

Supplementary Information

Synthesis of Quinolinediones by Catalyst-Free Formal Aza-[3+2+1] Cycloaddition of Enaminones, Aldehydes and Meldrum's Acid

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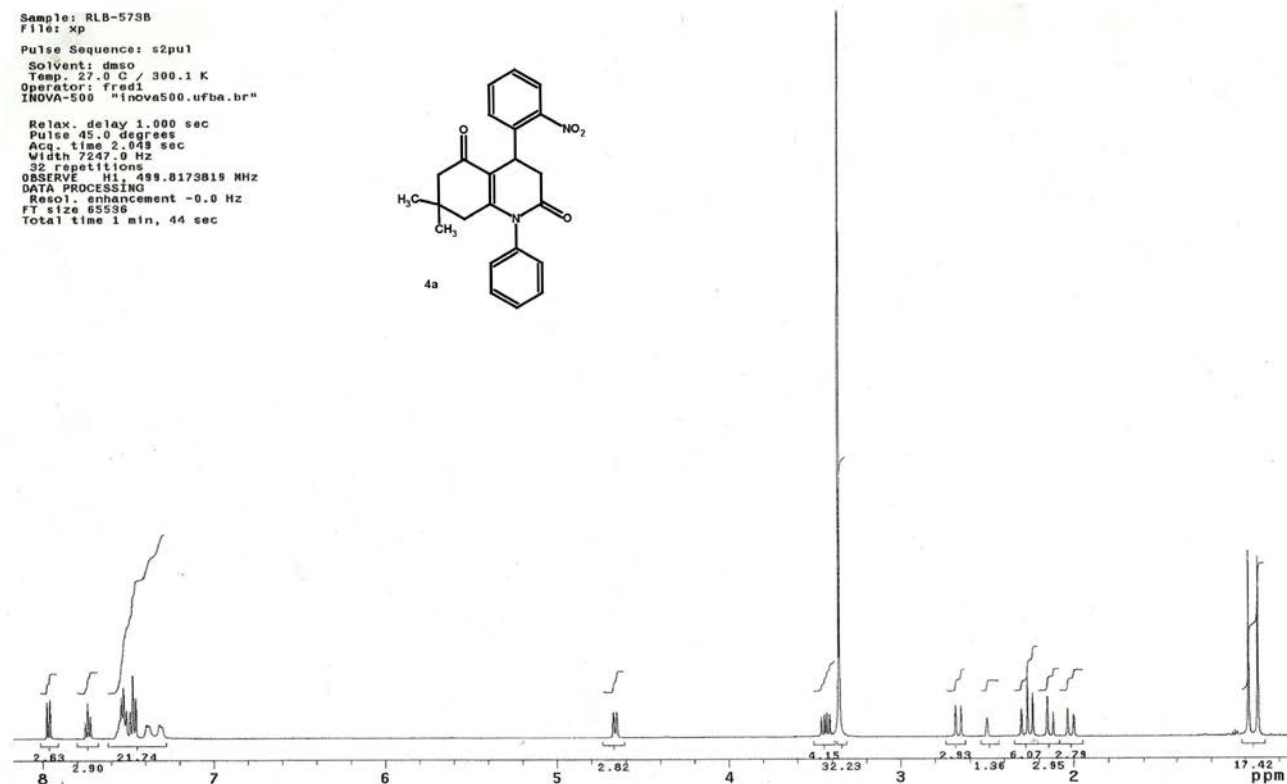
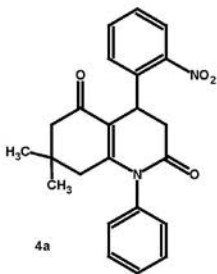


Figure S1. ¹H NMR (DMSO-D₆, 300 MHz) spectrum of **4a**.

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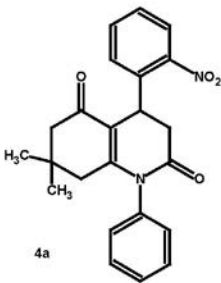


Figure S3. Expanded ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of compound **4a**.

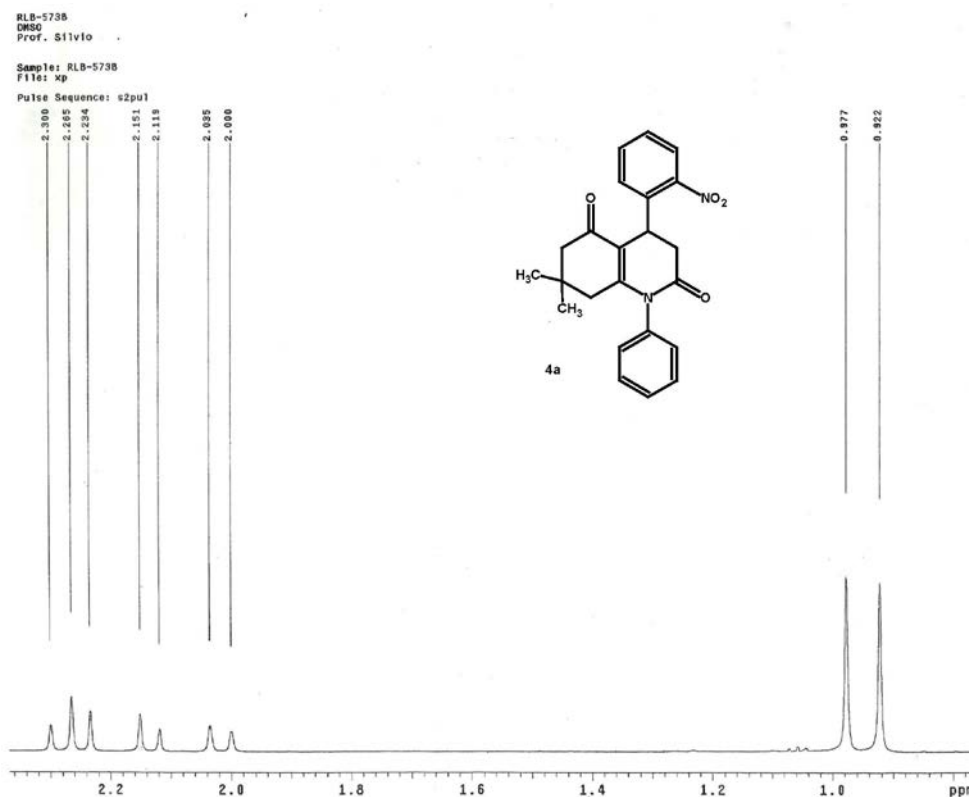


Figure S4. Expanded ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of compound 4a.

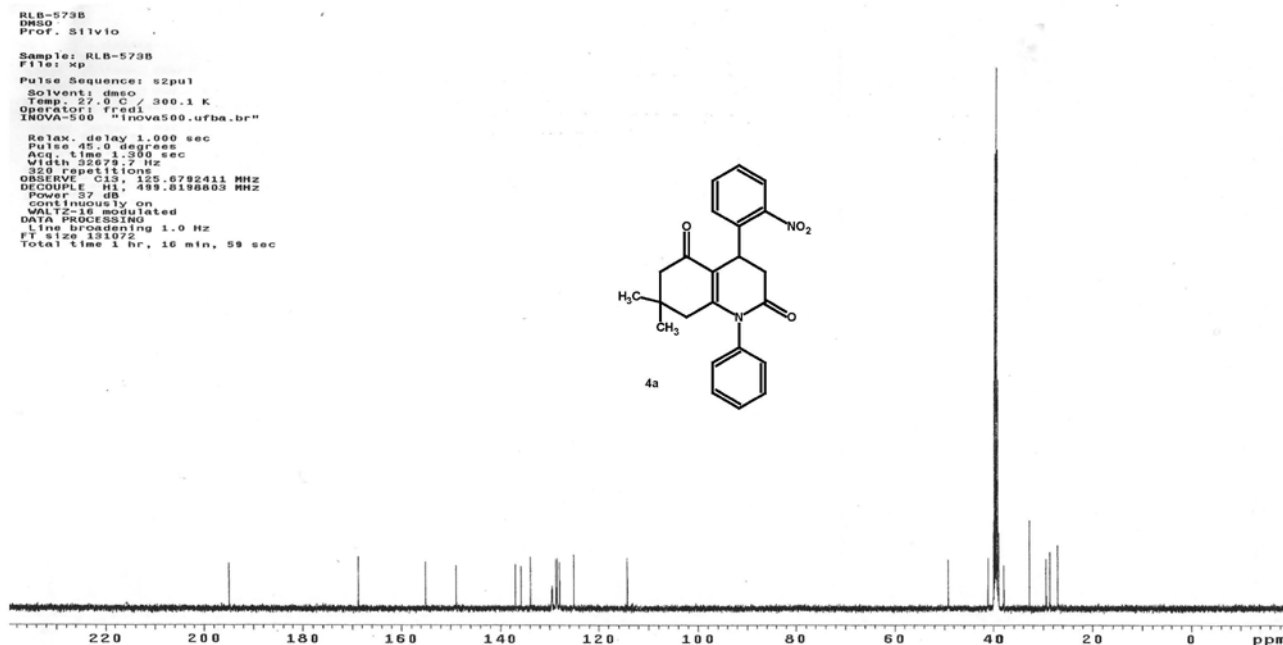
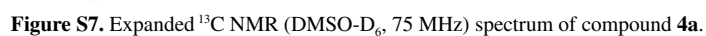
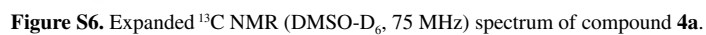


Figure S5. ^{13}C NMR (DMSO- D_6 , 75 MHz) spectrum of compound 4a.



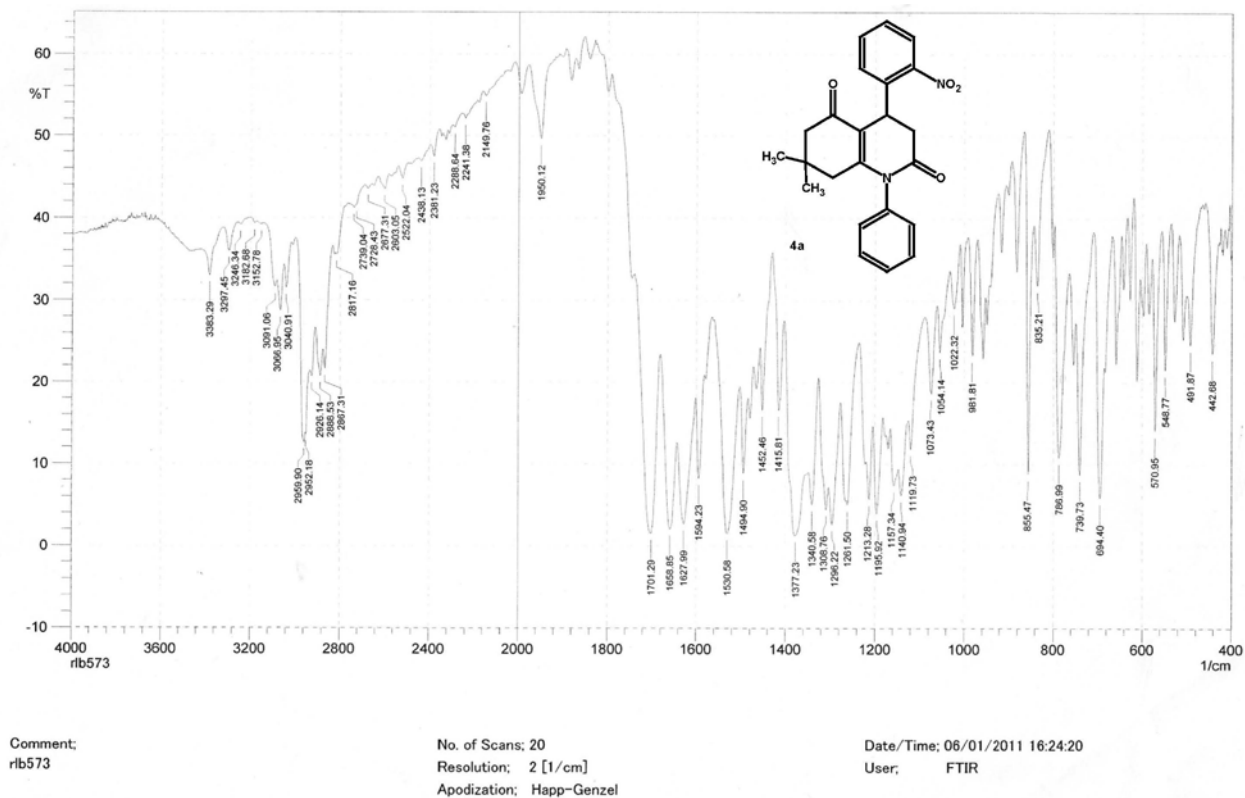


Figure S8. IR (KBr) spectrum of compound 4a.

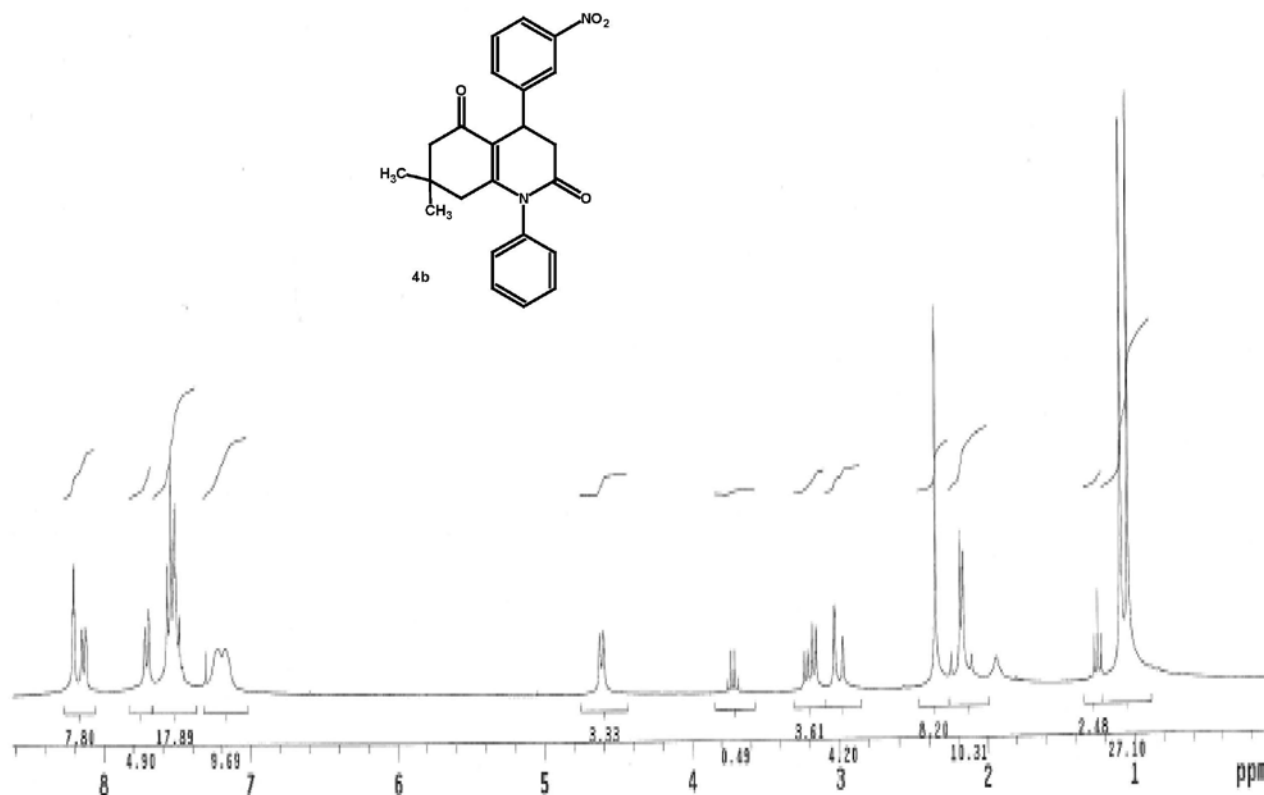
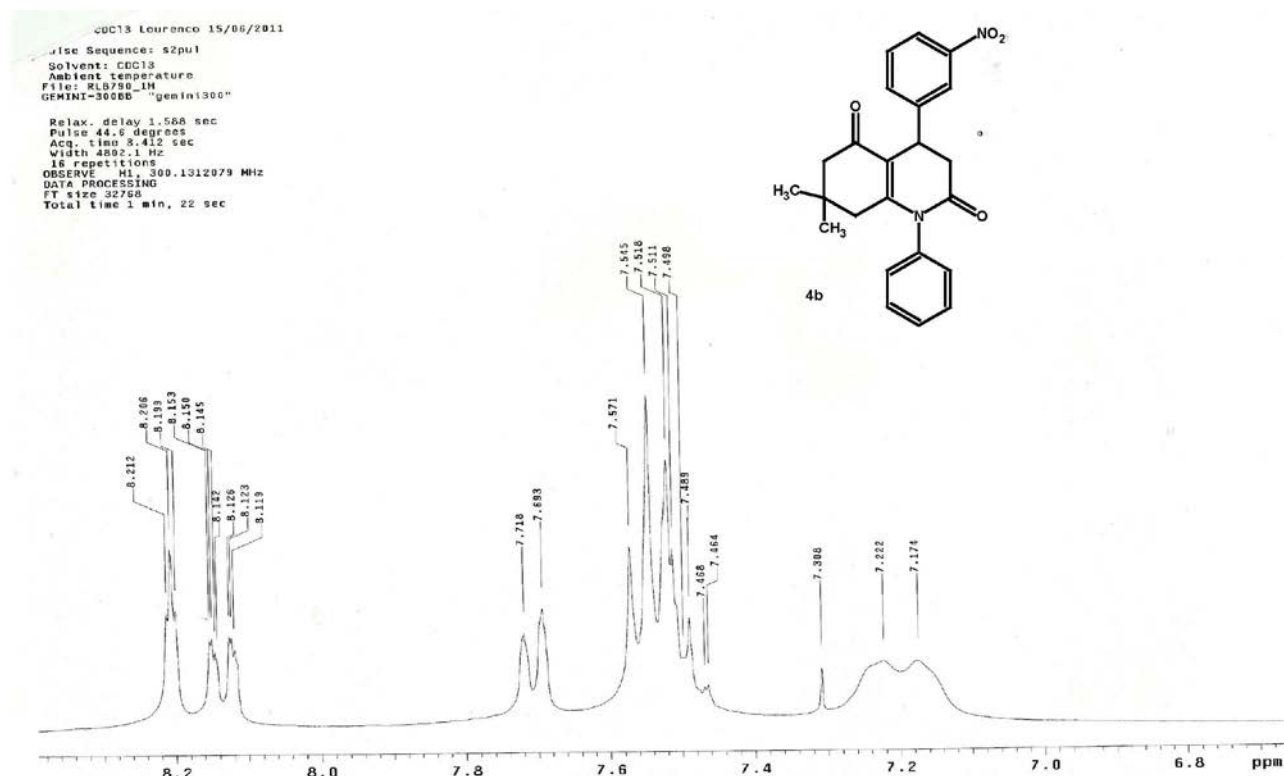
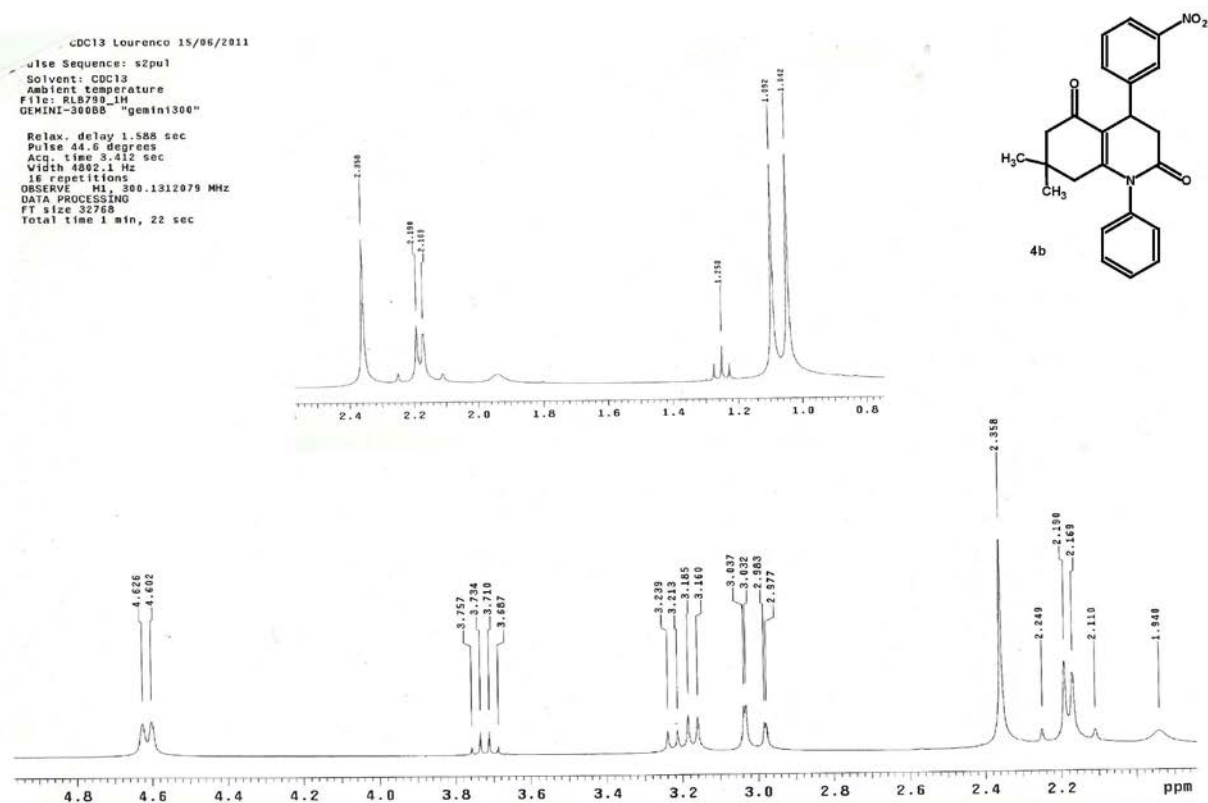


