Electronic Supplementary Material (ESI) for RSC Advances.

CCSO nano catalyzed solid phase synthesis of 3-oxo-5,6-disubstituted-2,3-dihydropyridazine-4-carbonitrile

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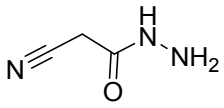
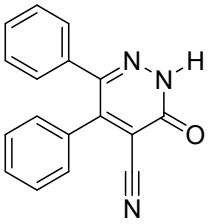
- Spectral data of compound 2a-2i.
- ^{1}H NMR spectra of compound (2a-2i) from page no. 5 to 11.
- ^{13}C NMR spectra of compound (2a-2h) from page no. 12 and 18.

2-cyanoacetohydrazide (1):

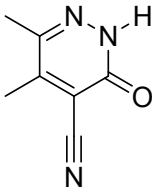


M.P.104-105°C; Yield: 0.17g (86%); FAB MS: m/z 100 (M+1); Elemental analysis for $C_3H_5N_3O$: Calcd: C, 36.36; H, 5.09; N, 42.41%; Found: C, 36.31; H, 5.06; N, 42.39%.

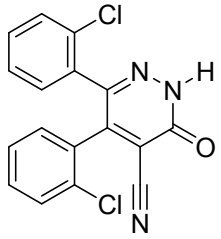
3-oxo-5,6-diphenyl-2,3-dihydropyridazine-4-carbonitrile (2a): M.P.270-272°C; Yield: 5.14g (94%); ^{1}H NMR (300MHz, $CDCl_3$): δ 7.08-7.45 (m, 10H, Ar-H), 11.62 (s, 1H, -NH); ^{13}C NMR (75MHz, $CDCl_3$): δ 113.4, 113.9, 127.9, 128.4, 128.5, 128.7, 129.1, 129.9, 133.3, 134.7, 145.9, 151.8, 157.5; FAB MS: m/z 274 (M+1); Elemental analysis for $C_{17}H_{11}N_3O$: Calcd: C, 74.71; H, 4.06; N, 15.38%; Found: C, 74.59; H, 4.01; N, 15.27%.



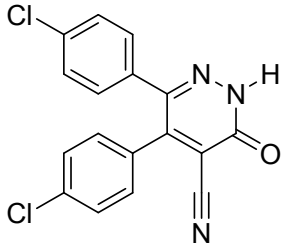
5,6-dimethyl-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2b) M.P. 209–211°C; Yield: 3.07g (94 %); ^{1}H NMR (300MHz, $Si(CH_3)_4$, $CDCl_3$): δ 2.34 (s, 3H, CH_3), 2.49 (s, 3H, CH_3), 11.28 (s, 1H, NH); FAB MS: m/z 150 (M+1); Elemental analysis for $C_7H_7N_3O$: Calcd: C, 56.37; H, 4.73; N, 28.17%; Found: C, 56.21; H, 4.68; N, 28.32%.



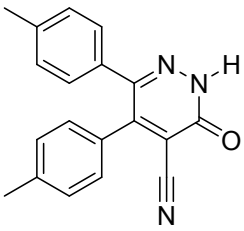
5,6-bis(2-chlorophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2c): M.P.263-265°C; Yield: 6.5g (95%);^{1}H NMR (300MHz, $CDCl_3$): δ 7.04-7.41 (m, 6H, Ar-H), 7.61-7.87 (d, 1H, Ar-H), 7.90-7.96 (d, 1H, Ar-H); ^{13}C NMR (75MHz, $CDCl_3$): δ 104.1, 112.3, 126.4, 126.8, 127.6, 128.1, 128.3, 129.5, 129.7, 134.0, 134.1, 137.5, 161.5, 164.6; FAB MS: m/z 342 (M+1); Elemental analysis for $C_{17}H_9Cl_2N_3O$: Calcd: C, 59.67; H, 2.65; N, 12.28%; Found: C, 59.60; H, 2.57; N, 12.24%.



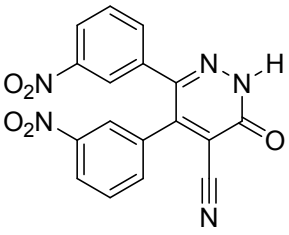
5,6-bis(4-chlorophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2d): M.P. 268-270°C; Yield: 6.36g (93%); ^{1}H NMR (300MHz, $CDCl_3$): δ 7.30-7.45 (m, 6H, Ar-H), 7.87-7.89 (d, 2H, Ar-H), 14.42 (s, 1H, -NH); ^{13}C NMR (75MHz, $CDCl_3$): δ 126.2, 130.5, 130.6, 131.2, 133.1, 167.2; FAB MS: m/z 342 (M+1); Elemental analysis for $C_{17}H_9Cl_2N_3O$: Calcd: C, 59.67; H, 2.65; N, 12.28%; Found: C, 59.61; H, 2.59; N, 12.20%.



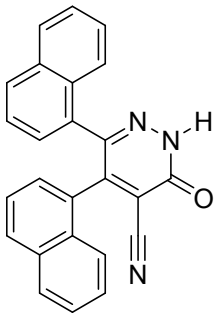
3-oxo-5,6-di-p-tolyl-2,3-dihydropyridazine-4-carbonitrile (2e): M.P.287-289°C; Yield: 5.54g (92%); ^{1}H NMR (300MHz, $CDCl_3$): δ 3.88 (s, 6H, 2 × CH_3), 7.12-7.36 (m, 6H, Ar-H), 7.51-7.65 (d, 1H, Ar-H), 7.73-7.98 (d, 1H, Ar-H), 12.37 (s, 1H, -NH); ^{13}C NMR (75MHz, $CDCl_3$): δ 20.3, 127.1, 127.3, 127.9, 128.1, 128.5, 128.8, 129.3, 130.1, 132.2, 132.8, 136.9, 142.9, 165.6, 167.5; FAB MS: m/z 302 (M+1); Elemental analysis for $C_{19}H_{15}N_3O$: Calcd: C, 75.73; H, 5.02; N, 13.94%; Found: C, 75.68; H, 4.99; N, 13.89%.



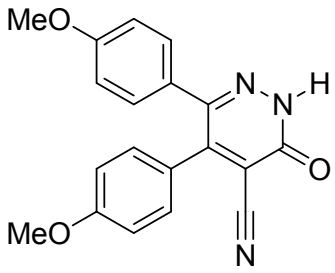
5,6-bis(3-nitrophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2f): M.P.302-305°C; Yield: 6.54g (90%); ^{1}H NMR (300MHz, DMSO): δ 7.07-8.73 (m, 8H, Ar-H), 14.41 (s, 1H, NH); ^{13}C NMR (75MHz, DMSO): δ 123.7, 127.3, 130.5, 135.4, 147.9, 166.0; FAB MS: m/z 364 (M+1); Elemental analysis for $C_{17}H_9N_5O_5$: Calcd: C, 56.20; H, 2.50; N, 19.28%; Found: C, 56.14; H, 2.45; N, 19.23%.



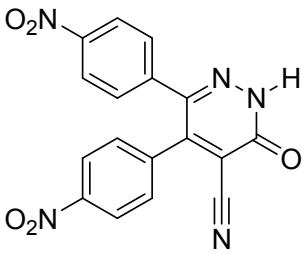
5,6-di(naphthalen-1-yl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2g): M.P.165-170°C; Yield: 7.09g (95%); ^{1}H NMR (300MHz, $CDCl_3$ + DMSO): δ 7.35-7.61 (m, 6H, Ar-H), 7.86-7.89 (d, 2H, Ar-H), 7.99-8.02 (d, 2H, Ar-H), 8.24-8.26 (d, 2H, Ar-H), 9.00-9.03 (d, 2H, Ar-H), 12.18 (s, 1H, NH); ^{13}C NMR (75MHz, $CDCl_3$ + few drops of DMSO): δ 124.3, 125.8, 125.9, 127.2, 127.5, 128.2, 130.2, 131.2, 132.8, 133.6, 165.0, 169.5; FAB MS: m/z 374 (M+1); Elemental analysis for $C_{25}H_{15}N_3O$: Calcd: C, 80.41; H, 4.05; N, 11.25%; Found: C, 80.37; H, 4.01; N, 11.19%.



