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Synthesis and Some Reactions of 2-[α-Benzoylaminostyryl]-6, 8-Dibromo-3,1-Benzoxazin-4(H)-one, Quinazolin-4(3H)-one and Chloroquinazoline Derivatives with Some Nucleophilic Reagents

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2-[α-Benzoylamino-*p*-chlorostyryl]-6, 8-dibromo-3, 1-benzoxazine-4(H)-one (II) has been prepared from N-[α-benzoylamino-*p*-chlorocinnamoyl]-3, 5-dibromoanthranilic acid (I). The benzoxazinone (II) was found to undergo ring opening reaction with different nitrogen nucleophiles to give (III). Similarly with secondary amines and hydrazines to give (IV) and (V). Reaction of (Va) with different aldehydes and acetylation gave bases (VI) and (VII) respectively. (II) reacted with hydrazoic acid to give the Schiff's benzimidazole derivative (VIII) beside the tetrazole derivative (IX). (II) also reacted with aromatic hydrocarbons under Friedel-Craft's conditions to give (X). (II) reacted with ethyl acetoacetate, as a carbon nucleophile, and maleic anhydride to give (XI) and (XII) respectively. Fusion of (II) with ammonium acetate gave the quinazolinone (XIII), while (XIII) condensed with formaldehyde in the presence of succinimide and/or benzamide to give Mannich bases (XIV); furthermore, alkylation of (XIII) with sodium ethoxide and methyl iodide gave (XIX) and (XX) respectively. On the other hand, chlorination of (XIII) with a mixture of $POCl_3/PCl_5$ gave (XVII); fusion of (XVII) with anthranilic acid gave (XVIII); also (XVII) reacted with both thiourea and hydrazoic acid to give (XIX) and (XX) respectively. Also (II) has been transformed to the thione (XXI) by the action of P_2S_5.

INTRODUCTION

The present investigation is a continuation for our comparative studies[1] on some new synthesised benzoxazinone, quinazolinone and chloroquinoline derivatives bearing a bulk moiety in position-2 with specific polar effect, with the aim of finding out a role for such groups on the stability of the benzoxazinone nucleus towards different nucleophiles.

RESULTS AND DISCUSSION

4-*p*-Chlorobenzylidene-2-phenyl oxazole-5-one reacted with dibromoanthranilic acid (equimolar amount) in acetic acid, to give N[α-benzoylamino-*p*-chlorocinnamoyl]-3-,5-dibromoanthranilic acid (I) in a good yield. The structure of (I) was confirmed by elemental analysis, NMR and the IR spectra (Table I). The above acid (I) was transformed easily to the corresponding 2-[α-benzoylamino-*p*-chlorostyryl]-3,1-4(H)-benzoxazin-4-one (II) upon its treatment with acetic anhydride[2]. The structure of (II) was confirmed on the basis of elemental analysis, NMR and the IR spectra (Table 1).

