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SYNTHESIS OF NOVEL ANALOGS 3,4- DIHYDRO-1*H*-QUINOLIN-2-ONE DERIVATIVES AS TYPICAL ANTIDEPRESSANT, SEDATIVE AND ANTI-PARKINSON AGENTS.

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ABSTRACT

This study aimed at synthesis of the potential Antidepressant and Sedative, anti-Parkinson's activity of structurally diverse derivatives of lead compound 3, 4-dihydro-1*H*-quinolin-2-one; synthesized via straightforward and efficient synthetic process. The structures of the compounds were characterized by spectral data (IR and 1H-NMR).

KEYWORDS

Potential, Antidepressant, Sedative, Anti-Parkinson's.

INTRODUCTION

Heterocyclic antidepressant drugs have greatly improved the outcome of depression. The heterocyclic antidepressants are the mainstay of antidepressant treatment and the development of new synthetic heterocyclic compounds as antidepressant, sedative, or analgesic agents has progressed considerably during the past decade. The present paper describes the synthesis of Structurally diverse analogs of lead compound 3,4- Dihydro-1*H*-Quinolin-2-One

EXPREMENTAL

Melting points were determined in open capillary tubes and are uncorrected. The time required for completion of the reaction was monitored by TLC using Silica gel-G plates and spots were exposed in UV chamber. IR spectra were recorded on a Perkin Elmer 1800 (FTIR) spectrometer 1H NMR spectra (DMSO) or ($CdCl_3$) were taken on a DRX-300 spectrometer (300 MHz) using TMS as internal standard and chemical shifts are expressed in δ ppm. All the NMR and I.R of the novel compounds from stage four to stage eight are mentioned in tabular format.

Detailed synthetic procedure-

Stage 1- synthesis of 7-(n-halo alkoxy)-2(1*H*)-quinolinone

(i) Synthesis of 7-(4'-bromobutoxy)-2(1*H*)-quinolinone[1]

1 mole of 7-hydroxy carbostryril and 4 moles respective dibromo butane and 1.1 moles of potassium carbonate are added in DMF and stirred till the reaction is completed. Completion of the reaction is checked on TLC (chloroform-methanol 8:2). Water is added to the reaction mixture and aqueous layer is extracted with ethyl acetate .The organic layer is subjected to vacuum distillation to get desire product. The recrystallised with ethanol. Yield-85%

(ii) Synthesis of 7-(3-chloropropoxy)-2(1H) quinolinone[2].
1 mole of 7-hydroxy carbostryril and 4 moles respective1- bromo-3-chloro propane and 2 moles of NaOH are taken in isopropyl alchohol and refluxed for 6-7 hrs. Completion of reaction is checked on TLC (chloroform-methanol 8:2).It is filtered hot and filtrate is cooled to 0^0-5^0C.The product is filtered and recrystallised in ethanol. Yield-85%

(iii) Synthesis of 7-(5-chloropentoxy)-2(1H) quinolinone[2].
1 mole of 7-hydroxy carbostryril and 4 moles respective 1-bromo-5-chloro pentane and 2 moles of NaOH are taken in isopropyl alcohol and refluxed for 6-7 hrs. Completion of reaction is checked on TLC (chloroform-methanol 8:2).It is filtered hot and filtrate is cooled to 0^0-5^0C.The product is filtered and recrystallised in ethanol. Yield-65%

(iv) Synthesis of 7-(6-chlorohexoxy)-2(1H) quinolinone[2].
In manner similar to above process this compound is synthesized.Yield-45%

(v) Synthesis of 7-(2-Chloroethoxy)-2(1H) quinolinone[1].
In round bottom flask 7-hydroxy carbostyril is taken. It is flushed with DMF. To the reaction mixture 1.1 mol of potassium carbonate is added. It is stirred for 1 hr. To these reaction mixture 3 moles of dibromo ethane is added. The reaction mixture is stirred for 20 hrs. Completion of reaction is checked on TLC (chloroform-methanol 8:2).After completion of reaction water is added to reaction mixture. It is extracted with ethyl acetate. Organic layer removed under vacuum. Oily layer obtained is stirred in hexane to get solid. It is recrystallised by ethanol.Yield-85%

Stage 2- synthesis of 1-aryl piperazine hydrochloride[3].
1 mole of aromatic amines and 1.2 moles bis-(2-chloro ethyl amine) hydrochloride is taken in xylene. PTSA is used as catalyst in this reaction and whole reaction mixture is refluxed in xylene for 48 hrs. Completion of reaction is checked on TLC (chloroform 100%).Reaction mixture is filtered off and washed with acetone to remove traces of un-reacted aromatic amine. The product is recrystallised using ethanol.Yield-80-85%

Stage 3- synthesis of indolo [2,3-b] indoloquinoxalines[4].
1 mole of isatine and 1 mole of o-phenylenediamine refluxed in glacial acetic acid for 5 hrs. Completion of reaction is checked on basis of TLC (chloroform: methanol 7:3). After completion of reaction reaction mixture is poured into water and filtered off. Product is recrystallised into DMF. Yield-90%

Stage 4- general synthesis of 7-[n-[4 -(substituted phenyl)-1-piperazinyl]alkoxy]-3,-dihydro-2(1H)-quinolinone[2].

