CHAPTER 5: EXPERIMENTAL DETAILS

5.0 General procedures

Organic solvents were distilled prior to use. Unless otherwise stated, all other organic solvents and reagents were used as received from commercial suppliers. Analytical Thin Layer Chromatography (TLC) was performed using MERCK 25 TLC plates 20 × 20 cm silica gel 60 F_{254} precoated aluminium plate. Spots were developed in an iodine chamber or viewed under ultra violet light and the ethyl acetate:hexane as the solvent or eluent.

Nuclear magnetic resonance (NMR) spectra were taken in deuterated chloroform on the JEOL FT-NMR Lambda 400 MHz and FT-NMR ECA 400 MHz spectrometer with tetramethylsilane (TMS) as internal standard. Chemical shifts are reported in ppm on δ scale, and the coupling constant is given in Hz.

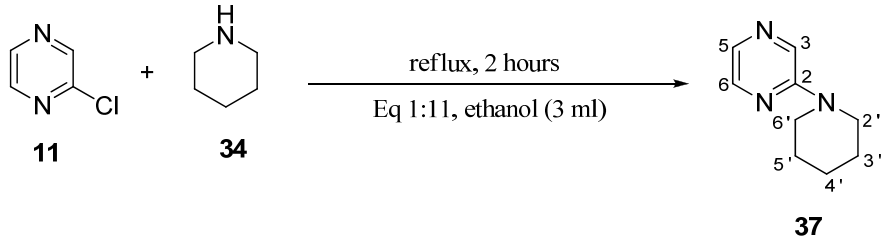
IR spectra were recorded on a Perkin-Elmer 1600 series FT-IR or a Perkin-Elmer RX1 FT-IR spectrophotometer. Melting point was carried out in glass capillaries recorded on a melting point apparatus Fargo MP-ID and are uncorrected. Mass spectroscopic analyses were performed at a Hewlett-Packard HP 6890 Series of GC System with mass selective indicator and GCMS QP5050A Shimadzu.

Fluorescence spectra were recorded by Fluorescence Spectrometer, Model F2000, Hitachi and Luminescence Spectrometer, Model LS 50B, Perkin Elmer. The measurements were recorded at room temperature at the same setting and quartz cells were used.

5.1 Preparation of pyrazine and quinoxaline derivatives

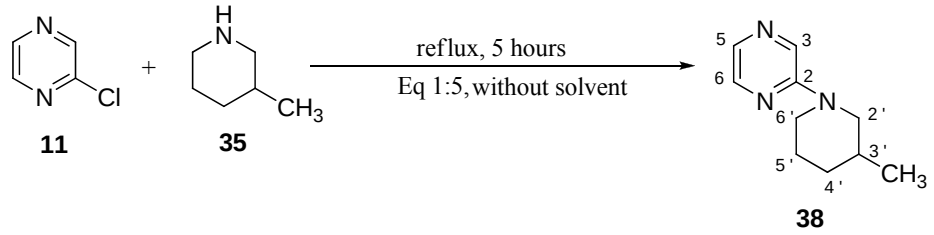
5.1.1 Pyrazine derivatives

5.1.1.1 Preparation of 2-*N*-piperidinopyrazine (37)



2-Chloropyrazine (0.40 ml, 4.516 mmoles) was added to a solution of piperidine (5.00 ml, 0.05 moles) in ethanol and the mixture was refluxed for 2 hours. The mixture was then cooled and the solvent was evaporated off. The residue was dissolved in water and then extracted with diethyl ether (3 × 10 ml). The ether extracts were washed with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Evaporation of the solvent gave the product, a yellowish liquid which was purified by washing with several portions of diethyl ether. (0.5606 g, 76%); IR (ν_{max}, cm^{-1}): 1672 (C=N), 1517 (C=C), 2935 (C-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.05 (1H, d, *J*=1.4 Hz, H-3), 7.96 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.69 (1H, d, *J*=2.6 Hz, H-6), 3.49 (4H, s, H-2', H-6'), 1.55 (6H, s, H-3', H-4', H-5'); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 155.0 (C-2), 141.6 (C-3), 131.7 (C-5), 130.9 (C-6), 45.4 (C-2', C-6'), 25.0 (C-5',C-3'), 24.4 (C-4'); GCMS: Found M^+=163.00; $C_9H_{13}N_3$ requires M^+=163.22

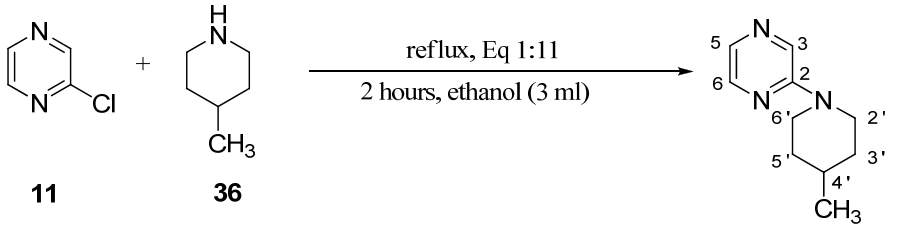
5.1.1.2 Preparation of 2-*N*-(3-methyl)piperidinopyrazine (38)



11 **35** **38**

2-Chloropyrazine (0.20 ml, 2.240 mmoles) was added to 3-methylpiperidine (1.33 ml, 0.011 moles) and refluxed in oil bath at 120°C - 140°C. The reaction was stopped when starting material could no longer be detected (5 hours) on TLC. The mixture was then cooled and dissolved in 10 ml of water. The aqueous layer was extracted with ether (3 × 10 ml). The ethereal layer was washed twice with water (10 ml) and dried over anhydrous sodium sulphate. Filtration and evaporation of the solvent gave the crude mixture which was purified using preparative thin layer chromatography. Ethyl acetate-hexane mixture (1:2) was used as the solvent system. (0.1337 g, 34%); IR (ν_{max}, cm^{-1}): 1673 (C=N), 1517 (C=C), 2927 (C-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.04 (1H, d, *J*=1.4 Hz, H-3), 7.94 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.66 (1H, d, *J*=2.6 Hz, H-6), 4.10 (2H, d, *J*=12 Hz, H-6'), 2.73 (1H, td, *J*=12.6 Hz, 2.9 Hz, H-2'), 2.41 (1H, t, *J*=10.9 Hz, H-2'), 1.42 (4H, m, H-5', H-4'), 1.03 (1H, m, H-3'), 0.86 (3H, d, *J*=6.5 Hz, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 155.0 (C-2), 141.7 (C-3), 131.8 (C-5), 131.1 (C-6), 52.2 (C-6'), 45.0 (C-2'), 33.1 (C-5'), 30.6 (C-4'), 24.8 (C-3'), 19.3 (CH_3); GCMS: Found M^+=177.00; $C_{10}H_{15}N_3$ requires M^+=177.25

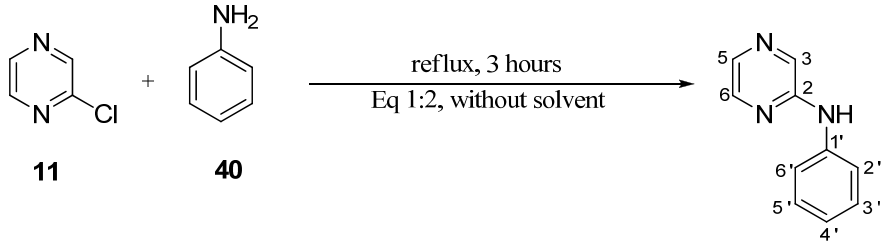
5.1.1.3 Preparation of 2-*N*-(4-methyl)piperidinopyrazine (39)



11 **36** **39**

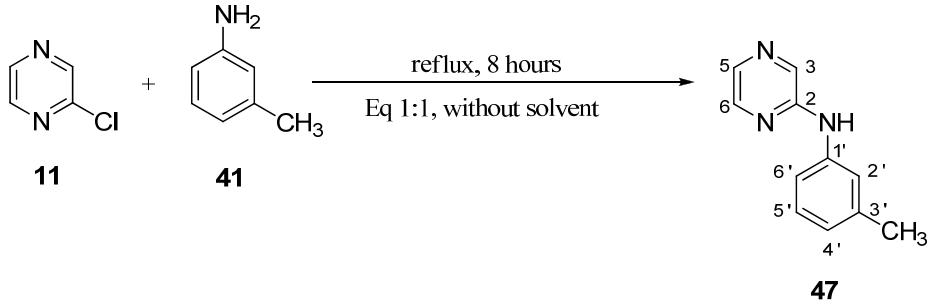
2-Chloropyrazine (0.20 ml, 2.24 mmoles) was added to 4-methylpiperidine (2.20 ml, 0.02 moles). The reaction mixture was refluxed in an oil bath for 2 hours. The mixture was then cooled and the solvent was evaporated off. The reaction mixture was dissolved in water and then extracted with diethyl ether (3 × 10 ml). The ether extracts were washed with water and dried over anhydrous sodium sulphate. Evaporation of solvent gave 2-*N*-(4-methyl)piperidinopyrazine, a yellowish liquid.(0.3049 g, 77%); IR (ν_{max}, cm^{-1}): 1673 (C=N), 1518 (C=C), 2924 (C-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.08 (1H, d, *J*=1.4 Hz, H-3), 7.98 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.69 (1H, d, *J*=2.6 Hz, H-6), 4.22 (2H, d, *J*=12 Hz, H-2'), 2.79 (2H, td, *J*=12.6 Hz, 2.4 Hz, H-6'), 2.60 (2H, d, *J*=12 Hz, H-3'), 1.54 (1H, m, H-4'), 0.99 (2H, m, H-5'), 0.85 (3H, d, *J*=4 Hz, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 155.1 (C-2), 141.7 (C-3), 131.9 (C-5), 131.2 (C-6), 45.0 (C-2', C-6'), 33.6 (C-3', C-5'), 31.0 (C-4'), 21.9 (CH_3); GCMS: Found M^+=177.00; $C_{10}H_{15}N_3$ requires M^+=177.25

5.1.1.4 Preparation of 2-*N*-anilinopyrazine (46)



2-Chloropyrazine (1.20 ml, 0.01 moles) was added to aniline (2.40 ml, 0.02 moles) and heated in an oil bath at 120°C-140°C for 3 hours. The reaction mixture was cooled and dissolved in minimum volume of water (~10 ml). The aqueous layer was extracted with ether (3 × 10 ml). The ether layer was washed with water and dried over anhydrous sodium sulphate. Filtration and evaporation of solvent gave crude product of 2-*N*-anilinopyrazine. Recrystallisation from chloroform gave pure 2-*N*-anilinopyrazine. M.p. 130°C - 132°C, (1.5837 g, 69%); IR (ν_{max}, cm^{-1}): 1624 (C=N), 1521 and 1499 (aromatic C=C), 3281 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.25 (1H, d, *J*=1.4 Hz, H-3), 8.11 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.97 (1H, d, *J*=2.6Hz, H-6), 7.42 (2H, d, *J*=7.8 Hz, H-2', H-6'), 7.34 (2H, t, *J*=7.3 Hz, H-3', H-5'), 7.09 (1H, t, *J*=7.3 Hz, H-4'), 6.74 (1H, s, N-H); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 152.3 (C-2), 141.9 (C-3), 139.1 (C-1'), 134.9 (C-5), 132.9 (C-6), 129.4 (C-2', C-6'), 123.6 (C-4'), 120.3 (C-3', C-5'); GCMS: Found M^+=171.00; $C_{10}H_9N_3$ requires M^+=171.21; X-Ray Diffraction results were summarised in the Chapter 3: Table 3.1

