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A convenient synthesis of chalcones, aminopyrimidines and phenylpyrazolines

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ABSTRACT

*Chalcones **(6a-f)** have been prepared by the condensation of ketone **(5)** and different aromatic aldehydes. These chalcones **(6a-f)** on treatment with guanidine hydrochloride and phenyl hydrazine hydrochloride in presence of alkali give aminopyrimidines **(7a-f)** and phenylpyrazolines **(8a-f)** respectively. All the newly synthesized compounds have been characterized on the basis of IR and ^{1}H NMR spectral data as well as physical data.*

Keywords: chalcones, aminopyrimidines, phenyl pyrazolines, spectral data.

INTRODUCTION

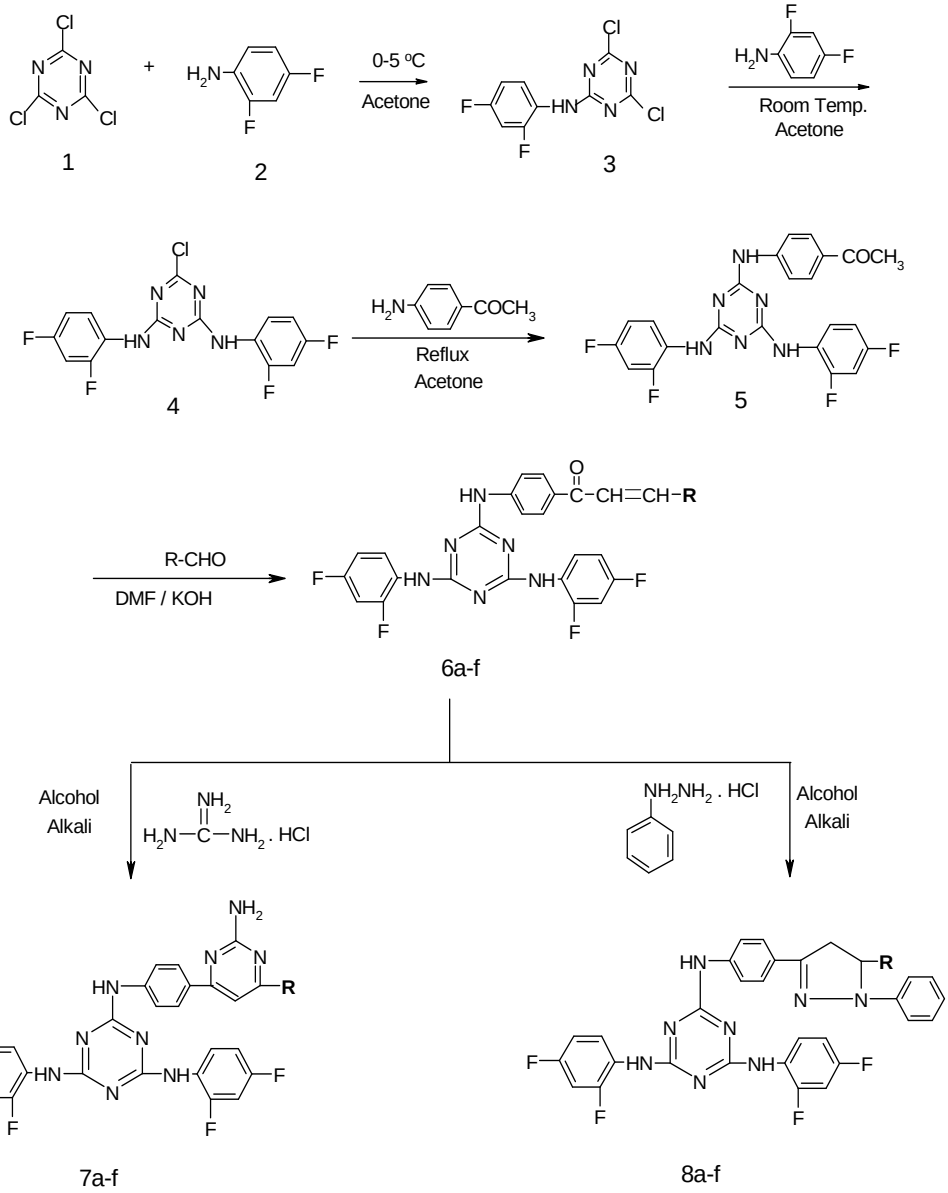
Chalcones and its derivatives have attracted particular interest during the last few decades due to use of such ring system as the core structure in many drug substances covering wide range of pharmacological application [1]. Chalcones are associated with diverse biological activities such as anticancer [2], anti-inflammatory [3], antiviral [4] and antibacterial [5] activities etc. Pyrimidine is a basic nucleus in DNA and RNA, it has been found to be associated with diverse biological activities such as antioxidant [6], antidepressant [7] and antiviral [8] activities etc. This biological significance of the pyrimidine derivatives had led us to the synthesis of substituted pyrimidines. Pyrazoline is five membered ring structure composed of three carbon atoms and two nitrogen atoms in adjacent positions. It has been reported that the heterocyclic compounds containing pyrazoline ring present a broad spectrum of biological activity such as tranquilizer [9], antifungal [10] and anti-inflammatory [11] activities etc. In view of the above and in continuation of our work [12-15] we herein report a new series of chalcones **(6a-f)**, aminopyrimidines **(7a-f)** and phenylpyrazolines **(8a-f)**.

