

Supplementary Information

SiO₂/ZnBr₂ mediated expeditious approach to 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-one derivatives in water under microwave irradiation

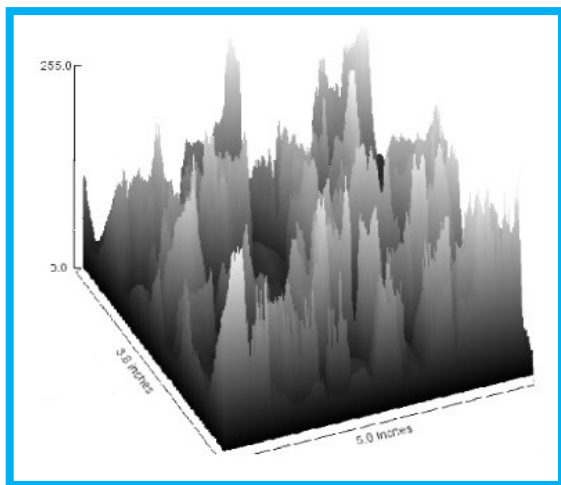
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Mayank Gupta^b

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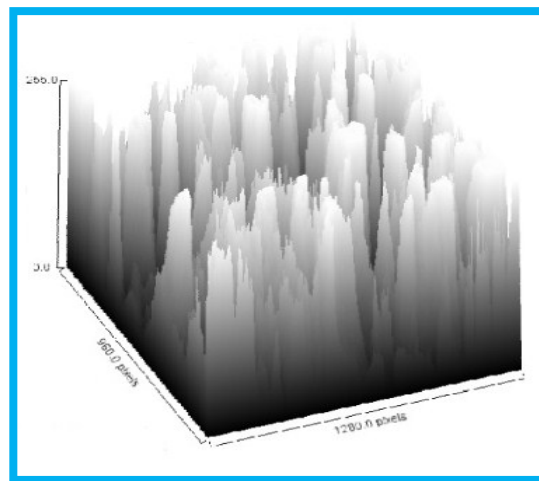
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^{*}*Corresponding author (Mehtab Parveen), E-mail: mehtab.organic2009@gmail.com; Tel: +91-9897179498*

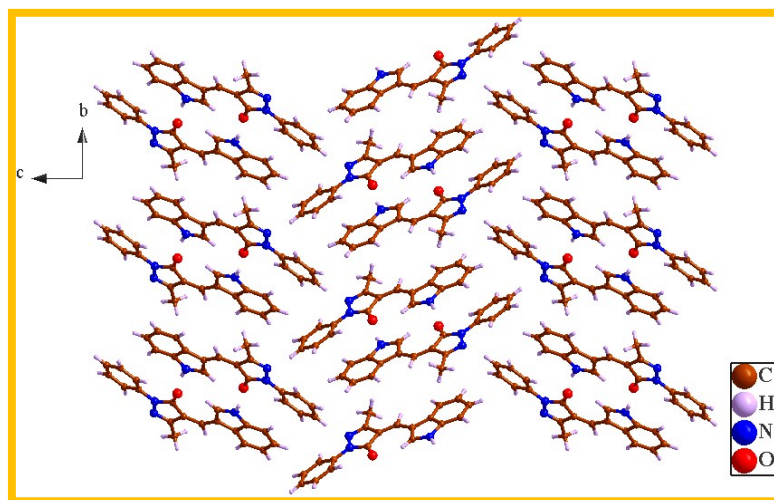


(a)

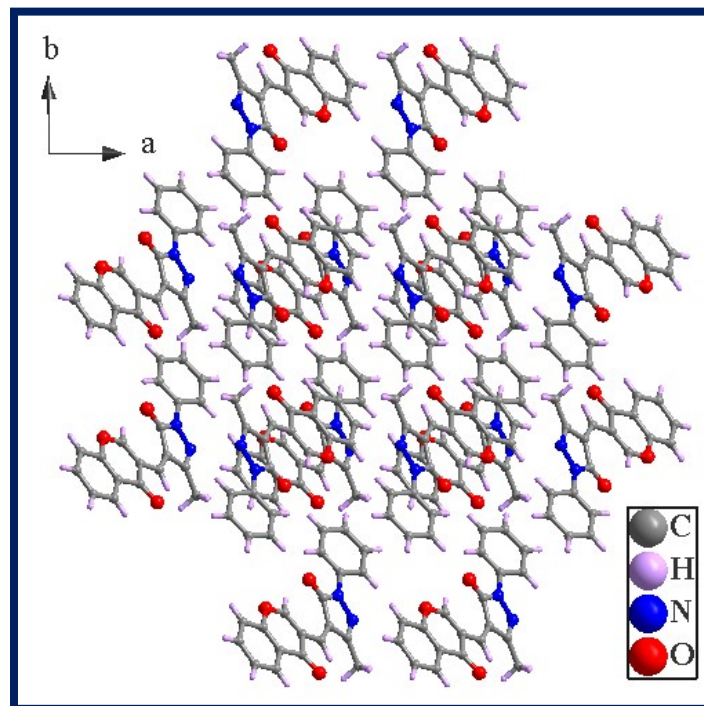


(b)

