Supporting Information for:

Construction of the Bacteriochlorin Macrocycle with Concomitant Nazarov Cyclization To Form the Annulated Isocyclic Ring: Analogues of Bacteriochlorophyll *a*

Shaofei Zhang and Jonathan S. Lindsey*

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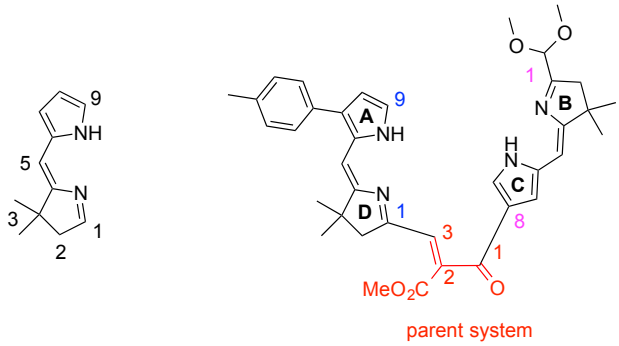
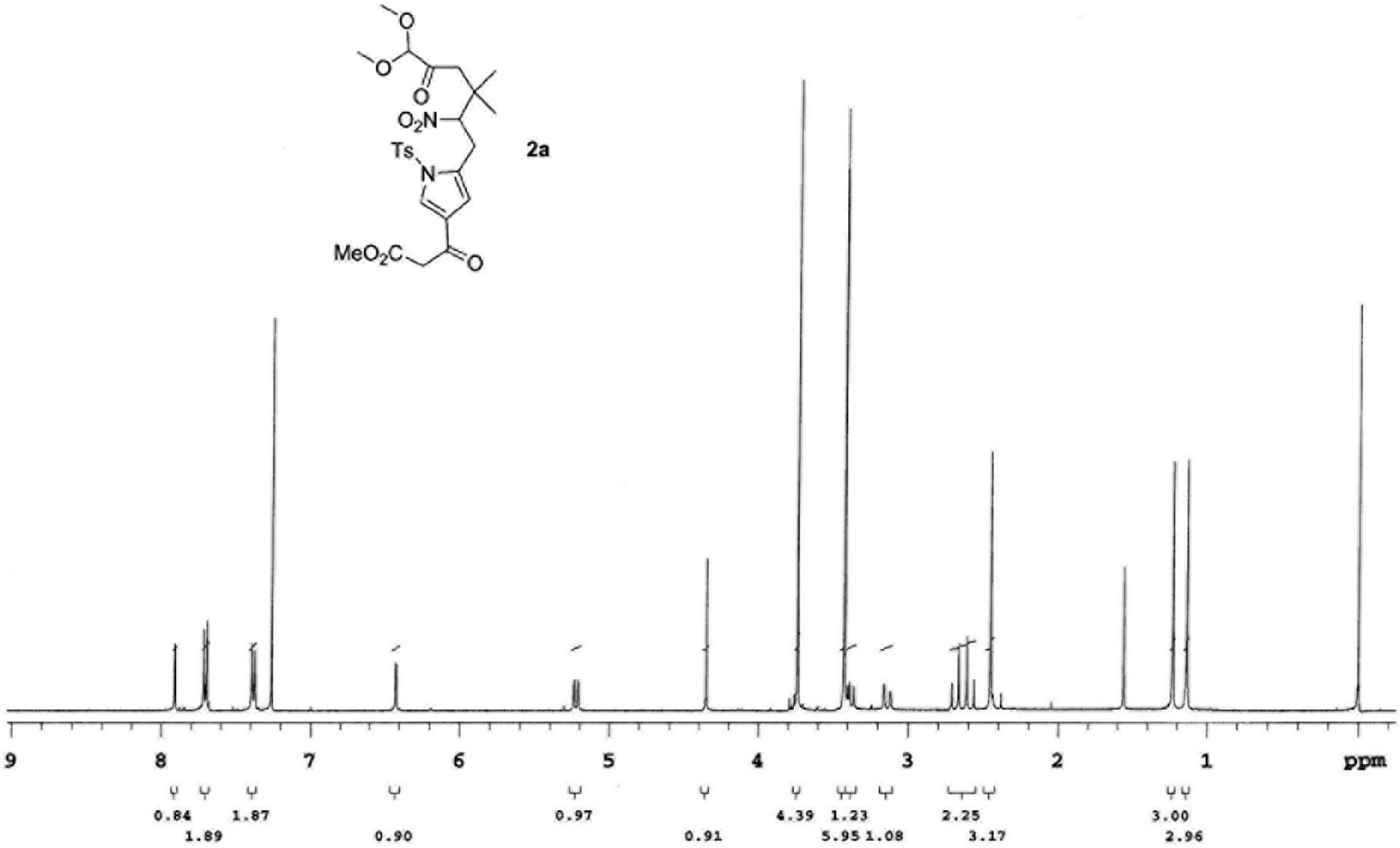
1. Nomenclature

Compounds of type **12** are unsaturated linear tetrapyrrole species with some similarity to bilins (e.g., bilirubin, biliverdin, urobilin, etc.), which are degradation products of chlorophylls. However, unlike bilins, ring C and ring D in **12** species are not joined via a methylene bridge. Herein, the linear species **12** are built from Western and Eastern halves. The Western and Eastern halves are named as 2,3-dihydro-3,3-dimethyldipyrrins in accord with the literature.[6] Such compounds are keystones in this synthetic route. Thus, the nomenclature for compounds of type **12** employed herein relies on the name of the propenone as the parent system, to which are attached the Western and Eastern halves as substituents. The parent propenone is shown in red in Chart S1. This nomenclature should provide an understandable name and be consistent with our previous nomenclature in chlorin and bacteriochlorin chemistry.

Chart S1. Nomenclature employed herein



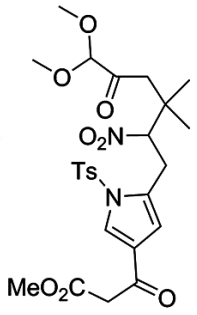
2. Spectral data


2a
9
8
7
6
5
4
3
2
1
ppm
0.84
1.89
1.87
0.90
0.97
0.91
4.39
1.23
5.95
1.08
2.25
3.17
3.00
2.96

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"

Pulse 39.0 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
50000 repetitions
OBSERVE C13, 75.3827063 MHz
DECOUPLE H1, 299.7932667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 29 hr, 20 min, 6 sec

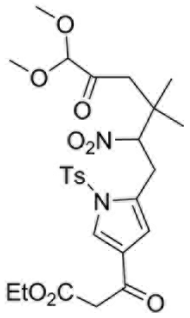


2a

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"

Relax. delay 1.000 sec
Pulse 36.0 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
64 repetitions
OBSERVE H1, 299.7918075 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 18 sec

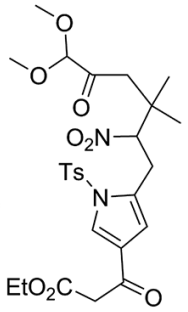


2b

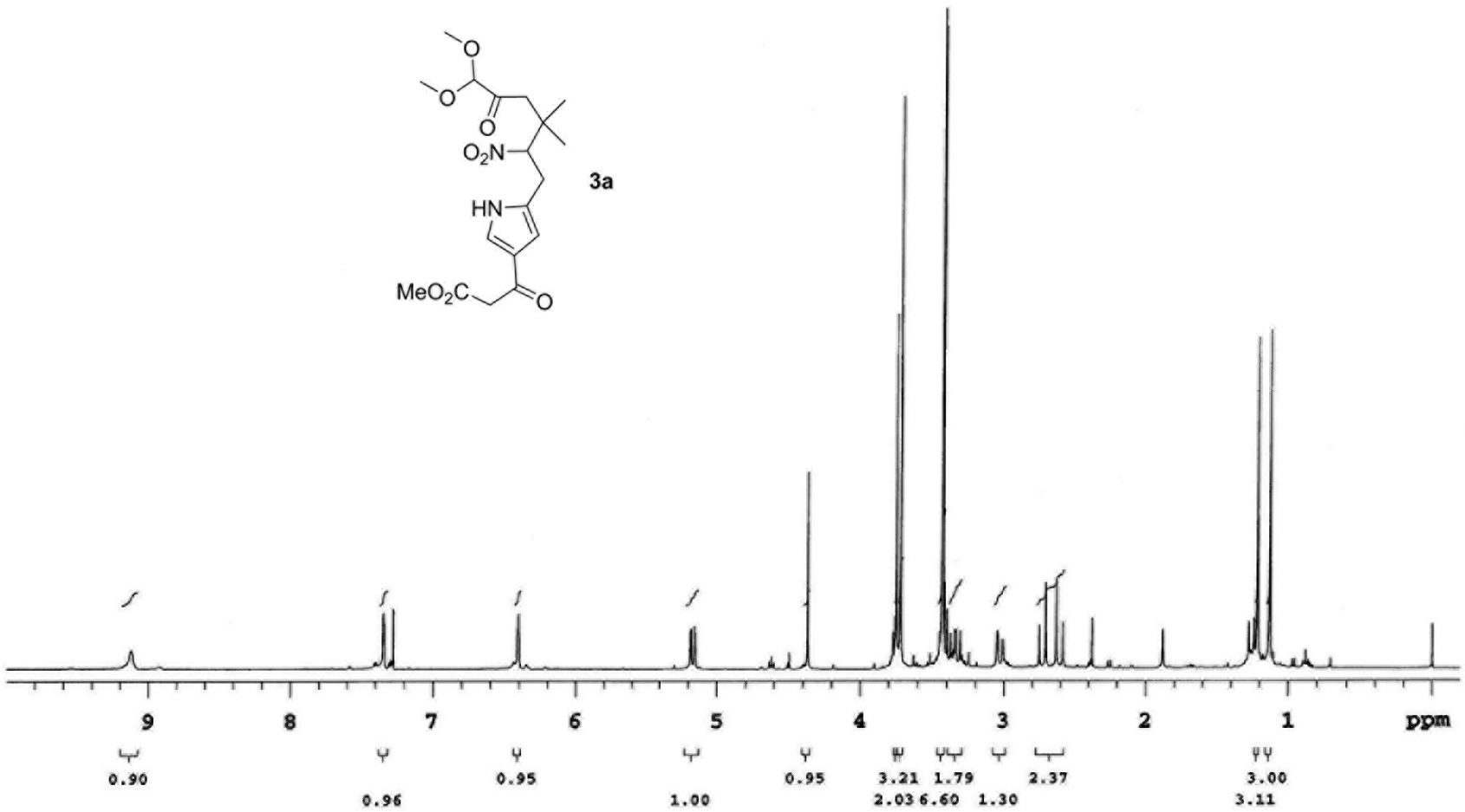
13C OBSERVE

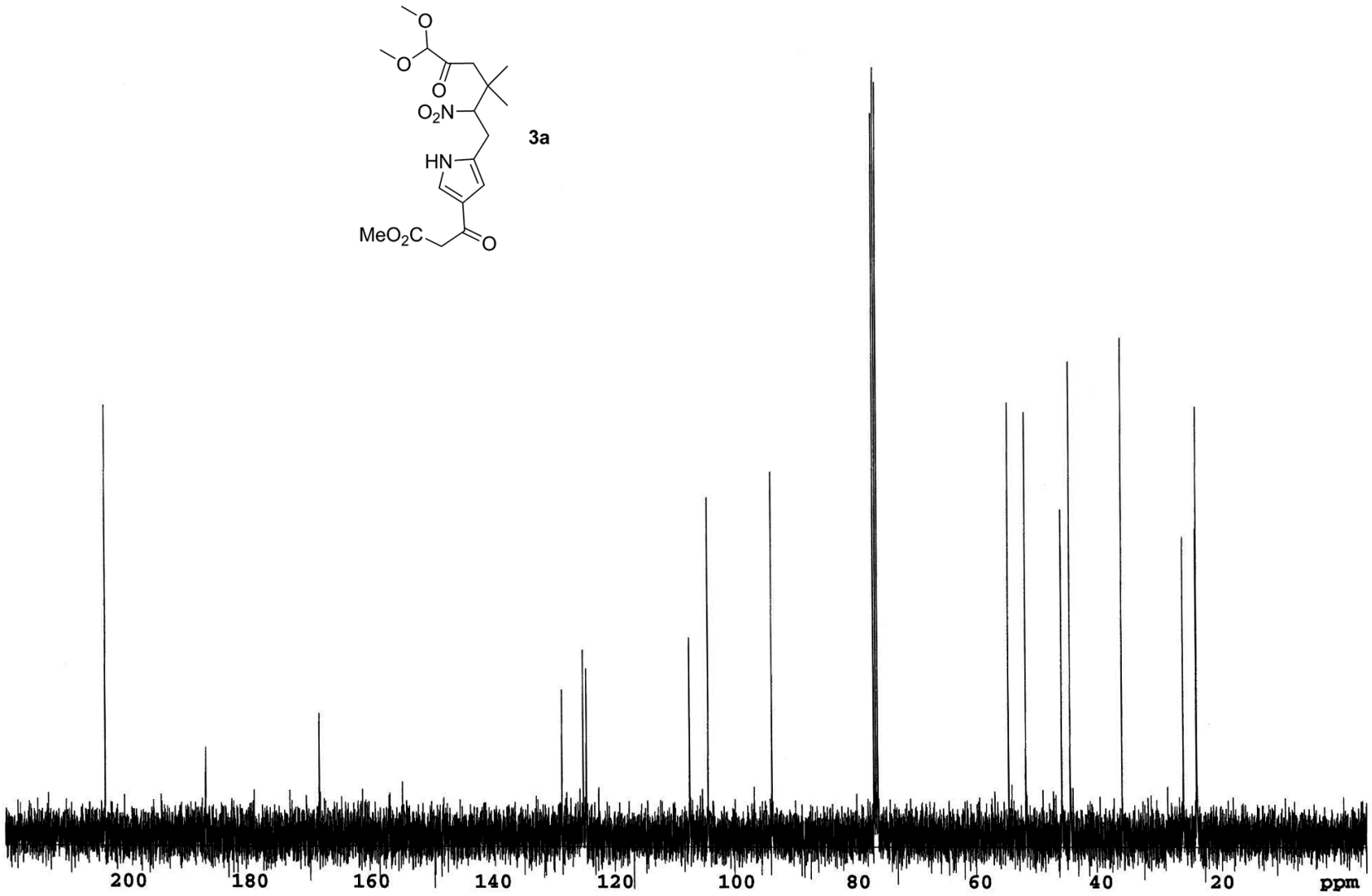
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"

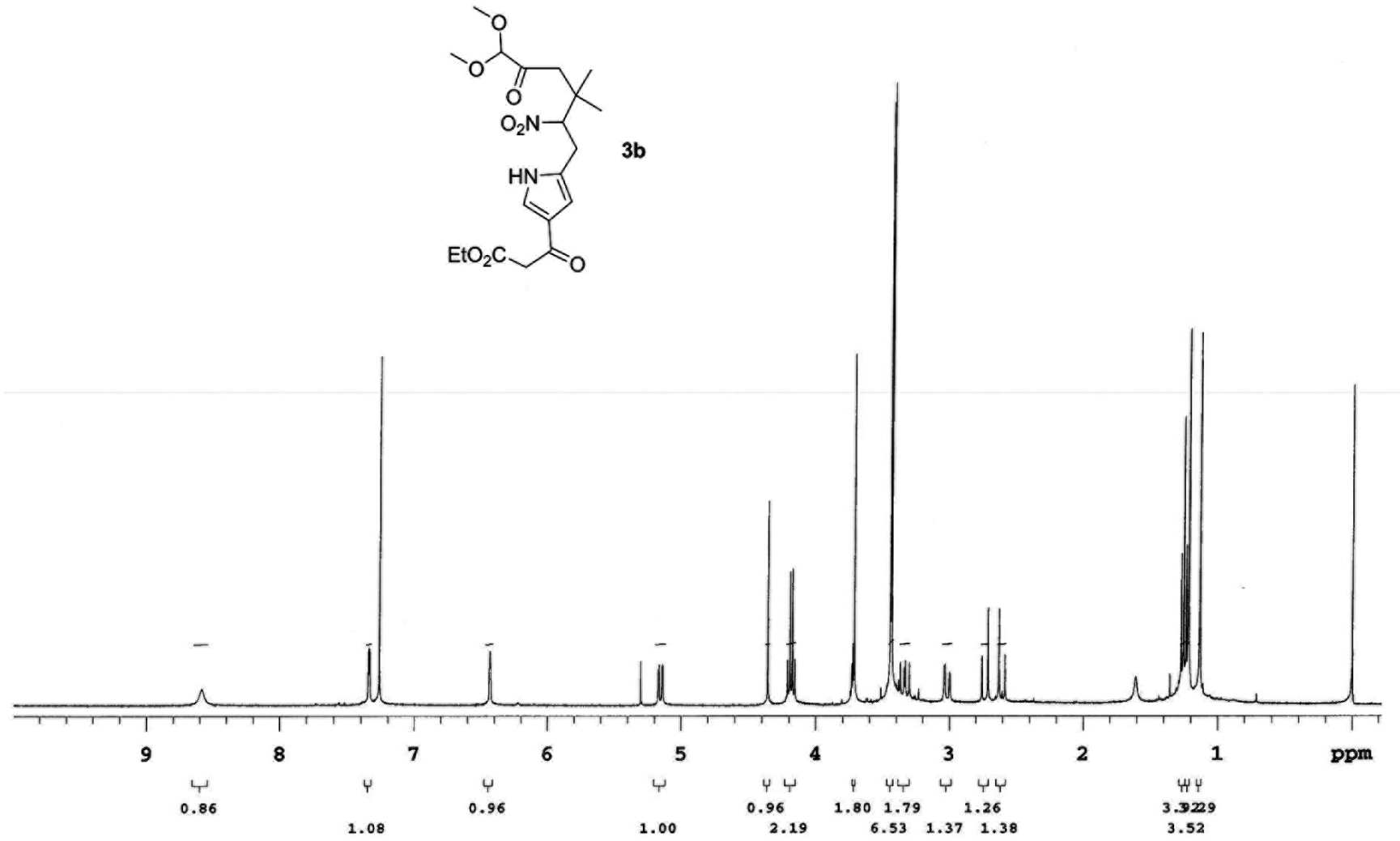
Pulse 39.0 degrees
Acq. time 1.815 sec
Width 18761.7 Hz
50000 repetitions
OBSERVE C13, 75.3826926 MHz
DECOUPLE H1, 299.7932667 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 29 hr, 20 min, 6 sec



2b


O
O
O
O2N
HN
MeO2C
O
3a
9
8
7
6
5
4
3
2
1
ppm
0.90
0.96
0.95
1.00
0.95
3.21
2.03
1.79
6.60
1.30
2.37
3.00
3.11

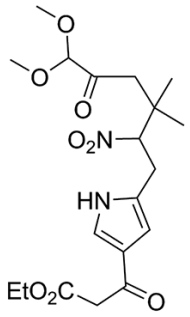

O
O
O
O_2N
HN
MeO_2C
O
3a
200
180
160
140
120
100
80
60
40
20
ppm


3b
9
8
7
6
5
4
3
2
1
ppm
0.86
1.08
0.96
1.00
0.96
2.19
1.80
1.79
6.53
1.37
1.26
1.38
3.52

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "ncsumerc400"

Pulse 29.0 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
20000 repetitions
OBSERVE C13, 100.6113782 MHz
DECOUPLE H1, 400.1266027 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 7 hr, 9 min, 47 sec

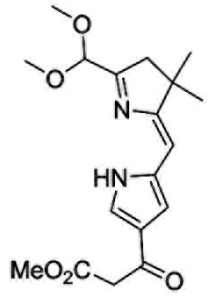


3b

STANDARD PROTON PARAMETERS

Sample Name:
shaofei
Data Collected on:
m300.chem.ncsu.edu-mercury300
Archive directory:
/home/vnmr1/vnmrsys/data
Sample directory:
N-300_calib_20160906_01
FidFile: methylester_dihydrodipyrrin_hnmr

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 10 2016



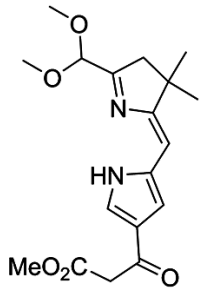
4a

Agilent Technologies

STANDARD PROTON PARAMETERS

Sample Name:
shaofei
Data Collected on:
m300.chem.ncsu.edu-mercury300
Archive directory:
/home/vnmr1/vnmrsys/data
Sample directory:
N-300_calib_20160906_01
FidFile: CARBON

Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 10 2016



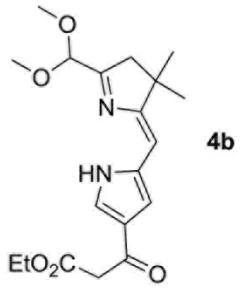
4a

Agilent Technologies

STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc300"

Relax. delay 1.000 sec
Pulse 36.0 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
128 repetitions
OBSERVE H1, 299.7918084 MHz
DATA PROCESSING
FT size 32768
Total time 6 min, 37 sec

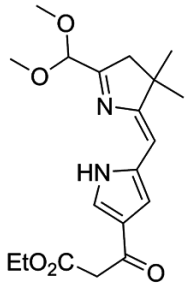


4b

13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "ncsumerc400"

Pulse 29.0 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
50000 repetitions
OBSERVE C13, 100.6113782 MHz
DECOUPLE H1, 400.1266027 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 17 hr, 54 min, 26 sec



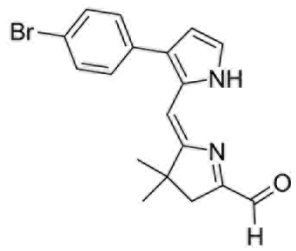
4b

Sample Name:
shaofei_934-7
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:

Sample directory:

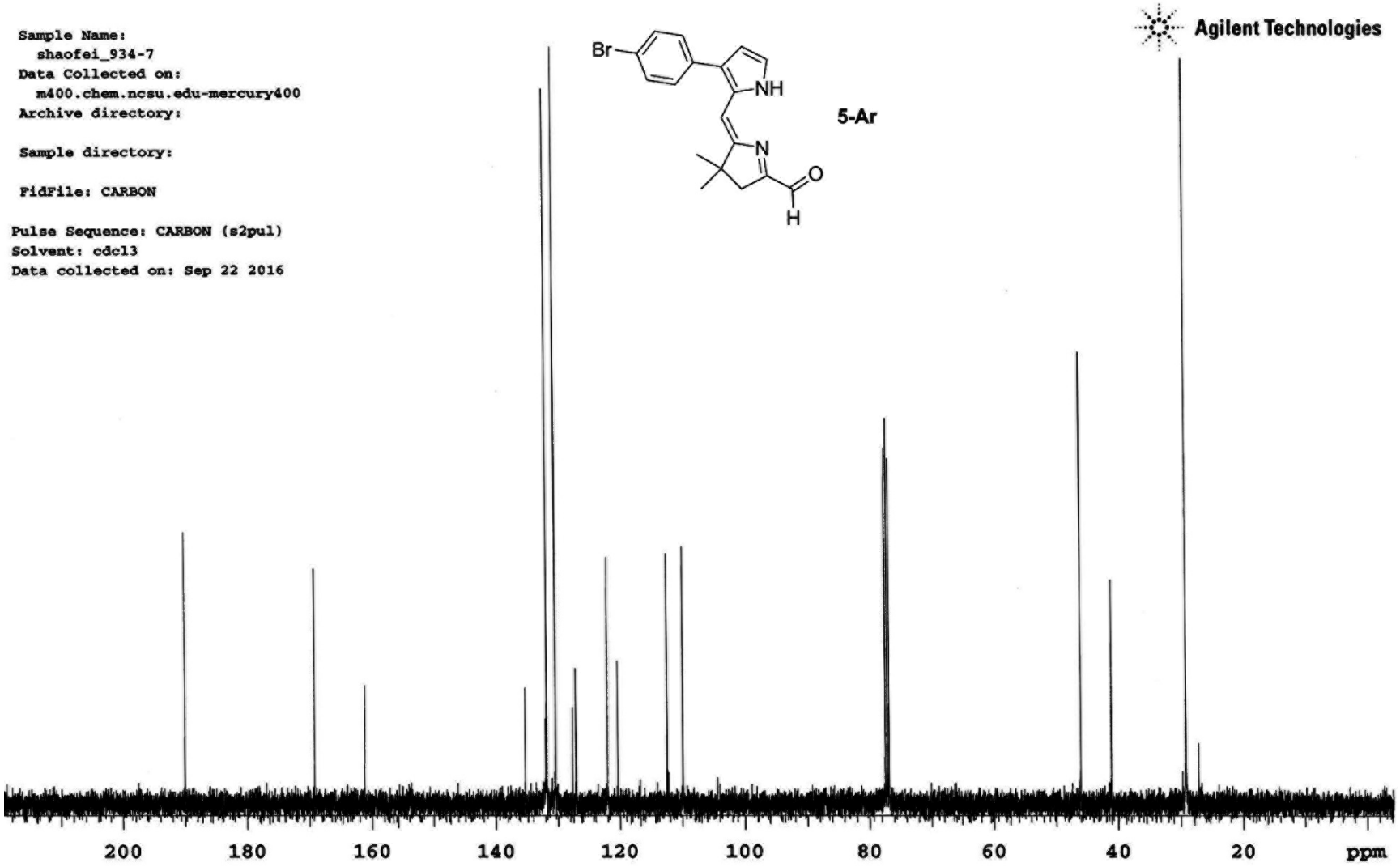
FidFile: 934-7h

Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2016



5-Ar

Agilent Technologies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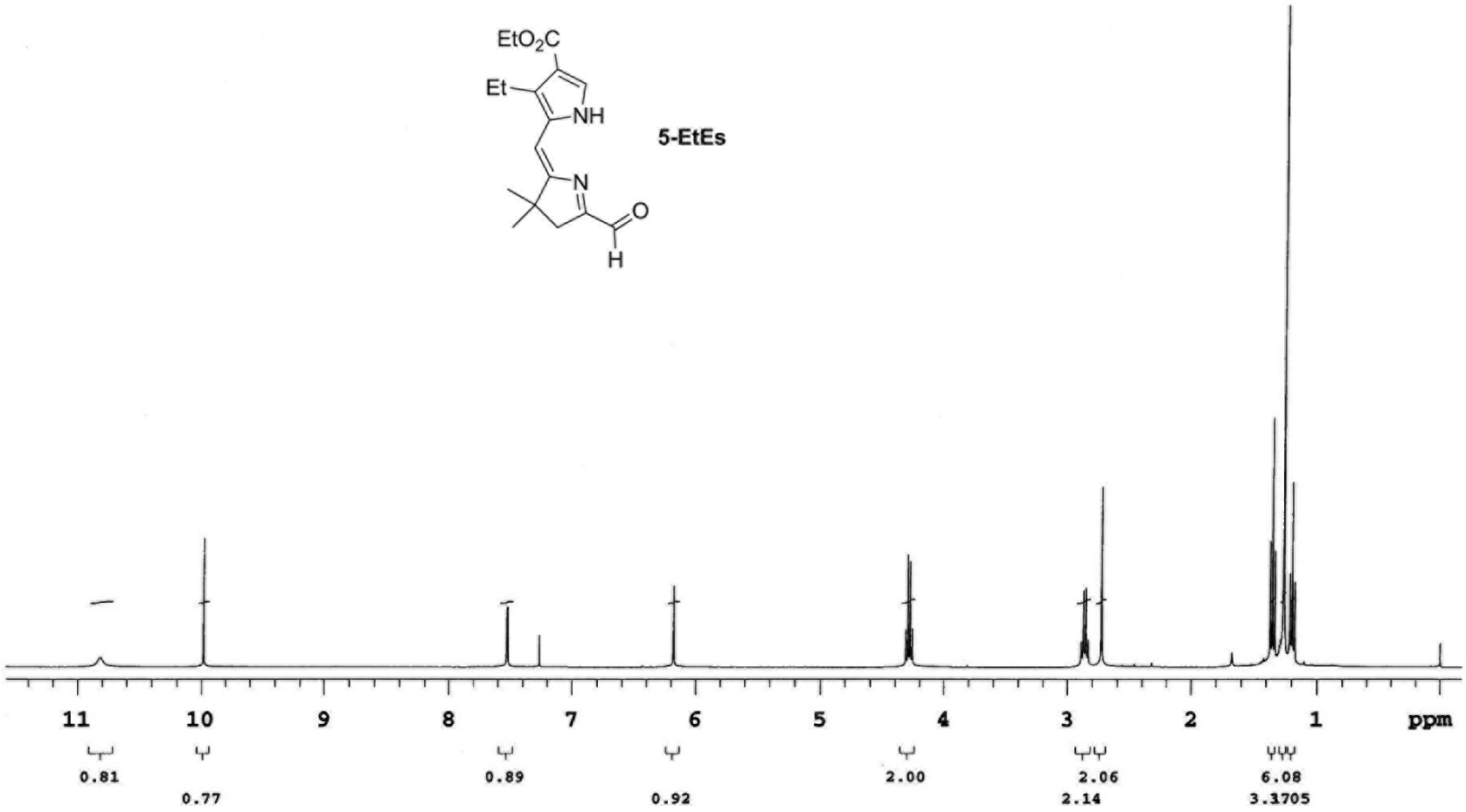
shaofei_934-7
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2016
Agilent Technologies
Br
NH
N
O
H
5-Ar
200
180
160
140
120
100
80
60
40
20
ppm


EtO2C
Et
NH
N
O
H
5-EtEs
11
10
9
8
7
6
5
4
3
2
1
ppm
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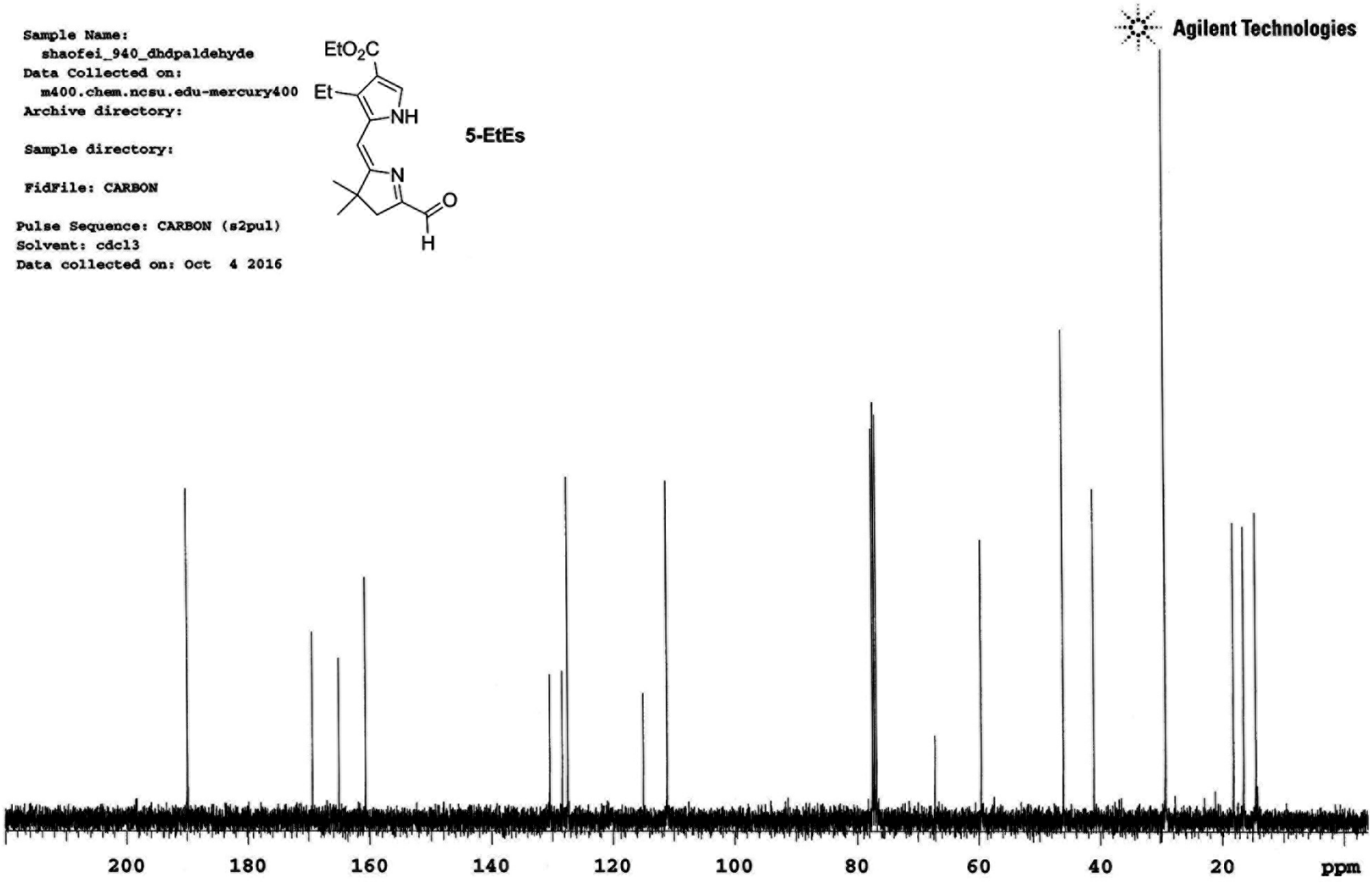
0.89
0.92
2.00
2.06
2.14
6.08
3.1705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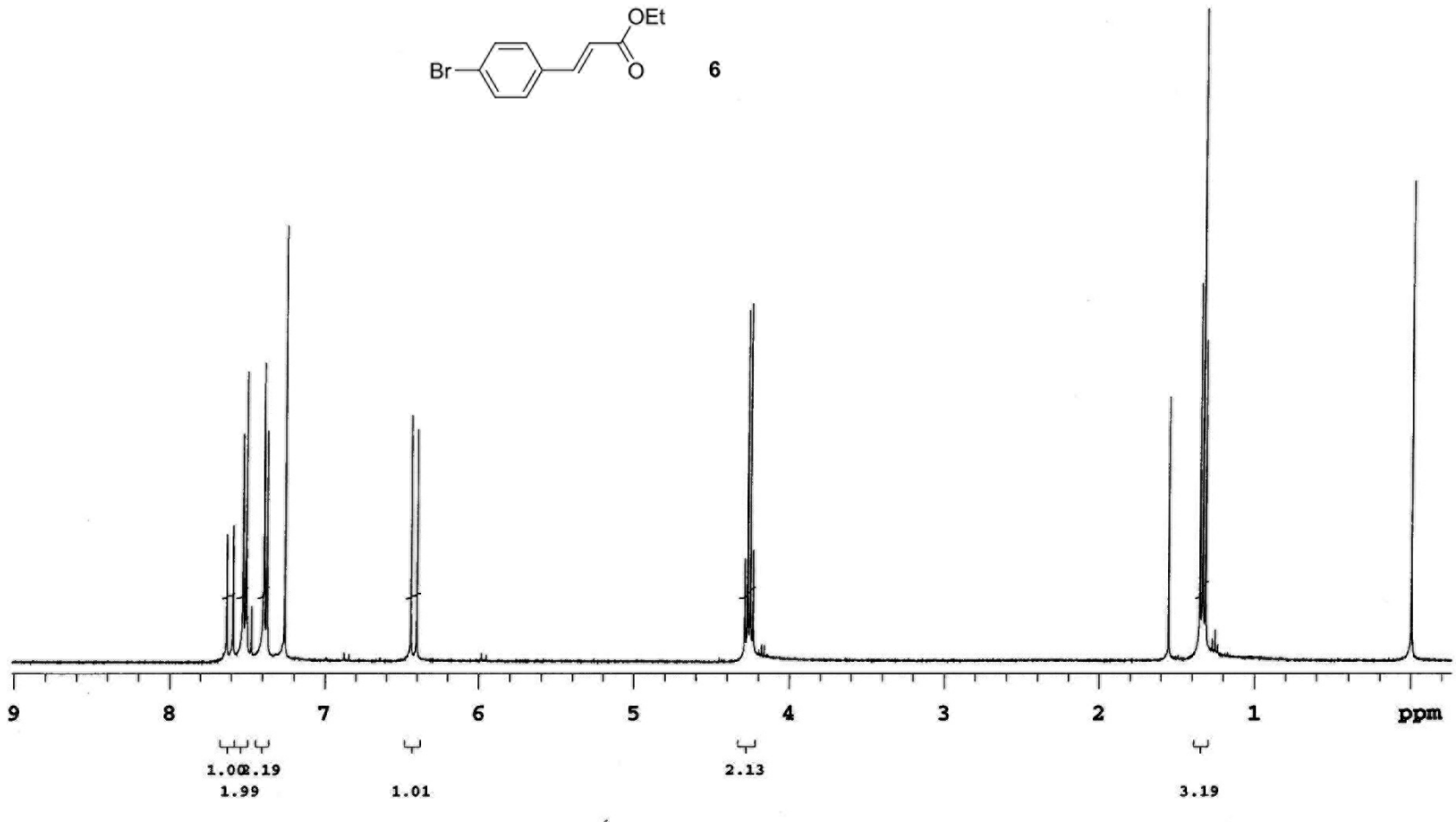
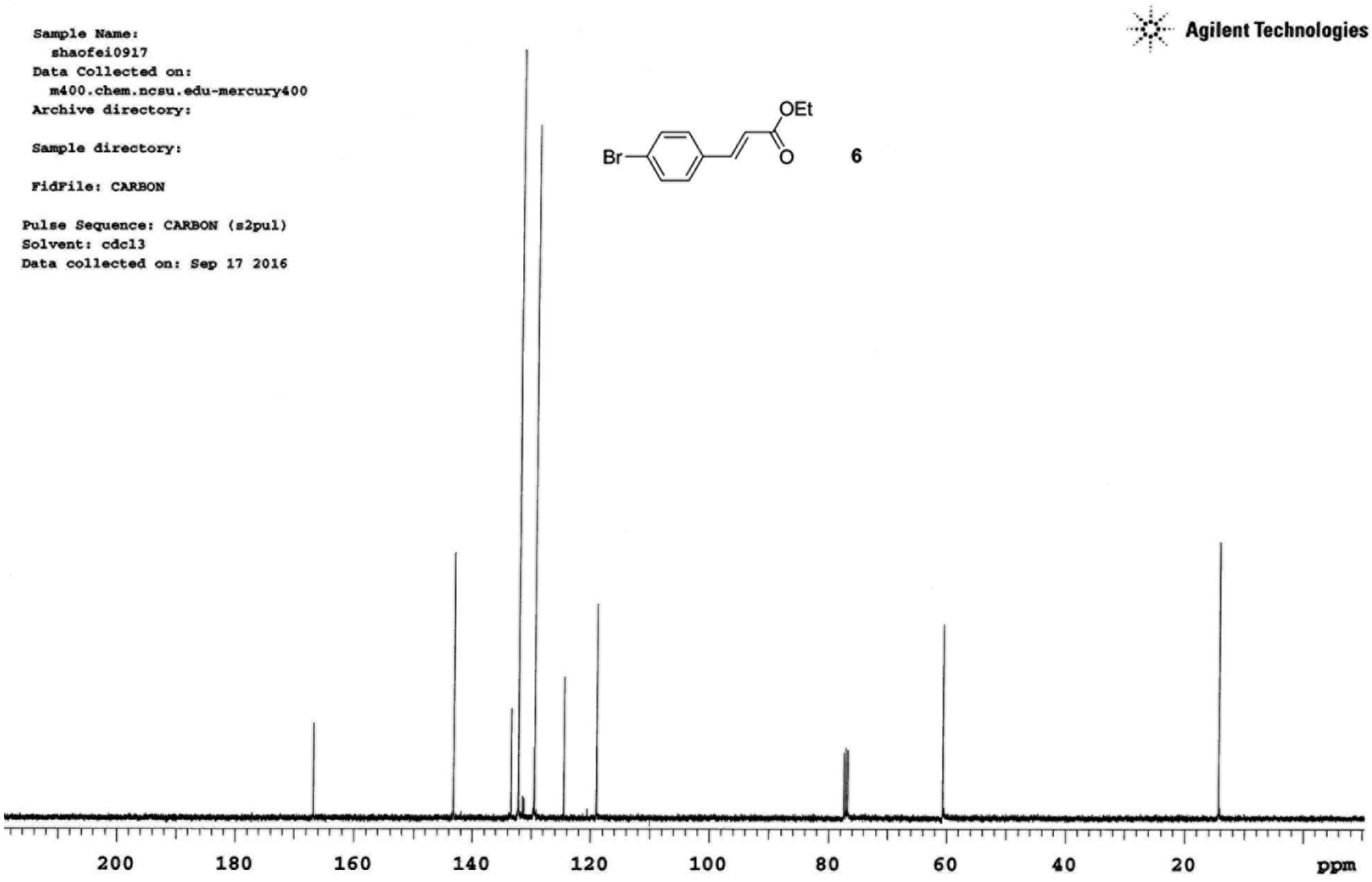
shaofei_940_dhdpaldehyde
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2016
EtO2C
Et
NH
N
O
H
5-EtEs
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm


OEt
Br
O
6
9
8
7
6
5
4
3
2
1
ppm
1.00
2.19
1.99
1.01
2.13
3.19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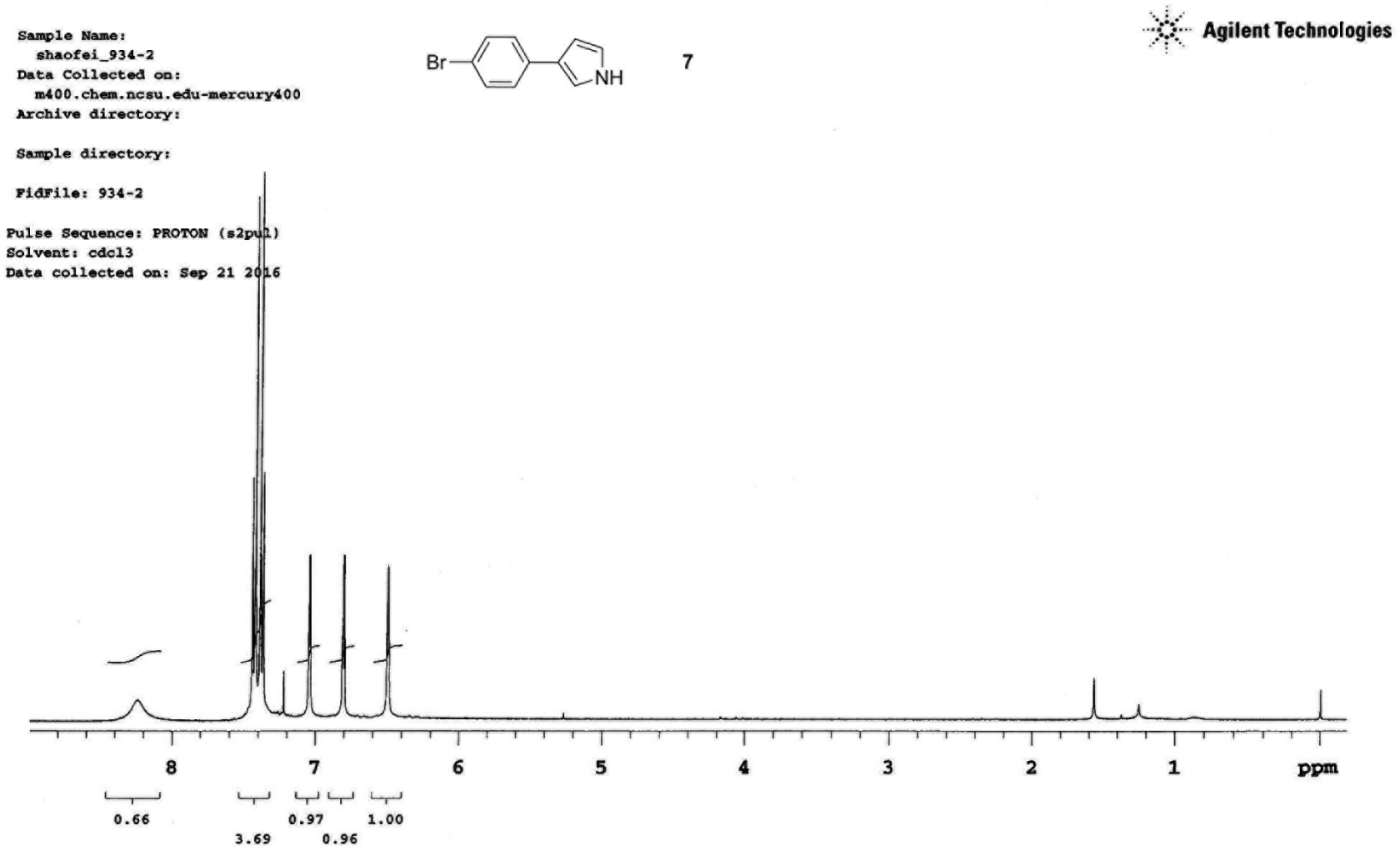
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 17 2016
Agilent Technologies
Br
OEt
O
6
200
180
160
140
120
100
80
60
40
20
ppm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shaofei_934-2
Data Collected on:
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Archive directory:
Sample directory:
FidFile: 934-2
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2016
Br
NH
7
Agilent Technologies
8
7
6
5
4
3
2
1
ppm
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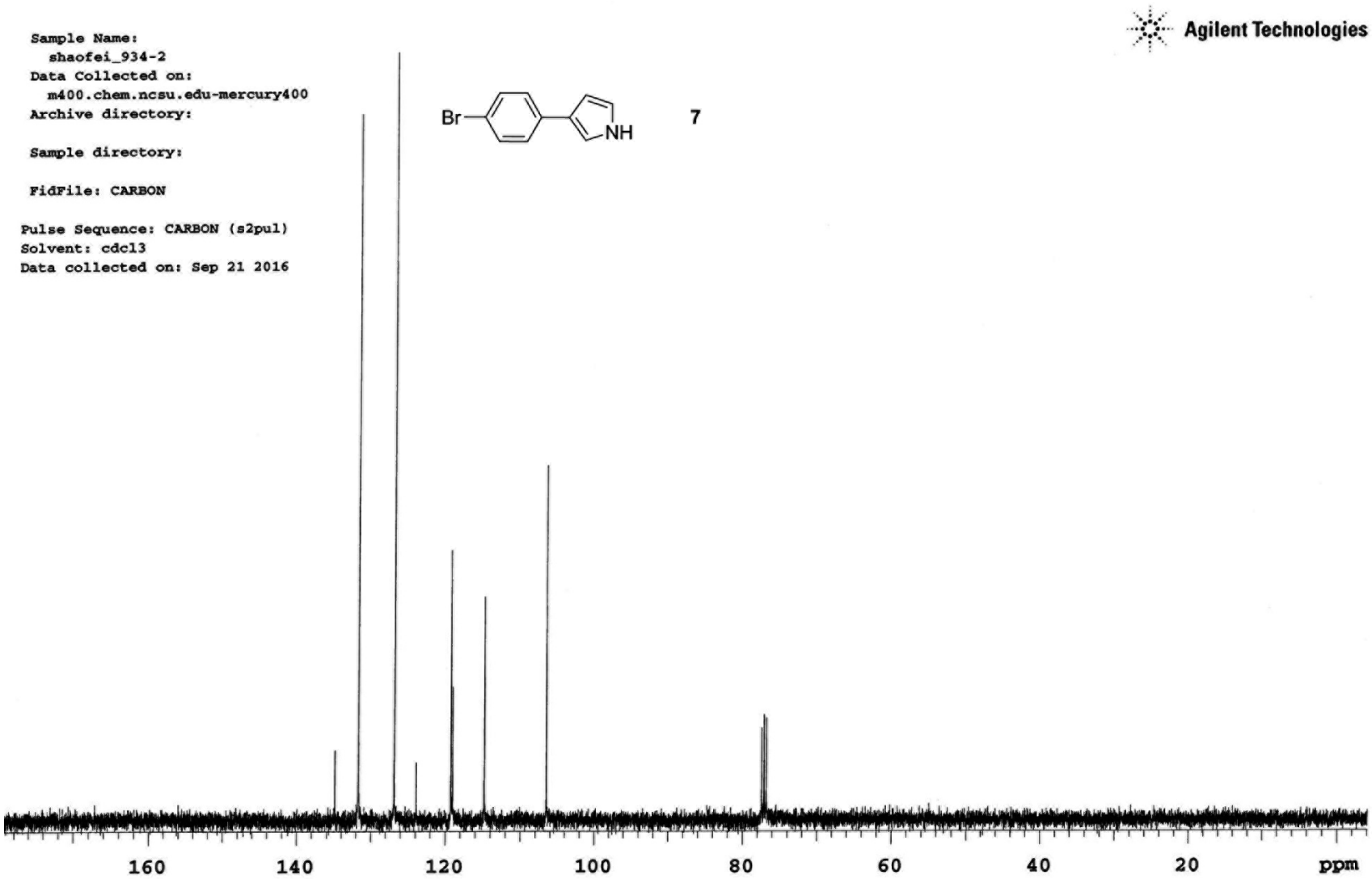
3.69
0.97
0.96
1.00


Agilent Technologies
Sample Name:
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Data Collected on:
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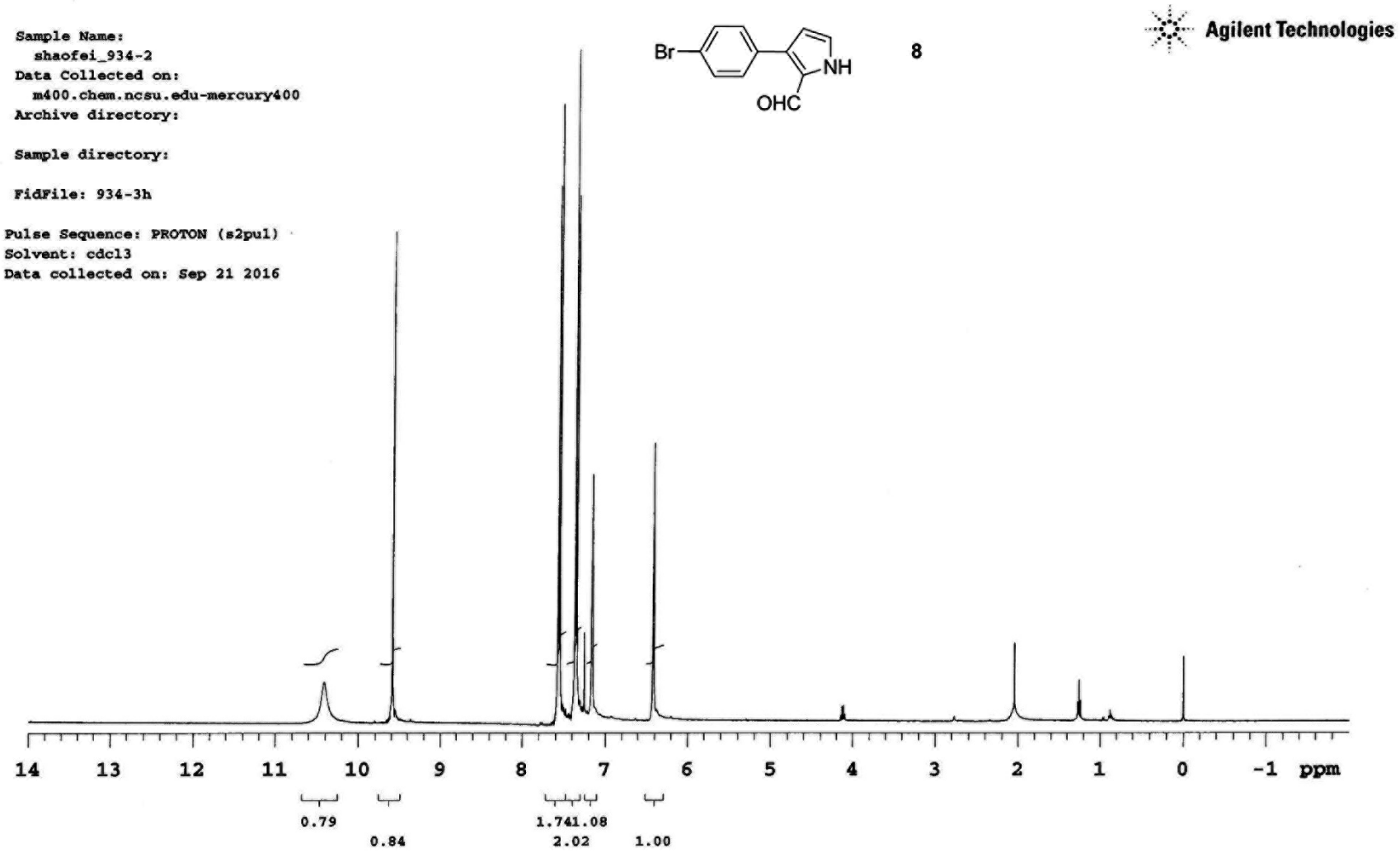
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2016
Br
NH
7
160
140
120
100
80
60
40
20
ppm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shaofei_934-2
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: 934-3h
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2016
Br
NH
OHC
8
Agilent Technologies
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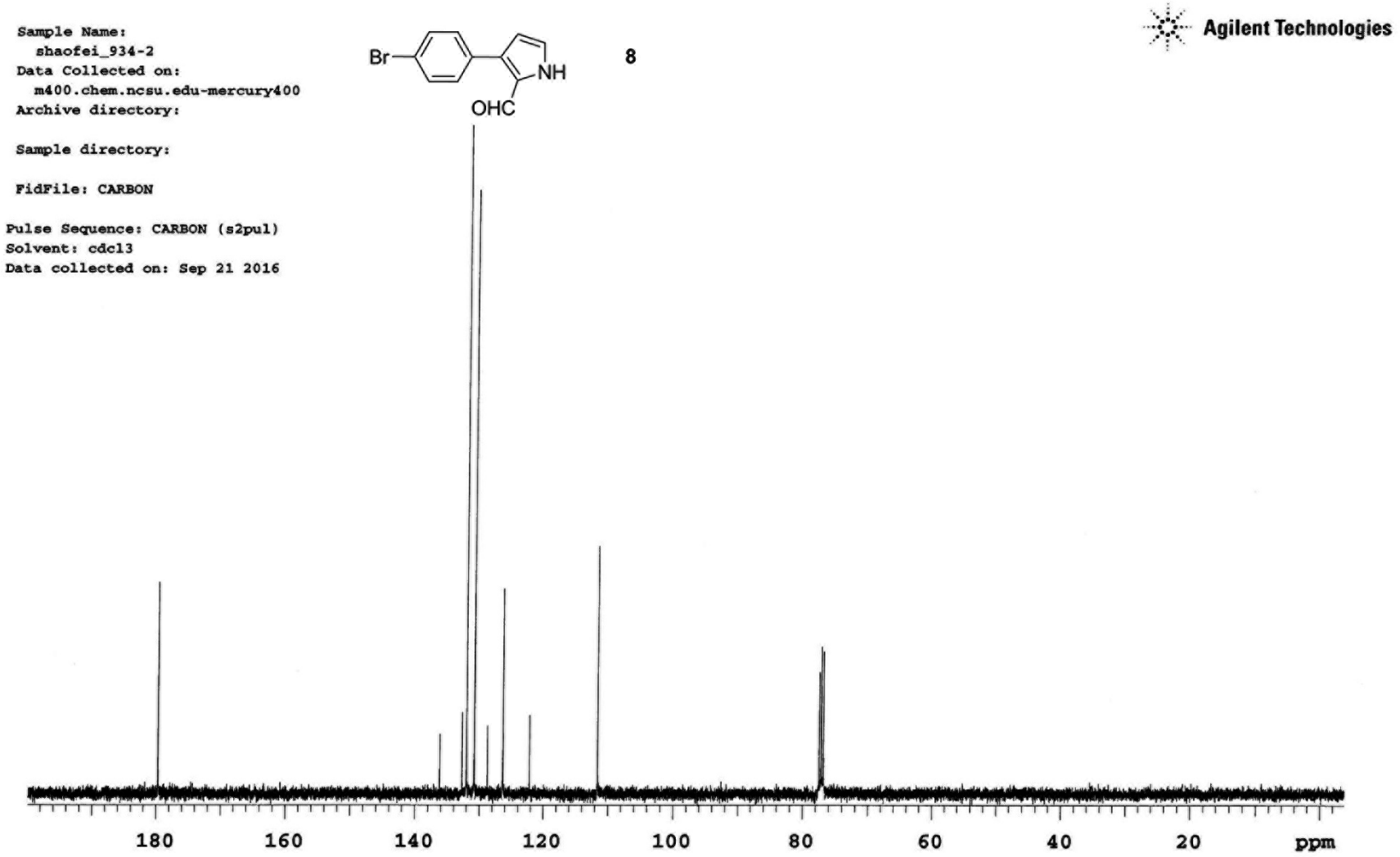
0.79
0.84
1.74
1.08
2.02
1.00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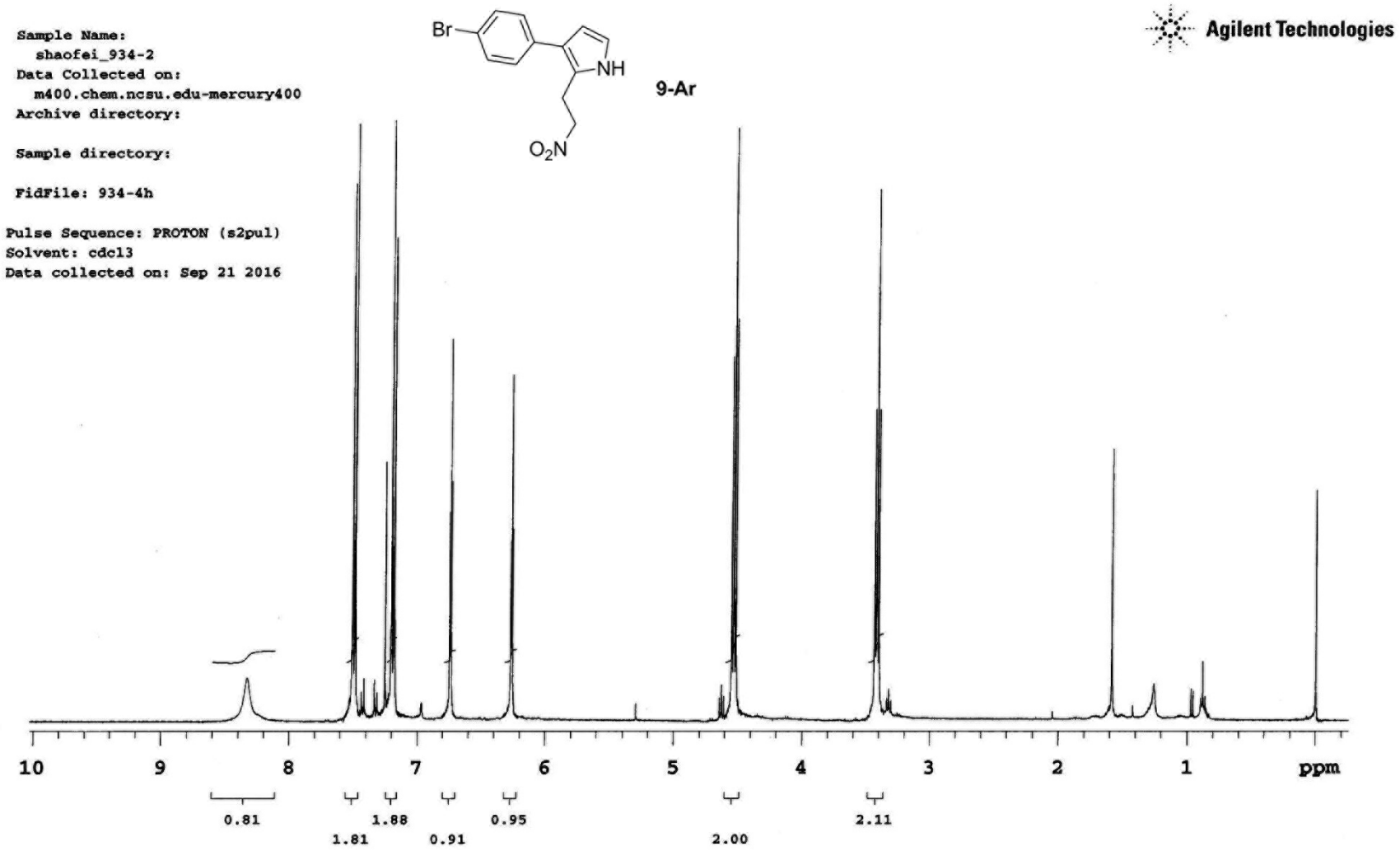
shaofei_934-2
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2016
Br
NH
OHC
8
Agilent Technologies
180
160
140
120
100
80
60
40
20
ppm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Data Collected on:
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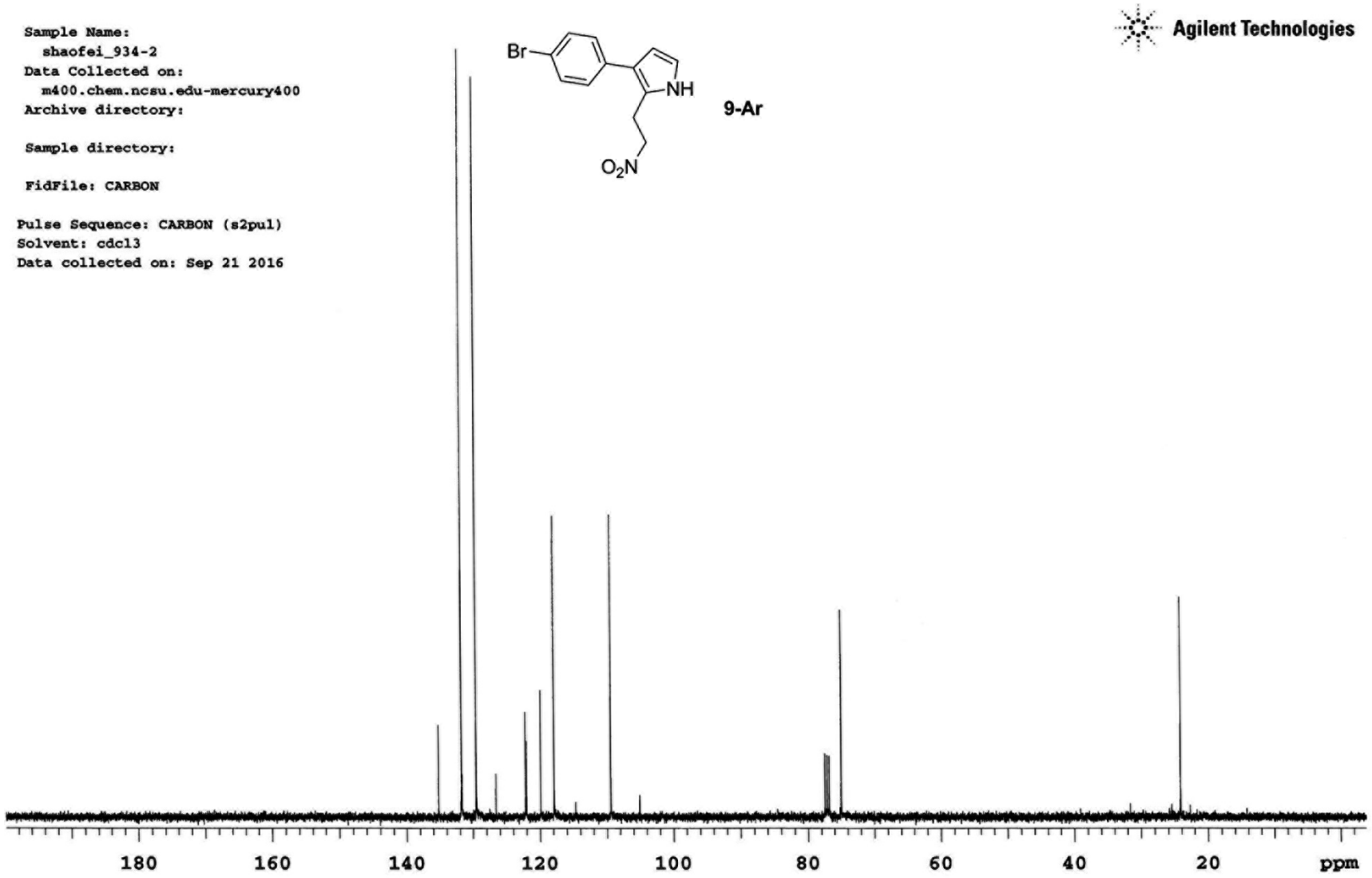
Archive directory:
Sample directory:
FidFile: 934-4h
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2016
Br
NH
O2N
9-Ar
Agilent Technologies
10
9
8
7
6
5
4
3
2
1
ppm
0.81
1.81
1.88
0.91
0.95
2.00
2.11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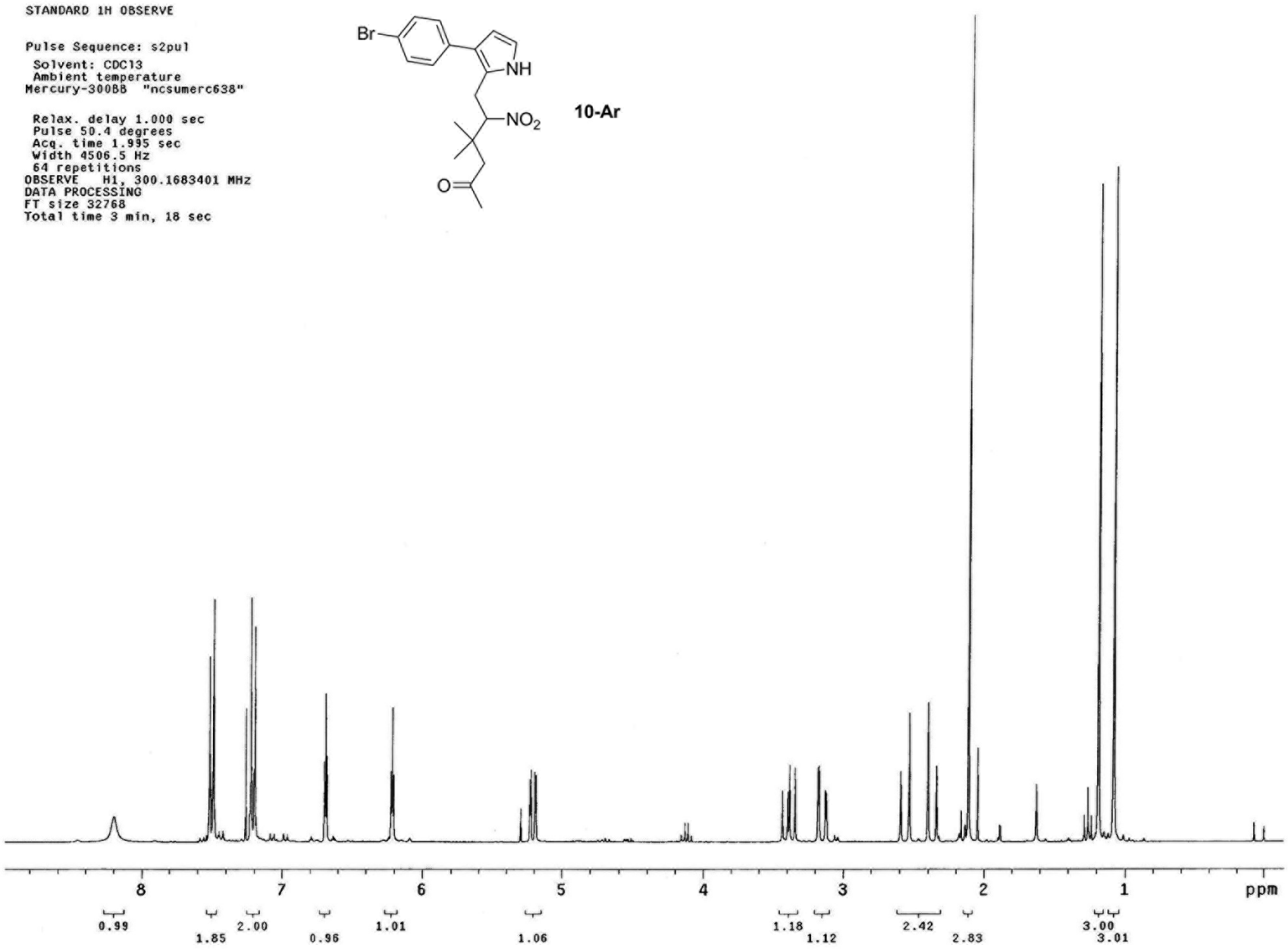
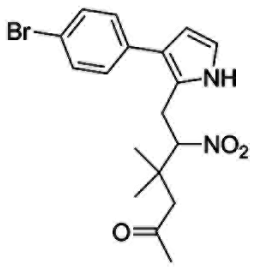
shaofei_934-2
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2016
Br
NH
O2N
9-Ar
Agilent Technologies
180
160
140
120
100
80
60
40
20
ppm