Figure S9. ¹H NMR (CDCl₃, 300 MHz) spectrum of compound 4b.

Figure S10. Expanded ^1H NMR (CDCl_3 , 300 MHz) spectrum of compound 4b.Figure S11. Expanded ^1H NMR (CDCl_3 , 300 MHz) spectrum of compound 4b.

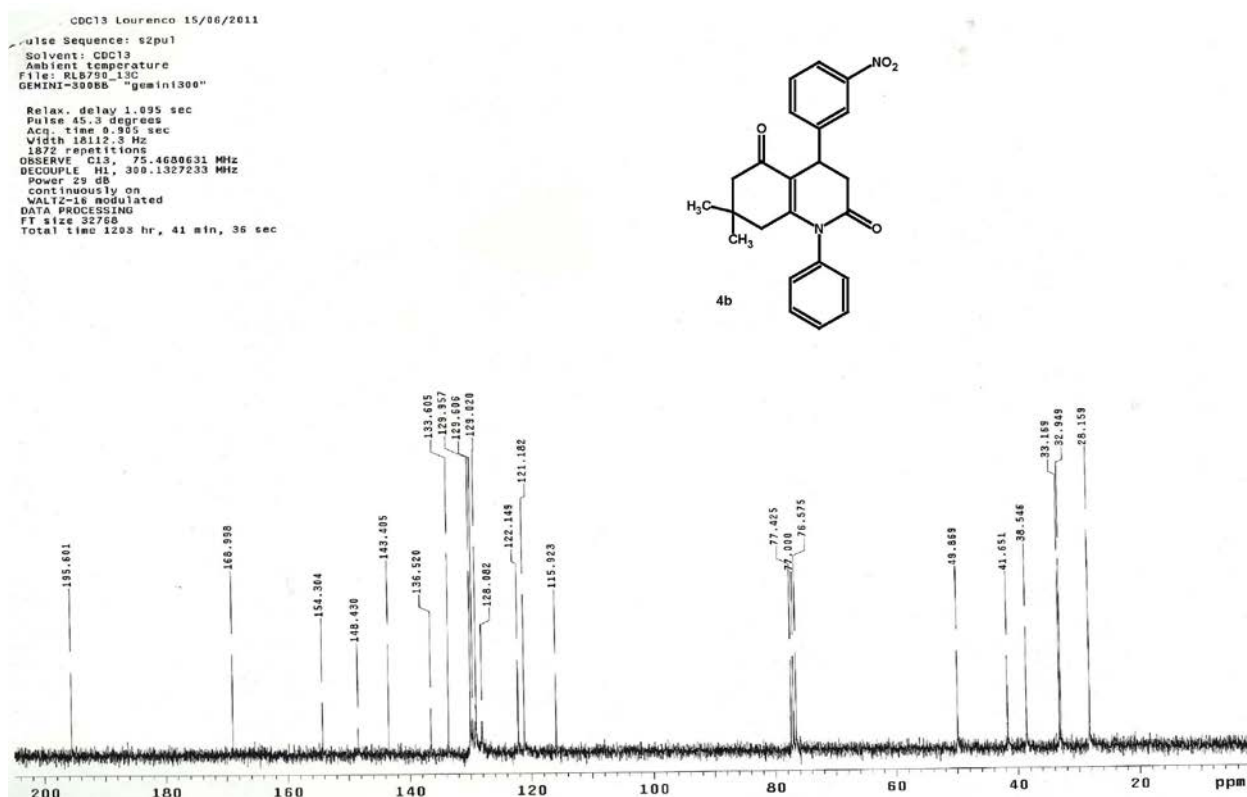
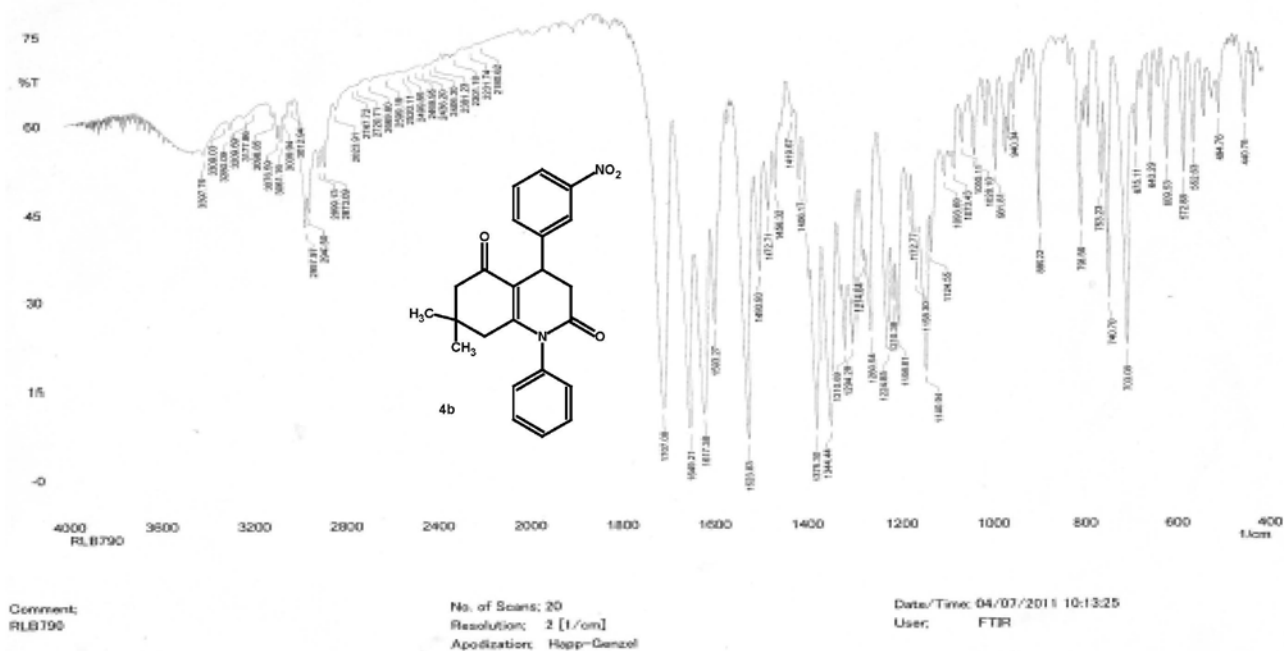
Figure S12. ^{13}C NMR (CDCl_3 , 75 MHz) spectrum of compound 4b.

Figure S13. IR (KBr) spectrum of compound 4b.

RLB789fr-1 CDCl₃ Lourenco 01/07/2011
Pulse Sequence: zgpg30
Solvent: CDCl₃
Ambient temperature
File: RLB789fr-1
GEMINI-30000 "gemin300"
Relax. delay 1.588 sec
Pulse 44.6 degrees
Acq. time 3.412 sec
Width 4092.1 Hz
16 repetitions
OBSERVE H1, 300.1312194 MHz
DATA PROCESSING
F1 size 32768
Total time 1 min, 22 sec

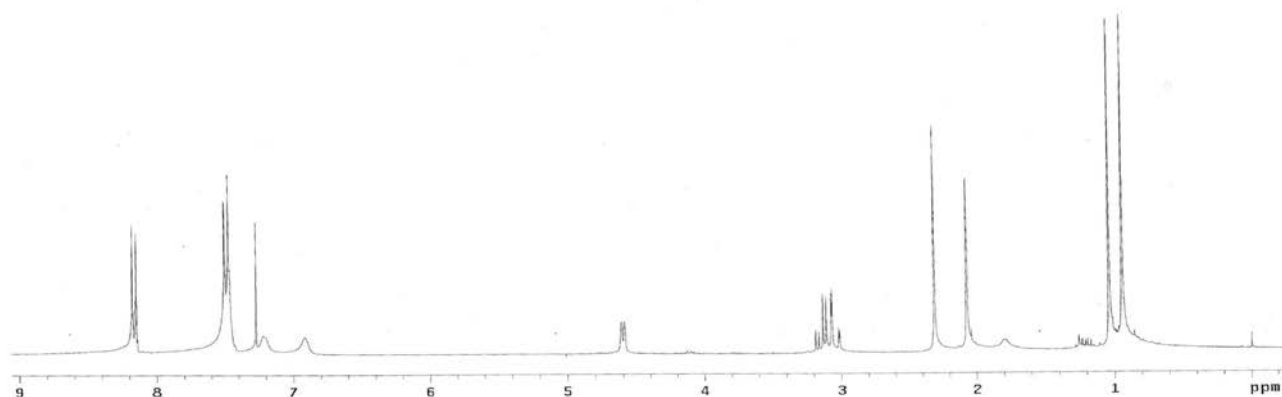
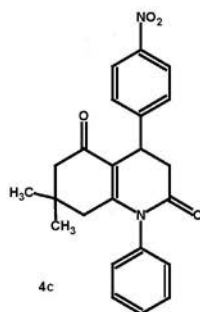


Figure S14. ¹H NMR (CDCl₃, 300 MHz) spectrum of compound **4c**.