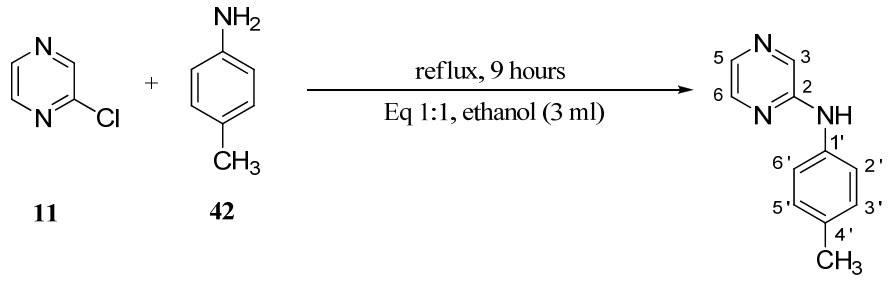
5.1.1.5 Preparation of 2-*N*-(*m*-methyl)anilinopyrazine (47)



11 41 47

2-Chloropyrazine (0.20 ml, 2.24 mmoles) was added to 3-methylaniline (0.24 ml, 2.24 mmoles) and heated in an oil bath for 8 hours. The reaction mixture was cooled and dissolved in minimum volume of water (~10 ml). The aqueous layer was extracted with ether (3 × 10 ml). The ether layer was washed with water and dried over anhydrous sodium sulphate. Filtration and evaporation of solvent gave 2-*N*-(*m*-methyl)anilinopyrazine, a dark brown liquid. (0.2506 g, 60.4%); IR (ν_{max}, cm^{-1}): 1601 (C=N), 1524 and 1492 (aromatic C=C), 3298 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.17 (1H, d, *J*=1.4 Hz, H-3), 8.02 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.88 (1H, d, *J*=2.6 Hz, H-6), 7.13 (3H, m, H-6', H-2', H-5'), 6.84 (1H, d, *J*=7 Hz, H-4'), 6.60 (1H, s, N-H), 2.28 (3H, s, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 152.4 (C-2), 141.9 (C-3), 139.3 (C-1'), 139.0 (C-3'), 134.7 (C-5), 132.8 (C-6), 129.2 (C-6'), 124.5 (C-2'), 121.0 (C-5'), 117.5 (C-4'), 21.5 (CH_3); GCMS: Found M^+=185.00; $C_{11}H_{11}N_3$ requires M^+=185.23

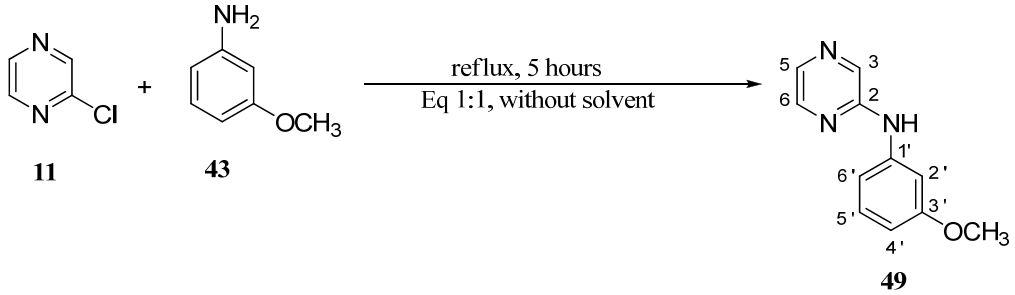
5.1.1.6 Preparation of 2-*N*-(*p*-methyl)anilinopyrazine (48)



11 **42** **48**

2-Chloropyrazine (0.20 ml, 2.24 mmoles), 4-methylaniline (0.2400 g, 2.24 mmoles) and ethanol (3.00 ml) were heated under reflux for 9 hours. The solvent was evaporated off under *vacuo* and 10 ml of water was added to the mixture. The mixture was extracted with ether (3 ×10 ml). The ether extracts were washed with water and dried over anhydrous sodium sulphate. Filtration and evaporation of the solvent gave the crude product, which was separated from traces of unreacted starting material using preparative thin layer chromatography to yield pure 2-*N*-(*p*-methyl) anilinopyrazine using ethyl acetate: hexane as the solvent. M.p. 102°C - 104°C, (0.0768 g, 19%), IR (ν_{max}, cm^{-1}): 1630 (C=N), 1514 and 1412 (aromatic C=C), 3296 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.19 (1H, d, *J*=1.4 Hz, H-3), 8.08 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.93 (1H, d, *J*=2.6 Hz, H-6), 7.26 (2H, d, *J*=8 Hz, H-2', H-6'), 7.16 (2H, d, *J*=8 Hz, H-3', H-5'), 6.62 (1H, s, N-H), 2.34 (3H, s, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 152.6 (C-2), 141.9 (C-1'), 136.3 (C-4'), 134.5 (C-3), 133.6 (C-5), 132.5 (C-6), 129.9 (C-2', C-6'), 121.0 (C-3', C-5'), 20.8 (CH_3); GCMS: Found M^+=185.00; $C_{11}H_{11}N_3$ requires M^+=185.23; X-Ray Diffraction results were summarised in the Chapter 3: Table 3.2

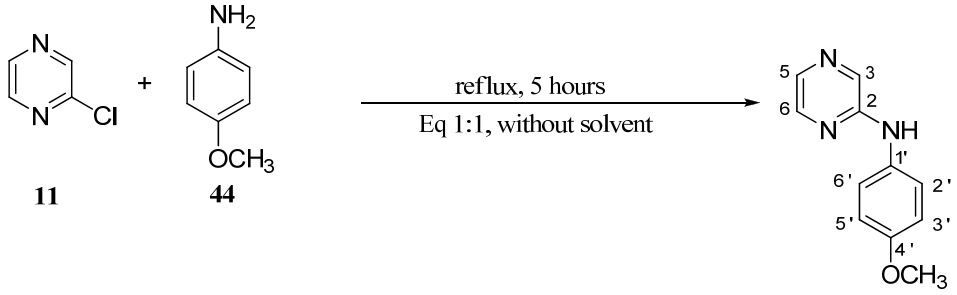
5.1.1.7 Preparation of 2-*N*-(*m*-methoxy)anilinopyrazine (49)



11 **43** **49**

2-Chloropyrazine (1.79 ml, 0.02 moles) was refluxed with *m*-anisidine (2.4630 g, 0.02 moles) for 5 hours at 120°-140°C. The mixture was cooled and extracted twice with chloroform (2 × 10 ml). The organic layer washed with water and dried over anhydrous sodium sulphate. Evaporation of solvent gave the product, 2-*N*-(*m*-methoxy)anilinopyrazine which was recrystallised from chloroform. M.p. 124°C - 126°C, (1.4013 g, 28%), IR (ν_{max}, cm^{-1}): 1605 (C=N), 1525 and 1458 (aromatic C=C), 3301 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.24 (1H, d, *J*=1.4 Hz, H-3), 8.09 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.95 (1H, d, *J*=2.6 Hz, H-6), 7.21 (1H, dd, *J*=8 Hz, 2.2 Hz, H-6'), 7.07 (1H, s, H-2'), 6.93 (1H, dd, *J*=7.8 Hz, 4 Hz, H-5'), 6.69 (1H, s, N-H), 6.61 (1H, dd, *J*=8Hz, 4Hz, H-4'), 3.79 (3H, s, OCH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 160.5 (C-3'), 152.1 (C-2), 141.8 (C-3), 140.2 (C-1'), 134.9 (C-5), 133.1 (C-6), 130.0 (C-6'), 112.4 (C-2'), 108.8 (C-5'), 106.1 (C-4'), 55.3 (OCH_3); GCMS: Found M^+=201.00; $C_{11}H_{11}N_3O$ requires M^+=201.22

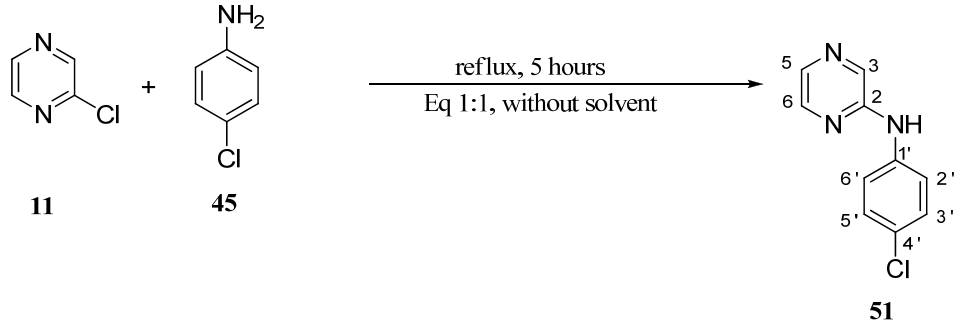
5.1.1.8 Preparation of 2-*N*-(*p*-methoxy)anilinopyrazine (50)



11 **44** **50**

2-Chloropyrazine (0.89 ml, 0.01 moles) and *p*-anisidine (1.2315 g, 0.01 moles) were refluxed for 5 hours at 120°C-140°C. The mixture was cooled. Water (10 ml) was added to the cooled mixture and the organic layer was extracted with chloroform (3×10 ml). The chloroform layer was washed with water and dried over anhydrous sodium sulphate. Chloroform was removed under reduced pressure, leaving a black residue of 2-*N*-(*p*-methoxy)anilinopyrazine which was recrystallised from chloroform. M.p. 118°C - 120°C, (1.4494 g, 72%), IR (ν_{max}, cm^{-1}): 1508 and 1428 (aromatic C=C), 3210 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.05 (1H, d, *J*=1.4 Hz, H-3), 7.96 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.83 (1H, d, *J*=2.6 Hz, H-6), 7.25 (2H, d, *J*=8 Hz, H-2', H-6'), 6.83 (2H, d, *J*=8 Hz, H-3', H-5'), 6.67 (1H, s, N-H), 3.74 (3H, s, OCH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 156.7 (C-4'), 153.0 (C-2), 141.6 (C-3), 134.0 (C-5), 132.2 (C-6), 131.6 (C-1'), 123.8 (C-2', C-6'), 114.6 (C-3', C-5'), 55.5 (OCH_3); GCMS: Found M^+=201.00; $C_{11}H_{11}N_3O$ requires M^+=201.22

