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Journal Name

ARTICLE

Electronic Supplementary Material (ESI) for RSC Advances.

DABCO-Based ionic liquids: green and recyclable catalysts for the synthesis of barbituric and thiobarbituric acid derivatives in aqueous media

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Supporting information

Catalogue

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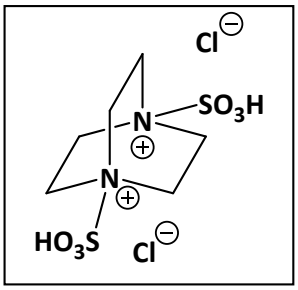
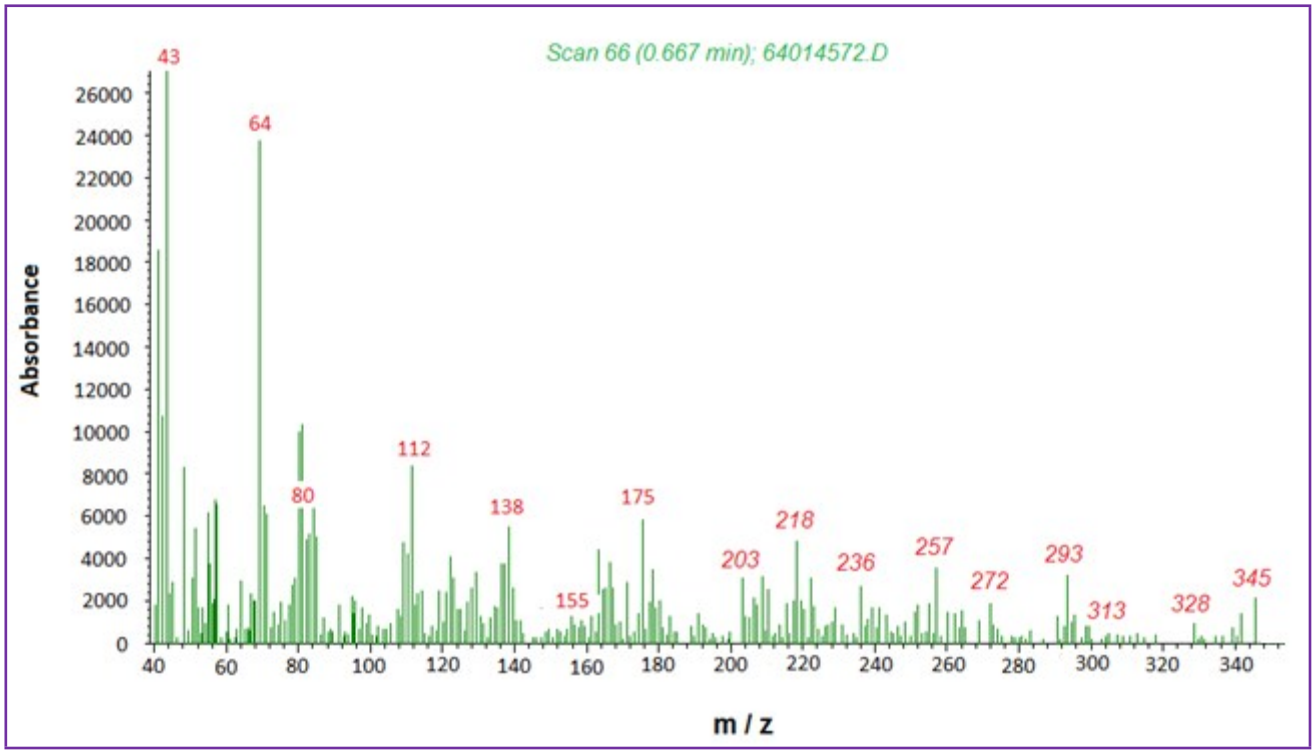
[DABCO](SO$_3$H)$_2$Cl$_2$:



M.p. 75 °C; MS: *m/z* = 345 (M^+); H_0 = 1.37; IR (KBr, cm^{-1}) υ_{max}: 3500-2800 (broad), 1179, 881, 850; ^{1}HNMR (400MHz, DMSO-d_6): δ (ppm) 3.58 (s, 6H), 7.72 (s, 1H); ^{13}CNMR (100MHz, DMSO-d_6): δ (ppm) 43.2.

Mass spectra



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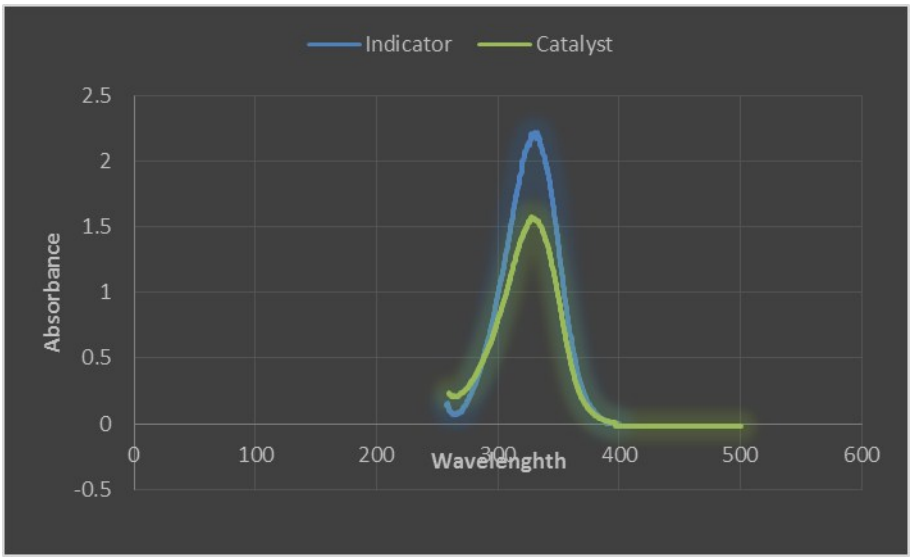
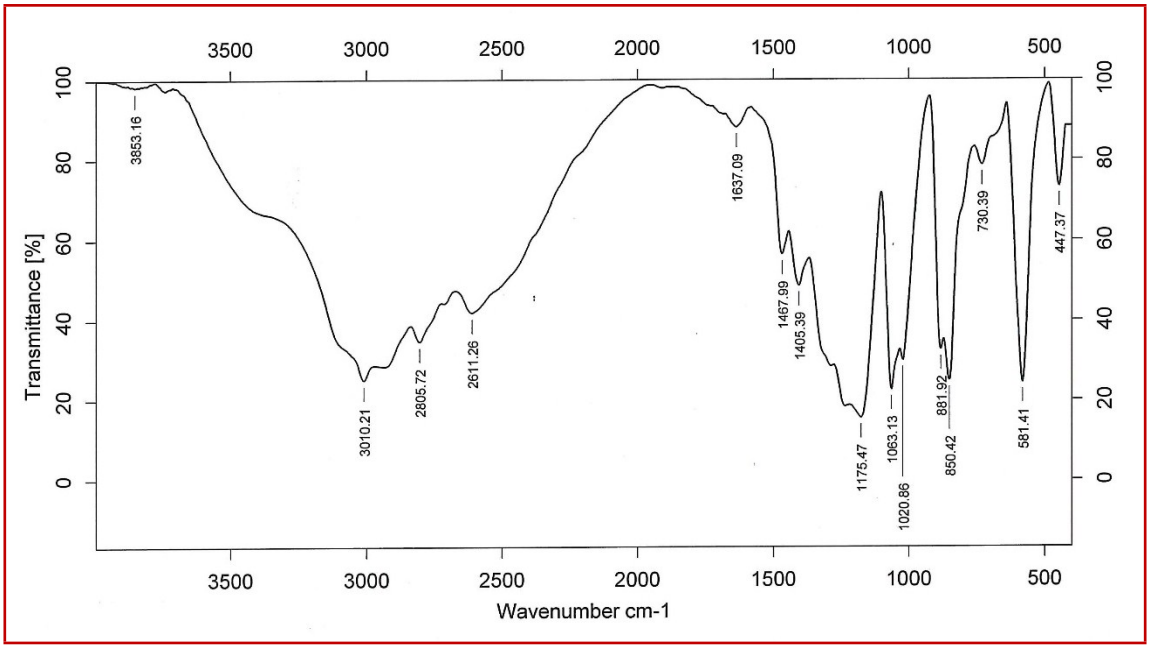
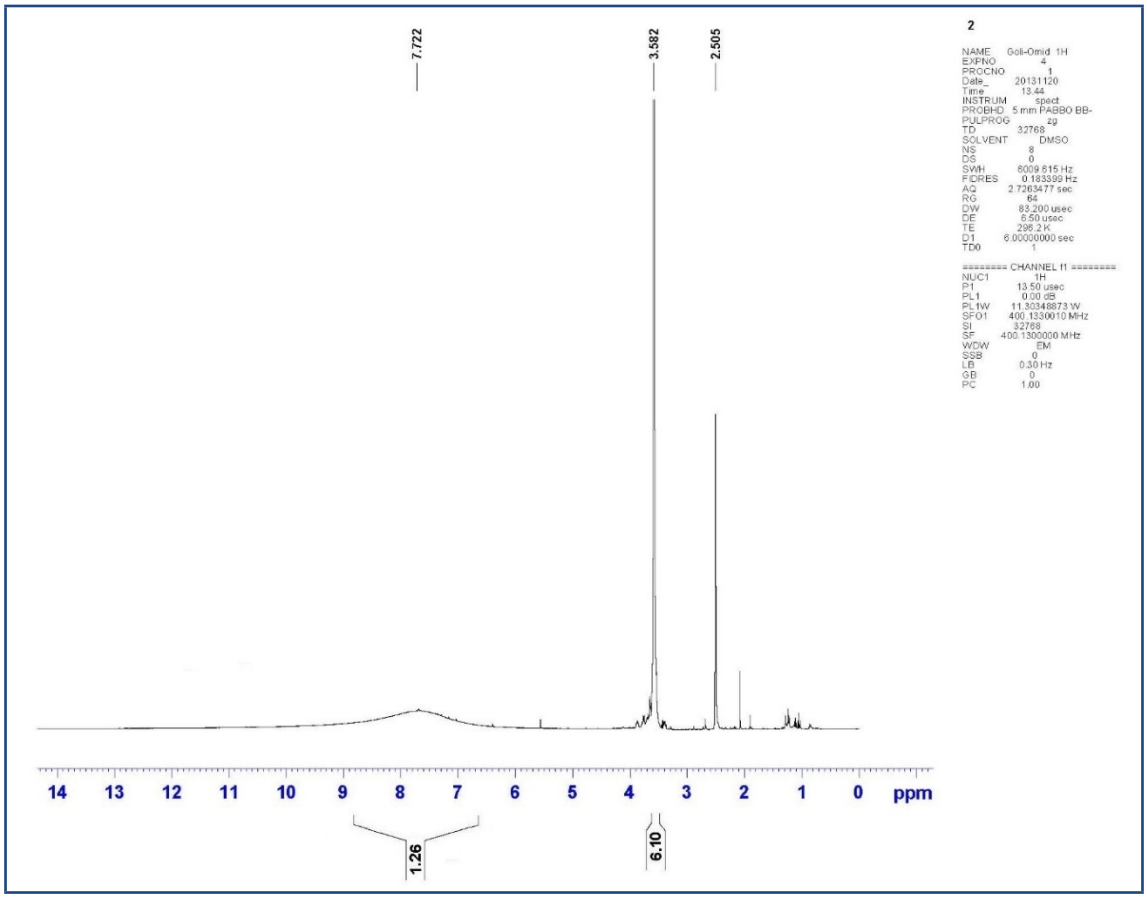
Hammett acidity