MATERIALS AND METHODS

Experimental

All melting points were determined in open capillary and are uncorrected. The IR spectra were recorded on a FTIR - 8400 spectrometer. ^{1}H NMR spectra on a Bruker Avance DPX 400 MHz

spectrometer with $CDCl_3$ as a solvent and tetramethylsilane (TMS) as internal standard. The chemical shifts are expressed in parts per million (ppm) downfield from the internal standard and signals are quoted as *s* (singlet) and *m* (multiplate). Analytical separation was conducted with silica Gel 60 F-254 (Merck) plates of 0.25 mm thickness eluted with toluene : acetone (10 : 2 v/v) and were visualized with UV (254 nm) or iodine to check the purity of the synthesized compounds.



SCHEME

Preparation of 2-(2', 4'-difluorophenylamino)-4, 6-dichloro-*s*-triazine (3)

2, 4-Difluoroaniline (0.01 mol, 1.29g in 10ml acetone) was added slowly to cyanuric chloride (0.01 mol, 1.845g in 30ml acetone) with constant stirring for 4 hours at 0 to 5 ^{0}C. Periodically, sodium carbonate solution (0.005 mol, 0.53g in 10 ml water) was added dropwise to neutralized

HCl evolved during the reaction. Finally, the content was poured into crushed ice. The solid seprated out was filtered, washed with water, dried and recrystallized from ethyl alcohol to give **(3)**.

Preparation of 2, 4-bis-(2', 4'-difluorophenylamino)-6-chloro-*s*-triazine (4)

2-4-Difluoroaniline (0.01 mol, 1.29g in 10ml acetone) was added slowly to compound **(3)** (0.01 mol, 2.77g in 35ml acetone) with constant stirring for 6 hours at room temperature. Periodically, sodium carbonate solution (0.005 mol, 0.53g in 10ml water) was added dropwise to neutralized HCl evolved during the reaction. Finally, the content was poured into crushed ice. The solid separated out was filtered, washed with water, dried and recrystallized from ethyl alcohol to give **(4)**.

Table-1 Characterization data of compounds (6a-f), (7a-f) and (8a-f)

Compound	R	M.P.	% Yield
6a	4-Methoxyphenyl	105°-108 °C	75
6b	2,5-Dimethoxyphenyl	96°-99 °C	69
6c	4-N,N-dimethylaminophenyl	109°-112 °C	76
6d	4-Nitrophenyl	106°-110 °C	69
6e	4-Fluorophenyl	112°-115 °C	66
6f	2,3-Dichlorophenyl	110°-114 °C	72
7a	4-Methoxyphenyl	100 °-104 °C	61
7b	2,5-Dimethoxyphenyl	140 °-143 °C	72
7c	4-N,N-dimethylaminophenyl	84 °-88 °C	59
7d	4-Nitrophenyl	107 °-111 °C	68
7e	4-Fluorophenyl	125 °-128 °C	55
7f	2,3-Dichlorophenyl	102 °-107 °C	51
8a	4-Methoxyphenyl	95 °-100 °C	61
8b	2,5-Dimethoxyphenyl	82 °-86 °C	59
8c	4-N,N-dimethylaminophenyl	110 °-115 °C	67
8d	4-Nitrophenyl	120 °-124 °C	58
8e	4-Fluorophenyl	92 °-97 °C	49
8f	2,3-Dichlorophenyl	90 °-94 °C	64

