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of Reliable Methods
for the Preparation
of Organic Compounds

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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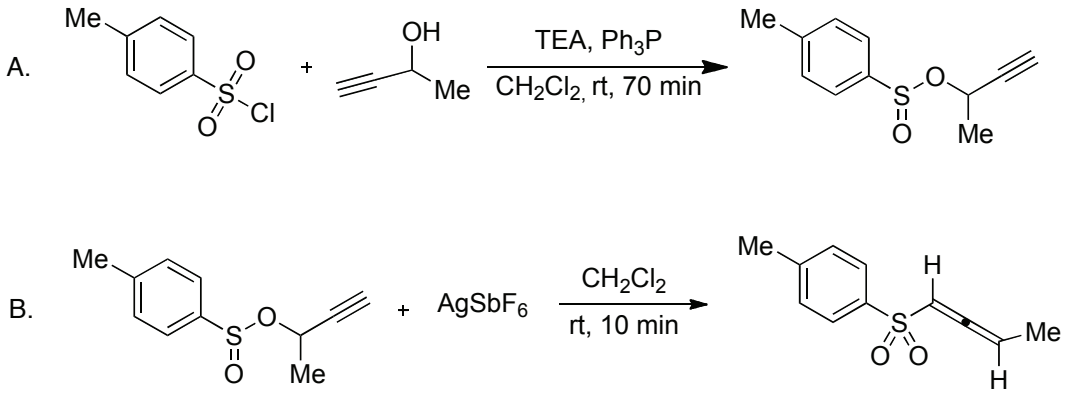
SILVER-CATALYZED REARRANGEMENT OF PROPARGYLIC SULFINATES: SYNTHESIS OF ALLENIC SULFONES



Submitted by Michael Harmata, Zhengxin Cai and Chaofeng Huang.[1]
Checked by Kay M. Brummond and Bo Wen.

1. Procedure

A. But-3-yn-2-yl 4-Methylbenzenesulfinate. A flame-dried 500-mL, three-necked, round-bottomed flask is equipped with a 5.0 cm magnetic stir bar, an internal thermometer, a 125-mL pressure-equalizing addition funnel, and a rubber septum containing nitrogen inlet and outlet needles. The flask is charged with tosyl chloride (9.55 g, 50.1 mmol, 1 equiv) (Note 1), dichloromethane (125 mL) (Note 2), and triethylamine (7.65 mL, 5.56 g, 55.0 mmol, 1.1 equiv) (Note 3), resulting in a colorless solution. The solution is cooled to 19 °C (internal temperature) in a water bath. To this solution is added, dropwise over 70 min by means of the addition funnel, a well mixed yellow solution of 3-butyn-2-ol (4.00 mL of 97% solution, 3.50 g, 50.0 mmol, 1 equiv) (Note 4) and triphenylphosphine (13.1 g, 50.0 mmol, 1 equiv) (Note 5) in 125 mL of dichloromethane, during which time the reaction solution turns a pale yellow color. The rate of the addition is adjusted so as to keep the temperature of the reaction mixture at 19 °C . The reaction is run under an inert nitrogen atmosphere and monitored by TLC. After the disappearance of starting material, the solution is transferred to a 500-mL round-bottomed flask and concentrated on a rotary evaporator (35 °C, 10 mmHg) to a volume of about 50 mL. Ether/hexanes (200 mL/1:4) (Notes 6 and 7) is added to the flask. Upon swirling by hand, a voluminous amount of white solid (triethylamine hydrochloride salt) forms, which is

filtered through a short silica gel column (40 g of silica in a 150-mL sintered glass funnel) (Note 8). Diethyl ether (150 mL) is used in three portions to rinse the flask and the solid. The filtrate is concentrated on a rotary evaporator to give a faint yellow oil. The crude oil is purified by silica gel column chromatography. The product is charged on a column (5 x 18 cm) of 150 g of silica gel and eluted with 1:4 ether/hexanes (1000 mL). Collection of 25 mL fractions begins immediately, and the desired product is obtained in fractions 15-38. The product-containing fractions are combined in a 1000-mL round-bottomed flask and concentrated in vacuo by rotary evaporation (35 °C, 10 mmHg) to afford 9.64 g (93%) of a 1:1 mixture of two diastereomers of but-3-yn-2-yl 4-methylbenzenesulfinate as a faint yellow (almost colorless) oil (Note 9).

B. 1-(Buta-1,2-dien-1-ylsulfonyl)-4-methylbenzene. A 250-mL round-bottomed flask equipped with a 3 cm magnetic stir bar is flame dried under a nitrogen flow. After the flask has cooled to room temperature, it is charged with silver hexafluoroantimonate (266 mg, 0.774 mmol, 0.02 equiv) (Note 10) and protected by a nitrogen atmosphere. The but-3-yn-2-yl 4-methylbenzenesulfinate (8.05 g, 38.7 mmol, 1 equiv) in 77 mL of dichloromethane (0.5 M) is introduced to the flask containing silver hexafluoroantimonate via cannula over 3 min. The reaction is run under a nitrogen atmosphere and monitored by TLC. After the disappearance of starting material (within 10 min), the contents are passed through a short silica gel plug (10 g silica gel in a 60 mL sintered glass filter funnel) and the pad of silica is rinsed with 100 mL of diethyl ether. After concentration of the filtrate on a rotary evaporator (35 °C, 10 mmHg), the residue is put on a high vacuum pump to remove trace amounts of solvent to provide 7.93 g (98.5%) of 1-(buta-1,2-dien-1-ylsulfonyl)-4-methylbenzene as a faint yellow oil. The oil is mixed with 50 mL of 1:4 diethyl ether/hexane (10 mL/40 mL) in a 100-mL round-bottomed flask. The solution is cooled to –20 °C and maintained at that temperature for 22 h, and the resulting crystals are collected by suction filtration on a 60-mL sintered glass funnel quickly so as to not allow the filtrate to warm too much. The flask and the crystals are rinsed using cold hexanes (100 mL). The crystals are transferred to a 100-mL, round-bottomed flask and dried under high vacuum pump to afford 7.73 g (96%) of a white solid (Note 11).