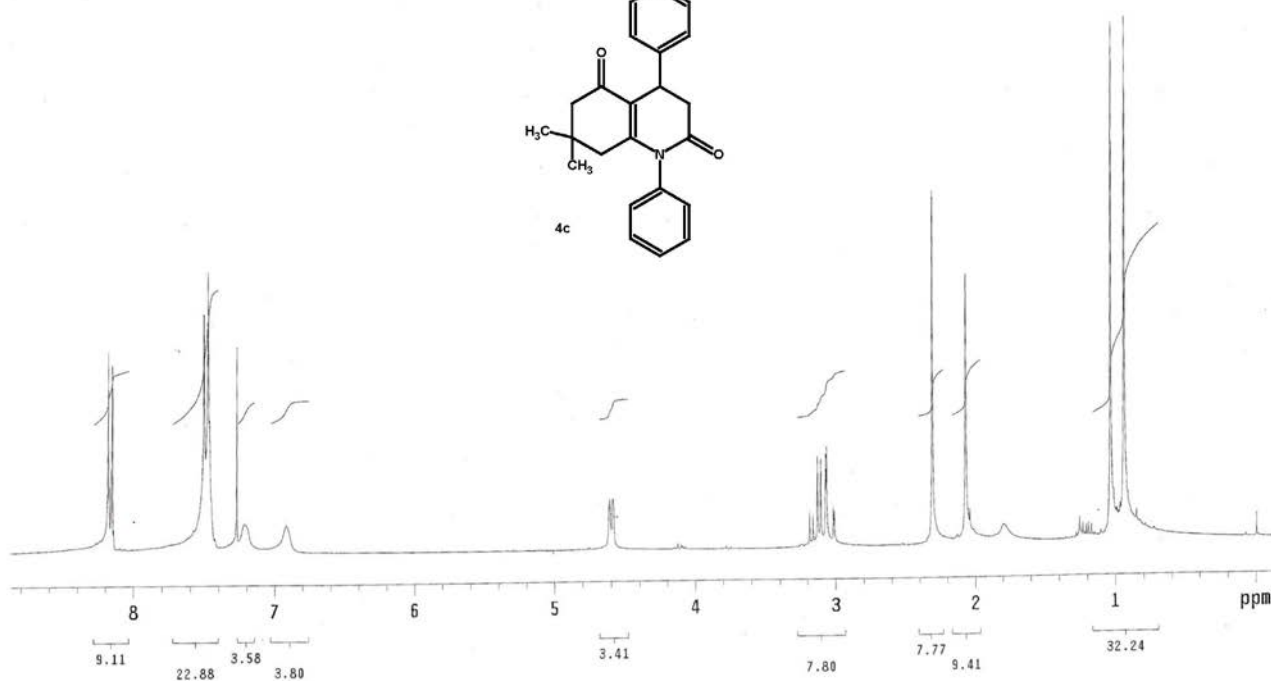
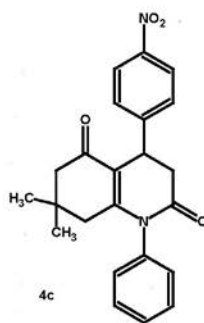
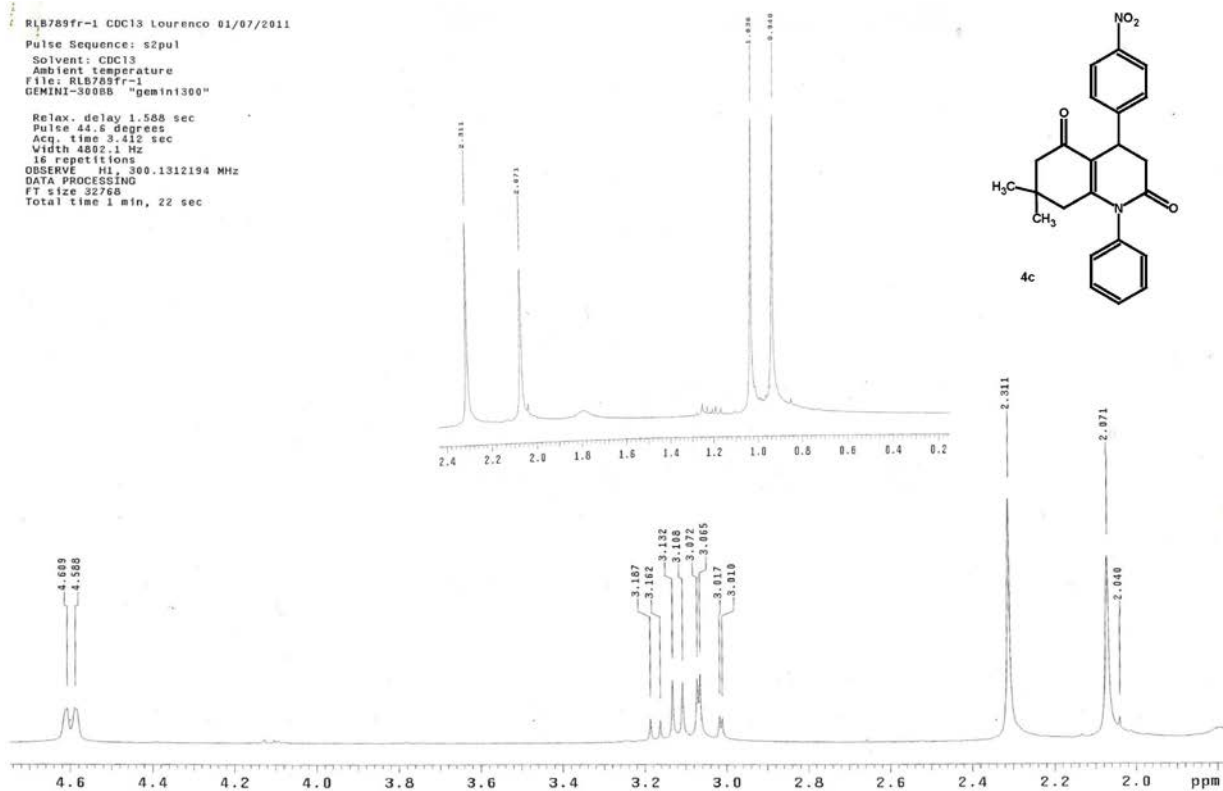
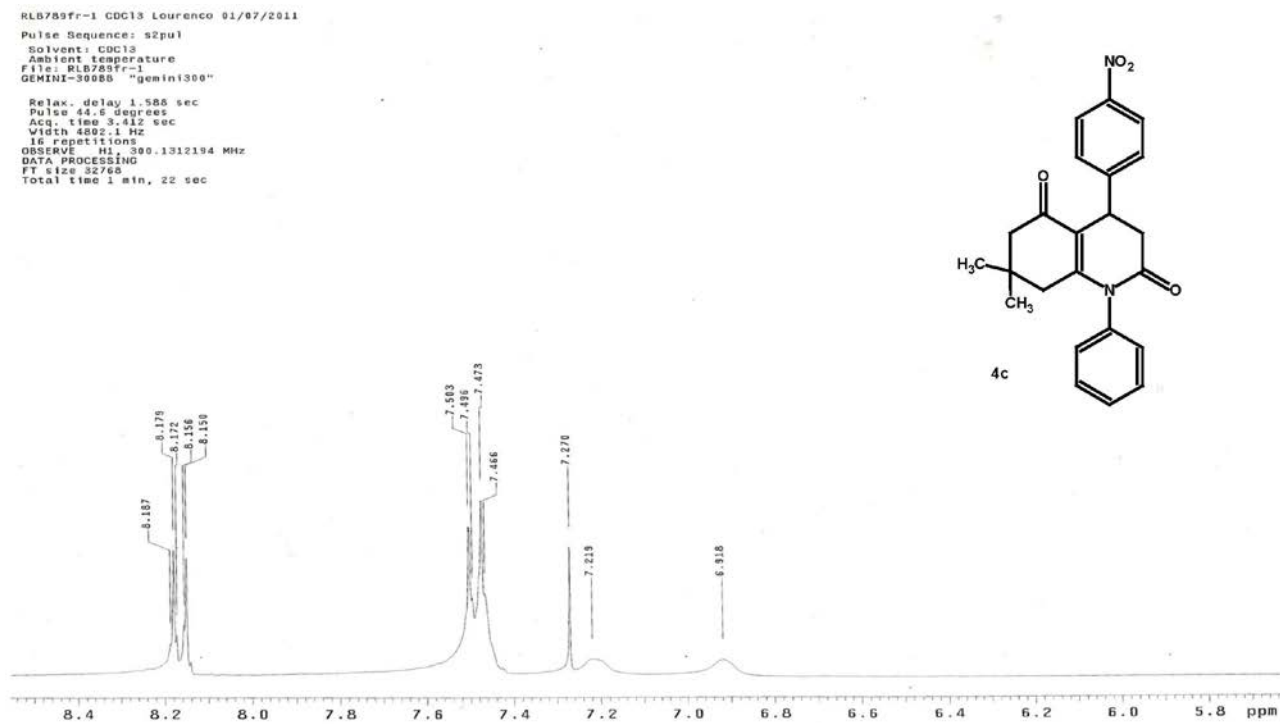
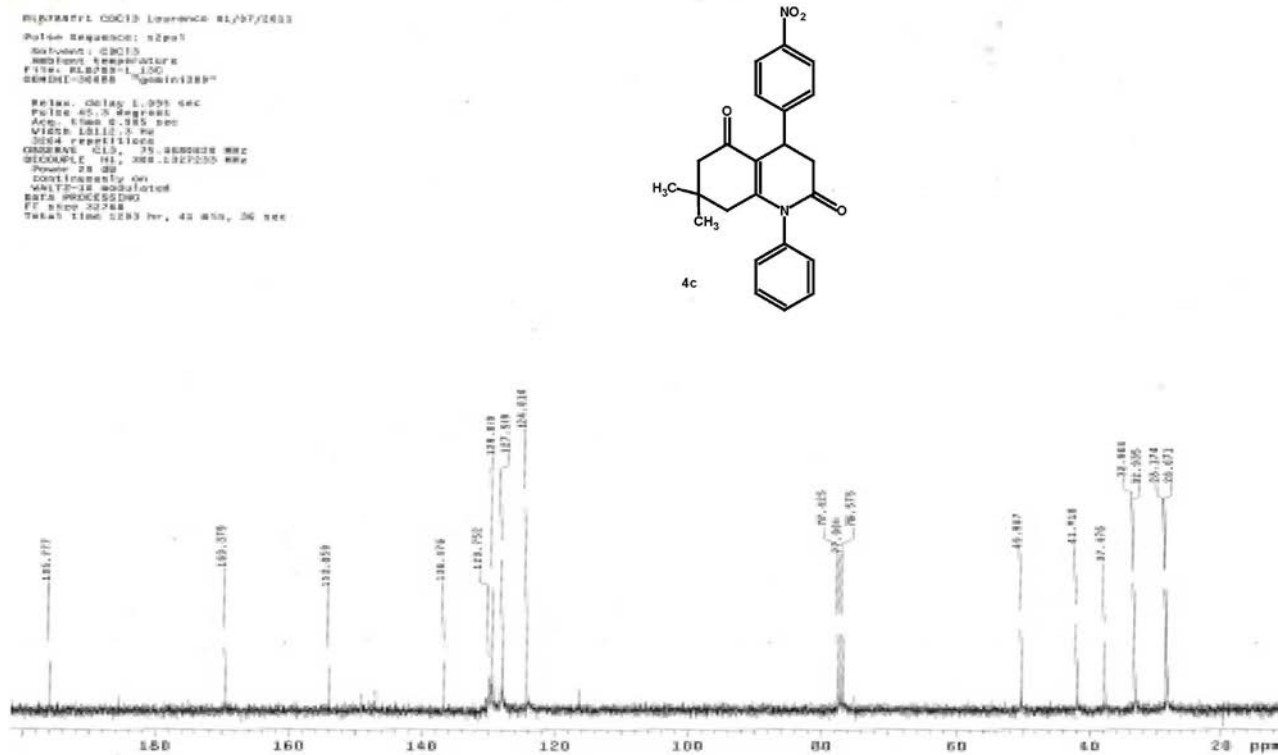
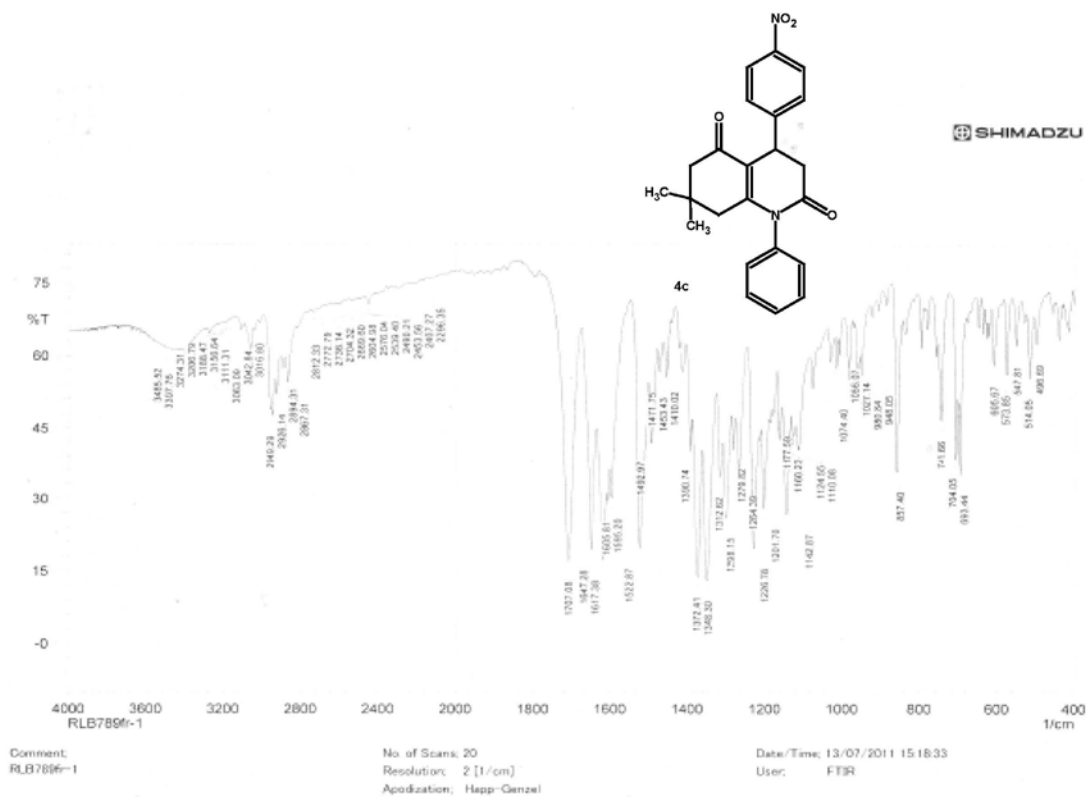
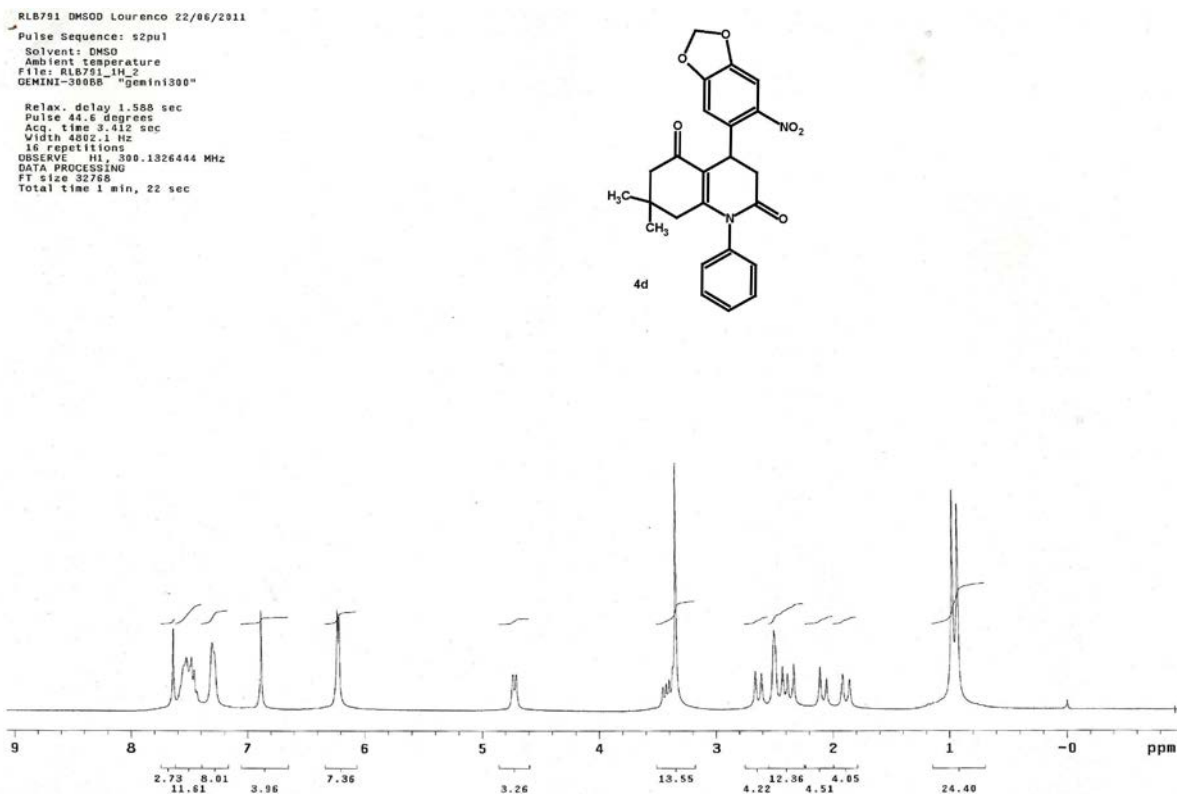
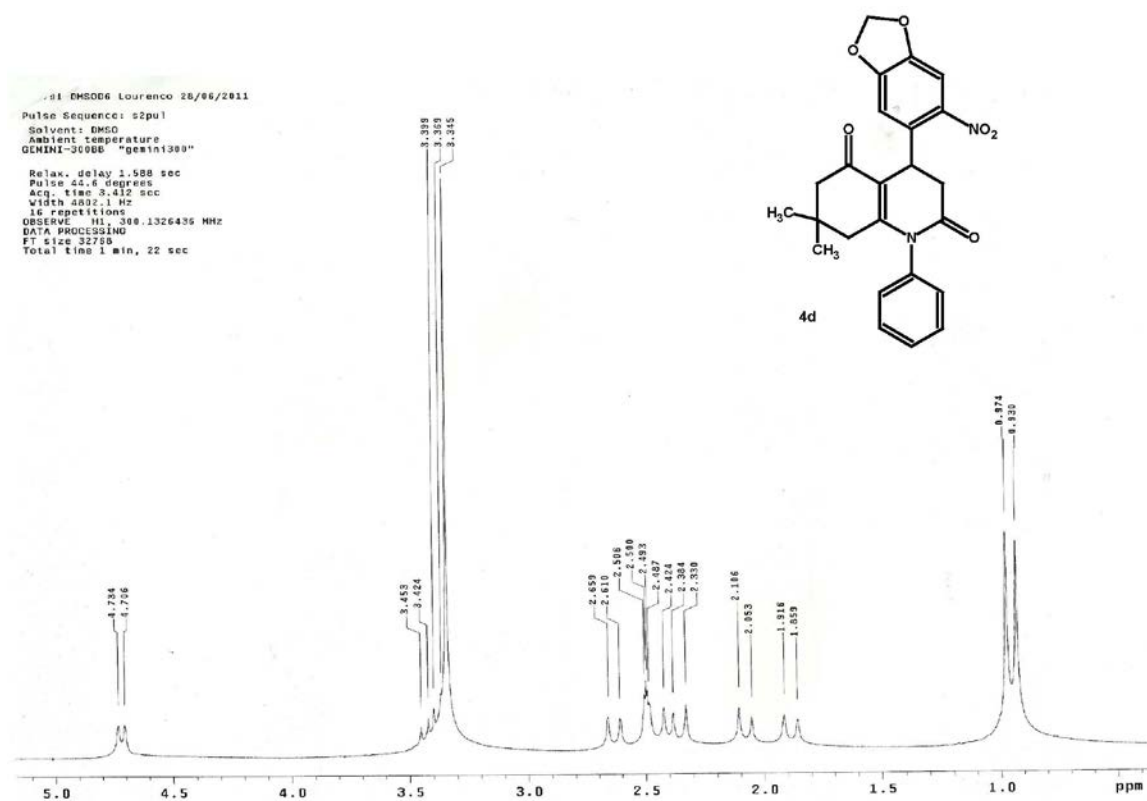
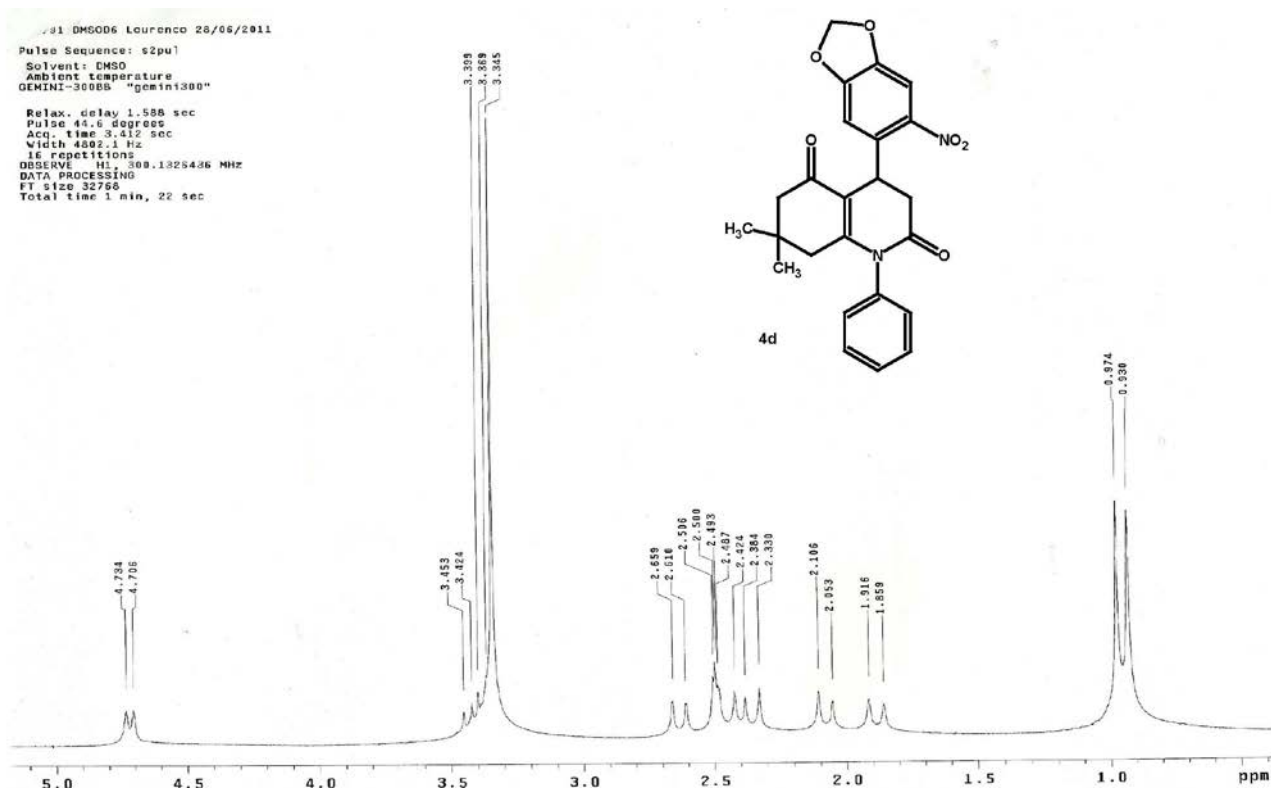
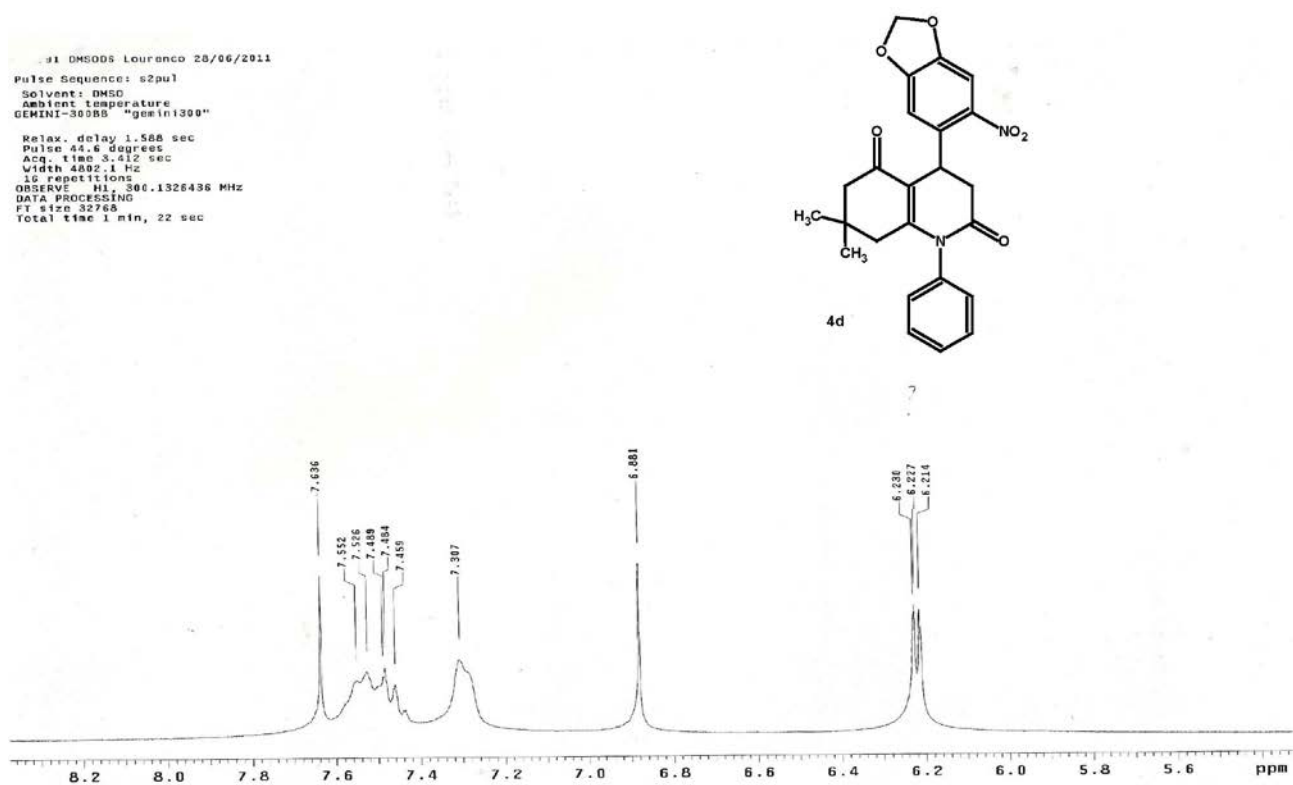