STANDARD 1H OBSERVE
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "ncsumerc638"
Relax. delay 1.000 sec
Pulse 50.4 degrees
Acq. time 1.995 sec
Width 4506.5 Hz
64 repetitions
OBSERVE H1, 300.1683401 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 18 sec
Br
NH
NO2
O
10-Ar
8
7
6
5
4
3
2
1
ppm
0.99
1.85
2.00
0.96
1.01
1.06
1.18
1.12
2.42
2.83
3.00
3.01

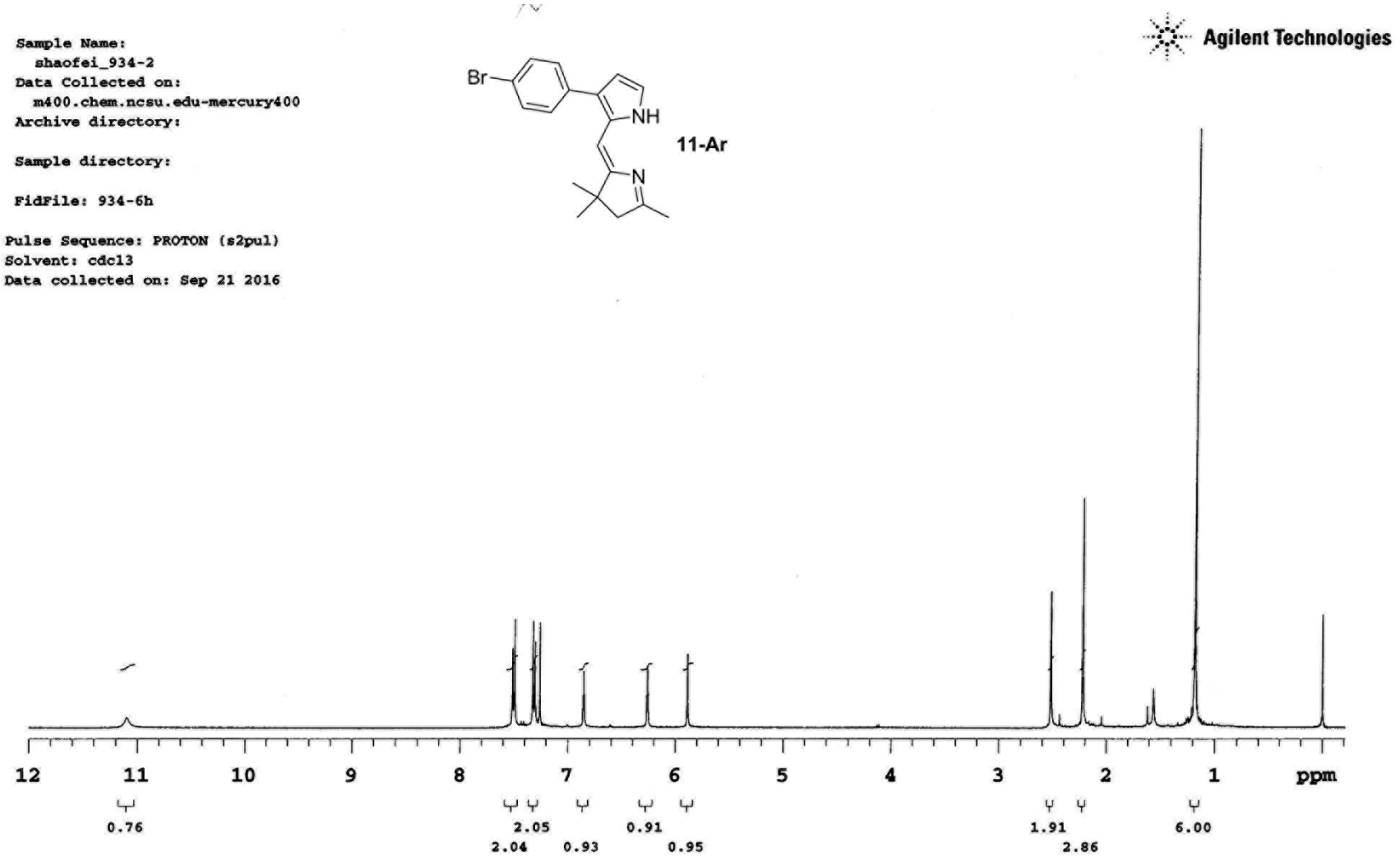
13C OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-400BB "ncsumerc400"

Pulse 81.2 degrees
Acq. time 1.199 sec
Width 25000.0 Hz
4096 repetitions
OBSERVE C13, 100.6113782 MHz
DECOUPLE H1, 400.1266027 MHz
Power 44 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 1 hr, 40 min, 6 sec



10-Ar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Data Collected on:
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Archive directory:
Sample directory:
FidFile: 934-6h
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2016
Agilent Technologies
Br
NH
N
11-Ar
12
11
10
9
8
7
6
5
4
3
2
1
ppm
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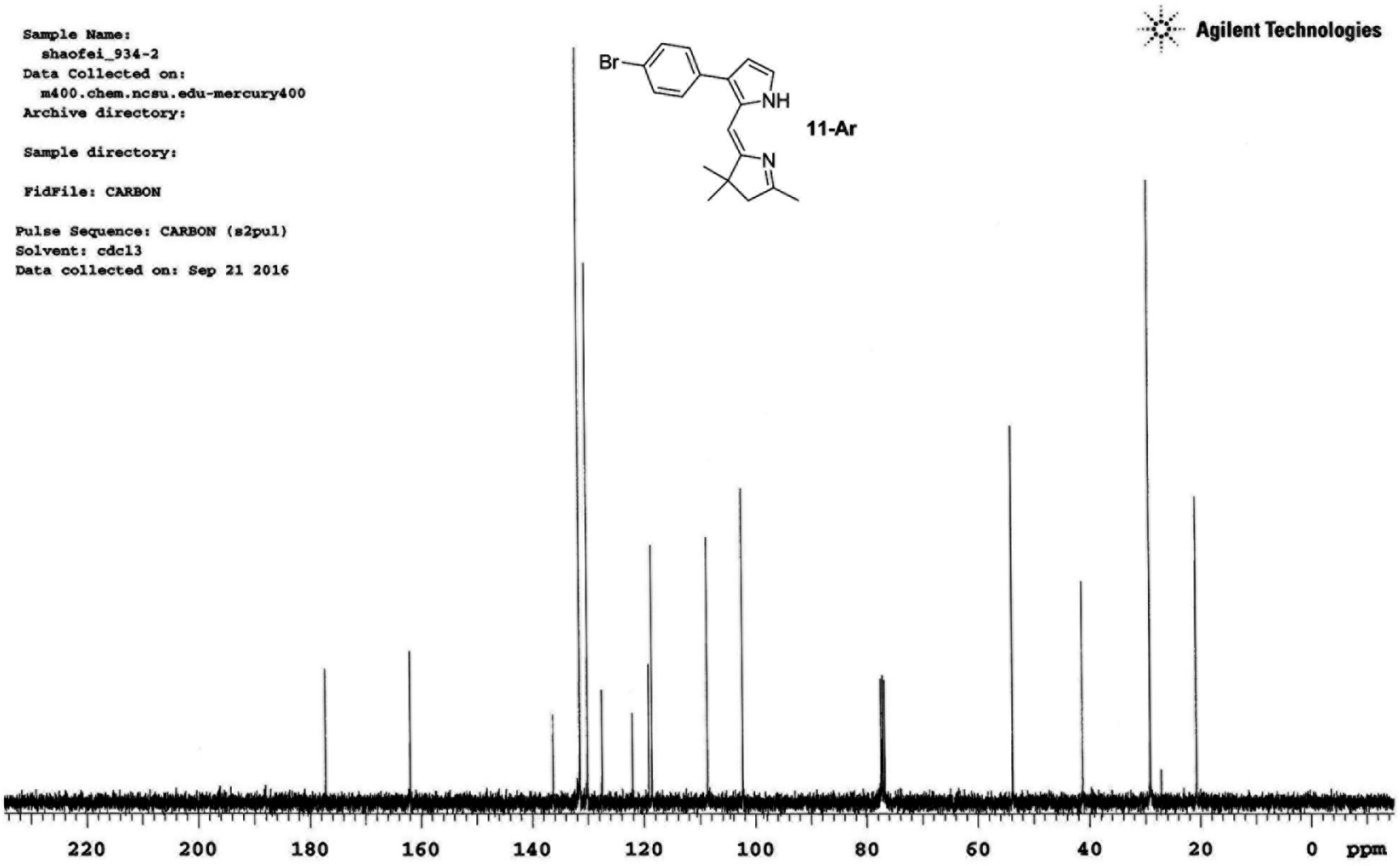
2.05
0.93
0.91
0.95
1.91
2.86
6.00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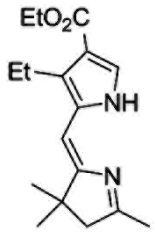
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 21 2016
Agilent Technologies
Br
NH
N
11-Ar
220
200
180
160
140
120
100
80
60
40
20
0
ppm

Sample Name:
shaofei_aldehyde_EtEs
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:

Sample directory:

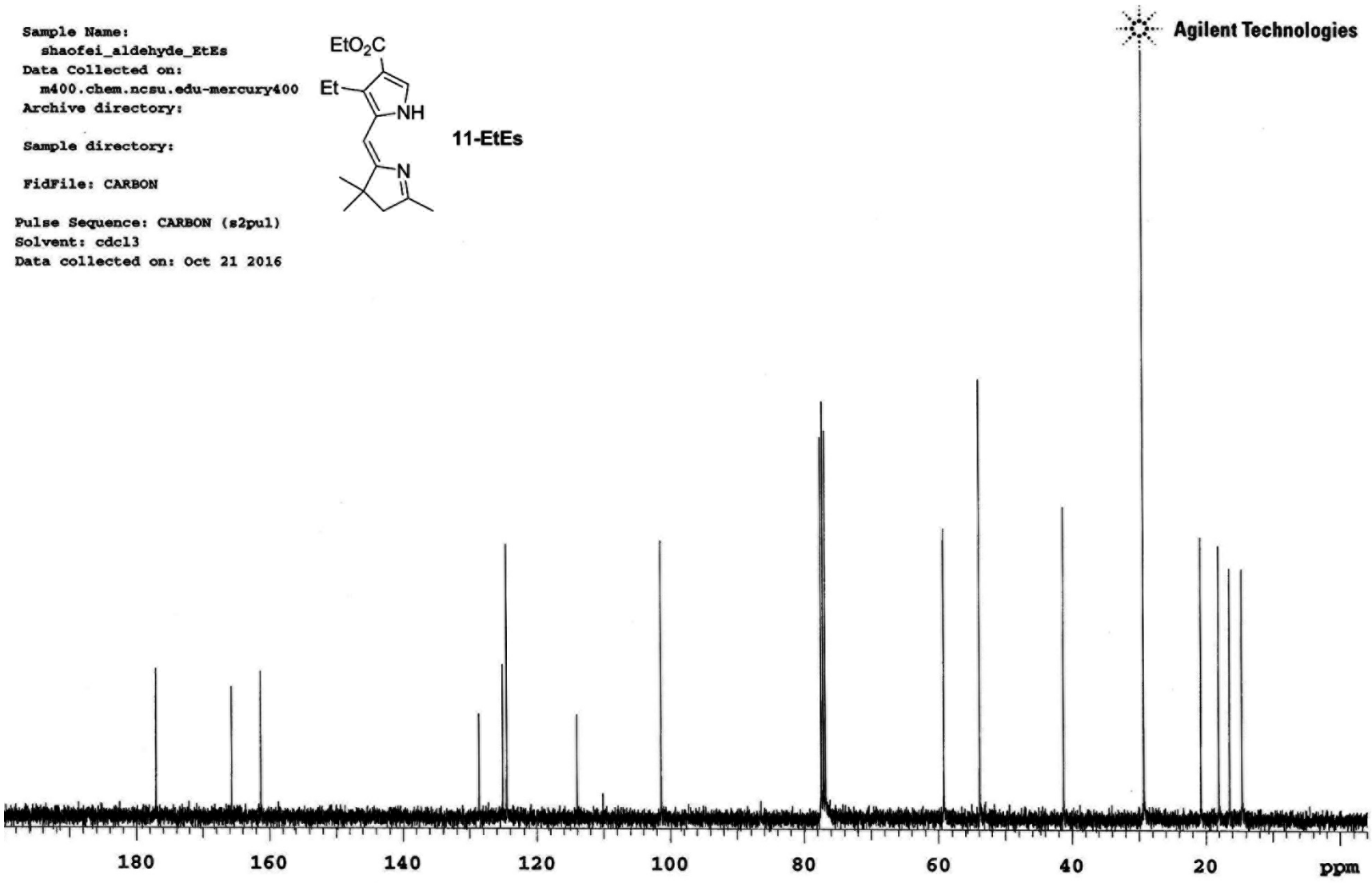
FidFile: dihydrodipyrrin_Me_EtEs_HNMR

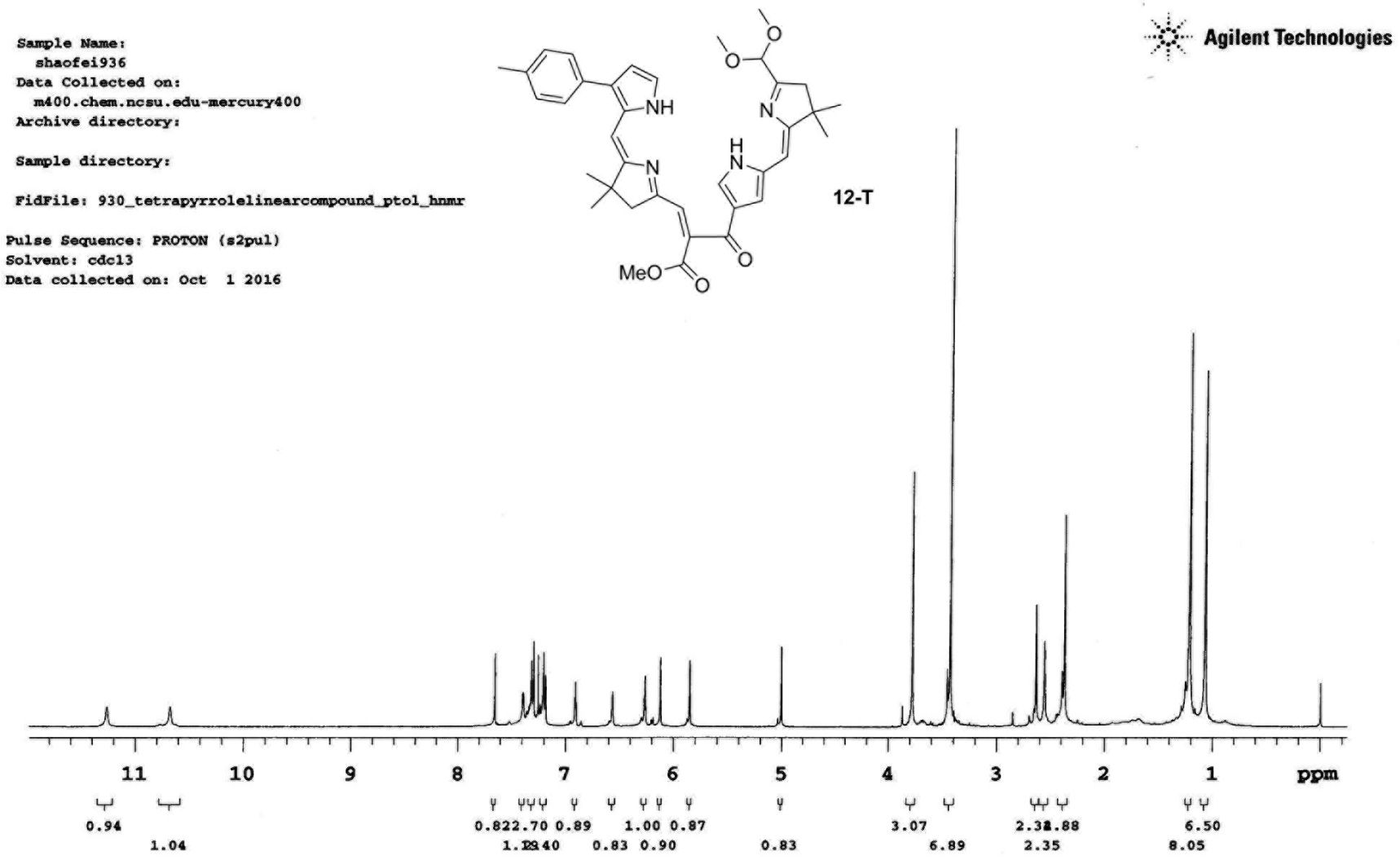
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 21 2016

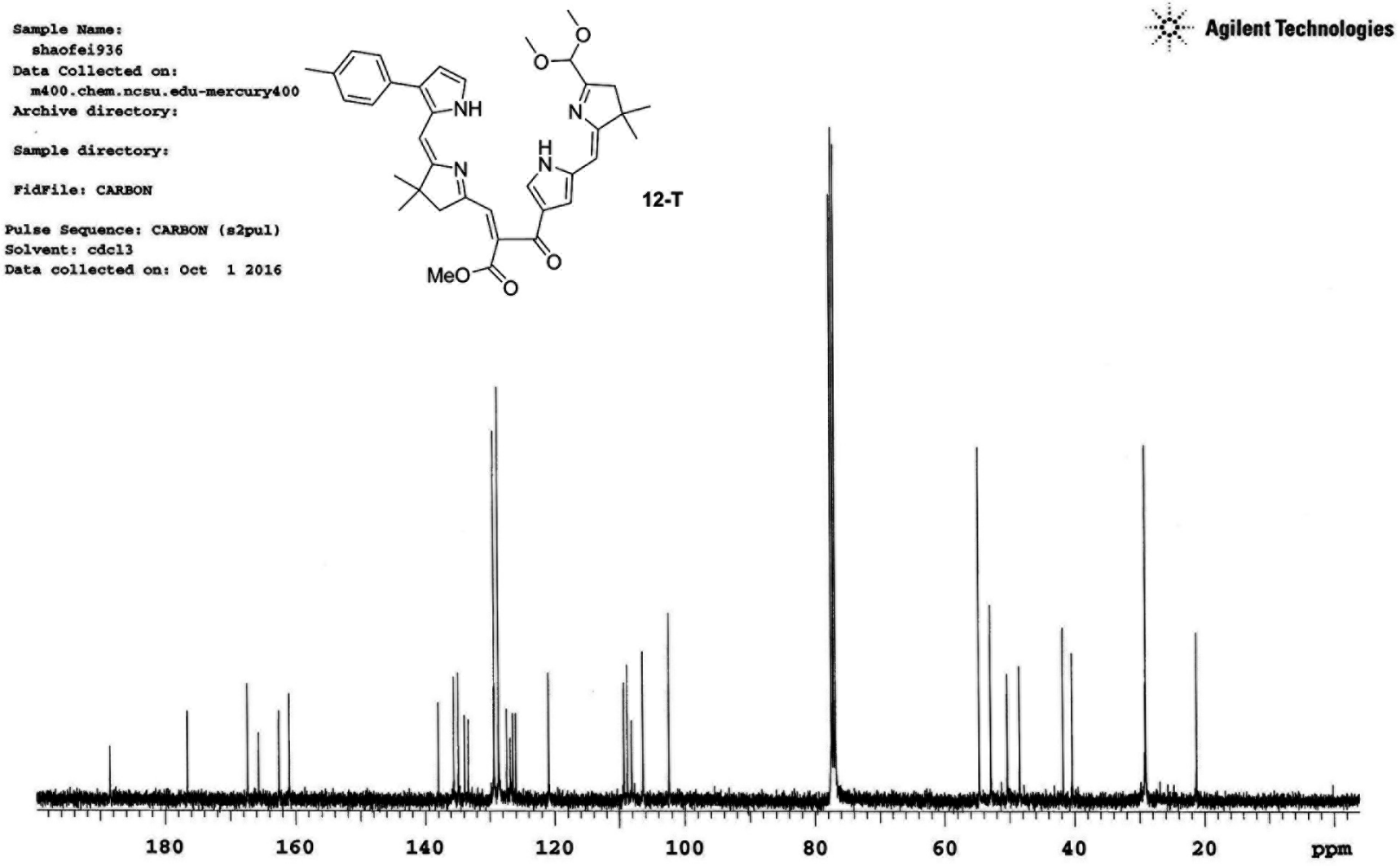


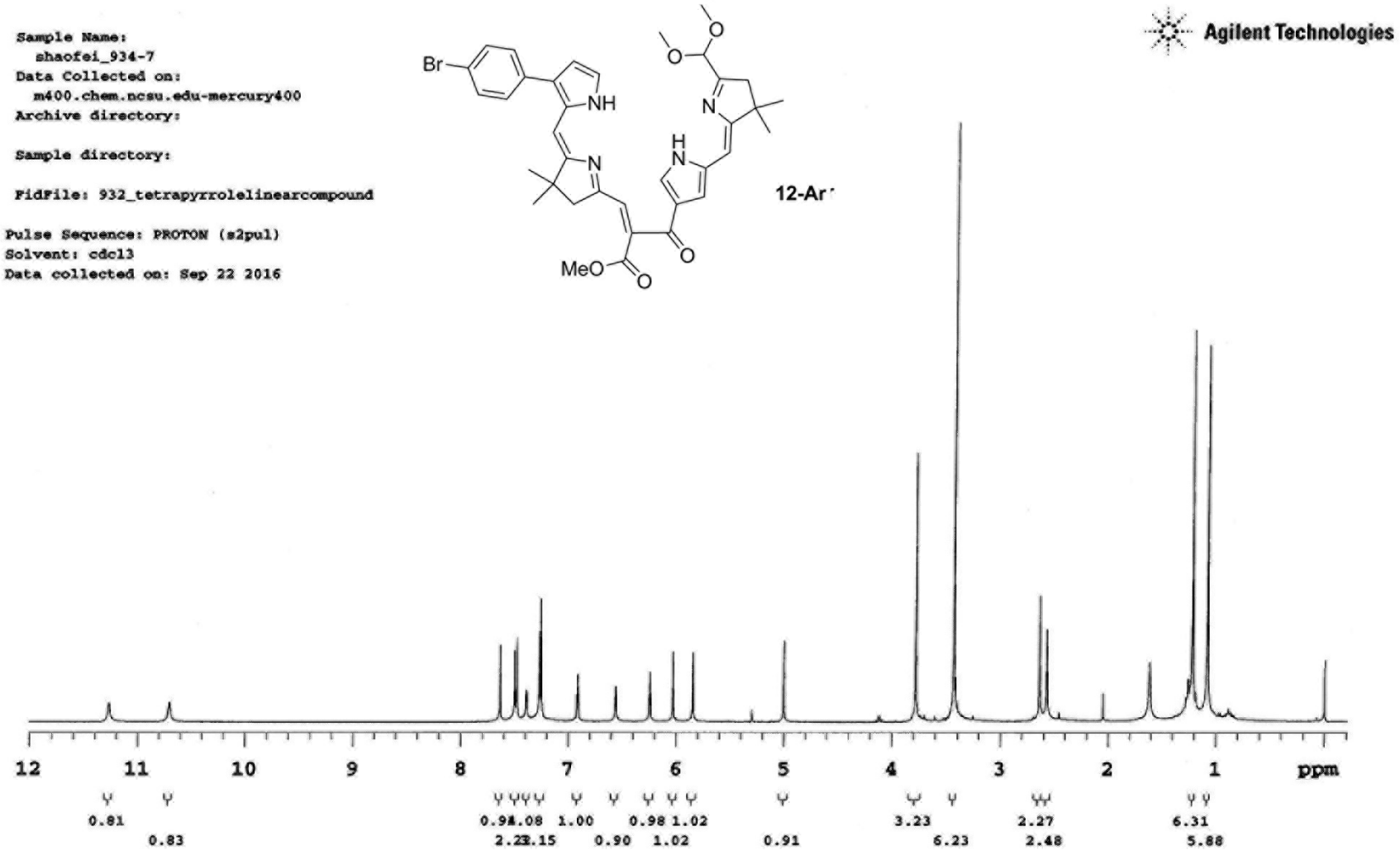
11-EtEs

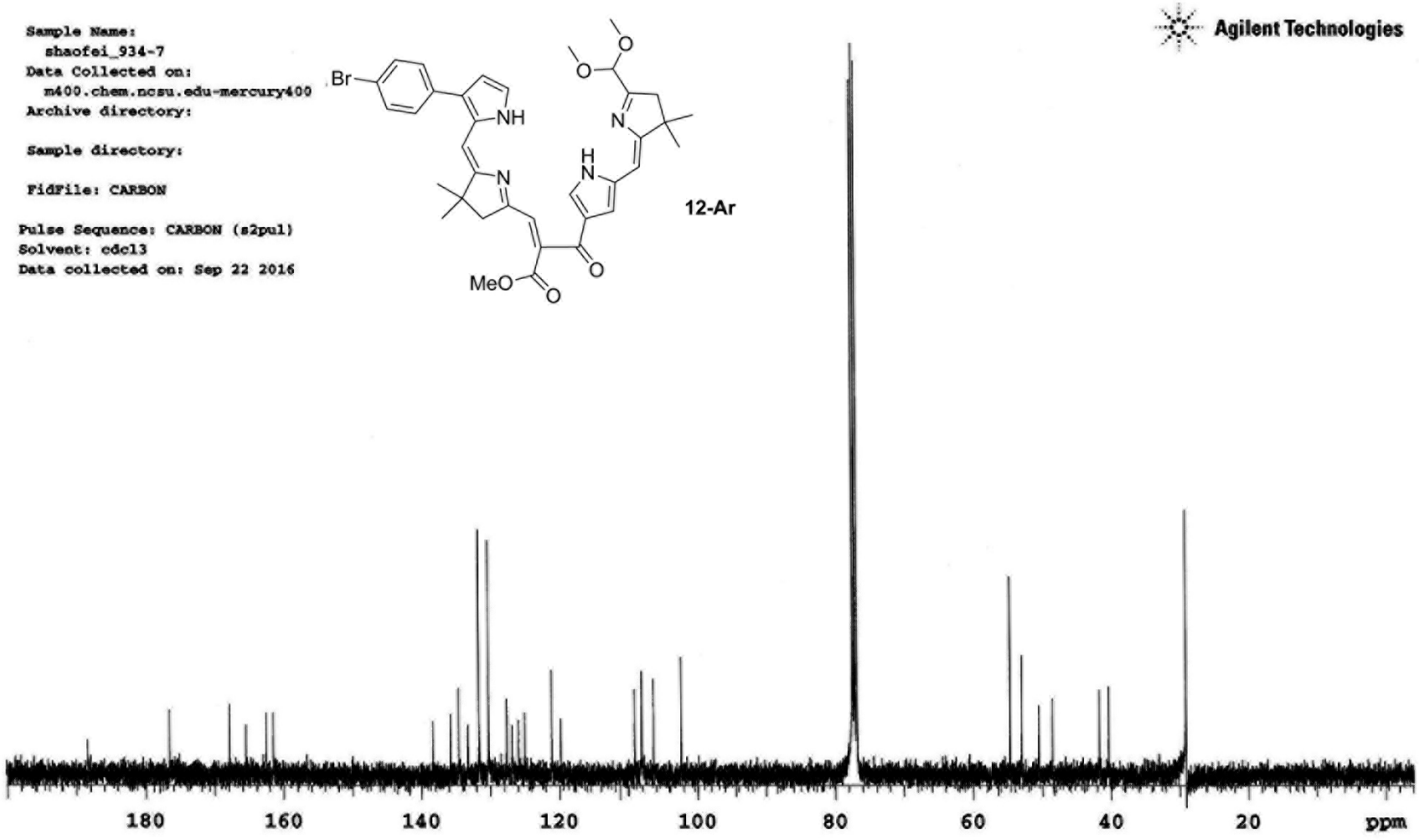
Agilent Technologies

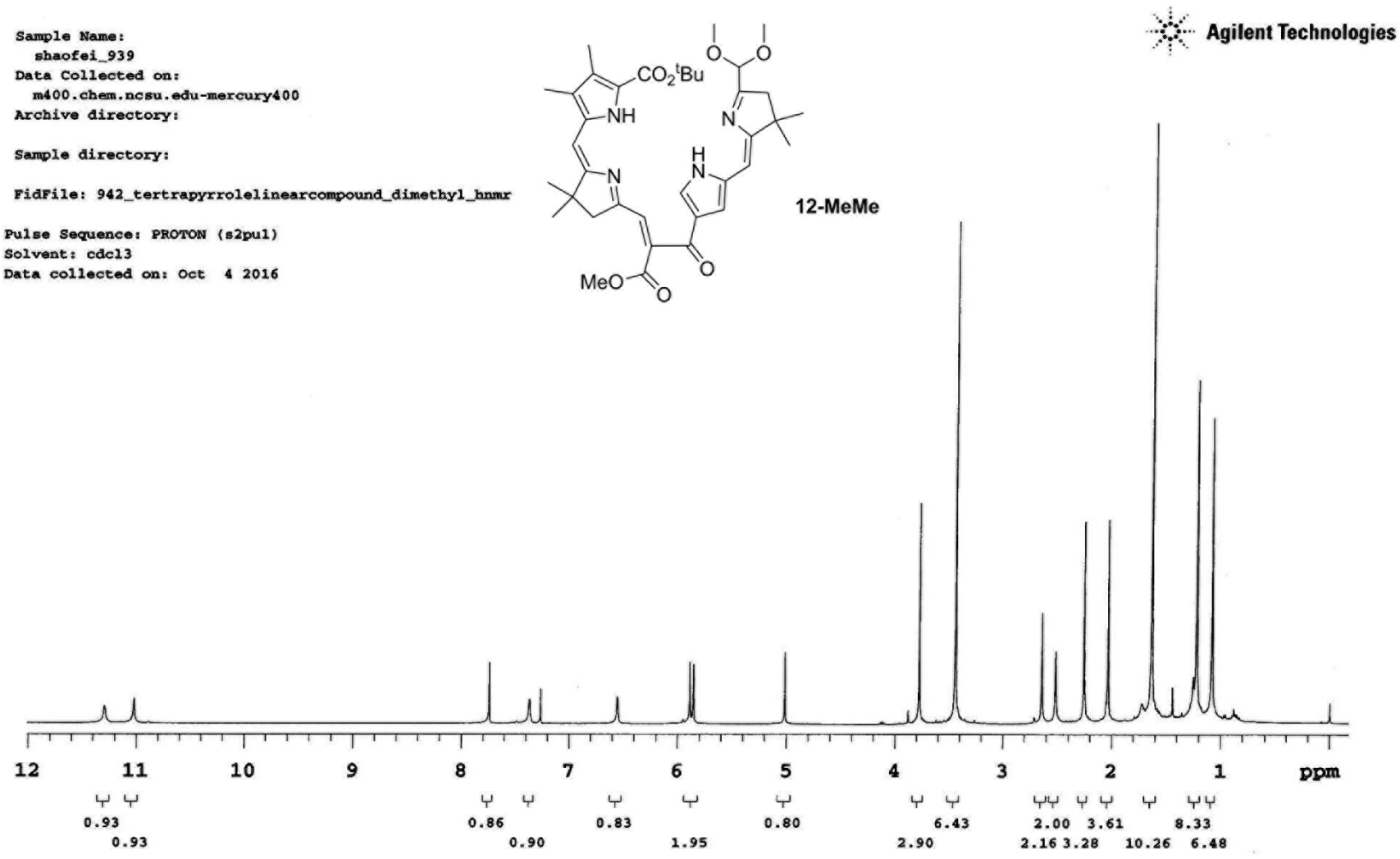

Sample Name:
shaofei_aldehyde_EtEs
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 21 2016
EtO2C
Et
NH
N
11-EtEs
Agilent Technologies
180
160
140
120
100
80
60
40
20
ppm


Sample Name:
shaofei936
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: 930_tetrapyrrolelinearcompound_ptol_hnmr
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2016
Agilent Technologies
12-T
11
10
9
8
7
6
5
4
3
2
1
ppm
0.94
1.04
0.82
2.70
0.89
1.00
0.87
0.83
0.90
0.83
3.07
6.89
2.35
8.05
6.50


Sample Name:
shaofei936
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 1 2016
Agilent Technologies
12-T
NH
N
H
N
N
O
O
O
O
MeO
O
180
160
140
120
100
80
60
40
20
ppm


Sample Name:
shaofei_934-7
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: 932_tetrapyrrolelinearcompound
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2016
Agilent Technologies
12-Ar'
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.81
0.83
0.94
1.08
1.00
0.98
1.02
2.23
3.15
0.90
1.02
0.91
3.23
6.23
2.27
2.48
6.31
5.88


Sample Name:
shaofei_934-7
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Sep 22 2016
Agilent Technologies
Br
NH
N
H
N
O
O
O
N
MeO
O
O
12-Ar
180
160
140
120
100
80
60
40
20
ppm

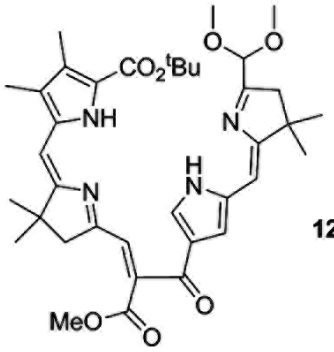

Sample Name:
shaofei_939
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: 942_tertrapyrrolelinearcompound_dimethyl_hnmr
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2016
Agilent Technologies
12-MeMe
12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.93 0.93 0.86 0.90 0.83 1.95 0.80 2.90 6.43 2.00 2.16 3.28 3.61 10.26 8.33 6.48