5,6-bis(4-methoxyphenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2h): M.P. 235-236°C, Yield: 6.45 g (93 %),^{1}H NMR(300MHz, $Si(CH_3)_4$, $CDCl_3$): δ 4.04 (s, 3H, OCH3), 7.19-8.47 (m, 8H, Ar-H),14.19(s,1H,-NH); ^{13}C NMR (75MHz, $CDCl_3$): δ 112.4, 125.9, 134.4, 164.9; FAB MS: m/z 334 (M+1); Elemental analysis for $C_{19}H_{15}N_3O_3$: Calcd: C, 68.46; H, 4.54; N, 12.61%; Found: C, 68.42; H, 4.56; N, 12.59%.

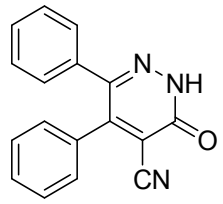


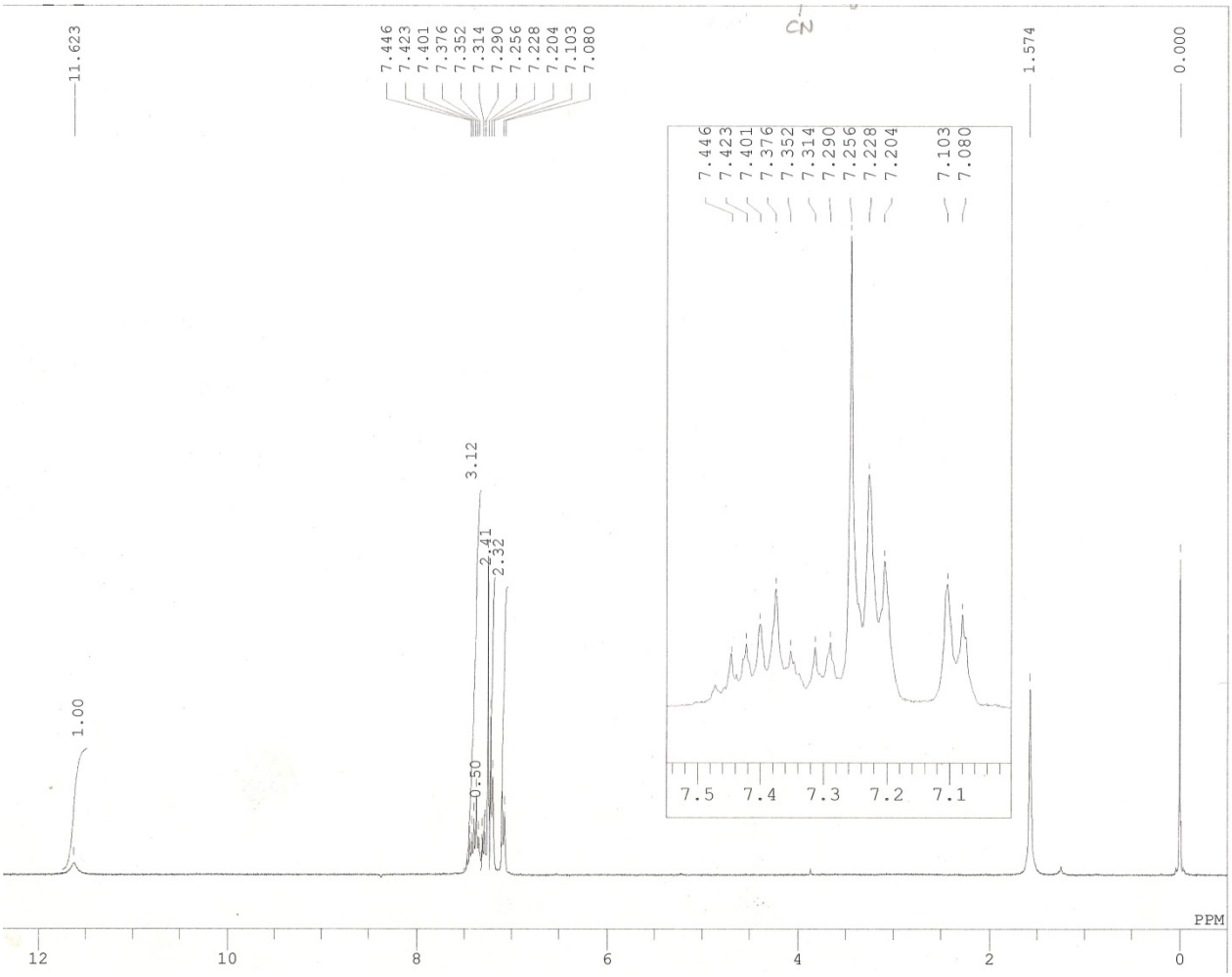
5,6-bis(4-nitrophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2i): M.P. 302-305°C;



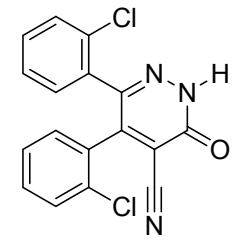
Yield: 6.93g (92 %); ^{1}H NMR (300MHz, $Si(CH_3)_4$, $CDCl_3$): δ 7.65-8.70 (m, 8H, Ar-H); ^{13}CNMR (75MHz, $CDCl_3$,): δ 122.75, 125.39, 126.39, 129.51, 130.08, 130.48, 131.60, 132.13, 132.94, 143.74, 147.71, 166.12, 166.77; FAB MS: m/z 364 (M+1); Elemental analysis for $C_{17}H_9N_5O_5$: Calcd: C, 56.20; H, 2.50; N, 19.28%; Found: C, 56.14; H, 2.45; N, 19.23%.

