2.5.1. Spectral data for compounds

2.5.1.1. Tetrahydro-3,7-diphenyl-[1,2,4] triazolo [1,2-a] [1,2,4] triazole-1,5-dithione (**3a**): white solid, m.p= 188-189 °C; R_f (petroleum ether:ethylacetate= 7:3 (v/v)) = 0.26; IR (KBr)/ ν (cm^{-1}): 3385, 3180, 1500, 1251; ^{1}H NMR (Acetone-d_6, 400 MHz)/ δ ppm:6.81 (s, 2H, CH), 7.38-7.42 (m, 10H, Ar-H), 11.42 (s, 2H, NH); ^{13}C NMR (DMSO-d_6, 100 MHz) δ (ppm): 73 (CH), 126.25 (CH), 127.76 (CH), 128.31 (CH), 129.73 (CH), 184.1 (C=S) ppm; $CHN_{Calculated}$ (%): C (58.89), H (4.29), N (17.18), S (19.63); CHN_{Found} (%): C (58.82), H (4.39), N (17.34), S (19.73).[17]

2.5.1.2. Tetrahydro-3,7-bis (3-nitrophenyl)-[1,2,4] triazolo [1,2-a] [1,2,4] triazole-1,5-dithione (**3b**): white solid, m.p= 190-191 °C; R_f (petroleum ether: ethyl acetate= 7:3 (v/v)) =0.15; IR (KBr)/ ν (cm^{-1}): 3215, 1616, 1529, 1491, 1352, 1247, 1161; ^{1}H NMR (Acetone-d_6, 400 MHz)/ δ ppm:7.25 (s, 2H, CH), 7.80 (t, *J*= 7.0 Hz, 2H, ArH), 8.00 (d, *J*= 7.0 Hz, 2H, ArH), 8.31 (d, *J*= 7.0 Hz, 2H, ArH), 8.39 (s, 2H, ArH), 10.38 (s, 2H, NH) ppm;); ^{13}C NMR (Acetone-d_6, 100 MHz) δ (ppm): 72.0 (CH), 119.43 (CH), 122.55 (CH), 129.88 (CH), 133.76 (CH), 145.50 (CH), 148.60 (C), 158.09 (C), 184.11 (C=S) ppm; $CHN_{Calculated}$ (%): C (46.02), H (2.2.87), N (20.13), S (15.62), O (15.36); CHN_{Found} (%): C (46.45), H (3.01), N (19.18), S (15.35), O (16.01).

2.5.1.3. Tetrahydro-3,7-bis (4-methoxyphenyl)-[1,2,4] triazolo [1,2-a] [1,2,4] triazole-1,5-dithione (**3c**): white solid, m.p= 160-162 °C; R_f (petroleum ether: ethyl acetate= 7:3 (v/v)) =0.18; IR (KBr)/ ν (cm^{-1}): 3390, 3157, 2960, 1613, 1508, 1249, 1173, 1028; ^{1}H NMR (Acetone-d_6, 400 MHz)/ δ ppm: 3.72 (s, 6H, OCH_3), 6.75 (s, 2H, CH), 6.99 (s, 4H, Ar-H), 7.28-7.30 (d, J= 6.8 Hz, 4H, Ar-H), 11.31 (s, 2H, NH) ppm; ^{13}C NMR (DMSO-d_6, 100 MHz) δ (ppm): 55.73 (CH3), 77.03 (CH), 114.76 (CH), 127.67 (CH), 130.0 (C), 160.35 (C), 184.0 (C=S); ppm; $CHN_{Calculated}$ (%): C (55.96), H (4.66), N (14.51), S (16.58); CHN_{Found} (%): C (55.93), H (4.76), N (14.72), S (16.68). [17]

