

PEG-assisted two-component approach for the facile synthesis of 5-aryl-1,2,4-triazolidine-3-thiones under catalyst-free conditions

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Experimental

Reagents and equipments

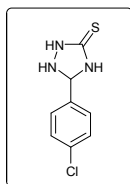
Variety of substituted aldehydes, thiosemicarbazides, polyethylene glycol and solvents were purchased from commercial sources (Sigma-Aldrich) and were used as received. Reactions were monitored by TLC using silica gel pre-coated plates with ethyl acetate/hexanes as the mobile phase. Melting points were determined with an electrothermal apparatus by open capillary method and are uncorrected. IR spectra were recorded on a Bruker FT-IR27 spectrophotometer using KBr optics. NMR spectra were recorded on a BRUKER DRX-300 spectrometer (300 MHz for ^1H NMR and 75 MHz for ^{13}C NMR) in DMSO and δ values are expressed in ppm using TMS as an internal standard.

Typical procedure for the synthesis of 5-aryl-[1,2,4]triazolidine-3-thione derivatives

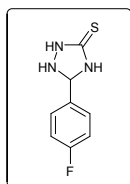
A mixture of aromatic aldehyde or cyclic ketone (2.0 mmol) and thiosemicarbazide (2.0 mmol) was taken in 0.5 ml of PEG-400. The resulting mixture was allowed to stir at 80 °C for the time specified in Table 3-5. After completion of the reaction (monitored by TLC), the reaction mixture was cooled to room temperature and treated with de-ionized water (10 mL), well-stirred for few minutes and the generated solid was filtered off. The crude product was recrystallized from 95% ethanol to afford the corresponding highly pure 1,2,4-triazolidine-3-

thione derivatives. The recovered PEG can be reused for further two more cycles with negligible loss of its efficiency.

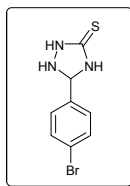
Spectral data for the synthesized compounds



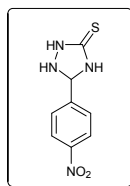
5-(4-Chlorophenyl)-1,2,4-triazolidine-3-thione (**3b**): White crystal, Mp. 206-208 °C; IR (KBr): 3435, 3282, 3165, 1598, 1524, 1466, 1363, 1282, 1224, 1165, 1086, 1013, 925, 871, 695, 513 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 7.46 (d, 2H, ArH, $J = 8.4$ Hz), 7.84 (d, 2H, ArH, $J = 8.4$ Hz), 8.02 (s, 1H, CH), 8.08 (s, 1H, NH), 8.24 (s, 1H, NH), 11.49 (s, 1H, NH). $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 129.08, 129.29, 133.62, 134.60, 141.35, 178.57; Anal. Calcd. for $\text{C}_8\text{H}_8\text{ClN}_3\text{S}$ (213.69): C, 44.97; H, 3.77; N, 19.66; Found; C, 44.68; H, 3.27; N, 19.02.



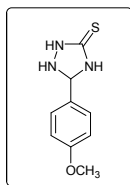
5-(4-Fluorophenyl)-1,2,4-triazolidine-3-thione (**3c**): White crystal, Mp. 172-174 °C; IR (KBr): 3462, 3288, 3165, 1599, 1522, 1464, 1396, 1285, 1168, 1089, 1065, 1010, 925, 815, 611, 511 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 7.46 (d, 2H, ArH, $J = 8.4$ Hz), 7.84 (d, 2H, ArH, $J = 8.4$ Hz), 8.02 (s, 1H, CH), 8.08 (s, 1H, NH), 8.25 (s, 1H, NH), 11.49 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 128.53, 129.16, 135.05, 134.78, 141.37, 179.04; Anal. Calcd. for $\text{C}_8\text{H}_8\text{FN}_3\text{S}$ (197.23): C, 48.72; H, 4.09; N, 21.30; Found; C, 47.96; H, 3.66; N, 20.79.



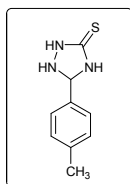
5-(4-Bromophenyl)-1,2,4-triazolidine-3-thione (**3d**): White crystal, Mp. 202-204 °C; IR (KBr): 3432, 3287, 3167, 1596, 1521, 1470, 1361, 1282, 1166, 1089, 1007, 926, 870, 698, 507 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 7.60 (d, 2H, ArH, $J = 8.7$ Hz), 7.78 (d, 2H, ArH, $J = 8.4$ Hz), 8.00 (s, 1H, CH), 8.08 (s, 1H, NH), 8.25 (s, 1H, NH), 11.49 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 128.84, 129.42, 134.12, 134.96, 141.53, 179.11; Anal. Calcd. for $\text{C}_8\text{H}_8\text{BrN}_3\text{S}$ (258.14): C, 37.22; H, 3.12; N, 16.28; Found; C, 37.06; H, 3.06; N, 16.01.



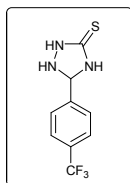
5-(4-Nitrophenyl)-1,2,4-triazolidine-3-thione (**3f**): Yellow solid, Mp. 226-228 °C; IR (KBr): 3308, 3138, 2984, 1594, 1538, 1442, 1396, 1265, 1196, 1081, 1009, 926, 861, 690, 511 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 7.59 (d, 2H, ArH, $J = 6.9$ Hz), 7.77 (d, 2H, ArH, $J = 6.9$ Hz), 8.01 (s, 1H, CH), 8.08 (s, 1H, NH), 8.25 (s, 1H, NH), 11.50 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 128.76, 129.57, 133.94, 135.26, 141.82, 178.85; Anal. Calcd. for $\text{C}_8\text{H}_8\text{N}_4\text{O}_2\text{S}$ (224.24): C, 42.85; H, 3.60; N, 24.99; Found; C, 42.08; H, 2.94; N, 24.17.



5-(4-Methoxyphenyl)-1,2,4-triazolidine-3-thione (**3g**): White crystal, Mp. 156-158 °C; IR (KBr): 3400, 3288, 3148, 1601, 1529, 1360, 1237, 1174, 1081, 1014, 948, 820, 602, 514 cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆): δ 3.78 (s, 3H, OCH₃), 6.97 (d, 2H, ArH, J = 8.7 Hz), 7.75 (d, 2H, ArH, J = 8.7 Hz), 7.92 (s, 1H, CH), 7.99 (s, 1H, NH), 8.12 (s, 1H, NH), 11.32 (s, 1H, NH); ¹³C-NMR (75 MHz, DMSO-d₆): 55.66, 114.18, 127.20, 129.41, 142.46, 161.55, 177.89; Anal. Calcd. for C₉H₁₁N₃OS (209.27): C, 51.65; H, 5.30; N, 20.08; Found; C, 51.46; H, 5.18; N, 19.22.

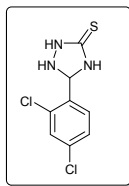


5-(*p*-Tolyl)-1,2,4-triazolidine-3-thione (**3h**): White crystal, Mp. 174-176 °C; IR (KBr): 3399, 3242, 3152, 1596, 1536, 1462, 1362, 1292, 1227, 1099, 950, 818, 627, 513 cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆): δ 2.31 (s, 3H, CH₃), 7.22 (d, 2H, ArH, J = 8.7 Hz), 7.69 (d, 2H, ArH, J = 8.4 Hz), 7.95 (s, 1H, CH), 8.01 (s, 1H, NH), 8.17 (s, 1H, NH), 11.38 (s, 1H, NH); ¹³C-NMR (75 MHz, DMSO-d₆): 23.08, 127.42, 129.90, 131.44, 140.12, 144.68, 178.23; Anal. Calcd. for C₉H₁₁N₃S (193.27): C, 55.93; H, 5.74; N, 21.74; Found; C, 55.68; H, 5.39; N, 21.31.

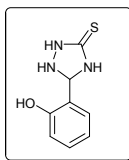


5-(4-(Trifluoromethyl)phenyl)-1,2,4-triazolidine-3-thione (**3i**): White crystal, Mp. 160-162 °C; IR (KBr): 3429, 3265, 3158, 1598, 1531, 1469, 1360, 1327, 1229, 1163, 1063, 1015, 932, 833, 596, 530 cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆): δ 7.74 (d, 2H, ArH, J = 8.7 Hz), 8.04 (d, 2H, ArH, J = 8.7 Hz), 8.10 (s, 1H, CH), 8.18 (s, 1H, NH), 8.34 (s, 1H, NH), 11.62 (s, 1H, NH); ¹³C-NMR (75 MHz,

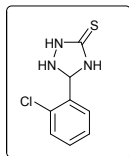
DMSO-d₆): 115.18, 123.69, 127.31, 130.18, 142.63, 162.15, 178.28; Anal. Calcd. for C₉H₈F₃N₃S (247.24): C, 43.72; H, 3.26; N, 17.00; Found; C, 43.58; H, 3.20; N, 16.88.



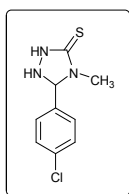
5-(2,4-Dichlorophenyl)-1,2,4-triazolidine-3-thione (**3k**): White solid, Mp. 230-232 °C; IR (KBr): 3452, 3286, 3168, 1602, 1527, 1469, 1360, 1287, 1225, 1160, 1073, 1017, 926, 876, 699, 528 cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆): δ 7.45 (d, 1H, ArH, J = 9Hz), 7.66 (d, 1H, ArH, J = 9Hz), 8.19 (s, 1H, ArH), 8.33 (s, 1H, CH), 8.36 (s, 1H, NH), 8.40 (s, 1H, NH), 11.66 (s, 1H, NH); ¹³C-NMR (75 MHz, DMSO-d₆): 128.98, 129.31, 133.14, 133.66, 134.78, 140.22, 141.52, 179.15; Anal. Calcd. for C₈H₇Cl₂N₃S (248.13): C, 38.72; H, 2.84; N, 16.93; Found; C, 38.64; H, 2.17; N, 16.24.



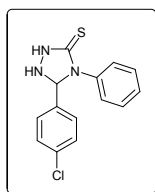
5-(2-Hydroxyphenyl)-1,2,4-triazolidine-3-thione (**3n**): White crystal, Mp. 216-218 °C; IR (KBr): 3438, 3318, 3174, 2989, 1608, 1538, 1484, 1362, 1272, 1205, 1112, 1059, 948, 827, 756, 625, 519 cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆): δ 6.87 (t, 2H, ArH, J = 9 Hz), 7.20 (s, 1H, CH), 7.92 (d, 2H, CH), 8.11 (s, 1H, NH), 8.37 (s, 1H, NH), 9.89 (s, 1H, OH), 11.38 (s, 1H, NH); ¹³C-NMR (75 MHz, DMSO-d₆): 116.13, 119.39, 120.35, 126.86, 131.21, 139.89, 156.47, 177.71; Anal. Calcd. for C₈H₉N₃OS (195.24): C, 49.21; H, 4.65; N, 21.52; Found; C, 48.99; H, 4.15; N, 21.08.



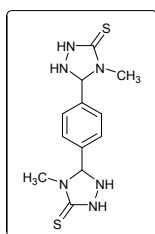
5-(2-Chlorophenyl)-1,2,4-triazolidine-3-thione (**3o**): White crystal, Mp. 198-200 °C; IR (KBr): 3415, 3250, 3153, 2983, 1610, 1519, 1465, 1375, 1290, 1226, 1126, 1045, 947, 869, 751, 607, 501 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 7.32 (m, 2H, ArH), 7.49 (m, 1H, ArH), 8.12 (s, 1H, CH), 8.31 (m, 1H, ArH), 8.28 (s, 1H, NH), 8.46 (s, 1H, NH), 11.62 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 118.24, 119.73, 120.51, 127.11, 131.57, 140.21, 156.62, 178.82; Anal. Calcd. for $\text{C}_8\text{H}_8\text{ClN}_3\text{S}$ (213.69): C, 44.97; H, 3.77; N, 19.66; Found; C, 44.24; H, 2.99; N, 18.93.



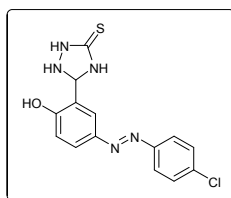
5-(4-Chlorophenyl)-4-methyl-1,2,4-triazolidine-3-thione (**3p**): White crystal, Mp. 178-180 °C; IR (KBr): 3421, 3307, 3138, 1594, 1539, 1441, 1359, 1265, 1195, 1081, 1006, 931, 858, 771, 694, 510 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 7.29 (s, 1H, NH), 7.37-7.65 (m, 8H, ArH), 7.95 (s, 1H, CH), 9.16 (s, 1H, NH), 10.48 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 128.12, 128.96, 129.08, 129.29, 132.31, 133.62, 134.60, 140.13, 141.35, 178.57; Anal. Calcd. for $\text{C}_{14}\text{H}_{12}\text{ClN}_3\text{S}$ (289.78): C, 58.03; H, 4.17; N, 14.50; Found; C, 57.88; H, 4.00; N, 13.97.



5-(4-Chlorophenyl)-4-phenyl-1,2,4-triazolidine-3-thione (**3q**): White crystal, Mp. 198-200 °C; IR (KBr): 3421, 3307, 3138, 1594, 1539, 1441, 1359, 1265, 1195, 1081, 1006, 931, 858, 771, 694, 510 cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆): δ 7.29 (s, 1H, NH), 7.37-7.65 (m, 8H, ArH), 7.95 (s, 1H, CH), 9.16 (s, 1H, NH), 10.48 (s, 1H, NH); ¹³C-NMR (75 MHz, DMSO-d₆): 128.12, 128.96, 129.08, 129.29, 132.31, 133.62, 134.60, 140.13, 141.35, 178.57; Anal. Calcd. for C₁₄H₁₂ClN₃S (289.78): C, 58.03; H, 4.17; N, 14.50; Found; C, 57.88; H, 4.00; N, 13.97.

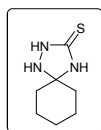


5,5'-(1,4-Phenylene)bis(4-methyl-1,2,4-triazolidine-3-thione) (**5b**): Yellow solid, Mp. >310 °C; IR (KBr): 3433, 3356, 3192, 1553, 1509, 1421, 1360, 1258, 1168, 1089, 1034, 935, 833, 768, 651, 534 cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆): δ 3.02 (s, 3H, CH₃), 7.82 (s, 4H, ArH), 8.04 (s, 1H, CH), 8.57 (s, 1H, NH), 11.55 (s, 1H, NH); ¹³C-NMR (75 MHz, DMSO-d₆): 27.28, 115.41, 125.12, 132.56, 179.72; Anal. Calcd. for C₁₂H₁₆N₆S₂ (308.43): C, 46.73; H, 5.23; N, 27.25; Found; C, 46.35; H, 5.06; N, 26.83.