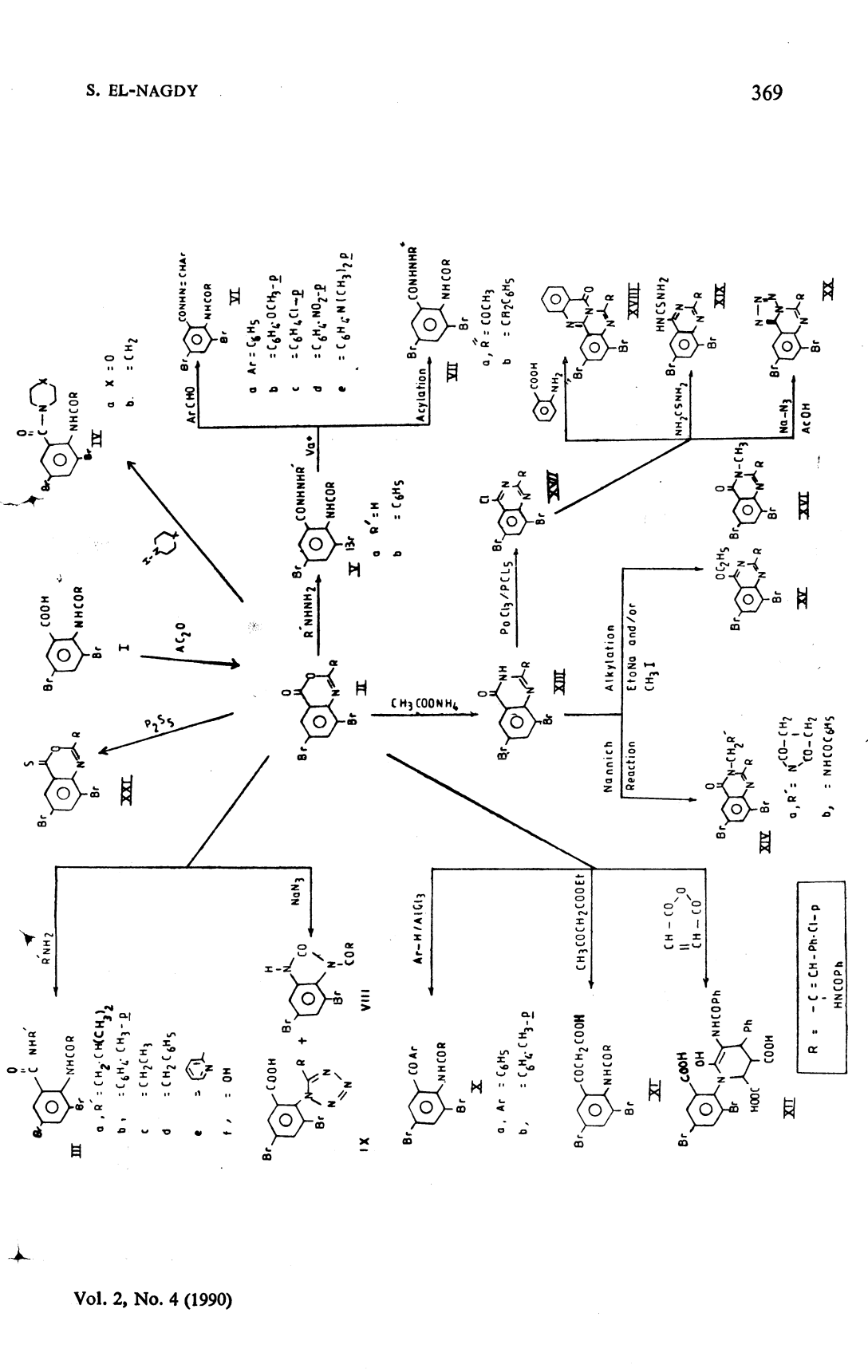

TABLE 1

PHYSICAL AND SPECTRAL DATA OF COMPOUNDS (I-XXI)

Compd	M. pt. (°C) (solvent)	Mol. formula (mol. wt.)	Analysis % Required/Found			IR (cm^{-1})			NMR
			C	H	N	$\nu_{NH, OH}$	$\nu_{C=O}$	$\nu_{C=N}$	
I	229–30 Acetic acid	$C_{23}H_{15}N_2O_4Br_2Cl$ (579.5)	47.63 47.28	2.59 2.45	4.83 4.60	3380–3310 broad	1690–1650, 1610		(DMSO-d_6) 7.5–8.6 (*m*, 11, *H*, Ar*H*), 6.8 (*s*, 1*H*, CH=), 10.2 (br., *s*, 2*H*, N*H*), 12 (br., *s*, 1, OH)
II	167–68 Acetic acid	$C_{23}H_{13}N_2O_3Br_2Cl$ (561.5)	47.55 47.25	2.32 2.20	4.99 4.75	3380–3200	1750–1680	1600	(DMSO-d_6) 7.6–8.8 (*m*, 11*H*, Ar*H*), 6.6 (br, *s*, 1H, CH=), 10.4 (br, *s*, 1H, NH)
IIIa	208–9 Ethanol	$C_{27}H_{24}N_3O_3Br_2Cl$ (634.5)	51.10 51.30	3.78 3.50	6.62 6.45	3370–3220	1670–1620		
IIIb	221–22 Ethanol	$C_{30}H_{22}N_3O_3Br_2Cl$ (668.5)	53.88 53.52	3.29 3.11	6.28 6.15	3360–3215	1665–1630		
IIIc	210–11 Ethanol	$C_{25}H_{20}N_3O_3Br_2Cl$ (606.5)	49.46 49.20	3.30 3.25	6.92 6.75	3390–3240	1670–1615		
IIId	171–72 Ethanol	$C_{30}H_{22}N_3O_3Br_2Cl$ (668.5)	53.85 53.56	3.29 3.12	6.28 6.15	3385–3235	1675–1625		(DMSO-d_6) 10.5 (br., 2H, N*H*CO), 9.2 (br., 1H, N*H*CH_2), 7.2–8.2 (m, 16H, Ar*H*), 6.6 (br, 1H, C*H*=), 4.5–4.8 (d, 2H, CH_2)
IIIe	153–54 Ethanol	$C_{28}H_{19}N_4O_3Br_2Cl$ (655.5)	51.26 51.42	2.90 2.70	8.54 8.36	3370–3210	1650–1610		
IIIf	227–28 Benzene	$C_{23}H_{16}N_3O_4Br_2Cl$ (594.5)	46.43 46.23	2.69 2.48	7.10 6.94	3420–3215 broad	1655–1605		

