

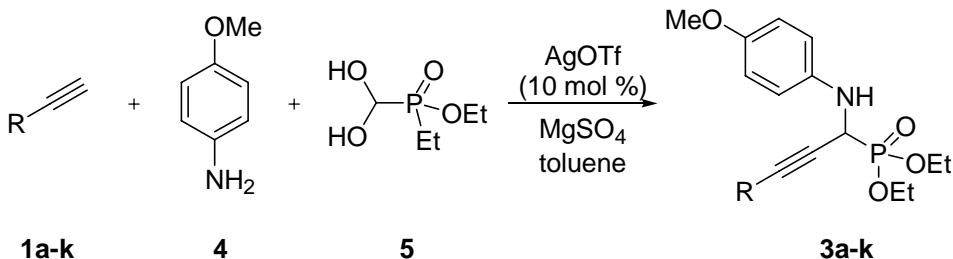
Silver(I) Triflate-Catalyzed Direct Synthesis of *N*-PMP Protected α -Aminopropargylphosphonates from Terminal Alkynes

Rajasekhar Dodda and Cong-Gui Zhao*

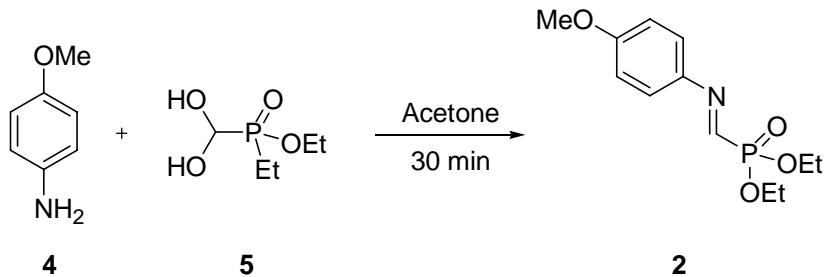
Department of Chemistry, University of Texas at San Antonio,
One UTSA Circle, San Antonio, Texas 78249-0698

Supporting Information

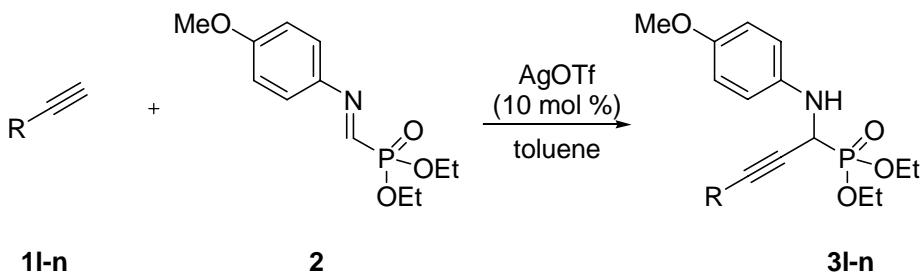
General Experimental Procedure for the One-Pot Synthesis of Compounds 3a-k: A solution of diethyl α -formylphosphonate hydrate (**5**, 92 mg, 0.5 mmol) and *p*-anisidine (**4**, 61.5 mg, 0.5 mmol) in anhydrous toluene (6.0 mL) was stirred at room temperature for 15 min. Afterwards anhydrous MgSO₄ (300 mg, 2.5 mmol) was added and the mixture was further stirred for 10 min. To the resulting mixture, AgOTf (12.8 mg, 0.05 mmol, 10 mol %) and the alkyne (**1a-k**, 0.75 mmol) were added sequentially while vigorous stirring. The mixture was further stirred for 6 h at room temperature. After removing the solvent, the crude product was directly subjected to flash chromatography (silica gel, 6:4 hexane/EtOAc) to give diethyl α -aminopropargylphosphonate (**3a-k**) as a pure compound, which has been characterized by ¹H and ¹³C NMR spectroscopy.



Preparation of diethyl (4-methoxyphenylimino)methylphosphonate (2**):** A mixture of diethyl α -formylphosphonate hydrate (**5**, 184 mg, 1.0 mmol) and *p*-anisidine (**4**, 123 mg, 1.0 mmol) in acetone (2 mL) was stirred at room temperature for 30 min. Afterwards the solvent was removed under reduced pressure at room temperature to give **2** as a colorless oil (268 mg, 99%).

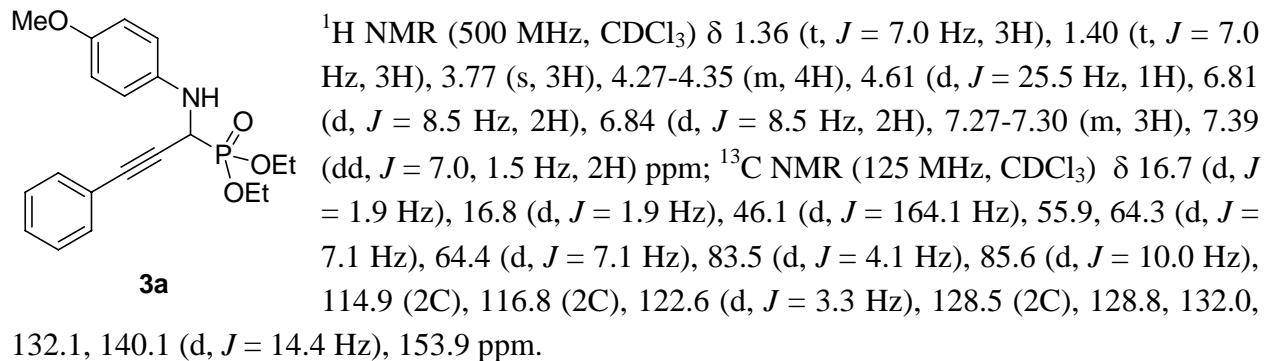


General Experimental Procedure for the Synthesis of Compounds **3l-n** : To a vigorously stirred mixture of the alkyne (**1l-n**, 0.75 mmol) and AgOTf (12.8 mg, 0.05 mmol, 10 mol % with respect to imine) in anhydrous toluene (3.0 mL) was added dropwise a solution of **2** (135.5 mg, 0.5 mmol) in toluene (3.0 mL) over a period of 1 h at room temperature. Upon the completion of the addition, the reaction mixture was further stirred for 24 h. After removing the solvent under reduced pressure, the crude product was purified by flash chromatography (silica gel, 6/4 hexane/EtOAc) to give the diethyl α -aminopropargylphosphonate (**3l-n**) as a pure compound, which has been characterized by ^1H and ^{13}C NMR spectroscopy.

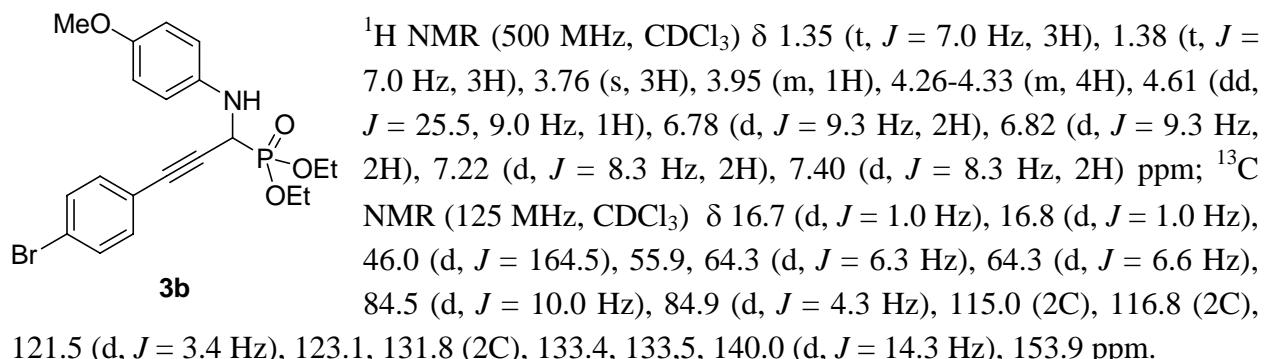


^1H and ^{13}C NMR data of compounds **4a-n** and **5**.

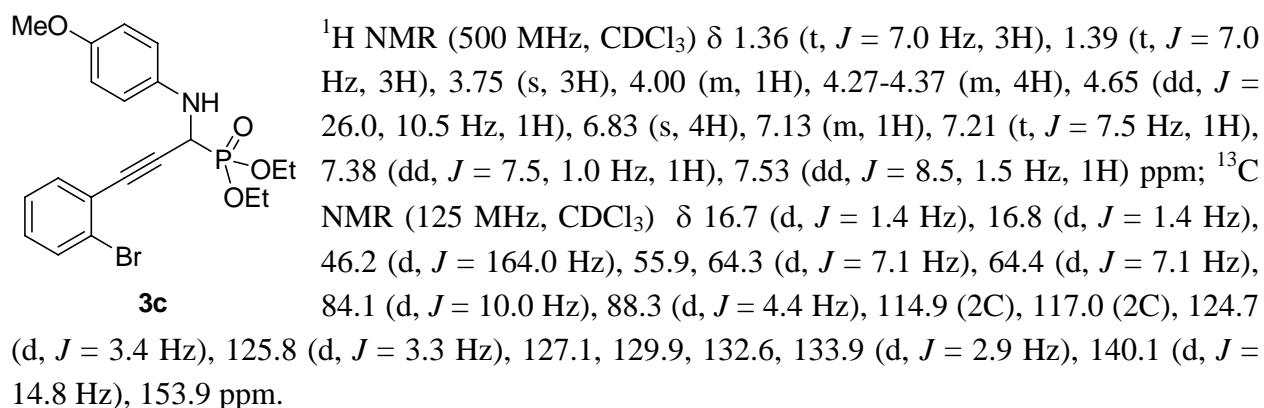
Diethyl 1-(4-methoxyphenylamino)-3-phenylprop-2-ynylphosphonate (**3a**)



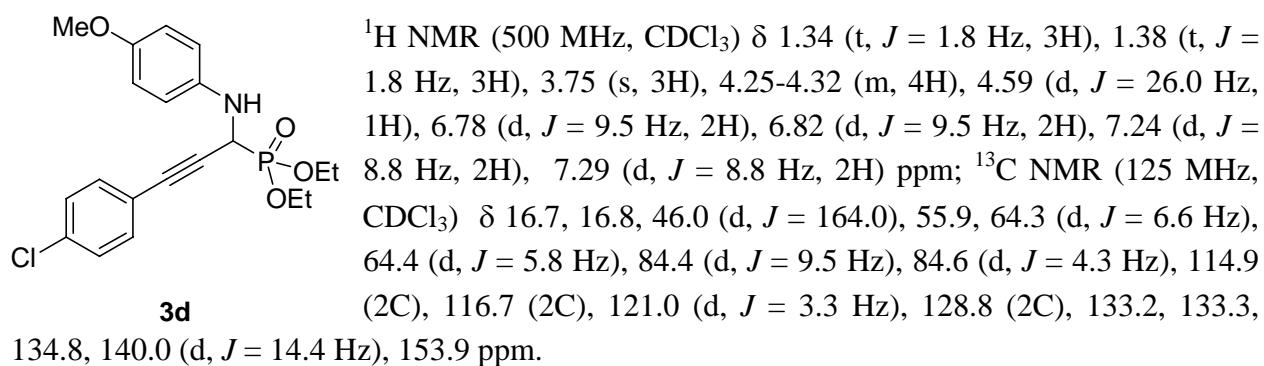
Diethyl 3-(4-bromophenyl)-1-(4-methoxyphenylamino)prop-2-ynylphosphonate (3b)



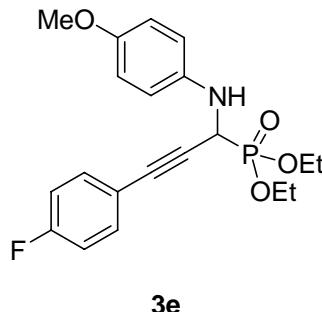
Diethyl 3-(2-bromophenyl)-1-(4-methoxyphenylamino)prop-2-ynylphosphonate (3c)



Diethyl 3-(4-chlorophenyl)-1-(4-methoxyphenylamino)prop-2-ynylphosphonate (3d)

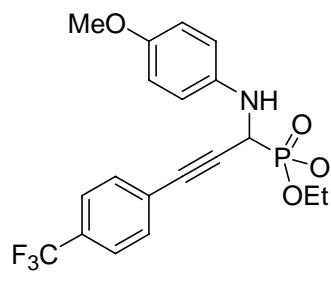


Diethyl 3-(4-fluorophenyl)-1-(4-methoxyphenylamino)prop-2-ynylphosphonate (3e)



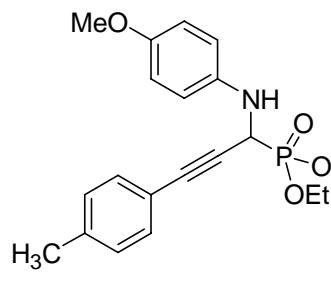
¹H NMR (500 MHz, CDCl₃) δ 1.35 (t, *J* = 7.0 Hz, 3H), 1.38 (t, *J* = 7.0 Hz, 3H), 3.76 (s, 3H), 3.96 (m, 1H), 4.24-4.34 (m, 4H), 4.56 (dd, *J* = 25.5, 10.5 Hz, 1H), 6.78 (d, *J* = 9.5 Hz, 2H), 6.83 (d, *J* = 9.5 Hz, 2H), 6.96 (t, *J* = 8.5 Hz, 2H), 7.35 (m, 2H) ppm; ¹³C NMR (125 MHz, CDCl₃) δ 16.7 (d, *J* = 0.9 Hz), 16.8, 46.0 (d, *J* = 164.1), 55.9, 64.3 (d, *J* = 7.1 Hz), 64.4 (d, *J* = 7.1 Hz), 83.3 (d, 5.7 Hz), 84.5 (d, *J* = 10.0 Hz), 114.9 (2C), 115.7, 115.9, 116.8 (2C), 118.6, 133.9 (d, *J* = 2.9 Hz), 134.0 (d, *J* = 2.9 Hz), 140.0 (d, *J* = 14.9 Hz), 153.9, 162.8 (d, *J* = 248.9 Hz) ppm.

Diethyl 1-(4-methoxyphenylamino)-3-(4-trifluoromethylphenyl)prop-2-ynylphosphonate (3f)



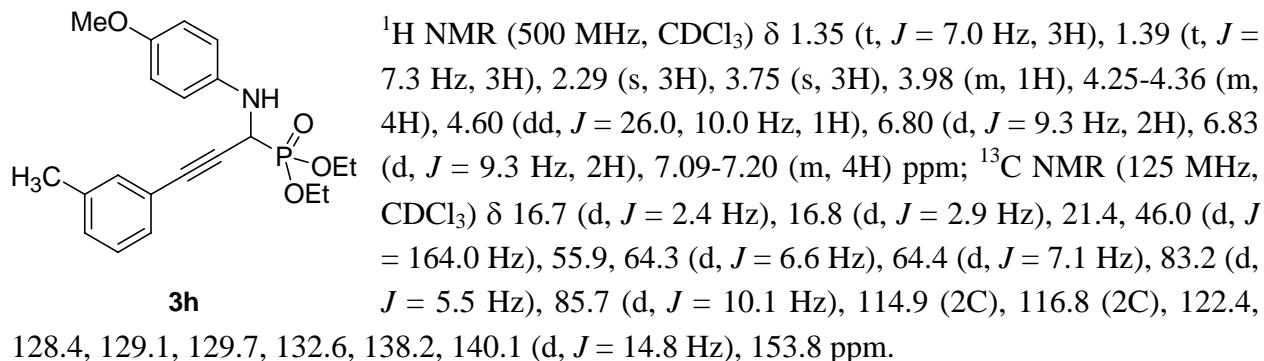
¹H NMR (500 MHz, CDCl₃) δ 1.36 (t, *J* = 6.3 Hz, 3H), 1.38 (t, *J* = 6.5 Hz, 3H), 3.76 (s, 3H), 4.26-4.34 (m, 4H), 4.64 (d, *J* = 26.0 Hz, 1H), 6.81 (d, *J* = 9.3 Hz, 2H), 6.83 (d, *J* = 9.3 Hz, 2H), 7.39 (m, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 7.5 Hz, 1H) ppm; ¹³C NMR (125 MHz, CDCl₃) δ 16.6, 16.7, 46.2 (d, *J* = 164.1 Hz), 55.9, 64.2 (d, *J* = 7.3 Hz), 64.4 (d, *J* = 6.8 Hz), 81.5 (d, *J* = 10.5 Hz), 89.5, 114.9 (2C), 116.9 (2C), 122.6 (q, *J* = 225.6 Hz), 126.0 (d, *J* = 5.3 Hz, 2C), 128.5, 131.6, 134.6 (d, *J* = 3.3 Hz, 2C), 140.0 (d, *J* = 14.4 Hz), 154.0 ppm.

