Electronic Supplementary Material (ESI) for RSC Advances

Organocatalysis by aprotic imidazolium zwitterion: A dramatic anion–cation cooperative effect on azide-nitrile cycloaddition

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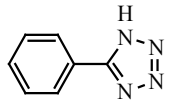
General: Melting points were determined on a glass disk with an electrical bath and are uncorrected. ^{1}H NMR spectra were determined on a Bruker 400 (400 MHz) spectrometer as solutions in DMSO-d_6. Chemical shifts are expressed in parts per million (δ) and are referenced to tetramethylsilane (TMS) as internal standard and the signals were reported as s (singlet), d (doublet), t (triplet), m (multiplet) and coupling constants *J* were given in Hz. ^{13}C NMR spectra were recorded at 100 MHz in $CDCl_3$ and DMSO-d_6 solution. TLC was done on silica gel coated glass slide (Merck, Silica gel G for TLC). IR spectra were taken as KBr plates. Commercially available substrates were freshly distilled before the reaction. Solvents, reagents and chemicals were purchased from Aldrich, Fluka, Merck, SRL, Spectrochem and Process Chemicals. All reactions involving moisture sensitive reactants were executed using oven dried glassware. Zwitterions were prepared using our reported method.[1]

Typical procedure for the synthesis of 5-phenyl-1*H*-tetrazole:

In a 50 mL round bottom flask benzonitrile nitrile (103 mg, 1 mmol), and NaN_3 (98 mg, 1.5 mmol) were taken in presence of zwitterionic-salt (22 mg, 10 mol%) and the whole mixture was stirred at 120 ^{o}C (oil bath) for 12h. After completion of the reaction, the reaction mixture, being cooled to room temperature was treated with 4 M HCl (20 mL) and stirred vigorously. The solid product was filtered under suction and then recrystallized from hot ethanol to obtain the pure product. The identity and purity of the product was confirmed by ^{1}H and ^{13}C NMR spectroscopic analysis.

Spectral and analytical data of all synthesized tetrazoles are presented in order of their entries (Table 2):

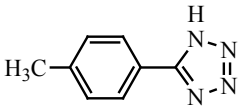
5-Phenyl-1*H*-tetrazole (3a):[2]



White solid, Yield: 123 mg, 84%; mp. 211-212 °C,

IR (KBr): 3207, 3000, 1611, 1562, 1493, 1466, 688 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 8.07-8.02 (m, 2H), 7.64-7.58 (m, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 155.2, 131.2, 129.4, 126.9, 124.1.

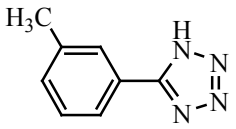
5-*p*-Tolyl-1*H*-tetrazole (3b):[3]



White solid, Yield: 131 mg, 82%; mp. 251-252 ^oC;

IR (KBr): 3440, 1595, 1562, 1485, 1412 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6): δ 7.92 (d, J = 8.0 Hz, 2H), 7.41 (d, J = 8.0 Hz, 2H), 2.38 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 155.3, 141.2, 129.9, 126.9, 121.2, 21.0.

5-*m*-Tolyl-1*H*-tetrazole (3c):[5]

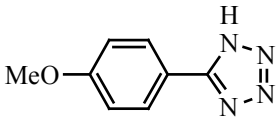


Off white solid, Yield: 130 mg, 81%; mp.143-144 °C;

IR (KBr): 3442, 1598, 1563, 1484, 1411 cm^{-1};

^{1}H NMR (400 MHz, DMSO-d_6): δ 7.86 (s, 1H), 7.82 (d, J = 7.6 Hz, 1H), 7.48 (t, J = 7.6 Hz, 1H), 7.39 (d, J = 7.6 Hz, 1H), 2.39 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d6): δ 155.2, 138.8, 131.9, 129.3, 127.4, 124.1, 124.0, 20.9.

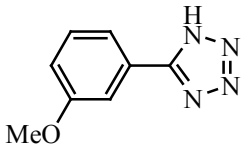
5-(4-Methoxyphenyl)-1*H*-tetrazole (3d):[2]



White solid, Yield: 134 mg, 76%; mp. 231-233 ^{o}C.

IR (KBr): 3200, 1298, 1184, 1035, 750 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.98 (dd, J_1 = 6.8 Hz, J_2 = 2.0 Hz), 7.16 (d, J = 8.8 Hz, 2H), 3.84 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 161.5, 154.7, 128.6, 116.3, 114.8, 55.4.

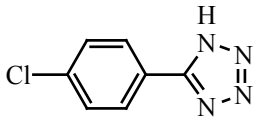
5-(3-Methoxyphenyl)-1*H*-tetrazole (3e):[6]



White solid, Yield: 132 mg, 75%, mp. 156-158 °C;

IR (KBr): 3199, 3161, 3043, 2985, 1325, 1280, 1153, 1041, 858, 742 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.63-7.50 (m, 3H), 7.16 (d, J = 8.0 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 159.7, 155.2, 130.6, 125.3, 119.2, 117.0, 112.1, 55.4.

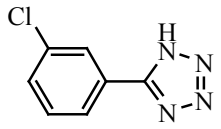
5-(4-Chlorophenyl)-1*H*-tetrazole (3f):[3]



White solid, Yield: 130 mg, 72%; mp. 261-263 °C;

IR (KBr): 3419, 2927, 1602, 1457, 1383, 1055, 765 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.88 (d, J = 8.0 Hz, 2H), 7.66 (d, J = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 150.1, 138.5, 134.1, 129.7, 129.4, 128.3.

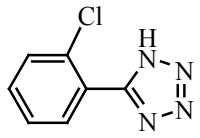
5-(3-Chlorophenyl)-1*H*-tetrazole (3g):[3]



White solid, Yield: 131 mg, 73%; mp. 137-138 °C;

IR (KBr): 3407, 2915, 2796, 2720, 1599, 1459, 1439, 1390, 1158, 1089, 1055, 870, 766 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 8.08 (s, 1H), 8.03-8.00 (m, 1H), 7.68-7.65 (m, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 154.8, 134.0, 131.4, 131.0, 126.6, 126.3, 125.6.

5-(2-Chlorophenyl)-1*H*-tetrazole (3h):[3]

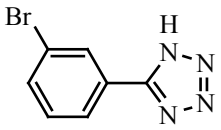


White solid, Yield: 130 mg, 72%; mp. 179-181 °C

IR (KBr): 3402, 2930, 1689, 1450, 1160, 1050, 760 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.81 (dd, J_1 = 7.6 Hz, J_2 = 1.6 Hz, 1H), 7.72 (dd, J_1 = 8.0, J_2 = 1.2 Hz, 1H), 7.64 (dt, J_1 = 8.0 Hz,

J_2 = 1.6 Hz, 1H), 7.57 (dt, J_1 =7.6 Hz, J_2 = 1.2 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 152.3, 131.6, 131.0, 130.7, 129.4, 126.8, 123.1.

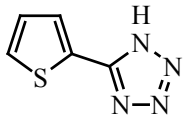
5-(3-Bromophenyl)-1*H*-tetrazole (3i):[4]



White solid, Yield: 168 mg, 75%; mp. 146-147 °C;

IR (KBr): 3424, 3066, 1714, 1604, 1557, 1092, 797 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 16.67 (br, 1H), 8.22-8.21 (m, 1H), 8.06 (d, J = 7.8 Hz, 1H), 7.82-7.79 (m, 1H), 7.58 (t, J = 7.92 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 155.1, 134.3, 132.0, 129.8, 127.0, 126.4, 122.8.

5-(Thiophen-2-yl)-1*H*-tetrazole (3j):



White solid, Yield: 123 mg, 81%; mp. 195 °C;

IR (KBr): 3076, 2950, 1759, 1593, 1505, 961, 718 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 16.88 (br, 1H), 7.90-7.89 (d, J = 5.04 Hz, 1H), 7.81-7.80 (d, J = 3.64 Hz, 1H), 7.29 (t, J = 4.04 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-d_6): δ 151.7, 130.8, 129.6, 129.0, 125.8. Anal. Cald. for $C_5H_4N_4S$: C, 39.46; H, 2.65; N, 36.82%. Found: C, 39.41; H, 2.58; N, 36.76%.

References:

