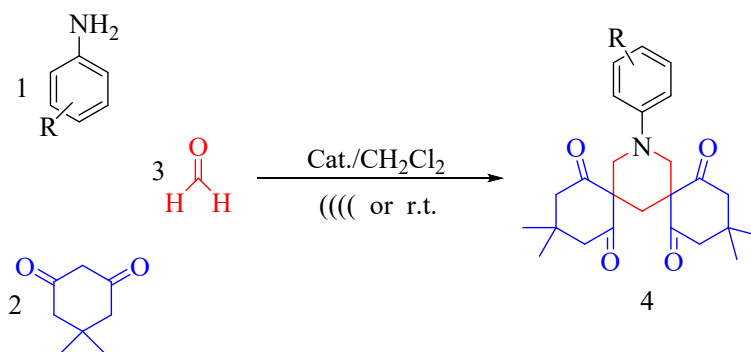


Synthesis of bis-spiro piperidines using nano γ -alumina supported Sb(V) under ultrasonic irradiation or room temperature conditions

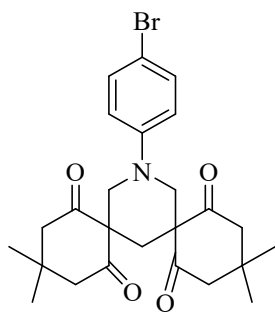
Maryam Aghamohammadsadegh,^a Abdolhamid Bamoniri,^{*a} and Bi Bi Fatemeh Mirjalili^b

^{*a} Department of Organic Chemistry, Faculty of Chemistry, University of Kashan, Kashan, I.R.Iran. E-mail: bamoniri@kashanu.ac.ir

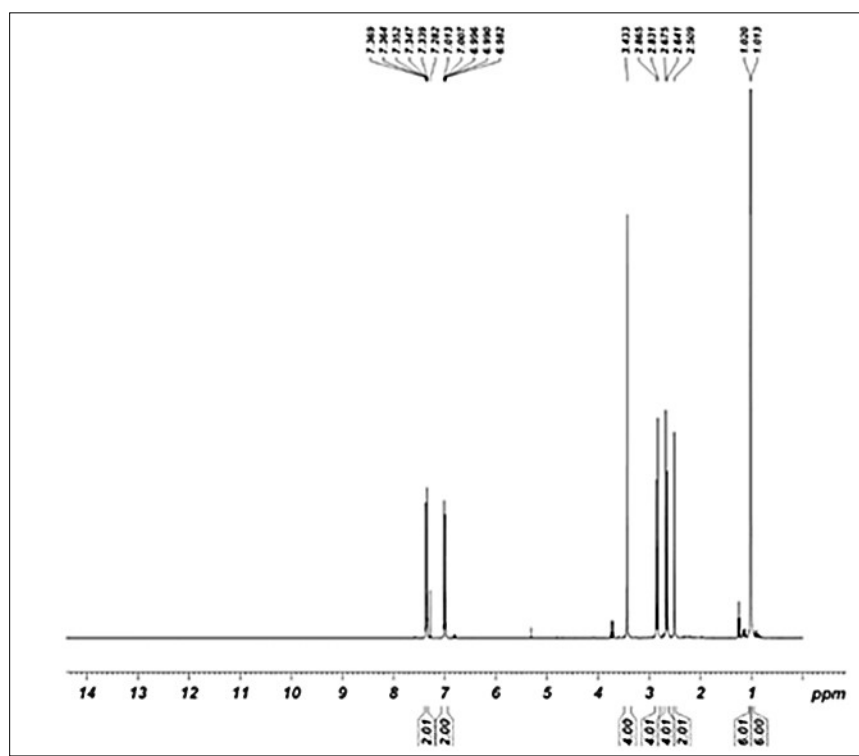
^b Department of Organic Chemistry, College of Science, Yazd University, Yazd, I.R.Iran.



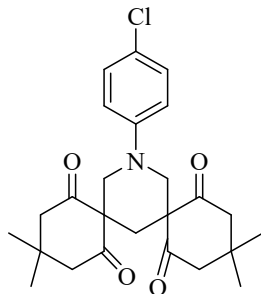
(15-(4-Boromophenyl)-3,3,11,11-tetramethyl-15-azadispiro[5.1.5.3]hexadecne-1,5,9,13-tetrone (table 4, Entry 1)



