Supplementary Material

Facile and efficient synthesis of 4$^{'}$- thioxo-1$^{'}$, 3, 3$^{'}$,4, 4$^{'}$, 6$^{'}$ -hexahydro-1*H*, 2$^{'}$*H*-spiro[naphthalene-2,5$^{'}$-pyrimidin]-1-ones in a three-component Mannich-type reaction
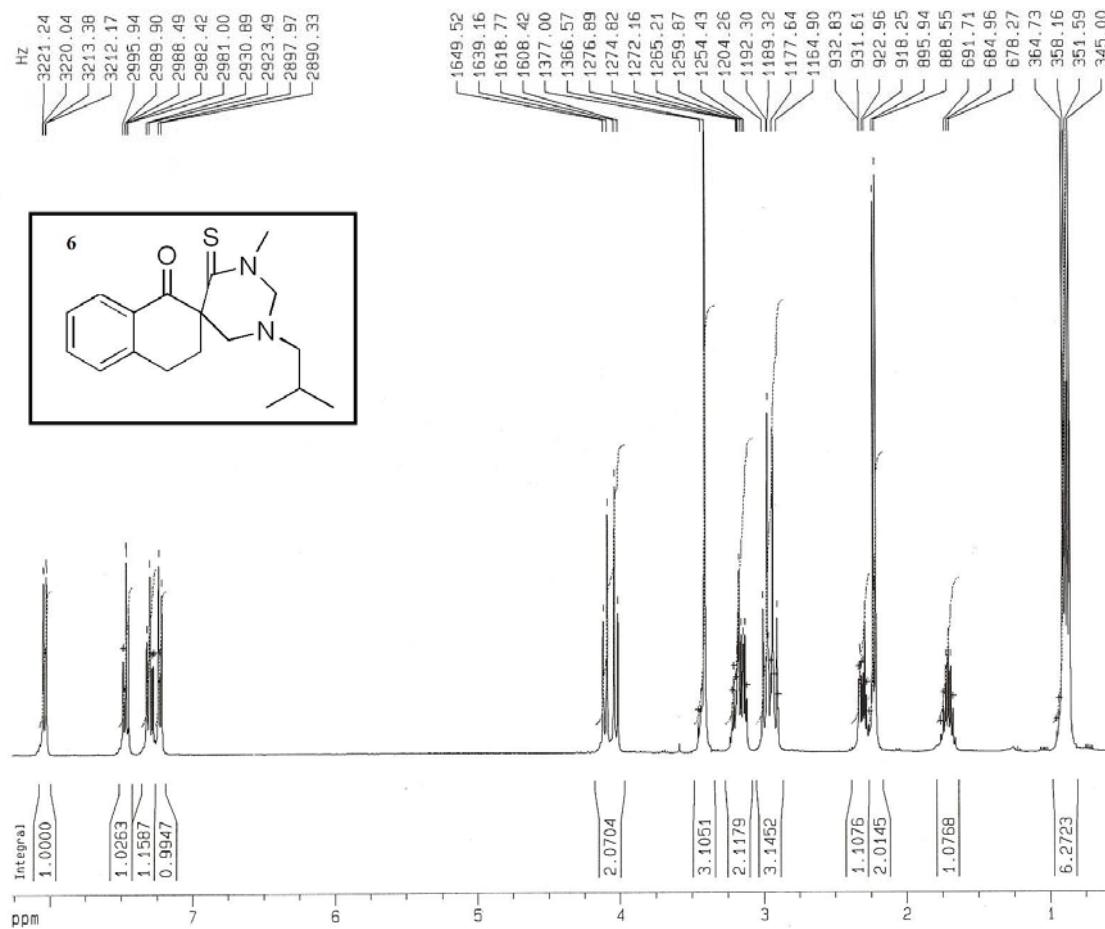
Tadeusz S. Jagodziński, Sławomir Westerlich

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Hz
3221.24
3220.04
3213.38
3212.17
2995.94
2989.90
2988.49
2982.42
2981.00
2930.89
2923.49
2897.97
2890.33

1649.52
1639.16
1618.77
1608.42
1377.00
1366.57
1276.89
1274.82
1272.16
1265.21
1259.87
1254.43
1204.26
1192.30
1189.32
1177.64
1164.90
932.83
931.61
922.96
918.25
895.94
888.55
691.71
684.96
678.27
364.73
358.16
351.59
345.00

Current Data Parameters
NAME   TJ_methyl_isobuthyl
EXPNO   1
PROCNO   1

F2 - Acquisition Parameters
Date_   20121112
Time   14.48
INSTRUM   spect
PROBHD   5 mm Multinu
PULPROG   zg30
TD   131072
SOLVENT   CDCl3
NS   16
DS   2
SWH   8003.201 Hz
FIDRES   0.061060 Hz
AQ   8.1887732 sec
RG   71.8
DW   62.475 usec
DE   89.25 usec
TE   300.0 K
D1   1.00000000 sec

======== CHANNEL f1 ========
NUC1   1H
P1   7.80 usec
PL1   0.00 dB
SFO1   400.1328009 MHz

F2 - Processing parameters
SI   32768
SF   400.1300021 MHz
WDW   no
SSB   0
LB   0.00 Hz
GB   0
PC   1.00

1D NMR plot parameters
CX   20.00 cm
CY   27.68 cm
F1P   8.272 ppm
F1   3309.77 Hz
F2P   0.537 ppm
F2   215.01 Hz
PPMCM   0.38672 ppm/cm
HZCM   154.73814 Hz/cm