Preparation of 2,4-bis-(2',4'-difluorophenylamino)-6-(4'-acetylphenylamino)-*s*-triazine (5)

4-Aminoacetophenone (0.01 mol) and compound **(4)** (0.01 mol) were dissolved in acetone (40ml). The reaction mixture was refluxed for 8 hours, cooled and poured into crushed ice. Periodically, sodium carbonate solution (0.005 mol, 0.53g in 10ml water) was added to neutralized HCl evolved during the reaction. The solid separated out was filtered, washed with water, dried and recrystallized from ethyl alcohol to give **(5)**.

Preparation of 2,4-bis-(2',4'-difluorophenylamino)-6-[4′-{5″-(4‴-methoxyphenyl)–2''-propenon-1''-yl} phenyl amino]-*s*-triazine (6a)

Compound **(5)** (0.01 mol) was dissolved in DMF (30ml) and 4-methoxybenzaldehyde (0.01 mol) was added to it. Then solution of KOH (5ml of 40%) was added to the reaction mixture with constant stirring at room temperature. The progress of the reaction was monitored on TLC plate. After completion the reaction mixture was poured into crushed ice and neutralized with HCl. The product separated out was filtered, washed with water, dried and recrystallized from ethyl alcohol.

Similarly the remaining compounds **(6b-f)** were prepared by this method.

Compound (6a) IR (KBr,cm^{-1}): 3305 (N-H str.), 3070 (=CH str.), 1649 (C=O str.), 1510 (C=C str.), 1025 (C-O-C str.), 808 (C-N str., *s*-triazine moiety), 1080 cm^{-1} (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.81 (3H, *s*, p-OCH_3), 6.42 (1H, d, -CO-CH=), 8.03 (1H, d, Ar-CH=), 6.98 – 7.91 (17H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{31}H_{22}F_4N_6O_2$: C, 63.48; H, 3.78; N, 14.33. Found: C: 63.50; H: 3.80; N: 14.35%.

Compound (6b) IR (KBr,cm^{-1}): 3308 (N-H str.), 3075 (=CH str.), 1650 (C=O str.), 1515 (C=C str.), 1020 (C-O-C str.), 810 (C-N str., *s*-triazine moiety), 1085 cm^{-1} (C-F str.); ^{1}H NMR ($CDCl_3$, δ, ppm): 3.82 (3H, *s*, o-OCH_3), 3.86 (3H, *s*, m-OCH_3), 6.41 (1H, d, -CO-CH=), 8.05 (1H, d, Ar-CH=), 6.98 – 7.92 (16H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{32}H_{24}F_4N_6O_3$: C, 62.34; H, 3.92; N, 13.63. Found: C: 62.32; H: 3.90; N: 13.60%.

Compound (6c) IR (KBr,cm^{-1}): 3303 (N-H str.), 3075 (=CH str.), 1652 (C=O str.) , 1510 (C=C str.), 1030 (C-O-C str.), 815 (C-N str., *s*-triazine moiety), 1083 cm^{-1} (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.21 (3H, *s*, N-CH_3), 3.27 (3H, *s*, N-CH_3), 6.39 (1H, d, -CO-CH=), 8.03 (1H, d, Ar-CH=), 6.98 – 7.91 (17H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{32}H_{25}F_4N_7O$: C, 64.10; H, 4.20; N, 16.35. Found: C: 64.05; H: 4.15; N: 16.33%.

Compound (6d) IR (KBr,cm^{-1}): 3304 (N-H str.), 3070 (=CH str.), 1651 (C=O str.) ,1515 (C=C str.), 1035 (C-O-C str.), 818 (C-N str., *s*-triazine moiety), 1084 cm^{-1} (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 6.41 (1H, d, -CO-CH=), 8.03 (1H, d, Ar-CH=), 6.95 – 7.91 (17H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{30}H_{19}F_4N_7O_3$: C, 59.90; H, 3.18; N, 16.30. Found: C: 59.87; H: 3.17; N: 16.28%.