1. (*a*) D. Kundu, R. K. Debnath, A. Majee and A. Hajra, *Tetrahedron Lett.,* 2009, **50**, 6998; (*b*) D. Kundu, A. Majee and A. Hajra, *Catal. Commun.,* 2010, **11**, 1157; (*c*) M. Rahman, A. K. Bagdi, D. Kundu, A. Majee and A. Hajra, *J. Heterocycl. Chem.* 2012, **49**, 1224.
2. Z. P. Demko and K. B. Sharpless, *J. Org. Chem*., 2001, **66**, 7945.
3. L. Lang, B. Li, W. Liu, L. Jiang, Z. Xu and G. Yin, *Chem. Commun.,* 2010, **46**, 448.
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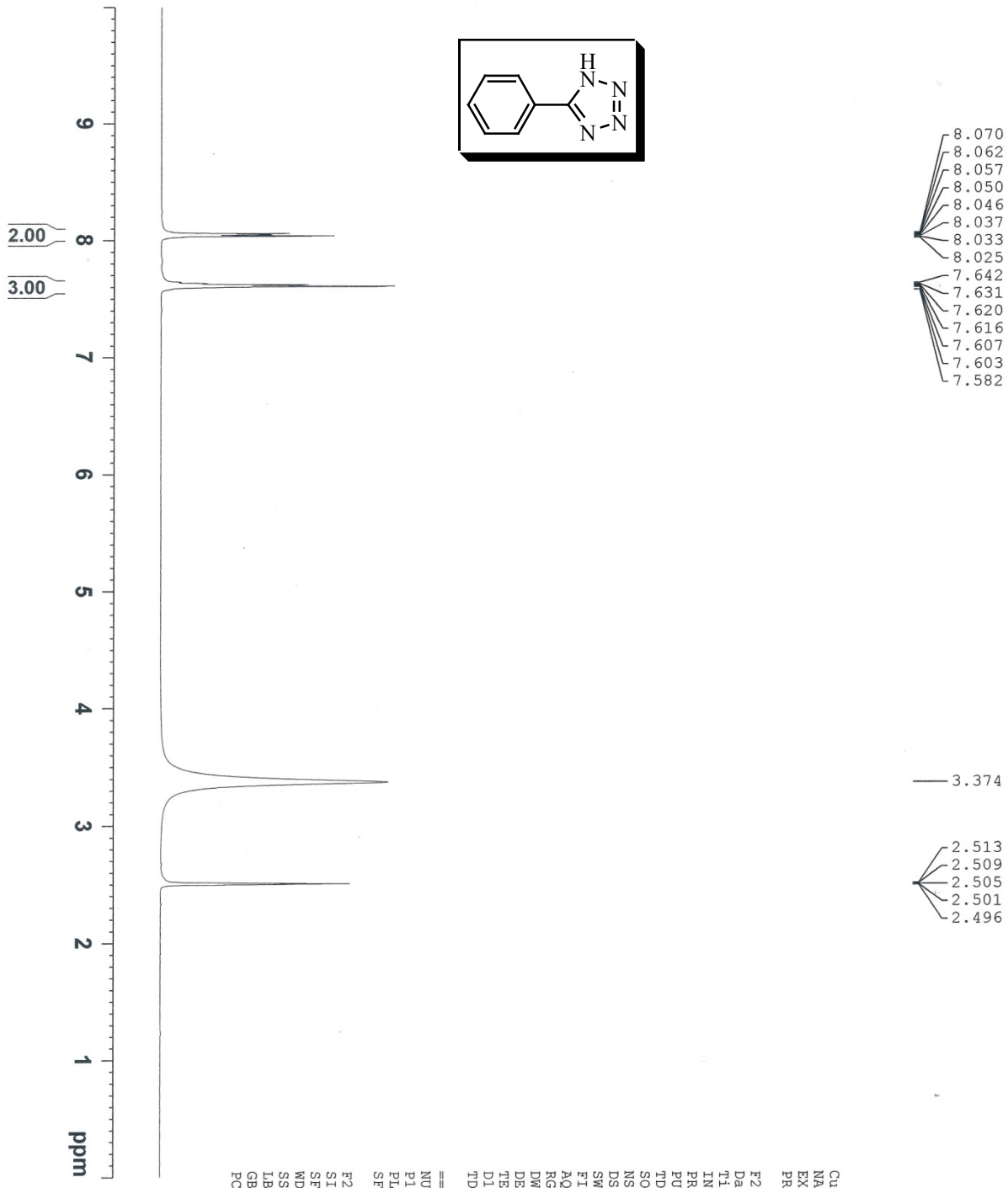
5. P. B. Palde and T. F. Jamison, *Angew. Chem., Int. Ed*., 2011, **50**, 3525.
6. D. Amantini, R. Beleggia, F. Fringuelli, F. Pizzo and L. Vaccaro, *J. Org. Chem.,* 2004, **69**, 2896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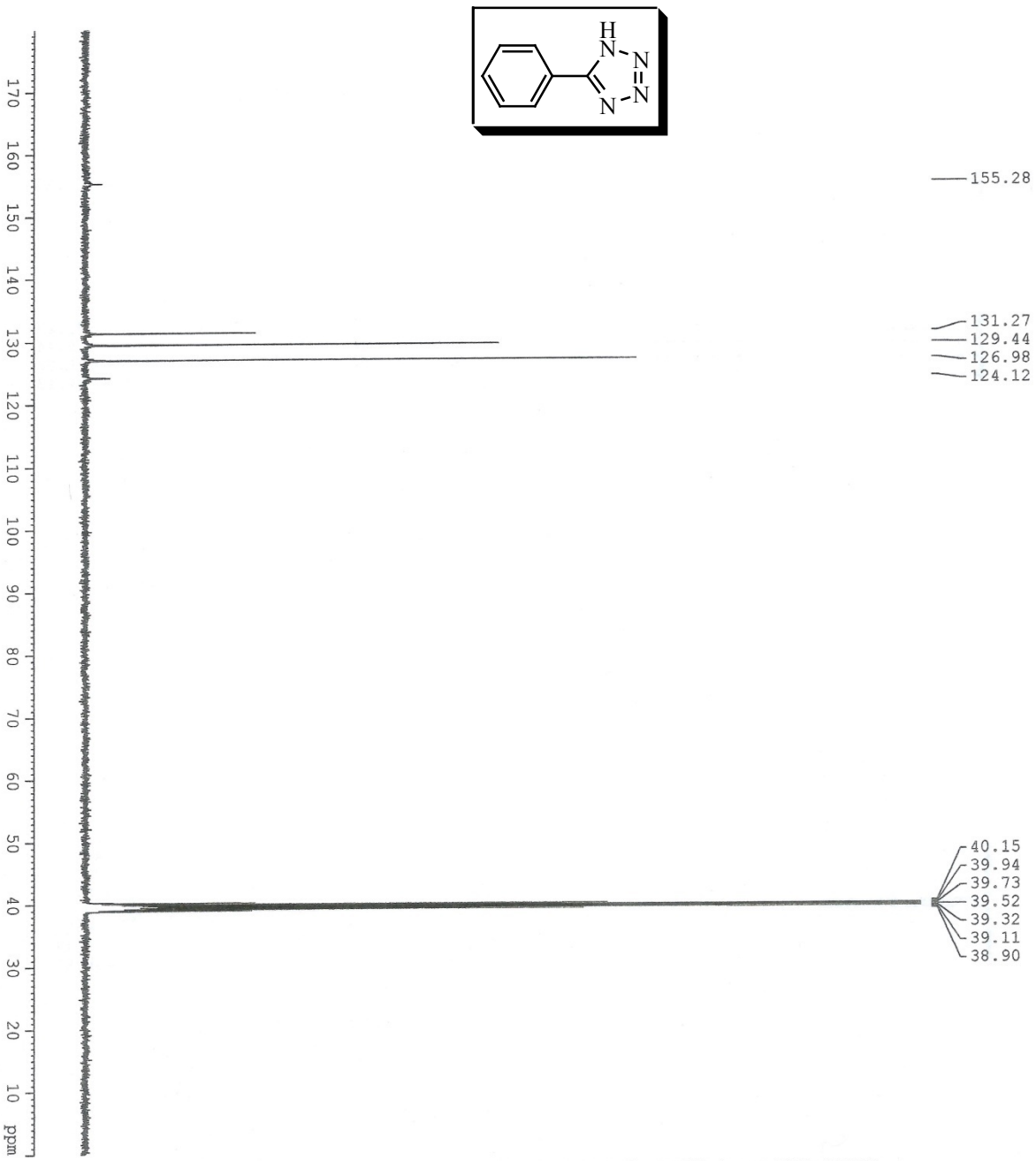

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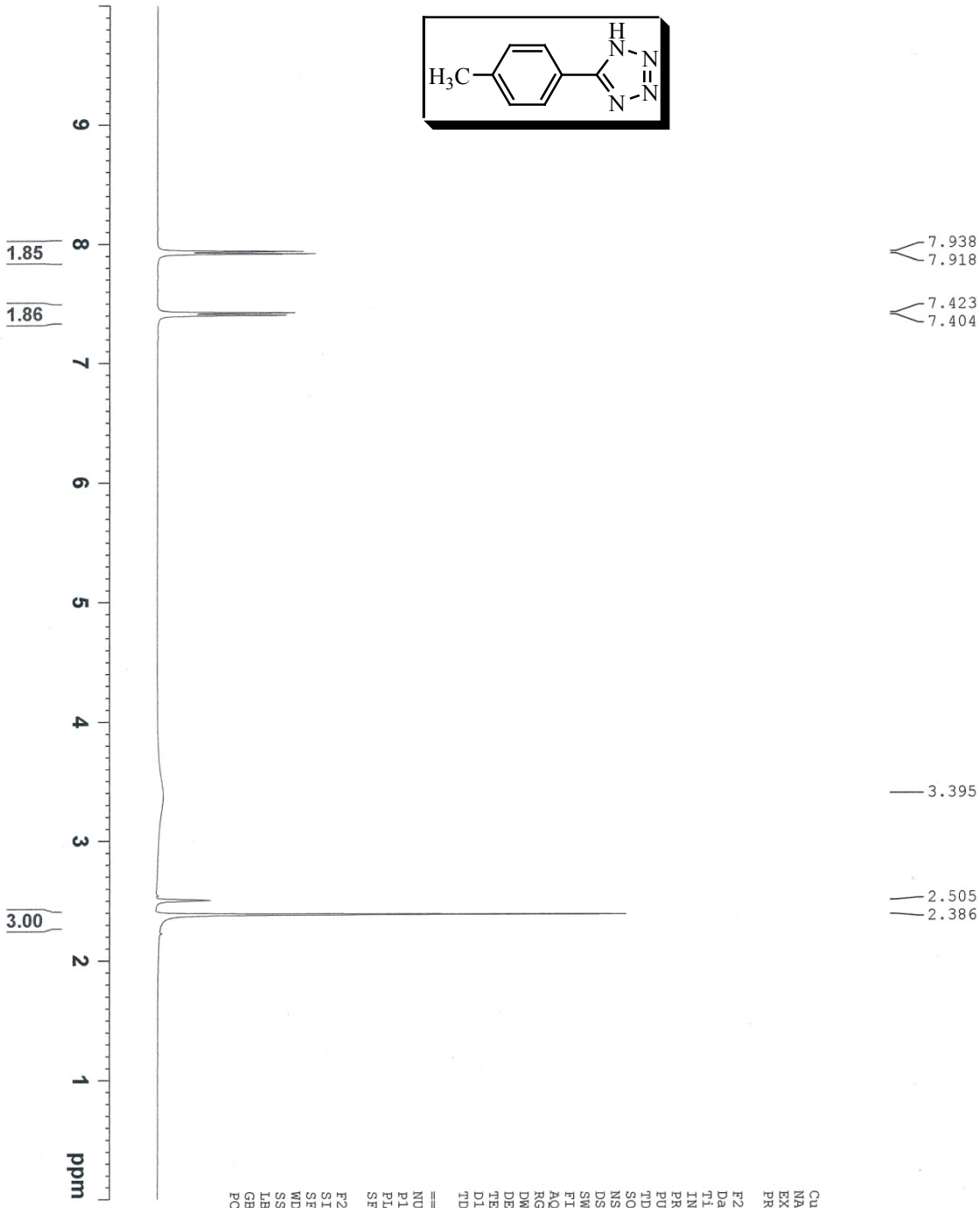

