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Synthesis and evaluation of some novel S-triazine based chalcones and their derivatives

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ABSTRACT

Some new chalcones, 2-4 bis (tetrahydro- 1,4-oxazine)-6-[4'-{3''-(substituted phenyl) -2''propenon-1''-yl} phenyl amino]-s-triazine (**6a-f**) have been achieved by the reaction between 2-4-bis- tetrahydro- 1,4-oxazine -6-(4'-acetylphenylamino)-s-triazine (**5**) and different aromatic, which on cyclisation with hydrazine hydrate in presence of acetic acid give acetyl pyrazolines (**7a-f**). Chalcones (**6a-f**) on cyclisation with guanidine hydrochloride in presence of alkali give aminopyrimidines (**8a-f**). The characterization of newly synthesised compounds has been done on the basis of IR, ¹H NMR spectral data as well as elemental analysis.

Keyword: Chalcones, Acetyl pyrazolines, Aminopyrimidines, Spectral data, Elemental analysis.

INTRODUCTION

Number of derivatives containing s-triazine nucleus have been reported as heterocyclic compounds [1]. They are applicable as reactive dyes and also are used as polymers and drugs [2,3]. The s-triazine based chalcones and their derivatives have their own importance in heterocyclic chemistry due to their good biological activities [4,5]. Chalcones have been studied extensively because of their wide range of biological activity. They are found to be effective as antibacterial [6], antifungal [7], anti-inflammatory [8] and anticancer [9] etc.... Synthesis and characterization of pyrazoline derivatives have been developing field within the realm of the heterocyclic chemistry for the past several decades because of their ready accessibility through synthesis and wide range of chemical reactivity. Pyrazoline derivatives have been found to possess wide range of therapeutic activities such as antifungal [10] and anticancer [11] etc.... In recent years, the chemistry of pyridines and their derivatives have gained increasing attention because substituted pyridines are associated with different types of biological activities. Literature survey reveals that pyrimidine is parent of a series of compounds that are important in medicinal and industrial chemistry. Pyrimidines are one of the most important classes of heterocyclic compounds with variety of biological activities. As pyrimidine is basic nucleus in DNA and RNA, it has been found to associate with diverse pharmacological activities such as anticancer [12], anti-inflammatory [13], antiviral [14], antibacterial [15], antioxidant [16],

neuroprotective agent [17], Antiulcer [18] and CNS stimulant [19] etc.... In continuation of our work [20-24], the scope for further studies on chalcones and its derivatives, we herein report some novel chalcones (**6a-f**), acetyl pyrazolines (**7a-f**) and aminopyrimidines (**8a-f**). The synthesised compounds were ascertained from spectral analysis.

MATERIALS AND METHODS

Experimental

All the melting points were determined in an open capillary and are uncorrected. The IR spectra were recorded on Perkin-Elmer spectrum BX series FT-IR spectrophotometer. ¹H NMR spectra on a Varian Gemini 400 MHz spectrometer with CDCl₃ as a solvent and TMS as internal reference. The chemical shifts are expressed in parts per million (ppm) downfield from the internal standard and signals are quoted as *s* (singlet), *d* (doublet) and *m* (multiplate). Thin Layer Chromatography (TLC) analytical separation was conducted with Silica Gel 60 F-254 (Merck) plates of 0.25mm thickness eluted with visualized with UV (254nm) or iodine to check the purity of the synthesised compounds. Elemental analyses were carried out on a Perkin-Elmer series ii 2400 equipment.

Preparation of 2- tetrahydro- 1,4-oxazine-4, 6-dichloro-s-triazine (3).

Tetrahydro- 1,4-oxazine (0.01 mole, 0.87g in 10 ml acetone) was added slowly to cyanuric chloride (0.01 mole, 1.845g in acetone 30 ml) with constant stirring for 7 hours at 0 to 5 °C. Periodically, sodium carbonate solution (0.005 mole, 0.53g in 10 ml water) was added drop wise to neutralise HCl evolved during the reaction. Finally the content was poured into crushed ice. The solid separated out was filtered, washed with water, dried and recrystallised from alcohol to give (**3**).

Preparation of 2-4-bis- tetrahydro- 1,4-oxazine -6-chloro-s-triazine (4).

Tetrahydro- 1,4-oxazine (0.01 mole, 0.87g in 10 ml acetone) was added slowly to compound (3) (0.01 mole, 2.35g in 35 ml acetone) with constant stirring for 9 hours at room temperature. Periodically, sodium carbonate solution (0.005 mole, 0.53g in10 ml water) was added drop wise to neutralise HCl evolved during the reaction. Finally the content was poured into crushed ice. The solid separated out was filtered, washed with water, dried and recrystallised from alcohol to give (4).

