

Electronic Supplementary Information

Synthesis of pyrrolidinone derivatives from aniline, an aldehyde and diethyl acetylenedicarboxylate in an ethanolic citric acid solution under ultrasound irradiation

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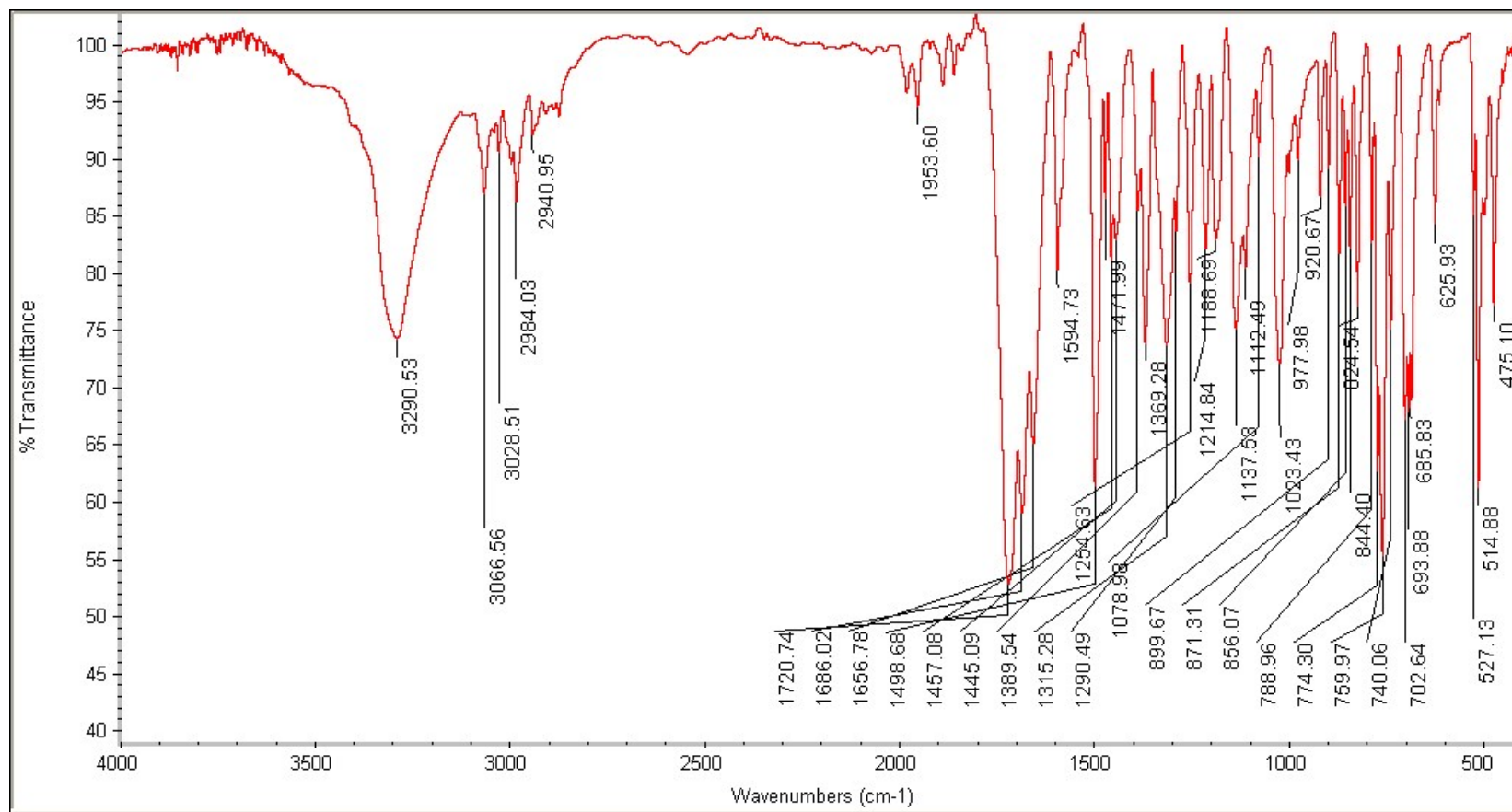


Fig. 1 FT-IR spectrum of Ethyl 4-hydroxy-5-oxo-1,2-diphenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4a**).

BpyA

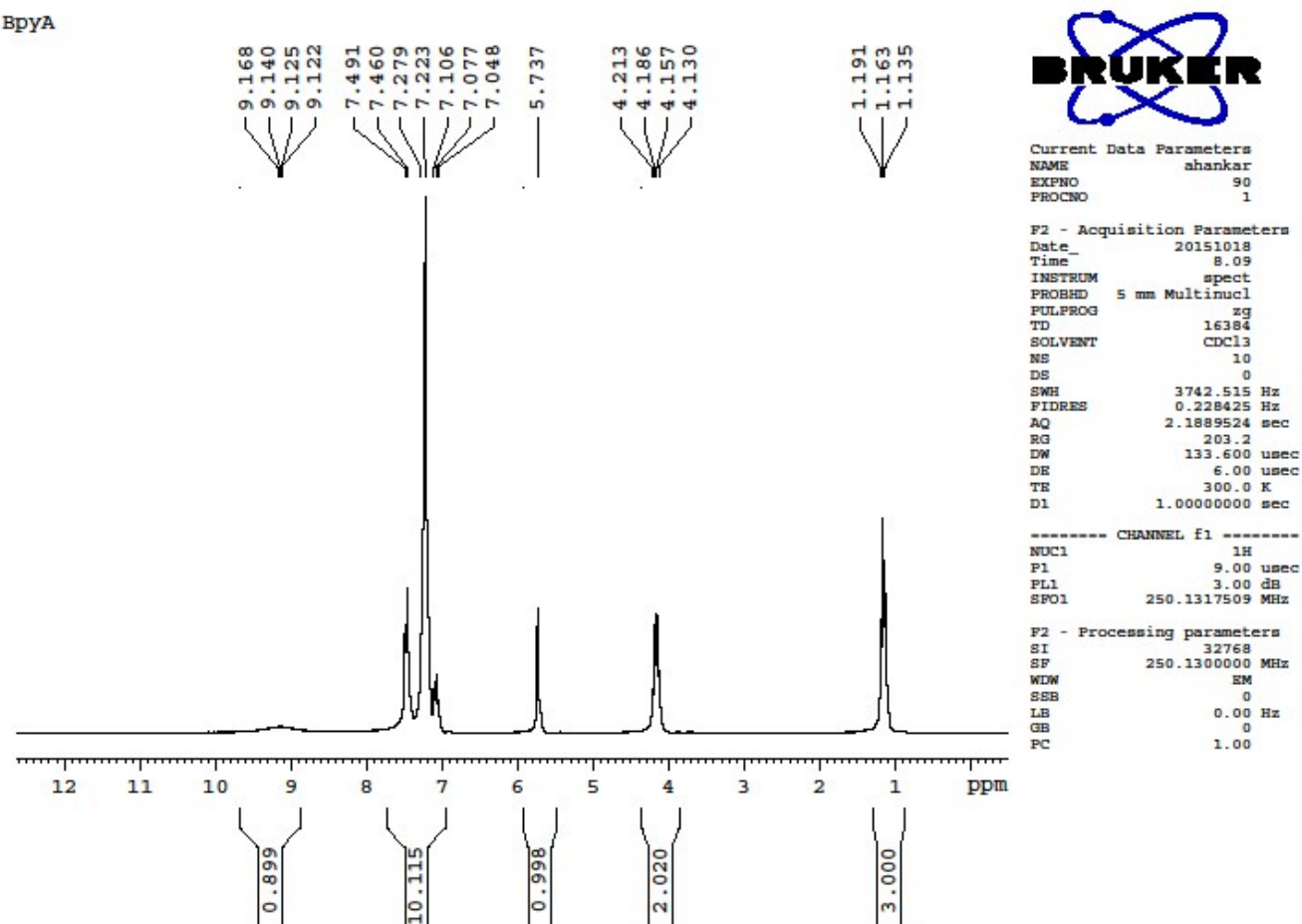
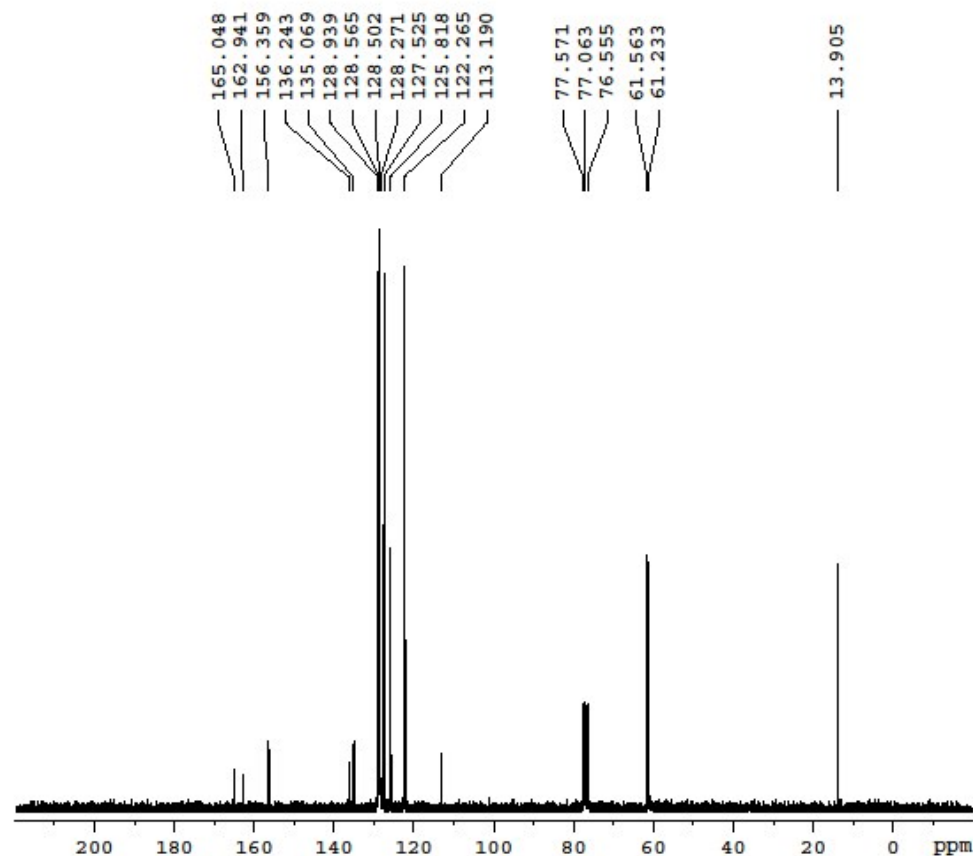


Fig. 2 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-5-oxo-1,2-diphenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4a**).

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Fig. 3 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-5-oxo-1,2-diphenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4a**).

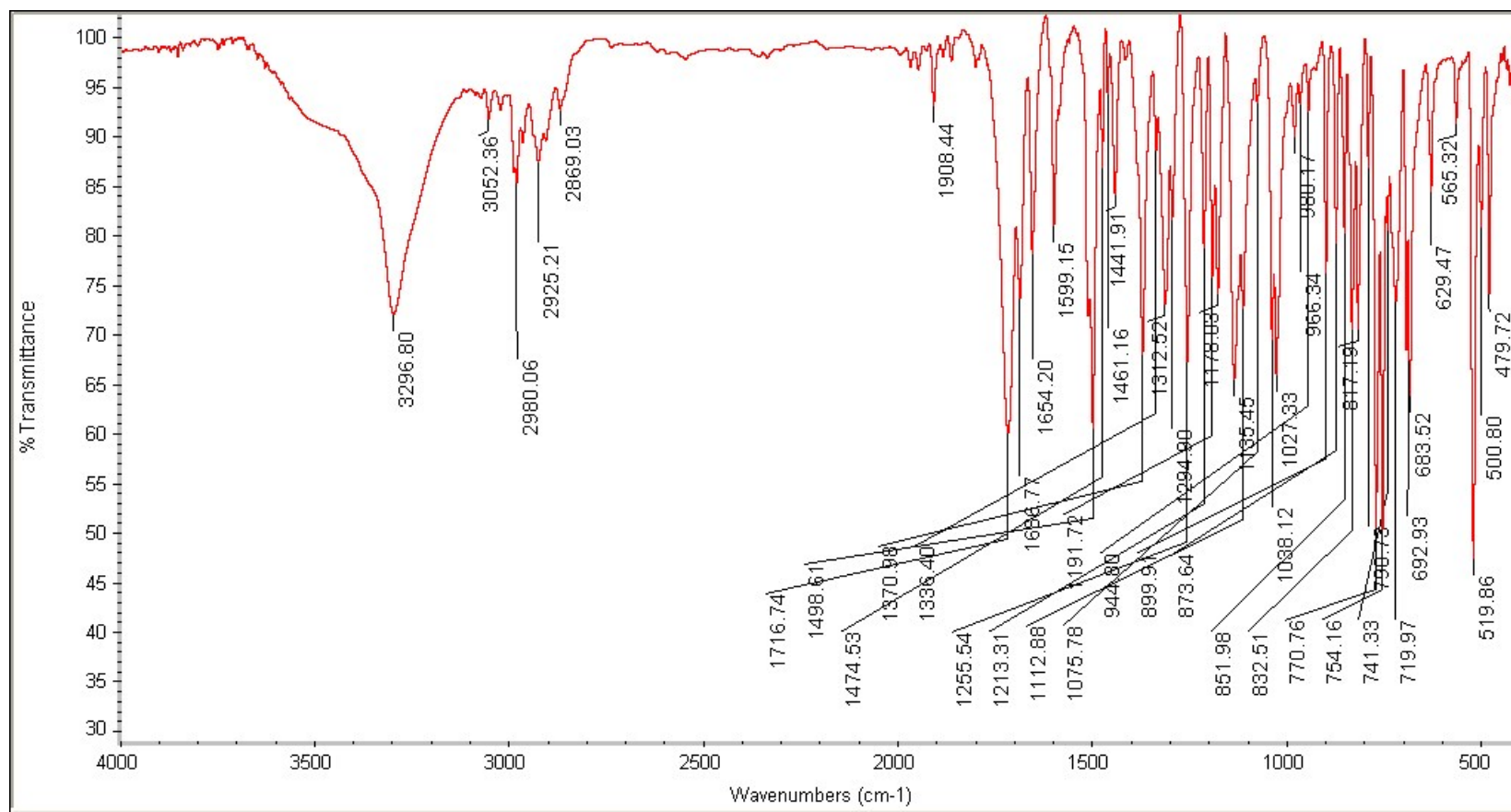


Fig. 4 FT-IR spectrum of Ethyl 4-hydroxy-5-oxo-1-phenyl-2-(p-tolyl)-2,5-dihydro-1H-pyrrole-3-carboxylate (**4b**).

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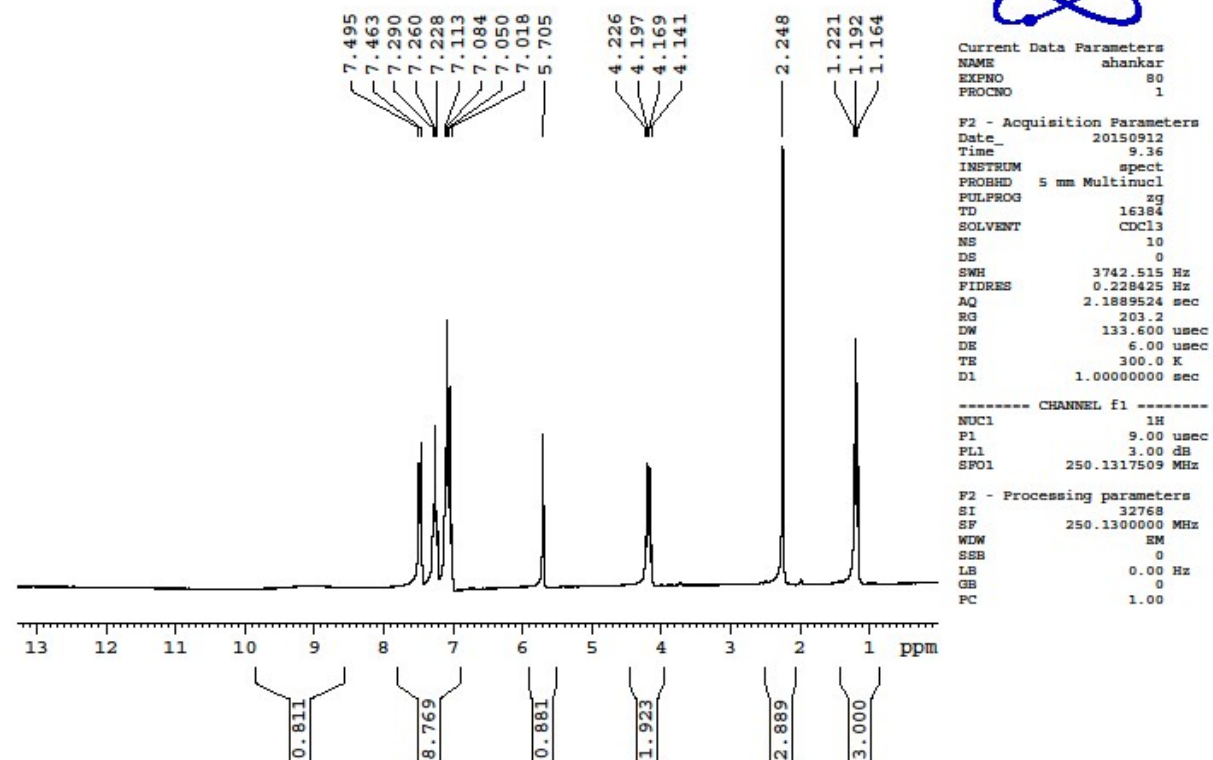
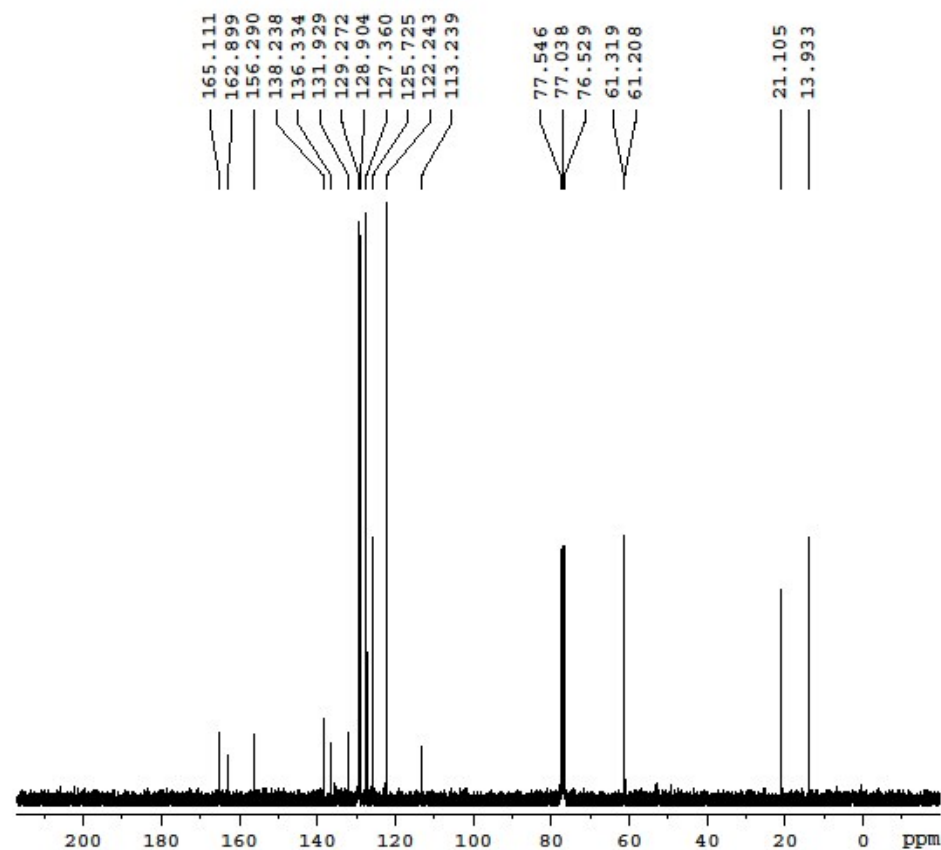


Fig. 5 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-5-oxo-1-phenyl-2-(p-tolyl)-2,5-dihydro-1H-pyrrole-3-carboxylate (**4b**)

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Fig. 6 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-5-oxo-1-phenyl-2-(p-tolyl)-2,5-dihydro-1H-pyrrole-3-carboxylate (**4b**).

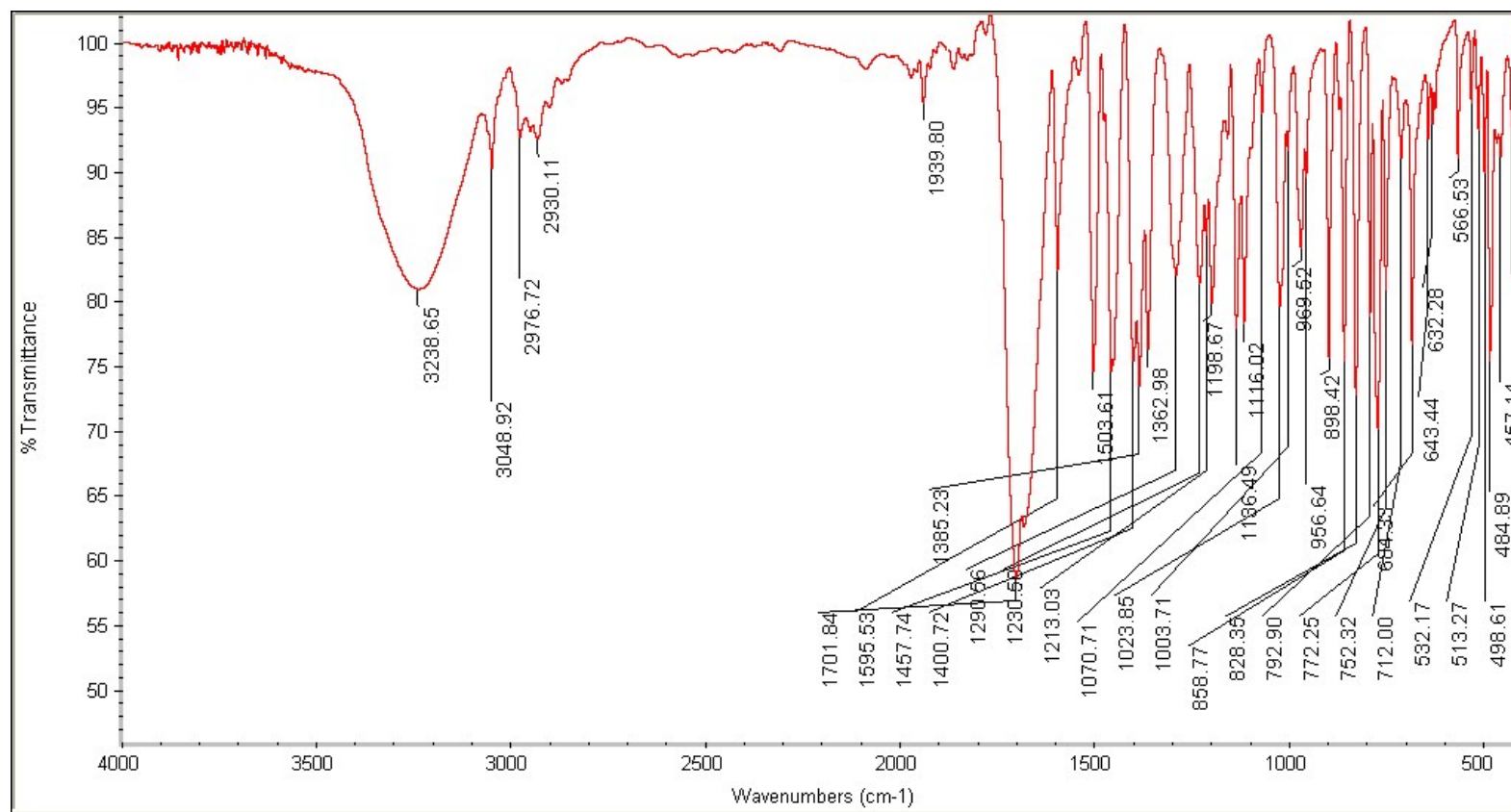


Fig .7 FT-IR spectrum of Ethyl 4-hydroxy-2-(naphthalen-2-yl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4c**).

naphtylF

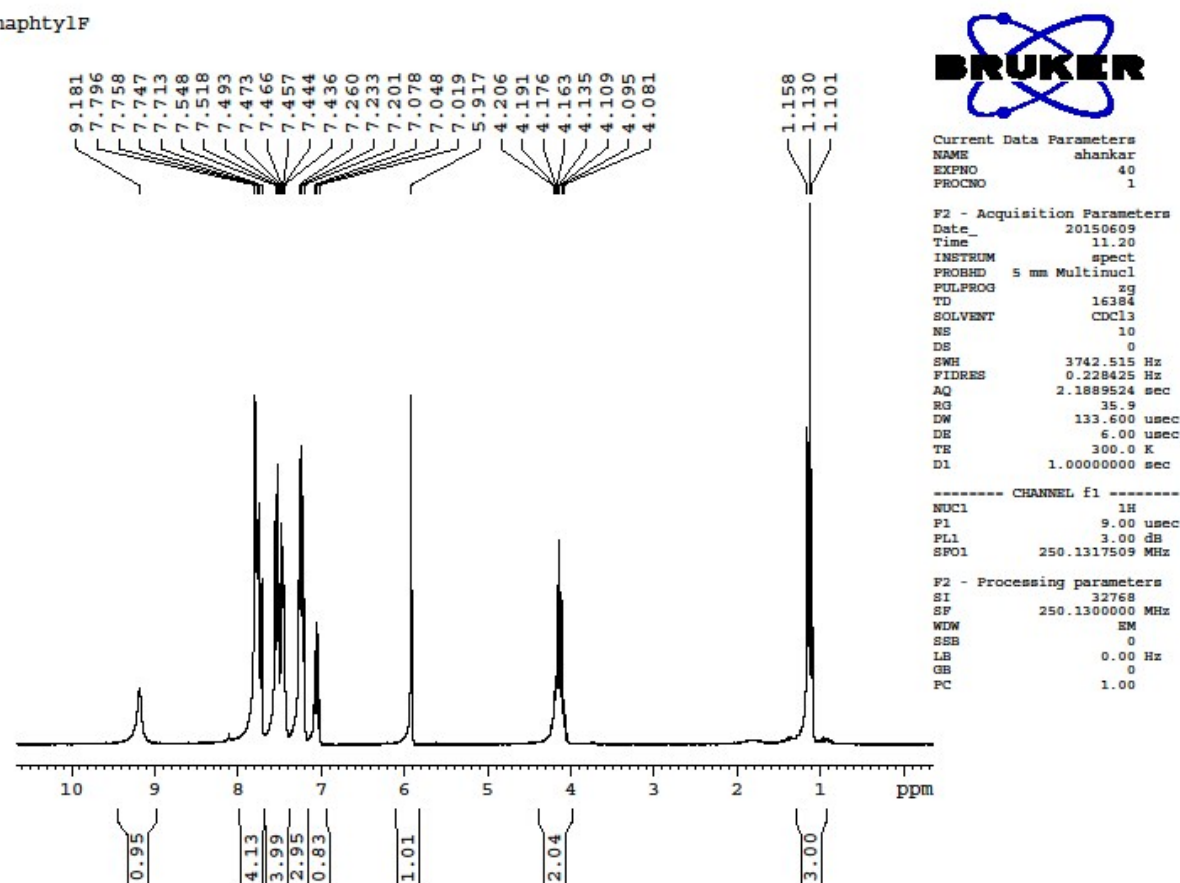
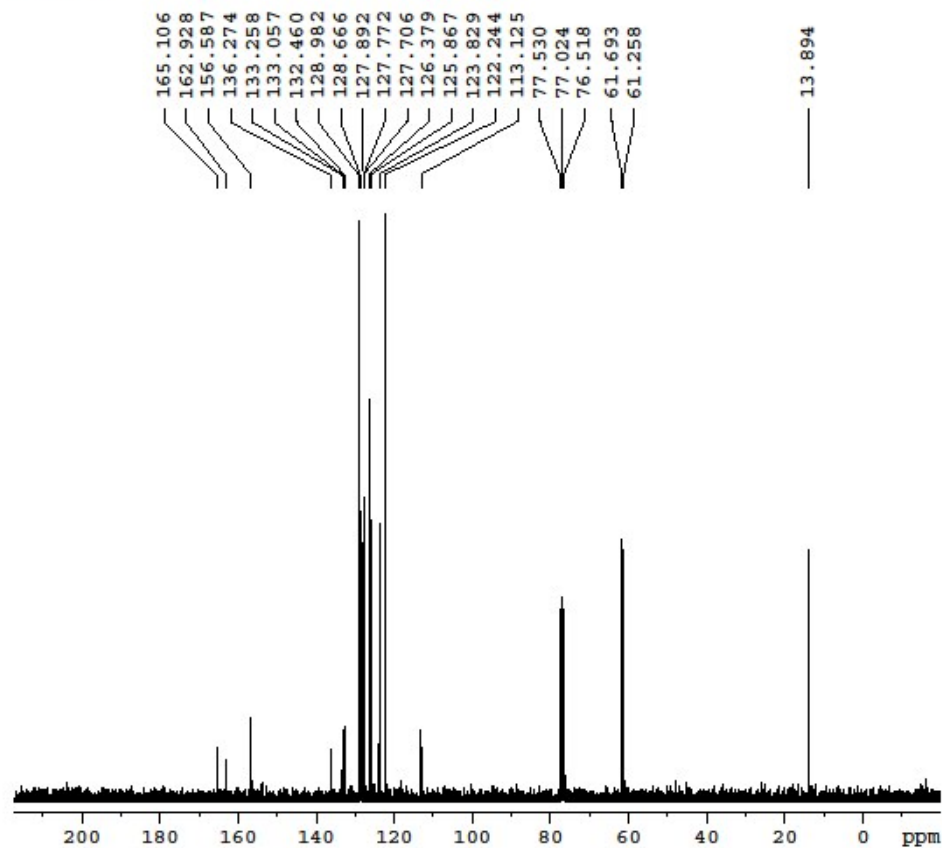


Fig. 8 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-2-(naphthalen-2-yl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4c**).