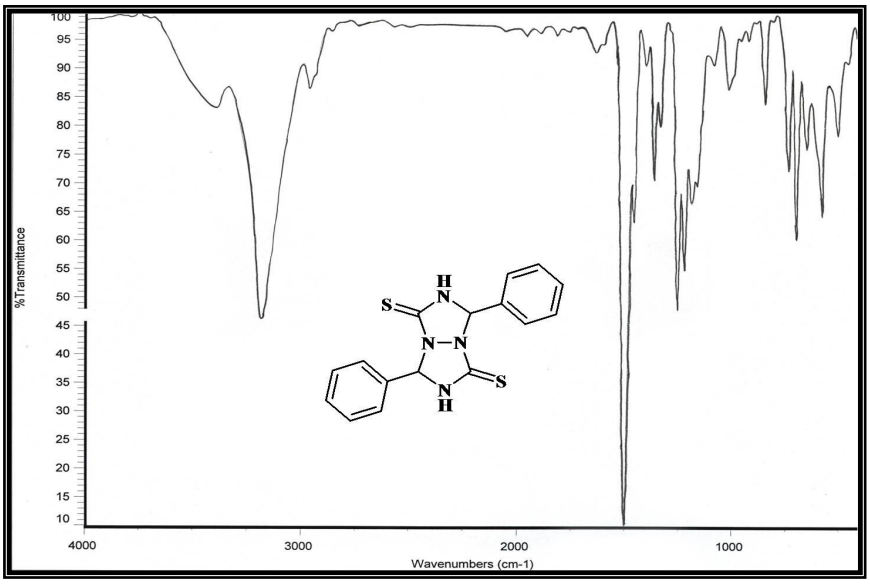
2.5.1.4. Tetrahydro-3,7-bis (2-chlorophenyl)-[1,2,4] triazolo [1,2-a] [1,2,4] triazole-1,5-dithione **(3d):**white solid, m.p= 197 °C; R_f (petroleum ether: ethyl acetate= 7:3 (v/v)) =0.32; IR (KBr) ν (cm^{-1}): 3433, 3183, 2929, 1498,1251; ^{1}H NMR (DMSO-d_6, 400 MHz) δ (ppm): 7.16 (s, 1H, CH), 7.34-7.36 (m, 1H, Ar-H), 7.43-7.47 (m, 2H, Ar-H), 7.54-7.61 (m, 1H, Ar-H), 11.48 (s, 1H, NH); ^{13}C NMR (DMSO-d_6, 100 MHz) δ (ppm): 74.86 (CH), 128.14 (CH), 128.63 (CH), 130.62 (CH), 131.87 (CH), 132.48 (C), 134.01 (C), 185.19 (C=S) ppm; $CHN_{Calculated}$ (%): C (48.60), H (3.04), N (14.18), S (16.20), Cl (17.72); CHN_{Found} (%): C (47.99), H (3.58), N (14.76), S (16.09), Cl (17.58).

*2.5.1.5.*Tetrahydro-3,7-bis (3-chlorophenyl)-[1,2,4] triazolo [1,2-a] [1,2,4] triazole-1,5-dithione (**3e**): white solid, m.p= 194-195 °C; R_f (petroleum ether: ethyl acetate= 7:3 (v/v)) = 0.35; IR (KBr)/ ν (cm^{-1}): 3427, 3191, 2927, 1501, 1247 ;^{1}H NMR (Acetone-d_6, 400 MHz)/ δ ppm: 7.07 (s, 1H, CH), 7.47-7.51 (m, 3H, Ar-H), 7.55 (s, 1H, Ar-H), 10.14 (s, 1H, NH); ^{13}C NMR (DMSO-d_6, 100 MHz) δ (ppm): 76.62 (CH), 124.88 (CH), 126.39 (CH), 128.12 (CH), 131.84 (CH), 134.15 (C), 139.76 (C), 184.47 (C=S) ppm; $CHN_{Calculated}$ (%): C (48.60), H (3.04), N (14.18), S (16.20), Cl (17.72); CHN_{Found} (%): C (48.32), H (3.13), N (14.31), S (16.29), Cl (17.81). [17]

2.5.1.6. Tetrahydro-3,7-bis (4-chlorophenyl)-[1,2,4] triazolo [1,2-a] [1,2,4] triazole-1,5-dithione (**3f**): white solid, m.p= 201-203 °C; R_f(petroleum ether: ethyl acetate)= 7:3 (v/v)) =0.31; IR (KBr)/ ν (cm^{-1}): 3438, 3168, 2925, 1629, 1492, 1249, 1157; ^{1}H NMR (Acetone-d_6, 400 MHz)/ δ ppm: 6.84 (s, 2H, CH), 7.39 (s, 4H, ArH), 7.51 (s, 4H, ArH), 11.48 (s, 2H, NH) ppm; ^{13}C NMR (DMSO-d_6, 100 MHz) δ (ppm): 75.50 (CH), 128.4 (CH), 130.45 (CH), 133.21 (CH), 133.8 (CH), 184.1 (C=S) ppm; $CHN_{Calculated}$ (%): C (48.60), H (3.04), N (14.18), S (16.20), Cl (17.72); CHN_{Found} (%): C (58.82), H (4.39), N (17.34), S (19.73). [17]

2.5.1.7. Tetrahydro-3,7-bis (3-hydroxyphenyl)-[1,2,4] triazolo [1,2-a] [1,2,4] triazole-1,5-dithione (**3g**): white solid, m.p= 165-167 °C; R_f (petroleum ether: ethyl acetate= 7:3 (v/v)) =0.12; IR (KBr)/ ν (cm^{-1}): 3423, 3177, 2958, 1601, 1505, 1250; ^{1}H NMR (Acetone-d_6, 400 MHz) δ (ppm): 6.95 (s, 4H, Ar-H), 7.24 (s, 1H, CH), 8.62 (s, 1H, OH), 9.97 (s, 1H, NH) ppm; ^{13}C NMR (Acetone-d_6, 100 MHz) δ (ppm): 73 (CH), 112.01 (CH), 113. 88 (CH), 119.03 (CH), 130.0 (CH), 145.50 (CH), 158.09 (C), 184. 0 (C=S) ppm; $CHN_{Calculated}$ (%): C (53.70), H (3.88), N (15.04), S (17.68), O (9.70); CHN_{Found} (%): C (53.76), H (3.58), N (15.34), S (17.74), O (9.66).

