

Supporting Information

Nano-zirconia as an excellent nano support for immobilization of sulfonic acid: A new, efficient and high recyclable heterogeneous solid acid nanocatalyst for multicomponent reactions

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Table S1. The experimental levels of variables in CCD for the synthesis of 2-amino-7,7-dimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile.

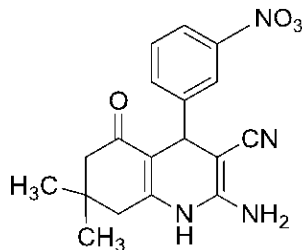
Independent variables	Levels				
	Lowest (-1.68)	Low (-1)	Central (0)	High (+1)	Highest (+1.68)
X ₁ : Catalyst amount (mg)	13.27	16	20	24	26.73
X ₂ : Temperature (°C)	83.18	90	100	110	116.81
X ₃ : Time (min)	6.59	10	15	20	23.40

Table S2. Conditions of predicted experiments in CCD for three effective factors in the synthesis of 2-amino-7,7-dimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carbonitrile accompanied to the observed responses.

Run	X ₁	X ₂	X ₃	Yield (%)
1	-1	+1	-1	54.85
2	+1	+1	-1	74.03
3	-1	+1	+1	72.82
4	0	-1	-1	68.66
5	0	+1.68	0	88.04
6	0	0	0	93.8
7	+1	+1	+1	89.55
8	0	0	0	94.42
9	+1.68	0	0	79.55
10	+1	-1	+1	82.86
11	0	0	0	93.56
12	-1	-1	-1	53.76
13	0	-1.68	0	80.72
14	-1	-1	+1	73.93
15	0	0	0	90.45
16	0	0	+1.68	85.31
17	0	0	0	91.76
18	0	0	-1.68	48.65
19	0	0	0	92.12
20	-1.68	0	0	56.36

Spectral data of some selected compound

3-(2-amino-3-cyano-7,7-dimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinolin-4-yl)phenyl nitrate (6d)



M. P.: 280-284 °C; IR (KBr, Cm^{-1}) ν_{max} : 3271, 3247, 3184, 2400, 1647, 1601, 1527, 1487, 1346, 1222, 1143.

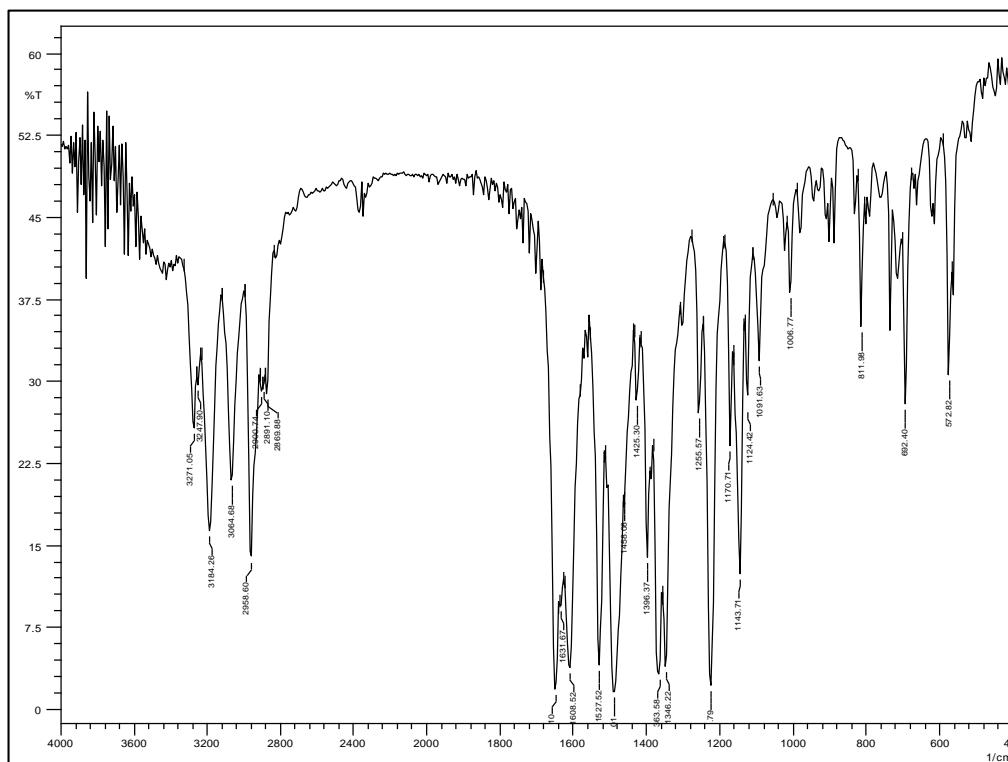
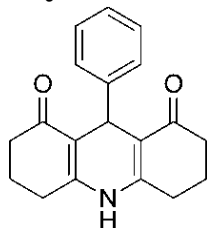


Fig. 1. FT-IR of (6d).

9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (7a)



M. P.: 280-282 °C; IR (KBr, Cm^{-1}) ν_{max} : 3273, 3196, 3080, 1624, 1601, 1483, 1218, 1140. ^1H NMR (400 MHz, DMSO): δ 1.90-2.09 (m, 4H), 2.26-2.28 (m, 2H), 2.30-2.38 (m, 2H), 2.51-2.64 (m, 4H), 4.79 (s, 1H), 7.05-7.09 (t, $J = 7.6$, 2H), 7.11-7.12 (d, $J = 8.4$ Hz, 1H), 7.23-7.31 (dd, $J = 8$, 24.2, 2H), 9.17 (s, 1H). Anal. Calc. for $\text{C}_{19}\text{H}_{19}\text{NO}_2$; C 77.79, H 6.53, N 4.77, O 10.91; Found: C 77.73, H 6.59, N 4.71, O 10.92.

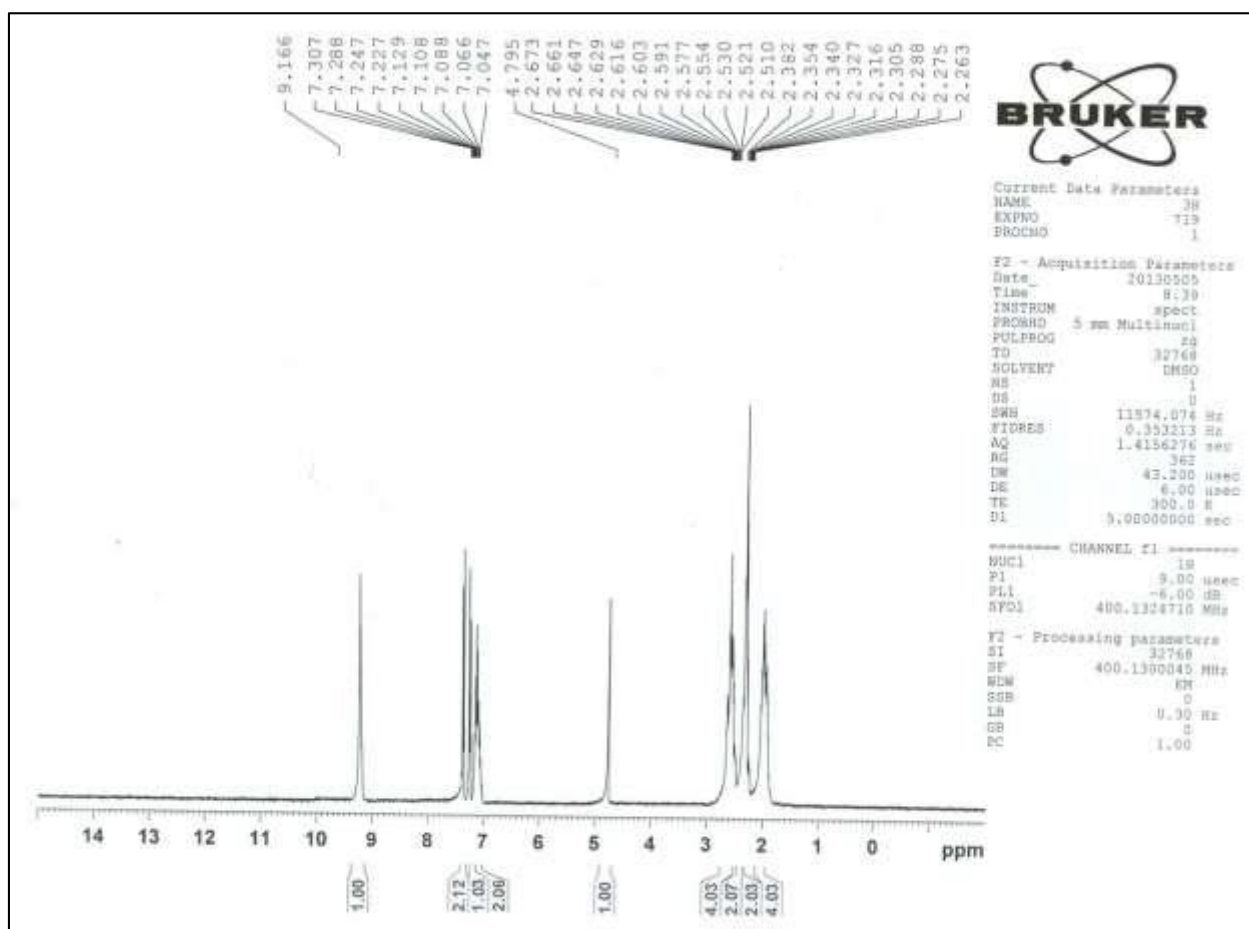
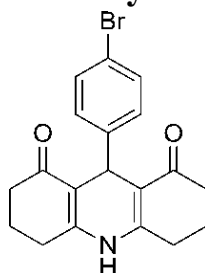


Fig. 2. ^1H NMR of (7a).

9-(4-bromophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (7c)



M. P.: 310-312 °C; IR (KBr, Cm^{-1}) ν_{max} : 3340, 3244, 2958, 2927, 1647, 1627, 1549, 1450, 1367, 1227, 1171. ^1H NMR (400 MHz, DMSO): δ 1.92-2.08 (m, 4H), 2.28-2.32 (m, 2H), 2.34-2.40 (m, 2H), 2.53-2.65 (m, 4H), 4.76 (s, 1H), 7.13-7.27 (m, 2H), 7.33-7.43 (dd, $J=30.6, 8.4$ Hz, 2H), 9.38 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d_6): δ 20.10, 20.27, 27.13, 31.39, 36.76, 36.90, 112.08, 117.66, 127.83, 129.43, 146.47, 151.59, 171.19, 194.58. Anal. Calc. for $\text{C}_{19}\text{H}_{18}\text{BrNO}_2$; C 61.30, H 4.87, N 3.76, O 8.60; Found: C 61.33, H 4.81, N 3.71, O 8.69.

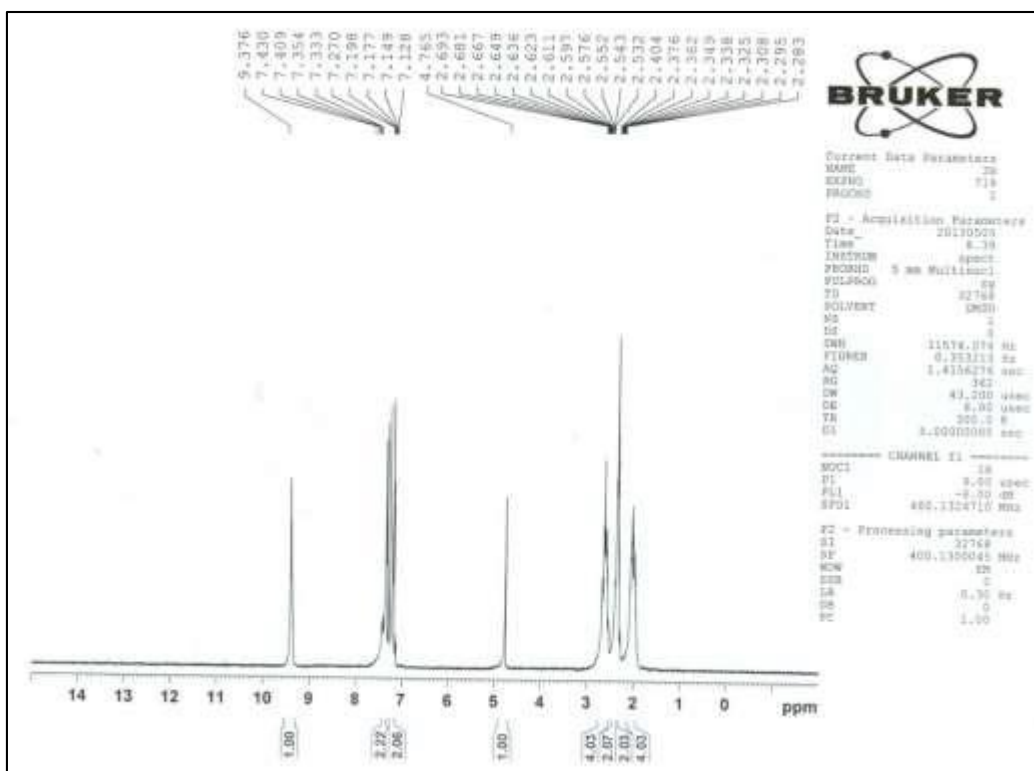


Fig. 3. ^1H NMR of (7c).

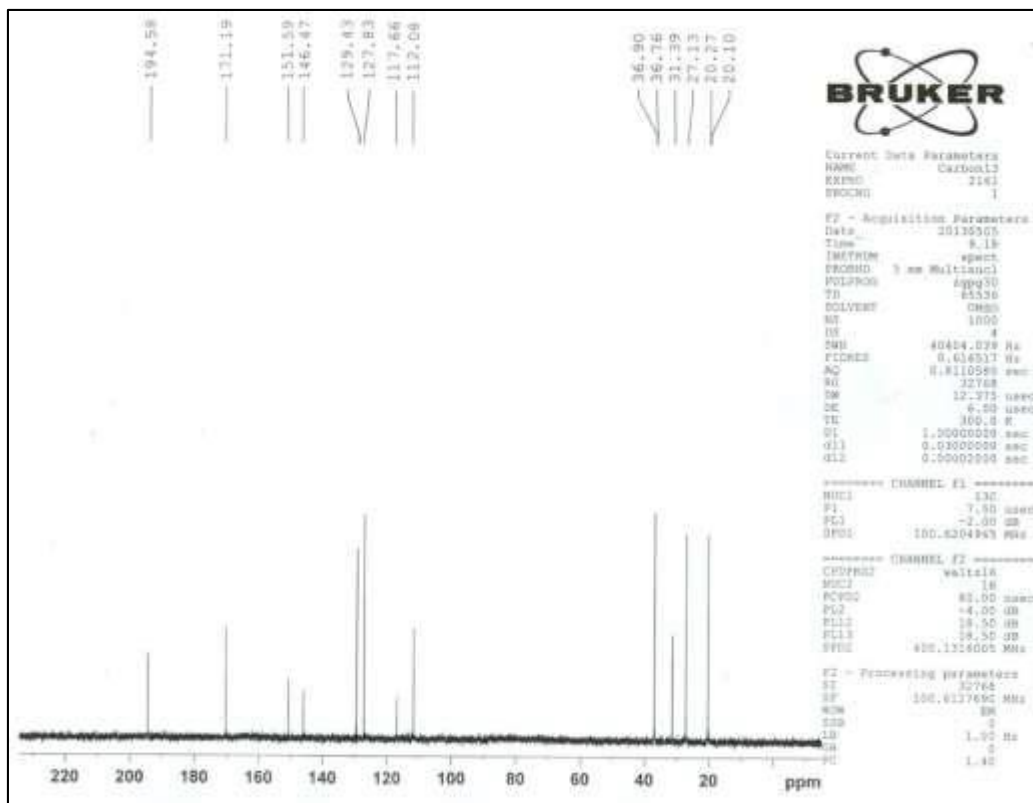
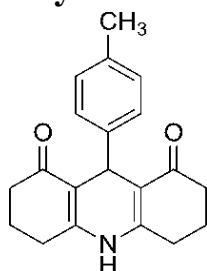


Fig. 4. ¹³C NMR of (7c).

9-(p-tolyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (7d)



M. P.: 254-256 °C; IR (KBr, Cm^{-1}) ν_{max} : 3286, 3203, 3068, 2943, 2887, 1639, 1608, 1458, 1364, 1232, 1176. ^1H NMR (400 MHz, DMSO): δ 1.96-2.07 (m, 4H), 2.26 (s, 3H), 2.29-2.42 (m, 4H), 2.52-2.69 (m, 4H), 4.79 (s, 1H), 7.03-7.05 (d, $J = 8$ Hz, 2H), 7.12-7.21 (d, $J = 7.6$ Hz, 2H), 9.18 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d^6): δ 20.31, 21.06, 27.15, 31.22, 36.98, 55.14, 113.52, 117.03, 128.25, 128.84, 129.33, 135.86, 146.36, 150.53, 169.57, 194.52. Anal. Calc. for $\text{C}_{20}\text{H}_{21}\text{NO}_2$; C 78.15, H 6.89, N 4.56, O 10.41; Found: C 78.19, H 6.83, N 4.58, O 10.45.

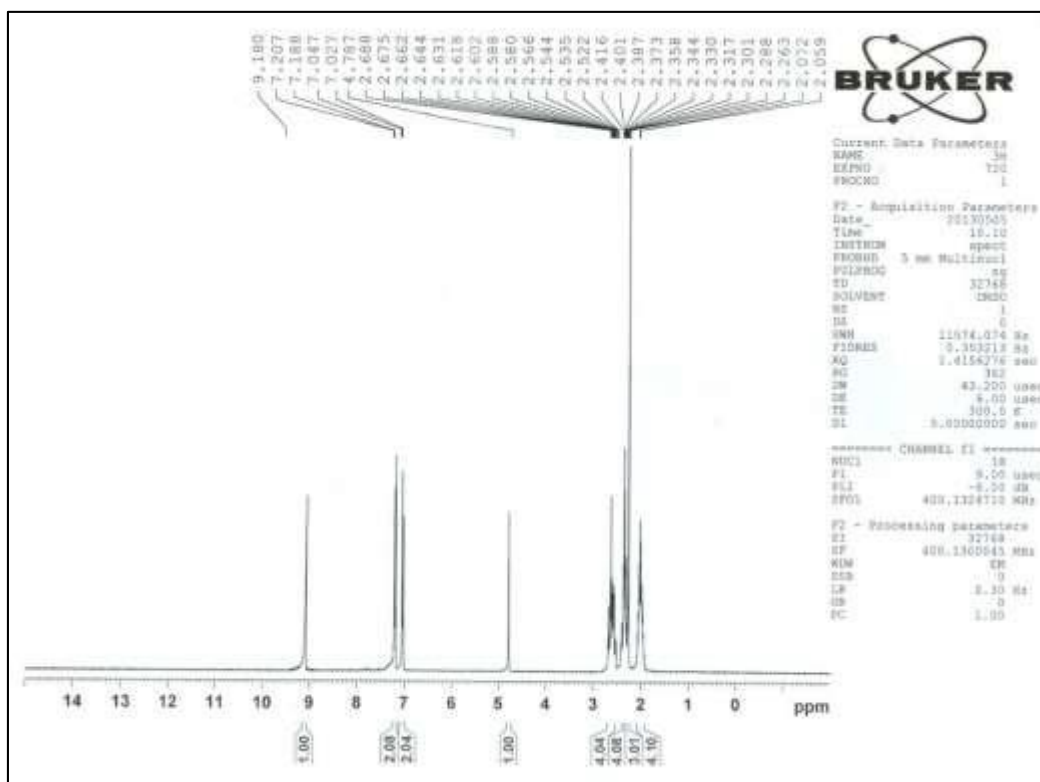


Fig. 5. ^1H NMR of (7d).

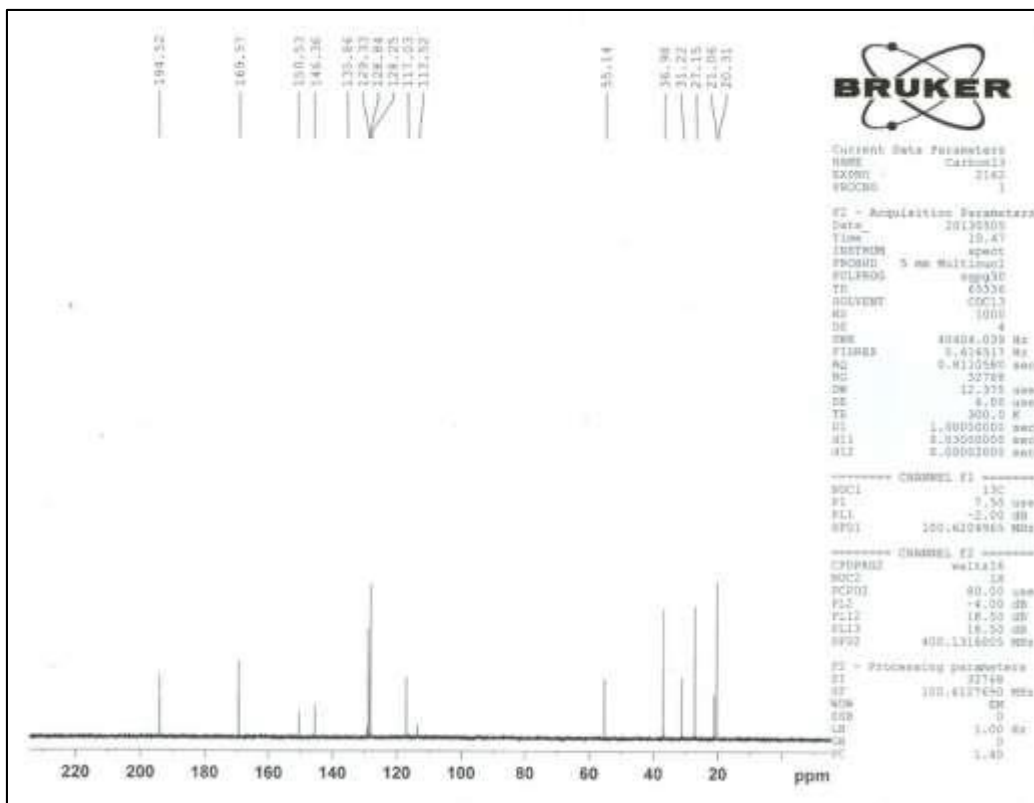
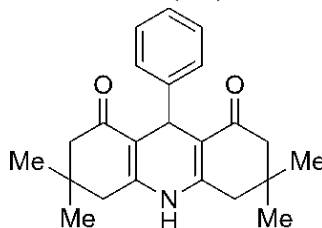


Fig. 6. ¹³C NMR of (7d).

3,3,6,6-tetra methyl-9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8-(2H,5H)-dione (8a)



M. P.: 290-291 °C; IR (KBr, Cm^{-1}) ν_{max} : 3279, 3209, 3063, 2955, 2932, 1636, 1605, 1481, 1365, 1219, 1142. ^1H NMR (400 MHz, DMSO): δ 0.84 (s, 6H), 0.99 (s, 6H), 1.96 (d, $J=21.5$ Hz, 2H), 2.15 (d, $J=21.5$ Hz, 2H), 2.30 (d, $J=22.8$ Hz, 2H), 2.40-2.49 (m, 2H), 4.8 (s, 1H), 7.00-7.14 (m, 5H), 9.28 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d_6): δ 26.45, 29.13, 32.14, 32.86, 50.24, 111.47, 125.46, 127.56, 127.63, 147.15, 149.32, 194.36. Anal. Calc. for $\text{C}_{23}\text{H}_{27}\text{NO}_2$; C 79.05, H 7.79, N 4.01, O 9.16; Found: C 79.08, H 7.73, N 4.06, O 9.11.

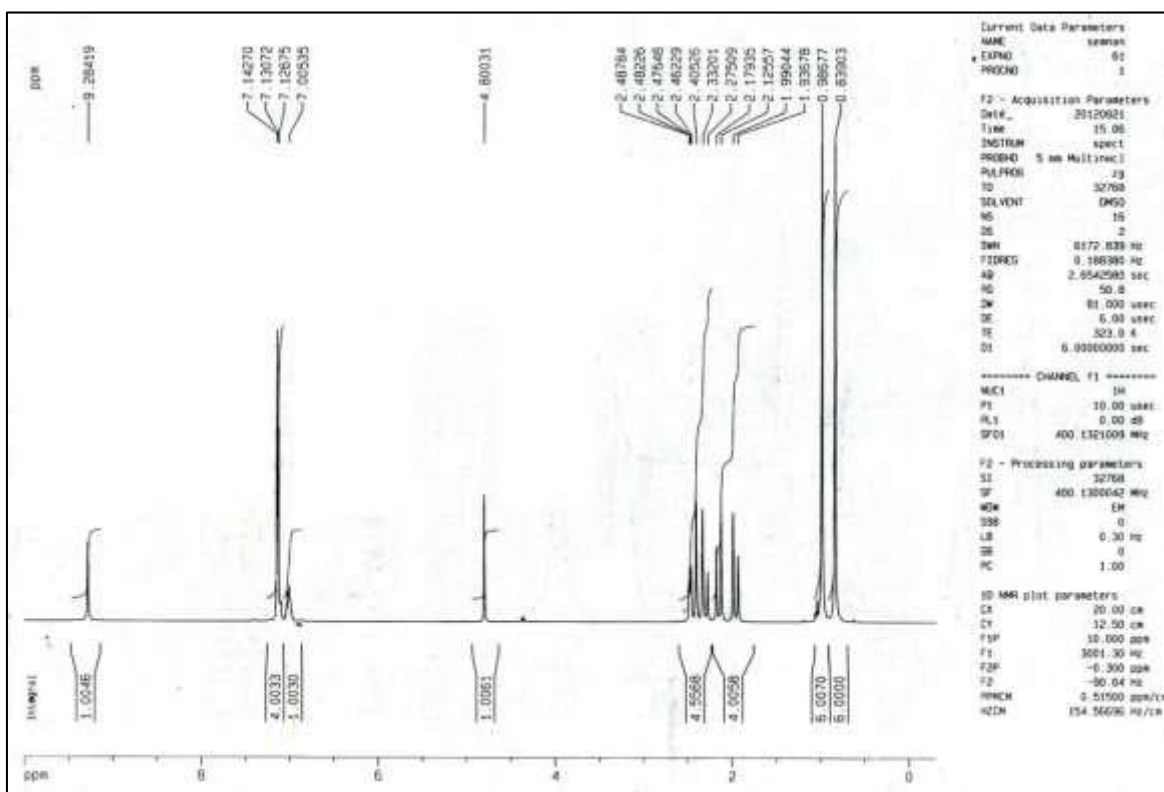


Fig. 7. ^1H NMR of (8a).

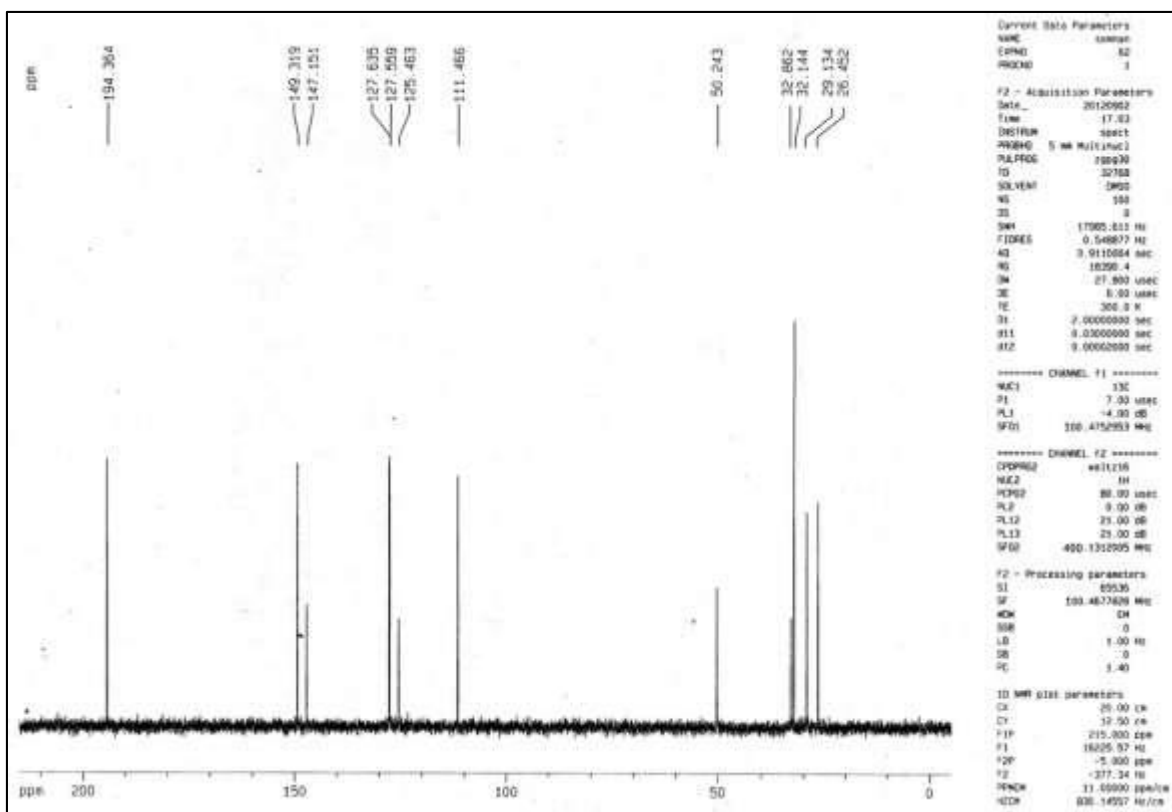
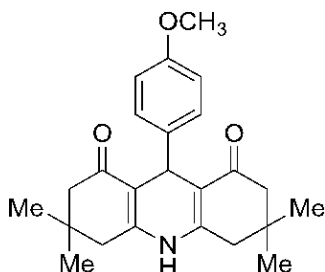


Fig. 8. ^{13}C NMR of (8a).

9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (8b)



M. P.: 274-276 °C; IR (KBr, cm^{-1}) ν_{max} : 3277, 3203, 3070, 2959, 2870, 1643, 1606, 1508, 1483, 1367, 1223, 1171. ^1H NMR (500 MHz, DMSO): δ 0.90 (s, 6H), 1.04 (s, 6H), 2.08 (d, $J=9.7$ Hz, 2H), 2.26 (d, $J=9.9$ Hz, 2H), 2.48-2.52 (m, 2H), 2.57 (d, $J=10.8$ Hz, 2H), 3.68 (s, 3H), 4.7 (s, 1H), 6.77-6.78 (t, $J=4.1$ Hz, 2H), 7.06-7.08 (t, $J=5.12$ Hz, 2H), 9.21 (s, 1H). Anal. Calc. for $\text{C}_{24}\text{H}_{29}\text{NO}_3$; C 75.96, H 7.70, N 3.69, O 12.65; Found: C 75.90, H 7.72, N 3.62, O 12.69.