Compound (6e) IR (KBr,cm^{-1}): 3310 (N-H str.), 3060 (=CH str.), 1653 (C=O str.) , 1505 (C=C str.), 1030 (C-O-C str.), 820 (C-N str., *s*-triazine moiety), 1087cm^{-1} (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 6.45 (1H, d, -CO-CH=), 8.03 (1H, d, Ar-CH=), 6.95 – 7.91 (17H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{30}H_{19}F_5N_6O$: C, 62.72; H, 3.33; N, 14.63. Found: C: 62.71; H: 3.31; N: 14.61%.

Compound (6f) IR (KBr,cm^{-1}): 3308 (N-H str.), 3070 (=CH str.), 1655 (C=O str.) , 1517 (C=C str.), 1035 (C-O-C str.), 810 (C-N str., *s*-triazine moiety), 801 cm^{-1} (C-Cl str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 6.43 (1H, d, -CO-CH=), 8.03 (1H, d, Ar-CH=), 6.95 – 7.91 (16H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{30}H_{18}F_4N_6OCl_2$: C, 57.62; H, 2.19; N, 13.44. Found: C: 57.60; H: 2.17; N: 13.40%.

Preparation of 2,4-bis-(2',4'-difluorophenylamino)-6-[4′-{2″-amino-6″-(4‴- methoxy phenyl)–pyrimidin - 4″-yl}phenylamino]-*s*-triazine (7a)

Compound **6a** (0.01 mol) was dissolved in ethyl alcohol (25ml) and guanidine hydrochloride (0.01 mol) was added to it. Then solution of KOH (5ml of 40%) was added to the reaction mixture and refluxed for 8 hours. The progress of the reaction was monitored on TLC plate. After completion the reaction mixture was then cooled and poured into crushed ice and neutralized with dilute HCl. The product separated out was filtered, washed with water, dried and recrystallized from ethyl alcohol to give **7a**.

Similarly the remaining compounds **(7b-f)** were prepared by this method.

Compound (7a) IR (KBr,cm^{-1}): 3405 (N-H str.), 3065 (=CH str.), 809 (C-N str., *s*-triazine moiety), 825 (C-H bending), 1645(C=N str, pyrimidinemoiety), 1266 (C-O-C str.), 1095 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.81 (3H, *s*, p-OCH_3), 5.1 (2H, *s*, -NH_2), 6.85 (1H, *s*, -CH=),

7.0 – 8.0 (17H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{32}H_{23}F_4N_9O$: C, 61.44; H, 3.71; N, 20.15. Found: C: 61.42; H: 3.70; N: 20.10%.

Compound (7b) IR (KBr,cm^{-1}): 3410 (N-H str.), 3070 (=CH str.), 810 (C-N str, *s*-triazine moiety), 830(C-H bending), 1642(C=N str, pyrimidinemoiety) 1267 (C-O-C str) 1098 (C-F str) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.81 (3H, *s*, o-OCH_3), 3.85 (3H, *s*, m-OCH_3), 5.2 (2H, *s*, -NH_2), 6.87 (1H, *s*, -CH=), 7.0 – 8.0 (16H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{33}H_{25}F_4N_9O_2$: C, 60.46; H, 3.84; N, 19.23. Found: C: 60.45; H: 3.82; N: 19.21%.

Compound (7c) IR (KBr,cm^{-1}): 3412 (N-H str.), 3072 (=CH str.), 812 (C-N str., *s*-triazine moiety), 832 (C-H bending), 1641 (C=N str, pyrimidinemoety)1270 (C-O-C str.), 1098 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.81 (3H, *s*, N-CH_3), 3.83 (3H, *s*, N-CH_3), 5.3 (2H, *s*, -NH_2), 6.83 (1H, *s*, -CH=), 7.0 – 8.0 (17H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{33}H_{26}F_4N_{10}$: C, 62.06; H, 4.10; N, 21.93. Found: C: 62.05; H: 4.08; N: 21.92%.

Compound (7d) IR (KBr,cm^{-1}): 3410 (N-H str.), 3071 (=CH str.), 815 (C-N str, *s*-triazine moiety), 830 (C-H bending), 1644 (C=N str, pyrimidinemoiety)1270 (C-O-C str.), 1087 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 5.1 (2H, *s*, -NH_2), 6.81 (1H, *s*, -CH=), 7.0 – 8.0 (17H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{31}H_{20}F_4N_{10}O_2$: C, 58.13; H, 3.15; N, 21.87. Found: C: 58.11; H: 3.12; N: 21.85%.