Integral
1.0000

1.0263
1.1587
0.9947

2.0704

3.1051

2.1179
3.1452

1.1076
2.0145

1.0768

6.2723

ppm   7   6   5   4   3   2   1

ppm
199.936
196.533

142.736
133.495
132.315
128.538
128.275
126.893

77.489
77.170
76.851
73.823

62.215
59.567
55.312

40.844
35.113
25.833
25.411
20.583
20.517

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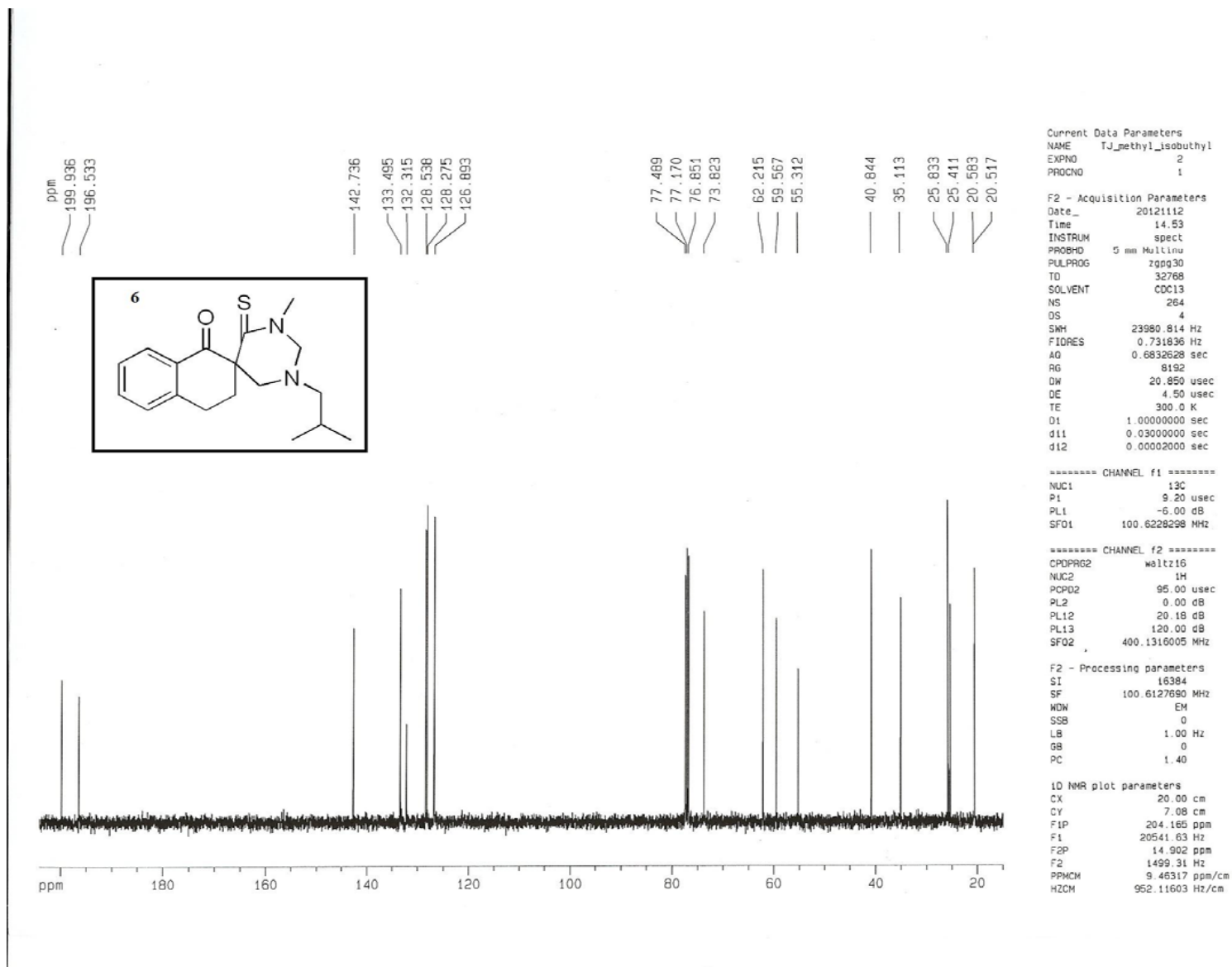