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Fig. 9 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-2-(naphthalen-2-yl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4c**).

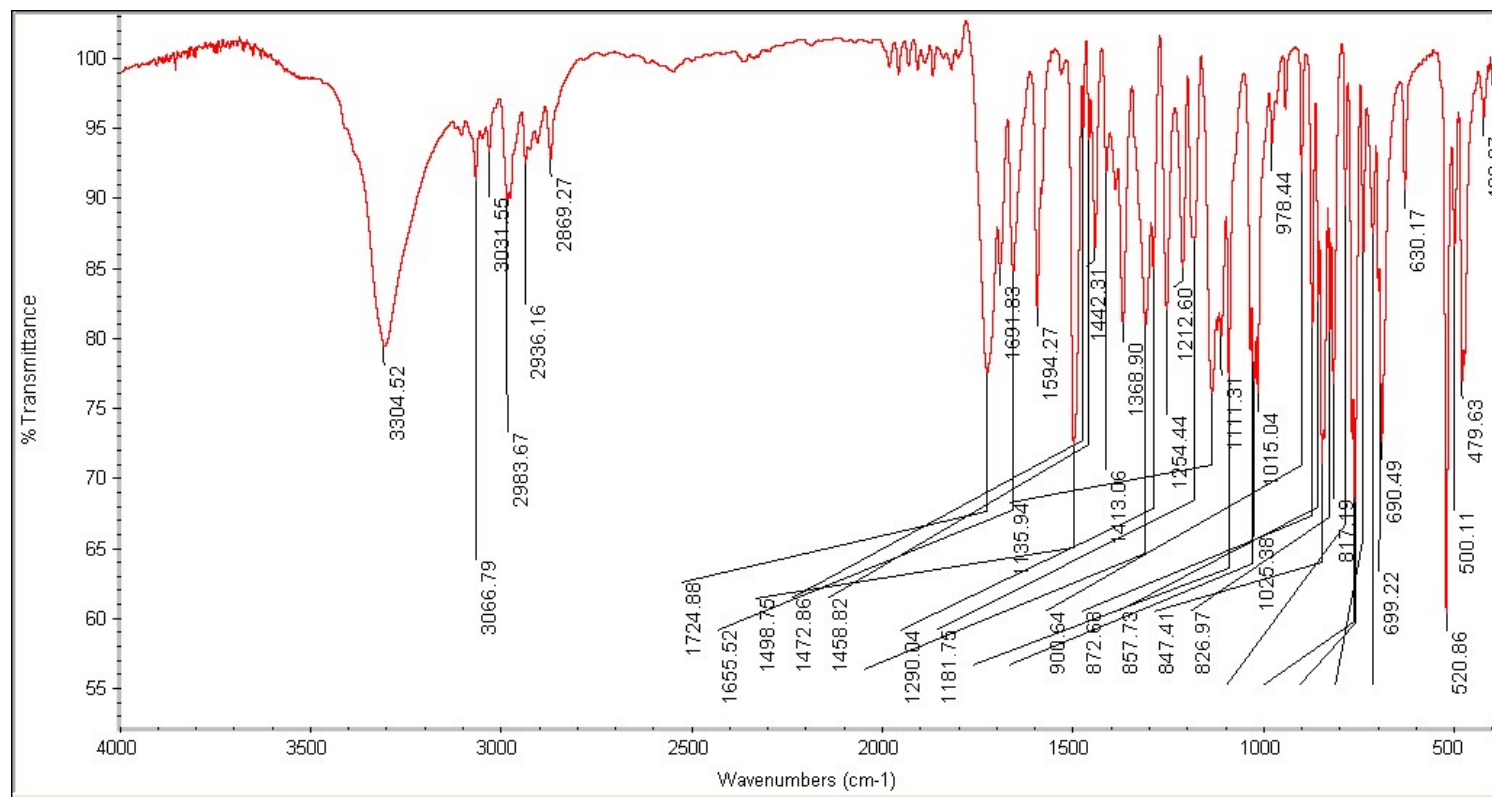


Fig. 10 FT-IR spectrum of Ethyl 2-(4-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4d**).

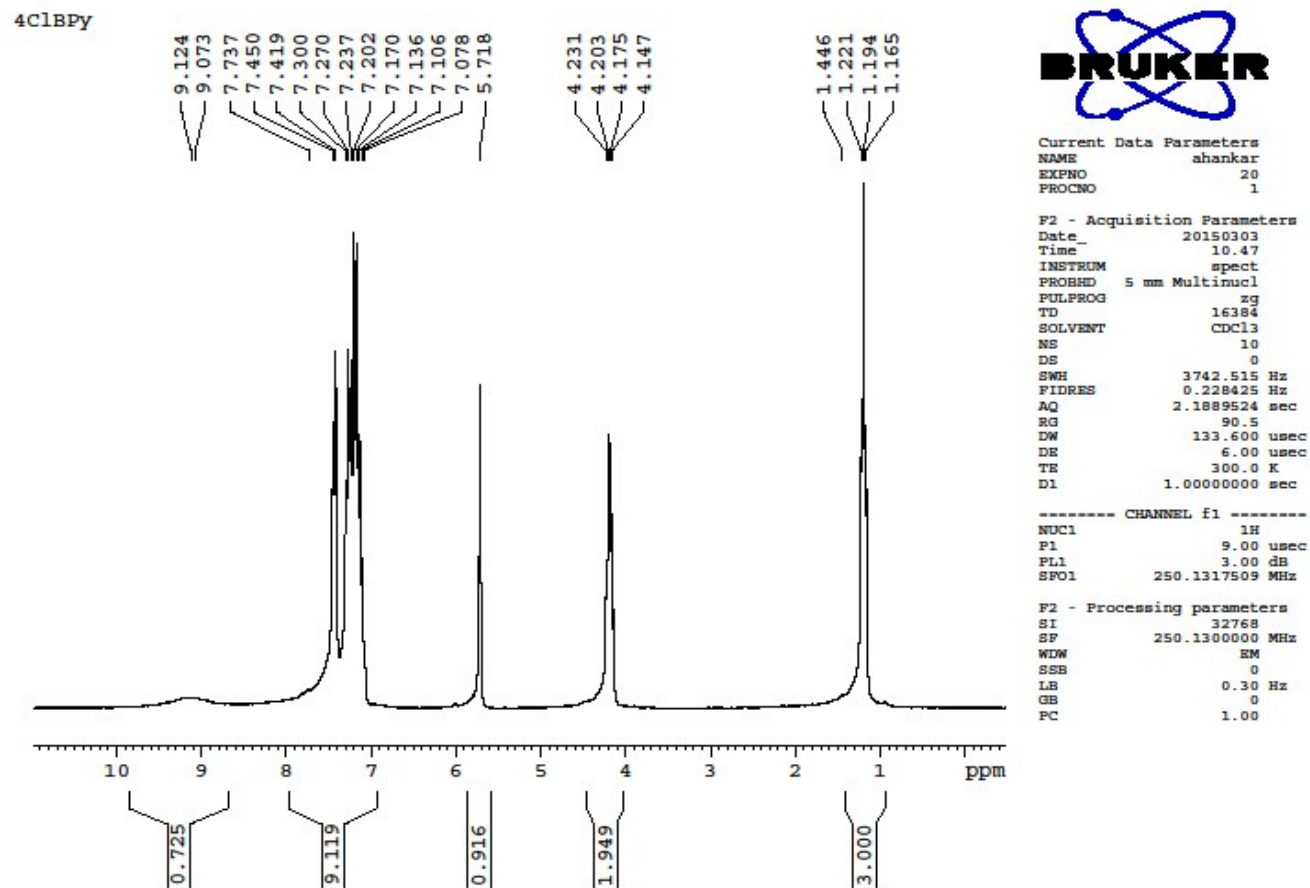


Fig. 11 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 2-(4-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4d**).

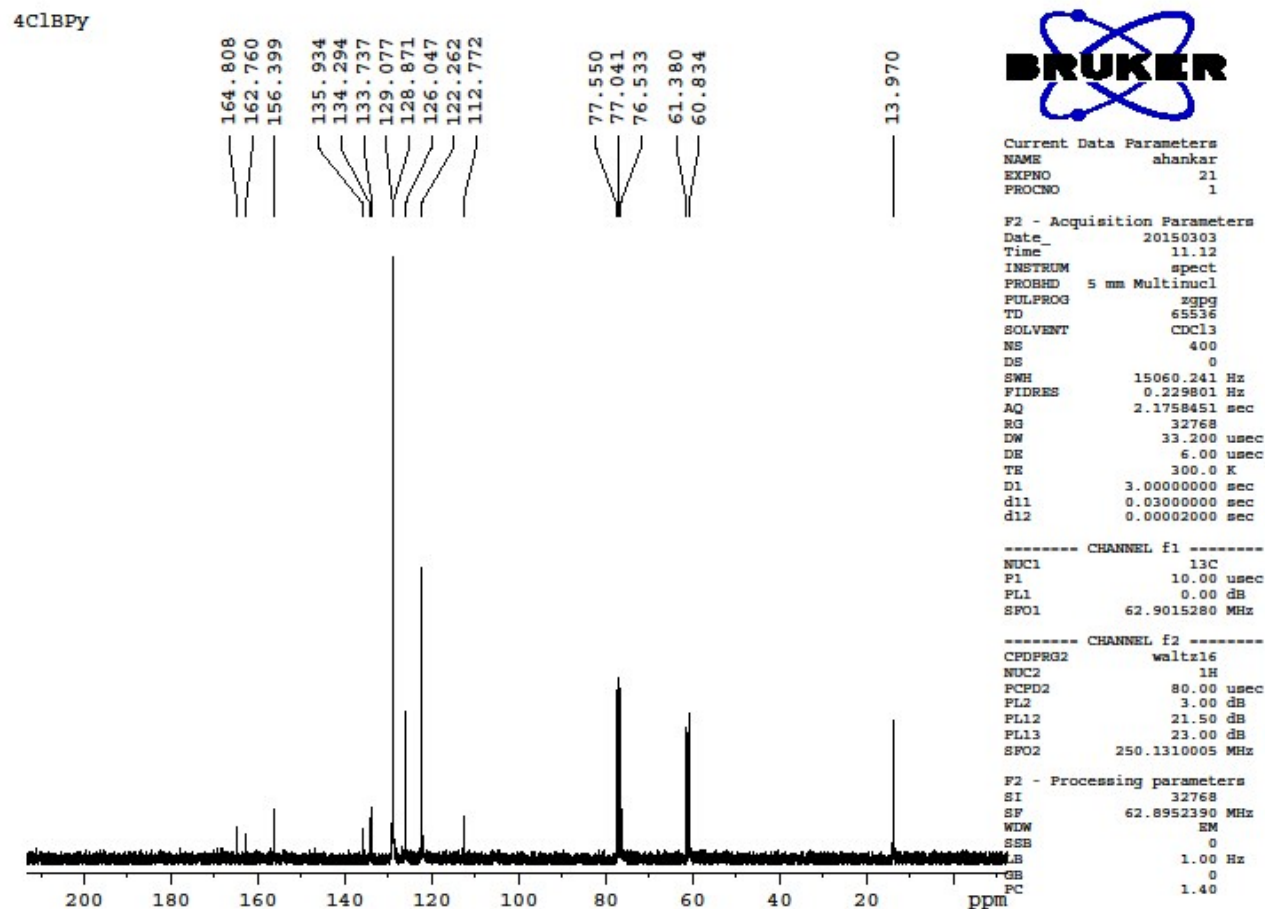


Fig. 12 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 2-(4-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (4d).

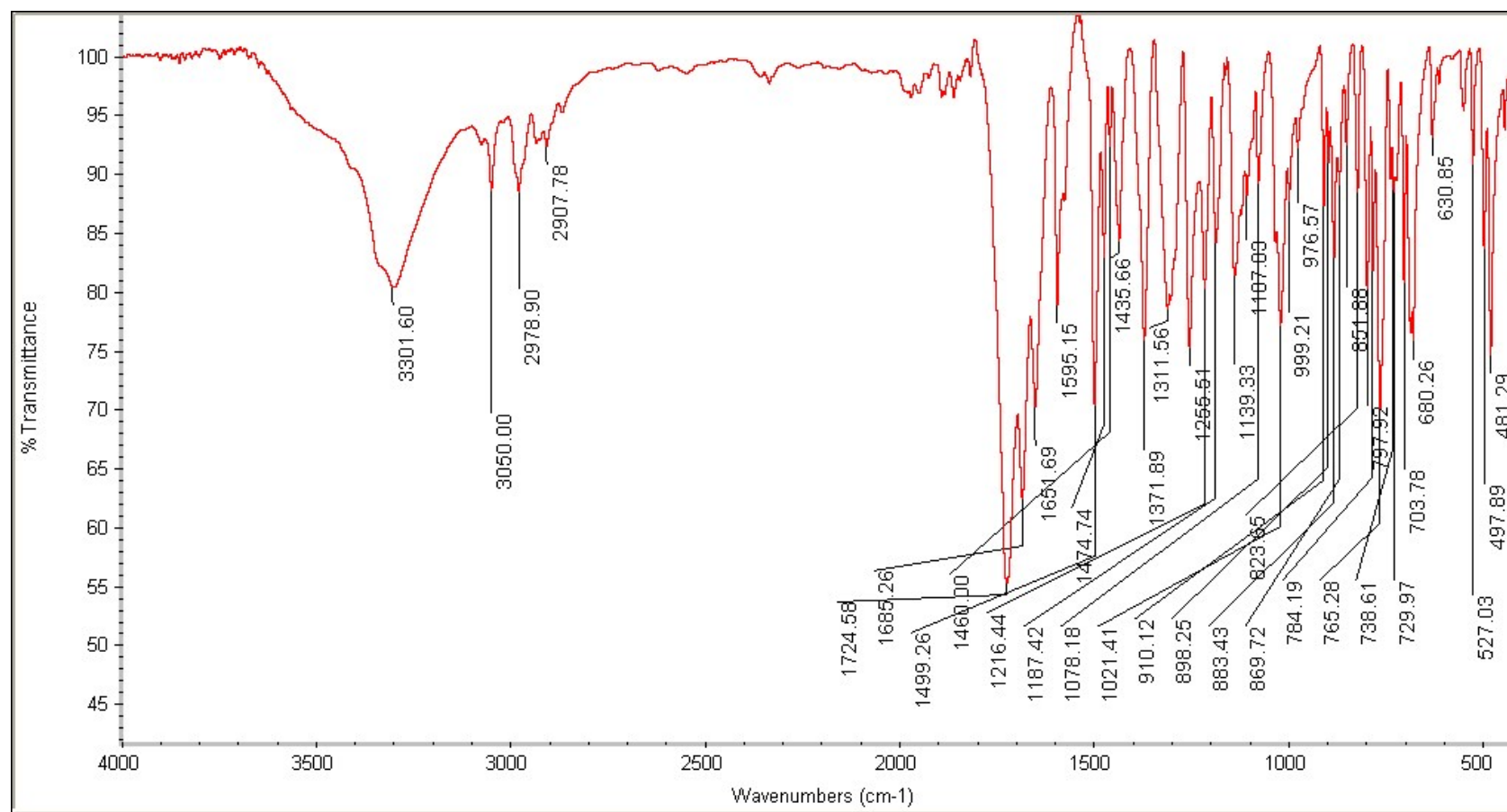


Fig. 13 FT-IR spectrum of Ethyl 2-(3-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4e**).

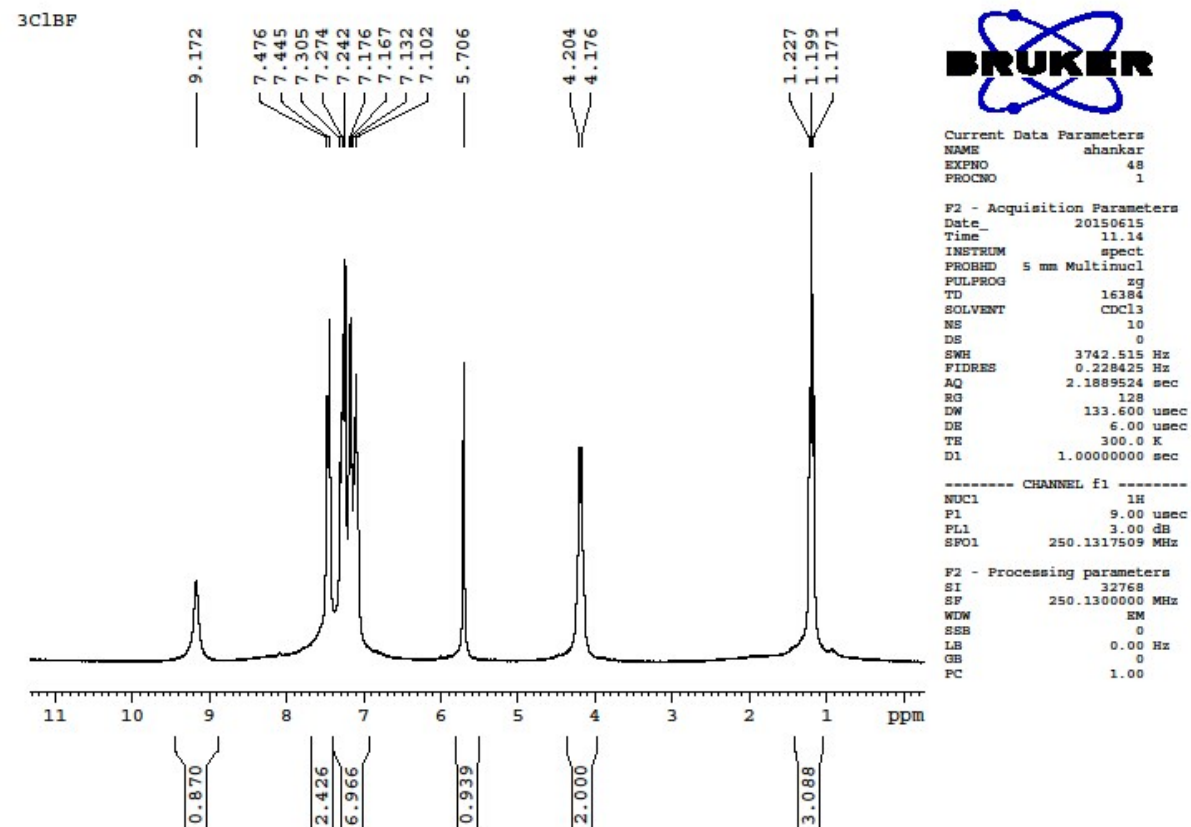
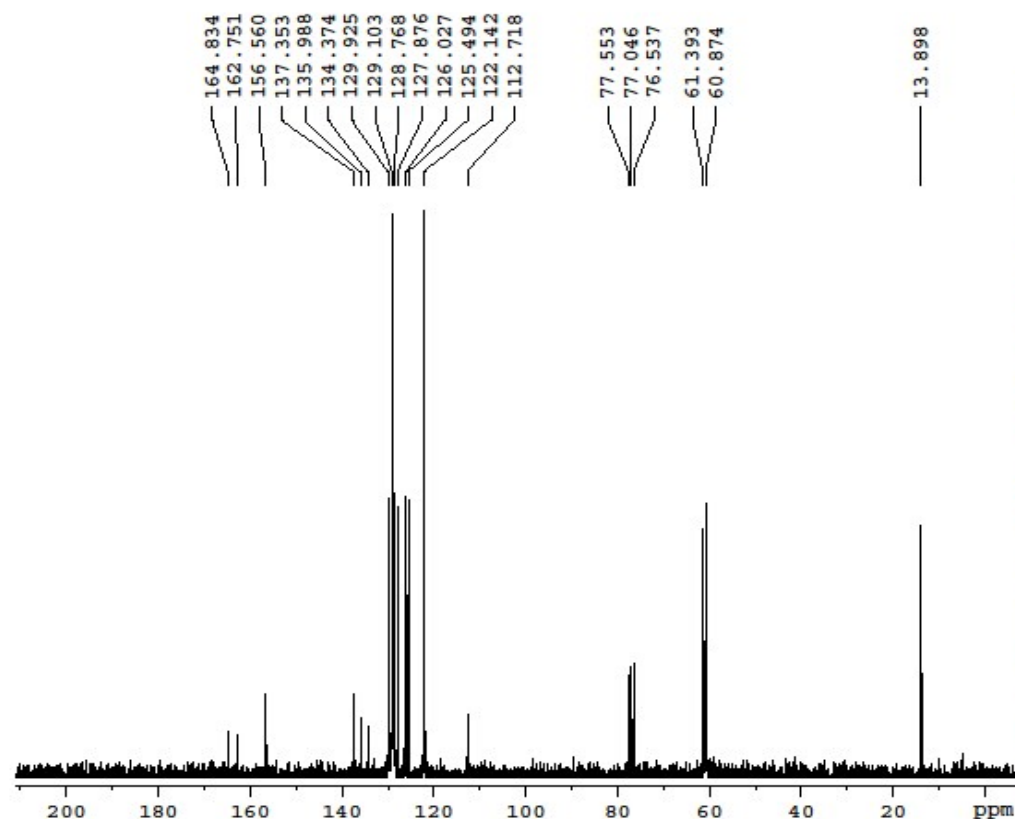


Fig. 14 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 2-(3-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4e**).

3CLBF



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Fig. 15 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 2-(3-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (4e).

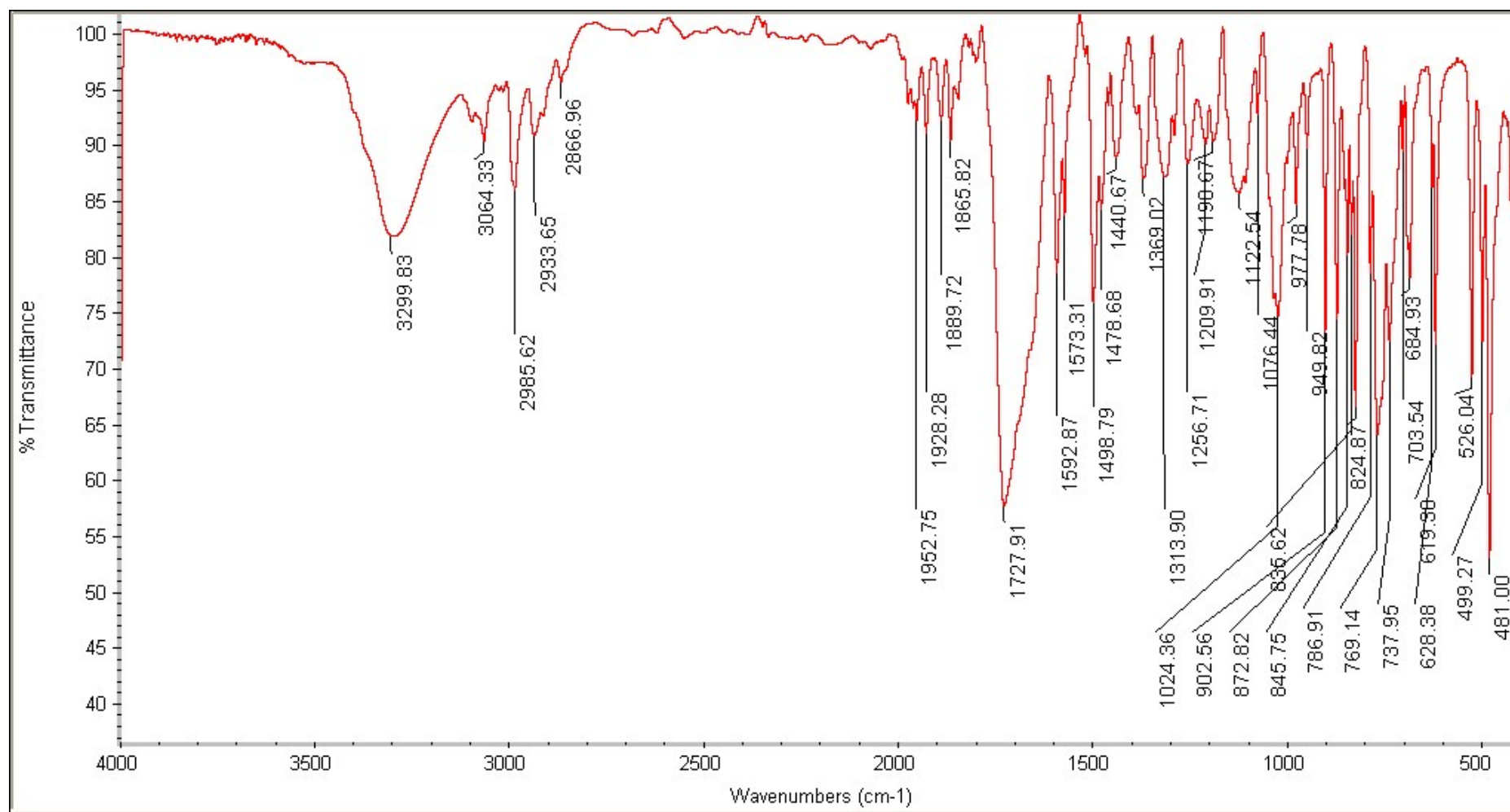


Fig. 16 FT-IR spectrum of Ethyl 2-(2-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4f**).