2.5.1.8. Tetrahydro-3,7-bis (3-hydroxy-4-methoxyphenyl)-[1,2,4] triazolo [1,2-a][1,2,4]triazole-1,5-dithione (**3h**): white solid, m.p= 177-180 °C; R_f (petroleum ether: ethyl acetate= 7:3 (v/v)) =0.06; IR (KBr)/ ν (cm^{-1}): 3422, 2929, 1510, 1278, 1133; ^{1}H NMR (Acetone-d_6, 400 MHz)/ δ ppm:3.81 (s, 6H, OCH_3), 6.86 (s, 2H, CH), 6.96-7.40 (m, 6H, ArH), 7.89 (s, 2H, OH), 9.88 (s, 2H, NH) ppm; ^{13}C NMR (Acetone-d_6, 100 MHz) δ (ppm): 55.40 (OCH_3), 77.09 (CH), 111.54 (CH), 112.79 (CH), 117.32 (CH), 129.98 (C), 146.91 (C), 148.37 (C), 185.00 (C=S); $CHN_{Calculated}$ (%): C (51.52), H (4.29), N (13.35), S (15.55), O (15.29); CHN_{Found} (%): C (51.34), H (4.83), N (12.88), S (14.98), O (15.97).



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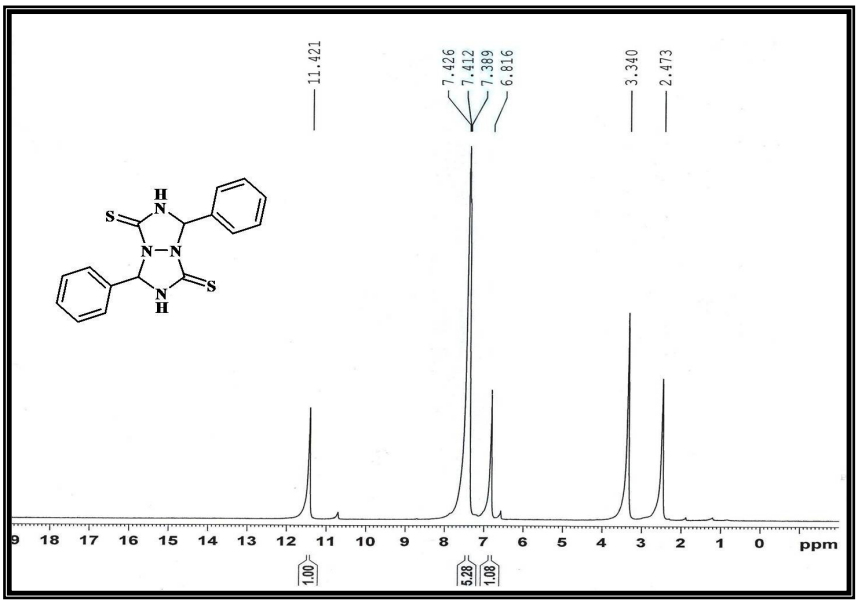

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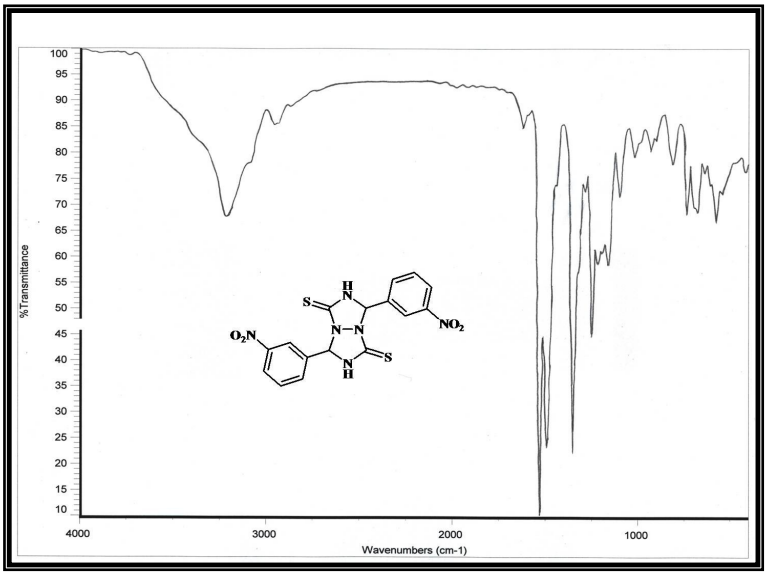