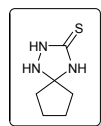


(*E*)-5(5-((4-Chlorophenyl)diazenyl)-2-hydroxyphenyl)-1,2,4-triazolidine-3-thione (**7a**): Yellow solid, Mp. >320 °C; IR (KBr): 3422, 3241, 3148, 2831, 1601, 1509, 1360, 1269, 1160, 1087, 1004, 948, 830, 779, 662, 508 cm⁻¹; ¹H-NMR (300 MHz, DMSO-d₆): δ 7.06 (s, 1H, ArH), 7.64 (d, 2H,

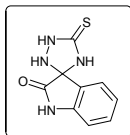
ArH, $J = 6.9$ Hz), 7.77 (s, 1H, CH), 7.89 (d, 2H, ArH, $J = 6.0$ Hz), 8.19 (d, 2H, ArH), 8.43 (s, 1H, NH), 8.59 (s, 1H, NH), 10.93 (s, 1H, OH), 11.50 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 116.86, 118.44, 121.14, 123.81, 129.48, 135.08, 138.37, 145.25, 150.62, 159.61, 163.59, 177.83; Anal. Calcd. for $\text{C}_{14}\text{H}_{21}\text{ClN}_5\text{OS}$ (333.80): C, 50.38; H, 3.62; N, 20.98; Found; C, 49.83; H, 2.96; N, 20.17.



1,2,4-Triazaspiro[4.5]decane-3-thione (**9a**): White crystal, Mp. 148-150 °C; IR (KBr): 3385, 3149, 2985, 2828, 1594, 1518, 1461, 1309, 1265, 1213, 1079, 1041, 873, 739, 591 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 1.54-1.60 (m, 6H, CH_2 , $J = 5.7$ Hz), 2.19-2.34 (t, 2H, CH_2 , $J = 5.7$ Hz), 2.50 (t, 2H, CH_2), 7.51 (s, 1H, NH), 7.96 (s, 1H, NH), 10.15 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 28.46, 25.63, 33.09, 163.64, 178.18; Anal. Calcd. for $\text{C}_7\text{H}_{13}\text{N}_3\text{S}$ (171.26): C, 49.09; H, 7.65; N, 24.54; Found; C, 48.57; H, 7.06; N, 23.86.

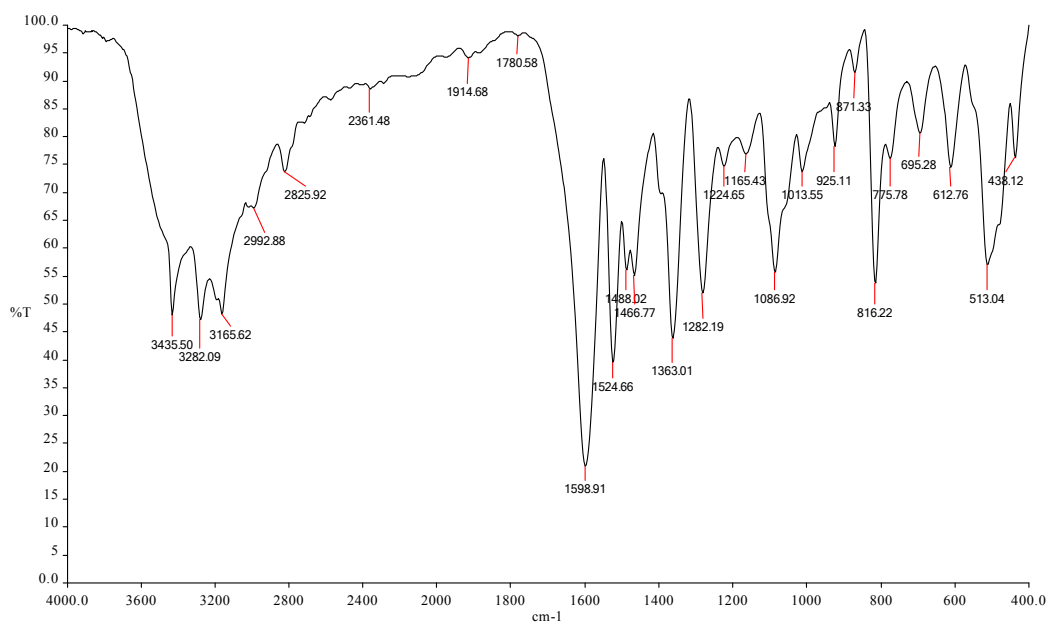


1,2,4-Triazaspiro[4.4]nonane-3-thione (**9b**): White crystal, Mp. 142-144 °C; IR (KBr): 3378, 3140, 2955, 2813, 1589, 1509, 1456, 1300, 1241, 1075, 1036, 861, 741, 577 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 1.60-1.78 (m, 4H, CH_2 , $J = 6.0$ Hz), 2.28-2.37 (m, 4H, CH_2 , $J = 7.2$ Hz), 7.47 (s, 1H, NH), 7.96 (s, 1H, NH), 9.82 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 28.46, 33.09, 163.64, 178.18; Anal. Calcd. for $\text{C}_6\text{H}_{11}\text{N}_3\text{S}$ (157.24): C, 45.83; H, 7.05; N, 26.72; Found; C, 45.11; H, 6.84; N, 26.07.

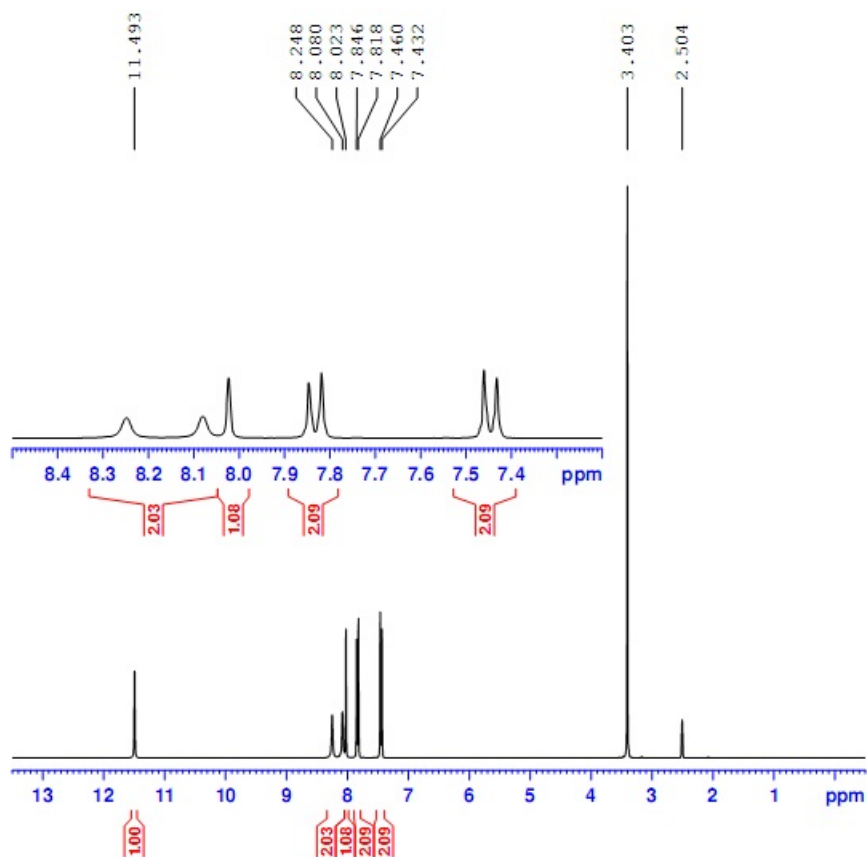


5'-Thioxospiro[indoline-3,3'-[1,2,4]-triazolidin]-2-one (**9c**): Yellow solid: Mp. 242-244 °C; IR (KBr): 3394, 3276, 3153, 1590, 1504, 1464, 1358, 1303, 1249, 1049, 895, 739, 584 cm^{-1} ; $^1\text{H-NMR}$ (300 MHz, DMSO-d_6): δ 6.94 (d, 1H, ArH, $J = 7.8$ Hz), 7.11 (t, 1H, ArH, $J = 7.5$ Hz), 7.37 (t, 1H, ArH, $J = 6.9$ Hz), 7.66 (d, 1H, ArH, $J = 7.5$ Hz), 8.69 (s, 1H, NH), 9.04 (s, 1H, NH), 11.21 (s, 1H, NH), 12.45 (s, 1H, NH); $^{13}\text{C-NMR}$ (75 MHz, DMSO-d_6): 111.48, 120.47, 121.40, 122.84, 131.74, 132.51, 142.79, 163.10, 179.16; Anal. Calcd. for $\text{C}_9\text{H}_8\text{N}_4\text{OS}$ (220.25): C, 49.08; H, 3.66; N, 25.44; Found; C, 48.85; H, 3.09; N, 24.97.

Spectrum Name: FTIR-PCT.sp



FT-IR Spectrum of compound **3b**



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Current Data Parameters
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PROCNO       1

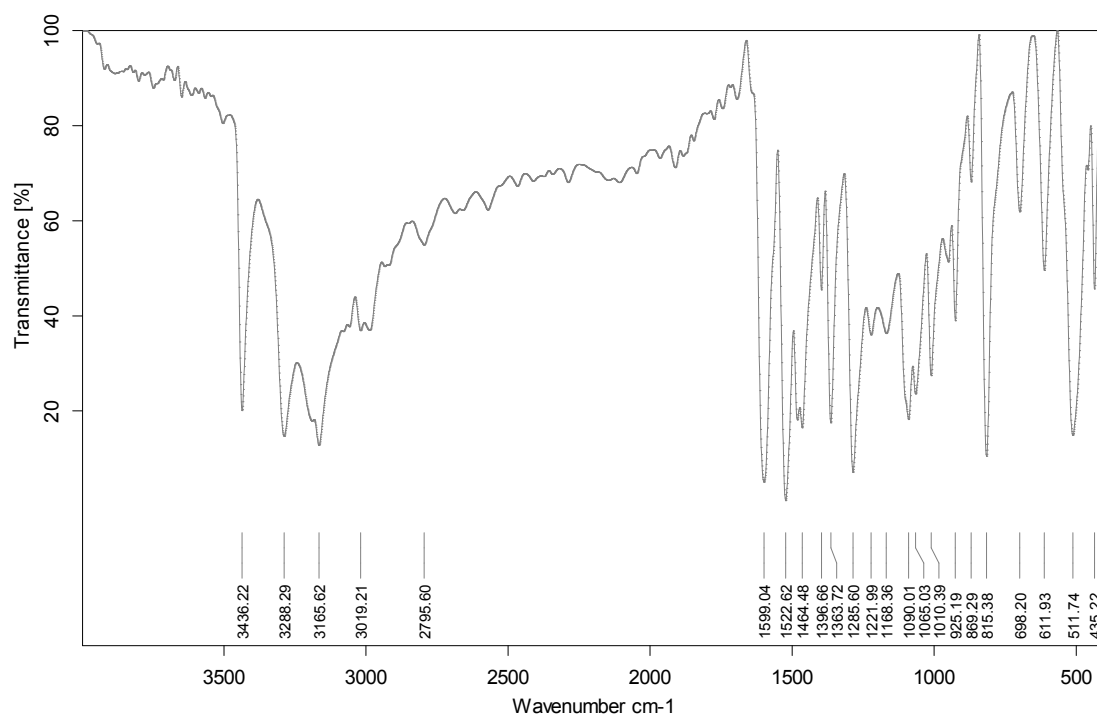
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FIDRES       0.094423 Hz
AQ           5.2953597 sec
RG           71.8
DW           80.800 usec
DE           6.00 usec
TE           300.0 K
D1           1.00000000 sec
TD0          1

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SFO1        300.1318534 MHz

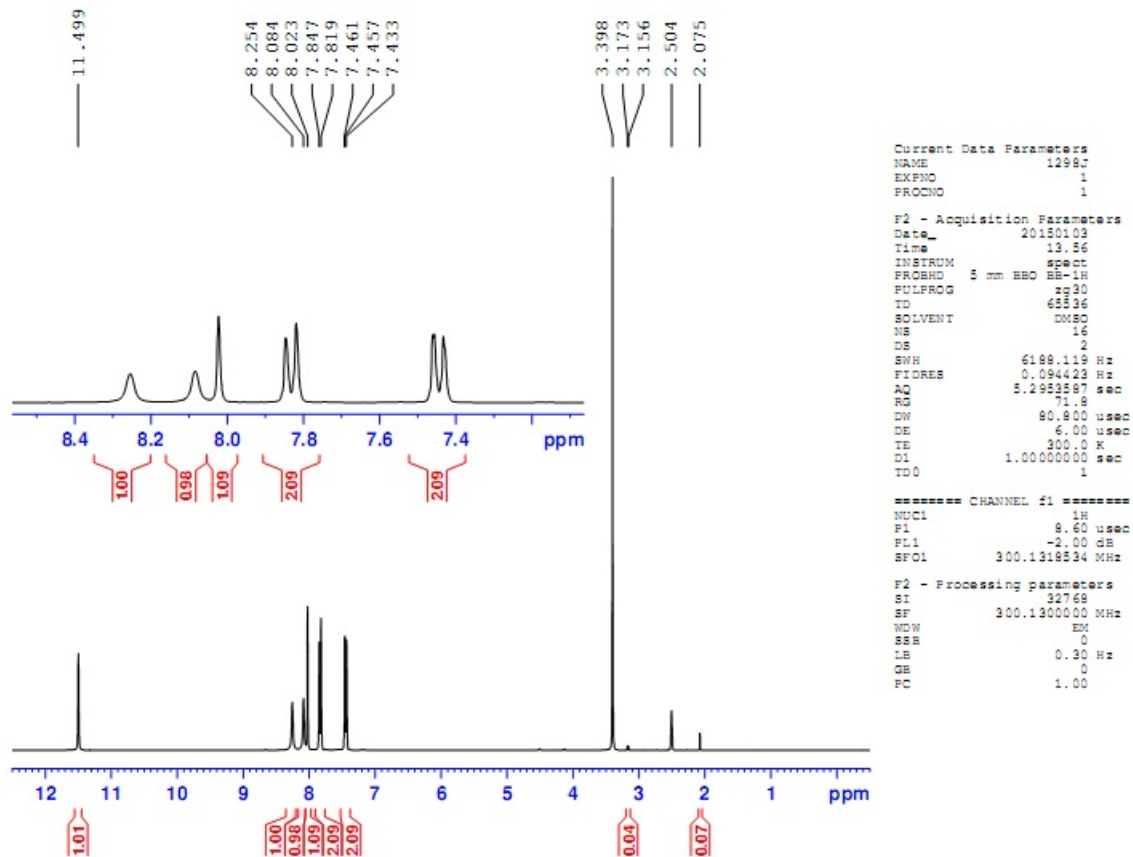
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¹H NMR Spectrum of compound **3b**