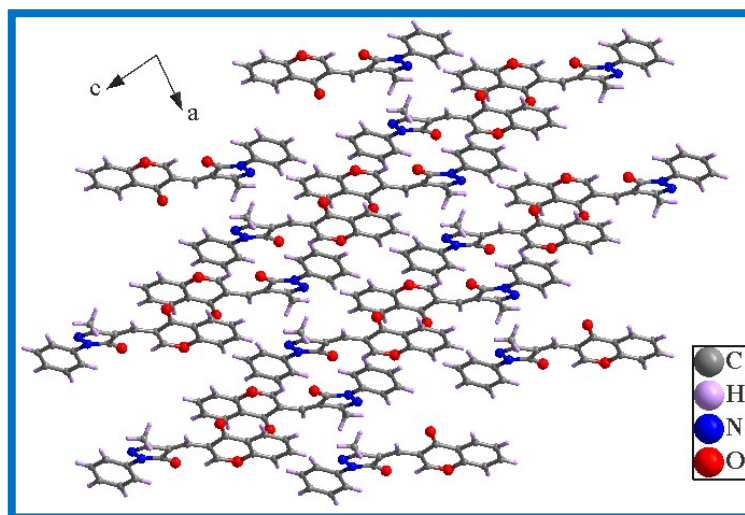
Fig. S1 3D Surface Plot of (a) pure SiO_2 (b) $\text{SiO}_2/\text{ZnBr}_2$ catalyst.



(a)

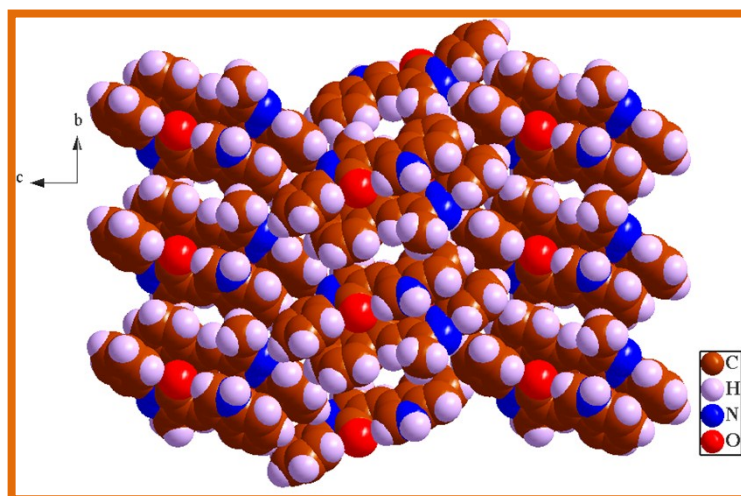


(b)

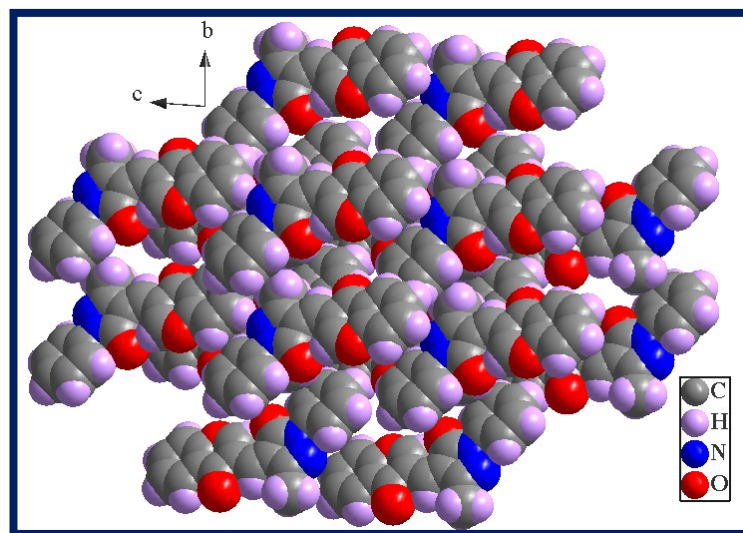


(c)

Fig. S2 Representation of 2D view along crystallographic (a) a-axis of compound **2a** (b) c-axis of compound **2d** (c) ac place of compound **2d**



(a)



(b)

Fig. S3 Representation of 2D view in CPK model along crystallographic a-axis of **(a)** compound **2a** **(b)** compound **2d**