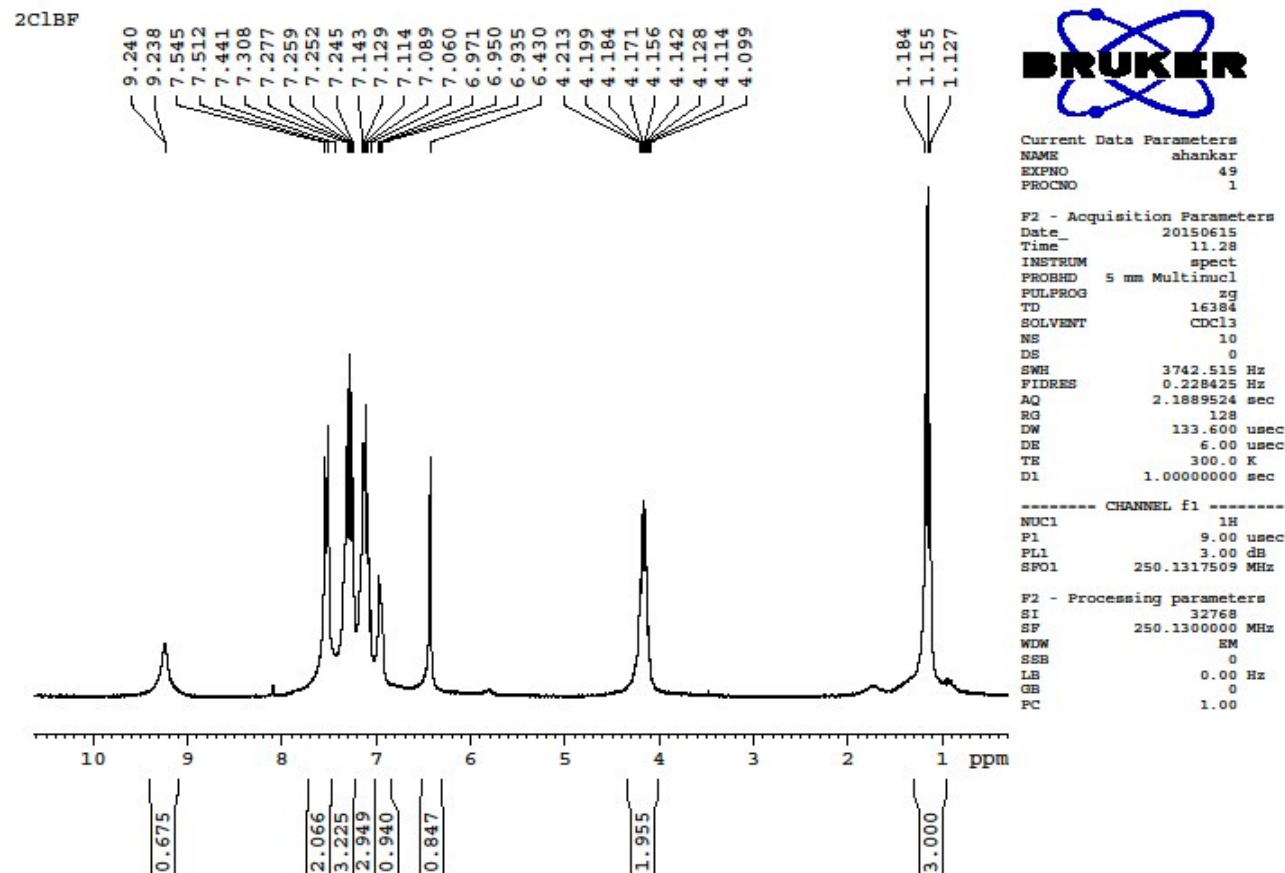


Fig. 17 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 2-(2-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4f**).

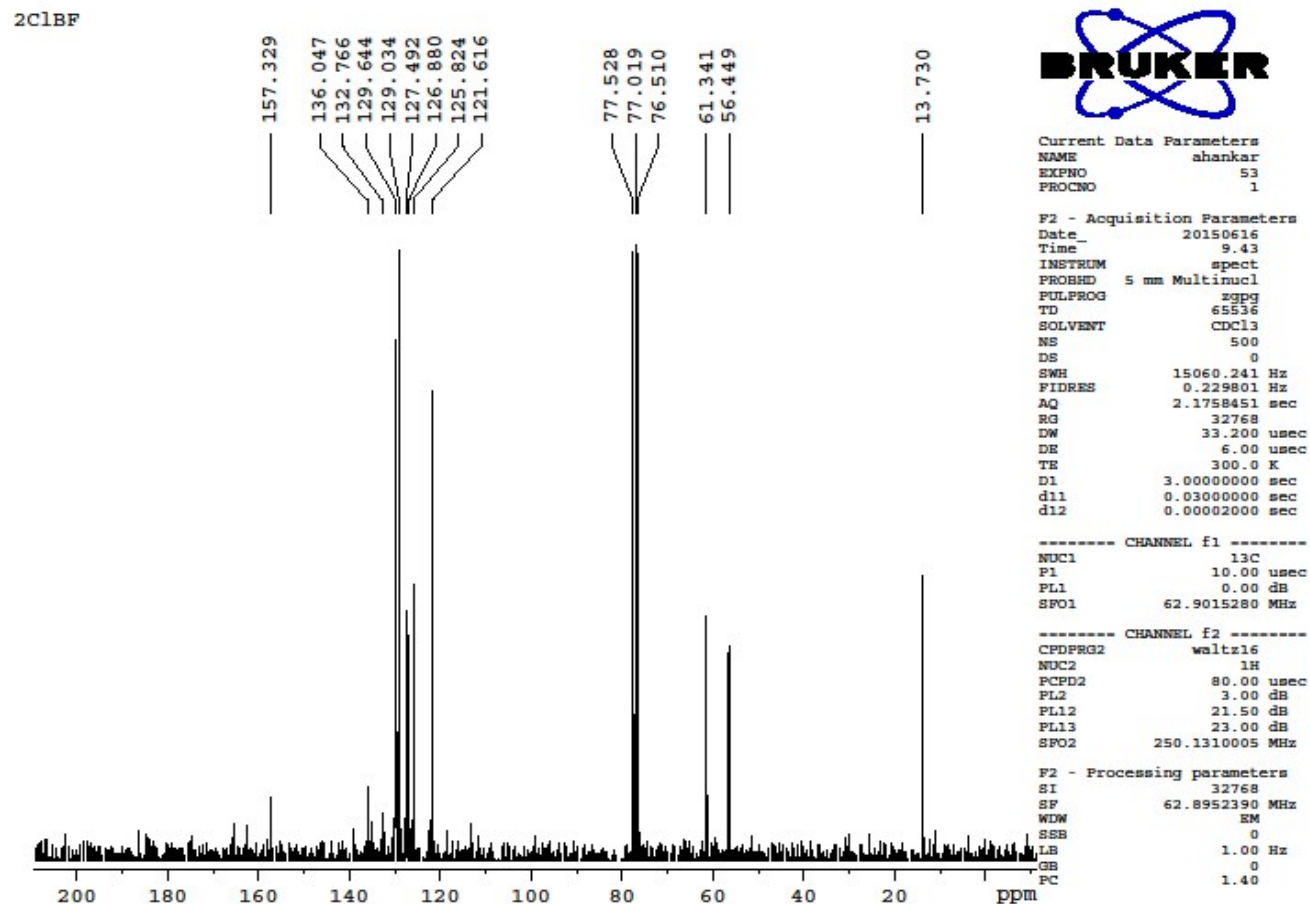


Fig. 18 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 2-(2-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4f**).

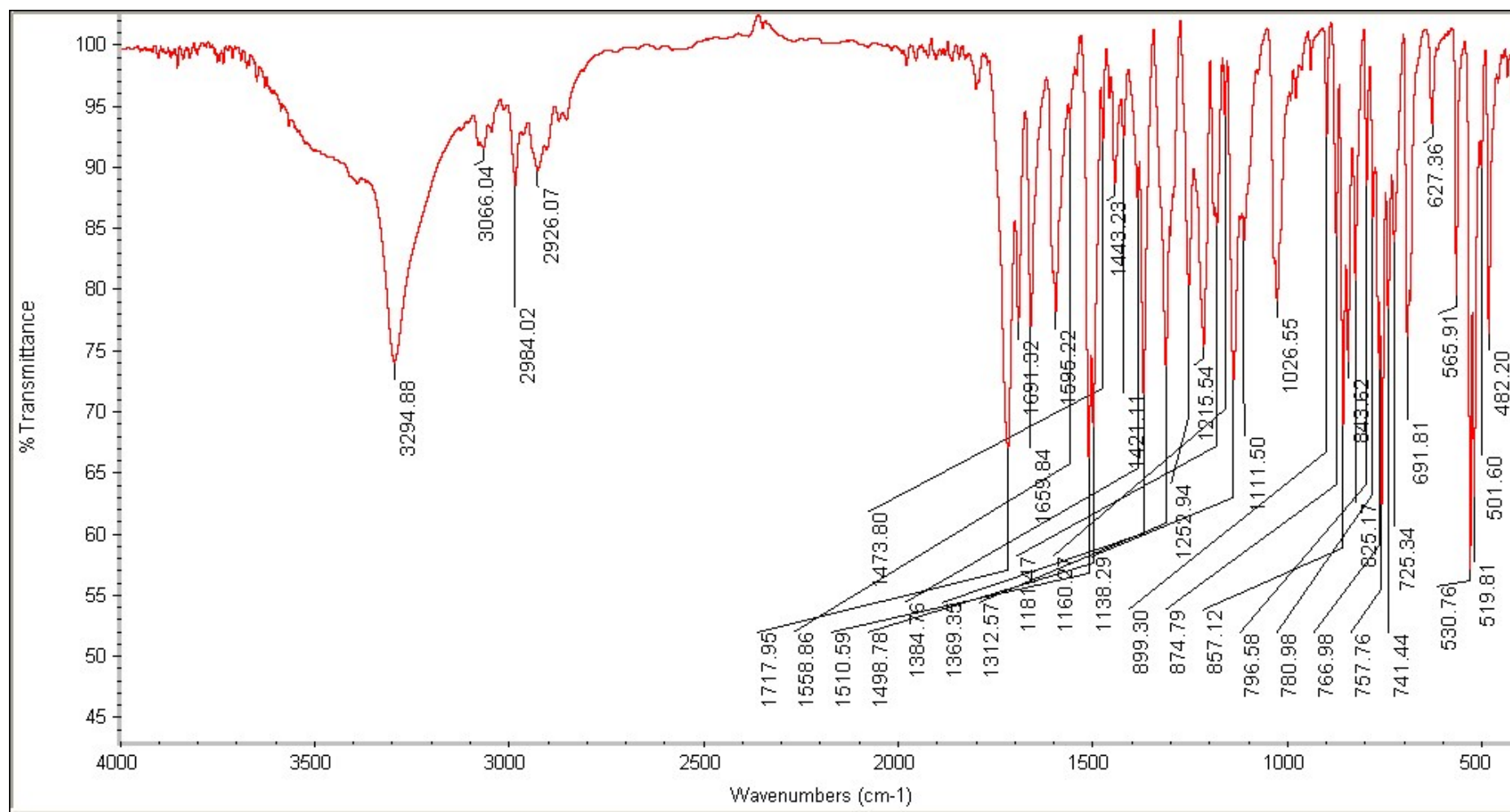


Fig. 19 FT-IR spectrum of Ethyl 2-(4-fluorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4g**).

4FBB

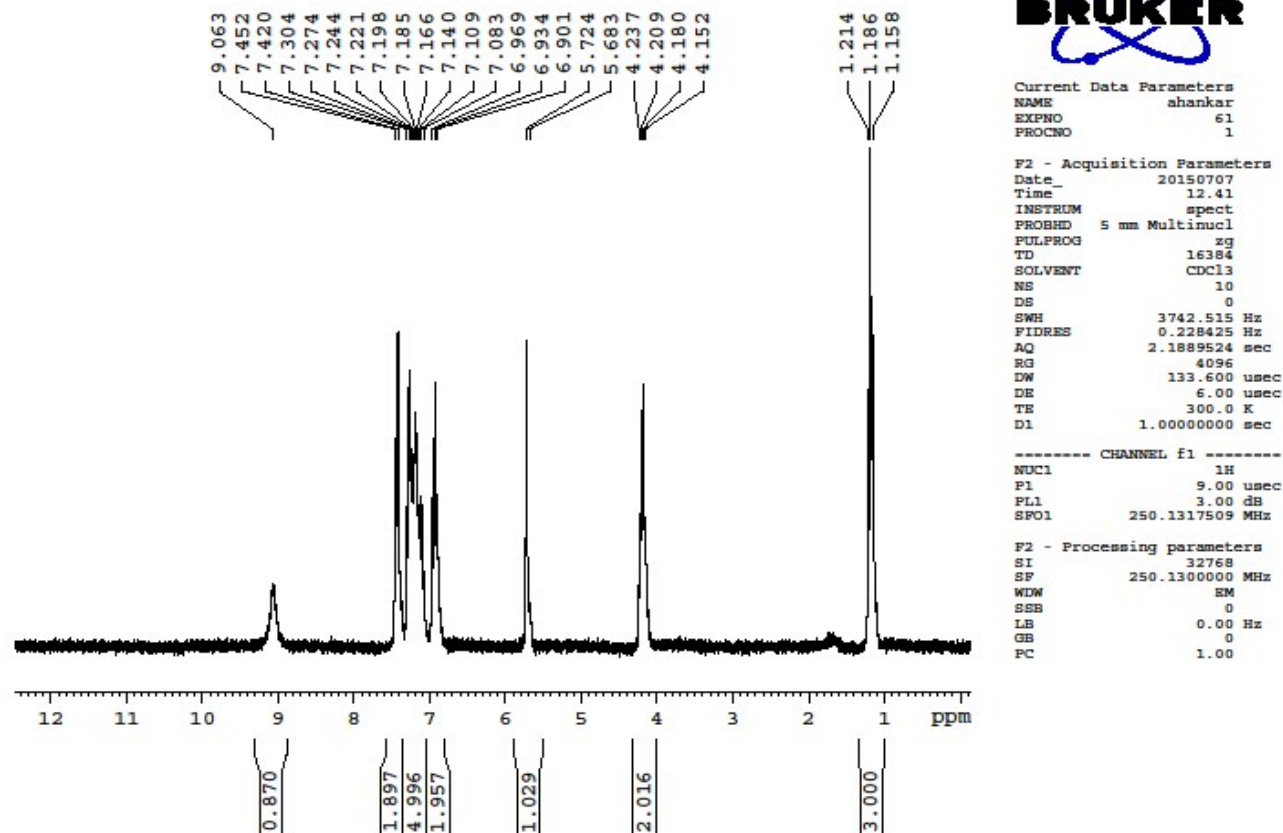
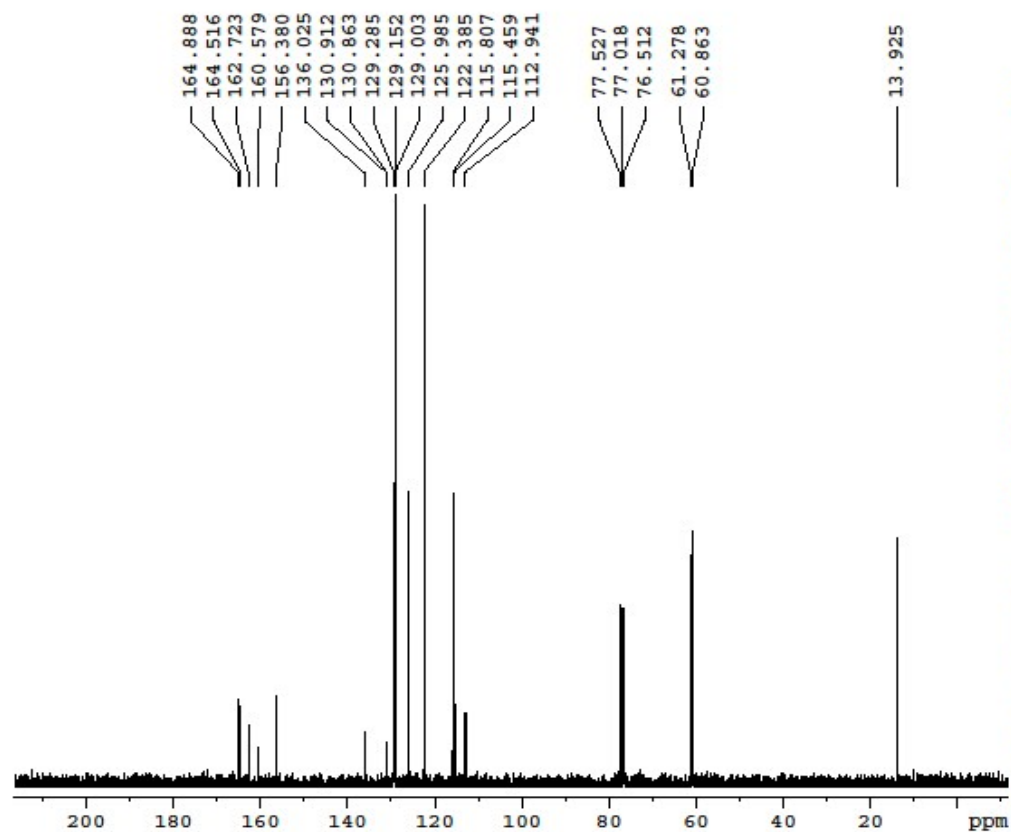


Fig. 20 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 2-(4-fluorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4g**).

4FBF



Current Data Parameters
NAME ahankar
EXPNO 64
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150713
Time 12.56
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 306
DS 0
SWH 15060.241 Hz
FIDRES 0.229801 Hz
AQ 2.1758451 sec
RG 32768
DW 33.200 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

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NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 62.9015280 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 21.50 dB
PL13 23.00 dB
SFO2 250.1310005 MHz

F2 - Processing parameters
SI 32768
SF 62.8952390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Fig. 21 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 2-(4-fluorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (4g).

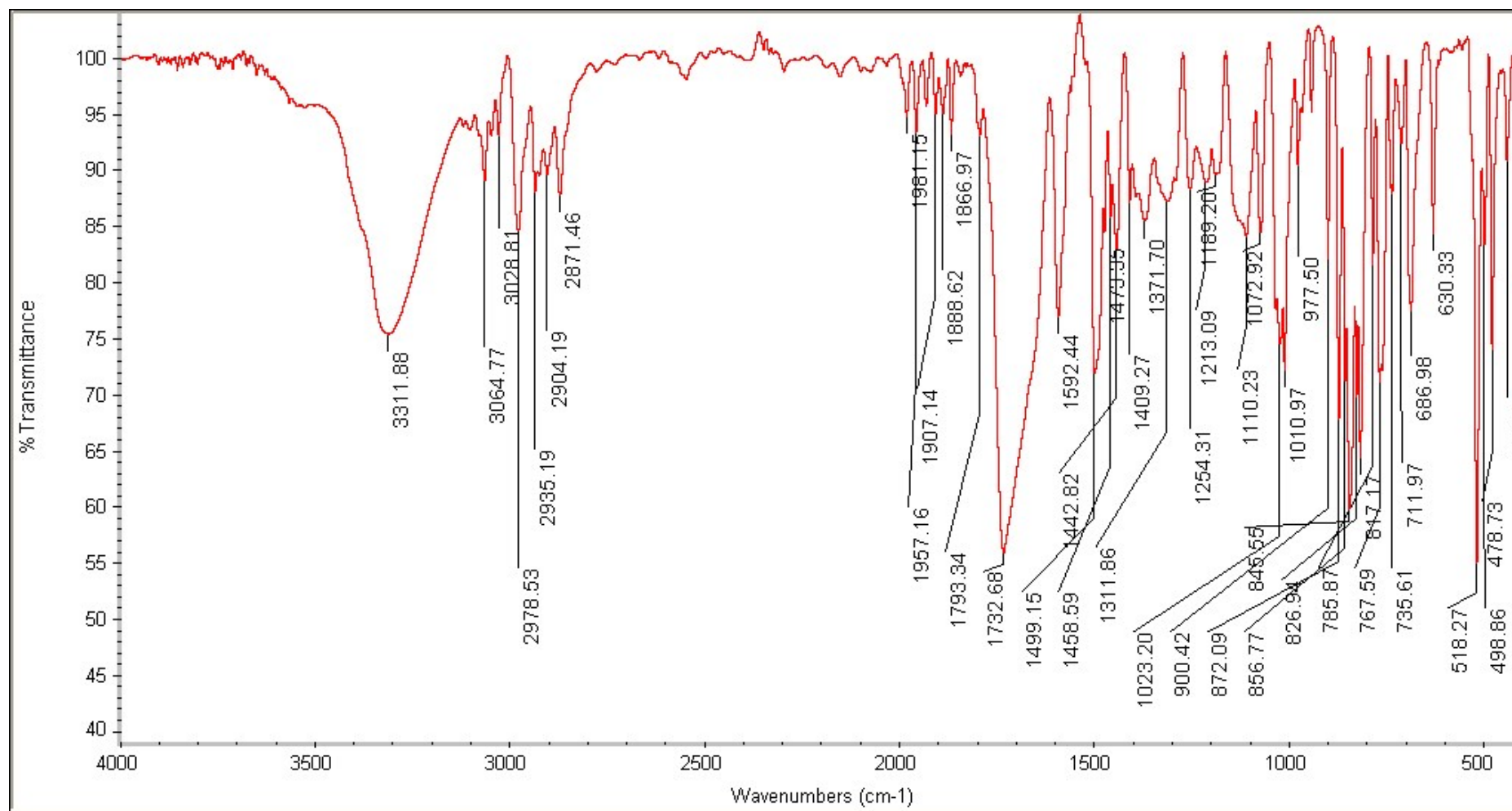


Fig. 22 FT-IR spectrum of Ethyl 2-(4-bromophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4h**).

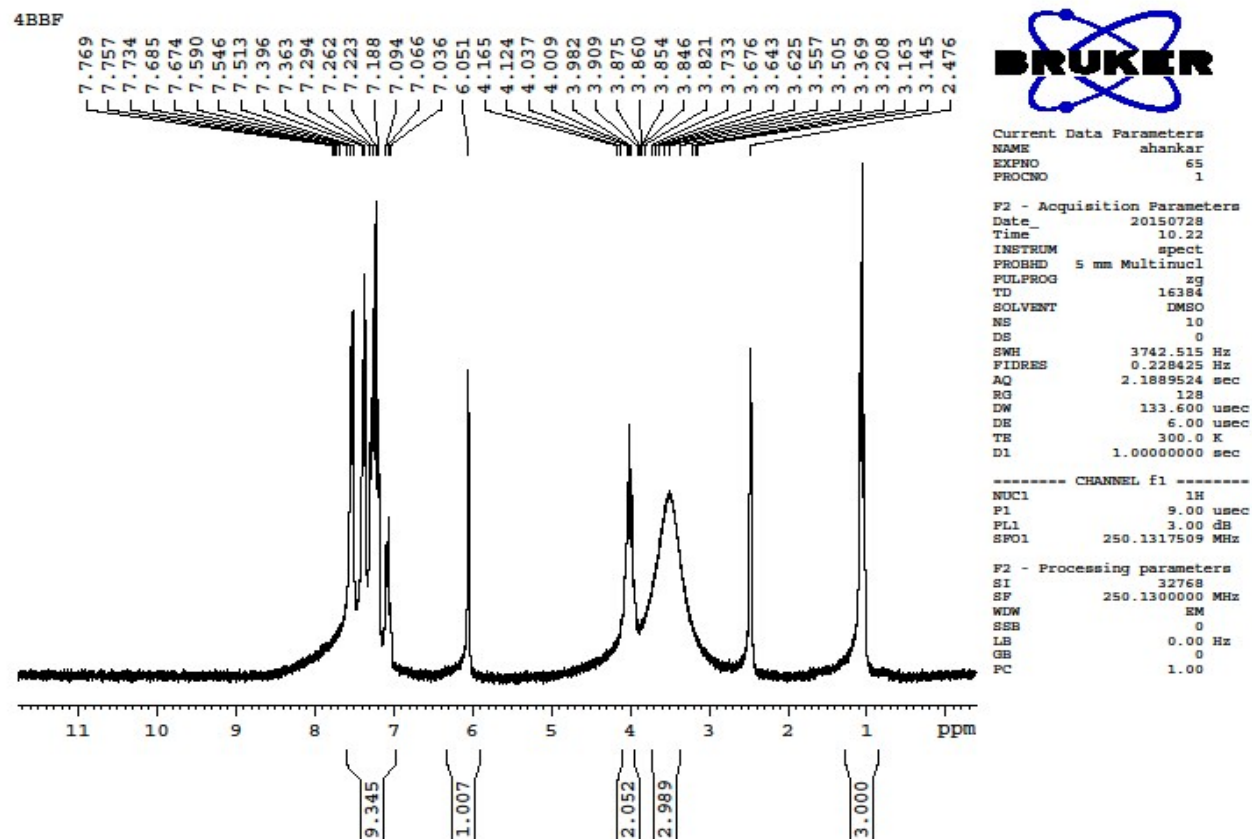


Fig. 23 ^1H NMR (250.13 MHz, DMSO-d_6) spectrum of Ethyl 2-(4-bromophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4h**).