2. Notes

1. *p*-Toluenesulfonyl chloride (99%) was purchased from Acros and used as received.

2. Dichloromethane was purified by passing through alumina using the Sol-Tek ST-002 solvent purification system directly before use.

3. Triethylamine (99%) was purchased from Aldrich and used as received.

4. 3-Butyn-2-ol (97%) was purchased from Aldrich and used as received.

5. Triphenylphosphine (99%) was purchased from Aldrich and used as received.

6. ACS reagent grade ether was purchased from Fischer and used as received.

7. ACS reagent grade hexanes was purchased from Fischer and used as received.

8. Silica gel, standard grade, was purchased from Sorbent Technologies, with 0.040 – 0.063 mm particle size.

9. For characterization purposes, the diastereomers were separated, but the product was used in the next step as a 1:1 mixture. The product displayed the following physical properties. Diastereomer 1: ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 1.59 (d, J = 6.5 Hz, 3 H), 2.39 (d, J = 2.5 Hz, 1 H), 2.42 (s, 3 H), 4.98 (dq, J = 2.0, 7.0 Hz, 1 H), 7.32 (d, J = 8.0 Hz, 2 H), 7.63 (d, J = 8.5 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$, 298 K) δ: 21.5, 23.7, 62.0, 74.4, 81.9, 125.4 (2 C), 129.6 (2 C), 141.6, 142.9. Diastereomer 2: ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 1.52 (d, J = 7.0 Hz, 3 H), 2.42 (s, 3 H), 2.64 (d, J = 2.5 Hz, 1 H), 5.02 (dq, J = 2.0, 7.0 Hz, 1 H), 7.34 (d, J = 8.0 Hz, 2 H), 7.64 (d, J = 8.0 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$, 298 K) δ: 21.5, 22.9, 63.8, 75.0, 82.2, 125.0 (2 C), 129.7 (2 C), 142.4, 142.9. IR (film) : 3291, 3250, 2989, 2935, 2118, 1596, 1446, 1330, 1138, 1019, 901, 813 cm^{-1}; TLC: R_f = 0.23, 0.29 (for two diastereomers respectively, SiO_2, Et_2O/hexanes, 1:4); MS *m/z* (relative intensity) : 209 (15%, M+H), 198 (10%), 215 (11%), 229 (100%), 241 (10%); HRMS *m/z* : calcd. for $C_{11}H_{12}O_2S$ [M+H] 209.0636, found 209.0636; Anal. calcd. for $C_{11}H_{12}O_2S$: C, 63.43; H, 5.81. Found C, 63.20; H, 5.84.

10. Silver hexafluoroantimonate (98%) was purchased from Aldrich and used as received.

11. The product displayed the following physical properties: mp 47–48 °C; ^{1}H NMR (500 MHz, $CDCl_3$, 298 K) δ: 1.78 (dd, *J* = 3.0, 7.5 Hz, 3 H), 2.44 (s, 3 H), 5.80 (dq, *J* = 6.0, 7.5 Hz, 1 H), 6.14 (dq, *J* = 6.0, 3.0, 1H), 7.33 (d, *J* = 8.0 Hz, 2 H), 7.77 (d, *J* = 8.5 Hz, 2 H); ^{13}C NMR (125 MHz, $CDCl_3$, 298 K) δ: 13.0, 21.6, 95.9, 100.7, 127.6 (2 C), 129.7 (2 C), 138.4, 144.3, 206.1; IR (film): 3020, 2925, 1954, 1596, 1317, 1146, 1085, 815, 767cm^{-1}. TLC: R_f = 0.35 (SiO_2, EtOAc/hexanes, 1:1) (Note 12); MS *m/z* (relative intensity) : 209 (57%, M+H), 201 (100%), 198 (10%), 212 (76%), , 218 (64%), 219 (46%); HRMS *m/z* : calcd. for $C_{11}H_{12}O_2S$ [M+H] 209.0636, found 209.0642; Anal. calcd. For $C_{11}H_{12}O_2S$: C, 63.43; H, 5.81. Found C, 63.69; H, 5.75.

12. ACS reagent grade ethyl acetate was purchased from Fischer and used as received.

Safety and Waste Disposal Information

All hazardous materials should be handled and disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

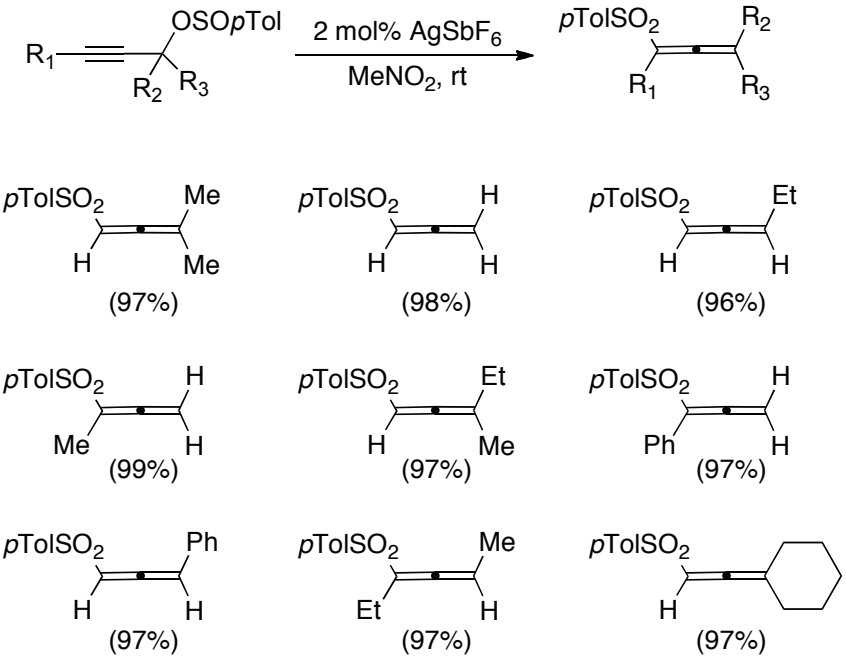
Allenic sulfones[2] have a rich chemistry that involves such diverse reactions as the carbon-accelerated Claisen rearrangement,[3] metal-catalyzed reactions[4] and DNA cleavage.[5] New, facile approaches to their synthesis are thus in demand.