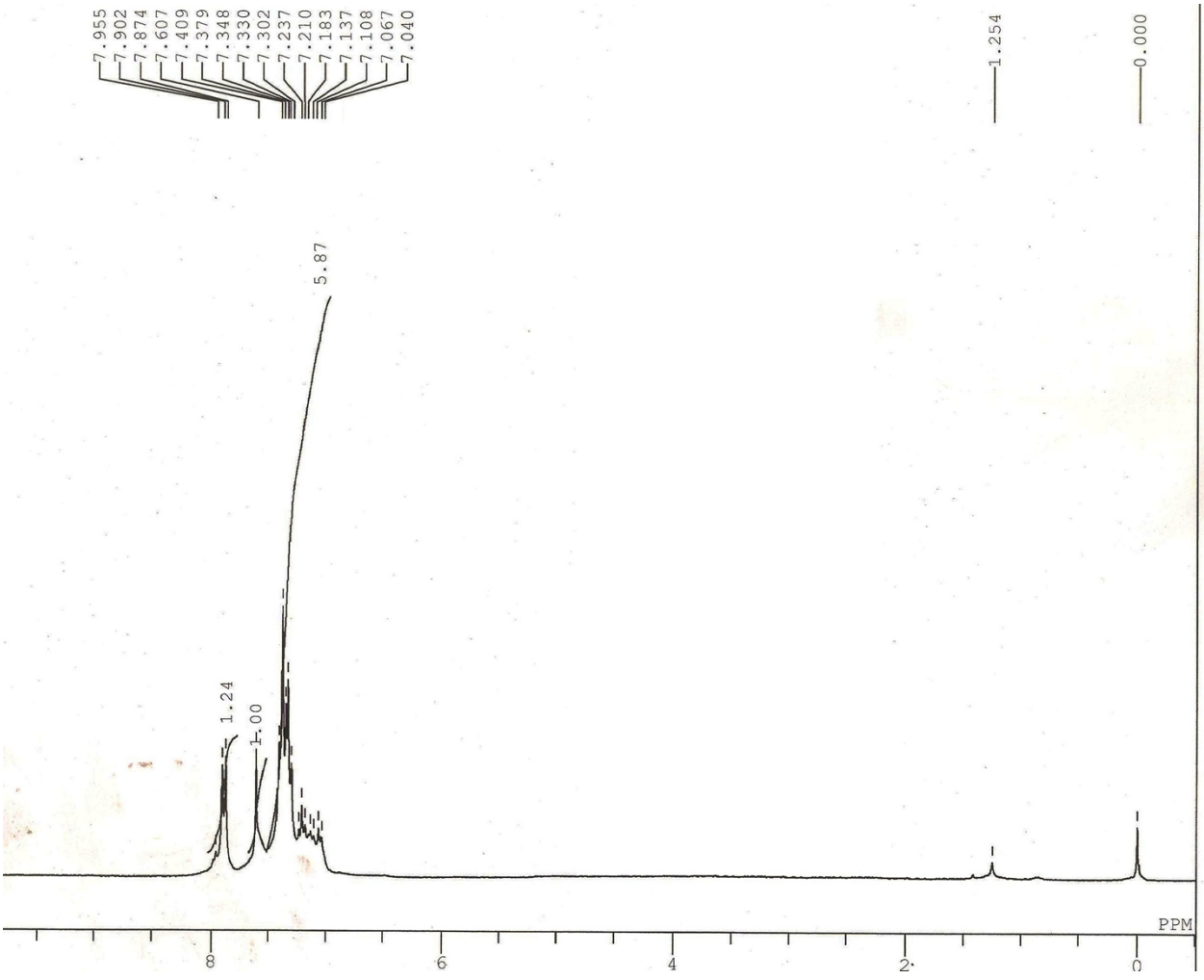
^{1}H NMR of 3-oxo-5,6-diphenyl-2,3-dihydropyridazine-4-carbonitrile (2a):



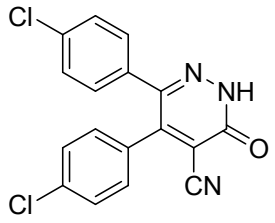


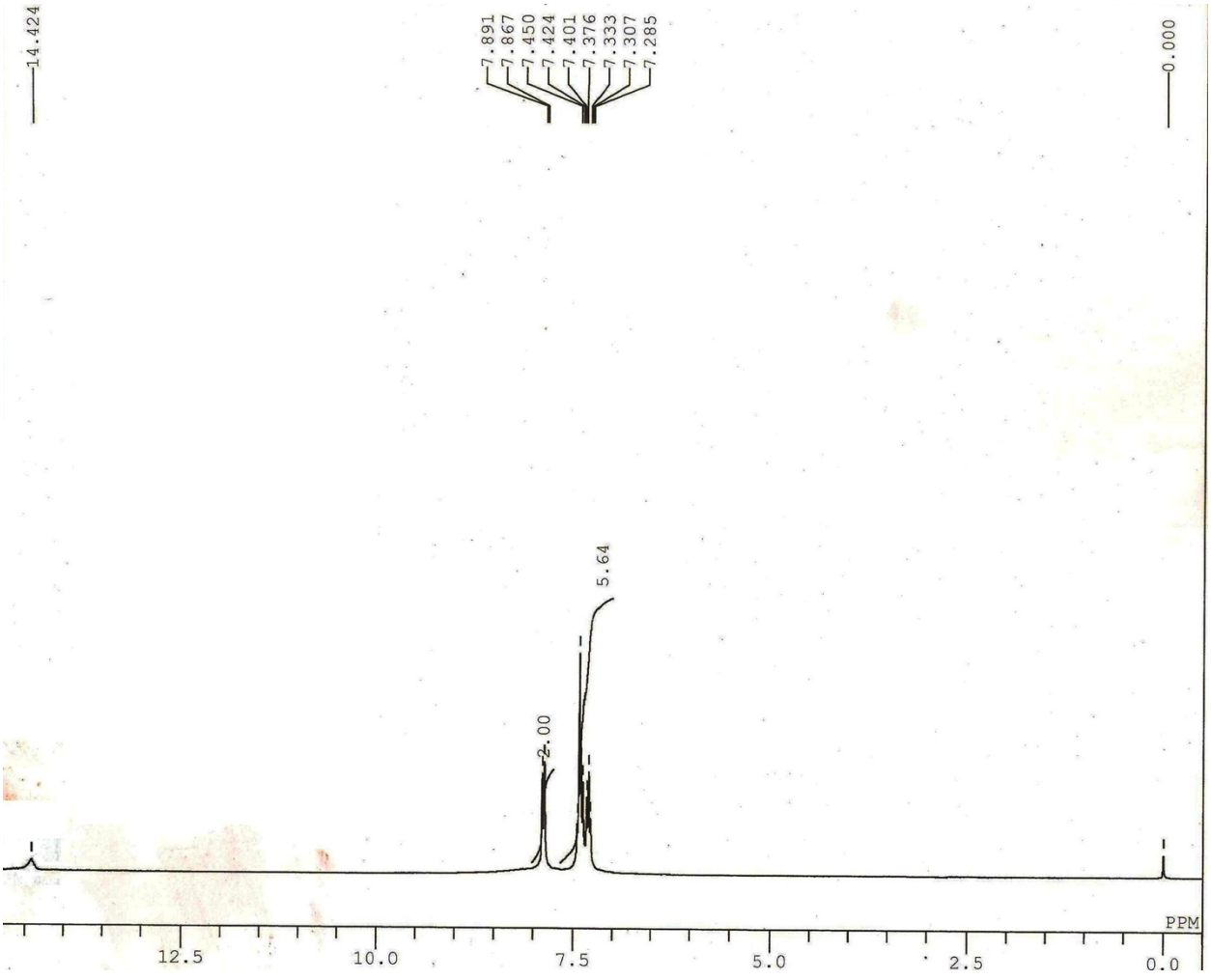
^{1}H NMR of 5,6-bis(2-chlorophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2c):



