

N-Alkylated 1,4-Diazabicyclo[2.2.2]octane–Polyethylene Glycol Melt as Deep Eutectic Solvent for the Synthesis of Fisher Indoles and 1*H*-Tetrazoles

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Thermal Study of DABCO-ILs (2, 4)

Figure-S1a shows overlap of thermogravimetric (TGA) graph and its first derivative (DTG) for compound **2**, and Figure-S1b shows overlap of TGA and DSC graphs. The compound remains stable on heating up to 200 °C. In the first step there is only a slight loss in weight of about 16% in the short temperature range of 200-220 °C. As can be seen from the DSC graph, this is an endothermic process and is probably due to the loss of water molecules. In the second step, also an endothermic process, there is a weight loss of 24% in the temperature range 220-260 °C, most likely due to loss of alkyl chain. From the DTG, the loss of water molecules is observed at 215 °C, and the first decomposition temperature is calculated to be 235 °C. The remaining weight loss (about 40%) occurs in the third and last gradual step in the temperature range 280-360 °C, the second decomposition temperature calculated from DTG is 315 °C.

Figure-S1c shows overlap of thermogravimetric (TGA) graph and its first derivative (DTG) for compound **4**, and Figure-S1d shows the overlap of TGA and DSC graphs. In the first step there is a weight loss of about 30% in the temperature range from ambient to 100 °C, the corresponding DSC data in this range suggests an endothermic process associated with this change. This weight loss is most likely due to loss of water molecules. After initial loss of water, the weight remains stable at heating up to 215 °C, after which a sharp decrease in weight (28%) is observed, this is the first decomposition temperature at 255 °C as indicated by the DTG. According to DSC, this weight loss is also an endothermic process and is probably due to loss of alkyl chains. Immediately after this step, a sharp weight loss of about 40% is observed. This corresponds to a second decomposition temperature of 300 °C as indicated by the DTG. Similar to previous step, this was also an endothermic process as can be seen from the DSC graph.

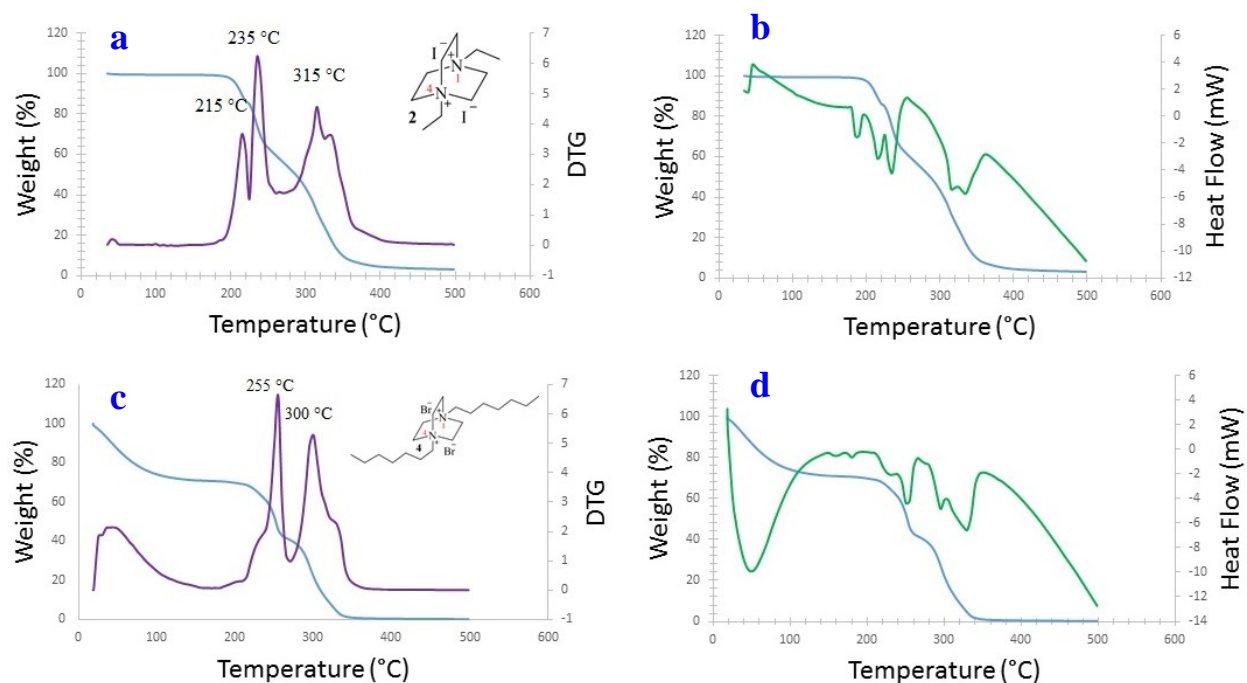


Figure-S1: a) Overlap of TGA (blue) and DTG (purple) graphs for compound **2**; b) Overlap of TGA (blue) and DSC (green) graphs for compound **2**; c) Overlap of TGA (blue) and DTG (purple) graphs for compound **4**; d) Overlap of TGA (blue) and DSC (green) graphs for compound **4**.