Current Data Parameters
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EXPNO   2
PROCNO   1
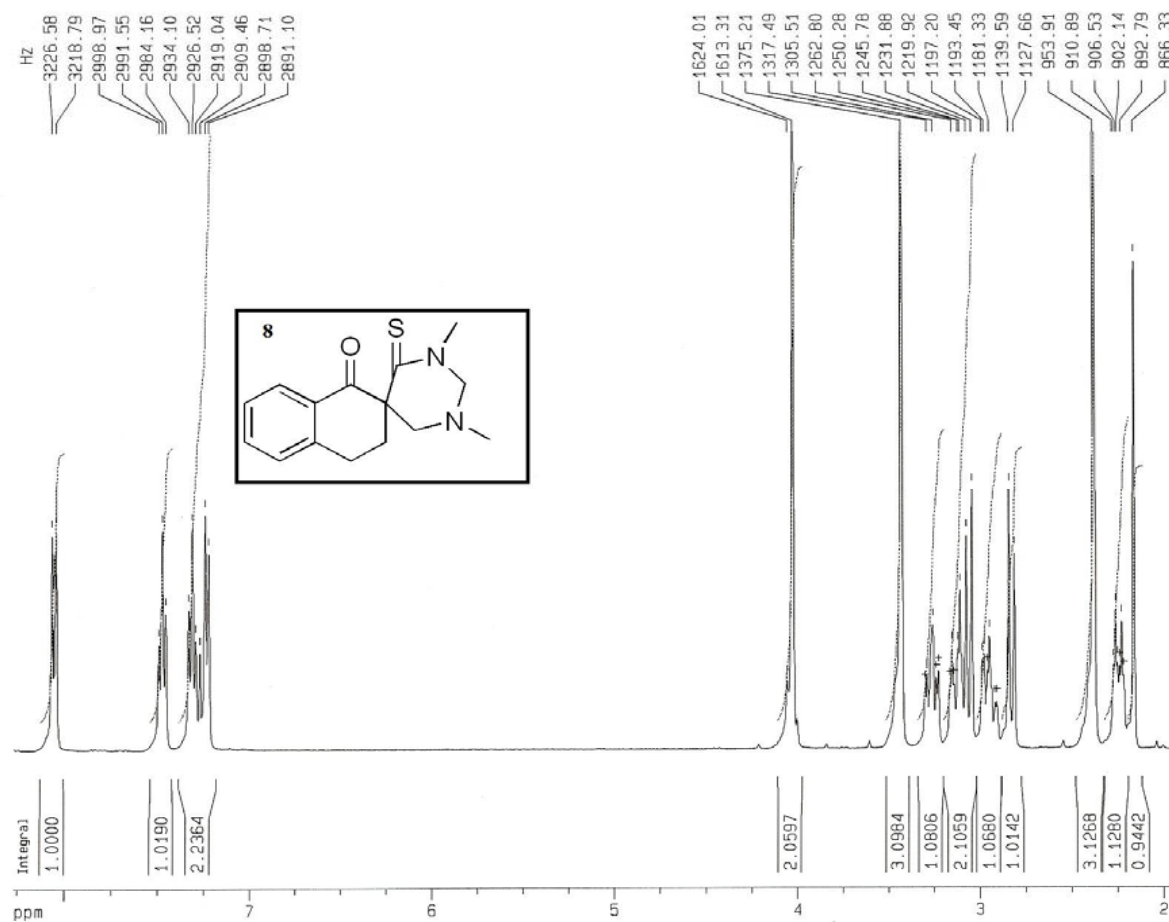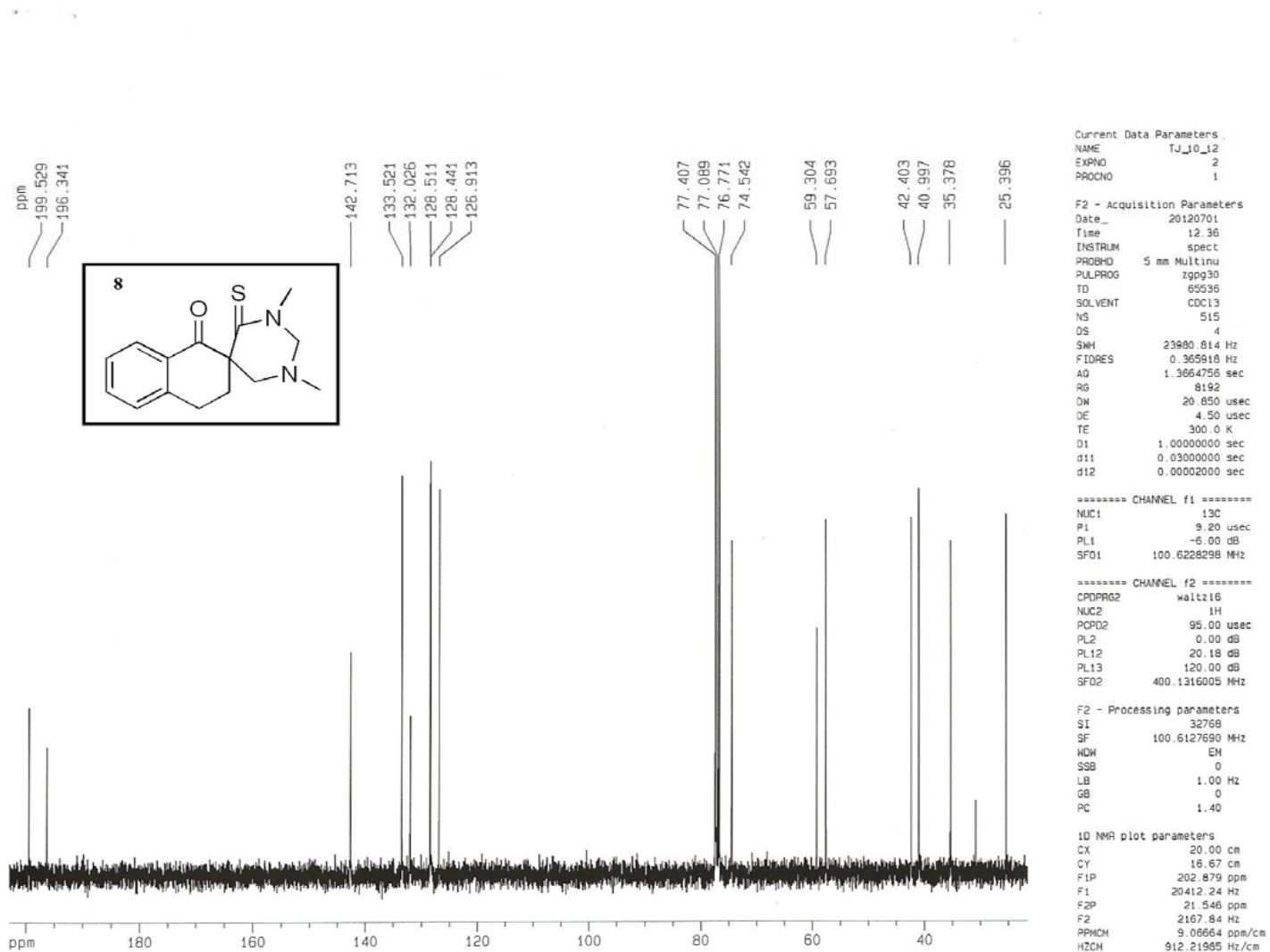
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Date_   20121112
Time   14.53
INSTRUM   spect
PROBHD   5 mm Multinu
PULPROG   zgpg30
TD   32768
SOLVENT   CDCl3
NS   264
DS   4
SWH   23980.814 Hz
FIDRES   0.731836 Hz
AQ   0.6832628 sec
RG   8192
DW   20.850 usec
DE   4.50 usec
TE   300.0 K
D1   1.00000000 sec
d11   0.03000000 sec
d12   0.00002000 sec

