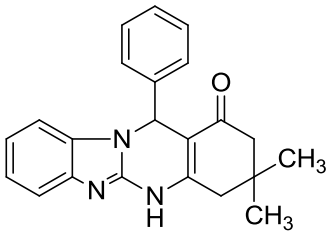
Electronic Supplementary Material (ESI) for RSC Advances.

Supporting information

A new type of SO_3H-functionalized magnetic-titania as a robust magnetically-recoverable solid acid nanocatalyst for multi-component reactions

Elham Tabrizian and Ali Amoozadeh*

3,3-dimethyl-12-phenyl-3,4,5,12-tetrahydrobenzo[4,5]imidazo[2,1-b]quinazolin-1(2H)-one



M. P.: 318-320 °C. FT-IR (KBr) ν cm^{-1} = 1569, 1647, 2956, 3367. ^{1}H NMR (300 MHz, DMSO-d^6): δ 1.06 (s, 3H, CH_3), 1.17 (s, 3H, CH_3), 2.23-2.37 (dd, J= 16.43, 30.48 Hz, 2H, CH_2), 2.67 (s, 2H, CH_2), 6.49 (s, 1H, CH), 7.09-7.65 (m, 9H, Ar-H), 10.87 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d^6) δ: 26.73, 26.43, 31.79, 35.92, 49.99, 58.15, 112.56, 119.77, 125.57, 125.67, 126.19, 127.44, 127.66, 128.13, 132.02, 132.87, 142.05, 158.48, 158.53, 162.57, 195.73.

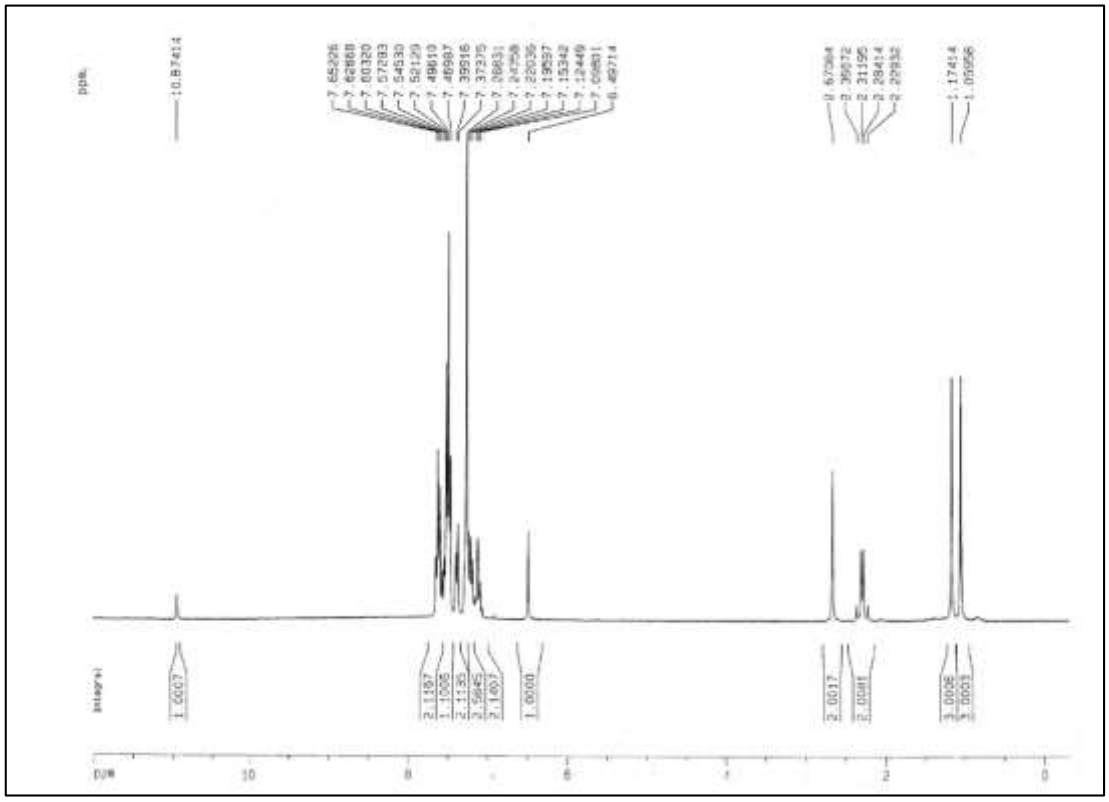
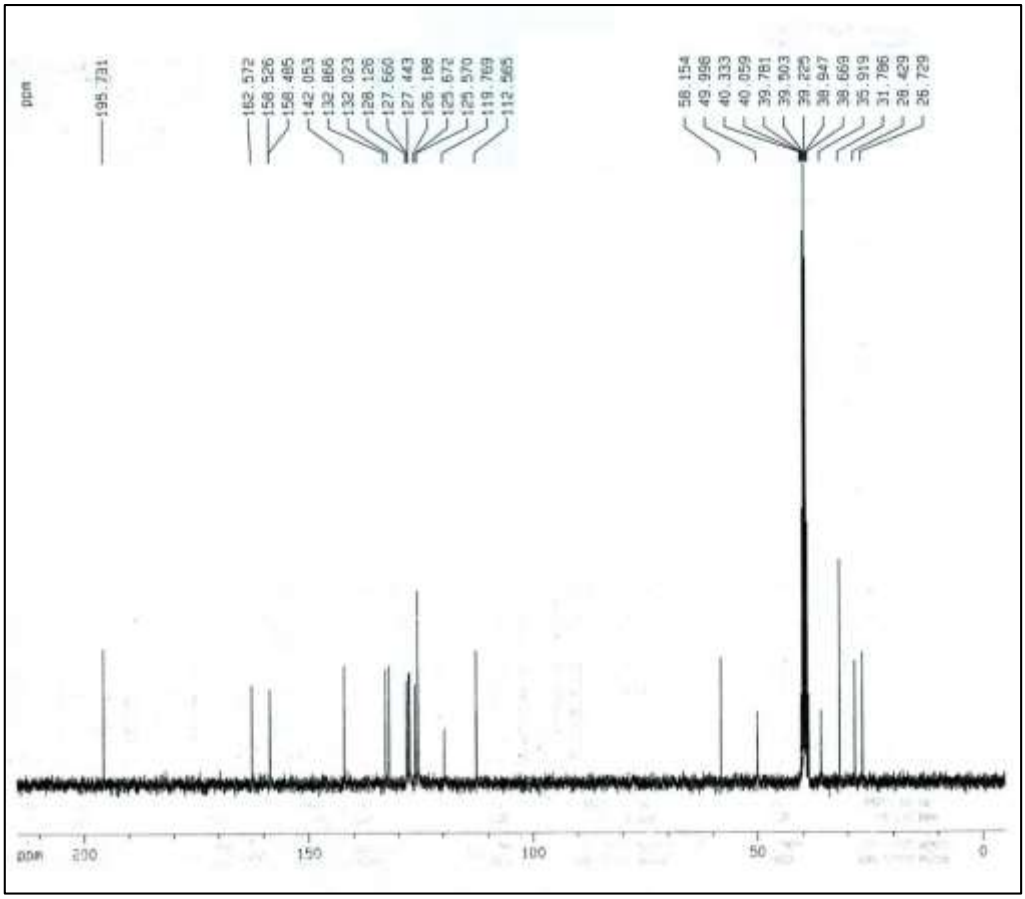


Fig. 1. ^{1}H NMR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132.023
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112.565
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49.996
40.333
40.059
39.781
39.503
39.225
38.947
38.669
35.919
31.786
28.429
26.729
ppm
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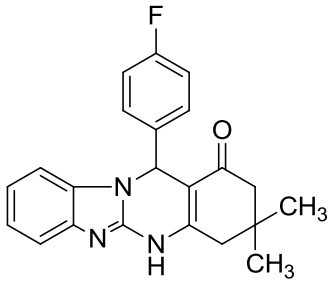
150
100
50
0

Fig. 2. ^{13}C NMR

12-(4-fluorophenyl)-3,3-dimethyl-3,4,5,12-tetrahydrobenzo[4,5]imidazo[2,1-b]quinazolin-1(2H)-one



M. P.: 325-326 °C. FT-IR (KBr) ν cm^{-1} = 1565, 1652, 2958, 3361. ^{1}H NMR (300 MHz, DMSO-d^{6}): δ 1.07 (s, 3H, CH_3), 1.18 (s, 3H, CH_3), 2.25-2.36 (dd, J= 11.23, 22.56 Hz, 2H, CH_2), 2.77 (s, 2H, CH_2), 6.48 (s, 1H, CH), 7.10-7.76 (m, 8H, Ar-H), 10.91 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d^{6}) δ: 26.83, 28.53, 31.87, 35.94, 50.20, 58.18, 112.36, 119.56, 125.47, 125.67, 126.28, 127.44, 127.66, 128.23, 132.42, 132.94, 142.15, 158.59, 158.73, 162.67, 196.01.