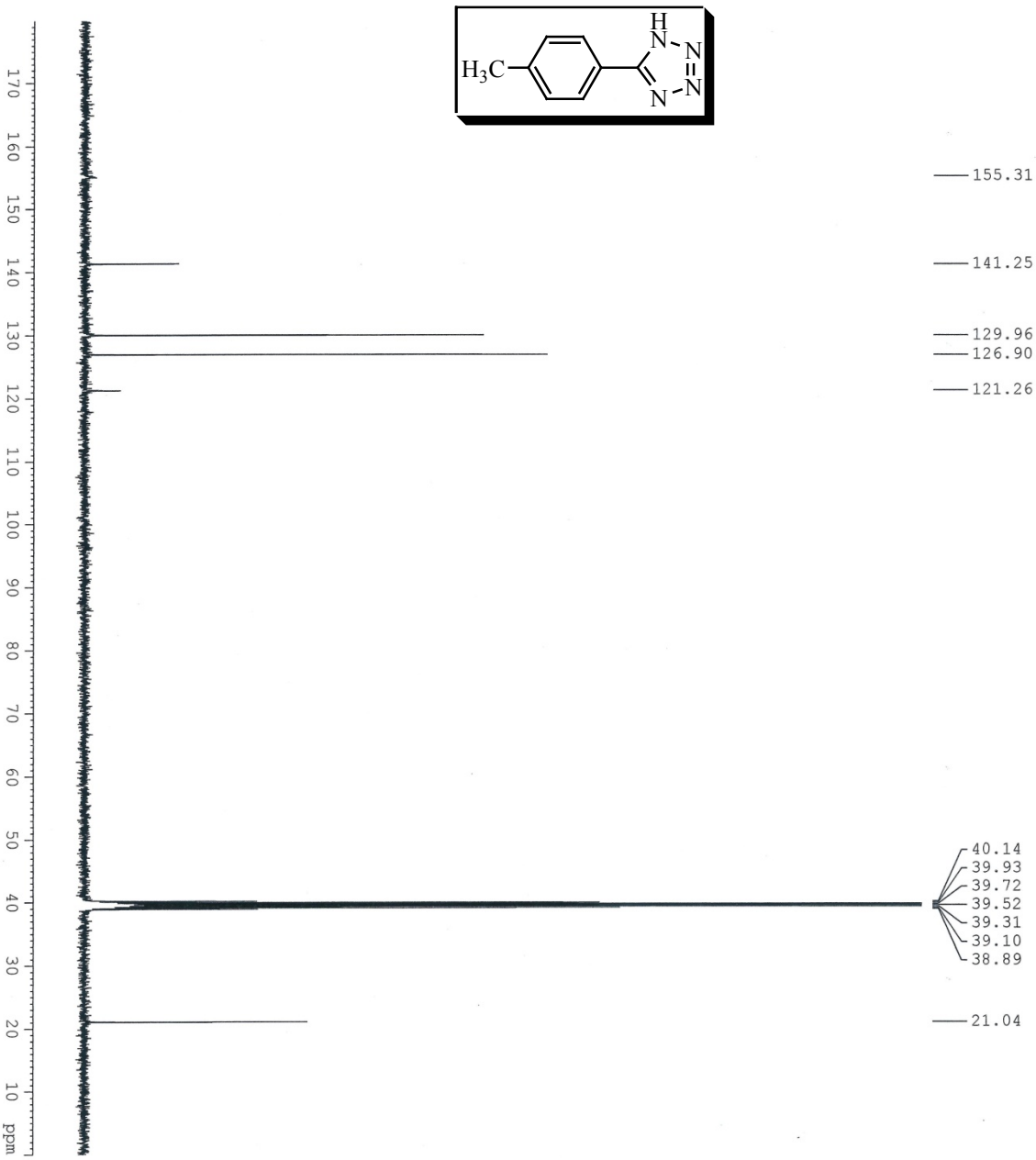


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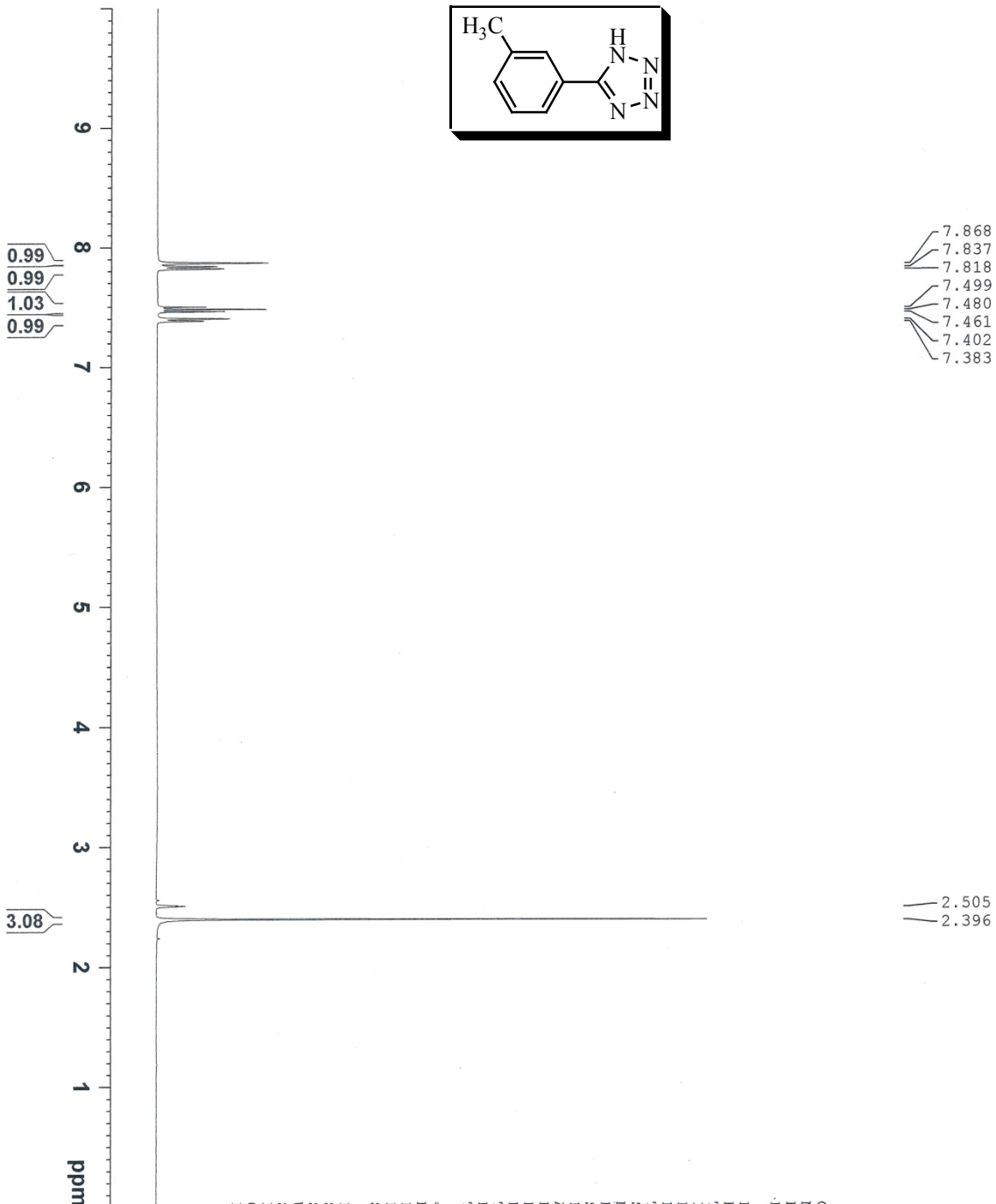


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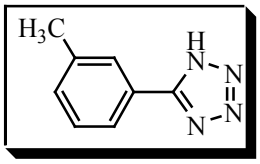


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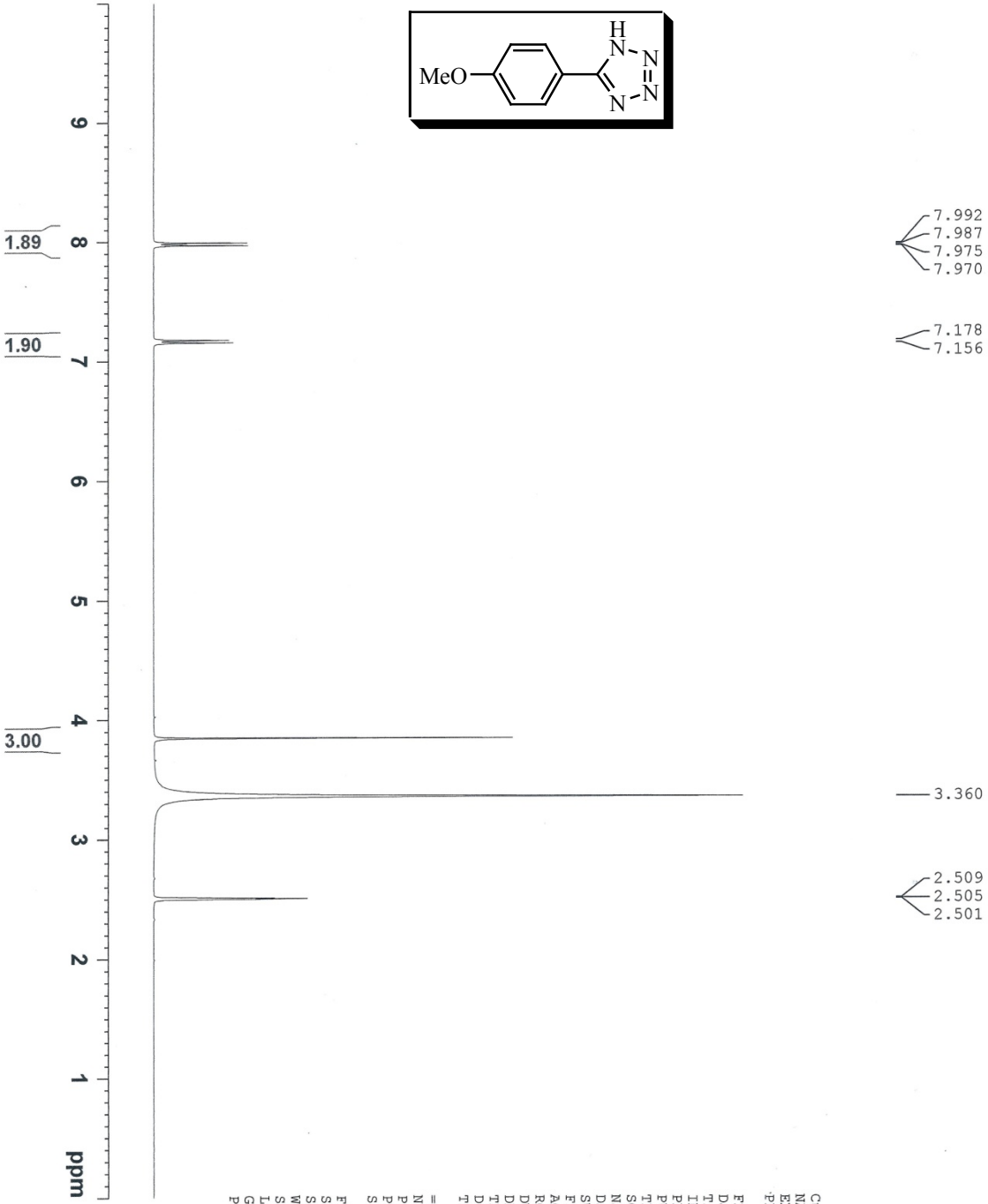