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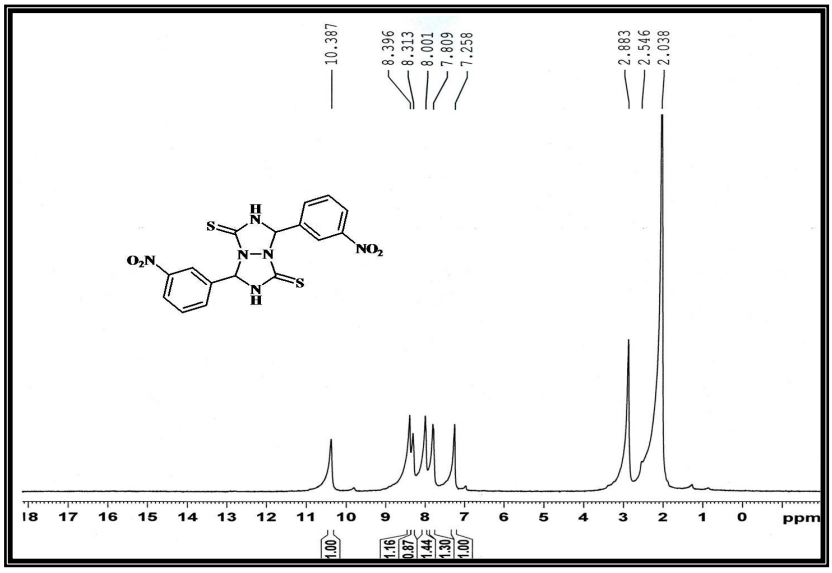

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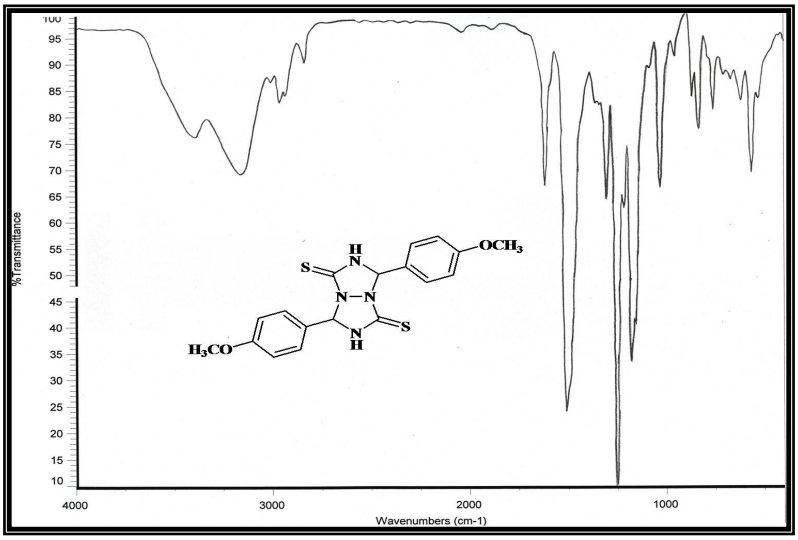

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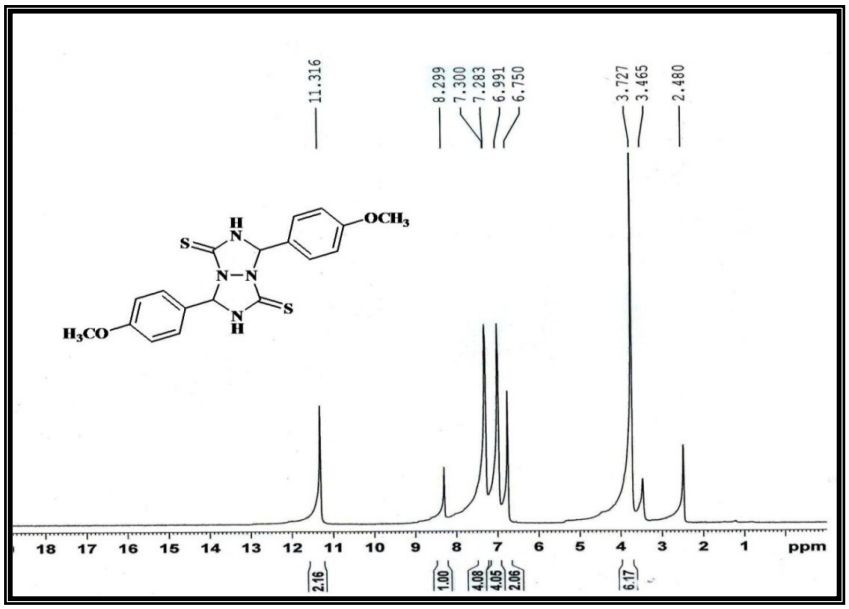