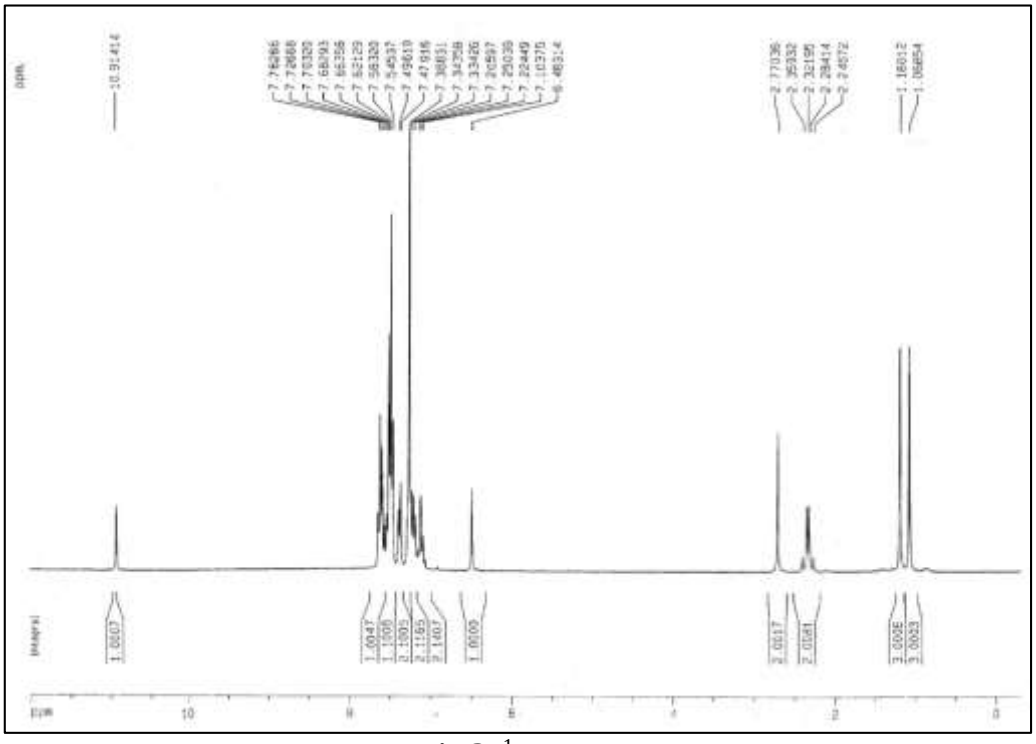
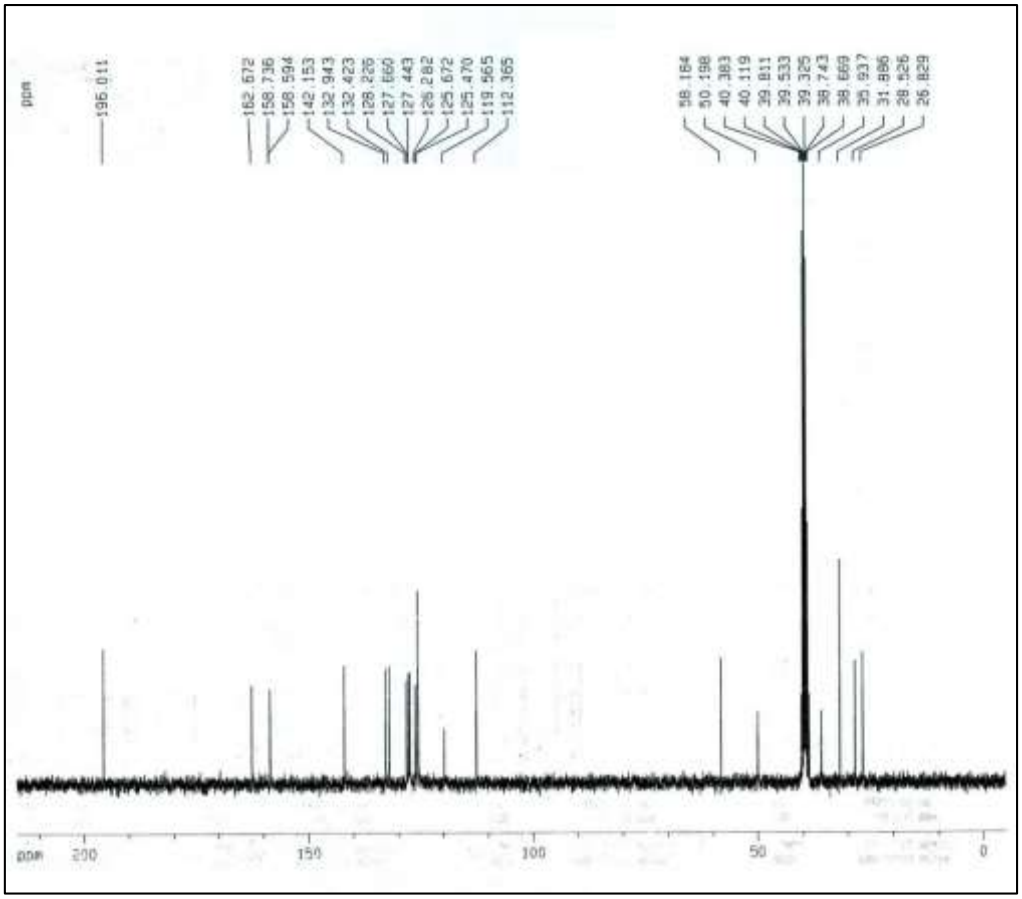


Fig 3. ^{1}H NMR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58.164
50.198
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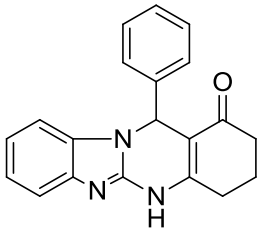
39.811
39.533
39.325
38.743
38.669
35.937
31.886
28.526
26.829
ppm
200
150
100
50
0

Fig 4. ^{13}C NMR

12-phenyl-3,4,5,12-tetrahydrobenzo[4,5]imidazo[2,1-b]quinazolin-1(2H)-one



M. P.: 310-312 °C. FT-IR (KBr) ν cm^{-1} = 1560, 1666, 2968, 3370. ^{1}H NMR (300 MHz, DMSO-d^6): δ 1.86-1.93 (m, 2H, CH_3), 2.23-2.29 (m, 2H, CH_3), 2.59-2.61 (d, J= 5.05 Hz, 2H, CH_2), 6.49 (s, 1H, CH), 7.09-7.90 (m, 9H, Ar-H), 11.11 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d^6) δ: 18.55, 19.73, 26.39, 30.14, 35.97, 49.99, 58.15, 112.64, 119.67, 125.56, 125.67, 126.28, 127.34, 127.54, 128.22, 132.13, 132.89, 142.08, 159.05, 159.09, 162.74, 196.04.

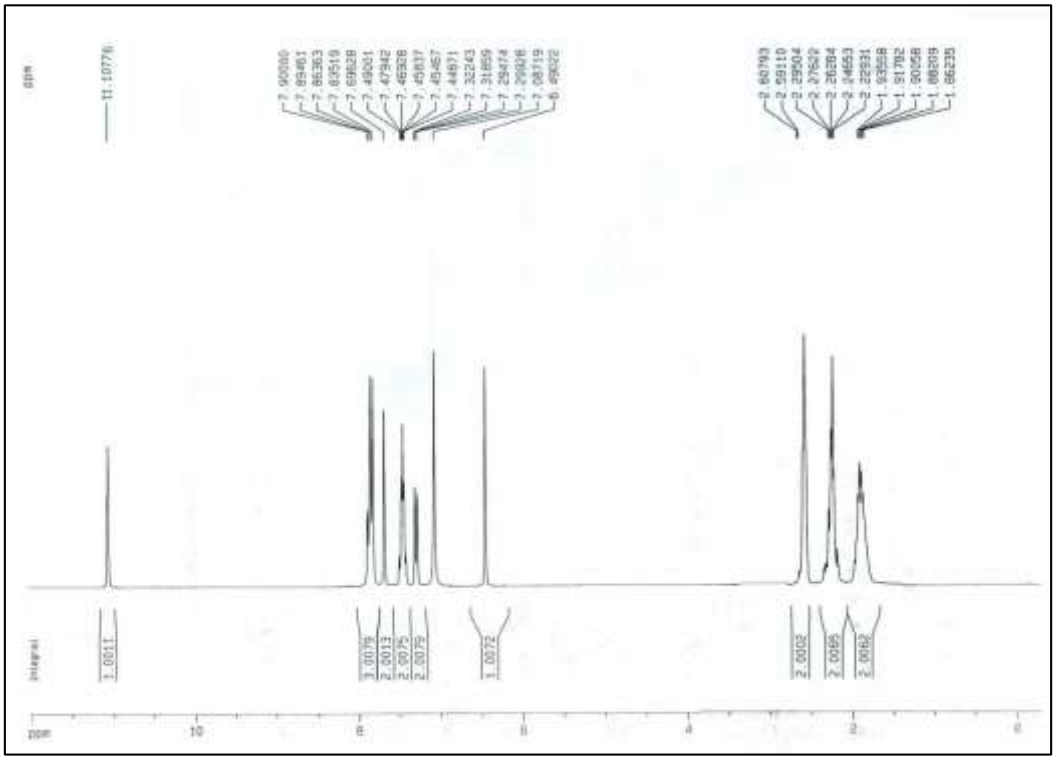
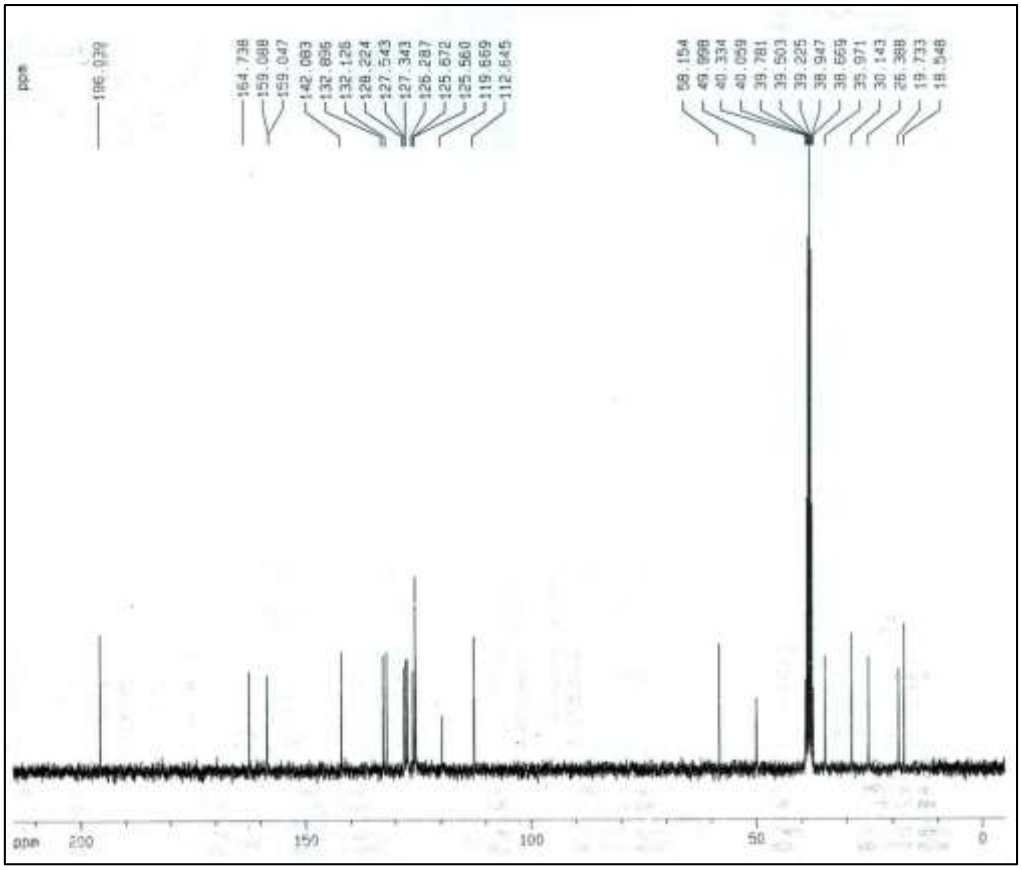