Diethyl 1-(4-methoxyphenylamino)-3-(4-methylphenyl)prop-2-ynylphosphonate (3g)

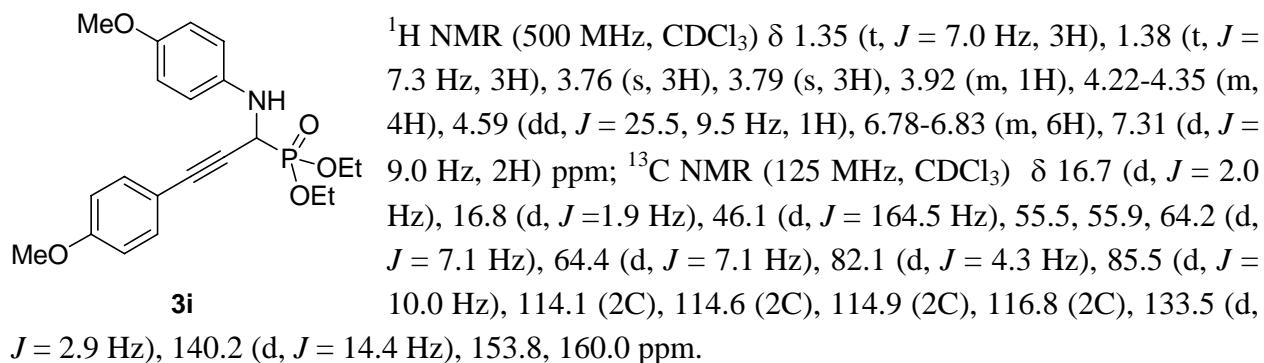


¹H NMR (500 MHz, CDCl₃) δ 1.35 (t, *J* = 7.0 Hz, 3H), 1.40 (t, *J* = 6.5 Hz, 3H), 2.32 (s, 3H), 3.76 (s, 3H), 3.96 (m, 1H), 4.25-4.35 (m, 4H), 4.59 (dd, *J* = 25.0, 10.0 Hz, 1H), 6.79 (d, *J* = 9.3 Hz, 2H), 6.83 (d, *J* = 9.3 Hz, 2H), 7.08 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8.5 Hz, 2H) ppm; ¹³C NMR (125 MHz, CDCl₃) δ 16.7 (d, *J* = 2.0 Hz), 16.8 (d, *J* = 1.9 Hz), 21.7, 46.1 (d, *J* = 164.5 Hz), 55.9, 64.3 (d, *J* = 7.1 Hz), 64.4 (d, *J* = 6.6 Hz), 82.8 (d, *J* = 4.3 Hz), 85.7 (d, *J* = 10.1 Hz), 114.9 (2C), 116.8(2C), 119.5 (d, *J* = 3.3 Hz), 129.2 (2C), 131.9, 132.0, 138.9, 140.2 (d, *J* = 14.3 Hz), 153.8 ppm.

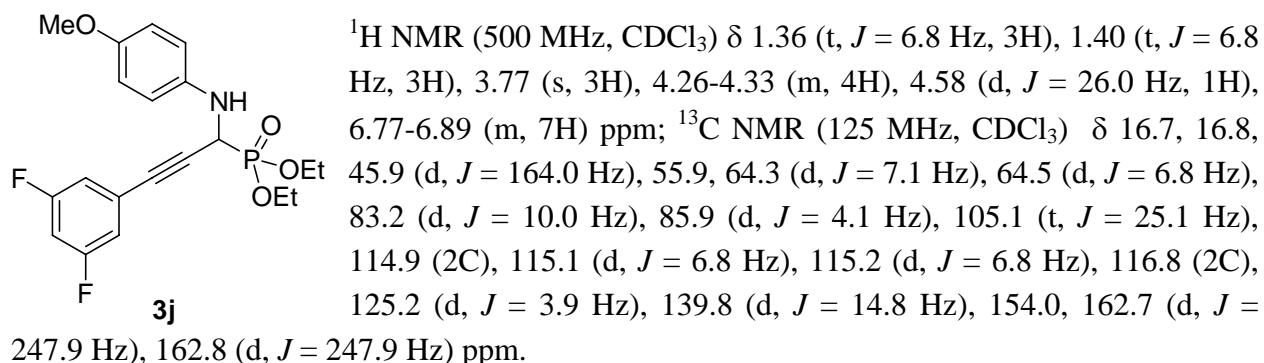
Diethyl 1-(4-methoxyphenylamino)-3-(3-methylphenyl)prop-2-ynylphosphonate (3h):



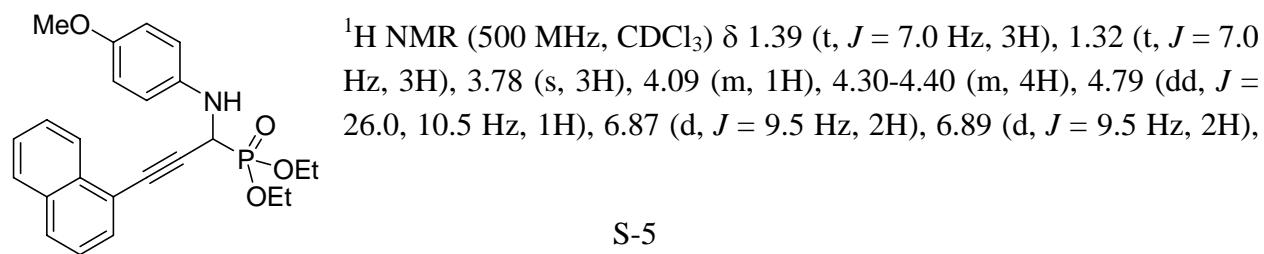
Diethyl 3-(4-methoxyphenyl)-1-(4-methoxyphenylamino)prop-2-ynylphosphonate (3i)



Diethyl 3-(3,5-difluorophenyl)-1-(4-methoxyphenylamino)prop-2-ynylphosphonate (3j)

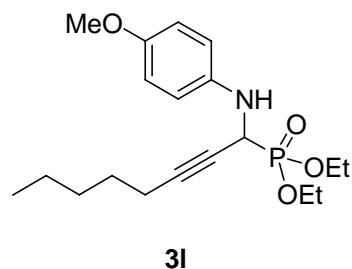


Diethyl 1-(4-methoxyphenylamino)-3-(naphth-1-yl)prop-2-ynylphosphonate (3k)



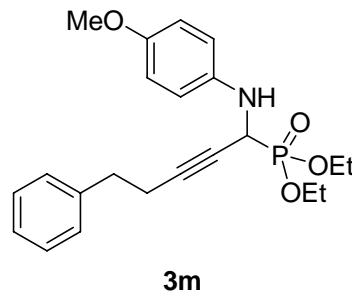
7.38 (t, $J = 8.5$ Hz, 1H), 7.46-7.50 (m, 2H), 7.61 (d, $J = 7.0$ Hz, 1H), 7.79-7.82 (m, 2H), 8.13 (m, 1H) ppm; ^{13}C NMR (125 MHz, CDCl_3) δ 16.8, 16.9, 46.5 (d, $J = 164.0$ Hz), 55.9, 64.3 (d, $J = 6.8$ Hz), 64.4 (d, $J = 6.7$ Hz), 83.9 (d, $J = 10.1$ Hz), 88.5 (d, $J = 4.4$ Hz), 115.0 (2C), 117.1 (2C), 120.2, 125.3, 126.4, 126.7, 127.1, 128.4, 129.3, 130.8, 133.3, 133.7, 140.2 (d, $J = 14.3$ Hz), 154.0 ppm.

Diethyl 1-(4-methoxyphenylamino)-oct-2-yanylphosphonate (3l)



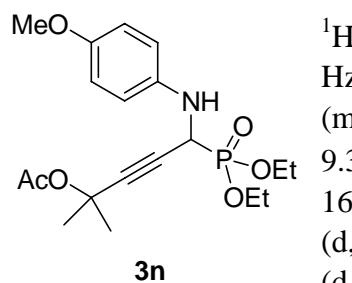
^1H NMR (500 MHz, CDCl_3) δ 0.86 (t, $J = 6.8$ Hz, 3H), 1.25-1.31 (m, 4H), 1.33 (t, $J = 6.5$ Hz, 3H), 1.33 (t, $J = 7.5$ Hz, 3H), 1.42-1.46 (m, 2H), 2.16-2.17 (m, 2H), 3.76 (s, 3H), 4.21-4.29 (m, 4H), 4.36 (d, $J = 25.0$ Hz, 1H), 6.75 (d, $J = 9.0$ Hz, 2H), 6.81 (d, $J = 9.0$ Hz, 2H) ppm; ^{13}C NMR (125 MHz, CDCl_3) δ 14.2, 16.7 (d, $J = 1.9$ Hz), 16.8 (d, $J = 1.9$ Hz), 19.0 (d, $J = 2.9$ Hz), 22.4, 28.3 (d, $J = 2.8$ Hz), 31.1, 45.5 (d, $J = 165.0$ Hz), 55.9, 64.0 (d, $J = 6.6$ Hz), 64.1 (d, $J = 7.1$ Hz), 74.1 (d, $J = 4.3$ Hz), 86.7 (d, $J = 10.0$ Hz), 114.8 (2C), 116.7 (2C), 140.2 (d, $J = 14.4$ Hz), 153.7 ppm.

Diethyl 1-(4-methoxyphenylamino)-5-phenylpent-2-yanylphosphonate (3m)



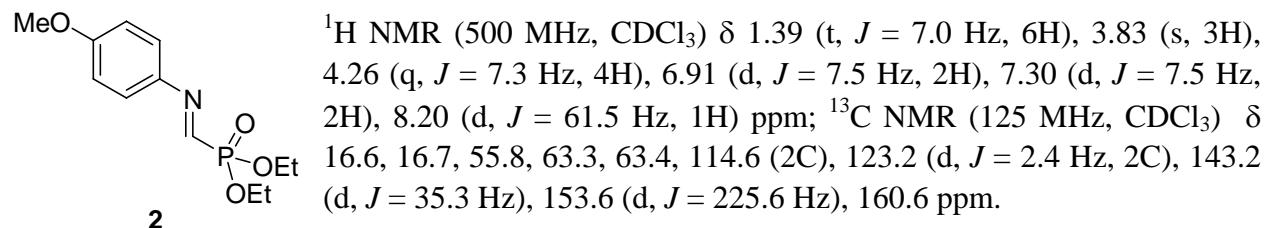
^1H NMR (500 MHz, CDCl_3) δ 1.31 (t, $J = 7.0$ Hz, 3H), 1.33 (t, $J = 7.0$ Hz, 3H), 2.46-2.50 (m, 2H), 2.77 (t, $J = 7.5$ Hz, 2H), 3.77 (s, 3H), 3.81 (m, 1H), 4.15-4.25 (m, 4H), 4.34 (dd, $J = 25.5, 10.5$ Hz, 1H), 6.71 (d, $J = 8.8$ Hz, 2H), 6.81 (d, $J = 8.8$ Hz, 2H), 7.14-7.27 (m, 5H) ppm; ^{13}C NMR (125 MHz, CDCl_3) δ 16.7, 16.8, 21.3 (d, $J = 2.8$ Hz), 34.8 (d, $J = 2.9$ Hz), 45.4 (d, $J = 165.0$ Hz), 55.9, 64.1 (d, $J = 6.8$ Hz), 64.2 (d, $J = 6.6$ Hz), 75.0 (d, $J = 3.9$ Hz), 85.6 (d, $J = 10.0$ Hz), 114.9 (2C), 116.7 (2C), 126.5, 128.6 (2C), 128.7 (2C), 140.2 (d, $J = 14.3$ Hz), 140.6, 153.7 ppm.

Diethyl 4-acetoxy- 1-(4-methoxyphenylamino)-4-methylpent-2-yanylphosphonate (3n)



^1H NMR (500 MHz, CDCl_3) δ 1.30 (t, $J = 7.0$ Hz, 3H), 1.36 (t, $J = 7.0$ Hz, 3H), 1.57 (s, 6H), 1.94 (s, 3H), 3.74 (s, 3H), 3.81 (m, 1H), 4.19-4.31 (m, 4H), 4.38 (d, $J = 25.0$ Hz, 1H), 6.73 (d, $J = 9.3$ Hz, 2H), 6.78 (d, $J = 9.3$ Hz, 2H) ppm; ^{13}C NMR (125 MHz, CDCl_3) δ 16.6 (d, $J = 2.4$ Hz), 16.7 (d, $J = 2.4$ Hz), 22.1, 28.9 (d, $J = 2.0$ Hz), 29.0 (d, $J = 1.5$ Hz), 45.6 (d, $J = 163.5$ Hz), 55.9, 64.2 (d, $J = 6.6$ Hz), 64.4 (d, $J = 6.8$ Hz), 72.0 (d, $J = 2.4$ Hz), 78.4 (d, $J = 3.9$ Hz), 86.9 (d, $J = 10.5$ Hz), 114.7 (2C), 117.0 (2C), 140.1 (d, $J = 13.8$ Hz), 153.8, 169.3 ppm.

Diethyl [(4-methoxyphenylimino)methyl]phosphonate (2)



VI-DR-PMPImine-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-PMPImine-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

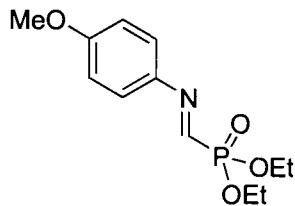
8 repetitions

OBSERVE H1, 499.6847200 MHz

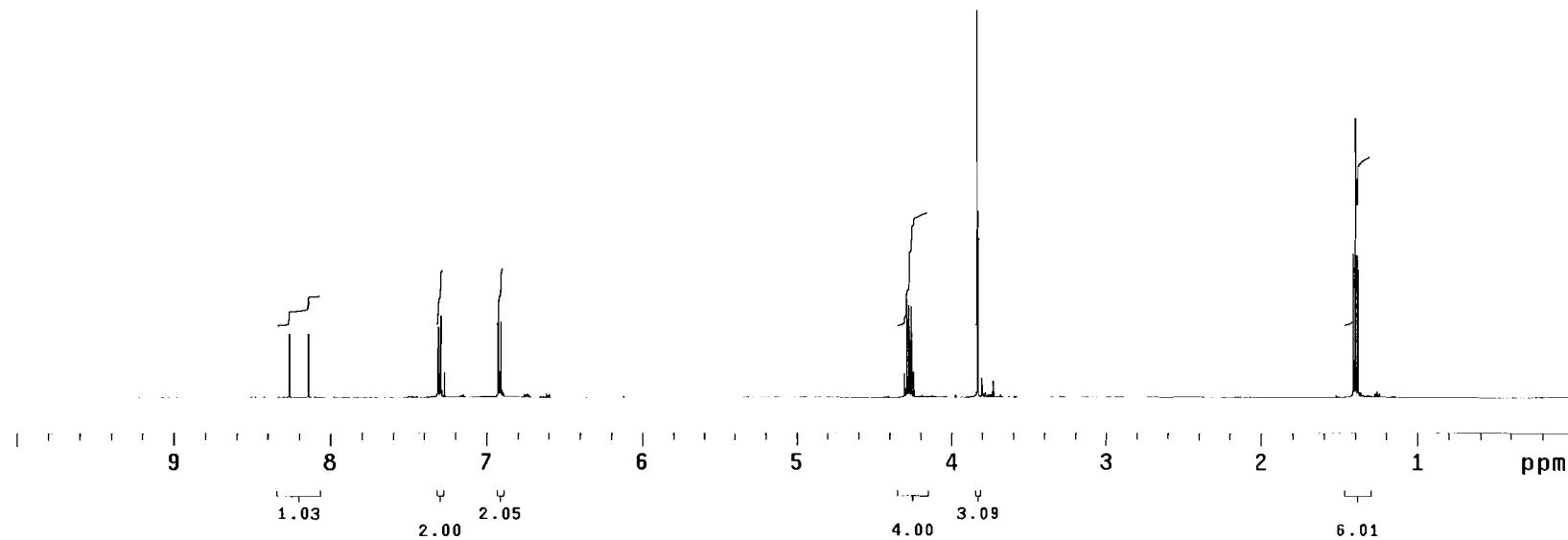
DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