======== CHANNEL f1 ========
NUC1   13C
P1   9.20 usec
PL1   -6.00 dB
SFO1   100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2   waltz16
NUC2   1H
PCPD2   95.00 usec
PL2   0.00 dB
PL12   20.18 dB
PL13   120.00 dB
SFO2   400.1316005 MHz

F2 - Processing parameters
SI   16384
SF   100.6127690 MHz
WDW   EM
SSB   0
LB   1.00 Hz
GB   0
PC   1.40

1D NMR plot parameters
CX   20.00 cm
CY   7.08 cm
F1P   204.165 ppm
F1   20541.63 Hz
F2P   14.902 ppm
F2   1499.31 Hz
PPMCM   9.46317 ppm/cm
HZCM   952.11603 Hz/cm

ppm   180   160   140   120   100   80   60   40   20

Hz
3326.58
3218.79
2998.97
2991.55
2984.16
2934.10
2926.52
2919.04
2909.46
2898.71
2891.10

1624.01
1613.31
1375.21
1317.49
1305.51
1262.80
1250.28
1245.78
1231.88
1219.92
1197.20
1193.45
1181.33
1139.59
1127.66
953.91
910.89
906.53
902.14
892.79
866.33

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Current Data Parameters
NAME	TJ_10_12
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters
Date_	20120701
Time	12.16
INSTRUM	spect
PROBHD	5 mm Multinu
PULPROG	zg30
TD	131072
SOLVENT	CDCl3
NS	32
DS	2
SWH	8003.201 Hz
FIDRES	0.061060 Hz
AQ	8.1887732 sec
RG	128
DW	62.475 usec
DE	89.25 usec
TE	300.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========
NUC1	1H
P1	7.80 usec
PL1	0.00 dB
SFO1	400.1328009 MHz

F2 - Processing parameters
SI	32768
SF	400.1300037 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

1D NMR plot parameters
CX	20.00 cm
CY	24.16 cm
F1P	8.268 ppm
F1	3308.13 Hz
F2P	1.918 ppm
F2	767.55 Hz
PPMCM	0.31747 ppm/cm
HZCM	127.02922 Hz/cm

Integral
1.0000
1.0190
2.2364
2.0597
3.0984
1.0806
2.1059
1.0680
1.0142
3.1268
1.1280
0.9442

ppm   7   6   5   4   3   2

Hz
3208.18
3205.52
3202.55
3132.99
3130.03
3127.48
2970.53
2963.24
2945.75
2938.52
2931.55
2919.07
2912.36
2904.68

