SYNTHESIS OF SOME NEW OXADIAZOLYL, TRIAZOLYL AND PYRIDOQUINOXALINE DERIVATIVES

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Abstract: Treatment of ethyl 3-methylquinoxaline-2-carboxylate **1** with hydrazine hydrate and ammonia gave the corresponding carbohydrazide and the carboxamide derivatives **2,3** respectively. The reaction of **2** with nitrous acid gave the carboazide **4**,which underwent Curtius rearrangement in boiling xylene to give pyrroloquinoxaline **5**. The oxadiazolylquinoxaline **8** and triazolylquinoxaline **14** derivatives were obtained through the reaction of **2** with CS_2 in pyridine and with phenyl isothiocyanate followed by subsequent cyclization of the intermediate **13** using KOH. Pyridoquinoxalines **16b** were produced from the reaction of **3** with aromatic aldehydes

Introduction

Recently it was reported that some benzo[f]pyrido[3,4-b]- and pyrido[3,4-b] quinoxalines have significant antitumor properties(1). Fernandez et al reported that some quinoxaline derivatives were used as antitumoral agent and they found that the anticancer activity was increased if one or two carbon atoms of the quinoxaline benzene ring are replaced by nitrogen atoms(2). On the other hand many substituted quinoxalines such as chloroquinoxalines, methoxyquinoxalines and metal salts of 2-mercaptoquinoxaline-1-oxide, have been tested and showed fungicidal, bactericidal and insecticidal activity (3-5). In view of the biological importance of quinoxaline derivatives and in continuation of our work in the synthesis of heterocyclic compounds containing quinoxaline moiety(6,7) in this article we reported the synthesis of some oxadiazolylquinoxaline, triazolylquinoxaline and pyridoquinoxaline derivatives hoping it may be biologically active.

Experimental

Melting points are uncorrected and were determined on a Mel-Temp 11 melting point apparatus, IR spectra were recorded on Pye-Unicam SP 3-100 spectrophotometer using KBr Wafer technique. ^{1}HNMR spectra were recorded on a Varian 390 90 MHz NMR spectrometer in the suitable

deuterated solvent, using TMS as internal standard .Elemental analysis were determined on Perkin-Elmer 240 C microanalyzer.

Ethyl 3-methylquinoxaline-2-carboxylate 1:

The titled compound was synthesized according literature procedure, mp. 74 °C; lit.(8) m.p. 72-73 °C.

3-Methyl-quinoxaline-2-carbohydrazide 2:

A mixture of compound 1(0.01 mol) and hydrazine hydrate (0.01 mol) in ethanol (30 ml) was refluxed for 3 h. The solid product was filtered off and recrystallised from ehanol as white crystals in 85% yield , mp.168-70 °C. Calcd $C_{10}H_{10}N_4O$: C, 59.40; H, 4.98 ;N, 27.71%; found: C,59.28; H, 5.10; N, 27.52%. IR:ν= 3450,3350,3200 (NH_2,NH), 1650(C=O); ^{1}HNMR($CDCl_3$): 2.95(CH_3), 4.25 (s, 2H, NH_2), 7.6-8.05 (m,4H,ArH), and 9.4(s,1H, NH).

3-Methylquinoxaline-2-carboxamide 3:

To a sample of compound 1 (0.01 mol) in ethanol (20 ml)ammonia solution (35%)(10 ml) was added, the mixture was stirred with warming at 60 °C for 3h., then alowed to stand for two hours. The solid product was collected and recrystallised from ethanol as white crystals in 65% yield, mp. 197 °C. Calcd. $C_{10}H_9N_3O$: C, 64.16; H, 4.85; N, 22.45 %, found: C,63.98; H, 5.02; N, 22.44 %. IR: ν= 3400, 3300 (NH_2), 1700 (C=O); ^{1}HNMR($CDCl_3$): 3.1 (s,3H,CH_3), 5.7(br. s, 2H,NH_2), 7.7-8.15 (m, 4H, ArH).

3-Methylquinoxaline-2-carboazide 4:

To a solution of 2 (0.01 mol) in acetic acid (20 ml.), sod. nitrite solution (2.0 gm. in 5 ml H_2O) was added dropwise with stirring, after addition was completed the mixture was stirred for 1 h and allowed to stand for additional 2 h . The solid product was collected in 75% yield, m.p. 120 °C dec., and used without further purification. Calcd. $C_{10}H_7N_5O$: C, 56.34; H, 3.31; N, 32.85%; found: C, 56.52; H, 3.48; N, 33.00%. IR:ν= 3050(CH aromatic), 2150(N_3),and at 1690(C=O); ^{1}HNMR ($CDCl_3$): 2.95 (s, 3H, CH_3), 7.7-8.3(m,4H,ArH).