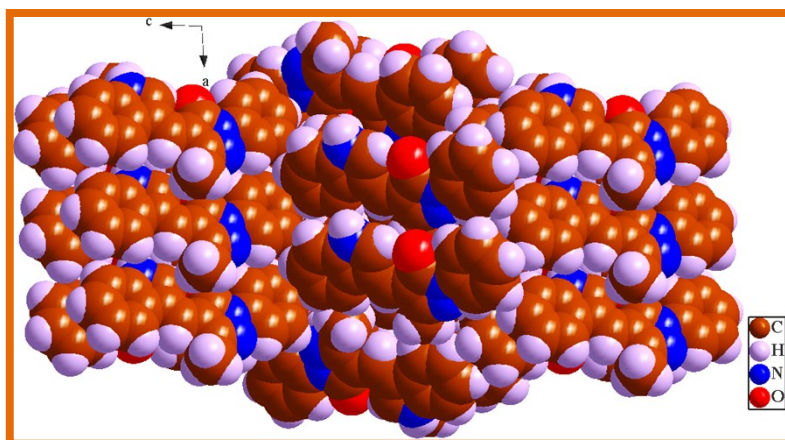


Fig. S4 Representation of 2D view in CPK model along crystallographic b-axis of compound **2a**

Table S1 Selected bond distances (Å) and bond angles (°) in compound **2a**

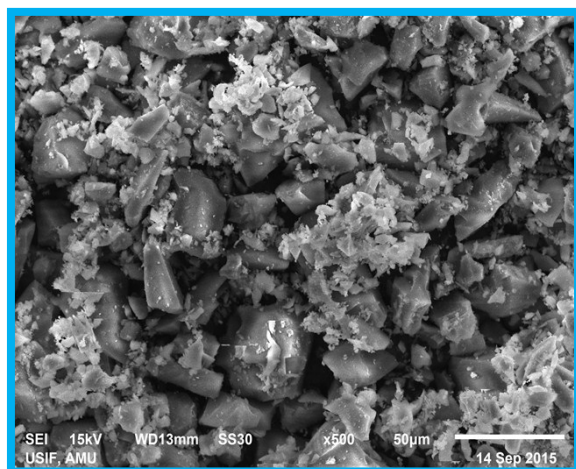
C1 N1 1.386(3)	C1 C2 1.394(2)	C1 C6 1.404(3)
C2 C3 1.375(3)	C3 C4 1.398(3)	C4 C5 1.382(3)
C5 C6 1.385(3)	C6 C7 1.456(2)	C7 C8 1.388(3)
C7 C9 1.420(3)	C8 N1 1.351(2)	C9 C10 1.368(3)
C10 C12 1.446(3)	C10 C11 1.465(3)	C11 O1 1.235(2)
C11 N2 1.383(2)	C12 N3 1.303(2)	C12 C13 1.483(3)
C14 C15 1.389(3)	C14 C19 1.393(3)	C14 N2 1.418(2)
C15 C16 1.388(3)	C16 C17 1.381(3)	C17 C18 1.379(3)
C18 C19 1.381(3)		
N1 C1 C2 130.18(18)	N1 C1 C6 107.75(16)	C2 C1 C6 122.06(18)
C3 C2 C1 117.55(18)	C2 C3 C4 121.09(18)	C5 C4 C3 121.04(19)
C4 C5 C6 119.02(18)	C5 C6 C1 119.21(17)	C5 C6 C7 134.22(18)
C1 C6 C7 106.55(17)	C8 C7 C9 130.71(18)	C8 C7 C6 106.01(16)
C9 C7 C6 123.28(18)	N1 C8 C7 109.92(17)	C10C9 C7 133.47(19)
C9 C10 C12 123.43(18)	C9 C10 C11 131.70(18)	O1C11 N2 126.06(17)
C15 C14 N2 121.29(16)	C19 C14 N2 119.04(17)	C8 N1 C1 109.77(17)
C11 N2 N3 112.50(14)	C11 N2 C14 130.25(16)	N3N2C14 117.08(14)
C12 N3 N2 106.44(15)		

CCDC Number for this molecule: **CCDC 1409997**

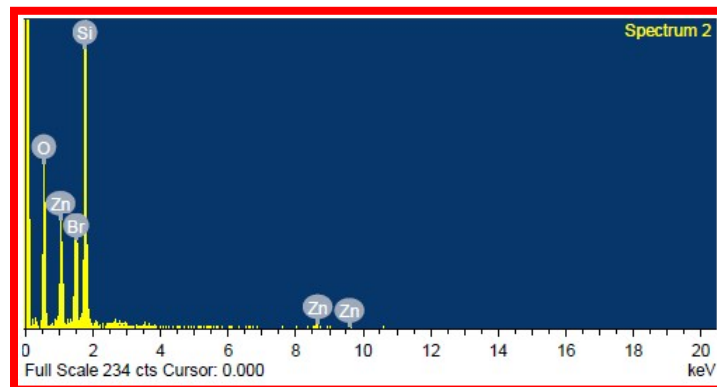
Table S2 Selected bond distances (Å) and bond angles (°) in compound **2d**