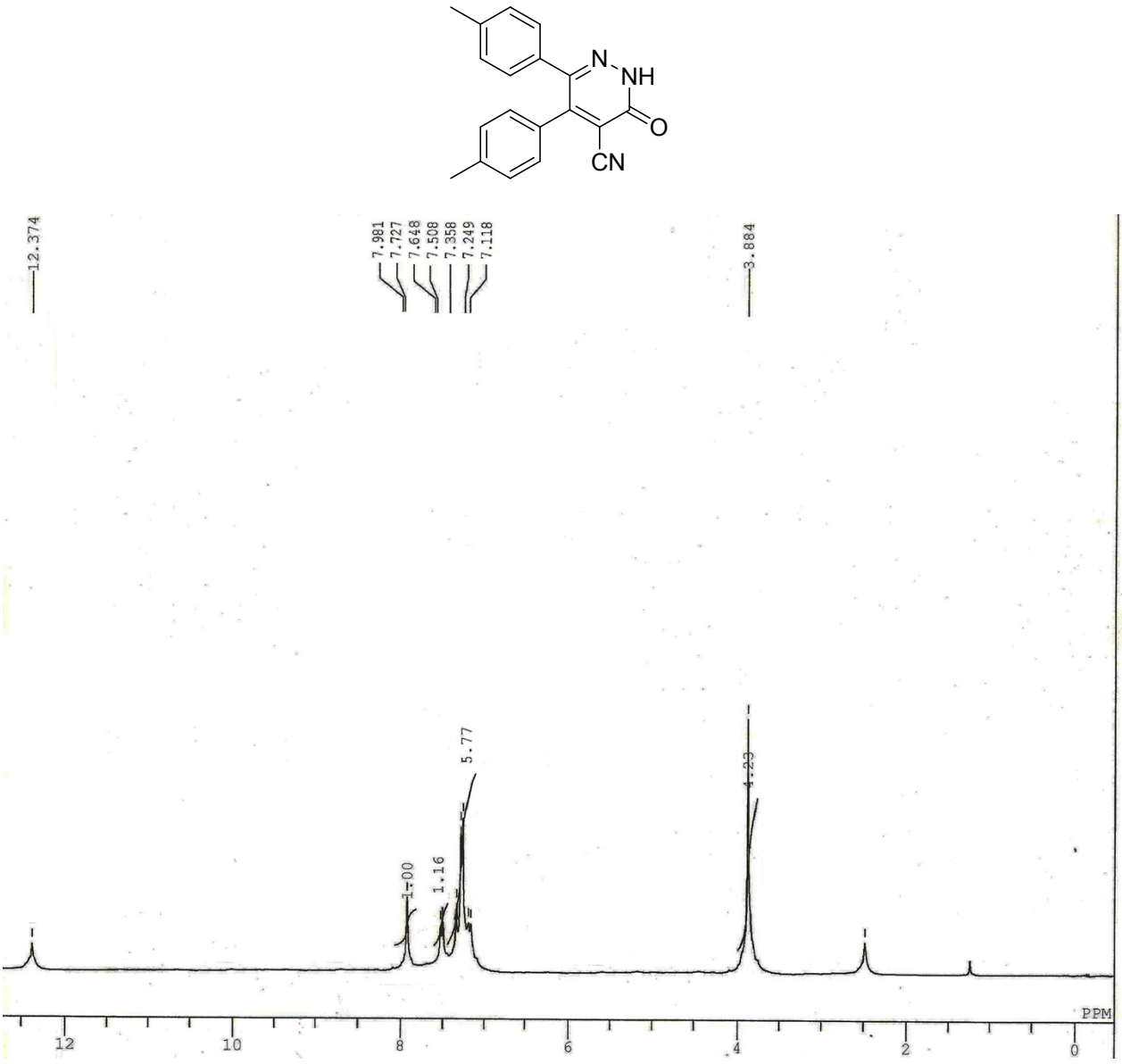


^{1}H NMR of 5,6-bis(4-chlorophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2d):

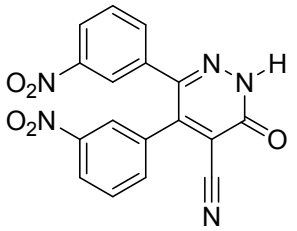


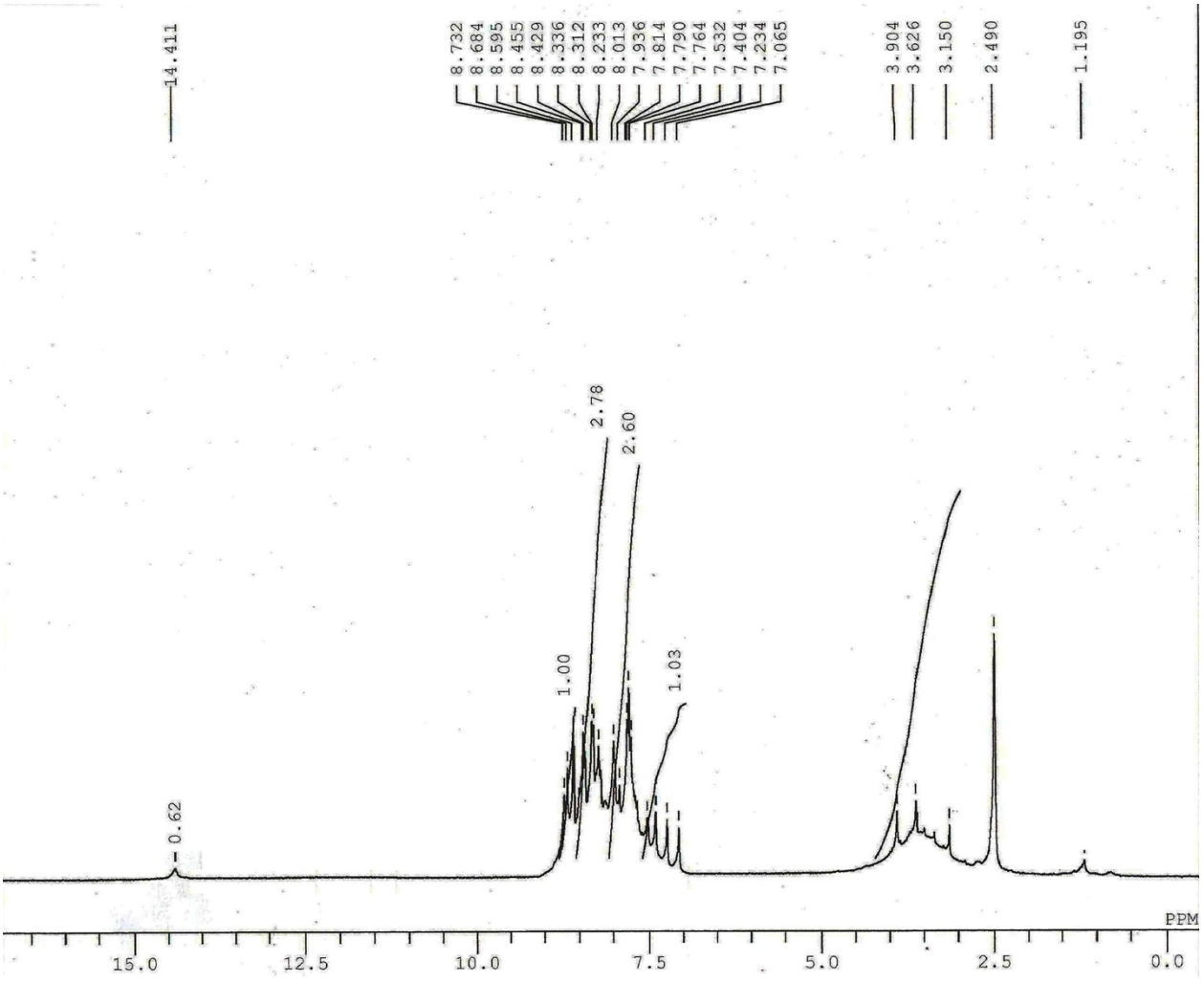


^{1}H NMR of 3-oxo-5,6-di-p-tolyl-2,3-dihydropyridazine-4-carbonitrile (2e):