2,3-Dihydropyrrolo[1H][2,3-b]quinoxalin-2-one 5:

A sample of compound 4 (0.5 g) in dry xylene (10 ml) was heated for 1 h, then allowed to cool. The solid product was colleted and recrystallized from dioxan as white crystals in 68% yield, m.p. >300 °C. Calcd. $C_{10}H_7N_3O$: C, 64.86; H, 3.81; N, 22.69%; found: C, 65.04; H, 3.72; N, 22.65%. IR: ν=3250 (NH), 1670(C=O); ^{1}HNMR(DMSO-d_6): 3.2(s,2H,CH_2), 7.7-8.3(m,4H,ArH), and 9.5 (s, 1H,NH)

Ethyl 2-methylquinoxaline-3-carbamate 6:

A sample of compound 4 (0.5 gm) was refluxed in ethanol(10 ml) for 3 h, then allowed to cool the solid product was filtered off and recrystallised from ethanol as white crystals in 70% yield, mp .130 °C.

Calcd. $C_{12}H_{13}N_3O_2$: C, 62.33; H, 5.67; N, 18.17%; found: C, 62.08; H, 5.60; N, 17.96 %. IR:ν= 3280 (NH), 1690(C=O); ^{1}HNMR($CDCl_3$): 1.35 (t, 3H, CH_3), 2.75 (s, 3H, CH_3), 4.3(q,2H, CH_2) and 7.6-8.00 (m, 4H, ArH), 11.65(s,1H,NH).

Reaction of carboazide 4 with amines:

A mixture of compound 4 (0.005 mol) and aromatic amine (0.005 mol) in xylene (20 ml) was refluxed for one hour, then allowed to cool. The solid product was filtered off and recrystallised from ethanol. The physical constants and spectral data of compound 7 are listed in table (1, 2).

2 Methyl-3(2-mercapto[1,3,4]oxadiazol-5-yl) quinoxaline 8:

To a sample of compound 2 (2 gm) in pyridine (15 ml), carbon disulfide (2 ml) was added . The mixture was refluxed untill the H_2S ceased (6 h), then allowed to cool. The solid product was collected and recrystallised from ethanol as yellow crystals in 75% yield, mp. 270 °C . Calcd: $C_{11}H_8N_4OS$: C, 54.09; H, 3.30; N, 22.94; S, 13.12 %, found: C, 53.88; H, 3.13; N, 23.15; S, 13.00 %. IR: ν=2900-2700 (SH); ^{1}HNMR (DMSO-d_6): 3.00(s,3H,CH_3), 4.5(s,1H,SH), 7.6-8.00(m,4H,ArH).

2 Methyl-3(2-substitutedthio[1,3,4]oxadiazxol-5-yl)quinoxalin 9:

A mixture of compound 8 (0.01 mol), α-halo compound (0.01 mol) and sod. acetate (0.012 mol) in ethanol (25 ml) was heated under reflux for 3 h, then allowed to cool. The solid product was collected, washed well with water and recrystallized from ethanol. The physical constants and spectral data of compounds 9a-i are listed in Table 1.

Table 1 physical constants of compounds 7a-c and 9a-i .