C1 O1 1.3414(18)	C1 C9 1.355(2)	C2 O1 1.3823(18)
C2 C3 1.385(2)	C2 C7 1.391(2)	C3 C4 1.379(2)
C4 C5 1.394(2)	C5 C6 1.375(2)	C6 C7 1.394(2)
C7 C8 1.464(2)	C8 O2 1.2272(18)	C8 C9 1.474(2)
C9 C10 1.447(2)	C10 C11 1.349(2)	C11 C13 1.454(2)
C11 C12 1.484(2)	C12 O3 1.2256(19)	C12 N1 1.378(2)
C13 N2 1.294(2)	C13 C14 1.491(2)	C15 C20 1.390(2)
C15 C16 1.396(2)	C15 N1 1.421(2)	C16 C17 1.385(2)
C17 C18 1.382(2)	C18 C19 1.384(2)	C19 C20 1.385(2)
O1 C1 C9 124.91(14)	O1 C2 C3 116.27(13)	O1 C2 C7 121.65(13)
C3 C2 C7 122.08(14)	C4 C3 C2 118.80(15)	C3 C4 C5 120.15(15)
C6 C5 C4 120.39(15)	C5 C6 C7 120.54(15)	C2 C7 C6 118.00(14)
C2 C7 C8 119.96(14)	C6 C7 C8 122.04(14)	O2 C8 C7 122.82(14)
O2 C8 C9 122.17(14)	C7 C8 C9 114.99(13)	C1 C9 C10 125.01(14)
C1 C9 C8 119.28(14)	C10 C9 C8 115.64(13)	C11 C10 C9 134.04(15)
O3 C12 N1 126.01(14)	O3 C12 C11 130.24(14)	N1 C12 C11 103.74(13)
N2 C13 C11 112.42(14)	N2 C13 C14 120.99(14)	C11 C13 C14 126.52(14)
C20 C15 C16 120.09(15)	C20 C15 N1 120.98(14)	C16 C15 N1 118.92(14)
C18 C19 C20 121.15(15)	C19 C20 C15 119.19(15)	C12 N1 N2 112.96(12)
C12 N1 C15 128.90(13)	N2 N1 C15 118.13(12)	C13 N2 N1 106.65(12)
C1 O1 C2 118.92(12)		

CCDC Number for this molecule: **CCDC 1432605**



(a)

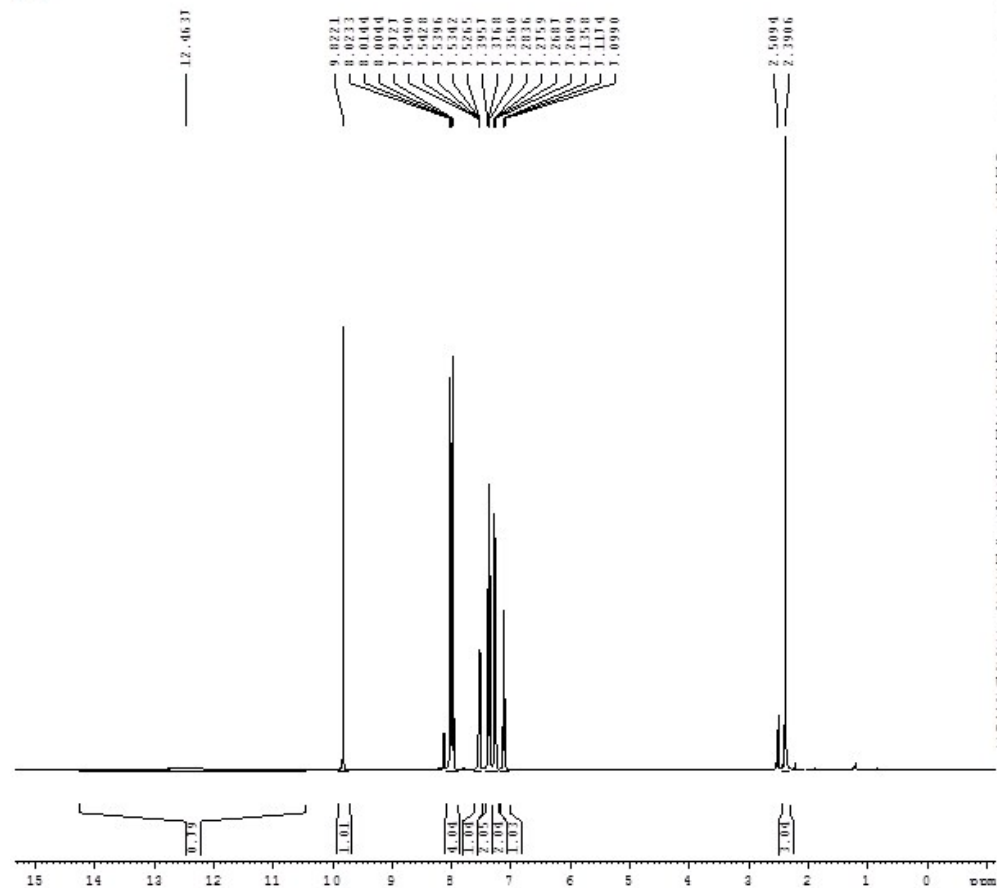


(b)