IVa	204–5 Ethanol	$C_{27}H_{22}N_3O_4Br_2Cl$ (648.5)	49.96 49.64	3.39 3.15	6.48 6.25	3280– 3140	1660– 1610		
IVb	193–4 Ethanol	$C_{28}H_{24}N_3O_3Br_2Cl$ (646.5)	51.97 51.75	3.71 3.65	6.50 6.24	3285– 3150	1655– 1600		
Va	227–28 Butanol	$C_{23}H_{17}N_4O_3Br_2Cl$ (593.5)	46.50 46.31	2.86 2.65	9.44 9.28	3380– 3180	1660– 1620		
Vb	219–20 Ethanol	$C_{29}H_{21}N_4O_3Br_2Cl$ (669.5)	51.98 51.72	3.14 3.00	8.36 8.18	3290– 3120	1655– 1615		(DMSO-d_6) 12.1 (br., 2H, N*H*CO), 10.4 (br., 1H, CON*H*NH), 9.8 (s, 1H, N*H*Ph), 6.8–7.9 (m, 17H, Ar*H* and olefinic)
VIa	206–7 Ethanol	$C_{30}H_{21}N_4O_3Br_2Cl$ (681.5)	52.82 52.60	3.10 2.98	9.54 9.37	3280– 3130	1660– 1630	1610	
VIb	243–44 Ethanol	$C_{31}H_{23}N_4O_4Br_2Cl$ (711.5)	52.28 52.39	3.23 3.15	7.87 7.65	3290– 3125	1655– 1620	1605	
VIc	211–12 Ethanol	$C_{30}H_{20}N_4O_3Br_2Cl_2$ (680.5)	52.90 52.78	2.94 2.70	8.23 8.15	3285– 3130	1600– 1625	1605	
VId	260–61 Ethanol	$C_{30}H_{20}N_5O_5Br_2Cl$ (726.5)	49.55 49.38	2.75 2.58	9.64 9.49	3280– 3130	1655– 1610	1590	
VIe	209–10 Ethanol	$C_{32}H_{26}N_5O_3Br_2Cl$ (724.5)	53.00 53.21	3.59 3.38	9.66 9.47	3340– 3220	1650– 1620	1595	
VIIa	198–99 Acetic acid	$C_{25}H_{19}N_4O_4Br_2Cl$ (635.5)	47.21 47.35	2.99 2.75	8.81 8.67	3380– 3240	1665– 1620		
VIIb	215–16 Ethanol	$C_{30}H_{23}N_4O_3Br_2Cl$ (683.5)	52.67 52.45	3.37 3.18	8.19 8.00	3370– 3250	1670– 1620		
VIII	189–90 Ethanol	$C_{23}H_{14}N_3O_3Br_2Cl$ (576.5)	47.88 47.61	2.43 2.31	7.29 7.12	3280– 3140	1755– 1675		(DMSO-d_6) 11.4 (br., 2*H*, N*H*CO amido and imidazole), 7.5–8.2 (*m*, 11*H*, Ar*H*), 6.7 (*s*, 1*H*, CH=)

TABLE 1 (contd.)