Preparation of 2-4-bis- tetrahydro- 1,4-oxazine -6-(4'-acetylphenylamino)-s-triazine (5).

4-Aminoacetophenone (0.01 mole) and compound (4) (0.01 mole) were dissolved in acetone (40 ml). The reaction mix ture was refluxed for 6 hours cooled and poured in to crushed ice. Periodically, sodium carbonate solution (0.005, 0.53g in 10 ml water) was added to neutralise HCl evolved during the reaction. The progress of the reaction was monitored on TLC plate. After completion, the solid separated out was filtered, washed with water, dried and recrystallised from alcohol to give (5).

Yield 79%, m.p. 208 °C IR (KBr) cm⁻¹: 1659 (-C=O), 1020 (C-O-C), 800 (C-N, *s*-triazine). ¹H NMR (CDCl₃): δ 2.6 (s, 3H, -COCH₃), δ 3.68 (t, 8H, oxazine ring), δ 3.72 (t, 8H, oxazine ring).

Preparation of 2-4 bis (tetrahydro- 1,4-oxazine) 6- [4'- { 3''-(4'''-methoxyphenyl) -2''- propenon-1''-yl } phenyl amino]-s-triazine (6a).

Compound (5) (0.01 mole) was dissolved in DMF (30 ml) and 4-methoxybenzaldehyde (0.01 mole) was added to it. Then solution of KOH (5 ml 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 neutralise with HCl. The product separated out was filtered, washed with water and dried. The crude solid product was purified by repeted recrytalisation from alcohol.

Similarly, the remaining compounds (6b-f) were prepared by this method. Their physical data are given in Table-1

Compound (6a) : IR (KBr) cm⁻¹: 1649 (-C=O), 1220 (C-O-C, ether), 806 (C-N, *s*-triazine), 786 (=CH). ¹H NMR (CDCl₃) : 3.52 (t, 8H, oxazine ring), 3.63 (t, 8H, oxazine ring), δ 3.92 (s, 3H, p-OCH₃), δ 6.70 (s, 1H, -NH), δ 7.1 - 7.8 (m, 8H, Ar-H), δ 8.40 (d, 1H, -CO-CH=), δ 8.7 (d, 1H, Ar-CH=). Anal. Calcd. for C₂₇H₃₀N₆O₄ : C, 64.53; N, 16.72; H, 6.01 Found : C, 64.35; N, 16.55; H, 6.00%.

Compound (6b) : IR (KBr) cm⁻¹: 1639 (-C=O), 1545 (C-NO₂), 1250 (C-O-C, ether), 801 (C-N, *s*-triazine), 797 (=CH). ¹H NMR (CDCl₃) : 3.42 (t, 8H, oxazine ring), 3.55 (t, 8H, oxazine ring), δ 6.77 (s, 1H, -NH), δ 7.2 - 7.8 (m, 8H, Ar-H), δ 8.33 {d, 1H, -CO-CH=), δ 8.62 (d, 1H, Ar-CH=). Calcd. for C₂₆H₂₇N₇O₅ : C, 60.34; N, 18.95; H, 5.68 Found : C, 60.30; N, 18.55; H, 5.52%.

Compound (6c) : IR (KBr) cm⁻¹: 1619 (-C=O), 1234 (C-O-C, ether), 809 (C-N, *s*-triazine), 820 (=CH). ¹H NMR (CDCl₃) : 3.39 (t, 8H, oxazine ring), 3.50 (t, 8H, oxazine ring), δ 3.89 (s, 3H, o-OCH₃), δ 4.09 (s, 3H, p-OCH₃), δ 6.70 (s, 1H, -NH), δ 7.1 - 7.8 (m, 7H, Ar-H), δ 8.23 (d, 1H, -CO-CH=), δ 8.52 (d, 1H, Ar-CH=). Anal. Calcd. for C₂₈H₃₂N₆O₅ : C, 63.15; N, 15.78; H, 6.05 Found : C, 63.10; N, 15.55; H, 6.02%.