Fig 5. ^{1}H NMR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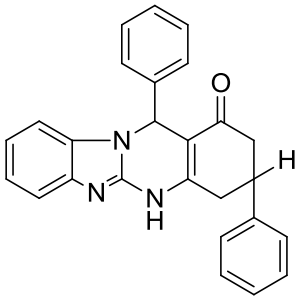
119.669
112.645
58.154
49.998
40.334
40.059
39.781
39.503
39.225
38.947
38.669
35.971
30.143
26.388
19.733
18.548
ppm
200
150
100
50
0

Fig 6. ^{13}C NMR

3,12-diphenyl-3,4,5,12-tetrahydrobenzo[4,5]imidazo[2,1-b]quinazolin-1(2H)-one



M. P: 332-335 °C. FT-IR (KBr) ν cm^{-1} = 1558, 1662, 2960, 3364. ^{1}H NMR (300 MHz, DMSO-d^6): δ 2.87-3.06 (m, 2H, CH_2), 3.23-3.48 (t, J= 5.01, 2H, CH_2), 3.51 (s, 1H, -CH), 6.41 (s, 1H, -CH), 6.42-7.35 (m, 14H, Ar-H), 10.93 (s, 1H, NH). ^{13}C NMR (75 MHz, DMSO-d^6) δ: 22.90, 23.43, 25.92, 33.52, 54.33, 107.01, 109.57, 116.78, 120.25, 121.54, 126.63, 126.89, 127.24, 127.86, 128.20, 140.95, 142.72, 159.07, 159.09, 162.81, 191.72.

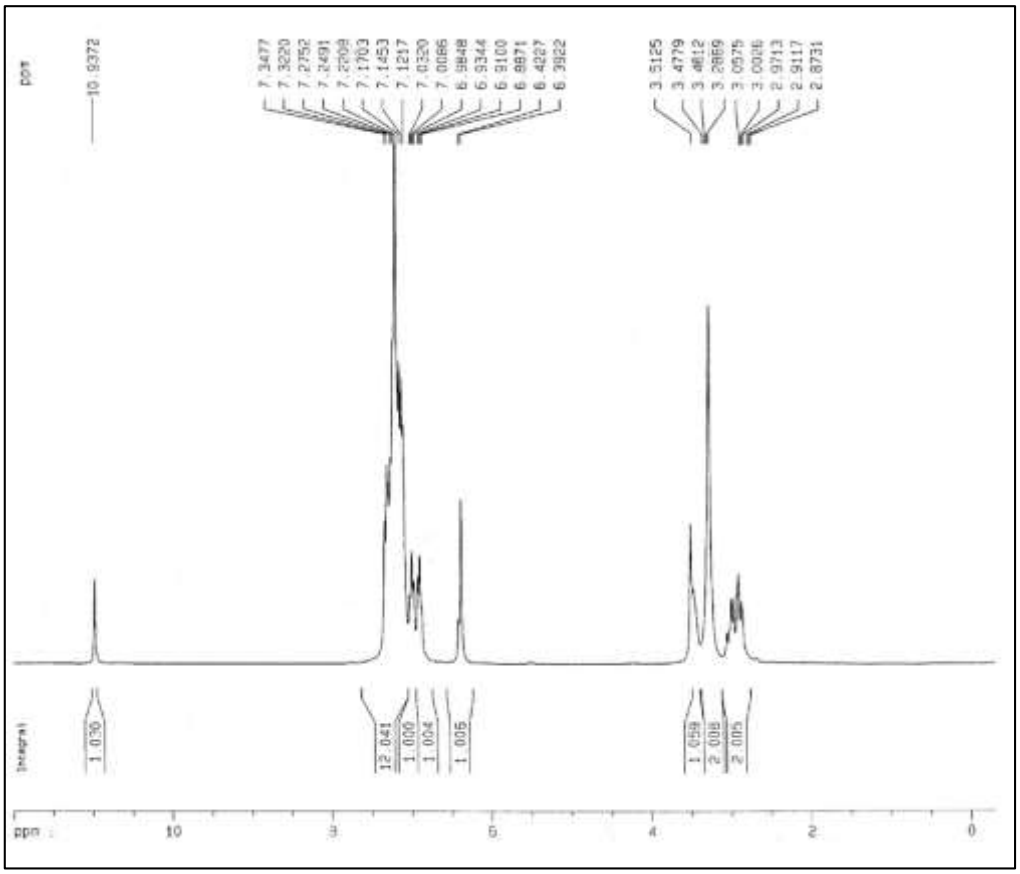
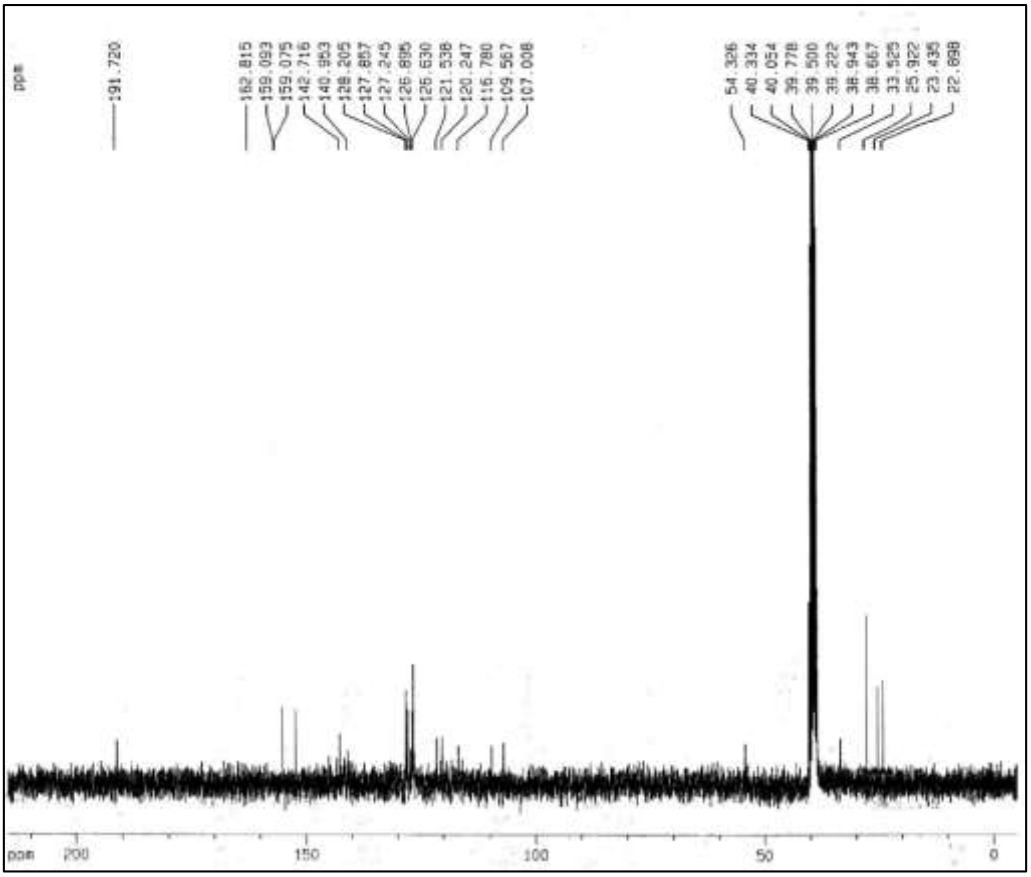