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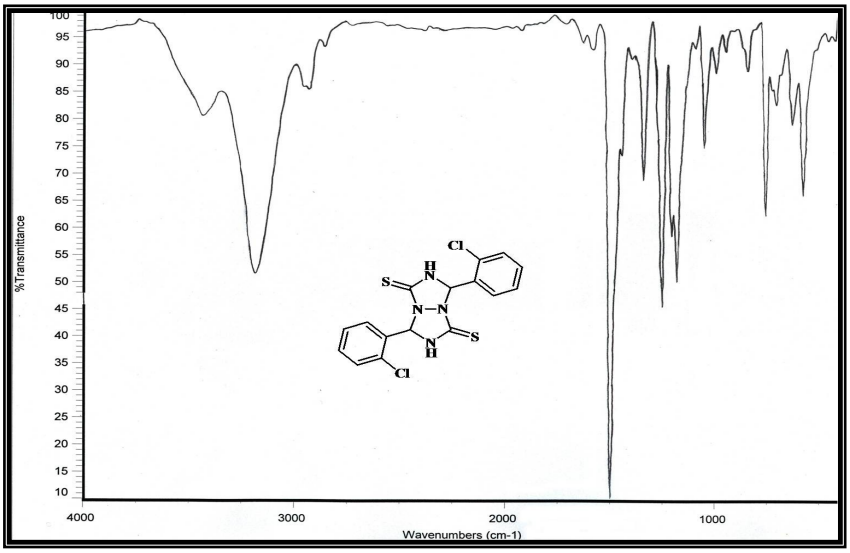

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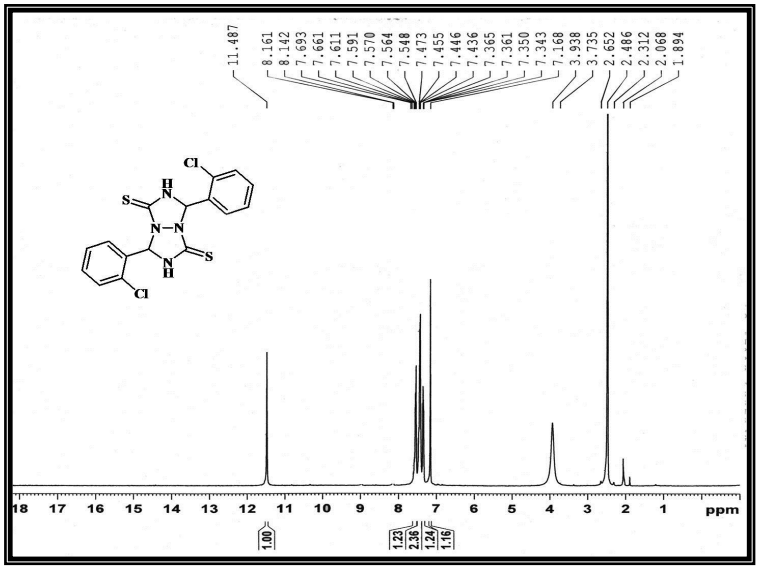
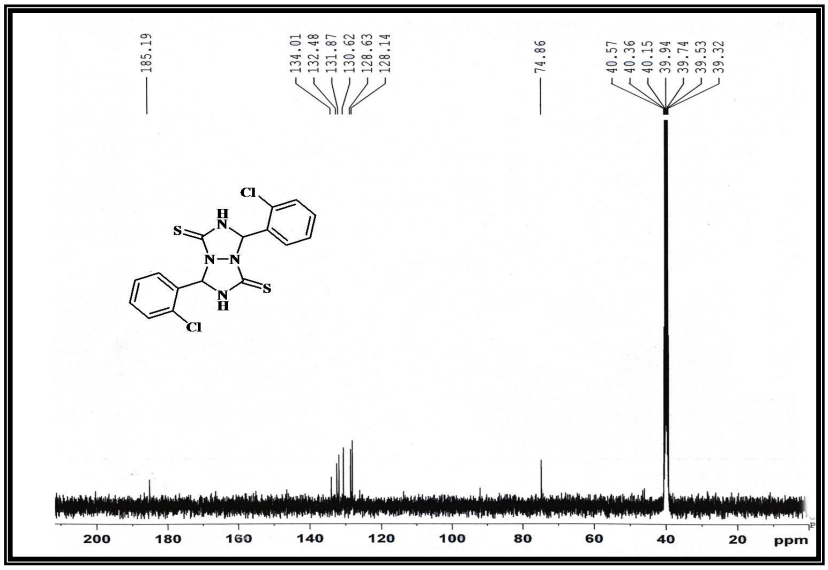