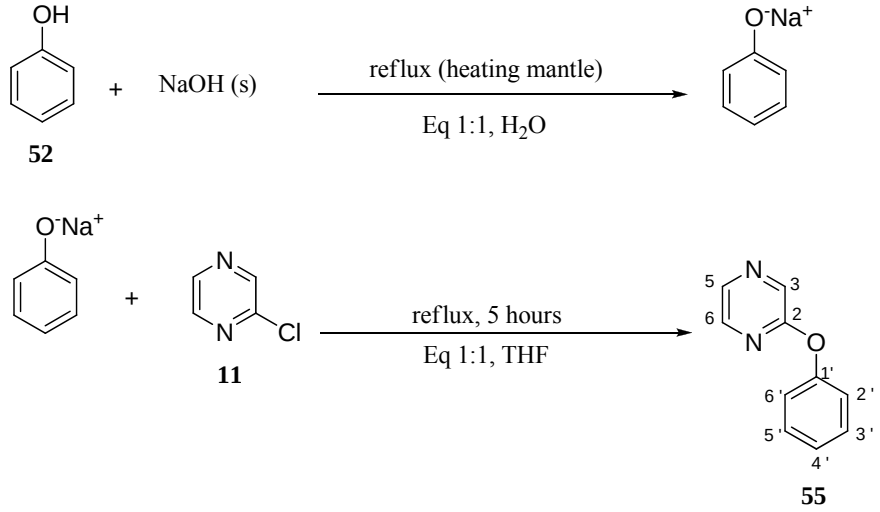
5.1.1.9 Preparation of 2-*N*-(*p*-chloro)anilinopyrazine (51)



11 **45** **51**

2-Chloropyrazine (1.79 ml, 0.02 moles) was added to *p*-chloroaniline (2.5506 g, 0.02 moles) and heated in oil bath at 120°C- 140°C. The reaction was stopped when starting material could no longer be detected on TLC (~5 hours). The mixture was cooled and water was added to the mixture. The mixture was extracted with chloroform (3 × 10 ml). The organic layer was washed with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Removal of the solvent followed by recrystallisation using chloroform gave the pure product, 2-*N*-(*p*-chloro)anilinopyrazine as yellowish crystals. M.p. 144°C - 146°C, (2.5690 g, 62%), IR (ν_{max}, cm^{-1}): 1602 (C=N), 1522 and 1491 (aromatic C=C), 3246 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.12 (1H, d, *J*=1.4 Hz, H-3), 8.04 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 7.93 (1H, d, *J*=2.6 Hz, H-6), 7.34 (2H, d, *J*=6.8 Hz, H-2', H-6'), 7.23 (2H, d, *J*=6.8 Hz, H-3', H-5'), 6.49 (1H, s, N-H); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 151.8 (C-2), 141.7 (C-3), 137.8 (C-4'), 135.2 (C-5), 133.2 (C-6), 129.3 (C-3', C-5'), 128.2 (C-1'), 121.2 (C-2', C-6'); GCMS: Found M^+=205.00; $C_{10}H_8N_3Cl$ requires M^+=205.64; X-Ray Diffraction results were summarised in the Chapter 3: Table 3.3

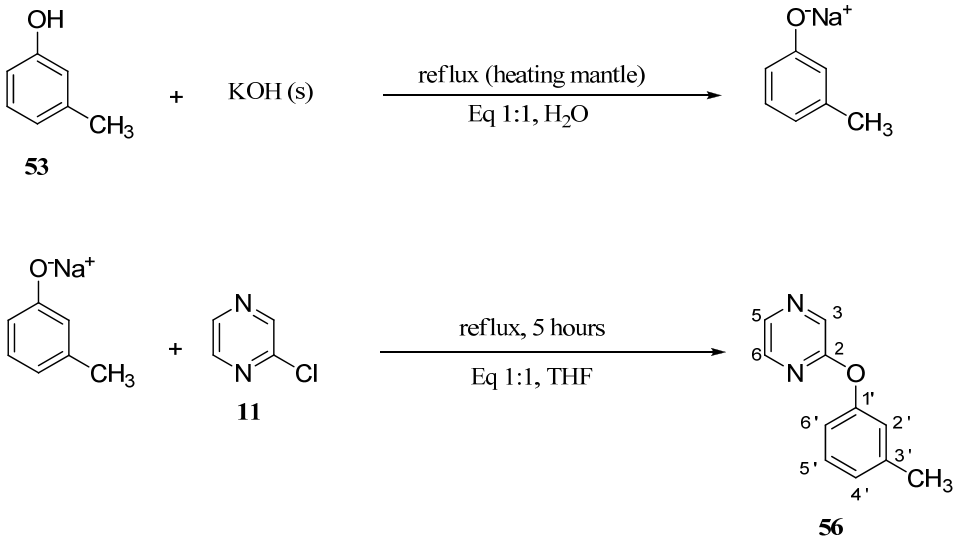
5.1.1.10 Preparation of 2-phenoxypyrazine (55)



Phenol (0.9411 g, 0.01 moles) was added to sodium hydroxide pellet (0.4 g, 0.01 moles) in minimum volume of water (5 ml). The mixture was heated until a dry white solid was formed. 2-Chloropyrazine (0.89 ml, 0.01 ml) in THF (5 ml) was then added to the dry white solid and refluxed for 5 hours. The mixture was cooled to room temperature and the solvent was removed under *vacuo*. Water (10 ml) and 5% sodium hydroxide solution (5 ml) was added to reaction mixture followed by extraction with chloroform (3 × 10 ml). The organic layer was washed twice with water (2 × 10 ml) and dried over anhydrous sodium sulphate. Filtration and evaporation of chloroform solvent gave crude product which was purified using column chromatography on silica using ethyl acetate-hexane mixture (1:3) as the solvent. Evaporation of the solvent gave 2-phenoxypyrazine. M.p. 52°C - 54°C, (0.2182 g, 76%), IR (ν_{max}, cm^{-1}): 1578 (C=N), 1534 and 1405 (aromatic C=C), 1284 and 1007 (C-O); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.40 (1H, d, *J*=1.4 Hz, H-3), 8.23 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 8.07 (1H, d, *J*=2.6 Hz, H-6), 7.38 (2H, dd, *J*=4 Hz, 2.2 Hz, H-2', H-6'), 7.21 (1H, t, *J*=7.5 Hz, H-4'), 7.12 (2H, d, *J*=8 Hz, C-3', C-5'); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 160.1 (C-1'), 152.9 (C-2), 141.0 (C-3), 138.4 (C-5), 135.8 (C-6), 129.7 (C-2', C-6'), 125.41 (C-

4'), 121.1 (C-3', C-5'); GCMS: Found M^+=172.00; $C_{10}H_8N_2O$ requires M^+=172.18; X-Ray Diffraction results were summarised in the Chapter 3: Table 3.4

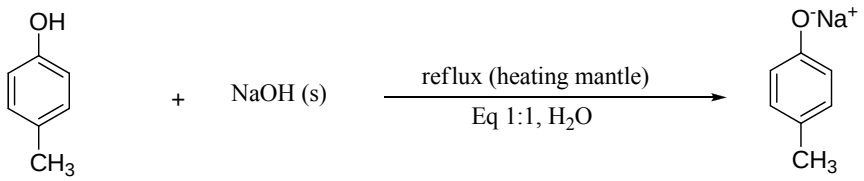
5.1.1.11 Preparation of 2-(*m*-methyl)phenoxypyrazine (56)



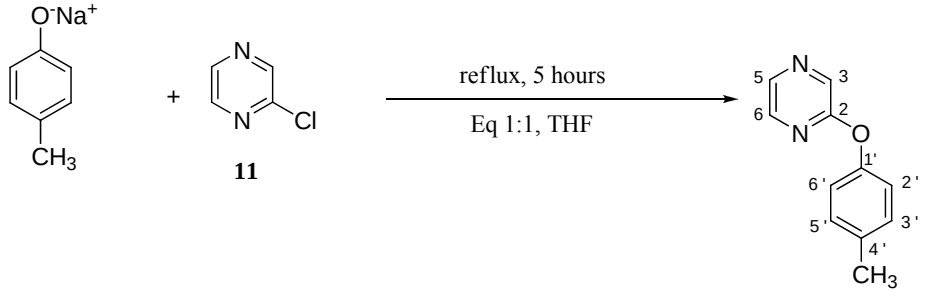
Potassium hydroxide pellet (1.1175 g, 0.02 moles) in water (5 ml) was stirred with *m*-cresol (2.09 ml, 0.02 moles) and the mixture was heated to yield a brown solid. 2-Chloropyrazine (1.79 ml, 0.02 moles) in THF (5 ml) was added to reaction mixture and refluxed for 5 hours. The mixture was cooled and the solvent was evaporated off. Water (10 ml) followed by 5% sodium hydroxide solution (5 ml) was added to the mixture and extracted with chloroform (3 × 10 ml). The organic extracts were washed with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Removal of chloroform followed by recrystallisation in ethyl acetate gave the pure 2-(*m*-methyl)phenoxypyrazine. M.p. 54°C - 56°C, (1.1154 g, 30%), IR (ν_{max}, cm^{-1}): 1612 (C=N), 1531 and 1466 (aromatic C=C), 1284 and 1007 (C-O); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.33 (1H, d, *J*=1.4 Hz, H-3), 8.17 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 8.03 (1H, d, *J*=2.6 Hz, H-6), 7.18 (1H, dd, *J*=11.8 Hz, 7.5 Hz, H-6'), 6.98 (1H, s, H-2'), 6.87 (2H, m, C-5', C-4'), 2.30 (3H, s, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 160.2 (C-1'), 152.9 (C-2), 141.1 (C-3), 140.0 (C-3'), 138.2 (C-5), 135.7 (C-6), 129.4

(C-6'), 126.2 (C-2'), 121.7 (C-5'), 118.1 (C-4'), 21.3 (CH_3); GCMS: Found M^+=186.00; $C_{11}H_{10}N_2O$ requires M^+=186.21

5.1.1.12 Preparation of 2-(*p*-methyl)phenoxypyrazine (57)



54



11

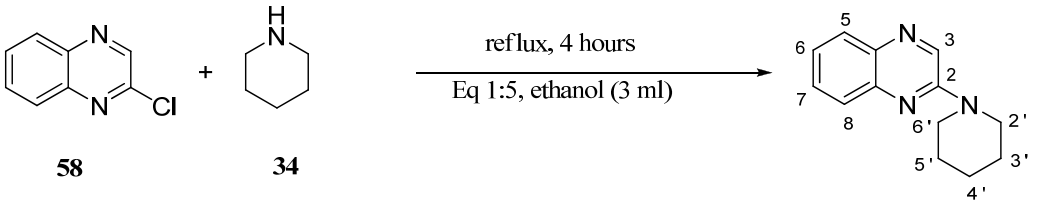
57

Sodium hydroxide pellet (0.2000 g, 5 mmoles) was dissolved in water (5 ml) and *p*-cresol (0.52 ml, 5 mmoles) was added to the solution and stirred for 10 minutes at room temperature. The mixture was then heated to form a brown solid. 2-Chloropyrazine (0.45 ml, 5 × 10^{-3} moles) in THF (5 ml) was added to reaction mixture and refluxed for 5 hours. The mixture was cooled and the solvent was evaporated off. The mixture was shaken thoroughly with water (5 ml) followed by addition of 5% of sodium hydroxide solution (5 ml). The mixture was then extracted three times with chloroform (3 × 10 ml). The combined chloroform layer was washed with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Evaporation of chloroform gave crude product which further recrystallised from ethyl acetate gave pure 2-(*p*-methyl) phenoxypyrazine. M.p. 44°C - 46°C, (0.2059 g, 22%), IR (ν_{max}, cm^{-1}): 1580 (C=N), 1507 and 1404 (aromatic C=C), 1287 and 1006 (C-O); ^{1}H NMR (ppm,