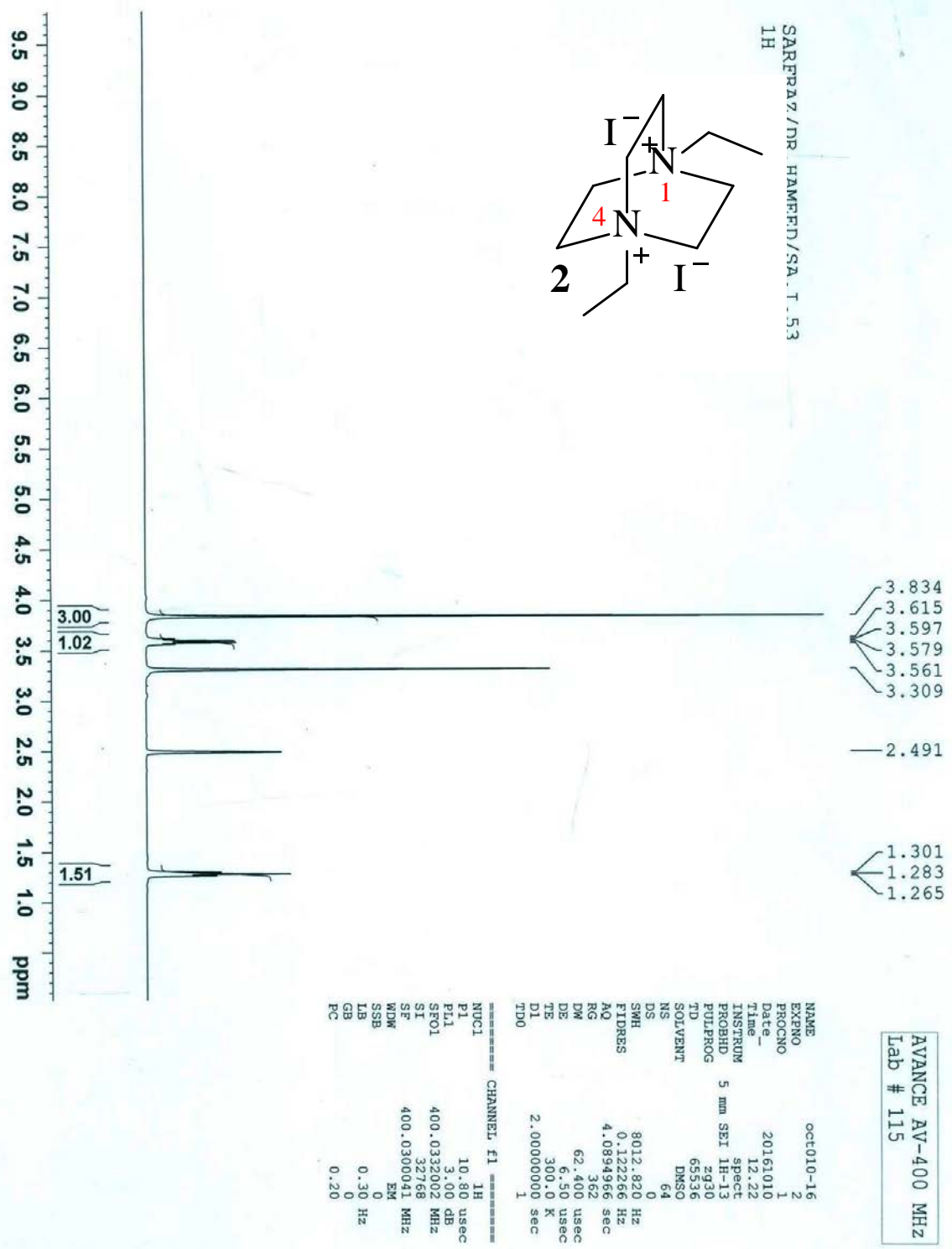


Figure-S2

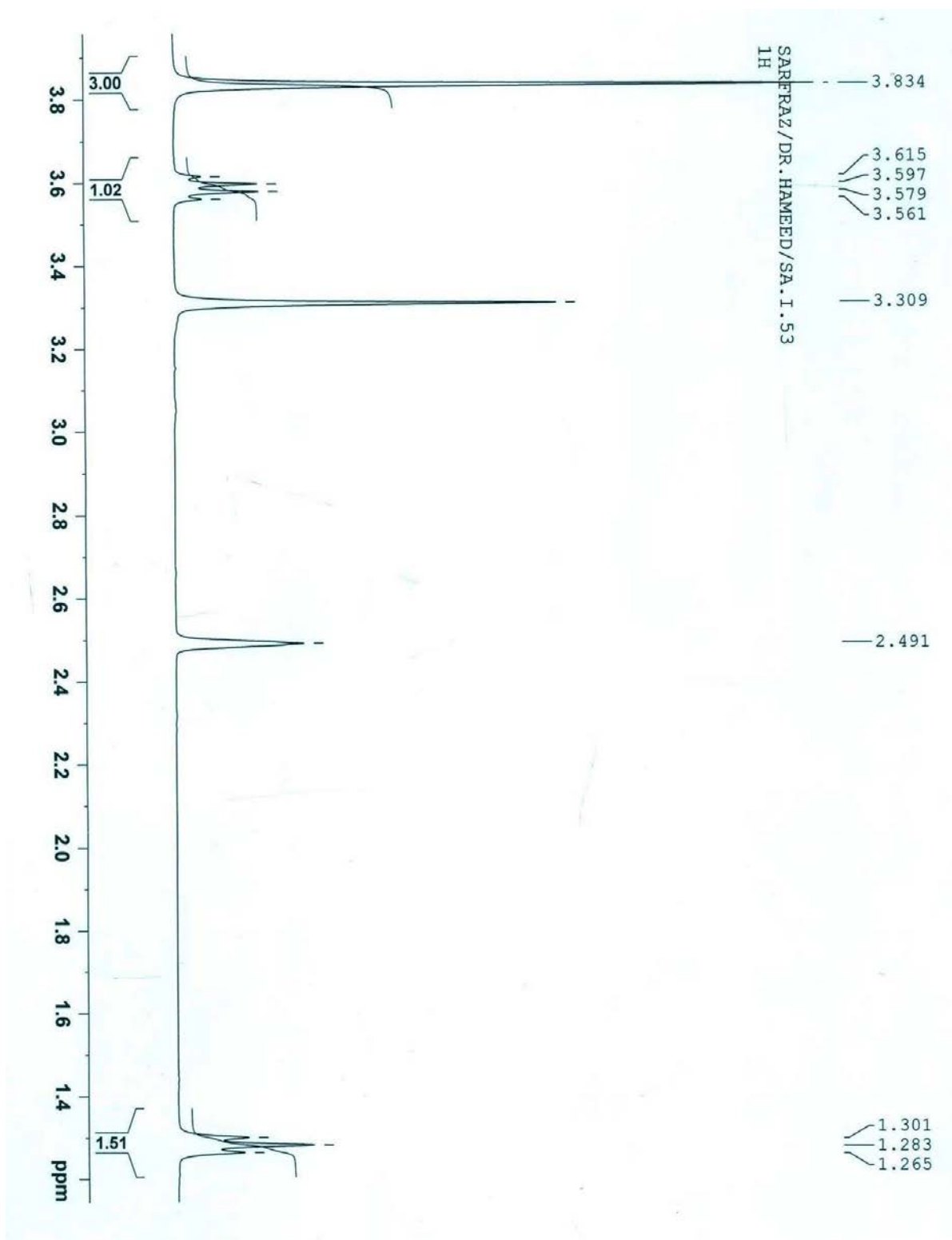


Figure-S2a

Sarfraz / Dr. A.Hameed / SA-I-53 / DMSO
BB

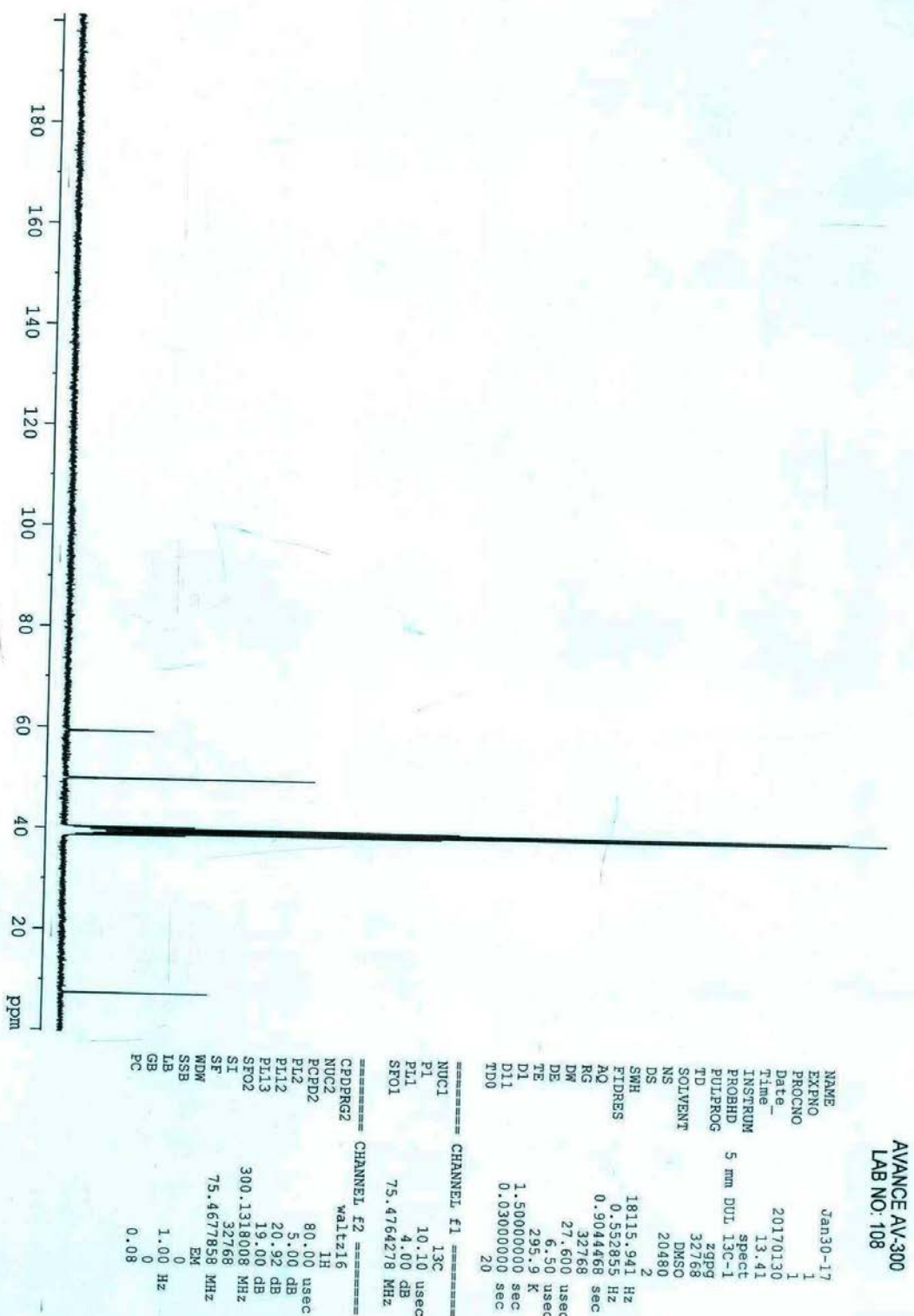


Figure-S3

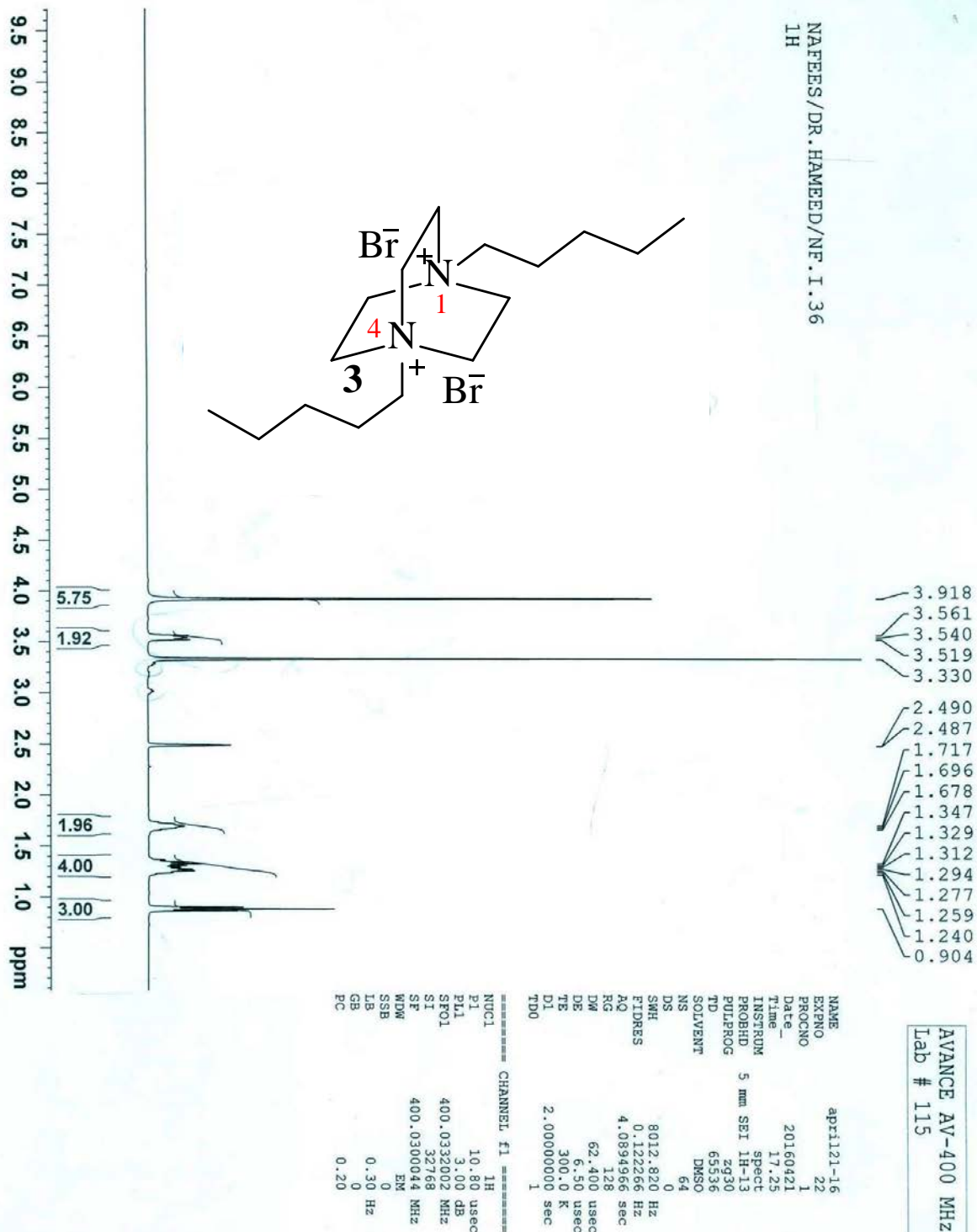


Figure-S4

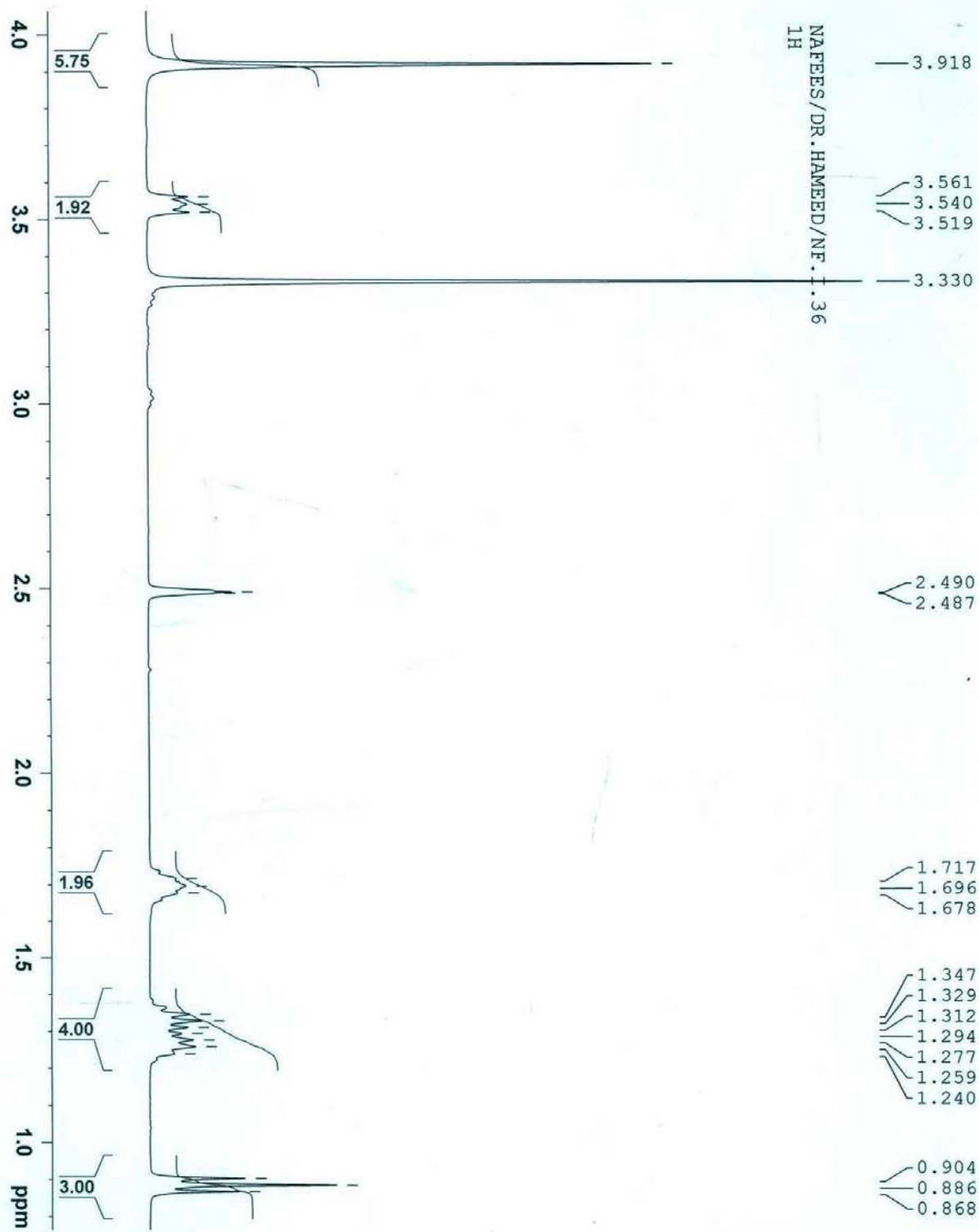


Figure-S4a

SARFARAZ/DR, ABDUL HAMEED/SA-I-32b/
ICCBS, U.O.K/BB

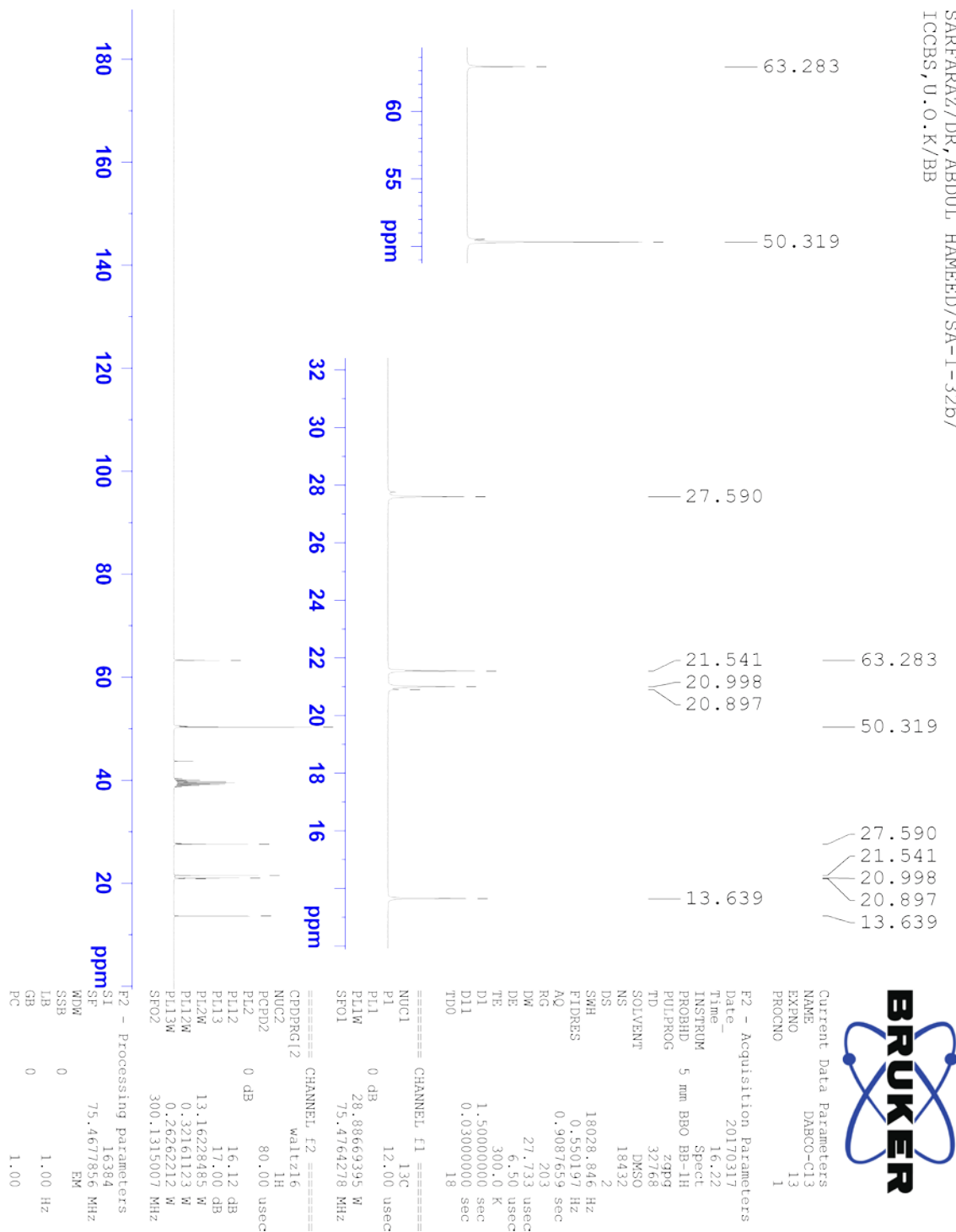


Figure-S5