Figure S15. ¹H NMR (CDCl₃, 300 MHz) spectrum of compound **4c**.

Figure S16. Expanded ^1H NMR (CDCl_3 , 300 MHz) spectrum of compound 4c.Figure S17. Expanded ^1H NMR (CDCl_3 , 300 MHz) spectrum of compound 4c.

Figure S18. ¹³C NMR (CDCl₃, 75 MHz) spectrum of compound **4c**.Figure S19. IR (film) spectrum of compound **4c**.

Figure S20. ¹H NMR (DMSO-D₆, 300 MHz) spectrum of 4d.Figure S21. Expanded ¹H NMR (DMSO-D₆, 300 MHz) spectrum of 4d.

Figure S22. Expanded ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of **4d**.Figure S23. Expanded ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of **4d**.

RLB791 DMSO Lourenco 22/06/2011
 Pulse Sequence: s2pu1
 Solvent: DMSO
 Ambient temperature
 GEMINI-300BB "gemin1300"
 Relax. delay 1.095 sec
 Pulse 45.3 degrees
 Acq. time 0.905 sec
 Width 16112.3 Hz
 5000 repetitions
 OBSERVE C13, 75.4683927 MHz
 DECOUPLE H1, 300.1341489 MHz
 Power 29 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 12036 hr, 56 min, 4 sec

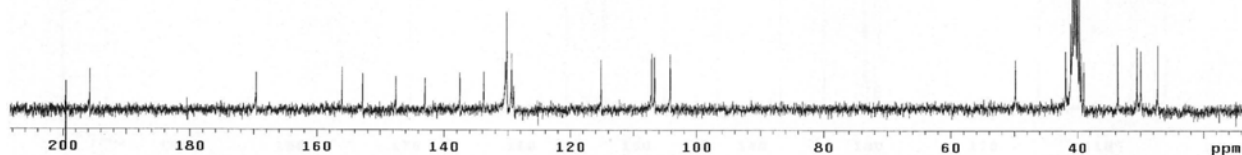
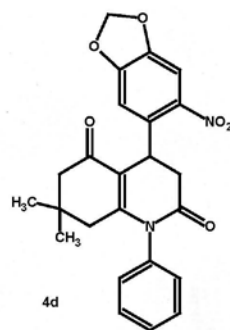


Figure S24. ^{13}C NMR (DMSO- D_6 , 75 MHz) spectrum of compound 4d.

RLB791 DMSO Lourenco 22/06/2011
 Pulse Sequence: s2pu1
 Solvent: DMSO
 Ambient temperature
 GEMINI-300BB "gemin1300"
 Relax. delay 1.095 sec
 Pulse 45.3 degrees
 Acq. time 0.905 sec
 Width 16112.3 Hz
 5000 repetitions
 OBSERVE C13, 75.4683927 MHz
 DECOUPLE H1, 300.1341489 MHz
 Power 29 dB
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 1.0 Hz
 FT size 32768
 Total time 12036 hr, 56 min, 4 sec

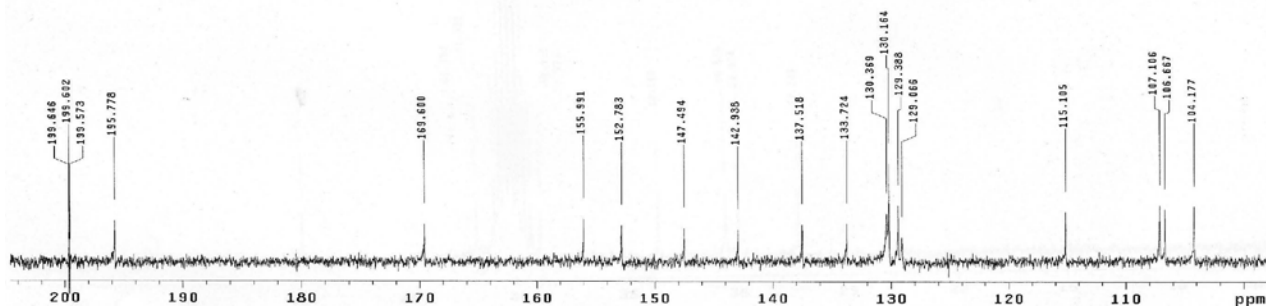
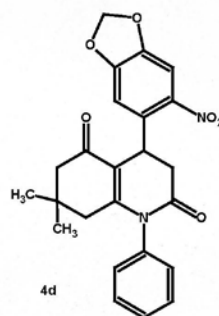
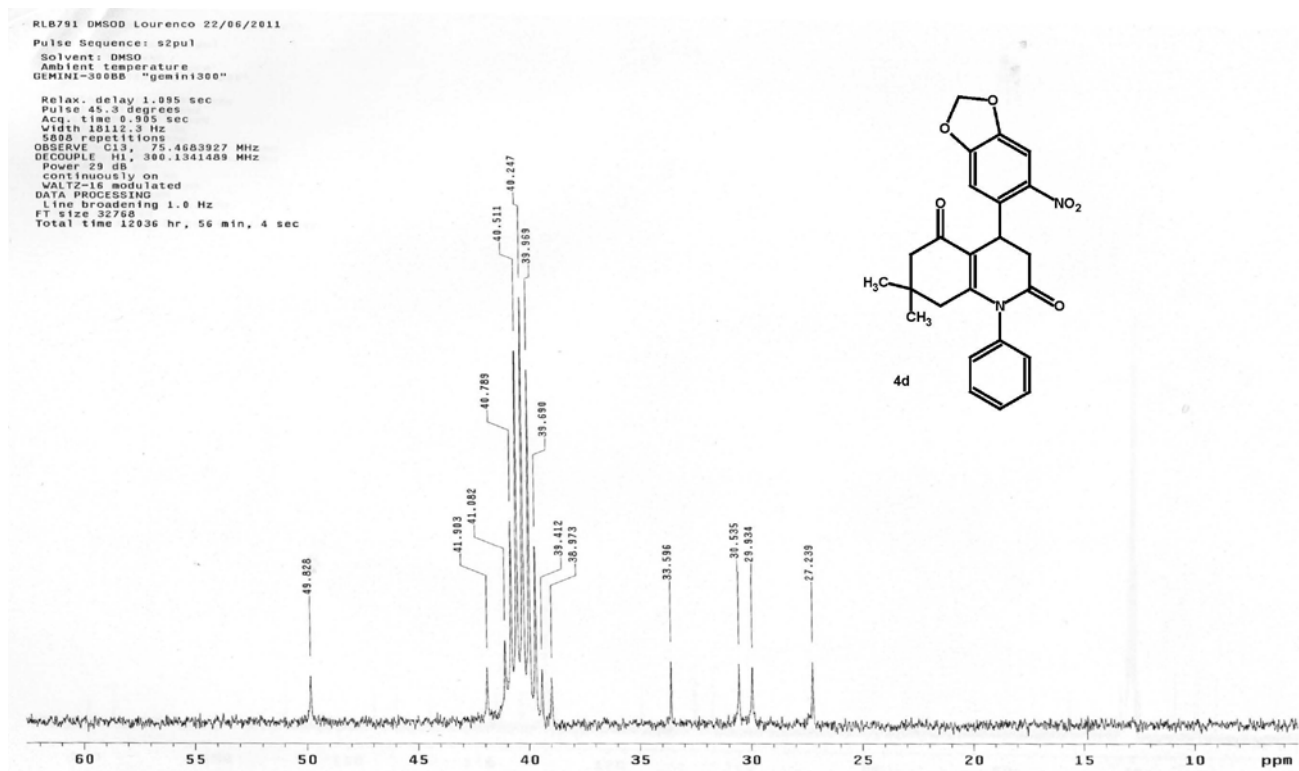
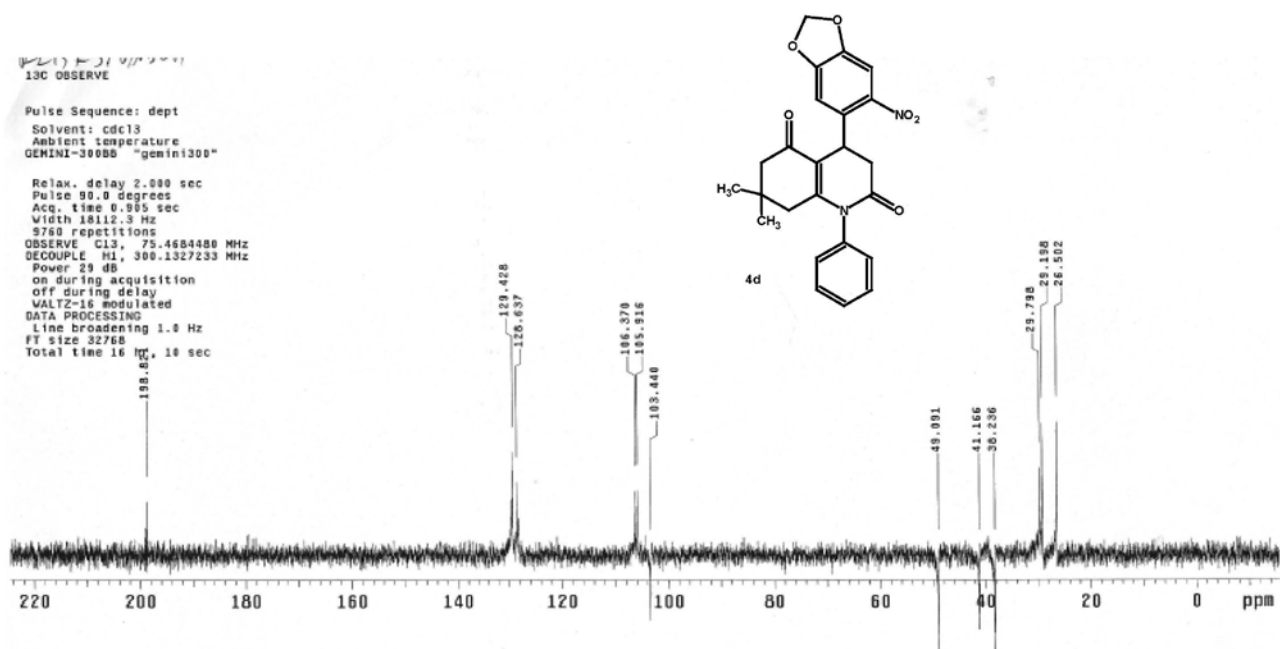
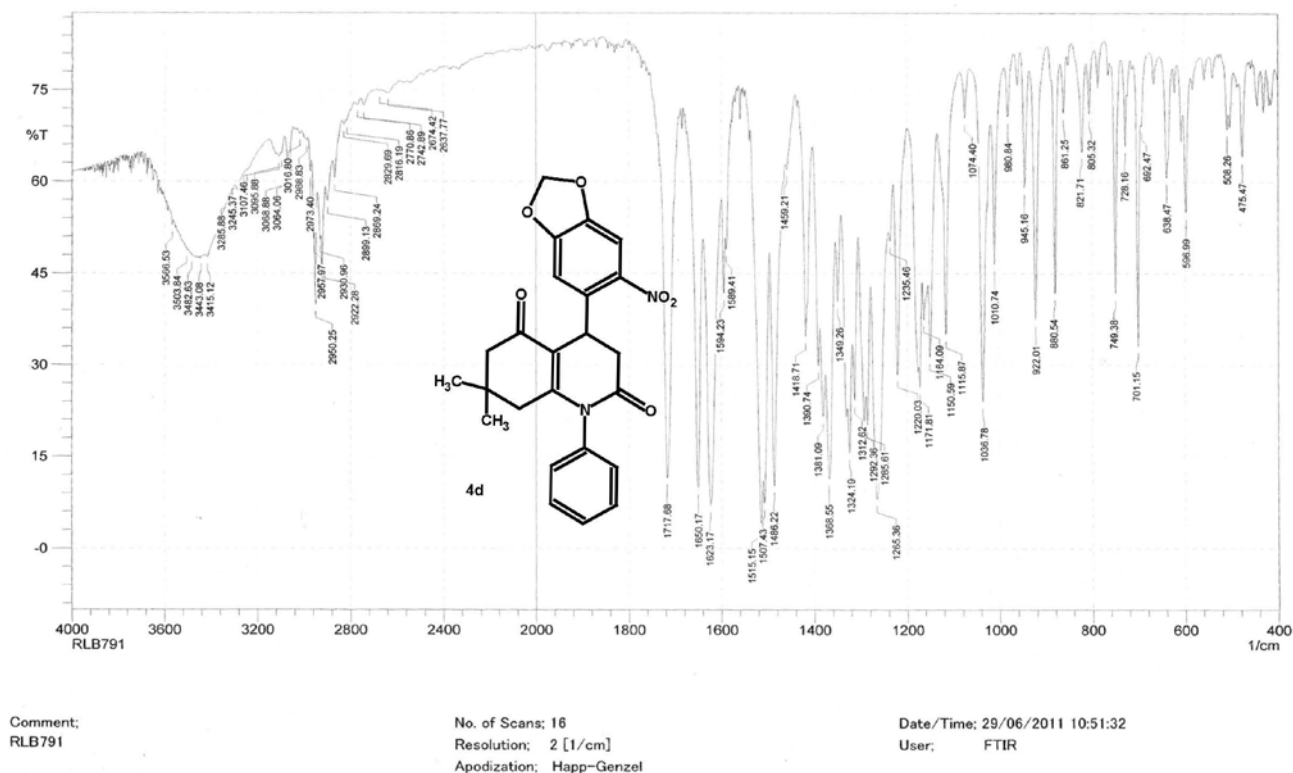
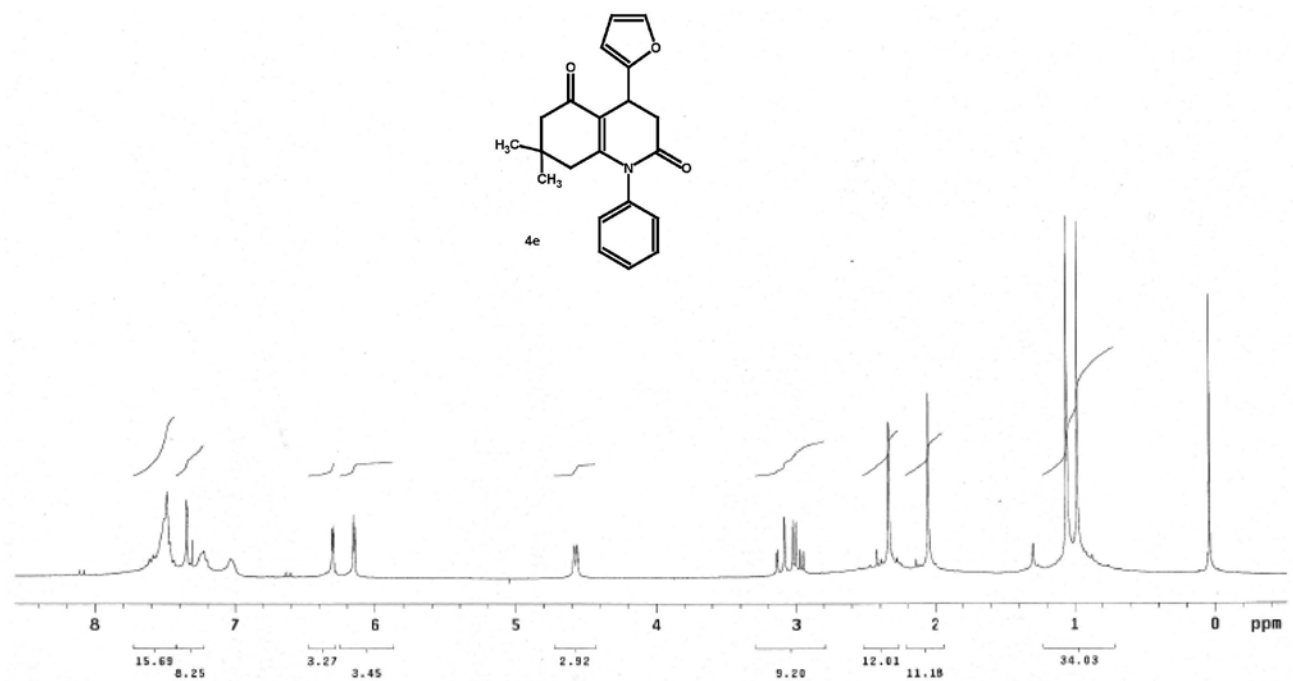


