511
1.7145
1.4427
1.4203
1.4119
1.4028
1.2980
1.2802
1.2624
Parameter Value
Title lzl-1-23-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.2
Pulse Sequence zg30
Number of Scans 6
Receiver Gain 62
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 4.0894
Acquisition Date 2015-03-24T08:52:51
Modification Date 2015-03-24T09:12:05
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
O
COOEt
COOEt
Cl
5Cc
2.00
1.94
4.38
0.98
2.00
2.04
2.10
4.14
6.28
f1 (ppm)

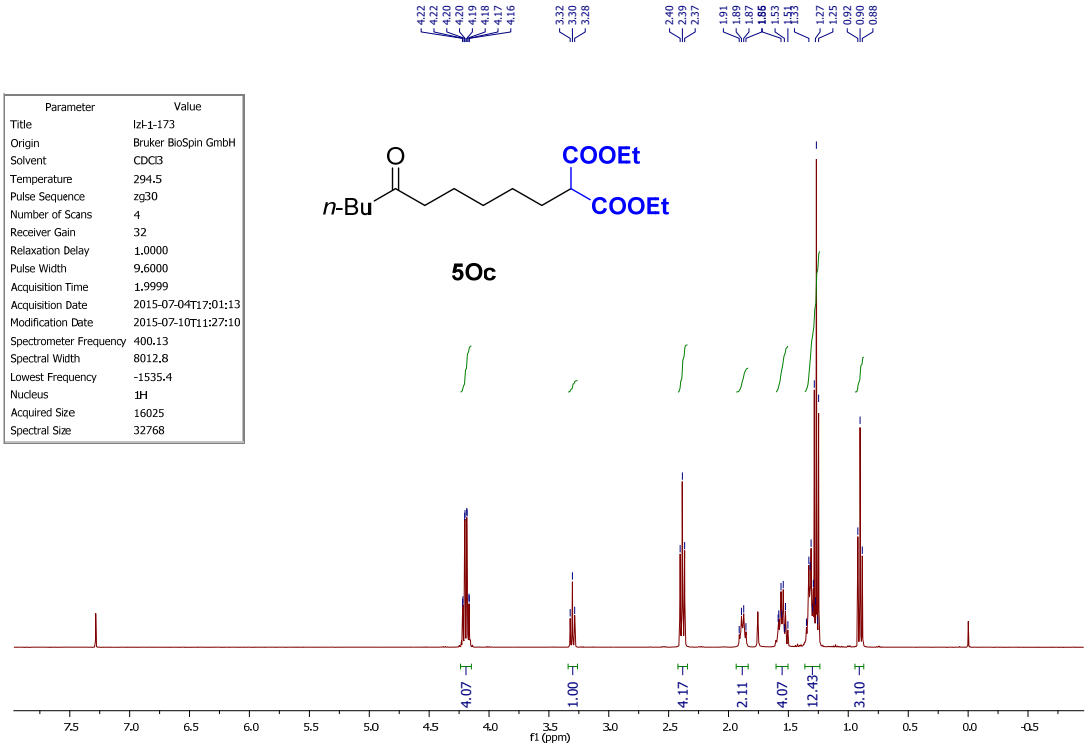

198.99
169.51
139.36
135.30
129.47
128.89
61.32
51.98
38.34
28.87
28.55
27.16
23.87
14.09
Parameter Value
Title lzl-1-23-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.4
Pulse Sequence zgpg30
Number of Scans 80
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-03-24T08:56:51
Modification Date 2015-03-24T09:12:06
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
COOEt
COOEt
Cl
5Cc
f1 (ppm)

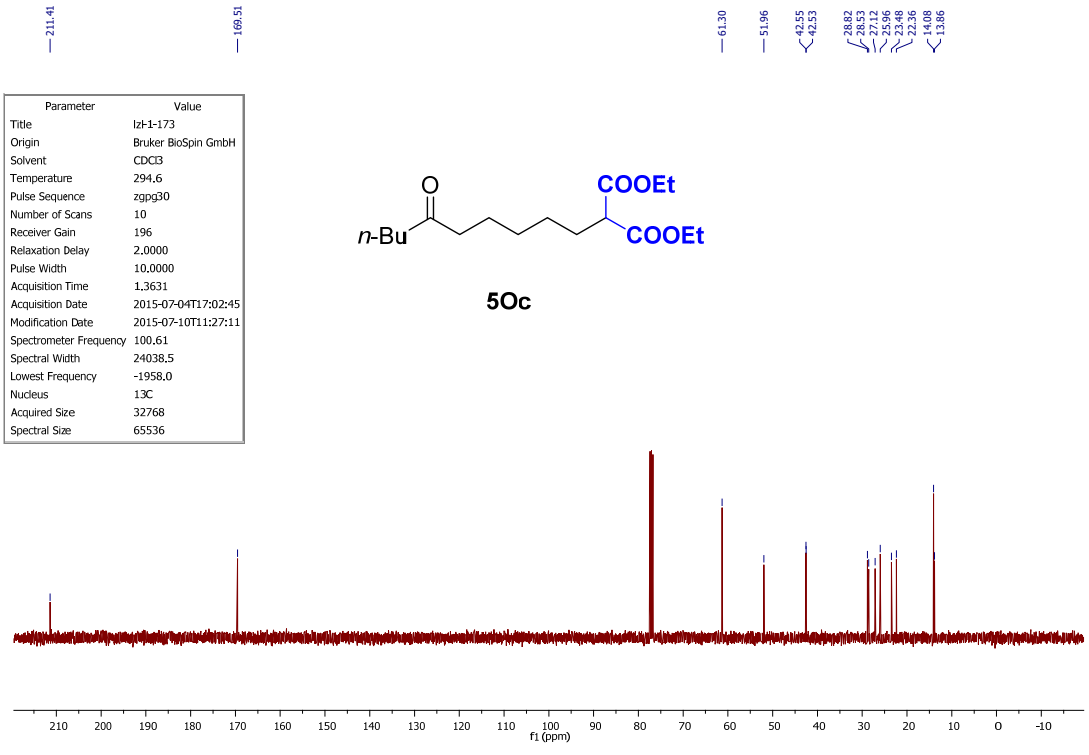

7.82
7.80
7.60
7.58
4.22
4.22
4.21
4.21
4.20
4.20
4.18
4.18
4.17
4.16
3.33
3.31
3.30
2.94
2.92
2.90
1.89
1.77
1.75
1.73
1.71
1.69
1.42
1.40
1.38
1.28
1.26
1.24
Parameter Value
Title lzl-1-79-pppp
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.6
Pulse Sequence zg30
Number of Scans 5
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-27T22:50:59
Modification Date 2015-04-28T09:03:55
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
COOEt
COOEt
Br
5Fc
2.11
2.00
4.23
0.98
2.17
2.06
2.21
4.21
6.25
f1 (ppm)

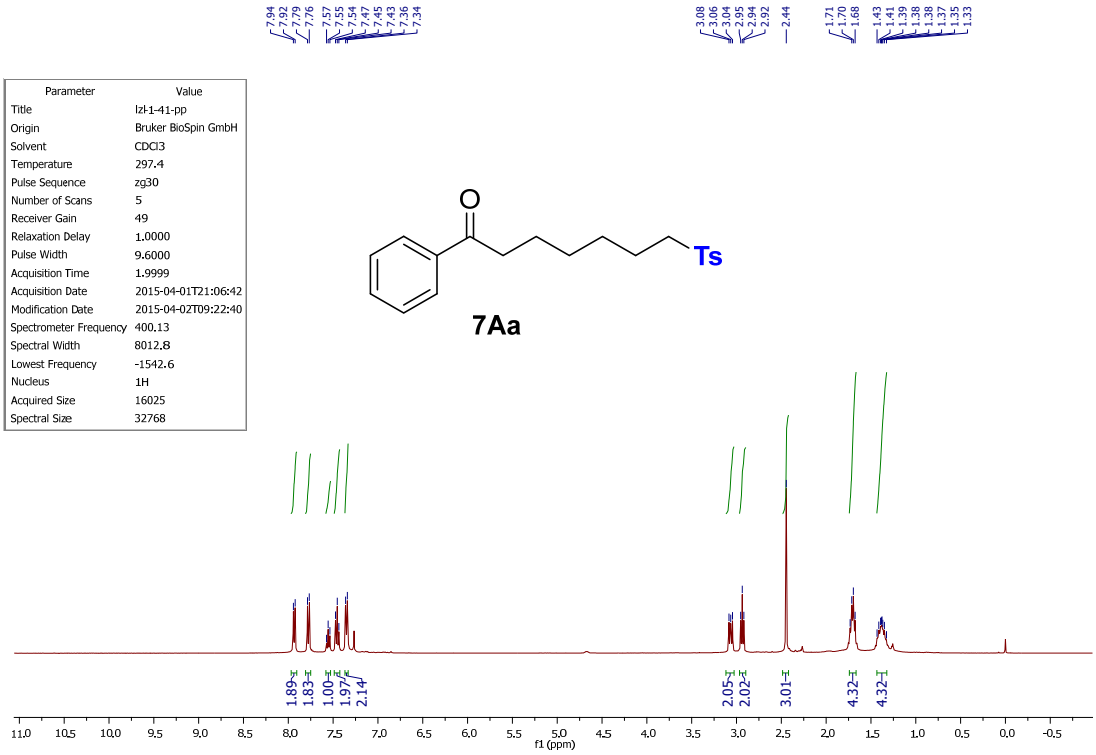

199.14
169.49
135.68
131.87
129.58
128.05
61.31
51.95
38.30
28.84
28.53
27.15
23.83
14.09
Parameter Value
Title lzl-1-79-ppp
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.6
Pulse Sequence zgpg30
Number of Scans 13
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-27T22:29:49
Modification Date 2015-04-28T09:03:56
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
COOEt
COOEt
Br
5Fc
f1 (ppm)