400 MHz, $CDCl_3$) δ_H: 8.45 (1H, d, *J*=1.4 Hz, H-3), 8.22 (1H, dd, *J*=2.6 Hz, 1.4 Hz, H-5), 8.08 (1H, d, *J*=2.6 Hz, H-6), 7.21 (2H, d, *J*=8 Hz, H-2', H-6'), 7.02 (2H, d, *J*=8 Hz, H-3', H-5'), 2.36 (3H, s, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 160.3 (C-1'), 150.5 (C-2), 140.9 (C-3), 138.1 (C-5), 135.6 (C-6), 135.0 (C-4'), 130.2 (C-3', C-5'), 120.9 (C-2', C-6'), 20.7 (CH_3); GCMS: Found M^+=186.00; $C_{11}H_{10}N_2O$ requires M^+=186.21

5.1.2 Quinoxaline derivatives

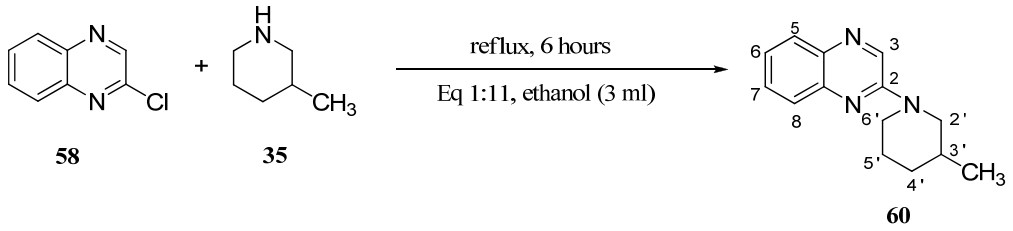
5.1.2.1 Preparation of 2-*N*-piperidinoquinoxaline (59)



58 **34** **59**

2-Chloroquinoxaline (0.3292 g, 0.002 moles) was added to a solution of piperidine (0.99 ml, 0.01 moles) in ethanol (3 ml) and the mixture was heated under reflux for 4 hours. The mixture was cooled and the solvent was evaporated off. A minimum volume of water was added and the organic product was extracted twice with diethyl ether (2 × 10 ml). The ether extracts were washed with water (2 × 10 ml) and dried over anhydrous sodium sulphate. Evaporation of the solvent gave the product, a yellowish solid. M.p. 54°C- 56°C, (0.3545 g, 83%), IR (ν_{max}, cm^{-1}): 1553 (C=N), 1582 (C=C), 2973 (C-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.57 (1H, s, H-3),7.83 (1H, d, *J*=8 Hz, H-8), 7.64 (1H, dd, *J*=8 Hz, H-5), 7.52 (1H, m, H-7), 7.33 (1H, m, H-6), 3.76 (4H, s, H-2', H-6'), 1.71 (6H, s, H-3', H-4', H-5'); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 152.4 (C-2), 141.8 (C-10), 136.4 (C-9), 136.0 (C-3), 129.9 (C-8), 128.5 (C-5), 126.3 (C-7), 124.2 (C-6), 45.8 (C-2', C-6'), 25.6 (C-3', C-5') 24.6 (C-4'); GCMS: Found M^+=213.00; $C_{13}H_{15}N_3$ requires M^+=213.28

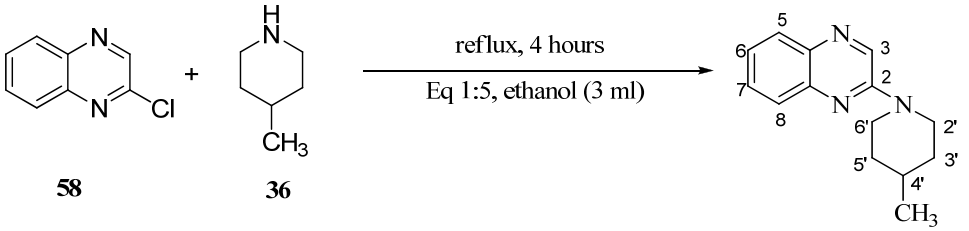
5.1.2.2 Preparation of 2-*N*-(3-methyl)piperidinoquinoxaline (60)



58 **35** **60**

2-Chloroquinoxaline(0.3292 g, 0.002 moles), 3-methylpiperidine (2.58 ml, 0.022 moles) and ethanol (3.00 ml) were heated under reflux until no longer starting material can be detected by thin layer chromatography (~6 hours). The mixture was cooled and ethanol was evaporated under *vacuo.* The residue was then dissolved in minimum volume of water (10 ml). The residual slurry was extracted with ether (3 × 10ml). The ethereal layer was washed with water and dried over anhydrous sodium sulphate. Evaporation of ether gave yellowish solid. M.p. 70°C- 72°C, (0.4342 g, 96%), IR (ν_{max}, cm^{-1}): 1552 (C=N), 1582 (C=C), 2869 (C-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.50 (1H, s, H-3), 7.76 (1H, d, *J*=8 Hz, H-8), 7.58 (1H, d, *J*=8 Hz, H-5), 7.45 (1H, m, H-7), 7.26 (1H, m, H-6), 4.33 (2H, t, *J*=3.4 Hz, H-6'), 2.89 (1H, td, *J*=12 Hz, 2.9 Hz, H-2'), 2.56 (1H, t, *J*=2.4 Hz, H-2'), 1.52 (4H, m, H-4', H-5'), 1.13 (1H, m, H-3'), 0.91 (3H, d, *J*=8 Hz, CH_3) ; ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 152.4 (C-2), 141.9 (C-10), 136.4 (C-9), 136.1 (C-3), 130.0 (C-8), 128.6 (C-5), 126.3 (C-7), 124.3 (C-6), 52.5 (C-6'), 45.4 (C-2') 33.2 (C-5'), 31.0 (C-4'), 25.1 (C-3'), 19.3 (CH_3); GCMS: Found M^+=227.00; $C_{14}H_{17}N_3$ requires M^+=227.30

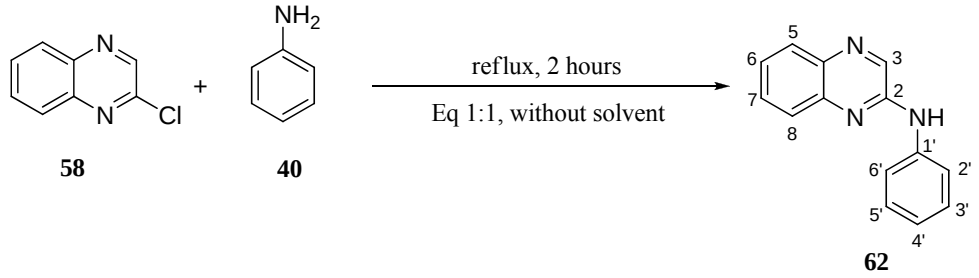
5.1.2.3 Preparation of 2-*N*-(4-methyl)piperidinoquinoxaline (61)



58 **36** **61**

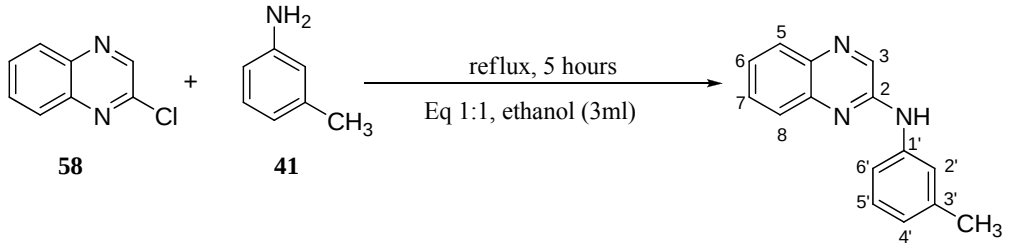
4-Methylpiperidine (1.18 ml, 0.01 moles) and ethanol (3.00 ml) were added to 2-chloroquinoxaline (0.3292 g, 0.002 moles) and the mixture was refluxed for 4 hours. The mixture was cooled and ethanol was evaporated off. The residue was then dissolved in minimum volume of water (10 ml). The slurry was extracted with ether (3 × 10 ml), washed with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Evaporation of ether gave light yellowish liquid. (0.3553 g, 78%), IR (ν_{max}, cm^{-1}): 1551 (C=N), 1578 (C=C), 2923 (C-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.49 (1H, s, H-3), 7.75 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.56 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.43 (1H, m, H-7), 7.24 (1H, m, H-6), 4.42 (2H, d, *J*=8 Hz, H-2'), 2.86 (2H, m, H-6'), 1.68 (2H, d, *J*=1.2 Hz, H-3'), 1.53 (1H, m, H-4',), 1.12 (2H, m, C-5'), 0.88 (3H, d, *J*=8 Hz, CH_3) ; ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 152.2 (C-2), 141.7 (C-10), 136.3 (C-9), 135.9 (C-3), 129.7 (C-8), 128.4 (C-5), 126.2 (C-7), 124.1 (C-6), 45.0 (C-6', C-2'), 33.71 (C-5', C-3') 31.1 (C-4'), 21.7 (CH_3); GCMS: Found M^+=227.00; $C_{14}H_{17}N_3$ requires M^+=227.31

5.1.2.4 Preparation of 2-*N*-anilinoquinoxaline (62)



2-Chloroquinoxaline (0.3295 g, 0.002 moles) was added to aniline (0.18 ml, 0.002 moles) and the mixture was heated in an oil bath at 140°C for 2 hours. The mixture was cooled, water (10 ml) was added to the mixture. The mixture was then extracted with ether (3 × 10 ml). The combined ethereal layer was washed with water and dried over anhydrous sodium sulphate. Ether was removed and pure product, 2-*N*-anilinoquinoxaline was obtained after purification using column chromatography using ethyl acetate:hexane (1:3) as the eluent. M.p. 132°C - 134°C, (0.1001 g, 23%), IR (ν_{max}, cm^{-1}): 1612 (C=N), 1521 and 1499 (aromatic C=C), 3304 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.45 (1H, s, H-3), 7.91 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.79 (1H, d, *J*=8 Hz, 1.2 Hz, H-5), 7.72 (2H, d, *J*=8 Hz, H-2', H-6'), 7.61 (1H, m, H-7), 7.45 (1H, m, H-6), 7.38 (2H, t, *J*=8.2 Hz, H-3', H-5'), 7.11 (1H, t, *J*=7.6 Hz, H-4'), 6.87 (1H, s, N-H); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 149.2 (C-2), 141.1 (C-1'), 139.1 (C-10), 138.3 (C-9), 137.8 (C-3), 130.3 (C-8), 129.2 (C-2', C-6'), 128.8 (C-5), 126.8 (C-7), 125.6 (C-6), 123.6 (C-4'), 119.9 (C-3', C-5'); GCMS: Found M^+=221.00; $C_{14}H_{11}N_3$ requires M^+=221.26