Fig. S5 (a) SEM micrograph (b) EDX spectrum of the recovered catalyst ($\text{SiO}_2/\text{ZnBr}_2$)

¹H and ¹³C NMR of Compounds (2a, 2d and 2m)

I-P



BRUKER
 AVANCE II 400 NMR
 Spectrometer
 SAIF
 Panjab University
 Chandigarh

Current Data Parameters
 NAME May14-2015
 EXPNO 11
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20150515
 Time 0.41
 INSTRUM spect
 PROBHD 5 mm PABBO BB-
 PULPROG zg30
 TD 65536
 SOLVENT DMSO
 NS 16
 DS 2
 SWH 12019.230 Hz
 FIDRES 0.183399 Hz
 AQ 2.7263477 sec
 RG 161
 DW 41.600 usec
 DE 6.00 usec
 TE 295.5 K
 D1 1.00000000 sec
 D10 1

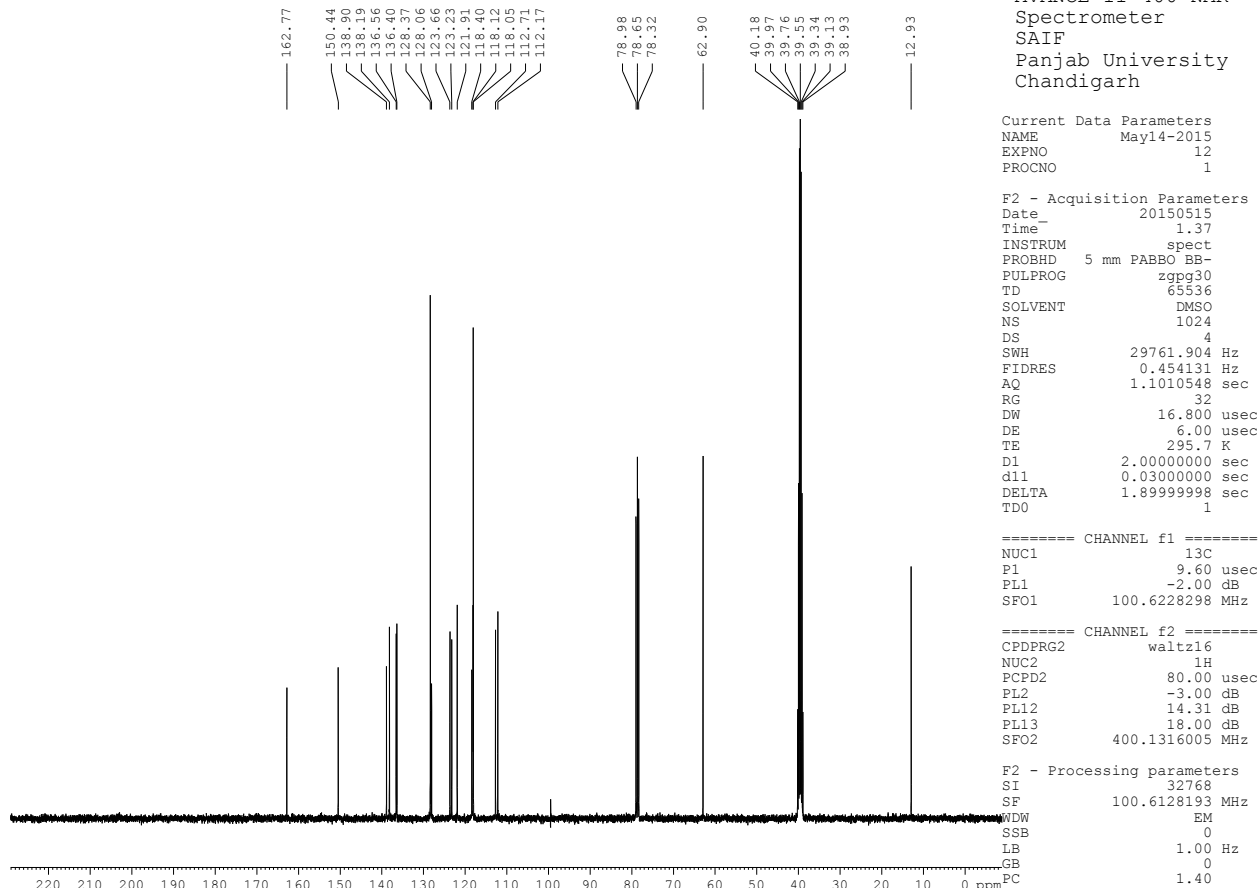
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 PL1 -3.00 dB
 SFO1 400.1324710 MHz