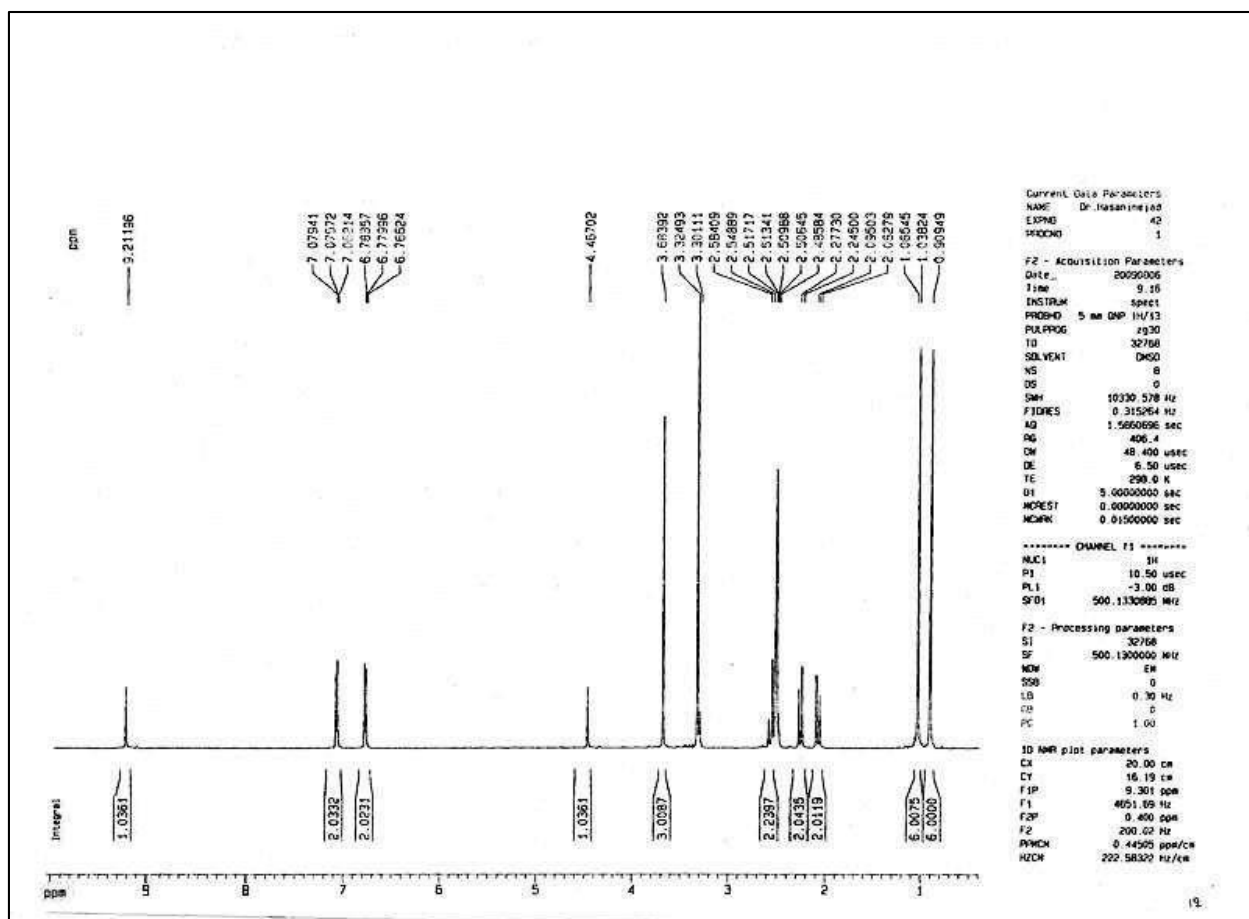
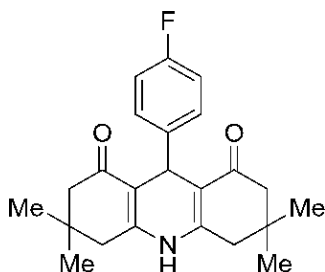


Fig. 9. ^1H NMR of (8b).

9-(4-fluorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (8e)



M. P.: 292-294 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3280, 3197, 3070, 2954, 2869, 1635, 1608, 1570, 1437, 1364, 1256, 1145. ^1H NMR (400 MHz, DMSO): δ : 0.99 (s, 6H), 1.11 (s, 6H), 2.15-2.26 (dd, J = 28, 16 Hz, 4H), 2.46 (s, 4H), 4.73 (s, 1H), 6.88-6.92 (t, J = 8.8 Hz, 2H), 7.24-7.27 (d, J = 10.8 Hz, 2H), 9.33 (s, 1H). ^{13}C NMR (100 MHz, DMSO- d_6): δ 27.29, 29.26, 31.22, 32.20, 50.73, 111.94, 126.07, 127.80, 127.87, 147.17, 149.30, 149.60, 194.46. Anal. Calc. for $\text{C}_{23}\text{H}_{26}\text{FNO}_2$; C 75.18, H 7.13, N 3.81, O 8.71; Found: C 75.13, H 7.18, N 3.78, O 8.76.

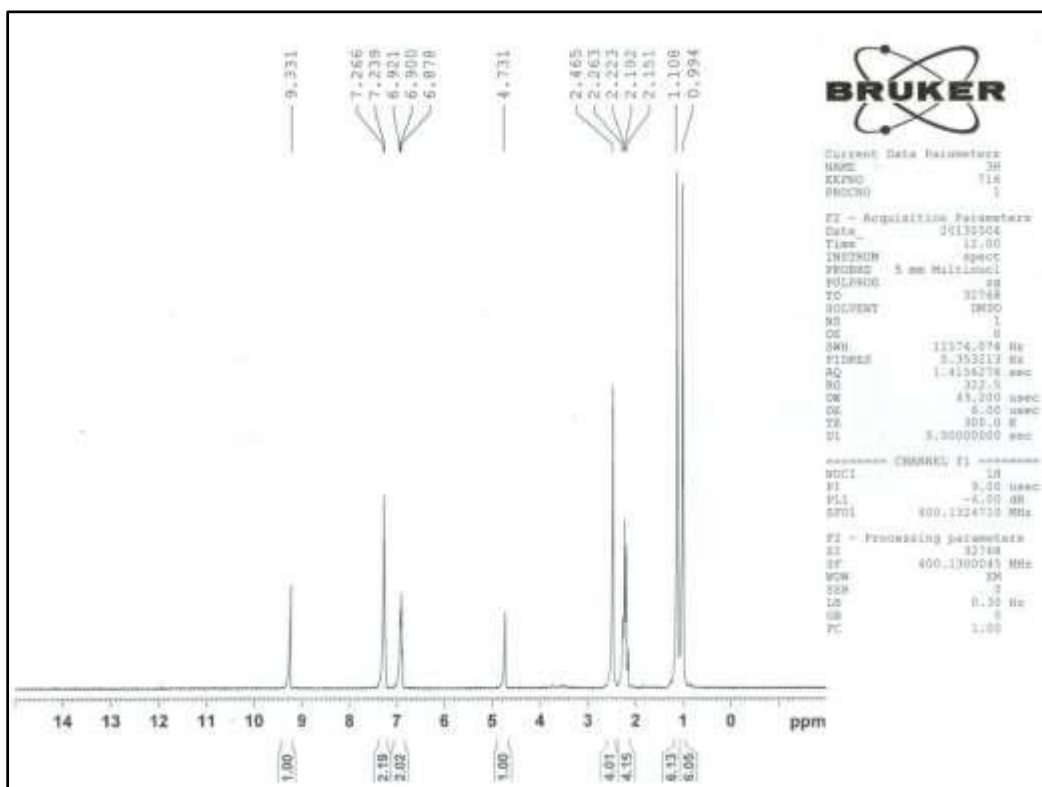


Fig. 10. ^1H NMR of (8e).

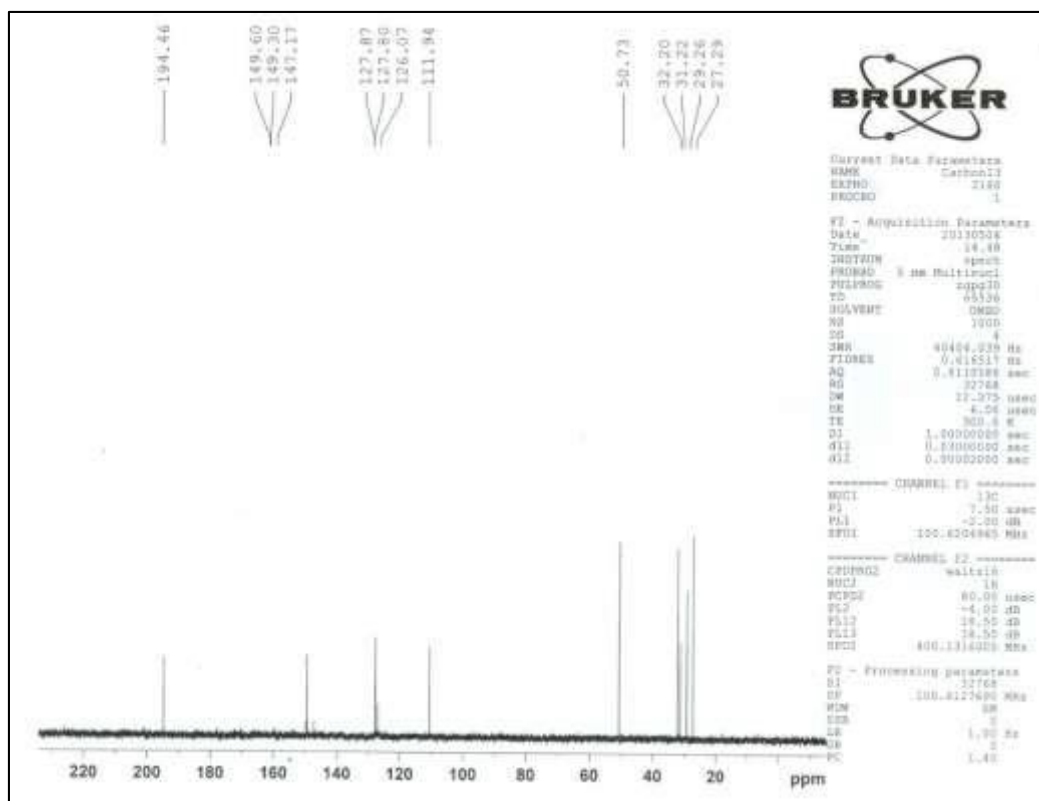
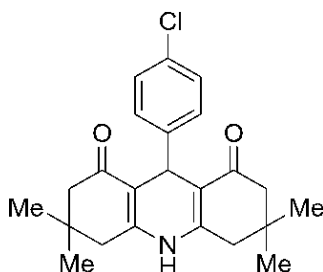


Fig. 11. ^{13}C NMR of (8e).

9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (8f)



M. P.: 299-302 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3288, 3205, 3068, 2956, 2869, 1643, 1608, 1475, 1363, 1223, 1171. $^1\text{H NMR}$ (500 MHz, DMSO): δ : 0.91 (s, 6H), 1.04 (s, 6H), 2.08-2.11 (d, $J=16$, 2H), 2.26-2.29 (d, $J=16$, 2H), 2.50-2.60 (dd, $J=14.65$, 4H), 4.51 (s, 1H), 7.18-7.20 (d, $J=8.2$ Hz, 2H), 7.28-7.29 (d, $J=8.2$ Hz, 2H), 9.32 (s, 1H). Anal. Calc. for $\text{C}_{23}\text{H}_{26}\text{ClNO}_2$; C 71.96, H 6.83, N 3.65, O 8.33; Found: C 71.90, H 6.89, N 3.59, O 8.32.

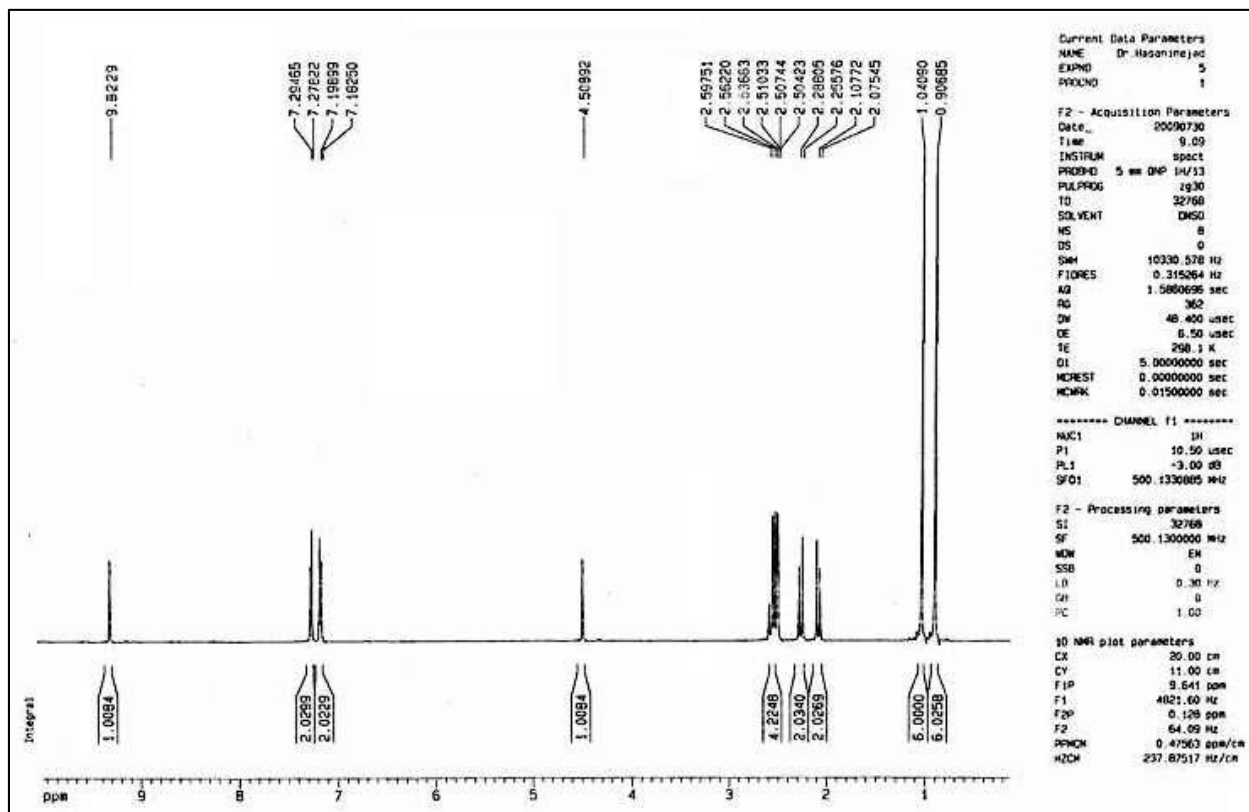
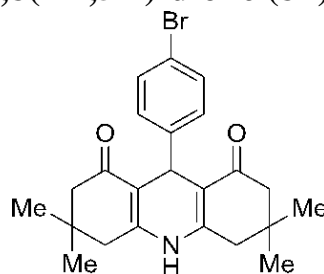


Fig. 12. $^1\text{H NMR}$ of (8f).

9-(4-bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (8h)



M. P.: 308-311 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3274, 3176, 3058, 2954, 2921, 1649, 1608, 1491, 1364, 1221, 1171. ^1H NMR (400 MHz, DMSO): δ : 1.03 (s, 6H), 1.23 (s, 6H), 2.24-2.30 (dd, $J=14.8, 8.8$ Hz, 4H), 2.44 (s, 4H), 4.84 (s, 1H), 7.16 (d, $J=6.8$ Hz, 2H), 7.43 (d, $J=17.6$ Hz, 2H), 9.30 (s, 1H). Anal. Calc. for $\text{C}_{23}\text{H}_{26}\text{BrNO}_2$; C 64.49, H 6.12, N 3.27, O 7.47; Found: C 64.52, H 6.17, N 3.31, O 7.49.

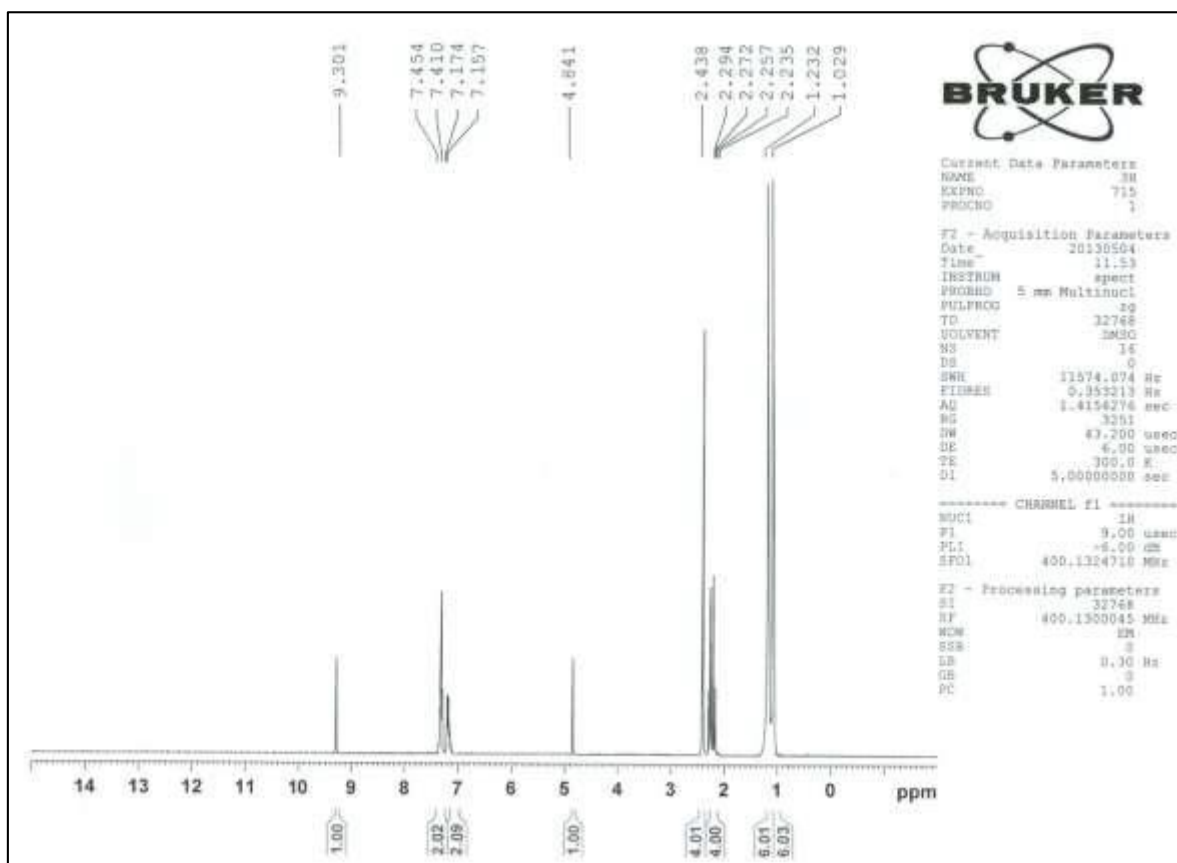
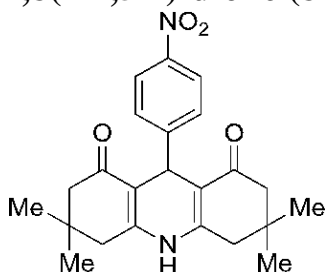


Fig. 13. ^1H NMR of (8h).

3,3,6,6-tetramethyl-9-(4-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (8i)



M. P.: 312-314 °C; FT-IR (KBr, cm^{-1}) ν_{max} : 3384, 2958, 2908, 1643, 1602, 1514, 1447, 1364, 1221, 1171. ^1H NMR (500 MHz, DMSO) δ : 0.91 (s, 6H), 1.05 (s, 6H), 2.08-2.11 (d, $J=6.5$ Hz, 2H), 2.27-2.30 (d, $J=6.5$ Hz, 2H), 2.50-2.63 (m, 4H), 4.63 (s, 1H), 7.45-7.48 (m, 2H), 8.10-8.12 (m, 2H), 9.32 (s, 1H). Anal. Calc. for $\text{C}_{23}\text{H}_{26}\text{N}_2\text{O}_4$; C 70.03, H 6.64, N 7.10, O 16.22; Found: C 70.08, H 6.69, N 7.07, O 16.26.

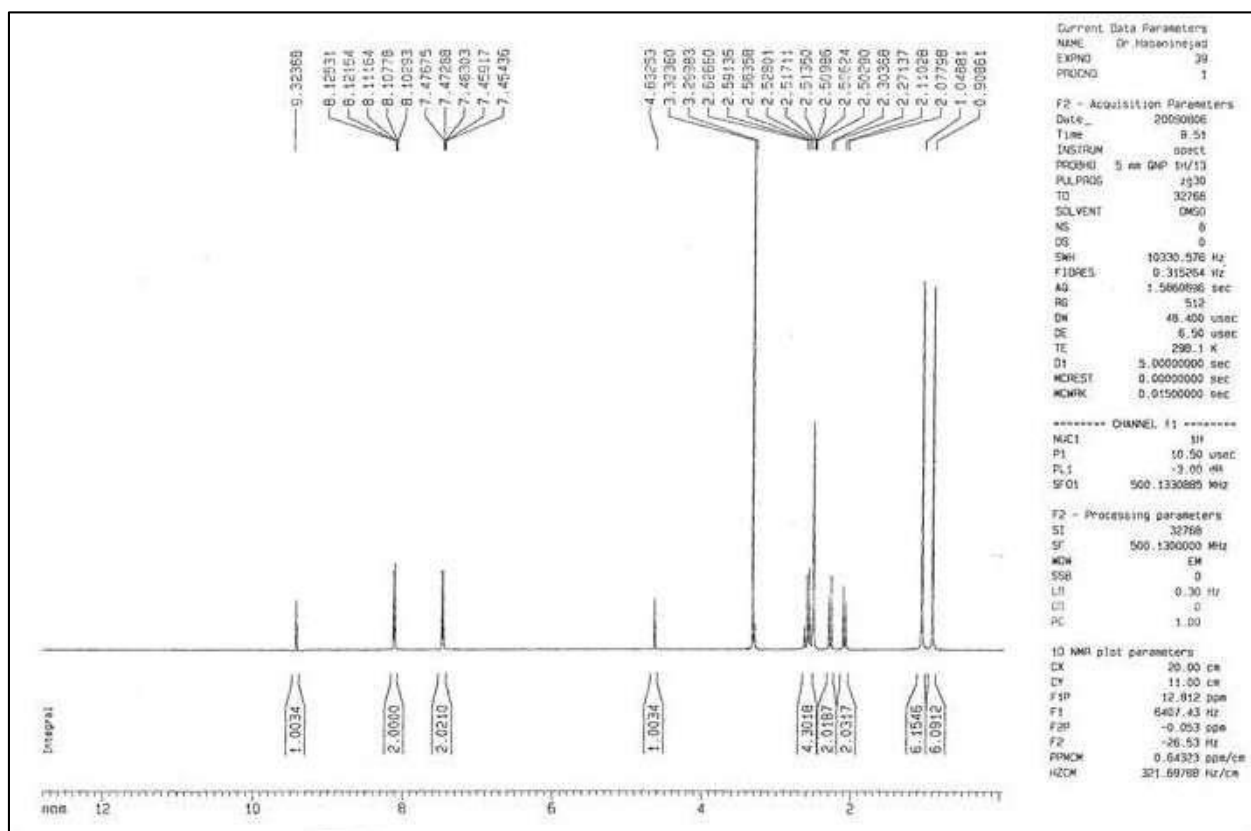
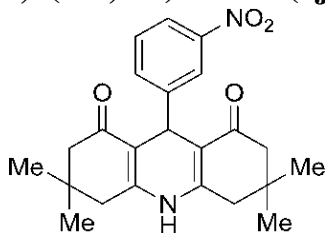


Fig. 14. ^1H NMR of (8i).

3,3,6,6-tetramethyl-9-(3-nitrophenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2H,5H)-dione (8j)



M. P.: 292-294 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3282, 3193, 3070, 2957, 2930, 2889, 2870, 1649, 1612, 1577, 1434, 1364, 1225, 1171. ^1H NMR (500 MHz, DMSO): δ : 0.91 (s, 6H), 1.05 (s, 6H), 2.09-2.12 (d, $J=4.4$ Hz, 2H), 2.27-2.30 (d, $J=6.4$ Hz, 2H), 2.55-2.63 (dd, $J=7.2$ Hz, 4H), 4.64 (s, 1H), 7.55-7.58 (t, $J=3.1$ Hz, 1H), 7.65-7.67 (d, $J=3$ Hz, 1H), 7.99-8.02 (d, $J=5.1$ Hz, 2H), 9.31 (s, 1H). Anal. Calc. for $\text{C}_{23}\text{H}_{26}\text{N}_2\text{O}_4$; C 70.03, H 6.64, N 7.10, O 16.22; Found: C 70.06, H 6.70, N 7.08, O 16.24.

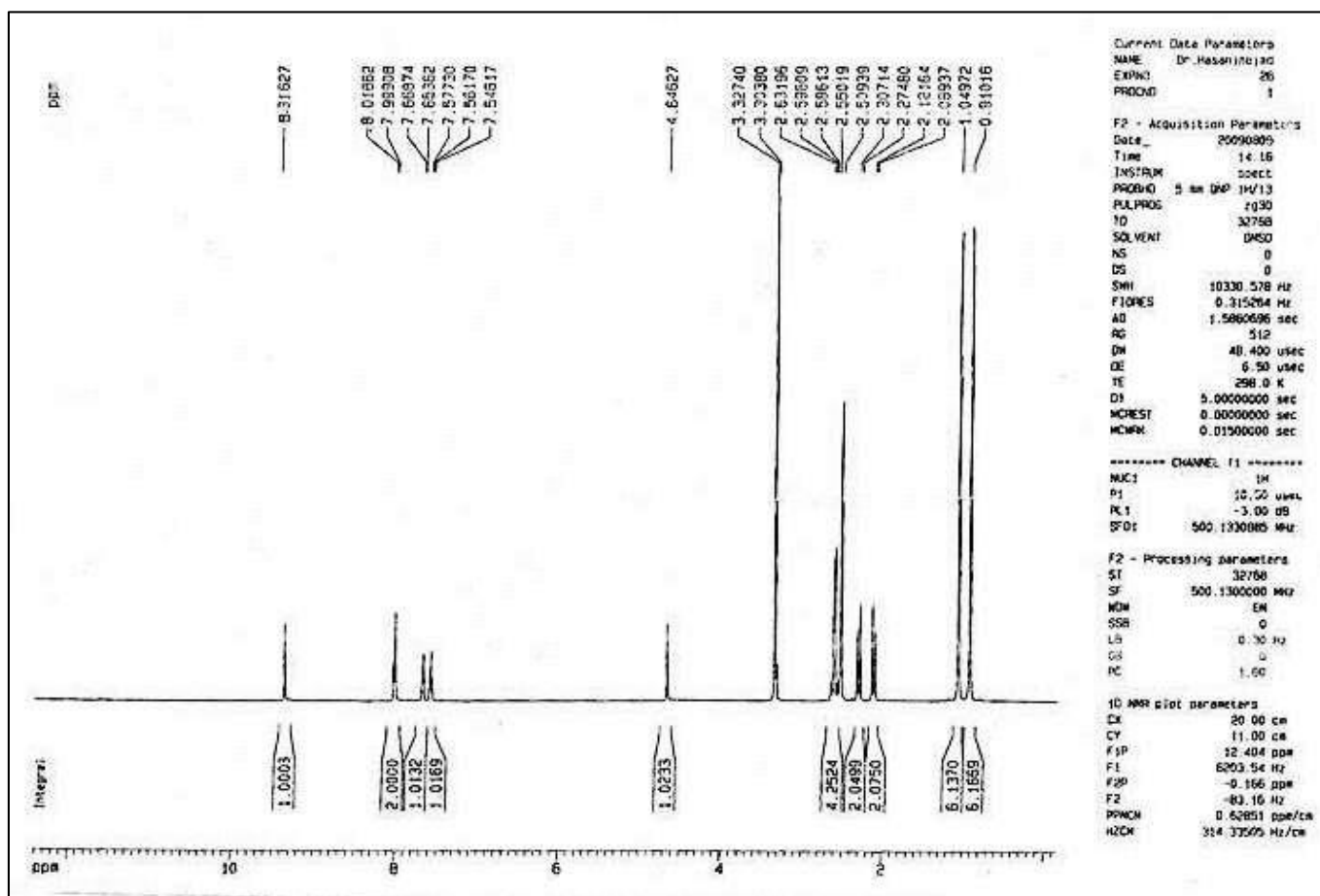
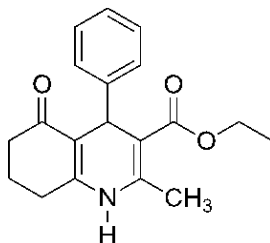


Fig. 15. ^1H NMR of (8j).

Ethyl 2-methyl-5-oxo-4-phenyl-4,6,7,8-tetrahydro-1H-quinoline-3-carboxylate (10a)



M.p. 239-241 °C (KBr, Cm^{-1}) ν_{max} : 3296 (NH), 1641 (C=O) (acid), 1608 (C=O) (ketone), 1488 (OC_2H_5) (ester) cm^{-1} ; δ/ppm (300 MHz, CDCl_3 -*d*) 1.17 (t, 3H, CH_3 - CH_2 -C=O), 1.59 (m, 2H, CH_2 - CH_2 - CH_2), 1.97 (m, 3H, CH_3 -Ph), 2.30-2.46 (m, 4H, CH_2 - CH_2), 4.05 (q, 2H, -O- CH_2 - CH_3), 5.01 (s, 1H, CH-Ph), 5.94 (s, 1H, NH), 7.08-7.31 (m, 5H, CH (Ph)).

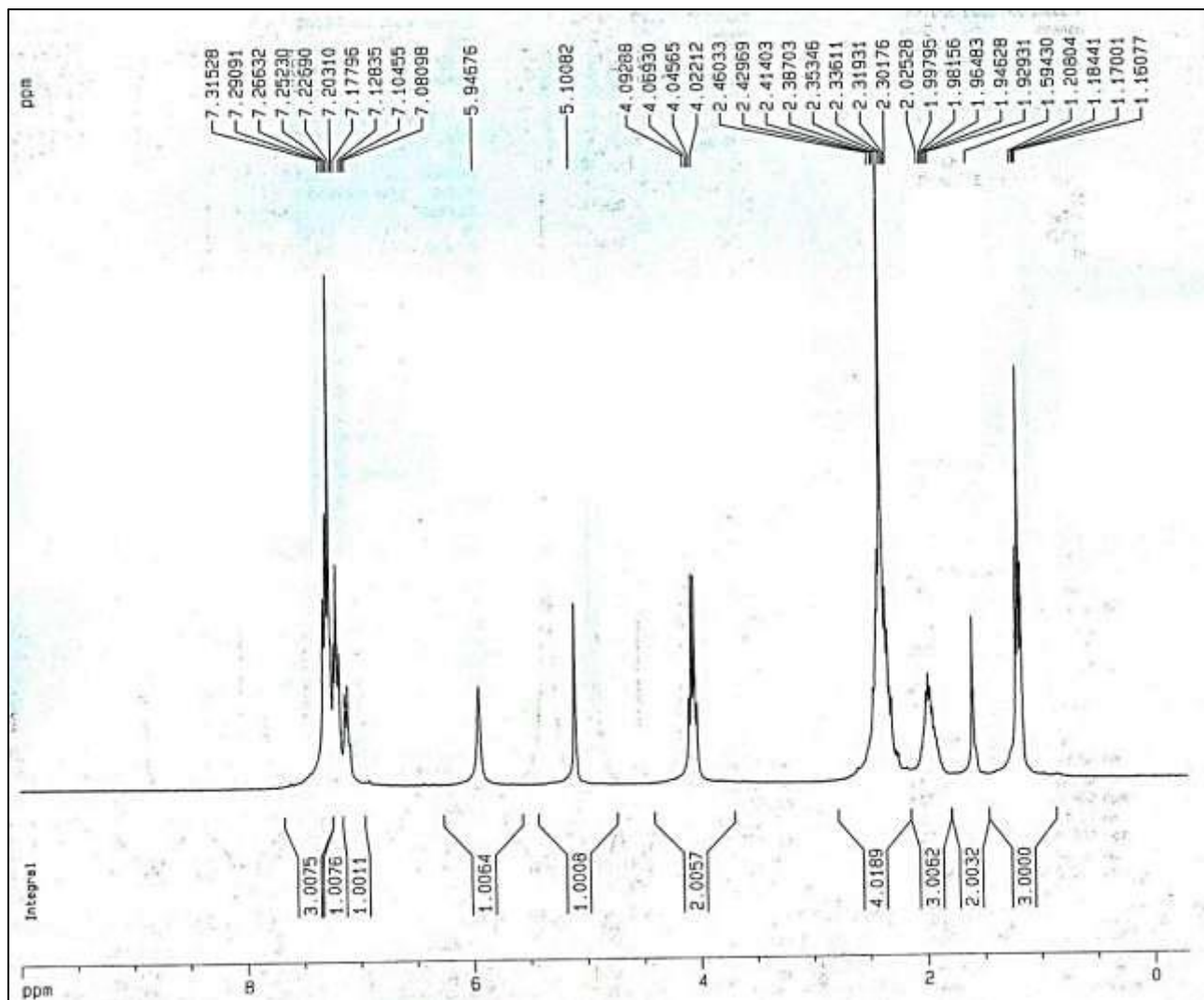
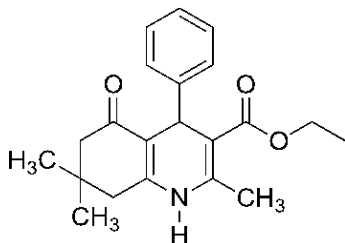


Fig. 16. ^1H NMR of (10a).