No.	M.P. °C	Yield	Molecular Formula	Analytical Data C H N S Cl	Spectral Data IR and ^{1}HNMR(DMSO-d_6)
7a	175	68	$C_{16}H_{14}N_4O$ (278.31)	69.05 5.07 20.13 68.86 4.88 20.34	3320,3210(2NH), 1680(C=O); ^{1}HNMR 2.70 (s, 3H,CH_3), 7.5-8.2 (m, 9H, ArH), 9.9 (s, 1H, NH), 12.5(s,1H,NH).
7b	280	65	$C_{17}H_{16}N_4O$ (292.34)	69.85 5.52 19.16 69.80 5.44 19.02	3350,3200 (2NH),1685 (C=O); ^{1}HNMR 2.5, 2.75(2s,6H,2CH_3),7.5-8.2 (m,9H,ArH), 9.8 (s, 1H,NH),12.3 (s, 1H, NH).
7c	286 subli	68	$C_{17}H_{16}N_4O_2$ (308.34)	66.22 5.23 18.17 66.44 5.00 17.98	3340,3230(2NH),16750(C=O); ^{1}HNMR 2.7, 3.35 (2s, 6H,2 CH_3), 7.5-8.2 (m,9H, ArH), 9.85(s,1H,NH),12.4(s,1H, NH).
9a	135	83	$C_{15}H_{14}N_4O_3S$ (330.36)	54.54 4.27 16.96 9.70 54.68 4.04 17.05 9.54	3050(CH aromatic), 2950 (CH aliphatic), 1720(C=O); ^{1}HNMR 1.5 (t, 3H, CH_3 ester), 2.75 (s,3H,CH_3),4.25(q,2H,CH_2),7.5-8.2 (m, 8H, ArH)
9b	243	72	$C_{13}H_{11}N_5O_2S$ (301.32)	51.82 3.86 23.24 10.64 51.86 4.02 23.16 10.60	3250,3150(NH_2),1690(C=O);^{1}HNMR 2.75 (s,3H,CH_3),4.35(s,2H,CH_2), 7.5-8.2 (m. 8H. ArH), 6.7 (s, 2H, NH_2).

Continued Table 1

9c	170	74	$C_{19}H_{15}N_5O_2S$ (377.42)	60.47 4.01 18.56 8.49 60.16 4.22 18.64 8.28	3250(NH), 1690(C=O); ^{1}HNMR:285 (s,3H, CH_3),4.35(s,2H,CH_2), 7.5-8.2 (m, 9H, ArH), 12.3 (s, 1H, NH).
9d	286	76	$C_{20}H_{19}N_5O_2S$	61.05 4.87 17.81 8.13	3200(NH), 1680(C=O); ^{1}HNMR(DMSO-d_6): 2.35(s,3H,CH_3),2.75(s,3H,CH_3),4.35(s,2H, CH_2),7.5-8.2(m,8H,ArH),12.25(s,1H, NH).
9e	215	70	$C_{20}H_{19}N_5O_3S$	58.67 4.68 17.10 7.83 58.90 4.82 16.90 8.04	3230(NH),1670(C=O);^{1}HNMR: 2.75 (s, 3H, CH_3), 3.5 (s, 3H, CH_3), 4.5 (s, 2H, S-CH_2), 7.5-8.2(m,8H,ArH),(12.35(s,1H, NH).
9f	235	80	$C_{21}H_{19}N_5O_3S$	55.14 3.90 16.92 7.75 8.57 55.00 4.12 17.08 8.00 8.42	3250(NH),1700,1690(2C=O);^{1}HNMR:2.75 (s,3H,CH_3),3.35(s,3H,CH_3),4.4 (s, 2H, CH_2), 7.5-8.2(m,8H,ArH),12.25(s,1H, NH).
9g	293	78	$C_{19}H_{16}ClN_5O_2S$	55.00 4.12 17.04 7.72 8.40 54.86 3.96 16.86 7.64 8.32	3150(NH),1690(C=O); ^{1}HNMR: 2.75 (s, 3H, CH_3), 4.45 (s, 2H,CH_2), 7.5-8.2 (m, 8H, ArH), 12.3 (s, 1H, NH).
9h	180	86	$C_{19}H_{16}N_4O_2S$	62.62 4.43 15.38 8.78 62.84 4.62 15.52 9.00	3050(CH aromatic), 2950 (CH aliphatic), 1690(C=O); ^{1}HNMR: 2.75 (s, 3H, CH_3), 4.40(s, 2H, CH_2), 7.5-8.2 (m, 9H, ArH).
9i	190	84	$C_{19}H_{15}ClN_4O_2S$	57.21 3.79 14.05 8.04 8.89 57.00 4.02 13.86 7.88 9.06	3050(CH aromatic),2950(CH aliphatic), 1690(C=O); ^{1}HNMR: 2.75 (s,3H, CH_3), 4.40 (s, 2H, S-CH_2),7.5-8.2 (m, 8H, ArH).