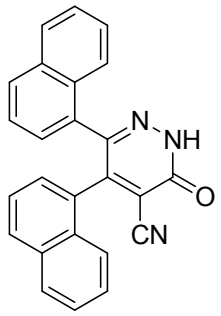


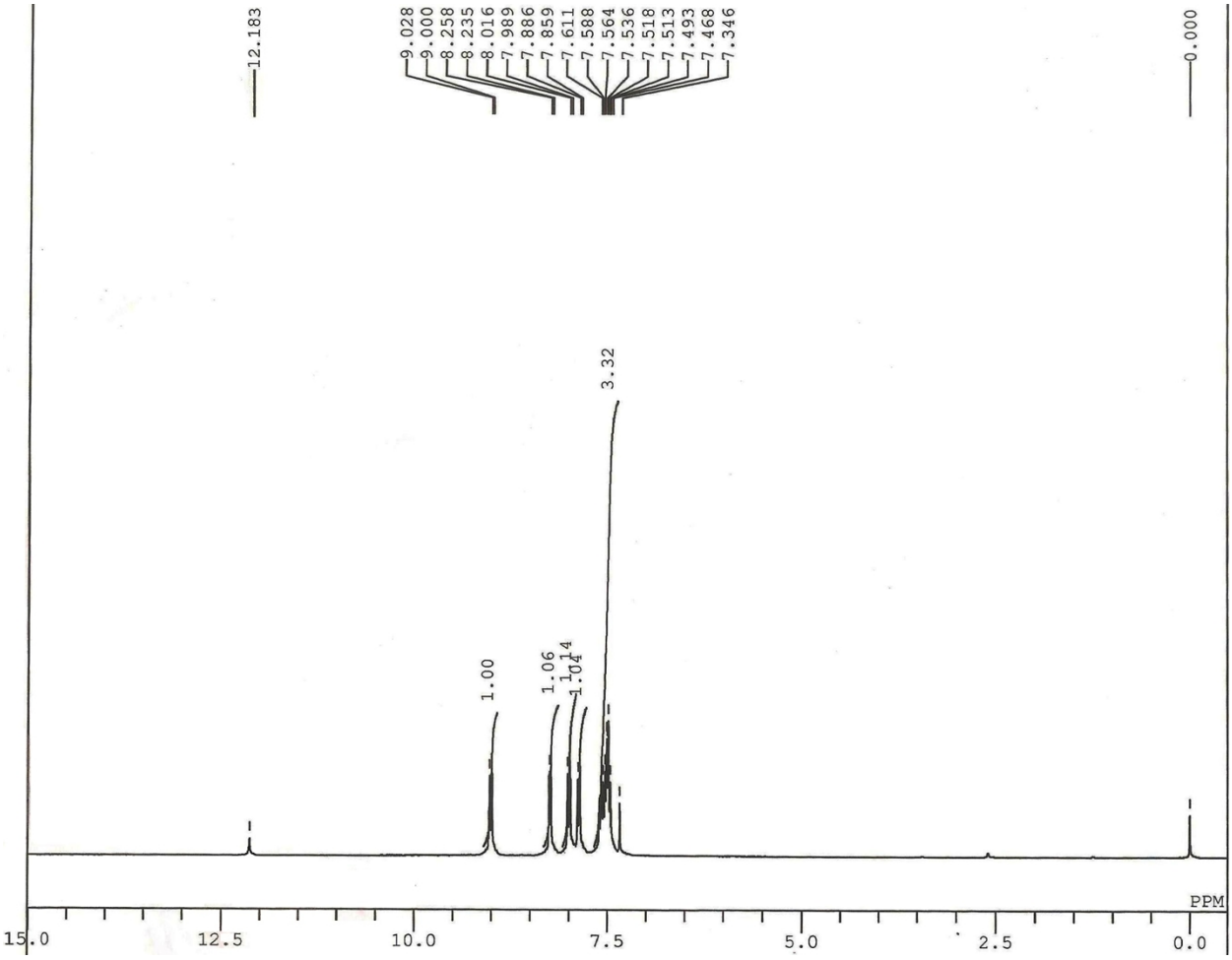
^{1}H NMR of 5,6-bis(3-nitrophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2f)



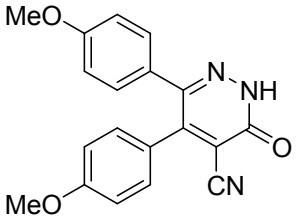


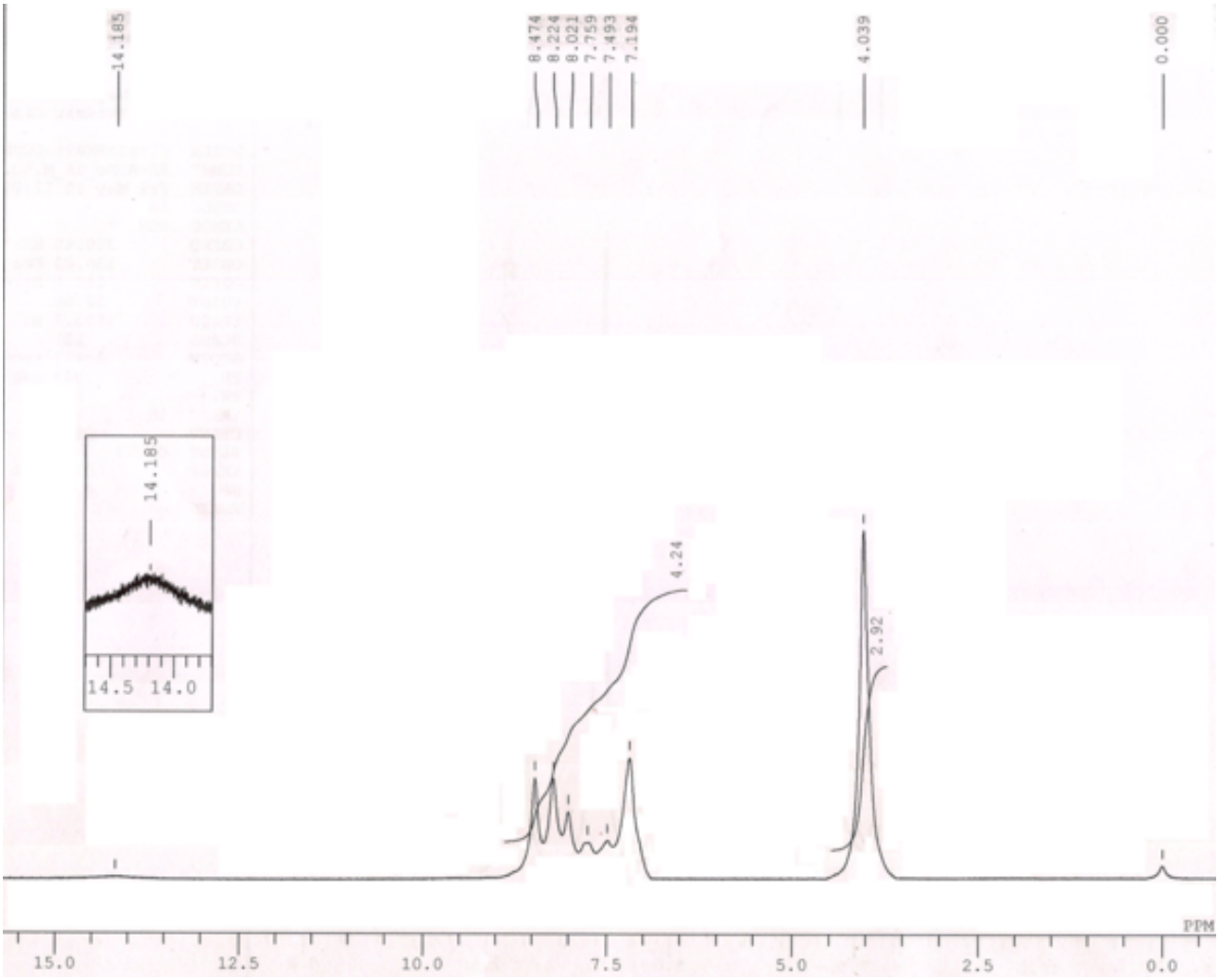
^{1}H NMR of 5,6-di(naphthalen-1-yl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2g):