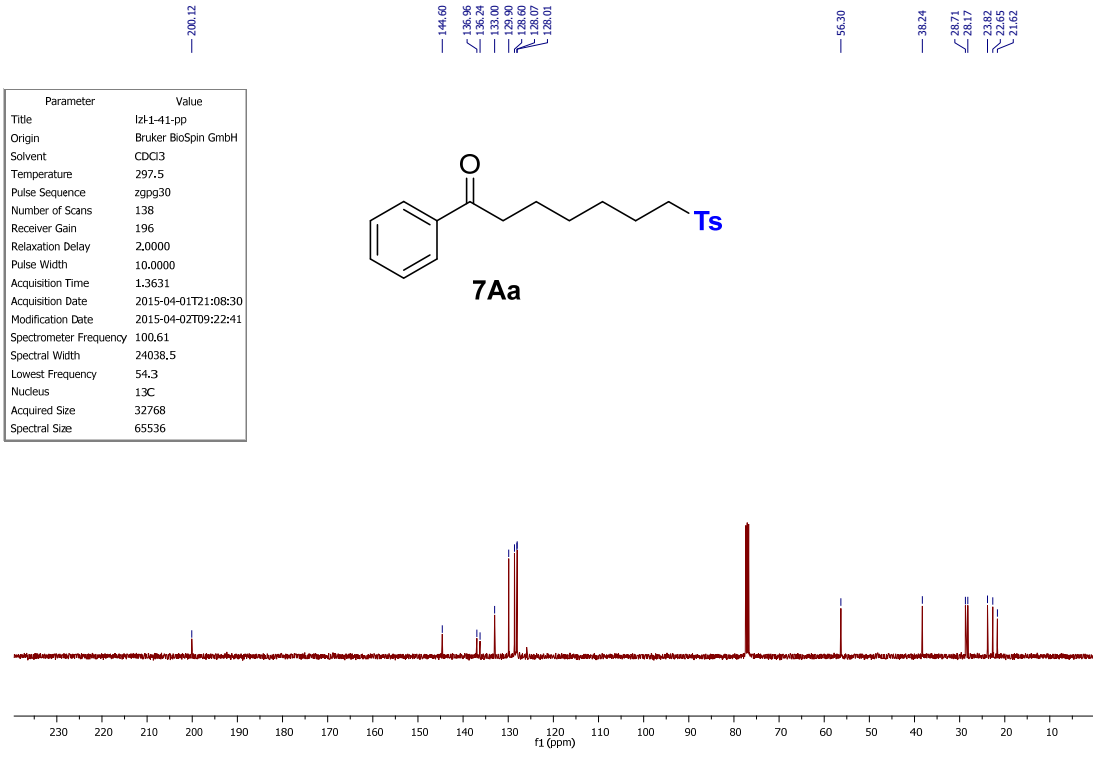

7.67
7.65
7.42
7.40
7.38
7.29
7.28
7.27
7.26
7.24
4.22
4.21
4.20
4.20
4.18
4.18
4.16
4.16
3.38
3.36
3.34
2.88
2.86
2.58
1.98
1.96
1.94
1.62
1.28
1.26
1.24
Parameter Value
Title lzl-3Qd
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.2
Pulse Sequence zg30
Number of Scans 8
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-08-13T21:46:02
Modification Date 2015-08-13T22:08:33
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
COOEt
COOEt
5Mc
1.00
1.05
2.17
4.28
1.06
2.24
3.14
2.26
2.23
6.34
f1 (ppm)

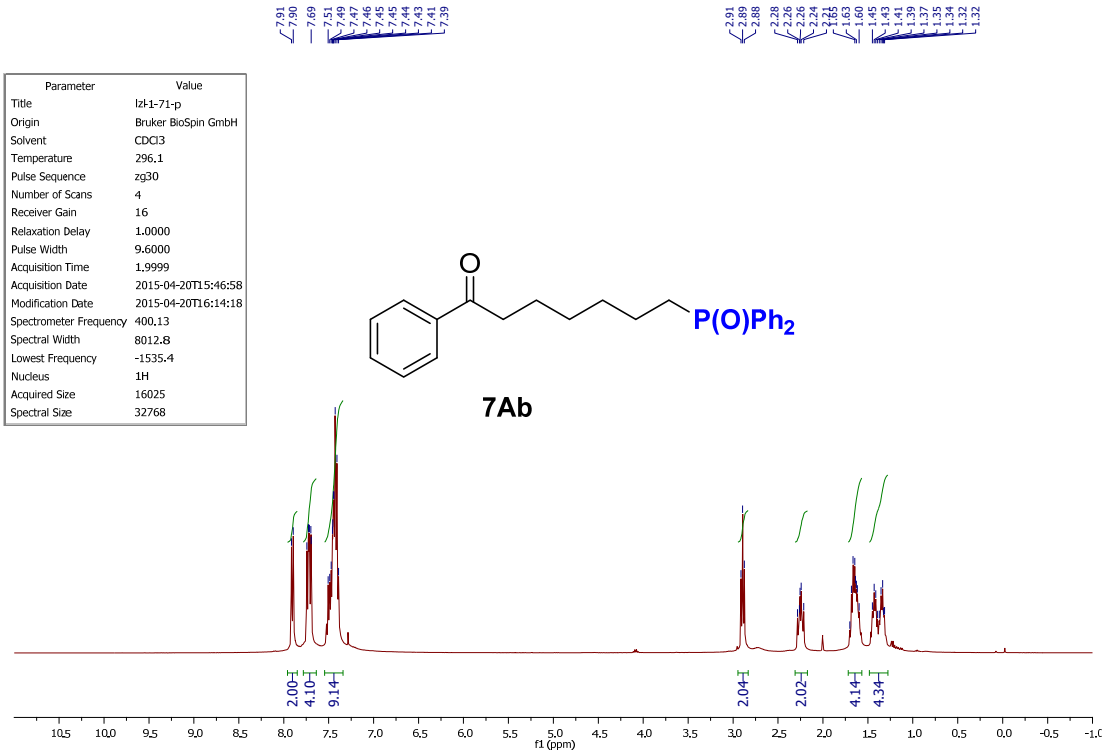

201.90
169.49
141.97
137.65
131.26
129.36
125.96
61.30
51.87
33.62
29.81
29.29
28.63
14.08
Parameter Value
Title lzl-3Qd
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.5
Pulse Sequence zgpg30
Number of Scans 21
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-08-13T21:48:50
Modification Date 2015-08-13T22:08:33
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
COOEt
COOEt
5Mc
f1 (ppm)