Compound (7e) IR (KBr,cm^{-1}): 3410 (N-H str.), 3072 (=CH str.), 816 (C-N str, *s*-triazine moiety), 832 (C-H bending), 1646 (C=N str, pyrimidinemoiety), 1271 (C-O-C str.), 1087 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 5.2 (2H, *s*, -NH_2), 6.82 (1H, *s*, -CH=), 7.1 – 8.1 (17H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{31}H_{20}F_5N_9$: C, 60.69; H, 3.29; N, 20.55. Found: C: 60.67; H: 3.27; N: 20.53%.

Compound (7f) IR (KBr,cm^{-1}): 3413 (N-H str.), 3074 (=CH str.), 818 (C-N str, *s*-triazine moiety), 835 (C-H bending), 1643 (C=N str, pyrimidinemoety), 1271 (C-O-C str.), 1087 (C-F str.), 780(C-Cl str.); ^{1}H NMR ($CDCl_3$, δ, ppm): 5.3 (2H, *s*, -NH_2), 6.85 (1H, *s*, -CH=), 7.1 – 8.1 (16H, *m*, Ar-H and -NH). Anal. Calcd. For $C_{31}H_{19}Cl_2F_4N_9$: C, 56.04; H, 2.88; N, 18.97. Found: C: 56.02; H: 2.85; N: 18.95%.

Preparation of 2,4-bis-(2',4'-difluorophenylamino)-6--[4'-{1''-phenyl-5''-(4'''-methoxyphenyl)-2''-pyrazolin-3''-yl} phenylamino]-*s*-triazine (8a)

Compound **6a** (0.01 mol) was dissolved in ethyl alcohol (25ml) and phenyl hydrazine hydrochloride (0.01 mol, 1.44 g) in 40 ml alcohol was refluxed for 10 hours in the presence of 40 % KOH (3ml). The progress of the reaction was monitored on TLC plate. After completion the reaction mixture was then cooled and poured into crushed ice and neutralized with dilute HCl. The product separated out was filtered, washed with water, dried and recrystallised from ethyl alcohol to give **8a**.

Similarly the remaining compounds **(8b-f)** were prepared by this method.

Compound (8a) IR (KBr,cm^{-1}): 3402 (N-H str.), 3060 (=CH str.), 809 (C-N str, *s*-triazine moiety), 1574 (C=N str, pyrazoline moiety), 2985 (C-H str, pyrazoline moiety), 1095 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.0 (1H, *dd*, -CHa-CH), 3.2 (1H, *dd*, -CHb-CH), 3.8 (3H, *s* , p-OCH_3), 7.0 – 8.0 (22H, *m*, Ar-H and -NH), 5.3 (1H, *dd*, -CH-CH_2-Ar-). Anal. Calcd. For $C_{37}H_{28}F_4N_8O$: C, 65.68; H, 4.17; N, 16.56. Found: C: 65.65; H: 4.15; N: 16.50%.

Compound (8b) IR (KBr,cm^{-1}): 3404 (N-H str.), 3065 (=CH str.), 811 (C-N str, *s*-triazine moiety), 1575 (C=N str., pyrazoline moiety), 2987 (C-H str., pyrazoline moiety), 1090 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.1 (1H, *dd*, -CHa-CH), 3.3 (1H, *dd*, -CHb-CH), 3.4 (3H, *s* , o-OCH_3), 3.6 (3H, *s*, p-OCH_3)7.0 – 8.0 (21H, *m*, Ar-H and -NH), 5.2 (1H, *dd*, -CH-CH_2-Ar-). Anal. Calcd. For $C_{38}H_{30}F_4N_8O_2$: C, 64.58; H, 4.28; N, 15.86. Found: C: 64.55; H: 4.25; N: 15.85%.