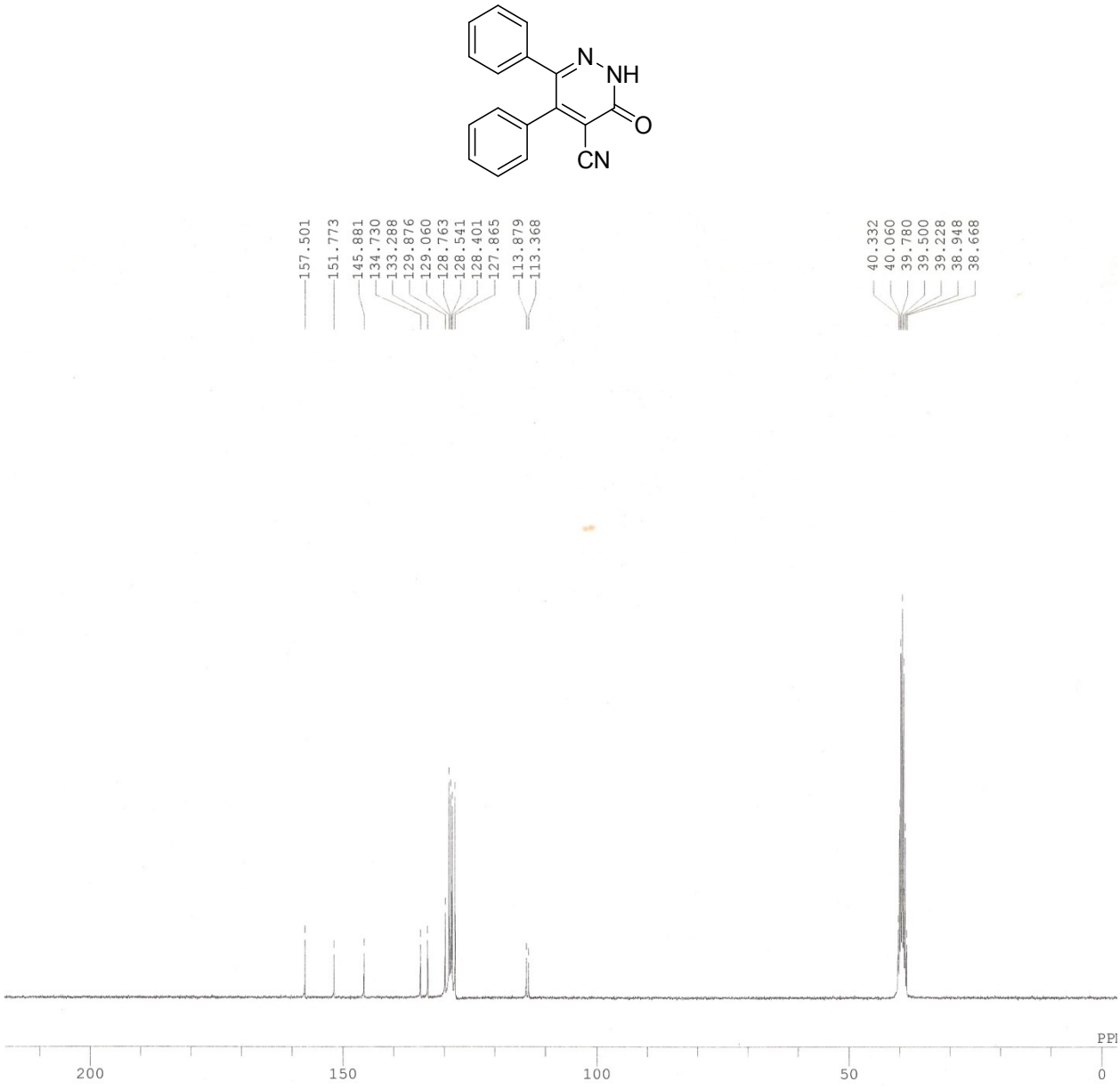


^{1}H NMR of 5,6-bis(4-methoxyphenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2h):

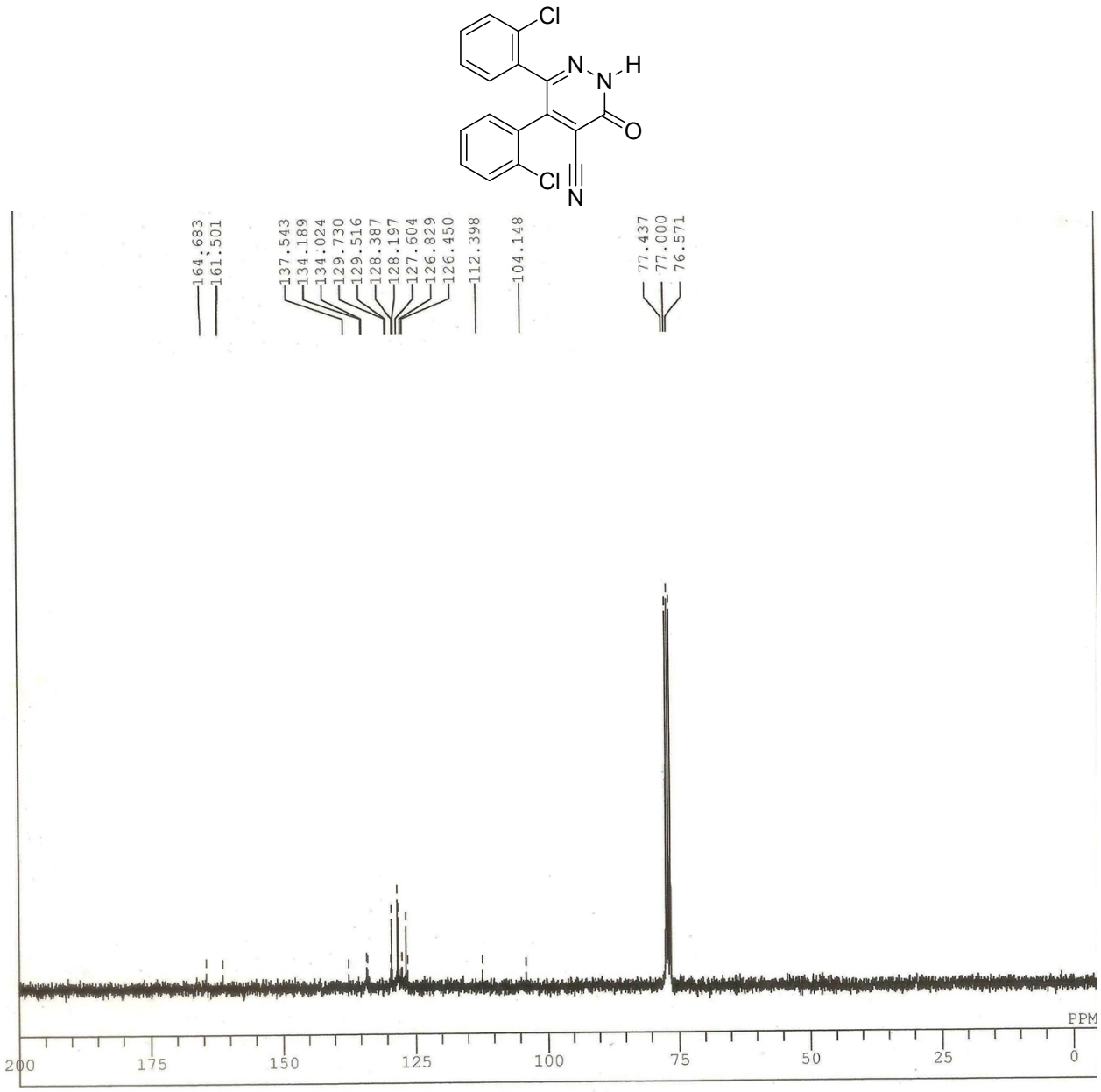




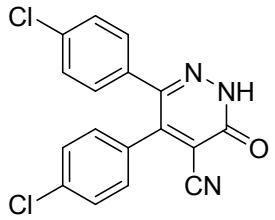
^{13}C NMR of 3-oxo-5,6-diphenyl-2,3-dihydropyridazine-4-carbonitrile (2a):

