Chapter 3

Synthesis and Characterization
3.1. Introduction

Pyrazole is an organic compound with the formula C\(_3\)H\(_3\)N\(_2\)H. It is a heterocycle characterized by a 5-membered ring of three carbon atoms and two adjacent nitrogen centers. Pyrazolines are the dihydro form of the pyrazoles with the molecular formula C\(_3\)H\(_6\)N\(_2\) and, depending upon the position of double bond, these may be classified as; 1-pyrazoline, 2-pyrazoline or 3-pyrazoline.

![Structure of different pyrazolines](image)

**Figure 3.1**: Structure of different pyrazolines

Out of the three tautomeric structures, 2-pyrazolines are the most stable and frequently used. 2-Pyrazolines can be considered as a cyclic hydrazine moiety which plays a crucial role in the development of theory in heterocyclic chemistry and is extensively used as useful synthon in organic synthesis. Pyrazolines are basic molecules and the ease of protonation is dependent on the position of double bonds. Unlike pyrazoles, which are stable to acid and bases, pyrazolines are less stable than corresponding pyrazoles. These are decomposed by hot water and can be easily oxidized and reduced. They can be converted into the pyrazoles by using mild oxidizing agents such as bromine or lead tetra acetate, however, their mild reduction give pyrazolidines. Also, pyrazolines undergo ring cleavage, rearrangement or side chain formation. 2-Pyrazoline is insoluble in water but soluble in propylene glycol because of its lipophilic character. 2-Pyrazolines, without any substituent at the N1 position, can react with benzaldehyde at high temperature (200°C) and in an inert atmosphere to give 4-benzylidene derivatives.\(^{176}\)

Pyrazoles are synthesized by the reaction of \(\alpha,\beta\)-unsaturated aldehydes/ketones with hydrazine, followed by subsequent dehydrogenation.

![Scheme 3.1: Synthesis of pyrazoles from chalcones](image)
Conventional approaches for the preparation of substituted pyrazoles involve either the construction of two C-N bonds by condensation of hydrazines with 1,3-dicarbonyl compounds or their 1,3-dielectrophilic equivalents or the generation of one C-N bond and one C-C bond by intermolecular [3+2]-cycloadditions of 1,3-dipoles to dipolarophiles.

\[
\begin{align*}
\text{O} & \quad \text{O} \\
\text{+ N}_2\text{H}_4 & \quad \text{\rightarrow (CH}_3\text{)_2C}_3\text{HN}_2\text{H + 2H}_2\text{O}} \\
\end{align*}
\]

**Scheme 3.2:** Synthesis of pyrazoles from diketones

Chalcones (1,3-diaryl-2-propen-1-ones), which can be easily obtained in high chemical yields by a Claisen Schmidt condensation of functionalized acetophenones and benzaldehydes, are adequate building blocks to prepare 3,5-diarylpyrazoles. A series of 4-alkyl-1,3,5-triarylpyrazoles were regioselectively obtained by oxidation of pyrazolines. These were in turn prepared by means of a cyclocondensation reaction between phenyl and 4-methoxy phenylhydrazine and chalcones, followed by alkylation at the C-4 position of the pyrazoline ring. On the other hand, chalcones underwent a regioselective cyclocondensation with phenyl or 6-fluorobenzothiazol-2-ylhydrazine in the presence of a catalytic amount of glacial acetic acid in refluxing ethanol, leading to the corresponding pyrazolines, which were subsequently oxidized with iodine (III) to pyrazoles in good yields. Starting from chalcones, 1-phenyl-3,5-triarylpyrazoles were also obtained in a one-pot procedure (70-88% yield) by reaction with phenylhydrazine in refluxing AcOH in the presence of one equivalent of elemental iodine.\(^{206-207}\)

The IR spectra of these compounds show the disappearance of C=C (olefinic) and N–H stretching bands at 1584–1608 and 3257–3366 cm\(^{-1}\) respectively due to the ring closure. The IR spectra of the compounds afford pyrazoline C=N stretching (1501-1576 cm\(^{-1}\)), C-H deformation (1362-1464 cm\(^{-1}\)), C5-N1 stretching (1069-1189 cm\(^{-1}\)), carbamoyl group N-H stretching (3112-3481 cm\(^{-1}\)) and C=O stretching (1315-1357 cm\(^{-1}\)) bands. In the \(^1\)H-NMR spectra, olefinic protons of chalcone appear as doublets at about 6.75 and 7.18 ppm respectively. After the ring closure, ring protons (HA and HB) of the final compounds appear at around 3.07 and 3.8 ppm as a doublet of doublet and also vicinal methine proton, HX show triplet/multiplet at about 5.4 ppm due to vicinal
coupling with the two magnetically nonequivalent protons of the methylene group HA (upfield H of CH₂) and HB (downfield H of CH₂). N-H protons of the carbamoyl group are seen at 10.1-10.3 ppm, generally as broad bands. The phenyl protons are observed at the expected chemical shifts and integral values. 212-214