Figure S25. Expanded ^{13}C NMR (DMSO- D_6 , 75 MHz) spectrum of compound 4d.

Figure S26. Expanded ^{13}C NMR (DMSO- D_6 , 75 MHz) spectrum of compound **4d**.Figure S27. DEPT-135 NMR (DMSO- D_6 , 75 MHz) spectrum of compound **4d**.

Figure S28. IR (KBr) spectrum of compound **4d**.Figure S29. ¹H NMR (CDCl₃, 300 MHz) spectrum of **4e**.

RLB227 CDCl3 Lourenco 31/03/2011
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File: RLB227_1MR
GEMINI-300BB "gemin300"

Relax. delay 1.588 sec
Pulse 44.6 degrees
Acq. time 3.612 sec
Width 4862.1 Hz
16 repetitions
OBSERVE H1, 300.1312079 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 22 sec

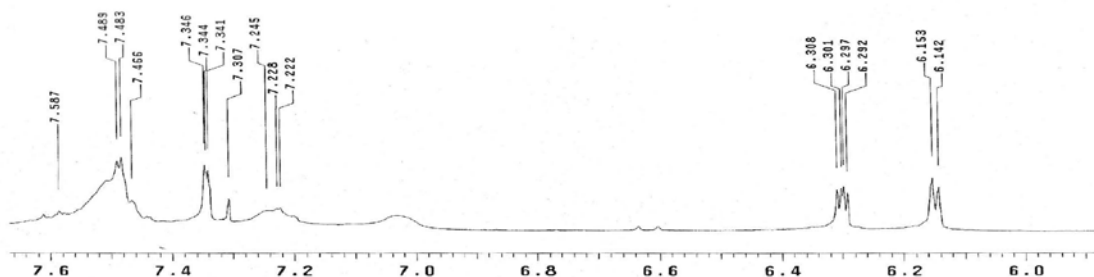
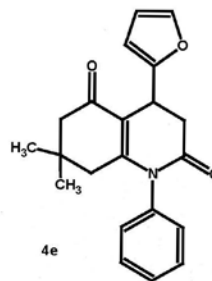


Figure S30. Expanded ^1H NMR (CDCl_3 , 300 MHz) spectrum of **4e**.

RLB227 CDCl3 Lourenco 31/03/2011

Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature

File: RLB227_1MR

GEMINI-300BB "gemin300"

Relax. delay 1.588 sec

Pulse 44.6 degrees

Acq. time 3.612 sec

Width 4862.1 Hz

16 repetitions

OBSERVE H1, 300.1312079 MHz

DATA PROCESSING

FT size 32768

Total time 1 min, 22 sec

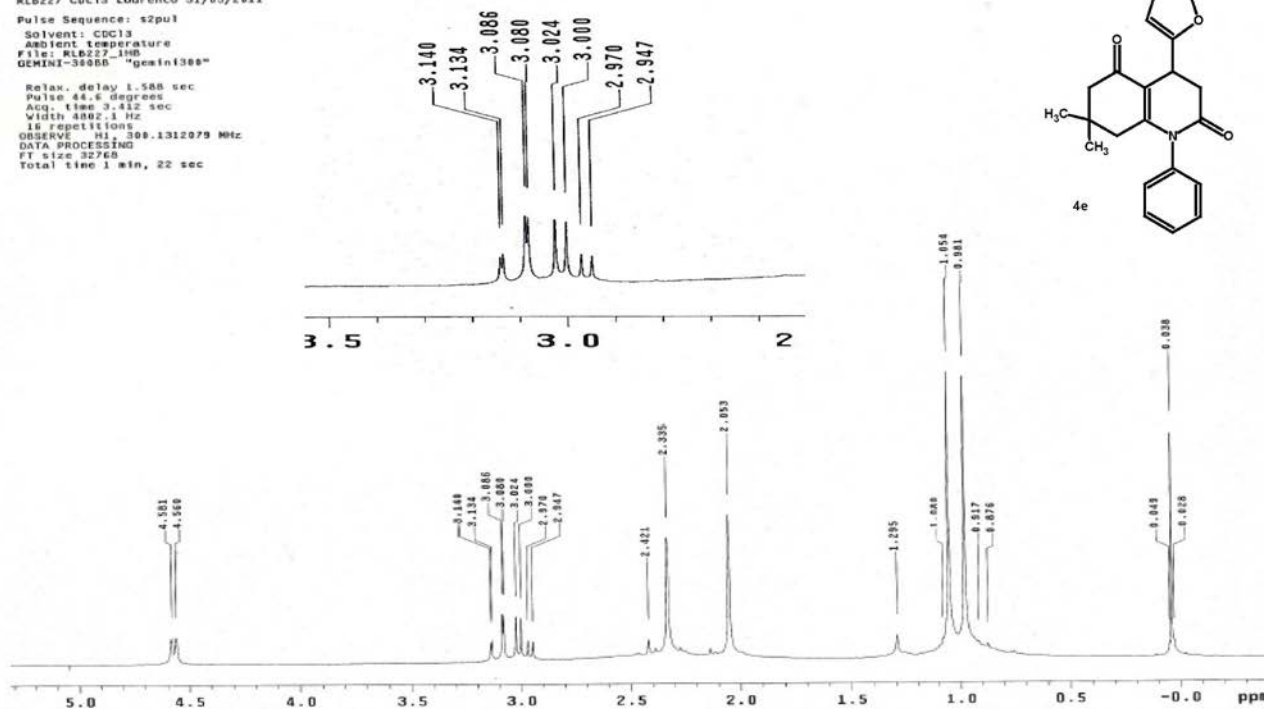
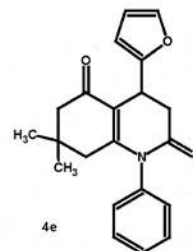


Figure S31. Expanded ^1H NMR (CDCl_3 , 300 MHz) spectrum of **4e**.

13C NMR
 File: 00000001
 Name: 00000001
 Date: 00/00/00
 Time: 00:00:00
 Solvent: CDCl3
 Concentration: 100 mg/mL
 Temperature: 25.00°C
 Frequency: 75.00 MHz
 Acquisition: 01.00
 Processing: 01.00
 F2: 50.00 MHz
 F1: 50.00 MHz
 T1: 5.00 s
 T2: 5.00 s
 T3: 5.00 s
 T4: 5.00 s
 T5: 5.00 s
 T6: 5.00 s
 T7: 5.00 s
 T8: 5.00 s
 T9: 5.00 s
 T10: 5.00 s
 T11: 5.00 s
 T12: 5.00 s
 T13: 5.00 s
 T14: 5.00 s
 T15: 5.00 s
 T16: 5.00 s
 T17: 5.00 s
 T18: 5.00 s
 T19: 5.00 s
 T20: 5.00 s
 T21: 5.00 s
 T22: 5.00 s
 T23: 5.00 s
 T24: 5.00 s
 T25: 5.00 s
 T26: 5.00 s
 T27: 5.00 s
 T28: 5.00 s
 T29: 5.00 s
 T30: 5.00 s
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 T35: 5.00 s
 T36: 5.00 s
 T37: 5.00 s
 T38: 5.00 s
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 T40: 5.00 s
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 T91: 5.00 s
 T92: 5.00 s
 T93: 5.00 s
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 T97: 5.00 s
 T98: 5.00 s
 T99: 5.00 s
 T100: 5.00 s

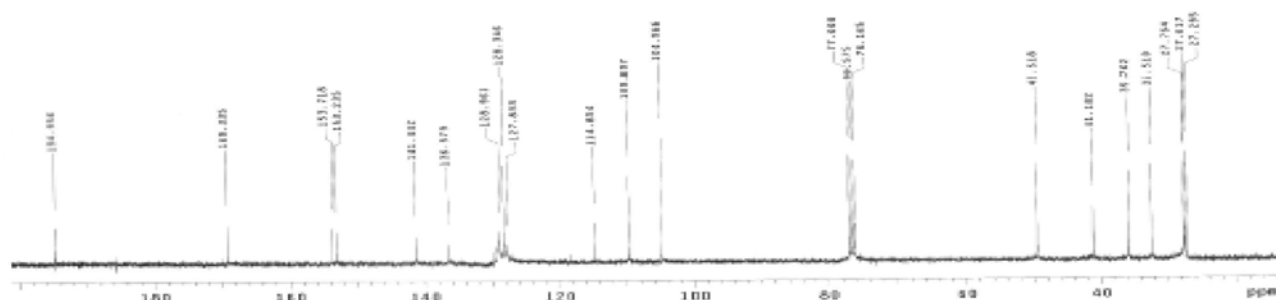
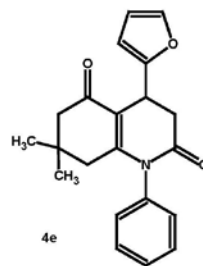


Figure S32. ^{13}C NMR(CDCl_3 , 75 MHz) spectrum of 4e.

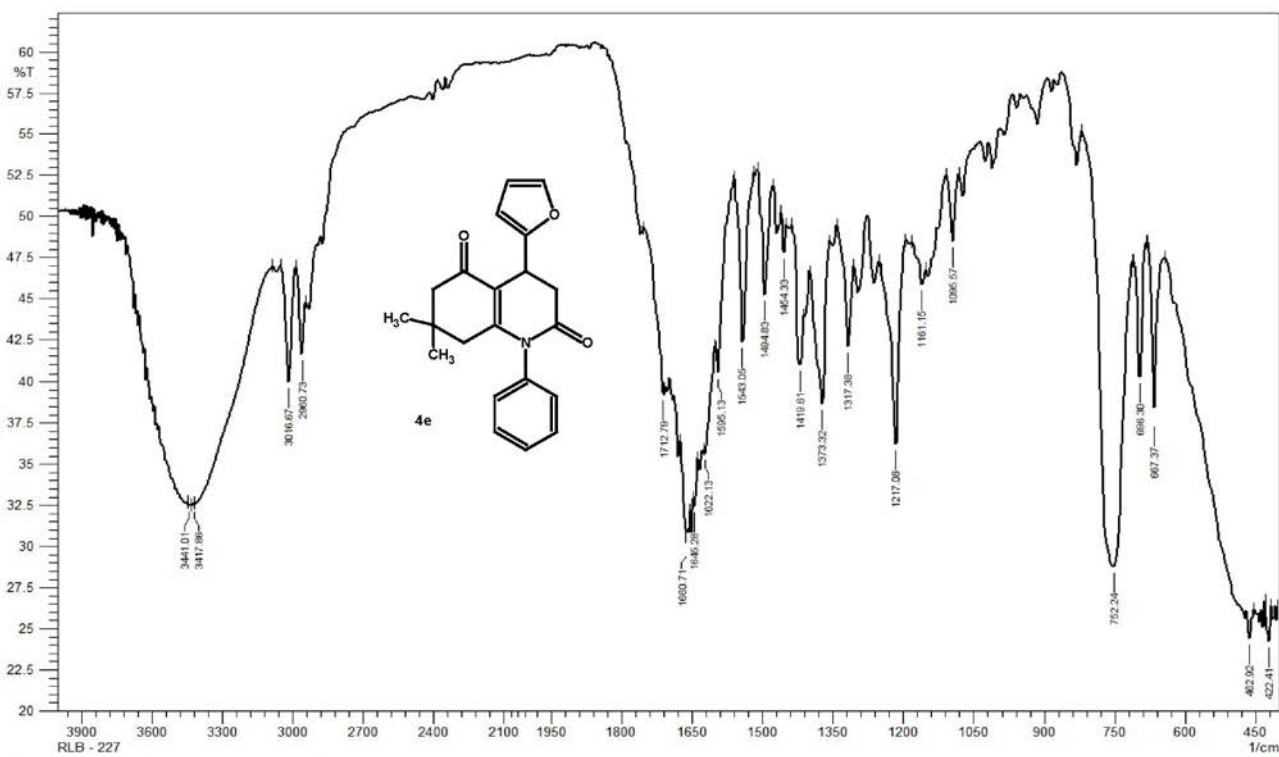
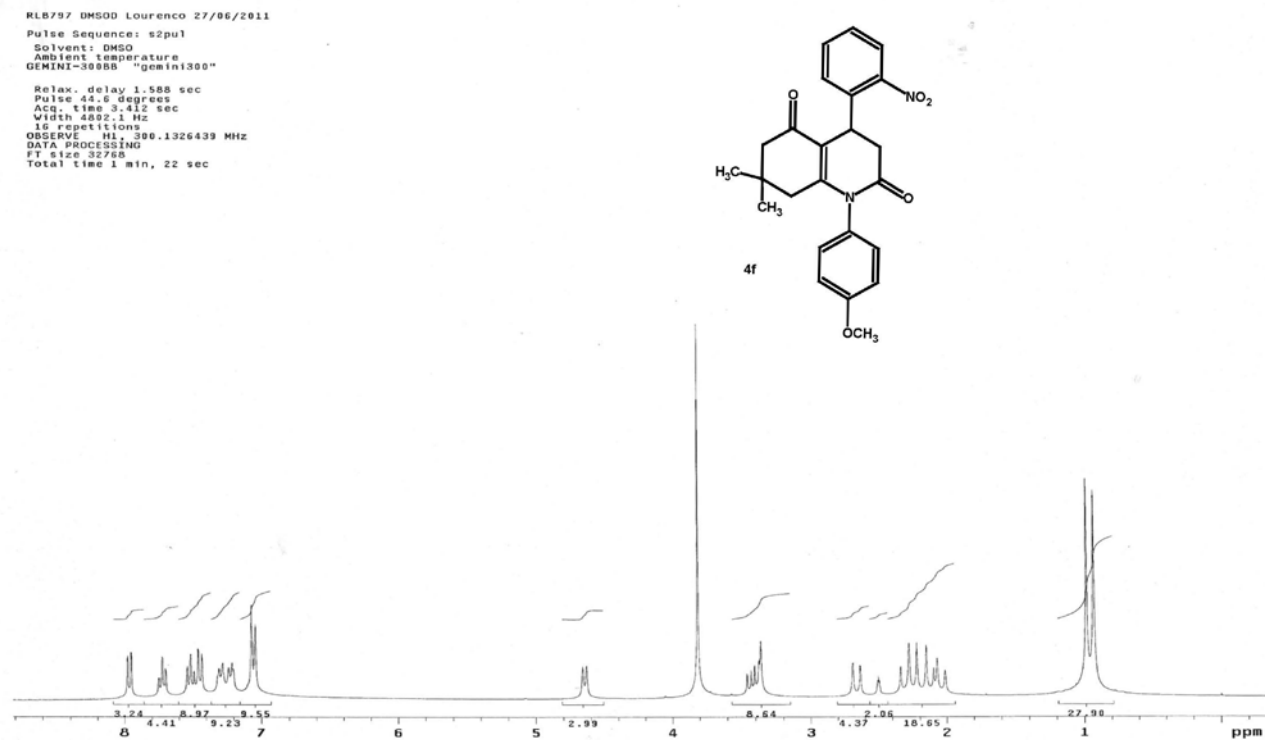
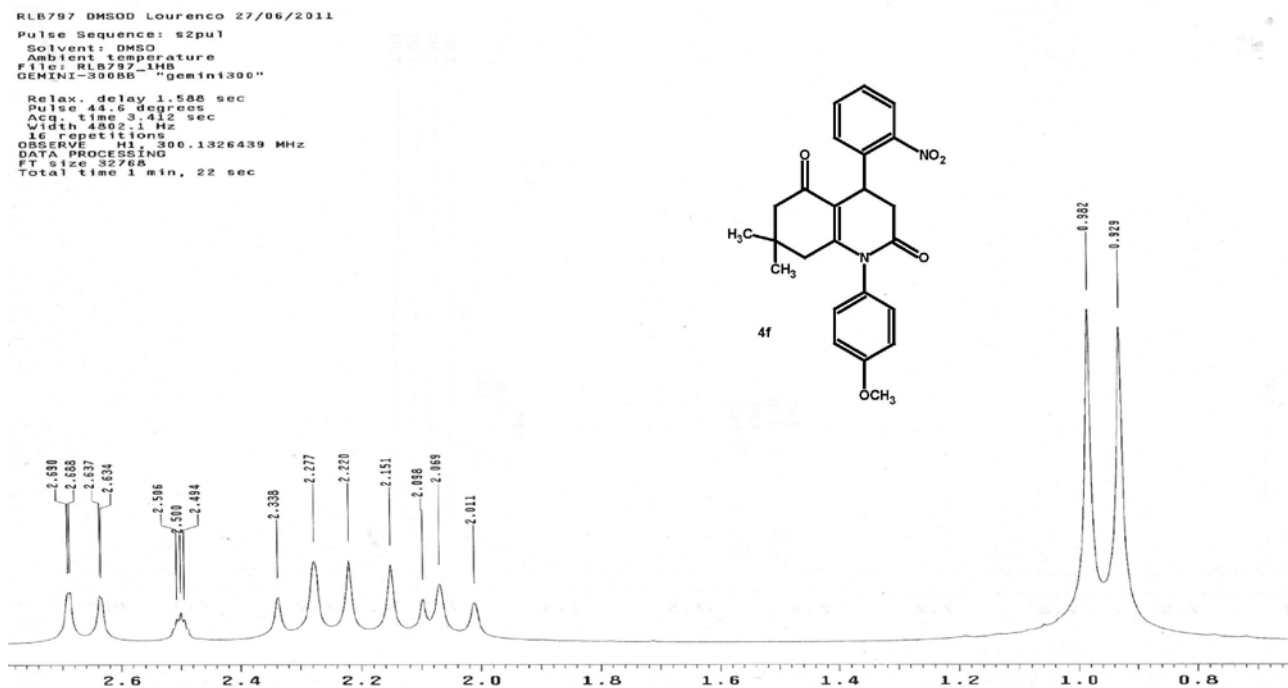


Figure S33. IR (film) spectrum of 4e.