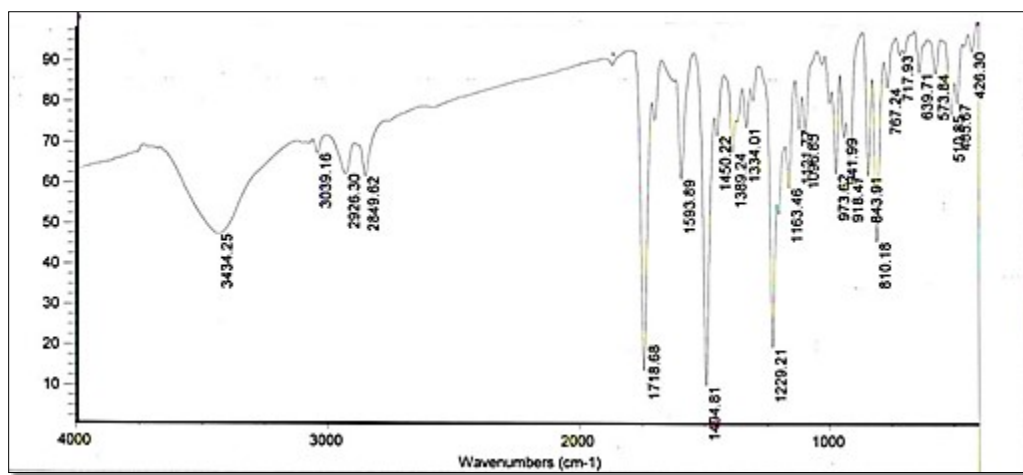
mp: 204-206 °C; FTIR (KBr) ν_{max} : 3039, 2924, 1725, 1600-1460, 1375, 1223, 1073 cm^{-1} . ^1H NMR (400 MHz, CDCl_3 , δ ppm): 1.01 (6H, s, CH_3), 1.02 (6H, s, CH_3), 2.50 (2H, s, CH_2), 2.65 (4H, d, $^2J_{\text{HH}}=13.6$ Hz, COCH_2), 2.84 (4H, d, $^2J_{\text{HH}}=13.6$ Hz, COCH_2), 3.43 (4H, s, NCH_2), 7.00 (2H, dd, $^3J_{\text{HH}}=6.8$, 2.4 Hz, ArH), 7.35 (2H, dd, $^3J_{\text{HH}}=6.8$ Hz, ArH).

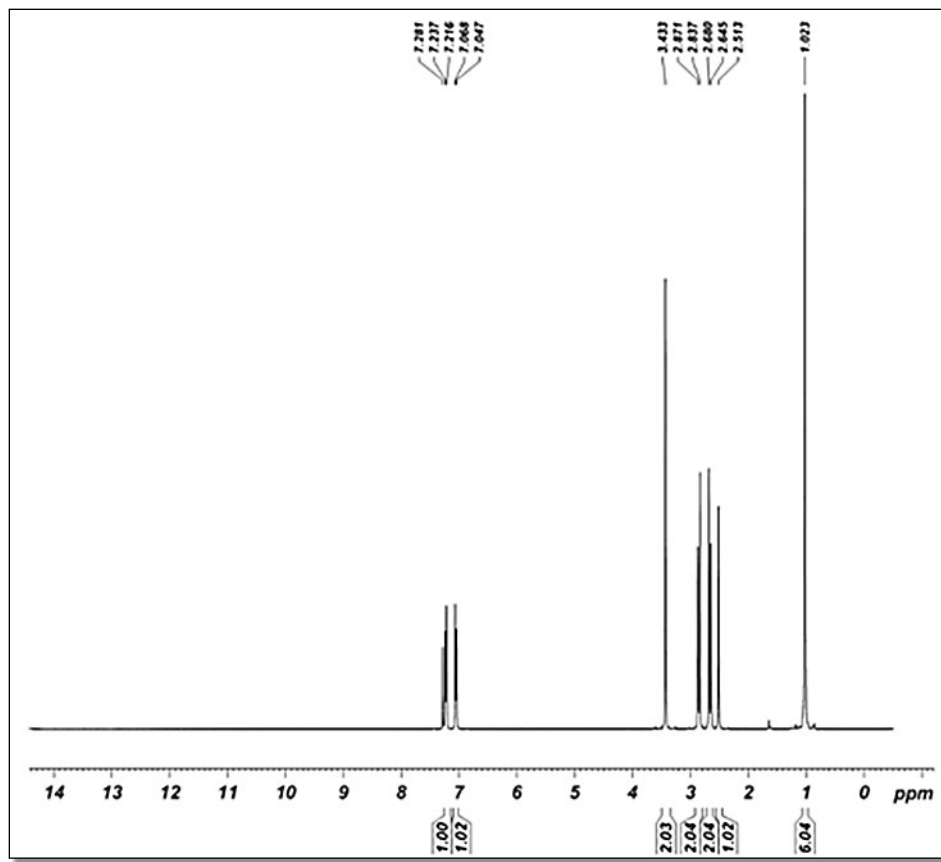


(15-(4-Chlorophenyl)-3,3,11,11-tetramethyl-15-azadispiro[5.1.5.3]hexadecne-1,5,9,13-tetrone (table 4, Entry 2)

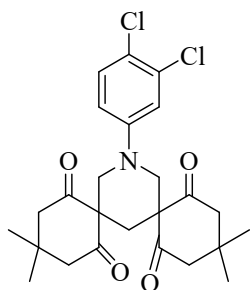


mp: 215-217 °C; FTIR (KBr) ν_{max} : 3039, 2926, 1718, 1593-1450, 1389, 1229, 767 cm^{-1} . ^1H NMR (400 MHz, CDCl_3 , δ ppm): 1.02 (12H, s, CH_3), 2.51 (2H, s, CH_2), 2.66 (4H, d, $^2J_{\text{HH}}=14$ Hz, COCH_2), 2.85 (4H, d, $^2J_{\text{HH}}=13.6$ Hz, COCH_2), 3.43 (4H, s, NCH_2), 7.05 (2H, d, $^3J_{\text{HH}}=8.4$ Hz, ArH), 7.22 (2H, d, $^3J_{\text{HH}}=8.4$ Hz, ArH).