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ICCBS, U.O.K/1H

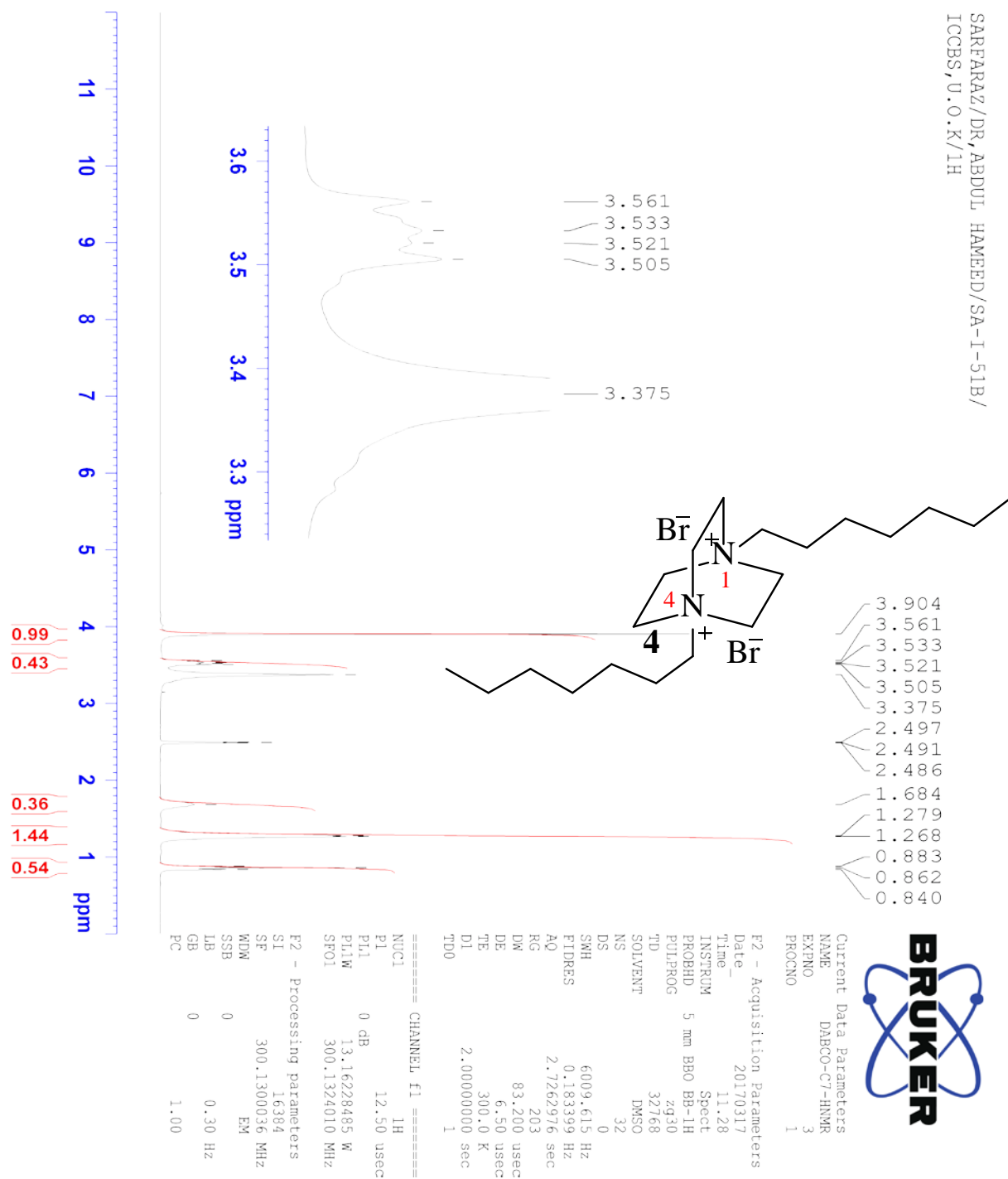


Figure-S6

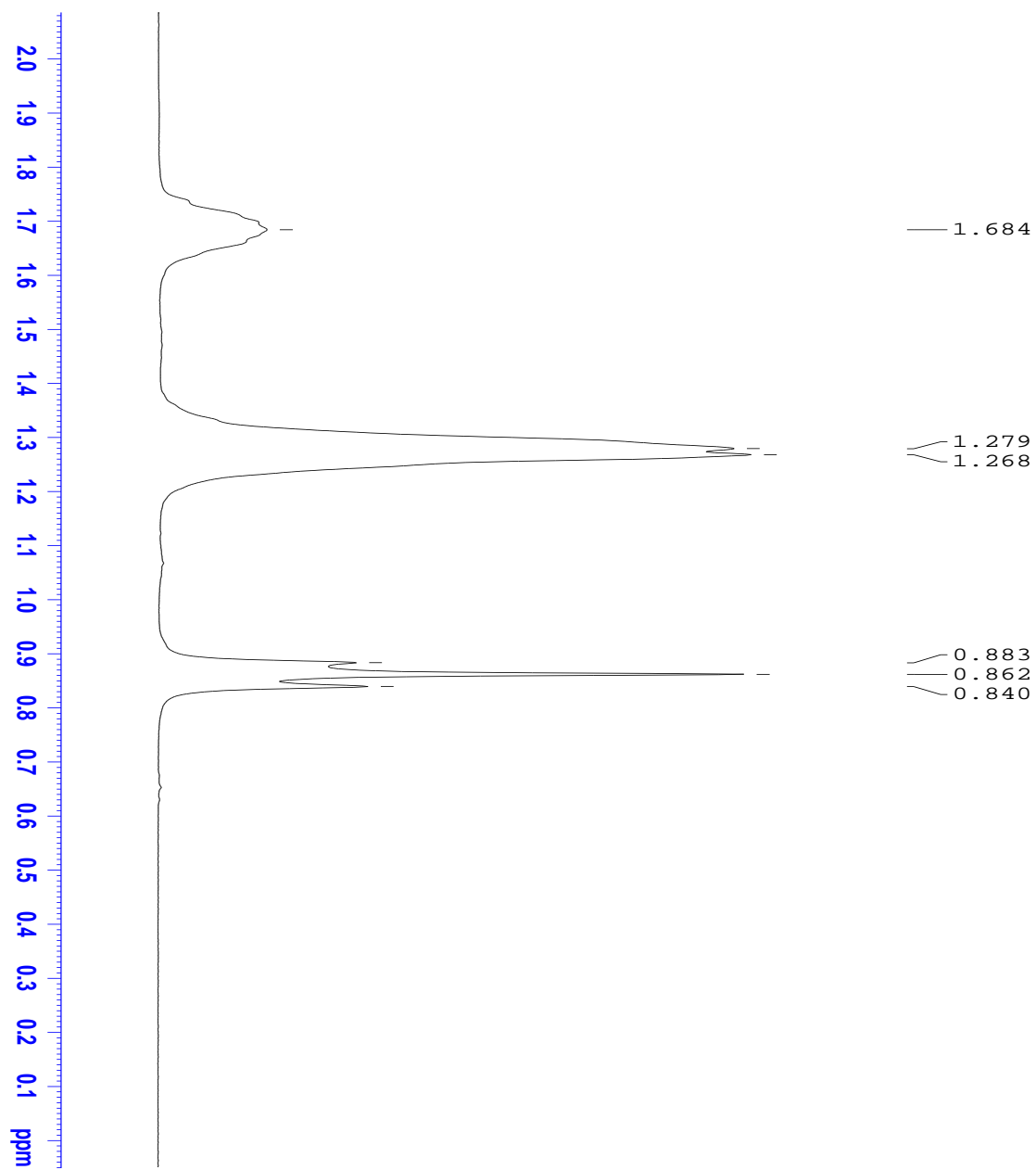


Figure-S6a

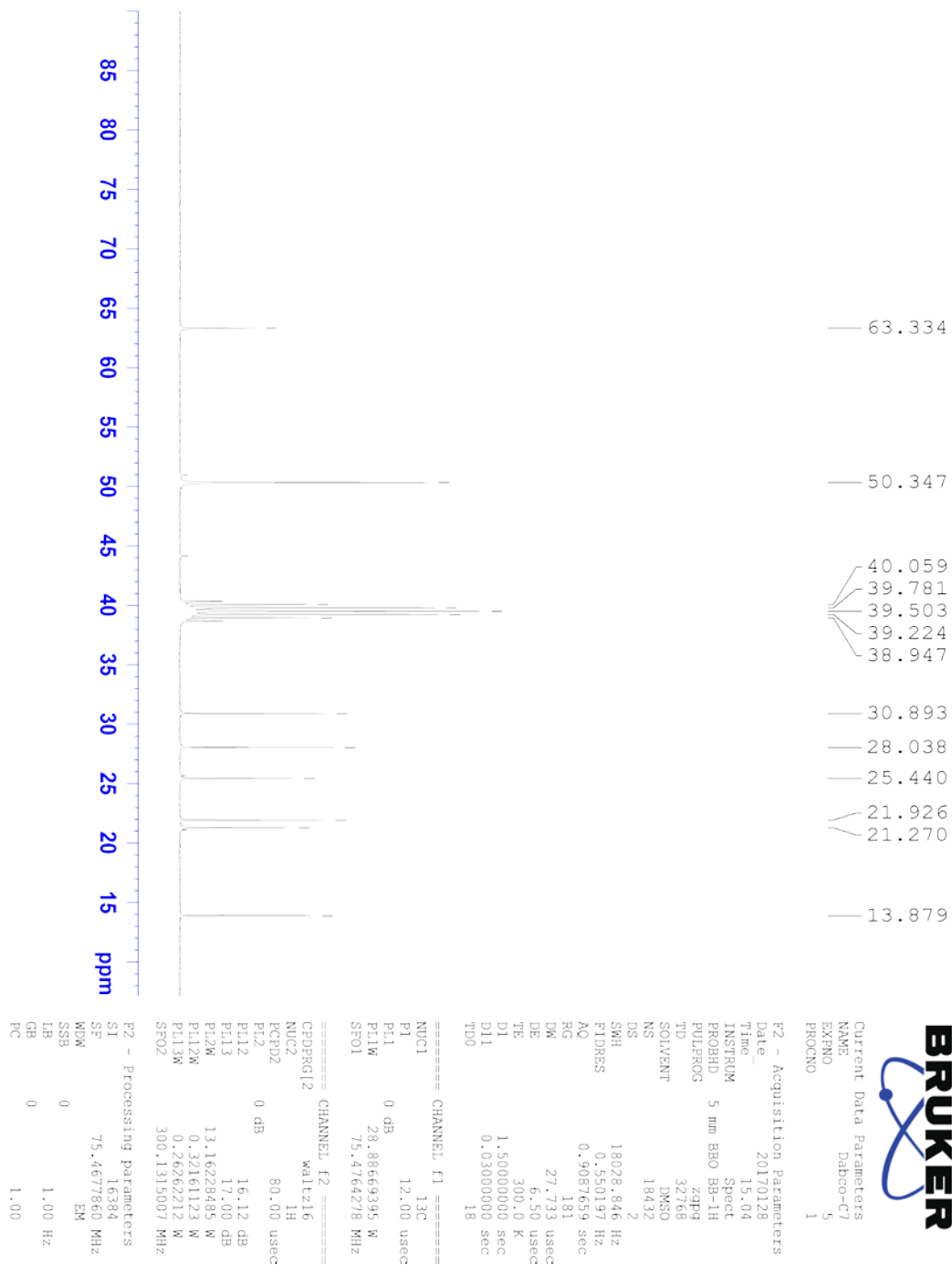


Figure-S7

SARFARAZ/DR.ABDUL HAMEED/SA-I-57/
ICCBS,U.O.K/

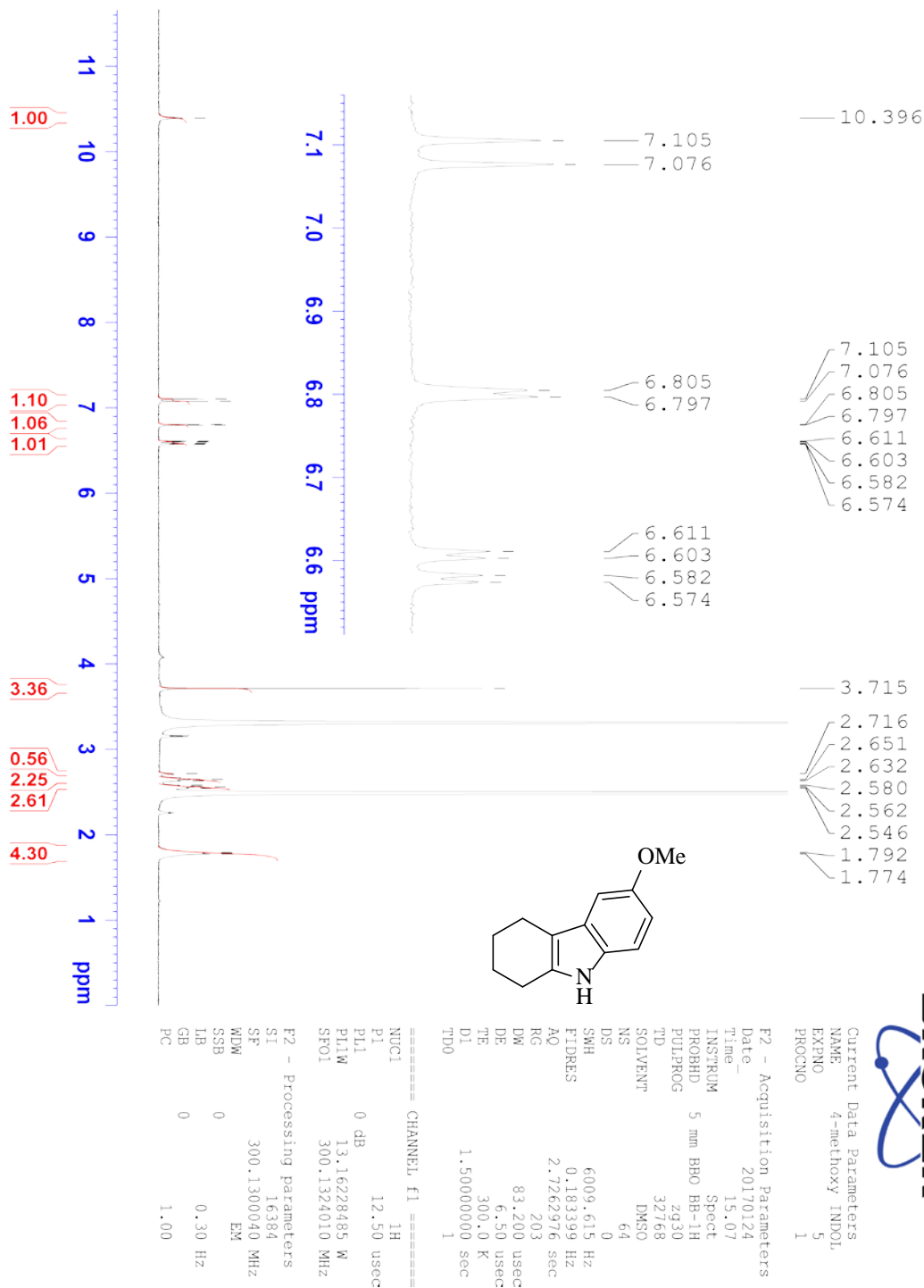


Figure-S8

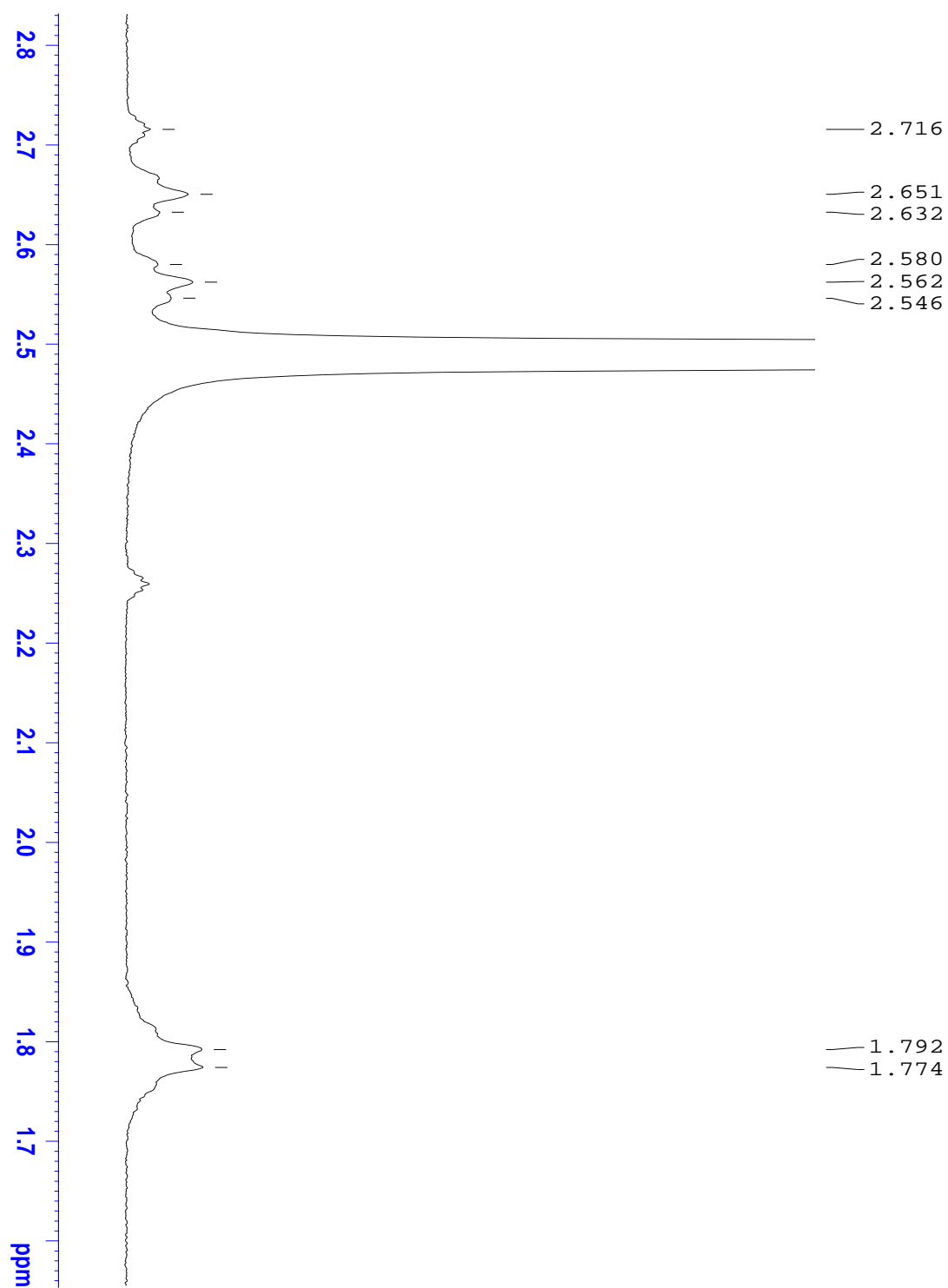


Figure-S8a

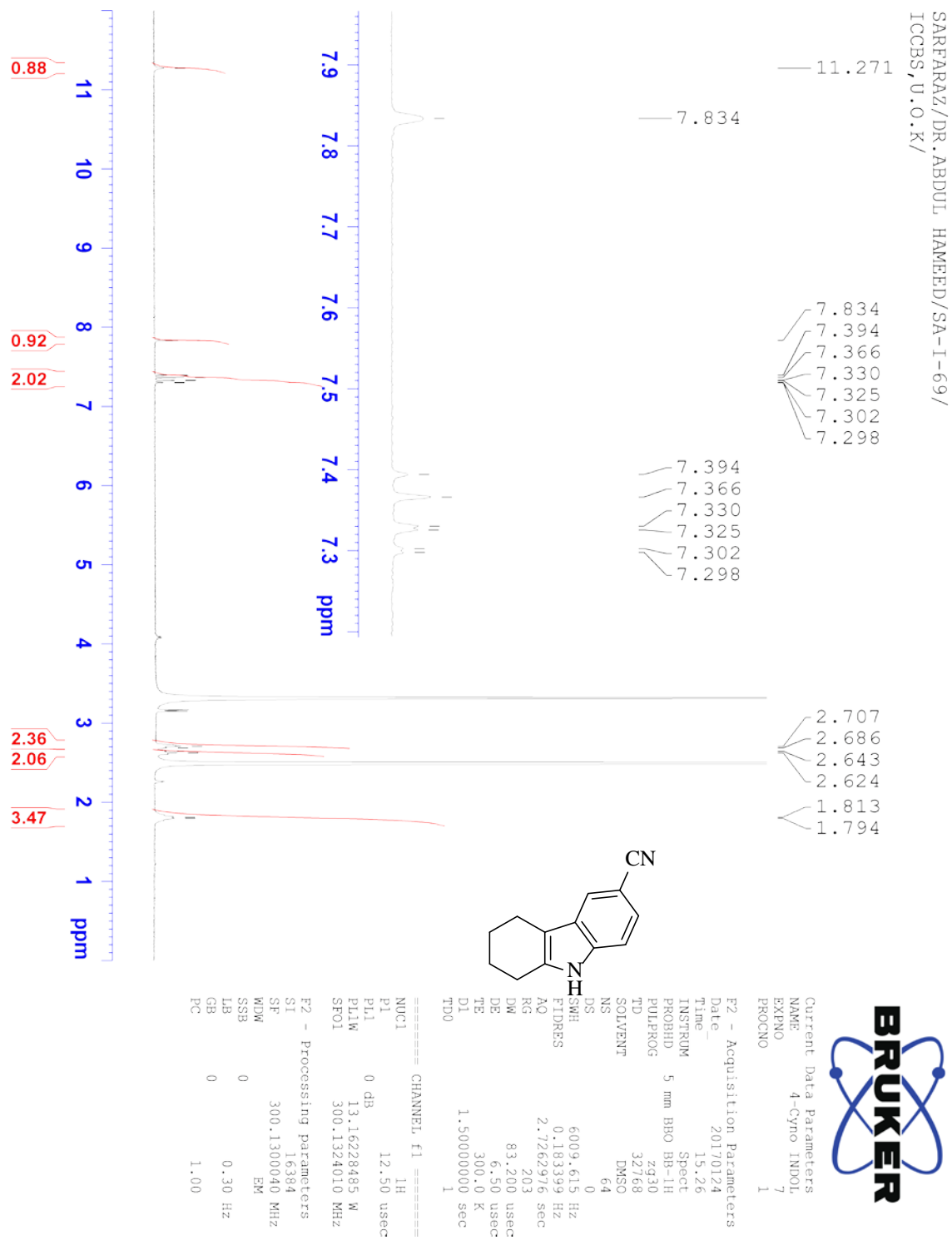


Figure-S9