Fig 7. ^{1}H NMR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127.245
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116.780
109.567
107.008
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40.054
39.778
39.500
39.222
38.943
38.667
33.525
25.922
23.435
22.698
ppm
200
150
100
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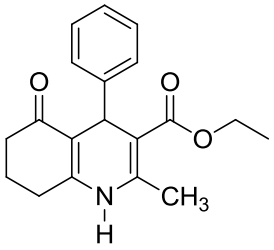
0

Fig 8. ^{13}C NMR

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



M.p. 240-241 °C FT-IR (KBr) 3296 (NH), 1641 (C=O) (acid), 1608 (C=O) (ketone), 1488 (OC_2H_5) (ester) cm^{-1}; δH/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)).

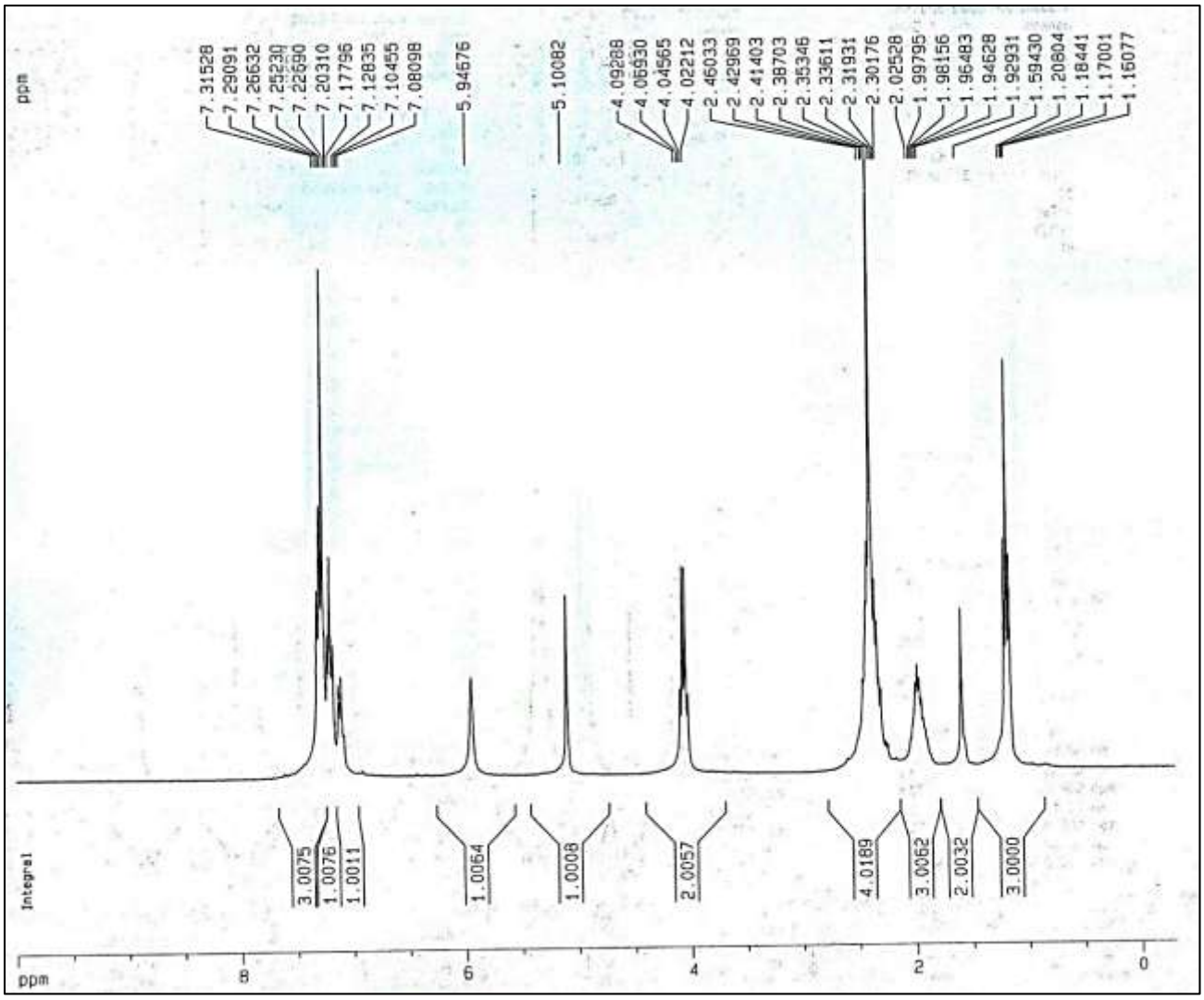
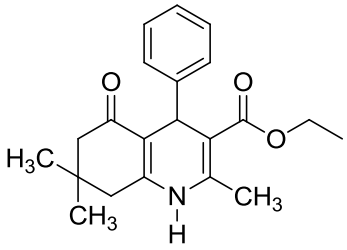


Fig. 9. ^{1}HNMR

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



M.p. 200-202 °C, FT-IR (KBr) 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.