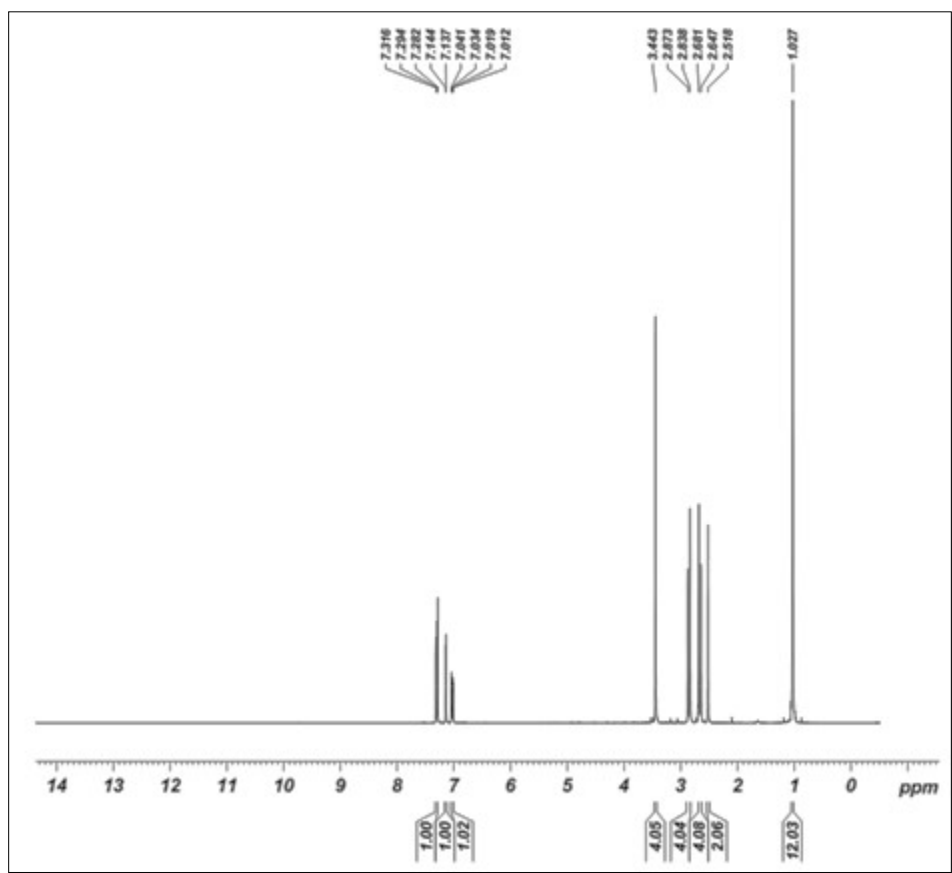
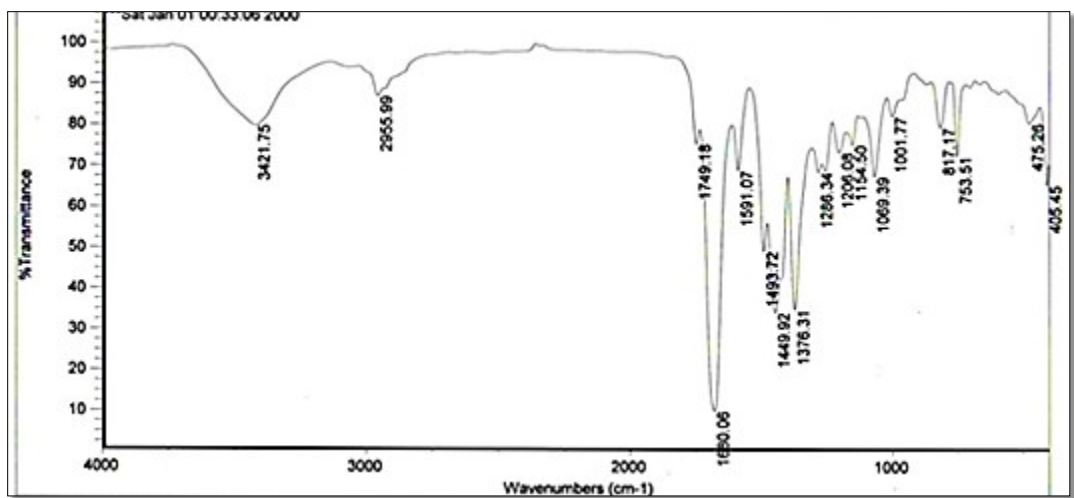




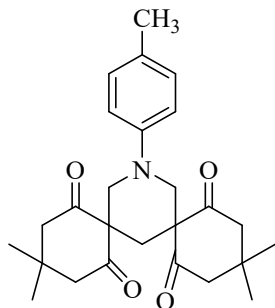
(15-(3,4-Dichlorophenyl)-3,3,11,11-tetramethyl-15-azadispiro[5.1.5.3]hexadecne-1,5,9,1-tetrone (table 4, Entry 3)