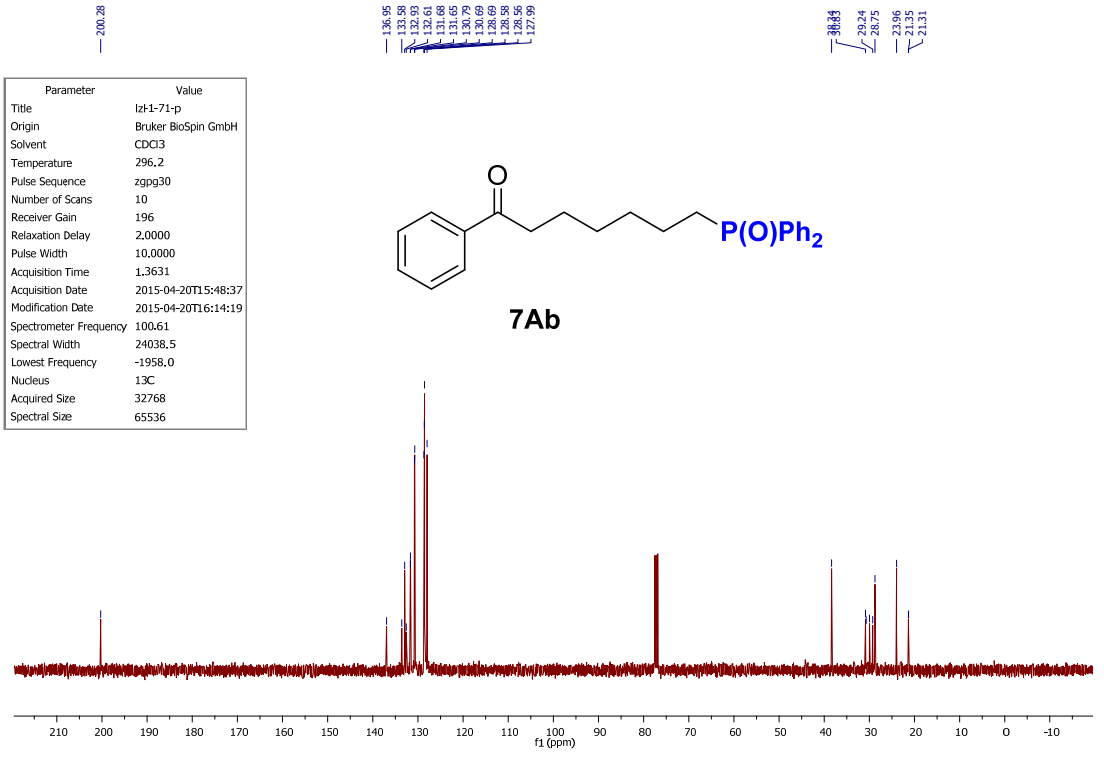

4.22
4.22
4.20
4.20
4.19
4.18
4.17
4.16
3.32
3.30
3.28
2.40
2.39
2.37
1.91
1.89
1.87
1.86
1.53
1.51
1.33
1.27
1.25
0.92
0.90
0.88
Parameter Value
Title lzl-1-173
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.5
Pulse Sequence zg30
Number of Scans 4
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-07-04T17:01:13
Modification Date 2015-07-10T11:27:10
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
COOEt
n-Bu
COOEt
5Oc
4.07
1.00
4.17
2.11
4.07
12.43
3.10
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
-0.5
f1 (ppm)

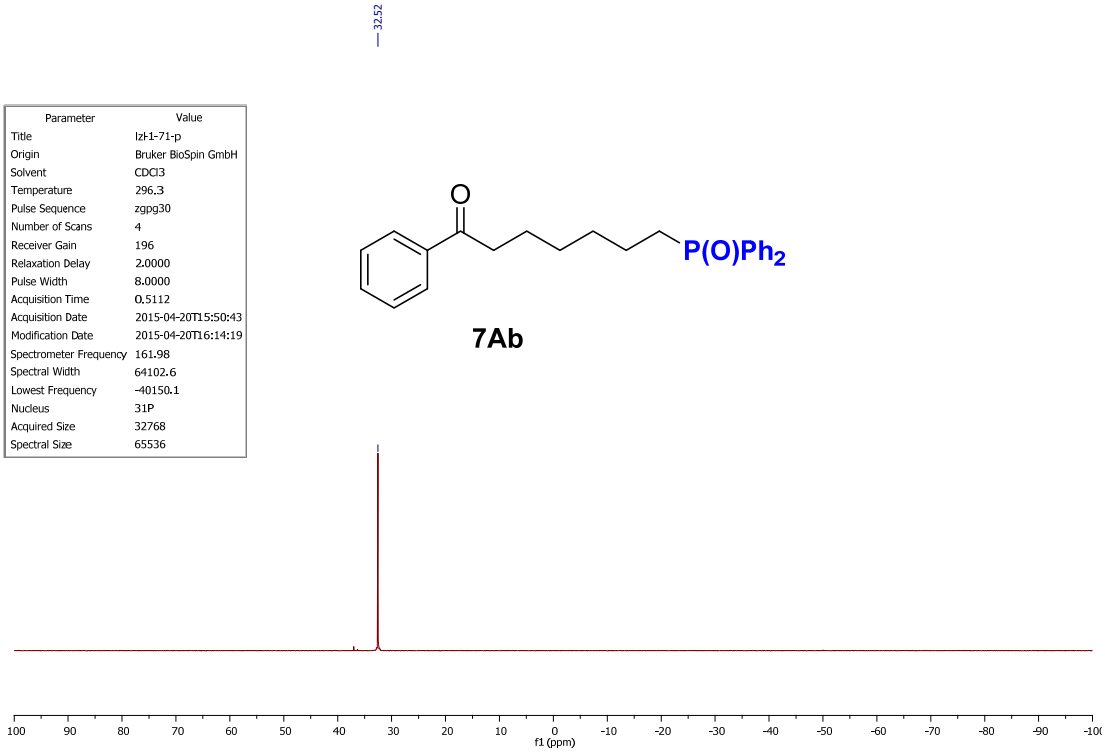

211.41
169.51
61.30
51.96
42.55
42.53
28.82
28.53
27.12
25.46
23.48
22.36
14.08
13.86
Parameter Value
Title lzl-1-173
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.6
Pulse Sequence zgpg30
Number of Scans 10
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-07-04T17:02:45
Modification Date 2015-07-10T11:27:11
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
COOEt
n-Bu
COOEt
5Oc
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

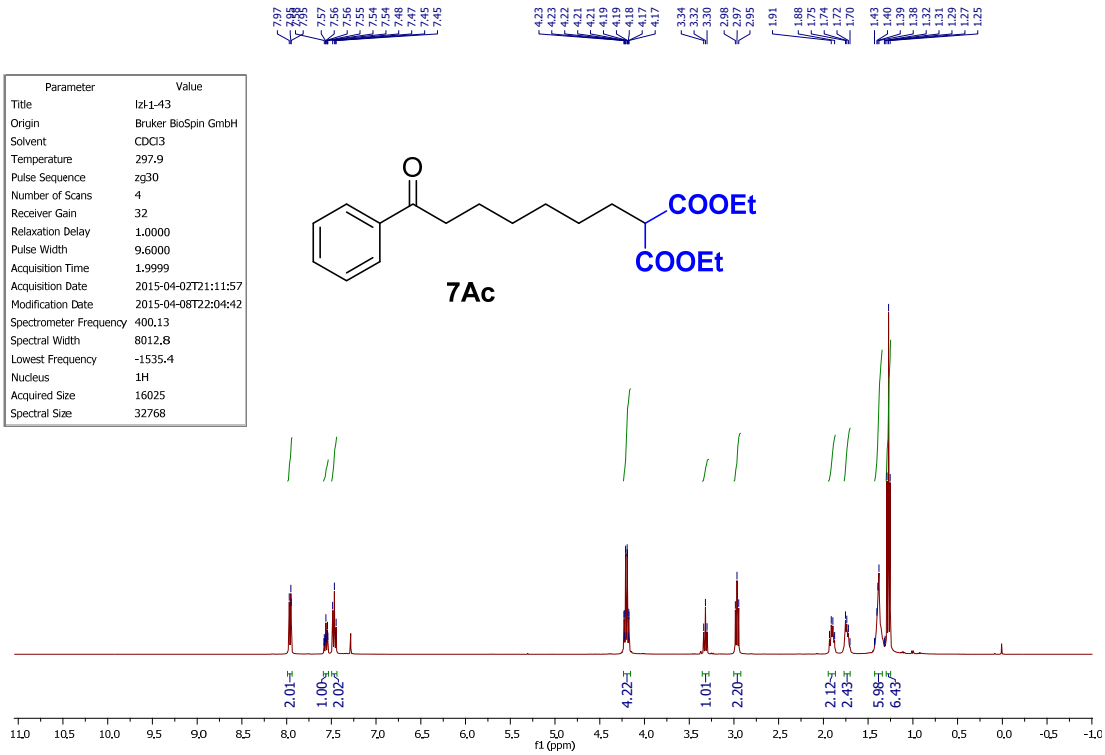

7.94
7.92
7.79
7.76
7.57
7.55
7.54
7.47
7.45
7.43
7.36
7.34
3.08
3.06
3.04
2.95
2.94
2.92
2.44
1.71
1.70
1.68
1.43
1.41
1.39
1.38
1.37
1.35
1.33
Parameter Value
Title lzl-1-41-pp
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 297.4
Pulse Sequence zg30
Number of Scans 5
Receiver Gain 49
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-01T21:06:42
Modification Date 2015-04-02T09:22:40
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1542.6
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
Ts
7Aa
1.89
1.83
1.00
1.97
2.14
2.05
2.02
3.01
4.32
4.32
11.0 10.5 10.0 9.5 9.0 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 -0.5
f1 (ppm)