1 mole of aryl piperazine, 1 mole of k_2CO_3, 1 mole of stage 1 product and 1 mole of NaI taken in acetonitrile and refluxed till the reaction is completed. TBAB is used as catalyst. Completion of reaction is checked on TLC (chloroform: methanol 8:2). Acetonitrile is subjected to vacuum and product is recrystallised using proper solvent.

Stage 5- General synthesis of 1-alkyl-7-[n-[4-(substituted phenyl)-1-piperazinyl] alkoxy]-3,-dihydro-2(1H)-quinolinone[2].

1 mole of stage 4 product is taken into round bottom flask, DMF is added as solvent, and 1mol of NaH added. It is stirred for half n hour, and then respective alkyl halide as added into DMF and stirred at room temperature for 8 hrs or till the reaction is completed. Completion of reaction is checked on TLC (chloroform: methanol 8:2).After completion of reaction water is added to reaction mixture and aqueous layer is extracted with ethyl acetate. Ethyl acetate is removed under vacuum and oily product is stirred with hexane rapidly to get solid material. These products are recrystallised using suitable solvent.

Stage 6- General of 1-alkyl-7-[4-[n(N-indolo[2,3-b] quinoxalines] alkoxy]-3,-dihydro-2(1H)-quinolinone[2].

1 mole of stage 3 product (indolo[2,3-b] quinoxalines) , 1 mole of K2CO3, 1 mole of 7-(n'-alkyl alkoxy)-2(1*H*)-quinolinone, and 1 mole of NaI along with TBAB as catalyst is taken round bottom flask and refluxed in acetonitrile till the reaction is completed. Completion of reaction is checked on TLC (chloroform: methanol 8:2). Acetonitrile is subjected to vacuum and product obtained is purified by column chromatography.

This product (1 mol) is taken in DMF, 1 mole of NaH is added into the mixture and respective alkyl halide is added. The reaction mixture is stirred at room temperature at about 8 hrs or till the reaction is completed. After completion of reaction water is added to reaction mixture and extracted with ethyl acetate. Ethyl acetate is subjected to vacuum, and oily product is washed with hexane to remove traces of alkyl halide. The solid product obtained is purified by column chromatography (chloroform –methanol 8:2)

Stage 7- Synthesis of N-(n-halo alkyl) isatine -7-[4-[4-(2,3-dichloro phenyl)-1-piperazinyl) butoxy]-3,4-dihydro-2(1H)-quinolinone[2].

1mole 7-[4-[4-(2,3-dichloro phenyl)-1-piperazinyl) butoxy]-3,4-dihydro-2(1H)-quinolinone (stage 4)and 1 mole of NaH is taken in DMF in round bottom flask, It is stirred for half n hour and 1 mole of N-(n-halo alkyl)isatine is added. The reaction mixture is stirred at room temperature for 8 hrs or till the reaction goes for completion. Completion of reaction is checked on TLC (chloroform: methanol 8:2). After completion of reaction water is added to reaction mixture and extracted with ethyl acetate. Ethyl acetate is subjected to vacuum, and oily product is stirred with hexane to get solid product. The solid product obtained is purified by column chromatography (chloroform –methanol 8:2).

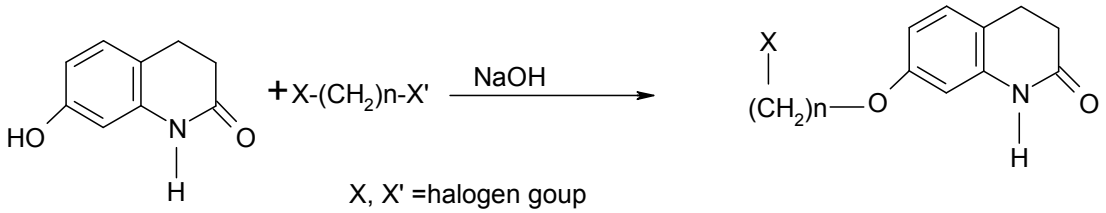
Stage 8- Synthesis of N-(3-chloropropyl) indolo [2,3-b] quinoxalines -7-[4-[4-(2,3-dichloro phenyl)-1-piperazinyl) butoxy]-3,4-dihydro-2(1H)-quinolinone[2].