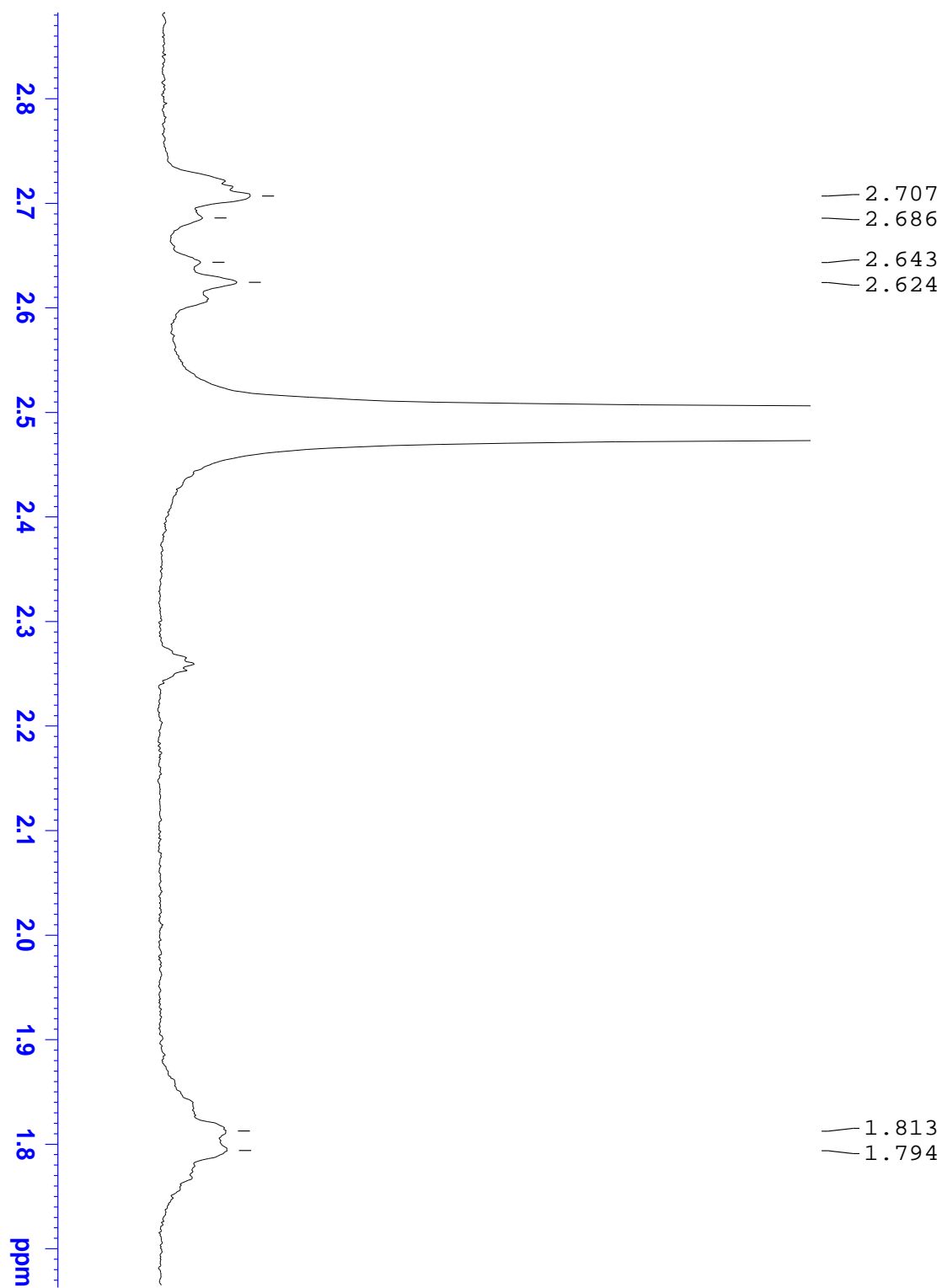


Figure-S9a

SARFARAZ/DR.ABDUL HAMEED/SA-I-66/
ICCBS,U.O.K/

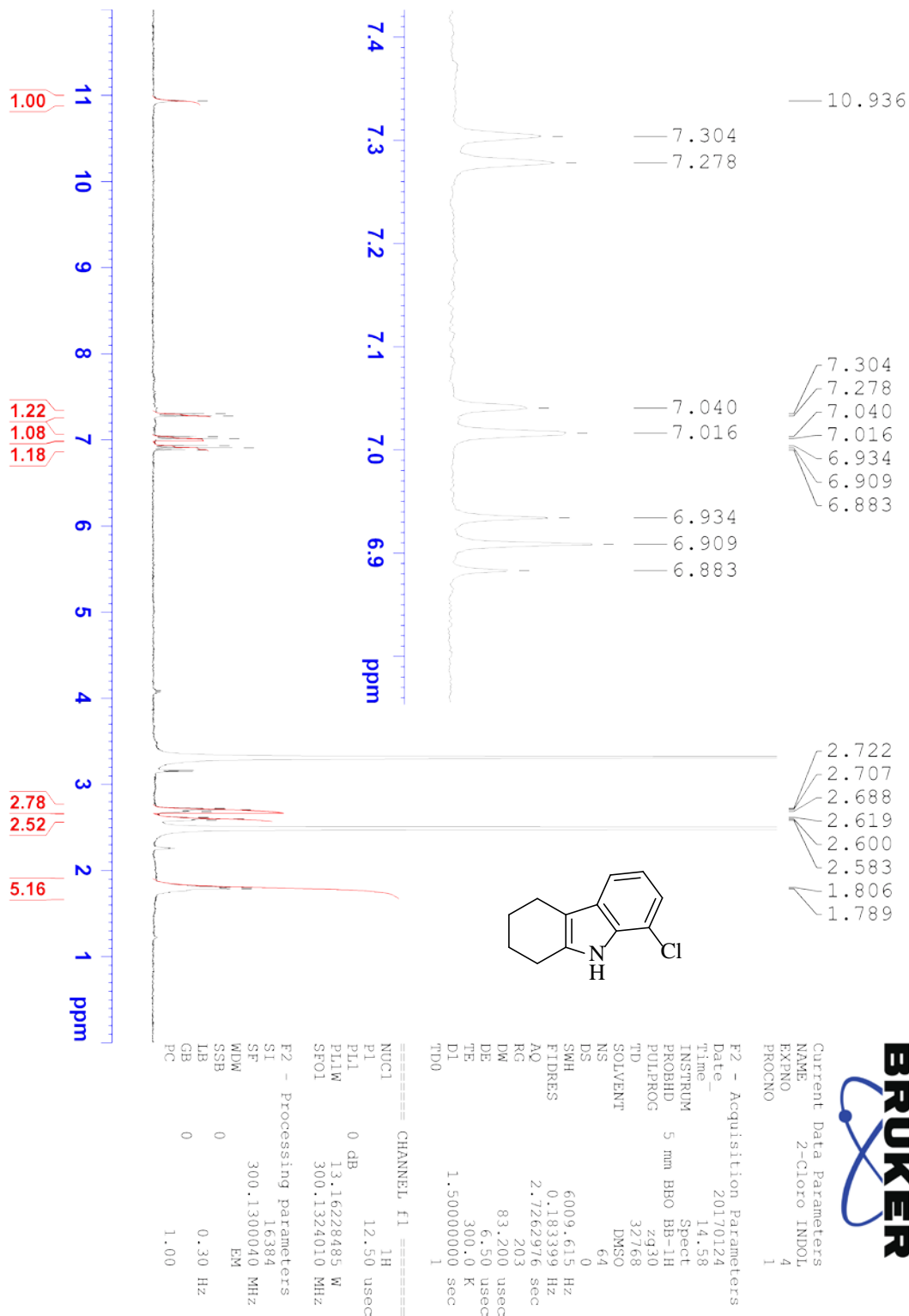


Figure-S10

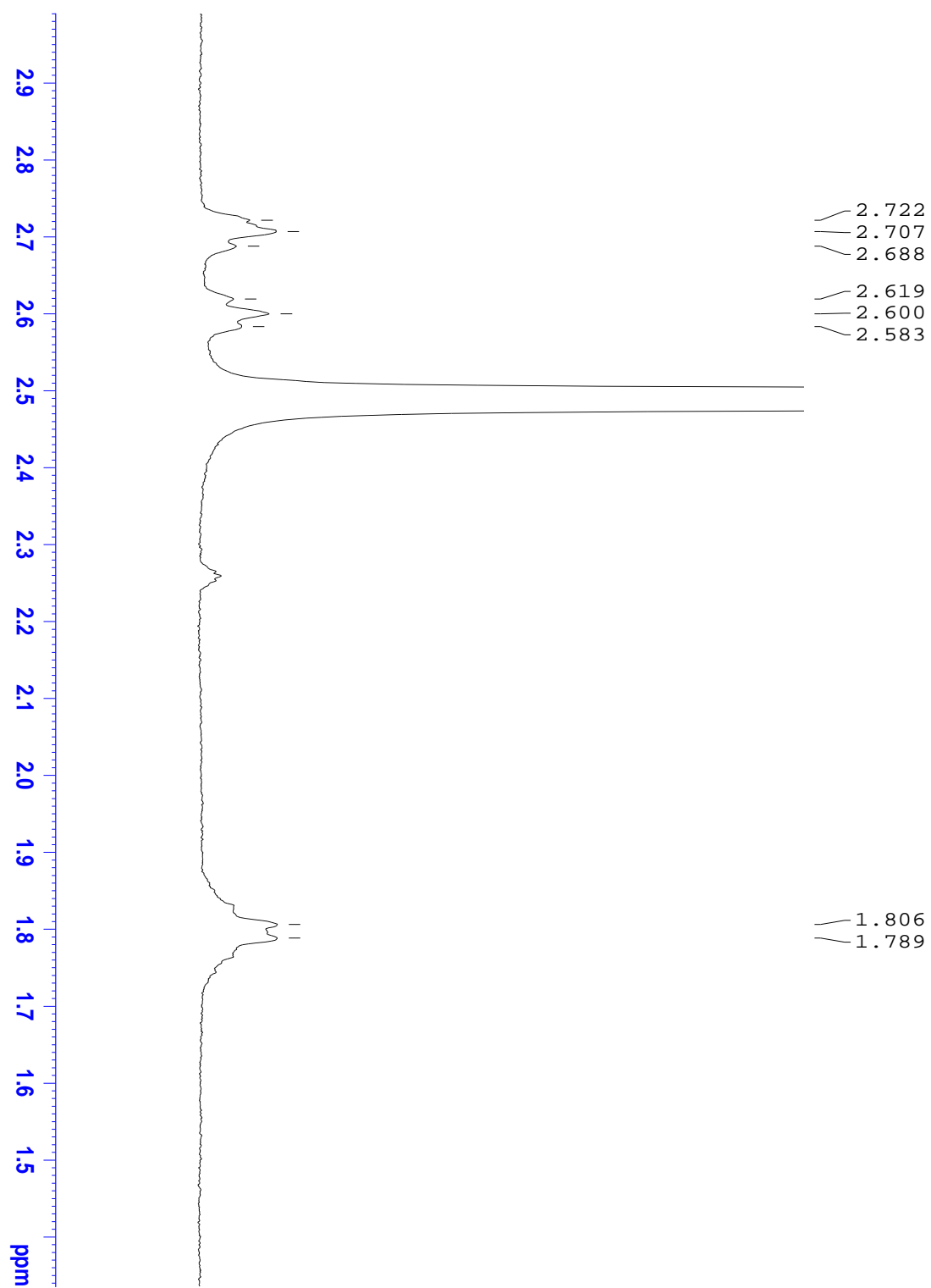


Figure-S10a

SARFARAZ/DR, ABDUL HAMEED/SA-I-85/
ICCBS, U.O.K/

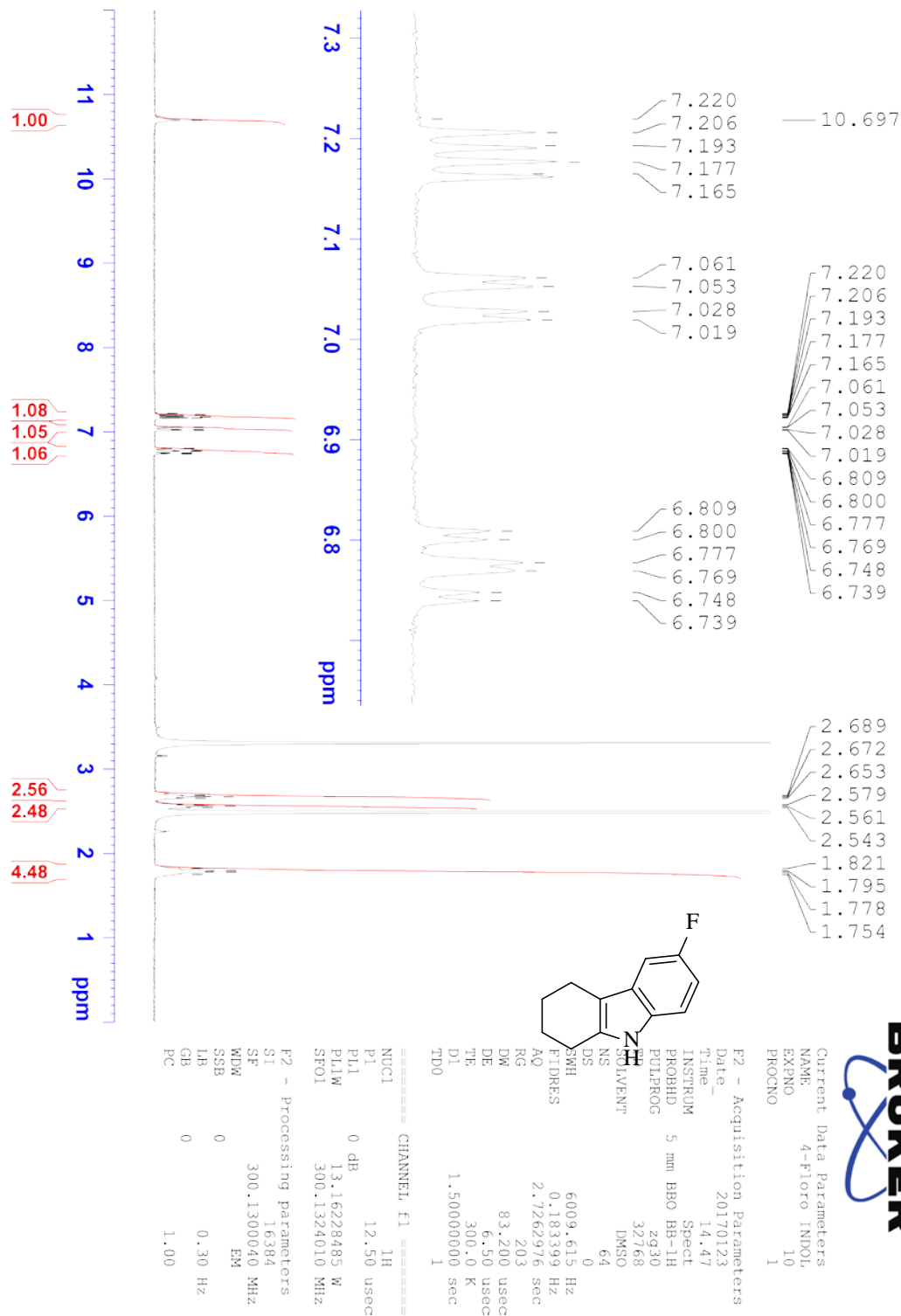


Figure-S11



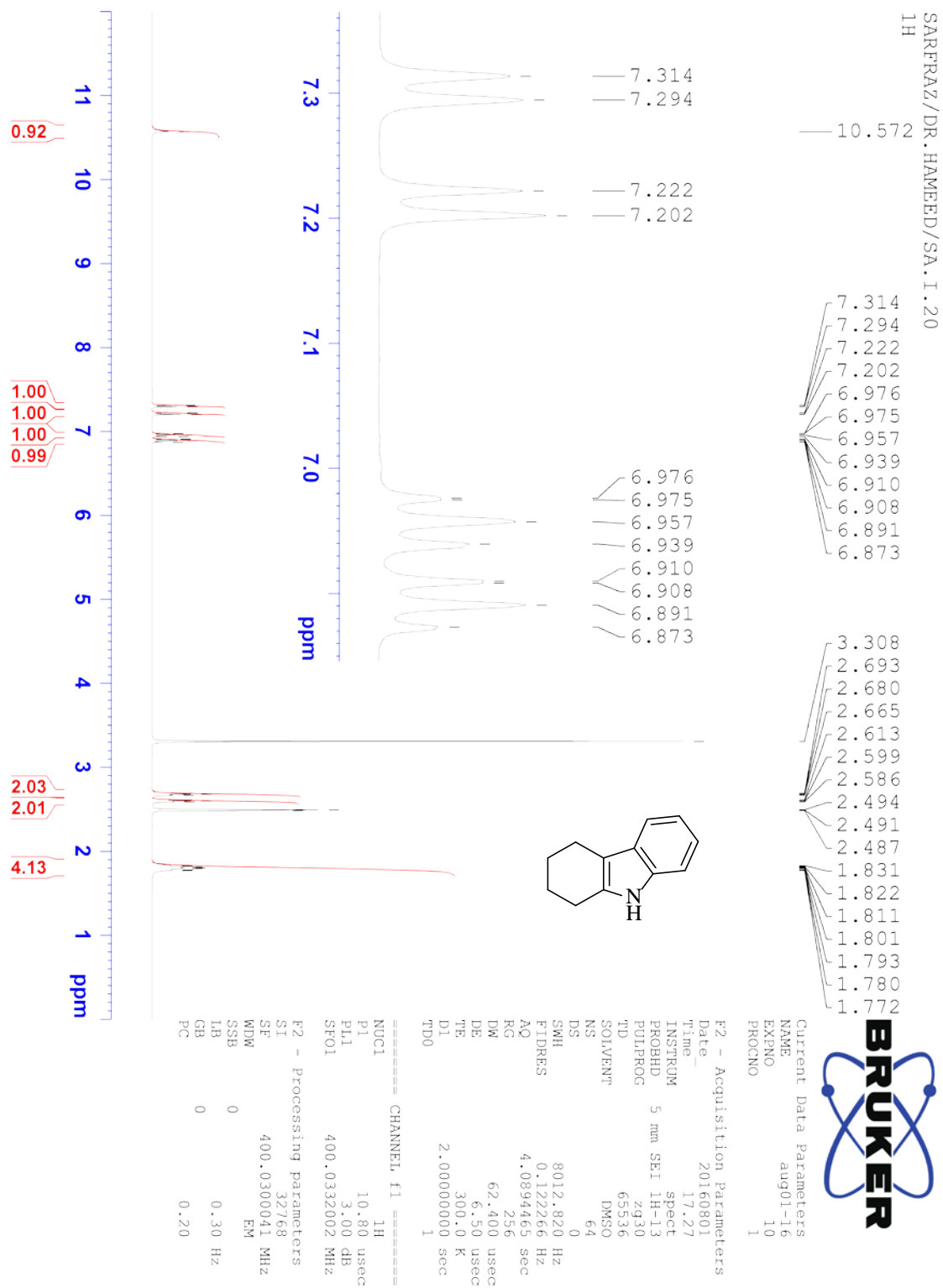


Figure-S12

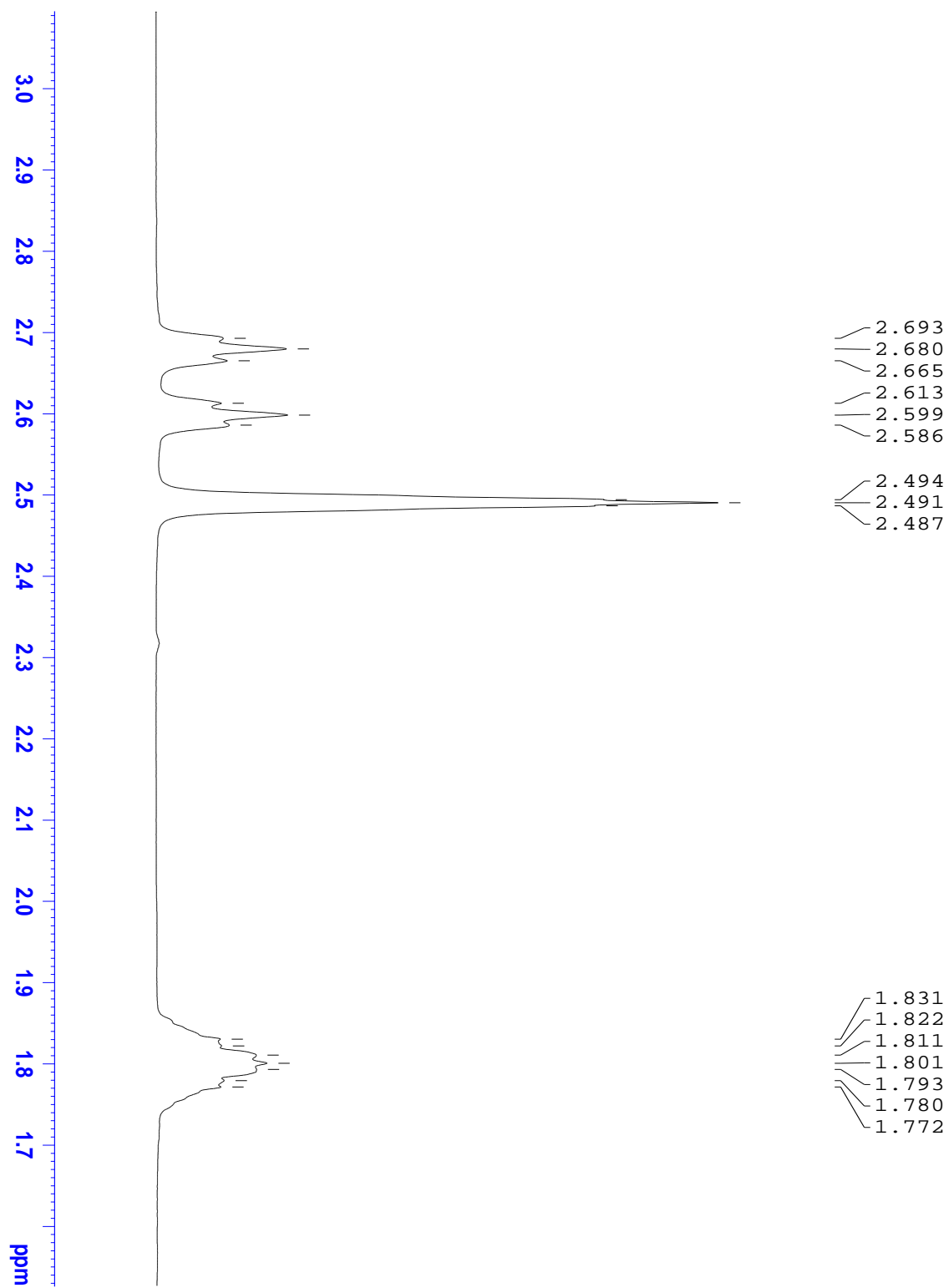


Figure-S12a