2-Methyl-3-[4-amino-3-mercapto[1,2,4]triazol-5-yl]quinoxaline 10:

A mixture of compund **8** (0.01mol) and hydrazine hydrate (0.012 mol) in pyridine (30 ml) was refluxed for 10 hrs., then allowed to cool. The solid product was collected and recrystallised from DMF as yellow crystals in 75% yield, mp. 295 °C. Calcd.$C_{11}H_{10}N_6S$:C, 51.15; H, 3.90; N, 32.54; 12.41; found:C, 51.06; H, 4.12; N, 32.30; S, 12.58%. IR:ν= 3400,3300(NH_2). and 2850-2700 (SH); ^{1}HNMR (DMSO-d_6): 3.0(s,3H,CH_3), 4.5 (s, 1H, SH), 6.5(s,2H,NH_2), 7.7-8.4(m,4H, ArH).

2-Methyl-3-[4-amino-3-ethoxycarbonylmethylthio[1,2,4]triazol-5-yl]quinoxaline 11:

A mixture of compound **10** (0. 01 mol), ethyl chloroacetate (0.01 mol), and sod. acetate (0.012 mol) in ethanol (20 ml) was refluxed for 3 h, then allowed to cool. The solid product was collected, washed well with water, and recrystallized from ethanol as white crystals in 76% yield, m.p. 147 °C.

Calcd. $C_{15}H_{16}N_6O_2S$:C, 52.13; H, 4.68; N, 24.40; S, 9.31; found:C, 51.92; H, 4.74; N, 24.56; S, 9.18%. IR:ν= 3350,3250(NH_2), 1700(C=O); ^{1}HNMR(DMSO-d_6): 1.0-1.4 (t,3H,CH_3), 2.95 (s, 3H, CH_3), 4.0-4.2 (q,2H,CH_2 ester), 4.45 (s,2H,SCH_2), 6.4(s, 2H, NH_2), 7.7-8.3(m, 4H,ArH).

3-(Methylquinoxalin-3-yl)-7[H]-([1,2,4]triazolo[3,4-b][1,3,4]thiadiazine 12:

A mixture of compound **10** (0.01 mol), phenacyl bromide (0.01 mol) and sod. acetate (0.012 mol) was refluxed for 4 h, then allowed to cool. The solid product was collected, washed with water and recrystallized from ethanol as white crystals in 70 % yield, m.p.270 °C. Calcd. $C_{19}H_{14}N_6S$: C, 63.67; H, 3.94; N, 23.45; S, 8.94%; found: C, 63.52; H, 4.15, N, 23.64; S, 9.06%. IR:ν= 3050(CH aromatic),

2950(CH-aliphatic), 1600 (C=N), and show disappearance of bands characteristic for NH_2 group in the starting materials; ^{1}HNMR(DMSO-d_6): 3.2 (s, 3H, CH_3), 4.4(s, 2H, CH_2), 7.3-8.2 (m, 9H,ArH).

N^1-(3-Methylquinoxalin-2-oyl)-N^2-phenyl-thiosemicarbazide 13:

To a sample of compound **2** (0.01 mol) in ethanol (20 ml), phenyl isothiocyanate (0.01 mol) was added, the mixture was refluxed for two hours, then allowed to cool. The solid oroduct was collected and recrystallized from ethanol to give compound **13** as white crystals in 78% yield mp.168 °C.

Calcd. $C_{17}H_{15}N_5OS$: C,60.52; H, 4. 48; N, 20. 76; S, 9.50%; found: C, 60.38; H, 4.68; N, 21.00; S, 9.62%. IR:ν= 3490, 3300-3100 (3NH), 1690 (C=O), 1250 (C=S).

2-Methyl-3-[3-mercapto-4-phenyl(1,2,4)triazol-5-yl]quinoxaline 14):

A sample of compound **13** (0.5 gm) was refluxed with alcoholic NaOH (10%,10ml) on water bath for 3 h, then allowed to cool and acidified with HCl. The solid product was collected and recrystallised from ethanol as white crystals in 72% yield, mp.148-150 °C. Calcd. $C_{17}H_{13}N_5S$: C, 63.93; H, 4.10; N, 21.93; S, 10.04%; found: C, 64.00; H,4.32; N, 22.04; S,10.18%. IR:ν= 3050(CH aromatic), 2850-2750(SH); ^{1}HNMR ($CDCl_3$): 3.1(s,3H,CH_3), 5.6 (s,1H,SH), 7.2-8.2 (m,9H, ArH).

2-Methyl-3-[3-substitutedthio-4-phenyl[1,2,4]triazol-5-yl]quinoxaline 15):

A mixture of compound **14** (0.01 mol), ethyl chloroacetate or phenacyl bromide (0.01 mol) and sod. acetate (0.012 mol) was refluxed for 4 h, then allowed to cool. The solid product was collected, washed with water and recrystallized from ethanol as white crystals.