Ethyl 2,7,7-trimethyl-5-oxo-4-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (11a)



M.p. 210-211 °C, (KBr, Cm^{-1}) ν_{max} : 3275 (NH), 3080 (CH), 1700 (C=O) (acid), 1610 (C=O) (ketone), 1480 (OC_2H_5) (ester), 1225 (OCH_3) (ether) cm^{-1} ; δH / ppm (400 MHz, CDCl_3 -*d*) 0.94 (s, 3H, CH_3), 1.08 (s, 3H, CH_3), 1.19-1.23 (t, $J = 6.8$ Hz, 3H, CH_3 - CH_2 -C=O), 2.14-2.35 (m, 4H,), 4.05-4.10 (q, $J = 7.2$, 2H, -O- CH_2 - CH_3), 5.05 (s, 1H, CH-Ph), 6.42 (s, 1H, NH), 7.09-7.13 (t, $J = 7.2$, 1H, CH (Ph)), 7.19-7.23 (t, $J = 7.2$, 2H, CH (Ph)), 7.28-7.33 (m, 2H, CH (Ph)); δC /ppm (100 MHz, CDCl_3) 195.67, 167.5, 148.5, 147.06, 143.4, 128.02, 127.88, 126.03, 112.08, 106.05, 50.73, 40.9, 36.6, 32.7, 29.45, 27.14, 19.34, 14.21.

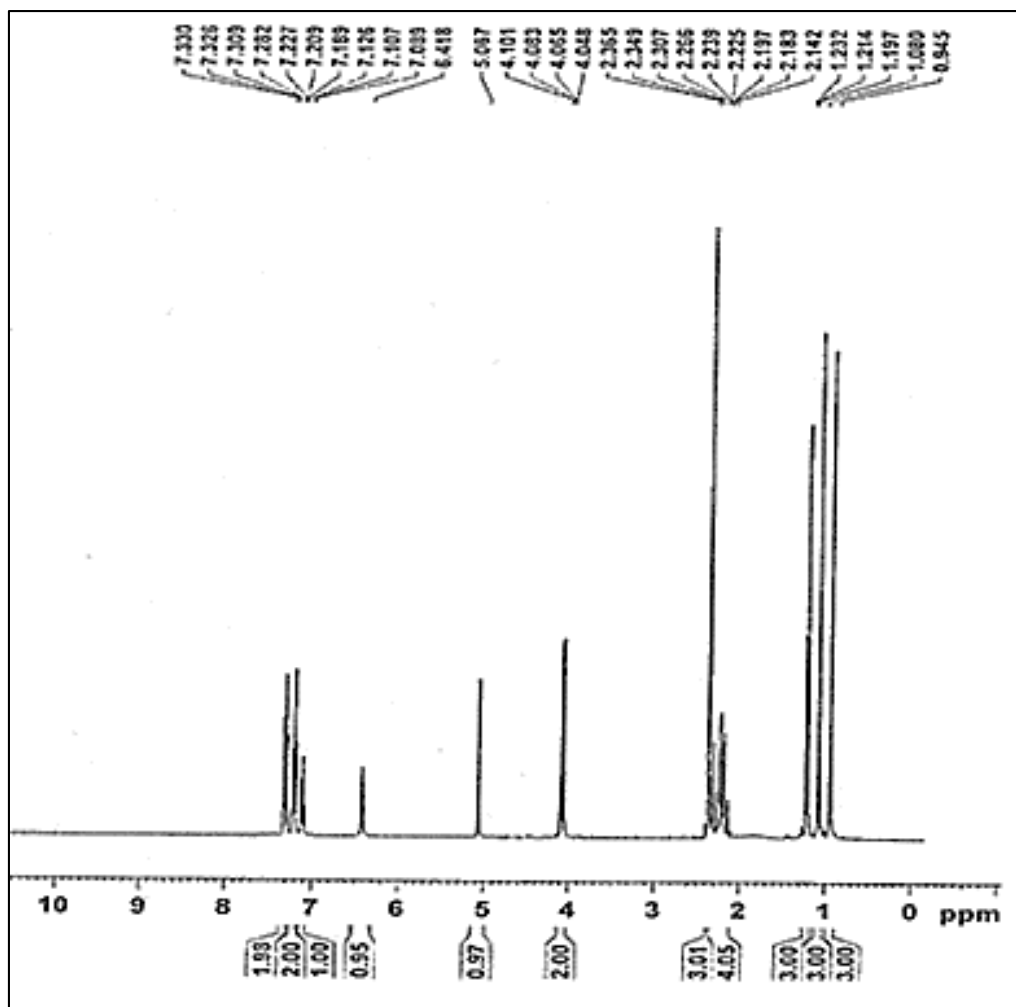


Fig. 17. ^1H NMR of (11a).

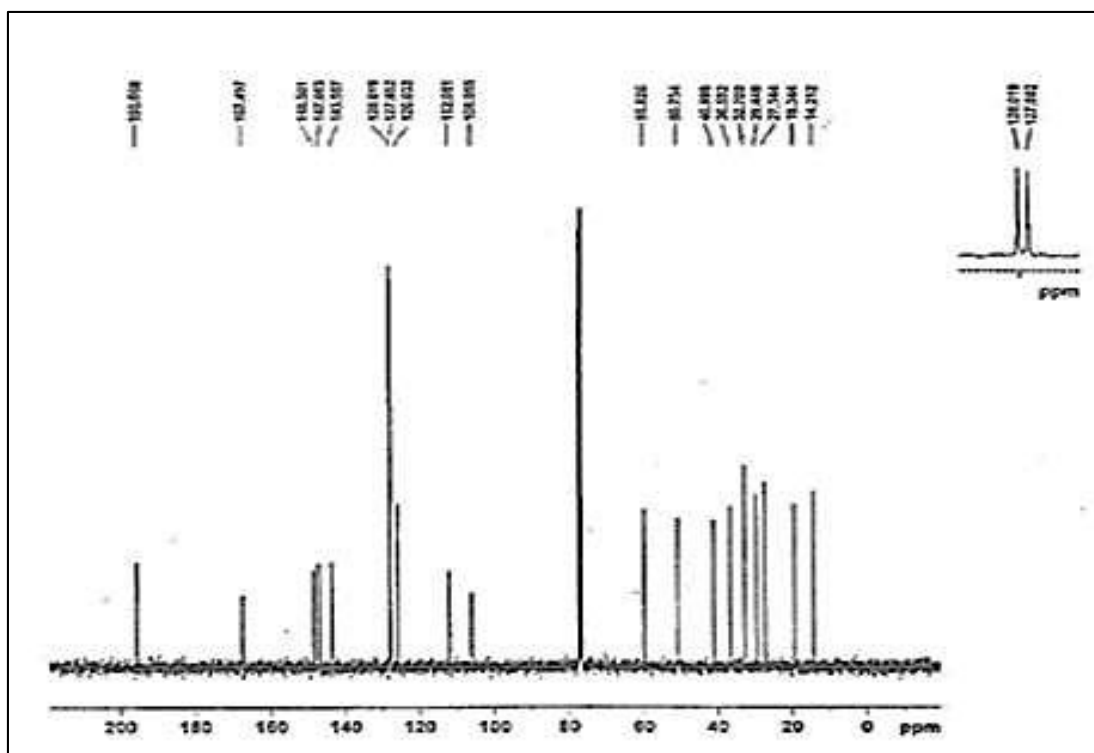
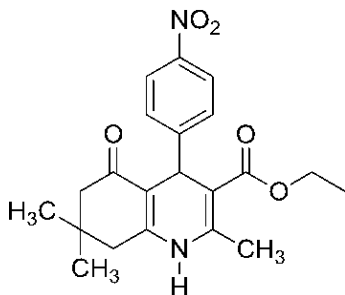


Fig. 18. ^{13}C NMR of (11a).

Ethyl 2,7,7-trimethyl-4-(4-nitrophenyl)-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (11d)



M.p. 207-209 °C, (KBr, Cm^{-1}) ν_{max} : 3278 (NH), 3076 (CH), 1690 (C=O) (acid), 1615 (C=O) (ketone), 1482 (OC_2H_5) (ester), 1218 (OCH_3) (ether) cm^{-1} ; δH /ppm (400 MHz, CDCl_3 -*d*) 0.77 (s, 3H, CH_3), 0.96 (s, 3H, CH_3), 1.04-1.09 (t, $J=9.6$ Hz, 3H, CH_3 - CH_2 -C=O), 1.90-2.22 (m, 2H, C- CH_2 -C=), 2.28 (s, 3H, CH_3), 2.38 (s, 1H), 2.43-2.46 (t, $J=2$, 1H), 3.30 (s, 1H), 3.88-3.95 (q, $J=9.6$, 2H, -O- CH_2 - CH_3), 4.93 (s, 1H, CH-Ph), 7.36-7.39 (d, $J=11.6$, 2H, CH (Ph)), 8.04-8.07 (d, $J=20.4$, 2H, CH (Ph)), 9.19 (s, 1H, NH); δC /ppm (100 MHz, CDCl_3) 194.32, 166.48, 161.96, 155.07, 150.17, 146.24, 145.73, 128.84, 123.24, 109.11, 102.46, 59.33, 50.14, 36.71, 32.22, 29.08, 26.52, 18.42, 14.16.

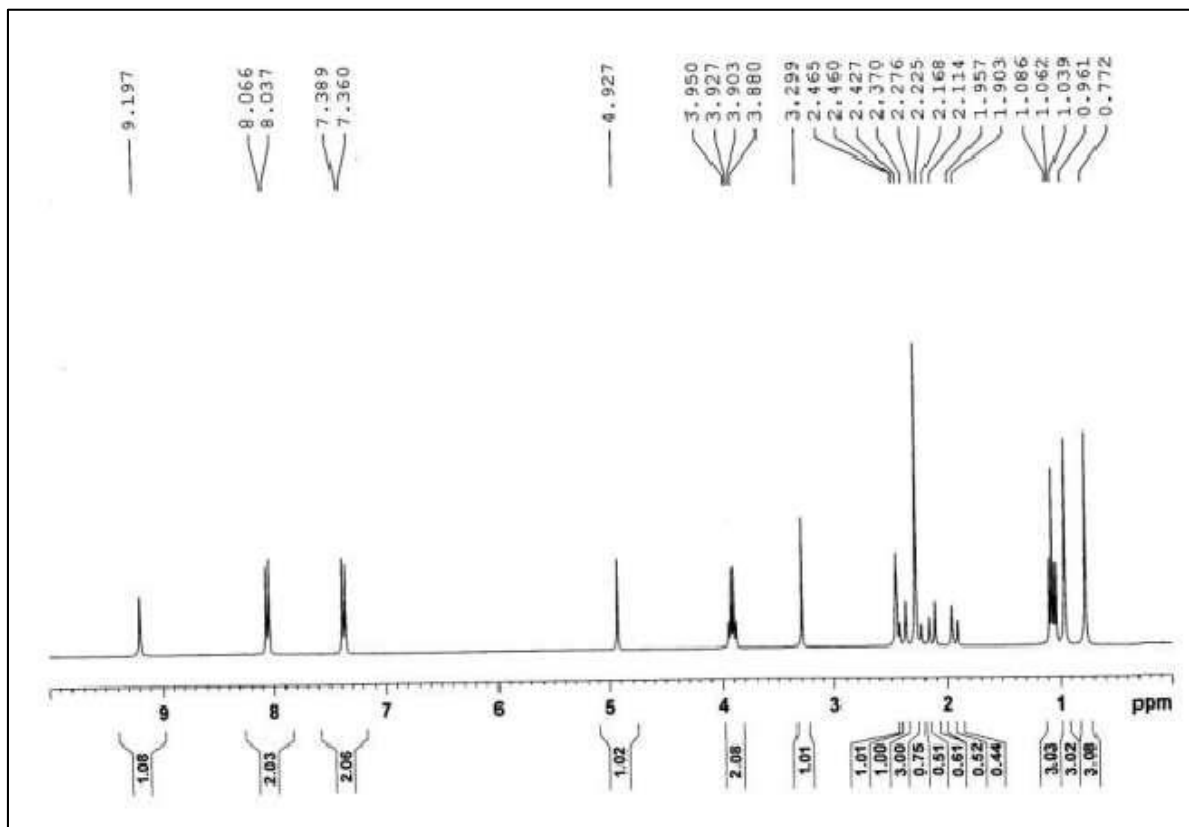


Fig. 19. ^1H NMR of (11d).

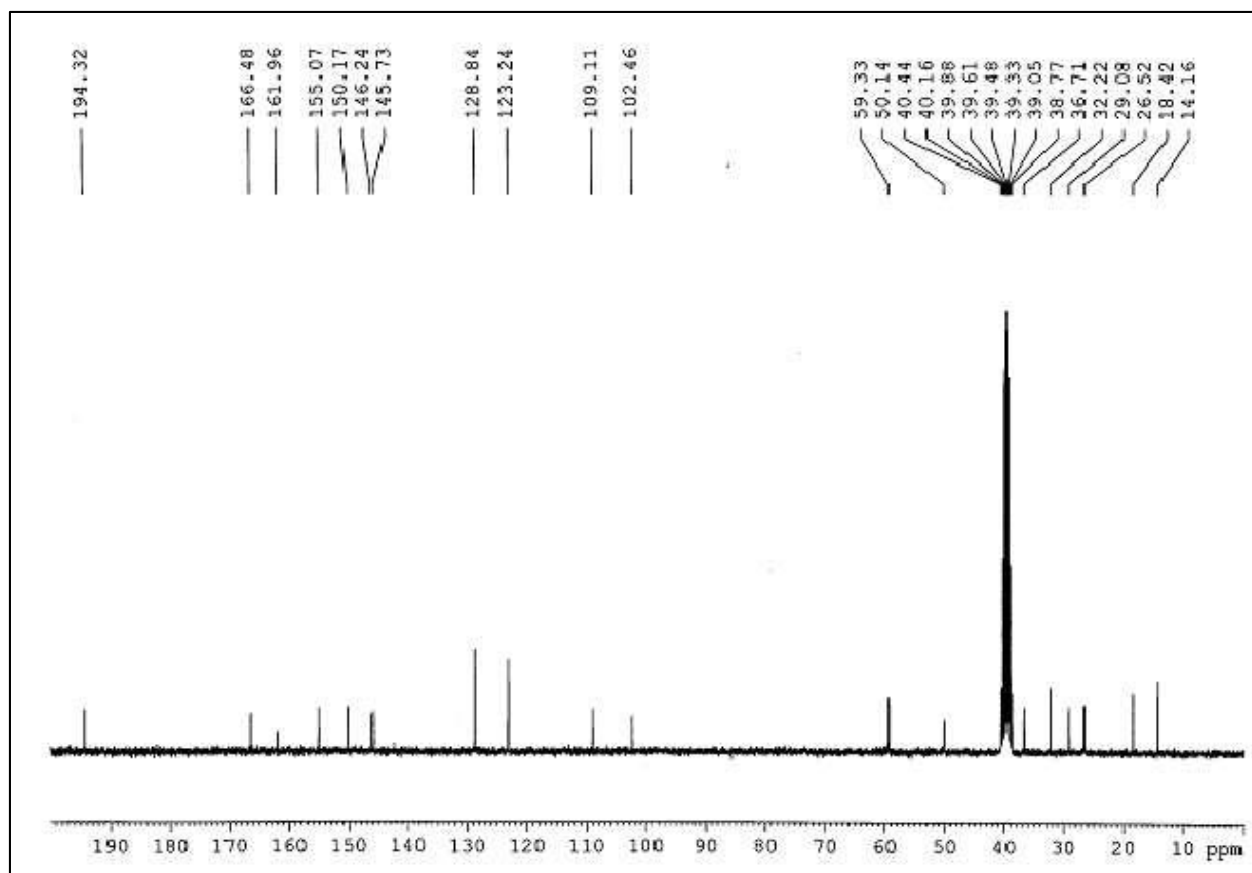
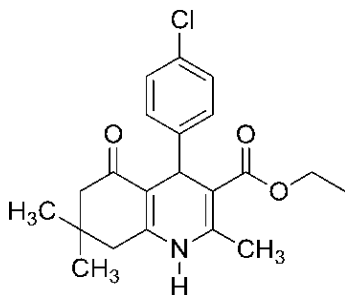


Fig. 20. ^{13}C NMR of (11d).

Ethyl 4-(4-chlorophenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (11f)



M.p. 243-245 °C, (KBr, Cm^{-1}) ν_{max} : 3275 and 3196 (NH), 3080 and 2963 (CH), 1711 (C=O) (acid), 1605 (C=O) (ketone), 1480 (OC_2H_5) (ester), 1221 (OCH_3) (ether) cm^{-1} ; $\delta\text{H}/\text{ppm}$ (400 MHz, CDCl_3 -*d*) 0.94 (s, 3H, CH_3), 1.09 (s, 3H, CH_3), 1.19-1.22 (t, $J=7.2$ Hz, 3H, $\text{CH}_3\text{-CH}_2\text{-C=O}$), 2.14-2.36 (m, 4H, $\text{C-CH}_2\text{-C=}$), 2.39 (s, 3H, N-C-CH_3), 4.04-4.09 (q, $J=7.2$ Hz, 2H, $-\text{O-CH}_2\text{-CH}_3$), 5.04 (s, 1H, CH-Ph), 6.12 (s, 1H, NH), 7.16-7.28 (m, 4H, CH (Ph)); $\delta\text{C}/\text{ppm}$ (100 MHz, CDCl_3) 195.49, 167.22, 148.12, 145.56, 143.62, 131.59, 129.45, 128.0, 111.90, 105.76, 59.92, 50.86, 41.08, 36.21, 32.72, 29.43, 27.11, 19.46, 14.21.

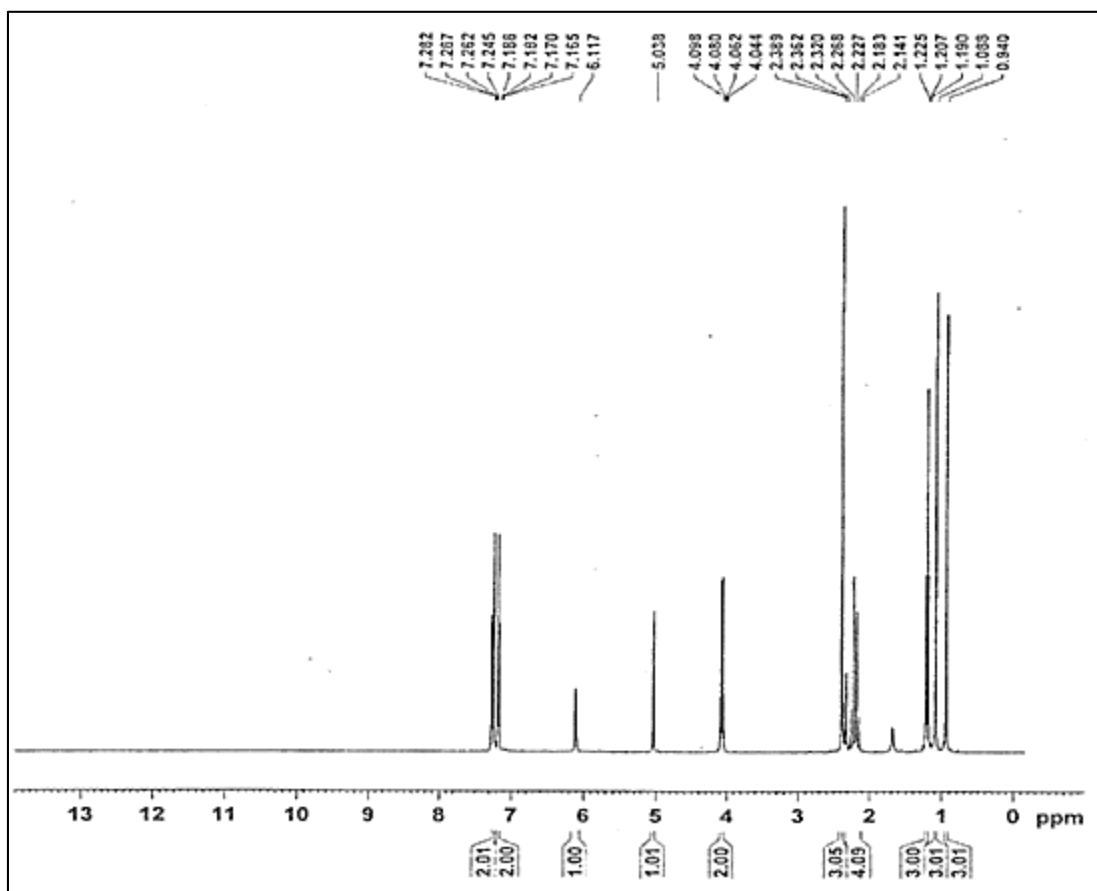


Fig. 21. ^1H NMR of (11f).

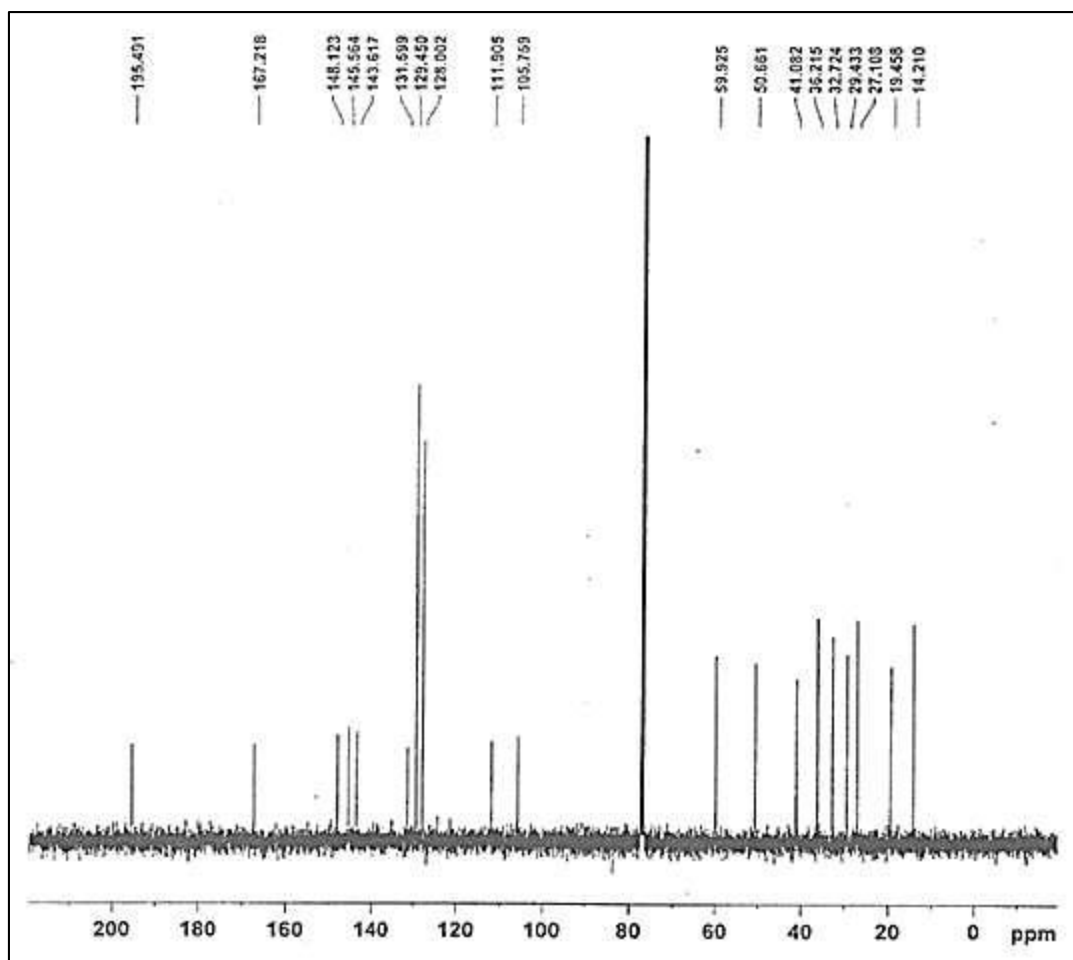
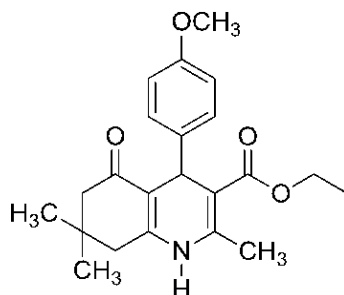


Fig. 22. ^{13}C NMR of (11f).

Ethyl 4-(4-methoxyphenyl)-2,7,7-trimethyl-5-oxo-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (11j)



M.p. 255-256 °C, (KBr, Cm^{-1}) ν_{max} : 3286 and 3112 (NH), 3080 and 2963 (CH), 1695 (C=O) (acid), 1615 (C=O) (ketone), 1498 (OC_2H_5) (ester), 1220 (OCH_3) (ether) cm^{-1} ; δH /ppm (400 MHz, CDCl_3 -*d*) 0.97 (s, 3H, CH_3), 1.09 (s, 3H, CH_3), 1.21-1.24 (t, $J = 7.2$ Hz, 3H, $\text{CH}_3\text{-CH}_2\text{-C=O}$), 2.15-2.37 (m, 4H, $\text{C-CH}_2\text{-C=}$), 2.39 (s, 3H, N-C-CH_3), 3.75 (s, 3H, $\text{H}_3\text{C-O-Ph}$), 4.05-4.10 (q, $J = 7.2$ Hz, 2H, $-\text{O-CH}_2\text{-CH}_3$), 5.01 (s, 1H, CH-Ph), 5.94 (s, 1H, NH), 6.73-6.77 (m, 2H, CH-Ar), 7.21-7.28 (m, 2H, CH-Ar); δC /ppm (100 MHz, CDCl_3) 195.52, 167.49, 157.75, 147.79, 142.99, 139.53, 128.98, 113.24, 112.45, 106.45, 59.83, 55.13, 50.66, 41.17, 35.67, 32.75, 29.43, 27.20, 19.49, 14.24.

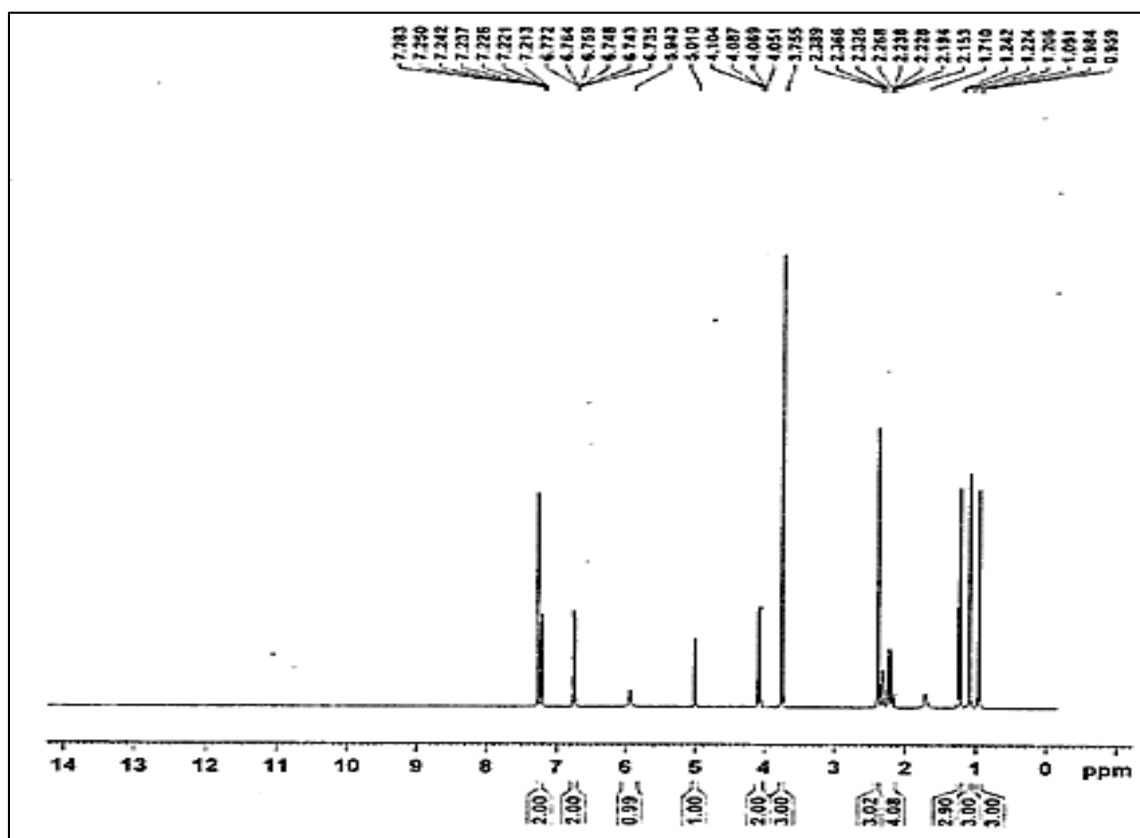


Fig. 23. ^1H NMR of (11j).