200.12
144.60
136.96
136.24
133.00
129.90
128.60
128.07
128.01
56.30
38.24
28.71
28.17
23.82
22.65
21.62
Parameter Value
Title lzl-1-41-pp
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 297.5
Pulse Sequence zgpg30
Number of Scans 138
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-01T21:08:30
Modification Date 2015-04-02T09:22:41
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency 54.3
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
Ts
7Aa
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

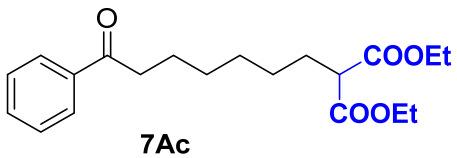

7.91
7.90
7.69
7.51
7.49
7.47
7.46
7.45
7.45
7.44
7.43
7.41
7.39
2.91
2.89
2.88
2.28
2.26
2.26
2.24
2.21
1.65
1.63
1.60
1.45
1.43
1.41
1.39
1.37
1.35
1.34
1.32
1.32
Parameter Value
Title lzl-1-71-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 296.1
Pulse Sequence zg30
Number of Scans 4
Receiver Gain 16
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-20T15:46:58
Modification Date 2015-04-20T16:14:18
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
P(O)Ph2
7Ab
2.00
4.10
9.14
2.04
2.02
4.14
4.34
f1 (ppm)


200.28
136.95
133.58
132.93
132.61
131.68
131.65
130.79
130.69
128.69
128.58
128.56
127.99
38.34
38.03
29.24
28.75
23.96
21.35
21.31
Parameter Value
Title lzl-1-71-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 296.2
Pulse Sequence zgpg30
Number of Scans 10
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-20T15:48:37
Modification Date 2015-04-20T16:14:19
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
7Ab
f1 (ppm)

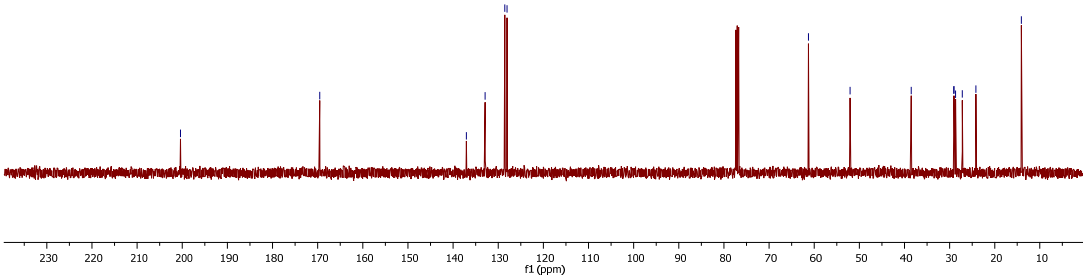

32.52
Parameter Value
Title lzl-1-71-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 296.3
Pulse Sequence zgpg30
Number of Scans 4
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 8.0000
Acquisition Time 0.5112
Acquisition Date 2015-04-20T15:50:43
Modification Date 2015-04-20T16:14:19
Spectrometer Frequency 161.98
Spectral Width 64102.6
Lowest Frequency -40150.1
Nucleus 31P
Acquired Size 32768
Spectral Size 65536
O
P(O)Ph2
7Ab
f1 (ppm)

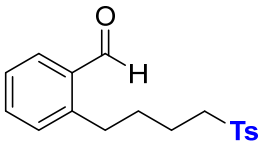

Parameter Value
Title lzl-1-43
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 297.9
Pulse Sequence zg30
Number of Scans 4
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-02T21:11:57
Modification Date 2015-04-08T22:04:42
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
COOEt
COOEt
7Ac
2.01
1.00
2.02
4.22
1.01
2.20
2.12
2.43
5.98
6.43
f1 (ppm)

Parameter	Value
Title	lzl-1-43
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	297.9
Pulse Sequence	zgpg30
Number of Scans	17
Receiver Gain	196
Relaxation Delay	2.0000
Pulse Width	10.0000
Acquisition Time	1.3631
Acquisition Date	2015-04-02T21:13:50
Modification Date	2015-04-08T22:04:42
Spectrometer Frequency	100.61
Spectral Width	24038.5
Lowest Frequency	54.3
Nucleus	13C
Acquired Size	32768
Spectral Size	65536