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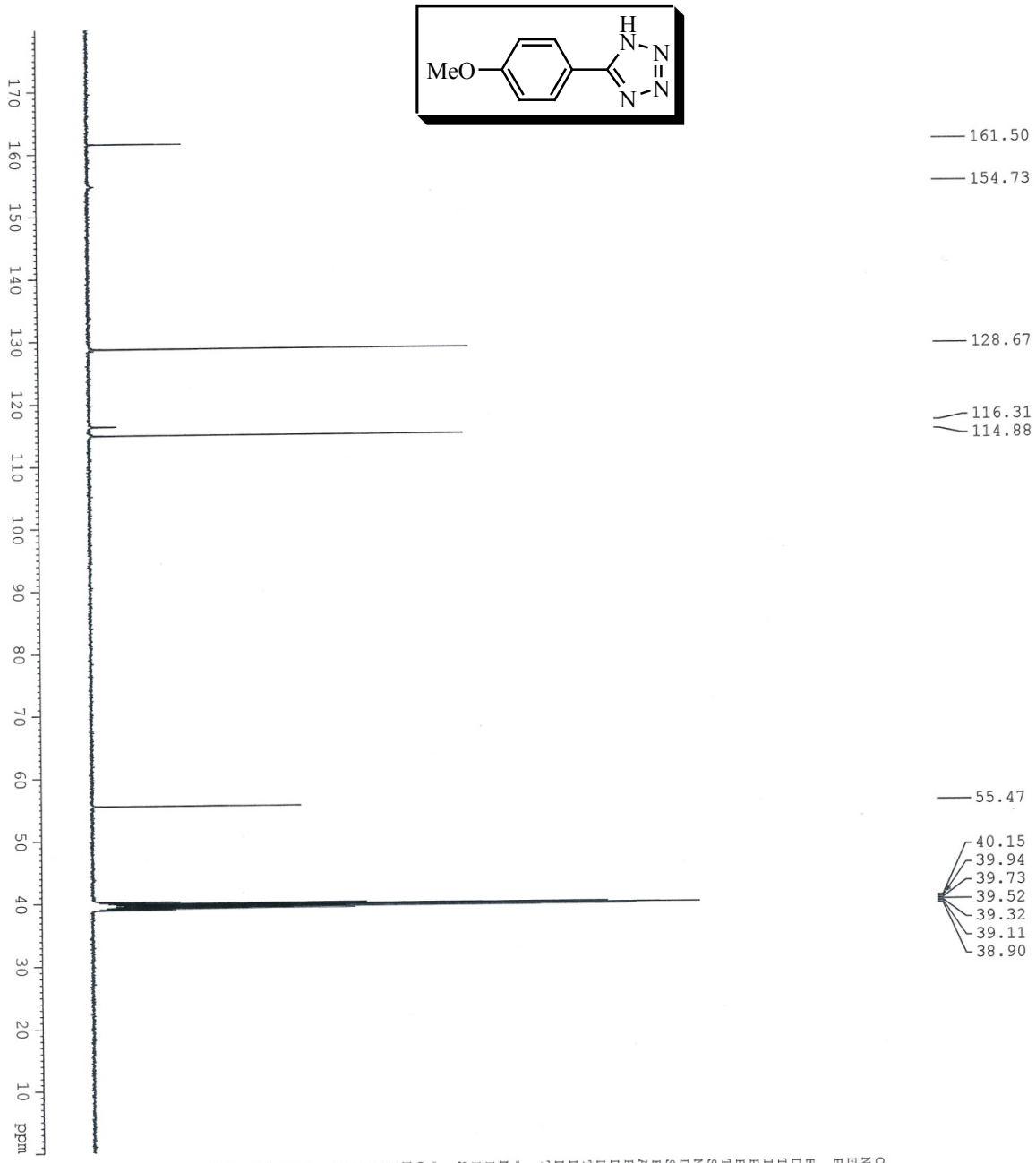


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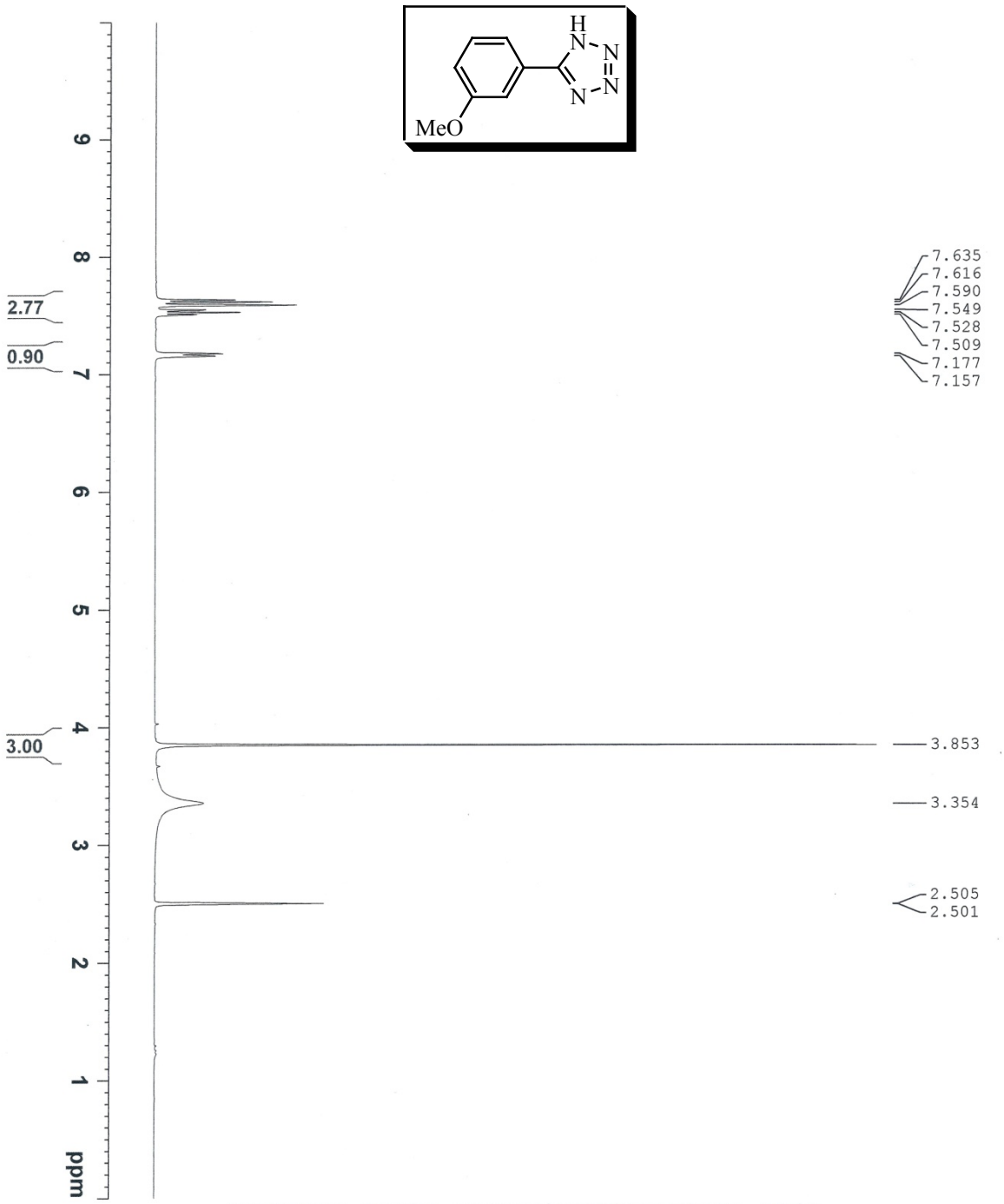


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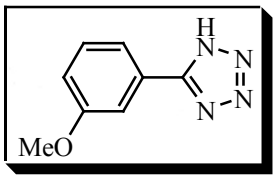
NUC1                     1H
P1                    14.75 usec
PLW1             11.99499989 W
SFO1            400.1524711 MHz

F2 - Processing parameters
SI                     16384
SF              400.1500000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