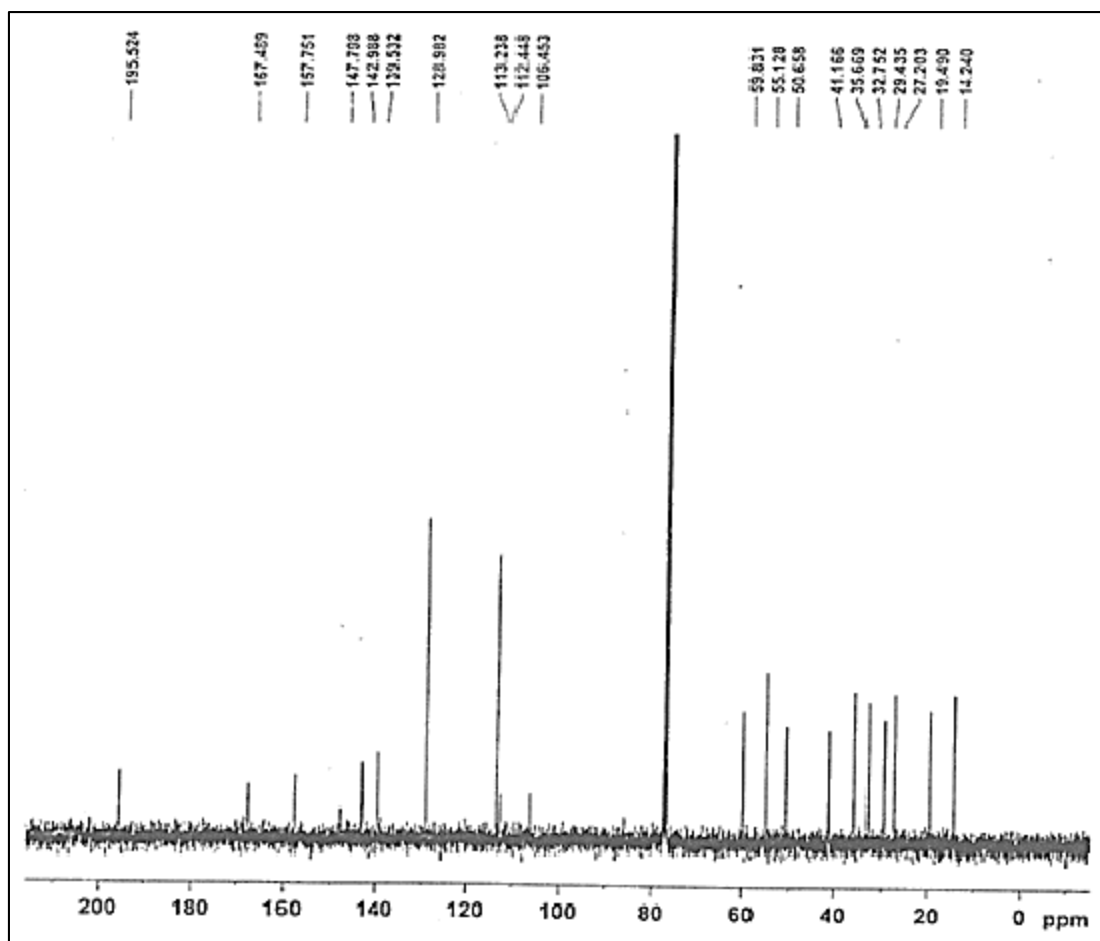
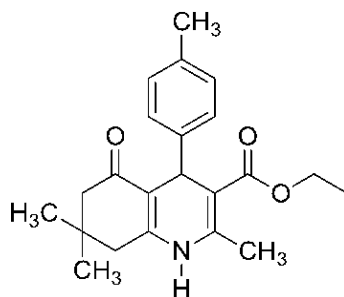


Fig. 24. ^{13}C NMR of (11j).

Ethyl 2,7,7-trimethyl-5-oxo-4-(p-tolyl)-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (11k)



M.p. 283-284 °C, (KBr, Cm^{-1}) ν_{max} : 3275 and 3207 (NH), 3085 and 2963 (CH), 1700 (C=O) (acid), 1610 (C=O) (ketone), 1494 (OC_2H_5) (ester), 1222 (OCH_3) (ether) cm^{-1} ; $\delta\text{H}/\text{ppm}$ (400 MHz, CDCl_3 -*d*) 0.96 (s, 3H, CH_3), 1.08 (s, 3H, CH_3), 1.21-1.25 (t, $J=7.2$ Hz, 3H, $\text{CH}_3\text{-CH}_2\text{-C=O}$), 2.14-2.24 (m, 3H, $\text{C-CH}_2\text{-C=}$), 2.27 (s, 3H, N-C-CH_3), 2.29-2.33 (t, $J=7.6$ Hz, 1H, $\text{C-CH}_2\text{-C=}$), 2.36 (s, 3H, $\text{H}_3\text{C-Ph}$), 4.05-4.10 (m, 2H, $-\text{O-CH}_2\text{-CH}_3$), 5.02 (s, 1H, CH-Ph), 6.18 (s, 1H, NH), 7.00-7.02 (d, $J=7.6$ Hz, 2H, CH-Ar), 7.19-7.21 (d, $J=8$ Hz, 2H, CH-Ar); $\delta\text{C}/\text{ppm}$ (100 MHz, CDCl_3) 195.64, 167.53, 148.15, 144.15, 143.27, 135.40, 128.61, 127.88, 112.28, 106.25, 59.83, 50.73, 41.1, 36.07, 32.72, 29.42, 27.23, 21.05, 19.4, 14.22.

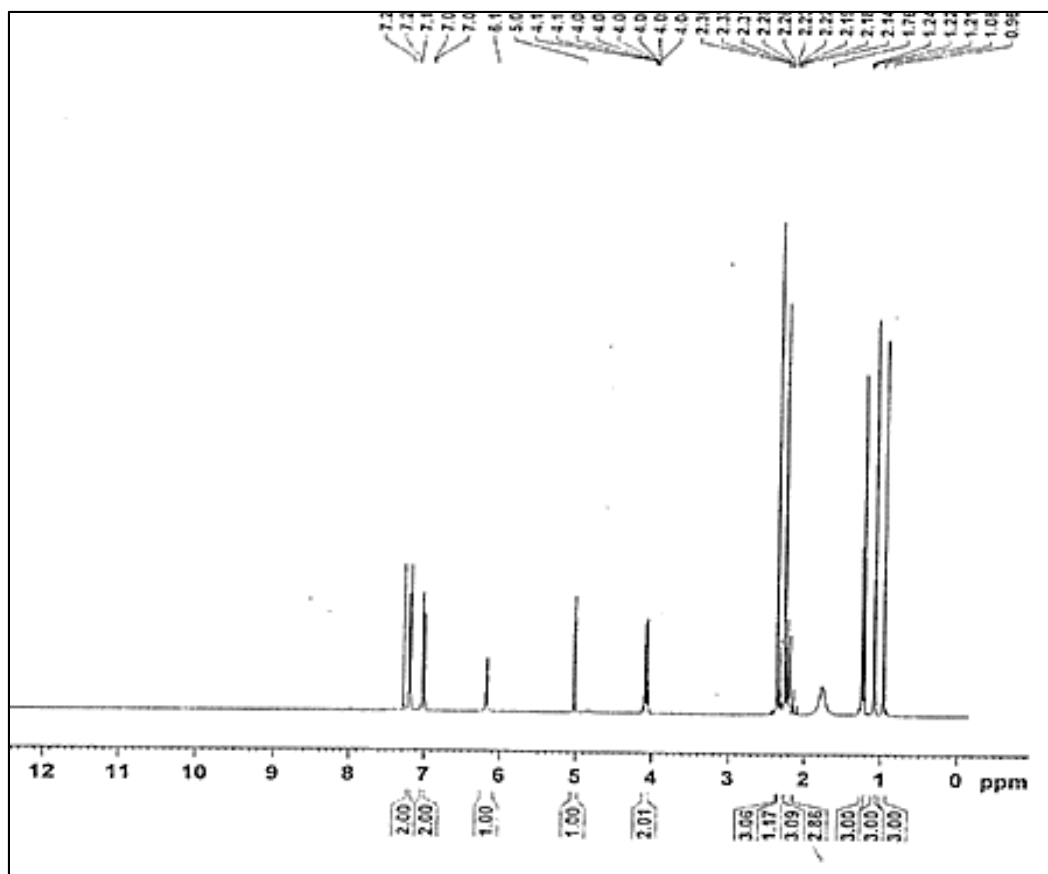


Fig. 25. ^1H NMR of (11k).

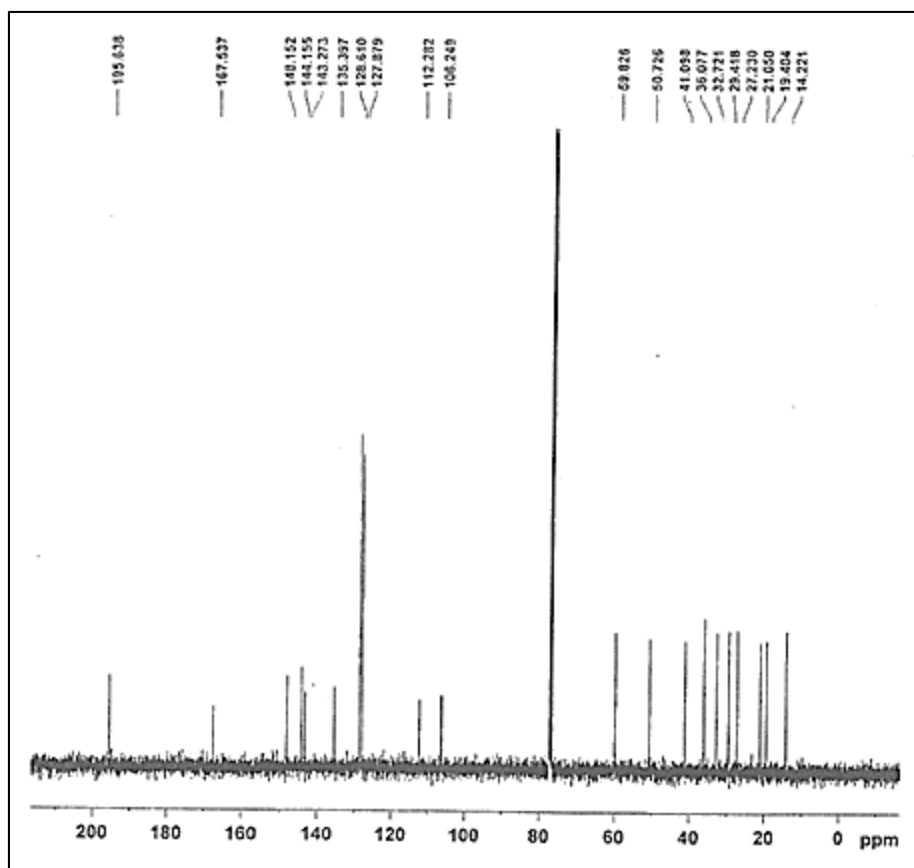
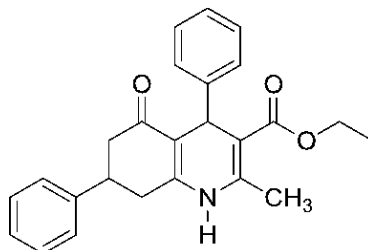


Fig. 26. ^{13}C NMR of (11k).

Ethyl 2-methyl-5-oxo-4,7-diphenyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (12a)



M.p. 213-215 °C, (KBr, Cm^{-1}) ν_{max} : 3276 (NH), 1701 (C=O) (acid), 1606 (C=O) (ketone), 1487 (OC_2H_5) (ester), cm^{-1} ; δ_{H} (400 MHz, $\text{DMSO-}d_6$) 1.14 (t, 3H, $\text{CH}_3\text{-CH}_2\text{-O-C=O}$), 2.32 (s, 3H, CH_3), 2.35 (dd, 1H, 8 H), 2.5 (d, 1H, 8 H), 2.59 (m, 1H, 7 H), 2.68 (dd, 1H, 6 H), 2.79 (dd, 1H, 6 H), 3.17 (s, 1H, NH), 4.0 (q, 2H, $\text{-O-CH}_2\text{-CH}_3$), 4.98 (s, 1H, 4 H), 7.11 (m, 1H, 4'H), 7.19-7.24 (q, 5H, 2', 4', 6', 2'', 6''H), 7.3-7.36 (m, 4H, 3', 5', 3'', 5'' H), 9.21 (s, 1H, NH) ppm; δ_{C} (100 MHz, $\text{DMSO-}d_6$) 194.4 (C-5), 167.3 (C= OOC_2H_5), 151.1 (C-2), 148.1 (C-1a), 145.3 (C-1''), 143.9 (C-1'), 128.9 (C-2' and C-6'), 128.3 (C-3' and C-5'), 127.9 (C-3'' and C-5''), 127.4 (C-2'' and C-6''), 127 (C-4''), 126.2 (C-4'), 111.2 (C-5a), 104.2 (C-3), 59.5 (O- $\text{CH}_2\text{-CH}_3$), 44.4 (C-6), 38.8 (C-4), 36.2 (C-8), 34 (C-7), 18.7 (1C, CH_3), 14.6 (1C, $\text{CH}_3\text{-CH}_2\text{O}$).

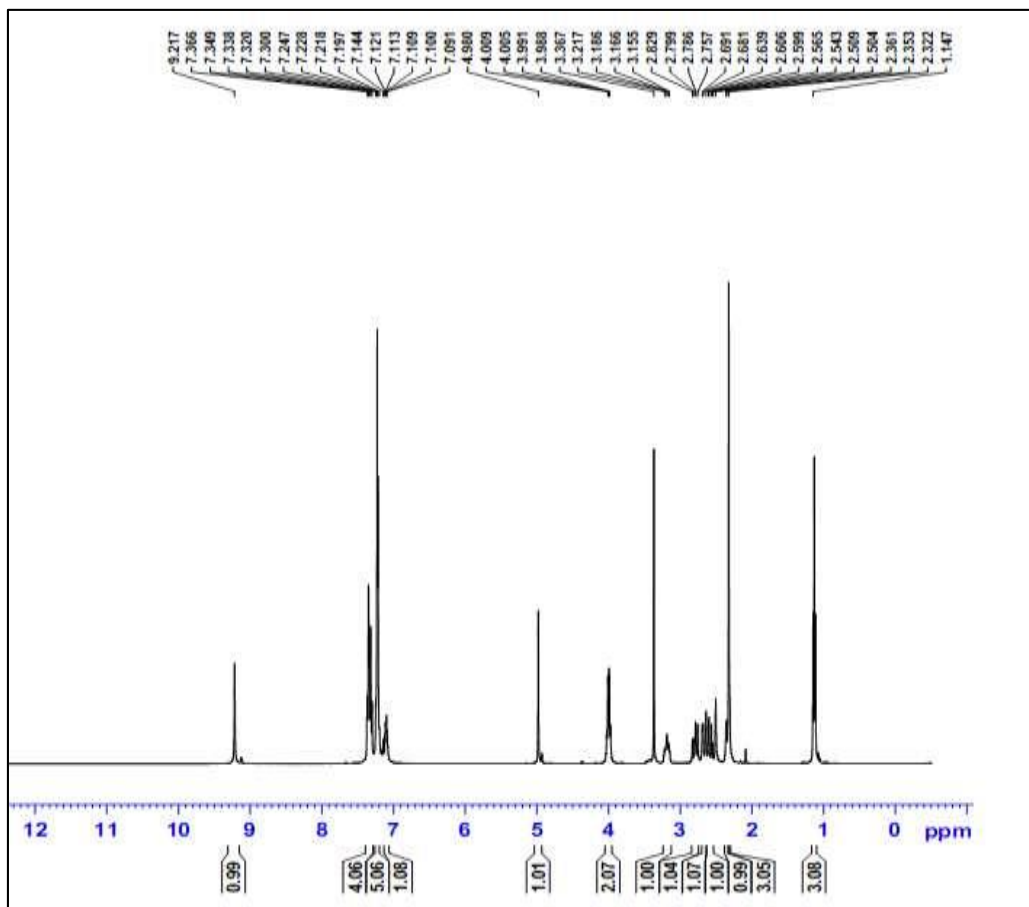


Fig. 27. ^1H NMR of (12a).

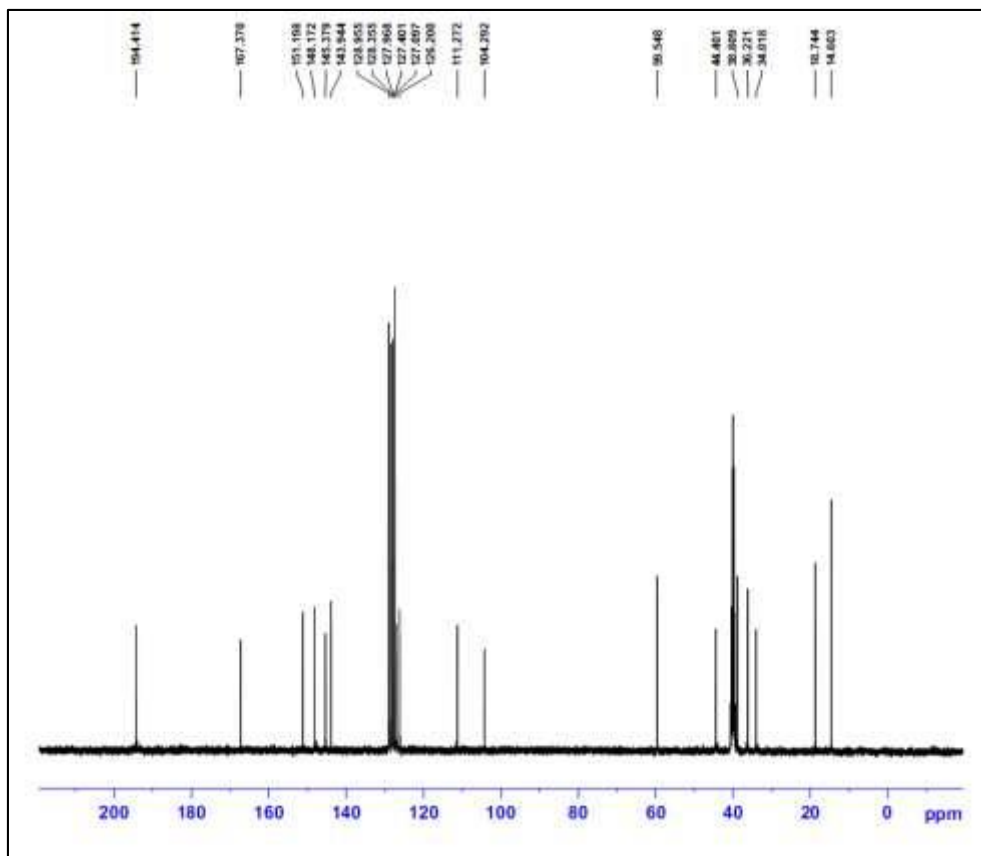
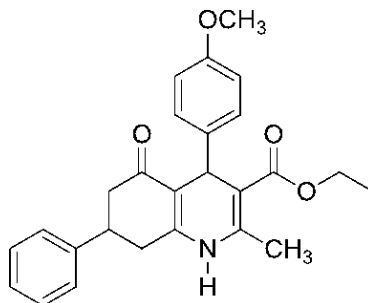


Fig. 28. ^{13}C NMR of (12a).

Ethyl 4-(p-methoxyphenyl)-2-methyl-5-oxo-7-phenyl-4, 6, 7, 8-tetrahydro-1H-quinoline-3-carboxylate (12b)



M.p. 236-238 °C, (KBr, Cm^{-1}) ν_{max} : 3280 (NH), 1689 (C=O) (acid), 1606 (C=O) (ketone), 1479 (OC₂H₅) (ester), 1222 (OCH₃) (ether) cm^{-1} ; δ_{H} / ppm (300 MHz, CDCl₃-d) 1.27 (m, 3H, CH₃-CH₂-C=O), 2.23-2.37 (m, 3H, CH₃-Ph), 2.38-2.52 (m, 5H, CH₂-CH-CH₂), 3.71 (s, 3H, CH₃-O-Ph), 4.07 (m, 2H, -O-CH₂-CH₃), 5.05 (m, 1H, CH-Ph), 6.71-7.12 (m, 4H, CH(p-OMe-Ph)), 6.96 (s, 1H, NH), 7.17-7.29 (m, 5H, CH (Ph)); δ_{C} /ppm (75 MHz, CDCl₃) 195.2, 167.5, 157.8, 150, 149.5, 143.4, 142.6, 139.7, 139.3, 128.8, 127, 126.6, 113.2, 106.2, 59.8, 55.1, 39.5, 38.8, 35.8, 34.4, 19.1, 14.2.

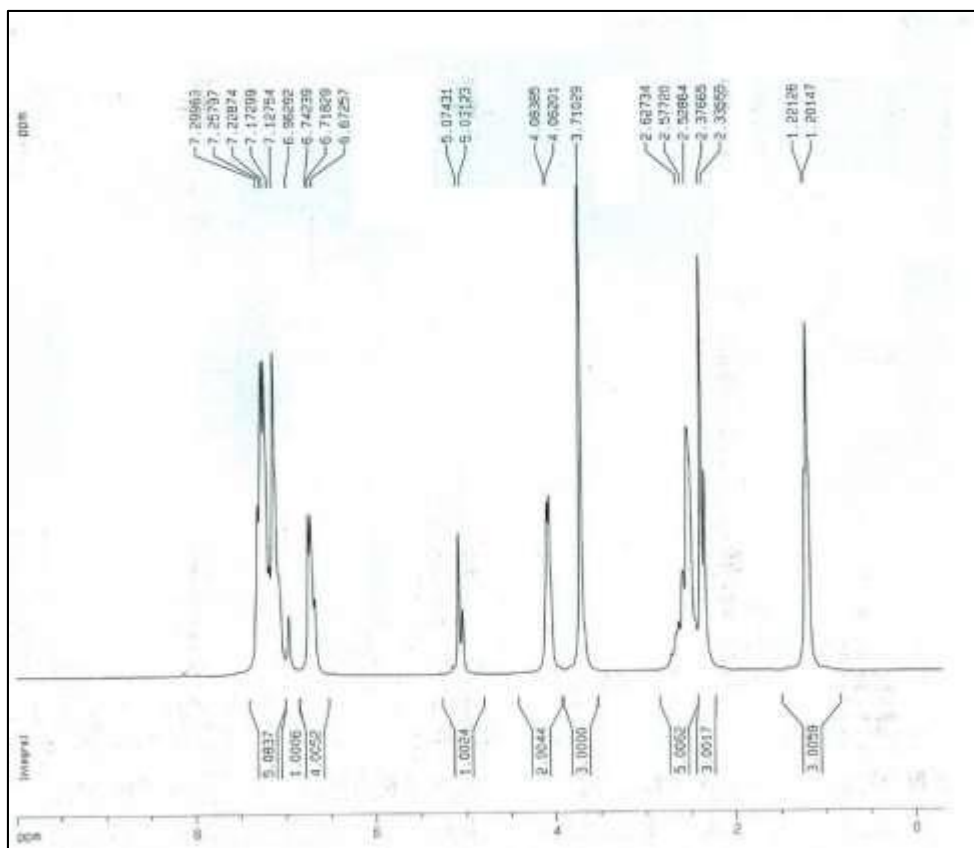


Fig. 29. ¹H NMR of (12b).

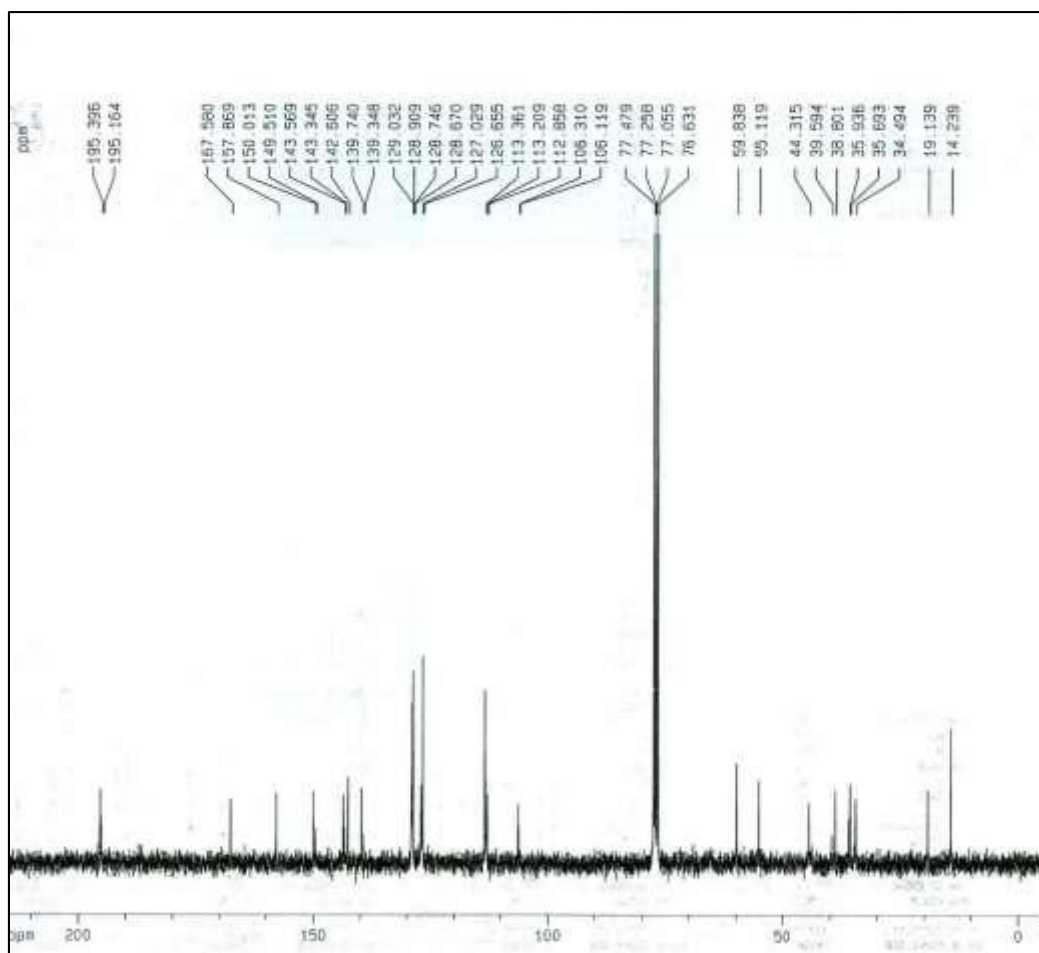
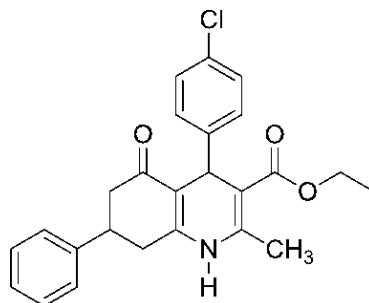


Fig. 30. ^{13}C NMR of (12b).

Ethyl 4-(4-chlorophenyl)-2-methyl-5-oxo-7-phenyl-1,4,5,6,7,8-hexahydroquinoline-3-carboxylate (12c)



M.p. 190-192 °C, (KBr, Cm^{-1}) ν_{max} : 3274 (NH), 1701(C=O) (acid), 1606 (C=O) (ketone), 1487 (OC₂H₅) (ester), 848 (C-Cl) cm^{-1} ; δ_{H} (400 MHz, DMSO-*d*₆) 1.12 (t, 3H, CH₃-CH₂-O-C=O), 2.3 (t, 1H, 8 H), 2.31 (s, 3H, CH₃), 2.5 (t, 1H, 8 H), 2.54–2.59 (7, 1H, 8 H), 2.59-2.66 (m, 1H, 6 H), 2.74-2.82 (m, 1H, 6 H), 3.17 (t, 1H, NH), 3.98 (q, 2H, -O-CH₂-CH₃), 4.90 (d, 1H, 4 H), 7.22-7.28 (m, 5H, 2', 6', 2'', 4'', 6'' H), 7.31–7.33 (m, 4H, 3', 5', 3'', 5'' H), 9.21 (d, 1H, NH) ppm; δ_{C} (100 MHz, DMSO-*d*₆) 194.2 (C-5), 167.1 (C=OOC₂H₅), 151.3 (C-2), 150.8 (C-1a), 147.1 (C-1''), 145.7 (C-1'), 143.8 (C-4'), 130.7 (C-2' and C-6'), 129.8 (C-3' and C-5'), 128.8 (C-3'' and C-5''), 128.1 (C-2'' and C-6''), 127.1 (C-4''), 111.0 (C-5a), 103.6 (C-3), 44.3 (O-CH₂-CH₃), 43.7 (C-6), 38.8 (C-4), 36.0 (C-8), 33.7 (C-7), 18.7 (1C, CH₃), 14.5 (1C, CH₃-CH₂O).

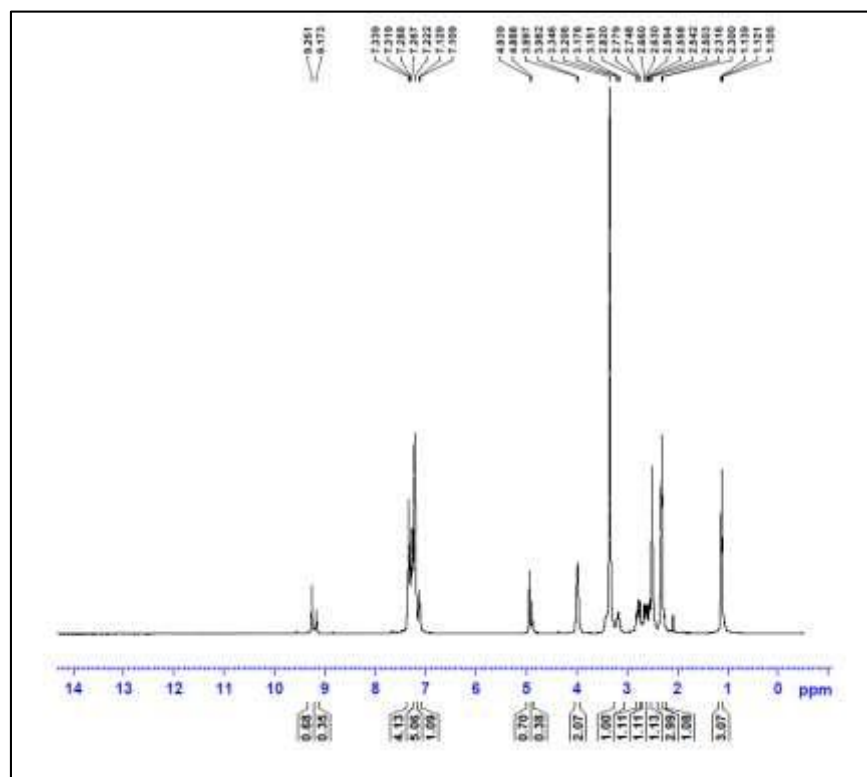


Fig. 31. ¹H NMR of (12c).