4BBF

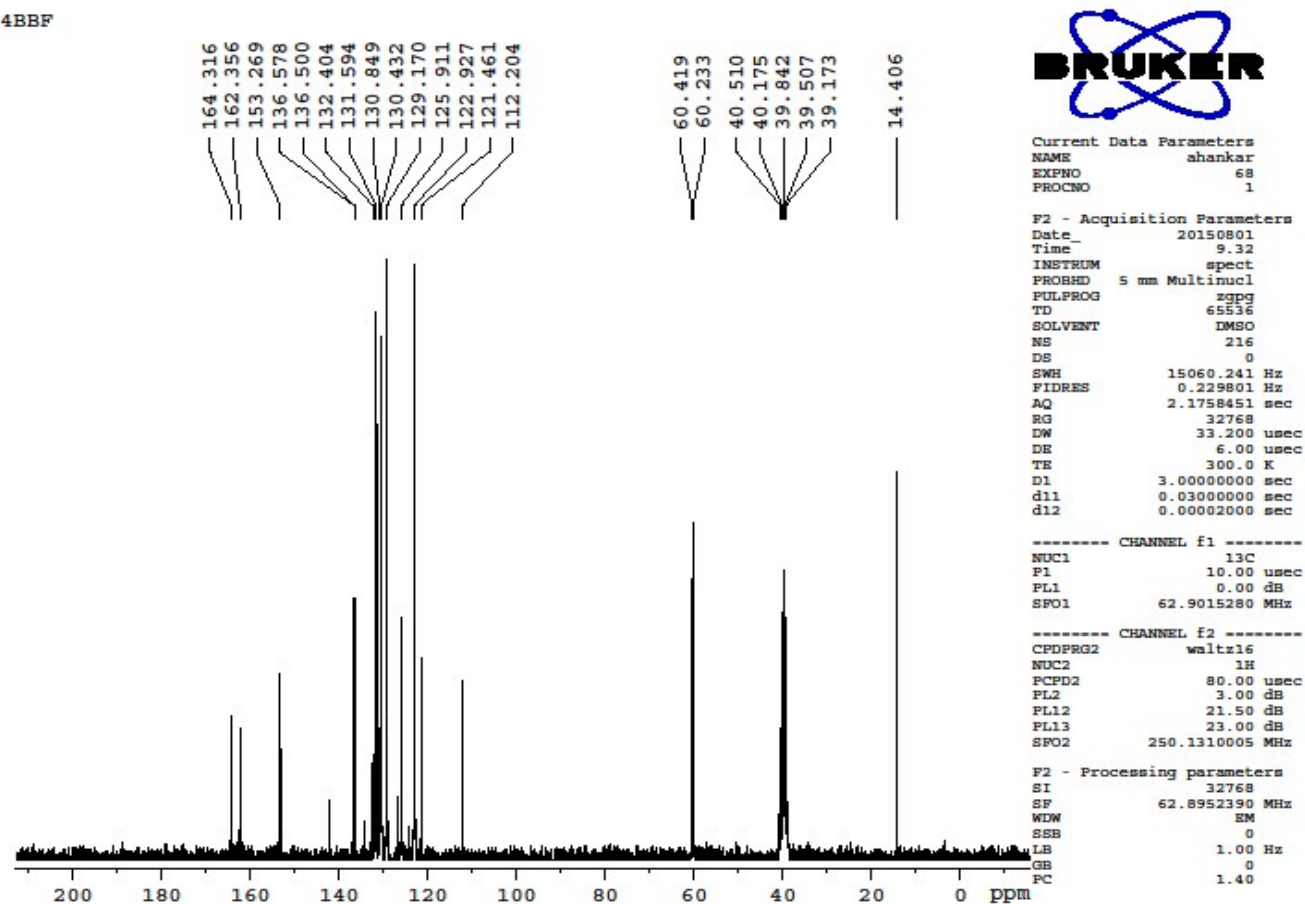


Fig. 24 ^{13}C NMR (62.90 MHz, DMSO- d_6) spectrum of Ethyl 2-(4-bromophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4h**).

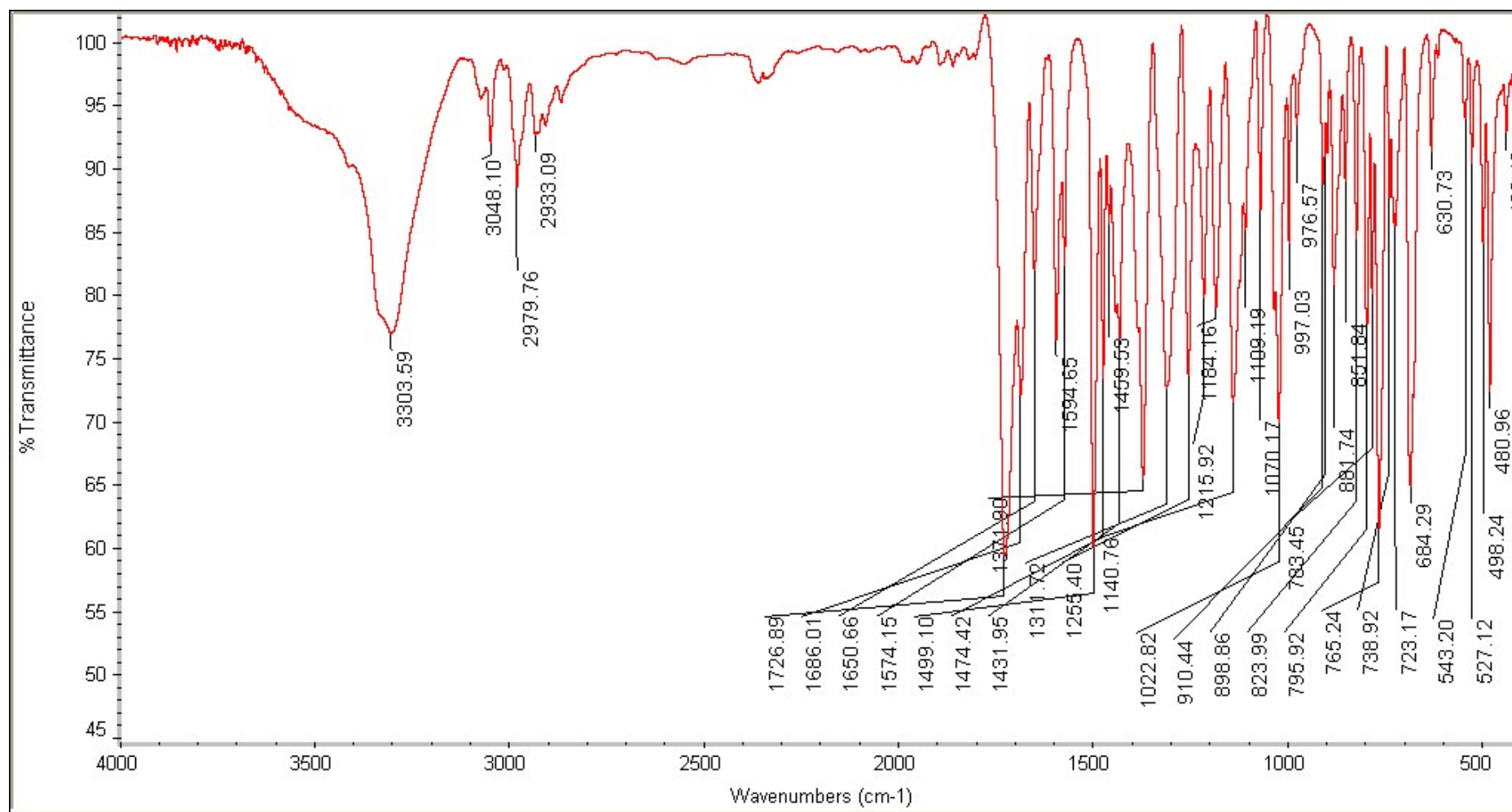


Fig. 25 FT-IR spectrum of Ethyl 2-(3-bromophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4i**).

3BBF

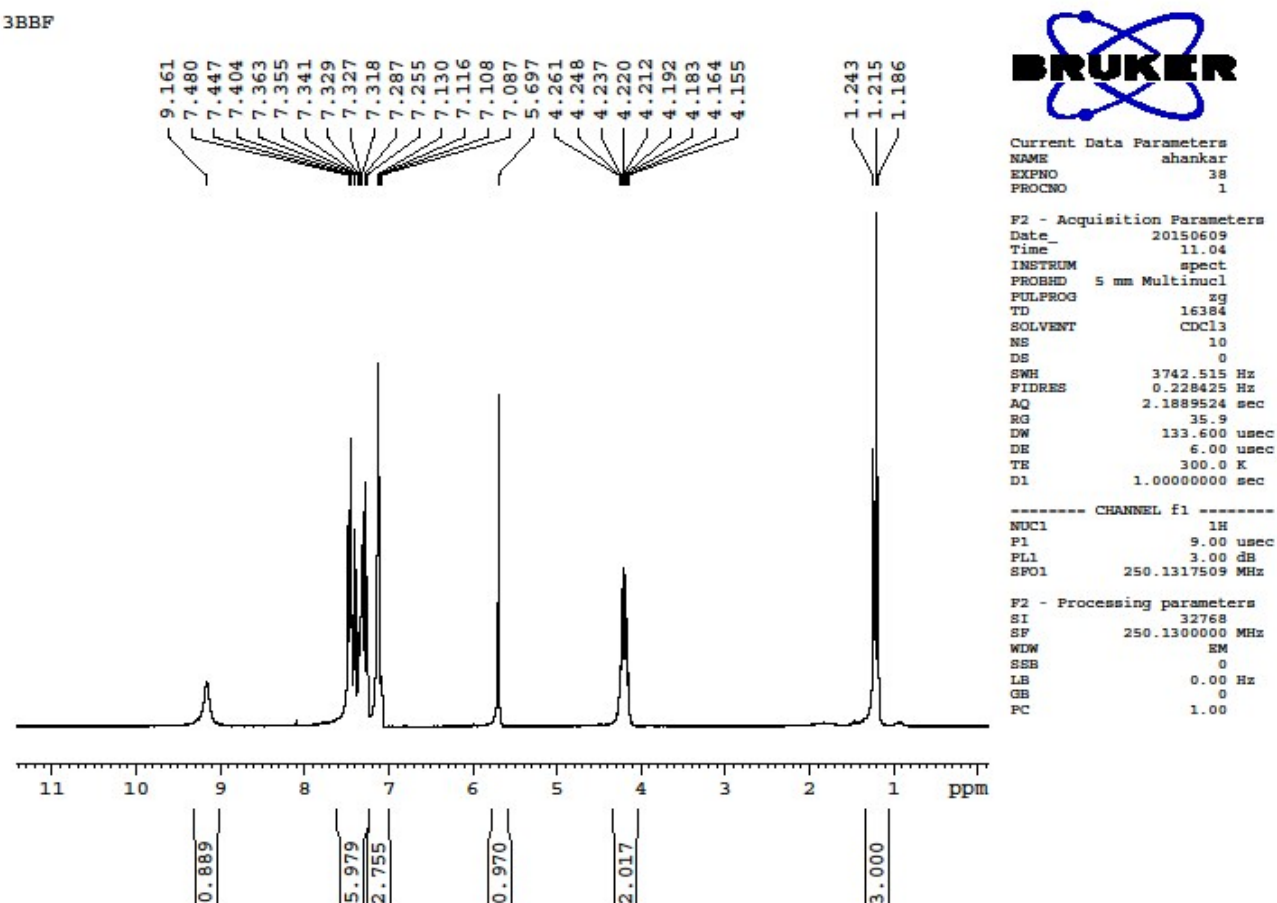


Fig. 26 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 2-(3-bromophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4i**).

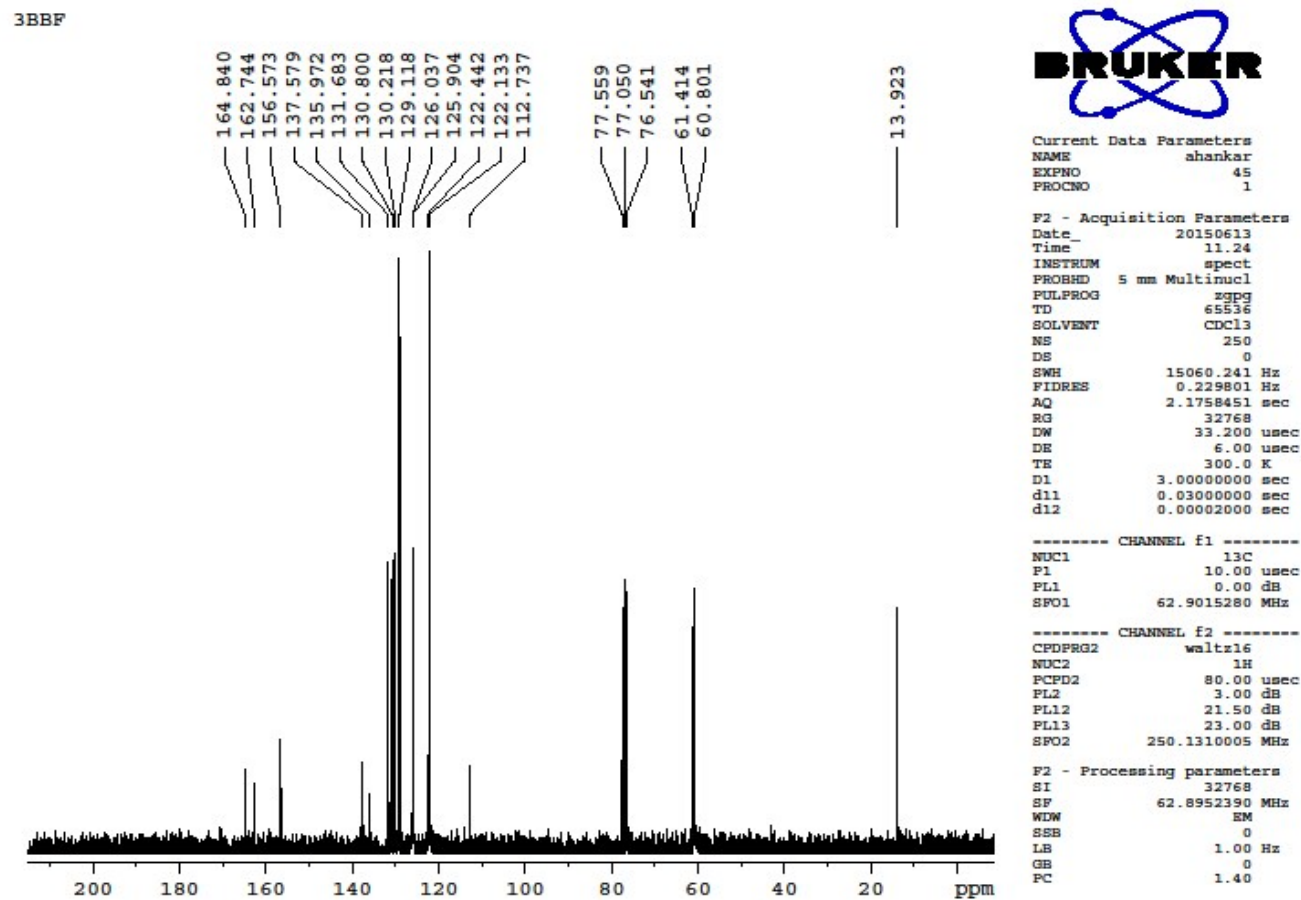


Fig. 27 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 2-(4-bromophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4i**).

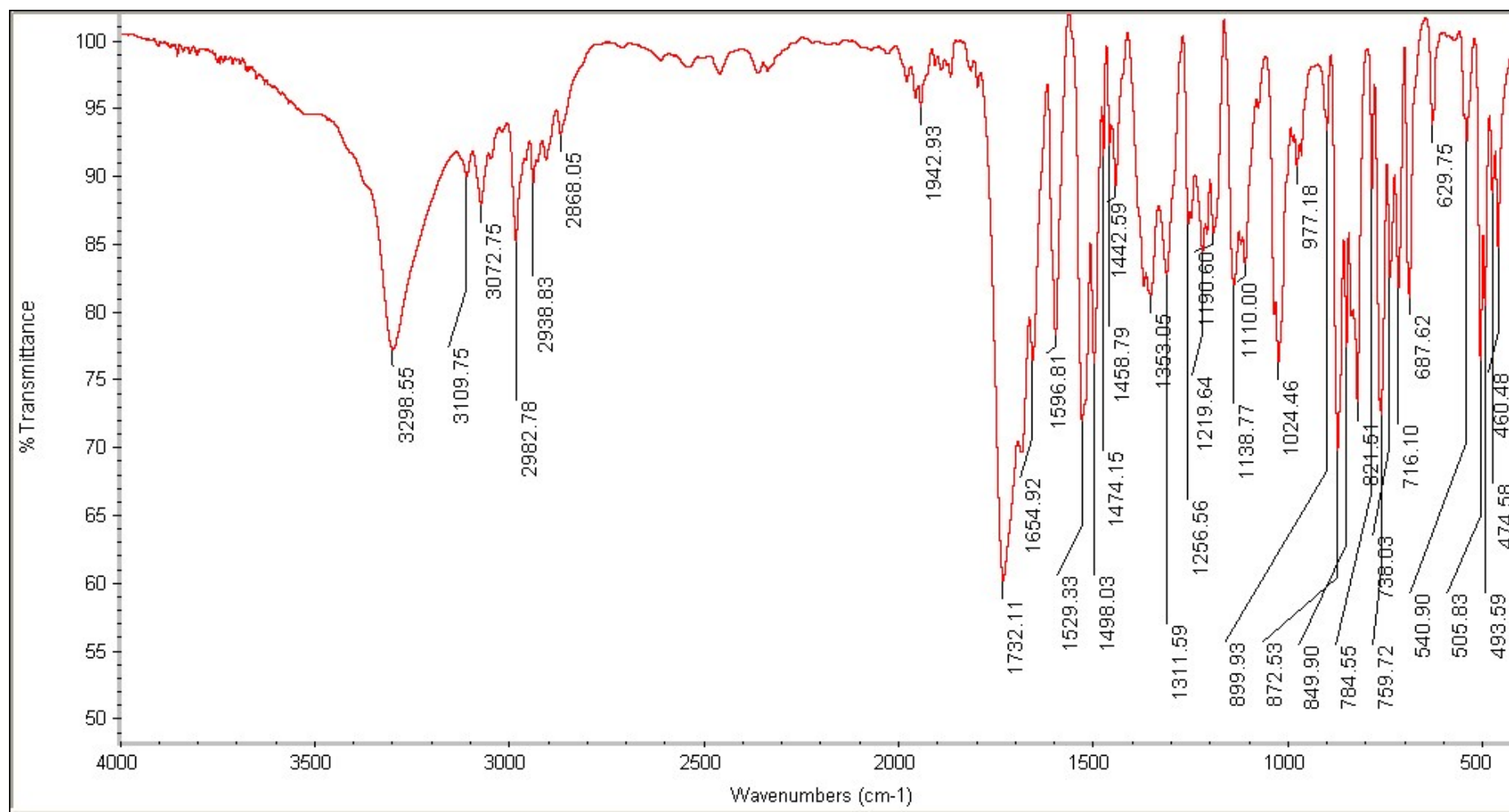
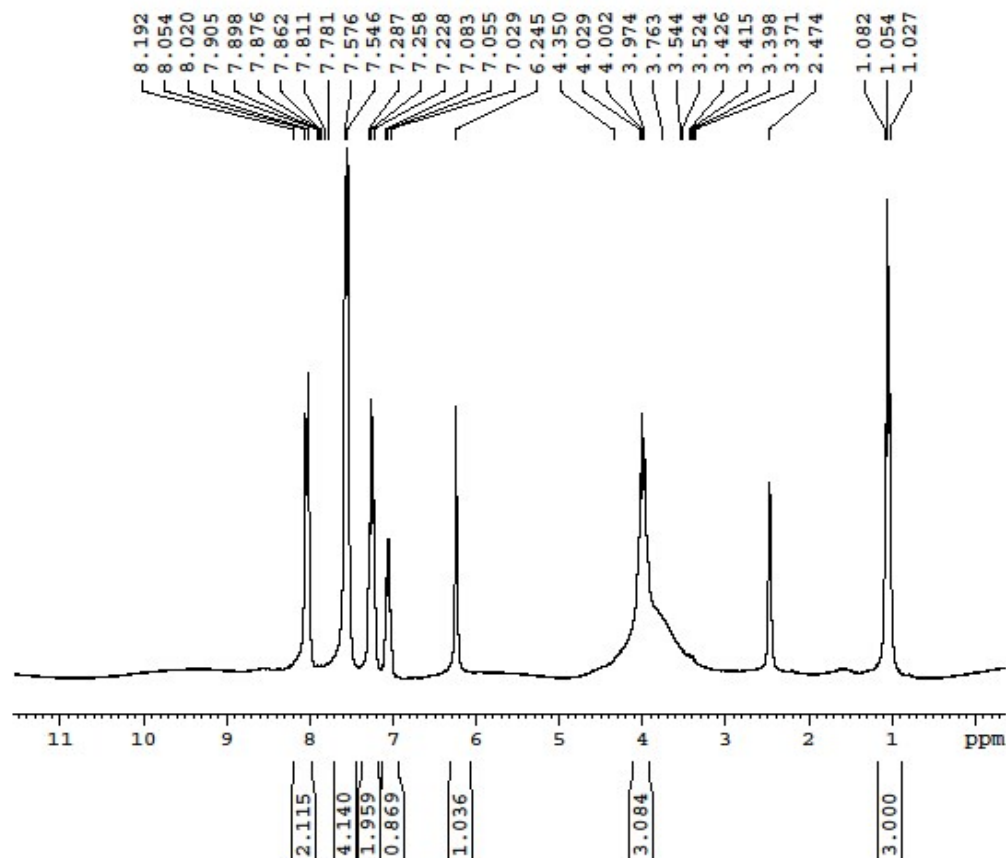


Fig. 28 FT-IR spectrum of Ethyl 2-(4-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4j**).

4NBF



Current Data Parameters
NAME ahankar
EXPNO 66
PROCNO 1

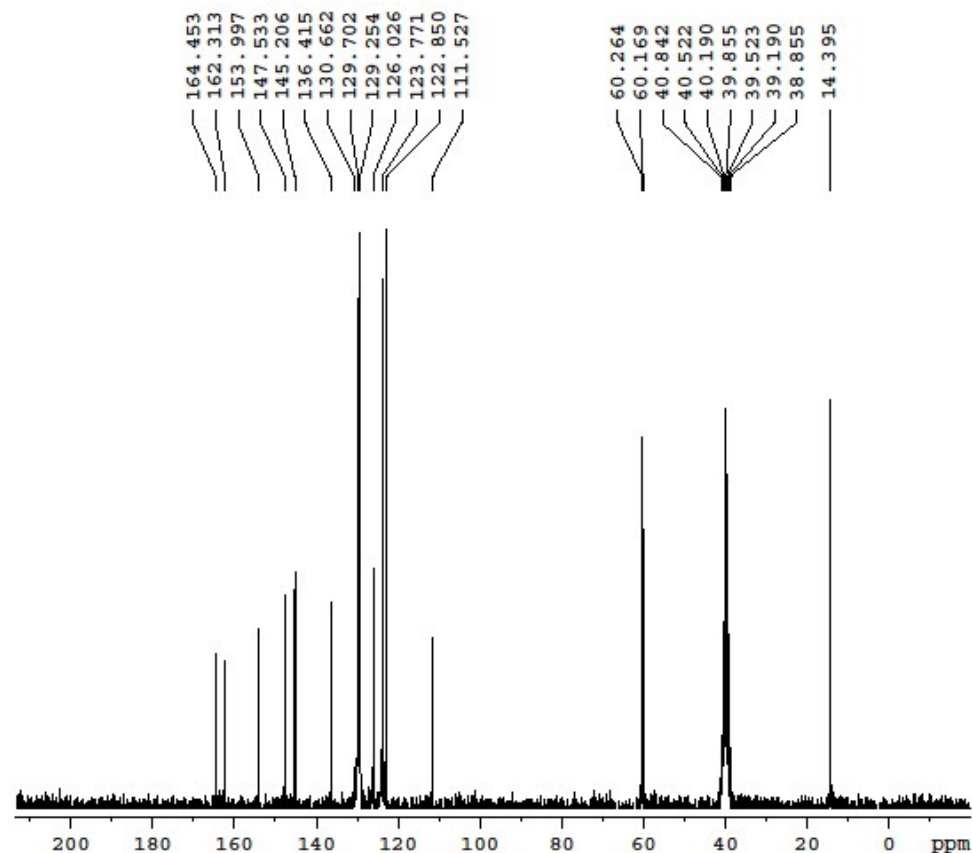
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PULPROG zg
TD 16384
SOLVENT DMSO
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DS 0
SWH 3742.515 Hz
FIDRES 0.228425 Hz
AQ 2.1889524 sec
RG 912.3
DW 133.600 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec

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P1 9.00 usec
PL1 3.00 dB
SFO1 250.1317509 MHz

F2 - Processing parameters
SI 32768
SF 250.1300000 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Fig. 29 ^1H NMR (250.13 MHz, DMSO-d_6) spectrum of Ethyl 2-(4-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4j**).

4NBF



Current Data Parameters
NAME ahankar
EXPNO 52
PROCNO 1

F2 - Acquisition Parameters
Date_ 20150616
Time 8.57
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PROBHD 5 mm Multinucl
PULPROG zgpg
TD 65536
SOLVENT DMSO
NS 213
DS 0
SWH 15060.241 Hz
FIDRES 0.229801 Hz
AQ 2.1758451 sec
RG 32768
DW 33.200 usec
DE 6.00 usec
TE 300.0 K
D1 3.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

----- CHANNEL f1 -----
NUC1 13C
P1 10.00 usec
PL1 0.00 dB
SFO1 62.9015280 MHz

----- CHANNEL f2 -----
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 21.50 dB
PL13 23.00 dB
SFO2 250.1310005 MHz

F2 - Processing parameters
SI 32768
SF 62.8952390 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Fig. 30 ^{13}C NMR (62.90 MHz, DMSO- d_6) spectrum of Ethyl 2-(4-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4j**).