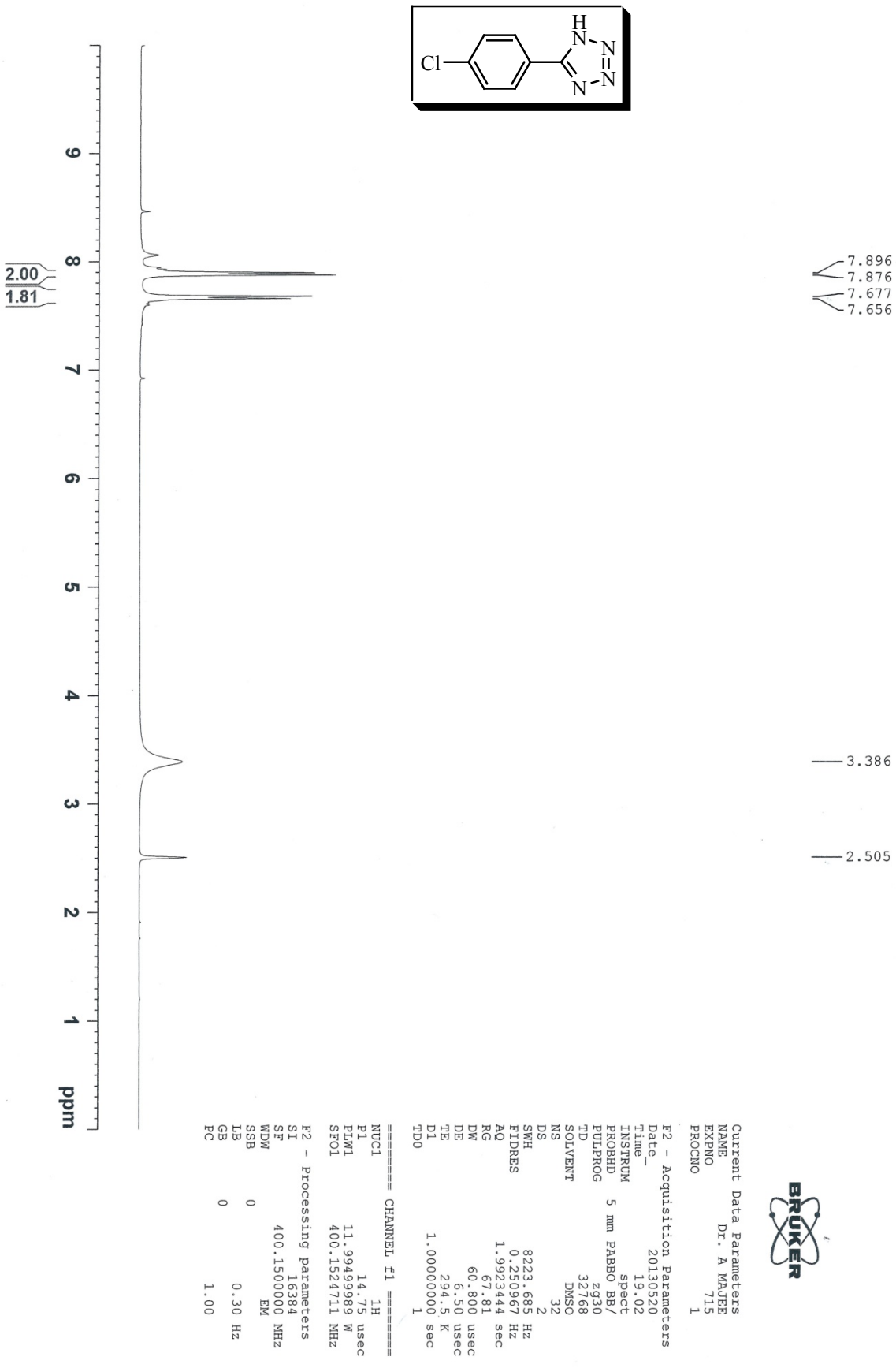

```
Current Data Parameters
NAME                  Dr. A MAJEE
EXPNO                         726
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20130521
Time_                       20.15
INSTRUM                      spect
PROBHD        5 mm PABBO BB/
PULPROG                  zgpg30
TD                            32768
SOLVENT                      DMSO
NS                              400
DS                                2
SWH                       24038.461 Hz
FIDRES                        0.733596 Hz
AQ                            0.6816244 sec
RG                               87.66
DW                              20.800 usec
DE                               6.50 usec
TE                              297.2 K
D1                        2.00000000 sec
D11                       0.03000000 sec
TD0                               1

========== CHANNEL f1 ==========
NUC1                            13C
P1                             8.90 usec
PLW1                  54.00000000 W
SFO1                  100.6278588 MHz

========== CHANNEL f2 ==========
CPDPRG2                     waltz16
NUC2                            1H
PCPD2                         80.00 usec
PLW2                  12.00000000 W
PLW12                  0.40792999 W
PLW13                  0.26107001 W
SFO2                  400.1516006 MHz

F2 - Processing parameters
SI                            16384
SF                    100.6178354 MHz
WDW                              EM
SSB                 0
LB                            1.00 Hz
GB                  0
PC                            1.40
```

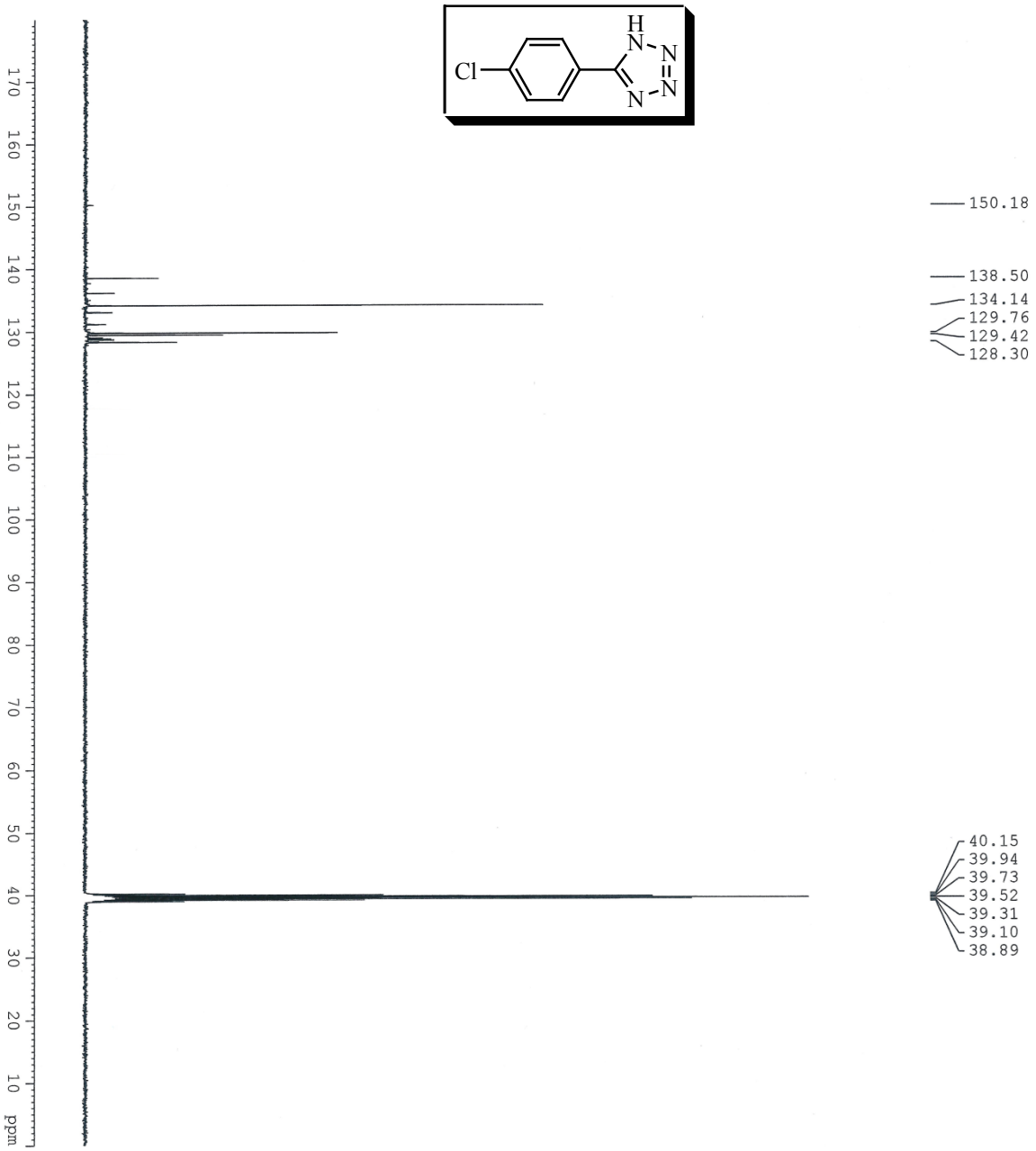


Current Data Parameters
NAME Dr. A MAJEE
EXPNO 715
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130520
Time 19.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.992344 sec
RG 67.81
DW 60.800 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.75 usec
PLW1 11.99499989 W
SFO1 400.1524711 MHz

F2 - Processing parameters
SI 16384
SF 400.1500000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