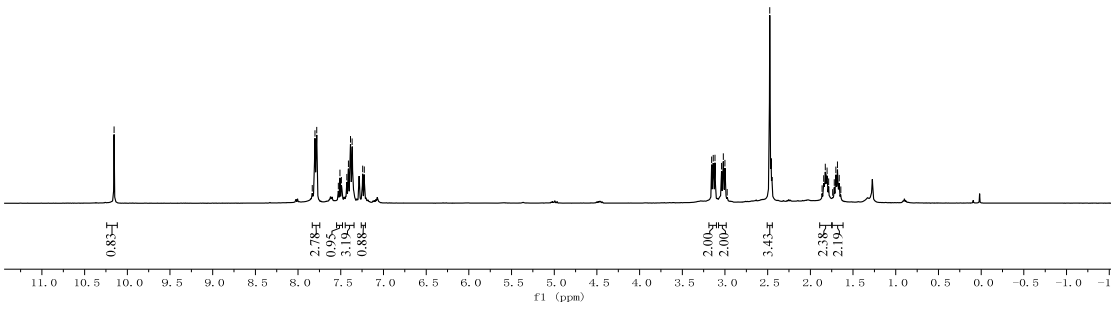


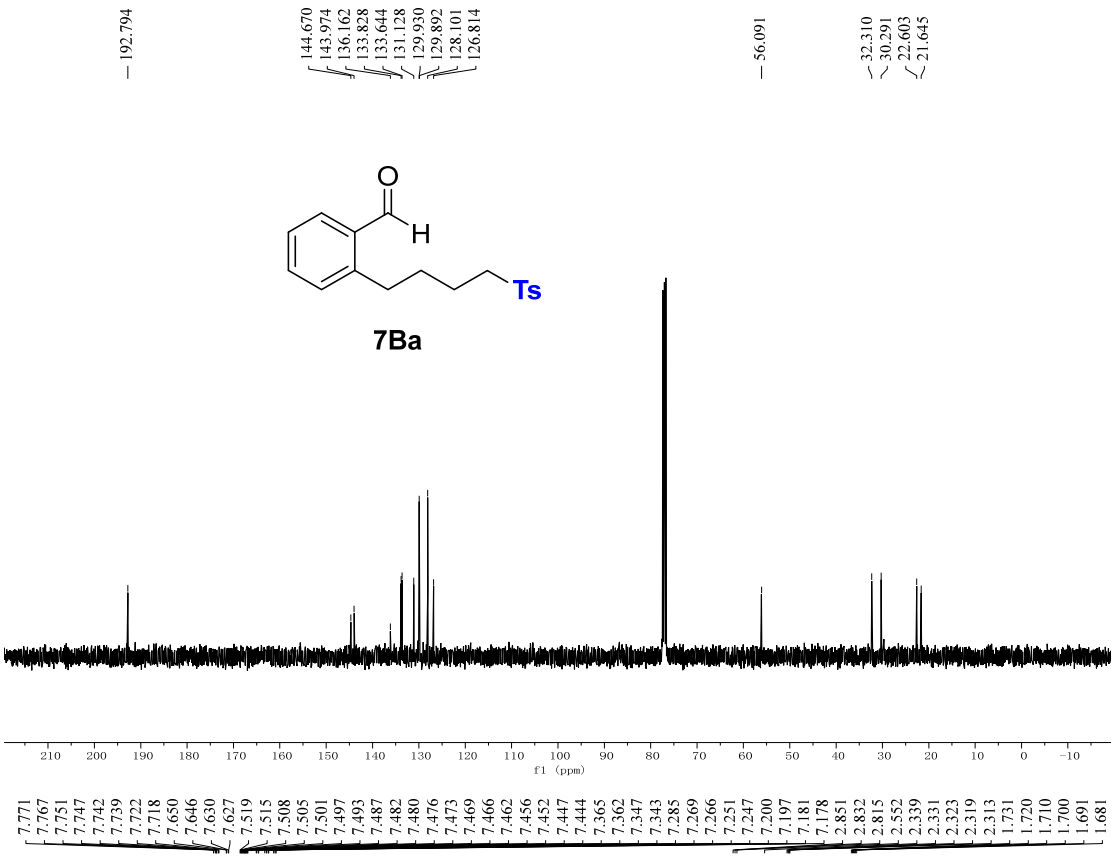
7Ac



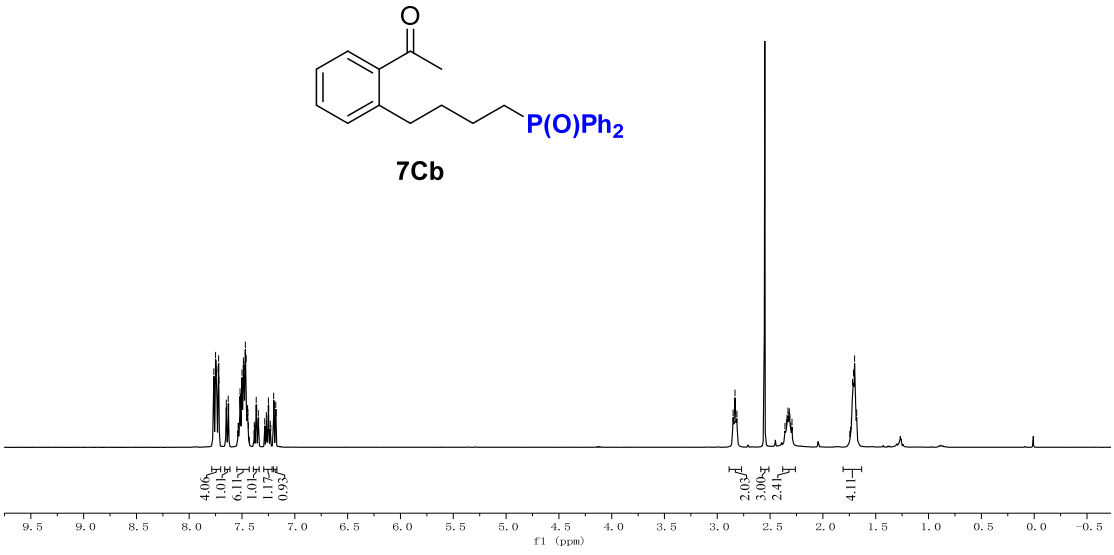


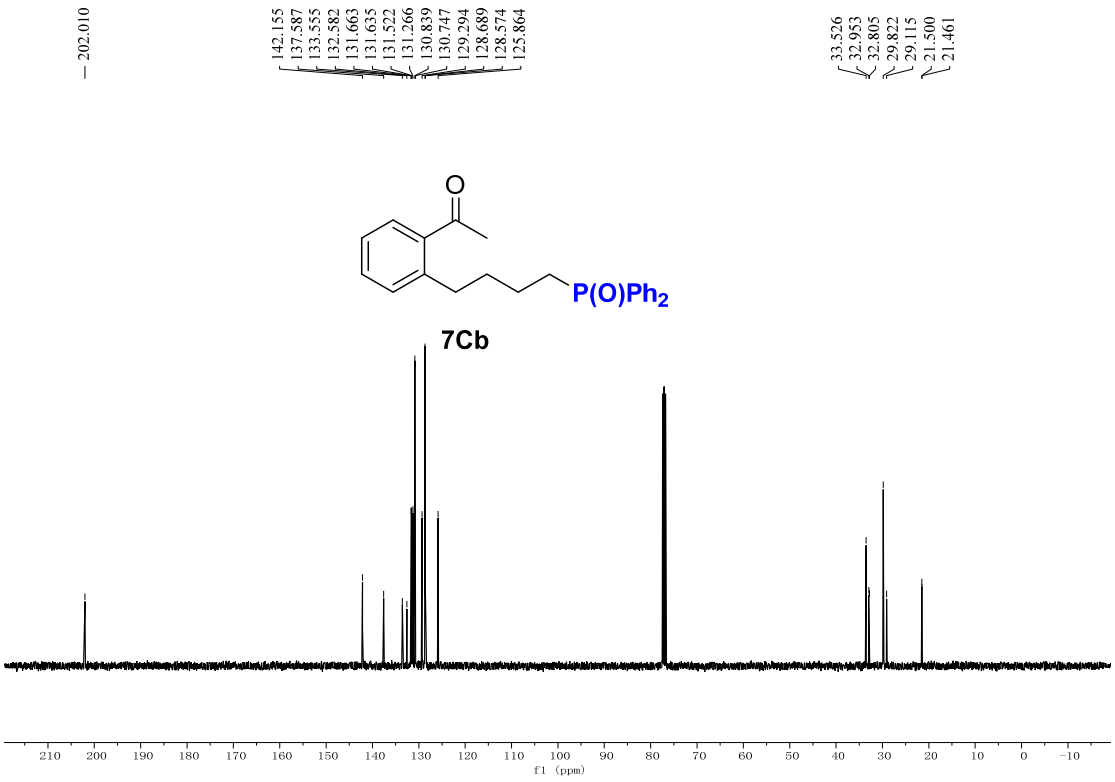
7Ba

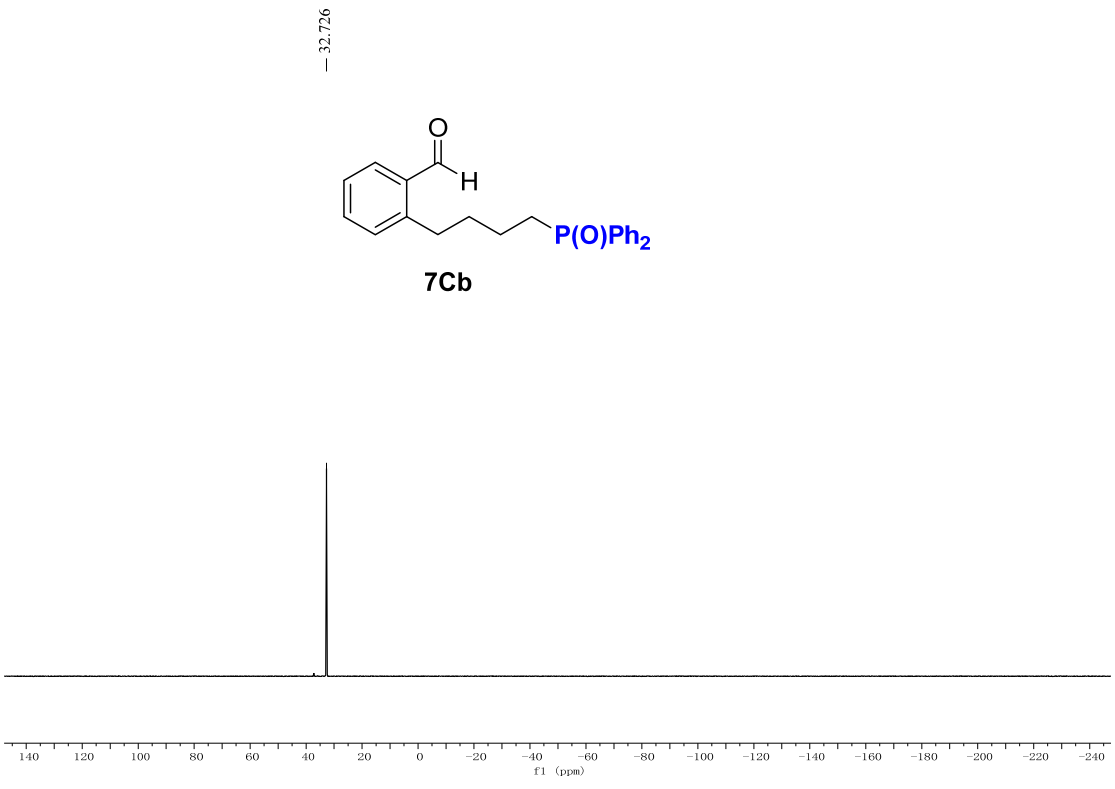


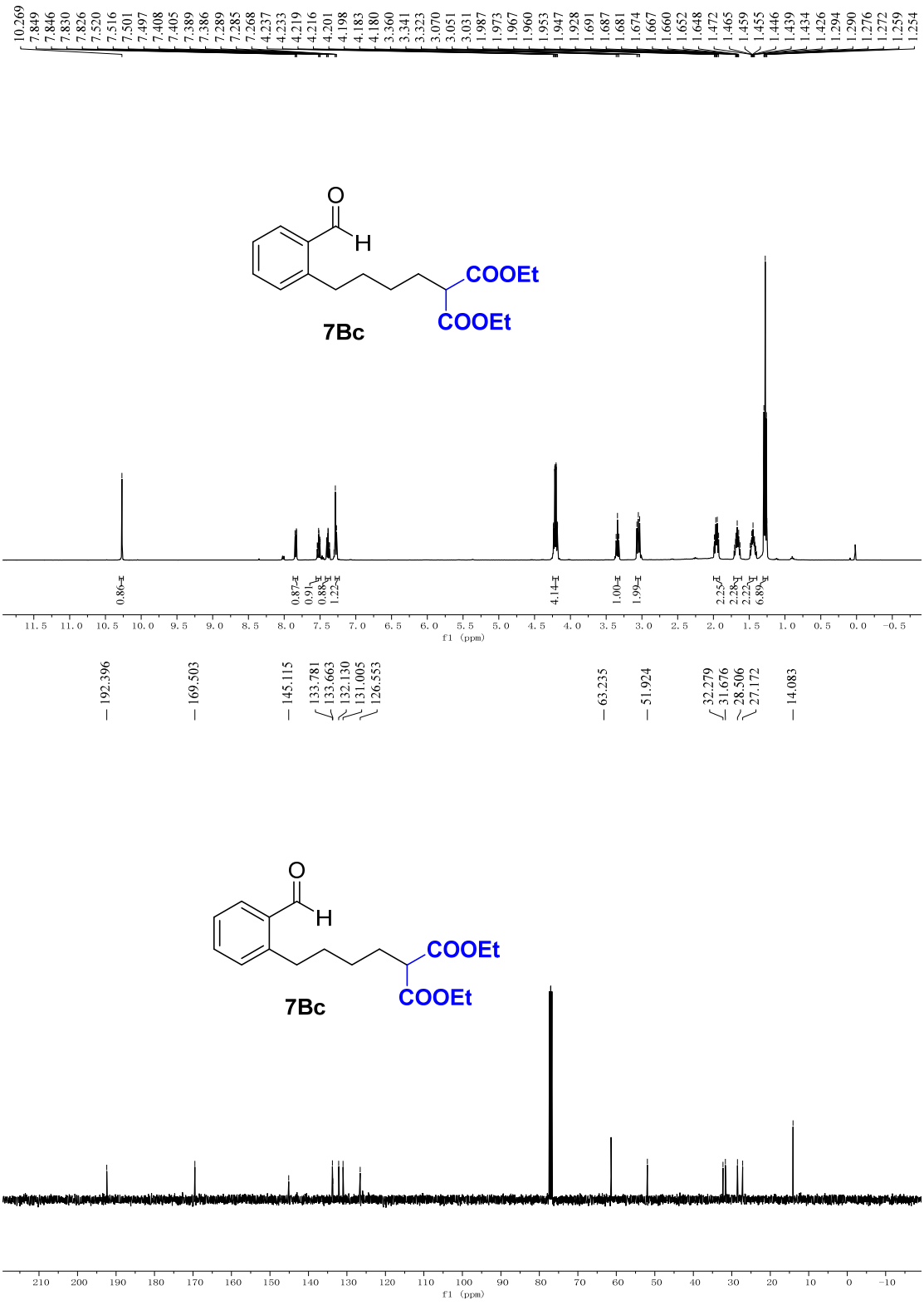

192.794
144.670
143.974
136.162
133.828
133.644
131.128
129.930
129.892
128.101
126.814
56.091
32.310
30.291
22.603
21.645
O
H
Ts
7Ba
f1 (ppm)

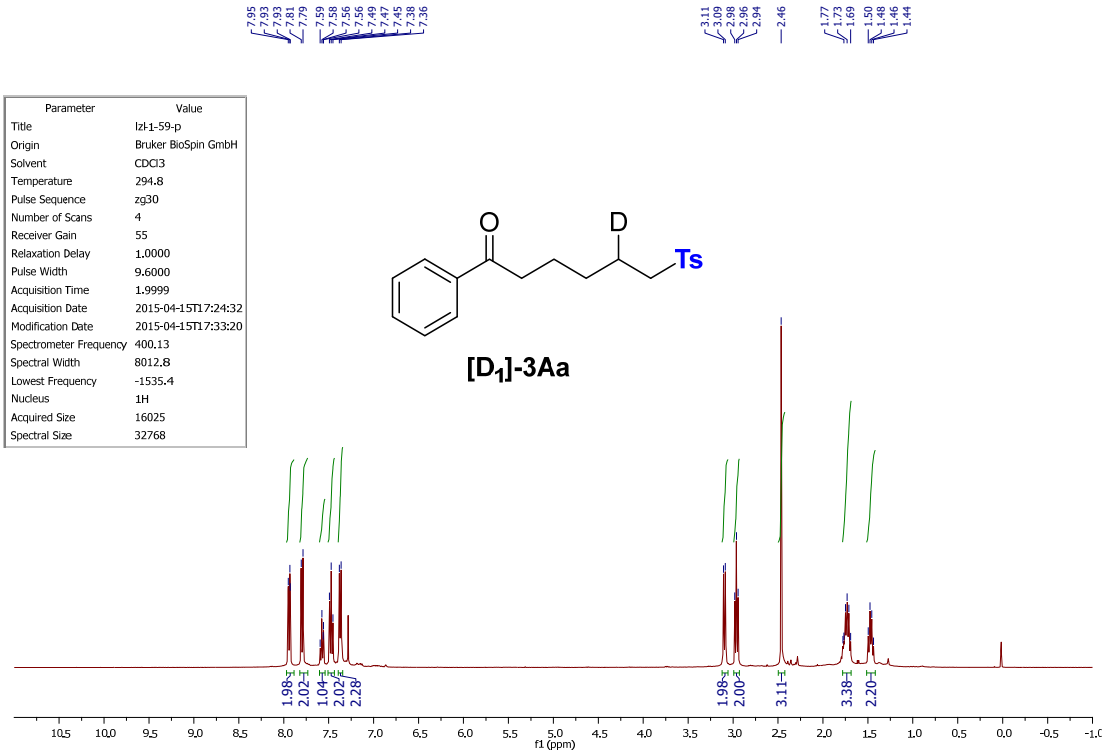
7.771
7.767