Compound (6d) : IR (KBr) cm⁻¹: 1638 (-C=O), 1259 (C-O-C, ether), 1020 (-C-F), 800 (C-N, *s*-triazine), 793 (=CH). ¹H NMR (CDCl₃) : 3.45 (t, 8H, oxazine ring), 3.60 (t, 8H, oxazine ring), δ 6.57 (s, 1H, -NH), δ 7.0 - 8.1 (m, 8H, Ar-H), δ 8.41 {d, 1H, -CO-CH=), δ 8.81 (d, 1H, Ar-CH=). Anal. Calcd. for C₂₆H₂₇FN₆O₃ : C, 63.66; N, 17.13; H, 5.54 Found : C, 63.60; N, 17.55; H, 5.52%.

Compound (6e) : IR (KBr) cm⁻¹: 1622 (-C=O), 1244 (C-O-C, ether), 806 (C-N, *s*-triazine), 802 (=CH), 786 (C-Cl). ¹H NMR (CDCl₃) : 3.39 (t, 8H, oxazine ring), 3.50 (t, 8H, oxazine ring), δ 6.70 (s, 1H, -NH), δ 7.1 - 7.8 (m, 7H, Ar-H), δ 8.23 (d, 1H, -CO-CH=), δ 8.52 (d, 1H, Ar-CH=). Anal. Calcd. for C₂₆H₂₆ Cl₂N₆O₃ : C, 57.68; N, 15.52; H, 4.84 Found : C, 57.55; N, 15.35; H, 4.72%.

Compound (6f) : IR (KBr) cm⁻¹: 1682 (-C=O), 1257 (C-O-C, ether), 802 (C-N, *s*-triazine), 818 (=CH). ¹H NMR (CDCl₃) : 3.29 (t, 8H, oxazine ring), 3.30 (t, 8H, oxazine ring), δ 3.42 (s, 6H, N-CH₃), δ 6.70 (s, 1H, -NH), δ 7.1 - 7.9 (m, 7H, Ar-H), δ 8.29 (d, 1H, -CO-CH=), δ 8.50 (d, 1H, Ar-CH=). Anal. Calcd. for C₂₈H₃₃N₇O₃ : C, 65.23; N, 19.02; H, 6.45 Found : C, 65.10; N, 19.00; H, 6.32%.

Preparation of 2,4-bis-(tetrahydro-1,4-oxazine)-6-[4''-{1''-acetyl-5''-(4'''-methoxyphenyl)-2''-pyrazoline-3''-yl} phenylamino]-s-triazine (7a)

Compound (**6a**) (0.01 mol) in minimum amount of glacial acetic acid and hydrazine hydrate (0.015 mol, 0.75g) was refluxed for 4 hours. The progress of the reaction was monitored on TLC plate. After completion, the reaction mixture was cooled and poured into crushed ice and the product separated out was filtered, washed with water and recrystallised from alcohol to give (**7a**).

Similarly, the remaining compounds (**7b-f**) were prepared by this method. Their physical data are given in **Table-1**

Compound (7a) : IR (KBr) cm⁻¹: 3272 (-NH), 1650 (C=O), 1613 (C=N, pyrazoline moiety), 1039 (C-O-C). ¹H NMR (CDCl₃) δ ppm : δ 2.5 (s, 3H, -COC<u>H₃</u>), 3.0 (dd, 1H, -C<u>H</u>a-CH-), 3.6 (d, 1H, -C<u>H</u>b-CH-), δ 3.58 (t, 8H, -CH₂), δ 3.78 (t, 8H, -CH₂), 3.7 (s, 3H, p-OC<u>H₃</u>), 5.8 (dd, 1H, -C<u>H</u>-CH₂), 6.8 to 7.8 (m, 9H, 8Ar-<u>H</u> and 1-N<u>H</u>). Anal. Calcd. for C₂₉H₃₄N₈O₄ : C, 62.35; N, 20.06; H, 6.13 Found : C, 62.32; N, 20.04; H, 6.11%.

Compound (7b) : IR (KBr) cm⁻¹: 3275 (-NH), 1640 (C=O), 1610 (C=N, pyrazoline moiety), 1075 (C-NO₂), 1045 (C-O-C). ¹H NMR (CDCl₃) δ ppm : δ 2.5 (s, 3H, -COC<u>H</u>₃), 3.0 (dd, 1H, -C<u>H</u>a-CH-), 3.6 (d, 1H, -C<u>H</u>b-CH-), δ 3.58 (t, 8H, -CH₂), δ 3.78 (t, 8H, -CH₂), 5.8 (dd, 1H, -C<u>H</u>-CH₂), 6.8 to 7.8 (m, 9H, 8Ar-<u>H</u> and 1-N<u>H</u>). Anal. Calcd. for C₂₈H₃₁N₉O₅ : C, 58.63; N, 21.98; H, 5.45 Found : C, 58.60; N, 21.94; H, 5.43%.