2



VI-DR-PMP-Imine-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature

User: 1-14-87

File: VI-DR-Imine-13C
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees

Acq. time 1.300 sec
Width 31409.5 Hz

1700 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

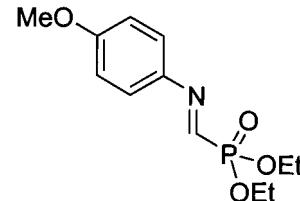
WALTZ-16 modulated

DATA PROCESSING

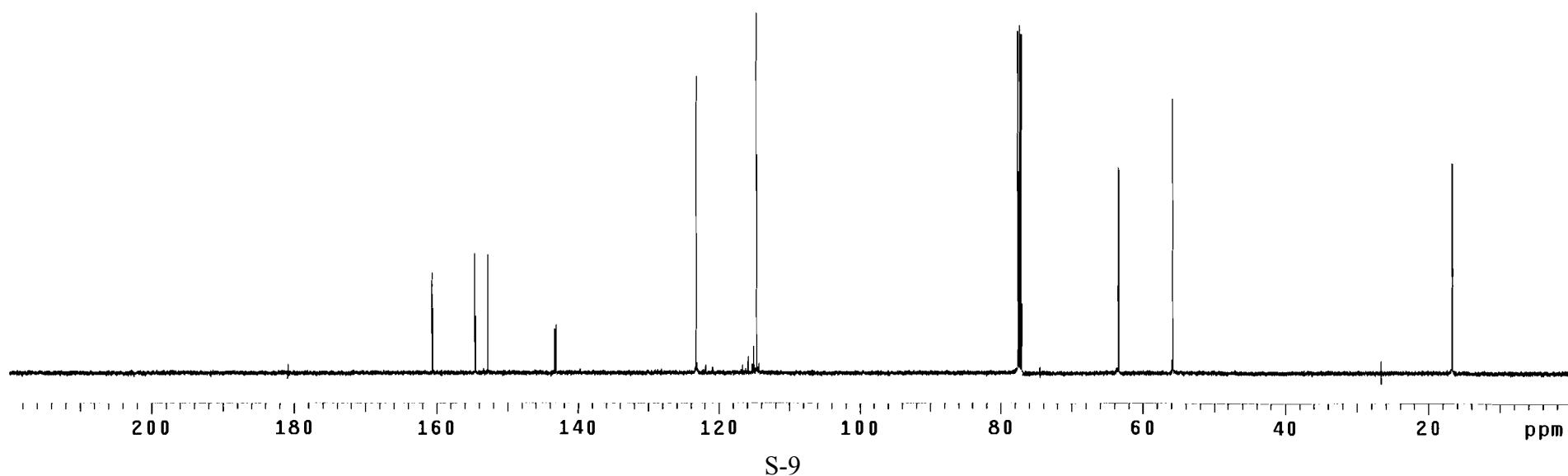
Line broadening 0.5 Hz

FT size 131072

Total time 1 hr, 5 min, 25 sec



2



S-9

VI-DR-11-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-11-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

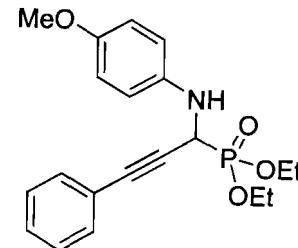
8 repetitions

OBSERVE H1, 499.6847200 MHz

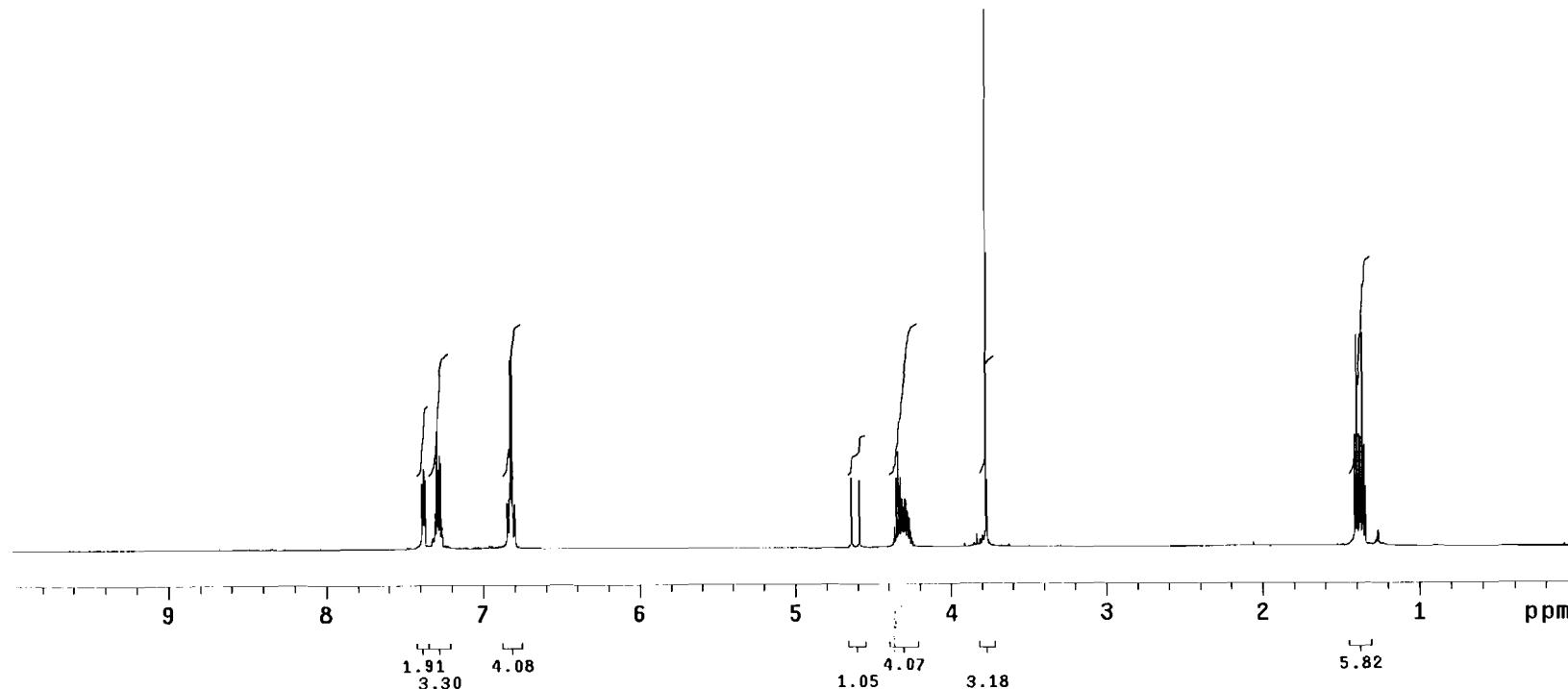
DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



3a



VI-DR-11-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature

User: 1-14-87

File: VI-DR-11-13C
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees

Acq. time 1.300 sec
Width 31409.5 Hz

20000 repetitions
OBSERVE C13, 125.6457928 MHz

DECUPLE H1, 499.6872185 MHz

Power 36 dB
continuously on

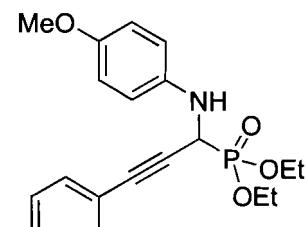
WALTZ-16 modulated

DATA PROCESSING

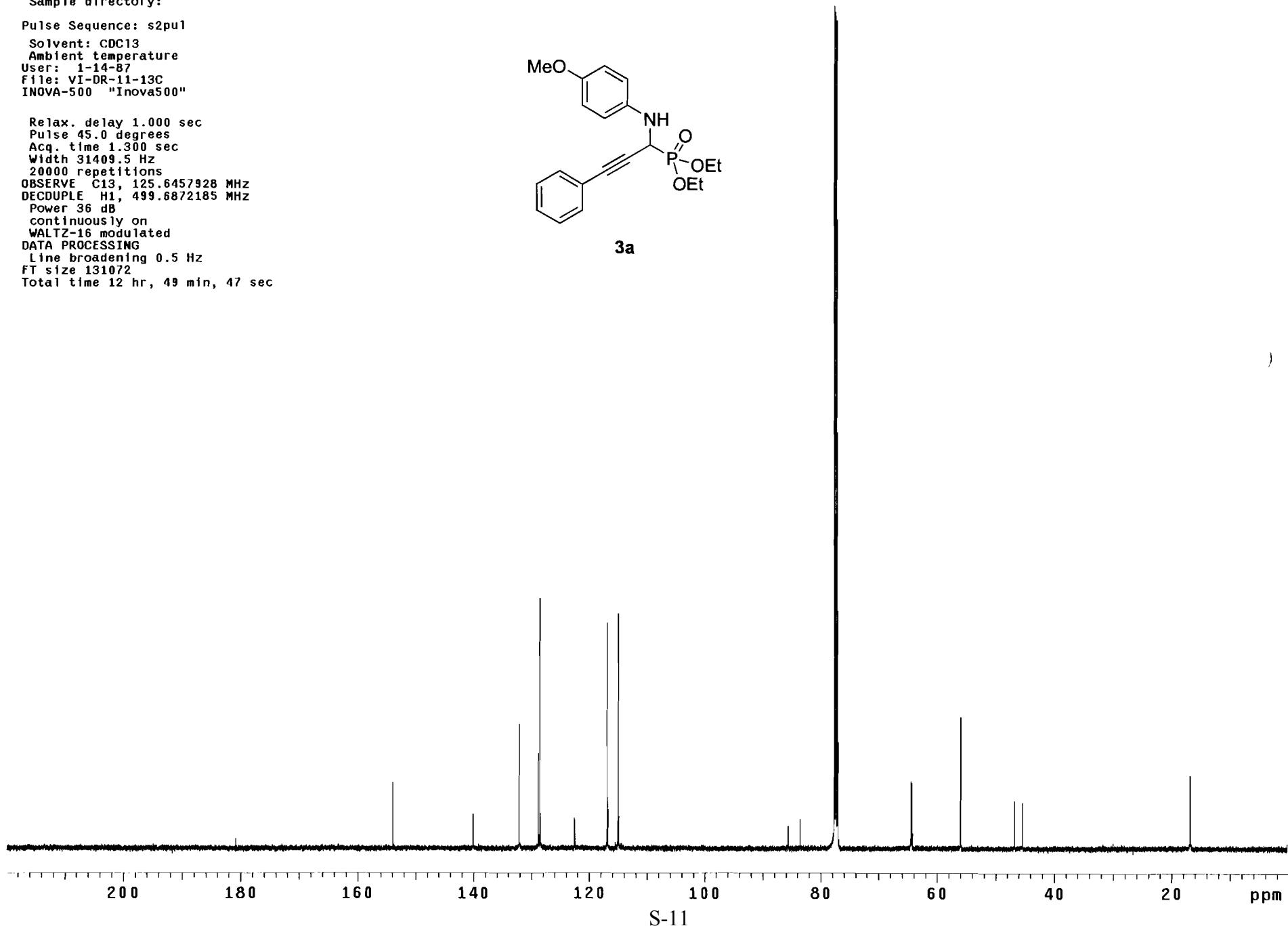
Line broadening 0.5 Hz

FT size 131072

Total time 12 hr, 49 min, 47 sec



3a



VI-DR-38-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-38-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

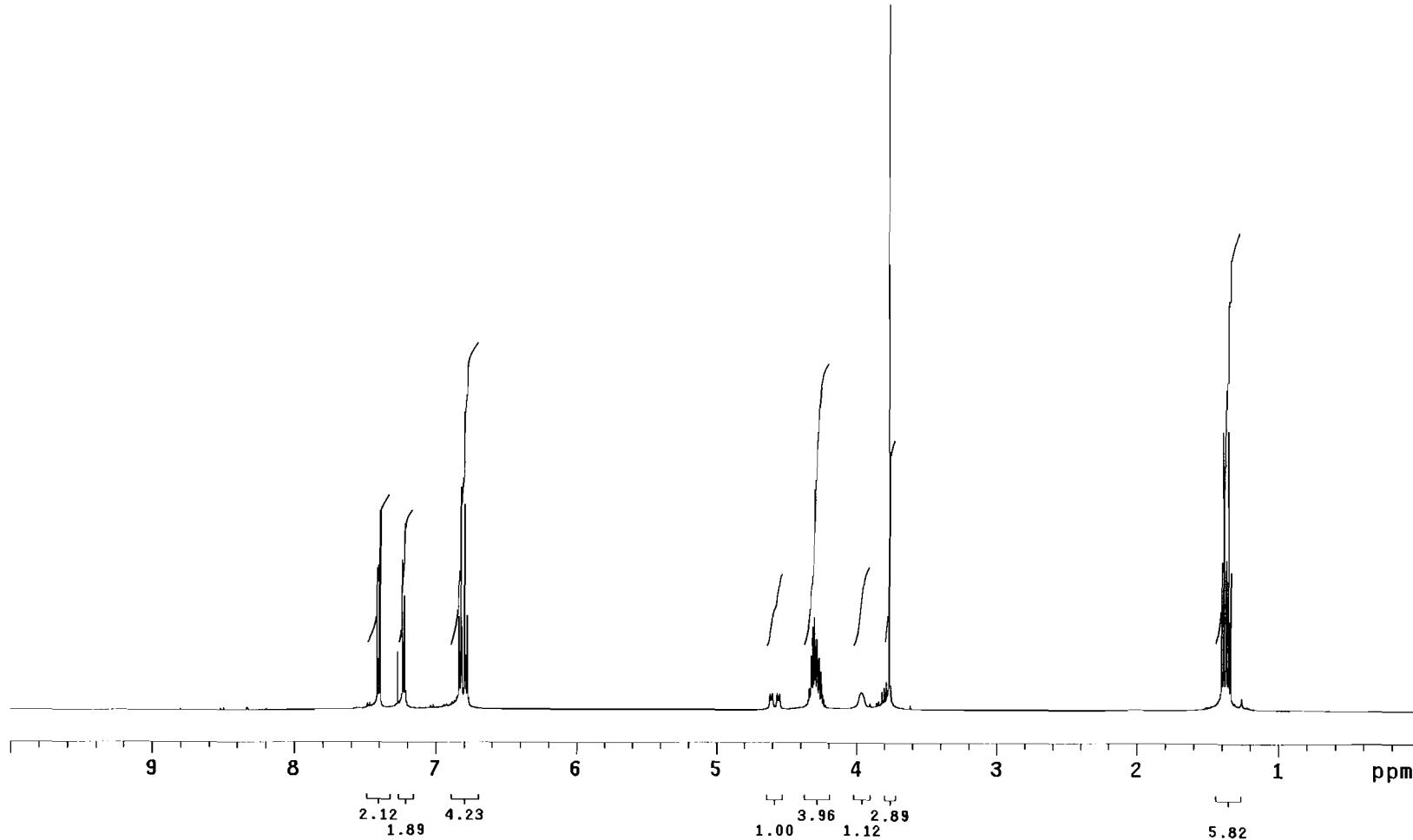
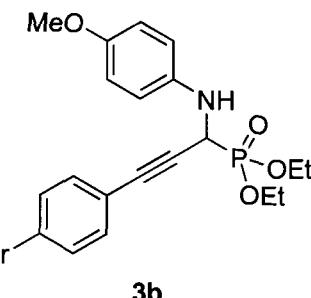
8 repetitions

OBSERVE H1, 499.6847200 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



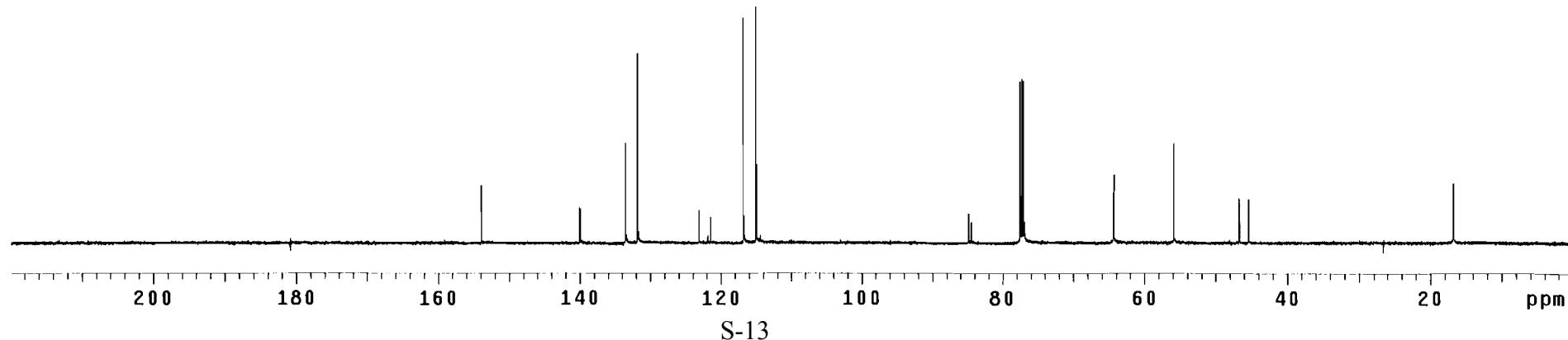
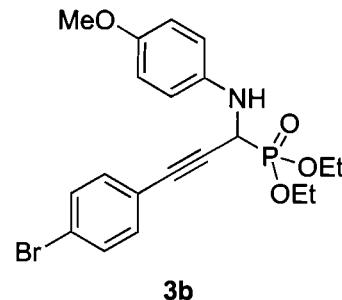
VI-DR-38-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
User: 1-14-87
File: VI-DR-38-13C
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 31409.5 Hz
1792 repetitions
OBSERVE C13, 125.6457928 MHz
DECOUPLE H1, 499.6872185 MHz
Power 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 2 hr, 33 min, 57 sec