15a, Yield 78%, m.p. 115-17 °C. Calcd. $C_{21}H_{19}N_5O_2S$:C,62.21;H, 4.72; N, 17.27; S, 7.91%; found: C, 62.00; H, 4.54; N, 17.48; S, 8.06%. IR:ν= 1700(C=O); ^{1}HNMR (DMSO-d_6):1 .25 (t, 3H, CH_3), 3.00 (s, 3H,CH_3), 4.1(q,2H,CH_2), 4.35 (s,2H,SCH_2), 7.65-8.2(m, 9H, ArH).

15b: Yield 72% as yellow crystals , m.p. 280 °C. Calcd. $C_{25}H_{19}N_5OS$: C, 68.63; H, 4.38; N, 16.01; S, 7.33%; found: C, 68.84; H, 4.22; N, 15.88; S, 7.47%. IR:ν= 1670(C=O; ^{1}HNMR(DMSO-d_6): 2.95 (s, 3H, CH_3), 4.2(s,2H,SCH_2), 7.6-8.3(m, 14H, ArH).

3-Aryl pyrido[3,4-b]quinoxalin-1[2H]one 16:

A mixture of **3** (0.01 mol), aromatic aldehyde (0.012 mol) and drops of piperidine was heated for 10 minutes then ethanol (20 ml) was added and refluxed for additional 1/2 hour, The solid product was separated from the hot mixture was collected and recrystallised from DMF as purple crystals.

16a, Yield 78%, mp.>300 °C. Calcd. $C_{17}H_{11}N_3O$: C,74.71; H,4.06; N,15.38%; found:C,74.68; H,3.88; N,15.50%; IR:ν=3250(NH), 1670(CO); ^{1}HNMR(CF_3COOD): 7.0(s,1H, CH pyridine), 7.4-8.4 (m, 9H, ArH)

16b, Yield 74%, mp.>300 °C. Calcd. $C_{18}H_{13}N_3O_2$: C,71.28; H, 4.32; N,13.58%; found: C,71.06; H, 4.52; N,13.40%. IR:ν=3280(NH), 1670(C=O); ^{1}HNMR (CF_3COOD): 3.2 (s,3H,OCH_3), 7.0 (s, 1H, CH pyridine), 7.45 -8.2 (m,8H,ArH).

3-(4-Methoxyphenyl)-3-phenylazopyrido[3,4-b]quinoxalin-1[2H]one 17:

To a cooled solution of **16b** (0.005 mol) in 10% alcoholic sodium hydroxide (30 ml), phenyl diazonium chloride was added dropwise with stirring during 1/4 h, after the addition was finished the stirring was continued for additional 2h. The solid product was collected and recrystallized from acetic acid as yellow crystals in 72% yield, m.p. 230 °C. Calcd. $C_{24}H_{17}N_5O_2$: C, 70.75; H, 4.21; N, 17.19%; found: C, 71.00; H, 4.04, N, 16.98%. IR:ν= 3180(NH), 1670(C=O), 1600 cm^{-1} (N=N); ^{1}HNMR (CF_3COOD): 3.2 (s, 3H,OCH_3),7.1-8.4 (m, 13H,ArH).

3-(p-Methoxyphenyl)-4-bromopyrido[3,4-b]quinoxalin-1[2H]one 18:

To a solution of **16b** (0.01 mol) in acetic acid (20 ml), bromine solution (0.01 mol) in acetic acid (10 ml) was added dropwise with stirring during 1/2 h. After the addition was finished the stirring was continued for 1h, then poured into cold water (20 ml). The solid product was collected and recrystallized from acetic acid as red crystals in 82% yield , m.p. 284-87 °C dec. Calcd. $C_{18}H_{12}BrN_3O_2$: C, 56.56; H, 3.16; Br, 20.91; N, 10.99%; found: C, 56.36; H, 3.00; Br, 21.04; N, 11.12%. IR:ν= 3240 (NH), 1680(C=O).