7.751
7.747
7.742
7.739
7.722
7.718
7.650
7.646
7.630
7.627
7.519
7.515
7.508
7.505
7.501
7.497
7.493
7.487
7.482
7.480
7.476
7.473
7.469
7.466
7.462
7.456
7.452
7.447
7.444
7.365
7.362
7.347
7.343
7.285
7.269
7.266
7.251
7.247
7.200
7.197
7.181
7.178
2.851
2.832
2.815
2.552
2.339
2.331
2.323
2.319
2.313
1.731
1.720
1.710
1.700
1.691
1.681

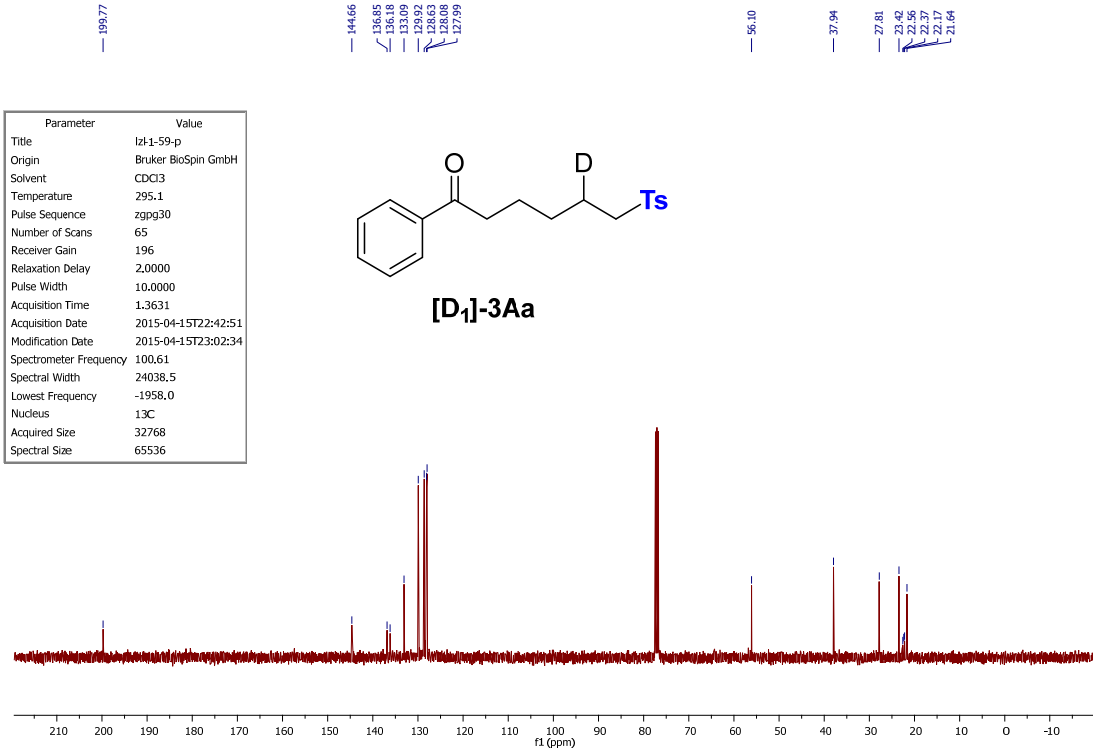

O
P(O)Ph2
7Cb
4.00
1.01
6.11
1.01
1.17
0.93
2.03
3.00
2.41
4.11
f1 (ppm)