1 mole 7-[4-[4-(2,3-dichloro phenyl)-1-piperazinyl) butoxy]-3,4-dihydro-2(1H)-quinolinone (stage 4)and 1 mole of NaH is taken in DMF in round bottom flask, it is stirred for half n hour and 1 mole of N-(3-chloropropyl) indolo[2,3-b] quinoxalines is added. The reaction mixture is

stirred at room temperature for 8 hrs or till the reaction goes for completion. Completion of reaction is checked on TLC (chloroform: methanol 8:2). After completion of reaction water is added to reaction mixture and extracted with ethyl acetate. Ethyl acetate is subjected to vacuum, and oily product is stirred with hexane to get solid product. The solid product obtained is purified by column chromatography (chloroform –methanol 8:2).

Reaction scheme-

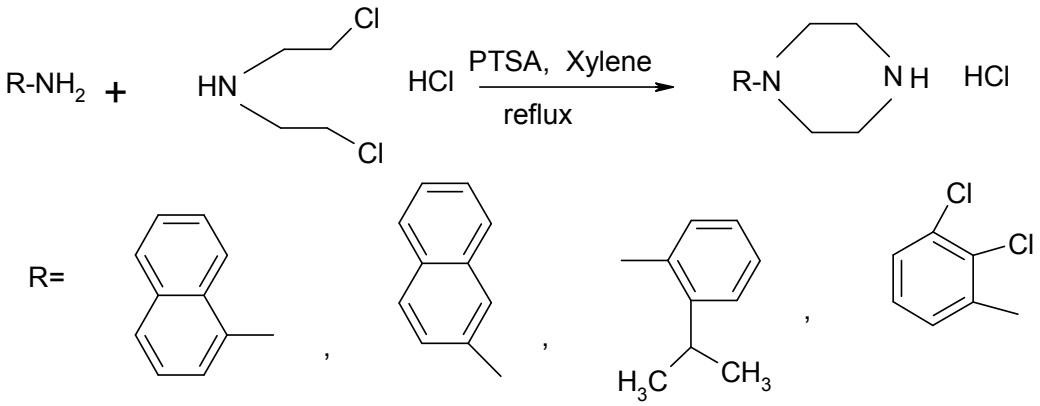
Stage 1-



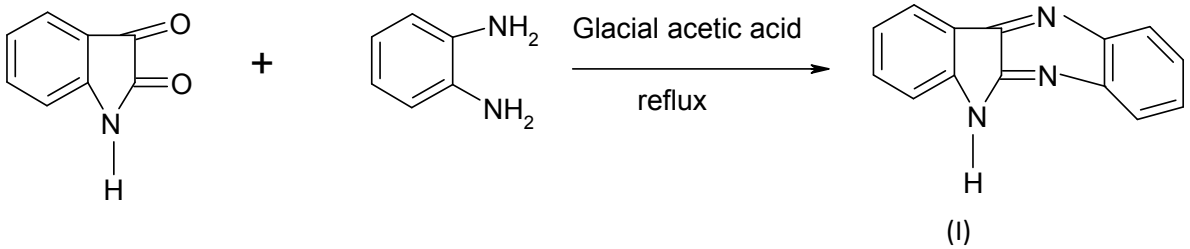
X, X' =halogen goup

X/X' = Cl or Br R= $(CH_2)_3$, $(CH_2)_4$, $(CH_2)_5$, $(CH_2)_6$

Stage 2-

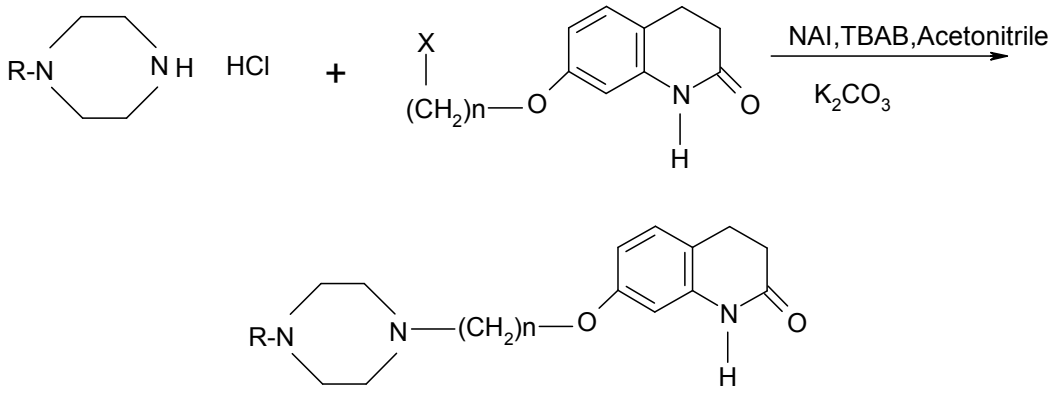


Stage 3-



(I)