The preparation of allenic sulfones has been accomplished thermally[6] and under the influence of various metal catalysts including those based on rhodium[7] and palladium.[8] The present method was discovered during a systematic study to use gold catalysts in the rearrangement of propargylic sulfinates to allenic sulfones. Although gold catalysts were effective, we found that the much more economical silver cation was as good or better in effecting the transformation.[9]

The synthesis of propargylic sulfinates used in this study is based on the method of Toru[10] and is related to one published by Sharpless.[11] This approach avoids the preparation of reactive sulfinyl chlorides and broadens the scope of sulfinate ester formation considerably, since many sulfonyl chlorides are either commercially available or very easy to prepare.

The procedure succeeds with a variety of propargylic alcohols. Primary, secondary and tertiary propargylic sulfinates rearrange upon exposure to silver cation in near quantitative yields (Figure 1). A chiral, non-racemic alcohol produced a chiral, non-racemic sulfone in the process with no apparent loss of stereochemical integrity. Overall the conversion from alcohol to sulfone is rapid and easy to perform and compares favorably with those methods that use more expensive metal catalysts.

Figure 1



1. Department of Chemistry, University of Missouri-Columbia, Columbia, MO, 65211, email: harmatam@missouri.edu. This work was supported by the National Science Foundation.

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11. (a) Klunder, J. M.; Sharpless, K. B. *J. Org. Chem.* **1987**, *52*, 2598-602. (b) Braverman, S.; Pechenick, T.; Zafrani, Y. *ARKIVOC (Gainesville, FL, United States)* **2004**, 51-63.

Appendix
Chemical Abstracts Nomenclature; (Registry Number)

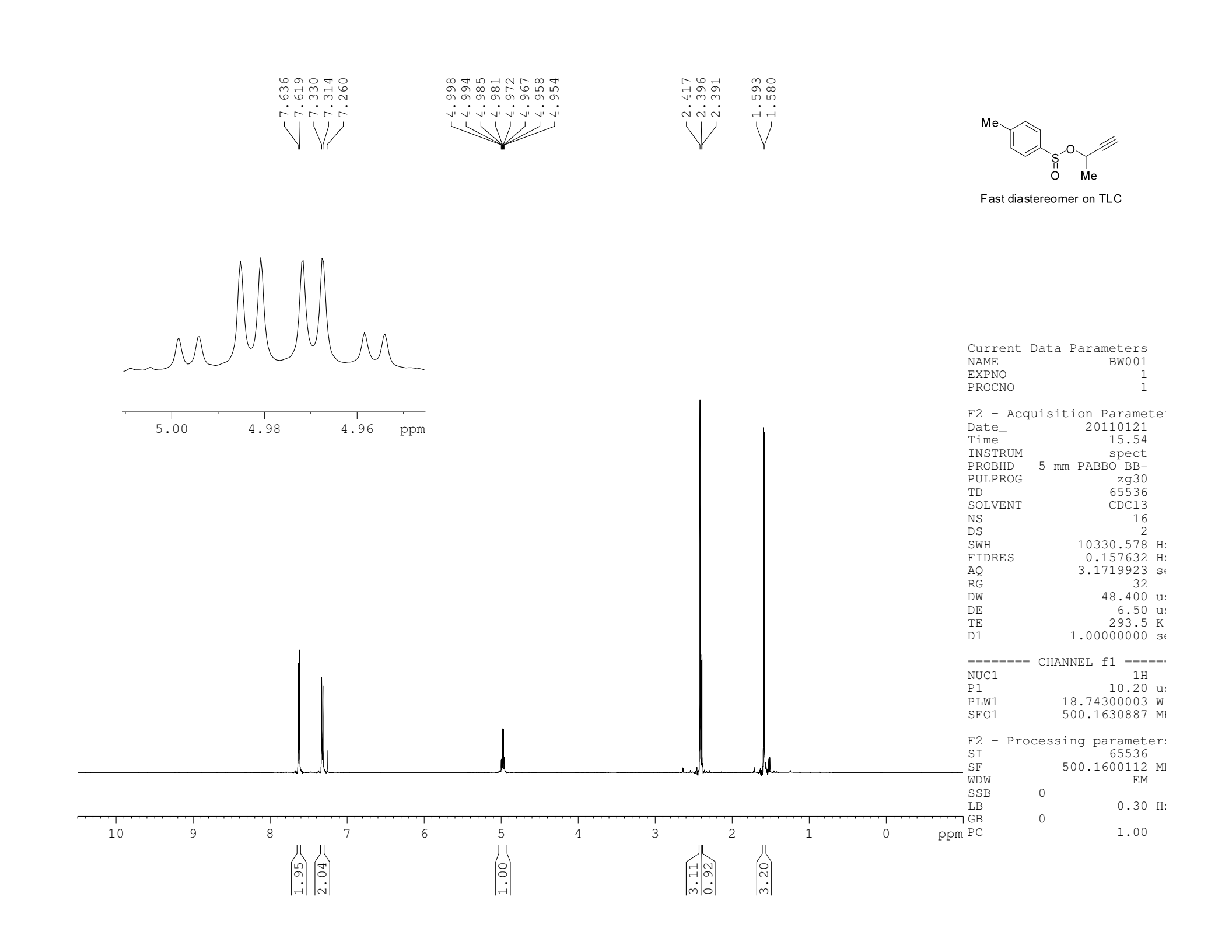
But-3-yn-2-yl 4-Methylbenzenesulfinate: Benzenesulfinic acid, 4-methyl-, 1-methyl-2-propyn-1-yl ester; (32140-54-8)
Tosyl chloride: Benzenesulfonyl chloride, 4-methyl-; (98-59-9)
Triethylamine: (121-44-8)
3-Butyn-2-ol; (2028-63-9)
Triphenylphosphine; (603-35-0)
1-(Buta-1,2-dien-1-ylsulfonyl)-4-methylbenzene: Benzene, 1-(1,2-butadien-1-ylsulfonyl)-4-methyl-; (32140-55-9)
Silver hexafluoroantimonate; (26042-64-8)