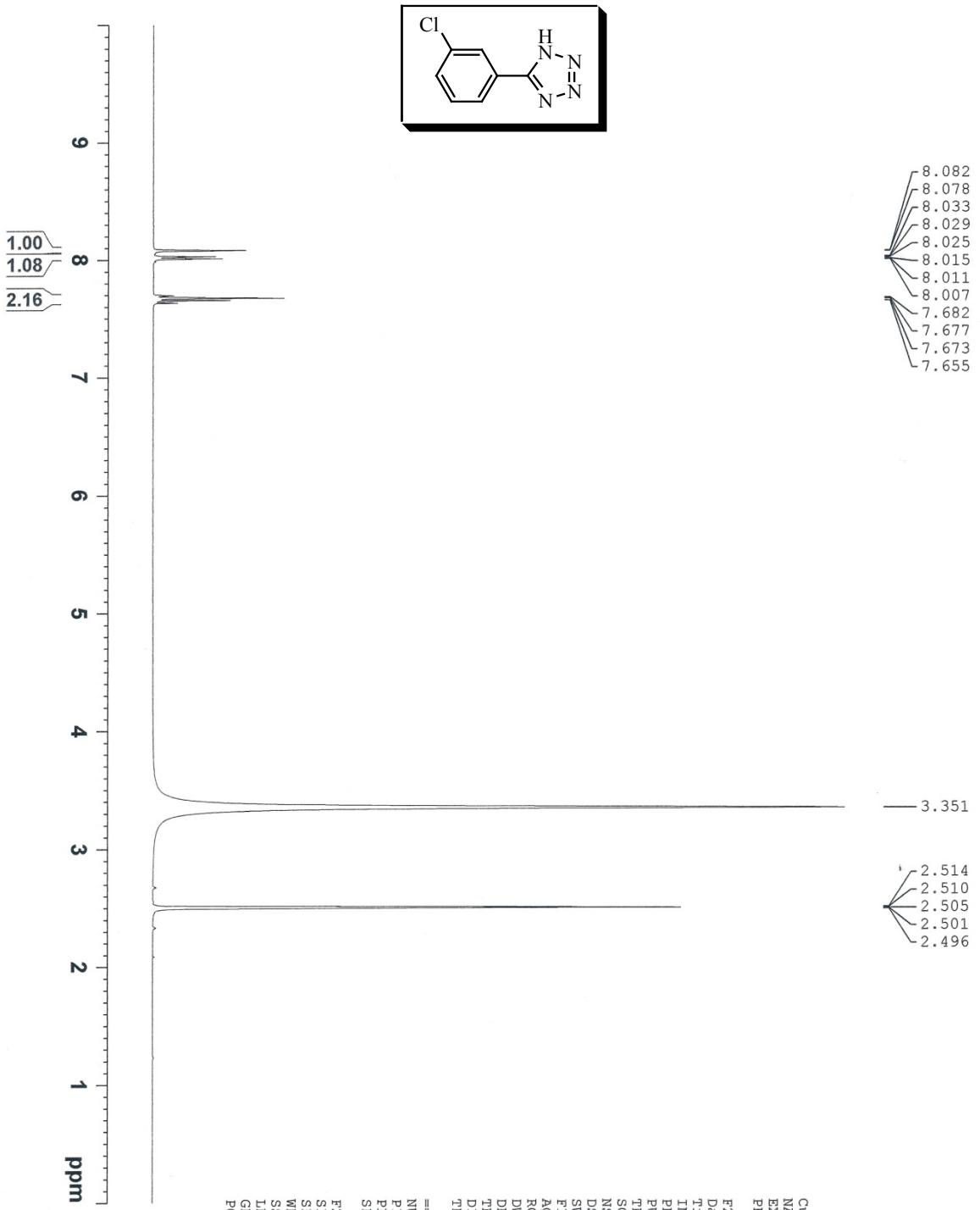

```
Current Data Parameters
NAME                 Dr. A MAJEE
EXPNO                       728
PROCNO                        1

F2 - Acquisition Parameters
Date_               20130522
Time_                   11.17
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                 zgpg30
TD                       32768
SOLVENT                  DMSO
NS                         400
DS                           2
SWH                  24038.461 Hz
FIDRES                0.733596 Hz
AQ                    0.6816244 sec
RG                         87.66
DW                      20.800 usec
DE                       6.50 usec
TE                     296.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        13C
P1                         8.90 usec
PLW1                 54.00000000 W
SFO1                100.6278588 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                        1H
PCPD2                    80.00 usec
PLW2                 12.00000000 W
PLW12                 0.40792999 W
PLW13                 0.26107001 W
SFO2                400.1516006 MHz

F2 - Processing parameters
SI                        16384
SF                  100.6178413 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```




```
Current Data Parameters
NAME                  Dr. A MAJEE
EXPNO                         713
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20130520
Time                        18.54
INSTRUM                      spect
PROBHD     5 mm PABBO BB/
PULPROG                     zg30
TD                         32768
SOLVENT                     DMSO
NS                             32
DS                              2
SWH                      8223.685 Hz
FIDRES                   0.250967 Hz
AQ                       1.9923444 sec
RG                          87.66
DW                         60.800 usec
DE                          6.50 usec
TE                         294.4 K
D1                       1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
NUC1                           1H
P1                          14.75 usec
PLW1                    11.99499989 W
SFO1                   400.1524711 MHz

F2 - Processing parameters
SI                          16384
SF                   400.1500000 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```