Figure S34. ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of **4f**.Figure S35. Expanded ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of **4f**.

RLB797 DMSO-D₆ Lourenco 27/06/2011
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: RLB797_1HR
GEMINI-3000S "gemin300"
Relax. delay 1.500 sec
Pulse 44.6 degrees
Acq. time 3.412 sec
Width 4862.1 Hz
16 repetitions
OBSERVE H1: 300.1326439 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 22 sec

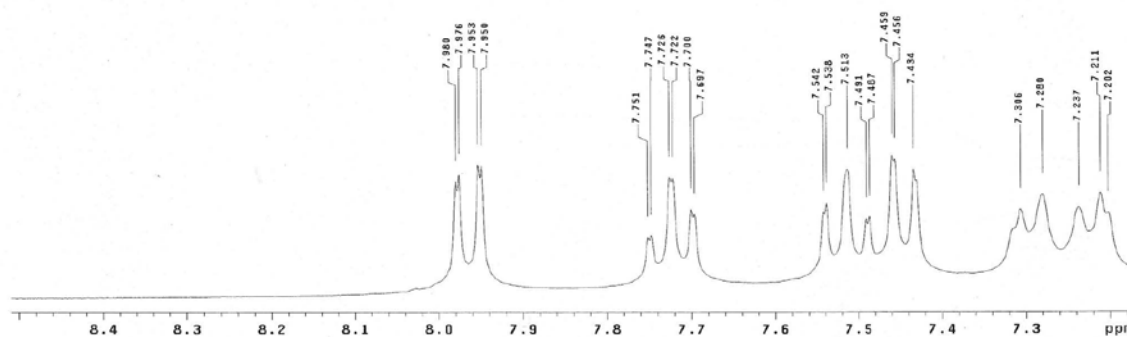
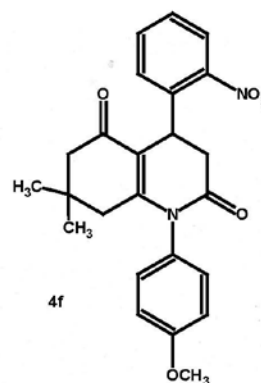


Figure S36. Expanded ¹H NMR (DMSO-D₆, 300 MHz) spectrum of 4f.

RLB797 DMSO-D₆ Lourenco 27/06/2011
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
File: RLB797_1HR
GEMINI-3000S "gemin300"
Relax. delay 1.500 sec
Pulse 44.6 degrees
Acq. time 3.412 sec
Width 4862.1 Hz
16 repetitions
OBSERVE H1: 300.1326439 MHz
DATA PROCESSING
FT size 32768
Total time 1 min, 22 sec

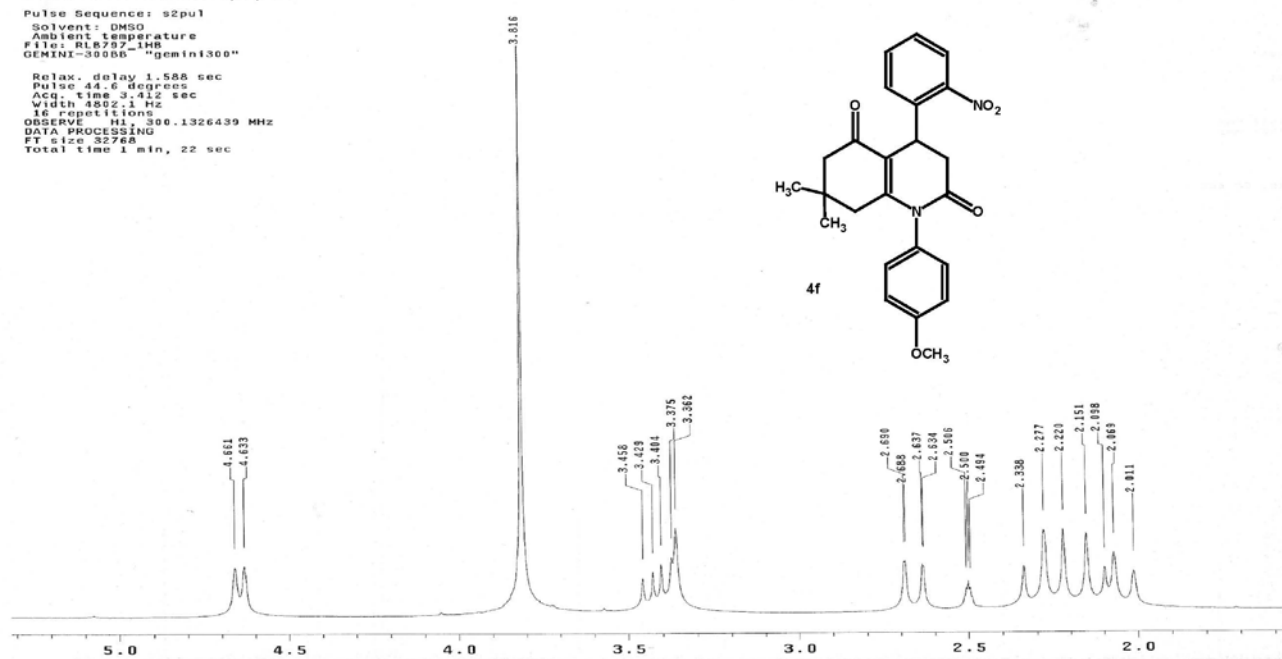
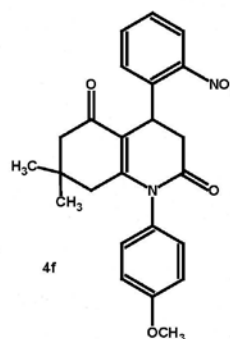


Figure S37. Expanded ¹H NMR (DMSO-D₆, 300 MHz) spectrum of 4f.

RLB797 DMSO-D₆ Lourenco 23/06/2011
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
GEMINI-36000 "gemin3600"
Relax. delay 1.095 sec
Pulse 45.3 degrees
Acq. time 0.305 sec
Width 16112.3 Hz
2280 repetitions
OBSERVE C13, 75.4684491 MHz
DECOUPLE H1, 399.1301489 MHz
Power 29 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 12036 hr, 56 min, 4 sec

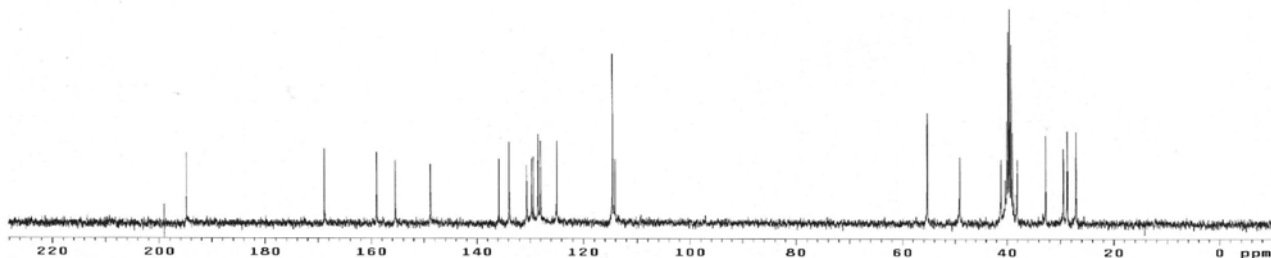
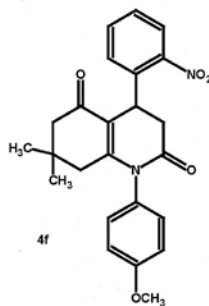


Figure S38. ¹³C NMR (DMSO-D₆, 75 MHz) spectrum of 4f.

RLB797 DMSO-D₆ Lourenco 23/06/2011
Pulse Sequence: s2pul
Solvent: DMSO
Ambient temperature
GEMINI-36000 "gemin3600"
Relax. delay 1.095 sec
Pulse 45.3 degrees
Acq. time 0.305 sec
Width 16112.3 Hz
2280 repetitions
OBSERVE C13, 75.4684491 MHz
DECOUPLE H1, 399.1301489 MHz
Power 29 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 32768
Total time 12036 hr, 56 min, 4 sec

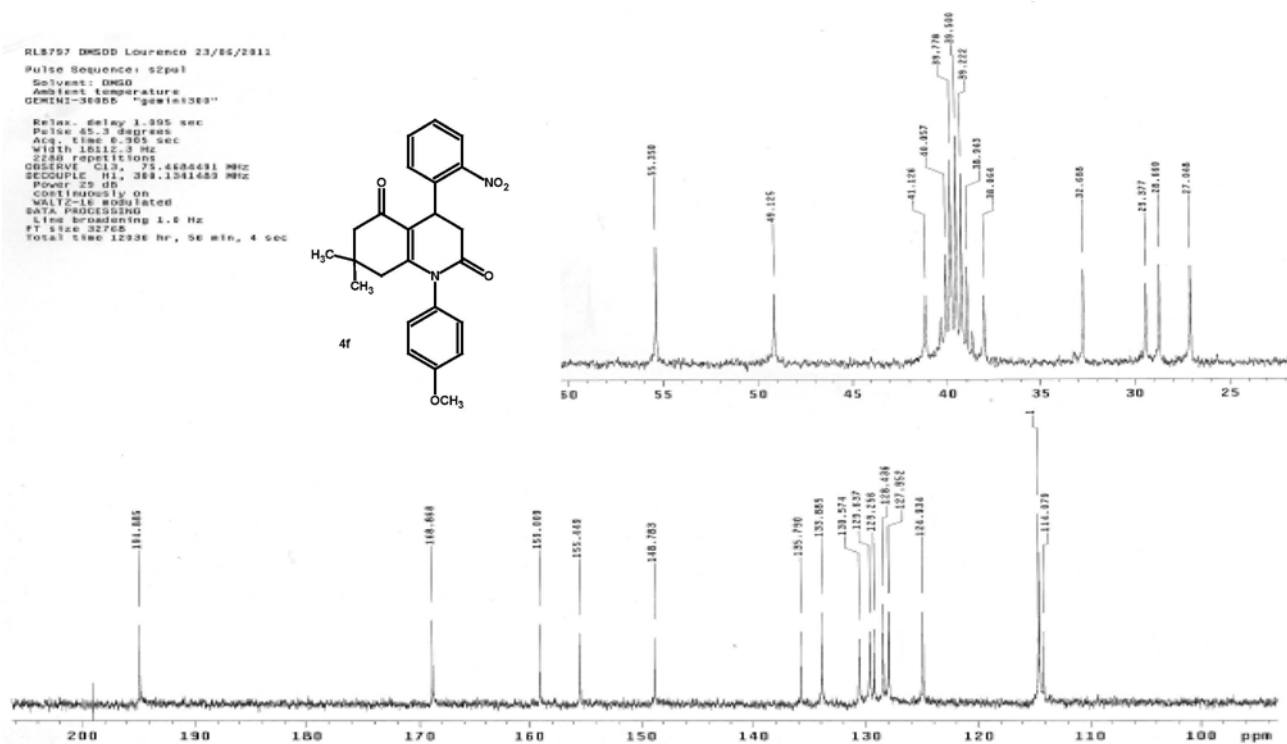
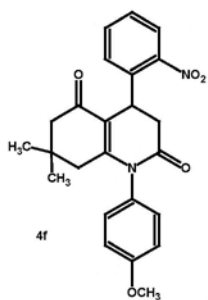


Figure S39. Expanded ¹³C NMR (DMSO-D₆, 75 MHz) spectrum of 4f.

RLB797 DEPT DMSO-D₆ Lourenco 27/06/2011

Pulse Sequence: dept

Solvent: dcd13

Ambient temperature

GEMINI-300BB "gemin1300"

Relax. delay 2.000 sec

Pulse 90.0 degrees

Acq. time 0.905 sec

Width 18112.3 Hz

1392 repetitions

OBSERVE C13, 75.4684491 MHz

DECOUPLE H1, 399.1327233 MHz

Power 29 dB

on during acquisition

off during delay

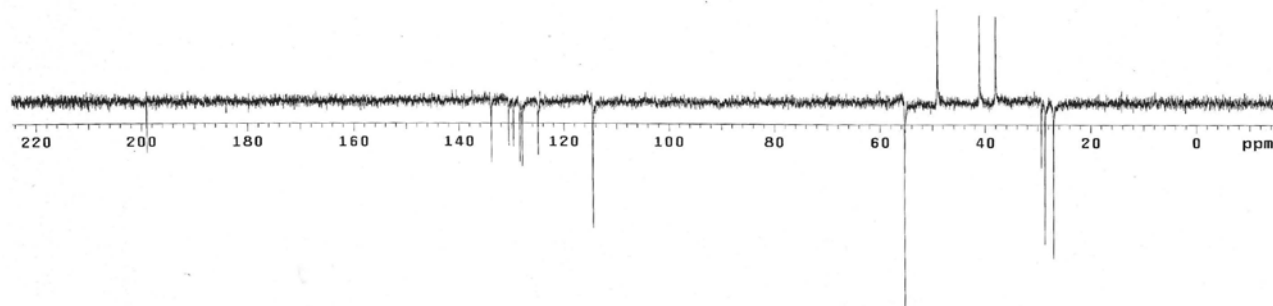
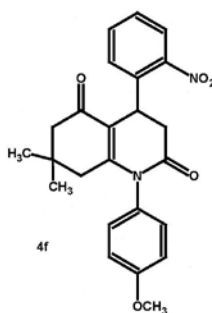
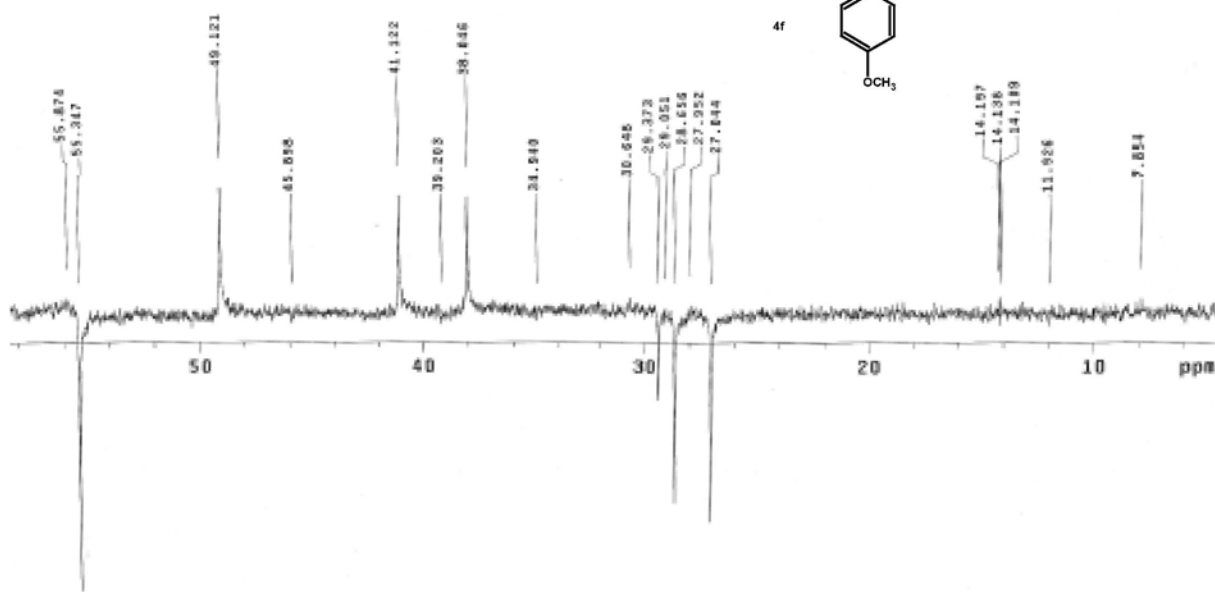
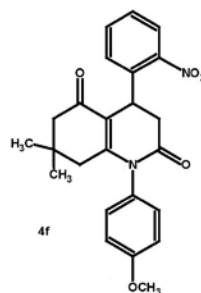
WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 32768

Total time 3 hr, 13 sec

Figure S40. DEPT 135 NMR (DMSO-D₆, 75 MHz) spectrum of 4f.Figure S41. Expanded DEPT 135 NMR (DMSO-D₆, 75 MHz) spectrum of 4f.