Compound (7c) : IR (KBr) cm⁻¹: 3260 (-NH), 1666 (C=O), 1611 (C=N, pyrazoline moiety), 1032 (C-O-C). ¹H NMR (CDCl₃) δ ppm : δ 2.5 (s, 3H, -COC<u>H₃</u>), 3.0 (dd, 1H, -C<u>H</u>a-CH-), 3.4 (d, 1H, -C<u>H</u>b-CH-), δ 3.58 (t, 8H, -CH₂), δ 3.60 (t, 8H, -CH₂), 3.7 (s, 3H, o-OC<u>H₃</u>), 3.79 (s, 3H, m-OC<u>H₃</u>), 5.8 (dd , 1H, -C<u>H</u>-CH₂), 6.8 to 7.8 (m, 9H, 8Ar-<u>H</u> and 1-N<u>H</u>). Anal. Calcd. for C₃₀H₃₆N₈O₅ : C, 61.21; N, 19.03; H, 6.16 Found : C, 61.20; N, 19.04; H, 6.11%.

Compound (7d) : IR (KBr) cm⁻¹: 3273 (-NH), 1640 (C=O), 1615 (C=N, pyrazoline moiety), 1070 (C-F), 1043 (C-O-C). ¹H NMR (CDCl₃) δ ppm : δ 2.54 (s, 3H, -COC<u>H₃</u>), 3.1 (dd, 1H, -C<u>H</u>a-CH-), 3.5 (d, 1H, -C<u>H</u>b-CH-), δ 3.66 (t, 8H, -CH₂), δ 3.78 (t, 8H, -CH₂), 5.9 (dd, 1H, -C<u>H</u>-CH₂), 6.8 to 7.8 (m, 9H, 8Ar-<u>H</u> and 1-N<u>H</u>). Anal. Calcd. for C₂₈H₃₁ FN₈O₃ : C, 61.53; N, 20.50; H, 5.72 Found : C, .60; N, 21.94; H, 5.43%.

Compound 7(e) : IR (KBr) cm⁻¹: 3270 (-NH), 1645 (C=O), 1605 (C=N, pyrazoline moiety), 780 (C-Cl), 1040 (C-O-C). ¹H NMR (CDCl₃) δ ppm : δ 2.49 (s, 3H, -COC<u>H₃</u>), 3.19 (dd, 1H, -C<u>H</u>a-CH-), 3.45 (d, 1H, -C<u>H</u>b-CH-), δ 3.60 (t, 8H, -CH₂), δ 3.72 (t, 8H, -CH₂), 5.89 (dd, 1H, -C<u>H</u>-CH₂), 6.7 to 7.9 (m, 9H, 8Ar-<u>H</u> and 1-N<u>H</u>). Anal. Calcd. for C₂₈H₃₁ FN₈O₃ : C,56.29; N, 18.75; H, 5.06 Found : C,56.20; N, 18.72; H, 5.03%.

Compound (7f) : IR (KBr) cm⁻¹: 3260 (-NH), 1666 (C=O), 1611 (C=N, pyrazoline moiety), 1032 (C-O-C). ¹H NMR (CDCl₃) δ ppm : δ 2.4 (s, 3H, -COC<u>H₃</u>), 3.09 (dd, 1H, -C<u>H</u>a-CH-), 3.38 (d, 1H, -C<u>H</u>b-CH-), δ 3.56 (t, 8H, -CH₂), δ 3.67 (t, 8H, -CH₂), 3.79 (s, 6H, -C<u>H₃</u>), 5.7 (dd, 1H, -C<u>H</u>-CH₂), 6.8 to 8.6 (m, 9H, 8Ar-<u>H</u> and 1-N<u>H</u>). Anal. Calcd. for C₃₀H₃₇N₉O₃ : C, 63.03; N, 22.05; H, 6.52 Found : C, 63.01; N, 22.04; H, 6.50%.

Preparation of 2,4-bis-tetrahydro- 1,4-oxazine-6-[4'-{2"-amino-6"-(4'''-methoxyphenyl)-pyrimidin-4"-yl} phenyl amino]-s-triazine(8a)

Compound (6a) (0.01 mol) was dissolved in alcohol (25 ml) and guanidine hydrochloride (0.01 mol) was added to it. Then solution of KOH (5 ml of 40%) was added to the reaction mixture and refluxed for 10 hours. The reaction mixture was cooled, poured into crushed ice and product separated out was filtered, washed with water, dried and recrystallised from alcohol to give (7a). Similarly, the remaining compounds (7b-f) were prepared by this method. Their physical data are given in Table-1.