BRUKER

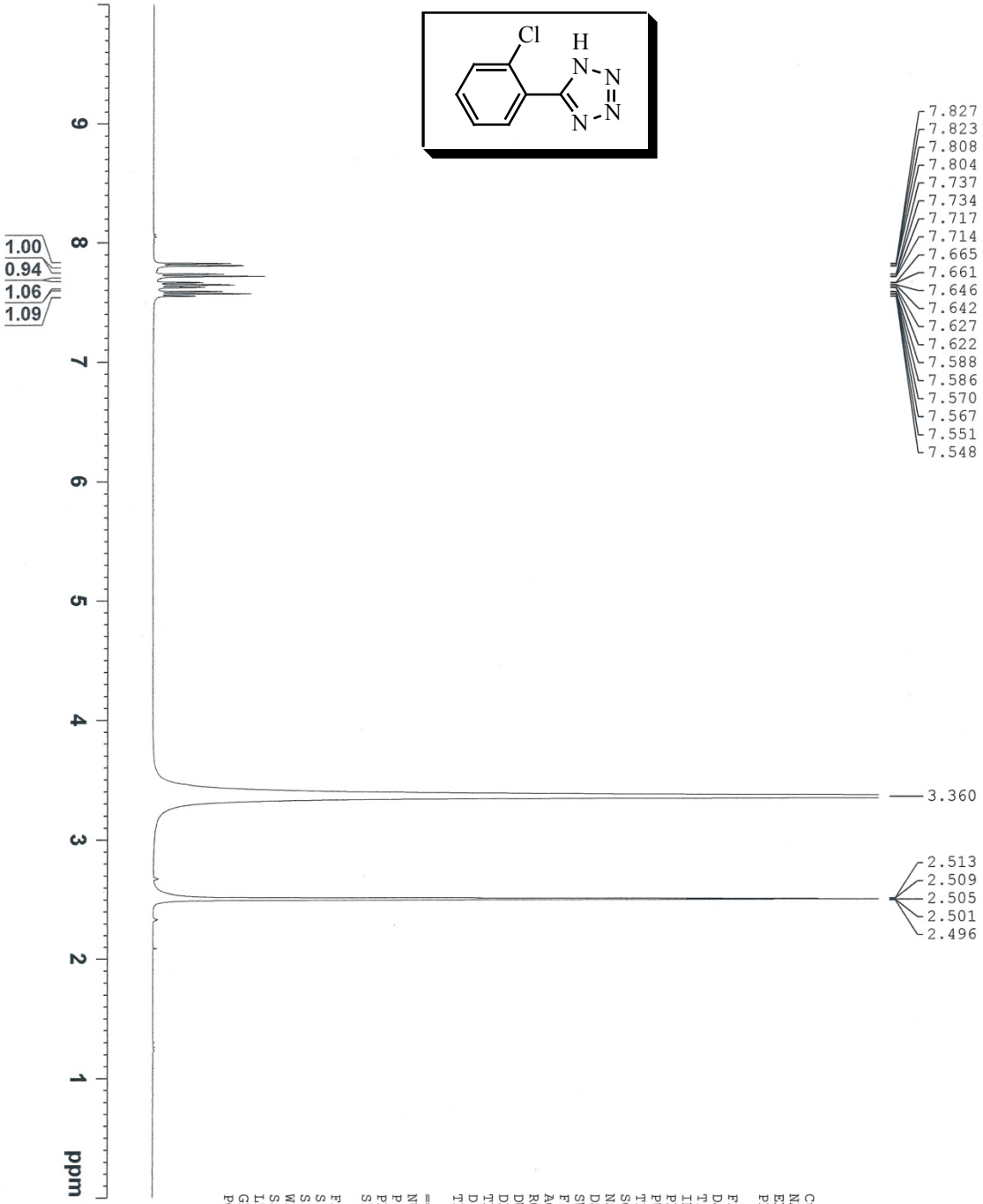
```
Current Data Parameters
NAME                  Dr. A MAJEE
EXPNO                        724
PROCNO                         1

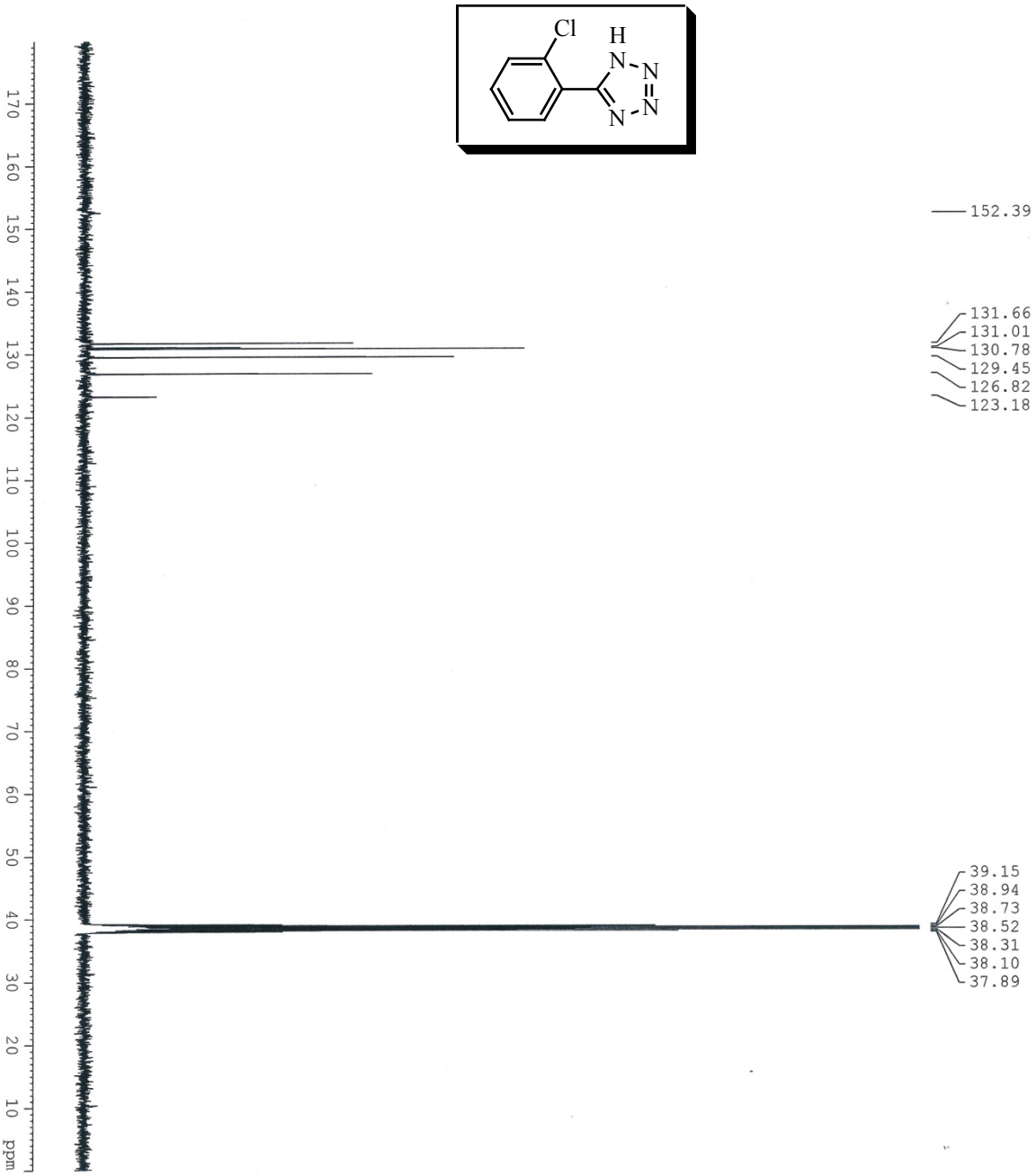
F2 - Acquisition Parameters
Date_                   20130521
Time                       18.48
INSTRUM                     spect
PROBHD     5 mm PABBO BB/
PULPROG                 zgpg30
TD                           32768
SOLVENT                   DMSO
NS                            400
DS                              2
SWH                     24038.461 Hz
FIDRES                      0.733596 Hz
AQ                          0.6816244 sec
RG                             87.66
DW                          20.800 usec
DE                           6.50 usec
TE                          297.5 K
D1                      2.00000000 sec
D11                     0.03000000 sec
TD0                             1

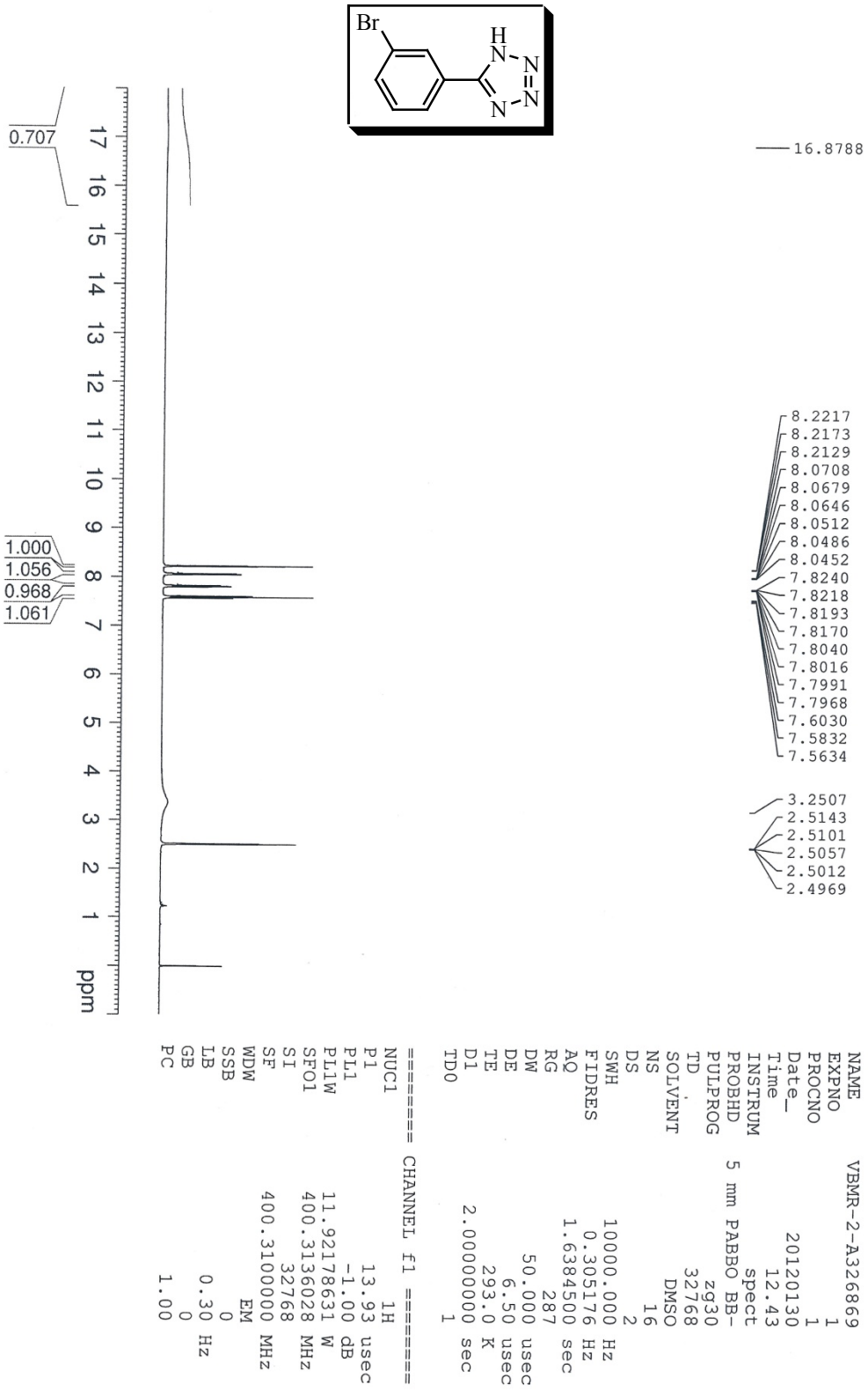
======== CHANNEL f1 ========
NUC1                          13C
P1                           8.90 usec
PLW1                54.00000000 W
SFO1                100.6278588 MHz

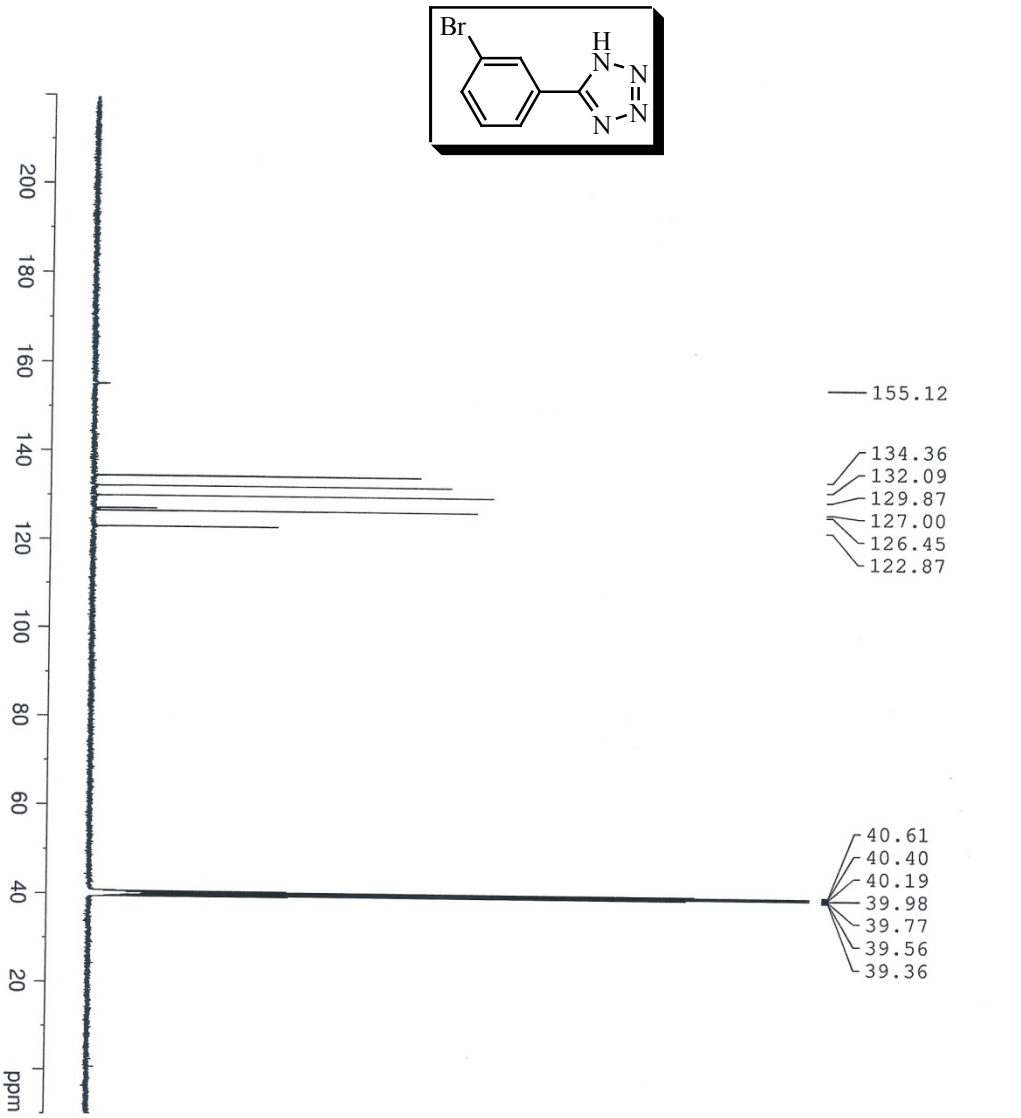
======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                       80.00 usec
PLW2                12.00000000 W
PLW12                0.40792999 W
PLW13                0.26107001 W
SFO2                400.1516006 MHz

F2 - Processing parameters
SI                          16384
SF                  100.6178353 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```





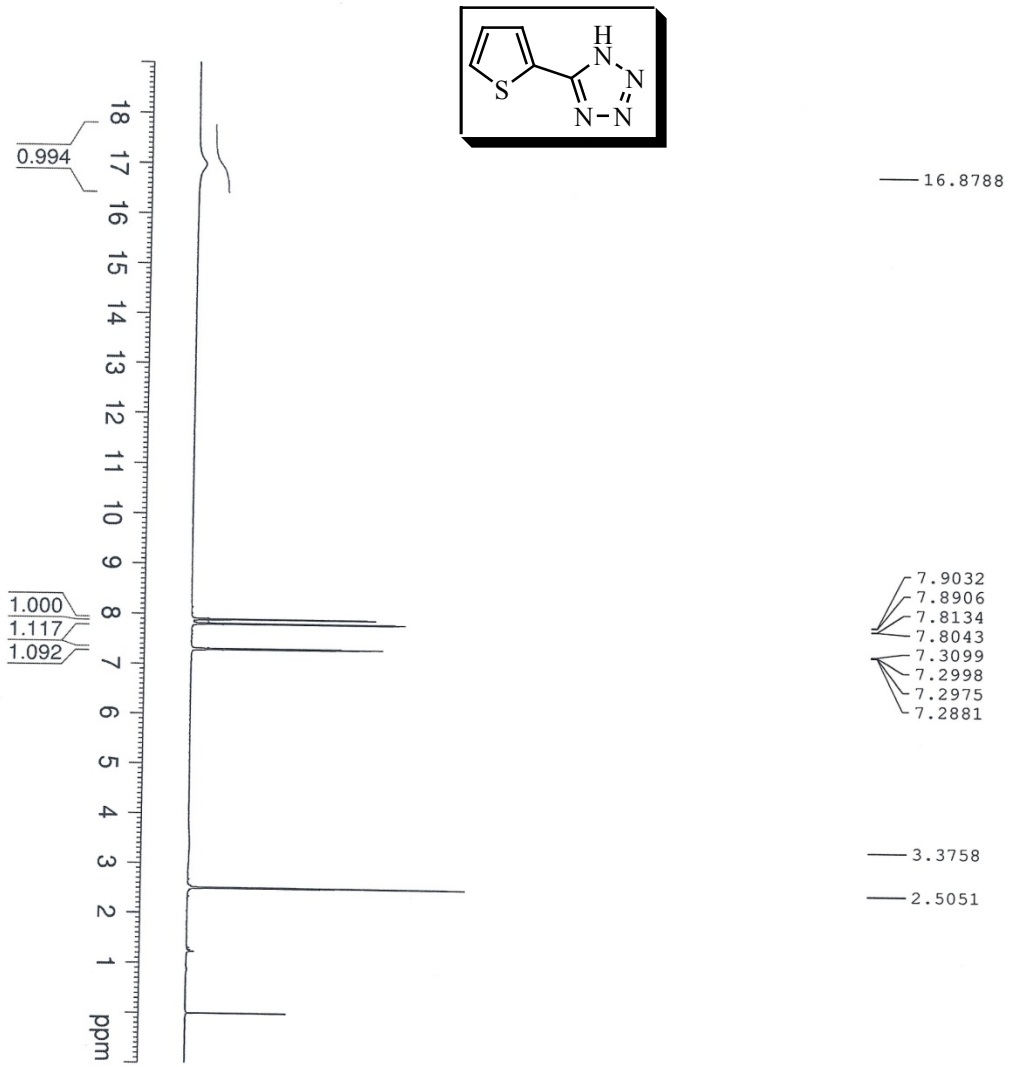