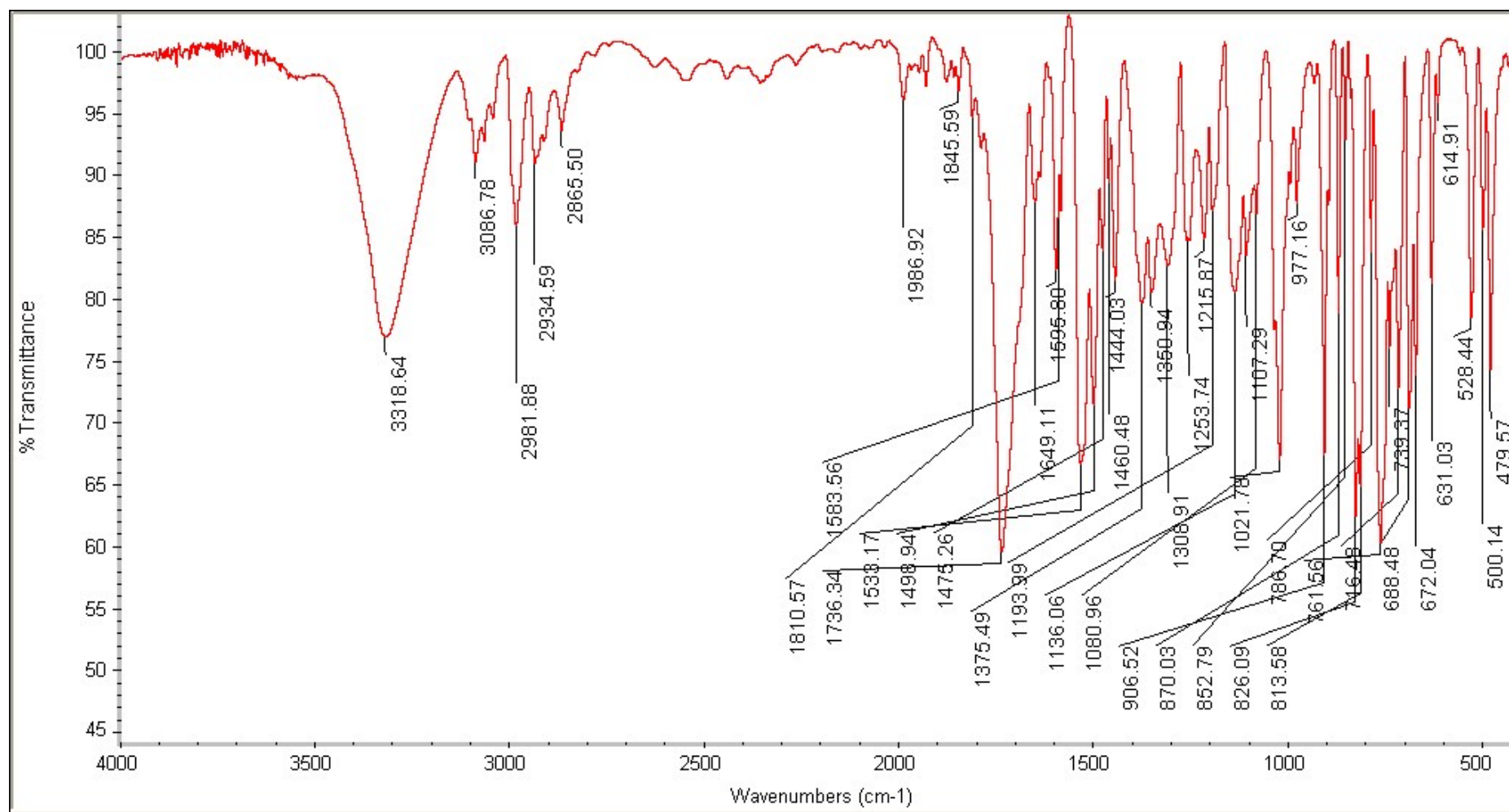


Fig. 31 FT-IR spectrum of Ethyl 2-(3-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4k**).

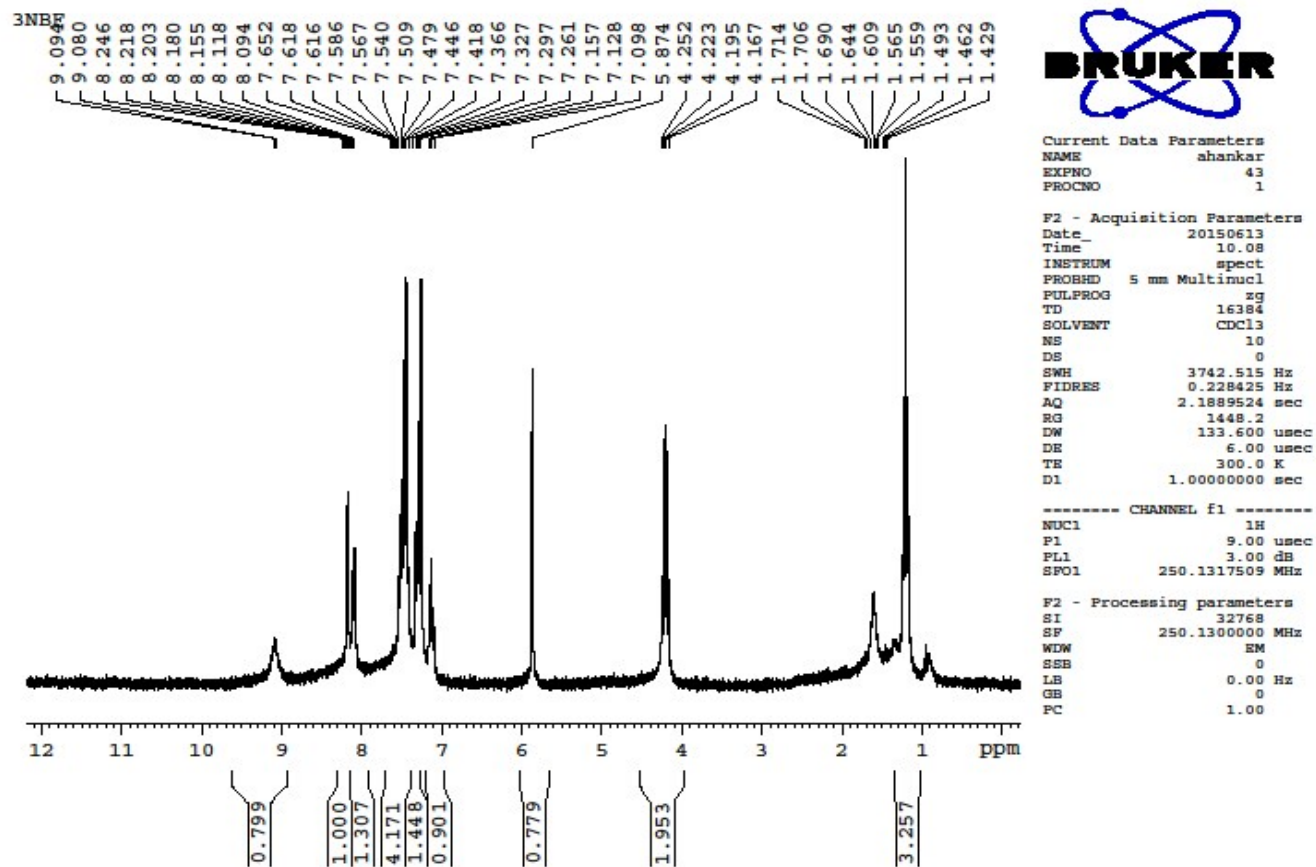


Fig. 32 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 2-(3-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4k**).

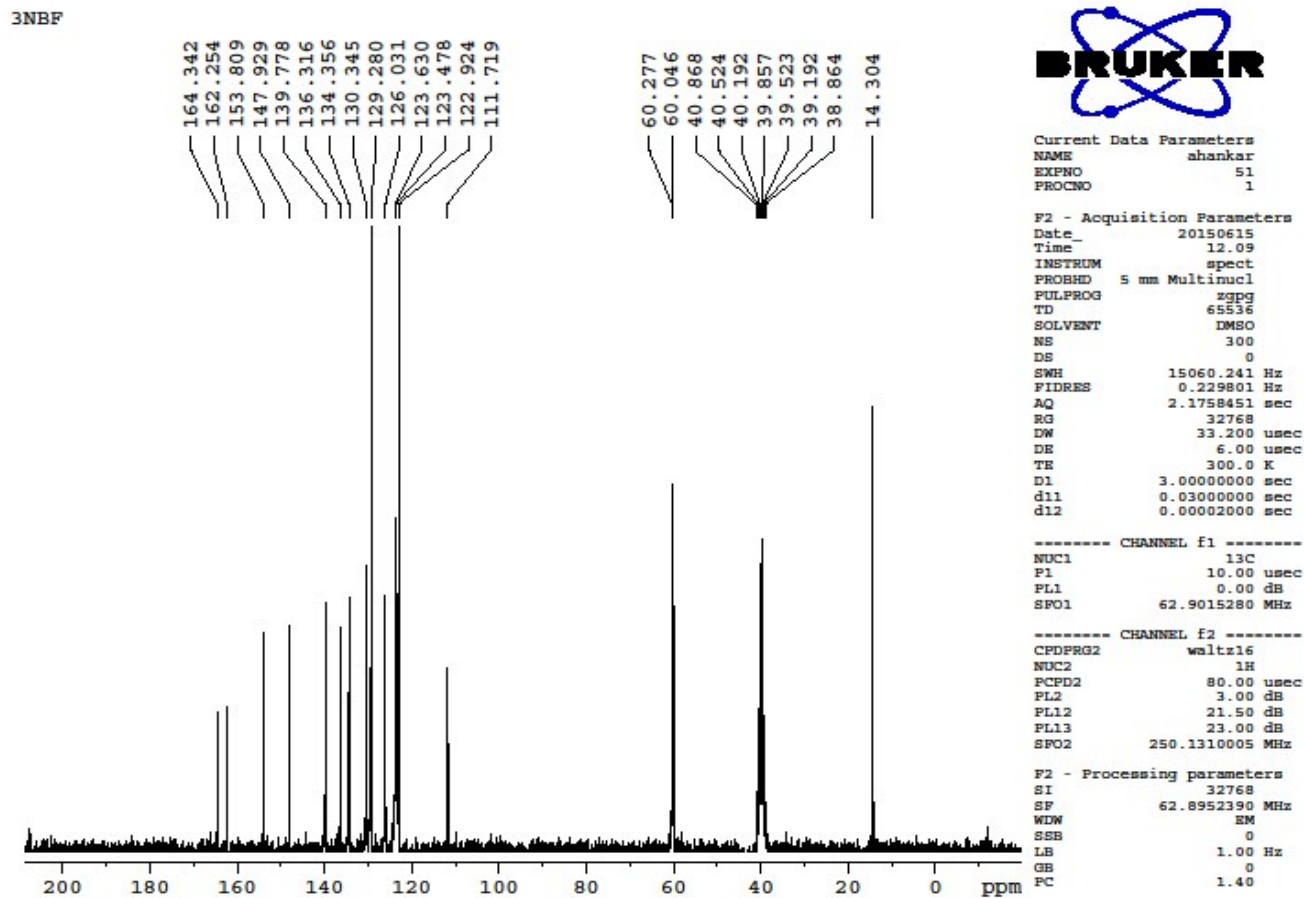


Fig. 33 ^{13}C NMR (62.90 MHz, DMSO-d_6) spectrum of Ethyl 2-(3-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4k**).

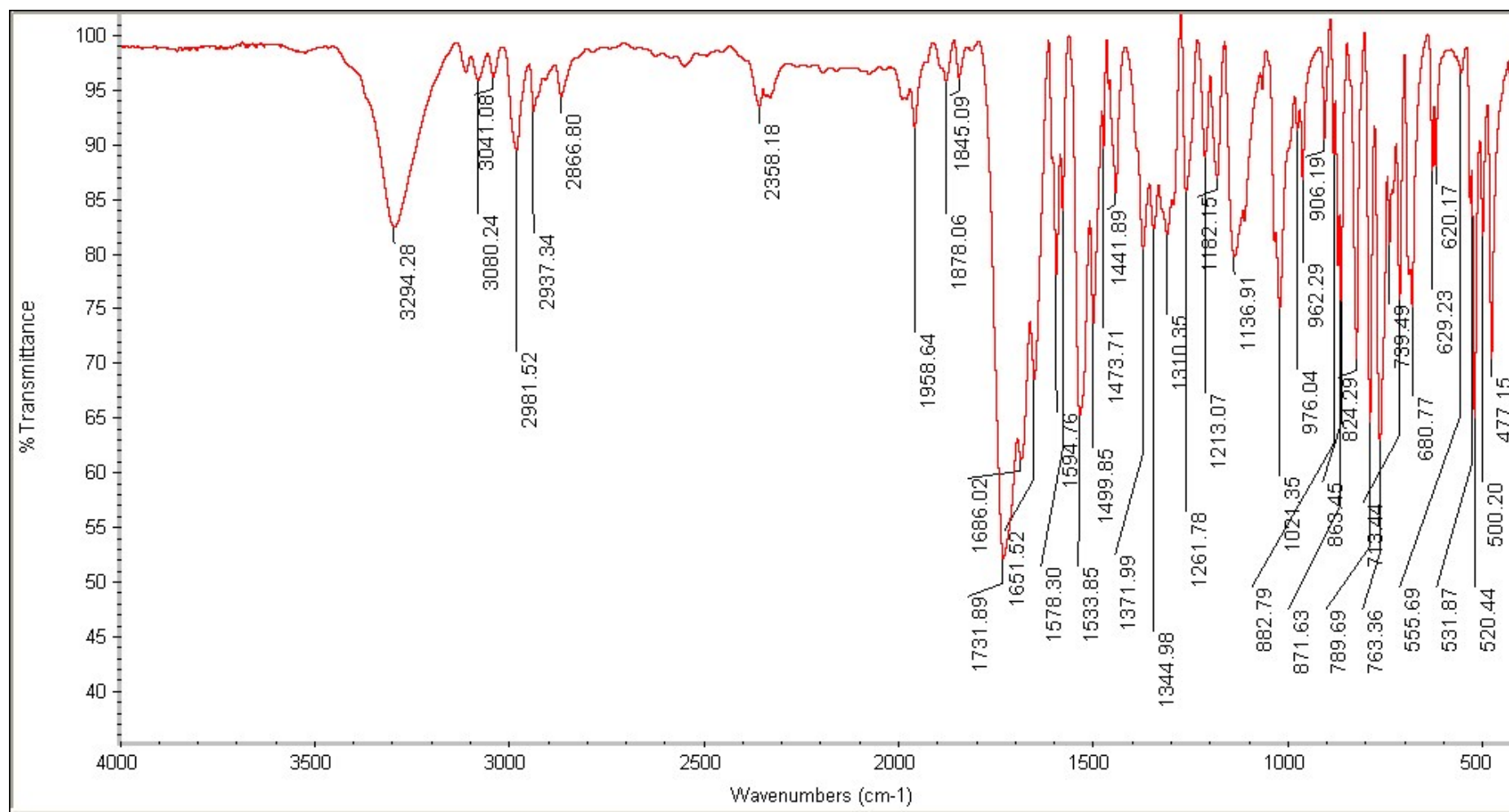


Fig. 34 FT-IR spectrum of Ethyl 2-(2-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4l**).

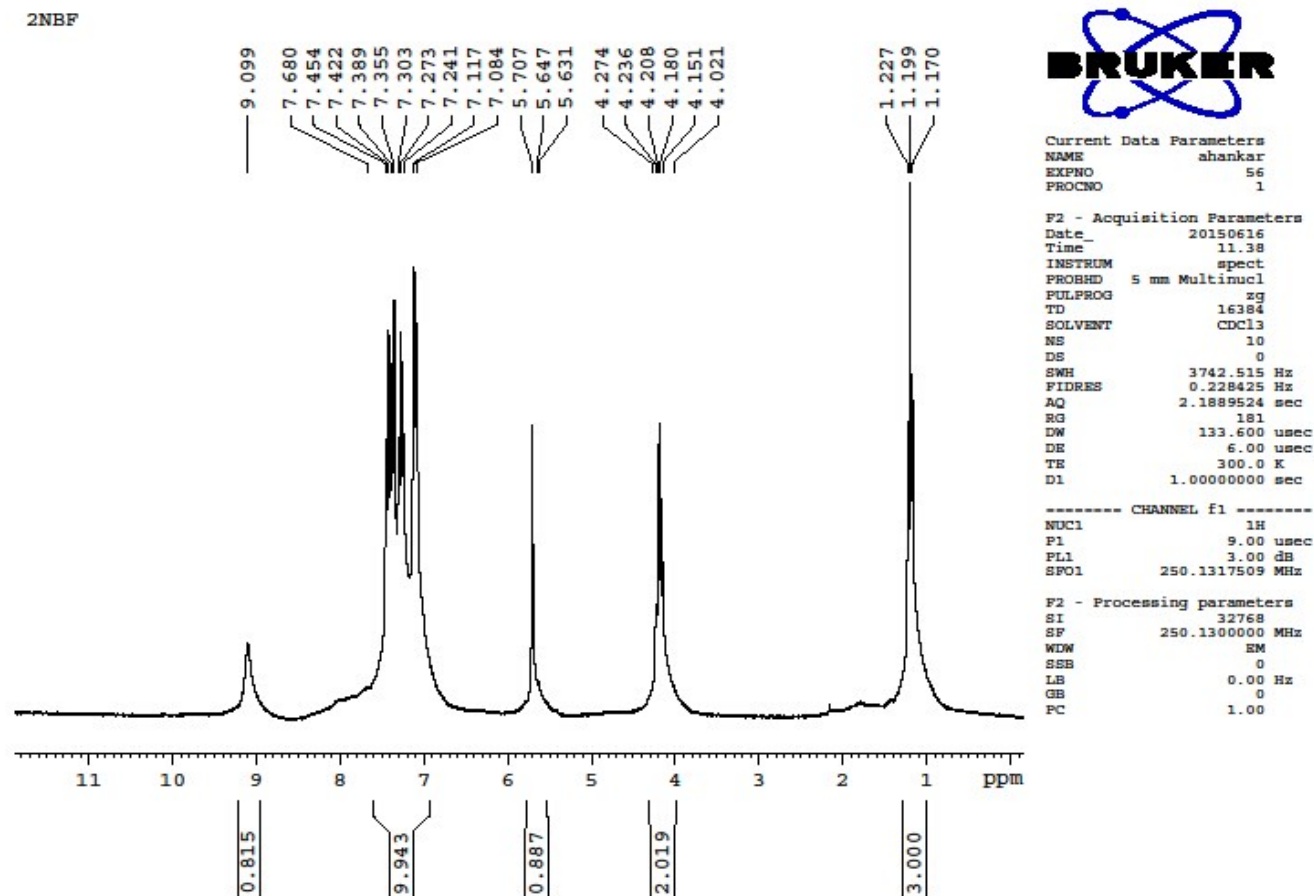


Fig. 35 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 2-(2-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4l**).

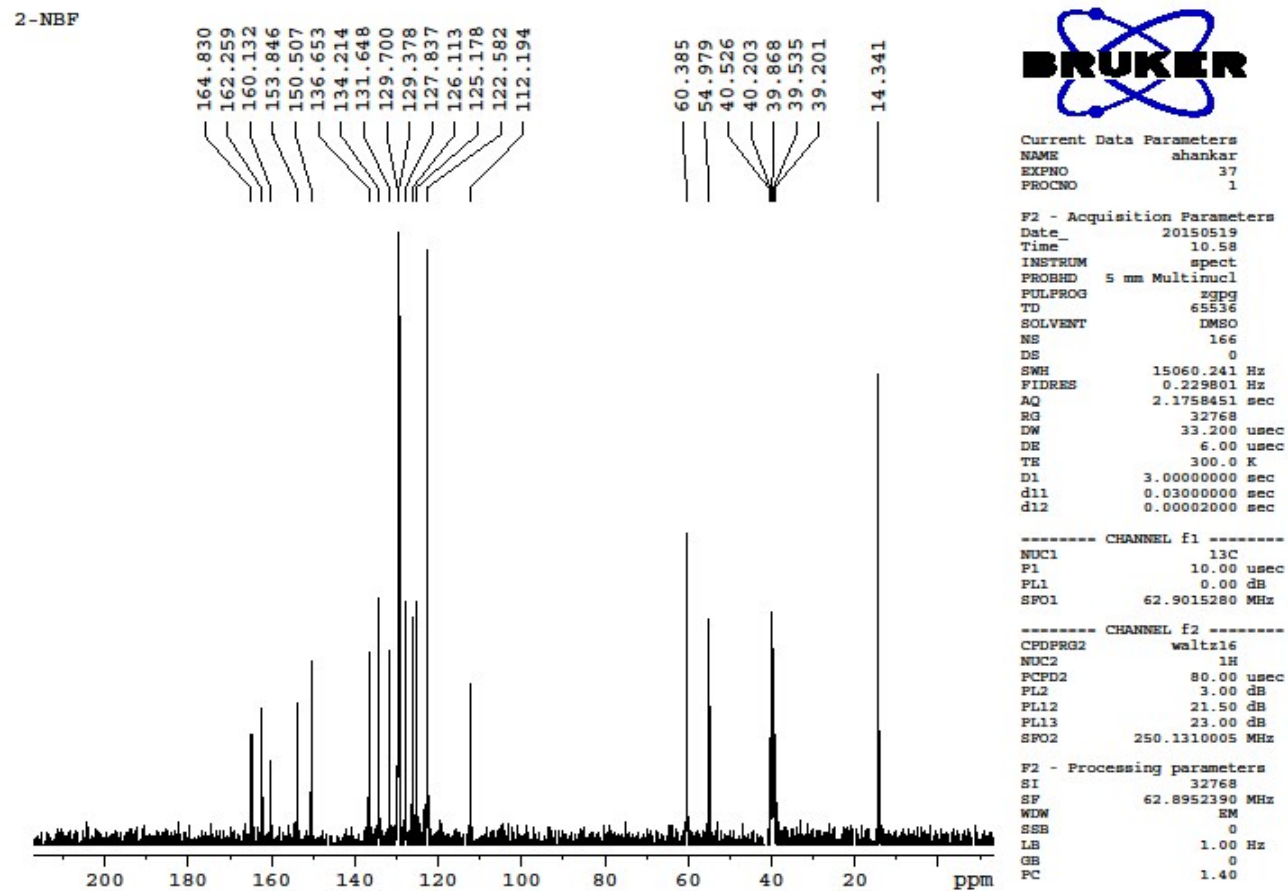


Fig. 36 ^{13}C NMR (62.90 MHz, DMSO-d_6) spectrum of Ethyl 2-(2-nitrophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4l**).

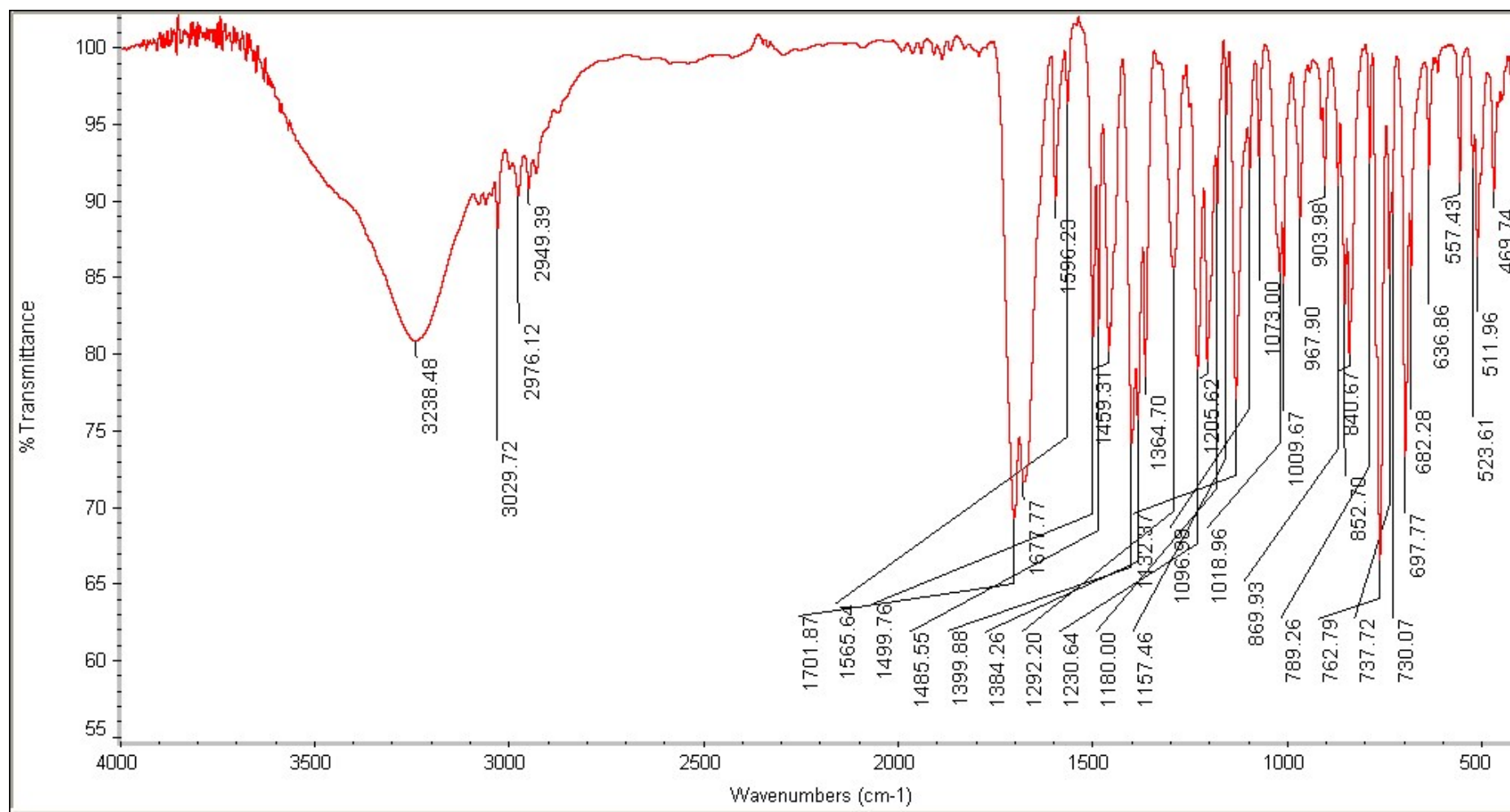


Fig. 37 FT-IR spectrum of Ethyl 2-([1,1'-biphenyl]-4-yl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4m**).