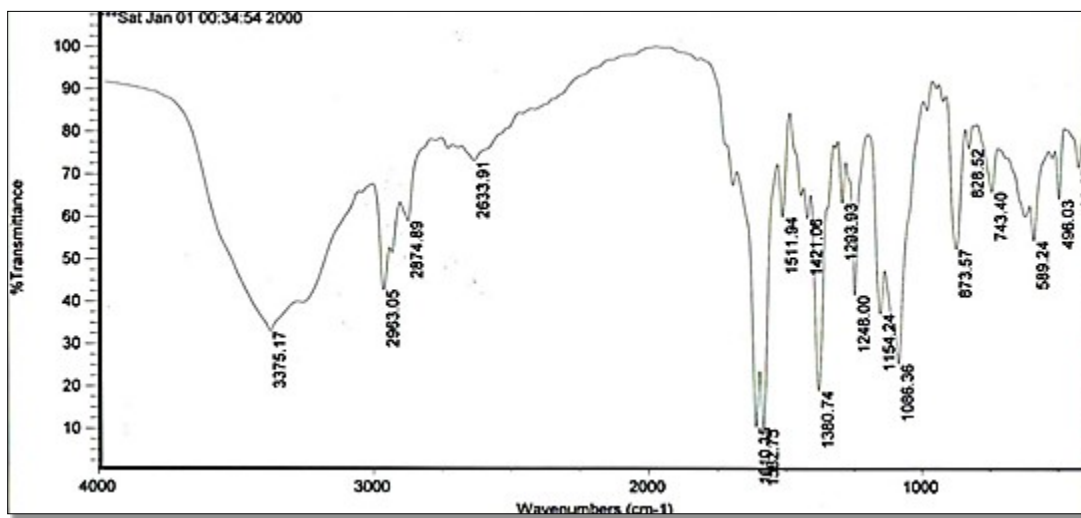
mp: 236-238 °C; FTIR (KBr) ν_{max} : 3050, 2955, 1749, 1660-1493, 1376, 1286, 1001 cm^{-1} . ^1H NMR (400 MHz, CDCl_3 , δ ppm): 1.02 (12H, s, CH_3), 2.51 (2H, s, CH_2), 2.66 (4H, d, $^2J_{\text{HH}}=13.6$ Hz, CH_2), 2.85 (4H, d, $^2J_{\text{HH}}=14$ Hz, COCH_2), 3.44 (4H, s, CH_2), 7.02 (1H, dd, $^3J_{\text{HH}}=8.8$, 2.8 Hz, ArH), 7.13 (1H, d, $^3J_{\text{HH}}=2.8$ Hz, ArH), 7.30 (1H, d, $^3J_{\text{HH}}=8.8$ Hz, ArH).

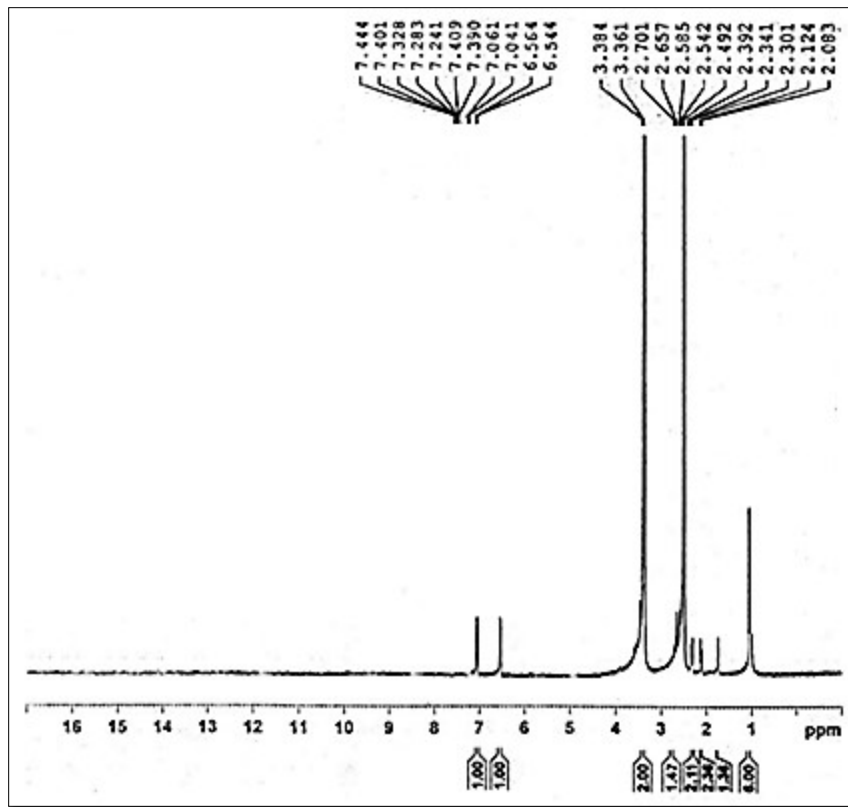


(15-(p-Tolyl)-3,3,11,11-tetramethyl-15-azadispiro[5.1.5.3]hexadecne-1,5,9,13-tetrone
(table 4, Entry 4)

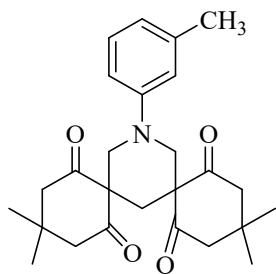


mp: 250-253°C; FTIR (KBr) ν_{max} : 3039, 2963, 1718, 1610-1421, 1380, 1248 cm^{-1} . ^1H NMR (400 MHz, DMSO-d_6 , δ ppm): 1.05 (12H, s, CH_3), 1.74 (2H, s, CH_2), 2.10 (4H, d, $^2J_{\text{HH}}=16.4$ Hz, CH_2), 2.32 (4H, d, $^2J_{\text{HH}}=16$ Hz, CH_2), 2.66 (3H, s, CH_3), 3.38 (4H, s, CH_2), 6.55 (2H, d, $^3J_{\text{HH}}=8$ Hz, ArH), 7.05 (2H, d, $^3J_{\text{HH}}=8$ Hz, ArH).