In past decade, interest in pyrazole chemistry has significantly increased because of its interesting properties. Pyrazole, is a motif found in a number of small molecules that possess a wide range of agricultural and pharmaceutical activities. Pyrazoles are used in supra-molecular and polymer chemistry, in the food industry, as cosmetic colorings, UV stabilizers; however, some others have liquid crystal properties. The discovery of this class of drugs provides an outstanding case history of modern drug development and also points out the unpredictability of pharmacological activity from structural modification of a prototype drug molecule. Substituted pyrazoles have also been applied as ligands for transition metal-catalyzed reactions. Pyrazoline interest has extended to the manufacturing of dyes and dye couplers. Accordingly, agrochemical, pharmaceutical, and chemical industries have a great interest in their synthesis.

Pyrazolines have a variety of medicinal applications, and their derivatives were found to possess potential anti-inflammatory, antipyretic-analgesic, tranquillizing, muscle relaxant, psychoanaleptic, antiepileptic, antidepressant, antimicrobial, antiparasitic, antitubercular, insecticidal, anti-hypotensive, anti-diabetic, and anaesthetic activities. Some of their analogs were also found to exhibit cytotoxic activity, antiviral, inhibitory activity of platelet aggregation, herbicidal activity, Nitric oxide synthase (NOS) inhibition and cannabinoid CB₁-receptor modulation.

Remarkably, the 1-arylpyrazole motif is present in drugs such as cyclooxygenase-2 (Cox-2; e.g. Celecoxib) inhibitors and protein kinase inhibitors, as well as in antifungal compounds. Some 1,5-diarylpyrazole derivatives exhibit inhibitory activities of the HIV-1 reverse transcriptase, whereas 1,3,5-triaryl-4-alkyl pyrazoles are efficient ligands for the estrogen receptor. 2-Pyrazolines display a broad spectrum of pharmacological activities and are present in a number of pharmacologically active molecules such as phenazone/ amidopyrene/ methampyrone (analgesic and antipyretic), azolid/ tandearil (anti-inflammatory), indoxacarb (insecticidal), anturane (uricosuric), etc. 171, 176, 207
3.2. Materials and methods

The chemicals and reagents were procured from Sigma Aldrich, and S. D. Fine Chemicals, Mumbai, India and pre-coated TLC sheets were purchased from Merck Chemicals, India and were used as such. Solvents were of reagent grade and were purified and dried by standard procedures. Microwave assisted synthesis was performed using Raga’s Scientific Microwave Systems (Ragatech, Pune, Maharashtra, India). Melting point was determined by open capillary method and are uncorrected. IR spectra were recorded on Bruker FT-IR, ALPHA-T (Eco-ATR) spectrophotometers, (Bruker, Tech. Pvt. Ltd., USA) and values are expressed in cm\(^{-1}\). \(^1\)H-NMR and \(^{13}\)C-NMR spectra were recorded on Bruker Avance-400, FTNMR spectrometer (Bruker, Tech. Pvt. Ltd., USA) at 400MHz and the chemical shifts are reported in parts per million (δ value), taking TMS (δ 0 ppm for \(^1\)H NMR) as the internal standard. Mass spectra were recorded on Waters UPLC-TQD Mass Spectrometer instrument (Waters Corporation, USA) using LC-ESI or APCI-MS Technique. Elemental analysis was performed on Perkin Elmer-2400, Series-II Analyzer (Waltham, Massachusetts, USA).

3.2.1. Synthesis

Synthesis of 1,3,5-trisubstituted-2-pyrazoline derivatives was carried out in three steps, using conventional methods (stirring and refluxing) as well as an environmentally benign green synthetic approach under the exposure of microwave irradiations. Microwave assisted organic synthesis is a technique which can be used to rapidly explore “chemical space” and increase the diversity of the compounds produced. It has been observed that all the conventionally heated reactions could be performed more conveniently using this technique, at a faster rate and in good yields.