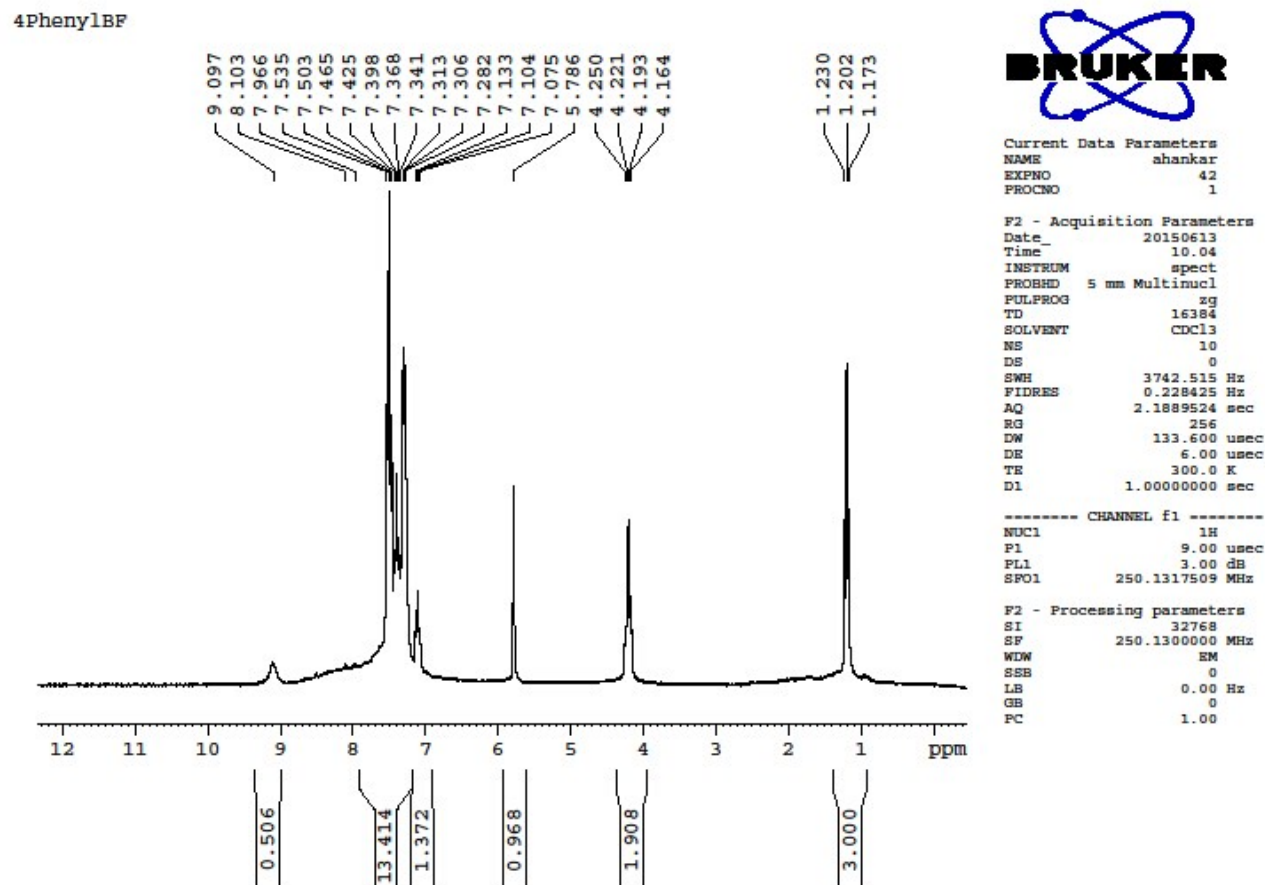


Fig. 38 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 2-([1,1'-biphenyl]-4-yl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4m**).

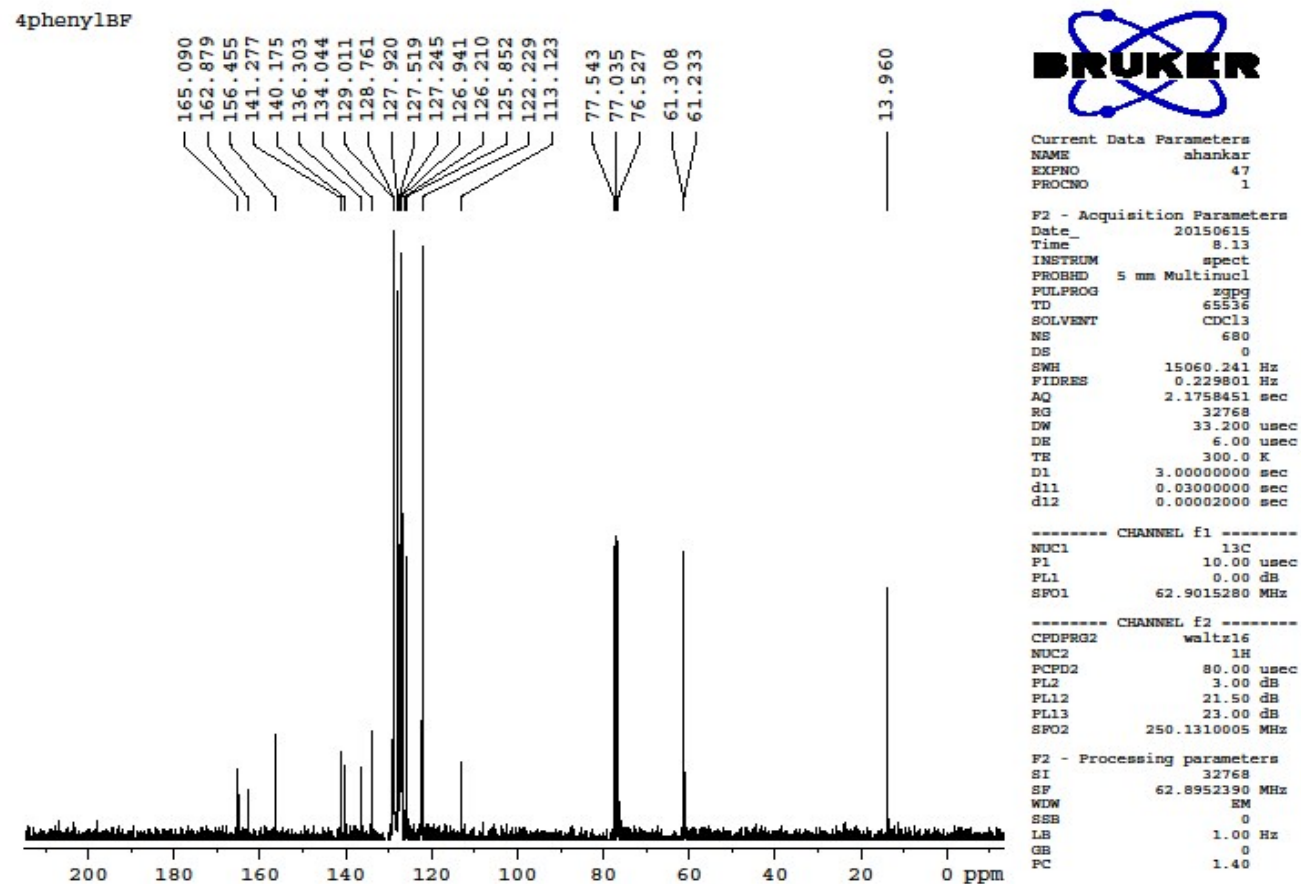


Fig. 39 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 2-([1,1'-biphenyl]-4-yl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4m**).

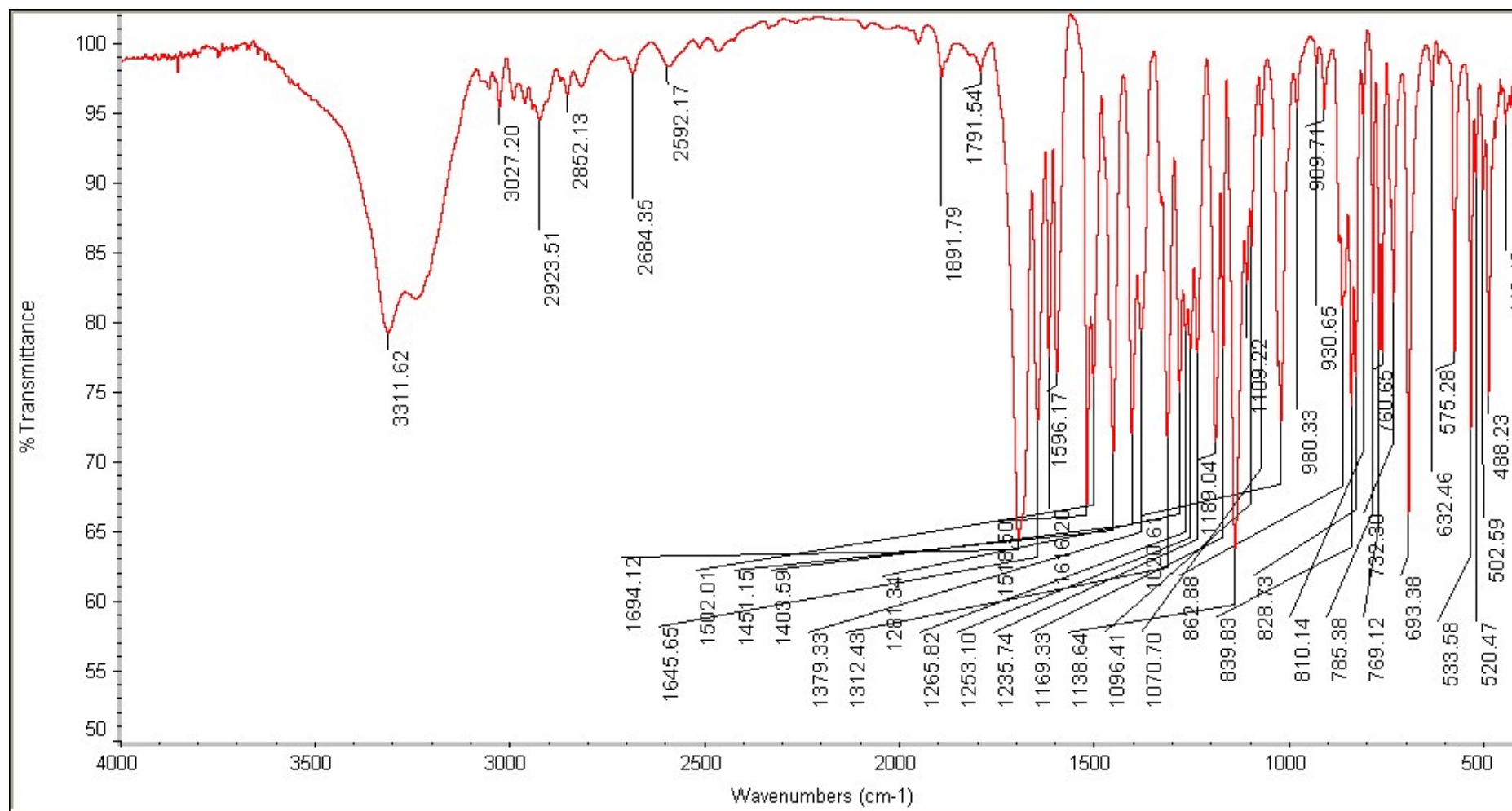


Fig. 40 FT-IR spectrum of Ethyl 4-hydroxy-2-(4-hydroxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4n**).

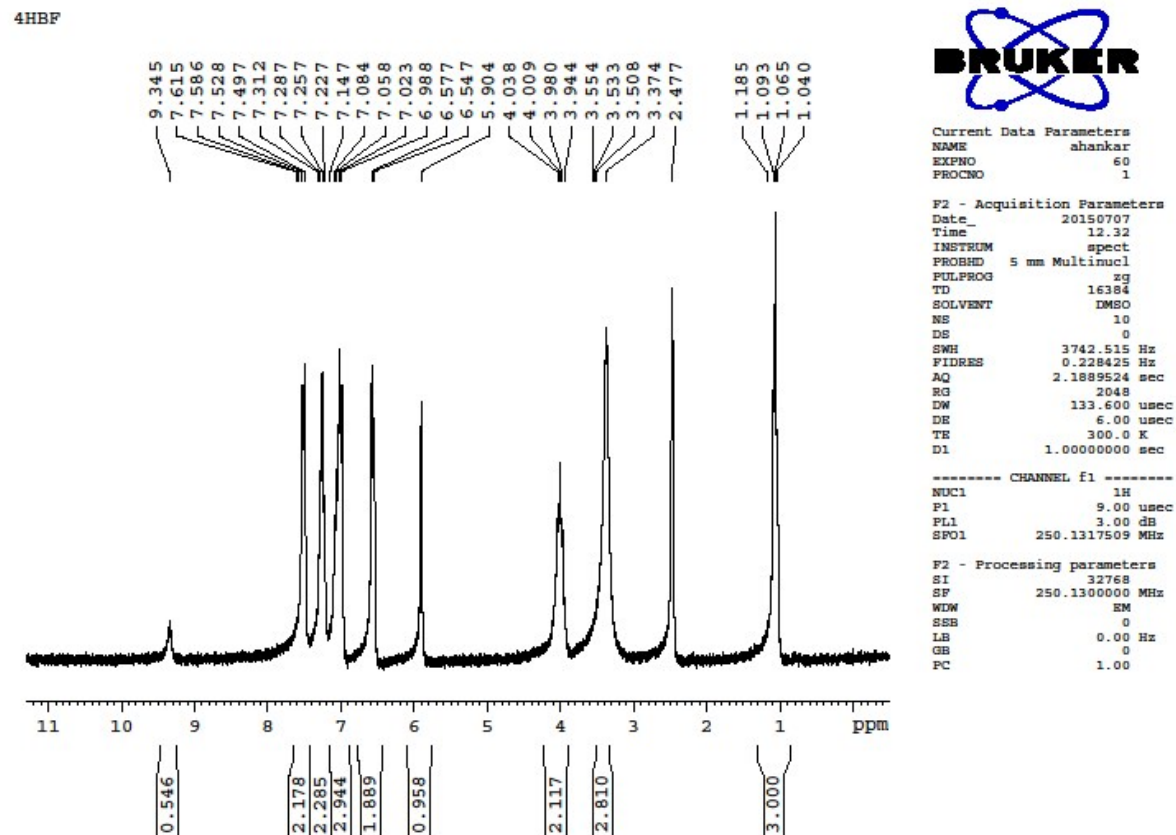


Fig. 41 ^1H NMR (250.13 MHz, DMSO-d_6) spectrum of Ethyl 4-hydroxy-2-(4-hydroxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4n**).

4HBF

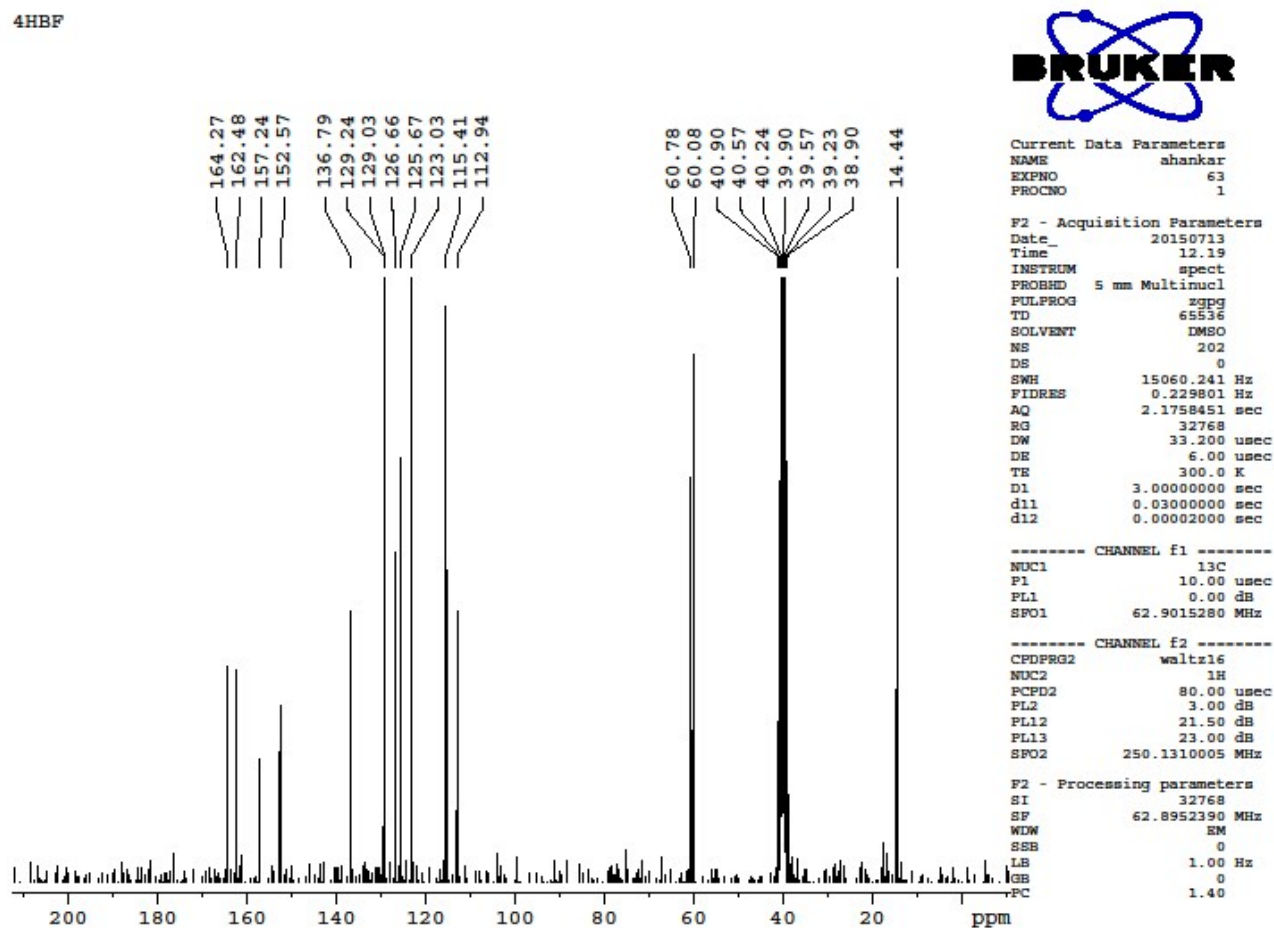


Fig. 42 ^1H NMR (62.90 MHz, DMSO-d_6) spectrum of Ethyl 4-hydroxy-2-(4-hydroxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4n**).

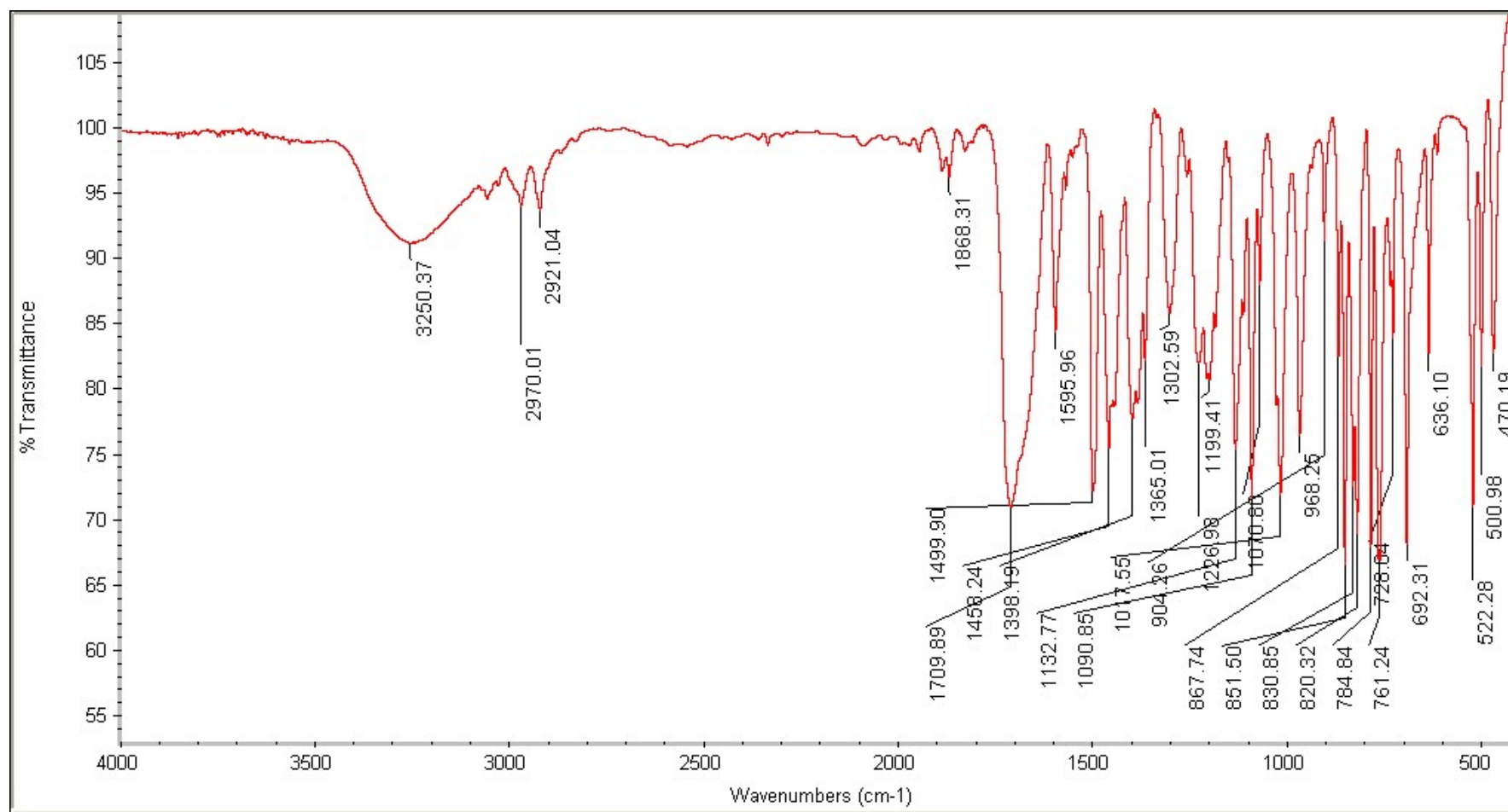


Fig. 43 FT-IR spectrum of Ethyl 4-hydroxy-2-(4-(methylthio)phenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4o**).

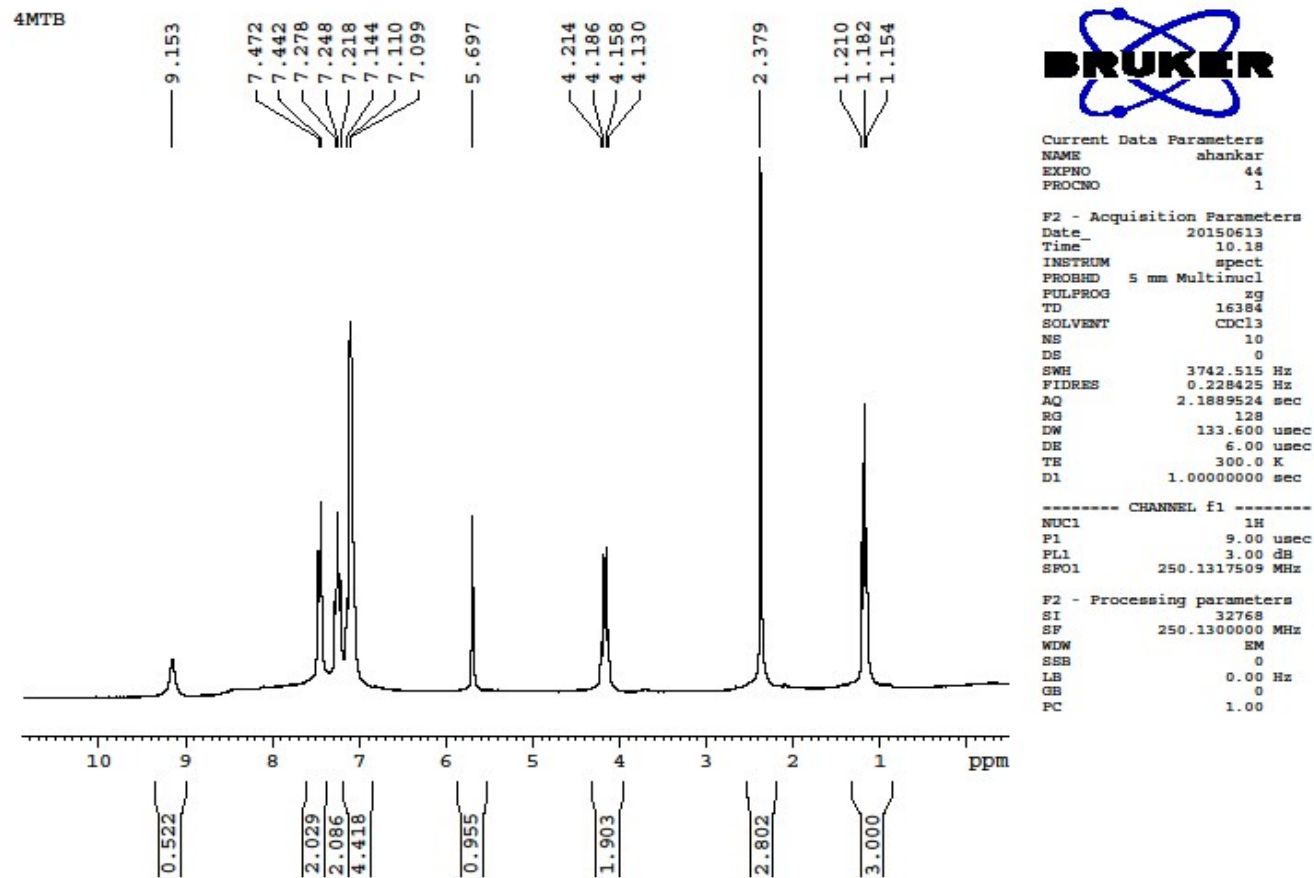


Fig. 44 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-2-(4-(methylthio)phenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**40**).