F2 - Processing parameters
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 SF 400.1300000 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

avtar saifpu@yahoo.co.in

¹H NMR of compound 2a

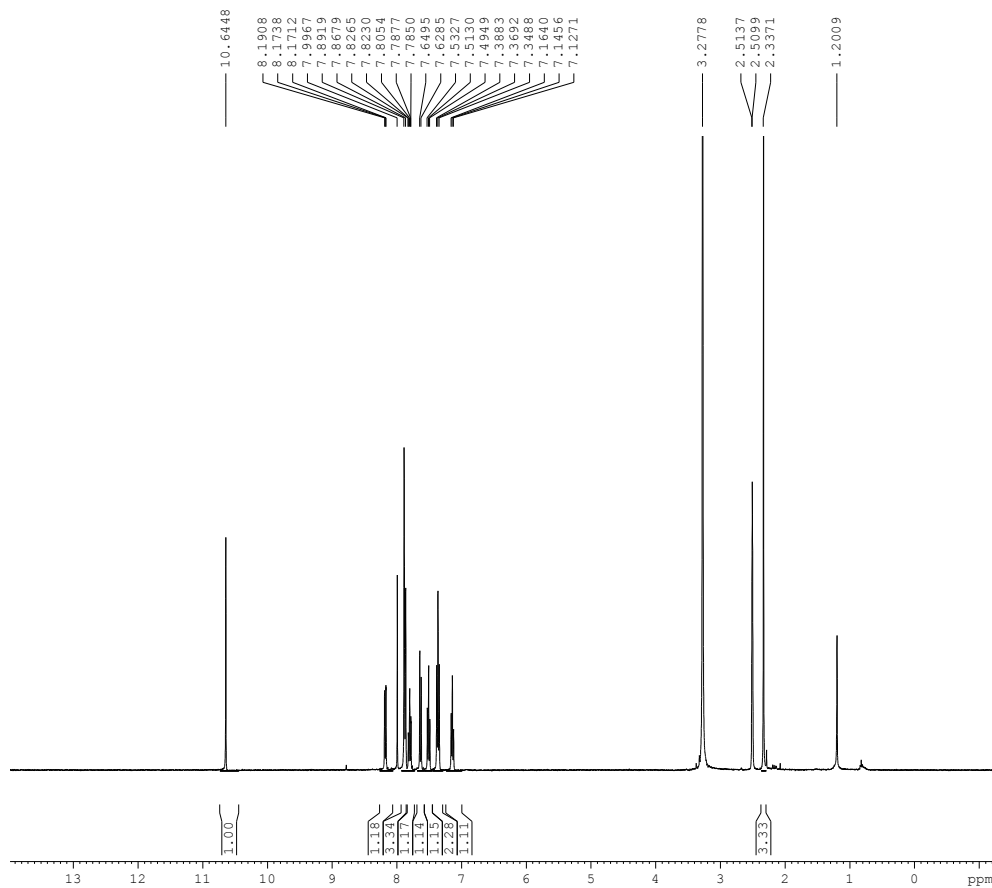
I-P



avtar_saifpu@yahoo.co.in

¹³C NMR of compound 2a

3F-P



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AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
NAME May14-2015
EXPNO 600
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150514
Time_ 23.40
INSTRUM spect
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PULPROG zg30
TD 65536
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DS 2
SWH 12019.230 Hz
FIDRES 0.183399 Hz
AQ 2.7263477 sec
RG 575
DW 41.600 usec
DE 6.00 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1