VI-DR-62-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-62-proton

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

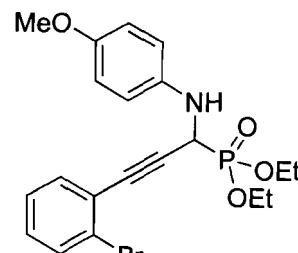
8 repetitions

OBSERVE H1, 499.6847200 MHz

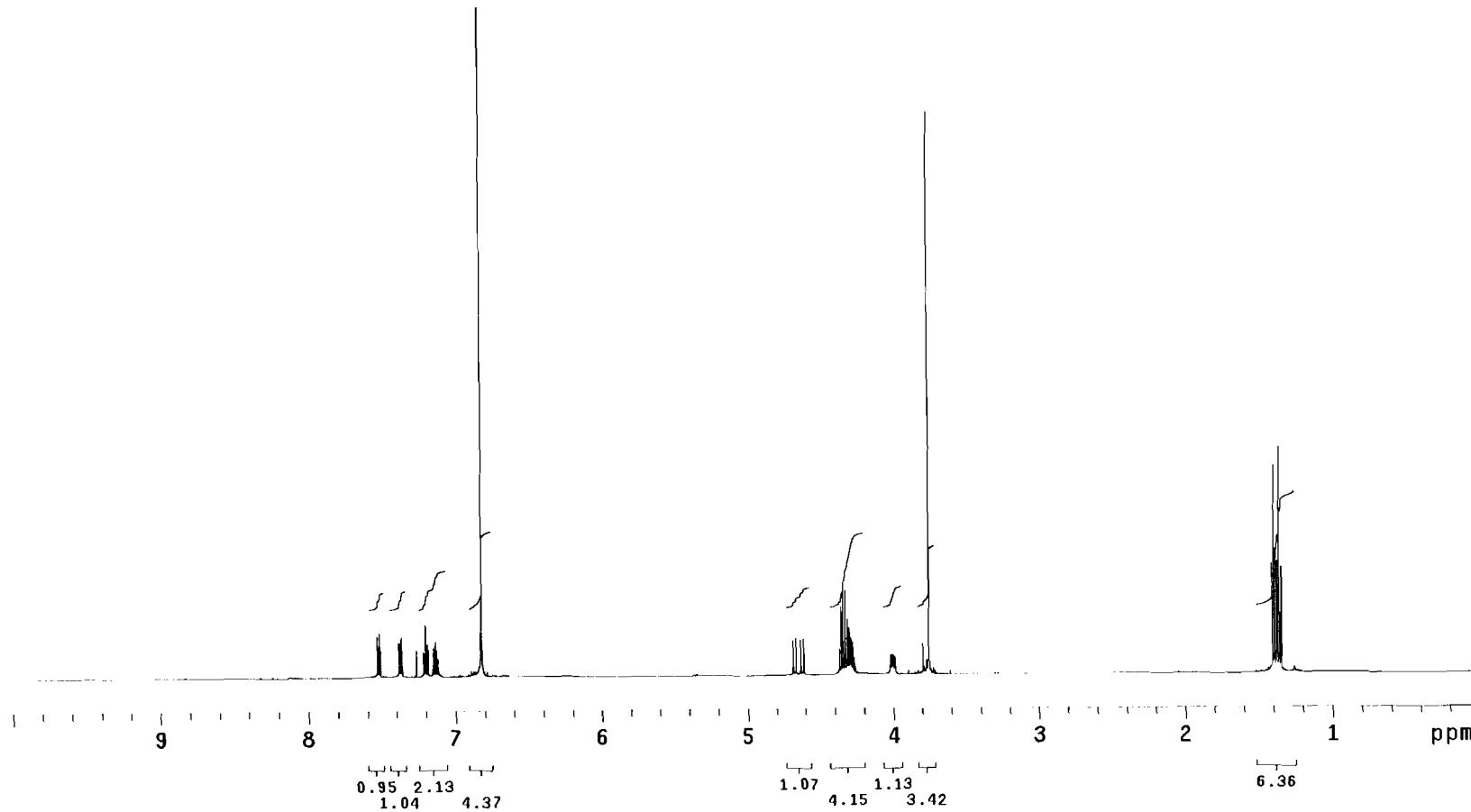
DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



3c



S-14

VI-DR-62-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: VI-DR-62-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

3000 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

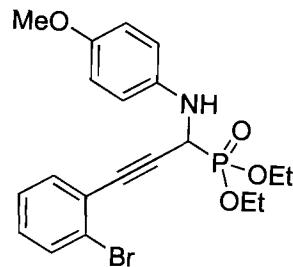
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DATA PROCESSING

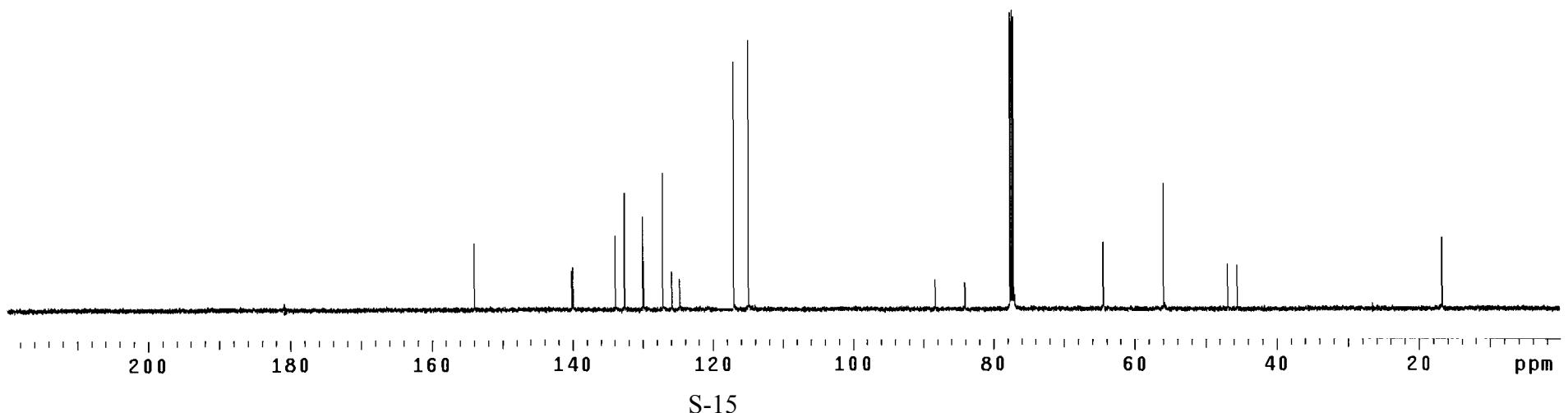
Line broadening 0.5 Hz

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Total time 1 hr, 55 min, 28 sec



3c



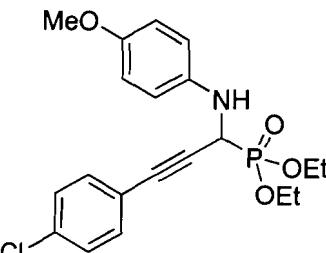
VI-DR-70-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:
File: PROTON

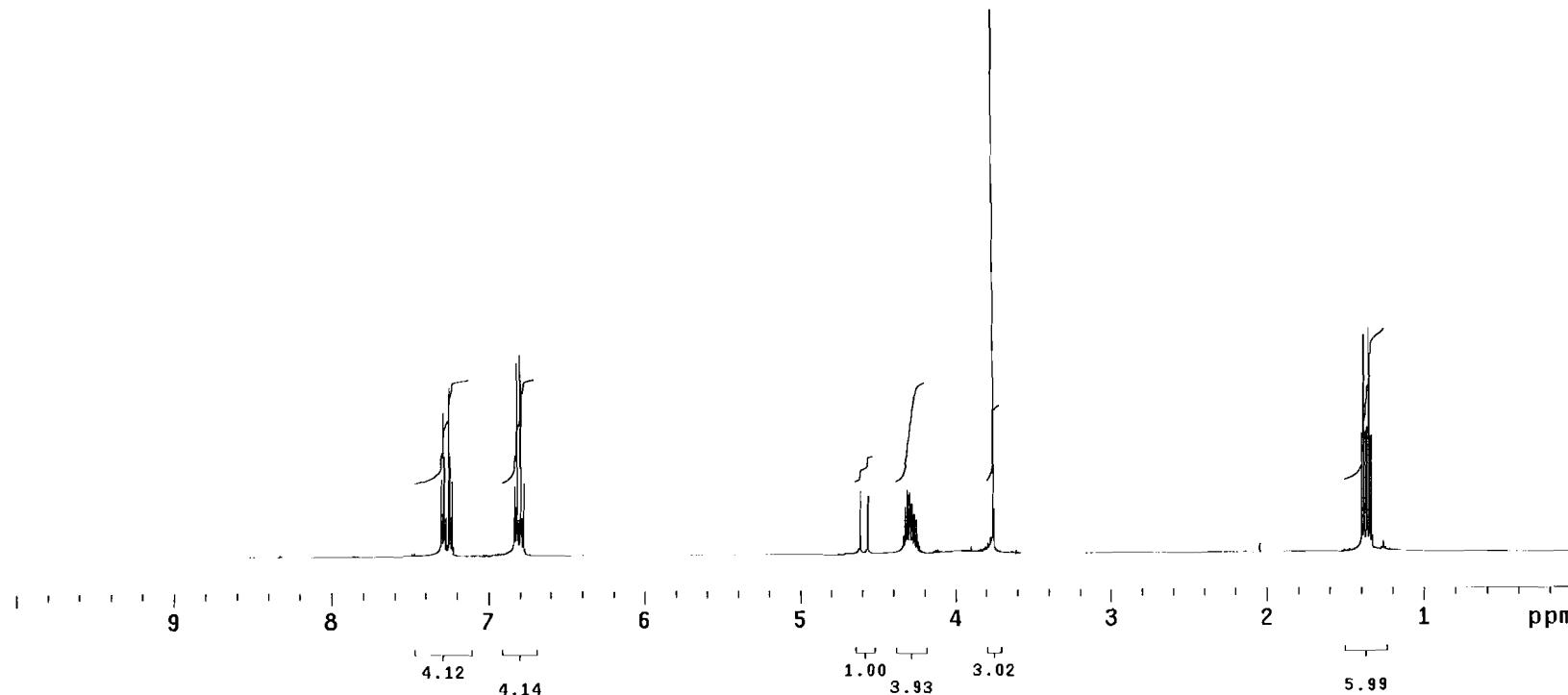
Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
4 repetitions
OBSERVE H1, 499.6847200 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 11 sec



3d



VI-DR-70-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

10000 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

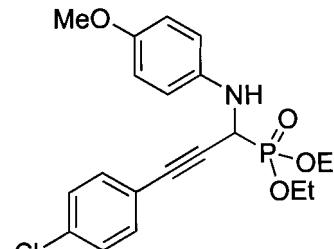
WALTZ-16 modulated

DATA PROCESSING

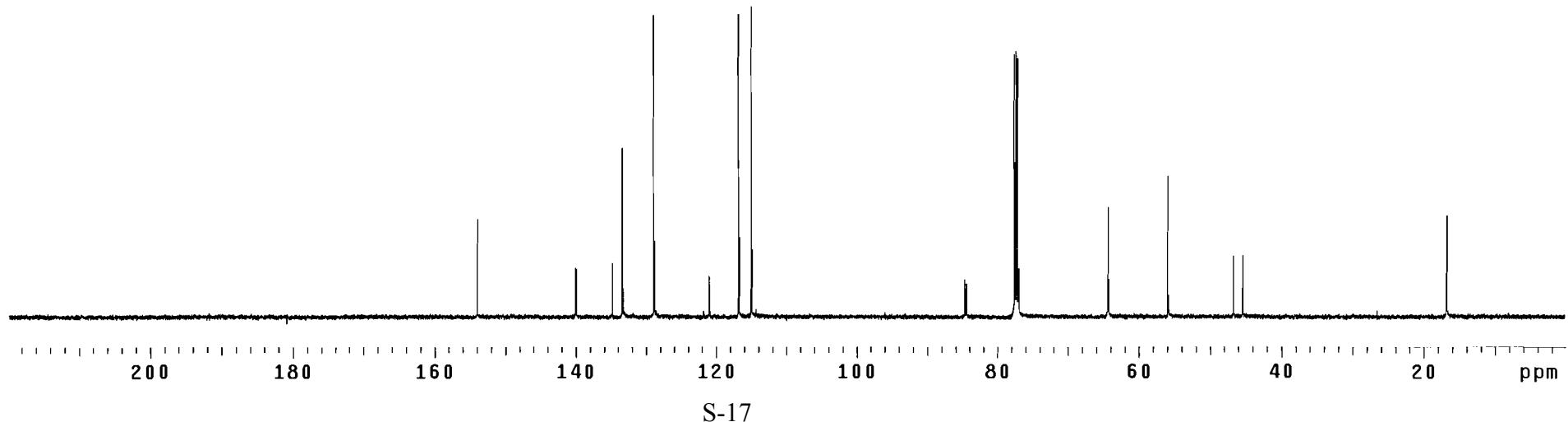
Line broadening 0.5 Hz

FT size 131072

Total time 6 hr, 24 min, 53 sec



3d



VI-DR-71-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-71-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

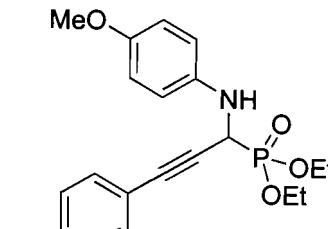
8 repetitions

OBSERVE H1, 499.6847200 MHz

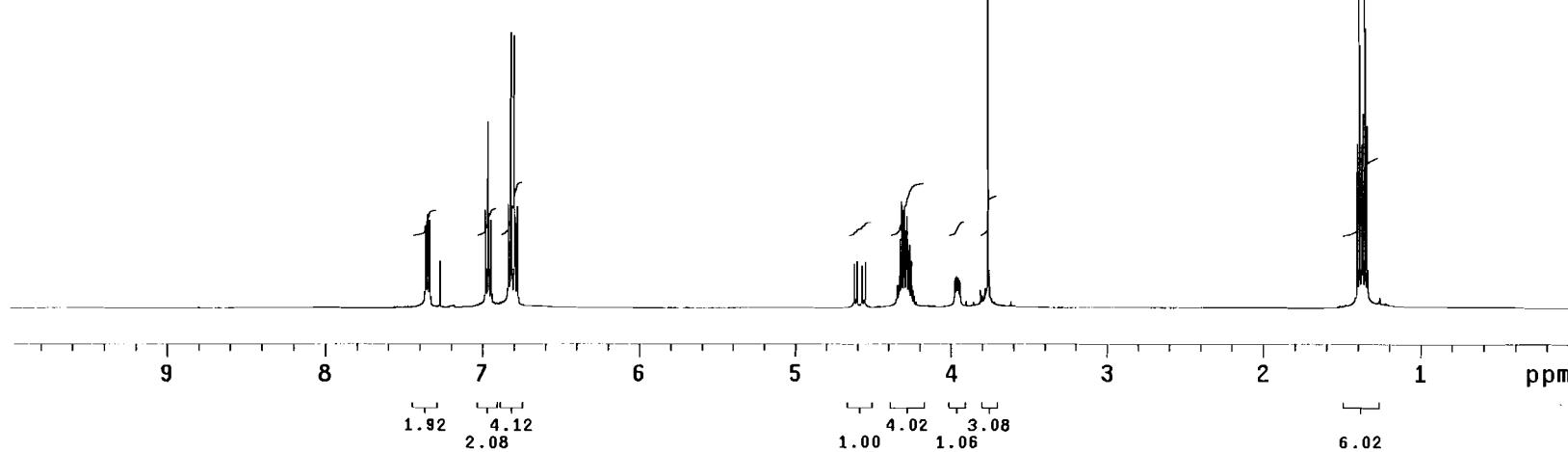
DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