Compd	M. pt. (°C) (solvent)	Mol. formula (mol. wt.)	Analysis % Required/Found			IR (cm^{-1})			NMR
			C	H	N	$\nu_{HN, OH}$	$\nu_{C=O}$	$\nu_{C=N}$	
IX	181–82 Ethanol	$C_{23}H_{14}N_5O_3Br_2Cl$ (604.5)	45.66 45.41	2.32 2.17	11.58 11.35	3450–3150 broad	1690–1655	1595	
Xa	117–18 Pet. ether (60–80 C)	$C_{29}H_{19}N_2O_3Br_2Cl$ (639.5)	54.42 54.13	2.97 2.74	4.38 4.21	3360–3280	1700–1655		
Xb	211–12 Xylene	$C_{30}H_{21}N_2O_3Br_2Cl$ (653.5)	55.10 55.30	3.21 3.12	4.28 4.15	3370–3220	1695–1640		(DMSO-d_6) 7.6–8.4 (*m*, 15*H*, Ar*H*), 6.9 (br., *s*, 1*H*, CH=), 1.7 (*s*, 3*H*, C*H*$_3$), 10.1 (br., 2*H*, N*H*CO)
XI	201–2 Benzene	$C_{25}H_{17}N_2O_5Br_2Cl$ (621.5)	48.27 47.92	2.74 2.59	4.51 4.38	3340–3210	1695 1675		(DMSO-d_6) 12.5 (s, 1H, OH), 11.6 (br, 2H, N*H*CO), 7.6–8.4 (m, 11H, Ar*H*), 6.9 (s, 1H, CH=), 3.5 (s, 2H, COC*H*$_2$)
XII	184–85 Xylene	$C_{27}H_{19}N_2O_8Br_2Cl$ (695.5)	46.59 46.38	2.73 2.59	4.03 3.88	3420–3270 broad	1690 1665		
XIII	278–79 Acetic acid	$C_{23}H_{14}N_3O_2Br_2Cl$ (560.5)	49.24 49.00	2.50 2.30	7.49 7.19	3370–3250	1705–1660	1590	(DMSO-d_6) 12.5 (br, 2*H*, N*H*CO amido and quinazolone), 7.4–8.5 (*m*, 12*H*, Ar*H* and olefinic)
XIVa	260–61 Acetic acid	$C_{28}H_{19}N_4O_4Br_2Cl$ (671.5)	50.00 49.71	2.83 2.65	8.34 8.21	3340–3190	1700–1665	1595	
XIVb	249–50 Ethanol	$C_{31}H_{21}N_4O_3Br_2Cl$ (693.5)	53.64 53.45	3.00 2.87	8.10 7.92	3320–3185	1710 1660	1600	

XV	305–6 Ethanol	$C_{25}H_{18}N_3O_2Br_2Cl$ (588.5)	50.98 50.71	3.10 3.00	9.52 9.39	3290– 3150	1650	1610	
XVI	135–36 Pet. ether (60–80°C)	$C_{24}H_{16}N_3O_2Br_2Cl$ (574.5)	50.13 49.88	2.79 2.57	7.31 7.20	3285– 3140	1670– 1640	1605	
XVII	324–25 Ethanol	$C_{23}H_{13}N_3OBr_2Cl_2$ (579)	47.67 47.45	2.25 2.11	7.75 7.15	3310– 3180	1650	1610	(DMSO-d_6) 12.1 (br, *s*, 1*H*, N*H*CO), 7.6–8.4 (*m*, 11*H*, Ar*H*), 6.8 (*s*, 1*H*, CH=)
XVIII	181–82 Pet. ether (60–80°C)	$C_{30}H_{17}N_4O_2Br_2Cl$ (661.5)	54.42 54.18	2.57 2.40	8.47 8.25	3320– 3170	1675– 1640	1605	
XIX	296–98 Benzene	$C_{24}H_{16}N_5OSBr_2Cl$ (618.5)	46.56 46.35	2.59 2.40	11.32 11.18	3380– 3225	1655	1600	
XX	183–84 Pet. ether (60–80°C)	$C_{23}H_{13}N_6OBr_2Cl$ (585.5)	47.14 47.30	2.22 2.11	14.35 14.16	3290– 3140	1650	1595	
XXI	129–30 Xylene	$C_{23}H_{13}N_2O_2SBr_2Cl$ (577.5)	47.79 47.54	2.25 2.18	4.85 4.69	3270– 3120	1660	1590	