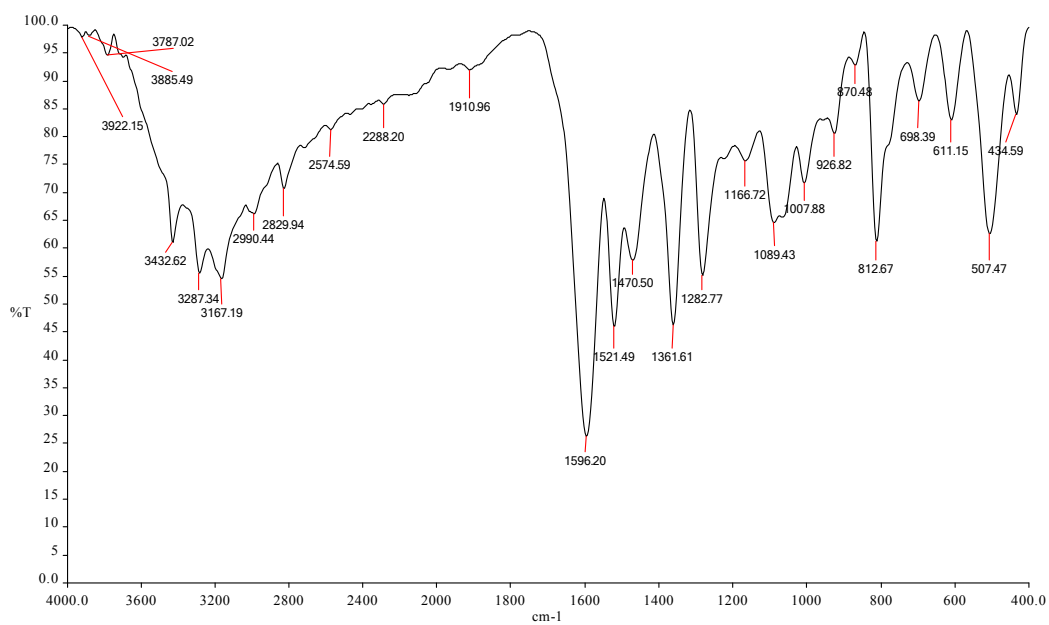


FT-IR Spectrum of compound 3c

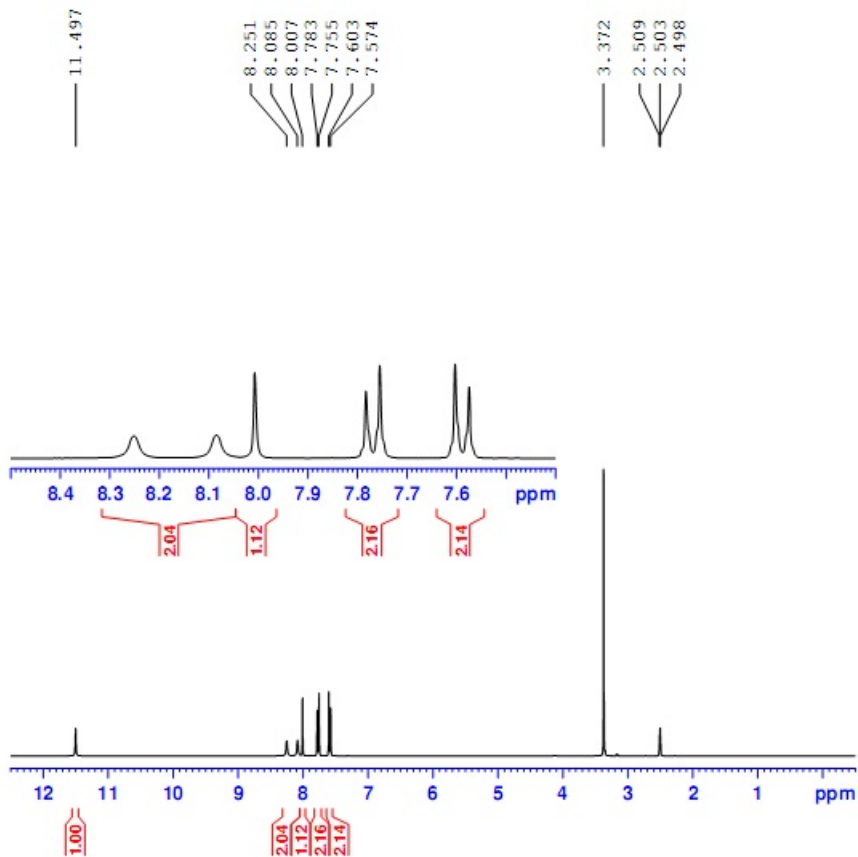


¹H NMR Spectrum of compound 3c

Spectrum Name: FTIR-BRTS.sp



FT-IR Spectrum of compound **3d**



```

Current Data Parameters
NAME      01590
EXPNO    1
PROCNO   1

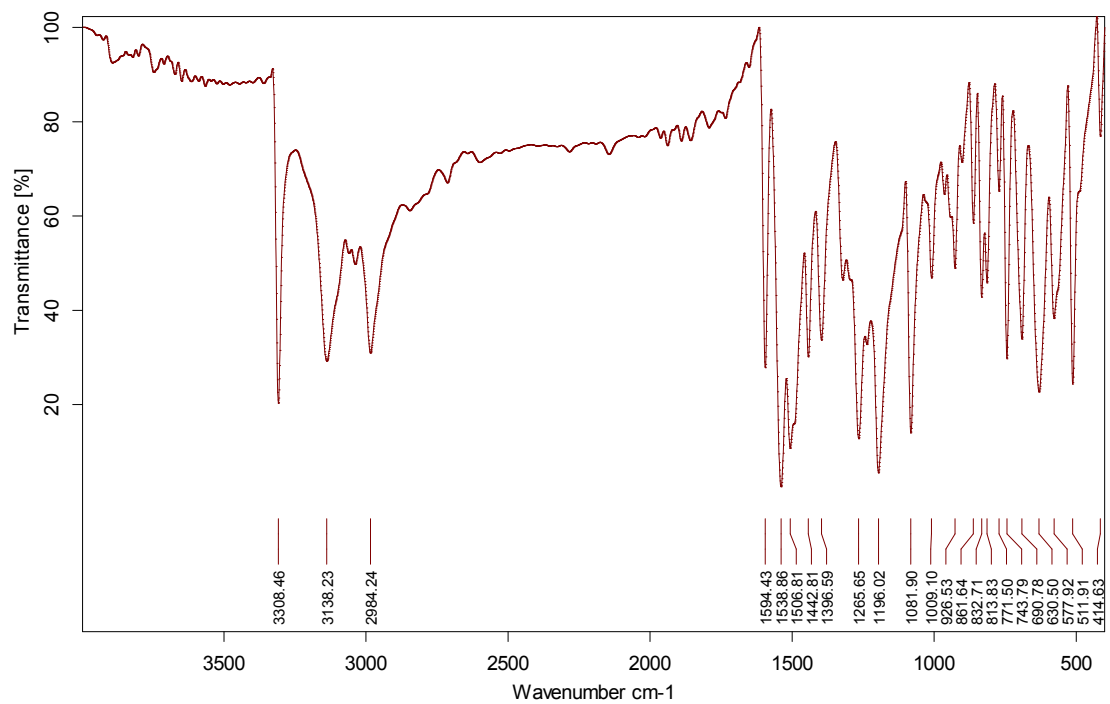
F2 - Acquisition Parameters
Date_    20180219
Time     10.36
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD        65536
SOLVENT  DMSO
NS        16
DS        2
SWH       6188.119 Hz
FIDRES    0.094423 Hz
AQ        5.2933587 sec
RG        181
DW        80.800 usec
DE        6.00 usec
TE        300.0 K
D1        1.00000000 sec
TD0       1

===== CHANNEL f1 =====
NUC1      1H
P1        8.60 usec
PL1       -2.00 dB
SFO1      300.1318534 MHz

F2 - Processing parameters
SI        32768
SF        300.1300000 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

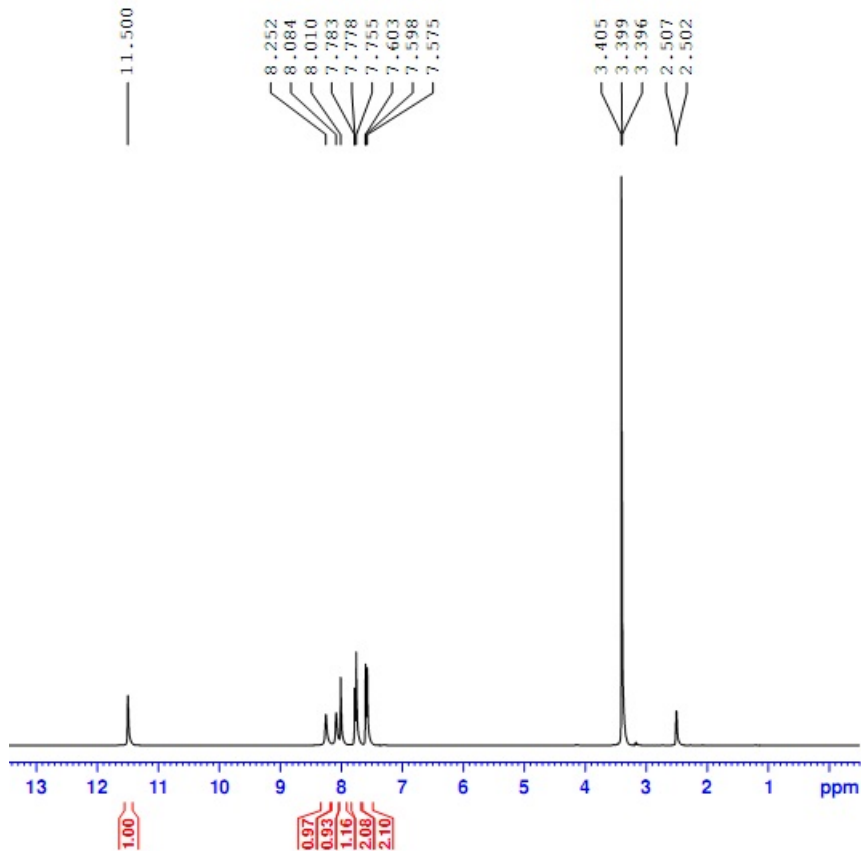
```