Sample Name:
shaofei_939
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:

Sample directory:

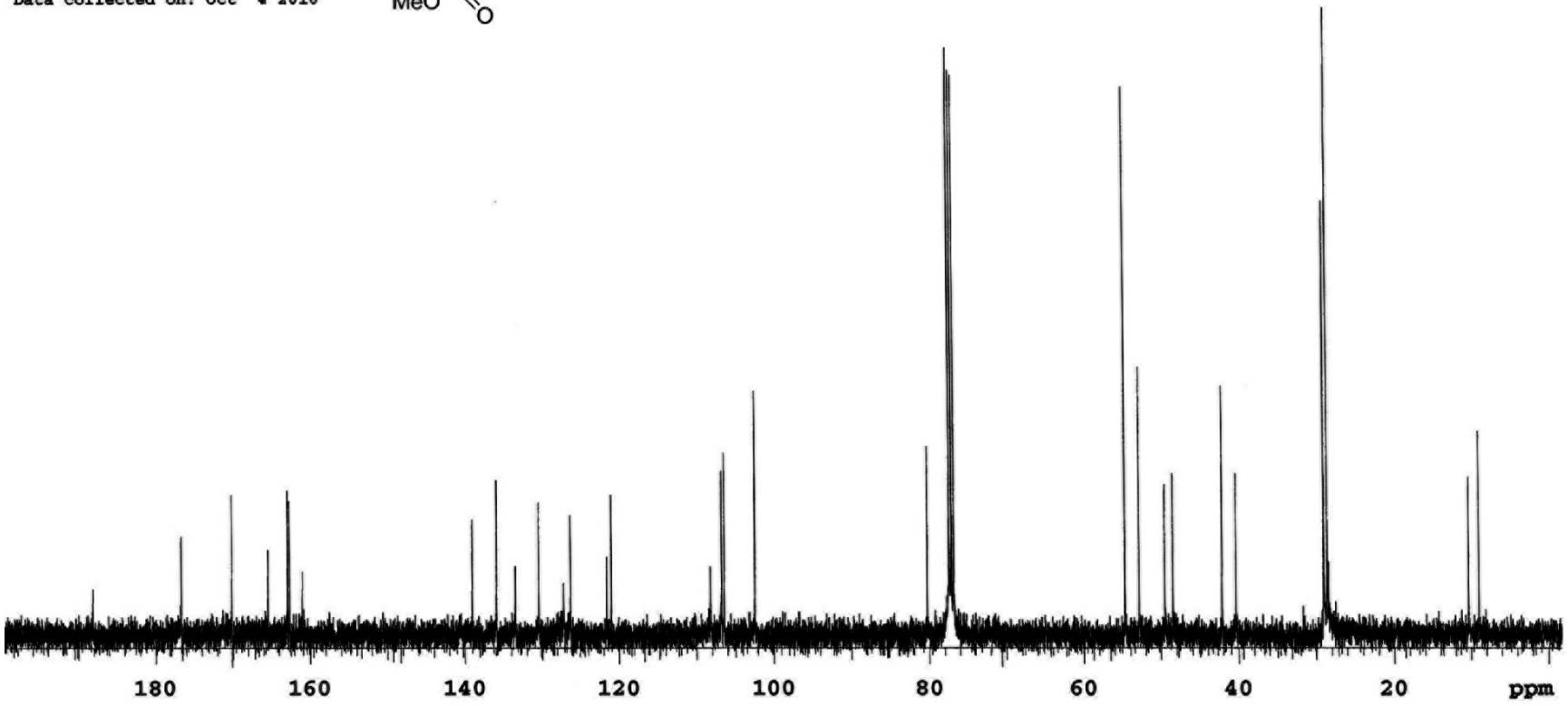
FidFile: CARBON

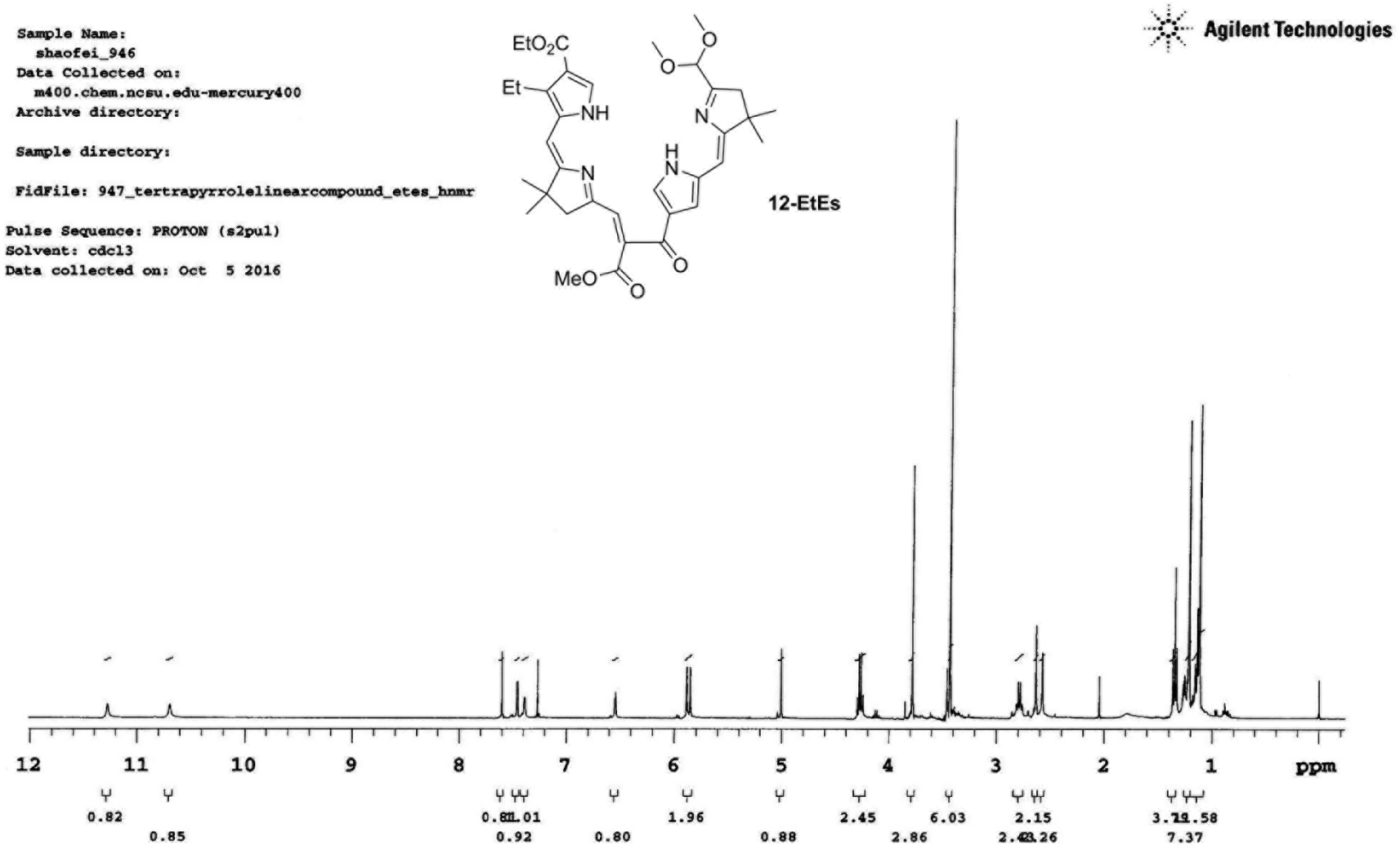
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 4 2016