Stage 4-



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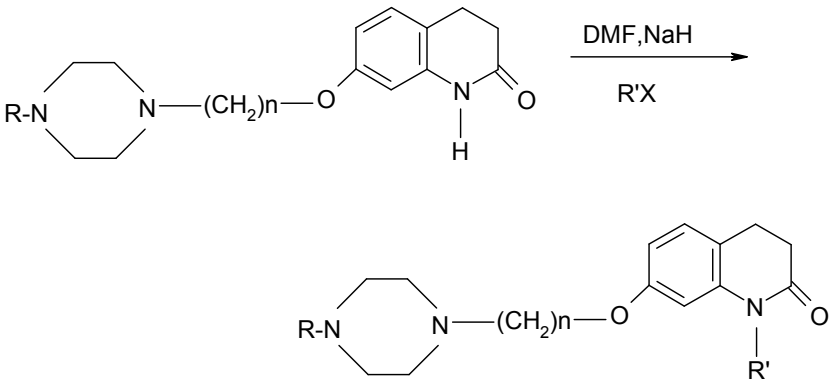
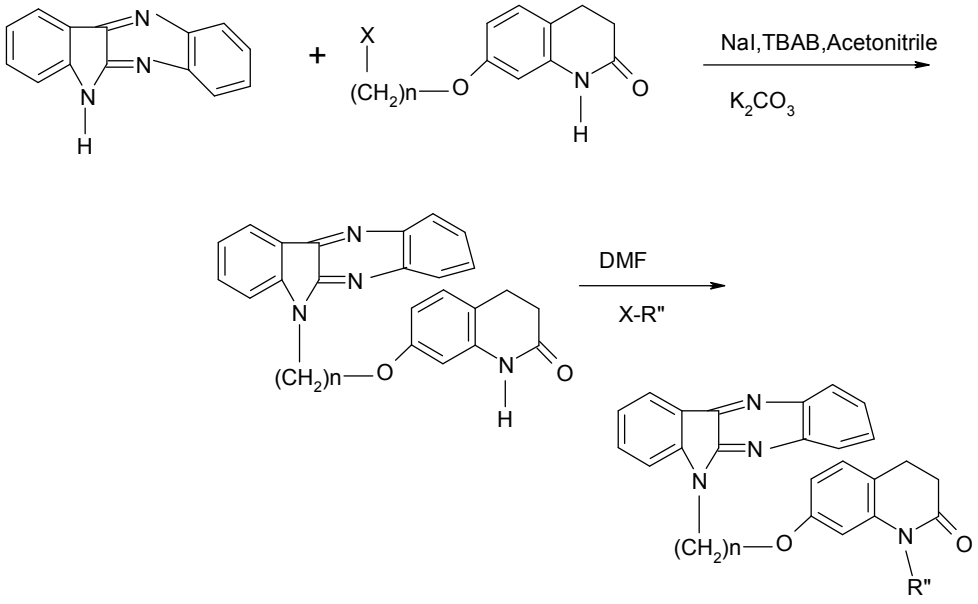
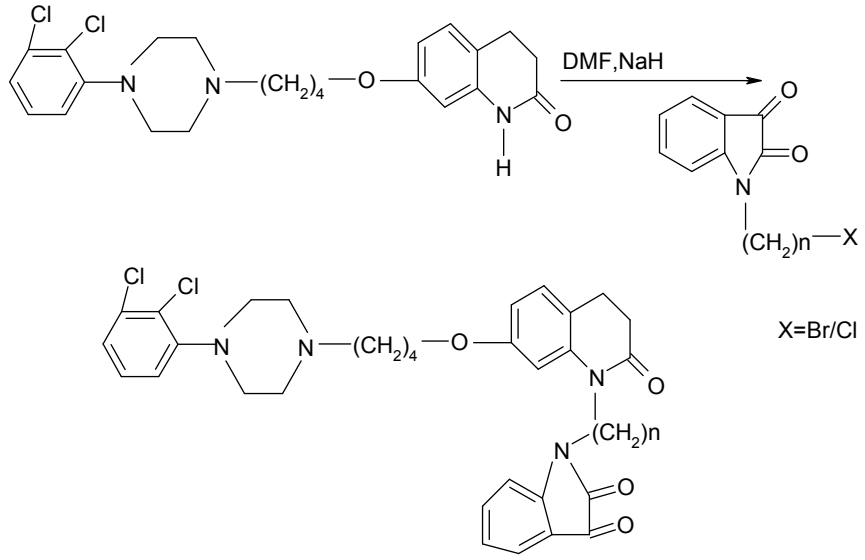