It was previously stated[2,3] that primary amines react with 2-substituted-3,1-benzoxazin-4(H)-ones in the presence of anhydrous zinc chloride to give 2,3-disubstituted-4-quinazolones. In the present study aminolysis of (II) with primary amines such as isobutylamine, *p*-toluidine, ethylamine, benzylamine, 2-aminopyridine and hydroxylamine hydrochloride in boiling ethanol or butanol gave the corresponding N-[α-benzoylamino-*p*-chlorocinnamoyl]-3,5-dibromoanthranilic acid anilides, (IIIa-f). Also, compound (II) reacted with the secondary amines, piperidine and morpholine to give N-[α-benzoylamino-*p*-chlorocinnamoyl]-3,5-dibromopiperidino- or morpholinoanthranilic acid (IVa & b).

Hydrazinolysis of II with hydrazine hydrate and phenylhydrazine in refluxing ethanol gave N-[α-benzoylamino-*p*-chlorocinnamoyl]-3,5-dibromo-anthranilic acid hydrazides, (Va & b). While condensation of (Va) with benzaldehyde, *p*-anisaldehyde, *p*-chlorobenzaldehyde, *p*-nitrobenzaldehyde and N,N-dimethyl *p*-aminobenzaldehyde in refluxing ethanol in presence of piperidine, afforded the Schiff's bases (VIa–e) respectively. Also, (Va) was subjected to acetylation with acetic anhydride and alkylation with benzyl chloride and gave (VII a & b) respectively.

Reaction of (II) with hydrazoic acid[4] (sodium azide in acetic acid) gave a mixture of benzimidazolone derivative (VIII) and tetrazole derivative (IX) which might be formed through ring opening either at position 2,3- to give (VIII) or at position 3,4- to give (IX).

Compound (II) reacts with aromatic hydrocarbons, namely, benzene and toluene in presence of anhydrous $AlCl_3$ under Friedel-Craft's conditions affording 6-aroyl-N-[α-benzoylamino-*p*-chlorocinnamoyl]-2,4-dibromoaniline (Xa & b) respectively. The reaction of (II) with active methylene as ethyl acetoacetate in a refluxing pyridine, and maleic anhydride in dry xylene through a cycloaddition reaction to give 2-[α-benzoylamino-*p*-chlorocinnamido]-3,5-dibromobenzoyl acetic acid (XI) and Diels-Alder adduct (XII) respectively.

The benzoxazinone (II) underwent ring closure on fusion with ammonium acetate at 170°C affording 2-[α-benzoylamino-*p*-chlorostyryl]-4(3H)-quinazolin-4-one (XIII) which exists in the lactam ⇌ lactim tautomeric equilibrium. (XIII) was condensed with formaldehyde, succinimide and/or benzamide on refluxing with a mixture of acetic acid and its anhydride to give 3-N-(substituted) quinazol-4-ones (Mannich bases) (XIVa & b) respectively. While the alkylation of (XIII) with sodium ethoxide and/or methyl iodide in presence of 0.2N sodium hydroxide, both in refluxing ethanol to give 2-substituted-4-ethoxyquinoline (XV) and 2-substituted 3 N-methylquinoline (XVI) respectively.

The reaction of (XIII) with a mixture of $POCl_3/PCl_5$ on a water bath gave 4-chloro-2-[α-benzoylamino-*p*-chlorostyryl]-4(3H)-quinazoline (XVII). On fusion of chloroquinazoline (XVII) with anthranilic acid afforded quinazoline derivative (XVIII), also chloroquinazoline (XVII) reacts with

thiourea in presence of sodium ethoxide afforded the corresponding 4-aminosubstituted quinazoline derivative (XIX), while (XVII) reacts with hydrazoic acid (sodium azide in acetic acid) to give tetrazoloquinoline derivative (XX).

The reaction of (II) with P_2S_5 in dry xylene has also been studied and gave the corresponding thione (XXI).