Compound (8a) : IR (KBr) cm⁻¹: 3398 (-NH₂), 1575 (C=N), 806 (C-N, *s*-triazine), ¹H NMR (CDCl₃) : δ 3.85 (s, 3H, p-OCH₃), δ 3.58 (t, 8H, –CH₂), δ 3.78 (t, 8H, –CH₂), 5.1 (s, 2H, -NH₂),

6.90 to 8.15 (m, 10H, 9H, Ar-H and 1H, -NH). Anal. Calcd. for $C_{28}H_{31}N_9O_3$: C, 62.09; N, 23.27; H, 5.77. Found : C, 62.07; N, 23.25; H, 5.76%.

Compound (8b) : IR (KBr) cm⁻¹: 3382 (-NH₂), 1564 (C=N), 1075 (C-NO₂), 800 (C-N, *s*-triazine), ¹H NMR (CDCl₃) : δ 3.46 (t, 8H, –CH₂), δ 3.69 (t, 8H, –CH₂), 5.4 (s, 2H, -NH₂), 6.90 to 8.15 (m, 10H, 9H, Ar-H and 1H, -NH). Anal. Calcd. for C₂₇H₂₈N₁₀O₄ : C, 58.27; N, 25.17; H, 5.07. Found : C, 58.24; N, 25.15; H, 5.03%.

Compound (8c) : IR (KBr) cm⁻¹: 3398 (-NH₂), 1575 (C=N), 806 (C-N, *s*-triazine), ¹H NMR (CDCl₃) : δ 3.75 (s, 3H, o-OCH₃), δ 3.80 (s, 3H, m-OCH₃), δ 3.50 (t, 8H, –CH₂), δ 3.75 (t, 8H, – CH₂), 5.23 (s, 2H, -NH₂), 6.90 to 8.15 (m, 9H, 8H, Ar-H and 1H, -NH). Anal. Calcd. for C₂₉H₃₃N₉O₄ : C, 60.94; N, 22.05; H, 5.81. Found : C, 60.90; N, 22.02; H, 5.79%.

Compound (8d) : IR (KBr) cm⁻¹: 3380 (-NH₂), 1562 (C=N), 1075 (C-F), 808 (C-N, *s*-triazine), ¹H NMR (CDCl₃) : δ 3.46 (t, 8H, –CH₂), δ 3.69 (t, 8H, –CH₂), 5.4 (s, 2H, -NH₂), 6.90 to 8.15 (m, 10H, 9H, Ar-H and 1H, -NH). Anal. Calcd. for C₂₇H₂₈FN₉O₂ : C, 61.24; N, 23.80; H, 5.33. Found : C, 61.20; N, 23.76; H, 5.31%.

Compound (8e) : IR (KBr) cm⁻¹: 3375 (-NH₂), 1562 (C=N), 787(C-Cl), 803 (C-N, *s*-triazine), ¹H NMR (CDCl₃) : δ 3.46 (t, 8H, –CH₂), δ 3.69 (t, 8H, –CH₂), 5.4 (s, 2H, -NH₂), 6.90 to 8.15 (m, 9H, 8H, Ar-H and 1H, -NH). Anal. Calcd. for C₂₇H₂₇Cl ₂N₉O₂ : C, 55.87; N, 21.27; H, 4.69. Found : C, 55.85; N, 21.24; H, 4.66%.