¹H NMR Spectrum of compound 3d



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IR Spectrum of compound 3f



```

Current Data Parameters
NAME          0159a
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20150224
Time         12.22
INSTRUM      spect
PROBHD       5 mm BBO BB-1H
PULPROG      zg30
TD           65536
SOLVENT      DMSO
NS           16
DS           2
SWH           6188.119 Hz
FIDRES       0.094423 Hz
AQ           5.2953587 sec
RG           128
DM           80.800 usec
DE           6.00 usec
TE           300.0 K
D1           1.00000000 sec
TD0          1

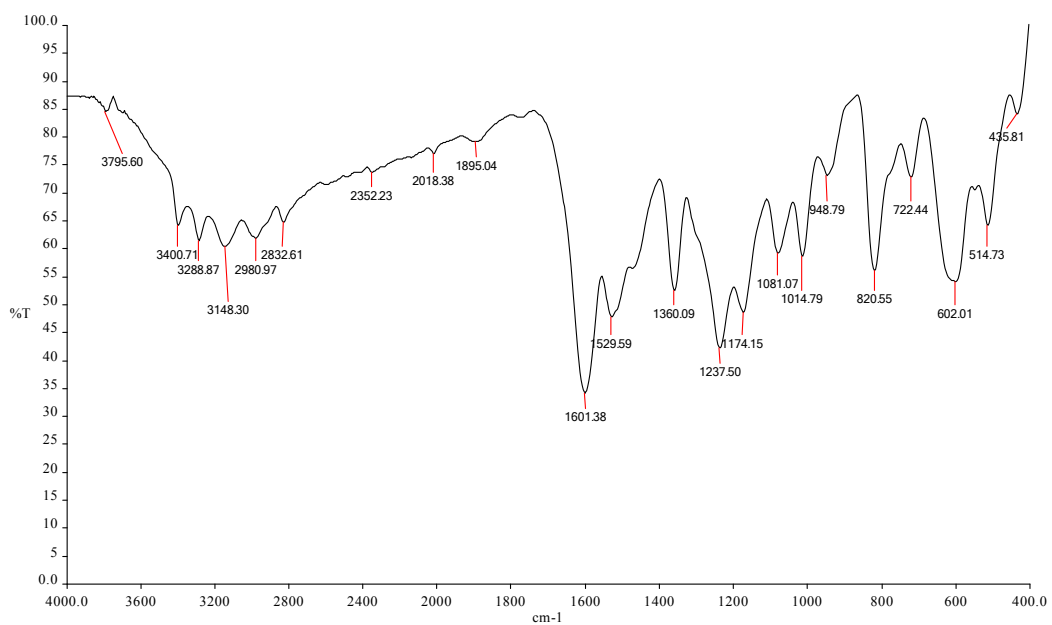
===== CHANNEL f1 =====
NUC1          1H
P1            8.60 usec
PL1          -2.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI            32768
SF           300.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

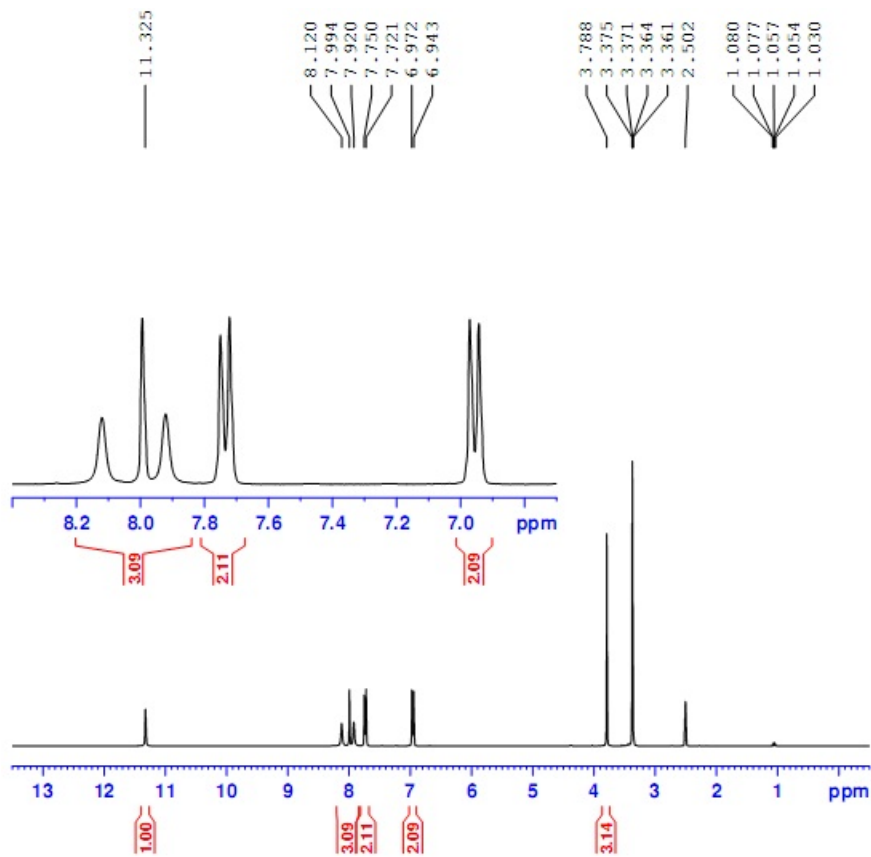
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¹H NMR Spectrum of compound **3f**

Spectrum Name: FTIR-ANTS.sp

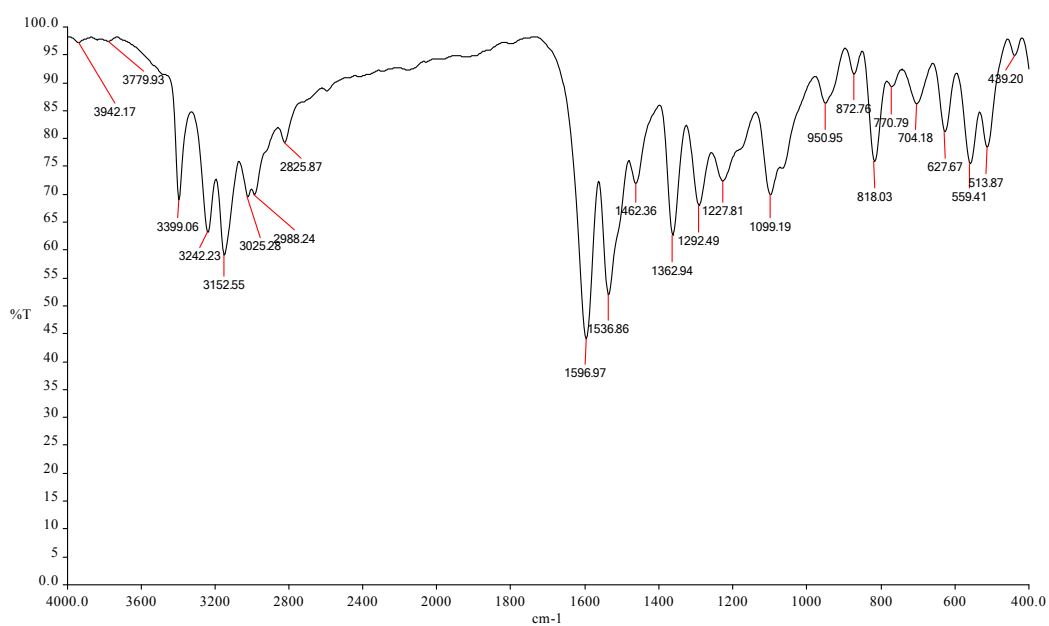


FT-IR Spectrum of compound **3g**

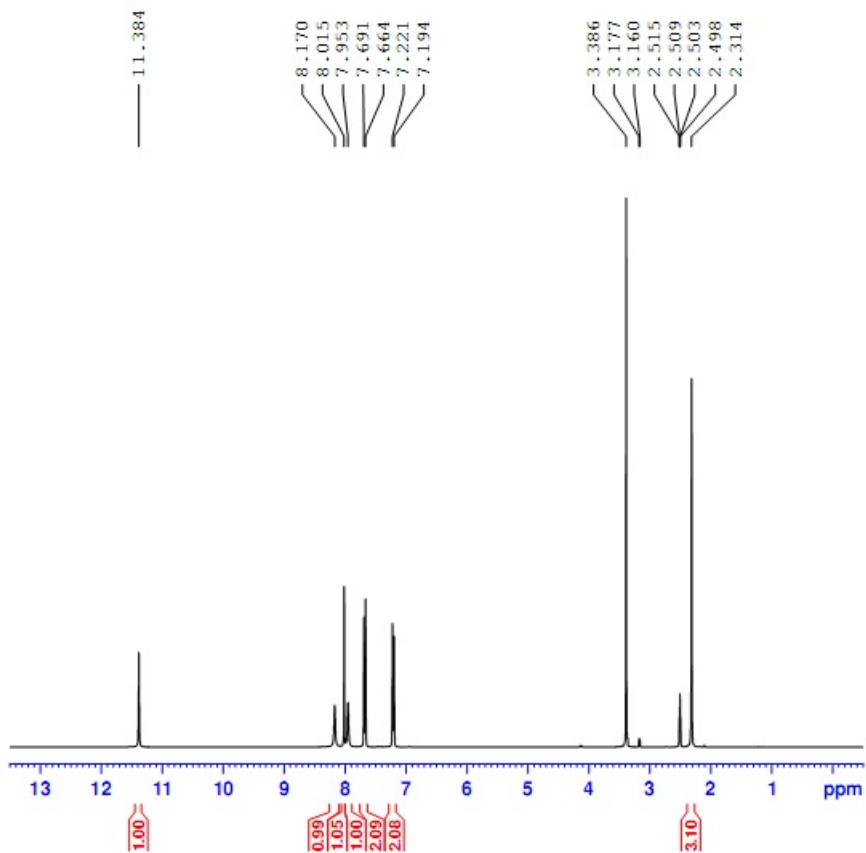


¹H NMR Spectrum of compound **3g**

Spectrum Name: FTIR-PTT.sp



FT-IR Spectrum of compound **3h**



```

Current Data Parameters
NAME          0159K
EXPNO         1
PROCNO        1

F2 - Acquisition Parameters
Date_         20150220
Time          13.58
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           6198.119 Hz
FIDRES        0.094423 Hz
AQ            5.2953987 sec
RG            71.8
DW            80.800 usec
DE            6.00 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

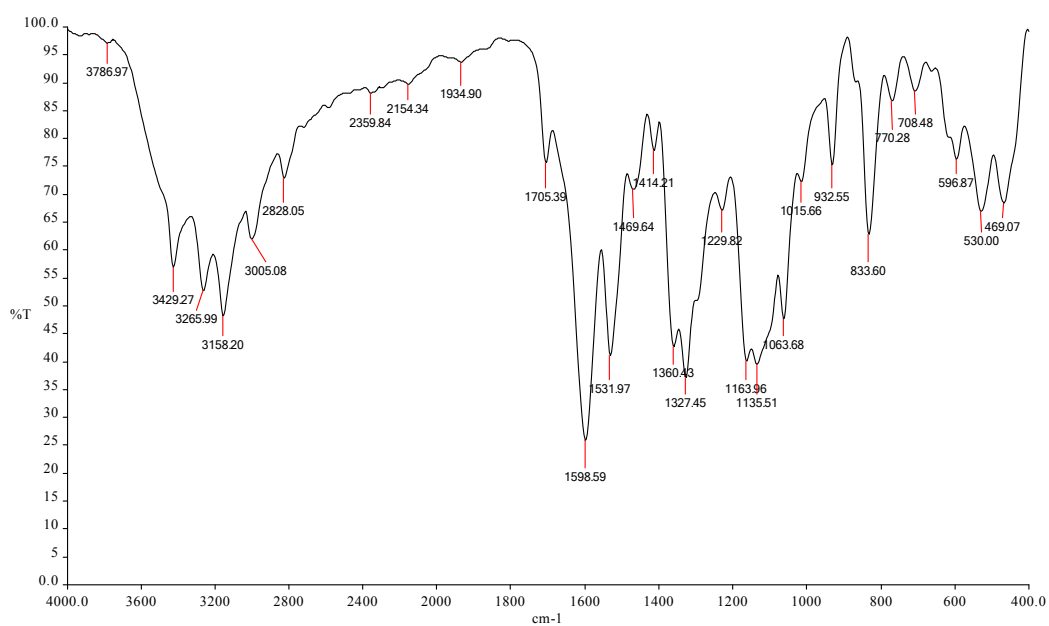
===== CHANNEL f1 =====
NUC1           1H
P1             8.60 usec
PL1            -2.00 dB
SFO1          300.1319534 MHz

F2 - Processing parameters
SI             32768
SF            300.1300000 MHz
WDW            EM
SSB            0
LB             0.30 Hz
GB             0
PC             1.00