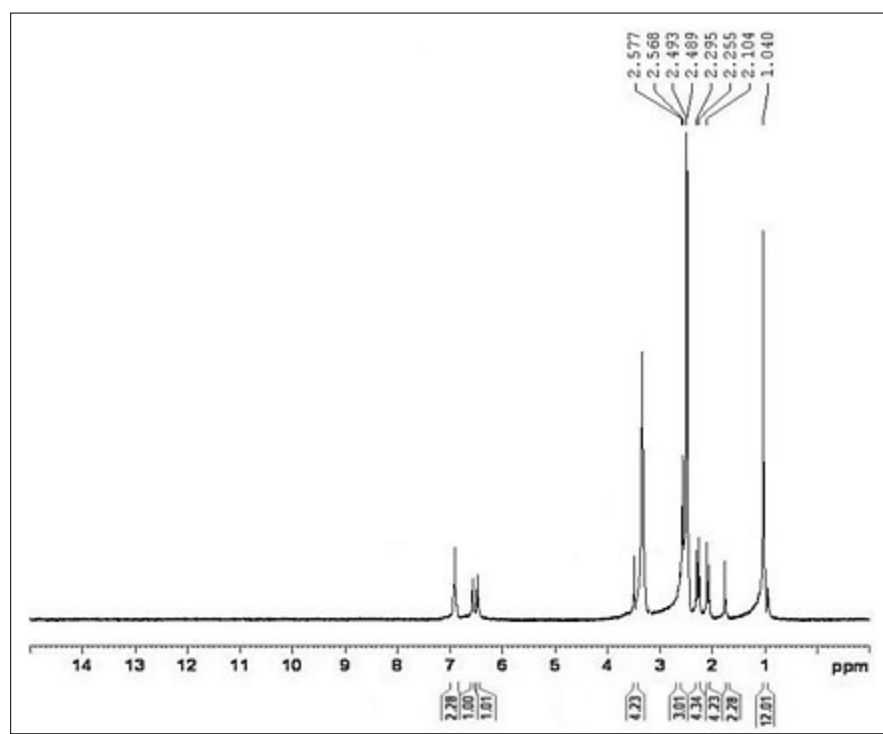
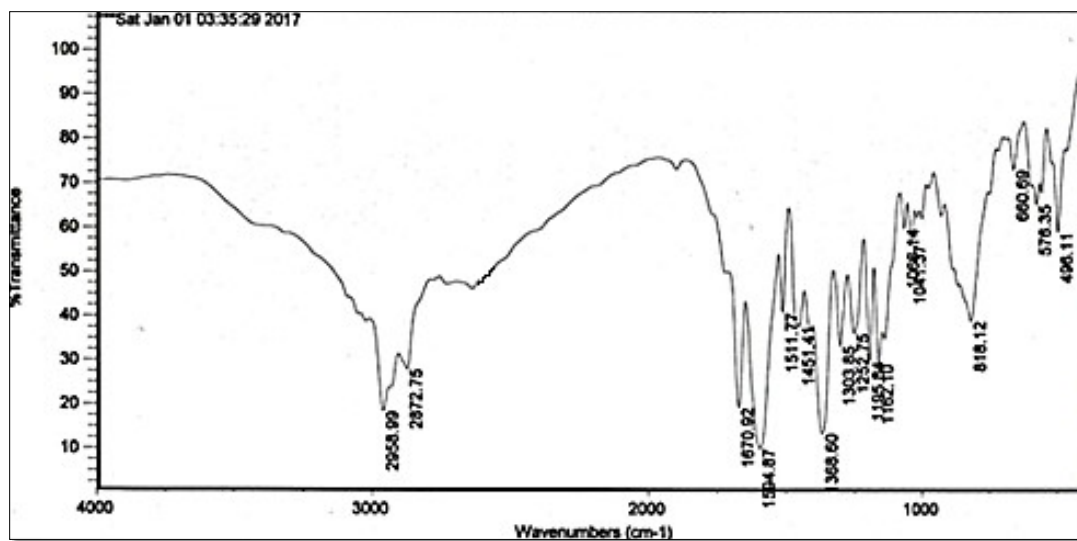




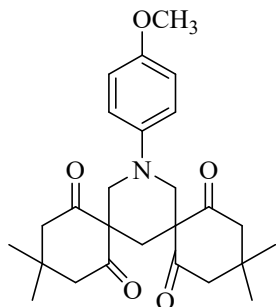
(15-(m-Tolyl)-3,3,11,11-tetramethyl-15-azadispiro[5.1.5.3]hexadecne-1,5,9,13-tetrone
(table 4, Entry 5)