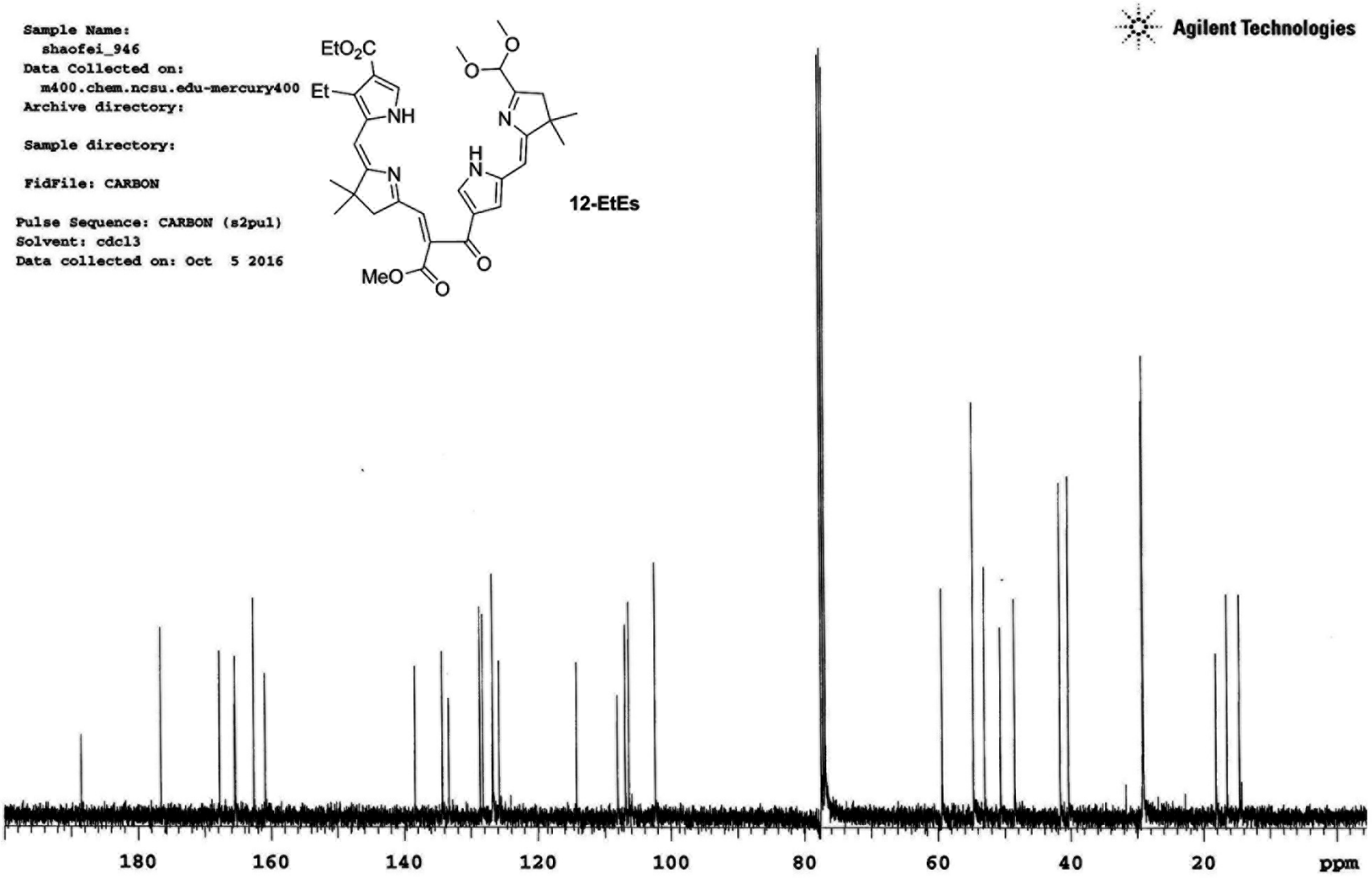


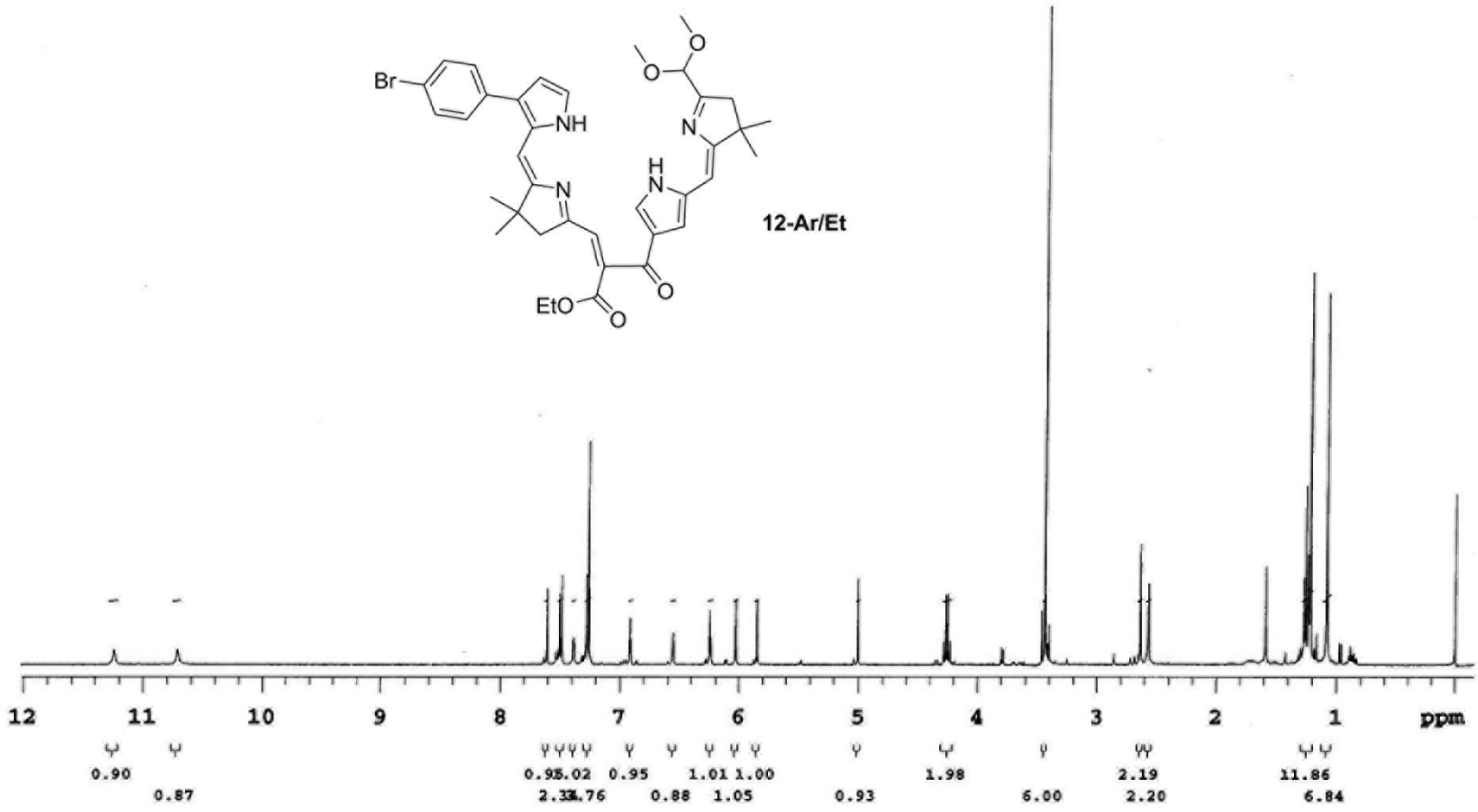
12-MeMe

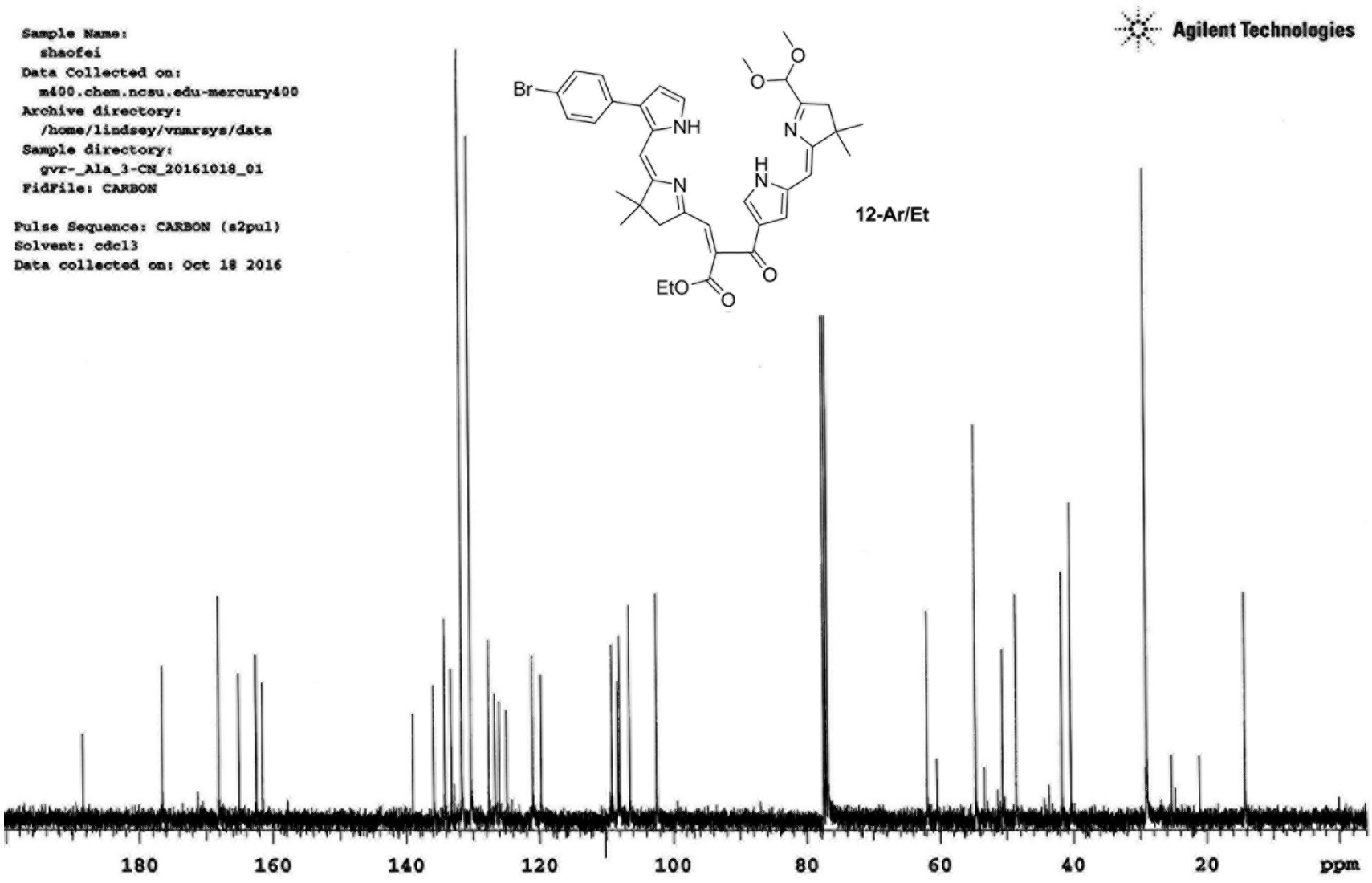
Agilent Technologies

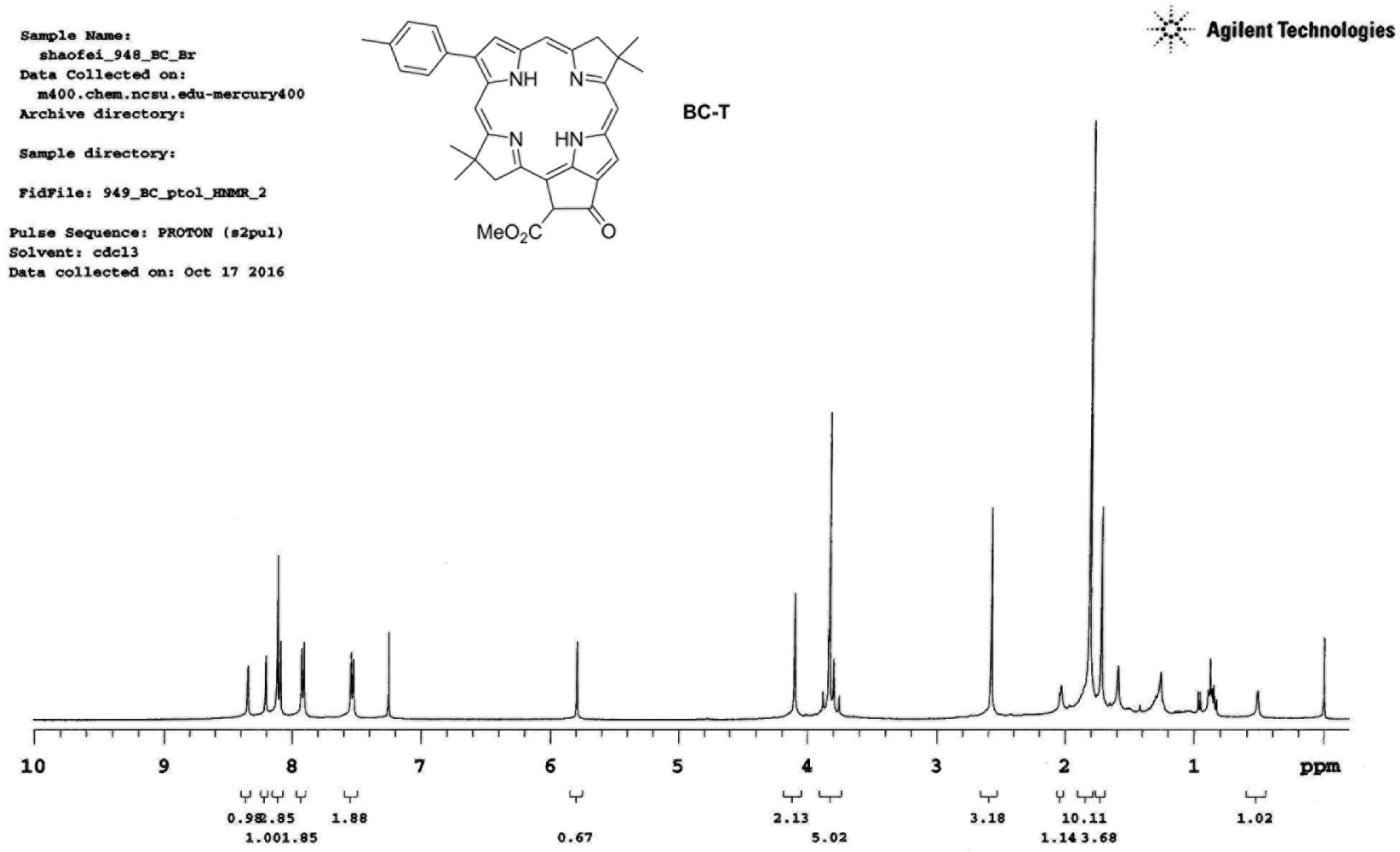


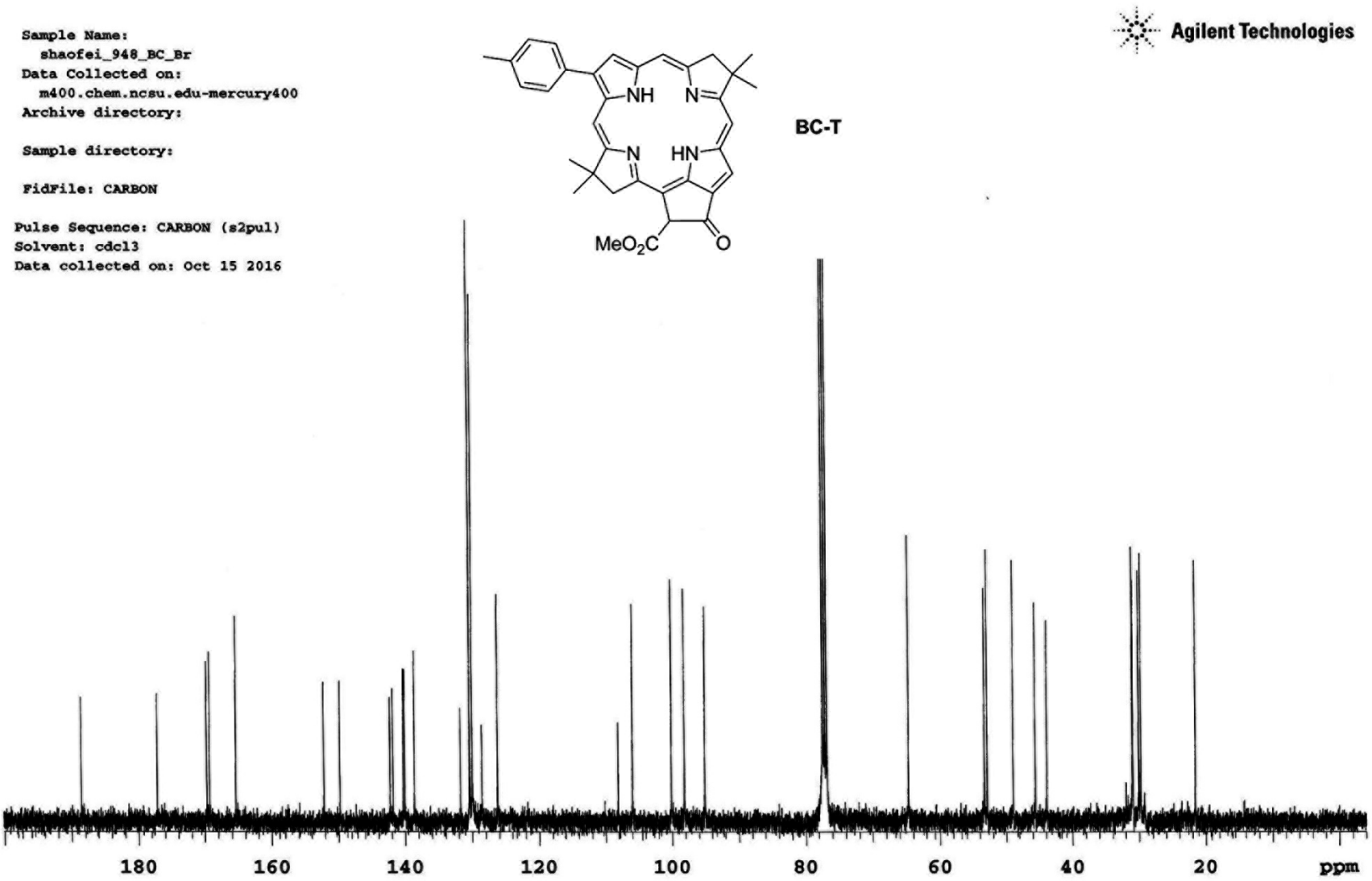

Sample Name:
shaofei_946
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: 947_tertrapyrrolelinearcompound_etes_hnmr
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 5 2016
Agilent Technologies
12-EtEs
12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.82 0.85 0.92 0.80 1.96 0.88 2.45 2.86 6.03 2.15 7.37

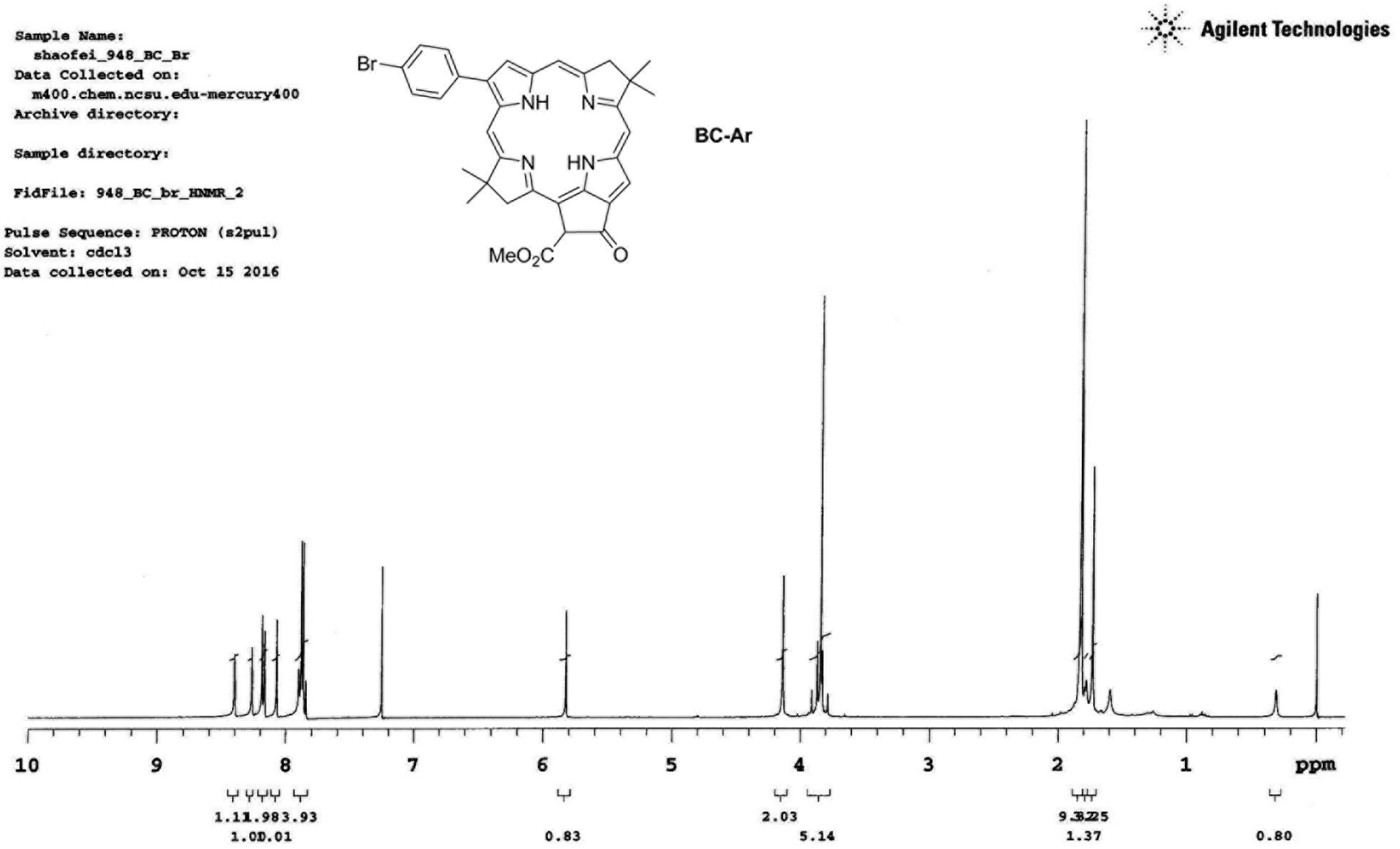

Sample Name:
shaofei_946
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 5 2016
Agilent Technologies
12-EtEs
180
160
140
120
100
80
60
40
20
ppm

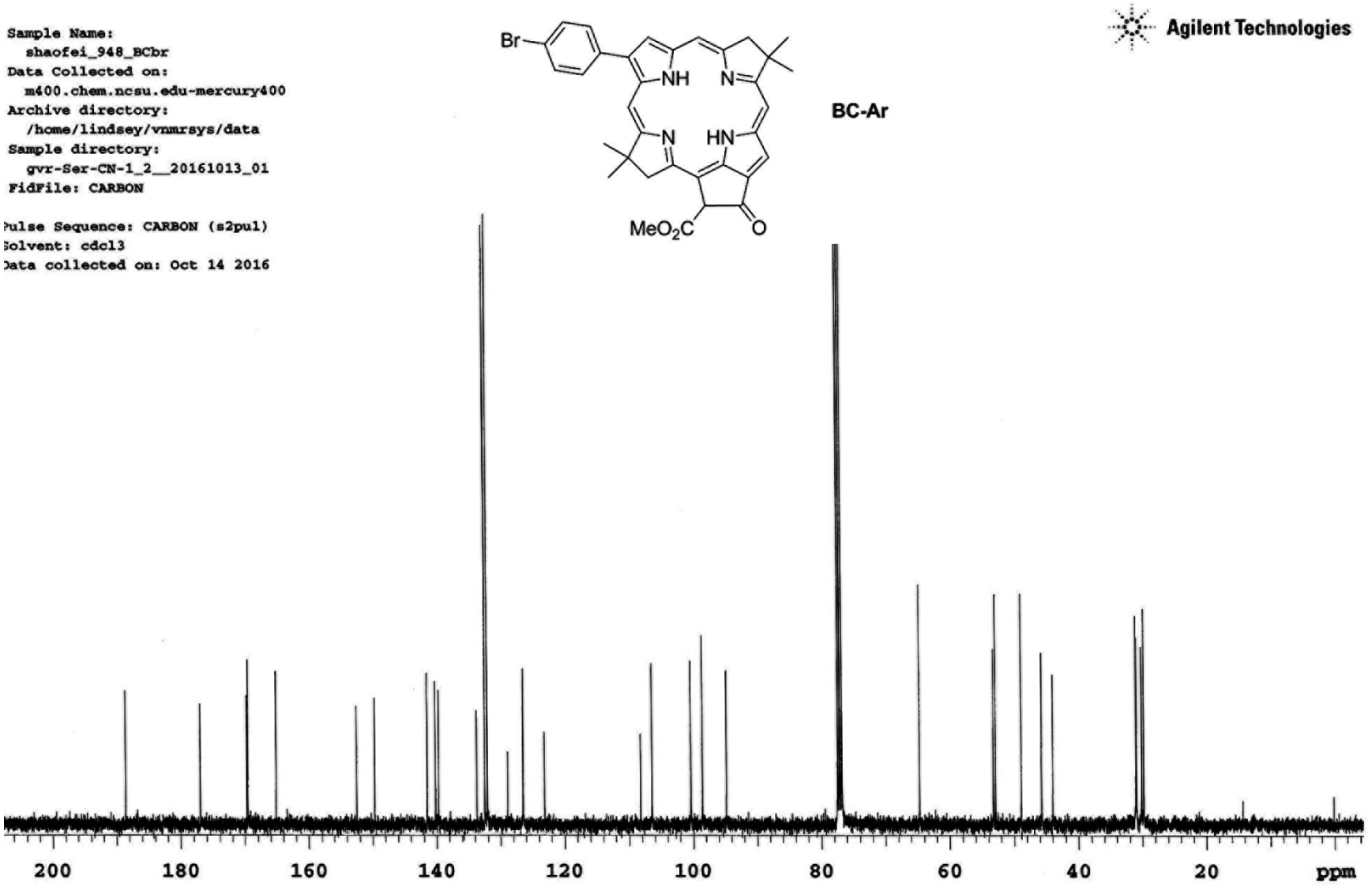

Br
NH
N
H
N
O
O
O
N
EtO
O
O
12-Ar/Et
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.90
0.87
0.95
5.02
0.95
1.01
1.00
2.33
4.76
0.88
1.05
0.93
1.98
6.00
2.19
2.20
11.86
6.84

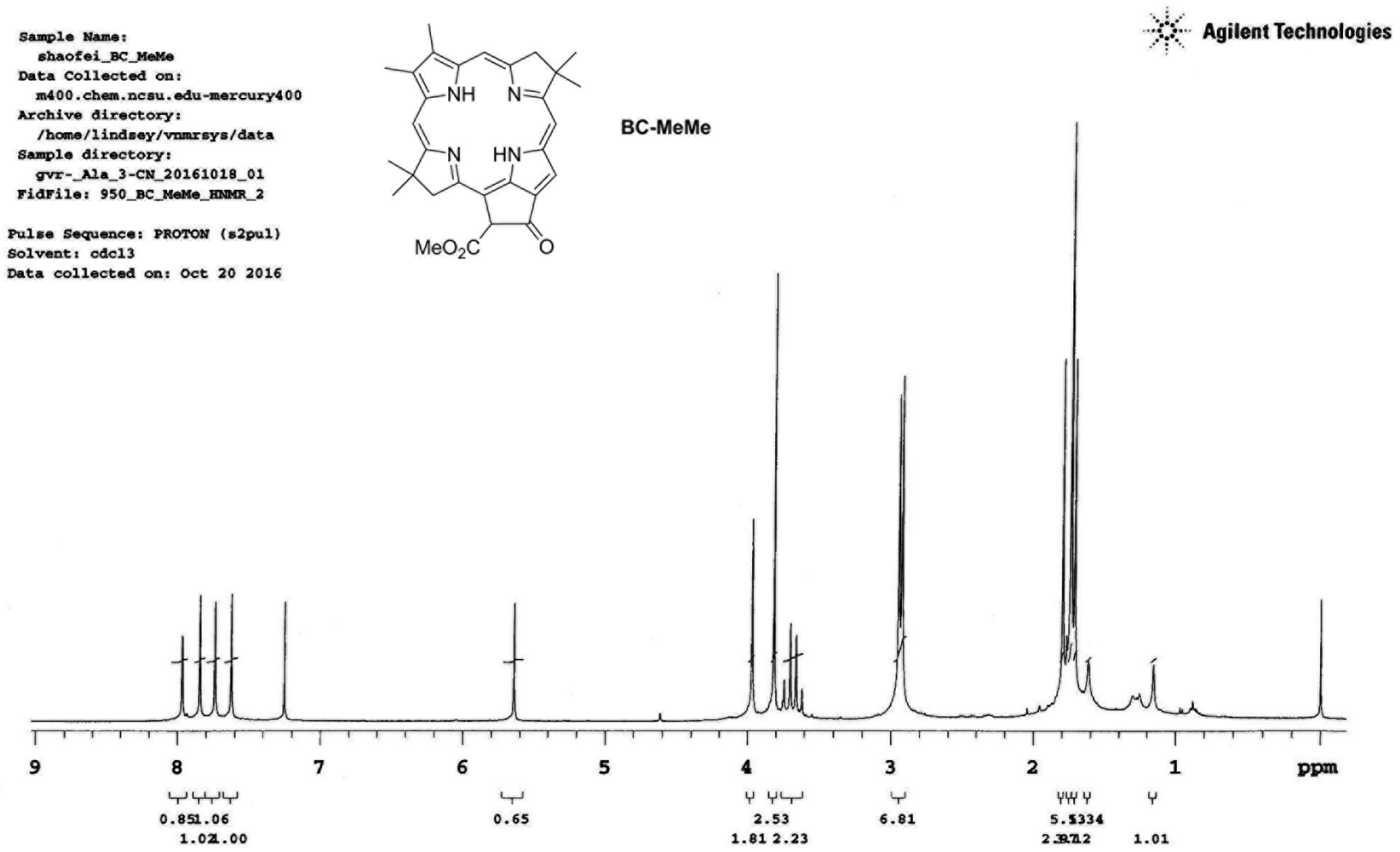

Sample Name:
shaofei
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
/home/lindsey/vnmrsys/data
Sample directory:
gvr-_Ala_3-CN_20161018_01
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 18 2016
Agilent Technologies
Br
NH
N
H
N
O
O
O
EtO
O
12-Ar/Et
180
160
140
120
100
80
60
40
20
ppm