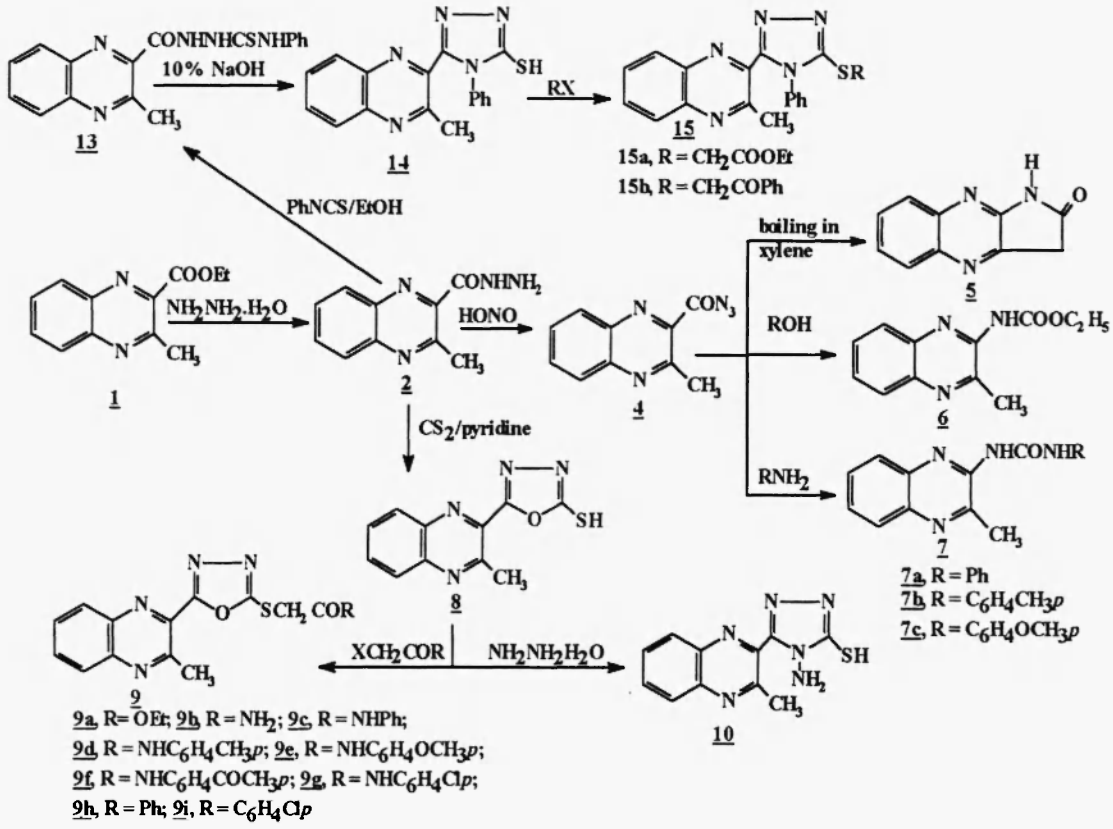
3-(4-Methoxyphenyl)-4-nitrosopyrido[3,4-b]quinoxalin-1[2H]one 19:

To a solution of compound **16b** (0.01 mol) in acetic acid (20 ml), sod. nitrite solution (0.1 mol) was added dropwise with stirring during 1/2 h. After the addition was finished the stirring was continued for 1h. The solid product was collected, washed with water and recrystallized from acetic acid as yellow crystals in 82% yield, m.p. 207 °C. Anal. Calcd. for $C_{18}H_{12}N_4O_3$: C, 65.06; H, 3.64; N, 16.86%; Found: C, 64.88; H, 3.88; N, 17.04%. IR:ν=3180(NH), 1680(C=O); ^{1}HNMR (CF_3COOD): 3.2 (s, 3H, OCH_3), 7.1-8.25 (m,8H,ArH).

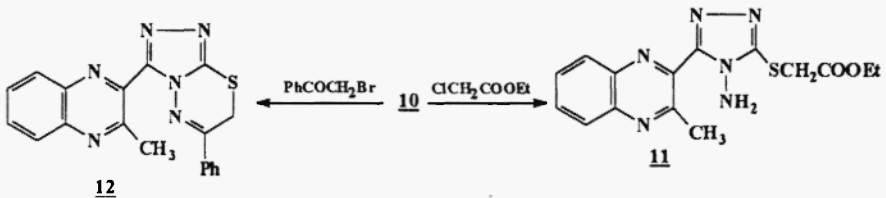
Results and Discussion

Ethyl 3-methylquinoxaline-2-carboxylate (8) **1** when refluxed with hydrazine hydrate or with ammonia solution in ethanol, 3-methylquinoxaline-2-carbohydrazide **2** or 3-methylquinoxaline-2-carboxamide **3** were obtained. The produced carbohydrazide **2** was used as verstile starting material for obtaining many other compounds . Compound **2** was converted into the corresponding carboazide **4** when treated with