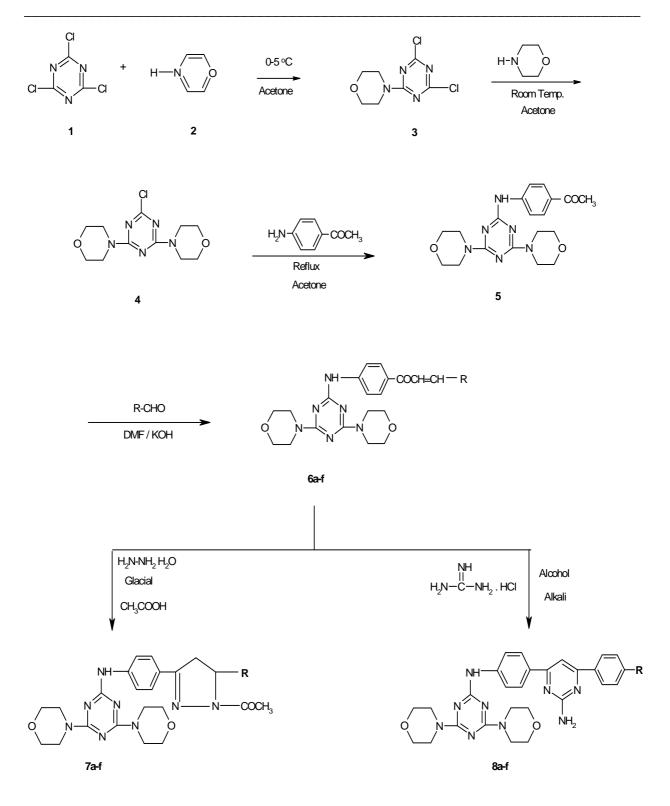
Compound (8f) : IR (KBr) cm⁻¹: 3398 (-NH₂), 1575 (C=N), 806 (C-N, *s*-triazine), ¹H NMR (CDCl₃) : δ 3.75 (s, 6H, -CH₃), δ 3.50 (t, 8H, -CH₂), δ 3.75 (t, 8H, -CH₂), 5.23 (s, 2H, -NH₂), 6.90 to 8.15 (m, 10H, 9H, Ar-H and 1H, -NH). Anal. Calcd. for C₂₉H₃₄N₁₀O₂ : C, 62.80; N, 25.25; H, 6.18. Found : C, 62.77; N, 25.23; H, 6.17%.

RESULTS AND DISCUSSION

Chalcones (**6a-f**) were prepared from cyanuric chloride. The IR spectrum of (**6a**) showed the characteristic band at 1649 cm⁻¹ due to >C=O group. The ¹H NMR spectrum of (**6a**) in CDCl₃ showed a doublet at δ 8.40 due to -CO-CH=. The aromatic cluster appeared at δ (7.1-7.8) with eight aromatic protons. The elemental analysis of compound (**6a**) was in good agreement with the molecular formula.

The product (7a) was obtained by the treatment of (6a) with hydroxylamine hydrochloride in presence of alkali. The IR spectrum of (7a) showed the characteristic band at 1575 cm⁻¹ due to – C=N group (isoxazole moiety). The IR spectrum of (7a) do not show any absorption bands in the region of 1700 -1600 cm⁻¹ which indicate the absence of –C=O group. The ¹H NMR spectrum of (7a) showed singlet at 5.8 due to -CH of isoxazole ring protons. The aromatic cluster appeared at δ (6.8-7.8) with eight aromatic protons. The elemental analysis of compound (7a) was in good agreement with the molecular formula.

Further the reaction of (6a) with malononitrile in presence of ammonium acetate give (8a). The IR spectrum of compound (8a) showed characteristic band at 3398 cm⁻¹ due to primary amine. It showed strong characteristic band at 1575 cm⁻¹ due to -C=N. The ¹H NMR spectrum of (8a) showed singlet at (5.1) due to -NH₂. The aromatic cluster appeared at δ (6.90-8.15) with ten protons.





Compounds	R	M.P.	% Yield
6a	4-Methoxyphenyl	145°-152 °C	75
6b	4-Nitrophenyl	160°-164 °C	69
6с	2,5-Dimethoxyphenyl	112°-118 °C	76
6d	4-Fluorophenyl	106°-110 °C	69
6e	2,3-Dichlorophenyl	112°-115 °C	66
6f	4-N,N-dimethylaminophenyl	262°-268 °C	72
7a	4-Methoxyphenyl	116°-121 °C	70
7b	4-Nitrophenyl	217°-223 °C	72
7c	2,5-Dimethoxyphenyl	123°-128 °C	59
7d	4-Fluorophenyl	257 °-261 °C	68
7e	2,3-Dichlorophenyl	125°-128 °C	55
7f	4-N,N-dimethylaminophenyl	101 °-106 °C	51
8a	4-Methoxyphenyl	98°-105 °C	65
8b	4-Nitrophenyl	86°-93 °C	59
8c	2,5-Dimethoxyphenyl	116°-122 °C	67
8d	4-Fluorophenyl	120°-126 °C	58
8e	2.3-Dichlorophenyl	92°-97 °C	49
8f	4-N,N-dimethylaminophenyl	110°-115 °C	64

Table-1 The physical data of synthesised compounds (6a-f), (7a-f) and (8a-f)

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