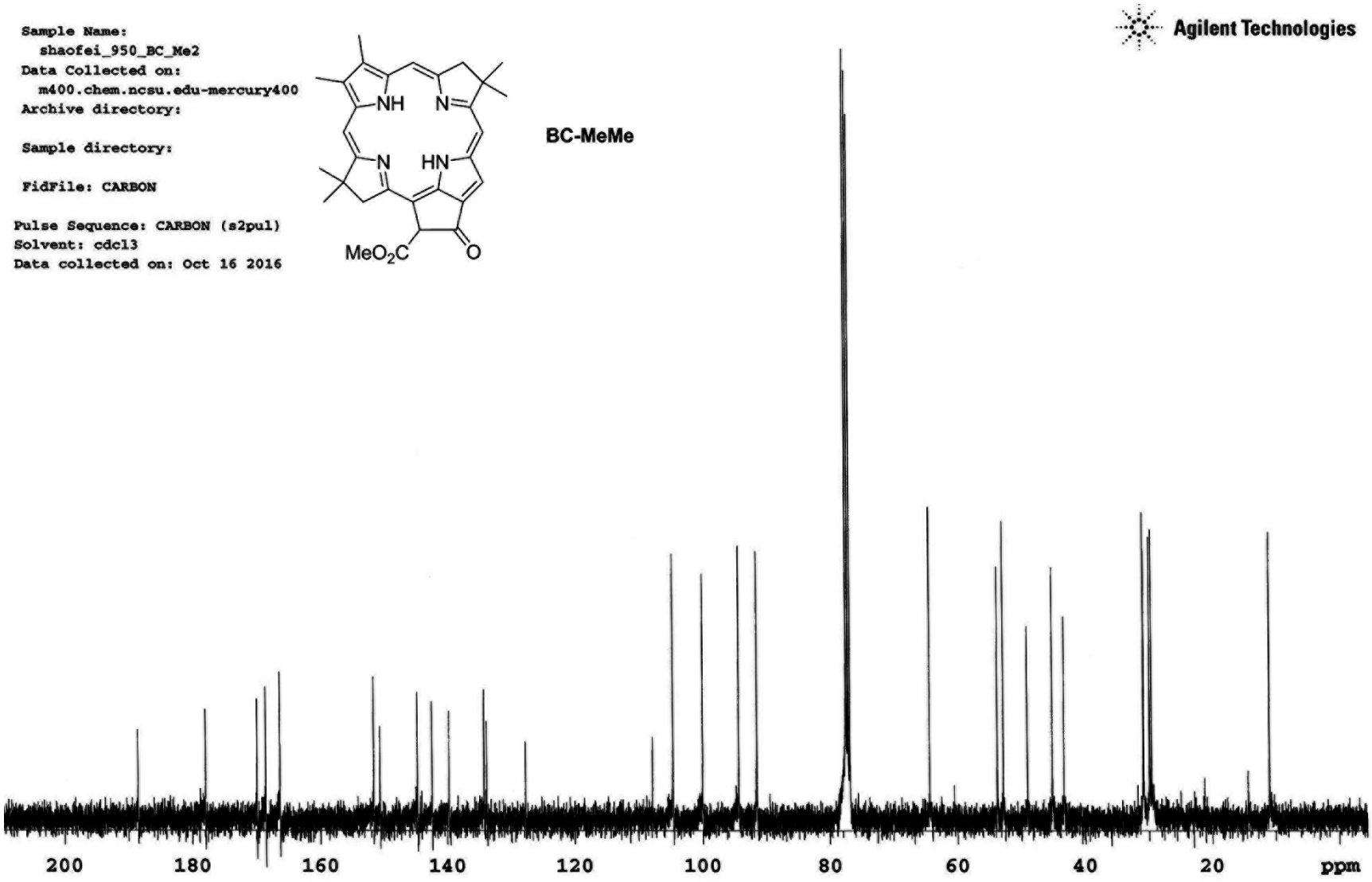

Sample Name:
shaofei_948_BC_Br
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: 949_BC_ptol_HNMR_2
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 17 2016
NH
N
N
HN
MeO2C
O
BC-T
Agilent Technologies
10
9
8
7
6
5
4
3
2
1
ppm
0.98
2.85
1.88
1.00
1.85
0.67
2.13
5.02
3.18
10.11
1.14
3.68
1.02

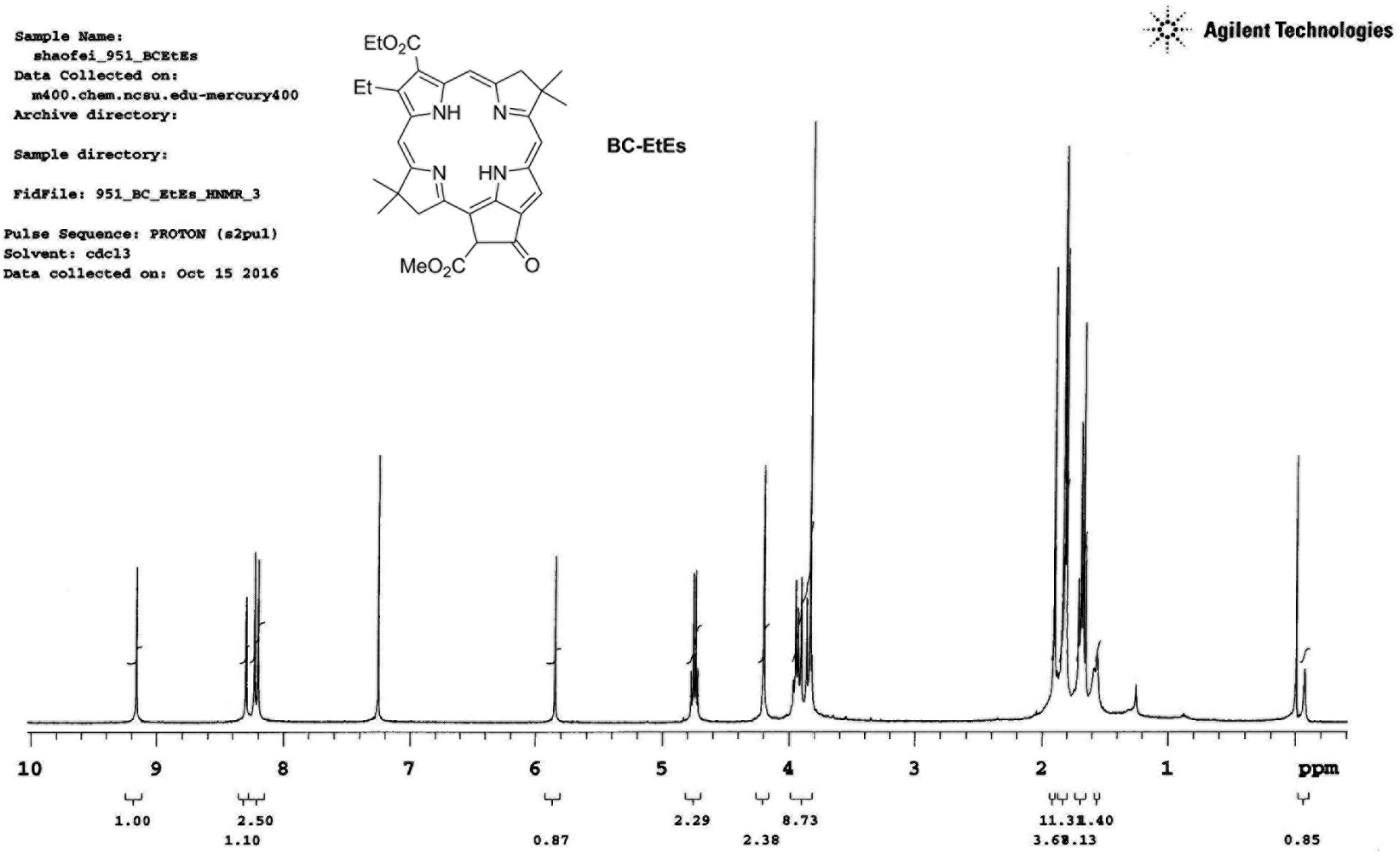

Sample Name:
shaofei_948_BC_Br
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 15 2016
Agilent Technologies
NH
N
N
HN
MeO2C
O
BC-T
180
160
140
120
100
80
60
40
20
ppm

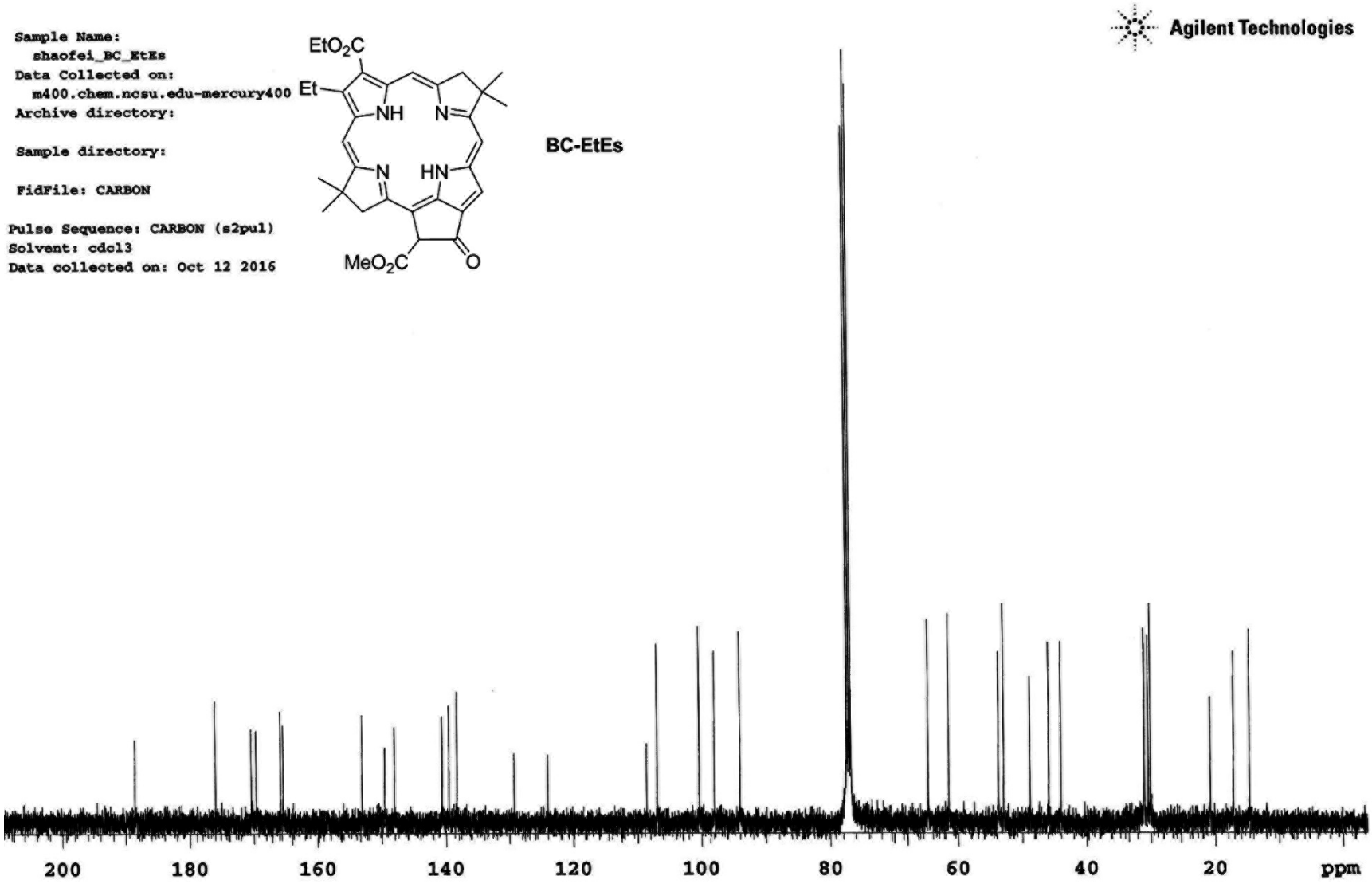

Agilent Technologies
Sample Name:
shaofei_948_BC_Br
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: 948_BC_br_HNMR_2
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 15 2016
Br
NH
N
N
HN
MeO2C
O
BC-Ar
10
9
8
7
6
5
4
3
2
1
ppm
1.11
1.98
3.93
1.00
1.01
0.83
2.03
5.14
9.32
25
1.37
0.80

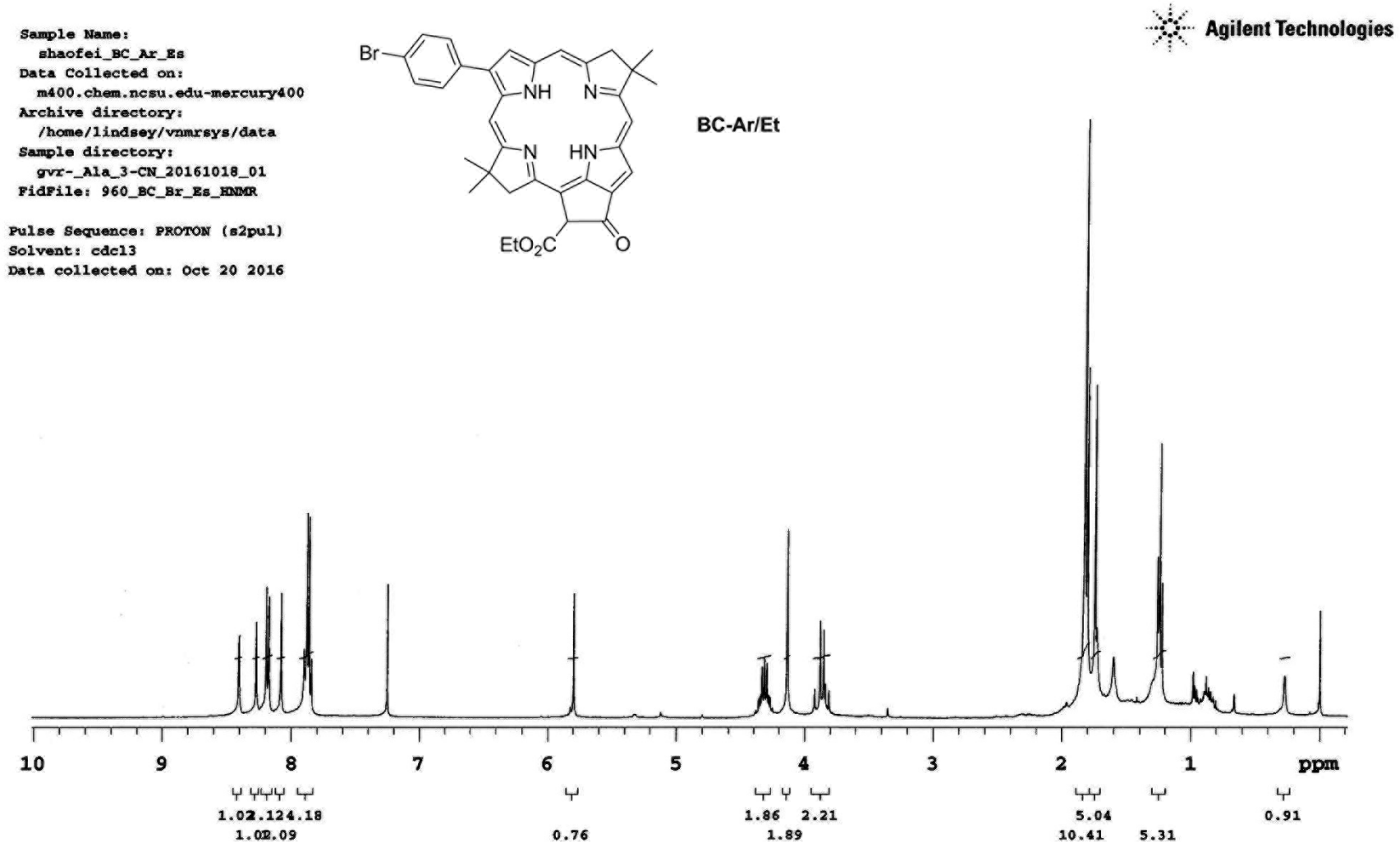

Sample Name:
shaofei_948_BCbr
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
/home/lindsey/vnmrsys/data
Sample directory:
gvr-Ser-CN-1_2__20161013_01
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 14 2016
Agilent Technologies
Br
NH
N
N
HN
MeO2C
O
BC-Ar
200
180
160
140
120
100
80
60
40
20
ppm

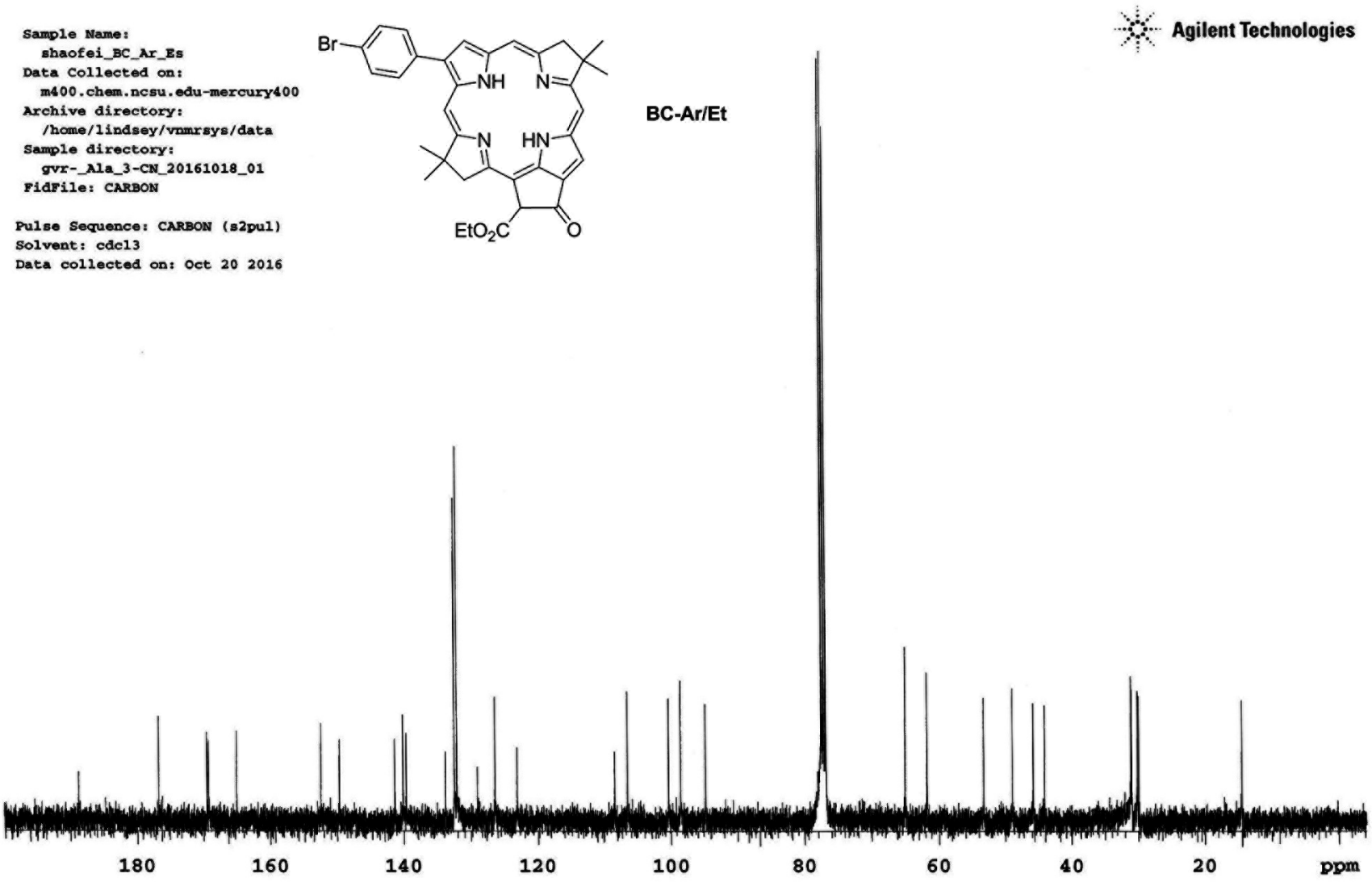

Sample Name:
shaofei_BC_MeMe
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
/home/lindsey/vnmrsys/data
Sample directory:
gvr-_Ala_3-CN_20161018_01
FidFile: 950_BC_MeMe_HNMR_2
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2016
NH
N
N
HN
MeO2C
O
BC-MeMe
Agilent Technologies
9
8
7
6
5
4
3
2
1
ppm
0.85
1.06
1.02
1.00
0.65
2.53
1.81
2.23
6.81
1.01


Sample Name:
shaofei_950_BC_Me2
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 16 2016
NH
N
N
HN
MeO2C
O
BC-MeMe
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm


Sample Name:
shaofei_951_BCEtEs
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: 951_BC_EtEs_HNMR_3
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 15 2016
EtO2C
Et
NH
N
N
HN
MeO2C
O
BC-EtEs
Agilent Technologies
10
9
8
7
6
5
4
3
2
1
ppm
1.00
2.50
1.10
0.87
2.29
2.38
8.73
11.31
1.40
3.68
8.13
0.85


Sample Name:
shaofei_BC_EtEs
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
Sample directory:
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 12 2016
EtO2C
Et
NH
N
N
HN
MeO2C
O
BC-EtEs
Agilent Technologies
200
180
160
140
120
100
80
60
40
20
ppm


Sample Name:
shaofei_BC_Ar_Es
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
/home/lindsey/vnmrsys/data
Sample directory:
gvr-_Ala_3-CN_20161018_01
FidFile: 960_BC_Br_Es_HNMR
Pulse Sequence: PROTON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2016
Agilent Technologies
BC-Ar/Et
Br
NH
N
N
HN
EtO2C
O
10
9
8
7
6
5
4
3
2
1
ppm
1.02
2.12
4.18
1.02
2.09
0.76
1.86
2.21
1.89
5.04
10.41
5.31
0.91


Sample Name:
shaofei_BC_Ar_Es
Data Collected on:
m400.chem.ncsu.edu-mercury400
Archive directory:
/home/lindsey/vnmrsys/data
Sample directory:
gvr-_Ala_3-CN_20161018_01
FidFile: CARBON
Pulse Sequence: CARBON (s2pul)
Solvent: cdcl3
Data collected on: Oct 20 2016
Br
NH
N
N
HN
EtO2C
O
BC-Ar/Et
Agilent Technologies
180
160
140
120
100
80
60
40
20
ppm