From the foregoing results we can conclude that the bulk of the substitutent at position-2 decreased the yield of the products in the above reactions and this is due to the steric hindrance as reported by other investigators.[5]

EXPERIMENTAL

Melting points are uncorrected. IR spectra were recorded in KBr on a Unicam SP 200 G, Pye Unicam 641749 or Beckman 922623 spectrophotometer and PMR spectra on a Varian 60 MHz using TMS as an internal standard. Characterisation data of all the compounds prepared are given in Table 1.

Action of 4-(*p*-chlorobenzylidene)-2-phenyl-oxazol-5-one on dibromoanthranilic acid; Formation of N-[α-benzoylamino-*p*-chlorocinnamoyl]-3,5-dibromoanthranilic acid (I)

A mixture of dibromoanthranilic acid (0.01 mol) and 4-(*p*-chlorobenzylidene)-2-phenyl oxazol-5-one (0-01 mol) in acetic acid (30 ml) was heated under reflux for 2 hrs. After cooling a solid separated out which was filtered and recrystallised from a proper solvent to give (I) (Table 1).

Action of acetic anhydride on (I); Formation of 2-[α-benzylamino-*p*-chlorostyryl]-6,8-dibromo-3,1-(4H)-benzoxazin-4-one (II)

A mixture of (I) (0.01 mol) and acetic anhydride (30 ml) was refluxed for 1 hr. After cooling a solid separated out which was filtered and recrystallised from a suitable solvent to give (II) (Table 1).

Action of amides and hydrazines on (II); Formation of (IIIa-f & Va and b)

A solution of (II) (0.01 mol) in ethyl alcohol or *n*-butanol (30 ml) was refluxed for 4 hrs with (0.01 mol) of amines, namely, isobutylamine, *p*-toluidine, ethylamine, benzylamine, 2-aminopyridine or hydroxylamine hydrochloride; or hydrazines, namely, hydrazine hydrate and phenylhydrazine. The separated solid was filtered, dried, and recrystallised from an appropriate solvent to give (IIIa–f & Va) and (Vb) (Table 1).

Action of piperidine or morpholine on (II); Formation of (IVa and b)

A solution of (II) (0.01 mol) and piperidine or morpholine (0.01 mol) in ethanol (30 ml) was heated under reflux for 3 hrs and concentrated. The

separated solid was filtered, dried and recrystallised from an appropriate solvent to give (IVa or IVb) (Table 1).

Condensation of (Va) with aromatic aldehydes; Formation of Schiff's bases (VIa–e)

A solution of (Va) (0.01 mol) and aromatic aldehydes, namely, benzaldehyde, *p*-anisaldehyde, *p*-chlorobenzaldehyde, *p*-nitrobenzaldehyde and N,N'-dimethyl *p*-aminobenzaldehyde in ethanol (40 ml) with few drops of piperidine was heated under reflux for 3 hrs. The separated solid was filtered, dried and recrystallised from a proper solvent to give (VIa–e) (Table 1).

Acetylation and benzylation of (Va); Formation of (VIIa and b)

Compound (Va) (0.01 mol) was heated with excess of acetic anhydride or benzyl chloride (25 ml) under reflux for 2 hrs. After cooling a solid separated out which was filtered, washed with ethanol, dried and recrystallised from a proper solvent to give (VIIa or VIIb) (Table 1).

Action of sodium azide on (II); Formation of (VIII and IX)

A mixture of (II) (0.01 mol) and sodium azide (0.015 mol) in boiling acetic acid (50 ml) was refluxed for 4 hrs and concentrated, a separated solid was filtered, dried and recrystallised from a proper solvent to give (VIII). The filtrate was diluted with water and acidified wito dilute HCl to give (IX) which was recrystallised from an appropriate solvent (Table 1).

Friedel-Crafts reaction on (II); Formation of (Xa and Xb)

To a solution of (II) (0.01 mol) in dry aromatic hydrocarbon (100 ml) benzene (or toluene), was added anhydrous $AlCl_3$ (0.04 mol) portionwise with stirring for 30 min. The stirring was continued and the reaction mixture was heated on a water bath for 3 hrs. The reaction mixture was then poured on ice-cold HCl. The organic layer was extracted with ether, washed with water and the ethereal layer distilled under reduced pressure. The solid obtained was recrystallised from a proper solvent to give (Xa) and (Xb) respectively (Table 1).