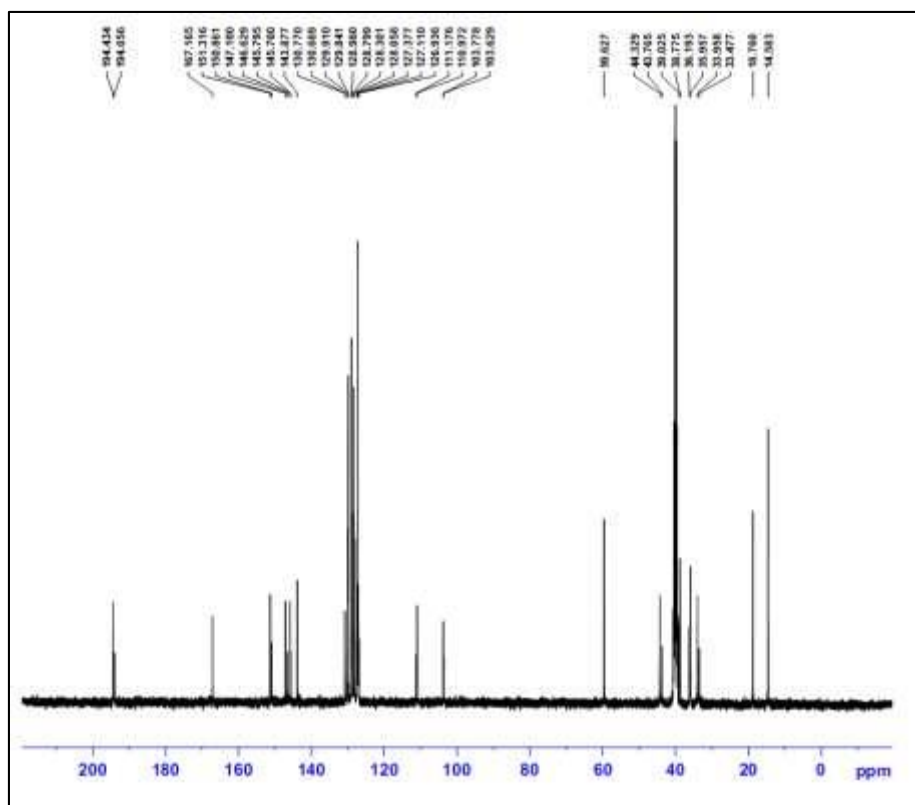
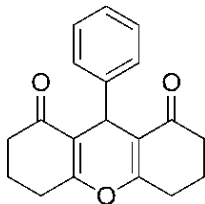


Fig. 32. ^{13}C NMR of (12c).

9-phenyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (13a)



M. P.: 213-215 °C; IR (KBr, cm^{-1}) ν_{max} : 3001, 1661, 1532, 1367, 1332, 1221, 1160, 1134, 1091, 841, 739. ^1H NMR (400 MHz, DMSO): δ 1.90-2.09 (m, 4H), 2.26-2.28 (m, 2H), 2.30-2.38 (m, 2H), 2.51-2.64 (m, 4H), 4.79 (s, 1H), 7.05-7.09 (t, $J=7.6$, 2H), 7.11-7.12 (d, $J=8.4$ Hz, 1H), 7.23-7.31 (dd, $J=8, 24.2$, 2H). ^{13}C NMR (100 MHz, $\text{DMSO}-d_6$): δ 20.42, 21.22, 27.19, 31.32, 37.08, 113.62, 117.13, 128.28, 128.93, 129.36, 135.97, 136.87, 141.76, 163.67, 196.62. Anal. Calcd for $\text{C}_{19}\text{H}_{18}\text{O}_3$: C, 77.53; H, 6.16; O, 16.31; Found: C, 77.58; H, 6.19; O, 16.37.

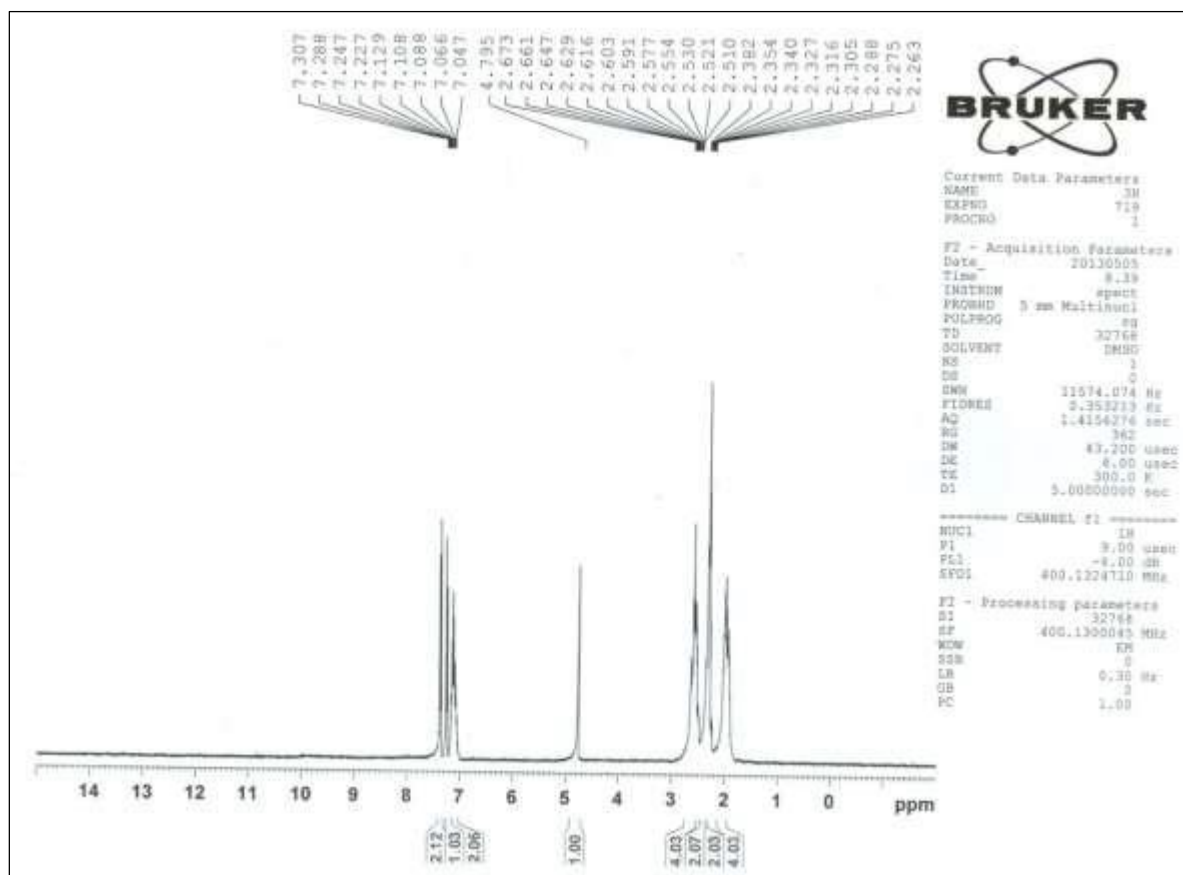


Fig. 33. ^1H NMR of 13a.

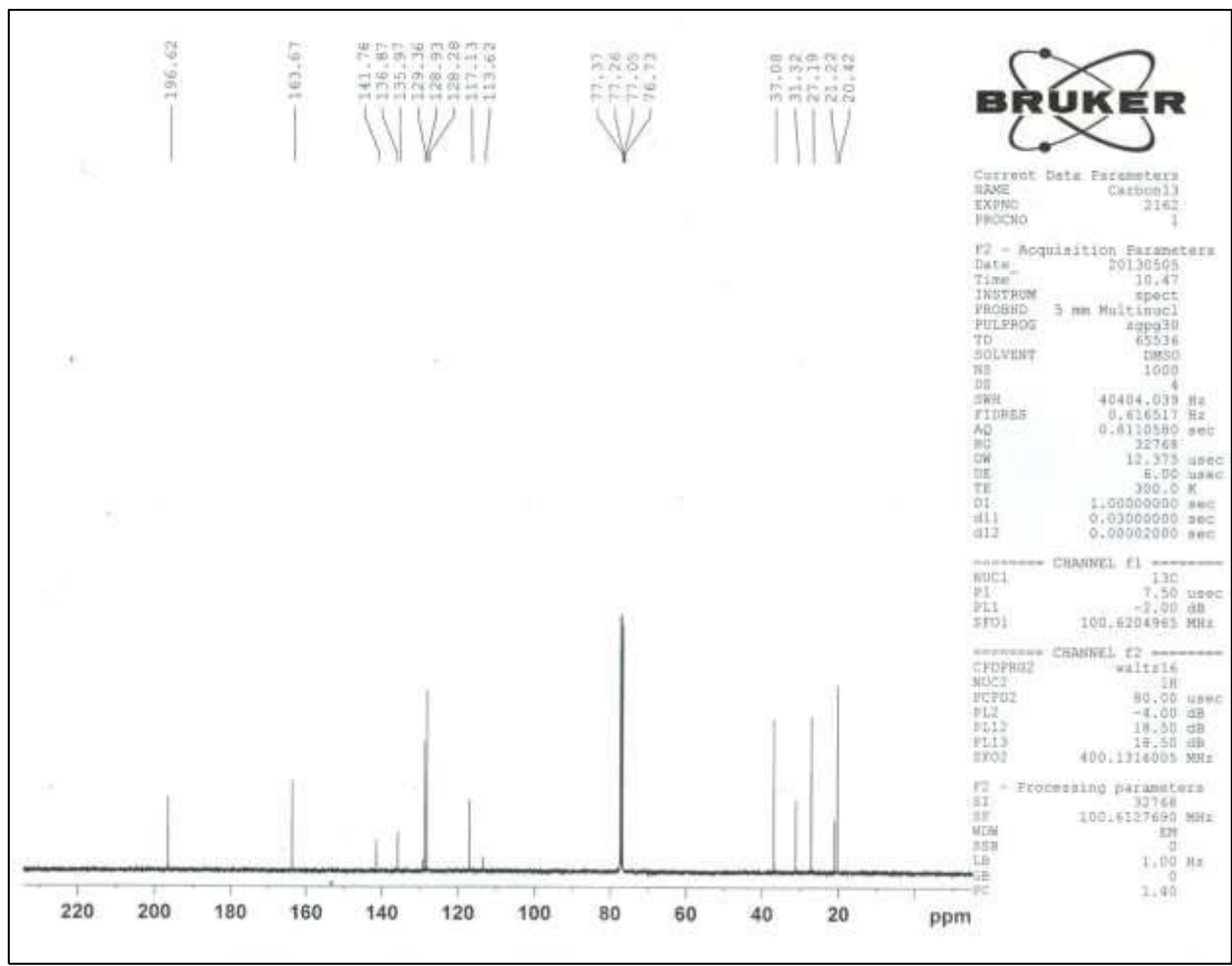
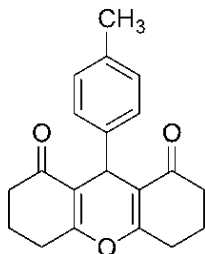


Fig. 34. ^{13}C NMR of 13a.

9-(p-tolyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (13b)



M. P.: 244-246 °C; IR (KBr, Cm^{-1}) ν_{max} : 2973, 1667, 1531, 1362, 1328, 1224, 1158, 1132, 1081, 832, 739. ^1H NMR (400 MHz, DMSO): δ 1.96-2.07 (m, 4H), 2.26 (s, 3H), 2.29-2.42 (m, 4H), 2.52-2.69 (m, 4H), 4.79 (s, 1H), 7.03-7.05 (d, $J = 8$ Hz, 2H), 7.12-7.21 (d, $J = 7.6$ Hz, 2H). ^{13}C NMR (100 MHz, DMSO- d^6): δ 20.31, 21.06, 27.15, 31.22, 36.98, 55.14, 113.52, 117.03, 128.25, 128.84, 129.33, 135.86, 136.88, 141.53, 163.77, 196.52. Anal. Calcd for $\text{C}_{20}\text{H}_{20}\text{O}_3$: C, 77.90; H, 6.54; O, 15.57; Found: C, 77.97; H, 6.59; O, 15.64.

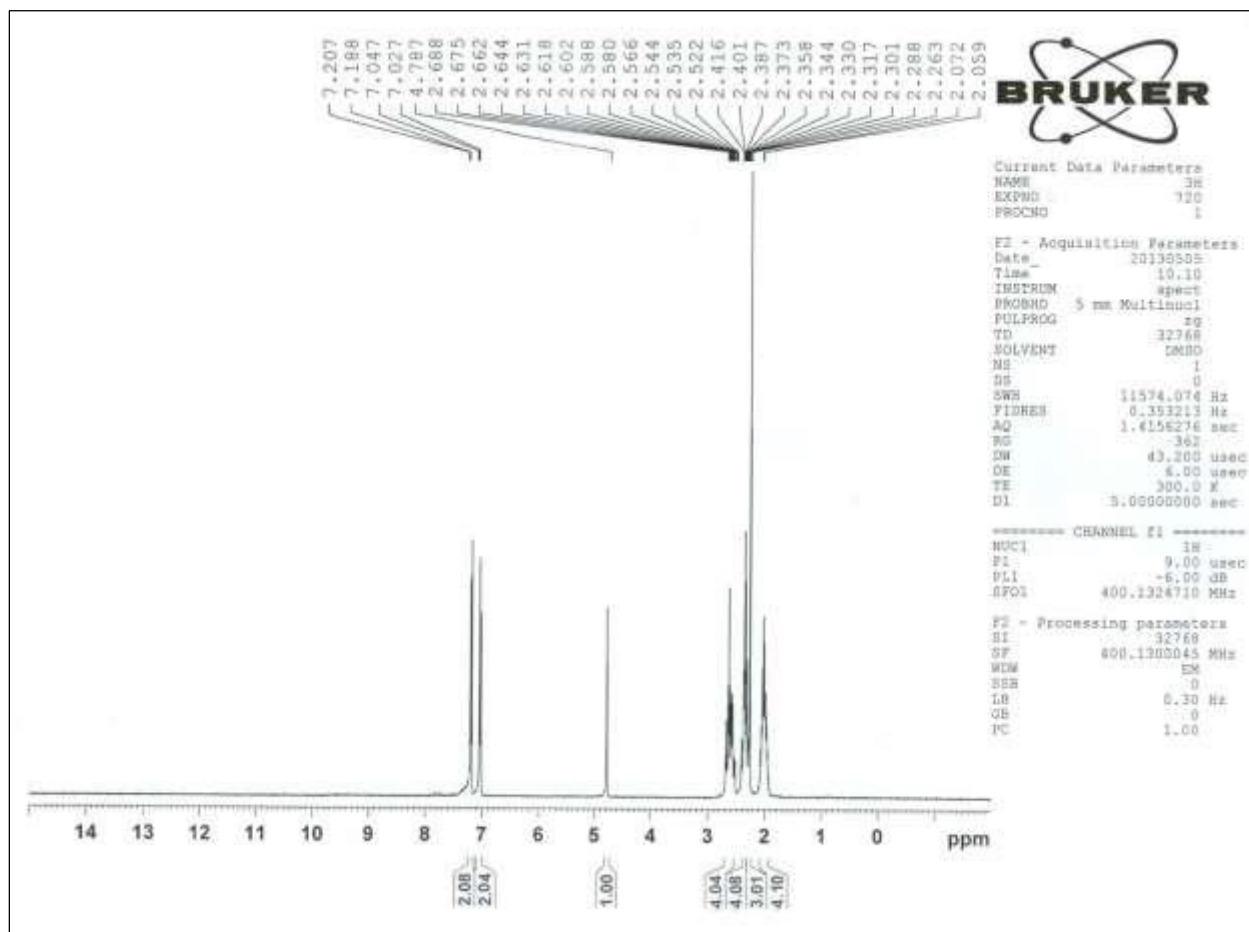


Fig. 35. ^1H NMR of 13b.

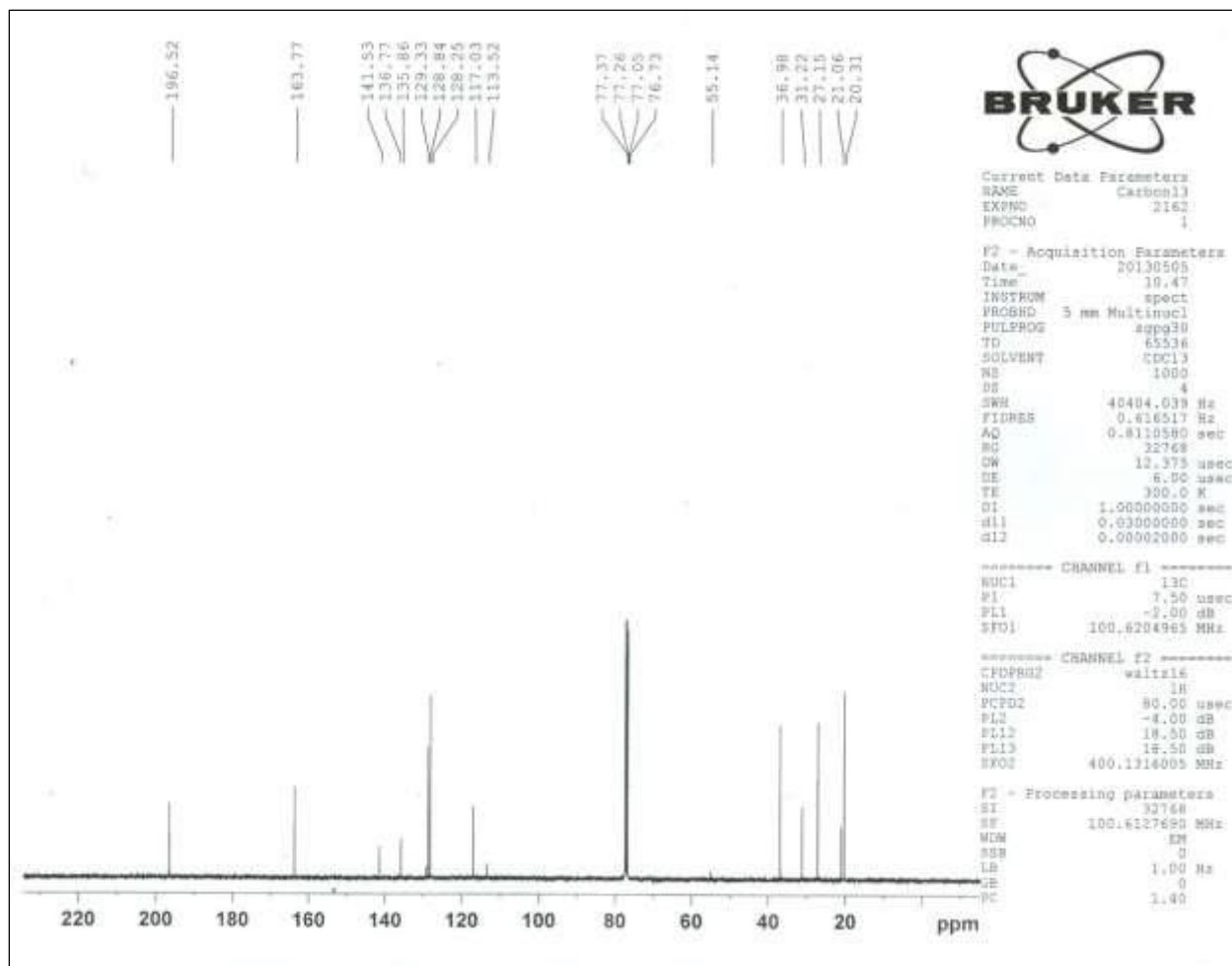
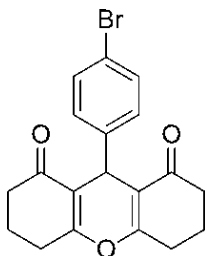


Fig. 36. ^{13}C NMR of 13b.

9-(4-bromophenyl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (13e)



M. P.: 222-225 °C; IR (KBr, cm^{-1}) ν_{max} : 2976, 1662, 1518, 1375, 1329, 1226, 1159, 1138, 1089, 829, 737. ^1H NMR (400 MHz, DMSO): δ 1.92-2.08 (m, 4H), 2.28-2.32 (m, 2H), 2.34-2.40 (m, 2H), 2.53-2.65 (m, 4H), 4.76 (s, 1H), 7.13-7.27 (m, 2H), 7.33-7.43 (dd, $J = 30.6, 8.4$ Hz, 2H). ^{13}C NMR (100 MHz, DMSO- d_6): δ 20.10, 20.27, 27.13, 31.39, 36.76, 36.90, 116.41, 120.28, 129.43, 130.21, 131.17, 131.66, 131.83, 133.08, 143.47, 158.49, 164.12, 196.51. Anal. Calcd for $\text{C}_{19}\text{H}_{17}\text{BrO}_3$: C, 61.14; H, 4.59; O, 12.86; Found: C, 61.19; H, 4.65; O, 12.93.

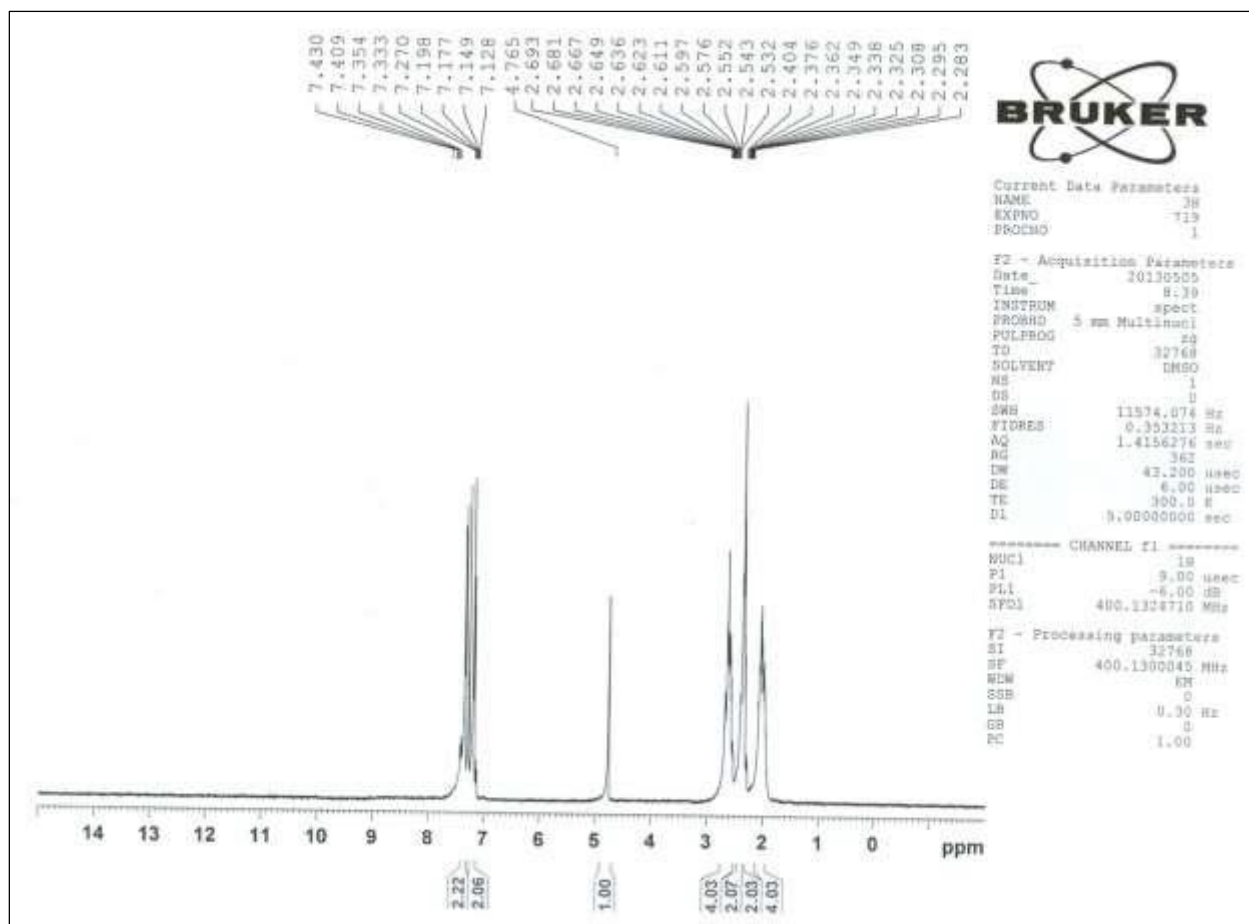


Fig. 37. ^1H NMR of 13e.

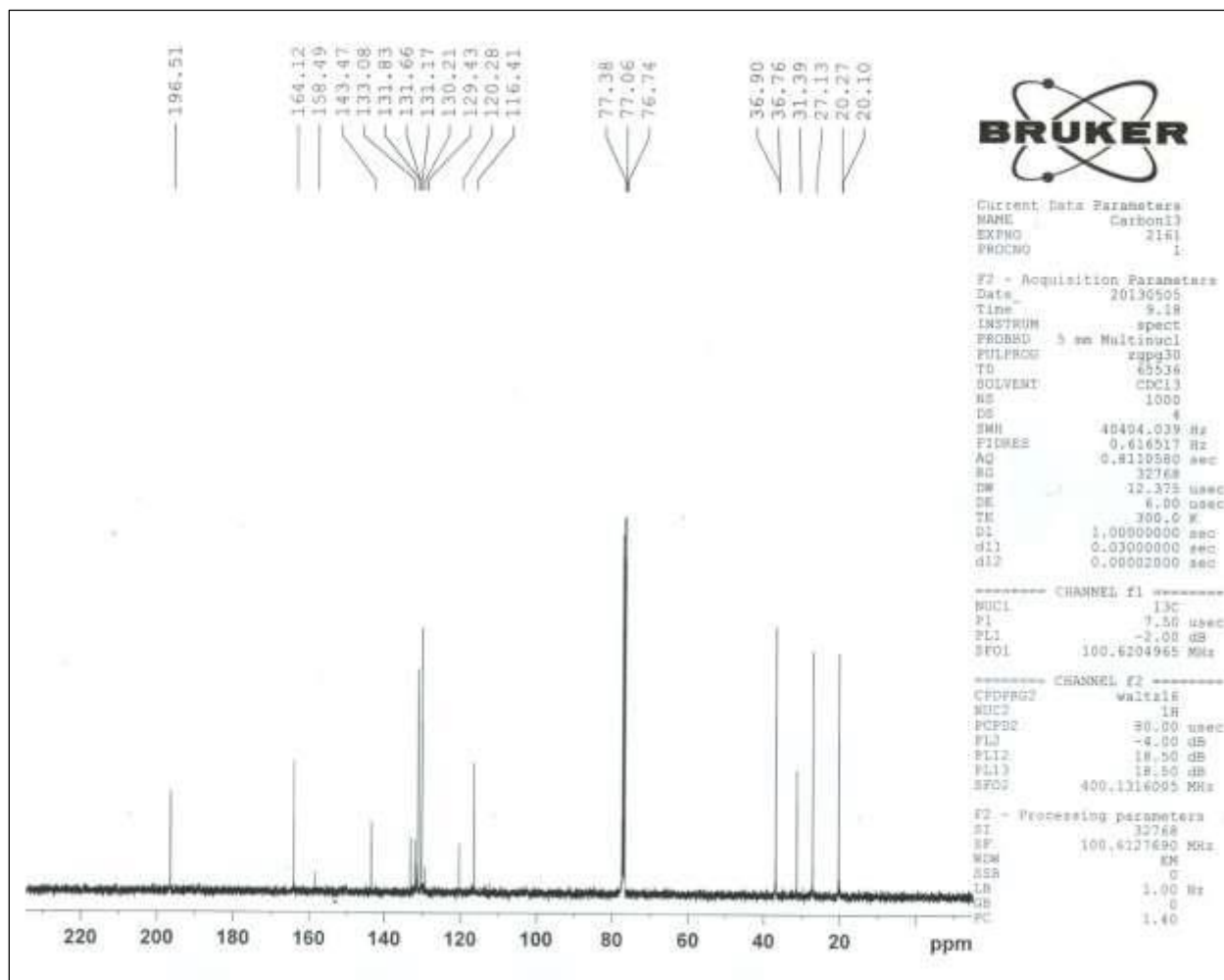
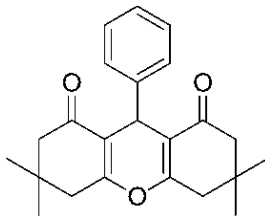


Fig. 38. ^{13}C NMR of 13e.

3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-phenyl-2H-xanthene-1,8(5H,9H)-dione (14a)



M. P.: 203-204 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 2950, 1660, 1648, 1360, 1200, 1162, 1141, 998, 694. ^1H NMR (500 MHz, $\text{DMSO}-d_6$): δ : 0.90 (s, 6H), 1.04 (s, 6H), 2.07-2.10 (d, $J=6.4$ Hz, 2H), 2.25-2.28 (d, $J=6.5$ Hz, 2H), 2.51-2.60 (m, 3H), 4.53 (s, 1H), 7.09-7.12 (t, $J=2.8$ Hz, 1H), 7.17-7.18 (d, $J=2.8$ Hz, 1H), 7.20-7.23 (t, $J=2.9$ Hz, 3H). ^{13}C NMR (DMSO , 125 MHz), δ : 26.45, 29.13, 32.14, 32.88, 50.24, 111.47, 125.46, 127.56, 127.63, 147.15, 149.32, 194.36. Anal. Calcd for $\text{C}_{23}\text{H}_{26}\text{O}_3$: C, 78.83; H, 7.48; O, 13.70; Found: C, 78.92; H, 7.59; O, 13.83.

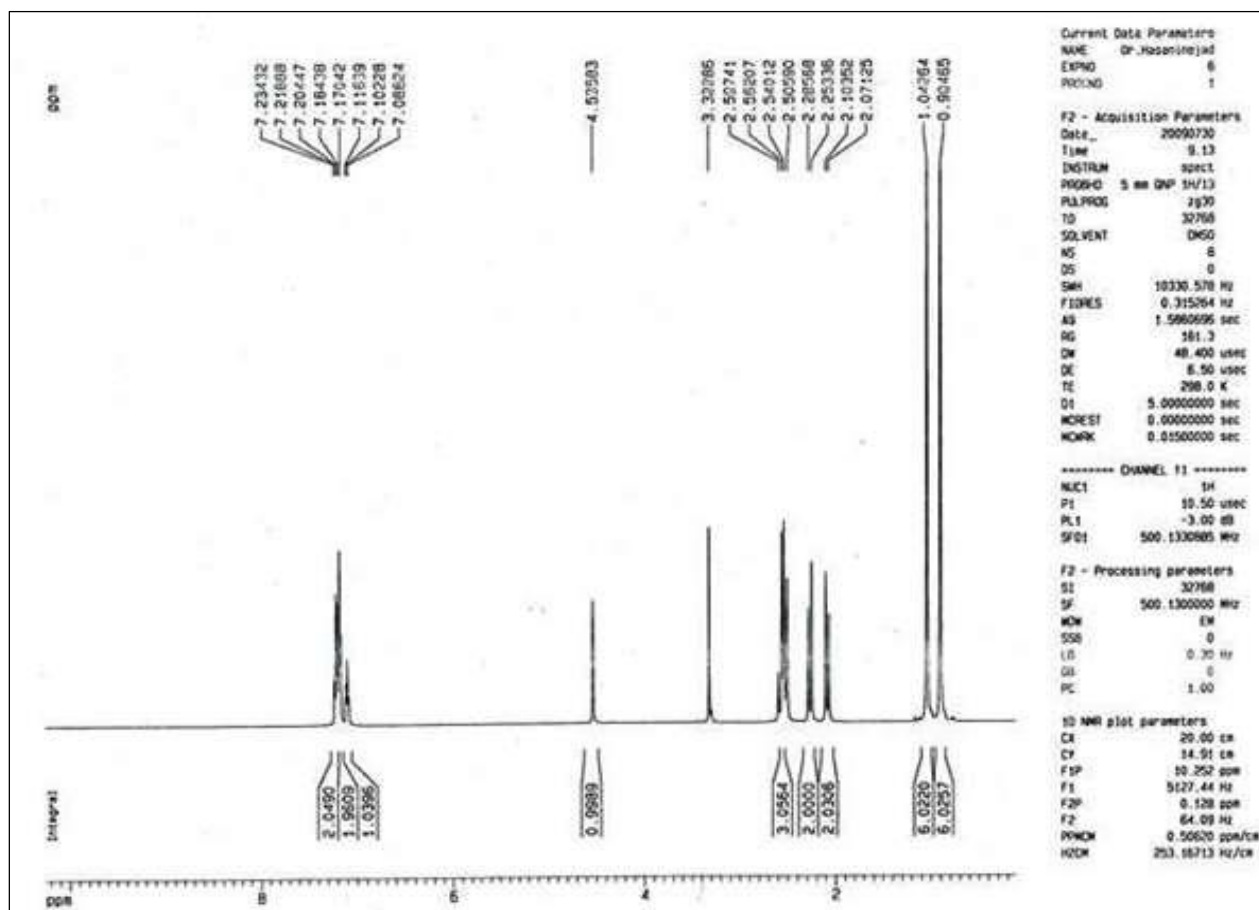


Fig. 39. ^1H NMR of 14a.