3e



VI-OR-71-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: i-14-87

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

2000 repetitions

OBSERVE C13, 125.6457928 MHz

DECOUPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

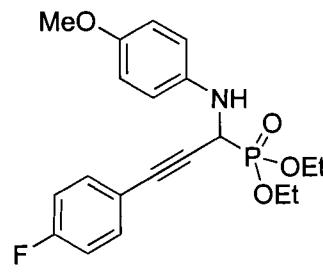
WALTZ-16 modulated

DATA PROCESSING

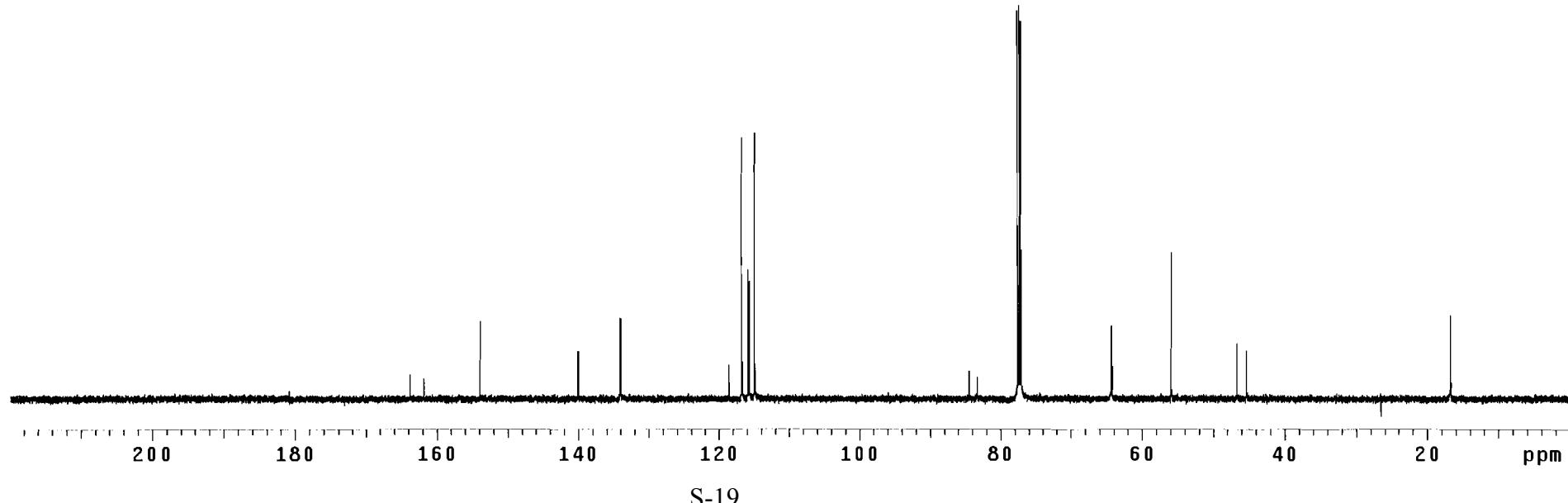
Line broadening 0.5 Hz

FT size 131072

Total time 1 hr, 16 min, 58 sec



3e



VI-DR-63-1H

Archive directory: /home/drajasek/vnmrsys/data

Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-CF3-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

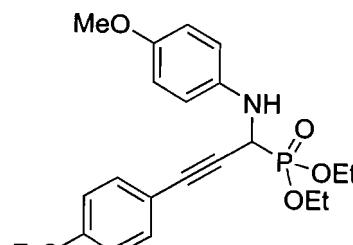
8 repetitions

OBSERVE H1, 499.6847200 MHz

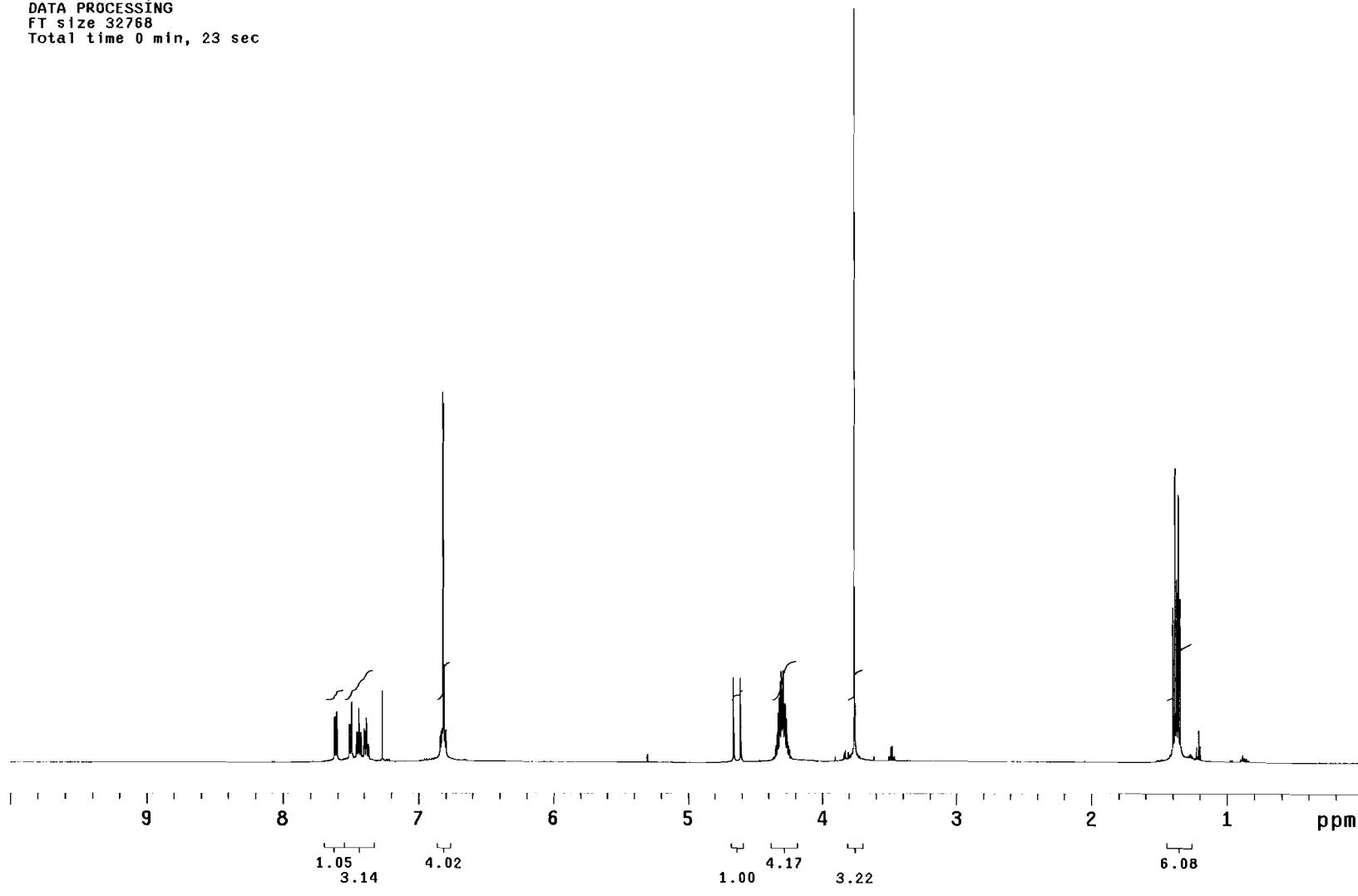
DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



3f



VI-DR 63-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: VI-DR-CF3-13C

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

3712 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

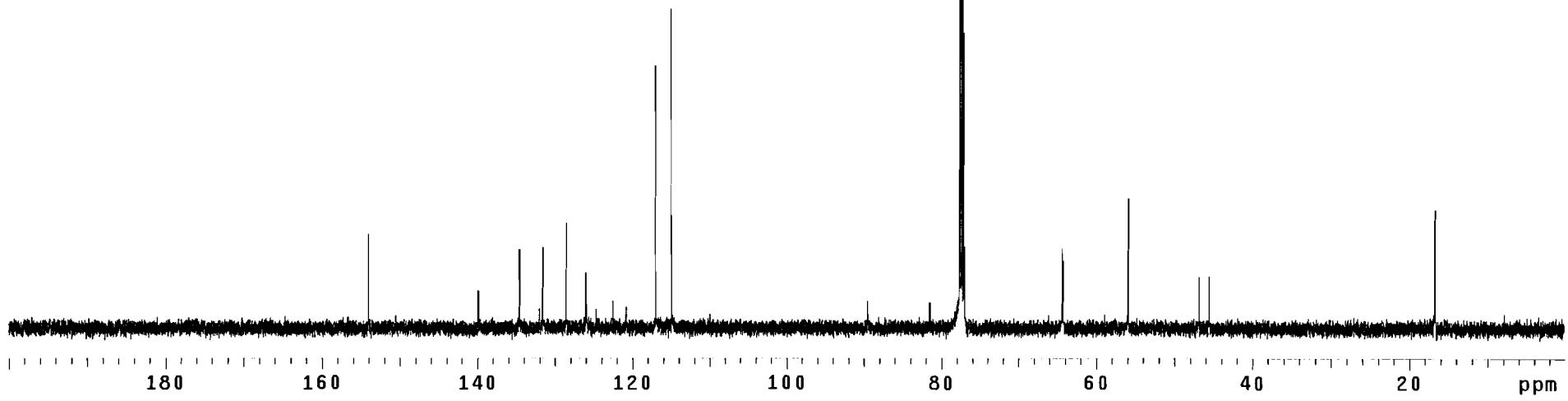
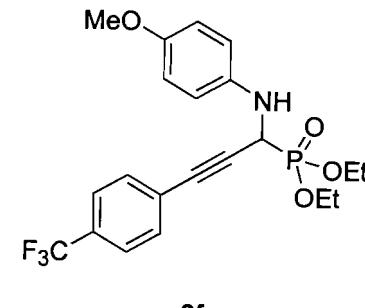
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 3 hr, 12 min, 26 sec



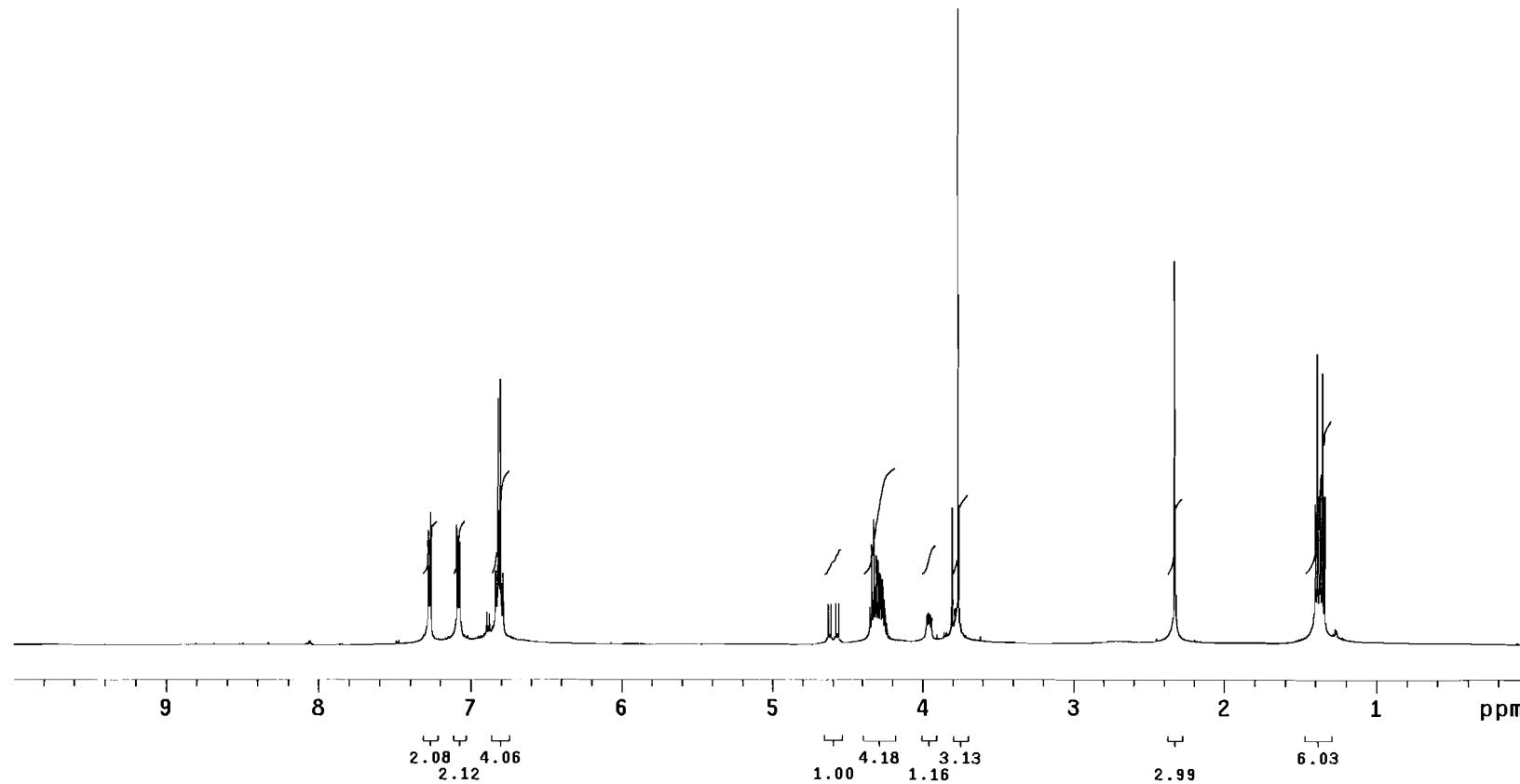
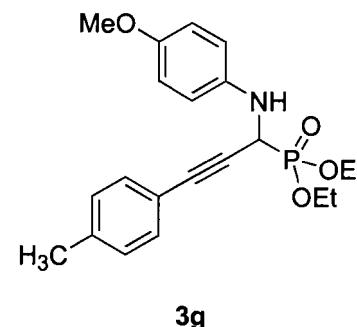
VI-DR-40-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: VI-DR-40-1H
INOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.892 sec
Width 7995.2 Hz
8 repetitions
OBSERVE H1, 499.6847200 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 23 sec