1785.24

1592.47

1370.43
1351.70

Current Data Parameters
NAME   TJ_23a_2012
EXPNO   1
PROCNO   1

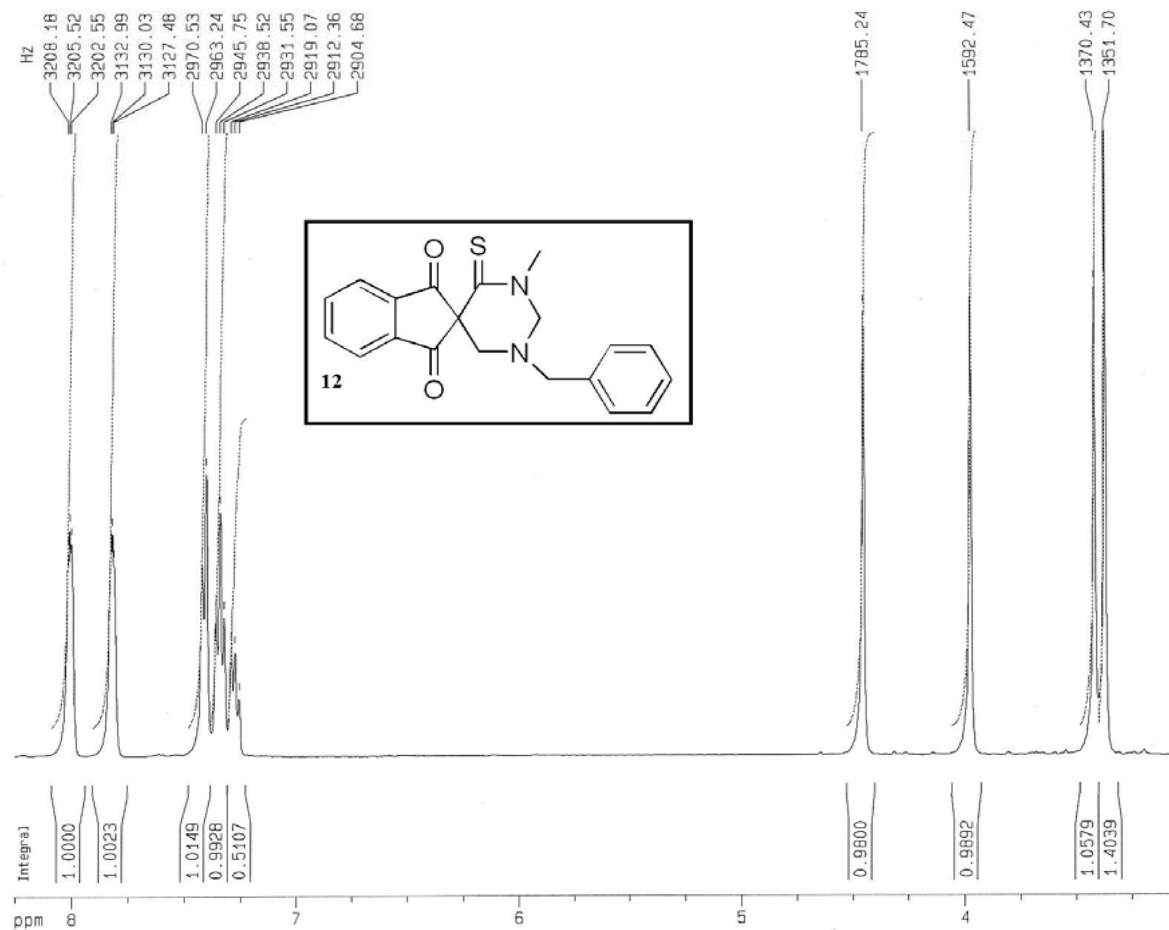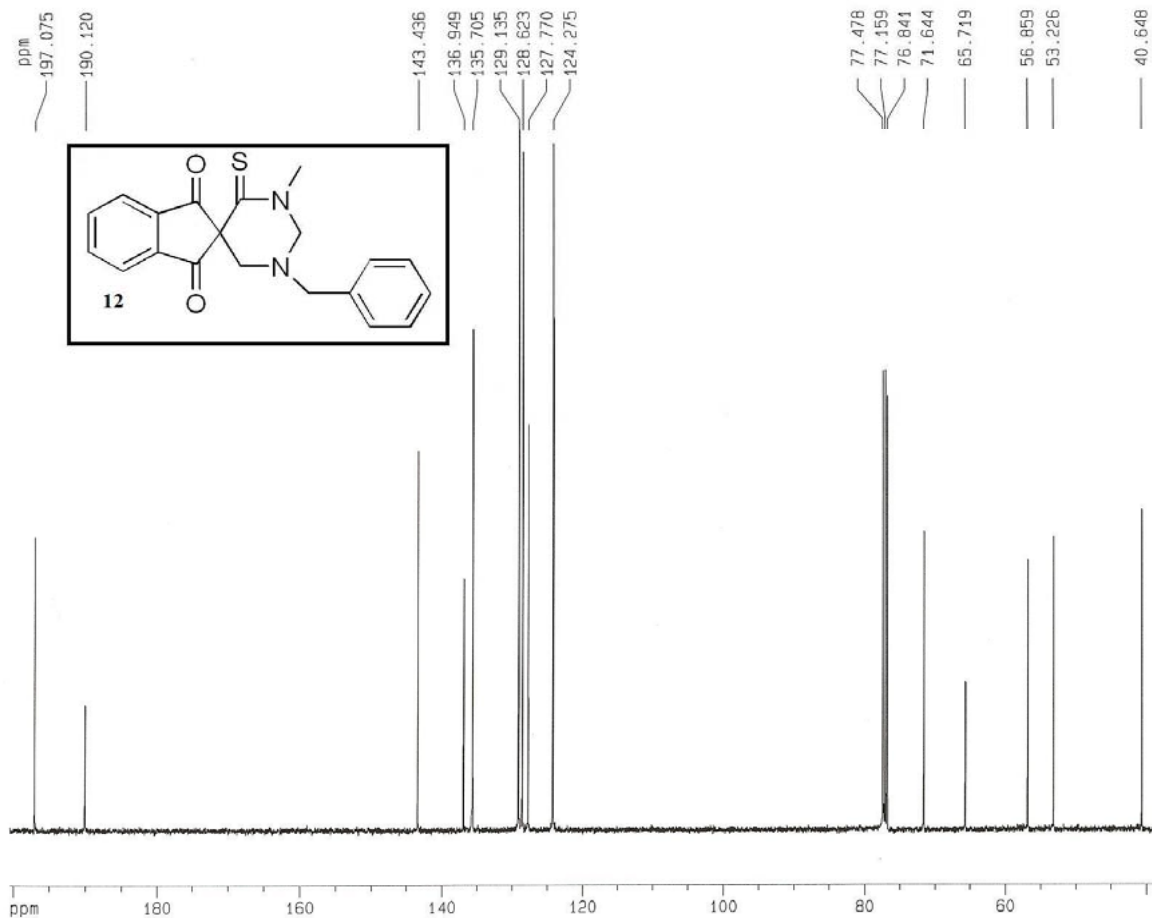F2 - Acquisition Parameters
Date_   20120625
Time   13.11
INSTRUM   spect
PROBHD   5 mm Multinu
PULPROG   zg30
TD   131072
SOLVENT   CDCl3
NS   18
DS   2
SWH   8003.201 Hz
FIDRES   0.061060 Hz
AQ   8.1887732 sec
RG   64
DW   62.475 usec
DE   89.25 usec
TE   300.0 K
D1   1.00000000 sec