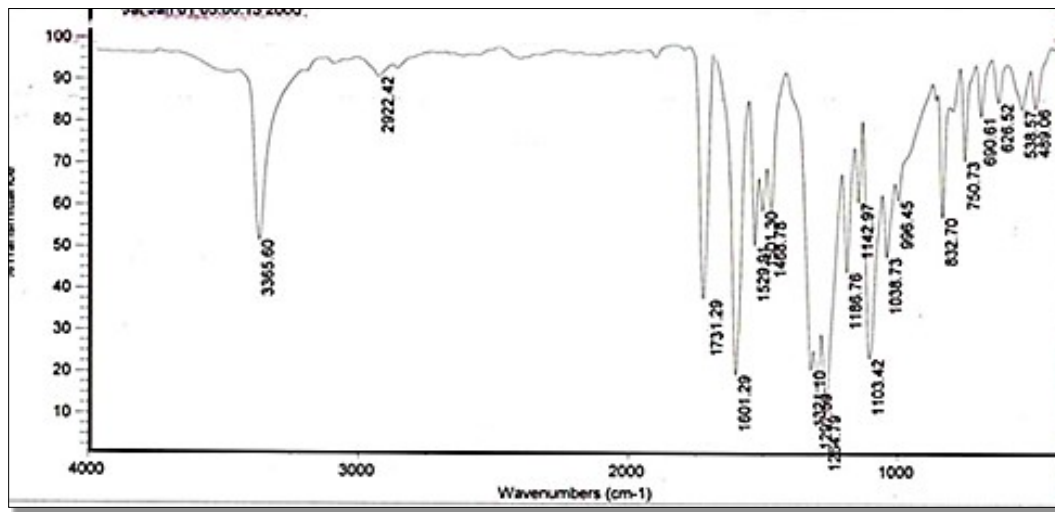
mp: 188-190 °C; FTIR (KBr) ν_{max} : 3015, 2958, 1667, 1596-1417, 1374, 1255 cm^{-1} . ^1H NMR (400 MHz, DMSO- d_6 , δ ppm): 1.04 (12H, s, CH_3), 1.75 (2H, s, CH_2), 2.12 (4H, d, $^2J_{\text{HH}}=16.4$ Hz, CH_2), 2.27 (4H, d, $^2J_{\text{HH}}=16.4$ Hz, CH_2), 2.57 (3H, s, CH_3), 3.4 (4H, s, CH_2), 6.54 (1H, t, $^3J_{\text{HH}}=8.4$ Hz, ArH), 6.64 (1H, d, $^3J_{\text{HH}}=8$ Hz, ArH), 6.85 (1H, d, $^3J_{\text{HH}}=8$ Hz, ArH), 6.90 (1H, s, ArH).

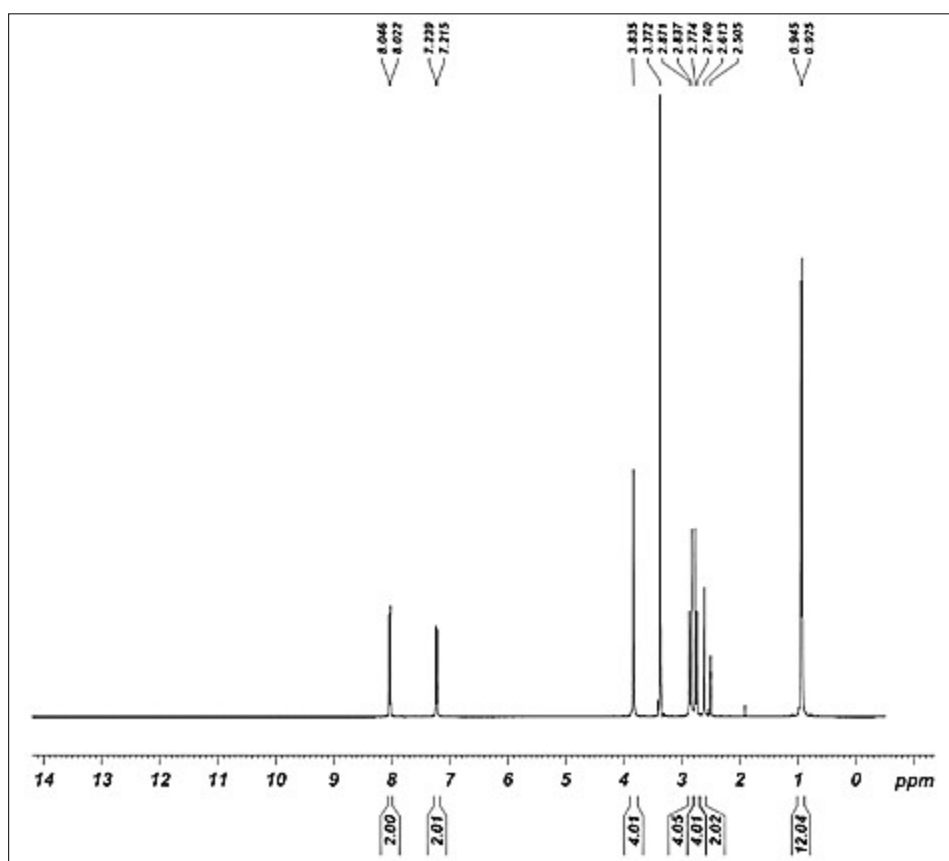
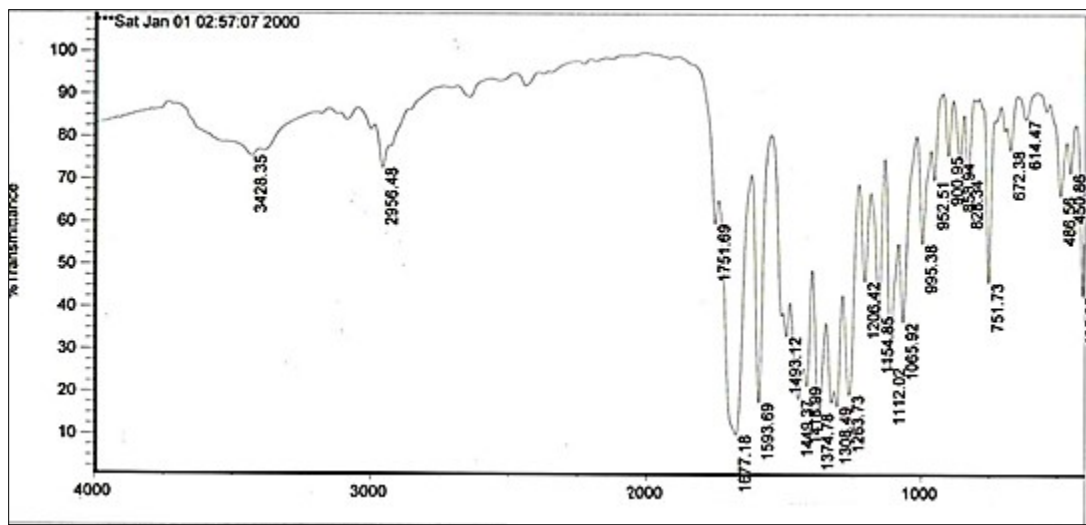