Compound (8c) IR (KBr,cm^{-1}): 3405 (N-H str.), 3065 (=CH str.), 810 (C-N str, *s*-triazine moiety), 1575 (C=N str, pyrazoline moiety), 2983 (C-H str, pyrazoline moiety), 1097 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.2 (1H, *dd*, -CHa-CH), 3.3 (1H, *dd*, -CHb-CH), 3.6 (3H, *s* , N-CH_3), 3.8 (3H, *s* , N-CH_3), 7.0 – 8.0 (22H, *m*, Ar-H and -NH), 5.4 (1H, *dd*, -CH-CH_2-Ar-). Anal. Calcd. For $C_{38}H_{31}F_4N_9$: C, 66.17; H, 4.53; N, 18.28. Found: C: 66.17; H: 4.52; N: 18.26%.

Compound (8d) IR (KBr,cm^{-1}): 3406 (N-H str.), 3066 (=CH str.), 809 (C-N str, *s*-triazine moiety), 1578 (C=N str, pyrazoline moiety), 2987 (C-H str, pyrazoline moiety), 1096 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.0 (1H, *dd*, -CHa-CH), 3.3 (1H, *dd*, -CHb-CH), 7.1 – 8.1 (22H, *m*, Ar-H and -NH), 5.5 (1H, *dd*, -CH-CH_2-Ar-). Anal. Calcd. For $C_{36}H_{25}F_4N_9O_2$: C, 62.52; H, 3.64; N, 18.23. Found: C: 62.50; H: 3.62; N: 18.20%.

Compound (8e) IR (KBr,cm^{-1}): 3407 (N-H str.), 3070 (=CH str.), 813 (C-N str, *s*-triazine moiety), 1580 (C=N str, pyrazoline moiety), 2987 (C-H str, pyrazoline moiety), 1098 (C-F str.) ; ^{1}H NMR ($CDCl_3$, δ, ppm): 3.1 (1H, *dd*, -CHa-CH), 3.3 (1H, *dd*, -CHb-CH), 7.2 – 8.2 (22H, *m*, Ar-H and -NH), 5.5 (1H, *dd*, -CH-CH_2-Ar-). Anal. Calcd. For $C_{36}H_{25}F_5N_8$: C, 65.06; H, 3.79; N, 16.86. Found: C: 65.05; H: 3.70; N: 16.80%.

Compound (8f) IR (KBr,cm^{-1}): 3408 (N-H str.), 3068 (=CH str.), 811 (C-N str, *s*-triazine moiety), 1578(C=N str, pyrazoline moiety), 2987 (C-H str, pyrazoline moiety), 1096 (C-F str.), 795 (C-Cl str.); ^{1}H NMR ($CDCl_3$, δ, ppm): 3.0 (1H, *dd*, -CHa-CH), 3.1 (1H, *dd*, -CHb-CH), 7.3 – 8.3 (21H, *m*, Ar-H and -NH), 5.5 (1H, *dd*, -CH-CH_2-Ar-). Anal. Calcd. For $C_{36}H_{24}Cl_2F_4N_8$: C, 60.43; H, 3.38; N, 15.66. Found: C: 60.40; H: 3.35; N: 15.60%.

RESULTS AND DISCUSSION

The IR spectrum of compound **6a** in KBr shows the characteristic band in the region of 1700-1647 cm^{-1} which indicate the presence of **-C=O** group. The IR spectrum of compound **7a** shows characteristic band in region of 1650-1600 cm^{-1} due to **–C=N** group. It also shows band in region the of 3500-3300 cm^{-1} due to **–NH_2** group. The IR spectrum of compound **8a** shows the characteristic band in the region of 1650-1570 cm^{-1} due to **(-C=N)**. The IR spectrum of compound **7a** and compound **8a** does not show any absorption band in the region of 1700- 1647 cm^{-1} which indicate the absence of **-C=O** group. ^{1}H NMR spectrum of compound **6a** shows doublet of **-CO-CH=** at δ 6.9 confirmed the presence of chalcone moiety. The ^{1}H NMR spectrum of compound **7a** shows sharp singlet of **–NH_2** at δ 5.1 confirmed the present of amino group in aminopyrimidine derivatives. The ^{1}H NMR spectrum of compound **8a** shows double doublet of **CH_a** at δ 3.0 and double doublet of **CH_b** at δ 3.2 confirmed the cyclisation in pyrazoline moiety. Result of IR and ^{1}H NMR analysis confirmed formation of desired products.

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