^{1}H NMR of 3d



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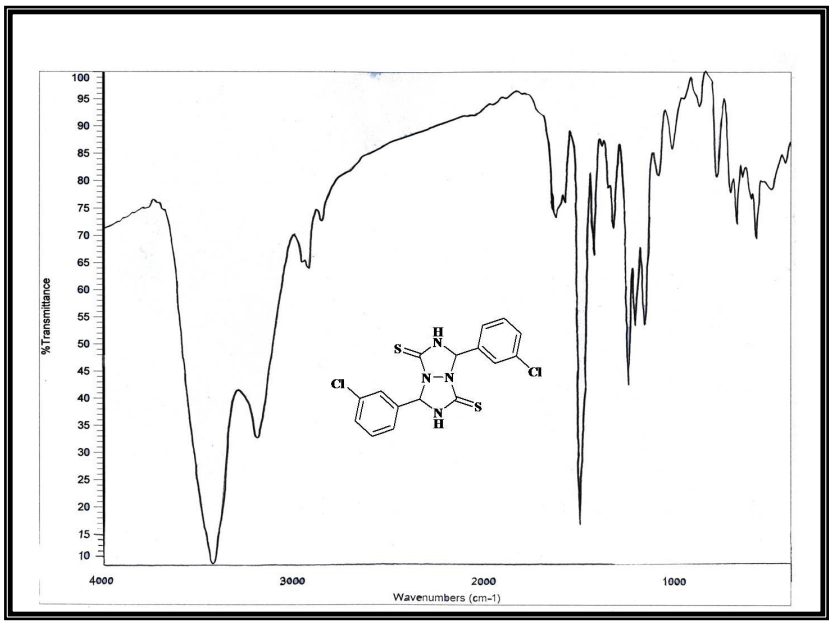

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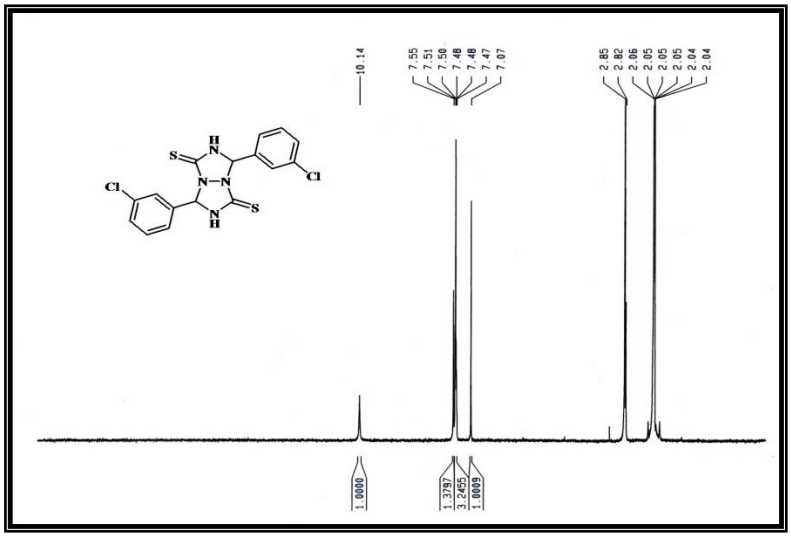

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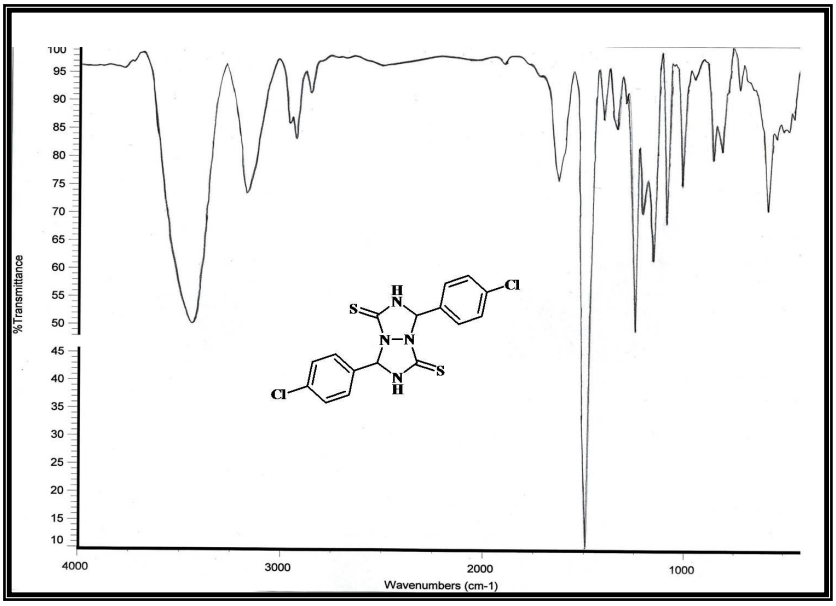