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ICCBS, U.O.K/

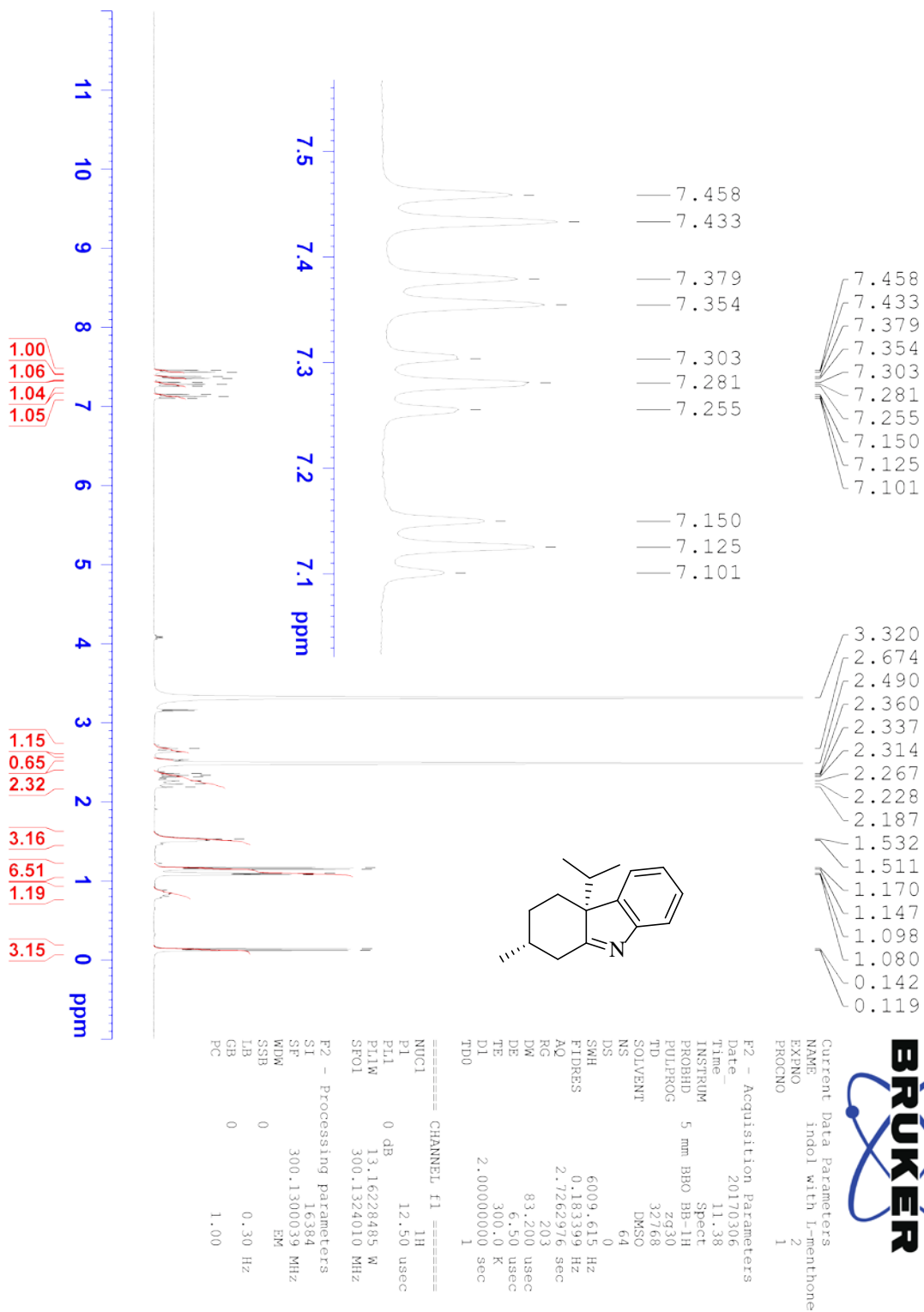


Figure-S13

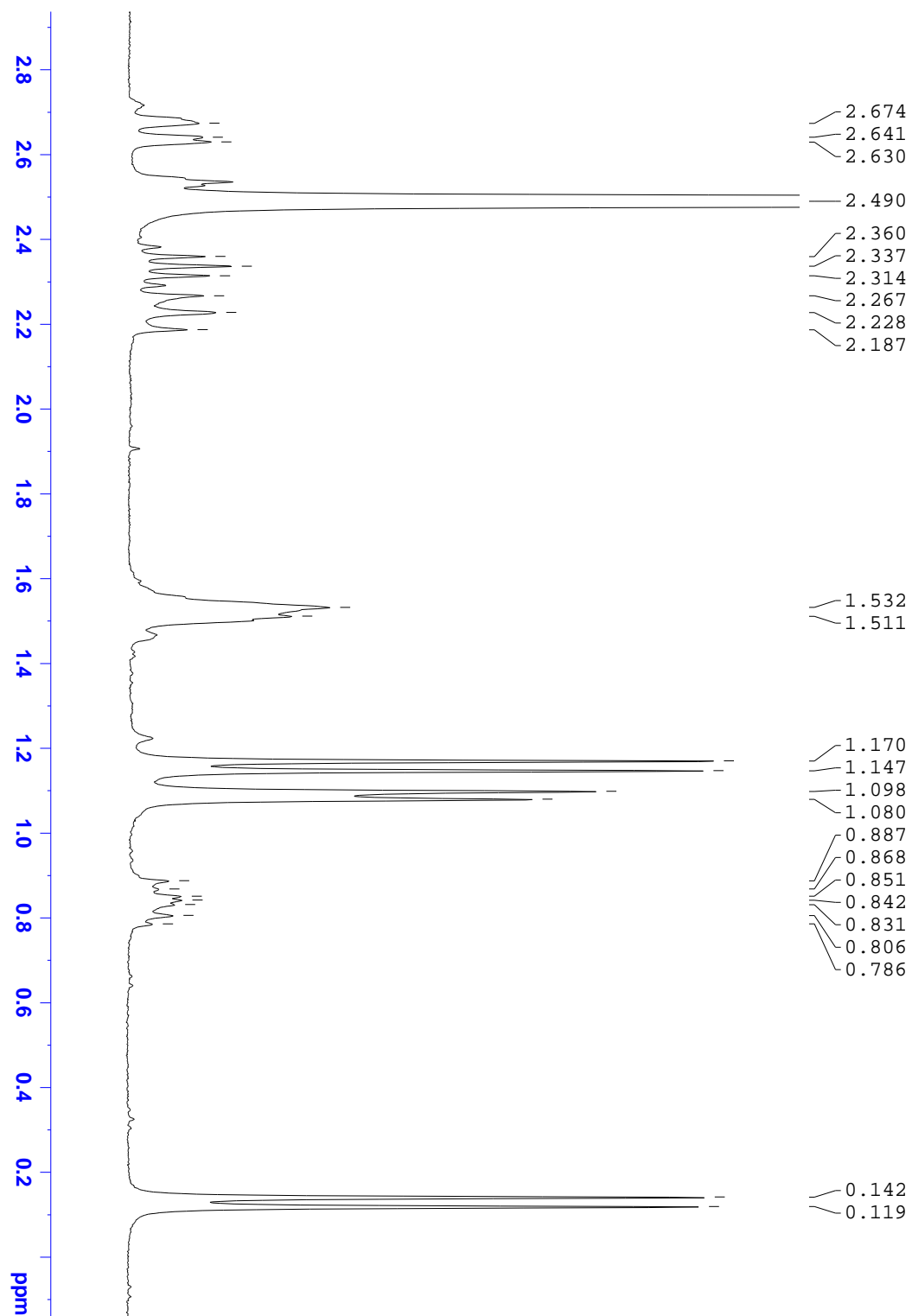
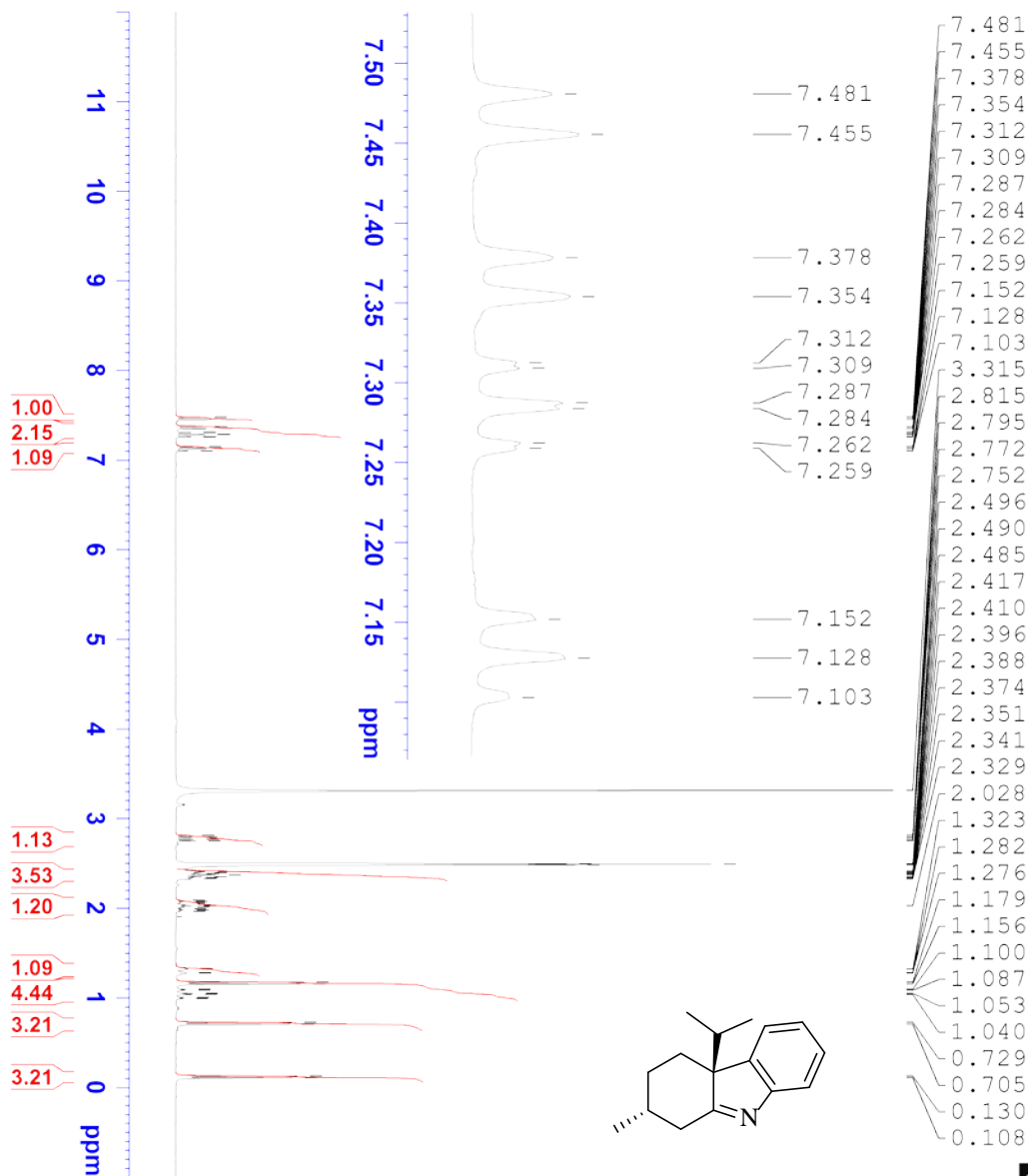


Figure-S13a

SARFARAZ/DR, ABDUL HAMEED/SA-I-97B/
ICCS, U.O.K/



BRUKER

Current Data Parameters
NAME indol with l-menthone
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170306
Time_ 11.49