Stage 6

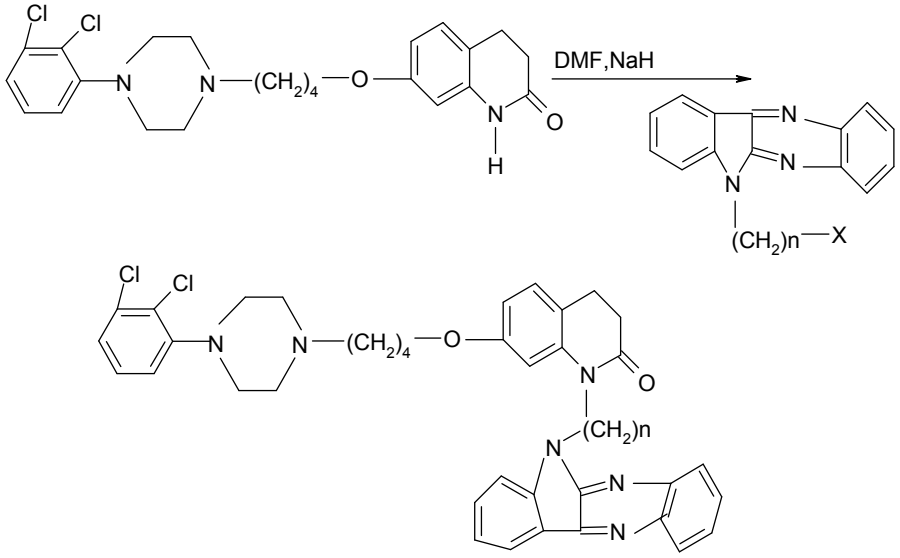


Stage 7-



n=2,

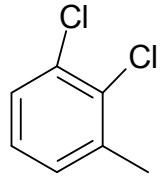
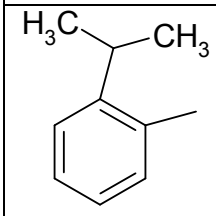
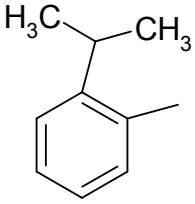
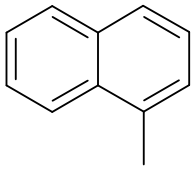
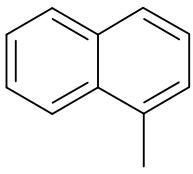
Stage 8-

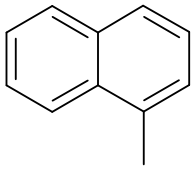
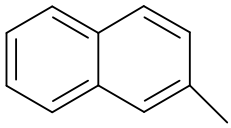
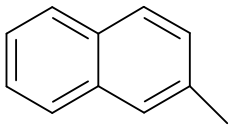
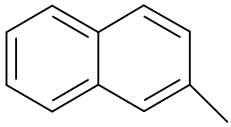


X=cl , n=3

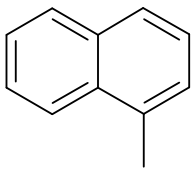
RESULT AND DISCUSSION
Table No 1 –

1A- NMR data of stage four compounds.