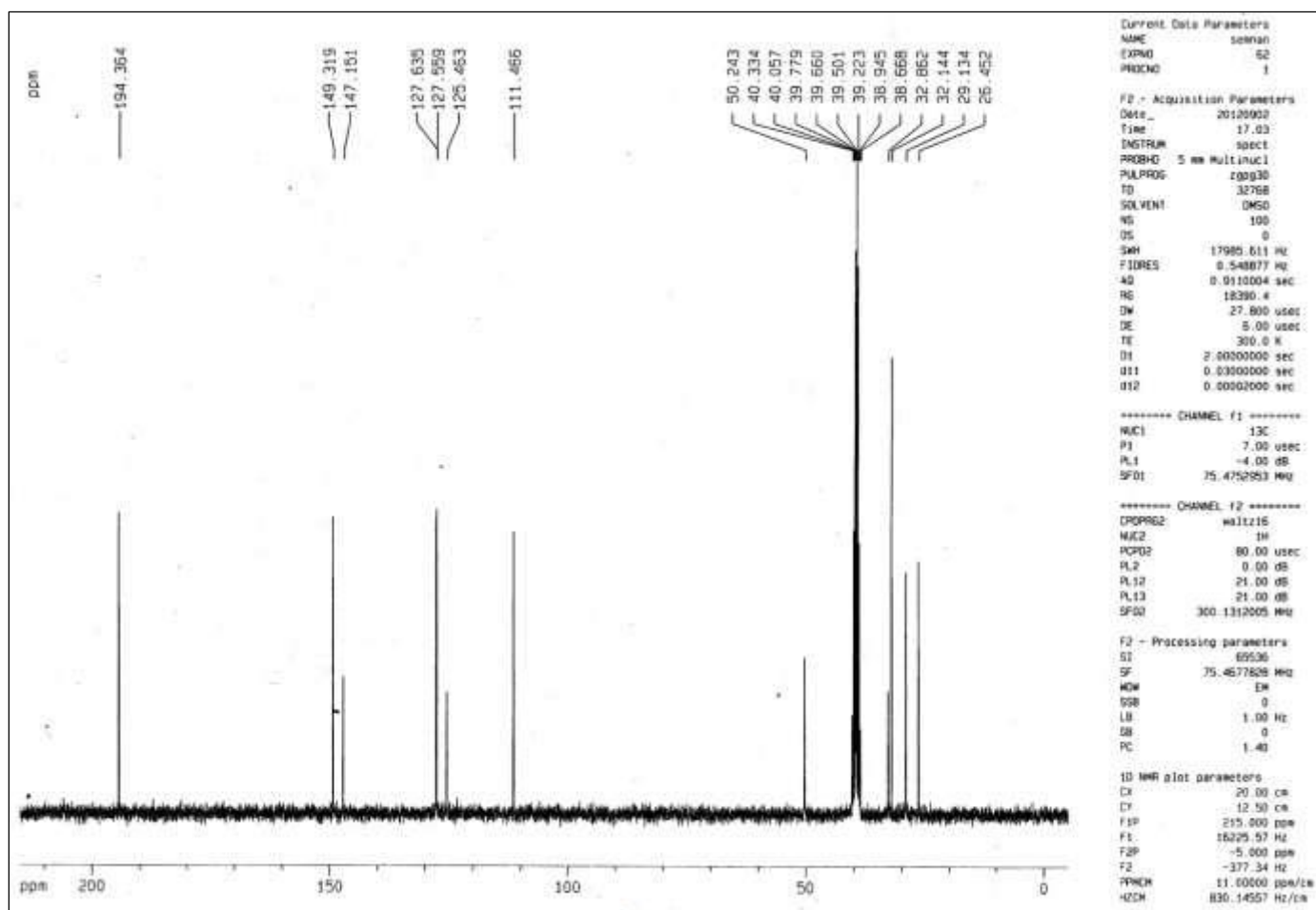
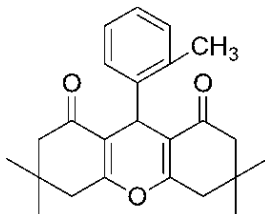


Fig. 40. ^{13}C NMR of 14a.

3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-o-tolyl-2H-xanthene-1,8(5H,9H)-dione (14b)



M. P.: 230-232 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 2910, 1660, 1620, 1350, 1200, 1160, 1020, 853. ^1H NMR (500 MHz, CDCl_3): δ : 0.88 (s, 6H), 1.04 (s, 6H), 2.03-2.06 (d, $J=6.5$ Hz, 2H), 2.24-2.27 (d, $J=6.5$ Hz, 2H), 2.48-2.51 (d, $J=6.5$ Hz, 2H), 2.57-2.60 (d, $J=7$ Hz, 2H) 2.71 (s, 3H), 4.63 (s, 1H), 7.09-7.11 (t, $J=5.6$ Hz, 1H), 7.17-7.18 (d, $J=2.8$ Hz, 1H) 7.20-7.23 (t, $J=6.0$ Hz, 2H). Anal. Calcd for $\text{C}_{24}\text{H}_{28}\text{O}_3$: C, 79.09; H, 7.74; O, 13.17; Found: C, 79.18; H, 7.89; O, 13.31.

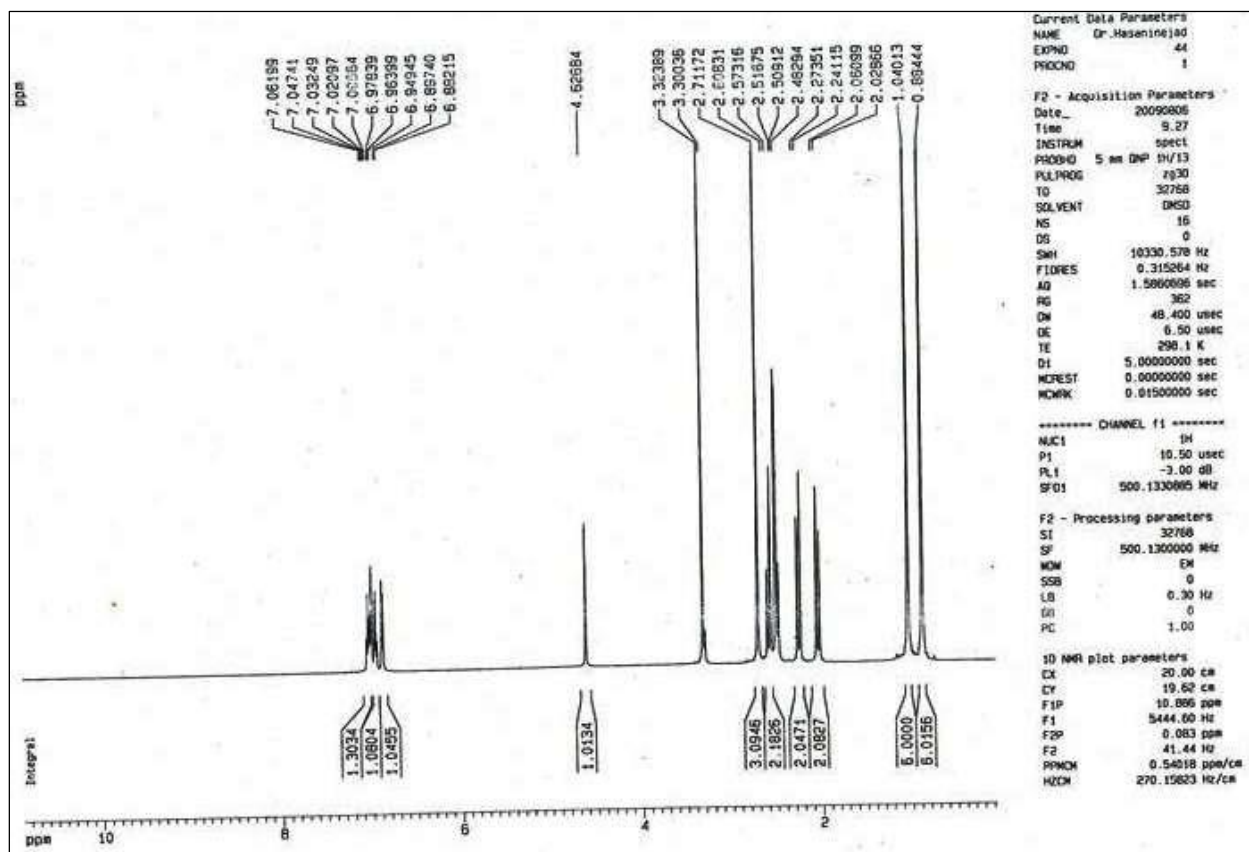
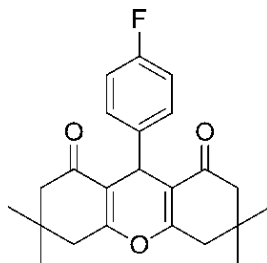


Fig. 41. ^1H NMR of 14b.

9-(4-fluorophenyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione
(14f)



M. P.: 206-207 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 2980, 1672, 1512, 1373, 1333, 1218, 1166, 1135, 1086, 835, 733. ^1H NMR (400 MHz, CDCl_3) δ : 0.99 (s, 6H), 1.11 (s, 6H), 2.15-2.26 (dd, $J=16$, 28.6 Hz, 4H), 2.46 (s, 4H), 4.73 (s, 1H), 6.88-6.92 (t, $J=8.8$ Hz, 2H), 7.24-7.27 (d, $J=10.8$ Hz, 2H). ^{13}C NMR (125 MHz, CDCl_3) δ : 27.22, 29.53, 32.00, 32.74, 50.05, 56.62, 60.79, 106.34, 115.16, 136.89, 140.71, 153.26, 163.56, 197.05. Anal. Calcd for $\text{C}_{23}\text{H}_{25}\text{FO}_3$: C, 74.98; H, 6.84; O, 13.03; Found: C, 74.93; H, 6.82; O, 13.09.

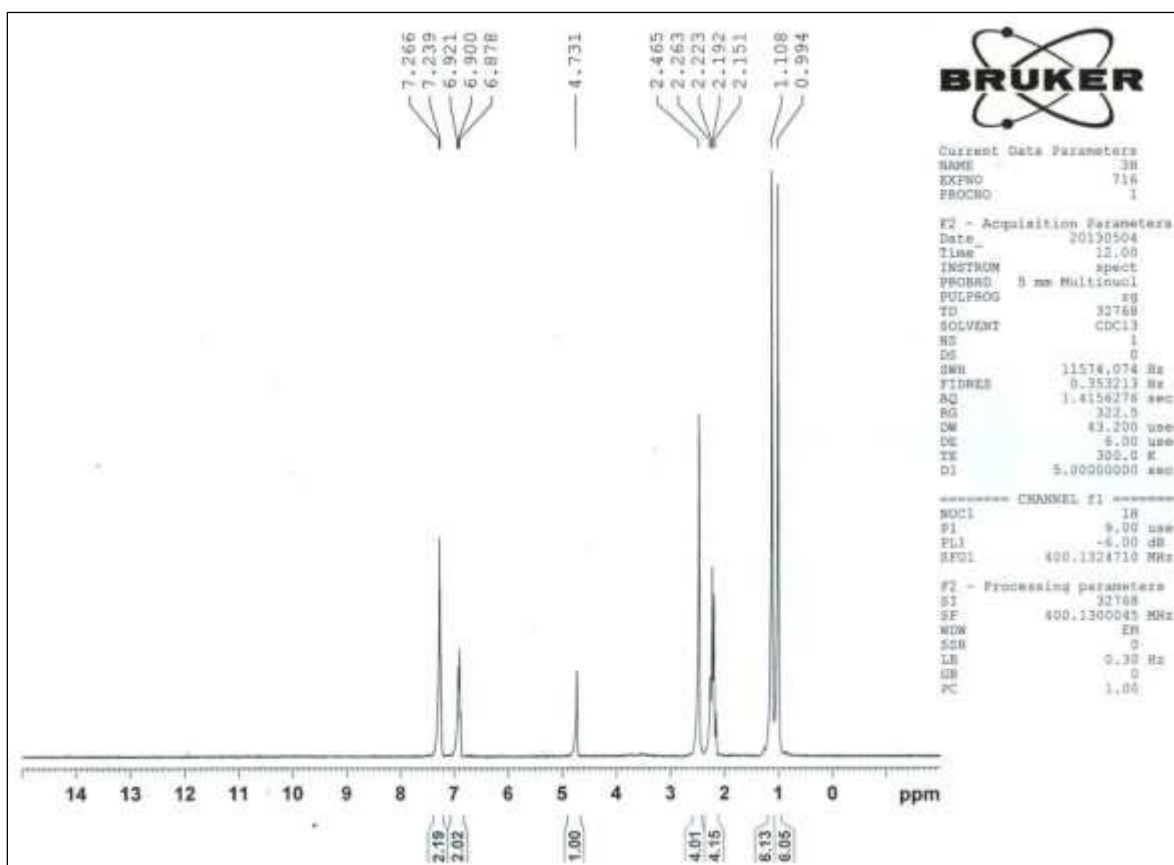


Fig. 42. ^1H NMR of 14f.

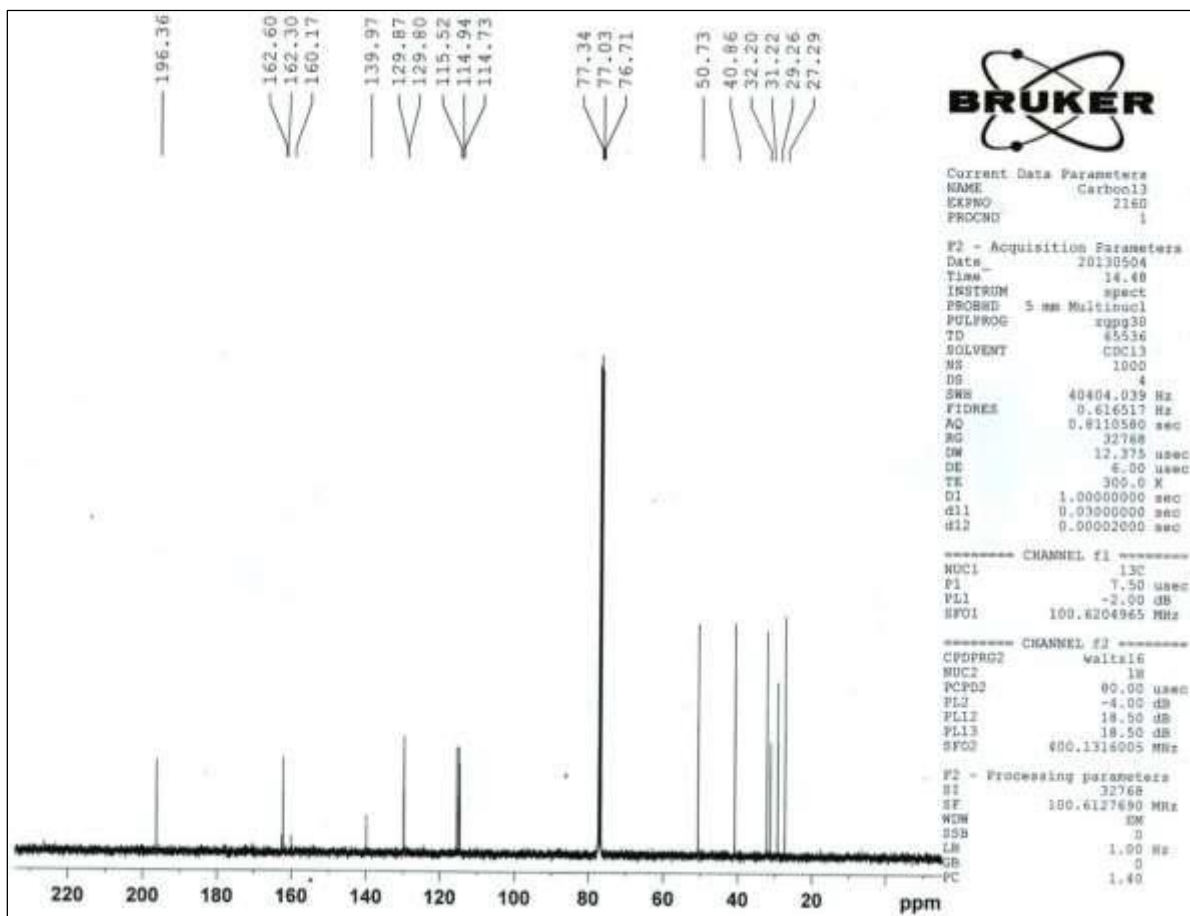
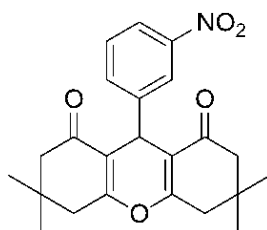


Fig. 43. ^{13}C NMR of 4f.

3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-(3-nitrophenyl)-2H-xanthene-1,8(5H,9H)-dione
(14j)



M. P.: 168-169 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 2950, 1659, 1619, 1520, 1360, 1200, 1180, 1000. ^1H NMR (500 MHz, CDCl_3): δ : 0.91 (s, 6H), 1.05 (s, 6H), 2.09-2.12 (d, $J=4.4$ Hz, 2H), 2.27-2.30 (d, $J=6.4$ Hz, 2H), 2.55-2.63 (dd, $J=7.2$ Hz, 4H), 4.64 (s, 1H), 7.55-7.58 (t, $J=3.1$ Hz, 1H), 7.65-7.67 (d, $J=3$ Hz, 1H), 7.99-8.02 (d, $J=5.1$ Hz, 2H). ^{13}C NMR (125 MHz, CDCl_3) δ : 27.24, 29.56, 32.03, 32.72, 50.02, 56.65, 60.77, 106.31, 115.13, 136.85, 140.75, 153.22, 163.89, 197.01. Anal. Calcd. for $\text{C}_{23}\text{H}_{25}\text{NO}_5$: C, 69.86; H, 6.37; N, 3.54; O, 20.23; Found: C, 69.91; H, 6.49; N, 3.68; O, 20.35.

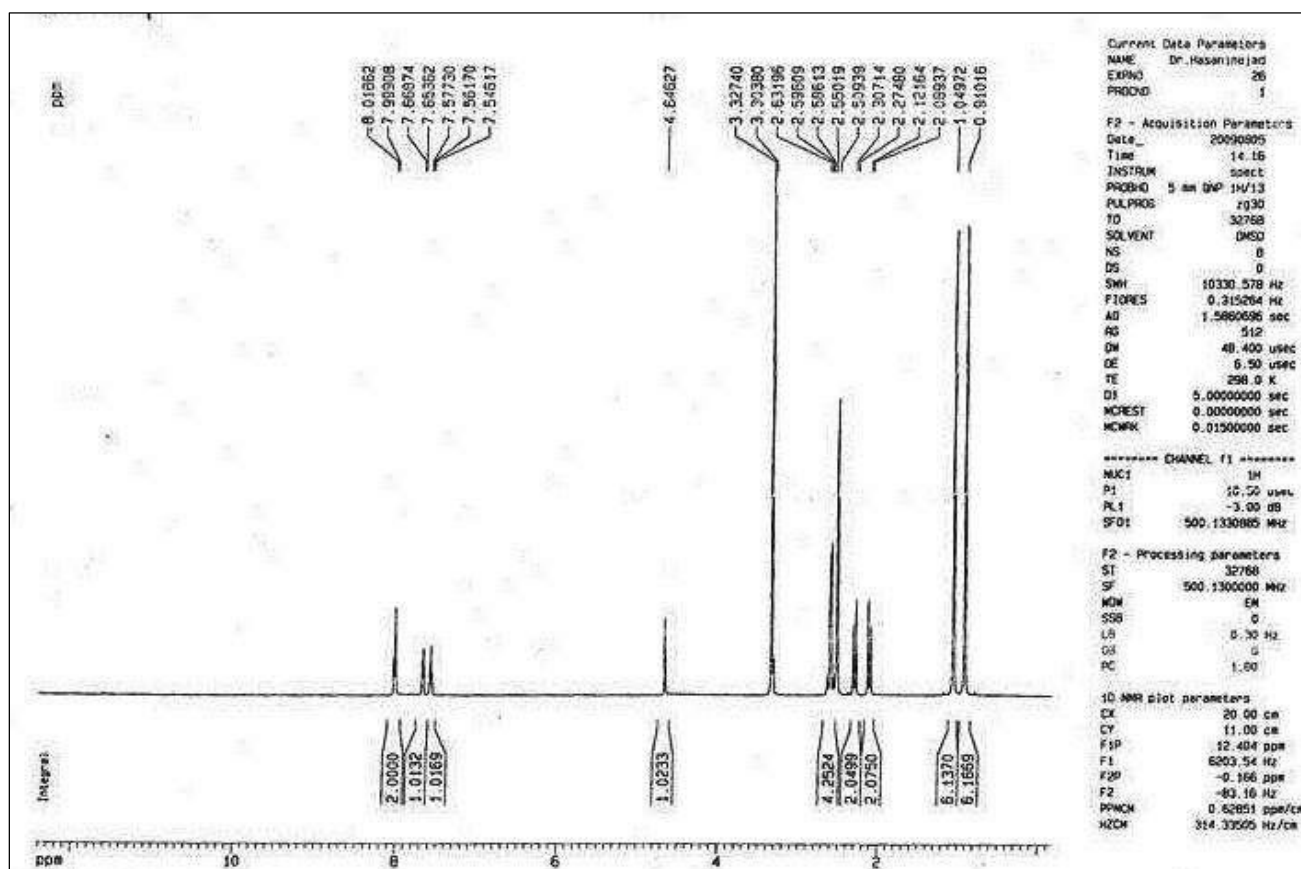


Fig. 44. ^1H NMR of 14j.

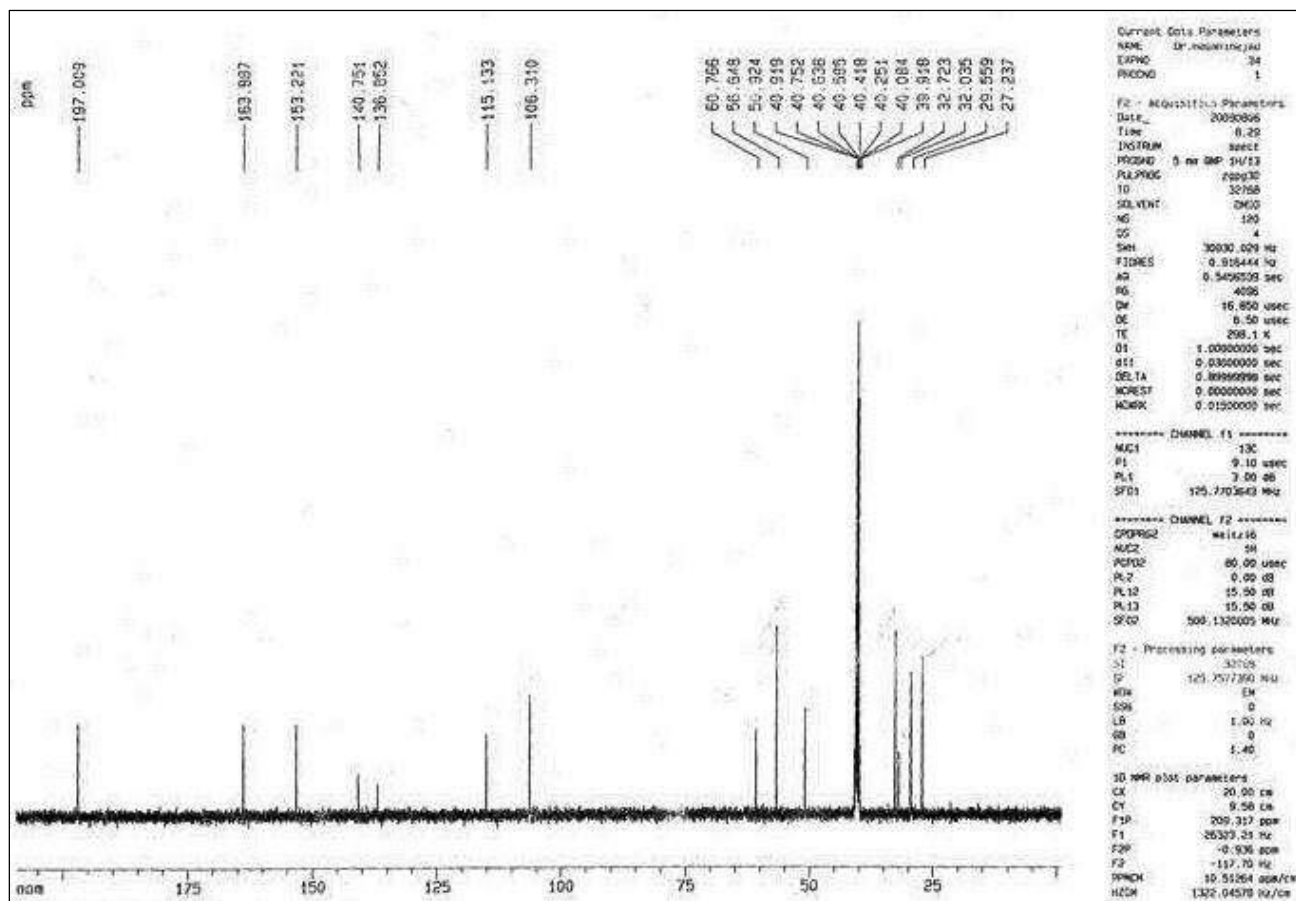
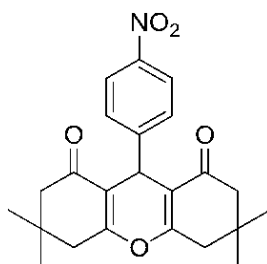


Fig. 45. ^{13}C NMR of 14j.

3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-9-(4-nitrophenyl)-2H-xanthene-1,8(5H,9H)-dione
(14k)



M. P.: 222-224 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 2950, 1658, 1614, 1508, 1360, 1340, 1196, 1160, 1136. ^1H NMR (500 MHz, CDCl_3) δ : 0.90 (s, 6H), 1.05 (s, 6H), 2.08-2.11 (d, $J=6.5$ Hz, 2H), 2.27-2.30 (d, $J=6.5$ Hz, 2H), 2.50-2.63 (m, 4H), 4.63 (s, 1H), 7.45-7.48 (m, 2H), 8.10-8.12 (m, 2H). ^{13}C NMR (125 MHz, CDCl_3) δ : 27.24, 29.57, 32.13, 32.74, 50.96, 56.69, 60.80, 106.33, 115.15, 136.86, 140.77, 153.26, 163.89, 197.11. Anal. Calcd for $\text{C}_{23}\text{H}_{25}\text{NO}_5$: C, 69.86; H, 6.37; N, 3.54; O, 20.23; Found: C, 69.91; H, 6.49; N, 3.68; O, 20.35.

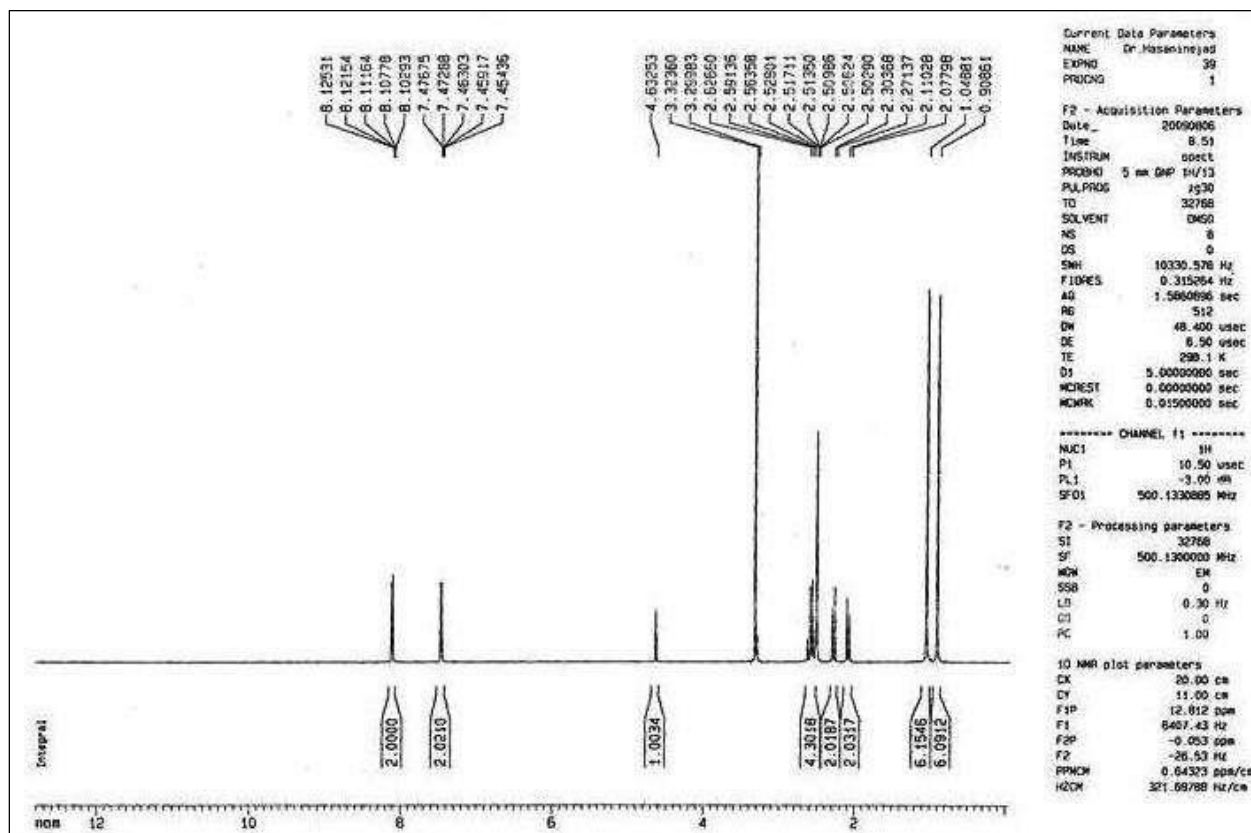


Fig. 46. ^1H NMR of 14k.