Michael Harmata was born in Chicago, Illinois in 1959. He received his A.B. degree from the University of Illinois-Chicago (1980) and his Ph.D. from the University of Illinois-Champaign/Urbana (1985). After an NIH postdoctoral fellowship at Stanford University, he began his independent career at the University of Missouri-Columbia (1986). He worked his way through the ranks since that time and was appointed the Norman Rabjohn Distinguished Professor of Chemistry in 2000. He was a Big 12 Faculty Fellow at the University of Texas-Austin (2006), was awarded a black belt in Taekwondo (2009) and was named the first Liebig Professor at the University of Giessen (2010).

Born in Huabei Oilfield, China, in 1982, Zhengxin Cai received his B.S. degree from School of Pharmaceutical Science and Technology, Tianjin University (2005), and has been studying organic synthesis at the University of Missouri under the direction of Michael Harmata since then. While focusing on organic synthesis, his interests extend to anything that is related to drug discovery and development. Besides "cooking" in the hood, he enjoys reading intellectually stimulating books and plays many kinds of sports, including chess, soccer, and table tennis.

Chaofeng Huang was born in Putian, China in 1978. He received his B.S. degree in Chemistry at Tongji University (Shanghai, China, 2001) and M.S. degree in Medicinal Chemistry at Shanghai Institute of Pharmaceutical Industry (Shanghai, China, 2004). In 2009, he earned his Ph.D. degree from the University of Missouri-Columbia. Currently, he is working as a postdoctoral fellow in the Department of Chemistry at the University of Pittsburgh. In October 2010, he will work as a postdoctoral research associate in the Mallinckrodt Institute of Radiology Department at the Washington University School of Medicine in St. Louis.