R	n	NMR (DMSO)
	4	1.62-1.52(m,2H,CH_2), 1.76-1.69(m,2H,CH_2), 2.35-2.27(t,4H,piperazine), 2.50(t,2H,CH_2-C=O), 2.85-2.70(m,2H,Ar-CH_2), 3.28(t,4H,piperazine), 3.41(t,2H,N-CH_2), 3.93-3.89(t,2H,O-CH_2), 6.55-6.42(m,2H,Ar-H), 7.05-7.02(d,1H,Ar-H), 7.27-7.10(m,1H,Ar-H), 7.35-7.33(d,2H,Ar-H),10.00(s,1H,N-H)
	2	1.22-1.15 ppm (d, 6H, -CH3), 2.60-2.43 (m, 2H, CO-CH2 of carbostyryl), 2.70-2.60 (m, 4H, CH2-N-CH2 of piperazine), 2.84-2.71 (t, 2H, -CH2), 3.10-2.86 (m, 3H, -CH2 of carbostyryl, -CH), 3.66-3.44 (m, 4H, CH2-N-CH2 of piperazine), 4.26-4.06 ppm(t, 2H, -CH2), 6.61-6.46 (d, 1H, -ArH), 6.71-6.62 (m, 2H, -ArH), 7.00-6.73 (d, 1H, -ArH), 7.39-7.13 (m, 3H, -ArH), 10.60 (s, 1H,N-H)
	3	1.29-1.05 ppm (d, 6H, -CH3), 1.92-1.75 (t, 2H, -CH2), 2.60-2.40 (m, 4H, CO-CH2 of carbostyryl, -CH2), 2.80-2.60 (m, 4H,CH2-N-CH2 of piperazine), 3.29-2.95 (m, 3H, -CH2 of carbostyryl, -CH), 3.82-3.48 (m, 4H, CH2-N-CH2 of piperazine), 4.25-4.04 (t, 2H, -CH2), 6.59-6.26 (d, 1H, -ArH), 6.95-6.70 (m, 2H, -ArH), 7.21-7.04 (d, 3H, -ArH), 7.39-7.21 (m, 1H, -ArH), 10.67(s,1H,N-H)
	3	1.95-1.86(m,2H,CH_2), 2.08-2.06(t,2H,Ar-CH_2),2.47-2.38(m,4H,piperazine), 2.80-2.75(t,CH_2-C=O), 3.20-3.30(s,4H,piperazine), 3.30(t,2H,N-CH_2), 4.04-3.94(m,2H,)-CH_2) , 6.51-6.44(m,2H,Ar-H), 7.06-7.03(d,1H,Ar-H), 7.15(d,1H,Ar-H), 7.27-7.22(t,1H,Ar-H),7.40-7.35(m,2H,Ar-H), 7.75-7.69(m,3H,Ar-H), 9.98(s,1H,N-H)
	4	1.79-1.65(m,4H,CH_2), 2.08-2.06(m,4H,piperazine), 2.70(s,2H,CH_2-C=0), 2.80-2.90(t,2H,Ar-CH_2), 3.00(s,4H,piperazine), 3.44-3.42(t,2H, N-CH_2), 4.00(t,2H,)-CH_2), 6.40-6.60(m,2H,Ar-H), 7.20-7.00(m,2H,Ar-H), 7.40-7.70(m,4H,Ar-H), 7.80-7.90(d,1H,Ar-H), 8.00-8.10(d,1H,Ar-H), 10.04(s,1H,N-H)

	6	1.55-1.43(m,4H,CH_2), 1.83-1.72(m,4H,CH_2), 2.46-2.43(t,2H, CH_2-C=0), 2.50(4H,piperazine), 2.89-2.84(t,2H,Ar-CH_2), 3.21(s,4H,piperazine),3.50(m,2H,N-CH_2), 4.03-3.98(t,2H,O-CH_2), 6.79-6.78(t,1H,Ar-H), 6.90-6.79(m,2H,Ar-H), 7.21-7.19(d,1H,Ar-H), 7.56-7.44(m,3H,Ar-H),7.69-7.66(d,1H,Ar-H), 7.94-7.91(m,1H,Ar-H), 8,15-8.12(d.1H,Ar-H), 8.89(s,1H,N-H)
	4	1.65-1.55(m,2H,CH_2), 1.82-1.69(m,2H,CH_2), 2.42-2.27(t,4H,piperazine), 2.60-2.50(t,2H,CH_2-C=O), 2.80-2.66(t,2H,Ar-CH_2), 3.10(s,4H,piperazine), 3.65-3.44(m,2H,N-CH2), 3.95-3.90(t,2H,O-CH_2), 5.51-6.44(m,2H,Ar-H), 7.05-7.02(d,2H,Ar-H), 7.14-7.13(d,2H,Ar-H), 7.27-7.22(t,2H,Ar-H), 7.40-7.34(t,2H,Ar-H), 7.75-7.69(t,3H,Ar-H), 9.99(s,1H,N-H)
	3	1.95-1.86(m,2H,CH_2), 2.43-2.28(t,2H,CH_2-C=O),2.55-2.50(t,4H,piperazine), 2.80-2.75(t,2H,Ar-CH_2), 3.02(s,4H,piperazine), 3.55-3.41(t,2H, N-CH2), 3.99-3.95(t,2H,O-CH_2), 6.52-6.45(m,2H,Ar-H), 7.11-7.03(m,2H,Ar-H), 7,59-7.38(m,4H,Ar-H), 7.88-7.85(d,2H,Ar-H), 8.11-8.08(d,2H,Ar-H), 10.01(s,1H,N-H)
	5	1.08-1.03(m,2H,CH_2), 1.54-1.46(m,2H,CH_2), 1.75-1.68(m,2H,CH_2), 2.43-2.38(m,4H,piperazine), 3.50-3.70(t,2H, CH_2-C=O),2.77-2.65(t,2H,Ar-CH_2), 3.02(s,4H,piperazine), 3.92-3.88(t,2H,O-CH_2), 6.50-6.44(m,2H,Ar-H), 7.05-7.02(dd,1H,Ar-H), 7.12-7.09(dd,1H,Ar-H), 7.59-7.39(m,4H,Ar-H), 7.89-7.86m,1H,Ar-H), 8.12-8.08(t,1H,Ar-H), 9.98(s,1H,N-H)