Action of ethyl acetoacetate on (II); Formation of (XI)

A solution of (II) (0.01 mol) and an active methylene compound, ethyl acetoacetate (0.03 mol) in pyridine (50 ml) was heated under reflux for 12 hrs. The reaction mixture was pured on ice-cold HCl, the separated solid was filtered, washed with dilute HCl and dried. The solid obtained was recrystallised from an appropriate solvent to give (XI) (Table 1).

Diels-Alder reaction on (II); Formation of (XII)

A mixture of (II) (0.01 mol), maleic anhydride (0.01 mol) and dry

xylene (50 ml) was heated under reflux for 10 hrs and filtered while hot; the filtrate was concentrated and cooled. The separated solid was recrystallised from a suitable solvent to give the Diels-Alder adduct (XII) (Table 1).

Action of ammonium acetate on (II); Formation of (XIII)

A mixture of (II) (0.01 mol) and ammonium acetate (0.015 mol) was heated at 180° C for 3 hrs, and poured into water, the resultant solid was filtered, washed with water and recrystallised from a proper solvent to give (XIII) (Table 1).

Mannich reaction on (XIII); Formation of (XIVa and b)

A mixture of (XIII) (0.01 mol), formaldehyde (5 ml) and an imide (succinimide) or amide (benzamide) in acetic acid/acetic anhydride (1 : 3, v/v) (50 ml) was refluxed for 5 hrs and cooled. The separated solid was recrystallised from a suitable solvent to give (XIVa and XIVb) (Table 1).

Alkylation of (XIII); Formation of 2-substituted-4-ethoxyquinazoline (XV) and 2-substituted 3 N-methylquinazolin-4-one (XVI)

A mixture of (XIII) (0.01 mol) and an excess of sodium ethoxide in ethanol or methyl iodide (0.015 mol) and sodium hydroxide (5 ml; 2N) in ethanol, was refluxed for 3 hrs and poured into dilute HCl. The product was filtered, dried and recrystallised from an appropriate solvent to give (XV) and (XVI) respectively (Table 1).

Action of $PCl_5/POCl_3$ on (XIII); Formation of 2-substituted 4-chloroquinazoline (XVII)

Compound (XIII) (0.01 mol) was heated with a mixture of $PCl_5/POCl_3$ (2 ml; 0.01 mol) in an oil bath at 180°C for 2 hrs. After cooling, the reaction mixture was poured into ice-water, the resultant solid was filtered, washed with water, dried and recrystallised from a proper solvent to give (XVII) (Table 1).

Condensation of (XVII) with anthranilic acid; Formation of quinazoline derivative (XVIII)

A mixture of (XVII) (0.01 mol) and anthranilic acid (0.015 mol) was heated at 170°C for 2 hrs and poured into water. The solid obtained was filtered, washed with hot water, dried and recrystallised from a proper solvent to give (XVIII) (Table 1).

Action of thiourea on (XVII); Formation of 4-aminosubstituted quinazoline derivative (XIX)

A mixture of (XVII) (0.01 mol), thiourea and an excess of sodium ethoxide in ethanol was refluxed for 4 hrs. The reaction mixture was

concentrated, poured into ice-cold HCl, the resultant solid was filtered, washed with water, dried and recrystallised from a suitable solvent to give XIX (Table 1).

Action of sodium azide on (XVII); Formation of substituted tetrazolo[1,5-c] quinazoline derivative (XX)

A mixture of (XVII) (0.01 mol) and NaN_3 (0.015 mol) in acetic acid (30 ml) was refluxed for 3 hrs. After concentration and cooling the product was filtered, washed with water and recrystallised from a proper solvent to give (XX) (Table 1).

Action of P_2S_5 on (II); Formation of thione (XXI)

A solution of (II) (0.01 mol) and P_2S_5 (0.02 mol) in dry xylene (100 ml) was refluxed for 2 hrs. The reaction mixture was filtered off upon hot and then concentrated. The product separated on cooling was crystallised from a proper solvent to give (XXI) (Table 1).

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