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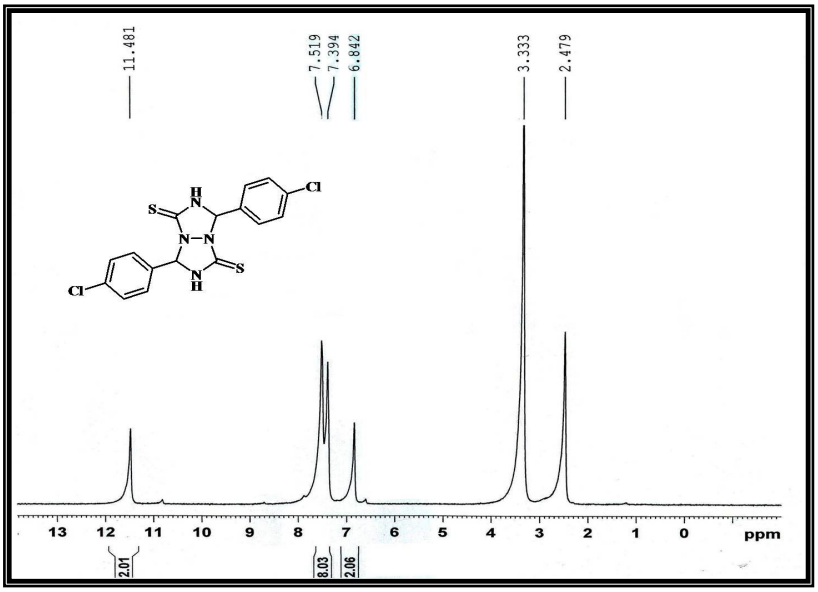

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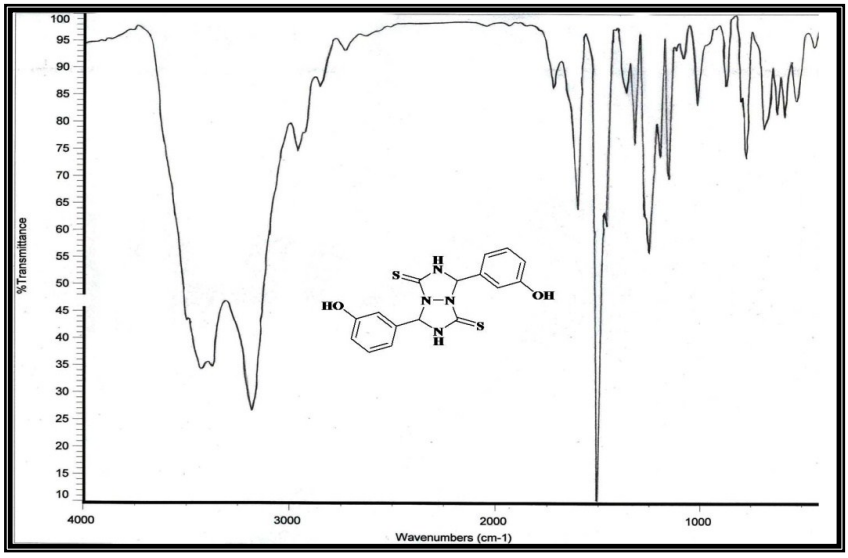
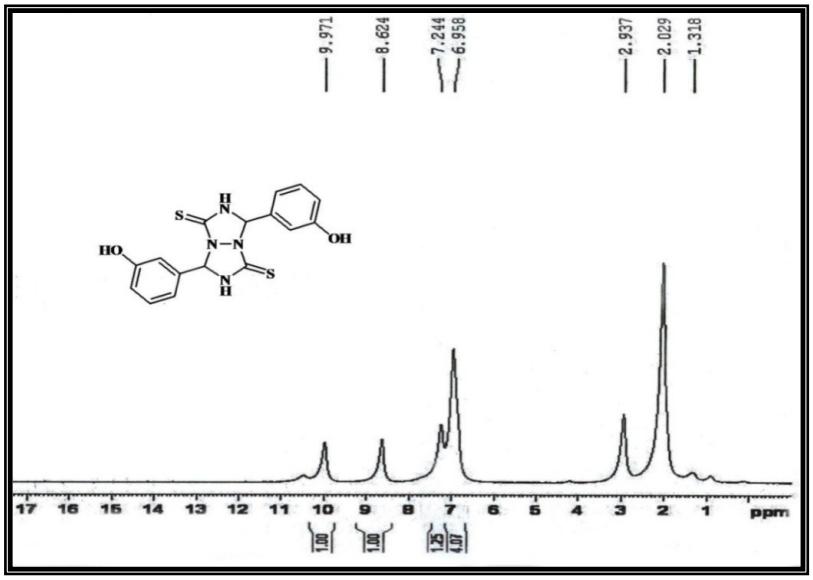
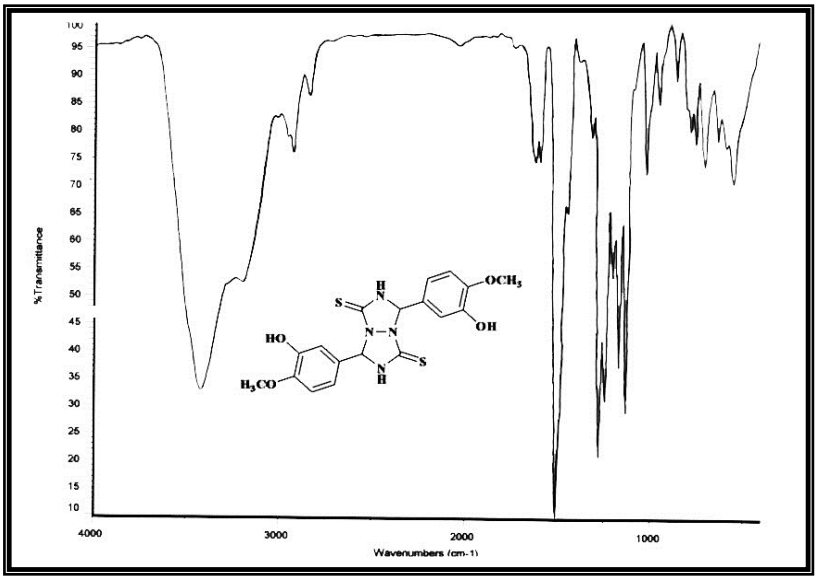