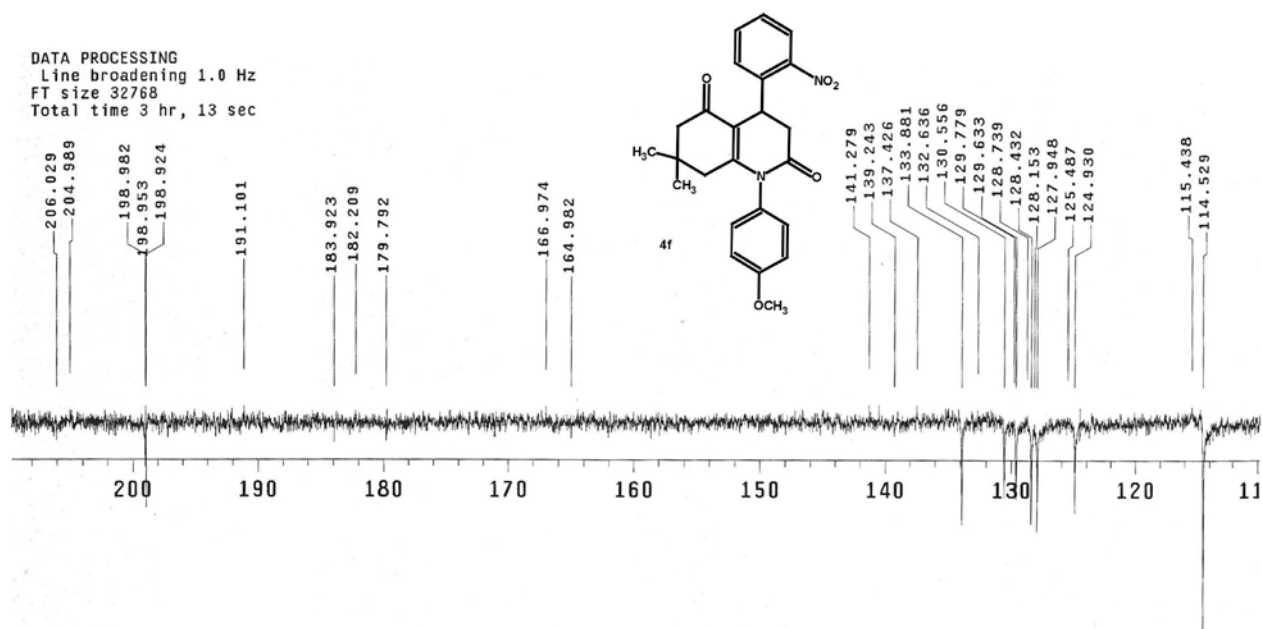


Figure S42. Expanded DEPT 135 NMR (DMSO-D₆, 75 MHz) spectrum of **4f**.

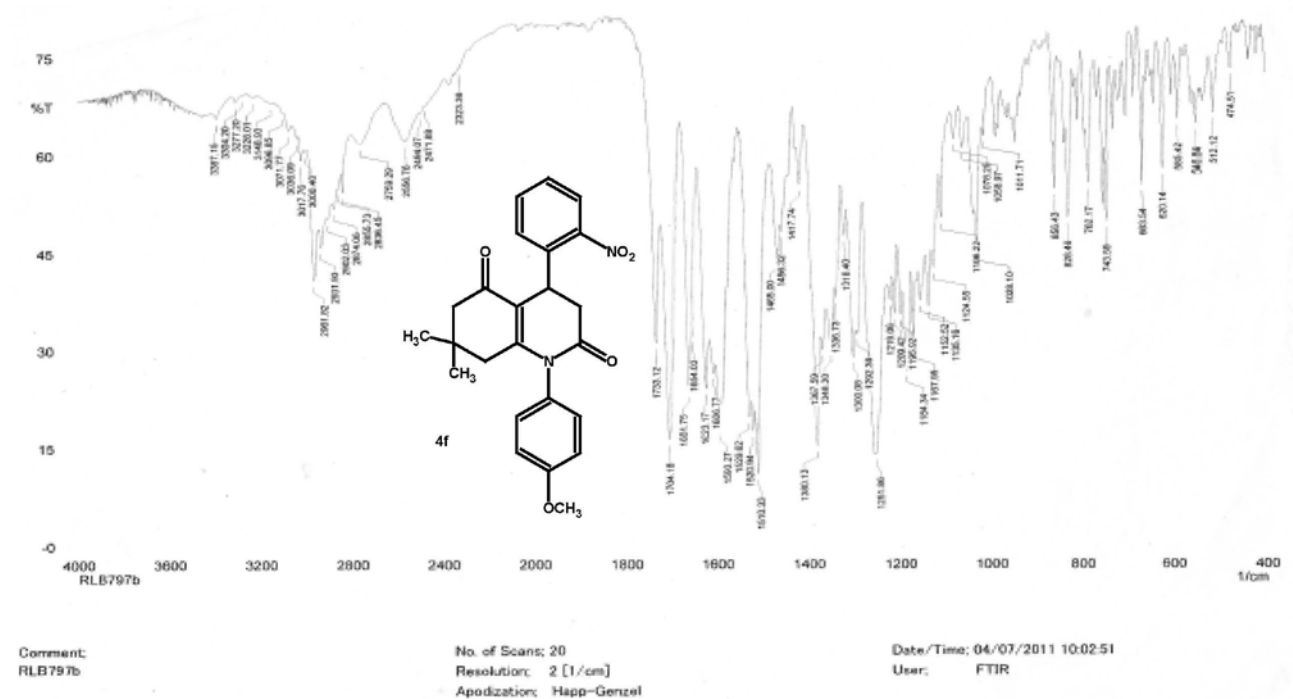


Figure S43. IR (KBr) spectrum of compound **4f**.

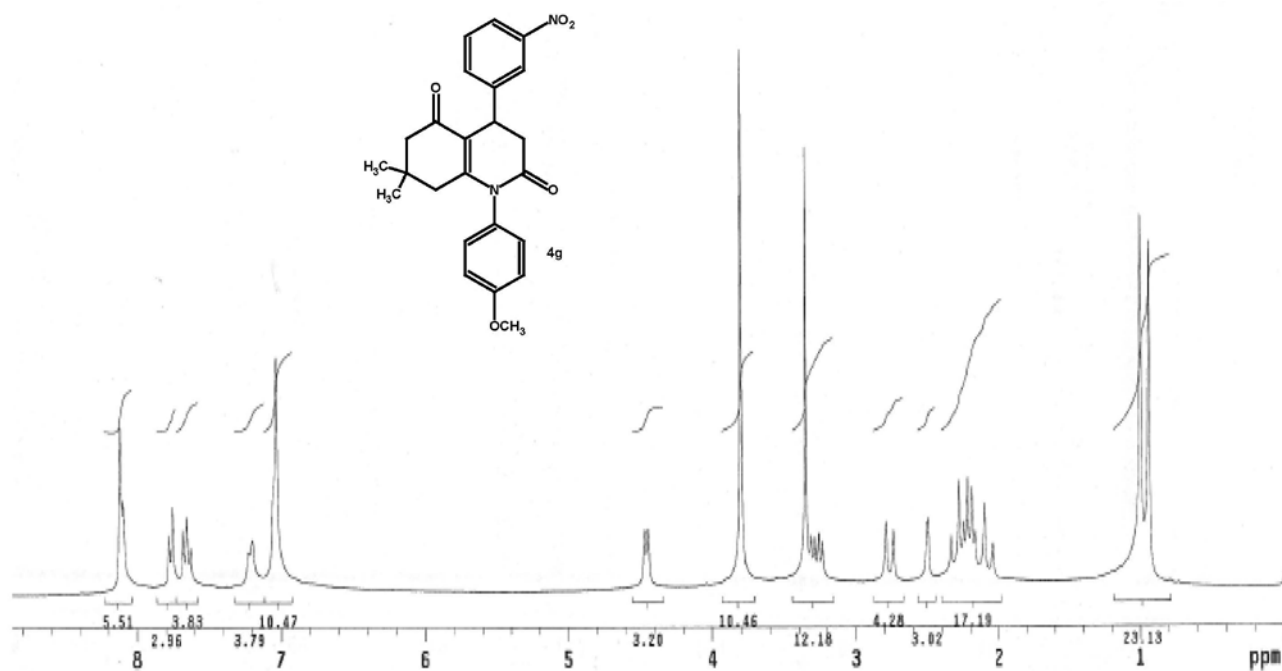


Figure S44. ¹H NMR (DMSO-D₆, 300 MHz) spectrum of 4g.

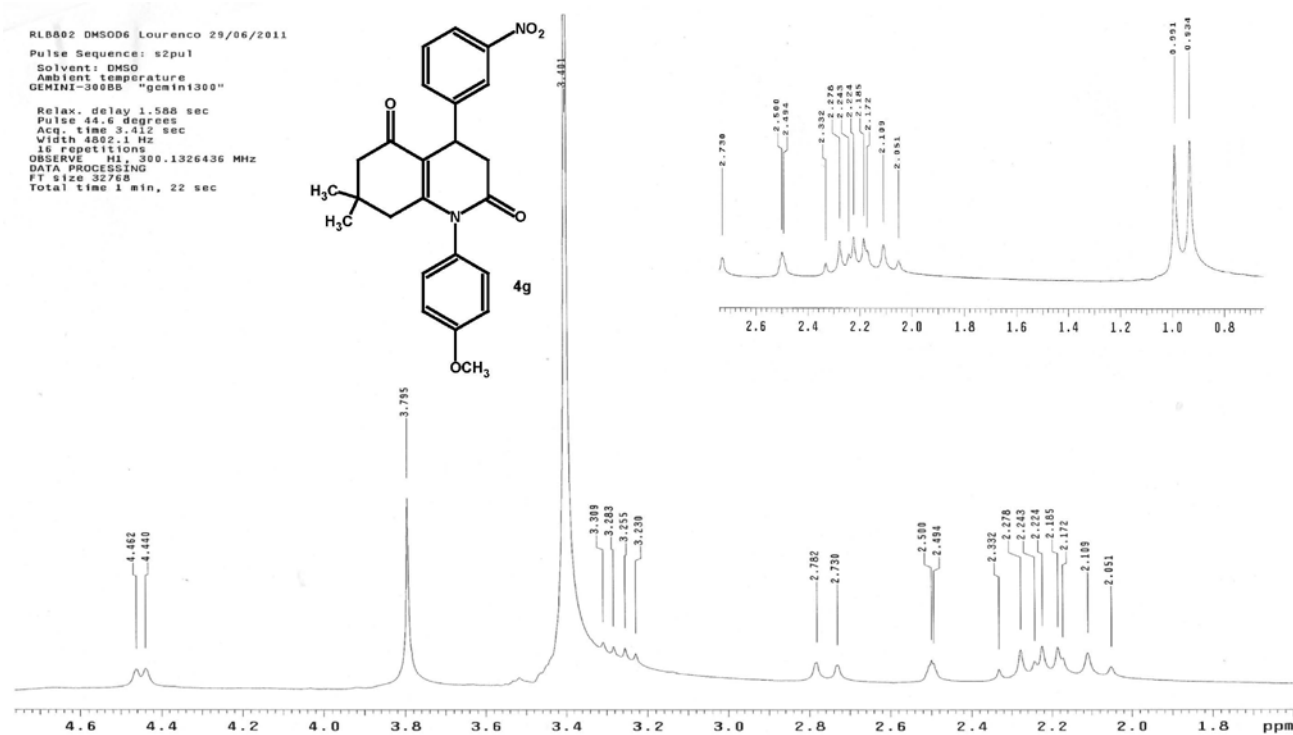


Figure S45. Expanded ¹H NMR (DMSO-D₆, 300 MHz) spectrum of 4g.