1B- NMR data of stage five compounds

R	n	R’	NMR(DMSO)
	3	C_2H_5	1.30-1.10(t,3H,CH_3), 2.00(m,2H,CH_2), 2.88-2.34(m,10H,piperazine, N-CH_2,Ar-CH_2, O=C-CH_2), 3.10(s,4H,piperazine), 4.03-3.92(m,2H,O=C-N-$\underline{CH_2}$-CH_3), 4.29-4.22(t,2H,O-CH_2), 6.61-6.47(m,2H,Ar-H), 7.03-7.05(d,2H,Ar-H), 7.68-7.24(m,4H,Ar-H), 7.89-7.86(t,1H,Ar-H), 8.12-8.09(d,1H,Ar-H), 10.00(s,1H,N-H)

1C- NMR data of stage six compounds

n	R''	NMR(DMSO)
3	CH_3	2.40-2.30(m,2H,CH_2), 2.50-2.40(t,2H,CH_2-C=O), 2.72-2.64(t,2H,Ar-CH_2), 3.10(s,3H,N-CH_3), 4.00(t,2H,N-CH_3), 4.60(t,2H,O-CH_2), 6.36(d,1H,Ar-H), 6.39(d,1H,Ar-H), 6.98-6.91(d,1H,Ar-H), 7.40-7.35(d,1H,Ar-H), 7.70-7.56(m,4H,Ar-H), 7.82-7.78(d,1H,Ar-H), 8.14-8.08(t,1H,Ar-H), 8.40(d,1H,Ar-H)
5	C_2H_5	1.20-1.10(t,3H,CH_3), 1.70-1.40(d,2H,CH_2), 1.90-1.70(d,2H,CH_2), 2.10-1.90(d,2H,CH_2), 2.80-2.60(s,4H,carbostyril CH_2), 4.10-3.80(t,N-CH_2, N-$\underline{CH_2}$-CH_3), 4.70-4.40(t,2H,O-CH_2), 6.60-6.40(t,2H,Ar-H), 7.00(dd,1H,Ar-H), 7.50-7.40(t,1H,Ar-H), 8.00-7.70(m,4H,Ar-H), 8.11-8.09(dd,1H,Ar-H), 8.27-8.25(dd,1H,Ar-H), 8.39-8.37(dd,1H,Ar-H)
4	C_4H_9	0.45-0.38(m,4H,CH_2),0.88-0.84(m,2H,CH_2), 1.32(d,2H,CH_3),2.20-2.00(m,4H,CH_2,N-CH_2), 2.40-2.60(t,2H,Ar-CH_2),3.20-3.00(t,2H,CH_2-N-C=O,carbostyril),3.70-3.90(t,2H,CH_2-N-C=O),4.63(t,2H,O-CH2), 6.30-6.00(m,2H,Ar-H), 6.90-6.80(m,1H,Ar-H), 7.30-7.10(m,1H,Ar-H), 7.90-7.40(m,6H,Ar-H), 8.20-8.00(dd,1H,Ar-H)

1D- NMR data of stage seven compounds

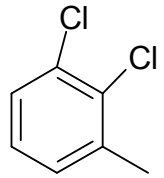
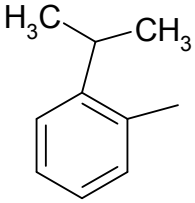
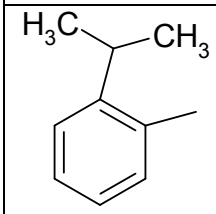
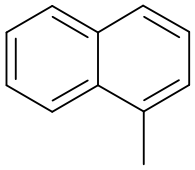
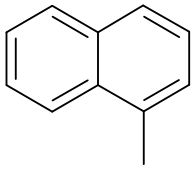
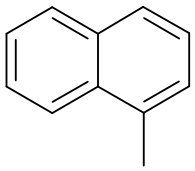
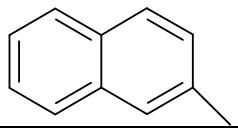
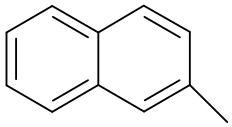
n	X	NMR($CDCl_3$)
2	Br	1.86-1.83(m,4H,CH_2), 2.52-2.41(t,2H,CH2-C=O, carbostyril), 2.65-2.60(m,6H,piperazine, Ar-CH_2), 2.97-2.88(m,4H,piperazine), 2.97(t,2H,N-CH2),3.09(s,4H,isatineCH_2) , 3.99-3.95(t,2H,O-CH_2), 6.33(d.1H,Ar-H), 6.55-6.52(m,1H,Ar-H), 7.00-6.95(m,1H,Ar-H),7.07-7.04(d,2H,Ar-H), 7.17-7.12(m,2H,Ar-H),7.28(s,1H,Ar-H),8.03-7.98(t,2H,Ar-H)