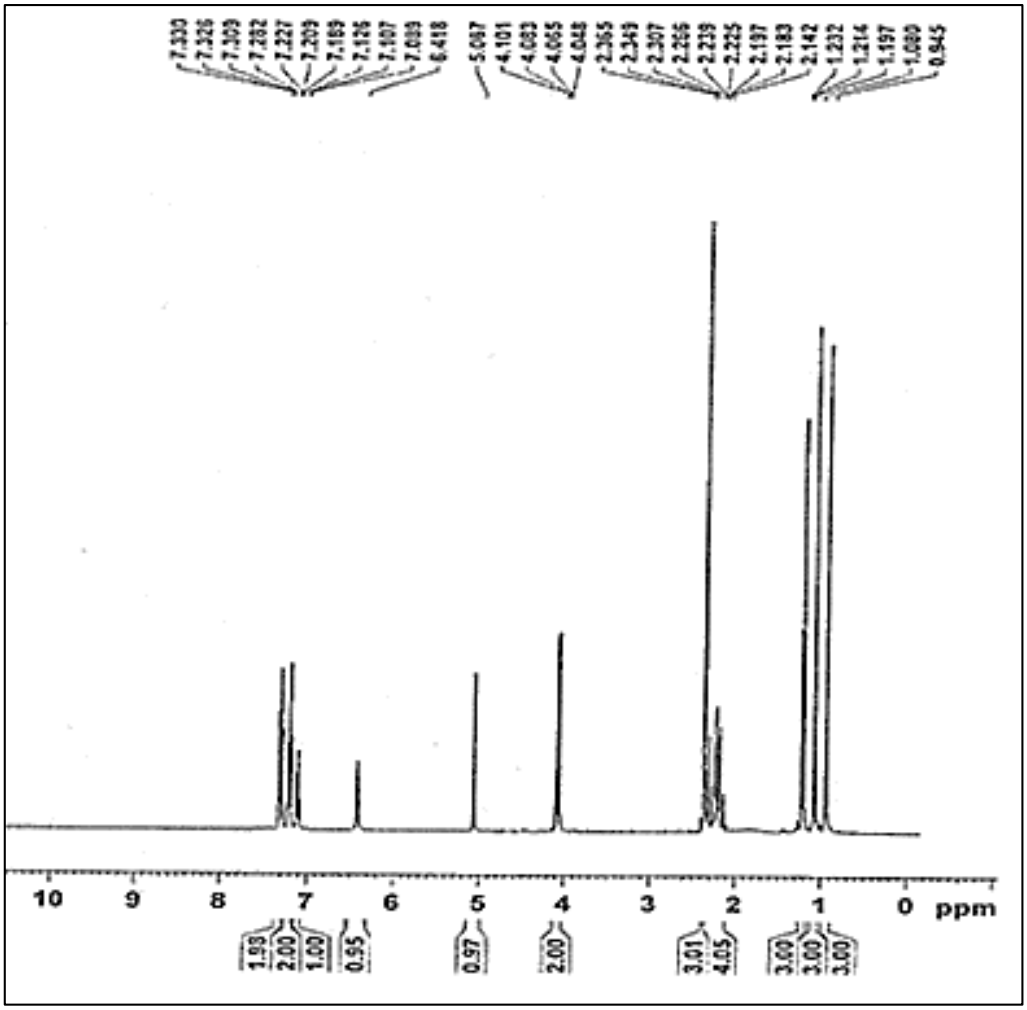


Fig. 10. ^{1}H NMR

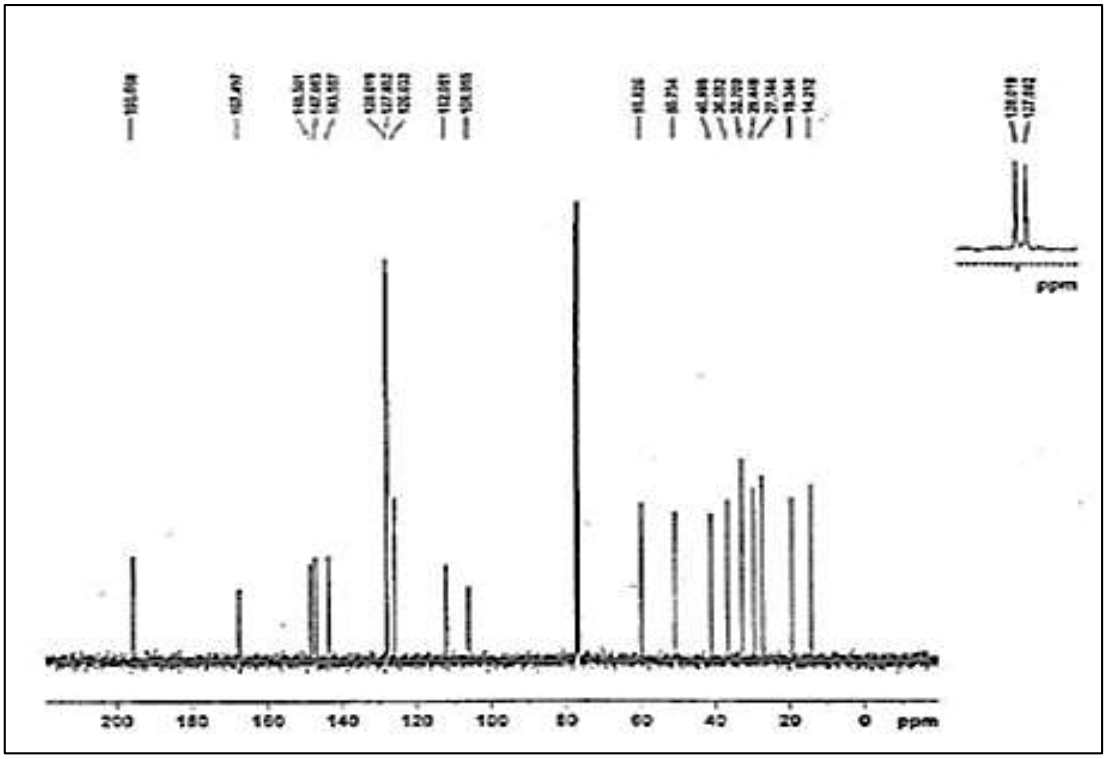
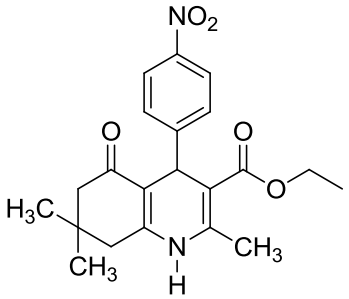


Fig. 11. ^{13}C NMR

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



M.p. 209-211 °C, FT-IR (KBr) 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.

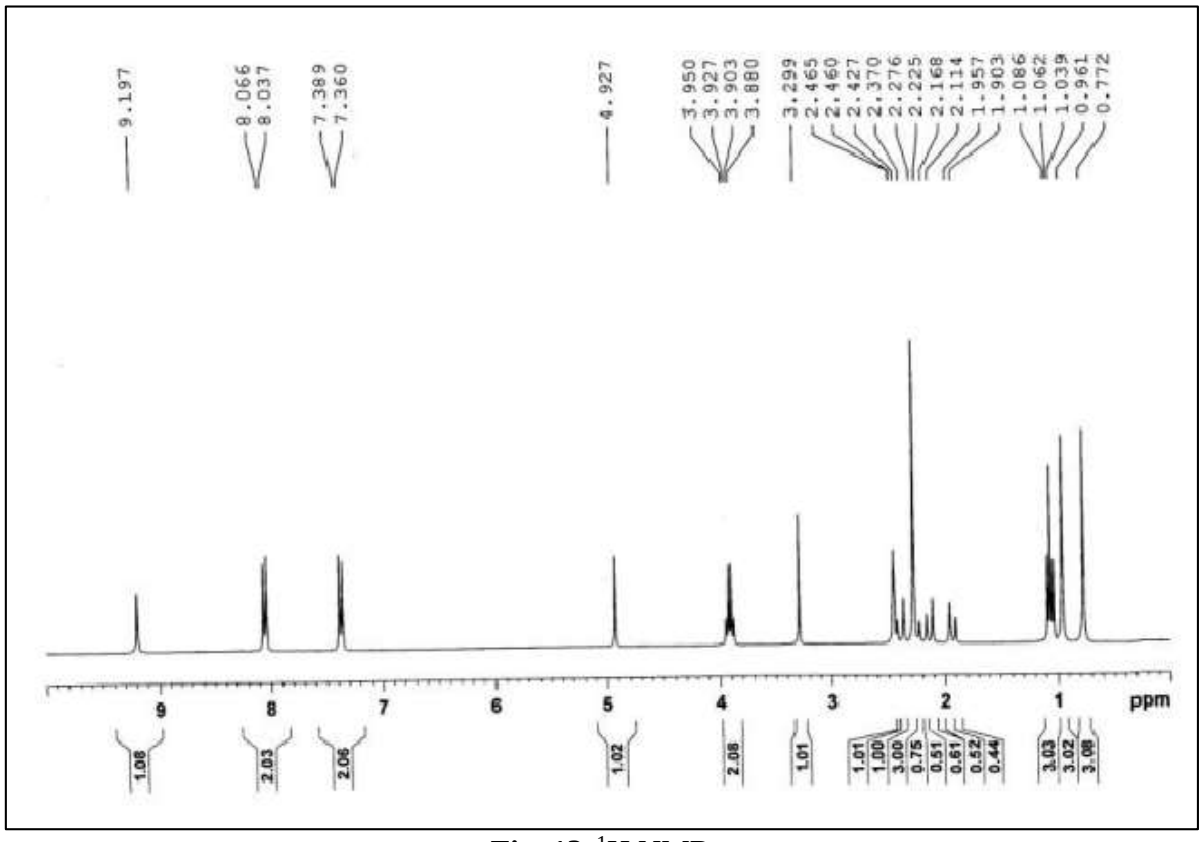


Fig. 12. ^{1}H NMR

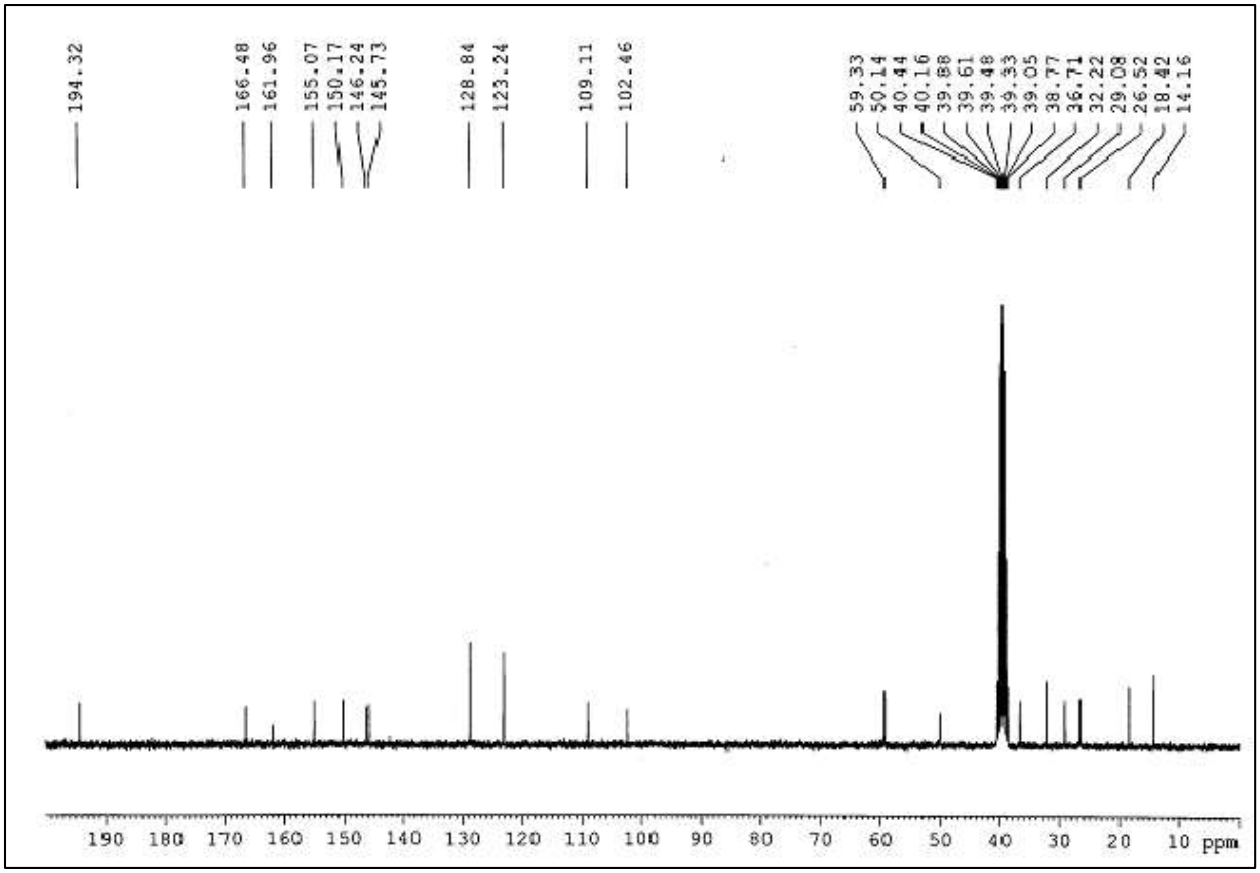
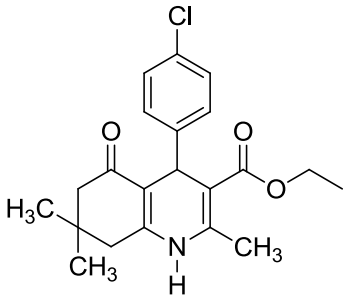