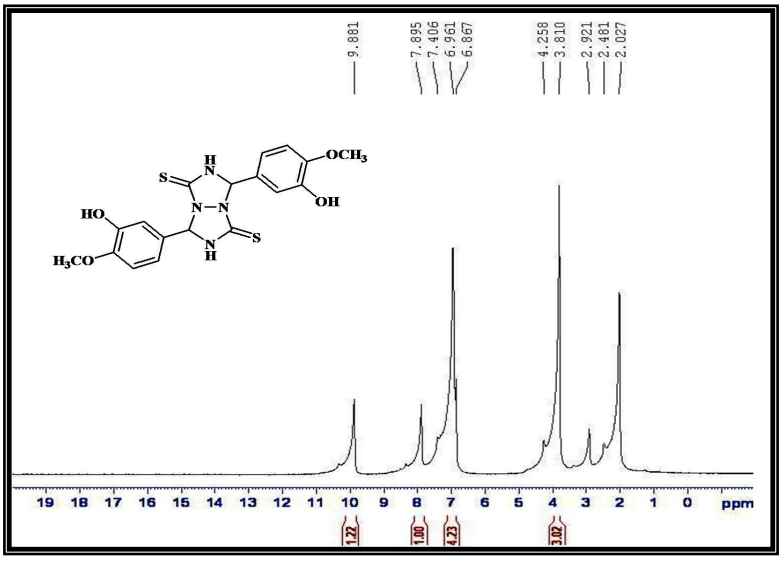
FT-IR of 3g



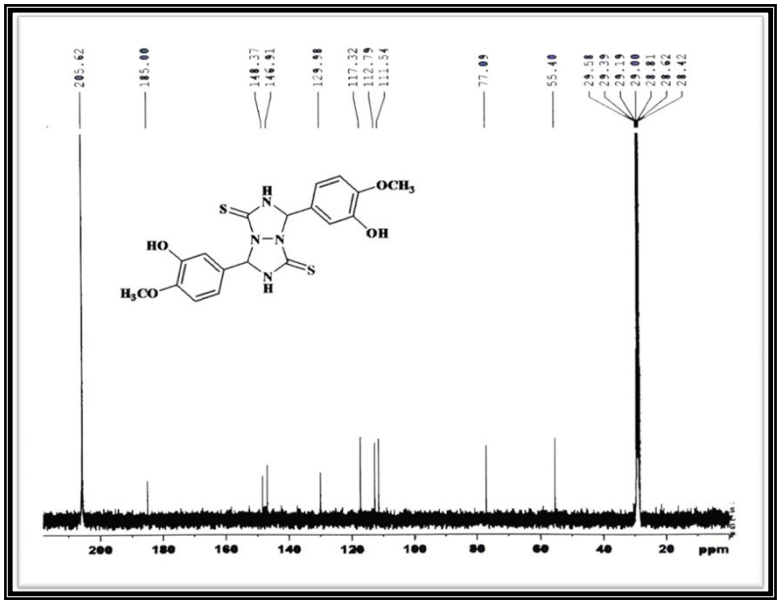
^{1}H NMR of 3g



FT-IR of 3h



^{1}H NMR of 3h



^{13}C NMR of 3h