======== CHANNEL f1 ========
NUC1   1H
P1   7.80 usec
PL1   0.00 dB
SFO1   400.1328009 MHz

F2 - Processing parameters
SI   32768
SF   400.1300087 MHz
WDW   no
SSB   0
LB   0.00 Hz
GB   0
PC   1.00

1D NMR plot parameters
CX   20.00 cm
CY   12.60 cm
F1P   8.255 ppm
F1   3303.13 Hz
F2P   3.039 ppm
F2   1215.97 Hz
PPMCM   0.26081 ppm/cm
HZCM   104.35828 Hz/cm

Integral
1.0000
1.0023
1.0149
0.9928
0.5107
0.9800
0.9892
1.0579
1.4039

ppm   8   7   6   5   4

Current Data Parameters
NAME   TJ_23aaa_2012
EXPNO   2
PROCNO   1

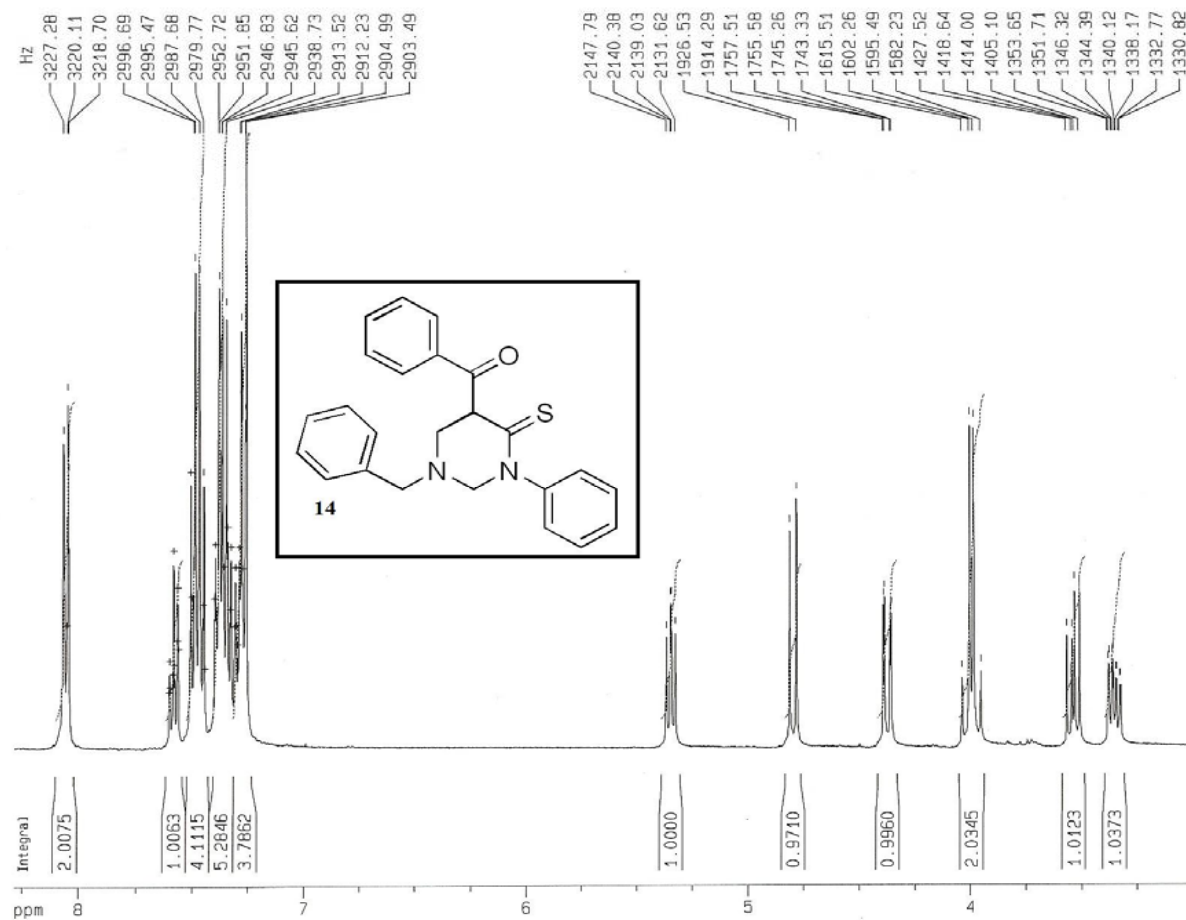F2 - Acquisition Parameters
Date_   20120627
Time   16.53
INSTRUM   spect
PROBHD   5 mm Multinu
PULPROG   zgpg30
TD   32768
SOLVENT   CDCl3
NS   3072
DS   4
SWH   23980.814 Hz
FIDRES   0.731836 Hz
AQ   0.6832628 sec
RG   5792.6
DW   20.850 usec
DE   4.50 usec
TE   300.0 K
D1   1.00000000 sec
d11   0.03000000 sec
d12   0.00002000 sec