FTIR



^{1}HNMR



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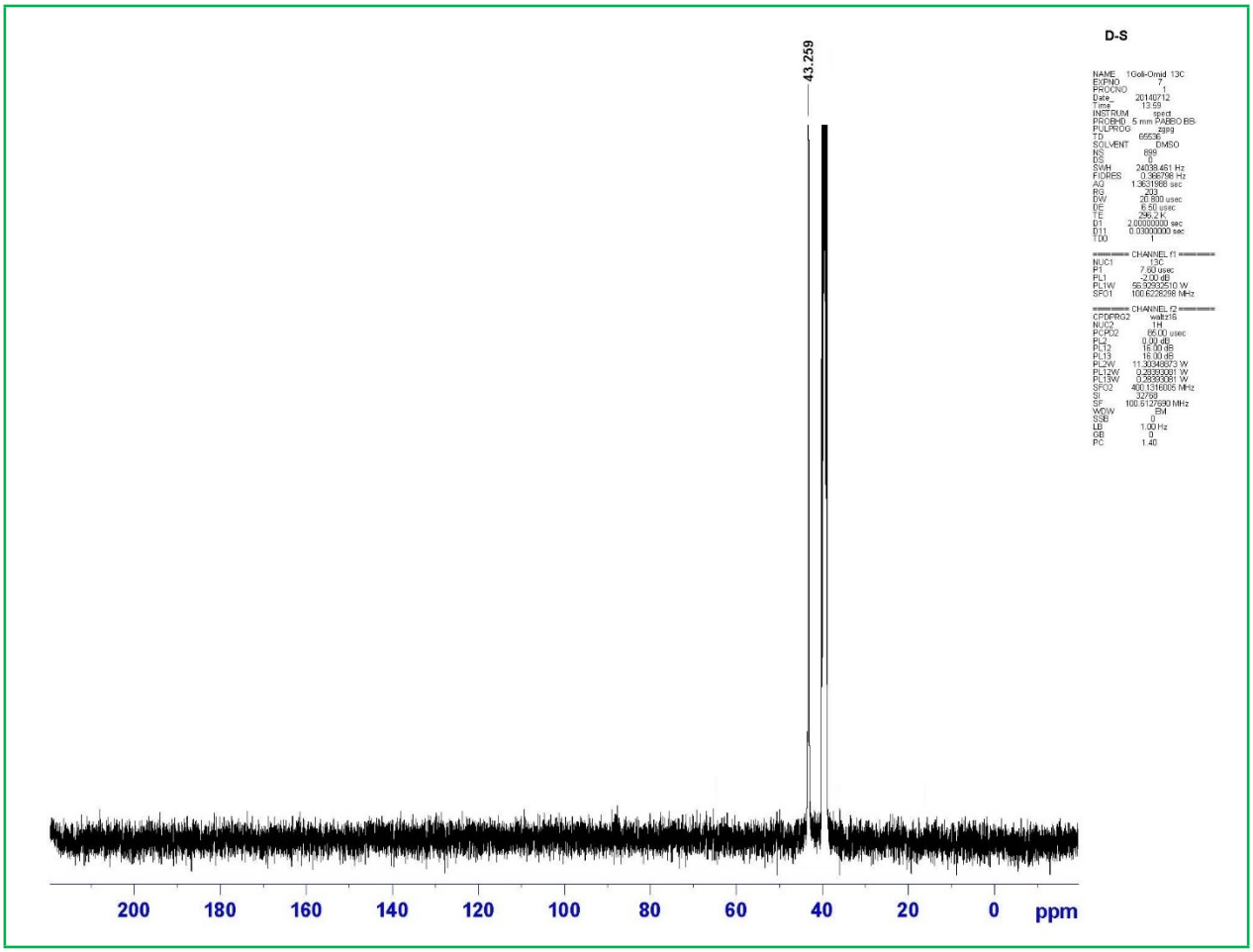
^{13}CNMR



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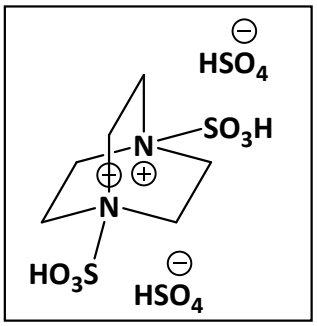
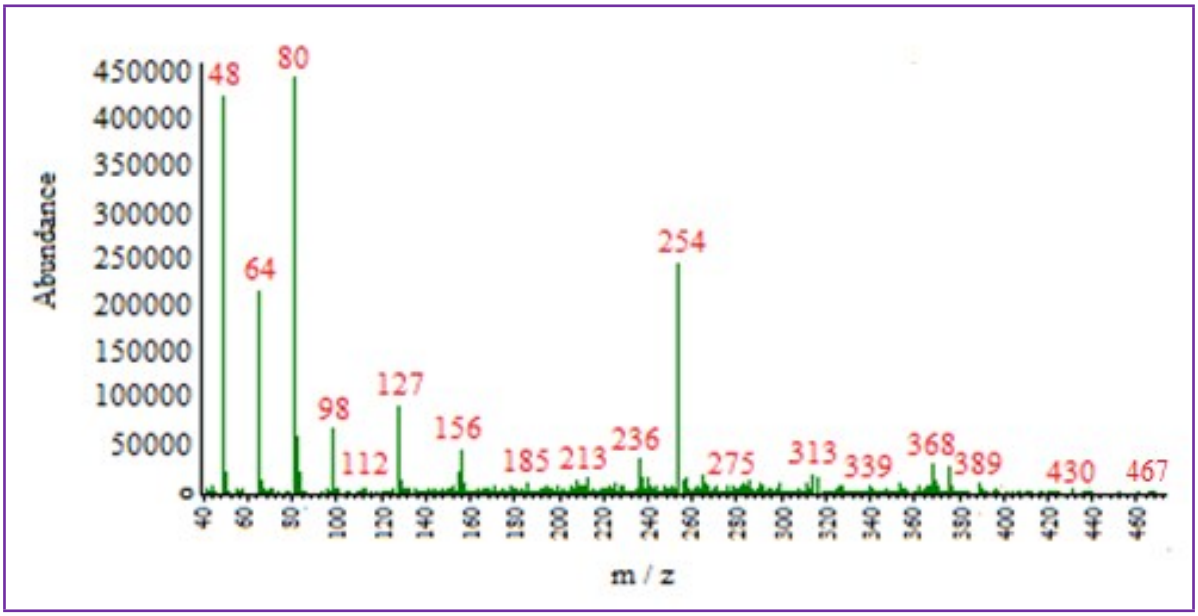
$[DABCO](SO_3H)_2(HSO_4)_2$:



M.p. 70 °C; MS: m/z = 467(M^+); H_0 = 0.43; FT-IR (KBr, cm^{-1}) υ_{max}: 3405, 3010, 2922, 1405, 1289, 1173, 1062, 1006, 855; ^{1}HNMR (400 MHz, DMSO-d_6): δ (ppm) 3.59 (s, 6H), 6.97 (s, 1H, SO_3H), 14.14 (s, 1H, HSO_4); ^{13}CNMR (100 MHz, DMSO-d_6): δ (ppm) 43.2.

Mass spectra



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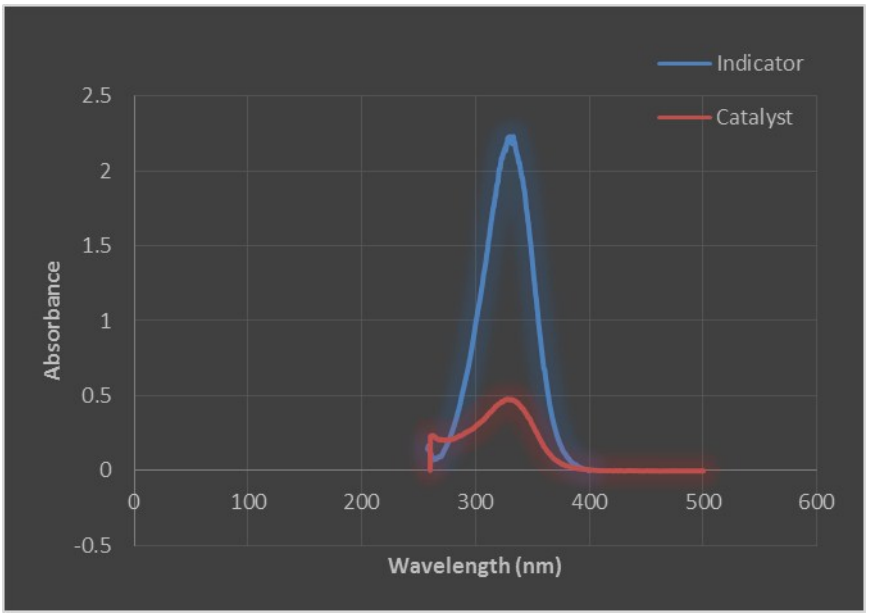
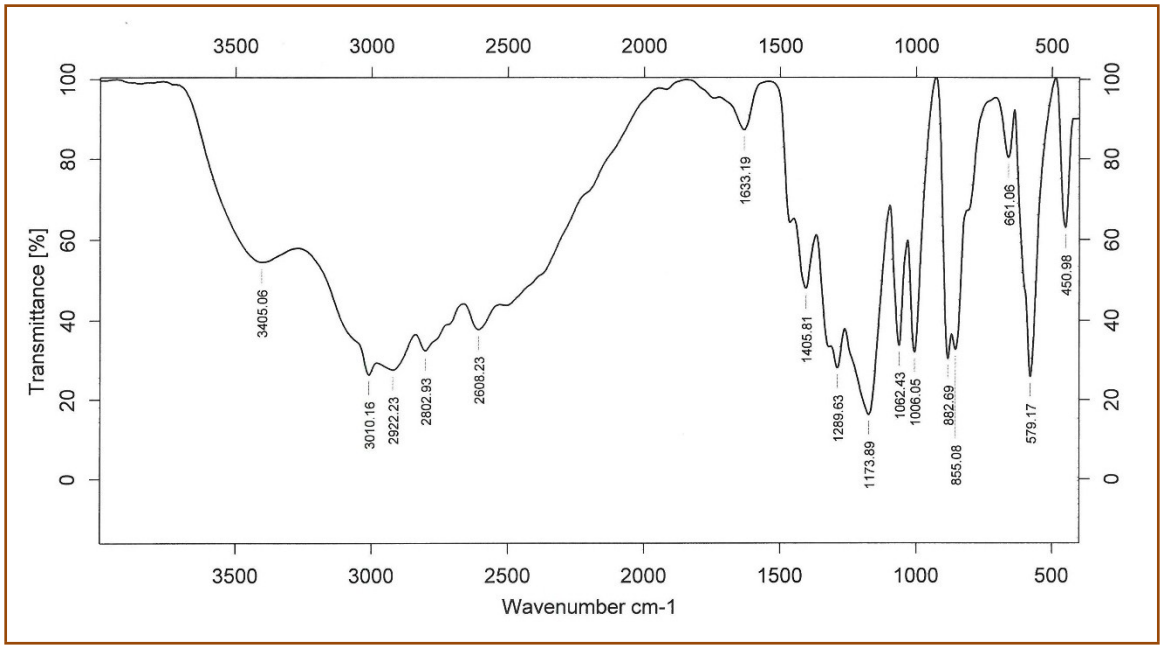
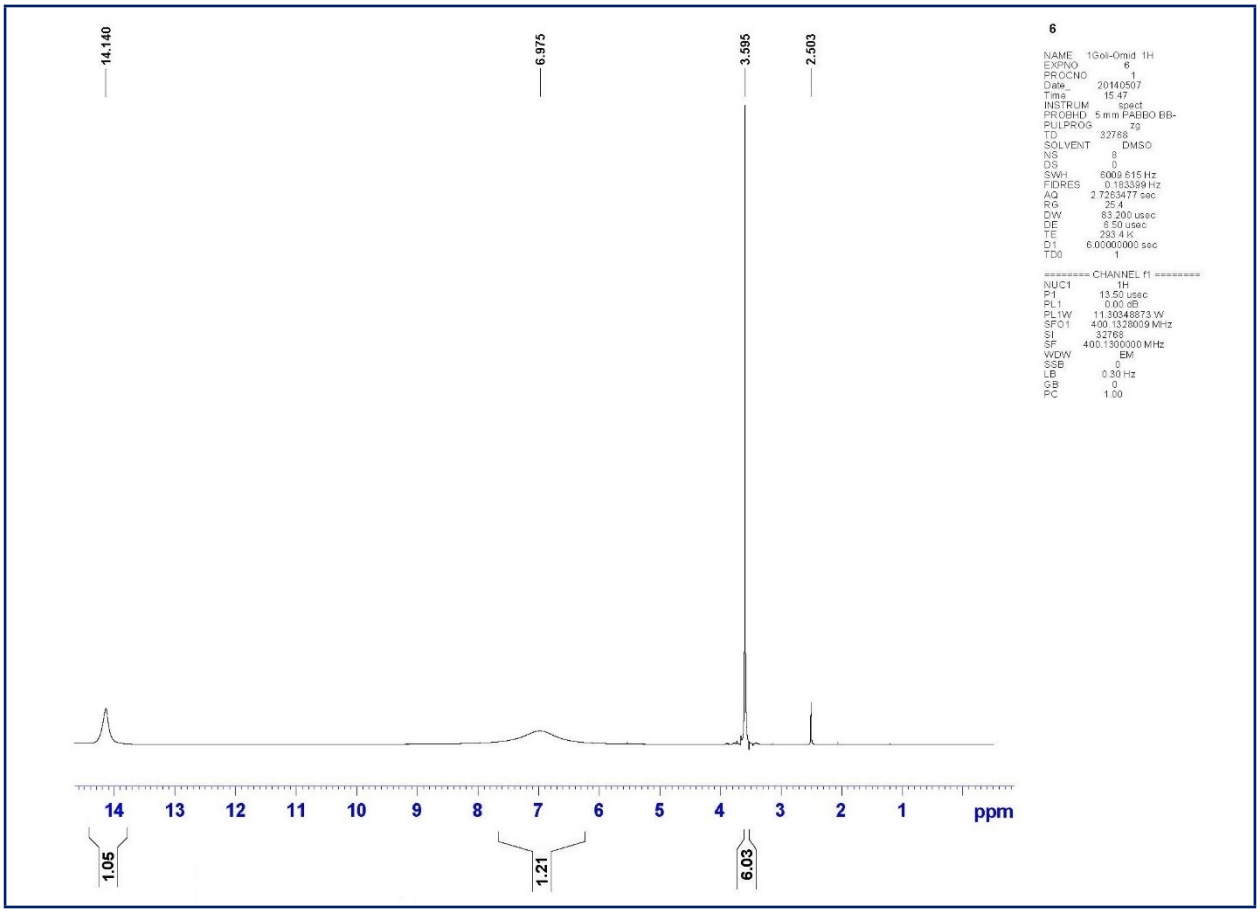
Hammet acidity