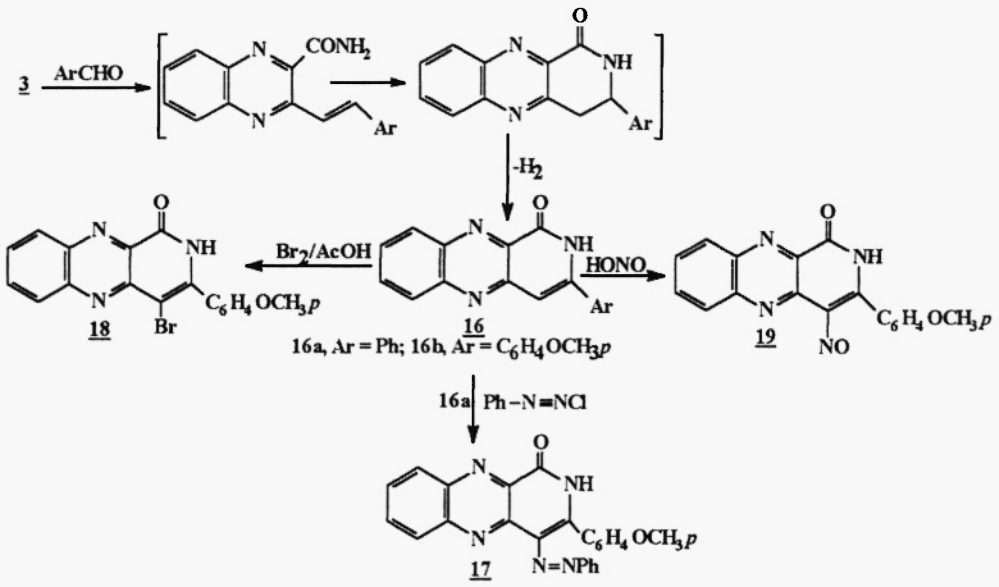
sodium nitrite in cold acetic acid. The carboazide **4** underwent Curtius rearrangement when boiled in xylene, ehanol and amines to give the pyrroloquinoxaline **5**. Ethyl 2-methylquinoxaline-3-carbamate (**6**) urea derivatives **7a-c** respectively. Treatment of **2** with carbon disulfide in pyridine led to the formation of oxadiazolyl derivative **8**, which was S-alkylated using halocompounds to give S-alkyl derivative **9a-i**.N-Aminomercaptotriazolylquinoxaline derivative **10** resulted from the condensation of **8** with hydrazine hydrate.



Mercaptotriazolyl compound **10** underwent S-alkylation using ethyl chloroacetate in refluxed ethanol in the presence of sodium acetate to give thioester derivative **11**, while in case of phenacyl bromide as alkylating agent, S-alkylation was occurred followed by cyclization under the reaction condition into the thiadiazino derivative **12**.



Another triazolylquinoxaline was obtained from the reaction of carbohydrazide **2** with phenyl isothiocyanate in ethanol to give the intermediate **13,** which upon heating in 10% sodium hydroxide solution was cyclized into **14**. This was easily S-alkylated with α-halocompounds to give S-alkyl. On the other hand the o-methylcarboxamide **3** was reacted with aromatic aldehydes in presence of catalytic amount of piperidine to produce the styryl derivative, which under the reaction condition was cyclized into pyridoquinoxaline **16** . The pyrido[3,4-b]quinoxaline derivative **16b** was submitted to electrophilic substitution reaction in the pyridine nucleus, this it was coupled with phenyldiazonium salt to give phenylazo derivative **17**, and also it reacted with bromine and nitrous acid to give compounds **18,19.**



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Erratum :

On page 581 (in the index of vol. 3, issue 6, 1997) for the manuscript entitled "Synthesis of sterically encumbered 2,9-diarylsubstituted phenanthrolines. Key building blocks for the preparation of mixed (bis-heteroleptic) phenanthroline copper(I) complexes" (referring page 493) and on the back cover page (table of contents, vol. 3, issue 6, 1997) authors should be read as :
M. Schmittel, U. Lüning, M. Meder, A. Ganz, C. Michel and M. Herderich