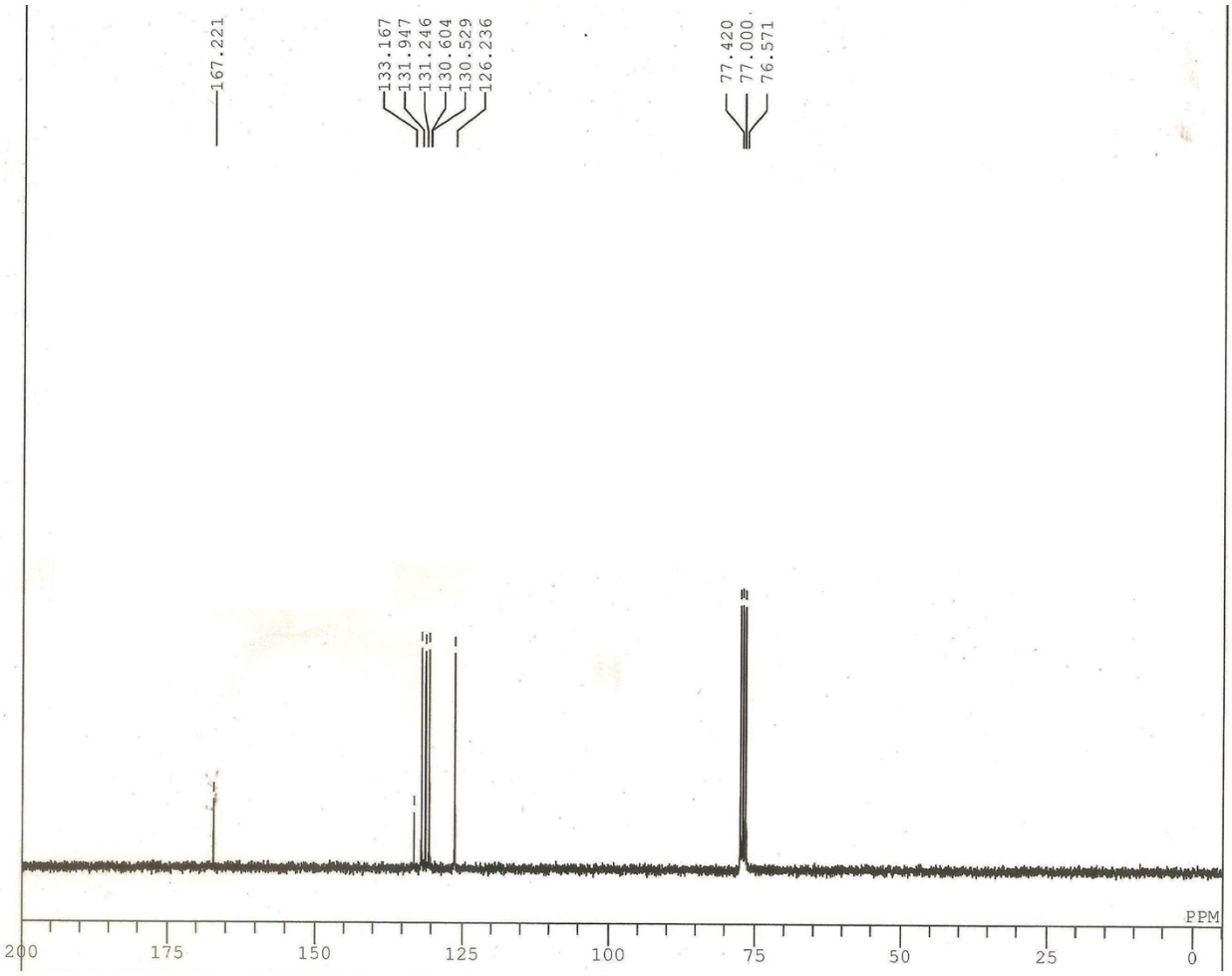


^{13}C NMR of 5,6-bis(2-chlorophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2c):

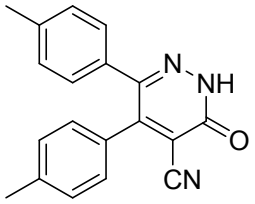


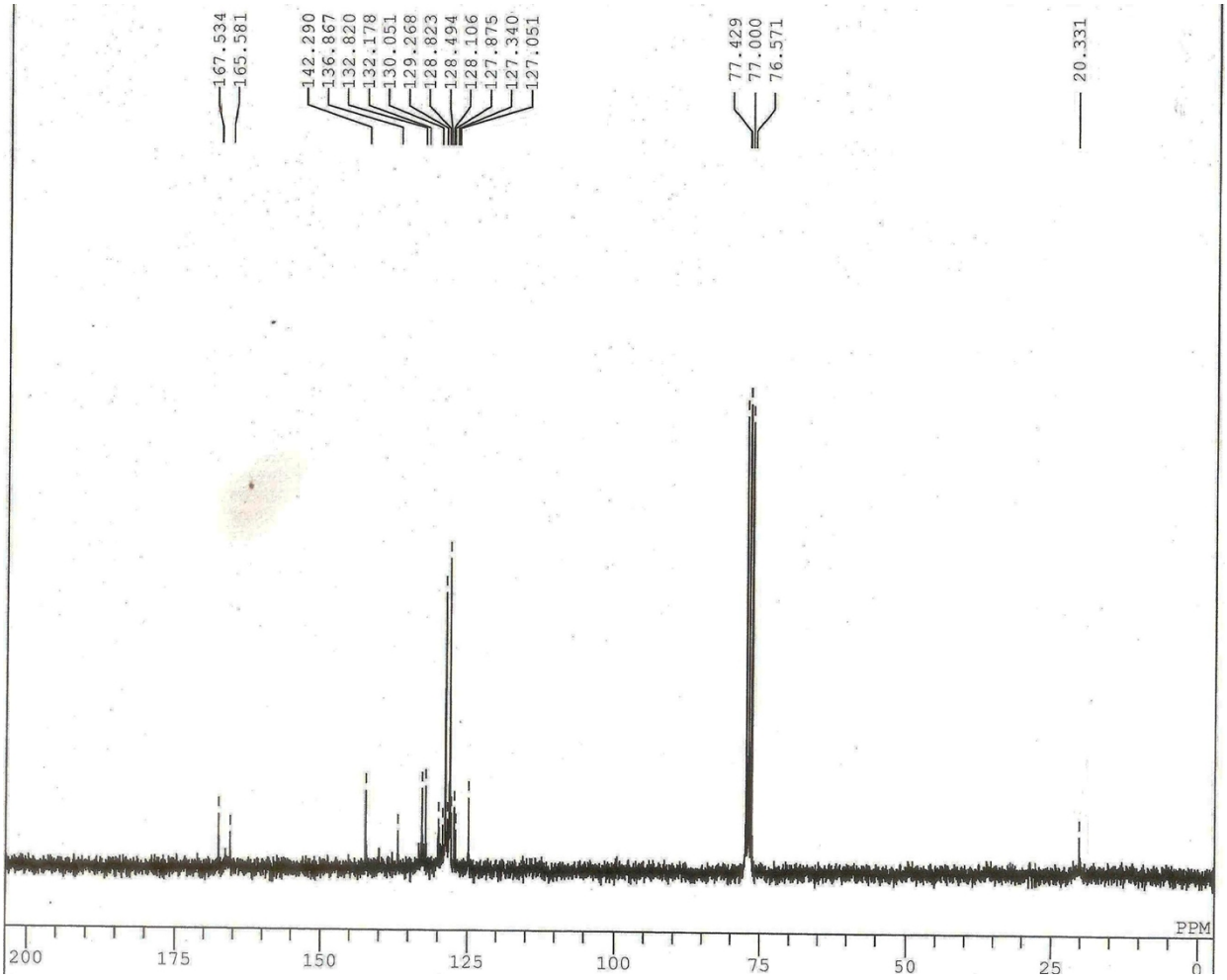
^{13}C NMR of 5,6-bis(4-chlorophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2d):