4MTB

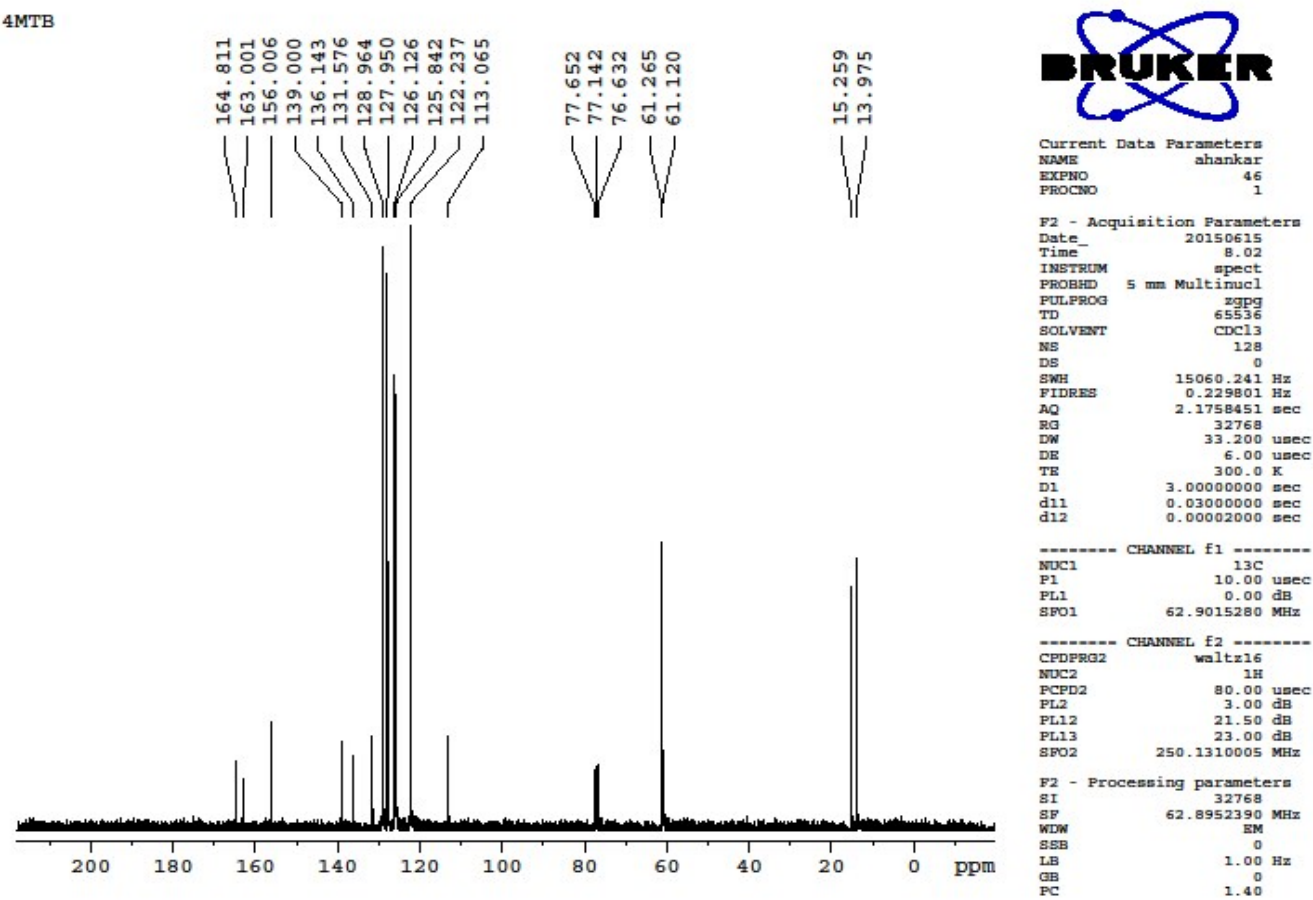


Fig. 45 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-2-(4-(methylthio)phenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4o**)

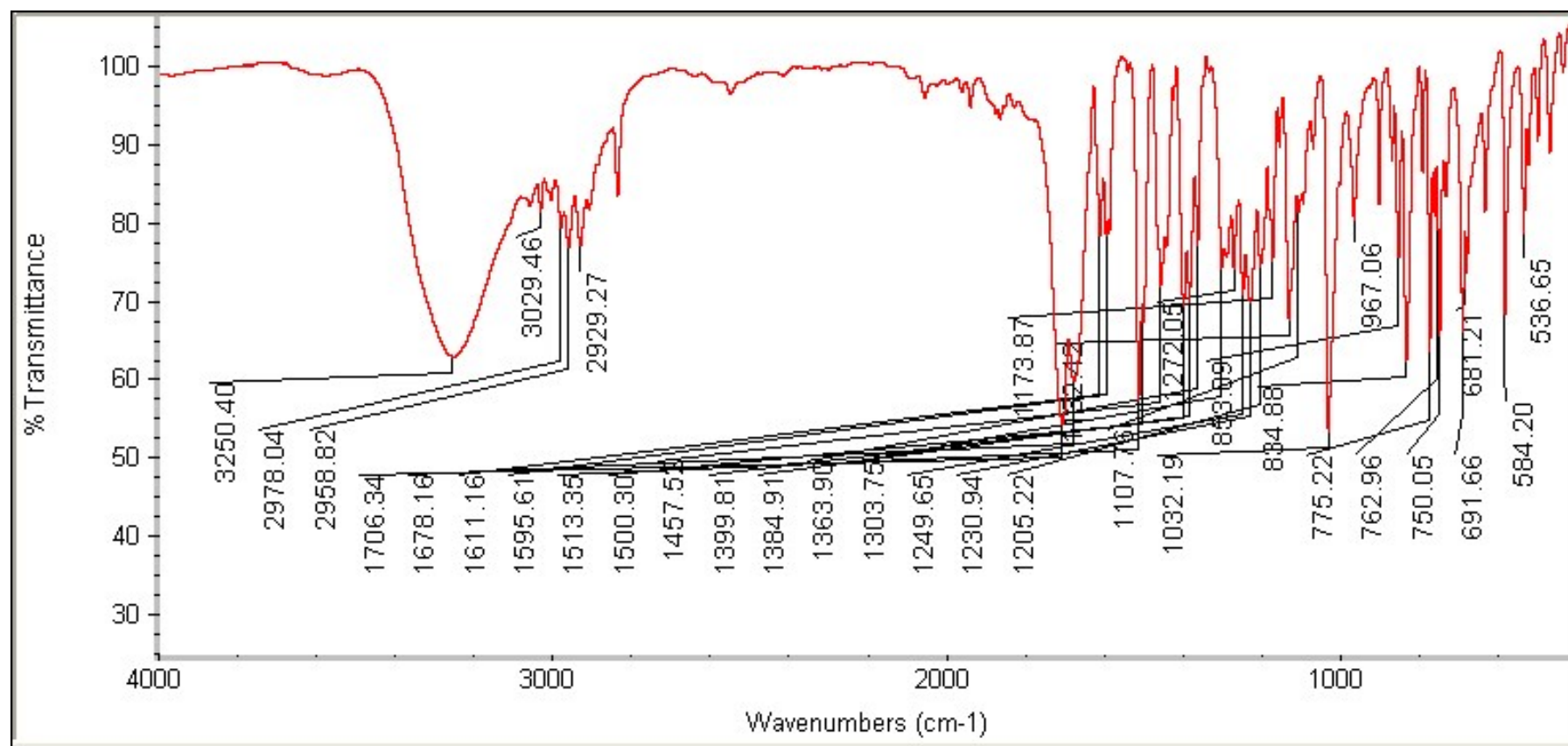


Fig. 46 FT-IR spectrum of Ethyl 4-hydroxy-2-(4-methoxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4p**).

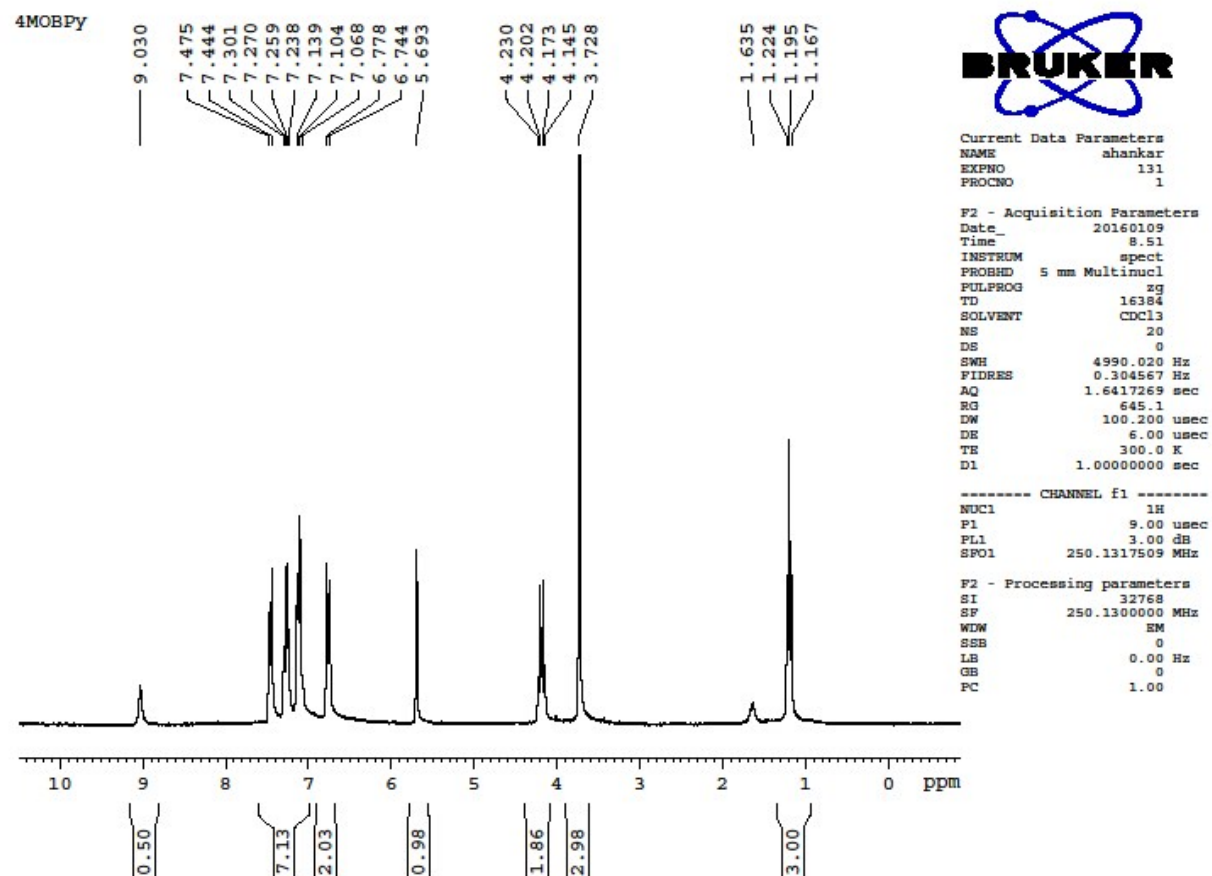


Fig. 47 ^1H NMR (250.13 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-2-(4-methoxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4p**)

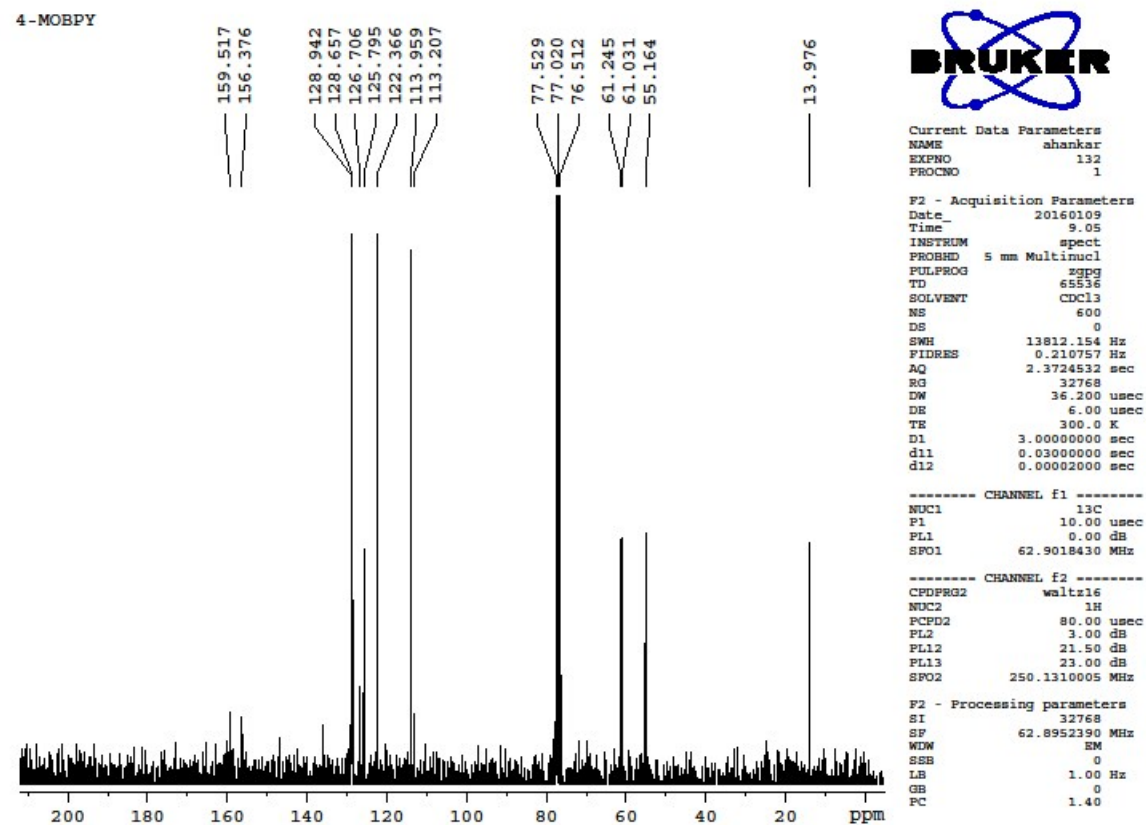


Fig. 48 ^{13}C NMR (62.90 MHz, CDCl_3) spectrum of Ethyl 4-hydroxy-2-(4-methoxyphenyl)-5-oxo-1-phenyl-2,5-dihydro-1H-pyrrole-3-carboxylate (**4p**)

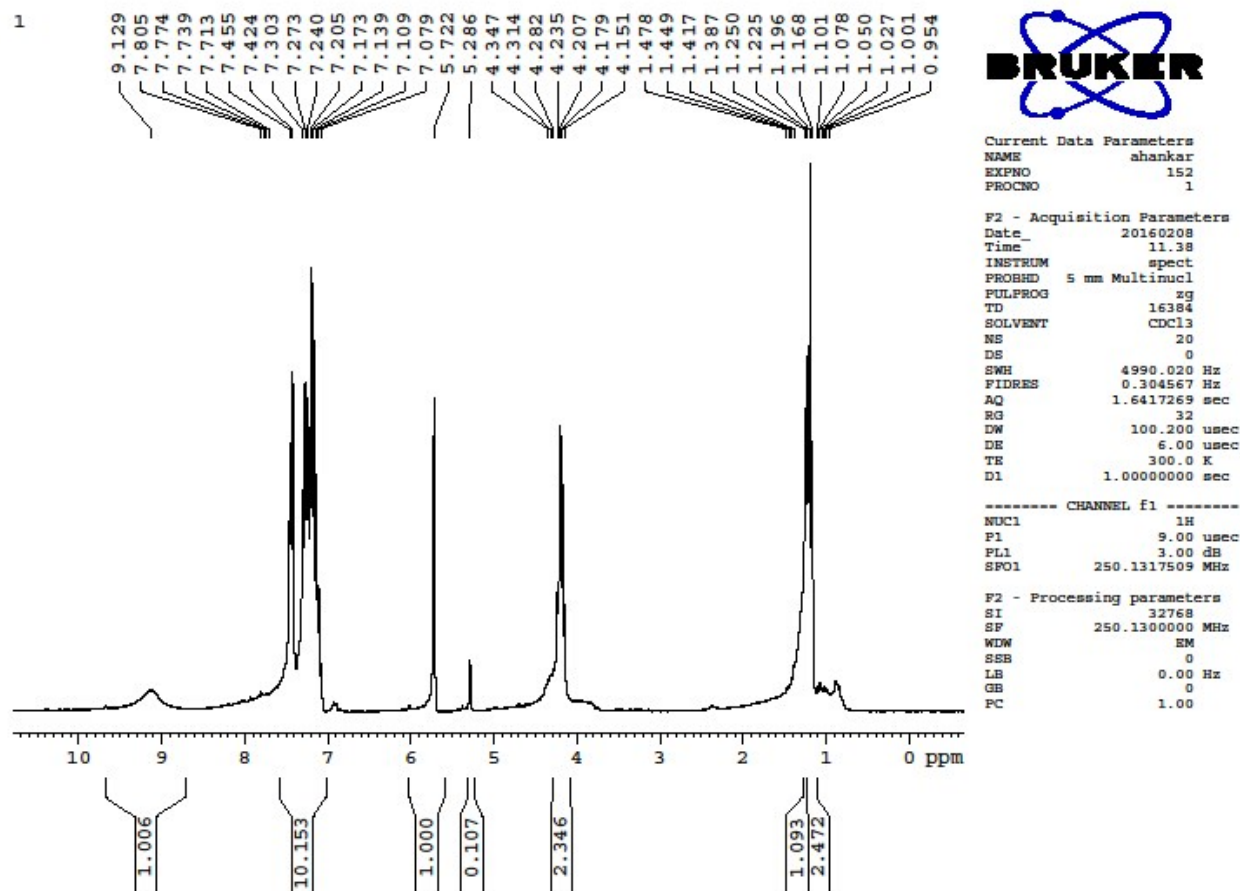


Fig. 49 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained under ultrasound irradiation conditions.

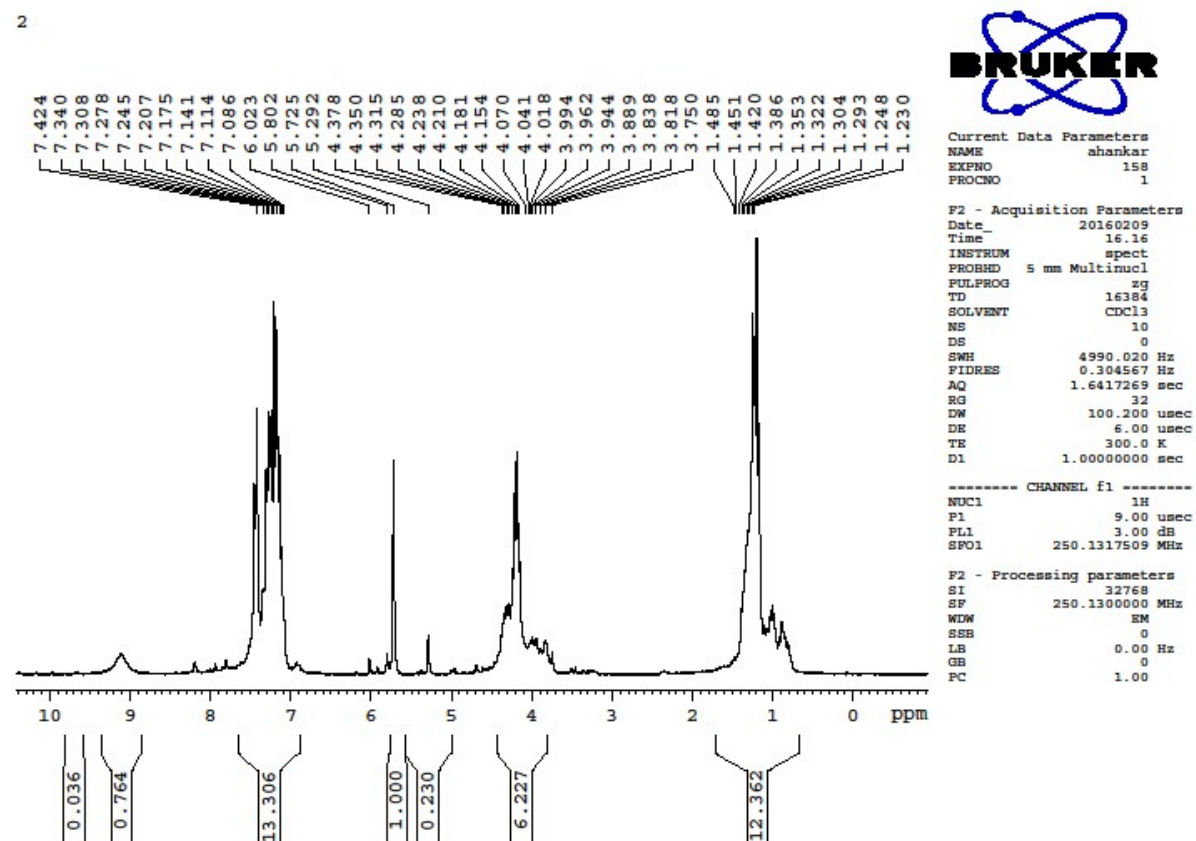


Fig. 50 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained at room temperature conditions.

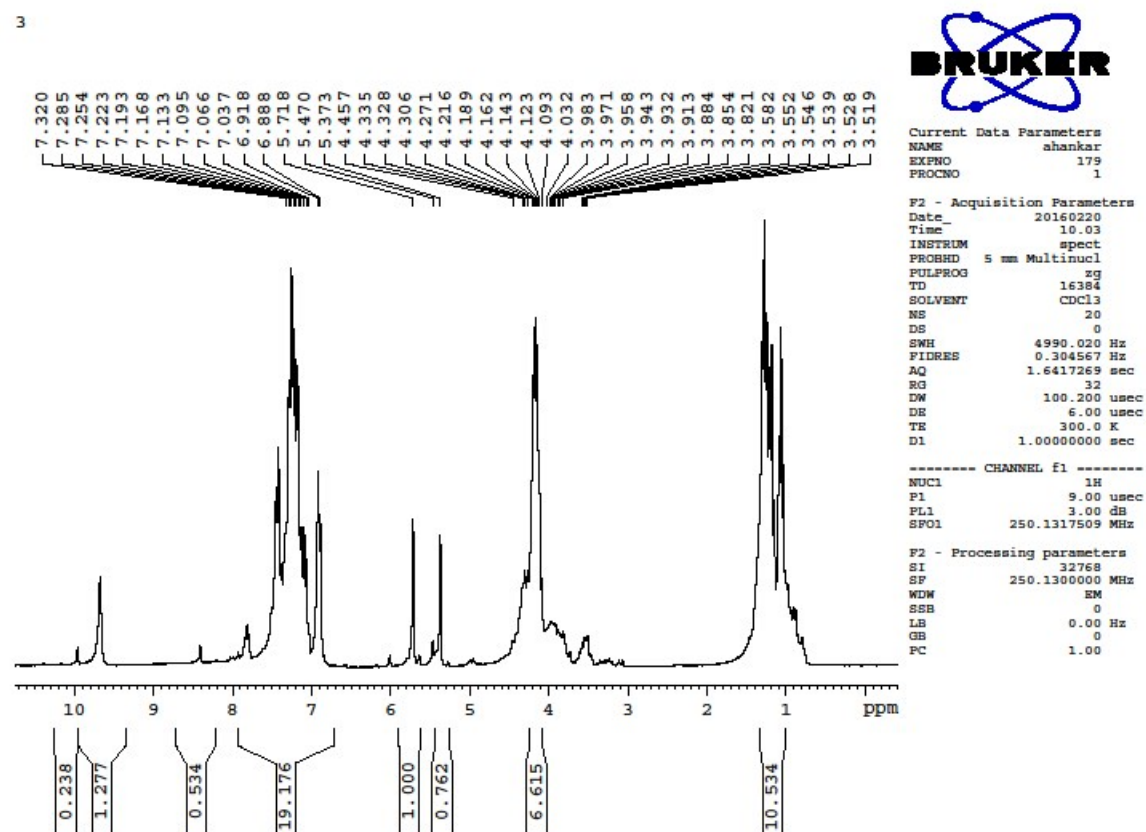


Fig. S1 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained at 40 °C for 15 min.

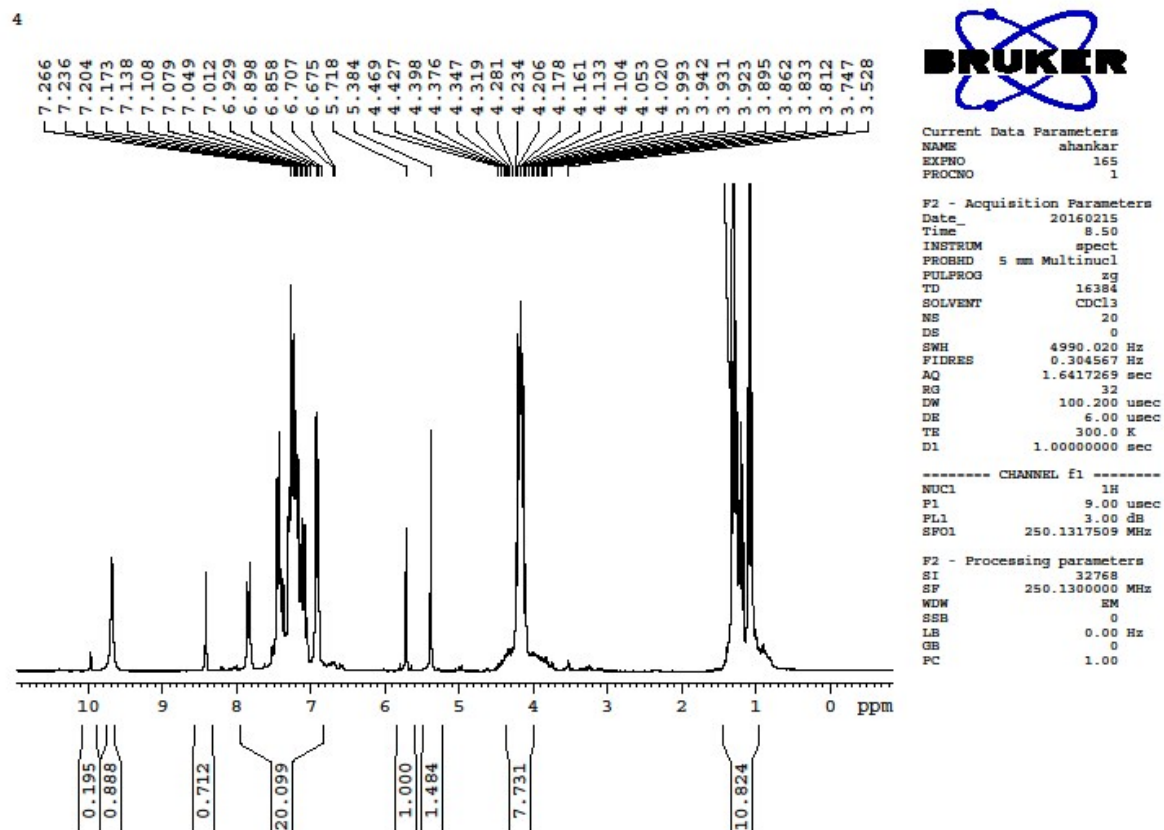


Fig. 52 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained at 40 °C for 30 min.

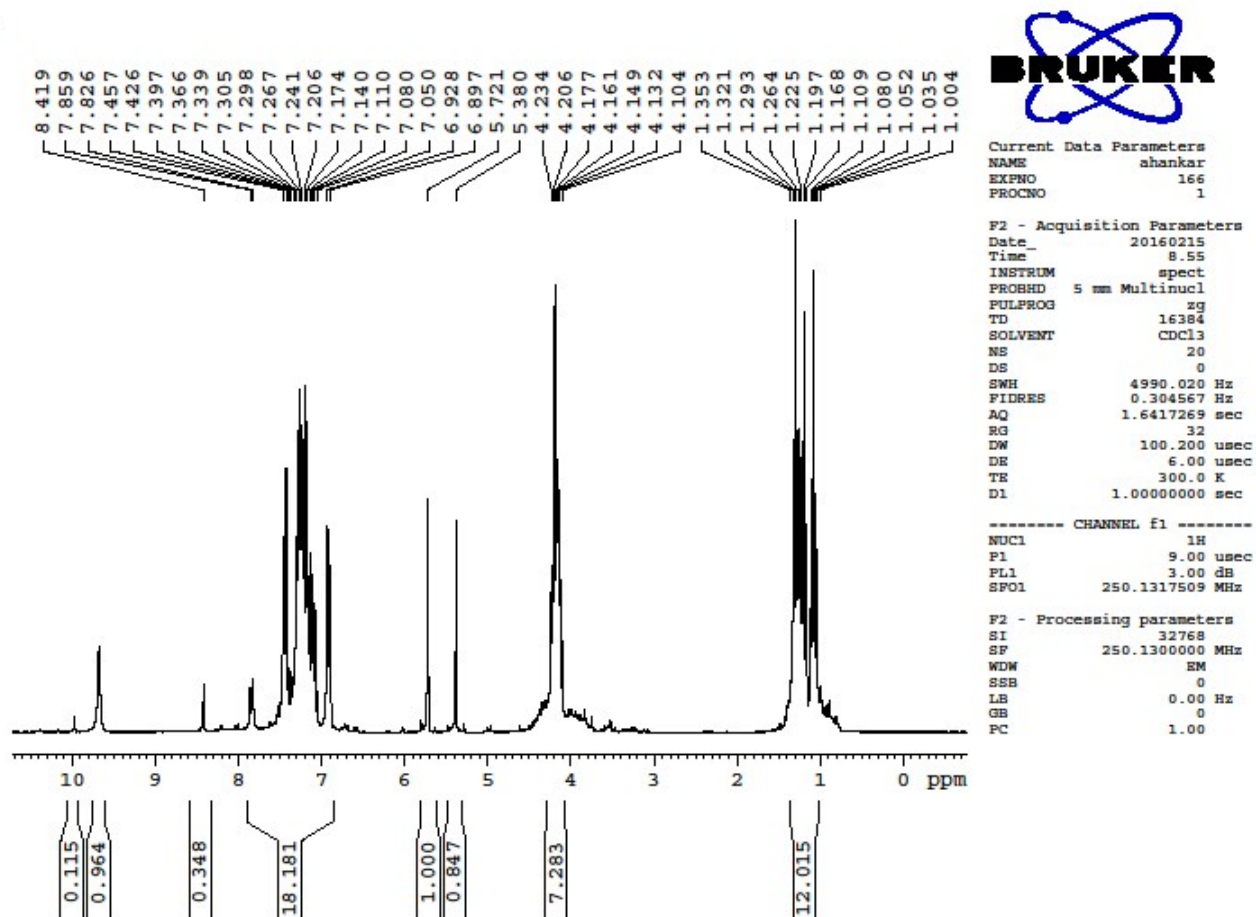


Fig. 53 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained at 40 °C for 60 min.