======== CHANNEL f1 ========
NUC1   13C
P1   9.20 usec
PL1   -6.00 dB
SFO1   100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2   waltz16
NUC2   1H
PCPD2   95.00 usec
PL2   0.00 dB
PL12   20.18 dB
PL13   120.00 dB
SFO2   400.1316005 MHz

F2 - Processing parameters
SI   16384
SF   100.6127690 MHz
WDW   EM
SSB   0
LB   1.00 Hz
GB   0
PC   1.40

1D NMR plot parameters
CX   20.00 cm
CY   14.76 cm
F1P   200.509 ppm
F1   20173.81 Hz
F2P   37.610 ppm
F2   3784.05 Hz
PPMCM   8.14497 ppm/cm
HZCM   819.48828 Hz/cm

Hz
3227.28
3220.11
3218.70
2996.69
2995.47
2987.68
2979.77
2952.72
2951.85
2946.83
2945.62
2938.73
2913.52
2912.23
2904.99
2903.49

2147.79
2140.38
2139.03
2131.62
1926.53
1914.29
1757.51
1755.58
1745.26
1743.33
1615.51
1602.26
1595.49
1582.23
1427.52
1418.64
1414.00
1405.10
1353.65
1351.71
1346.32
1344.39
1340.12
1338.17
1332.77
1330.82

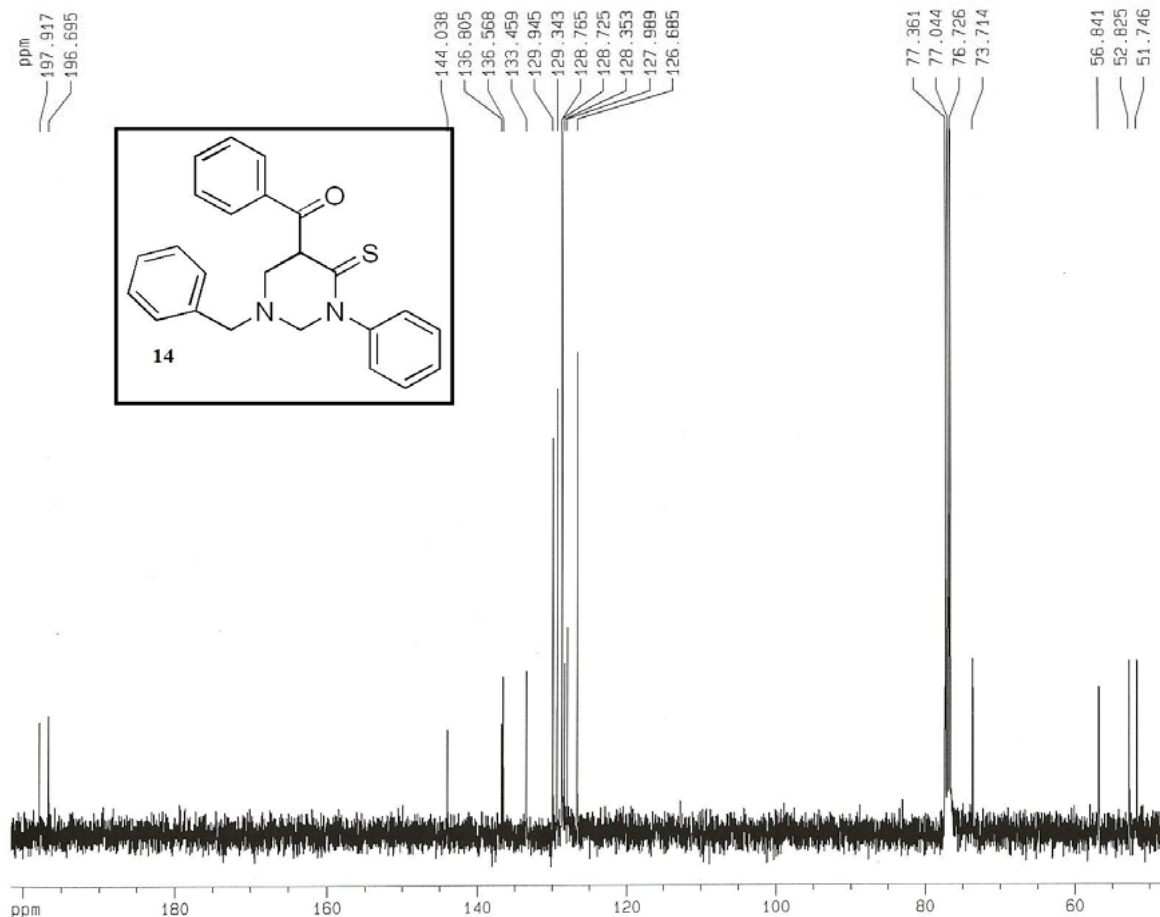Current Data Parameters
NAME   TJ_21_2012
EXPNO   1
PROCNO   1

F2 - Acquisition Parameters
Date_   20120810
Time   10.30
INSTRUM   spect
PROBHD   5 mm Multinu
PULPROG   zg30
TD   131072
SOLVENT   CDCl3
NS   32
DS   2
SWH   8003.201 Hz
FIDRES   0.061060 Hz
AQ   8.1887732 sec
RG   362
DW   62.475 usec
DE   89.25 usec
TE   300.0 K
D1   1.00000000 sec