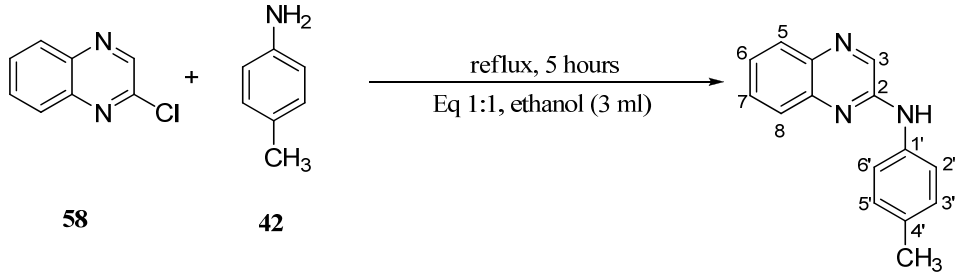
5.1.2.5 Preparation of 2-*N*-(*m*-methyl)anilinoquinoxaline (63)



58 **41** **63**

2-Chloroquinoxaline (0.3260 g, 0.002 moles) was dissolved in ethanol (3.00 ml) and added to *m*-toluidine (0.21 ml, 0.002 moles). The mixture was refluxed for 5 hours and cooled to room temperature. The solvent was evaporated and a minimum volume of water was added to the residue. The residual slurry was extracted with chloroform (3 × 10 ml). The chloroform extracts were washed with water and dried over anhydrous sodium sulphate. Evaporation of the solvent gave a crude product, pure 2-*N*-(*m*-methyl)anilinoquinoxaline was obtained after separating from the impurities using column chromatography, (mixture of ethyl acetate:hexane, 1:3, was used as the eluent. M.p. 88°C - 90°C, (0.1232 g, 42%), IR (ν_{max}, cm^{-1}): 1585 (C=N), 1552 (aromatic C=C), 3292 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.39 (1H, s, H-3), 7.84 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.71 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.53 (2H, m, H-7), 7.44 (1H, m, H-6), 7.41 (1H, s, H-2'), 7.37 (2H, d, *J*=6 Hz, H-6'), 7.19 (2H, t, *J*=7.8 Hz, H-4', H-5'), 6.87 (1H, d, *J*=8 Hz, N-H), 2.32 (3H, s, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 149.4 (C-2), 141.1 (C-1'), 139.1 (C-3'), 138.9 (C-10), 138.3 (C-3), 137.7 (C-9), 130.2 (C-8), 129.1 (C-5), 128.7 (C-7), 126.7 (C-6), 125.4 (C-2'), 124.5 (C-6'), 120.6 (C-5'), 117.2 (C-4'), 21.5 (CH_3); GCMS: Found M^+=235.00; $C_{15}H_{13}N_3$ requires M^+=235.28

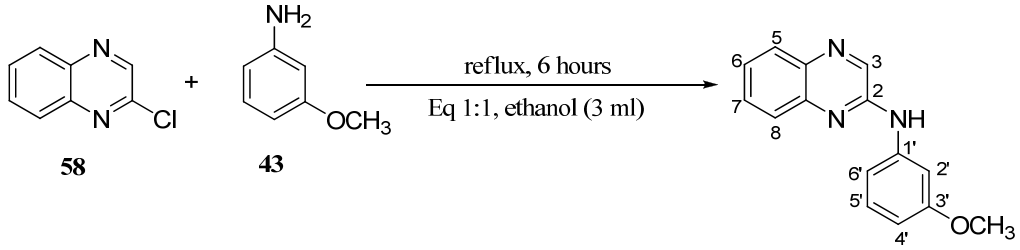
5.1.2.6 Preparation of 2-*N*-(*p*-methyl)anilinoquinoxaline (64)



58 **42** **64**

2-Chloroquinoxaline (0.3297 g, 0.002 moles) was dissolved in ethanol (3 ml) and *p*-toluidine (0.22 ml, 0.002 moles) was added to it. The reaction mixture was refluxed for 5 hours and cooled to room temperature. The solvent was evaporated off under *vacuo*. The minimum volume of water was added to the residue. The mixture was extracted three times with chloroform (3 × 10 ml). The organic layer was washed twice with water (2 × 10 ml) and dried over anhydrous sodium sulphate. Chloroform was removed and pure product was obtained after running through a column chromatography with ethyl acetate: hexane (1:3) as eluent. M.p. 150°C - 152°C, (0.2674 g, 57%), IR (ν_{max}, cm^{-1}): 1618 (C=N), 1544 and 1413 (aromatic C=C), 3296 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.41 (1H, s, H-3), 7.88 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.74 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.57 (1H, m, H-7), 7.53 (2H, d, *J*=8 Hz, H-2', H-6'), 7.41 (1H, m, H-6), 7.17 (2H, d, *J*=8 Hz, H-3', H-5'), 6.90 (1H, s, N-H), 2.34 (3H, s, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 149.5 (C-2), 141.0 (C-1'), 138.2 (C-3), 137.7 (C-10), 136.3 (C-9), 133.5 (C-4'), 130.2 (C-8), 129.8 (C-2', C-6'), 128.7 (C-5), 126.6 (C-7), 125.4 (C-6), 120.4 (C-3', C-5'), 20.8 (CH_3); GCMS: Found M^+=235.00; $C_{15}H_{13}N_3$ requires M^+=235.28; X-Ray Diffraction results were summarised in the Chapter 3: Table 3.5

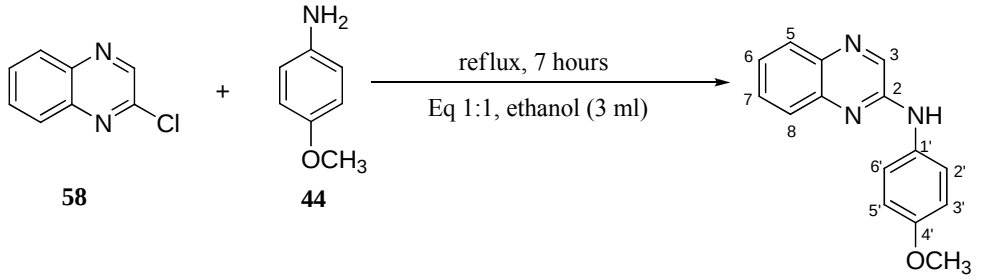
5.1.2.7 Preparation of 2-*N*-(*m*-methoxy)anilinoquinoxaline (65)



58 **43** **65**

2-Chloroquinoxaline (0.3292 g, 0.002 moles) and *m*-anisidine (0.22 ml, 0.002 moles) were refluxed for 5 hours. The mixture was cooled and water (10 ml) was added to the mixture. The organic layer was extracted with chloroform (3×10 ml). The chloroform layer was washed with water (3×10 ml) and dried over anhydrous sodium sulphate. Chloroform was removed under reduced pressure, leaving a brown residue of 2-*N*-(*m*-methoxy)anilinoquinoxaline which was recrystallised from chloroform. M.p. 128°C - 130°C, (0.0870 g, 18%), IR (ν_{max}, cm^{-1}): 1618 (C=N), 1496 (aromatic C=C), 3353 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.37 (1H, s, H-3), 7.83 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.71 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.53 (1H, m, H-7), 7.46 (1H, s, *J*=8 Hz, H-2'), 7.37 (1H, m, H-6), 7.17 (1H, d, *J*=4 Hz, H-6'), 7.08 (1H, dd, *J*=7.8 Hz, 1.2 Hz, H-5'), 6.59 (1H, dd, *J*=2.4 Hz, 1.7 Hz, H-4'), 3.74 (3H, s, OCH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 160.4 (C-3'), 149.0 (C-2), 140.5 (C-1'), 140.2 (C-10), 138.4 (C-3), 137.7 (C-9), 130.4 (C-8), 129.9 (C-5), 128.8 (C-7), 126.6 (C-2'), 125.7 (C-6), 112.2 (C-6'), 109.22 (C-5'), 105.9 (C-4'), 55.3 (OCH_3); GCMS: Found M^+=251.00; $C_{15}H_{13}N_3O$ requires M^+=251.28

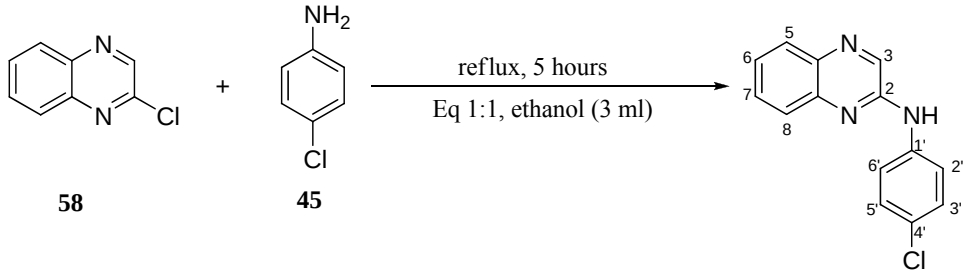
5.1.2.8 Preparation of 2-*N*-(*p*-methoxy)anilinoquinoxaline (66)



58 **44** **66**

2-Chloroquinoxaline (0.3292 g, 0.002 moles) was heated under reflux with *p*-anisidine (0.2441 g, 0.002 moles) for 7 hours at 120°-140°C. The mixture was cooled and water was added to the reaction mixture. The mixture was extracted twice with chloroform (2 × 10 ml). The organic layer was washed with water and dried over anhydrous sodium sulphate. Evaporation of solvent gave the product, 2-*N*-(*p*-methoxy)anilinoquinoxaline which was recrystallised from chloroform. M.p. 98°C - 100°C, (0.4234 g, 84%), IR (ν_{max}, cm^{-1}): 1617 (C=N), 1549 and 1511 (aromatic C=C), 3346 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.35 (1H, s, H-3), 7.87 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.71 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.58 (1H, m, H-7), 7.53 (2H, d, *J*=8 Hz, H-2', H-6'), 7.39 (1H, m, H-6), 6.91 (2H, d, *J*=8 Hz, H-3', H-5'), 3.80 (3H, s, OCH_3), 1.87 (1H, s, N-H); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 156.4 (C-4'), 149.8 (C-2), 141.1 (C-10), 138.0 (C-3), 137.6 (C-9), 131.9 (C-1'), 130.2 (C-8), 128.7 (C-5), 126.5 (C-7), 125.1 (C-6), 122.7 (C-2', C-6'), 114.5 (C-3', C-5'), 55.5 (OCH_3); GCMS: Found M^+=201.00; $C_{11}H_{11}N_3O$ requires M^+=201.22

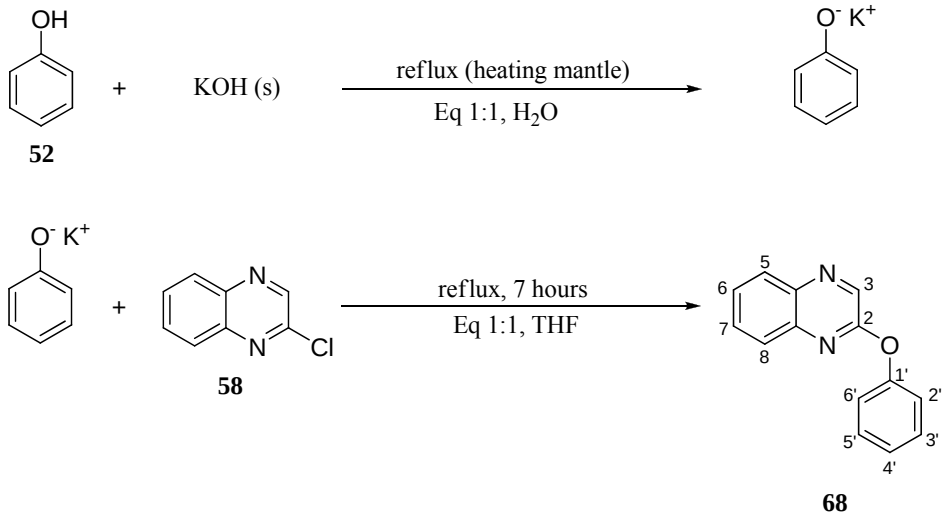
5.1.2.9 Preparation of 2-*N*-(*p*-chloro)anilinoquinoxaline (67)