VI-DR-40-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: VI-DR-40-13C

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

1792 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

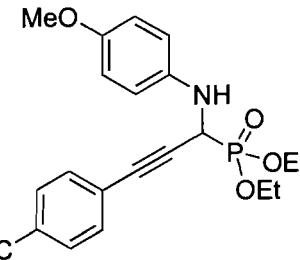
WALTZ-16 modulated

DATA PROCESSING

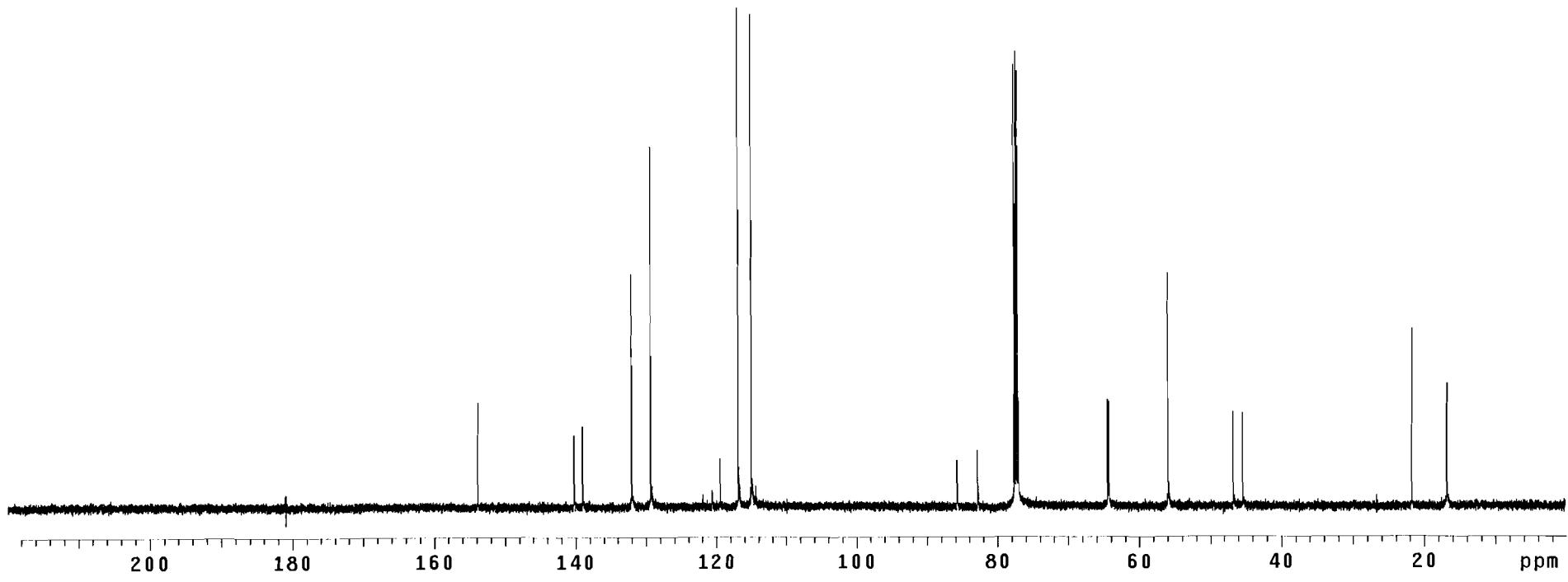
Line broadening 0.5 Hz

FT size 131072

Total time 6 hr, 24 min, 53 sec



3g



VI-DR-60-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-60-1Ha

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

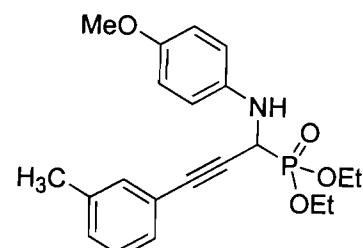
4 repetitions

OBSERVE H1, 499.6847200 MHz

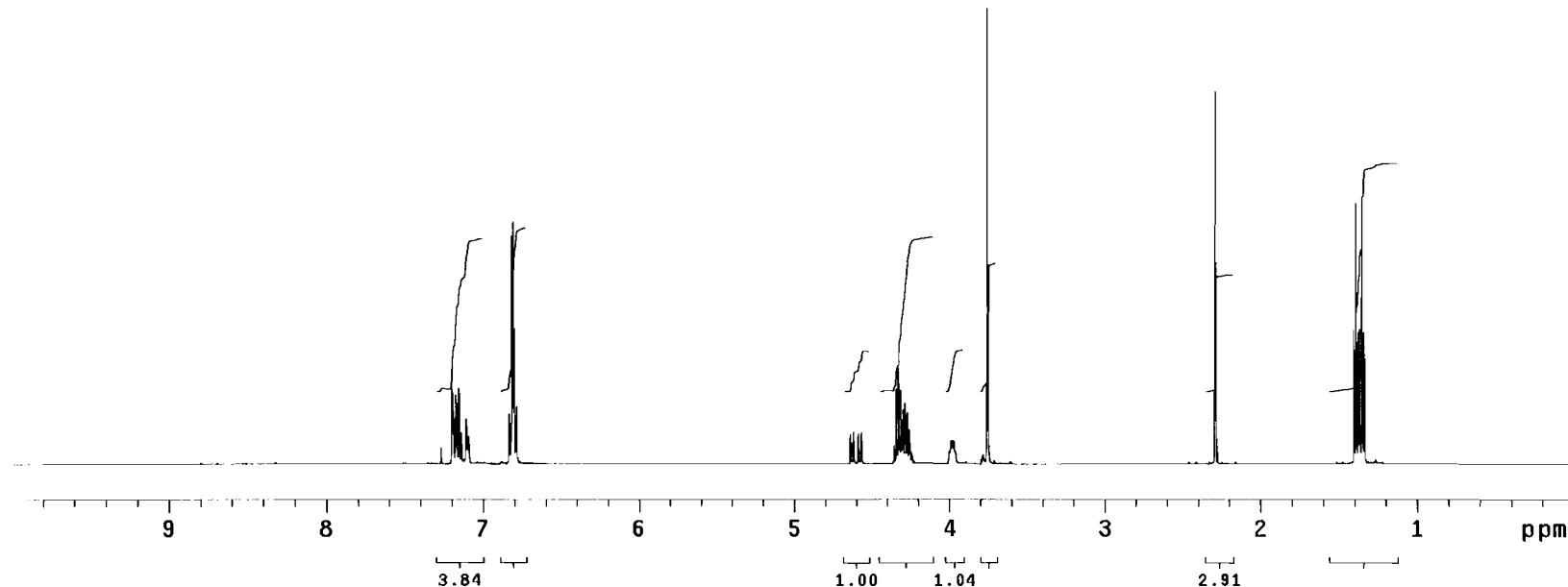
DATA PROCESSING

FT size 32768

Total time 0 min, 11 sec



3h



VI-DR-60-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: VI-DR-60-13Cb

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

1000 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

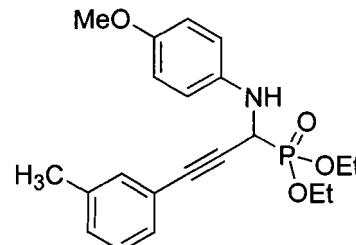
WALTZ-16 modulated

DATA PROCESSING

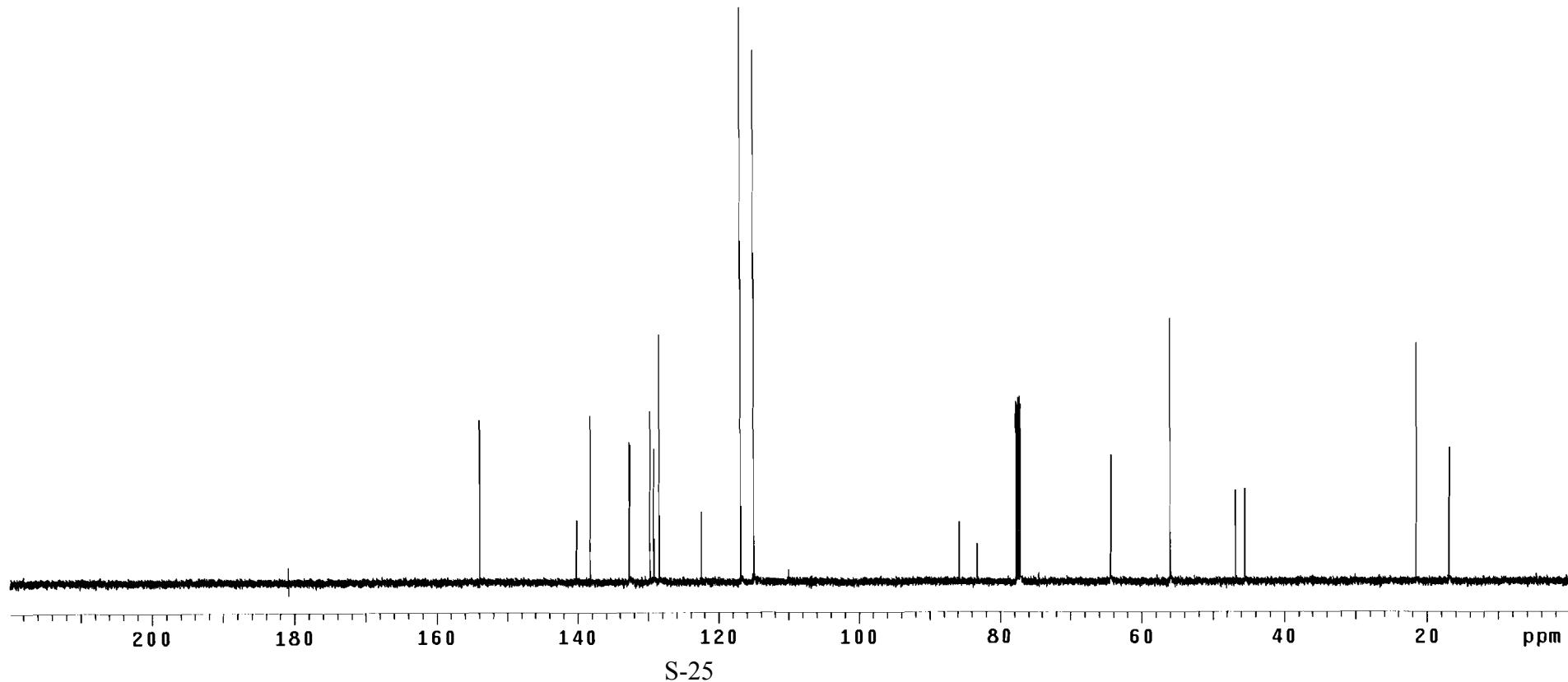
Line broadening 0.5 Hz

FT size 131072

Total time 38 min, 29 sec



3h



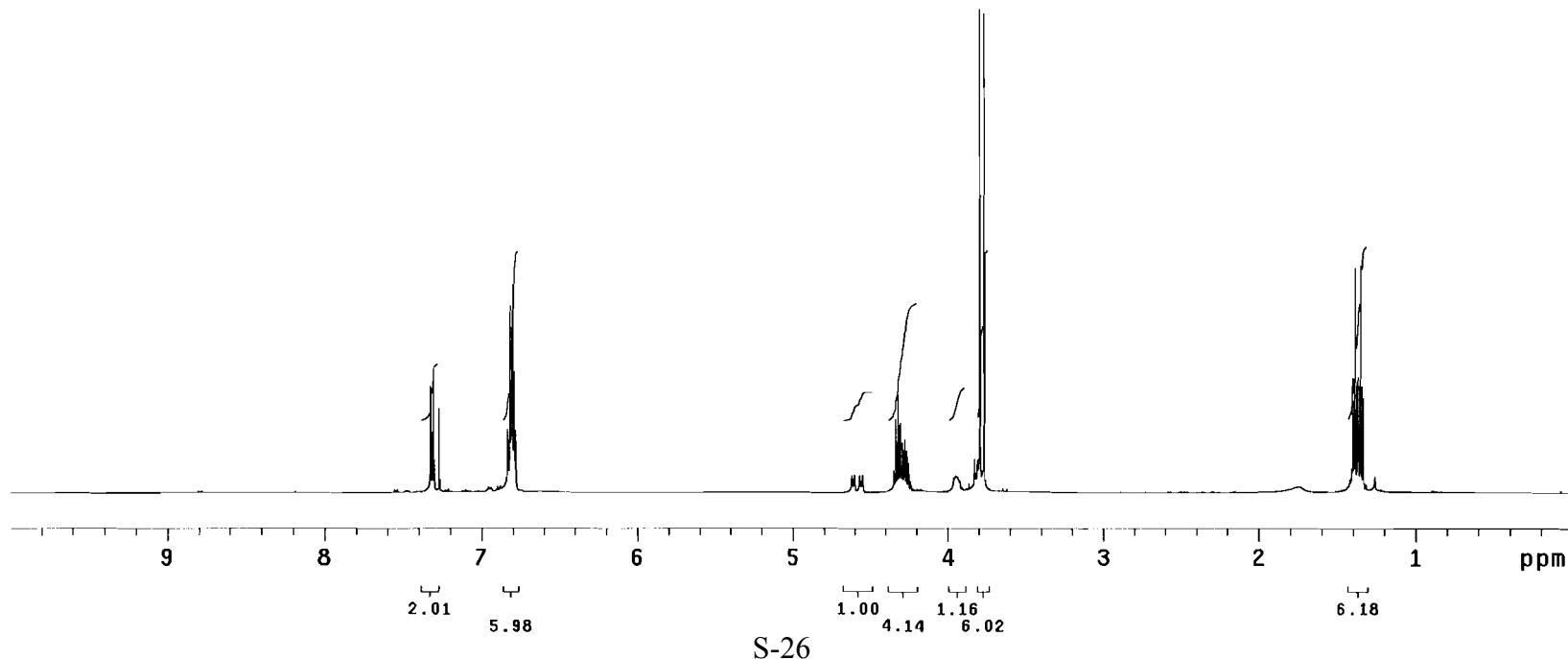
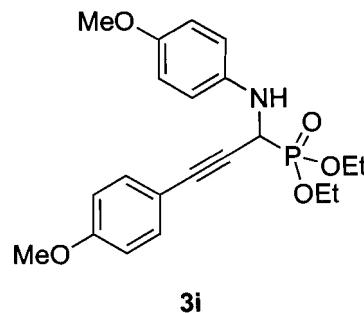
VI-DR-39-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃
Ambient temperature
File: VI-DR-39-1H
INNOVA-500 "Inova500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Aca. time 1.892 sec
Width 7995.2 Hz
4 repetitions
OBSERVE H1, 499.6847200 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 11 sec



VI-DR-39-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: VI-DR-39-13C

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

14000 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

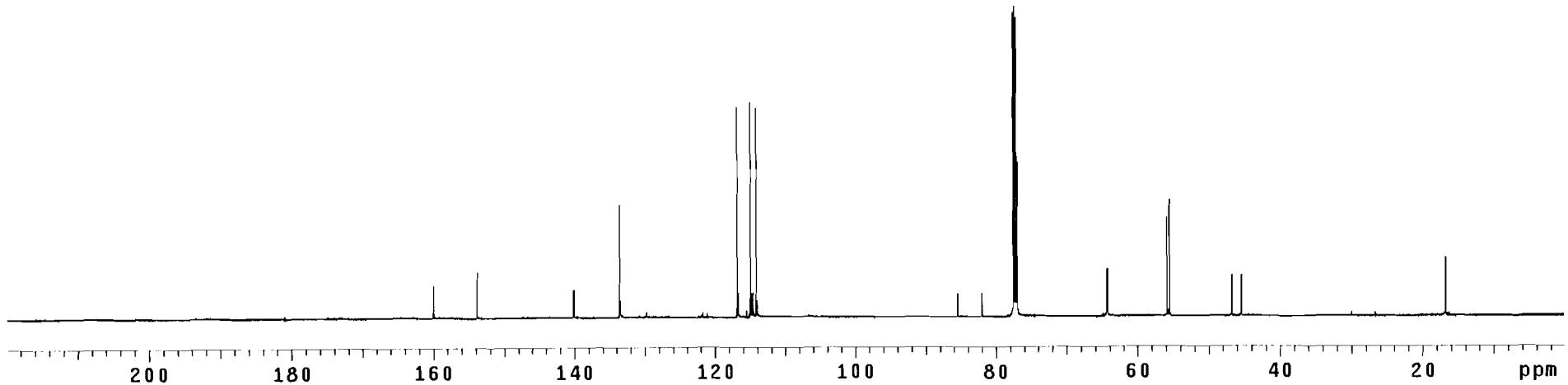
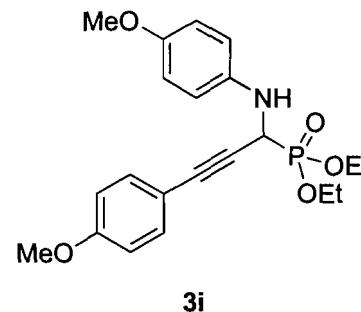
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 8 hr, 58 min, 51 sec



VI-DR-65-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-C6H4F2-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

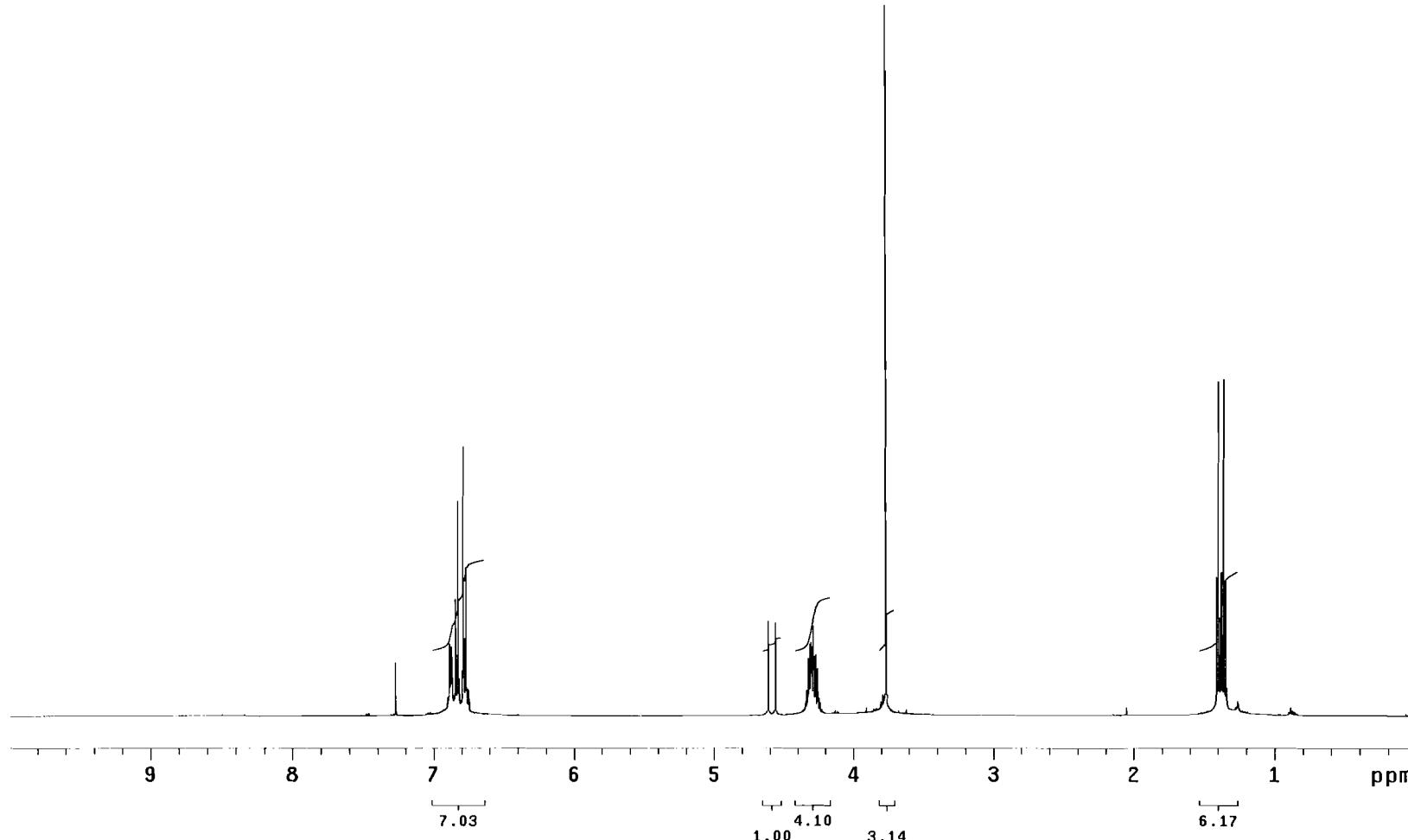
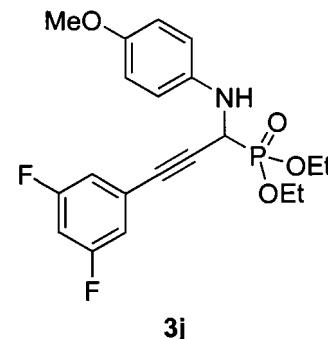
8 repetitions