FTIR



^{1}HNMR



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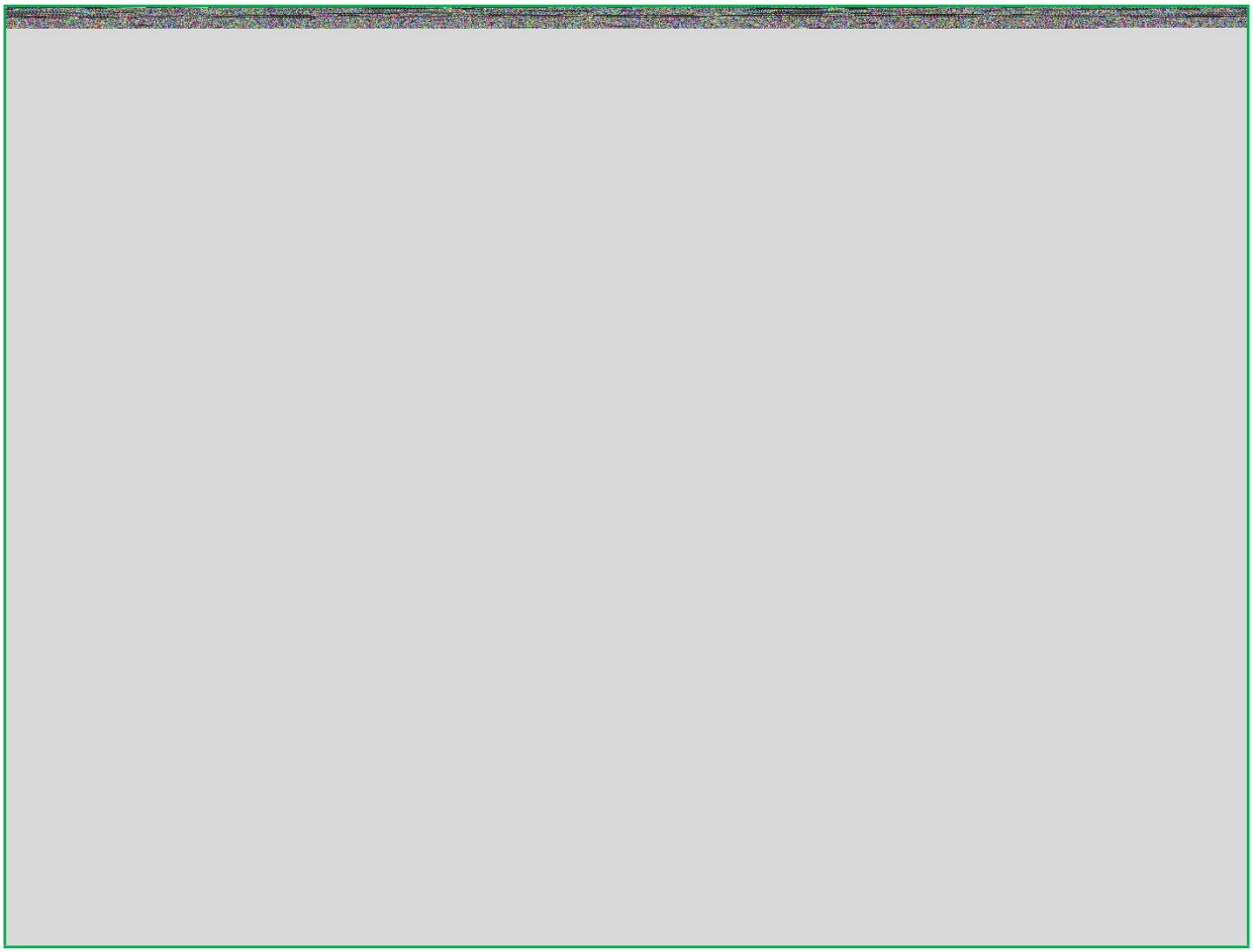
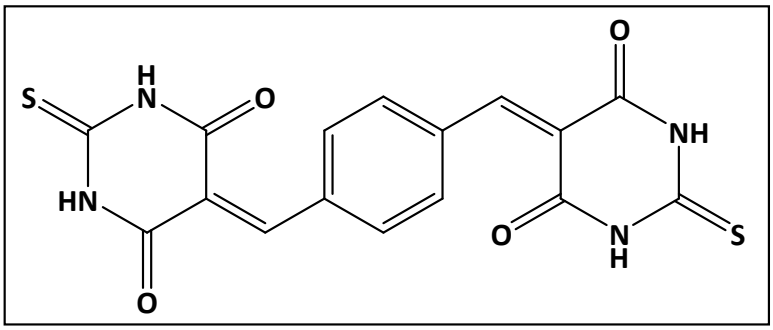
^{13}CNMR

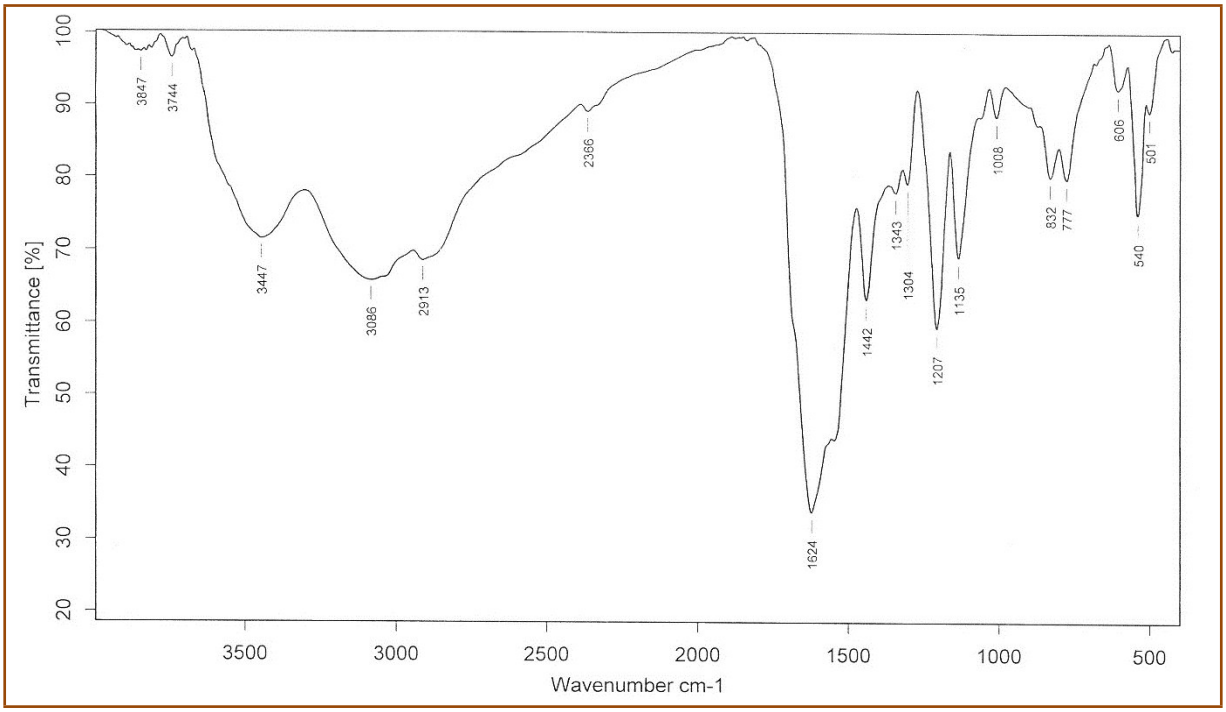


5,5'-(1,4-Phenylenebis(methanylylidene))bis(2-thioxodihydropyrimidine-4,6(1*H*,5*H*)-dione) (4x)



M.p. >300 ^{o}C; IR (KBr) ν_{max}/cm^{-1}: 3447, 3065, 2913, 1624, 1442; ^{1}HNMR (400 MHz, DMSO-d_6): δ (ppm) 8.05 (s, 4H), 8.29 (s, 2H, HC = C), 11.30 (NH, s, 2H), 11.45 (NH, s, 2H); ^{13}CNMR (100 MHz, DMSO-d_6): δ (ppm)120.9, 129.0, 132.3, 136.1, 150.6, 153.4, 161.9, 163.6.