Bo Wen received his B.S. degree in applied chemistry (2001) from Lanzhou University, China. He then worked in Lanzhou Institute of Chemical Physical, Chinese Academy of Science. In 2005, he moved to West Virginia University for graduate study with Professor Kung Wang, where his research was focused on the synthesis of helical and bowl-shaped polycyclic aromatic compounds. After completing his Ph.D. in 2010, he joined the group of Professor Kay Brummond at the University of Pittsburgh as a postdoctoral research associate and is currently working on the synthesis of biological active guaianolides.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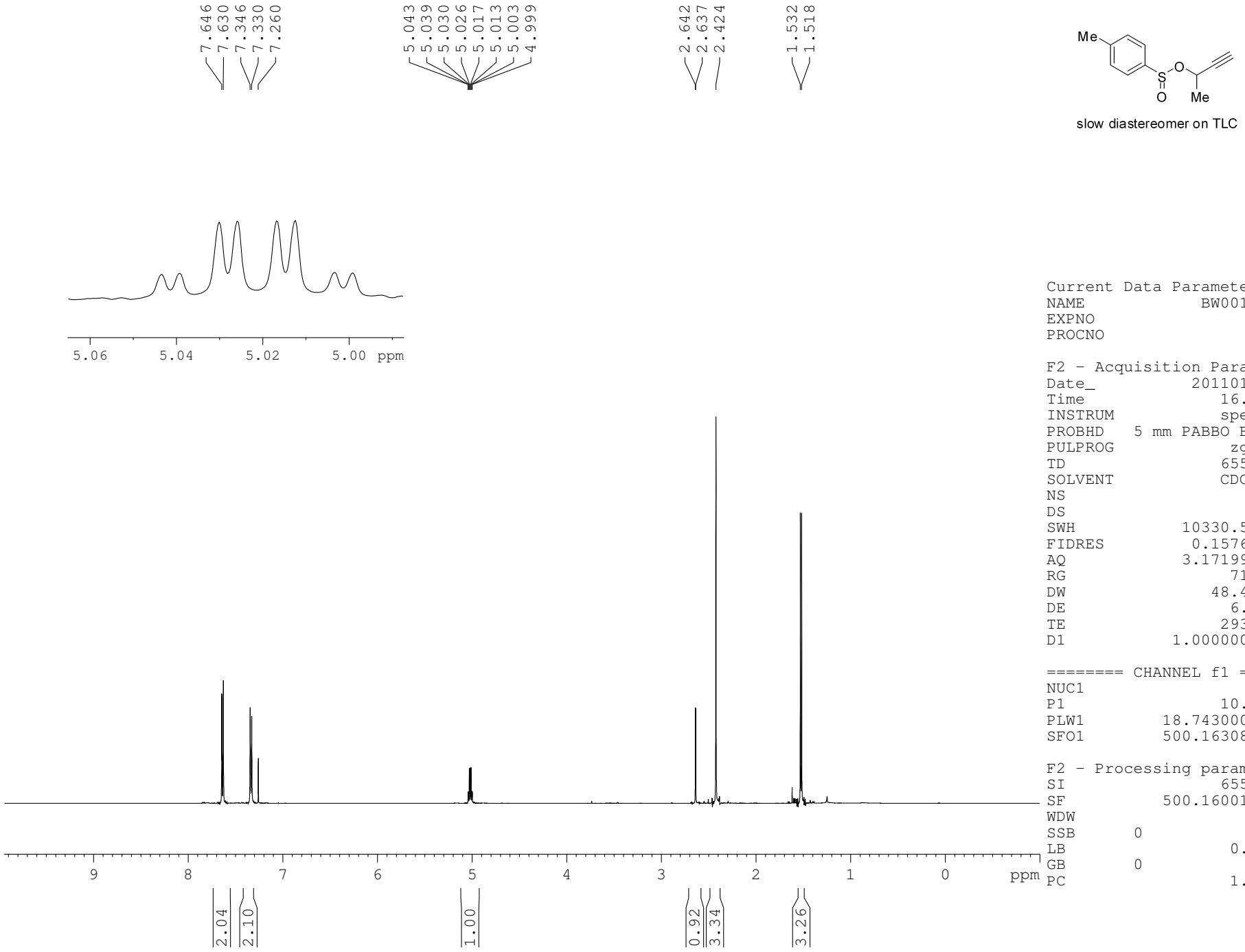
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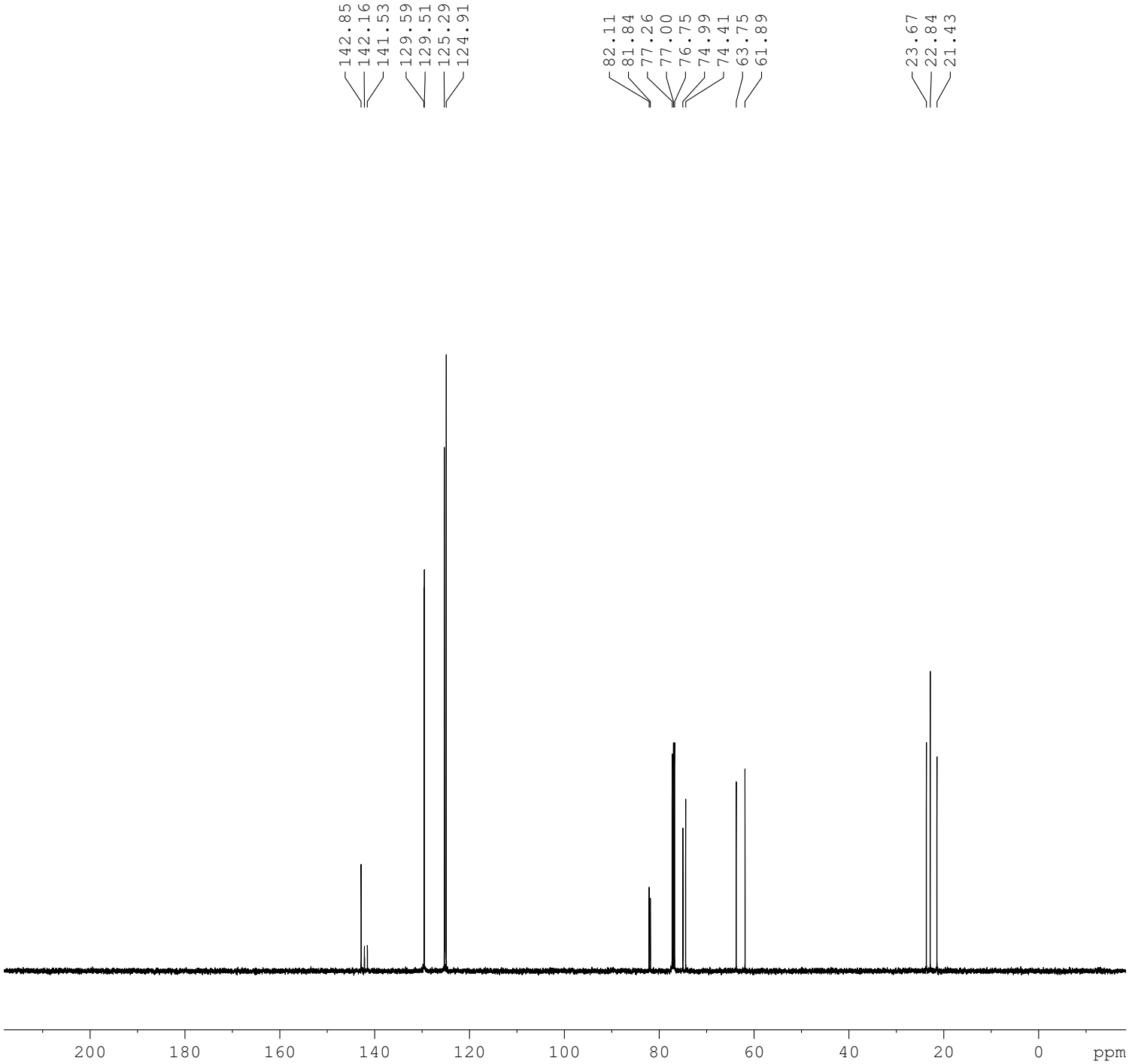

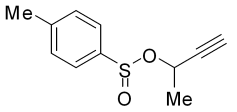

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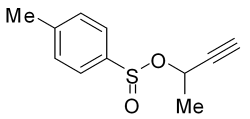