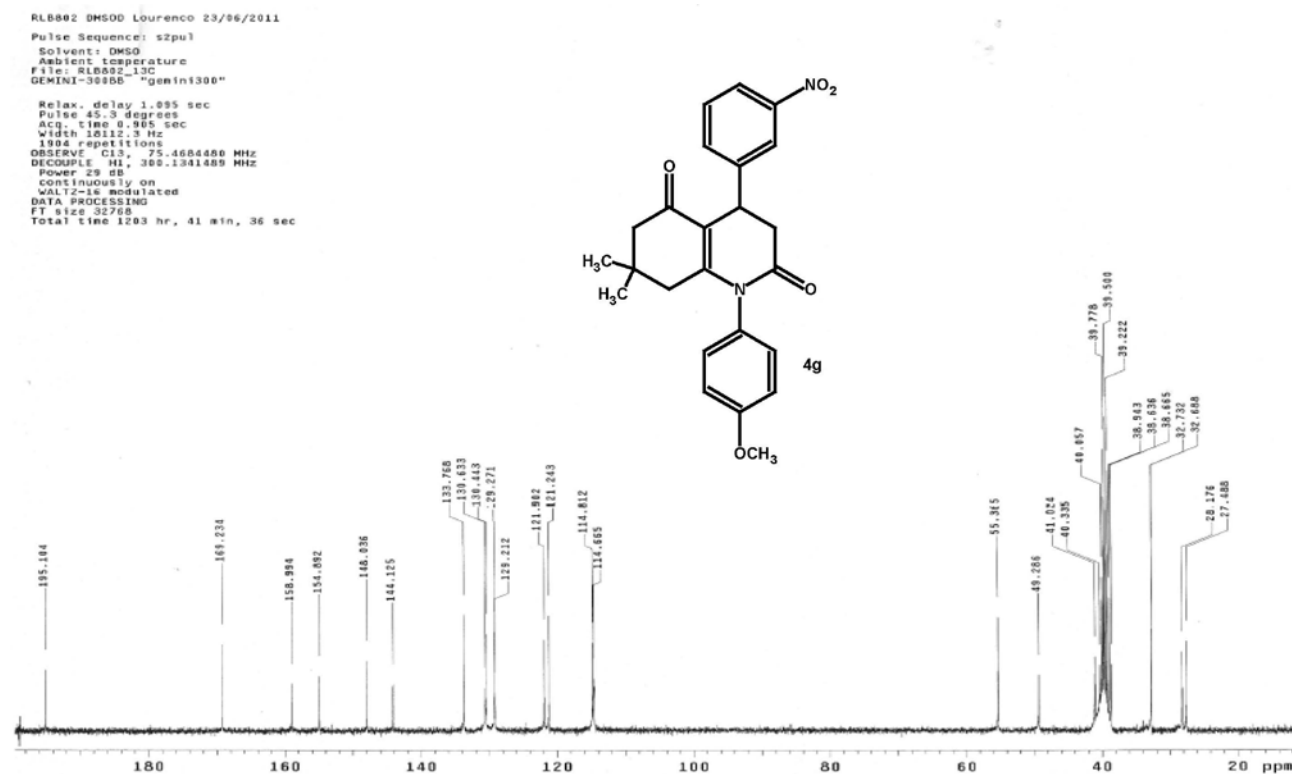
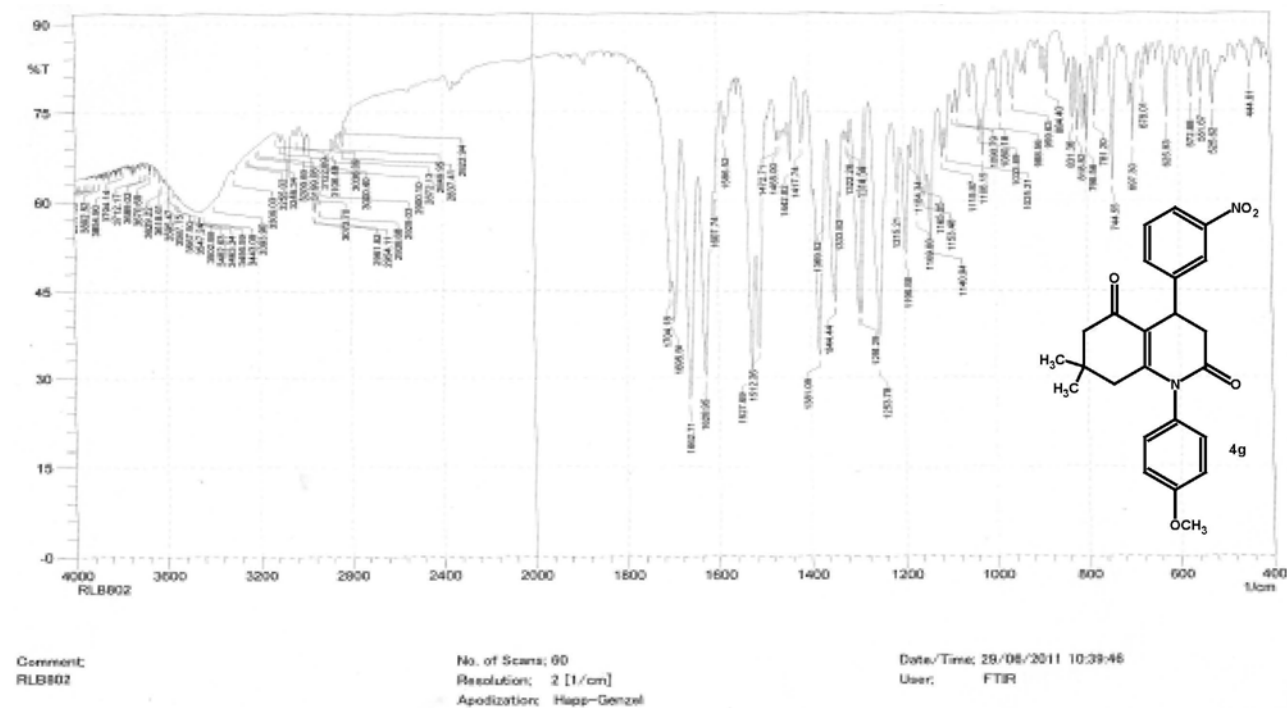
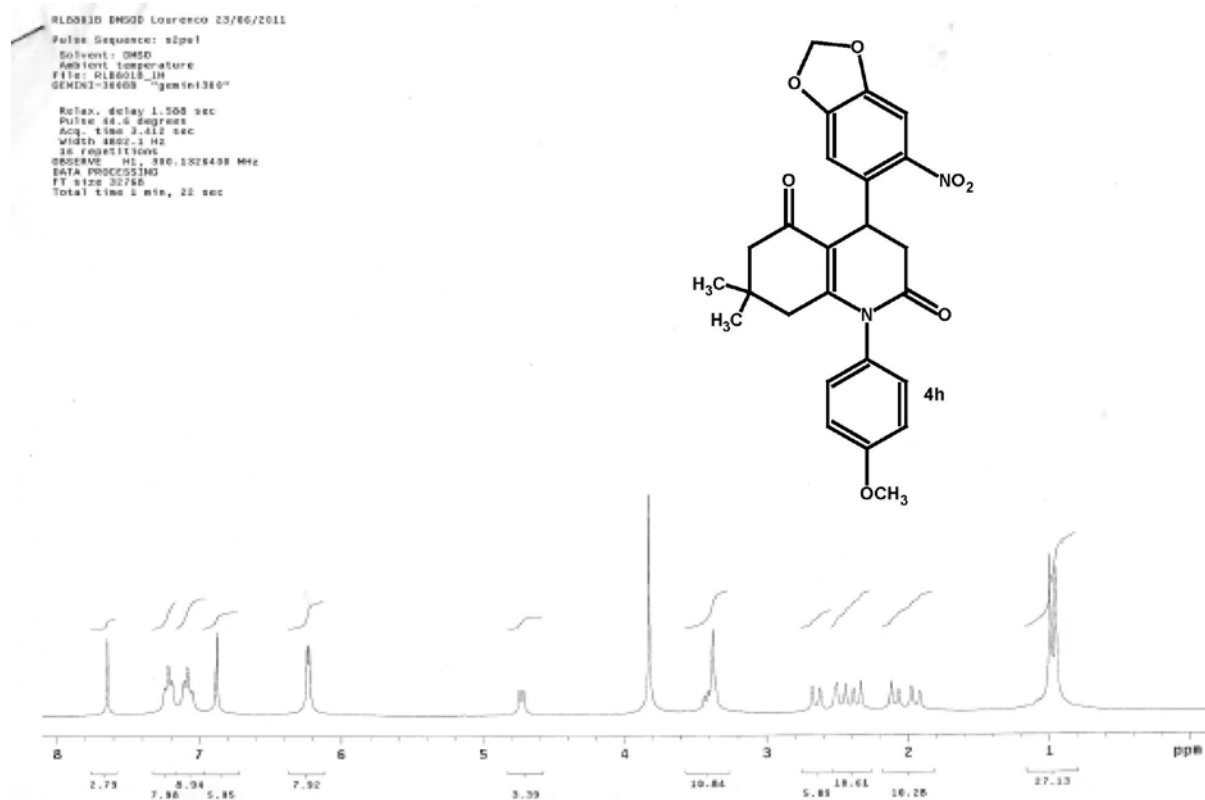
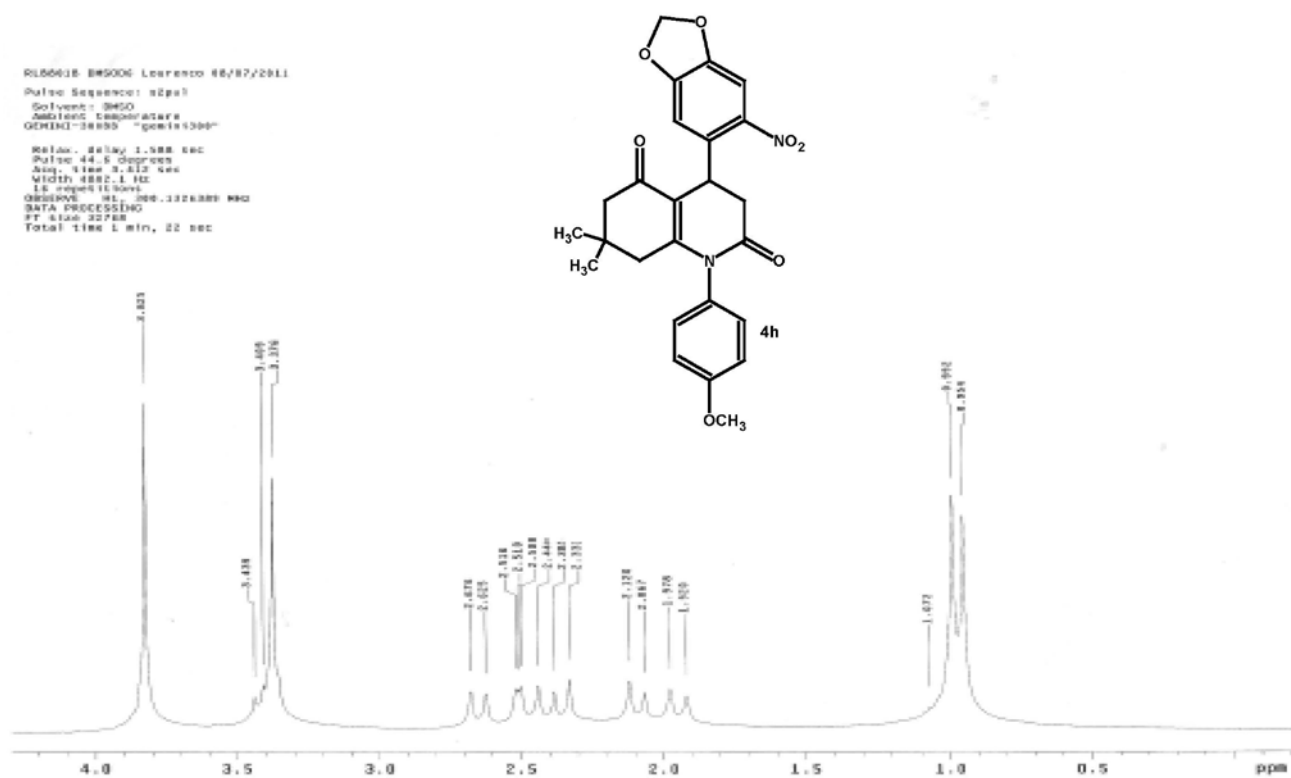
Figure S46. ^{13}C NMR (DMSO- D_6 , 75 MHz) spectrum of 4g.

Figure S47. IR (KBr) spectrum of compound 4g.

Figure S48. ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of 4h.Figure S49. Expanded ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of 4h.

RL8891B DMSD06 Lourenco 08/07/2011
 Pulse Sequence: s2pu1
 Solvent: DMSO
 Ambient temperature
 GEMINI-300BB "gemin300"
 Relax. delay 1.588 sec
 Pulse 44.6 degrees
 Acq. time 3.412 sec
 Width 4802.1 Hz
 16 repetitions
 OBSERVE H1, 300.132689 MHz
 DATA PROCESSING
 FT size 32768
 Total time 1 min, 22 sec

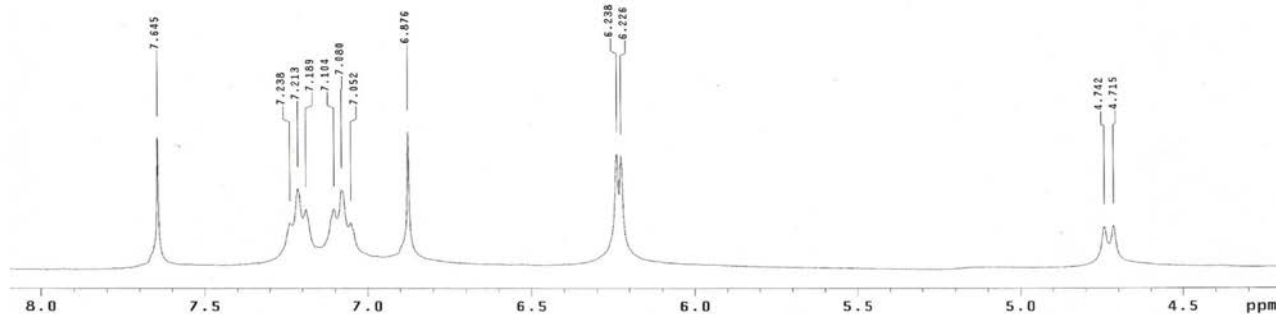
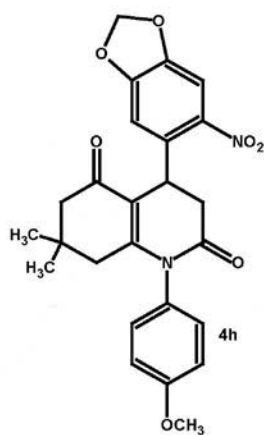


Figure S50. Expanded ^1H NMR (DMSO- D_6 , 300 MHz) spectrum of 4h.

RL8891B DMSD06 Lourenco 22/08/2011
 Pulse Sequence: s2pu1
 Solvent: DMSO
 Ambient temperature
 GEMINI-300BB "gemin300"
 Relax. delay 1.895 sec
 Pulse 45.5 degrees
 Acq. time 3.585 sec
 Width 18112.3 Hz
 166 repetitions
 OBSERVE C13, 75.465400 MHz
 DECOUPLE H1, 300.132689 MHz
 Power 23 dB
 continuously on
 GALT2-28 modulated
 DATA PROCESSING
 Line broadening 3.0 Hz
 FT size 32768
 Total time 12006 hr, 38 min, 4 sec

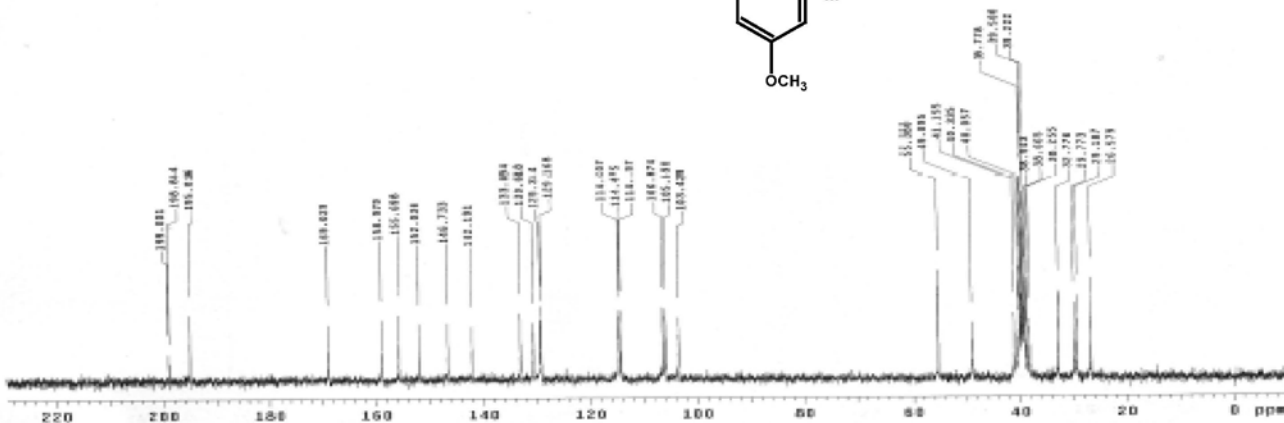
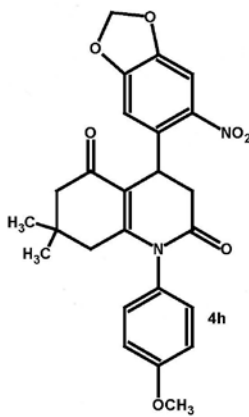
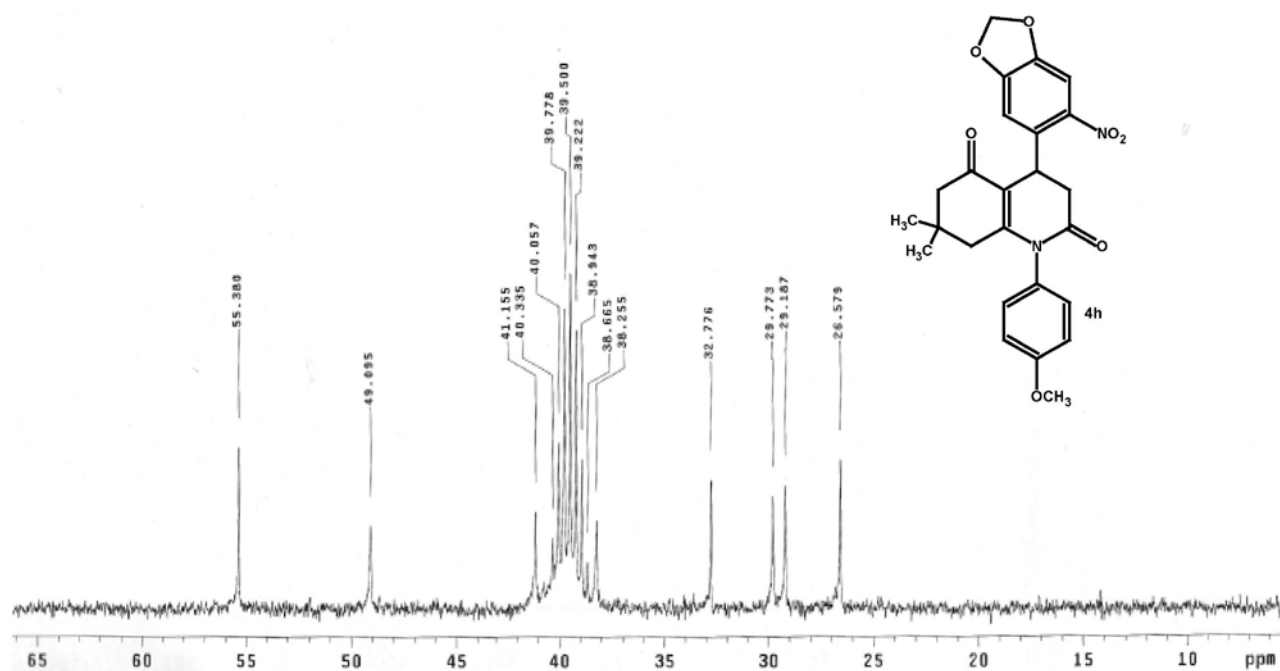
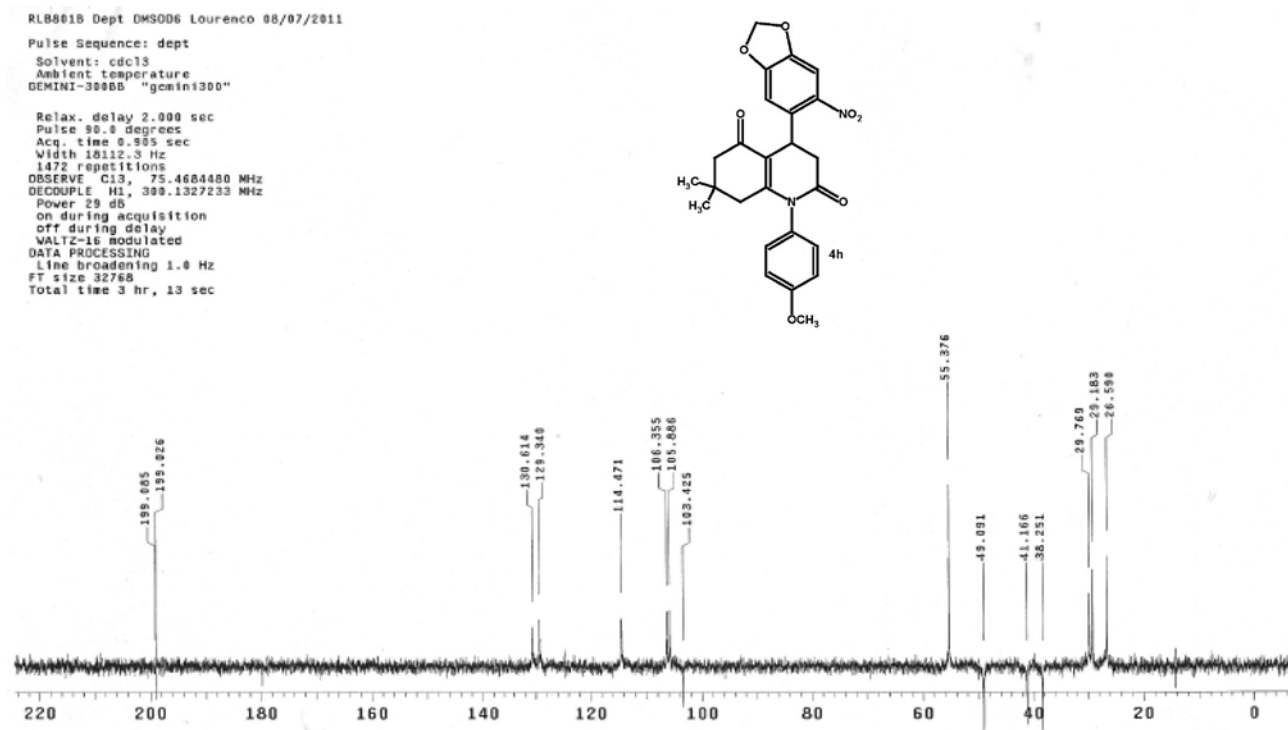


Figure S51. ^{13}C NMR (DMSO- D_6 , 75 MHz) spectrum of 4h.

Figure S52. Expanded ^{13}C NMR (DMSO- D_6 , 75 MHz) spectrum of 4h.Figure S53. DEPT ^{135}C NMR (DMSO- D_6 , 75 MHz) spectrum of 4h.