FTIR



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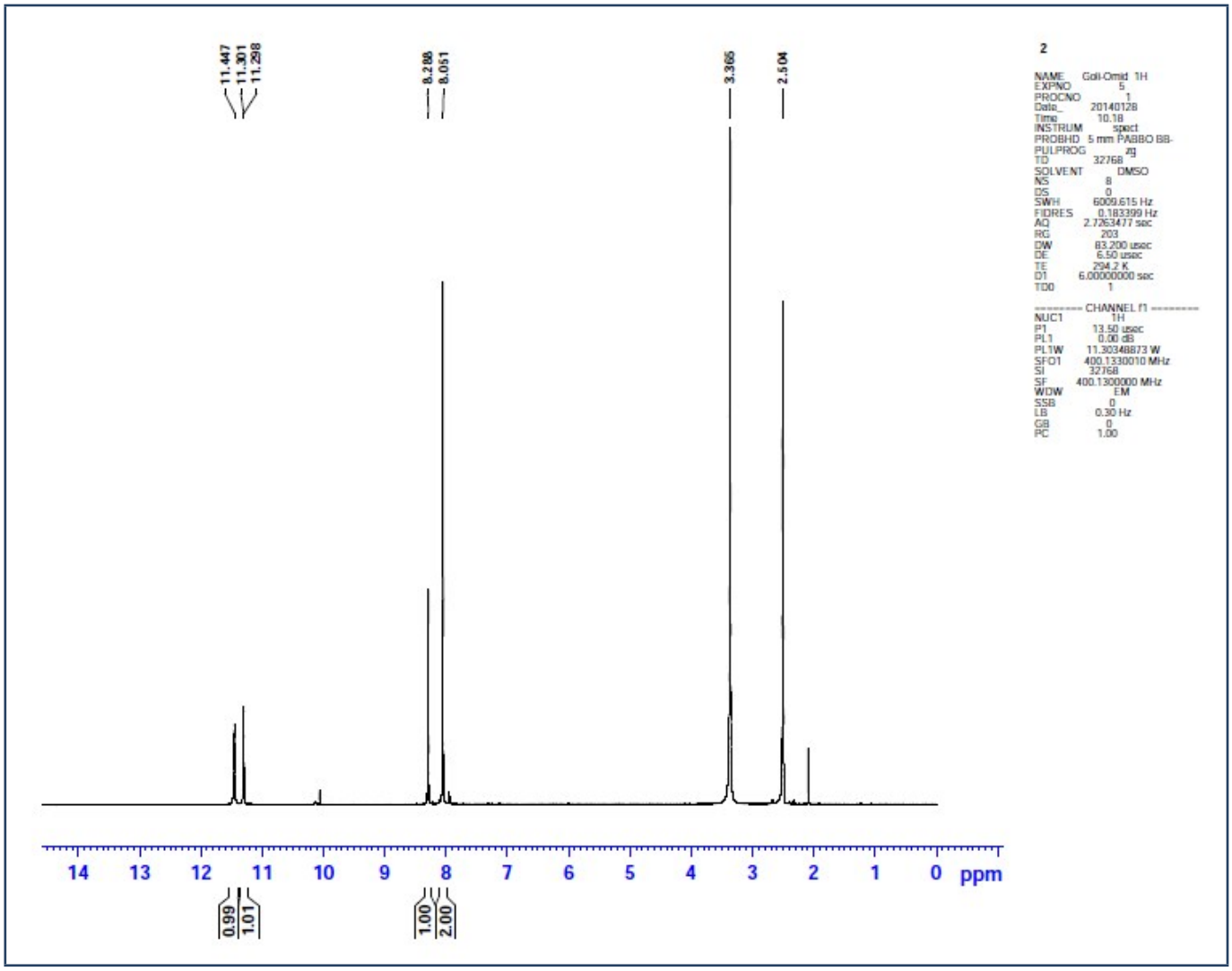
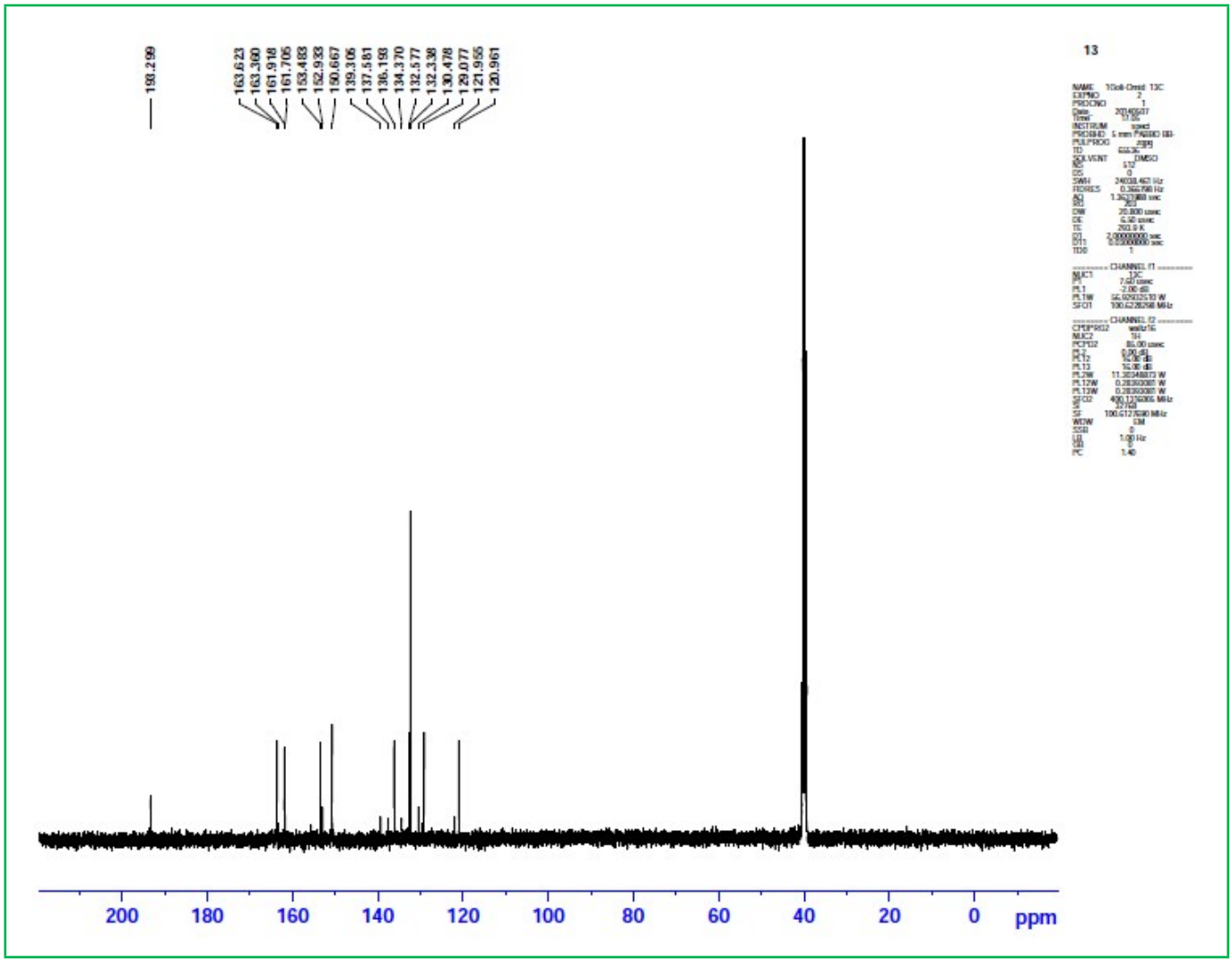
^{1}HNMR


11.447
11.301
11.298
8.288
8.051
3.365
2.504
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.99
1.01
1.00
2.00
2
NAME Goli-Omid 1H
EXPNO 5
PROCNO 1
Date_ 20140128
Time 10.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT DMSO
NS 8
DS 0
SWH 6009.615 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 203
DW 83.200 usec
DE 6.50 usec
TE 294.2 K
D1 6.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 13.50 usec
PL1 0.00 dB
PL1W 11.30348873 W
SFO1 400.1330010 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