6

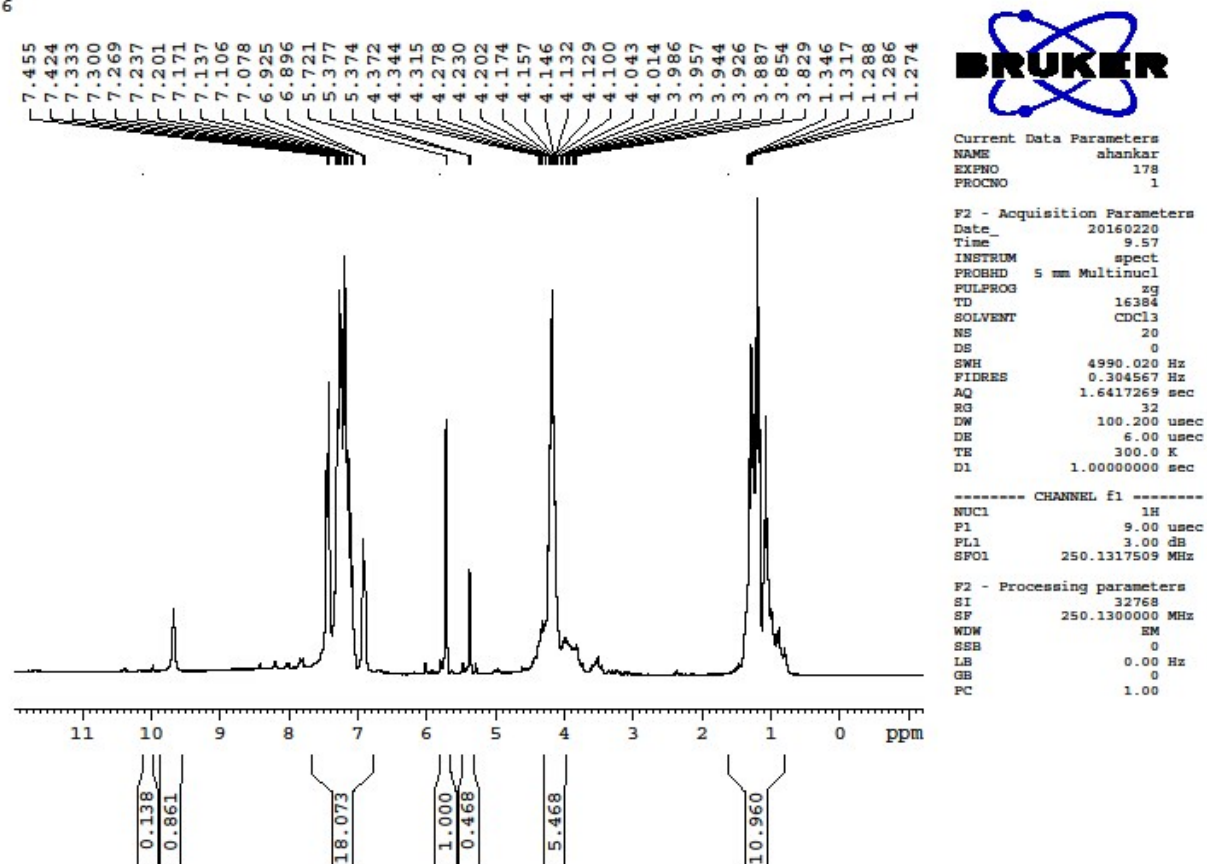


Fig. 54 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained in reflux conditions for 15 min.

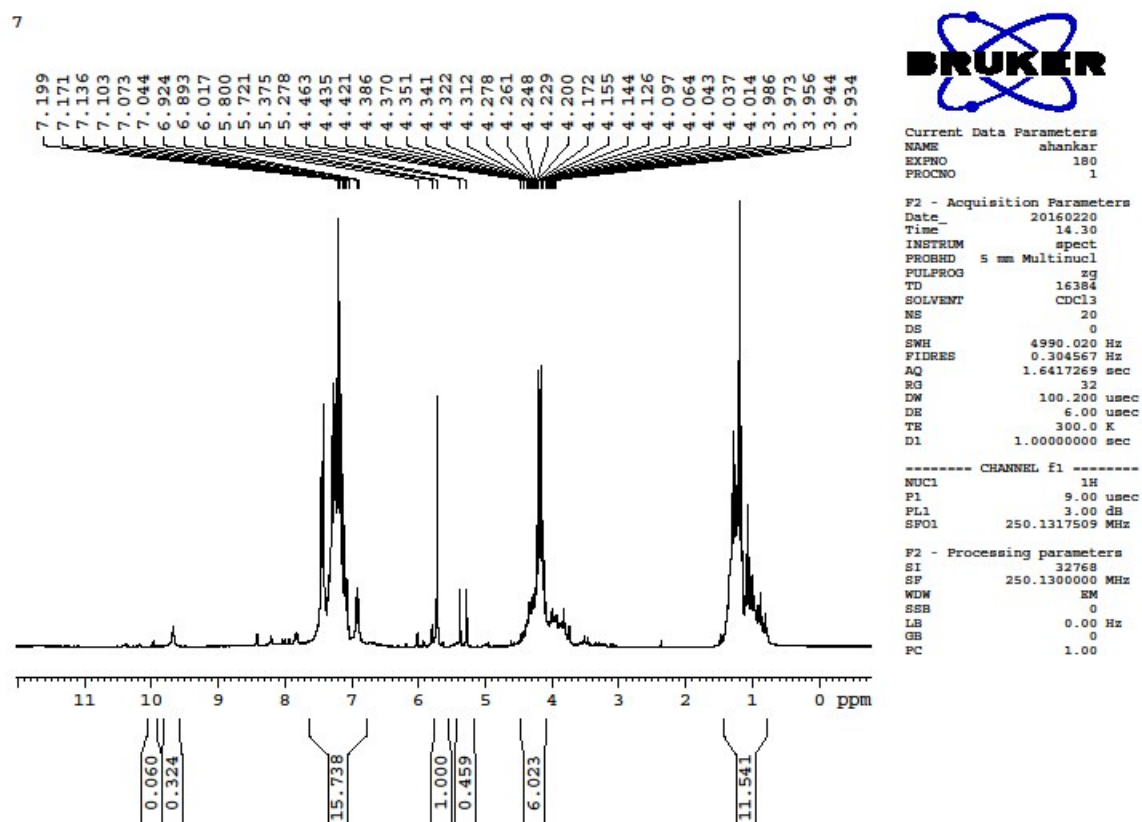


Fig. 55 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained in reflux conditions for 30 min.

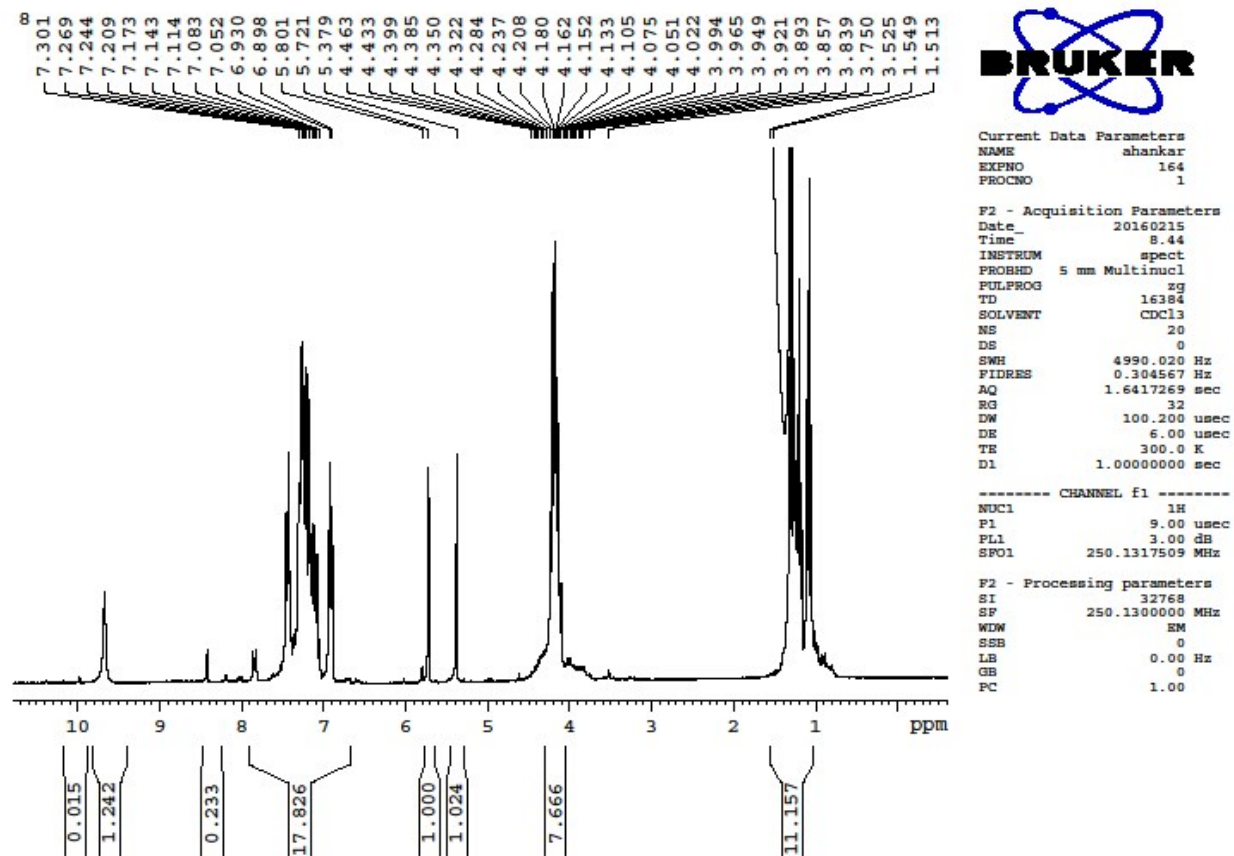


Fig. S6 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained in reflux conditions for 90 min.

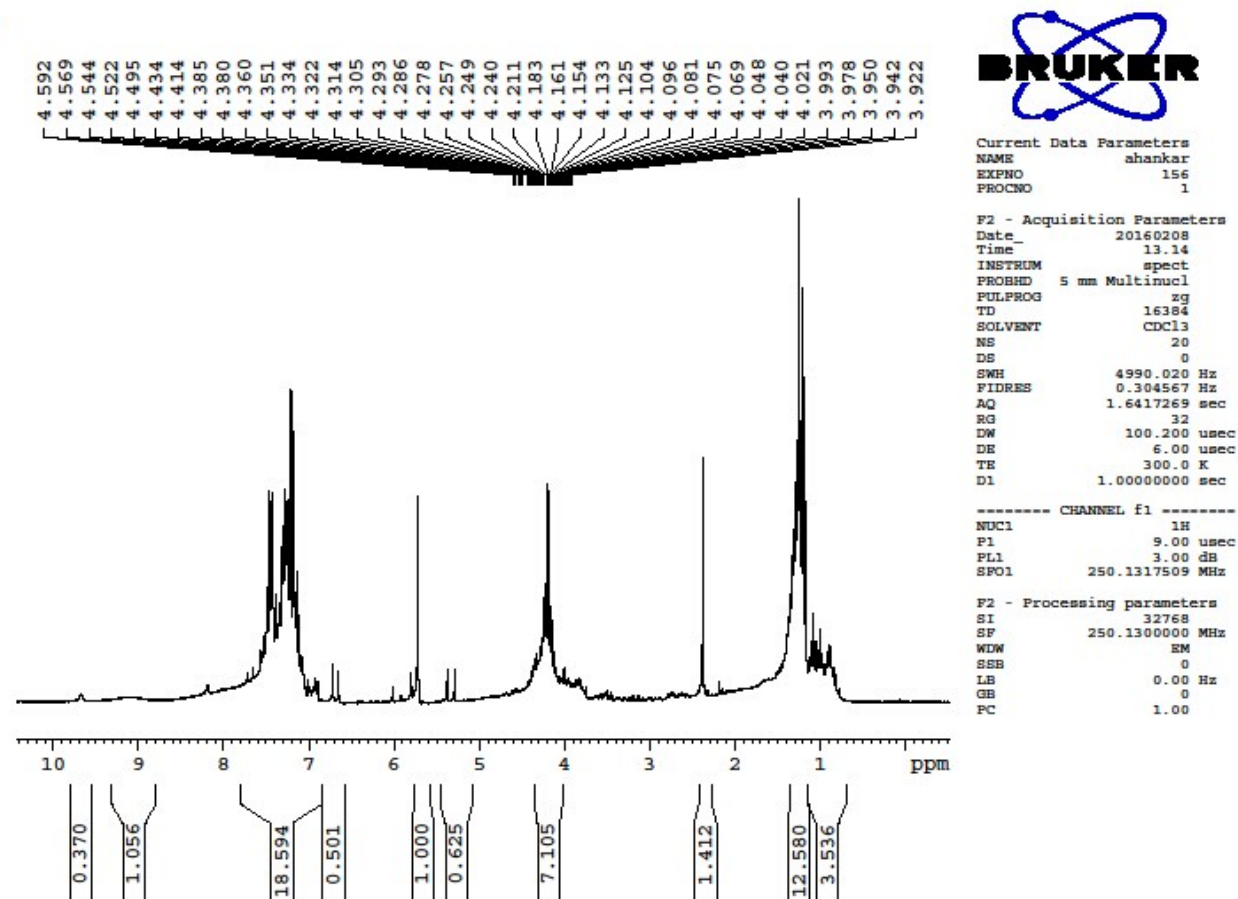


Fig. 57 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained in reflux conditions for 180 min.

10

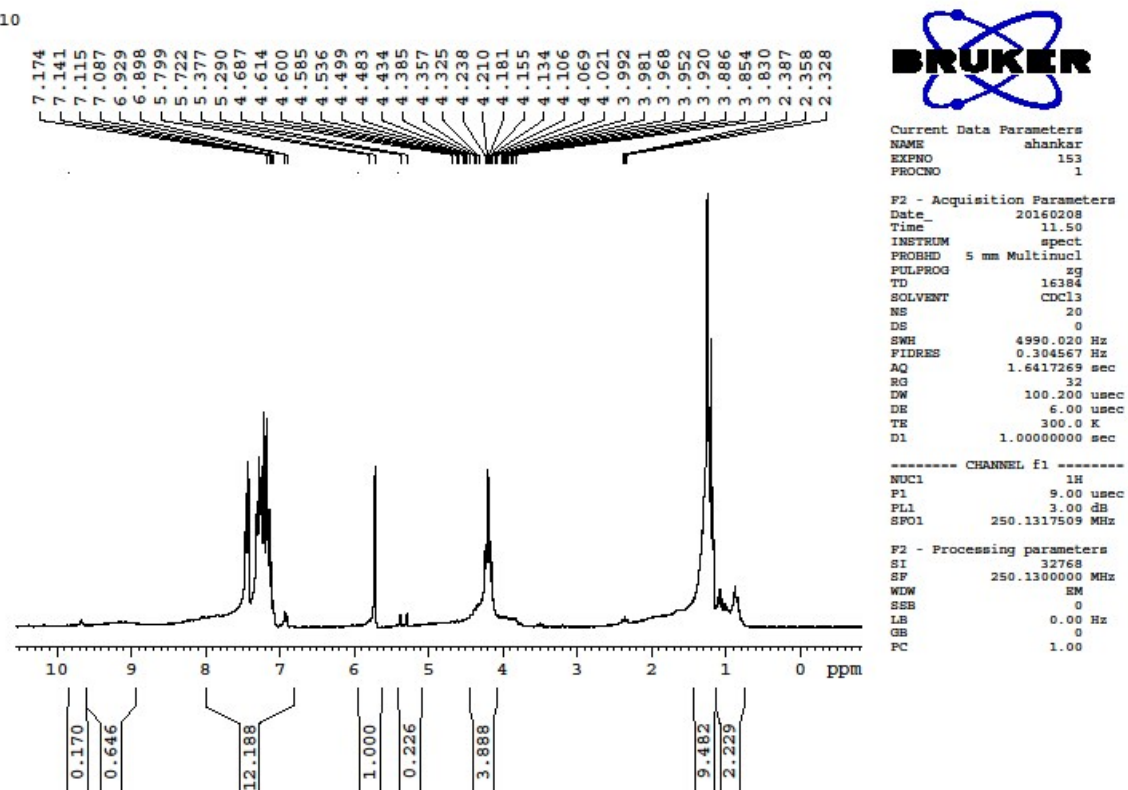


Fig. 58 ^1H NMR (250.13 MHz, CDCl_3) of the crude product **4d** that obtained in reflux conditions for 360 min.

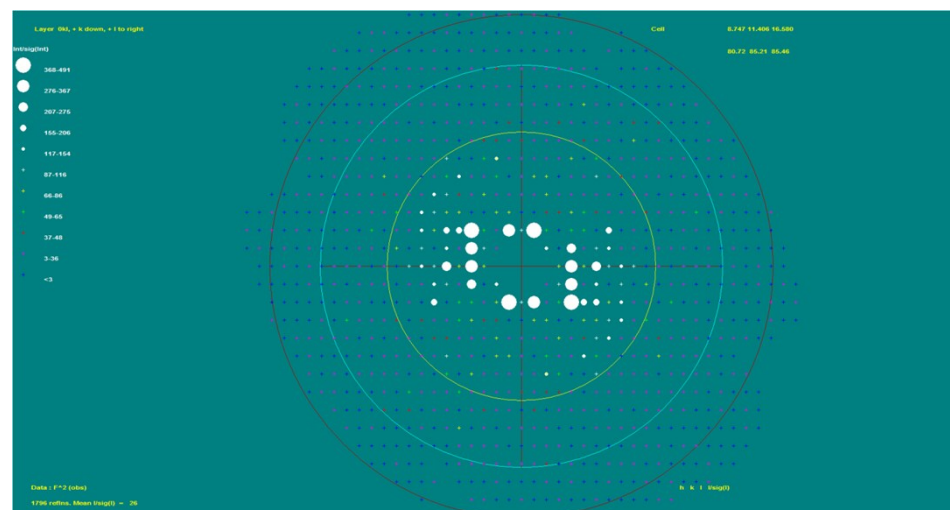


Fig. 59 Symmetry of diffraction pattern **0kl**.



Fig. 60 Symmetry of diffraction pattern **1kl**.

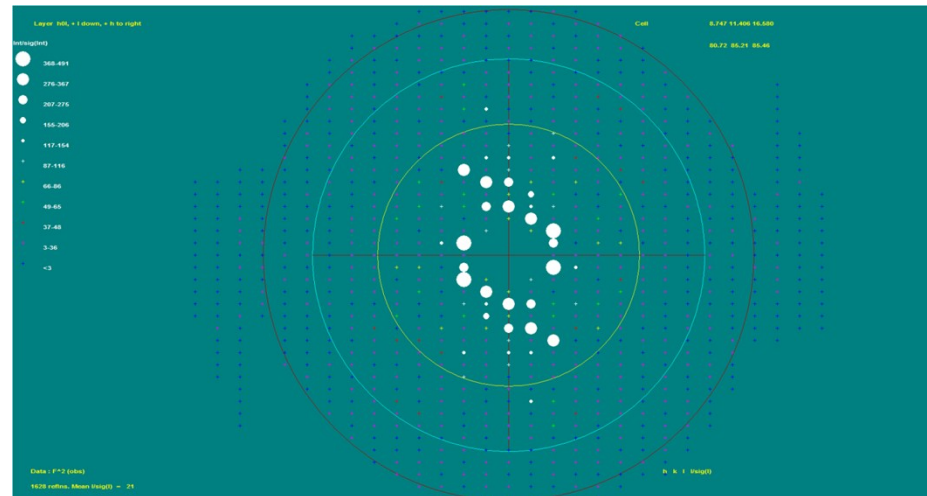


Fig. 61 Symmetry of diffraction pattern $h0l$.

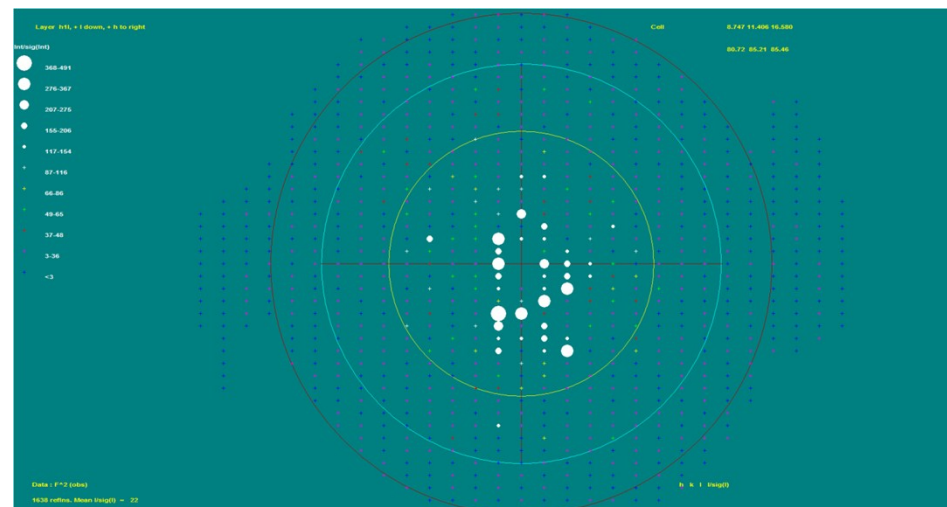


Fig. 62 Symmetry of diffraction pattern $h1l$.

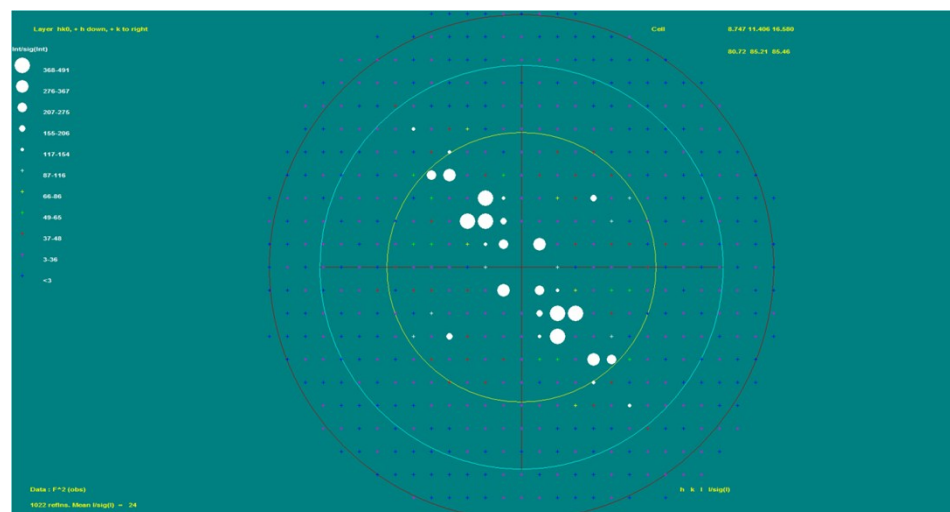


Fig. 63 Symmetry of diffraction pattern $hk0$.

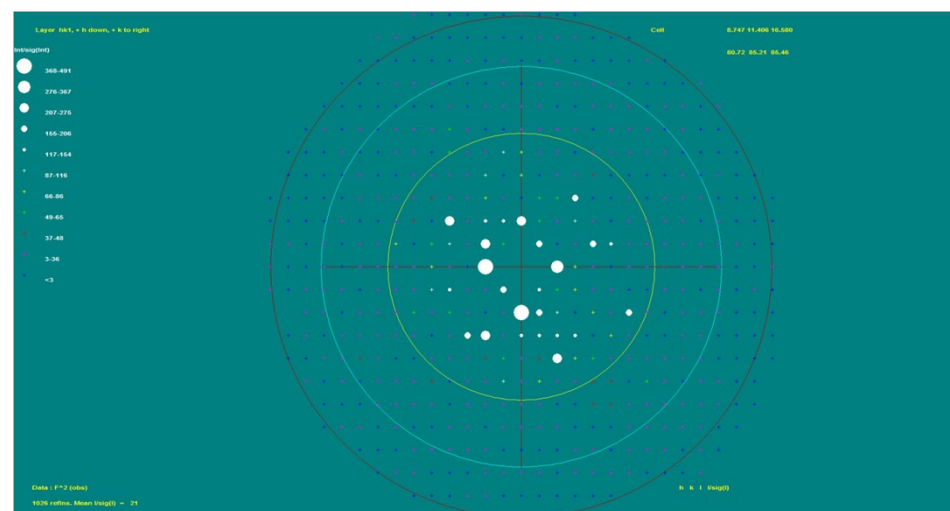


Fig. 64 Symmetry of diffraction pattern hkl .

Table S1 Selected geometric parameters (Å, °) for **4g**

Molecule A		Molecule B	
F1—C17	1.367(3)	F2—C36	1.370(3)
O1—C7	1.216(3)	O5—C26	1.218(3)
O2—C8	1.337(3)	O6—C27	1.331(3)
N1—C7	1.380(3)	N2—C26	1.375(3)
N1—C6	1.433(3)	N2—C25	1.421(3)
N1—C13	1.476(3)	N2—C32	1.484(3)
C7—N1—C6—C1	−16.0(4)	C26—N2—C25—C20	−19.4(4)
C11—O4—C10—C9	178.9(2)	C30—O8—C29—C28	−177.8(2)
C8—C9—C10—O4	−172.6(2)	C27—C28—C29—O8	−177.0 (2)
C10—O4—C11—C12	−173.5(2)	C29—O8—C30—C31	−175.9(2)
C7—N1—C13—C14	119.5(2)	C26—N2—C32—C33	124.4(2)
N1—C13—C14—C15	136.4(2)	N2—C32—C33—C38	137.7(2)
C9—C13—C14—C15	−108.1(2)	C28—C32—C33—C38	−105.8(3)