INSTRUM Spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 64
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7262976 sec
RG 203
DW 83.200 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.50 usec
PL1 0 dB
PL1W 13.16228485 W
SFO1 300.1324010 MHz

F2 - Processing parameters
SI 16384
SF 300.1300039 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure-S14

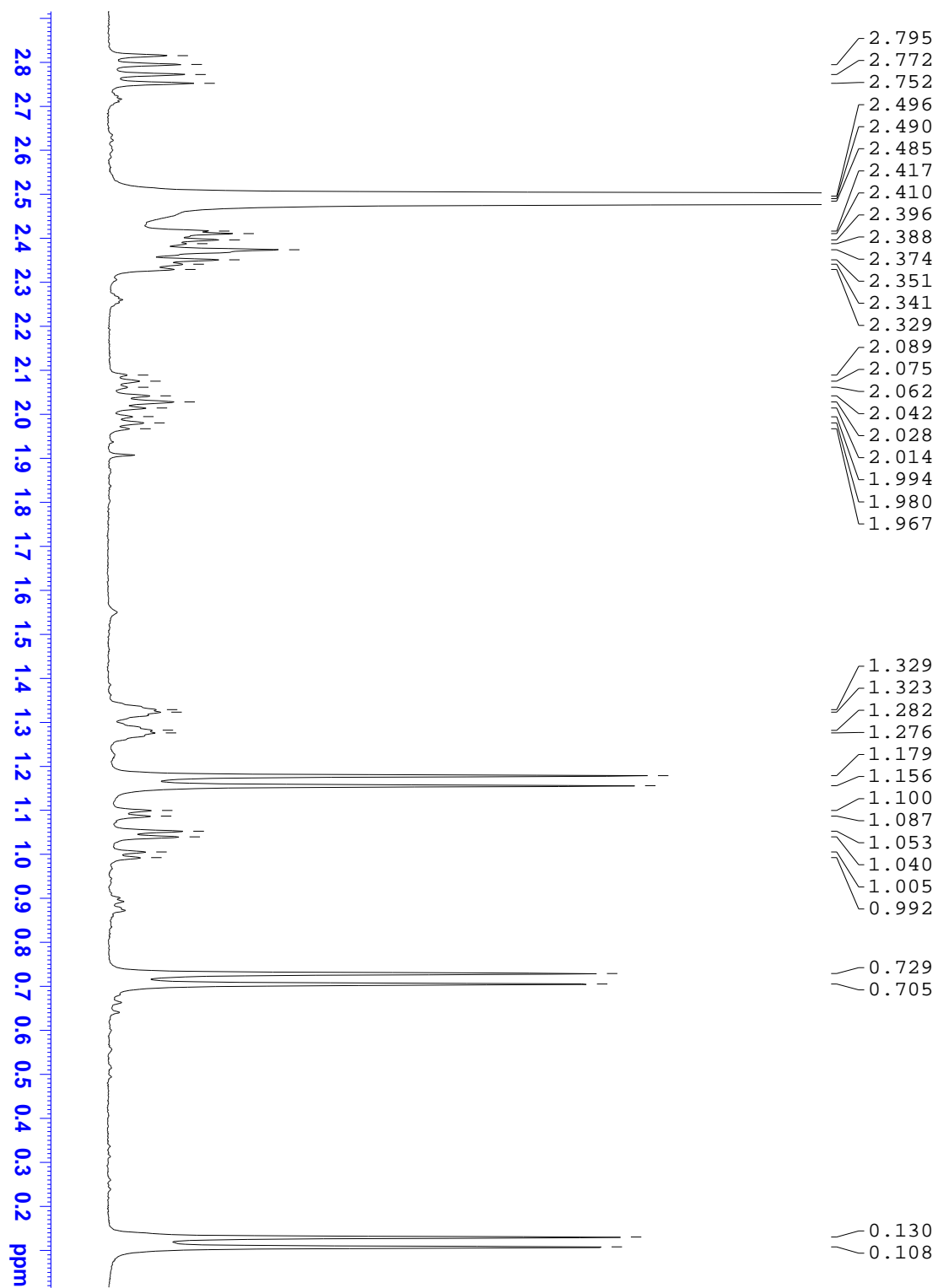
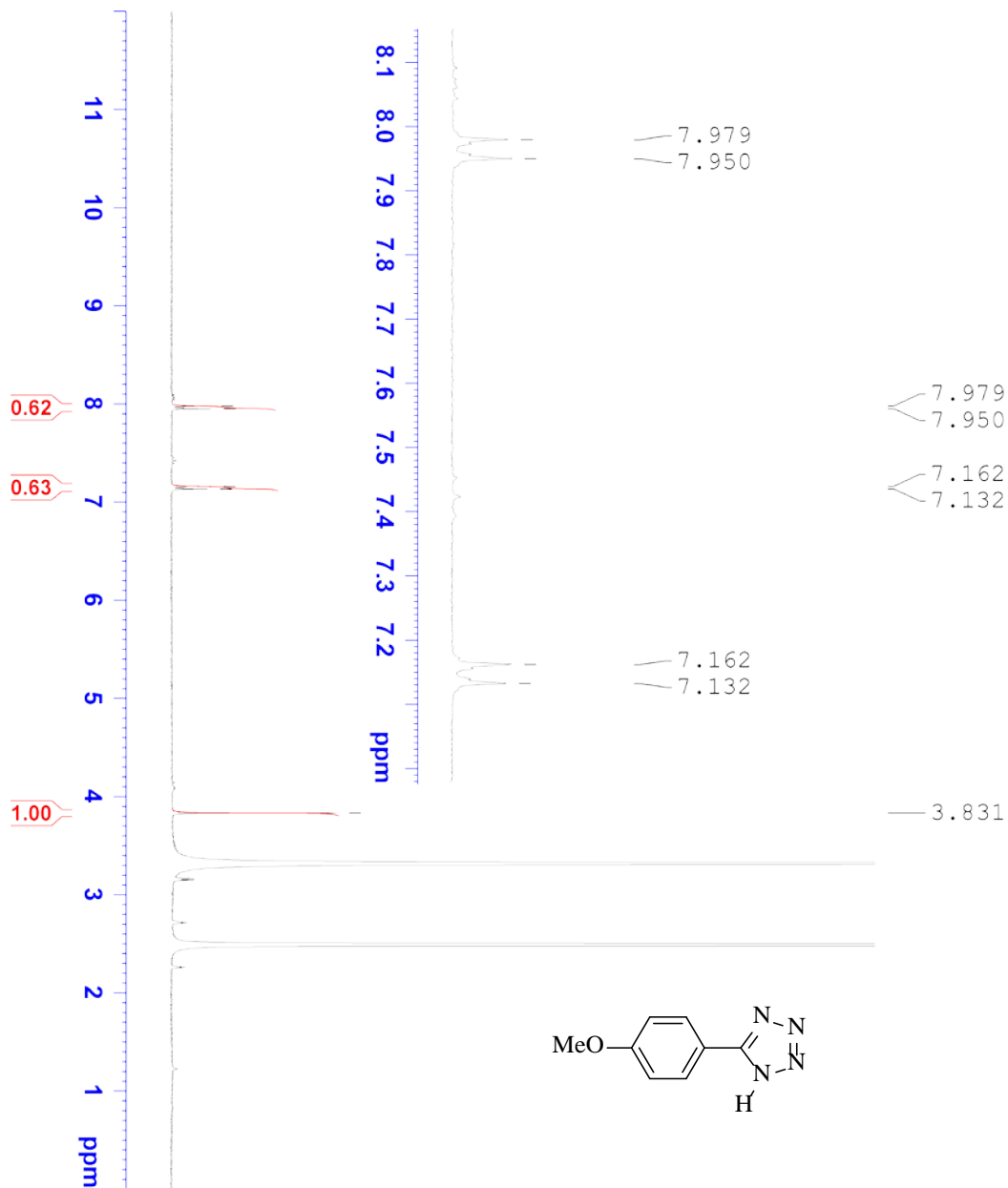