58 **45** **67**

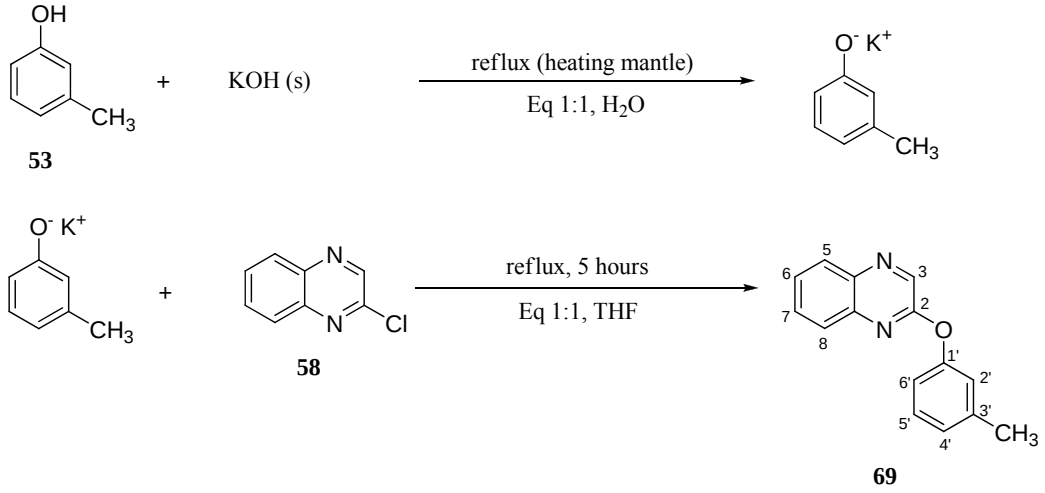
2-Chloroquinoxaline (0.3286 g, 0.002 moles) was dissolved in ethanol (3 ml) and *p*-chloroaniline (0.2510 g, 0.002 moles) was added to the reaction mixture refluxed for 5 hours. It was then cooled to room temperature. The solvent was evaporated off under *vacuo*. The minimum volume of water was added to the residue and the mixture was extracted with chloroform (3 × 10 ml). The organic layer was washed with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Chloroform was removed and the pure product was obtained after recrystallisation from chloroform to yield pure 2-*N*-(*p*-chloro)anilinoquinoxaline. M. p. 190°C - 192°C, (0.3645 g, 71%), IR (ν_{max}, cm^{-1}): 1619 (C=N), 1544 and 1498 (aromatic C=C), 3292 (N-H); ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.32 (1H, s, H-3), 7.84 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.72 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.65 (2H, d, *J*=8 Hz, H-3', H-5'), 7.55 (1H, m, H-7), 7.39 (1H, m, H-6), 7.26 (2H, d, *J*=8 Hz, H-2', H-6'), 6.91 (1H, s, N-H); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 148.9 (C-2), 140.9 (C-1'), 138.4 (C-3), 137.8 (C-10), 130.4 (C-9), 129.2 (C-5, C-8), 128.8 (C-3', C-5'), 128.2 (C-4'), 126.9 (C-7), 125.8 (C-6), 120.7 (C-3', C-5'); GCMS: Found M^+=255.00; $C_{14}H_{10}N_3Cl$ requires M^+=255.75; X-Ray Diffraction results were summarised in the Chapter 3: Table 3.6

5.1.2.10 Preparation of 2-phenoxyquinoxaline (68)



Phenol (0.18 ml, 0.002 moles), potassium hydroxide pellet (0.1120 g, 0.002 moles) and water (5 ml) were stirred for 10 minutes and the mixture was heated until a solid formed. 2-Chloroquinoxaline (0.3290 g, 0.002 moles) in THF was added to the solid and the reaction mixture was refluxed for 7 hours. The mixture was then cooled and the solvent was evaporated off. 5% sodium hydroxide (5 ml) was added to the residue followed by water (5 ml). The residual slurry was extracted with chloroform (3 × 10 ml). The chloroform extracts were washed with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Removal of chloroform followed by recrystallisation with chloroform gave the pure 2-phenoxyquinoxaline. M.p. 98°C - 100°C, (0.2705 g, 61%), IR (ν_{max}, cm^{-1}): 1597 (C=N), 1571 and 1489 (aromatic C=C), 1217 and 1019 (C-O), ^1H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.62 (1H, s, H-3), 7.98 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.68 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.51 (2H, m, H-6, H-7), 7.36 (2H, td, *J*=7.6 Hz, 1.6 Hz, H-2', H-6'), 7.18 (3H, m, H-3', H-5', H-4'); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 156.9 (C-2), 152.7 (C-1'), 139.9 (C-10), 139.6 (C-9), 139.1 (C-3), 130.3 (C-8), 129.6 (C-2', C-6'), 128.8 (C-5), 127.7 (C-7), 127.4 (C-6), 125.4 (C-4'), 121.3 (C-3', C-5'); GCMS: Found M^+=222.00; $C_{14}H_{10}N_2O$ requires M^+=222.25

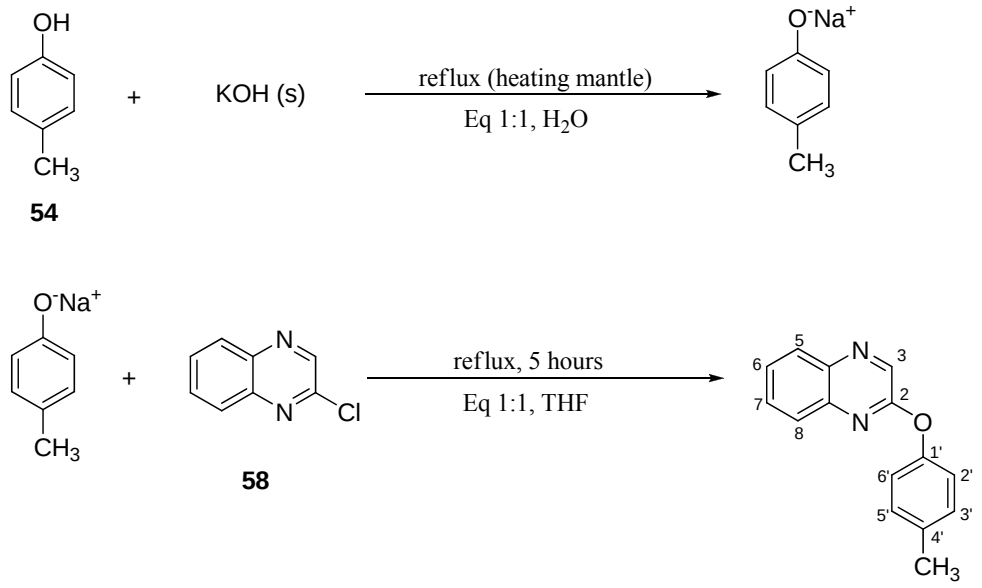
5.1.2.11 Preparation of 2-(*m*-methyl)phenoxyquinoxaline (69)



Phenol (0.21 ml, 0.002 moles) was added with potassium hydroxide pellet (0.1137 g, 0.002 moles) in minimum volume of water (5 ml) and heated to form a solid. 2-Chloroquinoxaline (0.3256 g, 0.002 moles) in THF (5 ml) was then added to the reaction mixture and refluxed for 5 hours. The mixture was cooled to room temperature and the solvent was removed under *vacuo*. Water (5 ml) was added to the residue followed by 5% sodium hydroxide solution (5 ml). The residual slurry was extracted with chloroform (3 × 10 ml). The organic layer was then washed three times with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Filtration and evaporation of the solvent gave a pure product, 2-(*m*-methyl)phenoxyquinoxaline which was obtained after recrystallisation from chloroform. M.p. 98°C - 100°C, (0.3097 g, 66%), IR (ν_{max}, cm^{-1}): 1610 (C=N), 1569 and 1486 (aromatic C=C), 1214 and 1140 (C-O), ^{1}H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.60 (1H, s, H-3), 7.97 (1H, dd, *J*=8 Hz, 1.2 Hz, H-8), 7.70 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.51 (2H, m, H-6, H-7), 7.25 (1H, td, *J*=7.3 Hz, 1.4 Hz, H-6'), 7.00 (3H, t, *J*=7.5 Hz, H-2', H-4', H-5'), 2.31 (3H, s, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 157.0 (C-2), 152.7 (C-1'), 140.0 (C-3'), 139.9 (C-10), 139.6 (C-9), 139.2 (C-3), 130.3 (C-8), 129.3 (C-5), 128.8 (C-7), 127.7 (C-6), 127.3 (C-6'), 126.2 (C-2'),

121.8 (C-5'), 118.3 (C-4'), 21.4 (CH_3); GCMS: Found M^+=236.00; $C_{15}H_{12}N_2O$ requires M^+=236.27

5.1.2.12 Preparation of 2-(*p*-methyl)phenoxyquinoxaline (70)



Phenol (0.21 ml, 0.002 moles) was added to pottasium hydroxide pellet (0.1270 g, 0.002 moles) in minimum volume of water (5 ml) and heated form a solid. 2-Chloroquinoxaline (0.3294 g, 0.002 moles) in THF (5 ml) was added to reaction mixture and refluxed for 5 hours. The mixture was cooled to room temperature and the solvent was removed under *vacuo*. Water (5 ml) was added to the residue, the mixture was shaken thoroughly with 5% of sodium hydroxide solution (5 ml) and extracted three times with chloroform (3 × 10 ml). The combined chloroform layer was washed with water (3 × 10 ml) and dried over anhydrous sodium sulphate. Evaporation of chloroform gave crude product which further recrystallised from ethyl acetate to give pure 2-(*p*-methyl)phenoxyquinoxaline. M.p. 88°C - 90°C, (0.3535 g, 75%), IR (ν_{max}, cm^{-1}): 1572 (C=N), 1498 and 1401 (aromatic C=C), 1215 and 1019 (C-O), ^1H NMR (ppm, 400 MHz, $CDCl_3$) δ_H: 8.68 (1H, s, H-3), 8.04 (1H, dd, *J*=8 Hz, 1.2 Hz,

H-8), 7.76 (1H, dd, *J*=8 Hz, 1.2 Hz, H-5), 7.58 (2H, m, H-6, H-7), 7.26 (2H, d, *J*=8 Hz, H-2', H-6'), 7.00 (2H, d, *J*=8 Hz, H-3', H-5'), 2.40 (3H, s, CH_3); ^{13}C NMR (ppm, 100 MHz, $CDCl_3$) δ_C: 157.0 (C-2), 150.4 (C-1'), 140.0 (C-10), 139.5 (C-9), 139.2 (C-3), 135.0 (C-4'), 130.3 (C-8), 130.1 (C-2', C6'), 128.8 (C-5), 127.7 (C-7), 127.3 (C-6), 121.1 (C-3', C-5'), 20.9 (CH_3); GCMS: Found M^+=236.00; $C_{15}H_{12}N_2O$ requires M^+=236.27

5.2 Fluorescence Measurements

Fluorescence spectra were recorded using Fluorescence Spectrometer, Model F2000, Hitachi (with the sensitivity is 2) and Luminescence Spectrometer, Model LS 50B, Perkin Elmer using 2.5 mm slits. Quinine sulphate (10 ppm in 0.1 N sulphuric acid) was used as the standard. All spectra were recorded at room temperature and are corrected for phototuberesponse and by substraction of the solvent background.