```
NAME                 VBMR-2-A326869
EXPNO                             2
PROCNO                            1
Date_                      20120131
Time                           1.54
INSTRUM                       spect
PROBHD     5 mm PABBO BB-
PULPROG                    zgpg30
TD                          32768
SOLVENT                      DMSO
NS                           2048
DS                              4
SWH                     24038.461 Hz
FIDRES                   0.733596 Hz
AQ                       0.6816244 sec
RG                            2050
DW                       20.800 usec
DE                        6.50 usec
TE                          293.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                             1

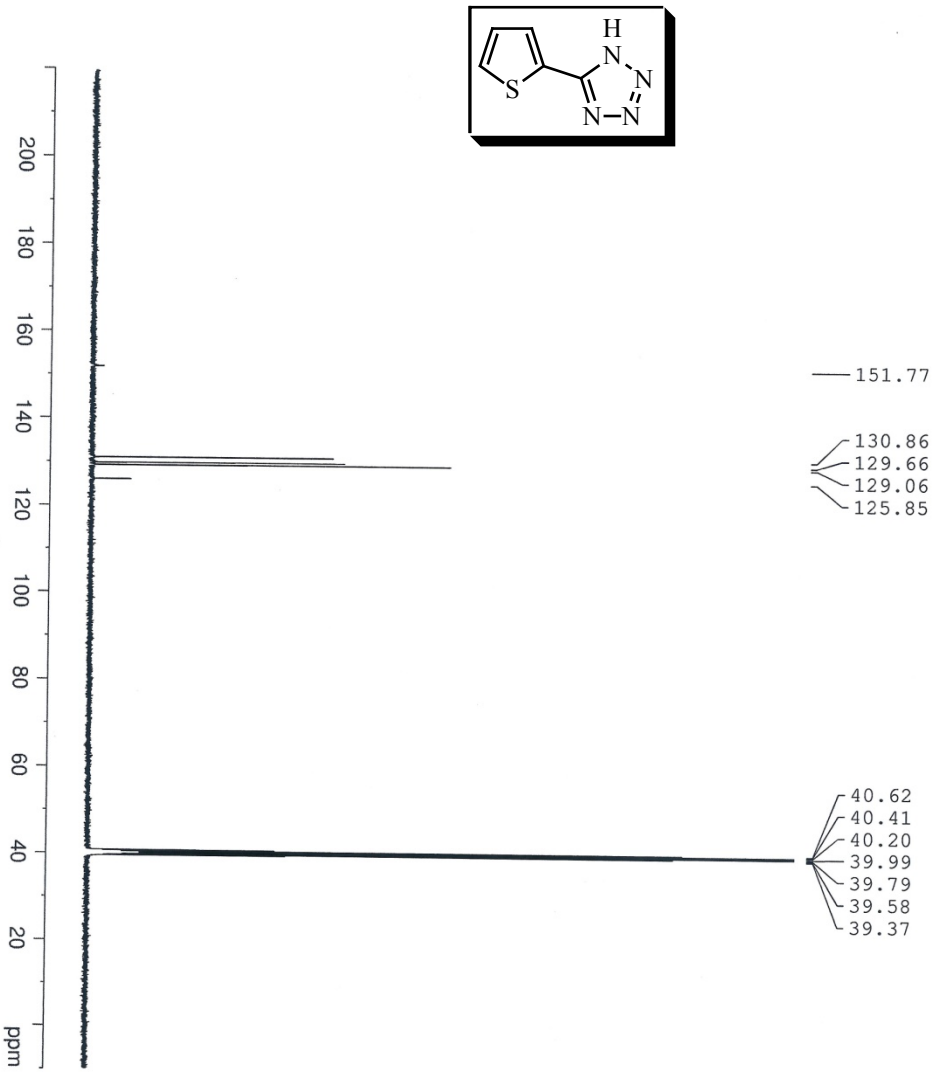
======== CHANNEL f1 ========
NUC1                          13C
P1                           7.75 usec
PL1                        -2.00 dB
PL1W                57.32743073 W
SFO1               100.6680954 MHz

======== CHANNEL f2 ========
CPDPRG2                   waltz16
NUC2                          1H
PCPD2                    80.00 usec
PL2                       -1.00 dB
PL12                      14.18 dB
PL13                      15.00 dB
PL2W                11.92178631 W
PL12W                0.36169401 W
PL13W                0.29946175 W
SFO2               400.3116012 MHz
SI                          32768
SF                 100.6580300 MHz
WDW                            EM
SSB                             0
LB                       1.00 Hz
GB                              0
PC                           1.40
```




```
NAME                 VBMR-1-A326868
EXPNO                             1
PROCNO                            1
Date_                      20120130
Time                       12.48
INSTRUM                       spect
PROBHD     5 mm PABBO BB-
PULPROG                        zg30
TD                            32768
SOLVENT                        DMSO
NS                               16
DS                                2
SWH                       10000.000 Hz
FIDRES                        0.305176 Hz
AQ                        1.6384500 sec
RG                              287
DW                           50.000 usec
DE                            6.50 usec
TE                          293.0 K
D1                        2.00000000 sec
TD0                               1

========== CHANNEL f1 ========
NUC1                           1H
P1                          13.93 usec
PL1                          -1.00 dB
PL1W                    11.92178631 W
SFO1                    400.3136028 MHz
SI                            32768
SF                      400.3100000 MHz
WDW                              EM
SSB                               0
LB                             0.30 Hz
GB                                0
PC                             1.00
```




```
NAME                 VBMR-1-A326868
EXPNO                             2
PROCNO                            1
Date_                      20120131
Time_                          0.17
INSTRUM                       spect
PROBHD     5 mm PABBO BB-
PULPROG                     zgpg30
TD                           32768
SOLVENT                      DMSO
NS                           2048
DS                              4
SWH                    24038.461 Hz
FIDRES                  0.733596 Hz
AQ                      0.6816244 sec
RG                           2050
DW                         20.800 usec
DE                          6.50 usec
TE                           293.0 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                               1

======== CHANNEL f1 ========
NUC1                           13C
P1                            7.75 usec
PL1                          -2.00 dB
PL1W                   57.32743073 W
SFO1                   100.6680954 MHz

======== CHANNEL f2 ========
CPDPRG2                    waltz16
NUC2                           1H
PCPD2                      80.00 usec
PL2                          -1.00 dB
PL12                         14.18 dB
PL13                         15.00 dB
PL2W                   11.92178631 W
PL12W                   0.36169401 W
PL13W                   0.29946175 W
SFO2                   400.3116012 MHz
SI                           32768
SF                     100.6580300 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```