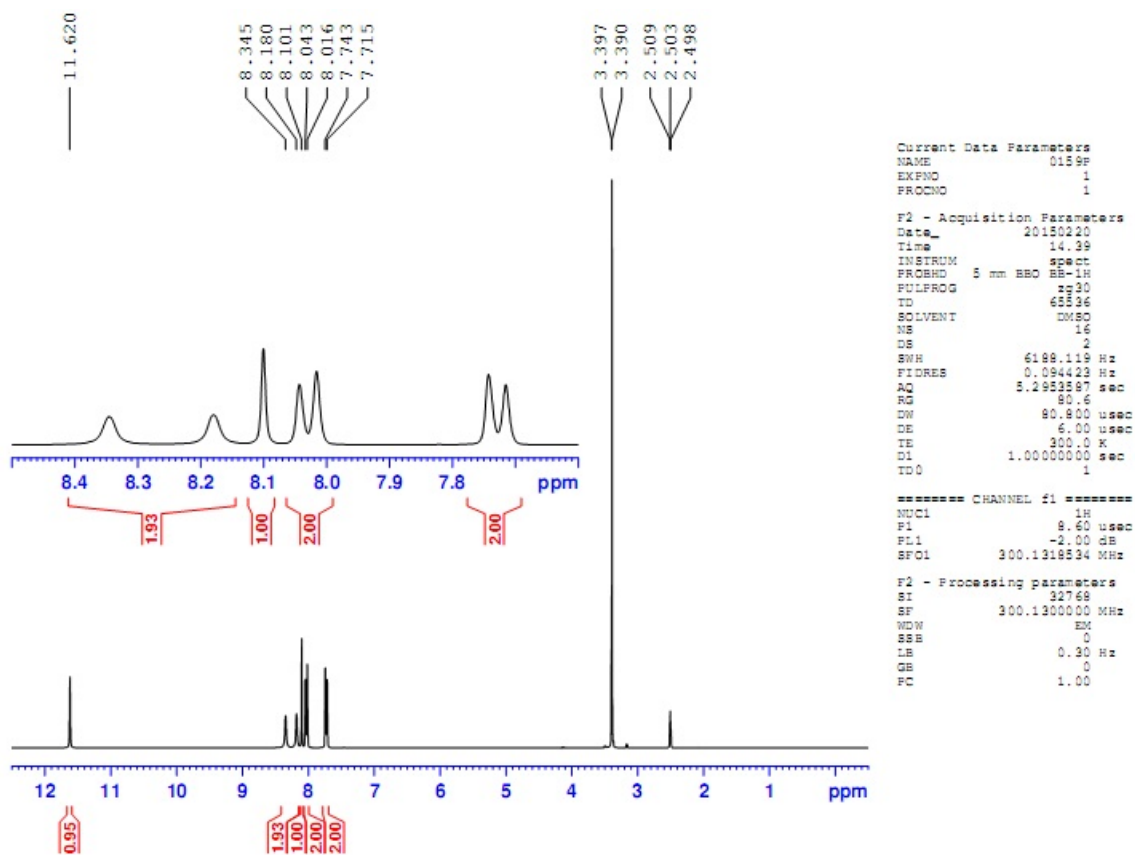
```

¹H NMR Spectrum of compound **3h**

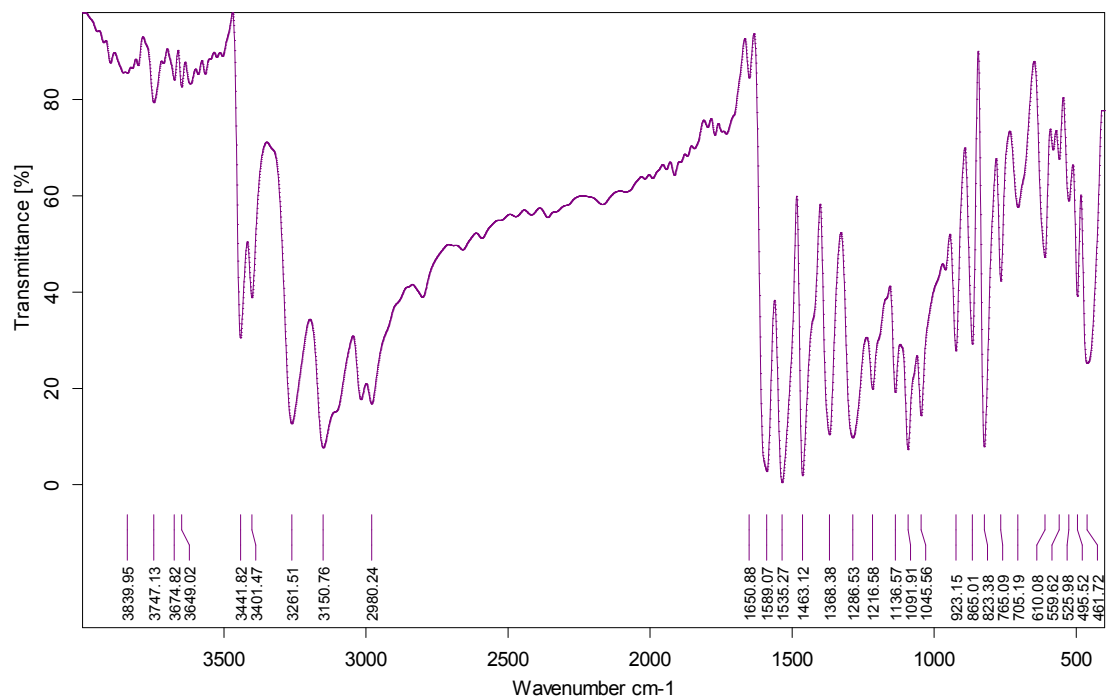
Spectrum Name: FTIR-TFT.sp



FT-IR Spectrum of compound **3i**

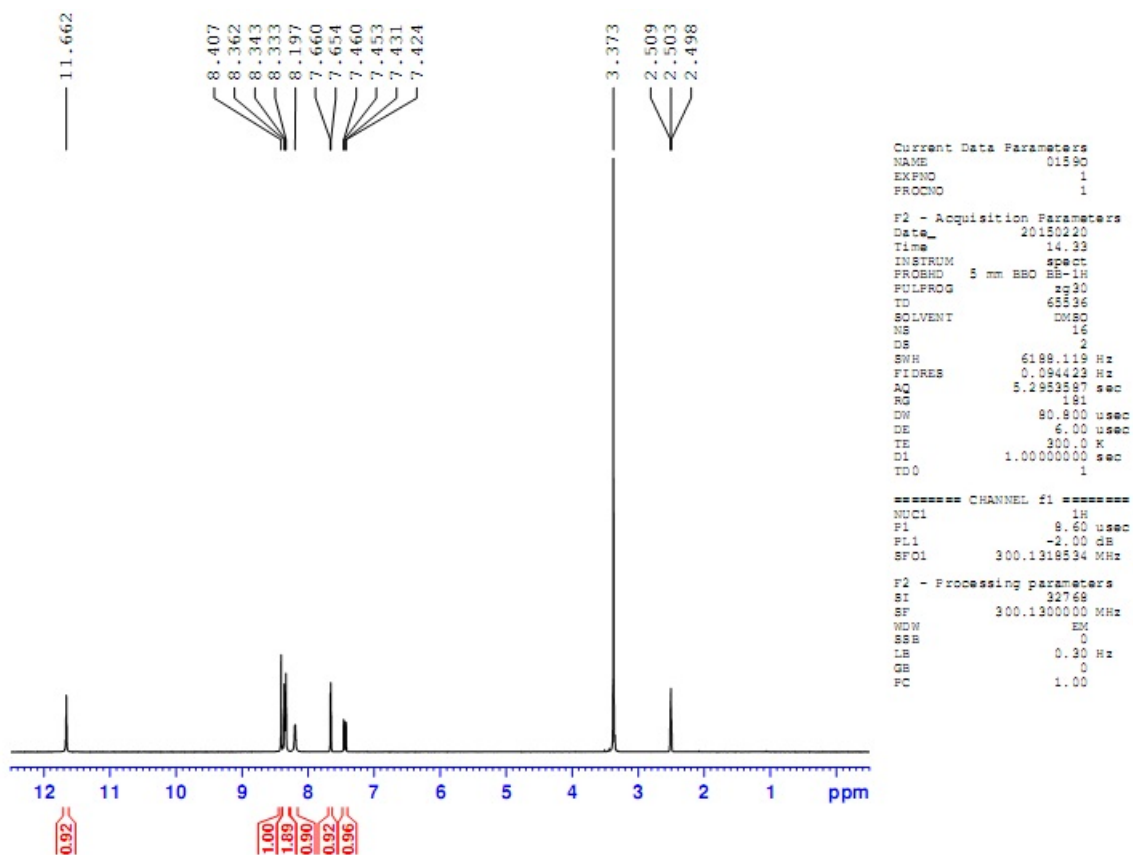


¹H NMR Spectrum of compound **3i**



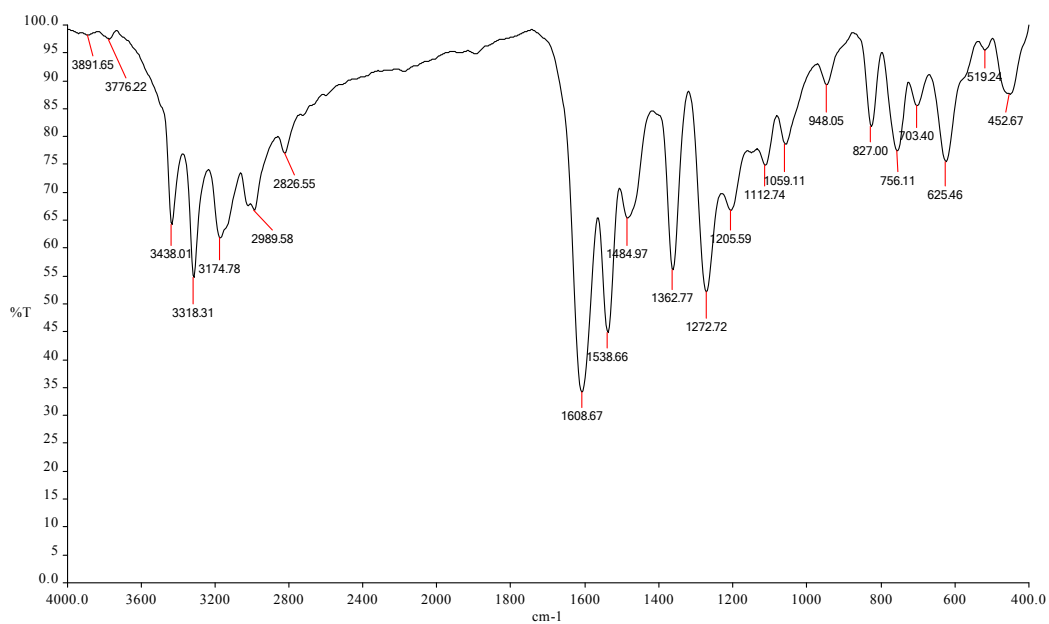
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FT-IR Spectrum of compound 3k

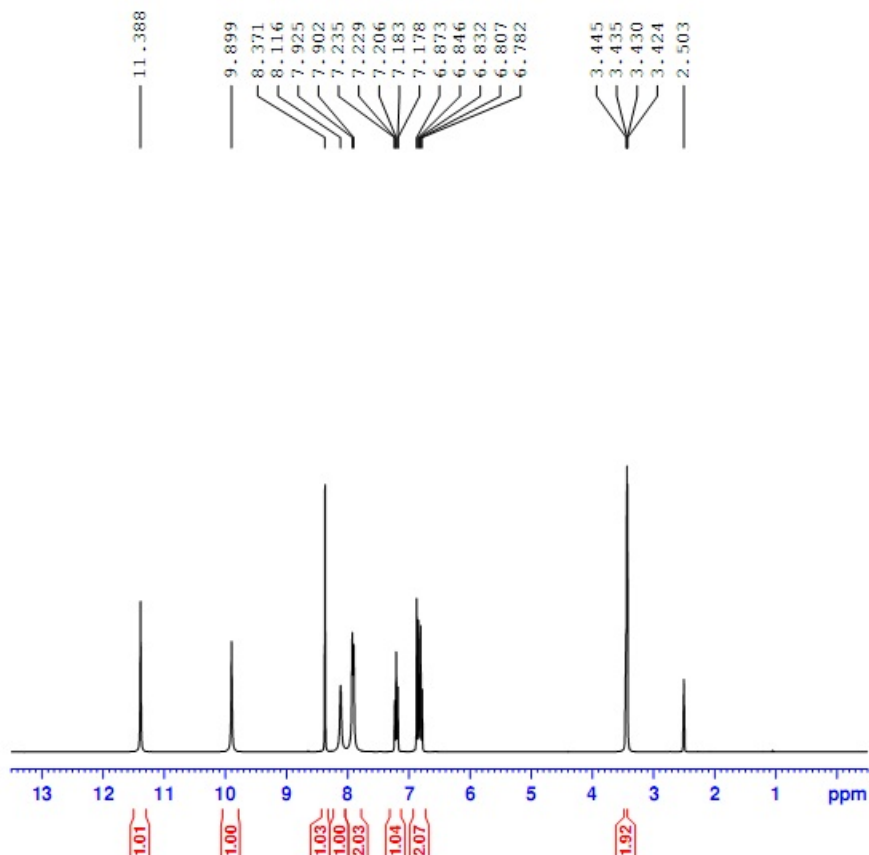


¹H NMR Spectrum of compound **3k**

Spectrum Name: FTIR-OHTS.sp



FT-IR Spectrum of compound **3n**



```

Current Data Parameters
NAME      1299B
EXPNO    1
PROCNO   1

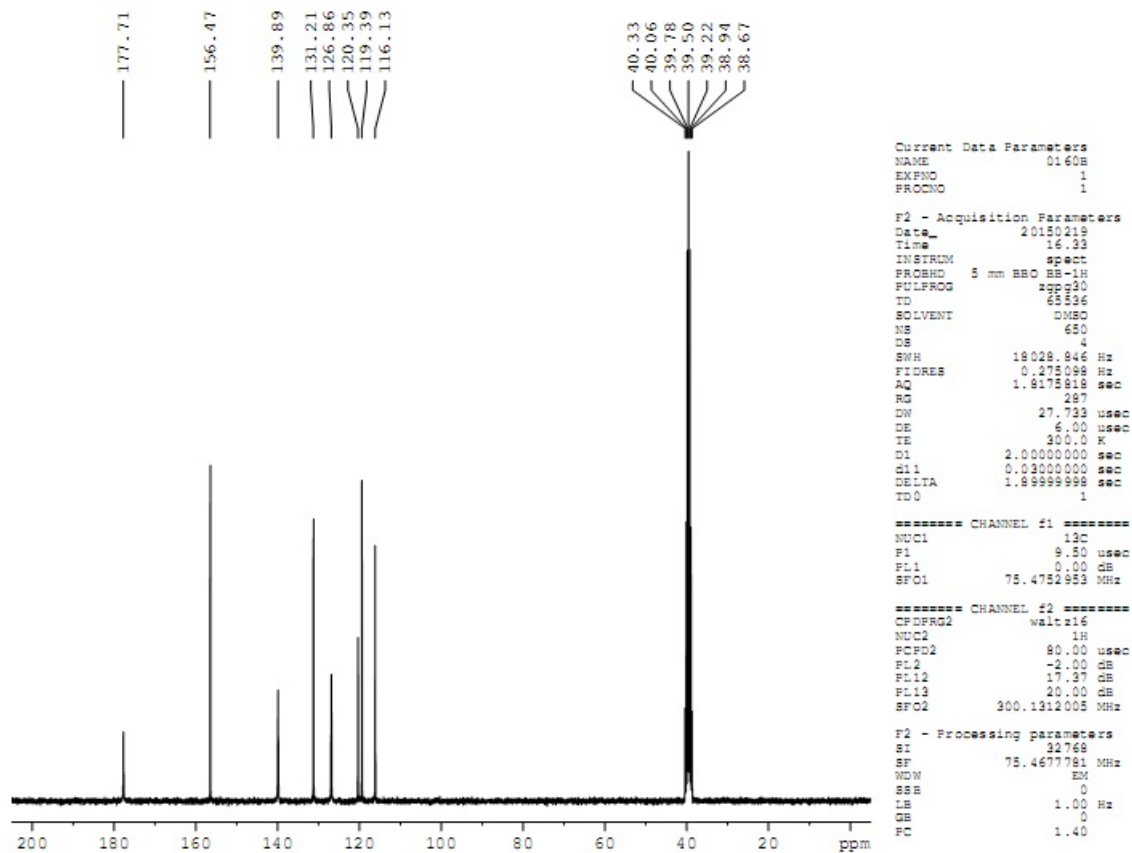
F2 - Acquisition Parameters
Date_    20150103
Time     11.28
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953597 sec
RG       101
DW       80.900 usec
DE       6.00 usec
TE       300.0 K
D1       1.00000000 sec
TD0      1

===== CHANNEL f1 =====
NUC1     1H
P1       9.60 usec
PL1      -2.00 dB
SFO1     300.1318534 MHz

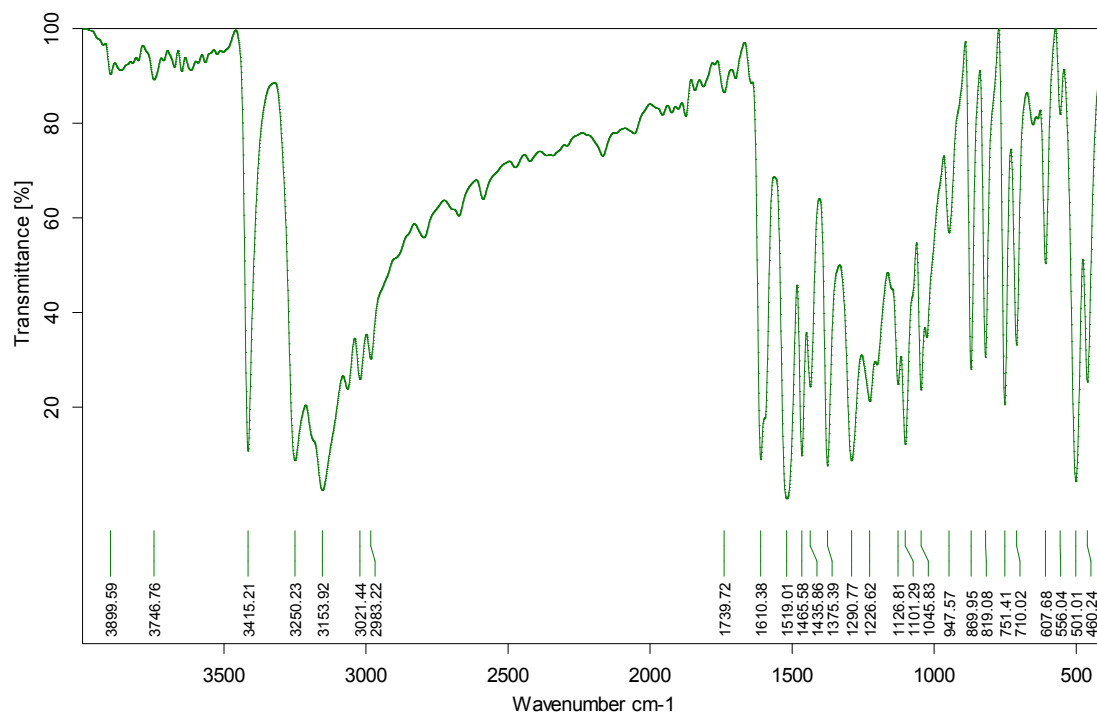
F2 - Processing parameters
SI       32768
SF       300.1300000 MHz
WDW      EM
SSB      0
LB       0.30 Hz
GB       0
PC       1.00

```

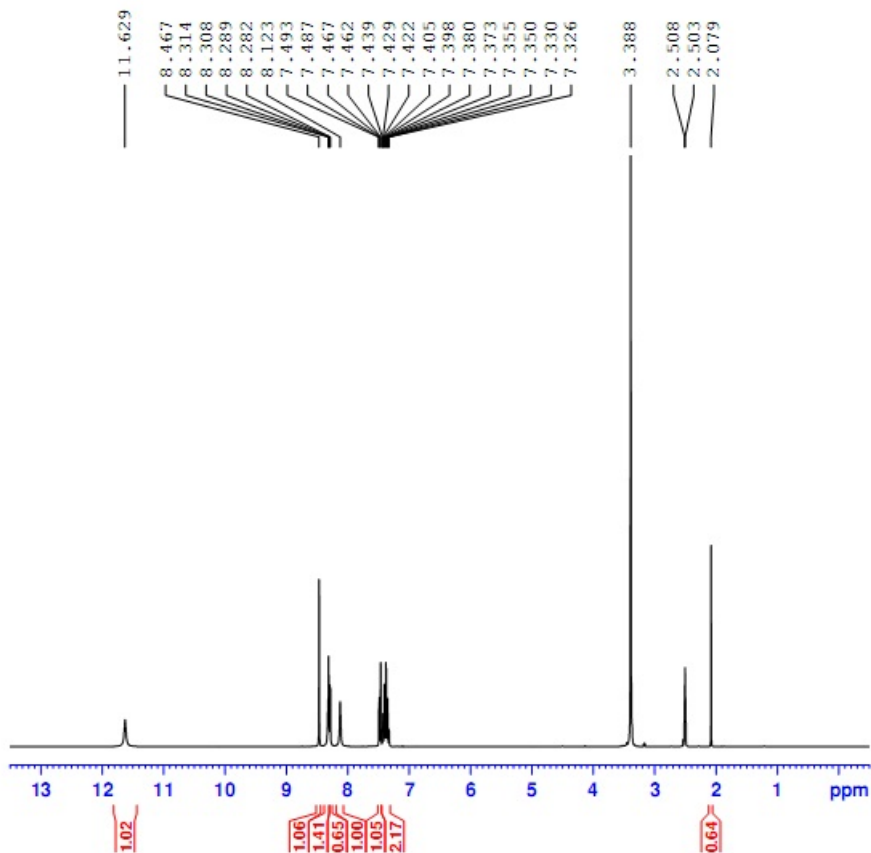
¹H NMR Spectrum of compound **3n**



¹³C NMR Spectrum of compound **3n**



FT-IR Spectrum of compound 3o