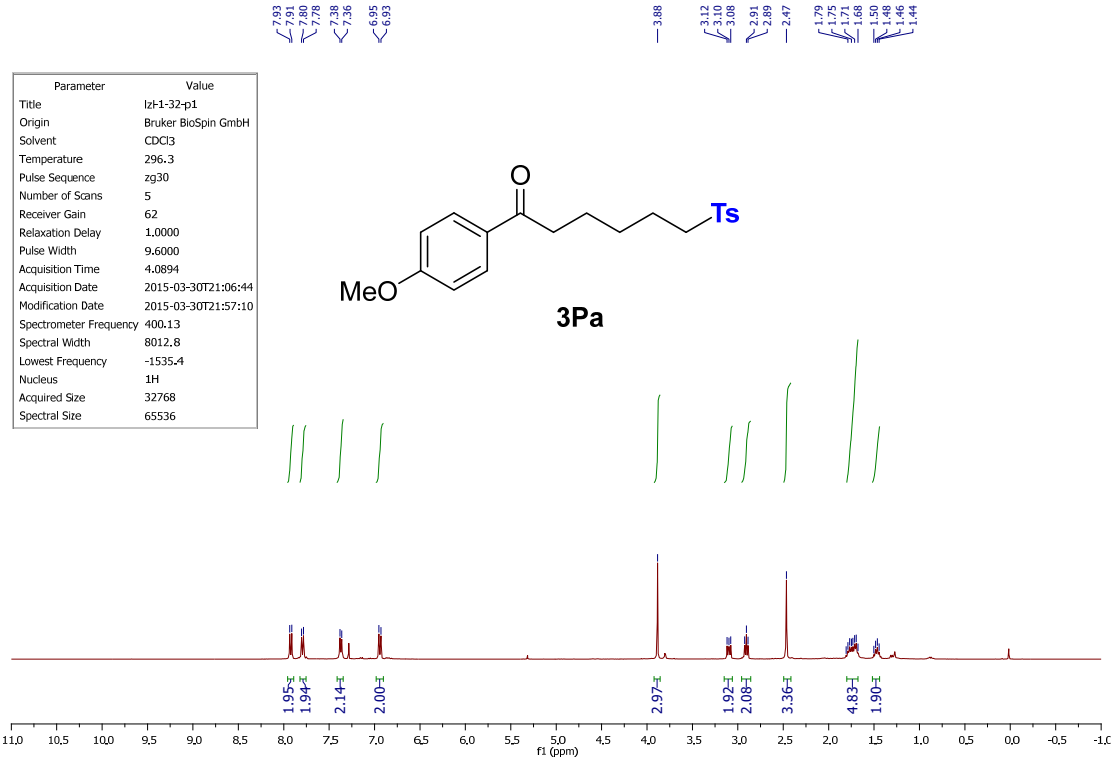

202.010
142.155
137.587
133.555
132.582
131.663
131.635
131.522
131.266
130.839
130.747
129.294
128.689
128.574
125.864
33.526
32.953
32.805
29.822
29.115
21.500
21.461
O
P(O)Ph2
7Cb
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10
f1 (ppm)