^{13}CNMR



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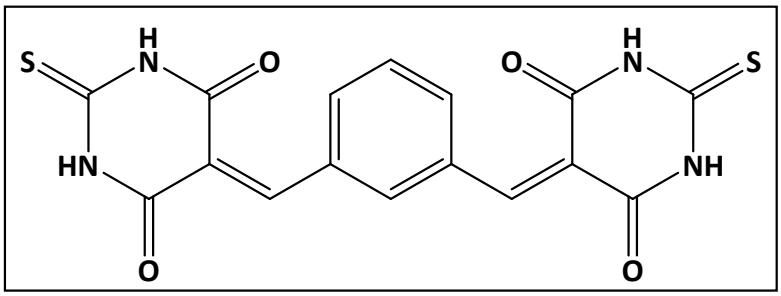
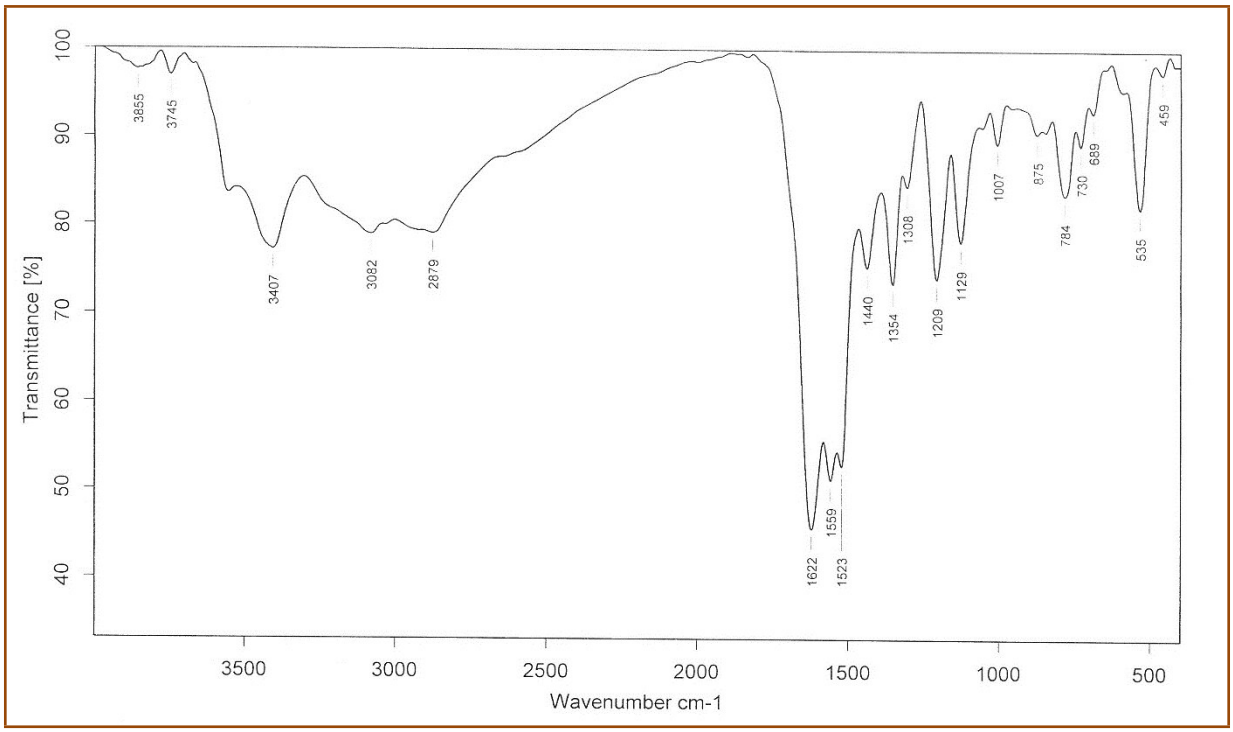
5,5'-(1,3-phenylenebis(methanylylidene))bis(2-thioxodihydropyrimidine-4,6(1*H*,5*H*)-dione) (4y)



M.p. >300 ^{o}C; IR (KBr) ν_{max}/cm^{-1}: 3407, 3062, 2879, 1622, 1559, 1523, 1442; ^{1}HNMR (400 MHz, DMSO-d_6): δ (ppm) 7.55 (t, J = 7.6, 1H), 8.20 (dd, J_1 = 7.8, J_2 = 1.6, 2H), 8.29 (s, 2H, HC=C), 8.49 (s, 1H), 11.29 (NH, s, 2H), 11.44 (NH, s, 2H); ^{13}CNMR (400 MHz, DMSO-d_6) (d, ppm) = 120.3, 128.0, 132.9, 135.9, 137.6, 150.6, 153.9, 161.9, 163.6.

FTIR



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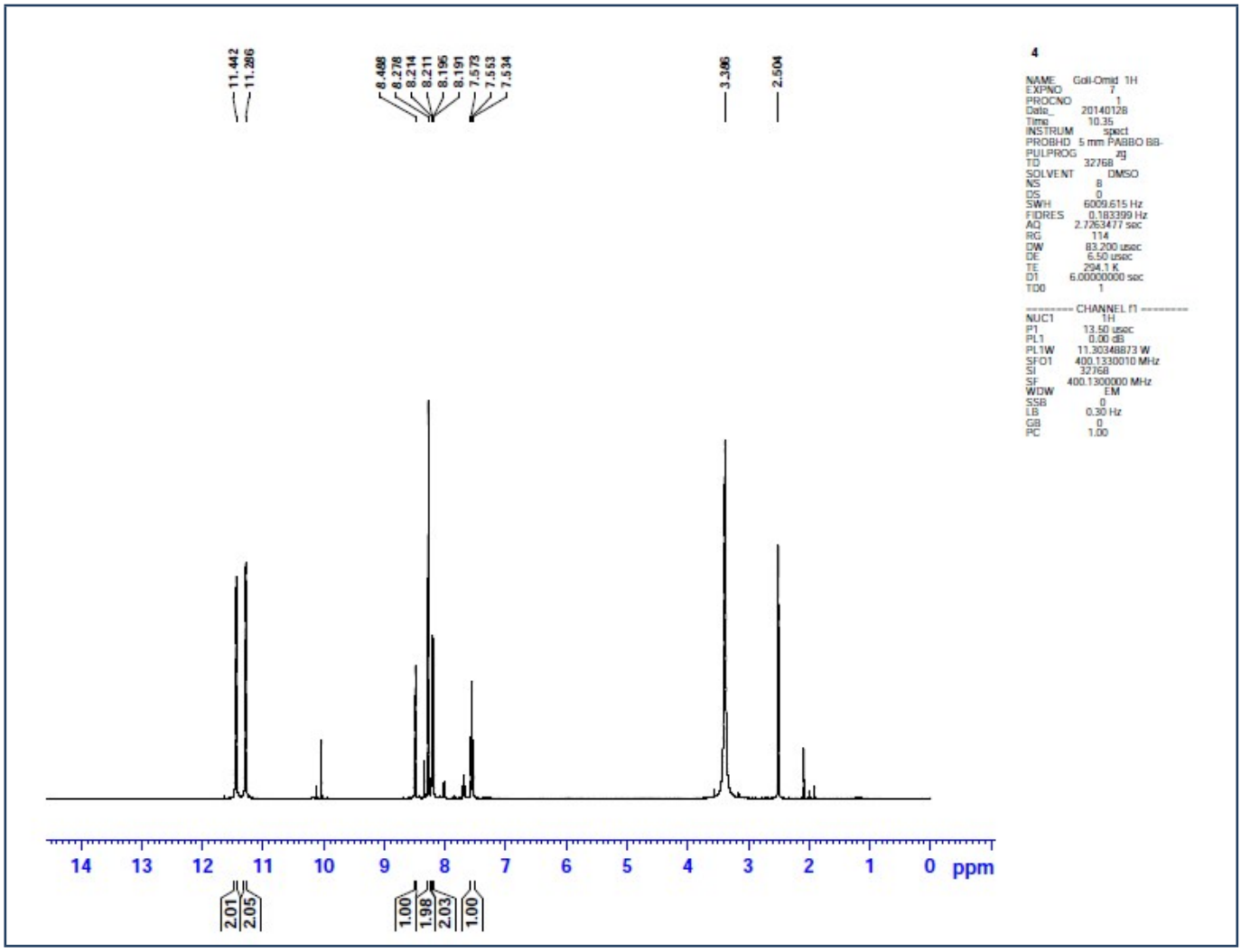
^{1}HNMR