```

Current Data Parameters
NAME          1298E
EXPNO         1
PROCNO        1

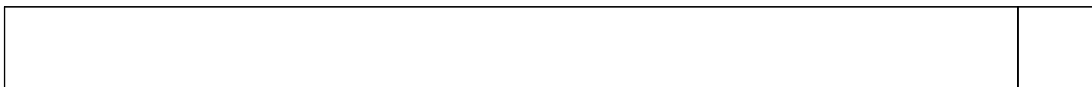
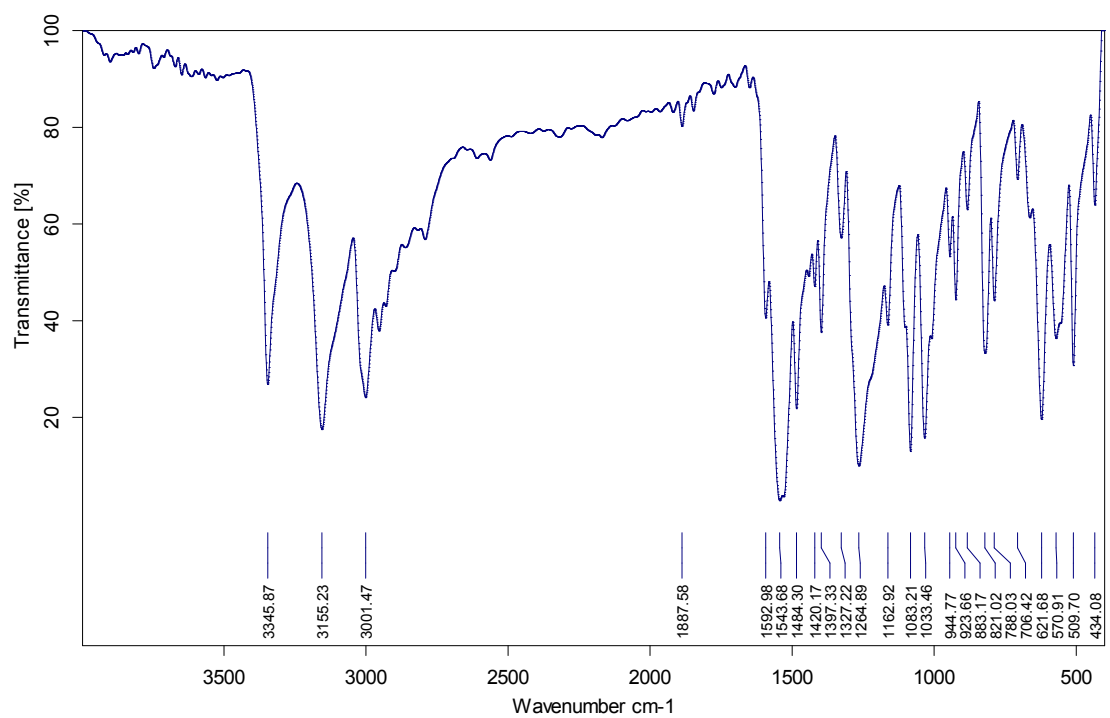
F2 - Acquisition Parameters
Date_         20150103
Time          12.12
INSTRUM       spect
PROBHD        5 mm BBO BB-1H
PULPROG       zg30
TD            65536
SOLVENT       DMSO
NS            16
DS            2
SWH           6188.119 Hz
FIDRES        0.094423 Hz
AQ            5.2953597 sec
RG            144
CW            80.800 usec
DE            6.00 usec
TE            300.0 K
D1            1.00000000 sec
TD0           1

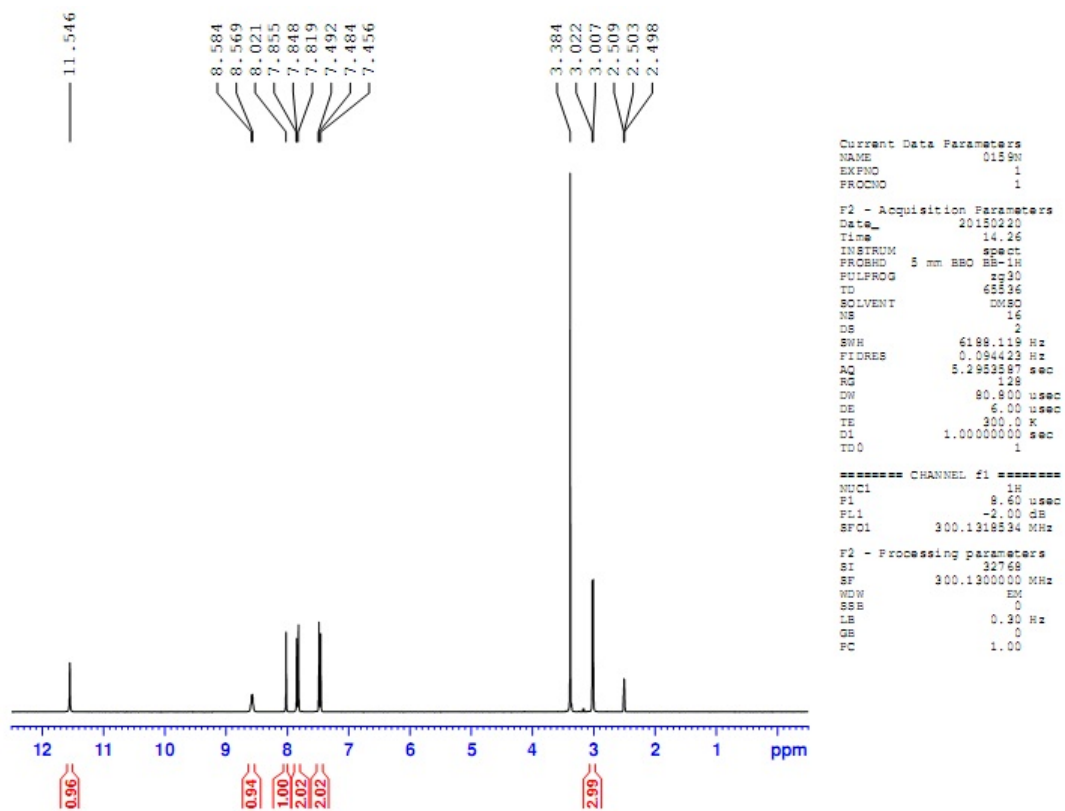
===== CHANNEL f1 =====
NUC1          1H
P1            8.60 usec
PL1           -2.00 dB
SFO1         300.1318534 MHz

F2 - Processing parameters
SI            32768
SF            300.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00

```

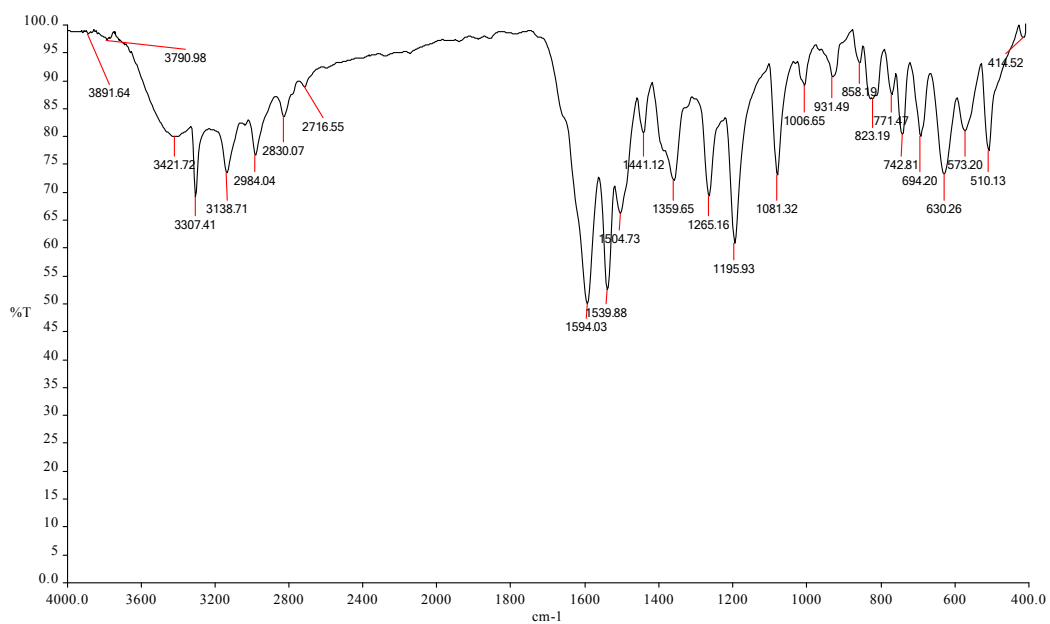
¹H NMR Spectrum of compound **3o**





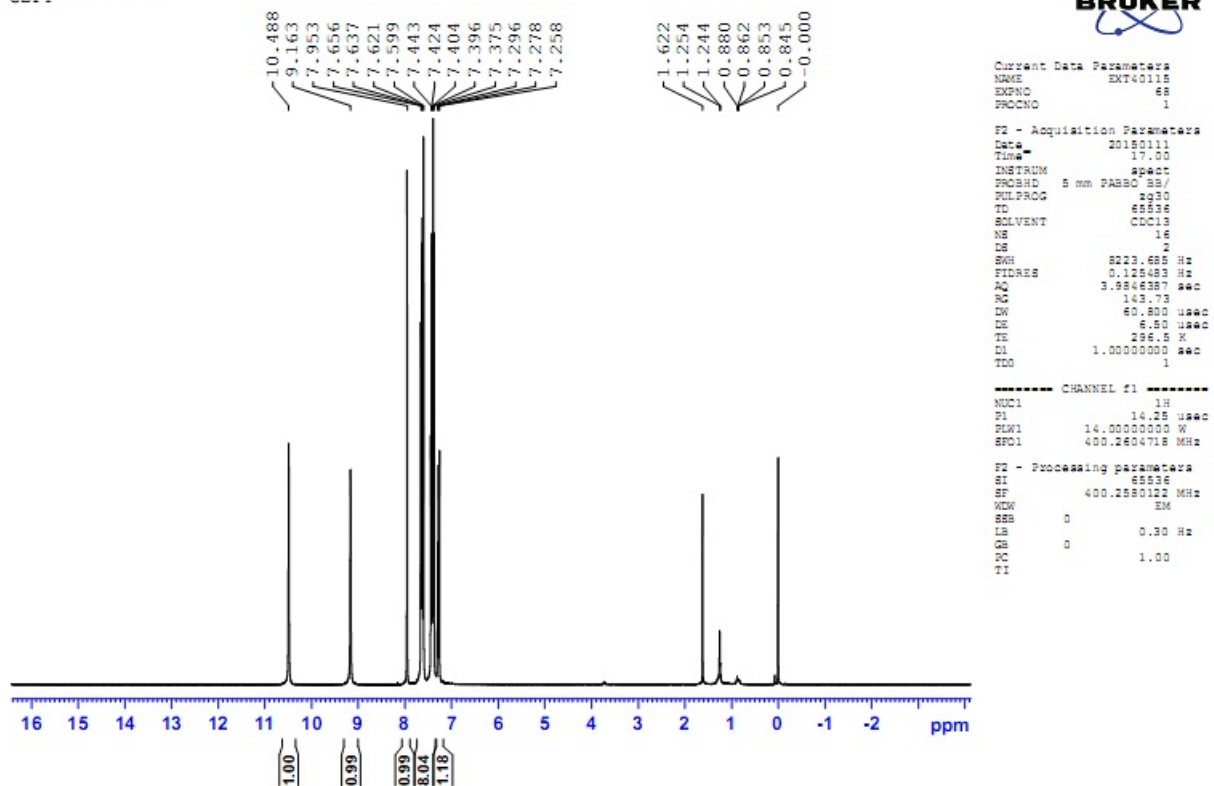
¹H NMR Spectrum of compound **3p**

Spectrum Name: FTIR-CBPT.sp

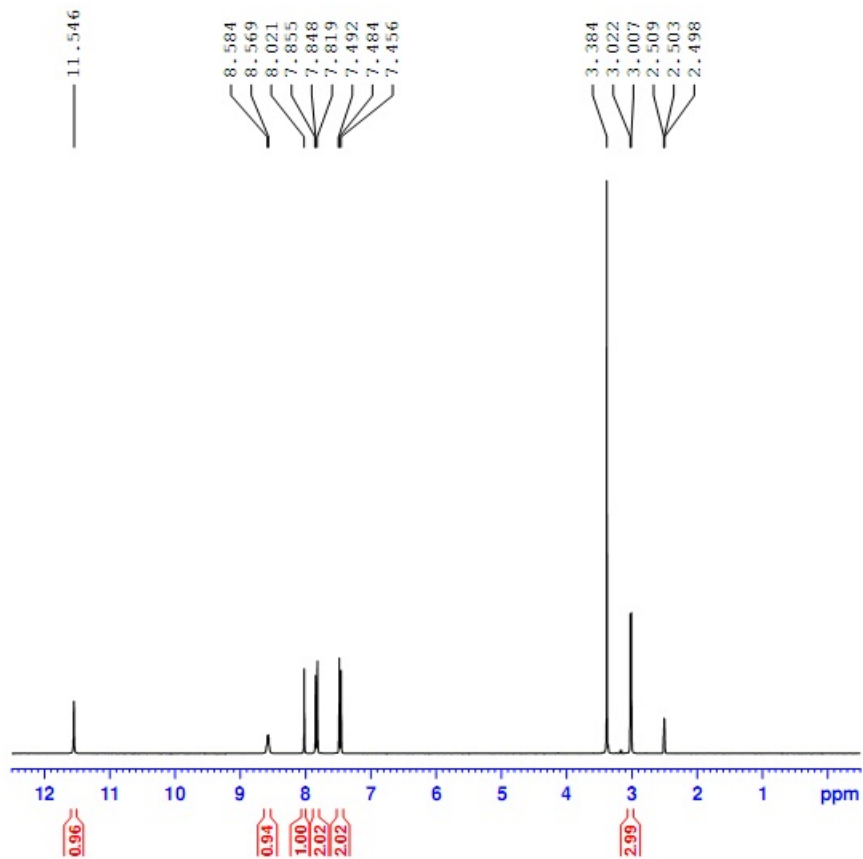


FT-IR Spectrum of compound **3q**

SIF VIT VELLORE
CBPT



¹H NMR Spectrum of compound **3q**