OBSERVE H1, 499.6847200 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



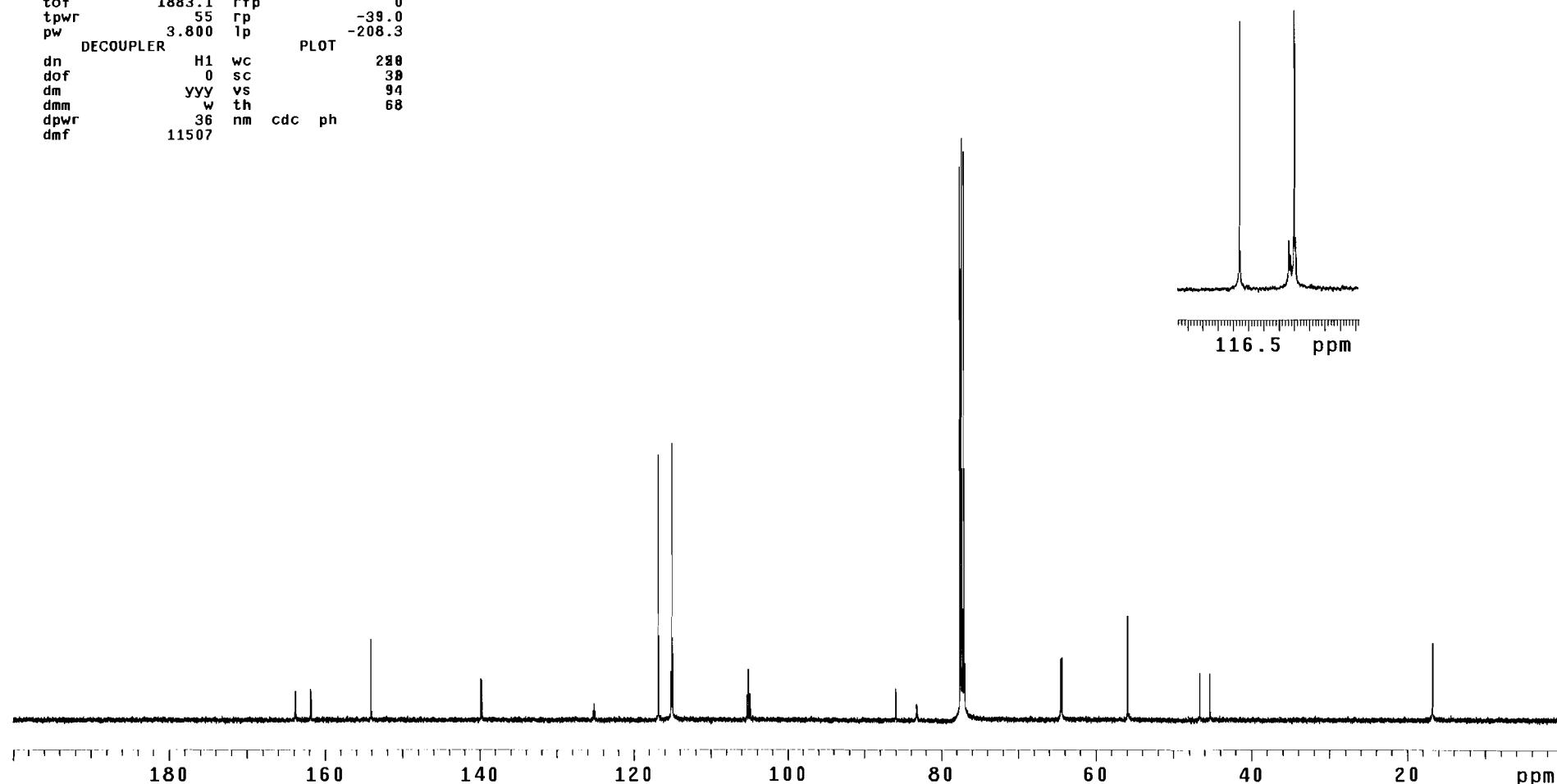
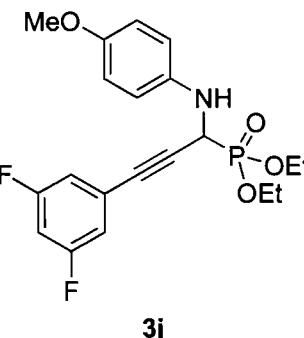
VI-DR-65-13C

exp1 s2pul

SAMPLE	SPECIAL
date Aug 14 2006	temp not used
solvent CDCl ₃	gain not used
file /export2/home/~	spin 16
/drajasek/vnmrsys/~/	hst 0.008
data/VI-DR-65-13C.~	pw90 7.600
fid alfa	10.000

ACQUISITION	FLAGS	
sw 31409.5	11	n
at 1.300	in	n
np 81694	dp	y
fb 17000	hs	nn
bs 64	PROCESSING	
d1 1.000	1b	0.50
nt 15000	fn	not used
ct 15000	DISPLAY	
TRANSMITTER	sp	14188.8
tn C13	wp	25182.3
sfrq 125.660	rf1	1883.7
tof 1883.1	rfp	0
tpwr 55	rp	-39.0
pw 3.800	lp	-208.3

DECOPPLER	PLOT	
dn H1	wc	288
dof 0	sc	38
dm YYY	vs	94
dmw W	th	68
dpwr 36	nm cdc ph	
dmf 11507		



VI-DR-66-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-66-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

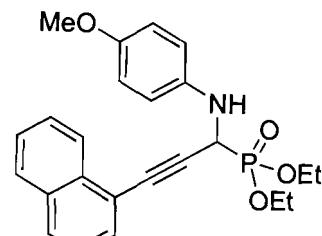
8 repetitions

OBSERVE H₁, 499.6847200 MHz

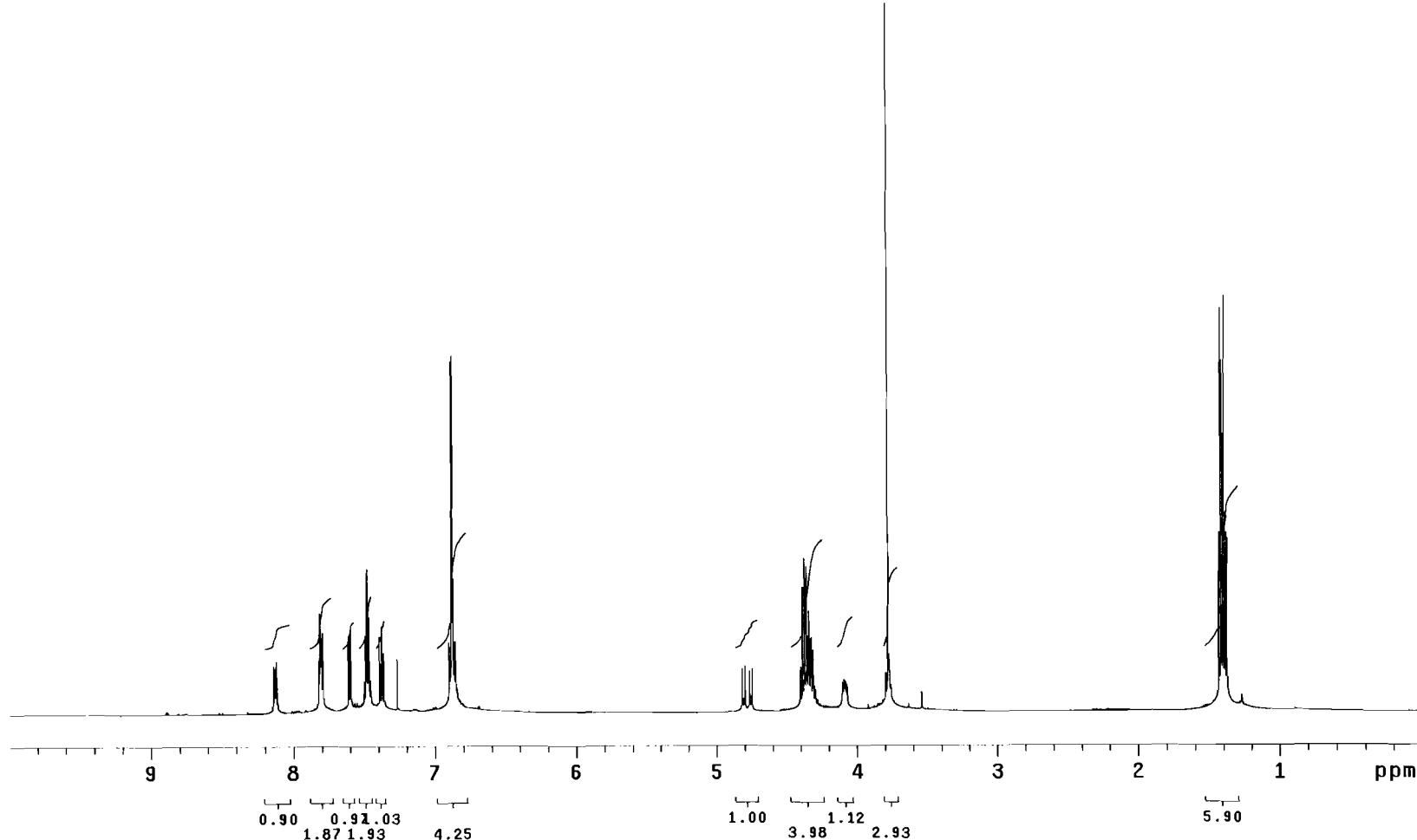
DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



3k



VI-DR-66-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

17000 repetitions

OBSERVE C13, 125.6457928 MHz

DECOUPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

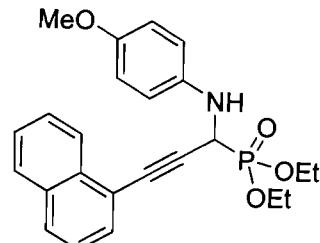
WALTZ-16 modulated

DATA PROCESSING

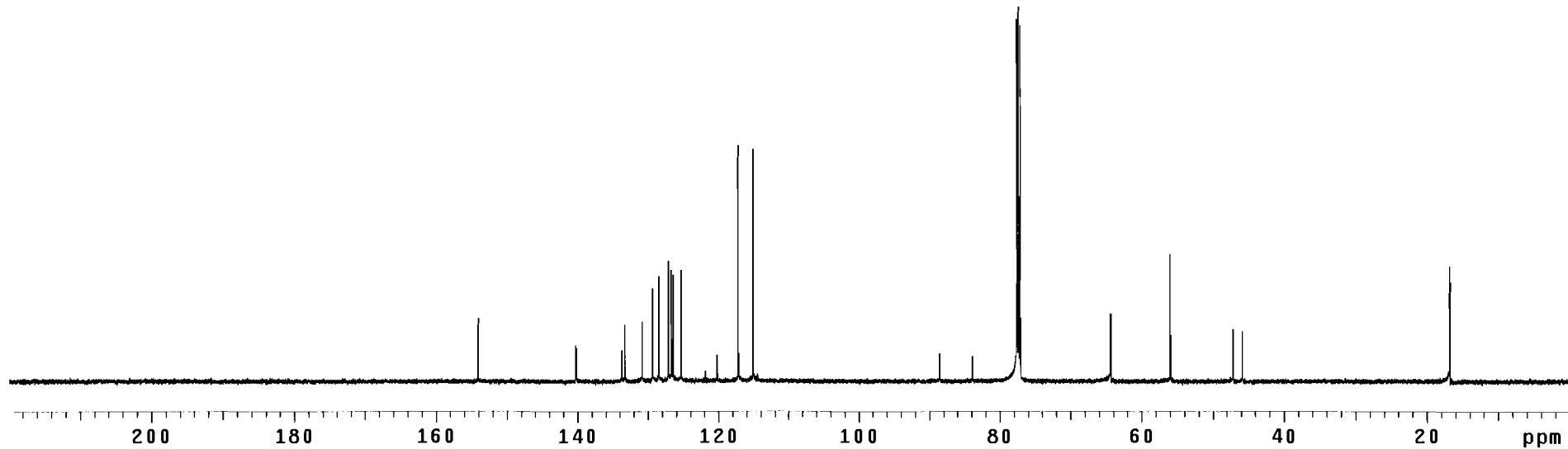
Line broadening 0.5 Hz

FT size 131072

Total time 10 hr, 54 min, 19 sec



3k



VI-DR-59-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-59-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

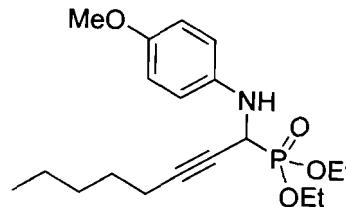
8 repetitions

OBSERVE H1, 499.6847200 MHz

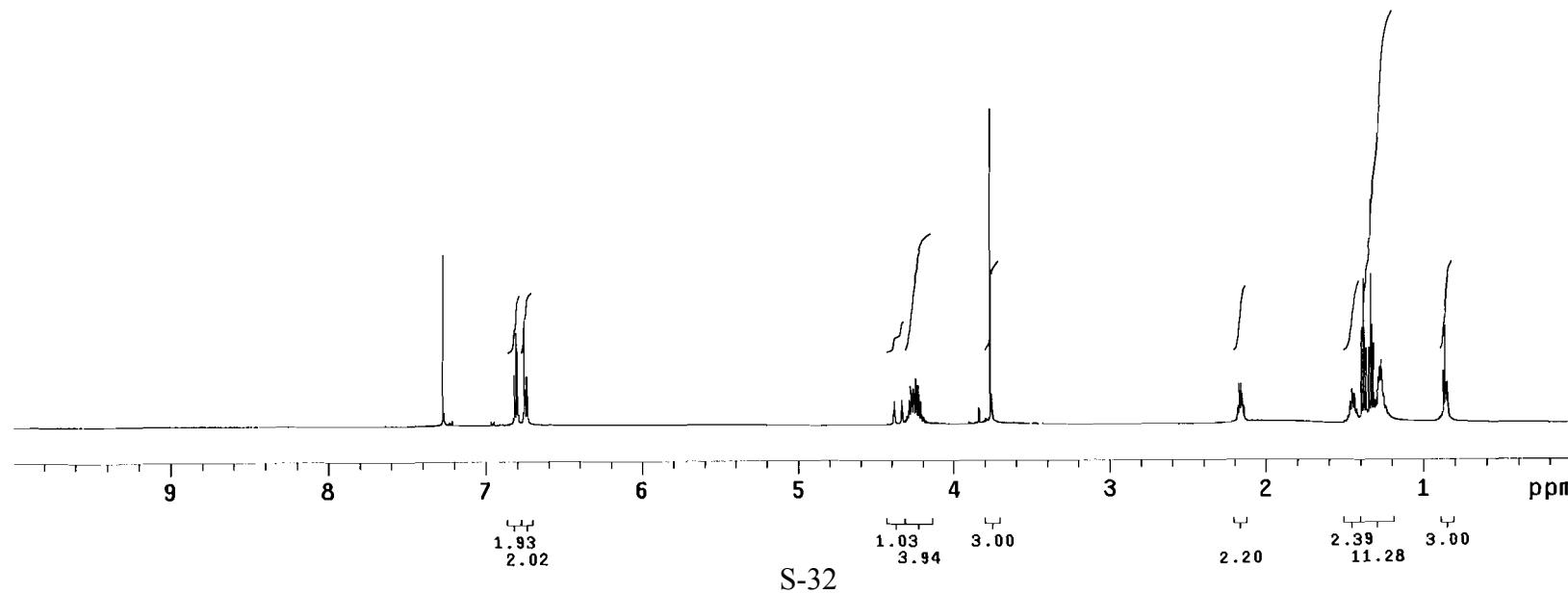
DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