Fig. 13. ^{13}C NMR

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



M.p. 245-247 °C, FT-IR (KBr) 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}; δH/ 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, CH_3-CH_2-C=O), 2.14-2.36 (m, 4H, C-CH_2-C=), 2.39 (s, 3H, N-C-CH_3), 4.04-4.09 (q, J= 7.2 Hz, 2H, -O-CH_2-CH_3), 5.04 (s, 1H, CH-Ph), 6.12 (s, 1H, NH), 7.16-7.28 (m, 4H, CH (Ph)); δC /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.

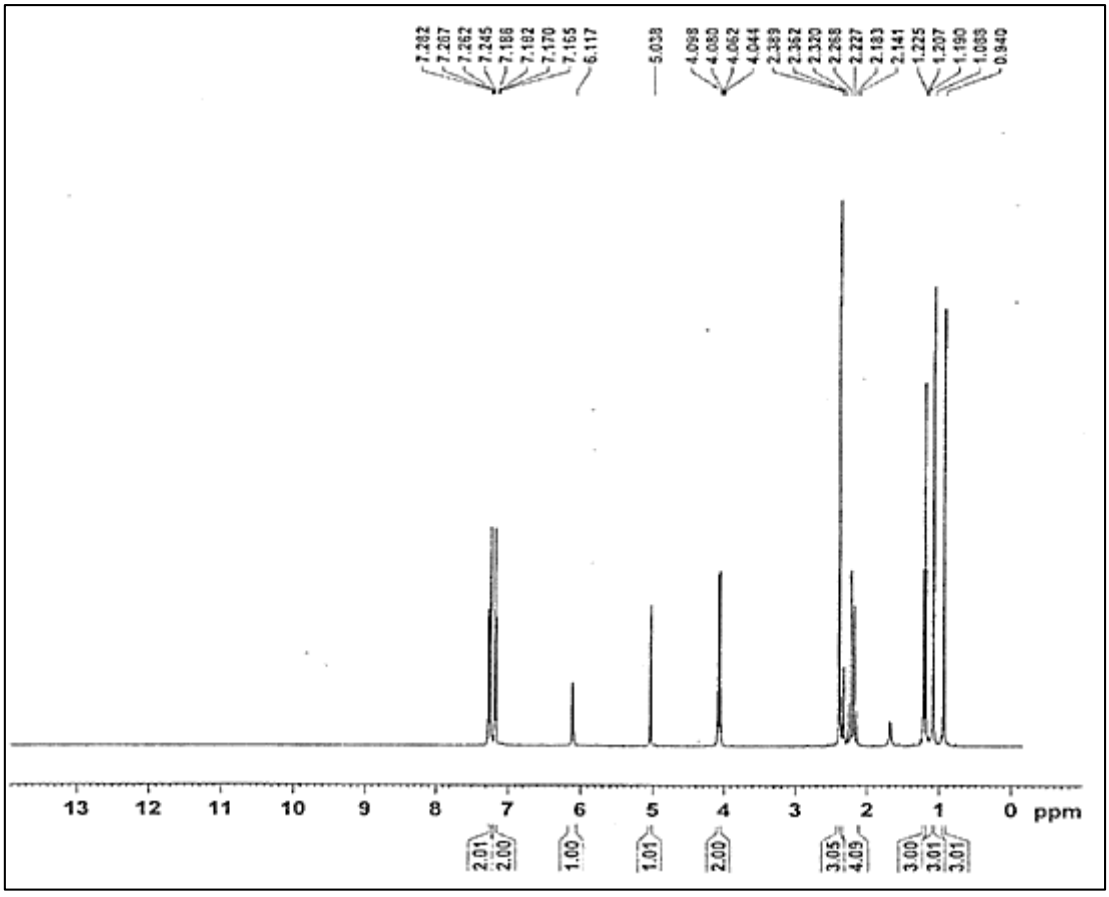


Fig. 14. ^{1}H NMR

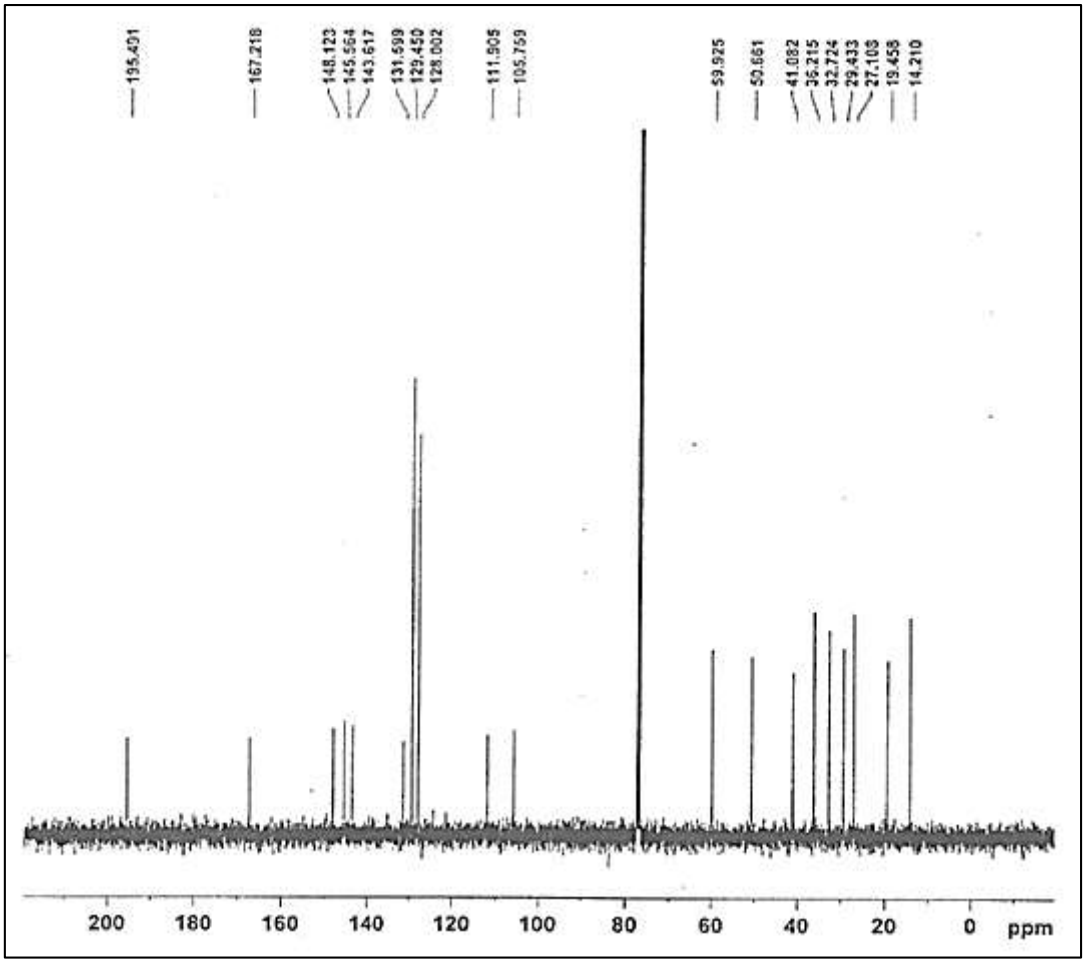
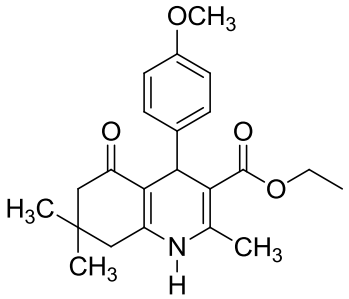


Fig. 15. ^{13}C NMR

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



M.p. 256-258 °C, FT-IR (KBr) 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, CH_3-CH_2-C=O), 2.15-2.37 (m, 4H, C-CH_2-C=), 2.39 (s, 3H, N-C-CH_3), 3.75 (s, 3H, H_3C-O-Ph), 4.05-4.10 (q, J= 7.2 Hz, 2H, -O-CH_2-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.