(15-(4-Methoxyphenyl)-3,3,11,11-tetramethyl-15-azadispiro[5.1.5.3]hexadecne-1,5,9,13-tetrone (table 4, Entry 6)

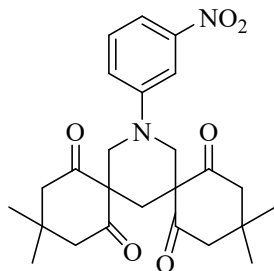


mp: 188–190 °C; FTIR (KBr) $\nu_{\max}$: 3030, 2922, 1731, 1601-1468, 1350, 1264, 1038, 1264 cm^{-1} . (400 MHz, DMSO- d_6 , δ ppm): 1.00 (12H, s, CH_3), 1.72 (2H, s, CH_2), 2.07 (4H, d, $^2J_{\text{HH}}=17.2$ Hz, CH_2), 2.23 (4H, d, $^2J_{\text{HH}}=17.2$ Hz, CH_2), 3.33 (3H, s, OCH_3), 3.66 (4H, s, CH_2), 6.75 (2H, d, $^3J_{\text{HH}}=7.2$ Hz, ArH), 7.05 (2H, d, $^3J_{\text{HH}}=6.4$ Hz, ArH).

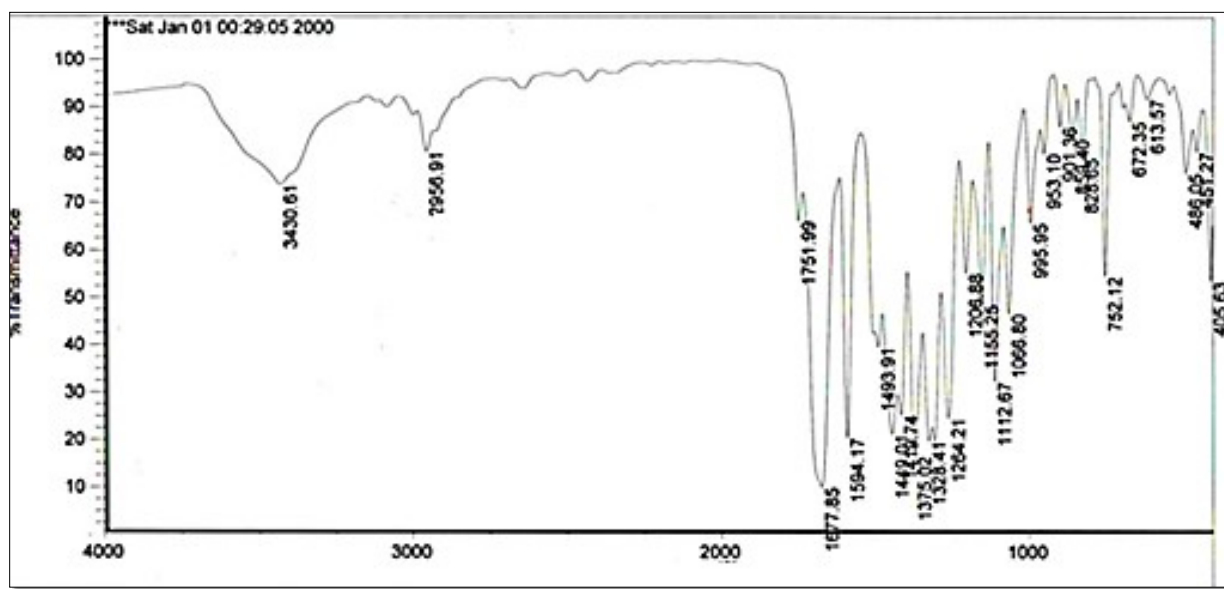


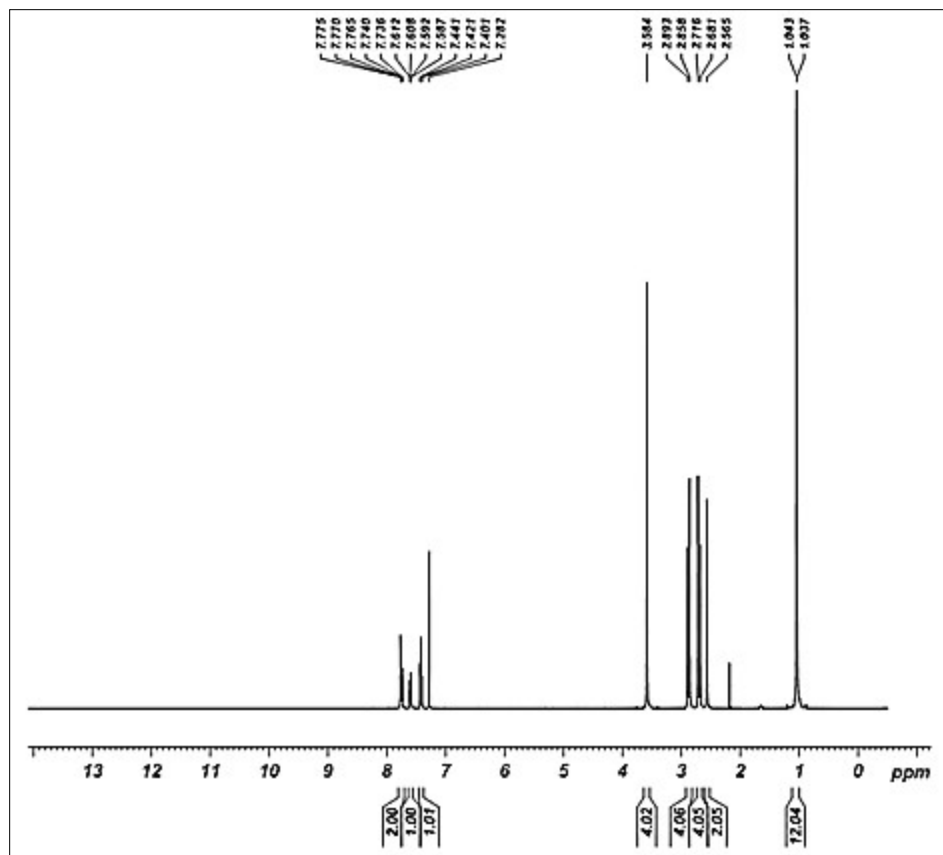


(15-(3-Nitrophenyl)-3,3,11,11-tetramethyl-15-azadispiro[5.1.5.3]hexadecne-1,5,9,13-tetrone (table 4, Entry 8)

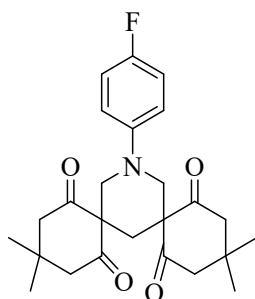