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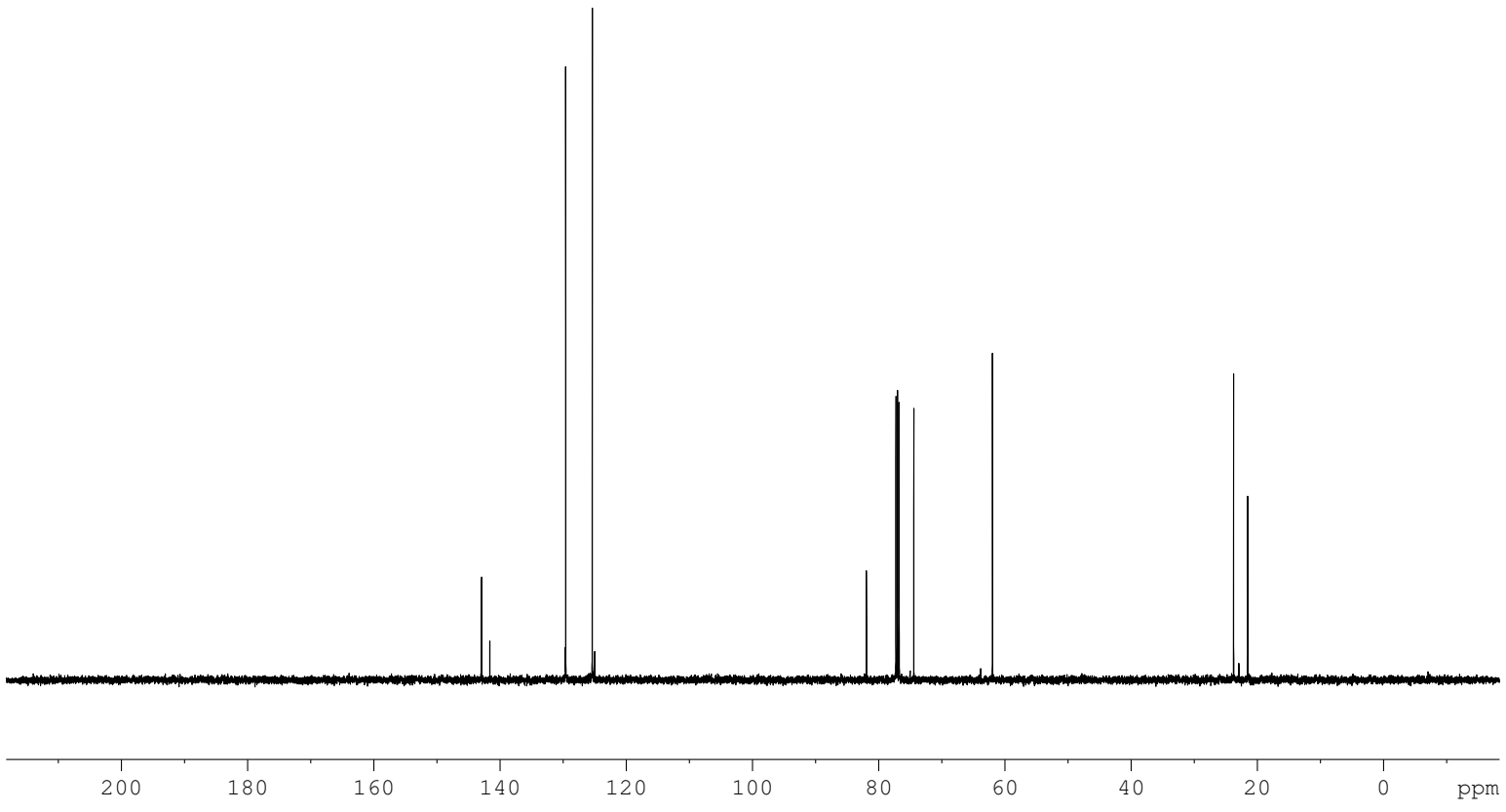


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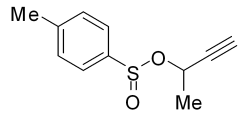

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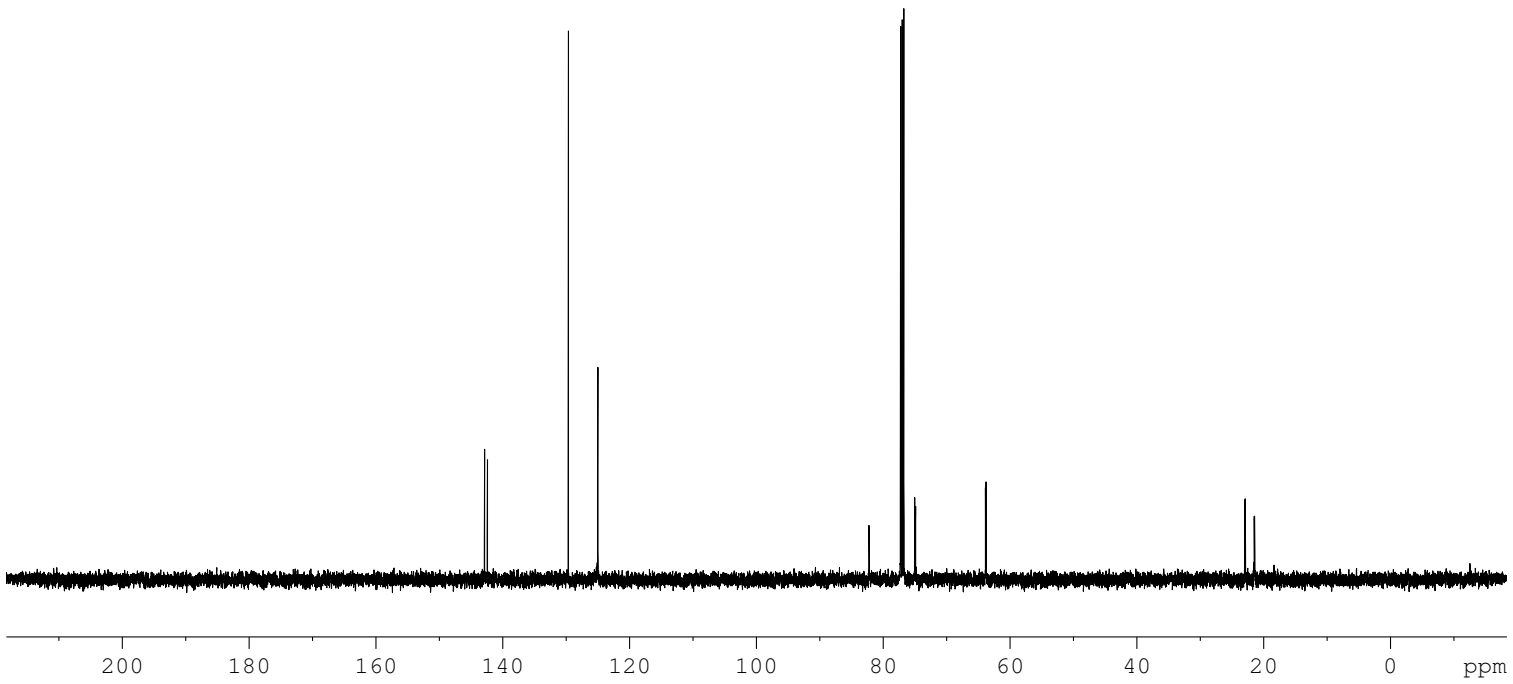