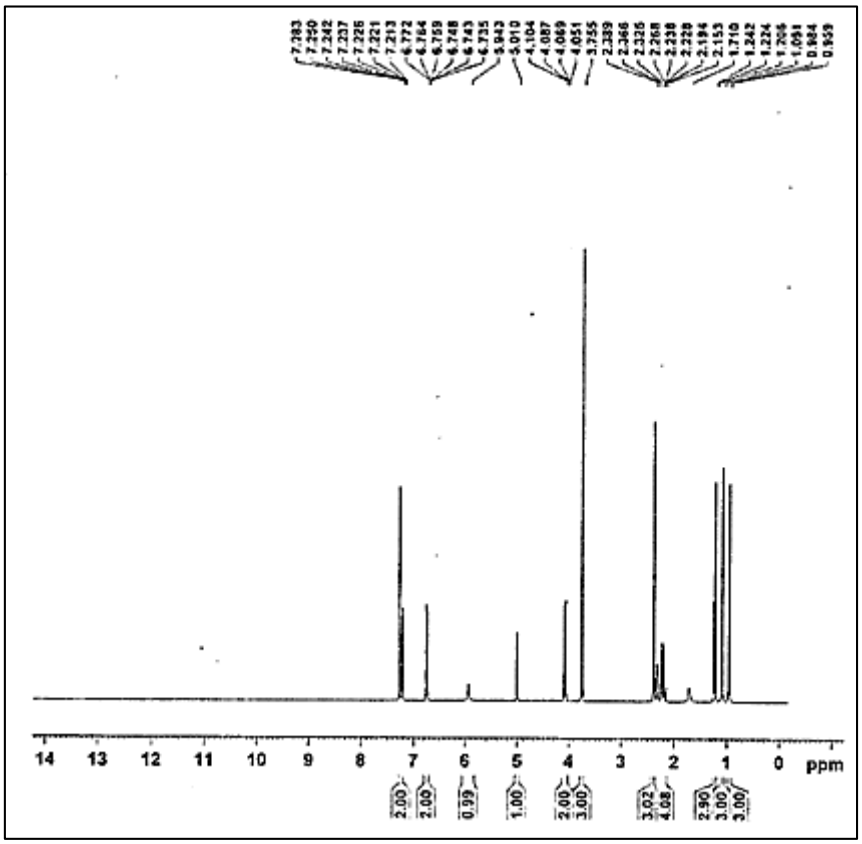


Fig. 16. ^{1}H NMR

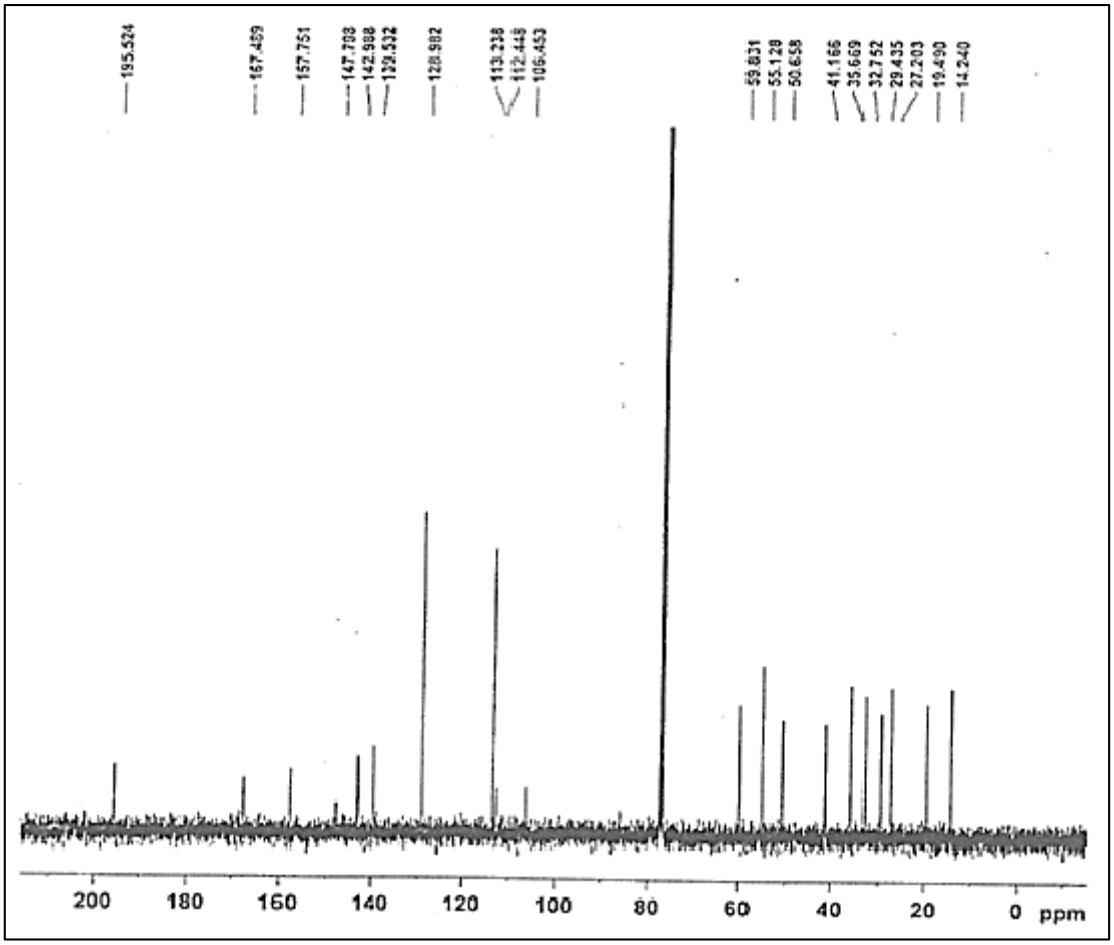
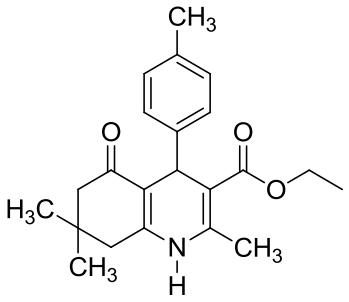


Fig. 17. ^{13}C NMR

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



M.p. 284-285 °C, FT-IR (KBr) 3275 and 3207 (NH), 3085 and 2963 (CH), 1700 (C=O) (acid), 1610 (C=O) (ketone), 1494 (OC_2H_5) (ester), 1222 (OCH3) (ether) cm–1; δH/ 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, CH_3-CH_2-C=O), 2.14-2.24 (m, 3H, C-CH_2-C=), 2.27 (s, 3H, N-C-CH_3), 2.29-2.33 (t, J= 7.6 Hz, 1H, C-CH_2-C=), 2.36 (s, 3H, H_3C-Ph), 4.05-4.10 (m, 2H, -O-CH_2-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); δC /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.