5.2.1 Study of fluorescence characteristics in various solvents and concentrations

The fluorescence spectra of all the compounds studied were measured in various solvents. Samples were prepared from stock solution (10^{-4} M) of the corresponding compound in tetrahydrofuran (THF), acetonitrile (CH_3CN), ethyl acetate (EtOAc) and ethanol (EtOH) to give concentrations of 10^{-5} M and 10^{-6} M.

Capped condition

2-*N*-Piperidinopyrazine (37)

Sensitivity = 2

Concentration = 6.1270 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	345	348	369	403	402	401	62.27	382.70	560.50
CH_3CN	348	348	371	411	415	419	51.64	262.40	460.60
EtOAc	344	345	362	404	407	408	26.30	159.80	441.70
EtOH	349	350	373	422	432	431	4.18	25.67	51.31

2-*N*-(3-Methyl)piperidinopyrazine (38)

Sensitivity = 2

Concentration = 5.5785 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	347	347	362	402	404	401	27.42	235.30	505.40
CH_3CN	347	348	354	414	414	414	11.31	84.34	364.80
EtOAc	345	346	356	401	404	405	15.32	119.10	398.10
EtOH	348	349	363	424	424	432	3.375	23.51	70.32

2-*N*-(4-Methyl)piperidinopyrazine (39)

Sensitivity = 2

Concentration = 5.5785 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	345	347	360	402	400	403	29.62	208.00	518.50
CH_3CN	347	348	360	411	415	415	17.80	130.30	399.60
EtOAc	343	345	358	404	404	404	21.88	149.60	399.10
EtOH	348	350	363	425	430	427	2.56	15.31	45.86

2-*N*-Anilinopyrazine (46)

Sensitivity = 2

Concentration = 5.8400 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	347	349	370	435	435	432	12.33	71.61	97.55
CH_3CN	348	358	377	386	405	414	2.25	3.49	10.09
EtOAc	334	349	371	379	438	438	5.25	11.72	18.54
EtOH	312	320	399	340	389	496	0.45	0.55	1.78

2-*N*-(*m*-Methyl)anilinopyrazine (47)

Sensitivity = 2

Concentration = 5.3987 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	350	350	369	436	438	440	8.35	45.61	71.39
CH_3CN	351	350	385	447	449	452	0.34	1.97	8.63
EtOAc	X	349	376	X	442	444	X	10.39	23.98
EtOH	X	X	409	X	X	456	X	X	149.10

X = non-fluorescent

2-*N*-(*p*-Methyl)anilinopyrazine (48)

Sensitivity = 2

Concentration = 5.3987 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	299	300	385	442	444	446	59.75	151.17	265.12
CH_3CN	X	X	397	X	X	447	X	X	54.05
EtOAc	X	X	390	X	X	448	X	X	58.60
EtOH	X	X	413	X	X	460	X	X	3.78

X = non-fluorescent

2-*N*-(*m*-Methoxy)anilinopyrazine (49)

Sensitivity = 2

Concentration = 4.9697 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	346	349	373	438	442	439	7.70	46.40	79.40
CH_3CN	349	350	382	433	435	448	0.60	3.62	12.11
EtOAc	X	349	377	X	441	439	X	8.66	25.77
EtOH	X	360	396	X	446	464	X	1.24	6.27

X = non-fluorescent

2-*N*-(*p*-Methoxy)anilinopyrazine (50)

Sensitivity = 2

Concentration = 4.9697 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	X	357	400	X	485	486	X	2.19	5.56
CH_3CN	X	463	458	X	490	487	X	2.57	3.74
EtOAc	X	461	471	X	482	556	X	0.99	2.88
EtOH	331	330	508	388	389	569	1.809	5.56	4.91

X = non-fluorescent

2-*N*-(*p*-Chloro)anilinopyrazine (51)

Sensitivity = 2

Concentration = 4.8629 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	345	348	367	432	436	434	8.14	49.88	70.13
CH_3CN	343	343	358	444	461	464	0.60	2.96	3.23
EtOAc	343	344	357	431	437	434	6.67	35.34	60.30
EtOH	347	352	383	383	447	466	0.20	0.33	0.53

2-Phenoxypyrazine (55)

Sensitivity = 2

Concentration = 5.8079 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	X	X	387	X	X	434	X	X	0.26
CH_3CN	X	305	320	X	385	387	X	1.36	1.99
EtOAc	X	X	314	X	X	340	X	X	3.78
EtOH	X	X	X	X	X	X	X	X	X

X = non-fluorescent

2-(*m*-Methyl)phenoxypyrazine (56)

Sensitivity = 2

Concentration = 5.3703 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	X	X	327	X	X	359	X	X	2.14
CH_3CN	X	307	313	X	409	374	X	0.45	1.53
EtOAc	X	X	309	X	X	339	X	X	3.97
EtOH	X	333	330	X	366	363	X	0.37	3.17

X = non-fluorescent

2-(*p*-Methyl)phenoxypyrazine (57)

Sensitivity = 2

Concentration = 5.3703 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	X	361	310	X	401	389	X	0.22	3.03
CH_3CN	X	304	313	X	442	444	X	0.26	0.67
EtOAc	X	X	311	X	X	340	X	X	3.29
EtOH	X	X	436	X	X	497	X	X	0.22

X = non-fluorescent

2-*N*-Piperidinoquinoxaline (59)

Sensitivity = 2

Concentration = 4.6886 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	382	381	404	437	438	439	6.77	44.17	84.38
CH_3CN	383	385	411	446	447	448	17.38	95.69	142.30
EtOAc	380	381	404	438	438	441	8.82	39.21	92.30
EtOH	385	392	418	450	452	454	6.85	34.68	42.68

2-*N*-(3-Methyl)piperidinoquinoxaline (60)

Sensitivity = 2

Concentration = 4.3990 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	380	382	405	437	437	439	7.54	48.63	83.90
CH_3CN	382	383	407	446	448	449	12.77	77.89	150.20
EtOAc	X	381	402	X	440	440	X	45.47	98.60
EtOH	385	389	408	452	452	454	4.07	28.30	68.15

X = non-fluorescent

2-*N*-(4-Methyl)piperidinoquinoxaline (61)

Sensitivity = 2

Concentration = 4.399 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	380	380	401	438	439	439	5.52	36.49	72.59
CH_3CN	381	384	395	444	44	446	8.02	51.42	115.90
EtOAc	379	381	405	435	437	438	7.21	45.56	69.24
EtOH	385	387	413	449	451	453	4.96	30.08	46.67

2-*N*-Anilinoquinoxaline (62)

Sensitivity = 2

Concentration = 4.5195 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	380	382	401	485	483	483	2.30	14.07	21.16
CH_3CN	382	325	314	411	395	411	1.29	5.94	10.69
EtOAc	X	384	405	X	484	486	X	7.11	9.09
EtOH	305	305	313	406	335	332	0.48	0.59	0.86

X = non-fluorescent

2-*N*-(*m*-Methyl)anilinoquinoxaline (63)

Sensitivity = 2

Concentration = 4.2503 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	381	385	410	486	488	472	2.06	9.29	14.43
CH_3CN	381	391	432	419	441	472	0.53	0.34	4.88
EtOAc	377	380	405	468	488	488	0.86	4.81	6.53
EtOH	398	397	441	449	483	482	0.15	0.40	1.83

2-*N*-(*p*-Methyl)anilinoquinoxaline (64)

Sensitivity = 2

Concentration = 4.2503 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	384	388	411	492	495	500	0.40	1.95	3.40
CH_3CN	X	X	X	X	X	X	X	X	X
EtOAc	358	383	399	400	493	496	0.90	0.87	2.13
EtOH	394	392	419	444	443	480	0.15	0.16	0.18

X = non-fluorescent

2-*N*-(*m*-Methoxy)anilinoquinoxaline (65)

Sensitivity = 2

Concentration = 3.9795 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	381	383	410	491	490	471	1.11	6.86	14.74
CH_3CN	X	407	414	X	469	471	X	0.38	3.09
EtOAc	379	384	412	484	486	469	0.83	4.04	5.82
EtOH	418	416	441	478	482	482	0.16	0.86	3.38

X = non-fluorescent

2-*N*-(*p*-Methoxy)anilinoquinoxaline (66)

Sensitivity = 2

Concentration = 3.9795 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	413	412	437	469	470	470	0.50	3.03	14.58
CH_3CN	409	409	434	467	471	472	0.25	1.96	12.98
EtOAc	369	410	435	407	471	470	0.63	0.89	10.44
EtOH	417	416	444	480	480	482	0.57	2.67	11.50

2-*N*-(*p*-Chloro)anilinoquinoxaline (67)

Sensitivity = 2

Concentration = 3.9100 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	380	382	400	464	483	481	1.84	11.53	20.41
CH_3CN	223	362	395	449	403	498	0.31	0.498	0.76
EtOAc	376	383	403	467	467	467	2.40	12.63	15.08
EtOH	387	385	408	435	461	465	0.14	0.335	0.47

2-Phenoxyquinoxaline (68)

Sensitivity = 2

Concentration = 4.4996 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	412	412	411	510	510	512	1.07	9.52	75.29
CH_3CN	380	380	395	427	448	449	0.51	2.42	15.81
EtOAc	404	404	407	504	506	507	0.66	4.66	37.93
EtOH	414	413	411	478	480	478	0.51	3.19	19.66

2-(*m*-Methyl)phenoxyquinoxaline (69)

Sensitivity = 2

Concentration = 4.2324 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	X	X	420	X	X	485	X	X	2.10
CH_3CN	X	347	345	X	386	438	X	1.08	1.48
EtOAc	X	X	348	X	X	381	X	X	1.78
EtOH	X	X	397	X	X	452	X	X	0.53

X = non-fluorescent

2-(*p*-Methyl)phenoxyquinoxaline (70)

Sensitivity = 2

Concentration = 4.2324 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	X	413	413	X	515	516	X	0.85	7.46
CH_3CN	X	334	345	X	485	487	X	0.18	0.47
EtOAc	341	338	380	397	430	440	0.58	1.52	6.40
EtOH	X	392	386	X	444	452	X	0.49	3.70

X = non-fluorescent

Uncapped condition

2-*N*-Piperidinopyrazine (37)