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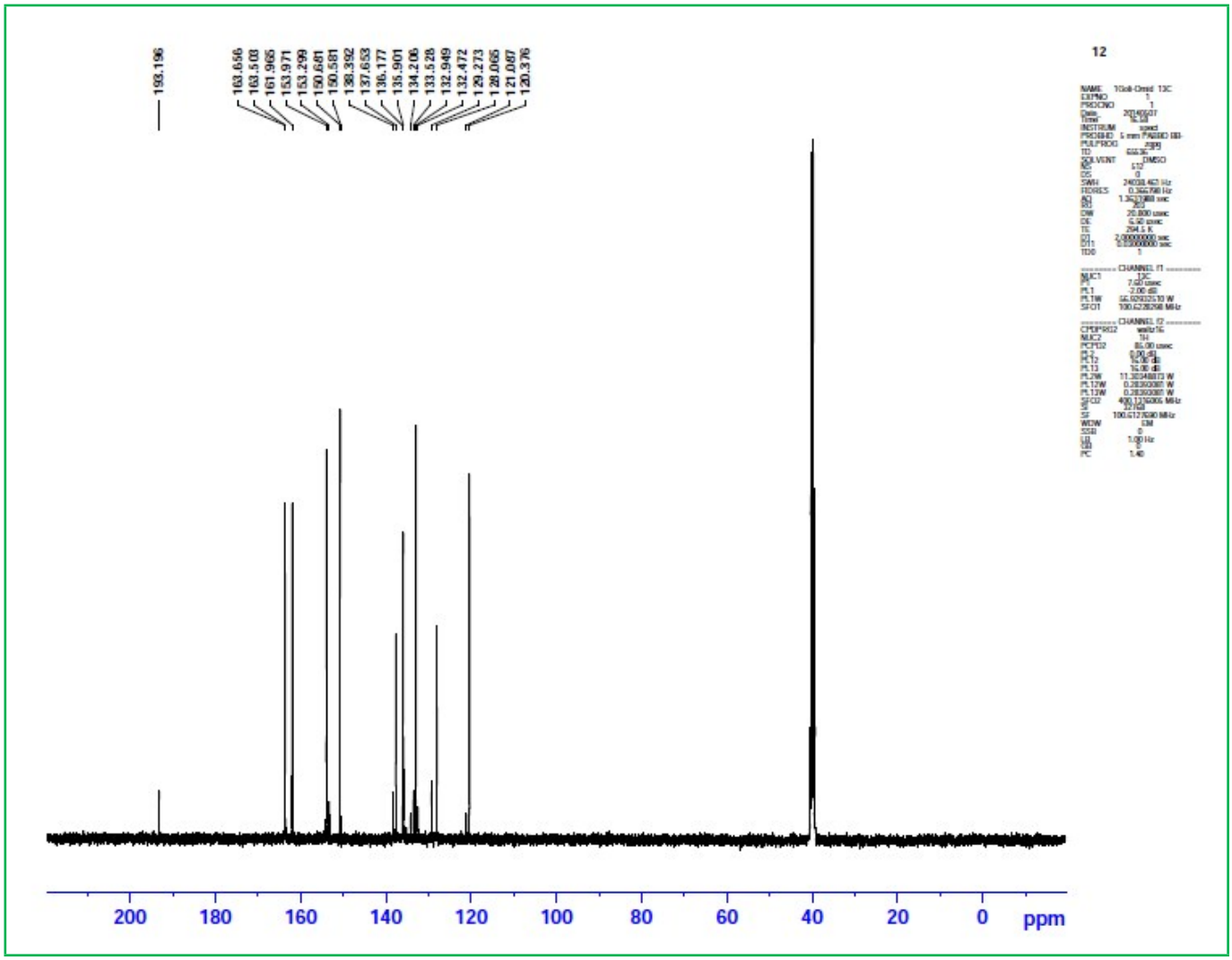
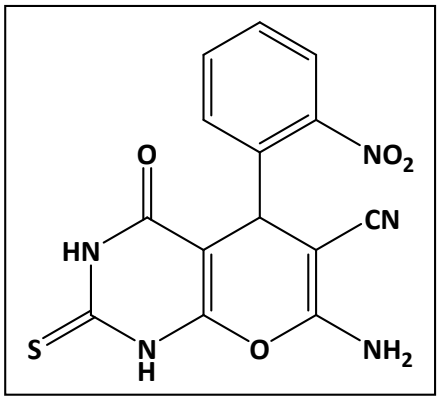
^{13}CNMR

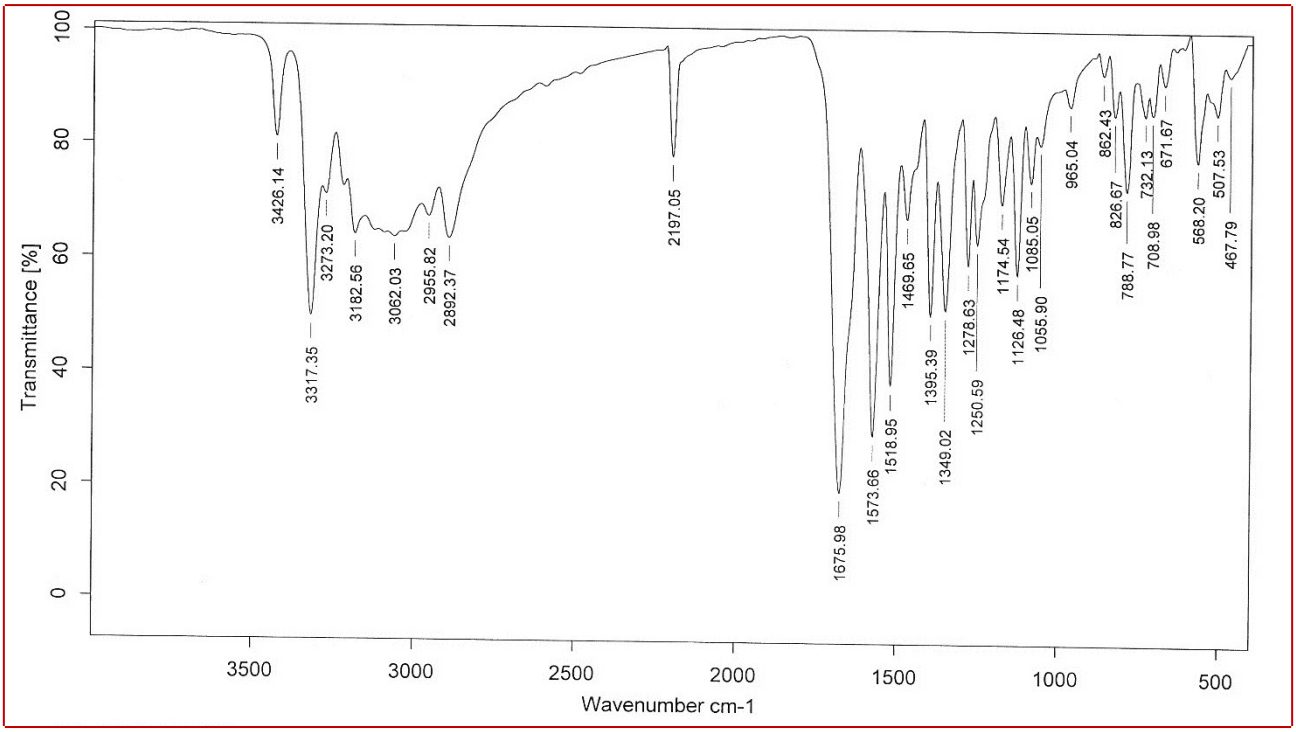

193.196
163.656
163.503
161.965
153.971
153.299
150.681
150.581
138.392
137.653
136.177
135.901
134.206
133.528
132.949
132.472
129.273
128.065
121.067
120.376
12
200
180
160
140
120
100
80
60
40
20
0
ppm

7-Amino-5-(2-nitrophenyl)-4-oxo-2-thioxo-1,3,4,5-tetrahydro-2*H*-pyrano[2,3-*d*]pyrimidine-6-carbonitrile (5r)



M.p. =242-246 ^{o}C; IR (KBr) ν_{max}/cm^{-1}:3426, 3062, 2197, 1675, 1573, 1518, 1349; ^{1}HNMR (400 MHz, DMSO-d$_6$): δ (ppm) 5.06 (s, 1H), 7.34 (s, 2H), 7.48 (dt, J_1 = 7.6 Hz, J_2 = 1.6 Hz, 1H), 7.54 (dd, J_1 = 8 Hz, J_2 = 1.2 Hz, 1H), 7.67 (dt, J_1 = 7.6 Hz, J_2 = 1.2 Hz, 1H), 7.86 (dd, J_1 = 8 Hz, J_2 = 1.2 Hz, 1H), 12.43 (NH, s, 1H), 13.67 (NH, s, 1H); ^{13}CNMR (100 MHz, DMSO-d$_6$): δ (ppm) 30.7, 56.9, 93.4, 119.0, 124.2, 128.6, 131.5, 133.9, 138.0, 149.7, 152.1, 158.6, 160.7, 174.4.