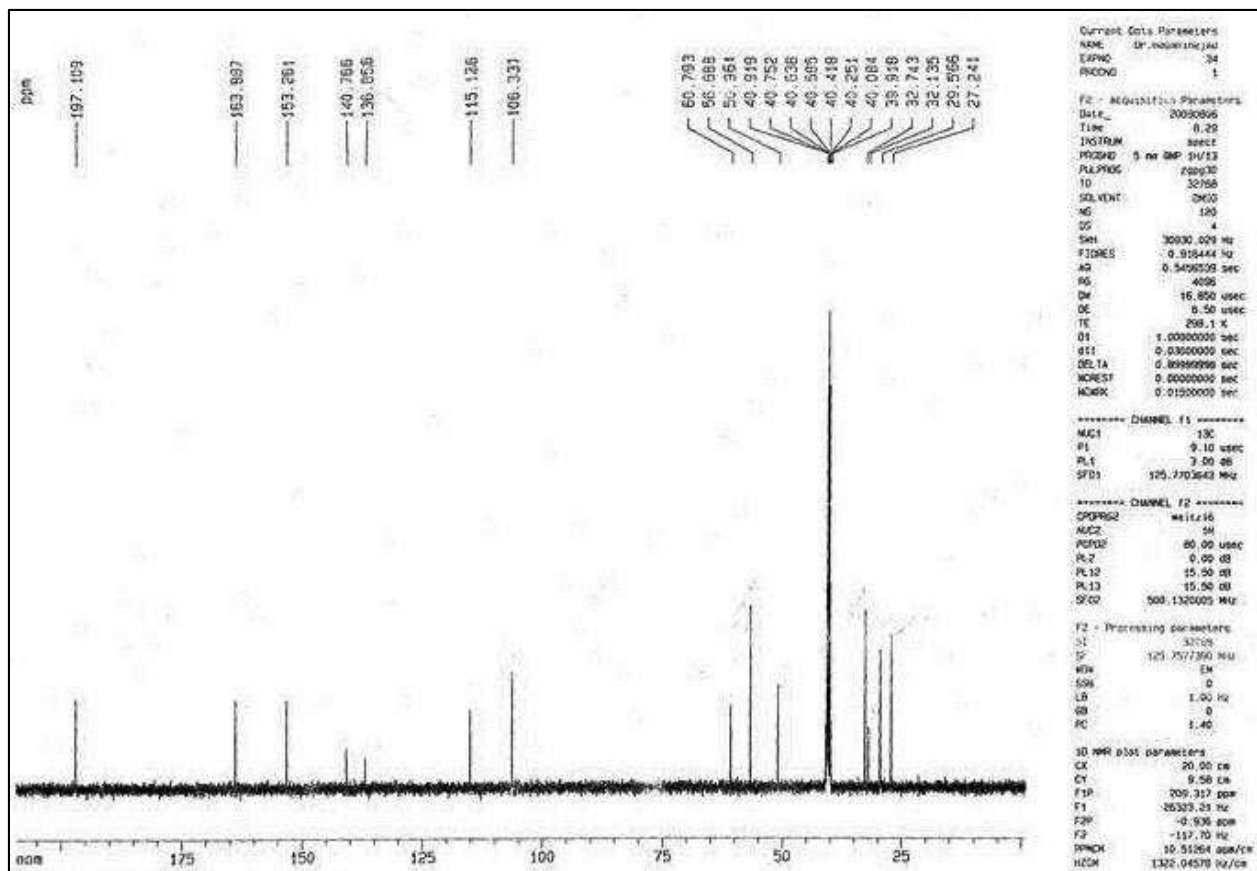
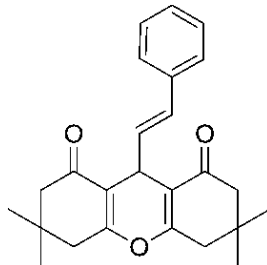


Fig. 47. ^{13}C NMR of 14k.

(E)-3,3,6,6-tetramethyl-9-styryl-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione (14l)



M. P.: 256-258 °C; FT-IR (KBr, cm^{-1}) ν_{max} : 3412, 3000, 1630, 1610, 1380, 1200, 1160, 1140, 1000, 958. ^1H NMR (500 MHz, CDCl_3) δ : 1.04-1.06 (d, 12H), 2.23-2.26 (d, J = 16.17 Hz, 2H), 2.30-2.33 (d, J = 16.02 Hz, 2H), 2.52 (s, 4H), 4.15-4.16 (d, J = 5.29 Hz, 1H), 6.16-6.22 (m, 2H), 7.19-7.28 (m, ArH, 5H). ^{13}C NMR (125 MHz, CDCl_3) δ : 27.68, 28.33, 29.41, 32.79, 50.98, 114.08, 126.82, 129.19, 129.45, 130.41, 132.21, 137.48, 164.38, 197.15. Anal. Calcd for $\text{C}_{25}\text{H}_{28}\text{O}_3$: C, 79.75; H, 7.50; O, 12.75; Found: C, 79.82; H, 7.52; O, 12.78.

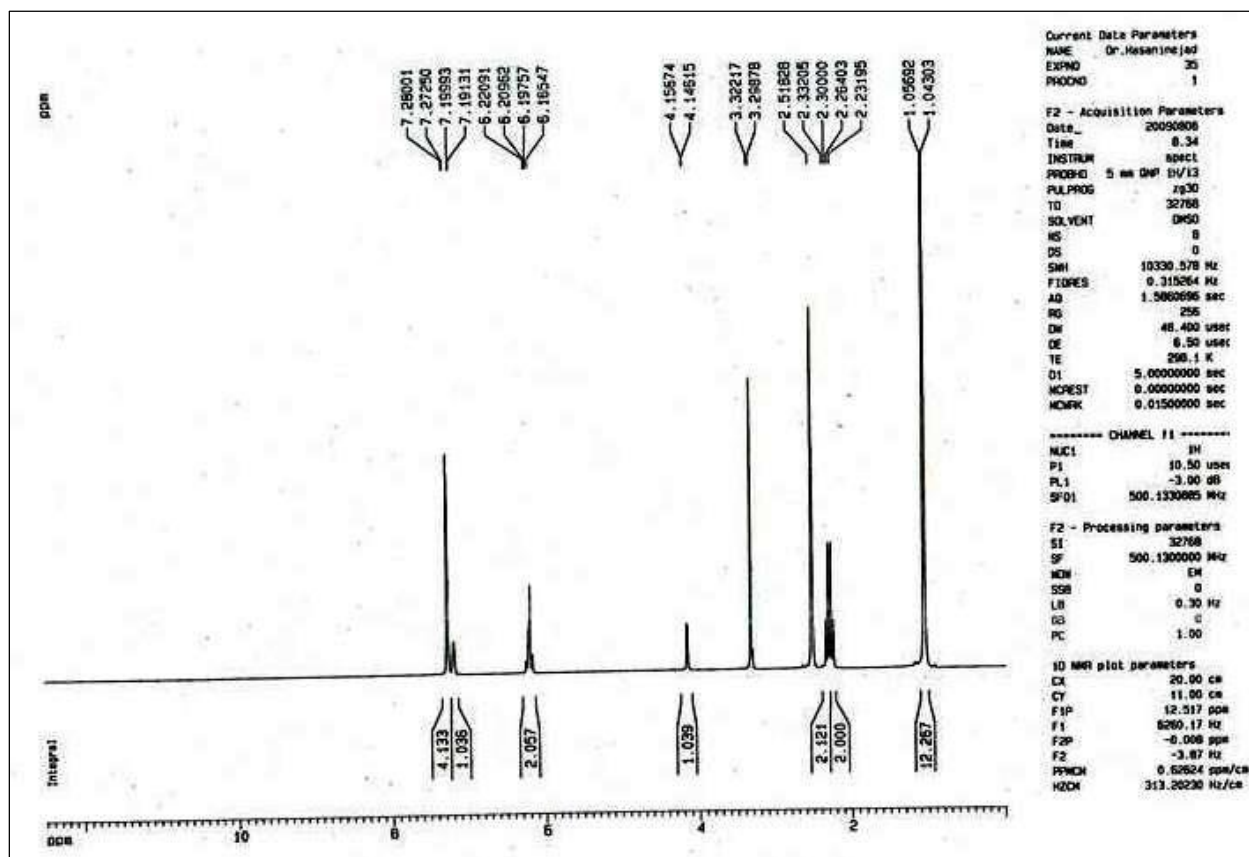


Fig. 48. ^1H NMR of 14l.

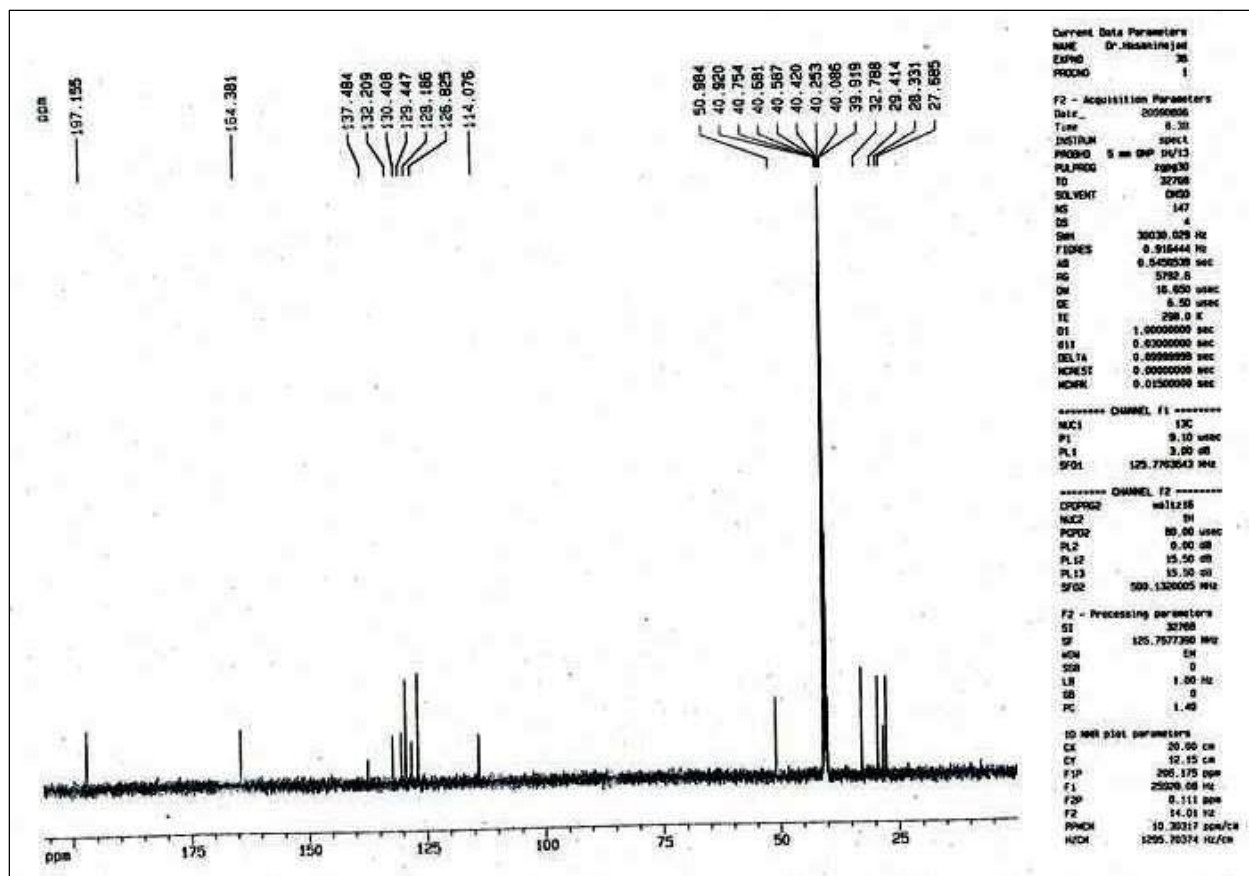
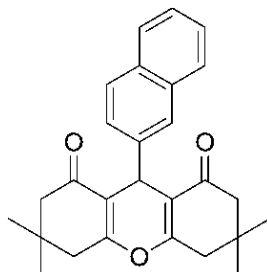


Fig. 49. ^{13}C NMR of 14l.

**3,3,6,6-tetramethyl-9-(naphthalen-2-yl)-3,4,5,6,7,9-hexahydro-1H-xanthene-1,8(2H)-dione
(14m)**



M. P.: 234-235 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3053, 2833, 1640, 1585, 1503, 1373, 1276, 1133, 813, 748. ^1H NMR (400 MHz, CDCl_3): δ : 0.99 (s, 6H), 1.12 (s, 6H), 2.13-2.06 (d, $J=6.5$ Hz, 2H), 2.24-2.27 (d, $J=6.5$ Hz, 2H), 2.48-2.51 (d, $J=6.5$ Hz, 2H), 2.57-2.60 (d, $J=7$ Hz, 2H) 2.71 (s, 3H), 4.63 (s, 1H), 7.09-7.11 (t, $J=5.6$ Hz, 1H), 7.17-7.18 (d, $J=2.8$ Hz, 1H) 7.20-7.23 (t, $J=6.0$ Hz, 2H). ^{13}C NMR (125 MHz, CDCl_3) δ : 21.47, 27.80, 29.69, 31.86, 32.62, 41.30, 51.20, 116.19, 128.66, 129.20, 136.17, 141.63, 162.51, 196.80. Anal. Calcd for $\text{C}_{27}\text{H}_{28}\text{O}_3$: C, 80.97; H, 7.05; O, 11.98; Found: C, 80.91; H, 7.09; O, 12.05.

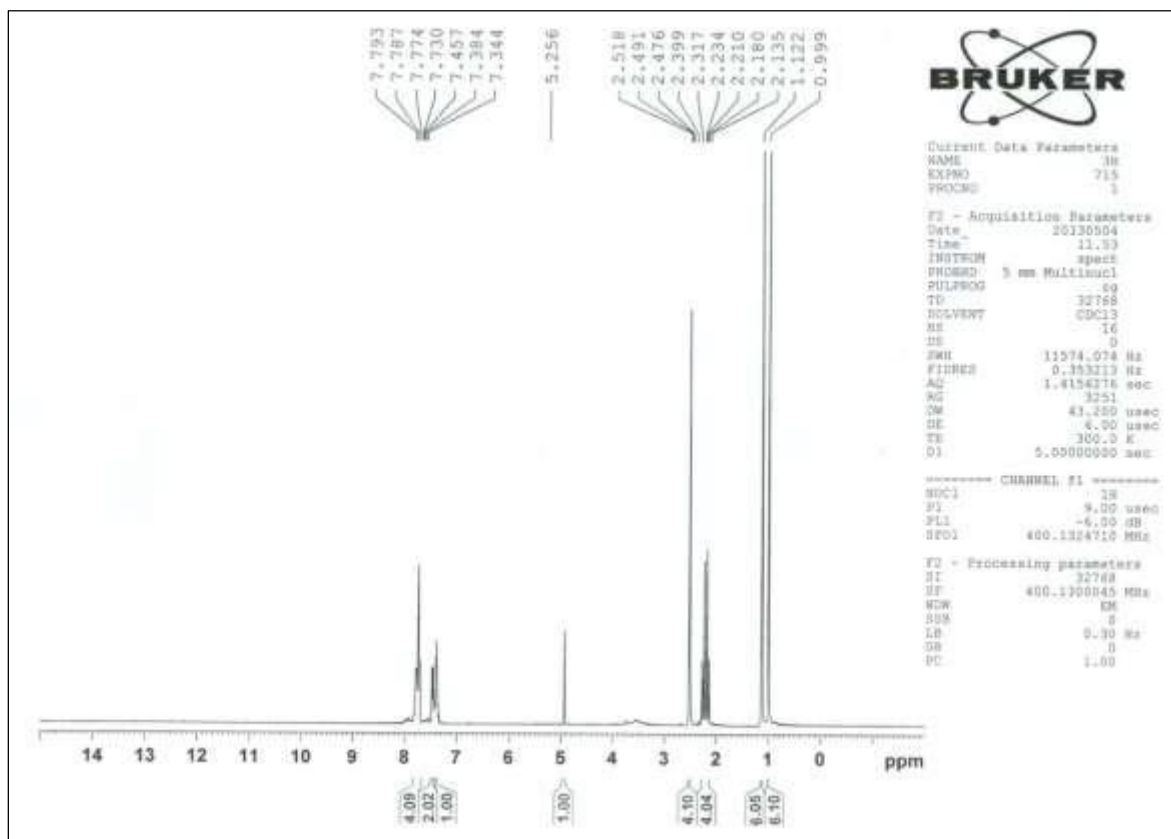


Fig. 50. ^1H NMR of 14m.

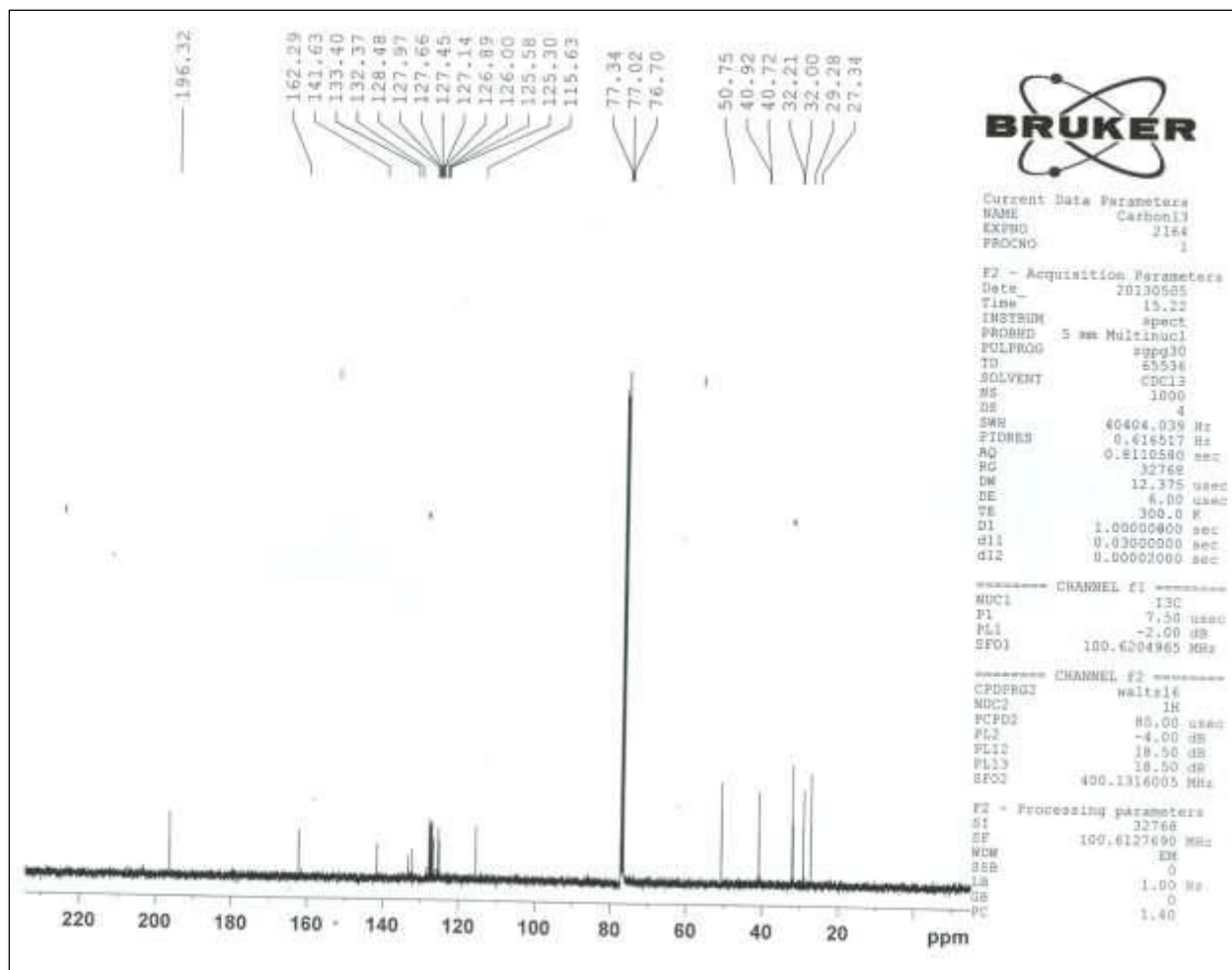
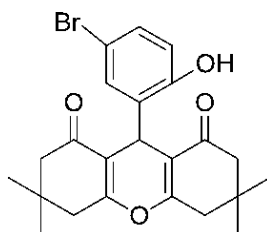


Fig. 51. ^{13}C NMR of 14m.

9-(5-bromo-2-hydroxyphenyl)-3,4,6,7-tetrahydro-3,3,6,6-tetramethyl-2H-xanthene-1,8(5H,9H)-dione (14n)



M. P.: 268-271 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3000, 1660, 1510, 1415, 1356, 1340, 912. ^1H NMR (500 MHz, CDCl_3) δ : 0.90 (s, 6H), 0.97 (s, 3H), 1.05 (s, 3H), 2.02-2.06 (d, $J=8.3$ Hz, 2H), 2.23-2.35 (dd, $J=6.3$, 4H), 2.51-2.56 (t, $J=3.9$, 2H), 5.54 (s, 1H), 6.94-6.96 (d, $J=3.4$ Hz, 1H), 7.04 (s, 1H), 7.27-7.29 (d, $J=3.4$ Hz, 2H), 10.56 (OH, 1H). ^{13}C NMR (125 MHz, CDCl_3) δ : 26.99, 28.41, 30.02, 32.48, 43.70, 51.22, 100.39, 111.30, 116.32, 118.61, 129.15, 130.56, 131.44, 149.83, 165.34, 196.05, 196.50. Anal. Calcd for $\text{C}_{24}\text{H}_{28}\text{O}_3$: C, 79.09; H, 7.74; O, 13.17; Found: C, 79.18; H, 7.89; O, 13.31.

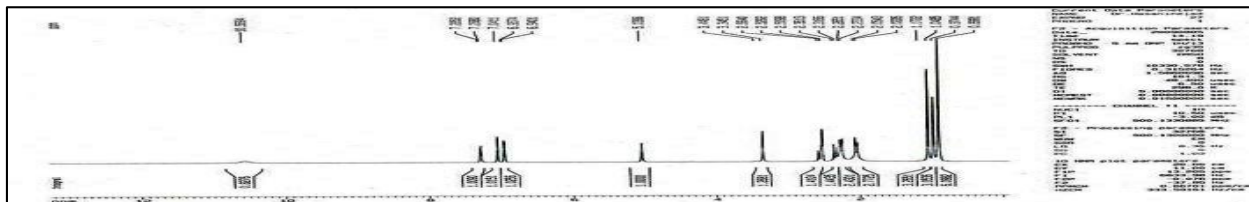


Fig. 52. ^1H NMR of **14n**.

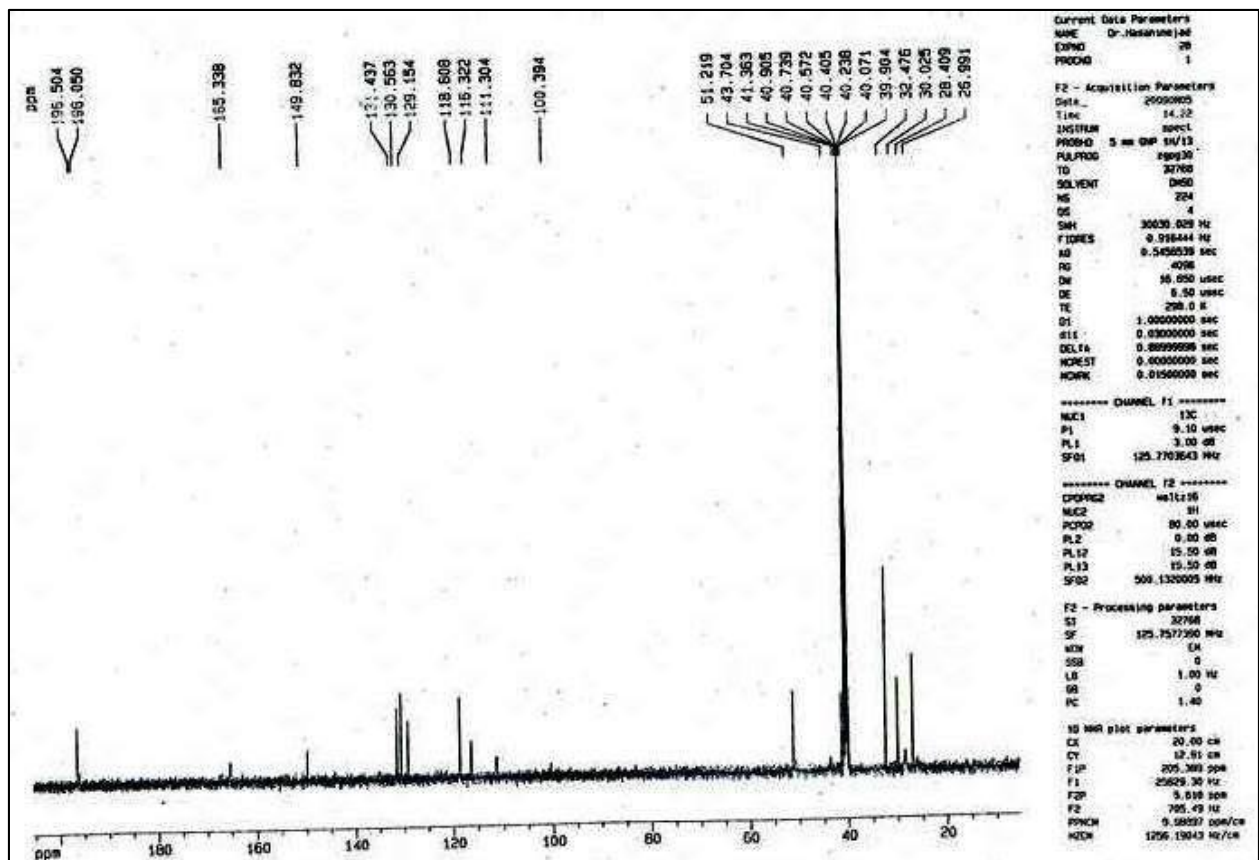
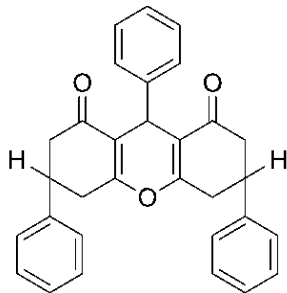


Fig. 53. ¹³C NMR of 14n.

3,4,6,7-tetrahydro-3,6,9-triphenyl-2H-xanthene-1,8(5H,9H)-dione (15a)



M. P.: 196-198 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3031, 1667, 1365, 1188, 1134, 995, 694. ^1H NMR (400 MHz, CDCl_3) δ : 2.57-2.75 (m, 4H), 2.80-3.01 (m, 4H), 3.30-3.55 (d, $J=3.9$ Hz, 2H), 4.92-4.95 (d, $J=3.5$ Hz, 1H), 7.18-7.45 (m, Ar-H, 15H). ^{13}C NMR (100 MHz, CDCl_3) δ : 31.98, 34.74, 38.26, 38.82, 43.89, 116.73, 126.65, 127.25, 128.39, 128.89, 142.11, 143.859, 144.25, 162.94, 195.59. Anal. Calcd for $\text{C}_{31}\text{H}_{26}\text{O}_3$: C, 83.38; H, 5.87; O, 10.75; Found: C, 83.47; H, 5.95; O, 10.83.

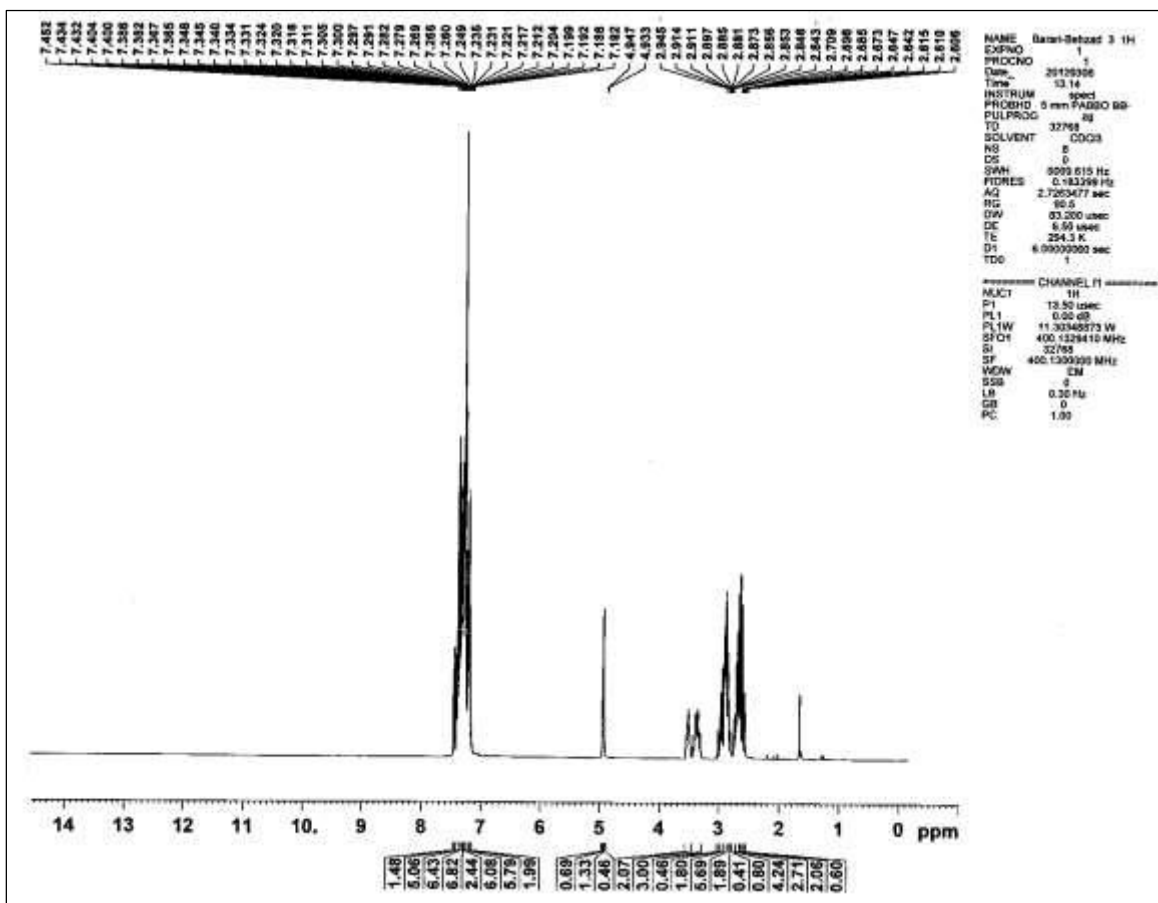


Fig. 54. ^1H NMR of 15a.