Figure-S14a

SARFARAZ/DR, ABDUL HAMEED/ SA-I-83
¹H

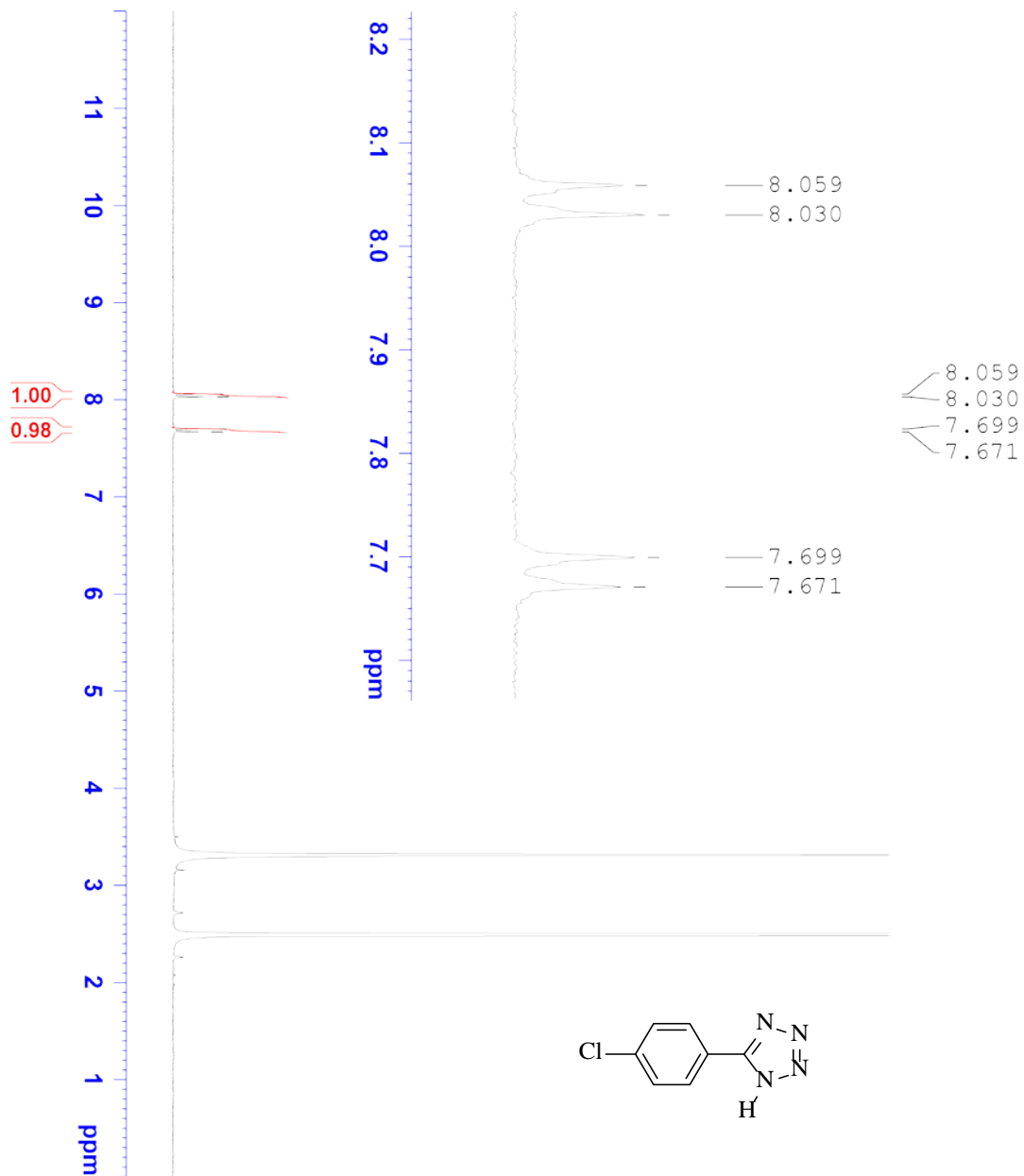


Current Data Parameters
 NAME: 5-(4-methoxyphenyl)-1H-tetrazole
 EXPTNO: 5
 PROCNO: 1
 P2 - Acquisition Parameters
 Date_: 20170131
 Time: 10.45
 INSTRUM: spect
 PROBU: 5 mm BBO BB-1H
 PULPROG: zgpg30
 TO: 32768
 SOLVENT: DMSO
 NS: 64
 DS: 0
 SWH: 6009.413 Hz
 F2: 500.136074 MHz
 AQ: 2.7262976 sec
 RG: 203
 DW: 83.200 usec
 DE: 6.50 usec
 TE: 300.0 K
 D1: 2.00000000 sec
 T100: 1
 CHANNEL f1
 NUC1: ¹H
 P1: 12.50 usec
 PL1: 0 dB
 F1A: 13.16228485 MHz
 SFO1: 300.13624010 MHz
 P2 - Processing parameters
 SI: 32768
 SF: 300.136074 MHz
 DS: 4
 SSB: 0
 GB: 0
 PC: 1.00



Figure-S15

SARFARAZ/DR, ABDUL HAMEED/SA-I-80/
/1H



Current Data Parameters
NAME 5-(4-chlorophenyl)-1H-tetrazole
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20170112
Time 5:44
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 64
DS 0
SS 0
AQ 6009.613350 Hz
ETDRES 0.181399 Hz
RG 2.7262976 sec
RG 203
FWD 83.200 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

CHANNEL f1 1H
NUC1 1H
P1 12.50 usec
PL1 0 dB
PR1W 13.16228485 M
SFO1 300.1324010 MHz

F2 - Processing parameters
SI 16384
SF 300.1300037 MHz
WDW EM
SSB 0
GB 0
PC 1.00



Figure-S16

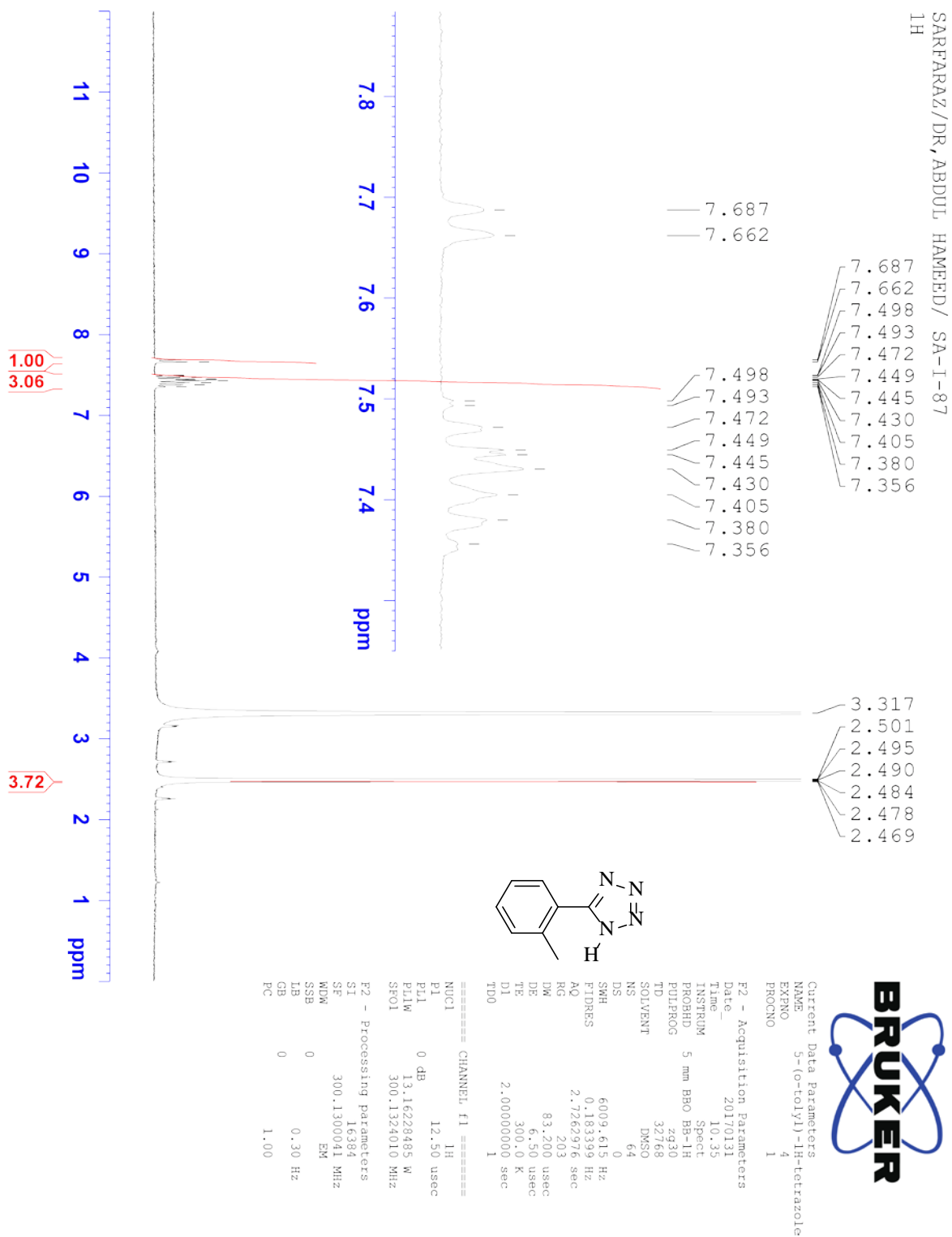
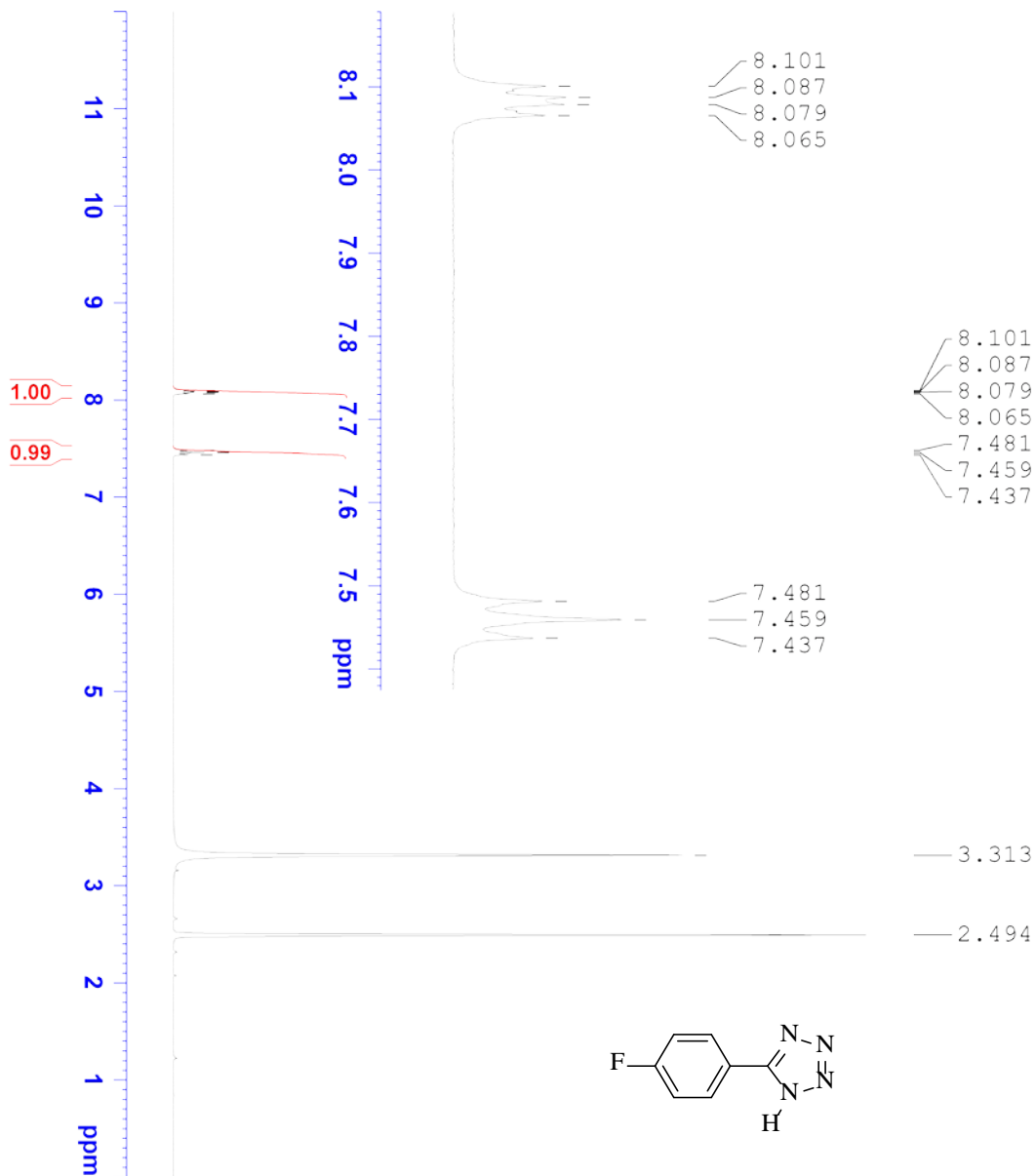