FTIR



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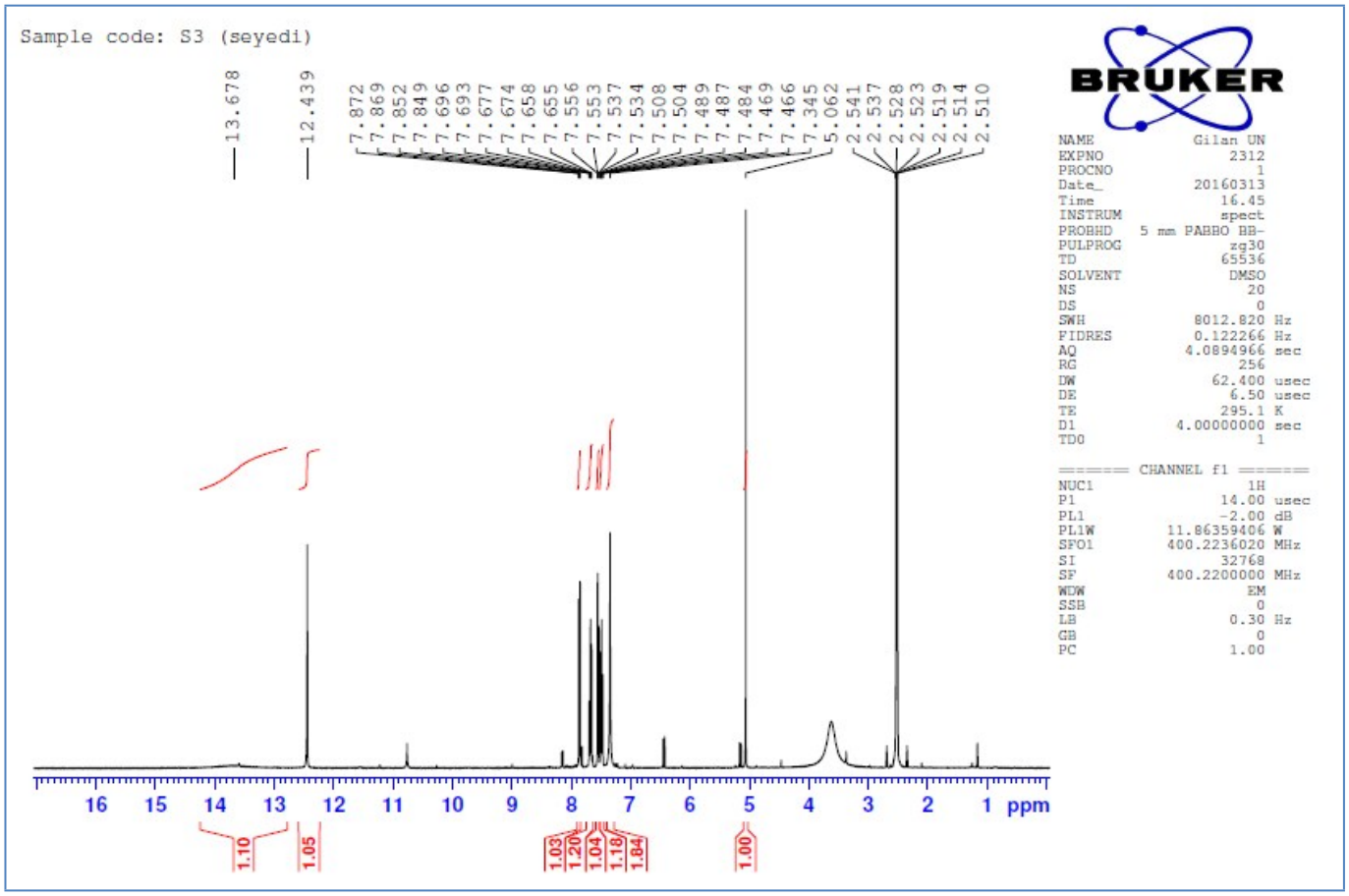
^{1}HNMR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7.869
7.852
7.849
7.696
7.693
7.677
7.674
7.658
7.655
7.556
7.553
7.537
7.534
7.508
7.504
7.489
7.487
7.484
7.469
7.466
7.345
5.062
2.541
2.537
2.528
2.523
2.519
2.514
2.510
BRUKER
NAME Gilan UN
EXPNO 2312
PROCNO 1
Date_ 20160313
Time 16.45
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT DMSO
NS 20
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 256
DW 62.400 usec
DE 6.50 usec
TE 295.1 K
D1 4.00000000 sec
TD0 1
====== CHANNEL f1 ======
NUC1 1H
P1 14.00 usec
PL1 -2.00 dB
PL1W 11.86359406 W
SFO1 400.2236020 MHz
SI 32768
SF 400.2200000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
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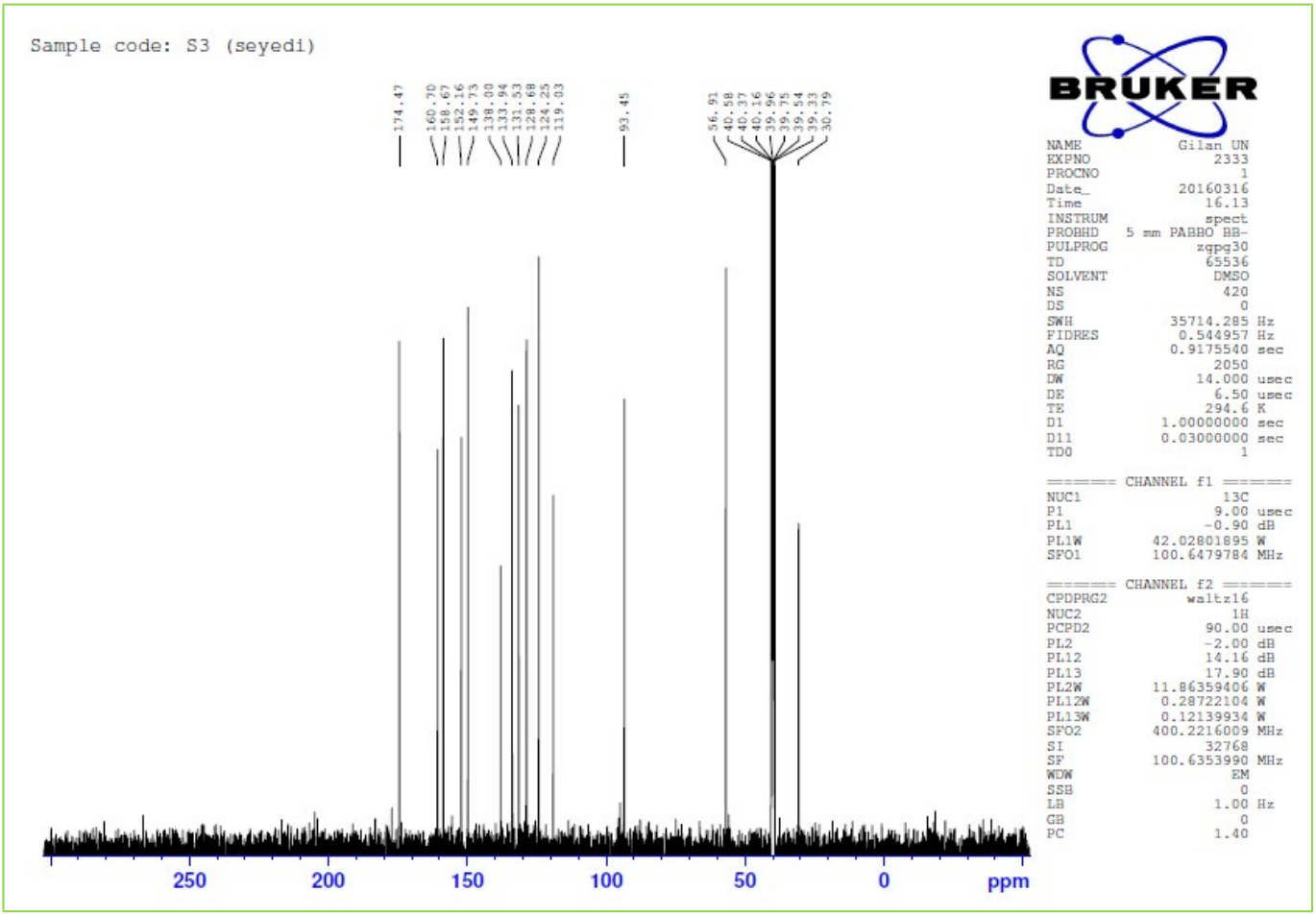
1.03
1.20
1.04
1.19
1.84
1.00

^{13}CNMR



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