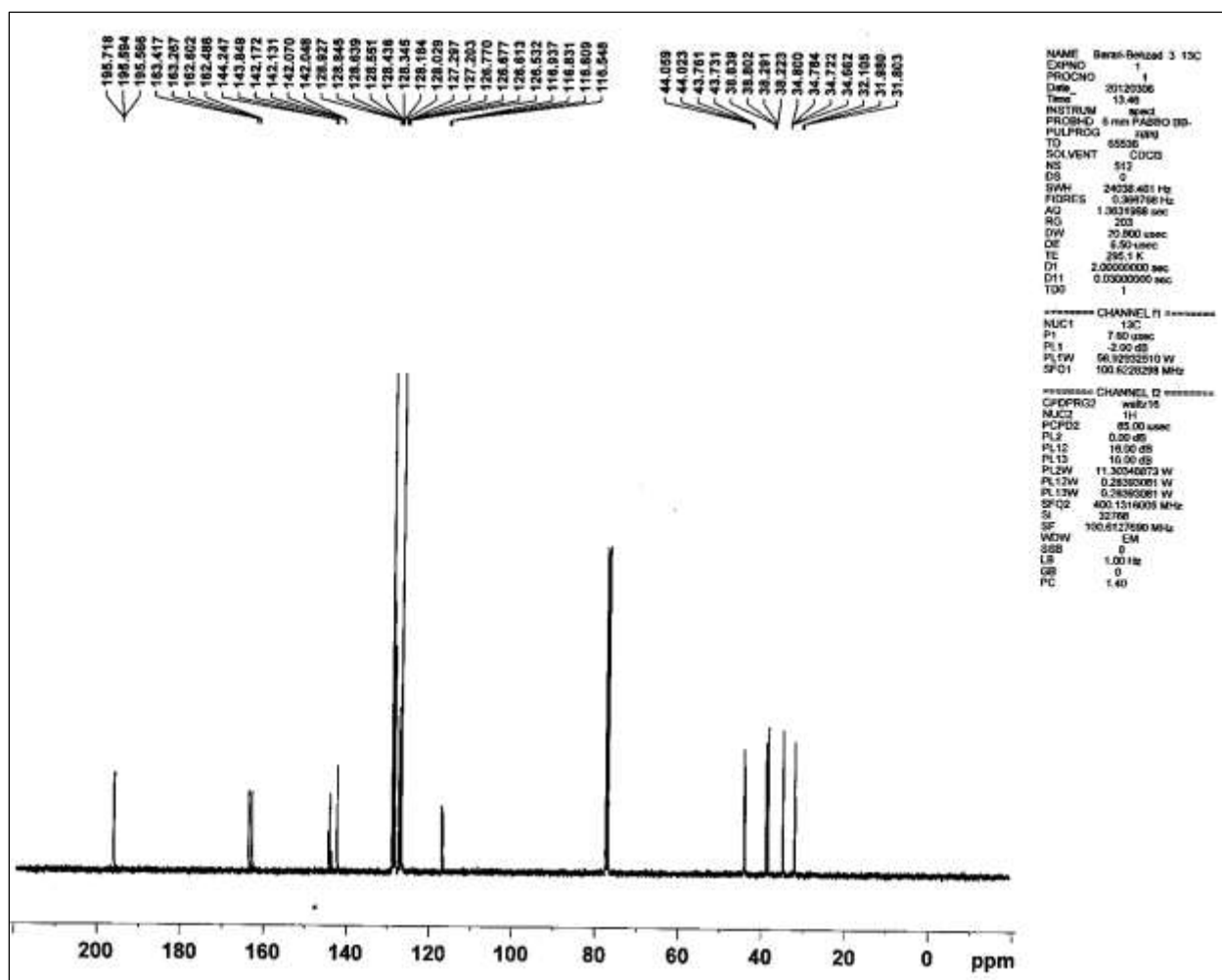
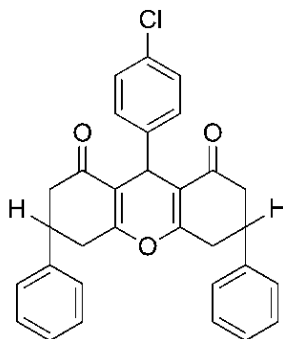


Fig. 55. ^{13}C NMR of 15a.

9-(4-chlorophenyl)-3,4,6,7-tetrahydro-3,6-diphenyl-2H-xanthene-1,8(5H,9H)-dione (15b)



M. P.: 234-236 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3031, 1666, 1357, 1188, 1134, 995, 756, 694. ^1H NMR (400 MHz, CDCl_3) δ : 2.56-2.76 (m, 4H), 2.81-3.00 (m, 4H), 3.30-3.54 (d, $J=3.9$ Hz, 2H), 4.88-4.89 (d, $J=2.75$ Hz, 1H), 7.17-7.40 (m, Ar-H, 14H). ^{13}C NMR (100 MHz, CDCl_3) δ : 31.59, 34.69, 38.28, 38.61, 43.79, 116.33, 126.70, 127.31, 128.30, 128.91, 129.96, 132.29, 141.92, 142.36, 142.77, 195.61. Anal. Calcd. for $\text{C}_{31}\text{H}_{25}\text{ClO}_3$: C, 77.41; H, 5.24; Cl, 7.37; O, 9.98; Found: C, 77.50; H, 5.33; Cl, 7.45; O, 10.08.

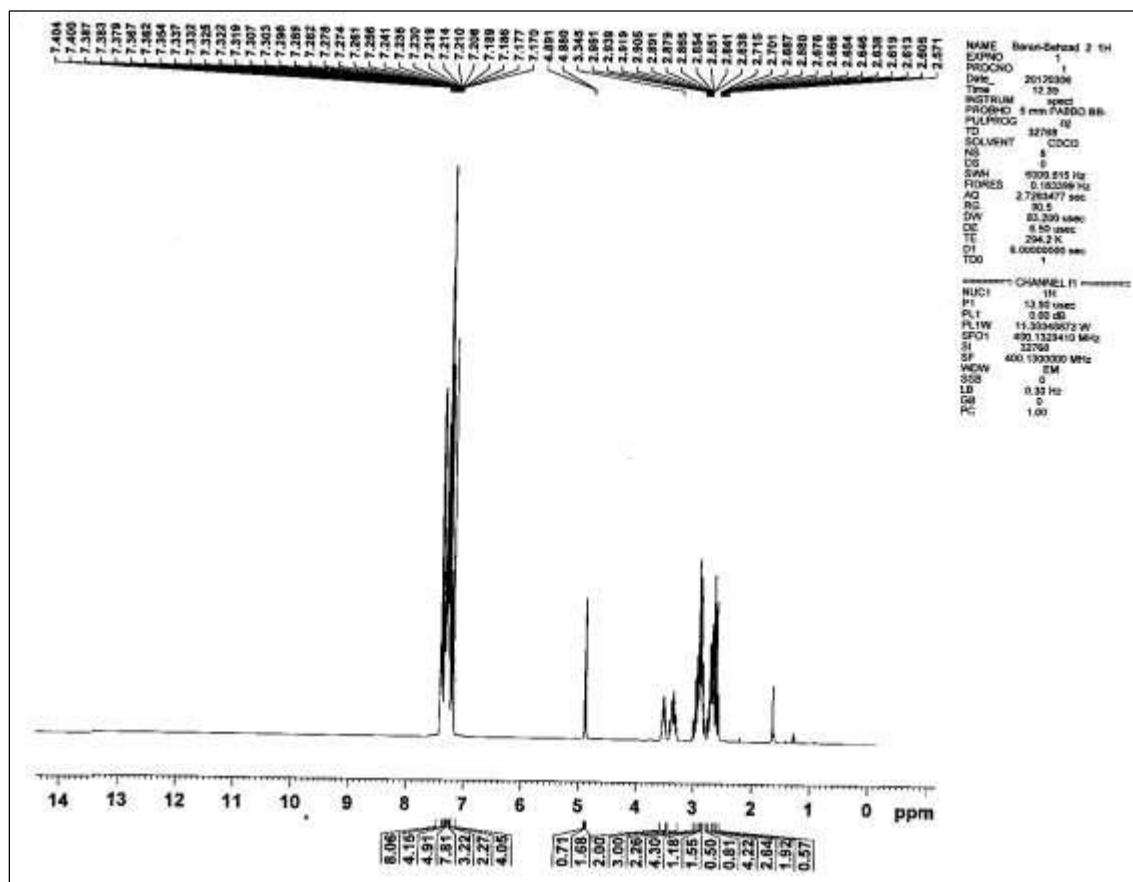


Fig. 56. ^1H NMR of 15b.

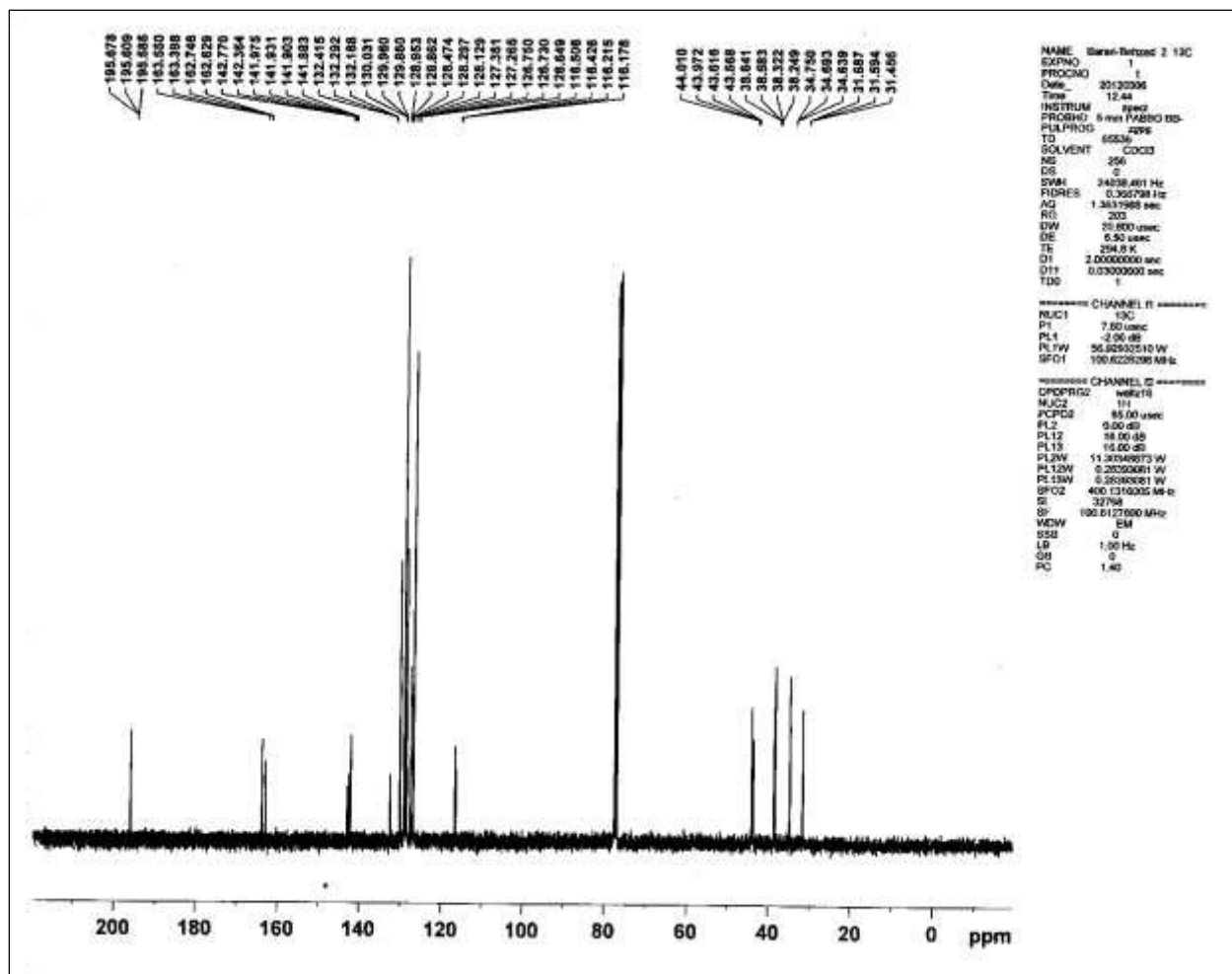
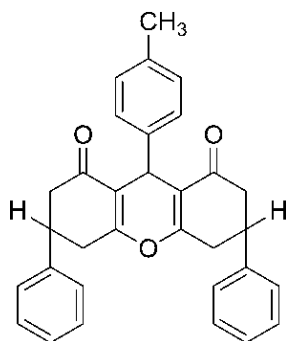


Fig. 57. ^{13}C NMR of 15b.

9-(4-Methylphenyl)-3,4,6,7-tetrahydro-3,6-diphenyl-2H-xanthene-1,8(5H,9H)-dione (15c)



M. P.: 204-208 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 2939, 1666, 1504, 1358, 1242, 1180, 1134, 1033, 987, 764, 702. ^1H NMR (400 MHz, CDCl_3) δ : 2.56-2.73 (m, 4H), 2.79-2.95 (m, 4H), 3.30-3.53 (d, $J=3.2$ Hz, 2H), 3.88 (s, 3H), 4.86-4.88 (d, $J=3.2$ Hz, 1H), 6.75-6.87 (m, Ar-H, 2H), 7.17-7.40 (m, Ar-H, 12H). ^{13}C NMR (100 MHz, CDCl_3) δ : 31.03, 34.70, 38.29, 38.83, 43.91, 113.56, 116.85, 126.72, 127.12, 128.88, 129.51, 136.39, 142.12, 158.20, 163.14, 195.78. Anal. Calcd. for $\text{C}_{32}\text{H}_{28}\text{O}_3$: C, 83.45; H, 6.13; O, 10.42; Found: C, 83.52; H, 6.20; O, 10.51.

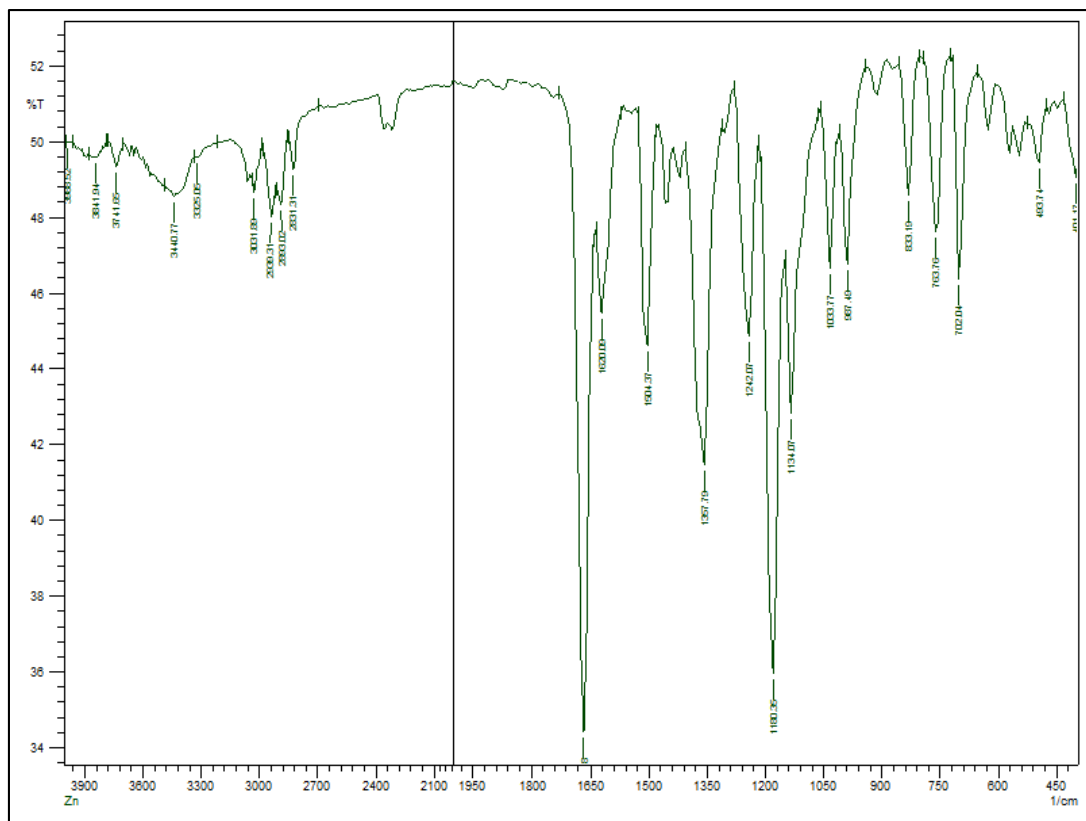


Fig. 58. FT-IR of 15c.

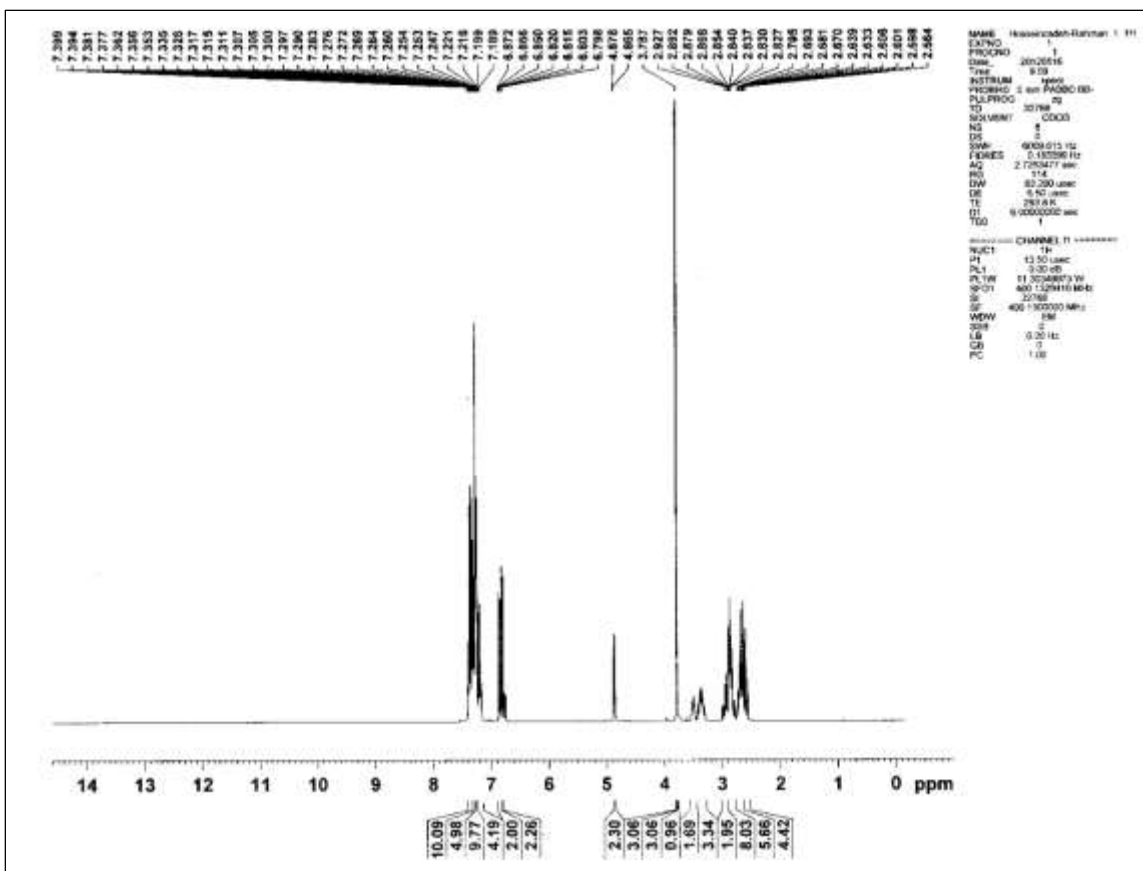


Fig. 59. ^1H NMR of 15c.

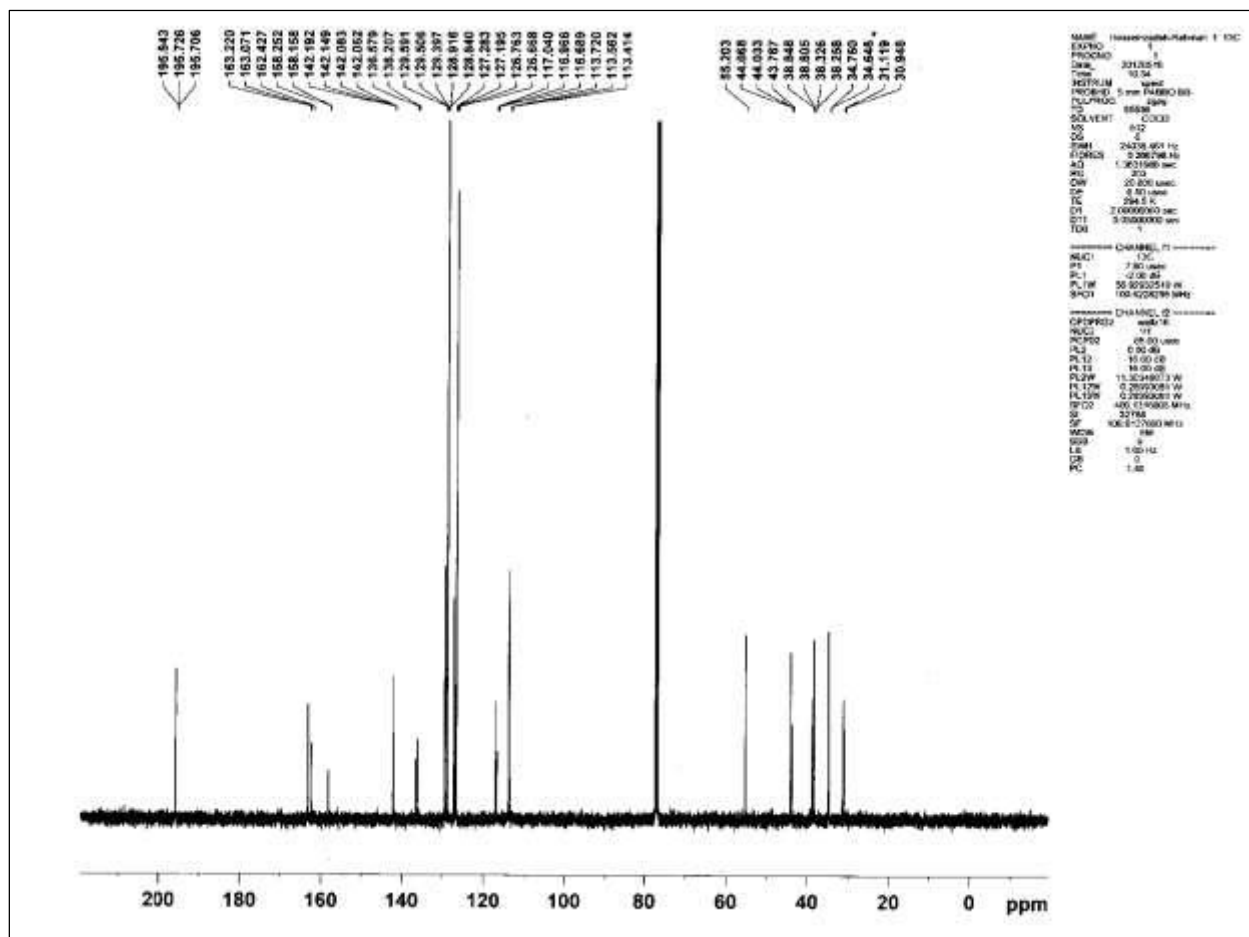
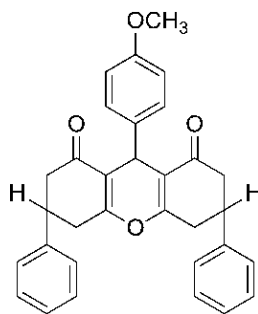


Fig. 60. ^{13}C NMR of 15c.

9-(4-Methoxyphenyl)-3,4,6,7-tetrahydro-3,6-diphenyl-2H-xanthene-1,8(5H,9H)-dione (15d)



M. P.: 212-215 °C; FT-IR (KBr, Cm^{-1}) ν_{max} : 3031, 1666, 1504, 1357, 1242, 1180, 1134, 987, 764, 702. ^1H NMR (400 MHz, CDCl_3) δ : 2.60-2.75 (m, 4H), 2.79-2.99 (m, 4H), 3.32-3.54 (d, $J=3.4$ Hz, 2H), 3.79-3.80 (d, $J=2.5$ Hz, 3H), 4.87-4.88 (d, $J=3$ Hz, 1H), 6.80-6.88 (m, Ar-H, 4H), 7.19-7.40 (m, Ar-H, 10H). ^{13}C NMR (100 MHz, CDCl_3) δ : 31.04, 34.71, 38.30, 38.81, 43.91, 113.64, 116.85, 126.72, 127.24, 128.88, 129.45, 136.40, 142.12, 158.21, 163.10, 195.77. Anal. Calcd for $\text{C}_{32}\text{H}_{28}\text{O}_4$: C, 80.65; H, 5.92; O, 13.43; Found: C, 80.74; H, 6.02; O, 13.56.

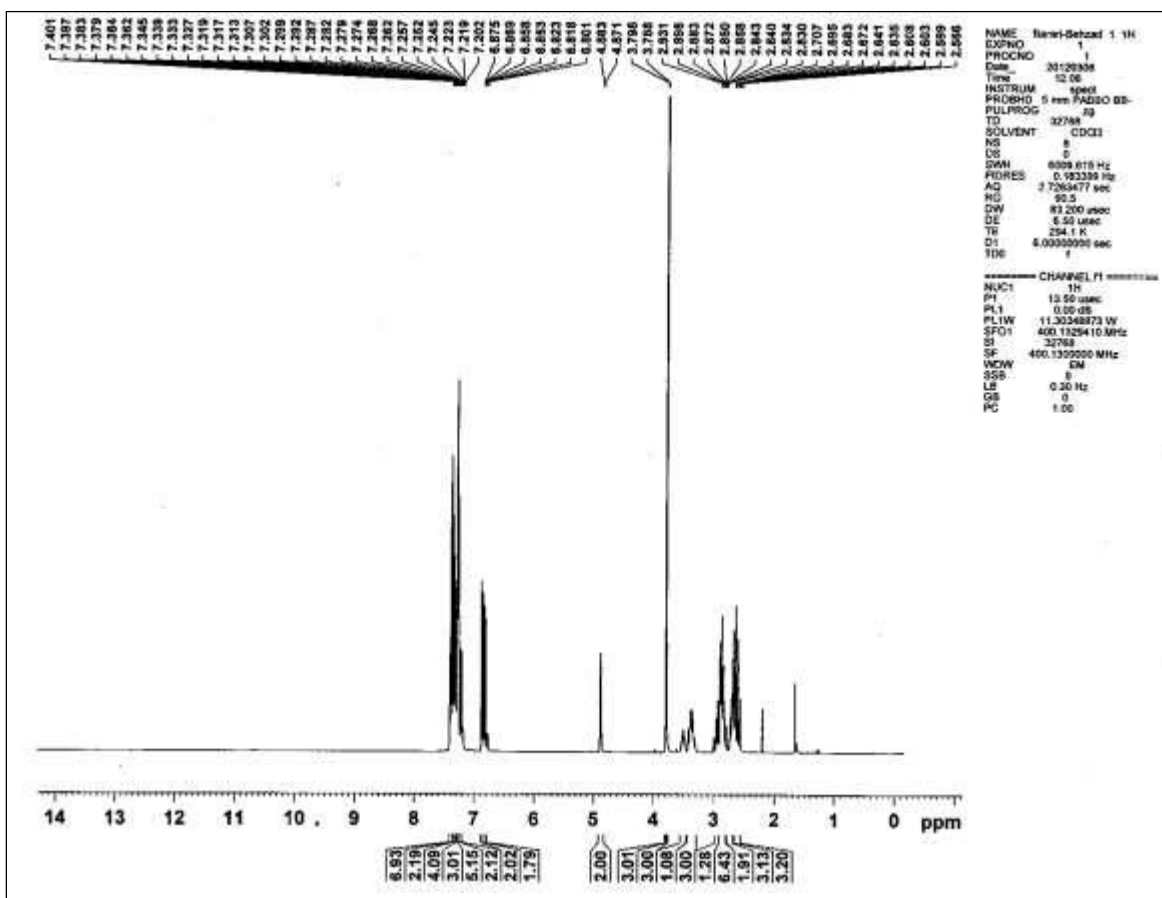


Fig. 61. ^1H NMR of 15d.

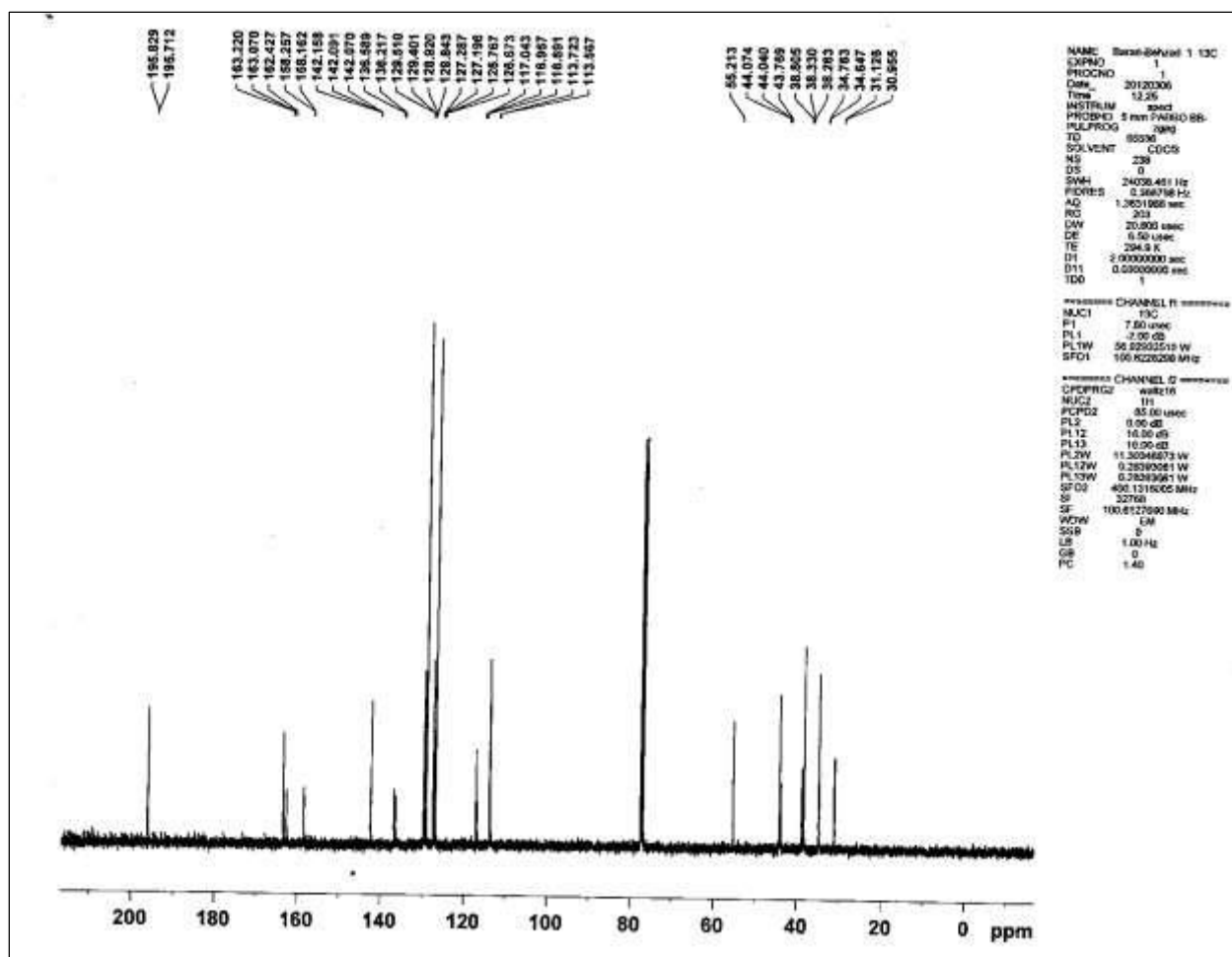


Fig. 62. ^{13}C NMR of 15d.