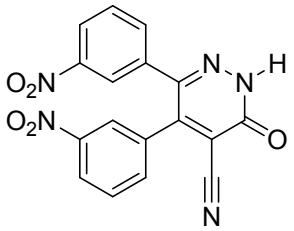


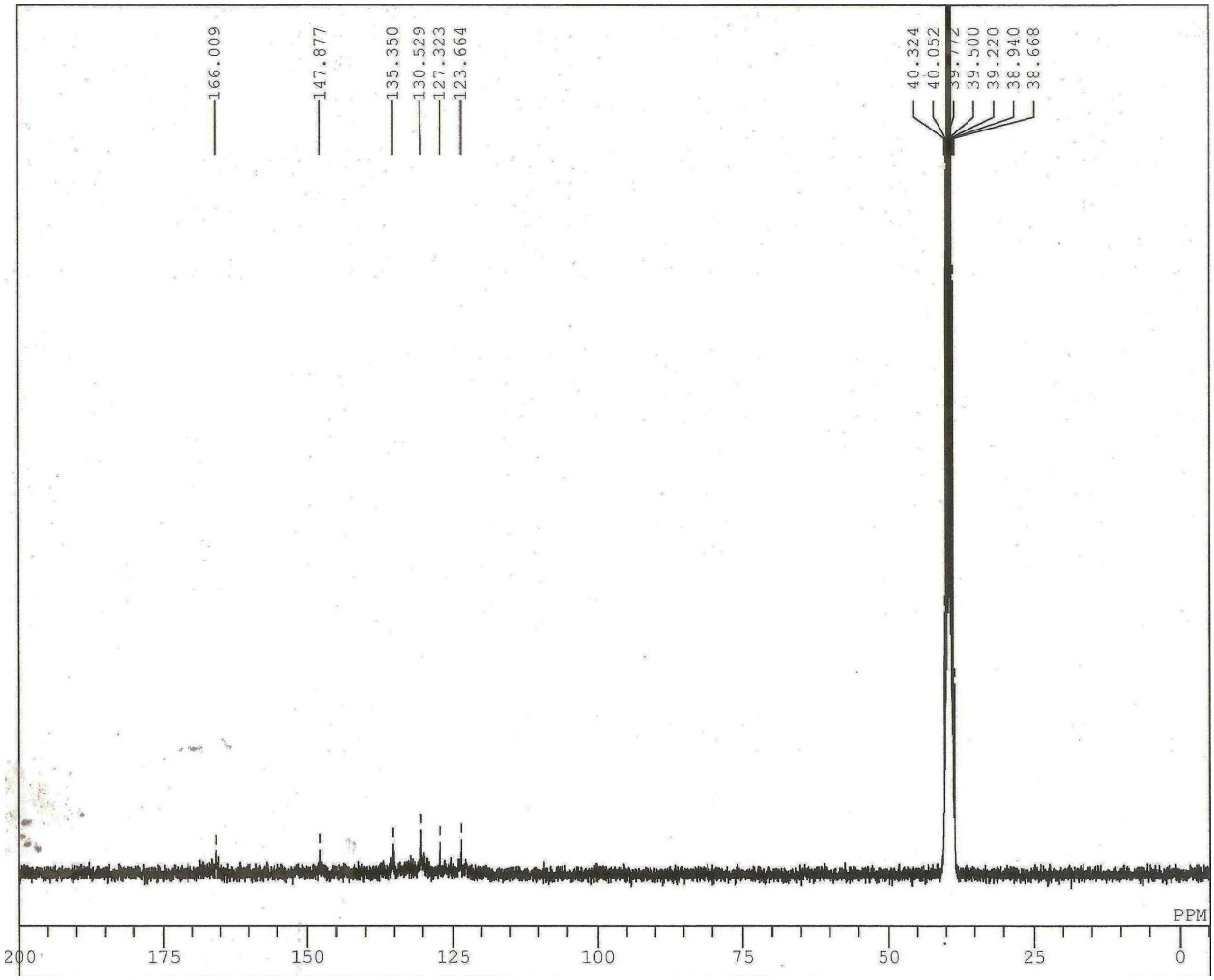
^{13}C NMR of 3-oxo-5,6-di-p-tolyl-2,3-dihydropyridazine-4-carbonitrile (2e):



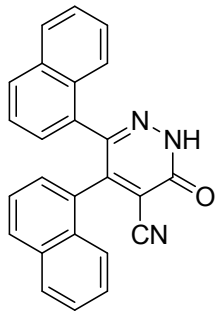


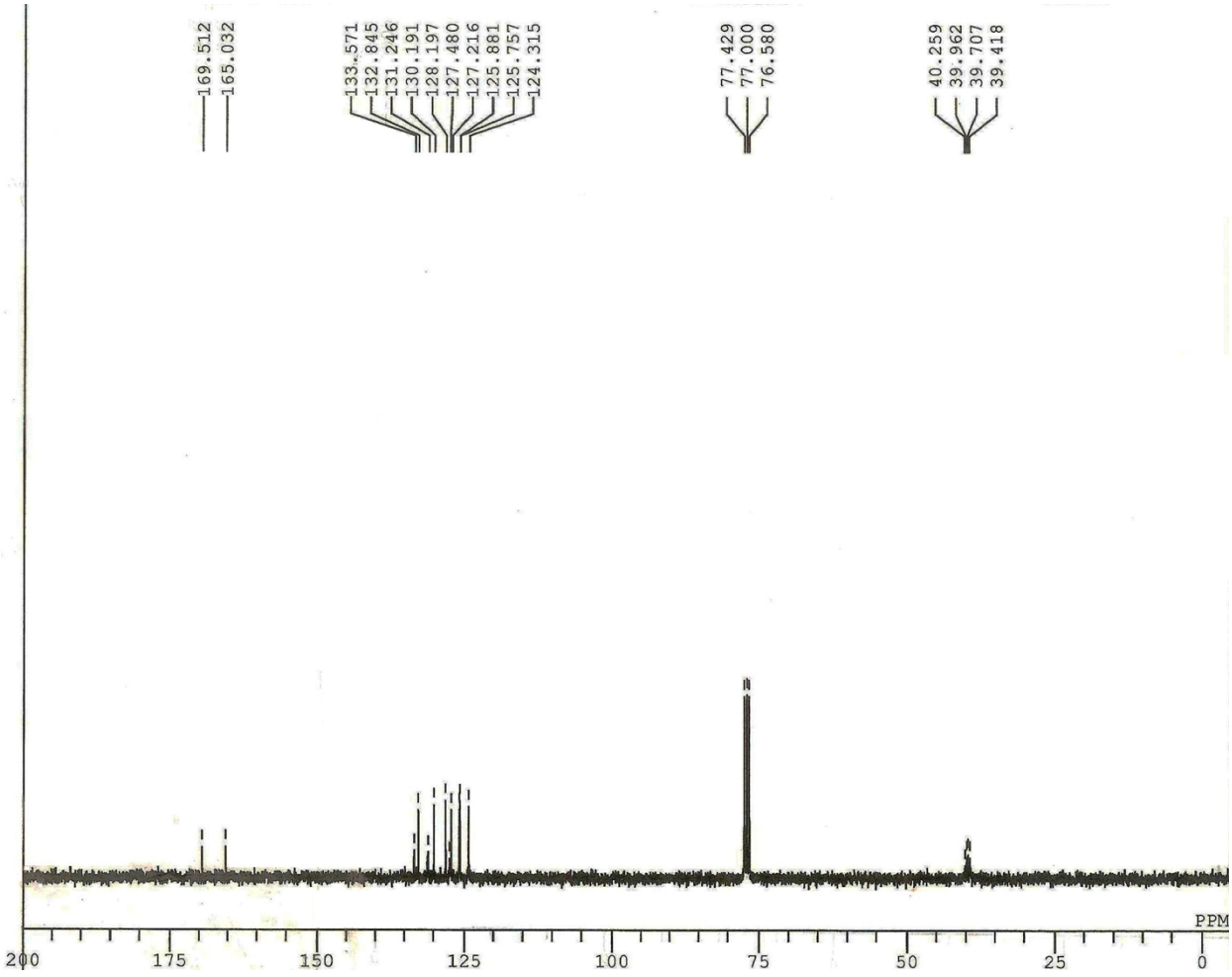
^{13}C NMR of 5,6-bis(3-nitrophenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2f)





^{13}C NMR of 5,6-di(naphthalen-1-yl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2g):





^{13}C NMR of 5,6-bis(4-methoxyphenyl)-3-oxo-2,3-dihydropyridazine-4-carbonitrile (2h)