======== CHANNEL f1 ========
NUC1   1H
P1   7.80 usec
PL1   0.00 dB
SFO1   400.1328009 MHz

F2 - Processing parameters
SI   32768
SF   400.1300110 MHz
WDW   no
SSB   0
LB   0.00 Hz
GB   0
PC   1.00

1D NMR plot parameters
CX   20.00 cm
CY   13.09 cm
F1P   8.286 ppm
F1   3315.30 Hz
F2P   2.997 ppm
F2   1199.34 Hz
PPMCM   0.26441 ppm/cm
HZCM   105.79771 Hz/cm

Integral
2.0075
1.0063
4.1115
5.2846
3.7862
1.0000
0.9710
0.9960
2.0345
1.0123
1.0373

ppm   8   7   6   5   4

ppm
197.917
196.695

144.038
136.805
136.568
133.459
129.945
129.343
128.765
128.725
128.353
127.989
126.685

77.361
77.044
76.726
73.714

56.841
52.825
51.746

14

Current Data Parameters
NAME   TJ_21_2012
EXPNO   2
PROCNO   1

F2 - Acquisition Parameters
Date_   20120810
Time   11.00
INSTRUM   spect
PROBHD   5 mm Multinu
PULPROG   zgpg30
TD   65536
SOLVENT   CDCl3
NS   1525
DS   4
SWH   23980.814 Hz
FIDRES   0.365918 Hz
AQ   1.3664756 sec
RG   13004
DW   20.850 usec
DE   4.50 usec
TE   300.0 K
D1   1.00000000 sec
d11   0.03000000 sec
d12   0.00002000 sec

======== CHANNEL f1 ========
NUC1   13C
P1   9.20 usec
PL1   -6.00 dB
SFO1   100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2   waltz16
NUC2   1H
PCPD2   95.00 usec
PL2   0.00 dB
PL12   20.18 dB
PL13   120.00 dB
SFO2   400.1316005 MHz

F2 - Processing parameters
SI   32768
SF   100.6127690 MHz
WDW   EM
SSB   0
LB   1.00 Hz
GB   0
PC   1.40

1D NMR plot parameters
CX   20.00 cm
CY   50.15 cm
F1P   201.593 ppm
F1   20282.84 Hz
F2P   47.910 ppm
F2   4820.39 Hz
PPMCM   7.68414 ppm/cm
HZCM   773.12250 Hz/cm

ppm   180   160   140   120   100   80   60

EURO EA
Elemental Analyzer

Method Filename : D:\Program Files\Callidus 2E3\Method\CHNS193.mth
Summarize Result : Percent
Configuration: C H N S
Date: 24/10/2012

Sample Name	N %	C %	H %	S %	Weight
BBOT	6.51	72.53	6.09	7.44	1.596
BBOT	6.606	72.807	6.242	7.547	1.863
BBOT	6.431	72.559	5.866	7.164	1.199
bbot	6.586	72.438	6.223	7.597	1.631
4	7.946	72.106	4.695	9.294	1.62
4	8.041	71.984	6.396	9.103	1.755
5	6.695	75.278	5.793	7.495	1.494
5	6.837	75.005	6.091	7.652	2.006
5	6.917	75.646	5.959	7.842	2.213
8	10.194	65.402	6.982	12.161	1.882
8	10.151	65.197	6.783	11.8	1.594
9	8.224	71.098	6.058	9.564	1.549
9	8.359	71.166	6.094	9.742	1.574
12	7.785	68.11	5.103	9.118	1.425
12	8.03	68.455	5.467	9.575	2.024
14	7.13	74.126	5.698	8.256	1.451
14	7.222	74.534	5.678	8.275	1.353

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EUROVECTOR
INSTRUMENTS & SOFTWARE

EURO EA
Elemental Analyzer

Method Filename :   D:\Program Files\Callidus 2E3\Method\CHNS196.mth
Summarize Result : Percent
Configuration: C H N S
Date: 27/11/2012

Sample Name	N %	C %	H %	S %	Weight
BBOT	6.473	72.423	5.909	7.376	1.432
BBOT	6.463	72.584	6.11	7.45	1.449
BBOT	6.596	72.583	6.262	7.495	1.575
bbot	6.649	72.788	6.488	7.578	1.954
3	7.483	73.031	5.845	8.055	1.677
3	7.331	72.971	7.392	8.423	1.587
3	7.853	72.047	7.526	8.454	2.772
3	7.568	71.562	7.38	8.545	2.422
7	7.348	72.47	7.43	8.339	2.245
7	7.302	72.682	7.314	8.445	1.421
7	7.247	72.058	7.286	8.381	1.458

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EUROVECTOR
INSTRUMENTS & SOFTWARE

EURO EA
Elemental Analyzer

Method Filename :   D:\Program Files\Callidus 2E3\Method\CHNS199.mth
Summarize Result : Percent
Configuration: C H N S
Date: 28/11/2012

Sample Name	N %	C %	H %	S %	Weight
BBOT	6.531	72.626	6.082	7.432	1.959
BBOT	6.518	72.584	6.087	7.443	1.588
BBOT	6.481	72.381	6.101	7.446	1.543
bbot	6.512	72.539	6.167	7.523	1.589
6	8.932	68.447	7.687	10.228	1.574
6	8.728	67.581	7.886	9.959	1.549
10	10.228	72.892	5.551	7.81	1.446
10	10.344	72.734	5.819	7.852	1.799
10	10.198	72.741	5.685	7.858	1.489
10	10.305	72.506	5.786	7.867	1.6

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