Figure-S17

SARFRAZ/DR.HAMEED/SI.I.82
¹H



BRUKER

Current Data Parameters
 NAME 5-(4-Fluorophenyl)-1H-tetrazole
 EXPNO 3
 PROCNO 1

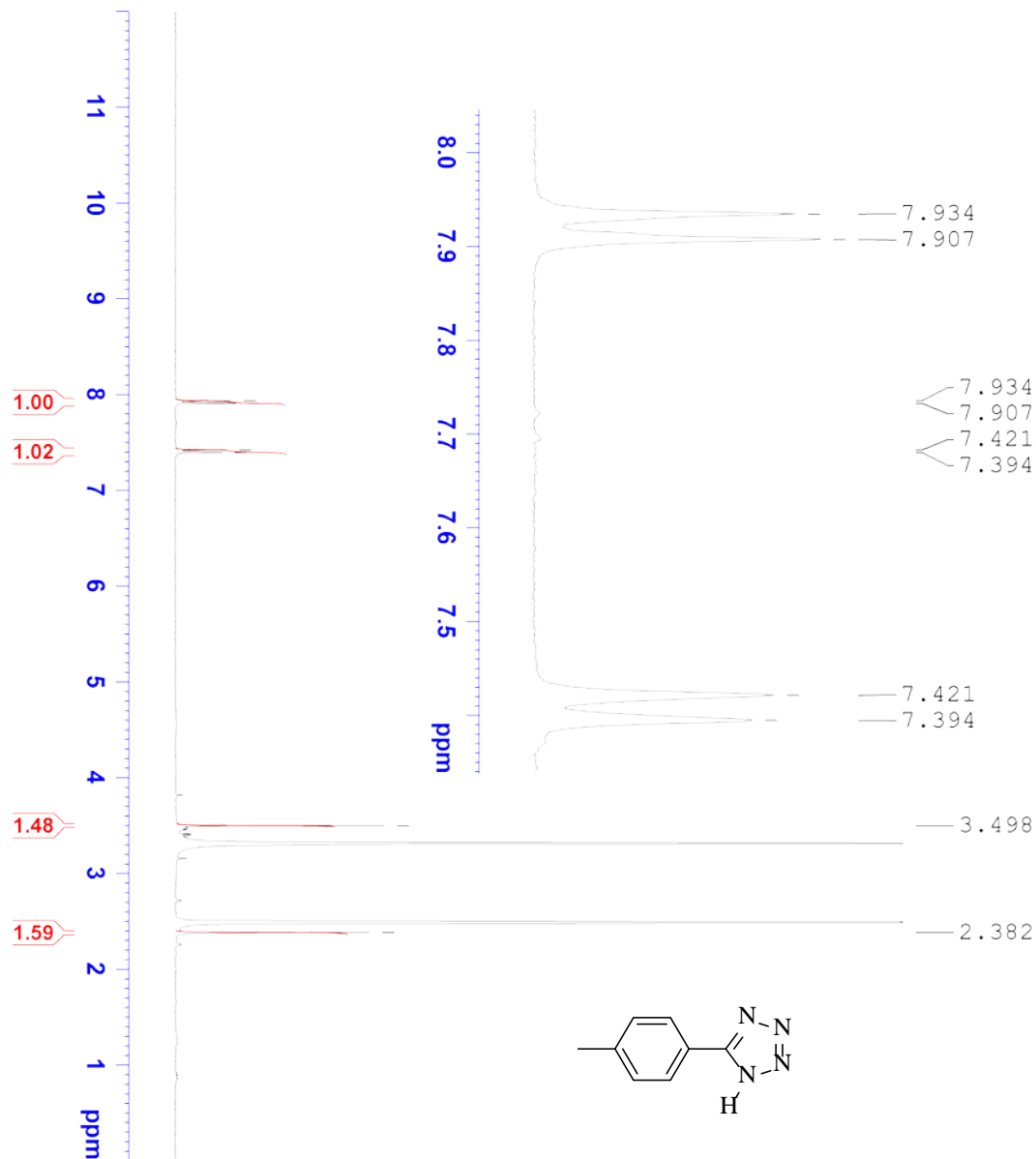
F2 - Acquisition Parameters
 Date_ 20170113
 Time_ 17.58
 INSTRUM spect
 PULPROG zgpg30
 TD 65536
 SOLVENT DMSO
 NS 64
 DS 4
 SWH 8012.800 Hz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 574.7
 DW 62.400 usec
 DE 6.50 usec
 TE 300.2 K
 D0 2.00000000 sec
 TDO 1

CHANNEL f1
 NUC1 ¹H
 P1 10.00 usec
 PL 3.00 dB
 SFO1 400.032002 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0100041 MHz
 WF 0
 WDS 0.30 Hz
 GB 0
 PC 1.00

Figure-S18

SARFARAZ/DR, ABDUL HAMEED/SA-I-86/
ICCBS, U.O.K/



Current Data Parameters
NAME 5-(p-tolyl)-1H-tetrazole
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters

Date_ 20170123
Time 14.55
INSTRUM Spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 64
DS 0
SWH 6009.615 Hz
FIDRES 0.193399 Hz
AQ 2.7262976 sec
RG 203
DW 83.200 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====

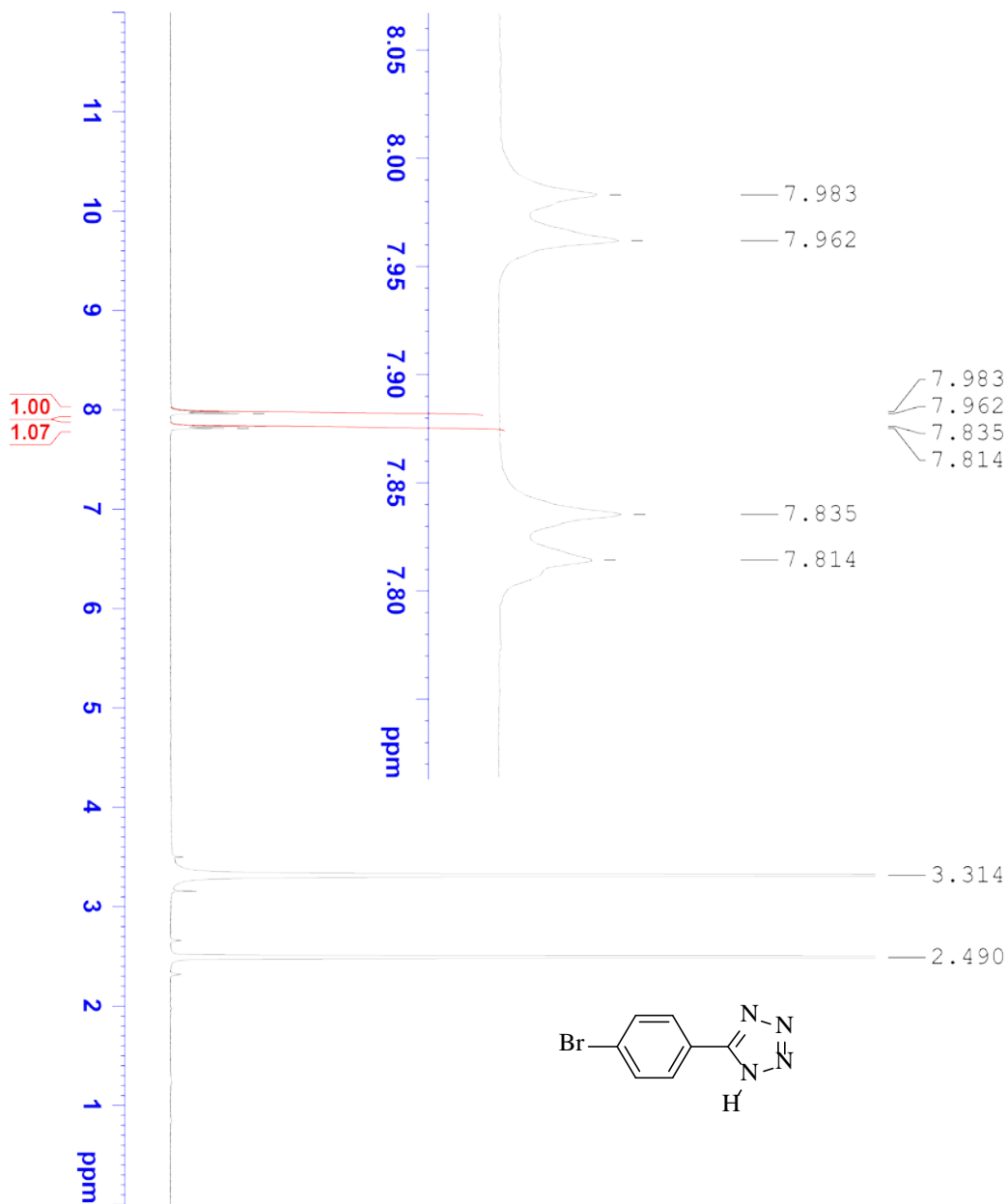
NUC1 1H
P1 12.50 usec
PL1 0 dB
PL1W 13.16228485 W
SFO1 300.1324010 MHz

F2 - Processing parameters

SI 16384
SF 300.1300040 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Figure-S19

SARFRAZ/DR.HAMEED/SI.I.81
¹H



BRUKER

Current Data Parameters
 NAME 5-(4-bromophenyl)-1H-tetrazole
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20170113
 Time 18.10
 INSTRUM spect
 PROBRD 5 mm SSI 1H-13
 PULPROG zgpg30
 TD 65536
 SFO1 400.132002 MHz
 FIDRES 0.122266 Hz
 AQ 4.0894465 sec
 RG 512
 DM 62.410 usec
 DE 1.450 usec
 TE 300.0 K
 D1 2.00000000 sec
 TD0 1

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.00 usec
 PL1 3.00 dB
 SFO1 400.032002 MHz

F2 - Processing parameters
 SI 32768
 SF 400.0100041 MHz
 NH 256
 SH 0
 FWHM 0.30 Hz
 LB 0
 GB 0
 PC 1.00

Figure-S20

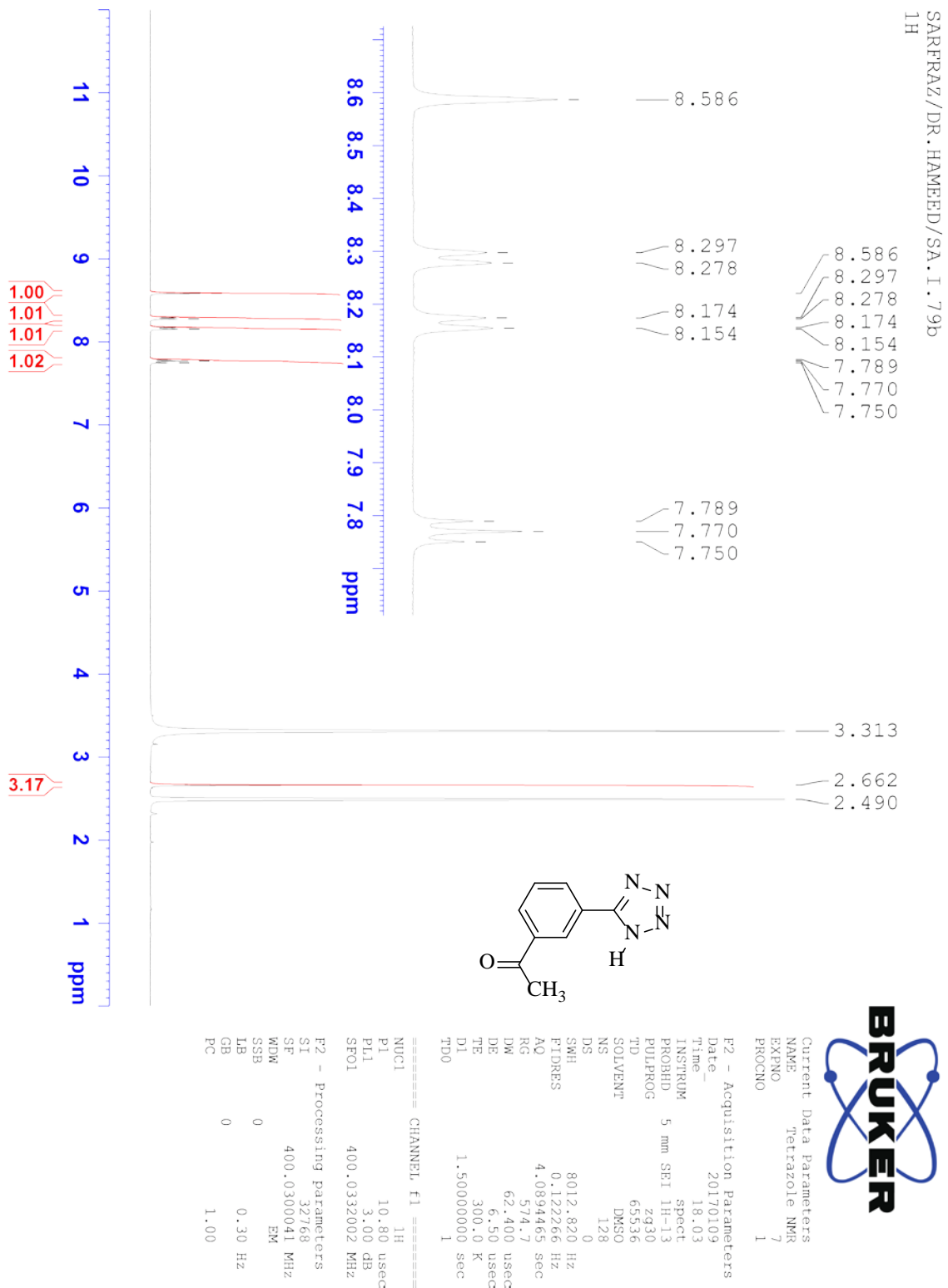


Figure-S21

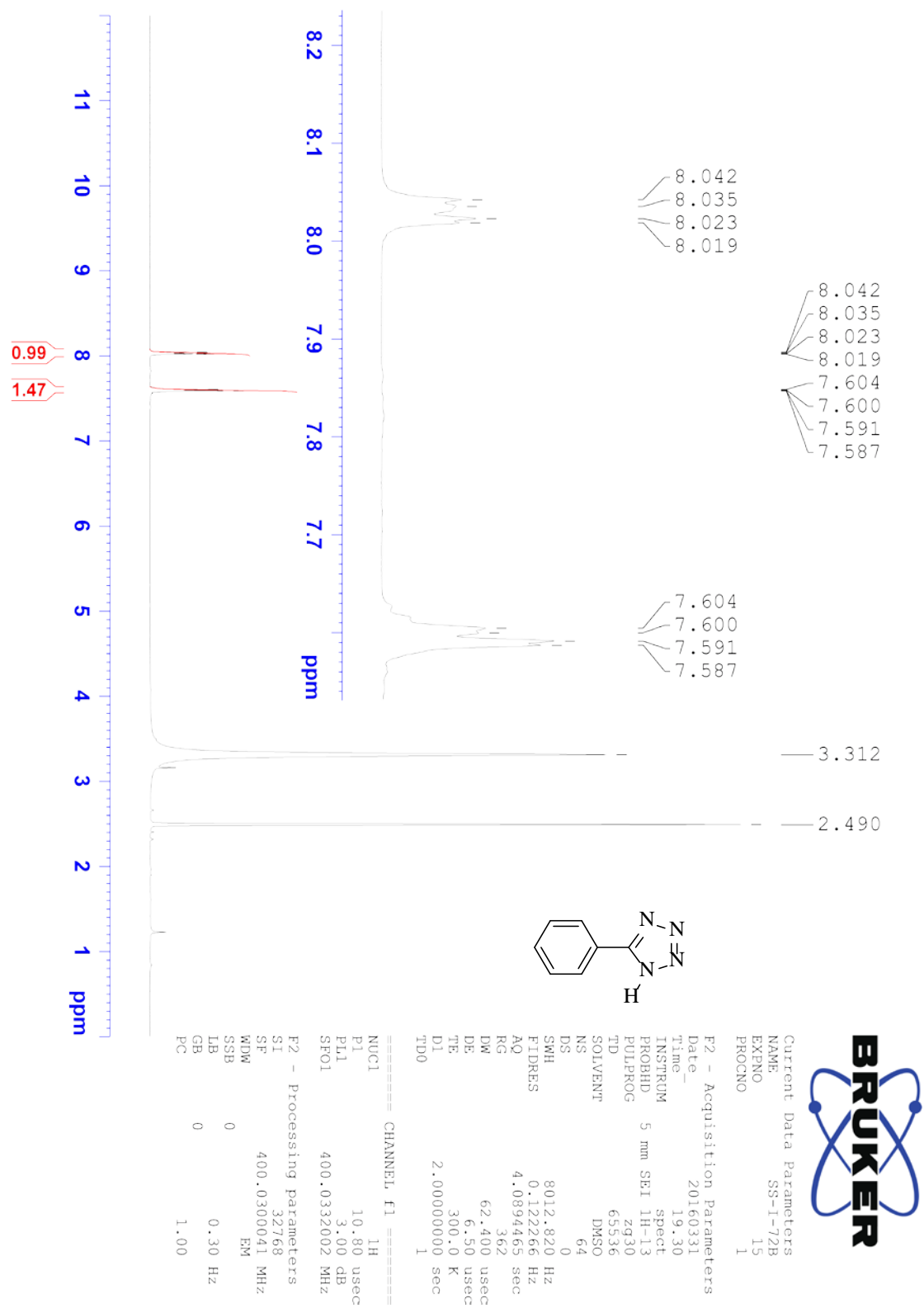


Figure-S22