```

Current Data Parameters
NAME      D159N
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20150220
Time     14.26
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zg30
TD       65536
SOLVENT  DMSO
NS       16
DS       2
SWH      6188.119 Hz
FIDRES   0.094423 Hz
AQ       5.2953587 sec
RG       128
DW       80.800 usec
DE       6.00 usec
TE       300.0 K
D1       1.00000000 sec
TD0      1

```

```

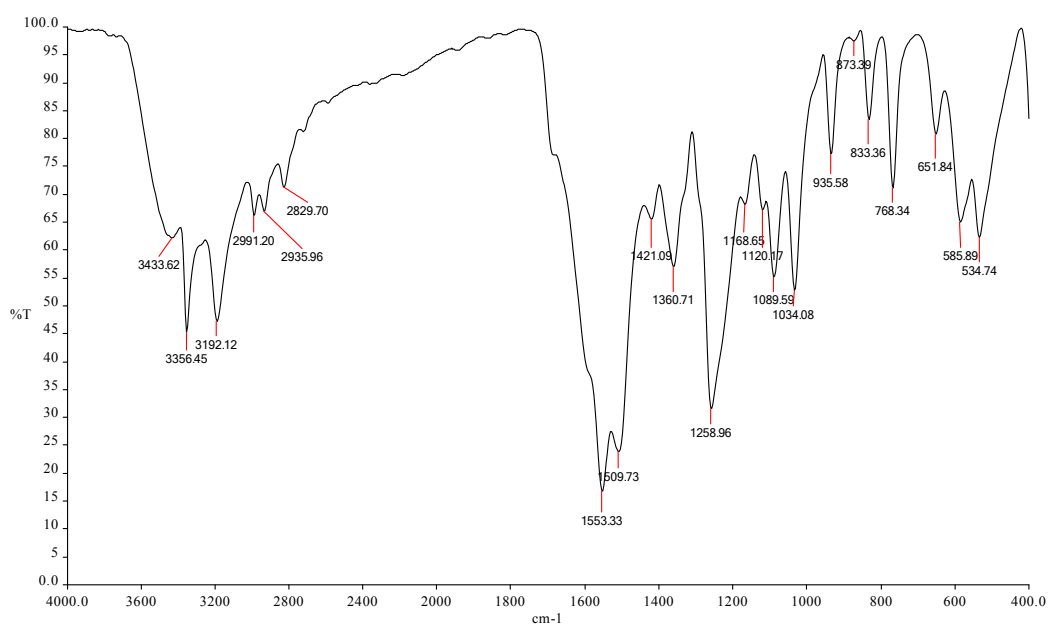
===== CHANNEL f1 =====
NUC1     1H
P1       8.60 usec
P11      -2.00 dB
SFO1     300.1318534 MHz

F2 - Processing parameters
SI       32768
SF       300.1300000 MHz
WDW      EM
SSB      0
LB       0.20 Hz
GB       0
PC       1.00