mp: 186–188 °C; FTIR (KBr) ν_{max} : 3030, 2956, 1751, 1677-1449, 1594, 1375, 1264 cm^{-1} . (400 MHz, CDCl_3 , δ ppm): 1.03 (6H, s, CH_3), 1.04 (6H, s, CH_3), 2.56 (2H, s, CH_2), 2.69 (4H, d, $^2J_{\text{HH}}=14.0$ Hz, COCH_2), 2.87 (4H, d, $^2J_{\text{HH}}=14.0$ Hz, COCH_2), 3.57 (4H, s, NCH_2), 7.42 (1H, t, $^3J_{\text{HH}}=8.0$ Hz, ArH), 7.59 (1H, dd, $^3J_{\text{HH}}=8.2$, 2.0 Hz, ArH), 7.75 (1H, dd, $^3J_{\text{HH}}=8.2$, 2.0, ArH).





(15-(4-Fluorine)-3,3,11,11-tetramethyl-15-azadispiro[5.1.5.3]hexadecne-1,5,9,13-tetrone
(table 4, Entry 9)



mp: 140–142 °C; FTIR (KBr) ν_{max} : 2924, 1698, 1598-1469, 1381, 1238, 1094 cm^{-1} . (400 MHz, DMSO- d_6 , δ ppm): 0.96 (6H, s, CH_3), 1.00 (6H, s, CH_3), 2.04 (2H, s, CH_2), 2.73 (4H, s, COCH_2), 2.82 (4H, s, COCH_2), 3.35 (4H, s, NCH_2), 7.28-7.37 (4H, m, ArH).