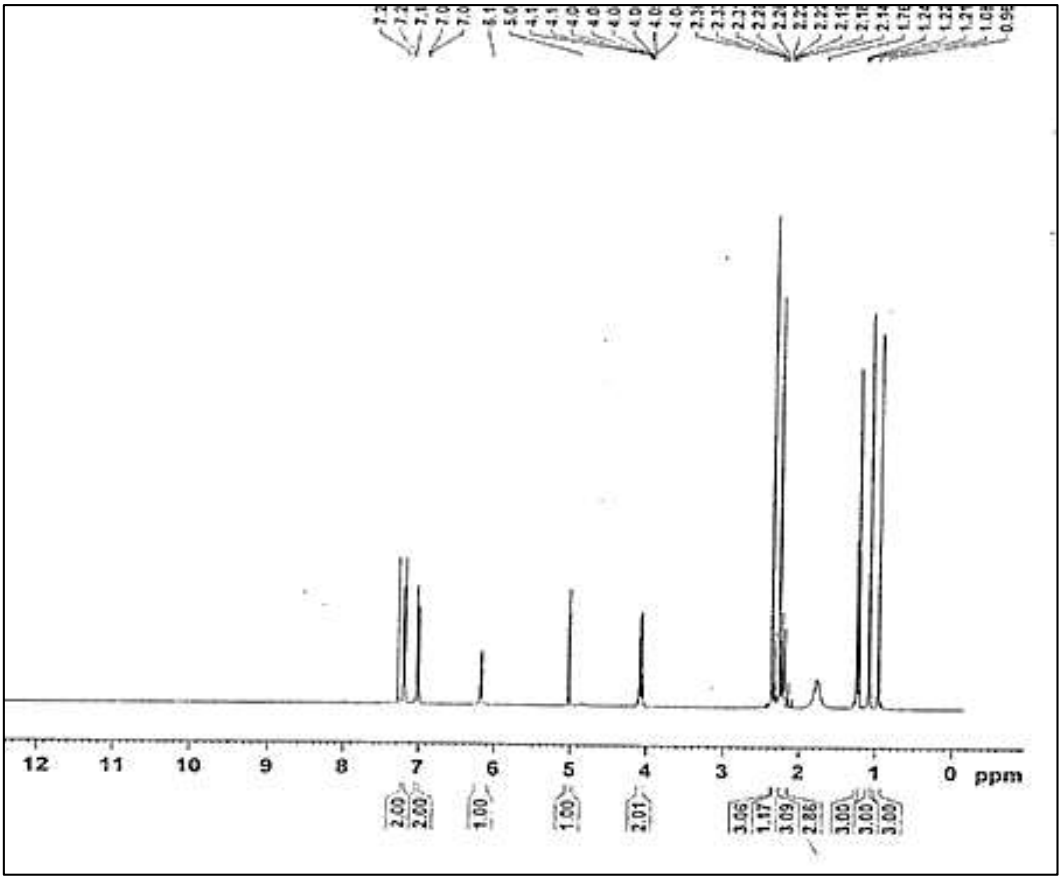


Fig. 18. ^{1}H NMR

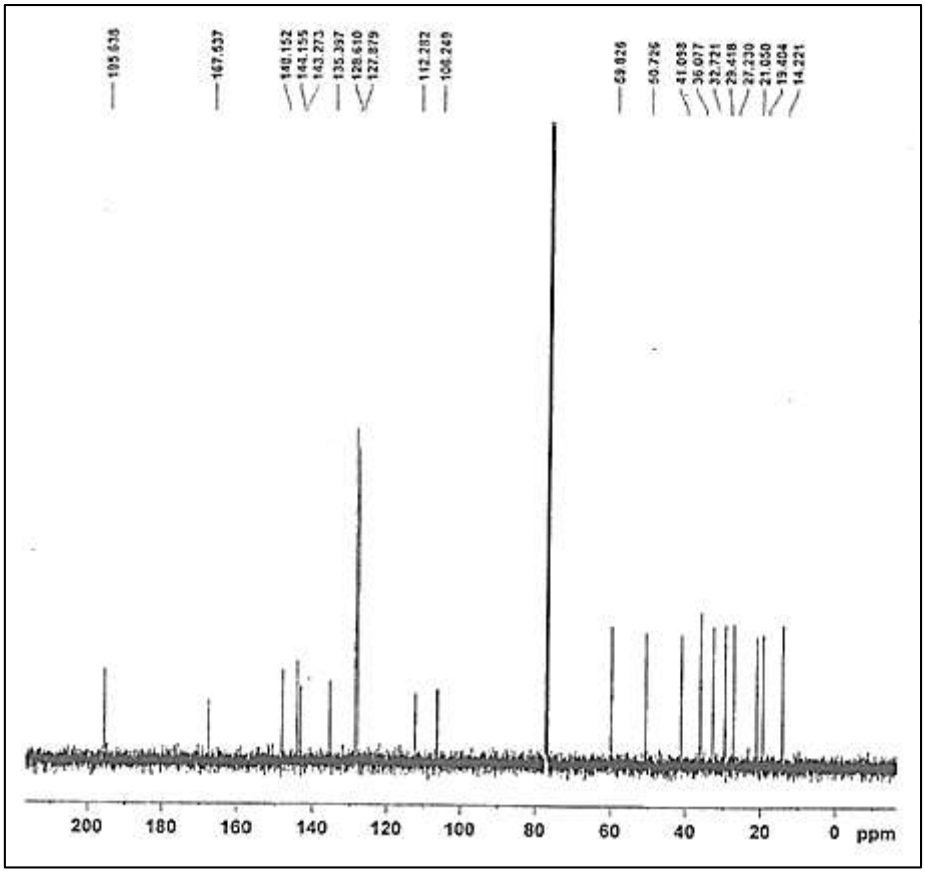
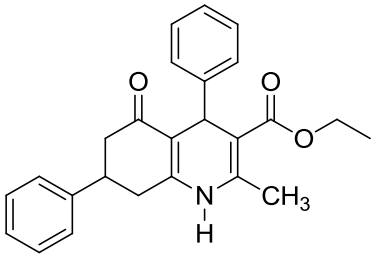


Fig. 19. ^{13}C NMR

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



M.p. 213-214 °C, ν_{max} (KBr) 3276 (NH), 1701 (C=O) (acid), 1606 (C=O) (ketone), 1487 (OC_2H_5) (ester), cm^{-1}; δ_H (400 MHz, DMSO-*d*$_6$) 1.14 (t, 3H, CH_3-CH_2-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, -O-CH_2-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, 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-CH_2-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, CH_3-CH_2O).

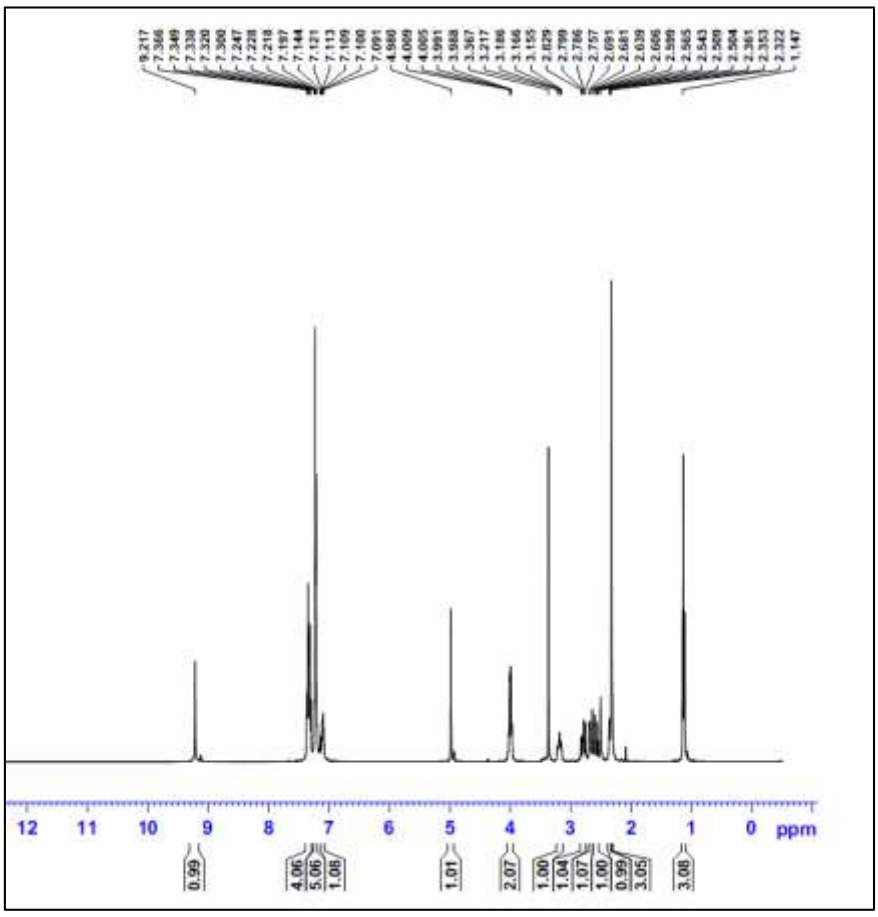


Fig. 20. ^{1}H NMR

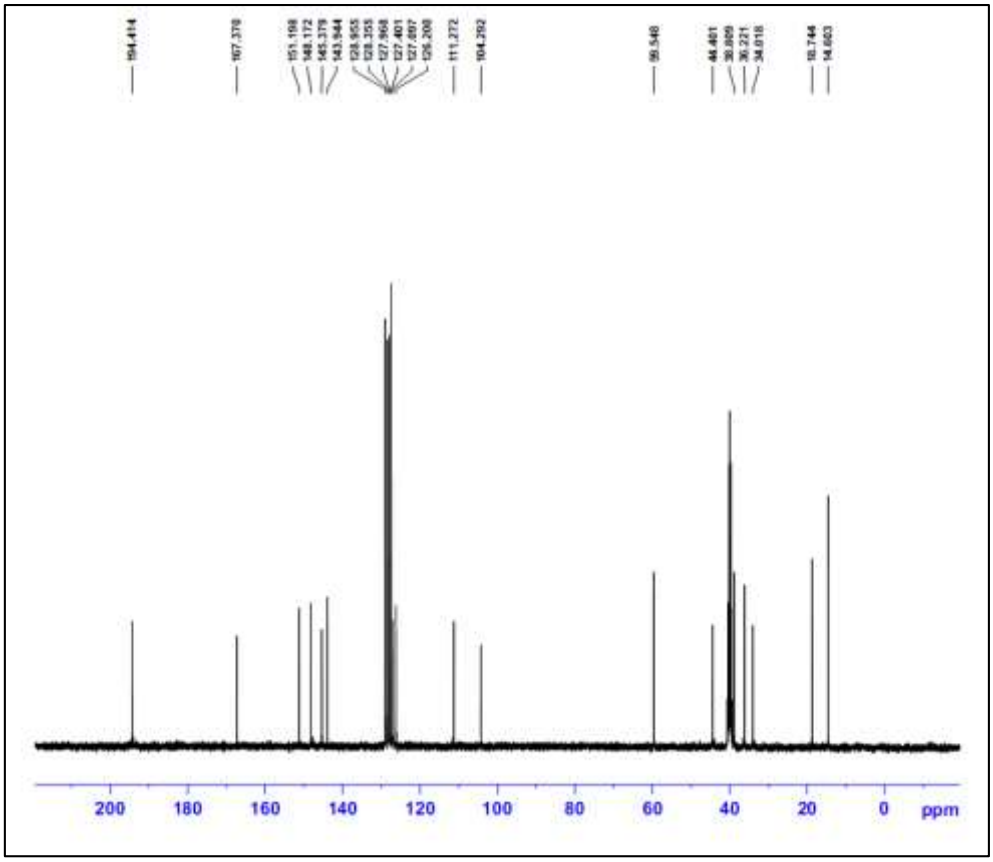


Fig. 21. ^{13}C NMR