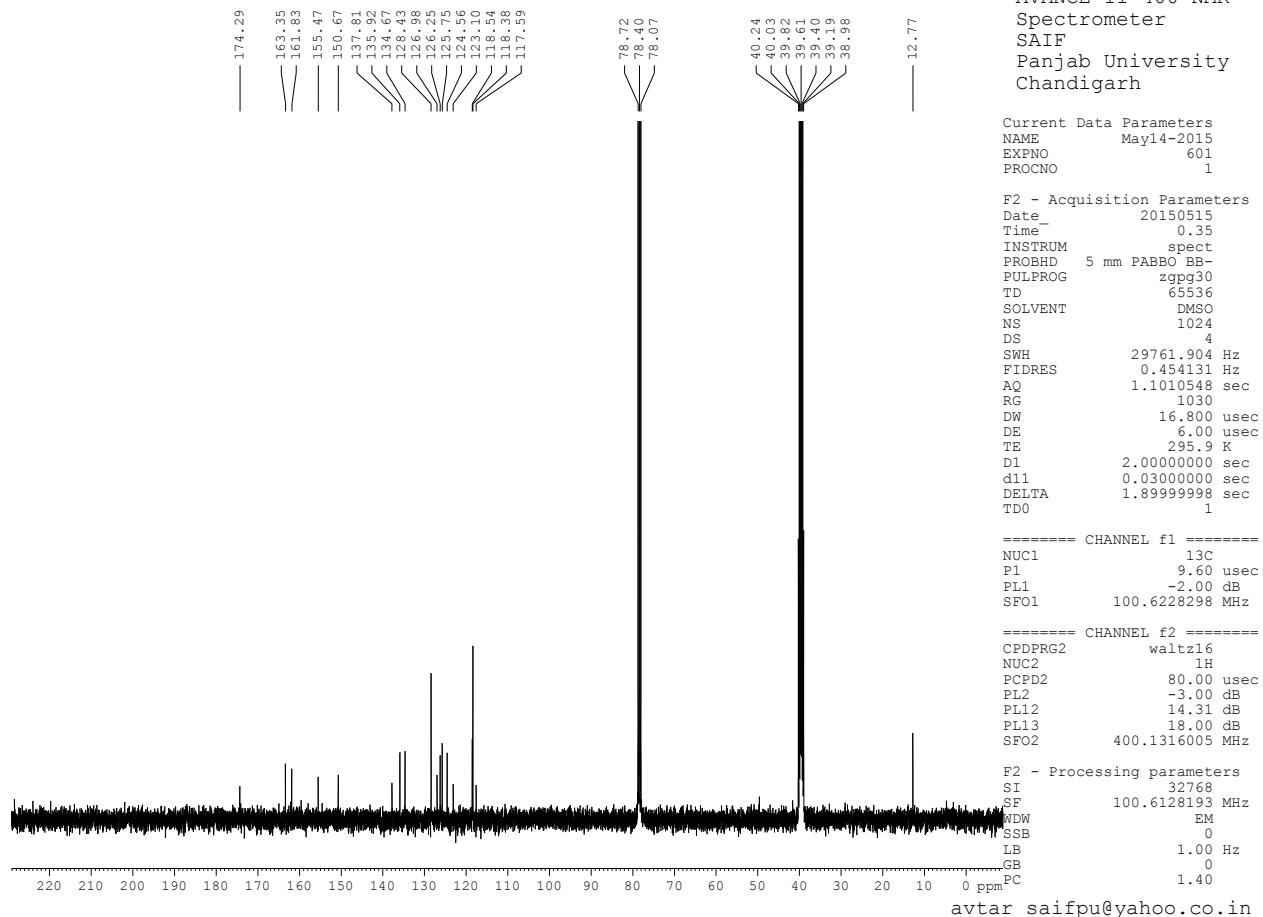
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PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
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SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