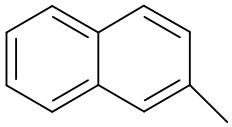
1E- NMR data of stage eight compounds

n	X	NMR(DMSO)
3	Cl	1.50-1.50(m,4H,CH_2),2.24(s,2H,CH_2),2.36-2.34(d,2H,CH_2),2.50-2.49(d,5H,CH_2piperazine,CH),2.772.74(d,2H,CH_2),2.93(s,4H,CH_2,piperazine-,4.04(t,4H,CH_2),4.63(d.3H,CH2,CH), 6.43(s,1H,Ar-H), 6.53-6.50(d,1H,Ar-H),7.11-7.08(t,2H,Ar-H),7.33-7.31(d,2H,Ar-H), 7.50-7.45(m,1H,Ar-H), 7.90-7.76(m,4H,Ar-H),8.15-8.12(d,1H,Ar-H). 8.25-(d,1H,Ar-H),8.45(d,1H,Ar-H)

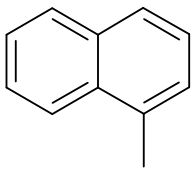
Table No 2-

2A- Melting point and I.R data of stage four compounds

R	n	M.P	I.Rcm^{-1}
	4		779(C-Cl str), 1049(C-N str), 1195(C-O str), 1627(aromatic C=C str), 1674(C=Ostr), 2808(sp3 C-H str), 3050(aromatic C-H str), 3193(N-H str)
	2	130-132^{0}C	1050(C-N str),1188(C-O str), 1589(aromatic C=C str), 1674(C=Ostr), 2823(sp3 C-H str),3047(aromatic C-H str), 3193(N-H str)
	3	210-212^{0}C	1050(C-N str), 1242(C-O str),1504,1604(aromatic C=C str),1681(C=Ostr), 2831(sp3 C-H str),3078(aromatic C-H str),3209(N-H str)
	3	205-208^{0}C	1050(C-N str),1195 (C-O str), 1596(aromatic C=C str), 1689(C=Ostr), 2839(sp3 C-H str), 3047(aromatic C-H str), 3201(N-H str)
	4	220-222^{0}C	1200(C-O str),1589(aromatic C=C str), 1674(C=O str),2893(sp3 C-H str),3055(aromatic C-H str), 3201(N-H str)
	6	196-198^{0}C	1200(C-O str),1600(aromatic C=C str), 1674(C=O str),2815(sp3C-H str), 3047(aromatic C-H str), 3193(N-H str)
	4	178-180^{0}C	1188(C-O str),1589(aromatic C=C str), 1674(C=O str),2869(sp3C-H str), 3047(aromatic C-H str), 3193(N-H str)
	3	165-167^{0}C	1188(C-O str), 1581(aromatic C=C str), 1674(C=O str), 2850(sp3C-H str), 3047(aromatic C-H str), 3193(N-H str)

	5	182-184^{0}C	1180(C-O str),1596(aromatic C=C str),1681(C=O str),2877(sp3C-H str),3062(aromatic C-H str), 3193(N-H str)

2B- Melting point and I.R data of stage five compounds

R	n	R'	M.P	I.Rcm^{-1}
	3	C_2H_5	120-122^{0}C	1200(C-O str), 1596(aromatic C=C str),1689(C=O str),2831(sp3C-H str), 3050(aromatic C-H str),

2C- Melting point and I.R data of stage six compounds

n	R''	M.P	I.Rcm^{-1}
3	CH_3	>225^{0}C	1056(C-N str), 1188(C-O str), 1589(Aromatic C=C str), 1674(C=O str), 2869(sp3 C-H str), 3050(aromatic C-H str),
5	C_2H_5	>225^{0}C	1080(C-N str), 1203(C-O str), 1589(Aromatic C=C str), 1666(C=O str), 2869(sp3 C-H str), 3055(aromatic C-H str),

2D- Melting point and I.R data of stage seven compounds

n	X	M.P	I.R cm^{-1}
2	Br	>225^{0}C	1049(C-N str), 1188(C-O str), 1589(Aromatic C=C str), 1681(C=O str), 2823(sp3 C-H str),3062(aromatic C-H str)

2E- Melting point and I.R data of stage eight compound

n	X	M.P	I.R cm^{-1}
3	Cl	175^{0}C-177^{0}C	1049(C-N str),1200(C-N str), 1600(aromatic C=C str), 1666(C=O str), 2877(sp3C-H str), 3055(aromatic C-H str)

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