3l



S-32

VI-DR-59-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: VI-DR-59-carbon

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

3328 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

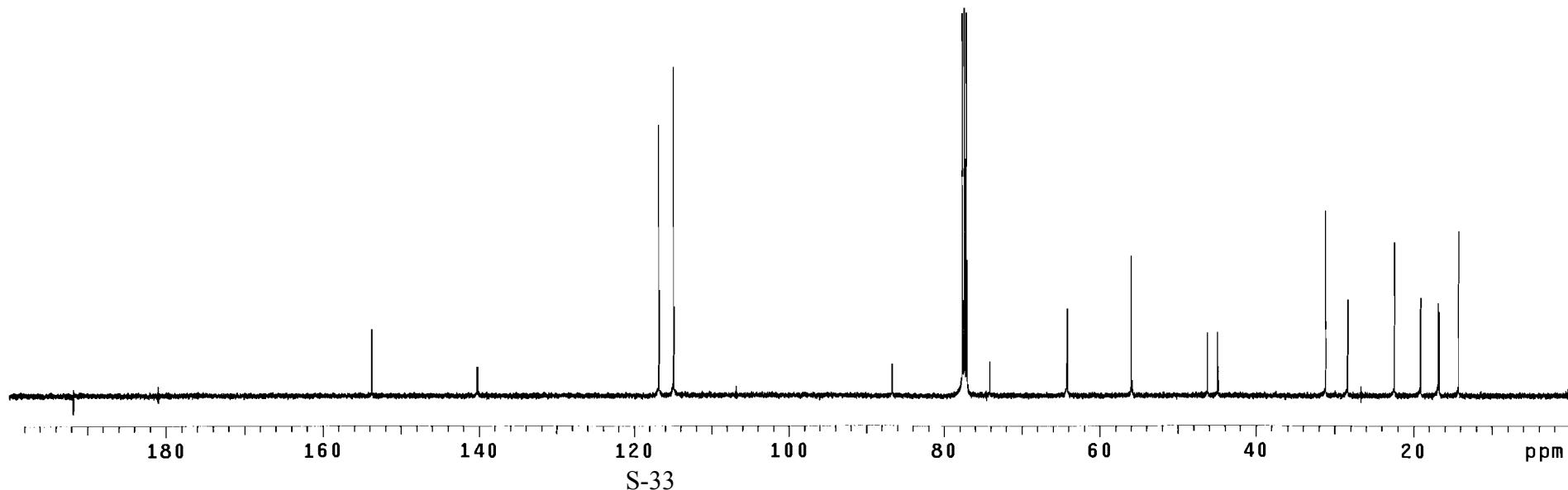
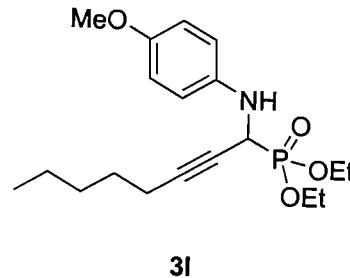
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 3 hr, 12 min, 26 sec



VI-DR-50-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

File: VI-DR-50-1H

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

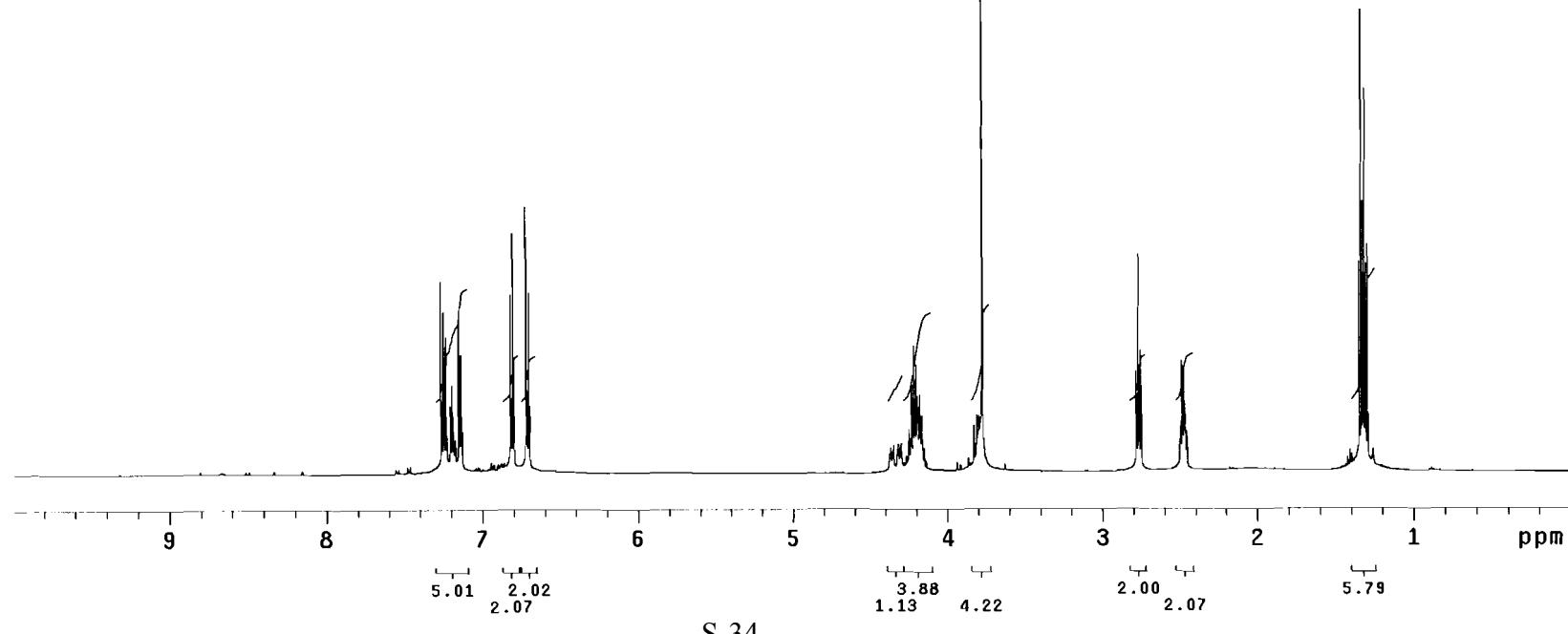
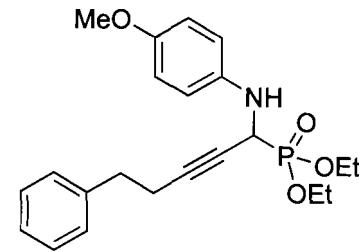
12 repetitions

OBSERVE H1, 499.6847200 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 34 sec



VI-DR-50-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

File: VI-DR-50-13C

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

13000 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

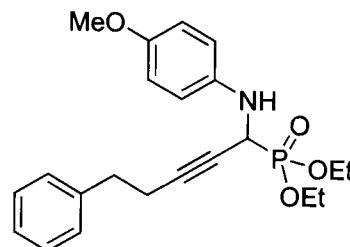
WALTZ-16 modulated

DATA PROCESSING

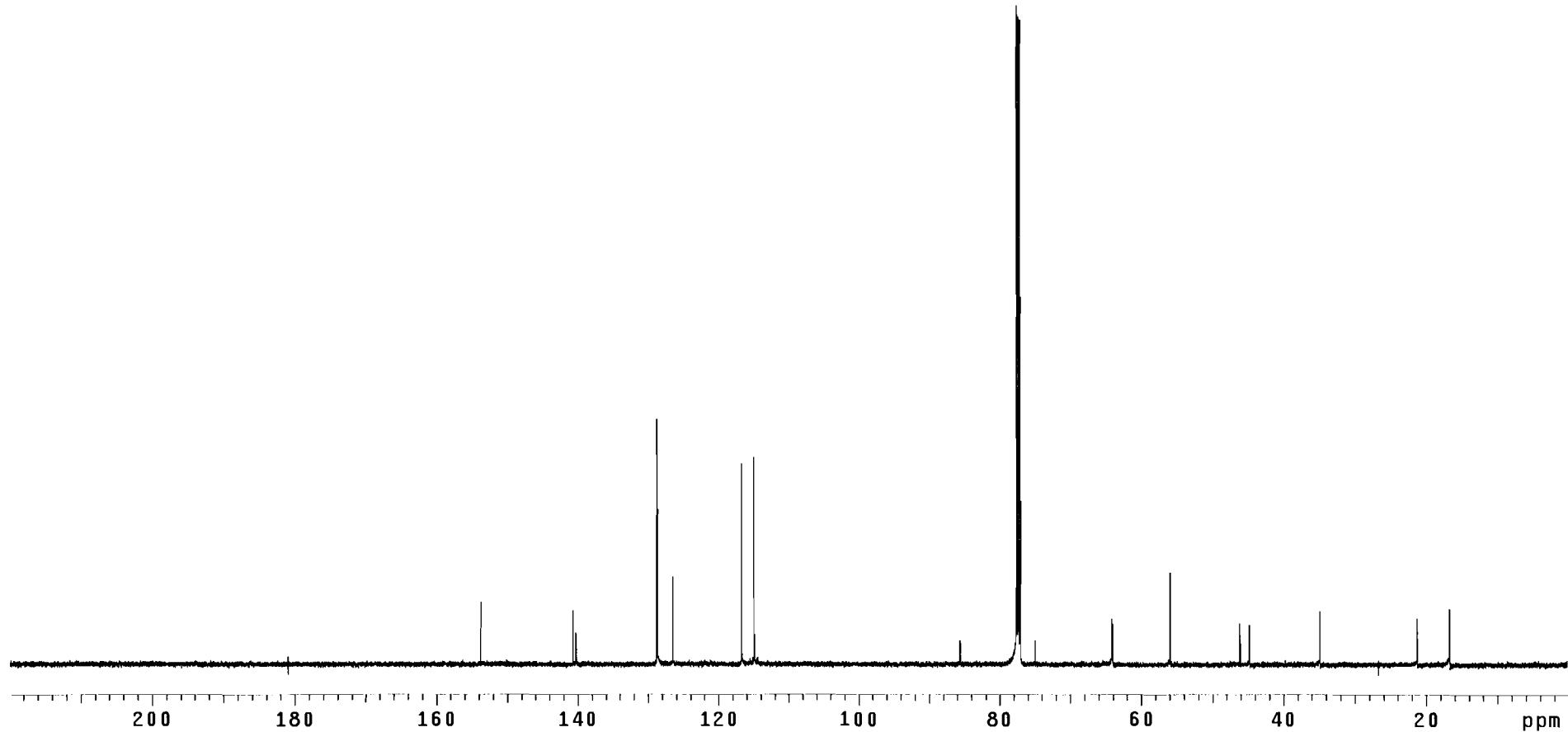
Line broadening 0.5 Hz

FT size 131072

Total time 8 hr, 20 min, 21 sec



3m



VI-DR-73-1H

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.892 sec

Width 7995.2 Hz

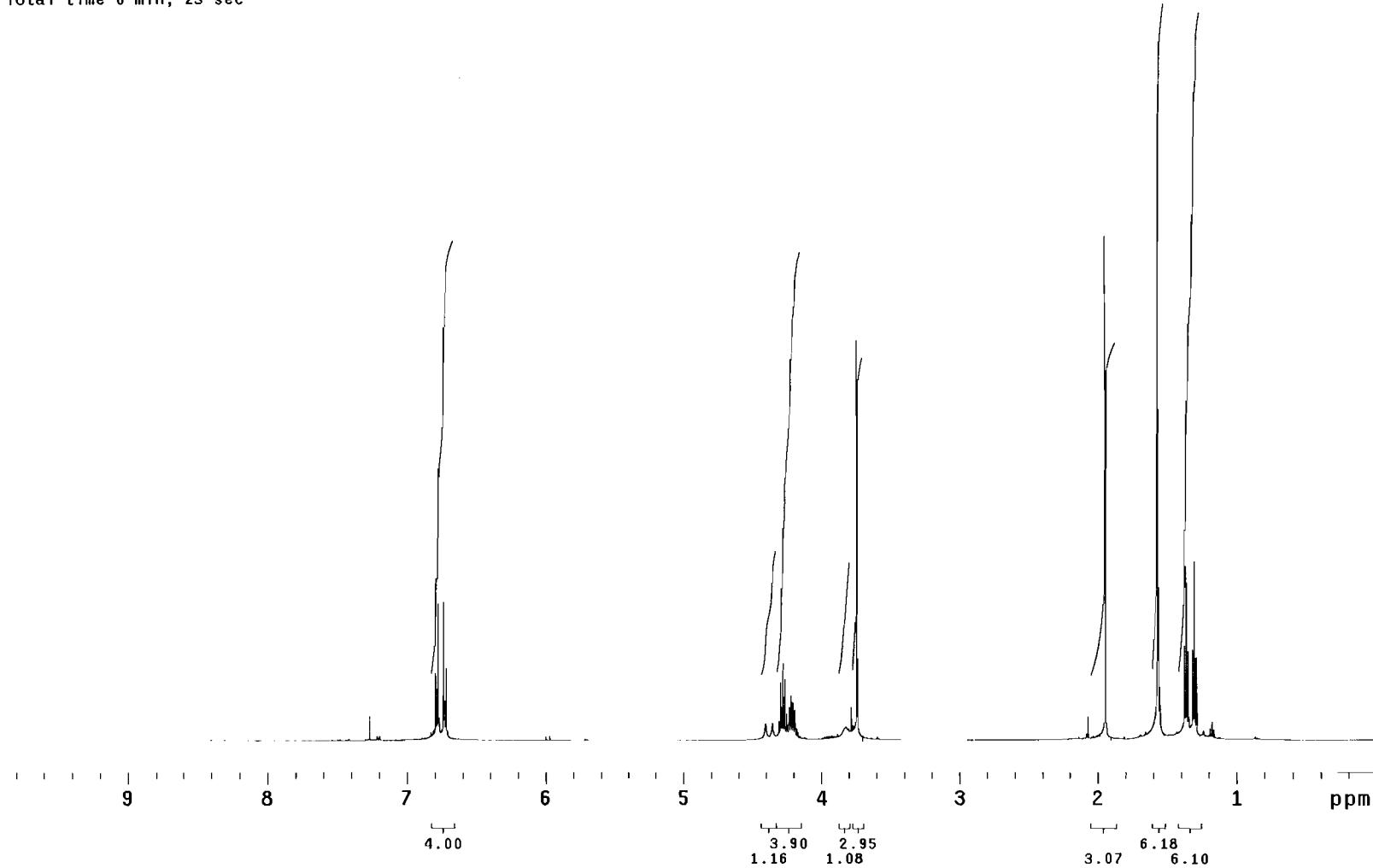
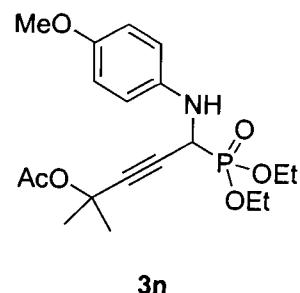
8 repetitions

OBSERVE H1, 499.6847200 MHz

DATA PROCESSING

FT size 32768

Total time 0 min, 23 sec



VI-DR-73-13C

Archive directory: /home/drajasek/vnmrsys/data
Sample directory:
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl₃

Ambient temperature

User: 1-14-87

INOVA-500 "Inova500"

Relax. delay 1.000 sec

Pulse 45.0 degrees

Acq. time 1.300 sec

Width 31409.5 Hz

12000 repetitions

OBSERVE C13, 125.6457928 MHz

DECOPLE H1, 499.6872185 MHz

Power 36 dB

continuously on

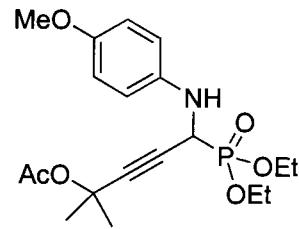
WALTZ-16 modulated

DATA PROCESSING

Line broadening 0.5 Hz

FT size 131072

Total time 7 hr, 41 min, 52 sec



3n