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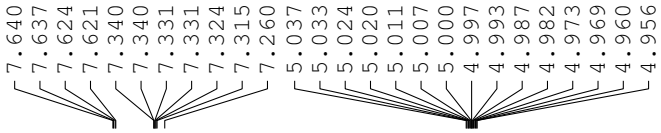
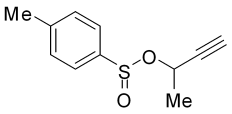
SFO1           125.7779080 M

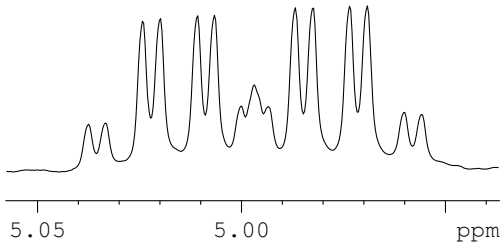
======== CHANNEL f2 =====
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 u
PLW2           18.74300003 W
PLW12           0.36736000 W
PLW13           0.18408000 W
SFO2          500.1620006 M

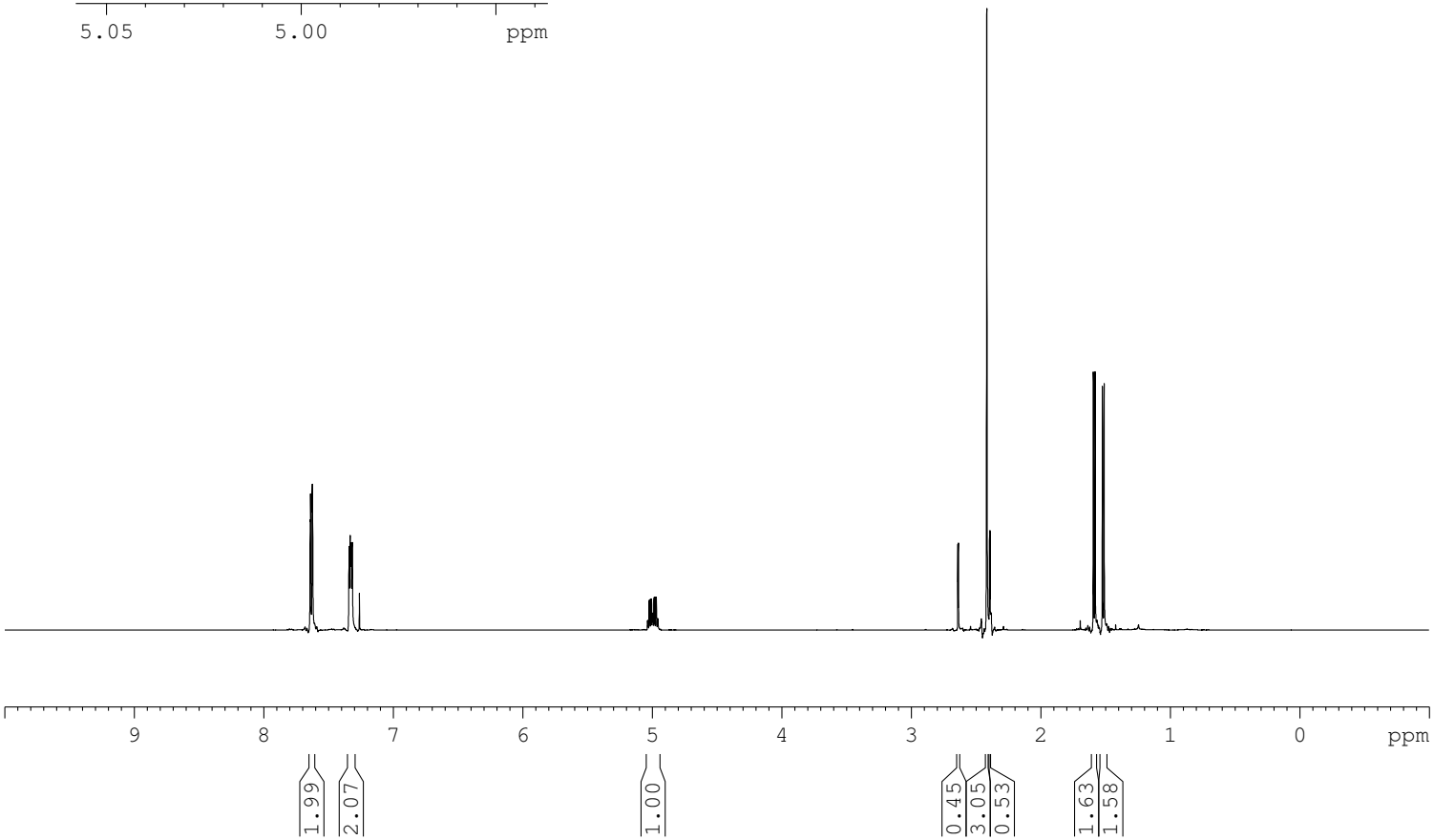
F2 - Processing parameter
SI                    32768
SF           125.7653353 M
WDW                     EM
SSB       0
LB                    1.00 H
GB        0
PC                    1.40
```





mixture of diastereomer





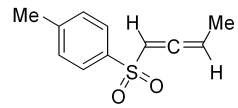

```
Current Data Parameters
NAME                  BW001-M
EXPNO                       1
PROCNO                      1