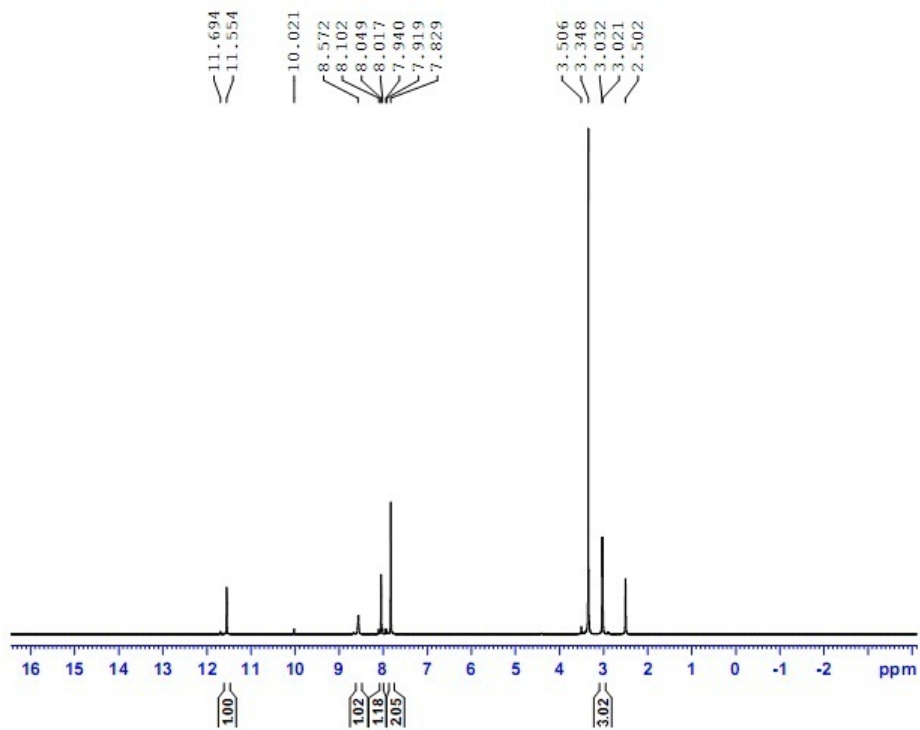
```

¹H NMR Spectrum of compound **3p**

Spectrum Name: FTIR-METS.sp



FT-IR Spectrum of compound **5b**



Current Data Parameters
NAME EXT41214
EXPNO 84
PROCNO 1

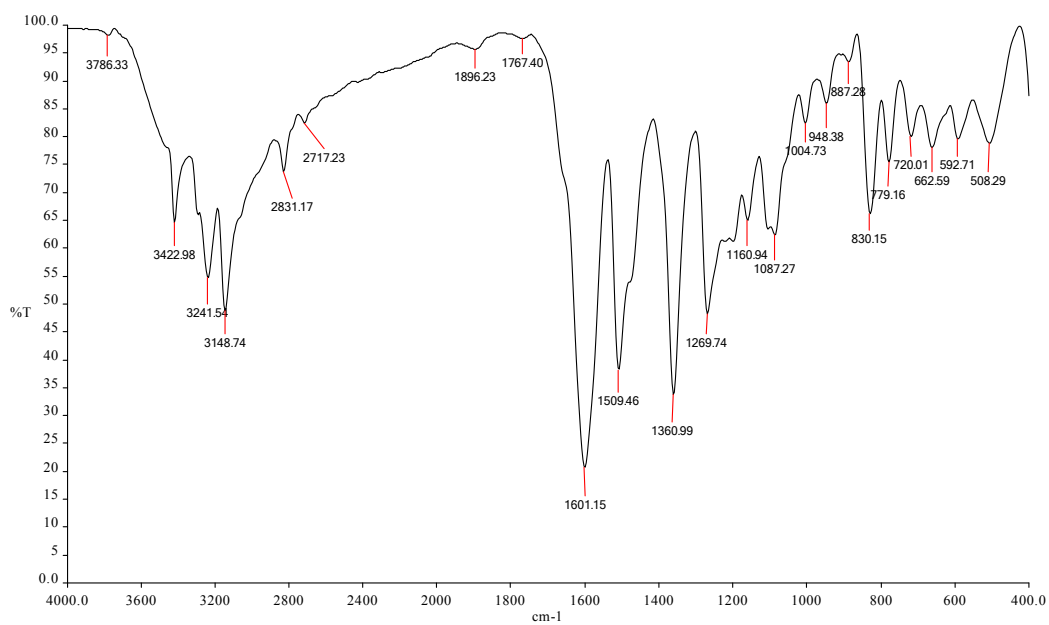
F2 - Acquisition Parameters
Date_ 20141214
Time_ 12.18
INSTRUM spect
PROBHD 5 mm DABBO BB/
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.112483 Hz
AQ 3.8846287 sec
RG 127.79
DW 60.800 usec
DE 6.50 usec
TE 297.5 K
D1 1.00000000 sec
TDO 1

----- CHANNEL f1 -----
NUC1 1H
P1 14.25 usec
PLW1 14.00000000 W
SFO1 400.2604718 MHz

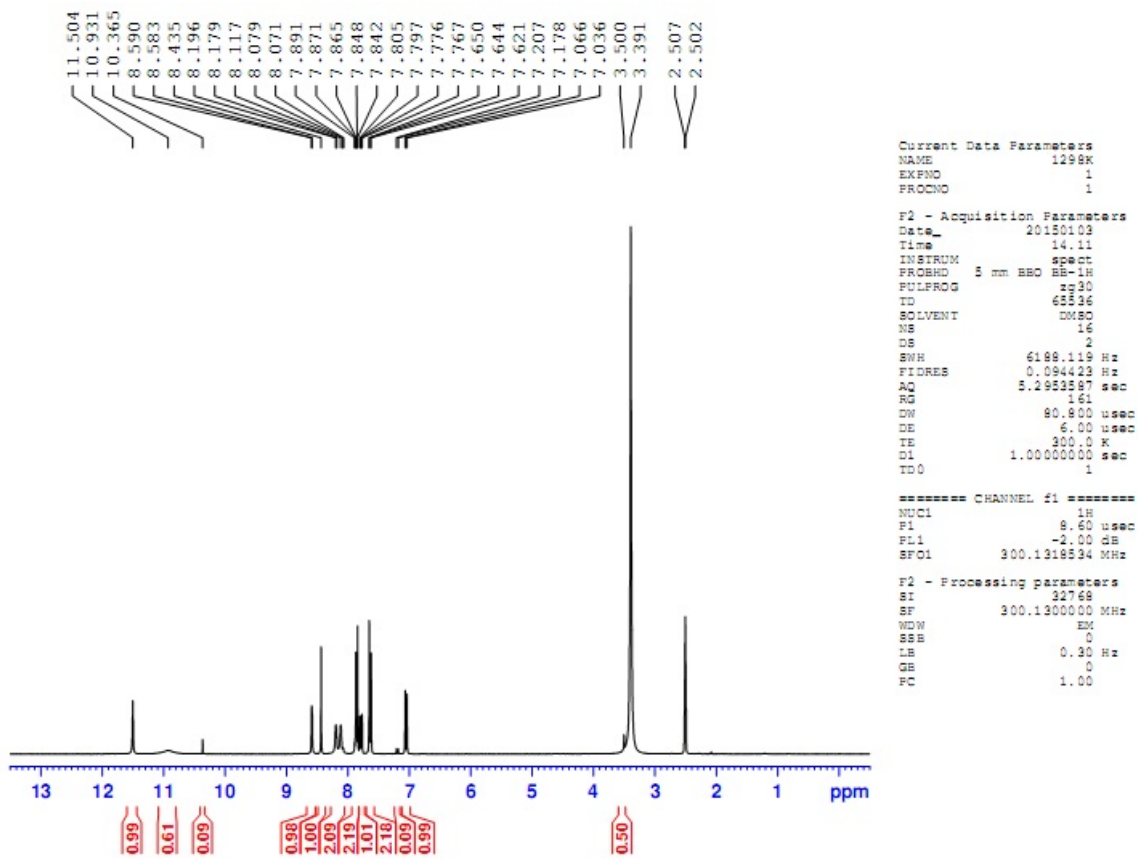
F2 - Processing parameters
SI 65536
SF 400.2580048 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TI

¹H NMR Spectrum of compound **5b**

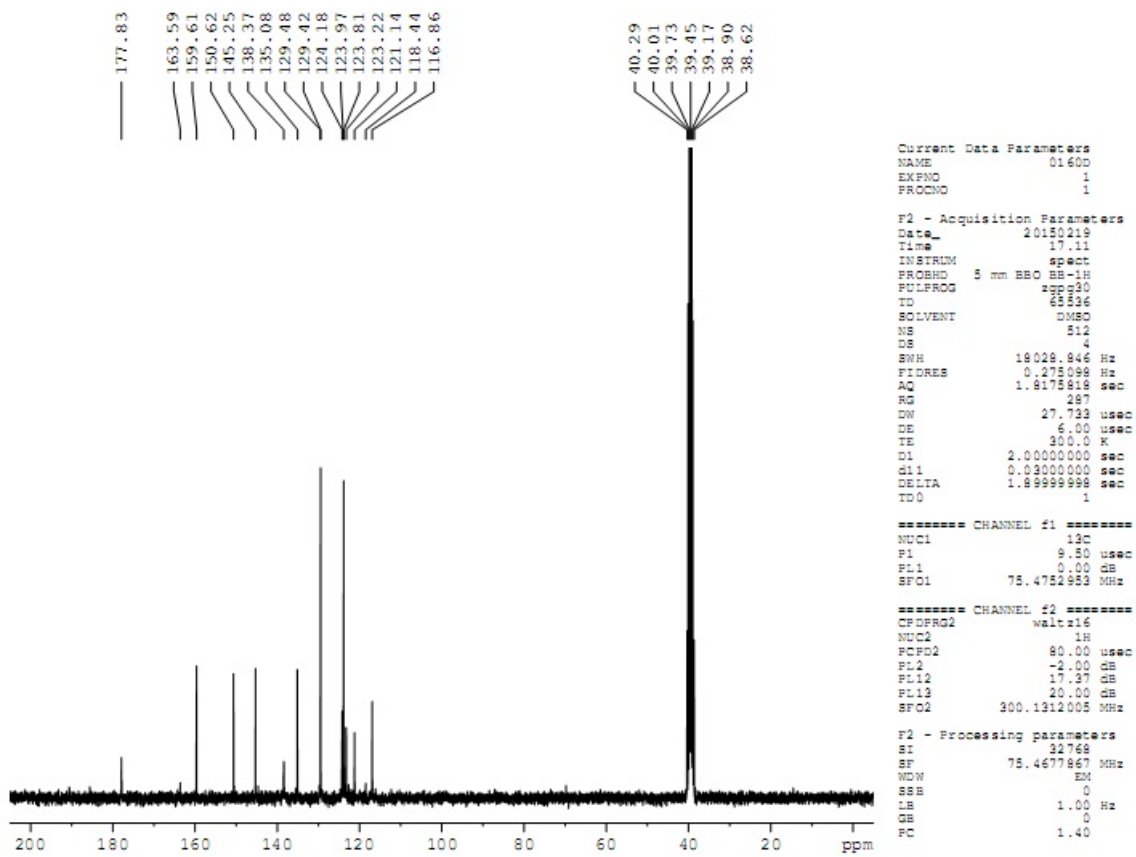
Spectrum Name: FTIR-AZT.sp



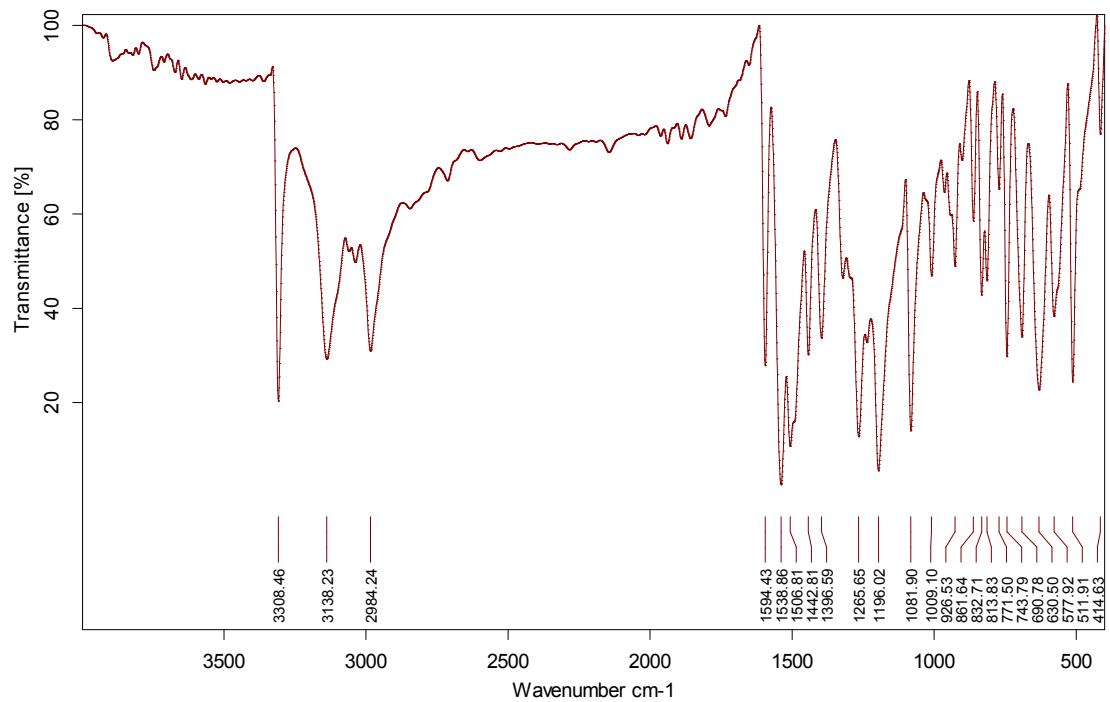
FT-IR Spectrum of compound **7a**



¹H NMR Spectrum of compound **7a**

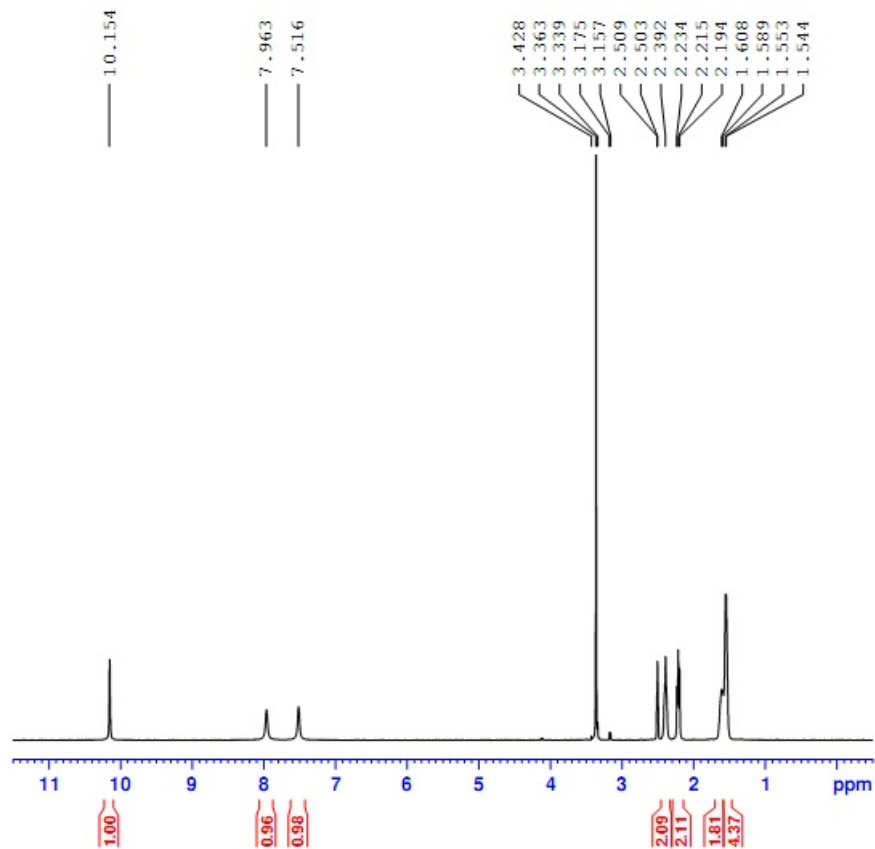


¹³C NMR Spectrum of compound **7a**



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FT-IR Spectrum of compound 9a



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Current Data Parameters
NAME          01890
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20150220
Time         13.50
INSTRUM      spect
PROBHD       5 mm BBO BB-1H
PULPROG      zg30
TD           65536
SOLVENT      DMSO
NS           16
DS           2
SWH          6198.119 Hz
FIDRES       0.094423 Hz
AQ           5.2953597 sec
RG           80.6
DN           80.800 usec
DE           6.00 usec
TE           300.0 K
D1           1.00000000 sec
TD0          1

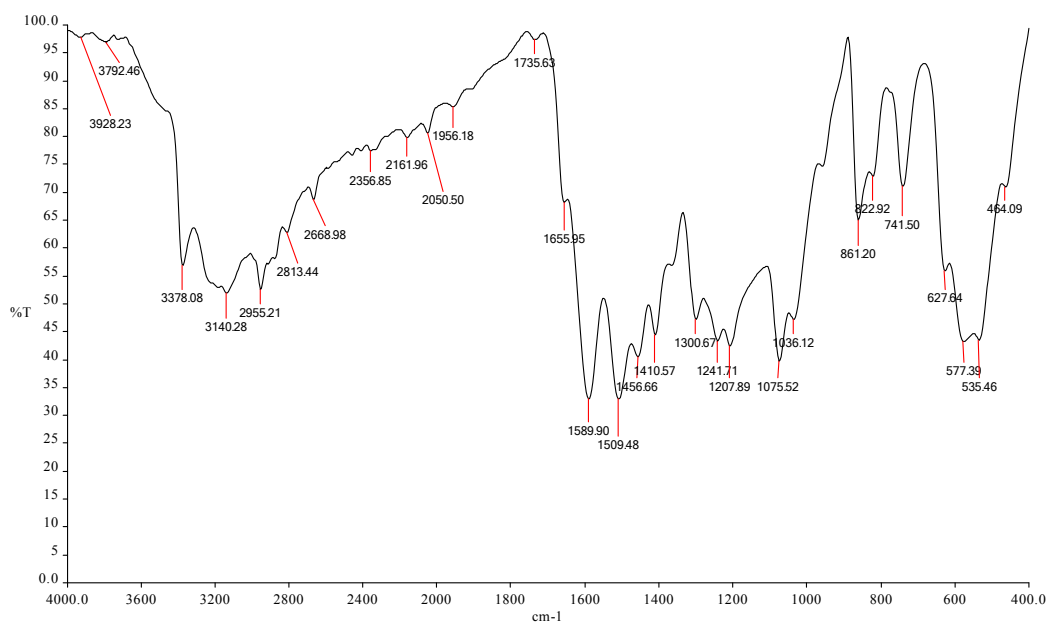
===== CHANNEL f1 =====
NUC1         1H
P1           9.60 usec
PL1         -2.00 dB
SFO1        300.1319534 MHz

F2 - Processing parameters
SI           32768
SF           300.1300000 MHz
WDW          EM
SSB          0
LB           0.10 Hz
GB           0
PC           1.00

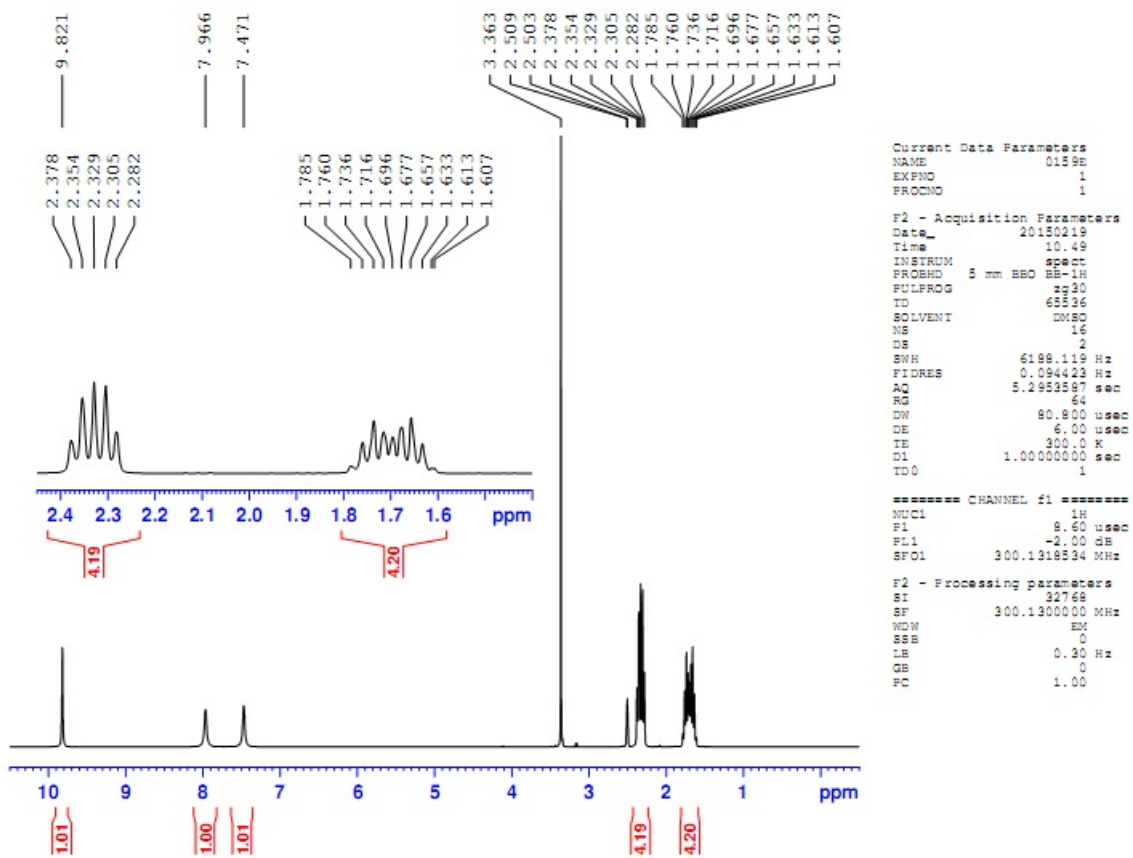
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¹H NMR Spectrum of compound **9a**

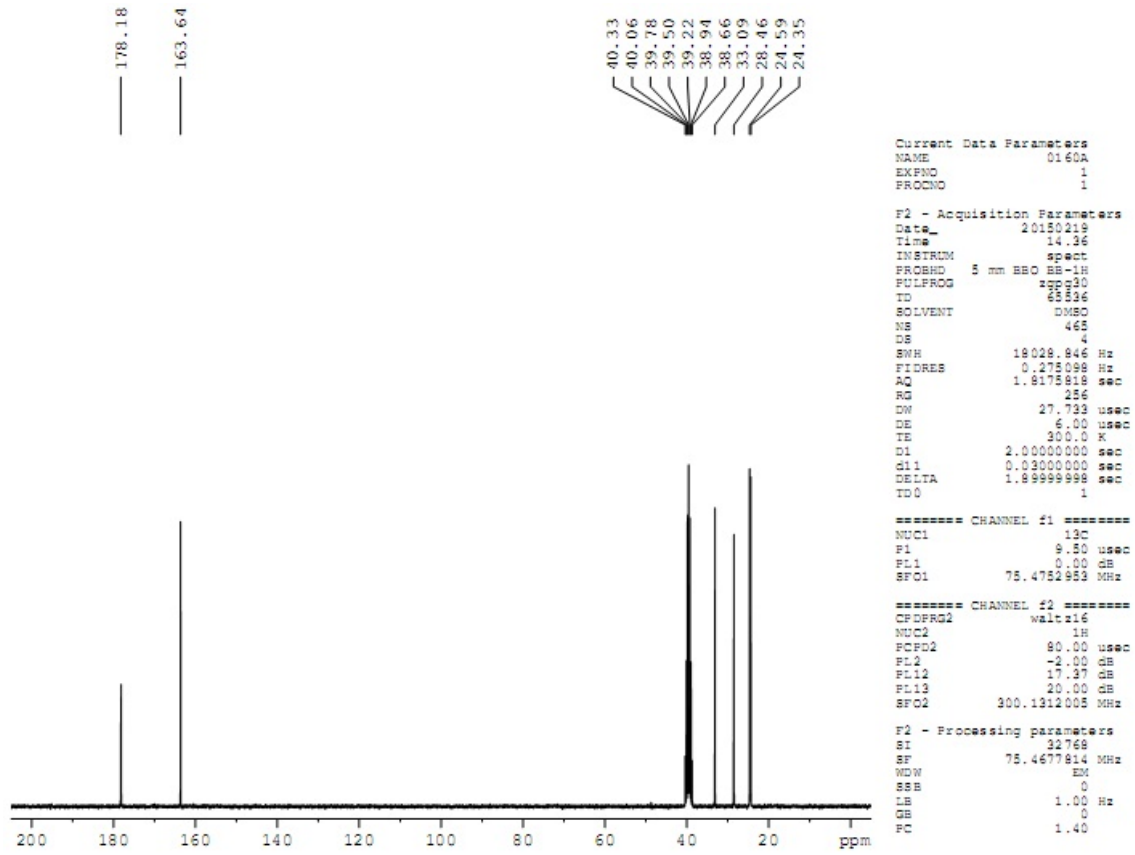
Spectrum Name: FTIR-CPTS.sp



FT-IR Spectrum of compound **9b**



¹H NMR Spectrum of compound 9b



¹³C NMR Spectrum of compound **9b**