avtar_saifpu@yahoo.co.in

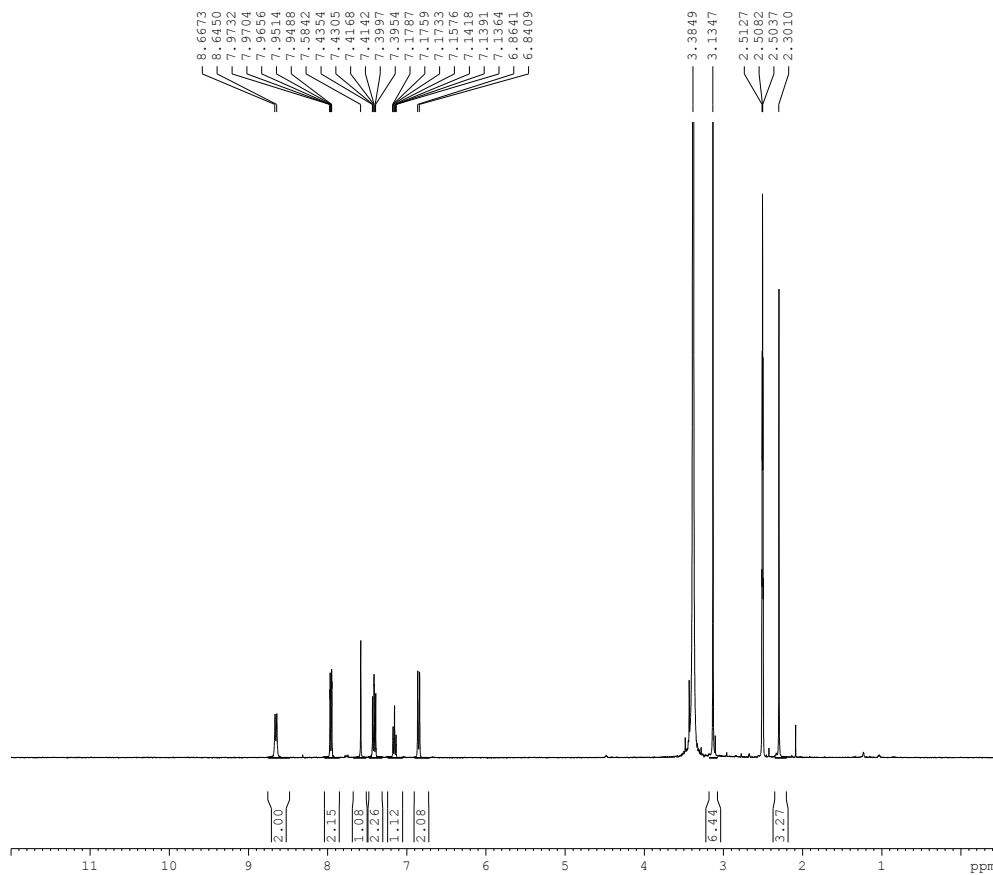
^1H NMR of compound **2d**

3F-P



^{13}C NMR of compound **2d**

NMe-P



BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
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EXPNO 420
PROCNO 1

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PULPROG zg30
TD 65536
SOLVENT DMSO
NS 16
DS 2
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FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 322
DW 60.800 usec
DE 6.00 usec
TE 293.8 K
D1 1.00000000 sec
TDO 1

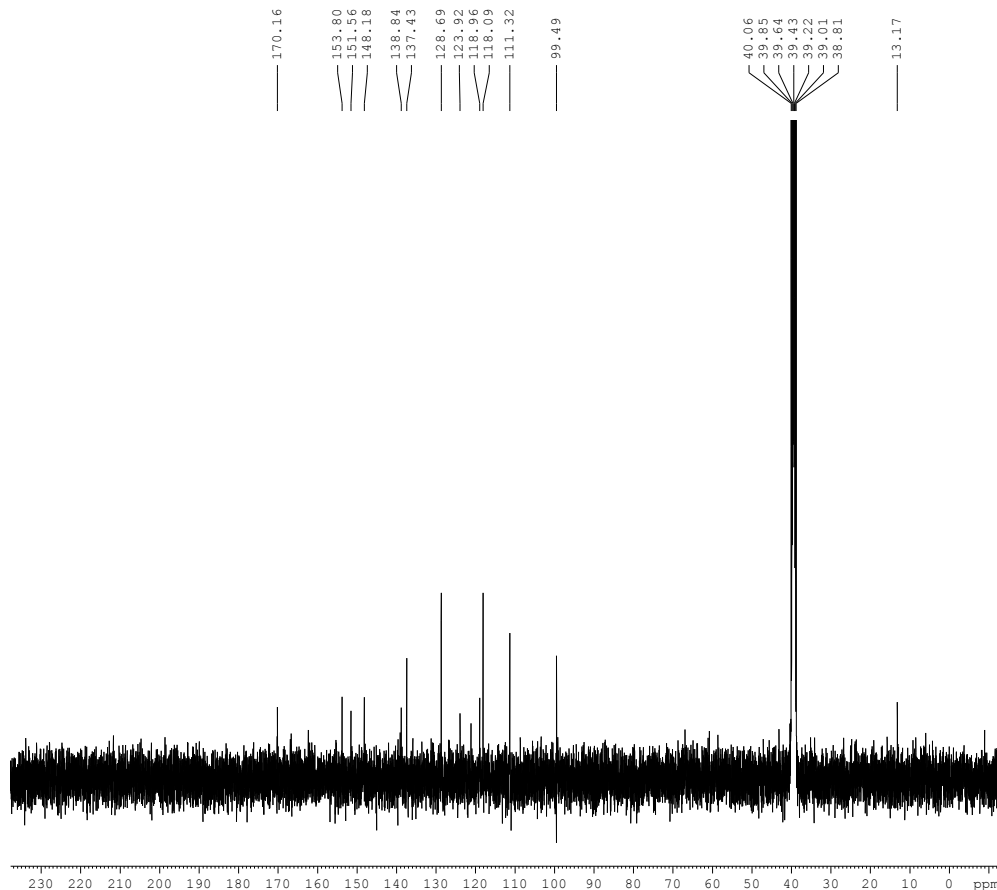
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PL1 -3.00 dB
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

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¹H NMR of compound **2m**

NMe-P



170.16
153.80
151.56
148.18
138.84
137.43
128.69
123.92
118.96
118.09
111.32
99.49

40.06
39.85
39.64
39.43
39.22
39.01
38.80
13.17

BRUKER
AVANCE II 400 NMR
Spectrometer
SAIF
Panjab University
Chandigarh

Current Data Parameters
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EXPNO 421
PROCNO 1

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FIDRES 0.454131 Hz
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RG 36
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DE 6.00 usec
TE 294.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

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SFO1 100.6228298 MHz

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PCPD2 80.00 usec
PL2 -3.00 dB
PL12 14.31 dB
PL13 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

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¹³C NMR of compound 2m