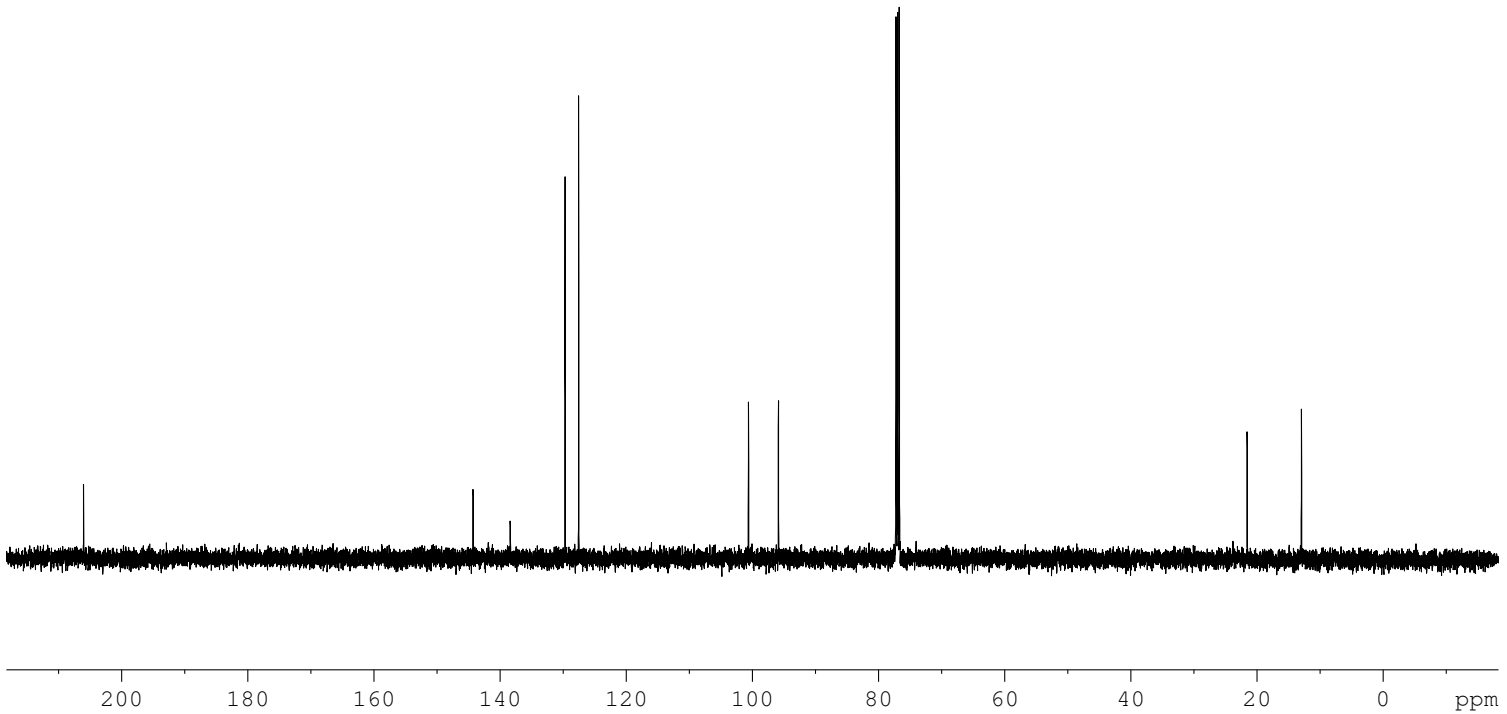
F2 - Acquisition Parameters
Date_               20110122
Time                   13.27
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH               10330.578 Hz
FIDRES             0.157632 Hz
AQ                3.1719923 sec
RG                        32
DW                   48.400 usec
DE                    6.50 usec
TE                   293.8 K
D1                1.00000000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    10.20 usec
PLW1            18.74300003 W
SFO1           500.1630887 MHz

F2 - Processing parameters
SI                     65536
SF             500.1600112 MHz
WDW                       EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     1.00
```

206.08
144.31
138.44
129.73
127.58
100.65
95.90
77.25
77.00
76.75
21.59
12.97





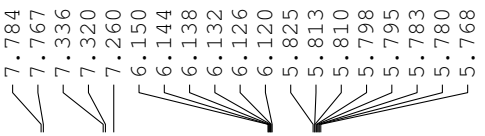

```
Current Data Parameters
NAME                  BW003
EXPNO                     2
PROCNO                    1

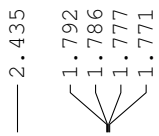
F2 - Acquisition Parameters
Date_              20110122
Time                  15.36
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     102
DS                       4
SWH              29761.904 Hz
FIDRES            0.454131 Hz
AQ               1.1010548 sec
RG                     203
DW                  16.800 use
DE                   6.50 use
TE                  294.2 K
D1              2.00000000 sec
D11             0.03000000 sec

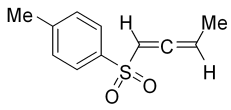
======== CHANNEL f1 =======
NUC1                   13C
P1                    8.80 use
PLW1         112.58000183 W
SFO1          125.7779080 MHz

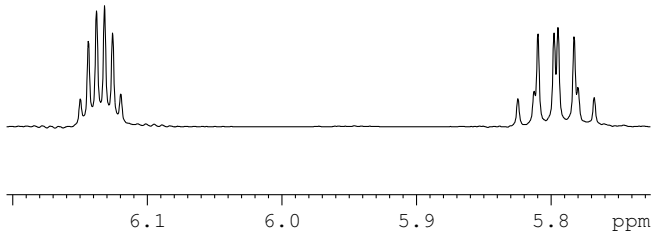
======== CHANNEL f2 =======
CPDPRG2           waltz16
NUC2                   1H
PCPD2                80.00 use
PLW2          18.74300003 W
PLW12          0.36736000 W
PLW13          0.18408000 W
SFO2          500.1620006 MHz

F2 - Processing parameters
SI                    32768
SF           125.7653373 MHz
WDW                     EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```










```
Current Data Parameters
NAME                  BW003
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110122
Time                  15.20
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH              10330.578 Hz
FIDRES            0.157632 Hz
AQ               3.1719923 sec
RG                       64
DW                  48.400 usec
DE                   6.50 usec
TE                   293.7 K
D1              1.00000000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                   10.20 usec
PLW1          18.74300003 W
SFO1          500.1630887 MHz

F2 - Processing parameters
SI                    65536
SF            500.1600112 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```