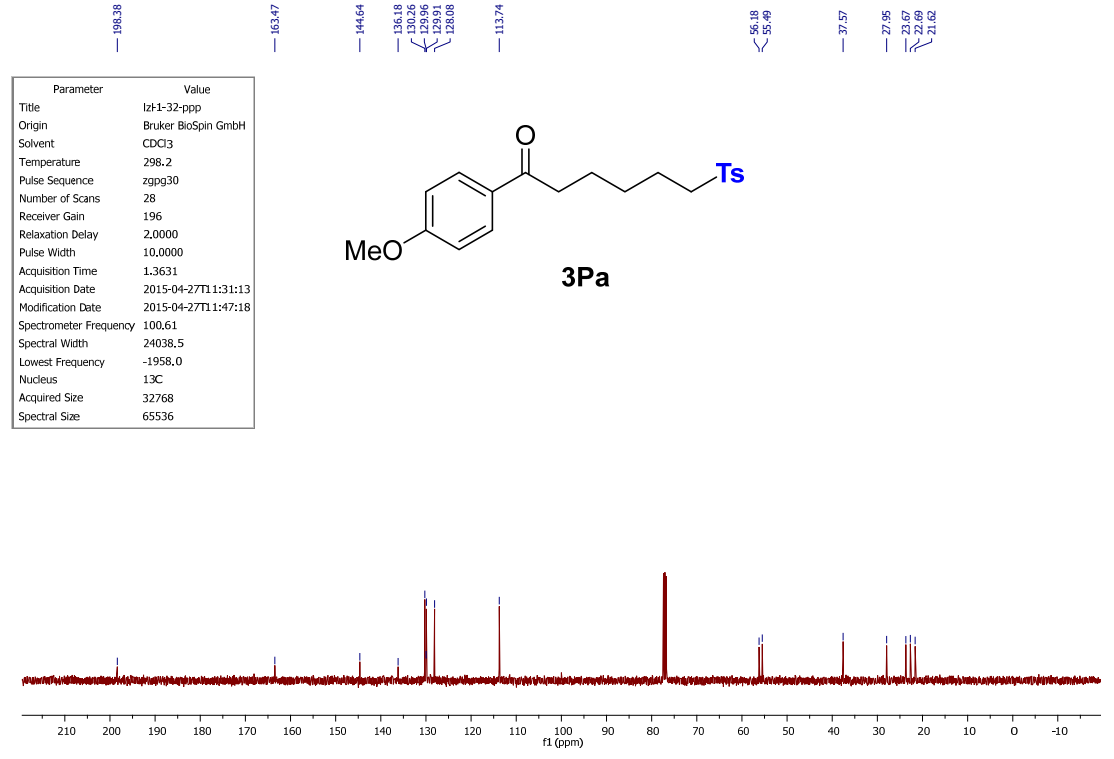

32.726
O
H
P(O)Ph2
7Cb
140 120 100 80 60 40 20 0 −20 −40 −60 −80 −100 −120 −140 −160 −180 −200 −220 −240
f1 (ppm)

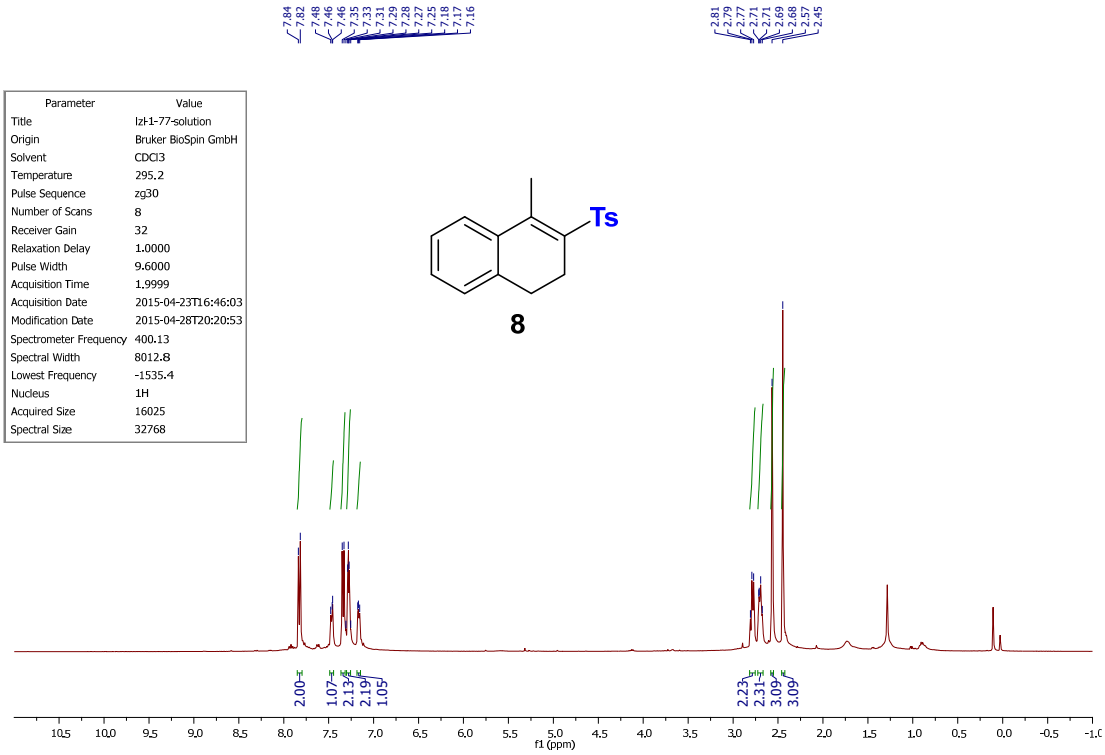

10.269
7.849
7.846
7.830
7.826
7.520
7.516
7.501
7.497
7.408
7.405
7.389
7.386
7.289
7.285
7.268
4.237
4.233
4.219
4.216
4.201
4.198
4.183
4.180
3.360
3.341
3.323
3.070
3.051
3.031
1.987
1.973
1.967
1.960
1.953
1.947
1.928
1.691
1.687
1.681
1.674
1.667
1.660
1.652
1.648
1.472
1.465
1.459
1.455
1.446
1.439
1.434
1.426
1.294
1.290
1.276
1.272
1.259
1.254
O
H
COOEt
COOEt
7Bc
0.86
0.87
0.91
0.88
1.22
4.14
1.00
1.99
2.25
2.28
2.22
6.89
f1 (ppm)
192.396
169.503
145.115
133.781
133.663
132.130
131.005
126.553
63.235
51.924
32.279
31.676
28.506
27.172
14.083
O
H
COOEt
COOEt
7Bc
f1 (ppm)

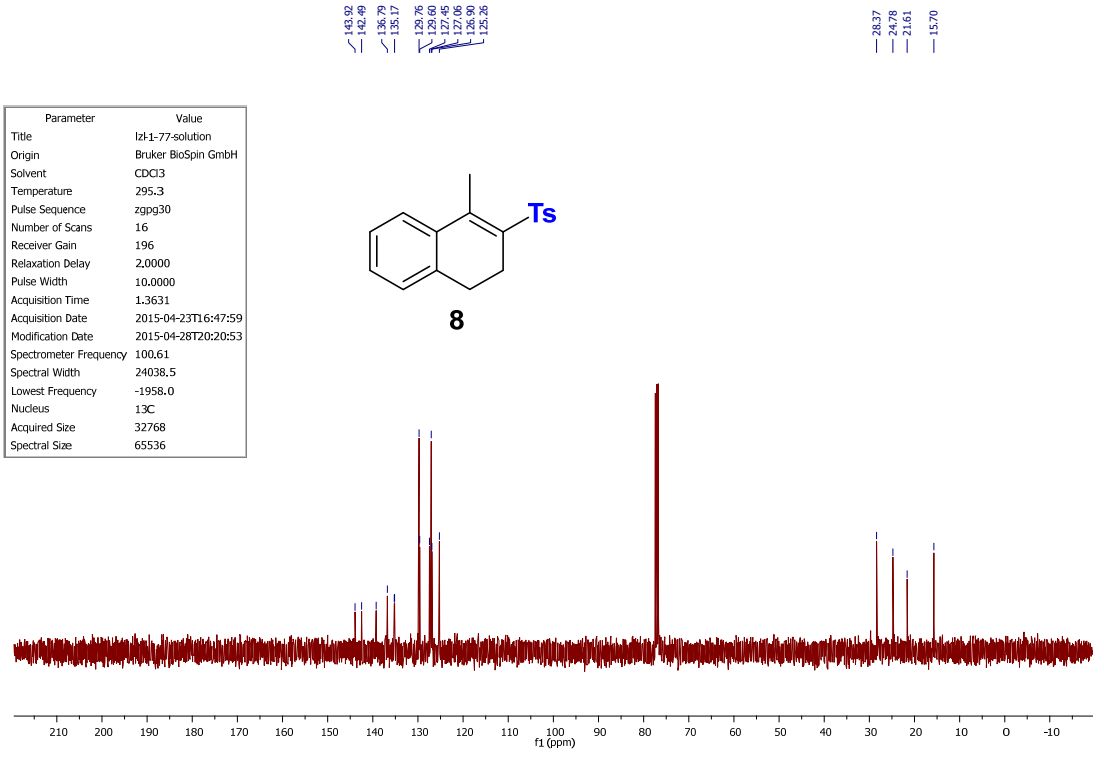

7.95
7.93
7.93
7.81
7.79
7.59
7.58
7.56
7.56
7.49
7.47
7.45
7.38
7.36
3.11
3.09
2.98
2.96
2.94
2.46
1.77
1.73
1.69
1.50
1.48
1.46
1.44
Parameter Value
Title lzl-1-59-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 294.8
Pulse Sequence zg30
Number of Scans 4
Receiver Gain 55
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-15T17:24:32
Modification Date 2015-04-15T17:33:20
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
O
D
Ts
[D1]-3Aa
1.98
2.02
1.04
2.02
2.28
1.98
2.00
3.11
3.38
2.20
f1 (ppm)


199.77
144.66
136.85
136.18
133.09
129.92
128.63
128.08
127.99
56.10
37.94
27.81
23.42
22.56
22.37
22.17
21.64
Parameter Value
Title lzl-1-59-p
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.1
Pulse Sequence zgpg30
Number of Scans 65
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-15T22:42:51
Modification Date 2015-04-15T23:02:34
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
D
Ts
[D1]-3Aa
f1 (ppm)


7.93
7.91
7.80
7.78
7.38
7.36
6.95
6.93
3.88
3.12
3.10
3.08
2.91
2.89
2.47
1.79
1.75
1.71
1.68
1.50
1.48
1.46
1.44
Parameter Value
Title lzl-1-32-p1
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 296.3
Pulse Sequence zg30
Number of Scans 5
Receiver Gain 62
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 4.0894
Acquisition Date 2015-03-30T21:06:44
Modification Date 2015-03-30T21:57:10
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 32768
Spectral Size 65536
O
Ts
MeO
3Pa
1.95
1.94
2.14
2.00
2.97
1.92
2.08
3.36
4.83
1.90
f1 (ppm)


198.38
163.47
144.64
136.18
130.26
129.96
129.91
128.08
113.74
56.18
55.49
37.57
27.95
23.67
22.69
21.62
Parameter Value
Title lzl-1-32-ppp
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.2
Pulse Sequence zgpg30
Number of Scans 28
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-27T11:31:13
Modification Date 2015-04-27T11:47:18
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
O
Ts
MeO
3Pa
f1 (ppm)


7.84
7.82
7.48
7.46
7.46
7.45
7.33
7.31
7.29
7.28
7.27
7.25
7.18
7.17
7.16
2.81
2.79
2.77
2.71
2.71
2.69
2.68
2.57
2.45
Parameter Value
Title lzl-1-77-solution
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.2
Pulse Sequence zg30
Number of Scans 8
Receiver Gain 32
Relaxation Delay 1.0000
Pulse Width 9.6000
Acquisition Time 1.9999
Acquisition Date 2015-04-23T16:46:03
Modification Date 2015-04-28T20:20:53
Spectrometer Frequency 400.13
Spectral Width 8012.8
Lowest Frequency -1535.4
Nucleus 1H
Acquired Size 16025
Spectral Size 32768
Ts
8
2.00
1.07
2.13
2.19
1.05
2.23
2.33
3.09
3.09
f1 (ppm)


143.92
142.49
136.79
135.17
129.76
129.60
127.45
127.06
126.90
125.26
28.37
24.78
21.61
15.70
Parameter Value
Title lzl-1-77-solution
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.3
Pulse Sequence zgpg30
Number of Scans 16
Receiver Gain 196
Relaxation Delay 2.0000
Pulse Width 10.0000
Acquisition Time 1.3631
Acquisition Date 2015-04-23T16:47:59
Modification Date 2015-04-28T20:20:53
Spectrometer Frequency 100.61
Spectral Width 24038.5
Lowest Frequency -1958.0
Nucleus 13C
Acquired Size 32768
Spectral Size 65536
Ts
8
f1 (ppm)