Sensitivity = 2

Concentration = 6.1270 x 10^{x} M, where 10^{x} M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	345	348	369	402	403	401	60.30	382.50	535.70
CH_3CN	348	348	371	411	417	417	51.00	240.70	440.00
EtOAc	344	345	362	402	407	407	24.08	156.20	440.00
EtOH	349	350	373	418	430	431	3.89	25.17	51.26

2-*N*-Anilinopyrazine (46)

Sensitivity = 2

Concentration = 5.8400 x 10^{x} M, where 10^{x} M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	347	349	370	434	434	432	11.55	64.02	90.62
CH_3CN	348	358	377	386	403	412	2.24	3.31	9.47
EtOAc	334	349	371	378	437	438	5.24	11.63	17.96
EtOH	312	320	399	339	386	492	0.44	0.54	1.64

2-*N*-Piperidinoquinoxaline (59)

Sensitivity = 2

Concentration = 4.6886 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	382	381	404	437	437	438	6.66	44.07	83.40
CH_3CN	383	385	411	447	447	449	17.15	94.02	140.6
EtOAc	380	381	404	439	437	440	8.70	38.85	91.02
EtOH	385	392	418	453	453	453	6.70	33.39	42.26

2-*N*-Anilinoquinoxaline (62)

Sensitivity = 2

Concentration = 4.5195 x 10^x M, where 10^x M = 10^{-6} M, 10^{-5} M, 10^{-4} M

Solvent	Excitation wavelength (nm)			Fluorescence wavelength (nm)			Intensity		
	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M	10^{-6} M	10^{-5} M	10^{-4} M
THF	380	382	401	483	483	482	2.22	13.33	20.30
CH_3CN	382	325	314	411	393	411	1.29	5.79	9.71
EtOAc	X	384	405	X	485	482	X	6.64	8.32
EtOH	305	305	313	403	336	332	0.44	0.56	0.84

X = non-fluorescent

Uncapped condition

Sensitivity = 2

Concentration = 10^{-4} M

Compound	Solvent	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
2-*N*-(3-methyl)piperidinopyrazine **(38)**	Tetrahydrofuran	362	403	502.50
	Acetonitrile	354	416	356.20
	Ethyl acetate	356	406	382.40
	Ethanol	363	429	67.750
2-*N*-(4-methyl)piperidinopyrazine **(39)**	Tetrahydrofuran	360	401	466.40
	Acetonitrile	360	412	390.50
	Ethyl acetate	358	406	398.00
	Ethanol	363	430	44.18
2-*N*-(*m*-methyl)anilinopyrazine **(47)**	Tetrahydrofuran	369	438	67.69
	Acetonitrile	385	455	8.57
	Ethyl acetate	376	440	23.74
	Ethanol	409	457	146.83
2-*N*-(*p*-methyl)anilinopyrazine **(48)**	Tetrahydrofuran	385	447	168.34
	Acetonitrile	397	447	49.66
	Ethyl acetate	390	449	54.15
	Ethanol	413	462	3.65
2-*N*-(*m*-methoxy)anilinopyrazine **(49)**	Tetrahydrofuran	373	439	71.56
	Acetonitrile	382	452	11.84
	Ethyl acetate	377	440	24.89
	Ethanol	396	461	6.07
2-*N*-(*p*-methoxy)anilinopyrazine **(50)**	Tetrahydrofuran	400	485	5.37
	Acetonitrile	458	487	3.63
	Ethyl acetate	471	552	1.15
	Ethanol	508	568	4.62
2-*N*-(*p*-chloro)anilinopyrazine **(51)**	Tetrahydrofuran	367	436	68.94
	Acetonitrile	358	467	2.17
	Ethyl acetate	357	435	58.23
	Ethanol	383	469	0.55
2-phenoxypyrazine **(55)**	Tetrahydrofuran	387	434	0.17
	Acetonitrile	320	388	1.84
	Ethyl acetate	314	340	3.765
	Ethanol	X	X	X
2-(*m*-methyl)phenoxypyrazine **(56)**	Tetrahydrofuran	327	359	2.05
	Acetonitrile	313	376	1.47
	Ethyl acetate	309	339	3.62
	Ethanol	330	363	3.14
2-(*p*-methyl)phenoxypyrazine **(57)**	Tetrahydrofuran	310	392	3.01
	Acetonitrile	313	443	0.65
	Ethyl acetate	311	339	3.22
	Ethanol	436	497	0.19

X = non-fluorescent

Compound	Solvent	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
2-*N*-(3-methyl)piperidinoquinoxaline **(60)**	Tetrahydrofuran	405	439	81.89
	Acetonitrile	407	448	144.60
	Ethyl acetate	402	440	95.25
	Ethanol	363	429	67.75
2-*N*-(4-methyl)piperidinoquinoxaline **(61)**	Tetrahydrofuran	401	439	72.54
	Acetonitrile	395	446	115.20
	Ethyl acetate	405	438	68.25
	Ethanol	413	454	46.63
2-*N*-(*m*-methyl)anilinoquinoxaline **(63)**	Tetrahydrofuran	410	473	14.20
	Acetonitrile	432	472	1.51
	Ethyl acetate	405	489	6.47
	Ethanol	441	482	1.57
2-*N*-(*p*-methyl)anilinoquinoxaline **(64)**	Tetrahydrofuran	411	502	3.28
	Acetonitrile	318	422	0.48
	Ethyl acetate	399	497	2.09
	Ethanol	419	480	0.16
2-*N*-(*m*-methoxy)anilinoquinoxaline **(65)**	Tetrahydrofuran	410	471	13.93
	Acetonitrile	414	471	2.70
	Ethyl acetate	412	488	4.08
	Ethanol	418	482	3.26
2-*N*-(*p*-methoxy)anilinoquinoxaline **(66)**	Tetrahydrofuran	437	471	10.19
	Acetonitrile	434	471	11.98
	Ethyl acetate	435	470	7.81
	Ethanol	444	480	9.46
2-*N*-(*p*-chloro)anilinoquinoxaline **(65)**	Tetrahydrofuran	400	478	19.77
	Acetonitrile	395	499	0.71
	Ethyl acetate	403	469	14.62
	Ethanol	408	467	0.46
2-phenoxyquinoxaline **(68)**	Tetrahydrofuran	411	511	71.31
	Acetonitrile	395	449	15.55
	Ethyl acetate	407	506	30.92
	Ethanol	411	479	17.53
2-(*m*-methyl)phenoxyquinoxaline **(69)**	Tetrahydrofuran	420	483	1.93
	Acetonitrile	345	441	1.40
	Ethyl acetate	348	387	1.78
	Ethanol	397	450	0.37
2-(*p*-methyl)phenoxyquinoxaline **(70)**	Tetrahydrofuran	413	516	7.28
	Acetonitrile	345	487	0.45
	Ethyl acetate	380	440	0.01
	Ethanol	386	451	3.39

4.2.1 Study of fluorescence characteristics with time

The fluorescence spectra of the selected derivatives studied were measured with time. The measurements were taken in every 20 minutes interval.

2-(*p*-Methyl)phenoxypyrazine (57)

Slit: 2.5 mm

Concentration: 5.3703 x 10^{-4} M

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	310	286	65.53
	20		391	62.77
	40		385	61.93

2-*N*-(*p*-Methyl)anilinoquinoxaline (64)

Slit: 2.5 mm

Concentration: 4.2503 x 10^{-4} M

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	411	500	4.92
	20		502	4.89
	40		516	4.74

2-*N*-(*p*-Methoxy)anilinoquinoxaline (66)

Slit: 2.5 mm

Concentration: 3.9795 x 10^{-4} M

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	437	471	32.23
	20		473	31.15
	40		472	29.67

2-Phenoxyquinoxaline (68)

Slit: 2.5 mm

Concentration = 4.4996 x 10^{-4} M,

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	387	428	6.24
	20		429	4.80
	40		427	4.45

2-(*m*-Methyl)phenoxyquinoxaline (69)

Slit: 2.5 mm

Concentration = 4.2324 x 10^{-4} M,

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	420	516	501.99
	20		516	495.03
	40		515	485.83

2-*N*-(3-Methyl)piperidinopyrazine (38)

Slit: 2.5 mm

Concentration = 5.5785 x 10^{-6} M

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
Ethyl acetate	Immediately	345	405	476.37
	20		404	468.87
	40		405	465.65

2-*N*-(4-Methyl)piperidinopyrazine (39)

Slit: 2.5 mm

Concentration = 5.5785 x 10^{-6} M,

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	343	405	635.91
	20		404	632.91
	40		404	632.31

2-*N*-(*p*-Methyl)anilinoquinoxaline (64)

Slit: 2.5 mm

Concentration = 4.2503 x 10^{-6} M

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	384	425	5.76
	20		424	3.73
	40		425	3.41

2-*N*-(*m*-Methoxy)anilinoquinoxaline (65)

Slit: 2.5 mm

Concentration = 3.9795 x 10^{-6} M

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	381	489	19.21
	20		481	19.18
	40		489	18.89

2-*N*-(*m*-Methoxy)anilinoquinoxaline (65)

Slit: 2.5 mm

Concentration = 3.9795 x 10^{-6} M

Solvent	Duration (min)	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
THF	Immediately	380	472	20.83
	20		473	23.98
	40		472	25.29

4.2.2 Study of the effect of acidity and basicity on fluorescence characteristics

The fluorescence spectra of the selected derivatives were measured in ethanol with the concentration of 10^{-4}M in acidic and basic conditions respectively.

2-*N*-Piperidinopyrazine (37)

Sensitivity = 2

Concentration = 6.1270 x 10^{-4} M

Conditions	pH	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
acid	2	359	439	106.20
	3	365	433	269.70
neutral	7	373	432	287.50
base	11	368	431	277.90
	12	368	433	259.10

2-*N*-Anilinopyrazine (46)

Sensitivity = 2

Concentration = 5.8400×10^{-4} M

Conditions	pH	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
acid	2	382	433	3.79
	3	393	492	6.49
neutral	7	399	493	12.25
base	11	395	491	7.71
	12	393	490	7.48

2-Phenoxypyrazine (55)

Sensitivity = 2

Concentration = 5.8079×10^{-4} M

Conditions	pH	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
acid	2	314	344	3.30
	3	320	353	3.89
neutral	7	277	320	4.00
base	11	377	489	1.39
	12	385	494	1.15

2-*N*-Piperidinoquinoxaline (59)

Sensitivity = 2

Concentration = 4.6886×10^{-4} M

Conditions	pH	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
acid	2	409	455	143.00
	3	410	455	157.80
neutral	7	418	455	159.40
base	11	410	455	154.20
	12	409	455	158.60

2-*N*-Anilinoquinoxaline (62)

Sensitivity = 2

Concentration = 4.5195×10^{-4} M,

Conditions	pH	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
acid	2	336	454	2.10
	3	419	483	2.36
neutral	7	313	482	5.17
base	11	422	483	2.60
	12	418	483	2.62

2-Phenoxyquinoxaline (68)

Sensitivity = 2

Concentration = 4.4996×10^{-4} M

Conditions	pH	Excitation wavelength (nm)	Fluorescence wavelength (nm)	Intensity
acid	2	391	478	65.15
	3	393	480	63.87
neutral	7	411	452	27.35
base	11	413	481	68.64
	12	413	481	67.76