In the first step, substituted aldehydes and ketones were reacted in a basic medium to form the chalcone derivatives (1a-1t), through Claisen-Schmidt condensation as given in Scheme 3.3. Now these suitably substituted chalcone derivatives (1a-1t) were heterocyclized into 3,5-disubstituted-2-pyrazoline derivatives (2a-2t), using hydrazine hydrate in excess as given in Scheme 3.4. Finally, the N1 position of the 3,5-disubstituted-2-pyrazoline intermediates was substituted using 4-nitrobenzenesulfonylchloride (3a-3t), 4-methoxybenzenesulfonylchloride (4a-4t) and 4-chlorobenzenesulfonylchloride (5a-5t)
reagent in the final step of the reaction, as given in **Scheme 3.5.** Progress of the reactions was monitored by thin layer chromatography (TLC) on precoated silica gel G plates, using iodine vapors and UV light as the visualizing agents.

### 3.2.1.1. General procedure for the synthesis of chalcone derivatives (Step 1)

**Conventional method:** To a solution of different ketones (0.01 M) and suitably substituted aldehydes (0.01 M) in ethanol (10 mL), aqueous solution of potassium hydroxide (60%) was added drop wise and with continuous stirring at 0°C over a period of 15min-2hrs. The reaction mixture was stirred at a low temperature (0-10°C) for about 24-48 hrs, with occasional shaking. After completion of the reaction, it was poured into ice-cold water and then neutralized to pH 2 using 6 N hydrochloric acid. The yellow colored intermediates (chalcones) obtained were filtered, washed, dried, and re-crystallized from methanol.52, 53, 181, 184, 216

![Conventional Method](image)

**Where R:**

- ![ification](image)

**and R':**

- ![ification](image)

**Scheme 3.3:** Synthesis of chalcones derivatives (1a-1t)

**Microwave assisted organic synthesis (MAOS):** Different aromatic/ hetero-aromatic ketones (0.01 M) and suitably substituted aldehydes (0.01 M) were reacted, in the presence of hydro-alcoholic solution of KOH (60%, 10mL), under microwave irradiation (MWI: 120-280W, 60-230s). The reaction mixture was poured into ice-cold water and
then neutralized to pH 2 using 6 N hydrochloric acid. The yellow colored intermediates (chalcones) obtained were filtered, washed, dried, and re-crystallized from methanol.²¹⁷

3.2.1.2. General procedure for the synthesis of 3,5-disubstituted-2-pyrazoline derivatives (Step II)

Conventional method: Appropriate chalcone derivatives were treated with 10 times excess of hydrazine hydrate (80%) in dry ethanol and refluxed for 3–6 hrs. The hot reaction mixture was then poured into ice-cold water. The separated out solid was filtered, washed, dried and re-crystallized from ethanol/acetone/ethyl acetate to afford the respective pyrazoline.¹⁸¹, ¹⁸⁴

![Conventional Method](image)

**Scheme 3.4:** Synthesis of 3,5-disubstituted-2-pyrazoline derivatives (2a-2t)

Microwave assisted organic synthesis (MAOS): Appropriate chalcone derivatives were treated with 10 times excess of hydrazine hydrate (80%) in dry ethanol, under the exposure of microwave irradiation (MWI: 240-350W; 50-400s). The reaction mixture was then poured into ice-cold water. The separated out solid was filtered, washed, dried and re-crystallized from ethanol/acetone/ethyl acetate to afford the respective pyrazolines.¹⁸⁹, ²¹⁸

3.2.1.3. General procedure for the synthesis of 1,3,5-trisubstituted-2-pyrazoline derivatives (Step III)

Conventional method: Appropriately substituted 3,5-disubstituted-2-pyrazolines (0.001M) were reacted with 0.002M of 4-nitrobenzenesulfonylchloride (for compounds,
3a-3t), 4-methoxybenzenesulfonyl chloride (for compounds 4a-4t) and 4-chlorobenzenesulfonyl chloride (for compounds 5a-5t) by stirring, taking tetra hydro furan (10 mL) as the solvent. Stirring was continued for 1-4 hrs. After completion of reaction, the reaction mixture was poured on a petri plate and solvent was evaporated to dryness. The sticky crude product was re-precipitated using acetonitrile or methanol. Recrystallization was done with acetonitrile or methanol to obtain the pure product.

Scheme 3.5: Synthesis of 1,3,5-trisubstituted-2-pyrazoline derivatives

(3a-3t, 4a-4t and 5a-5t)

Microwave assisted organic synthesis (MAOS): Appropriately substituted 3,5-disubstituted-2-pyrazolines (0.001M) were reacted with 0.002M of 4-nitrobenzenesulfonyl chloride (for compounds, 3a-3t), 4-methoxybenzenesulfonyl chloride (for compounds, 4a-4t) and 4-chlorobenzenesulfonyl chloride (for compounds, 5a-5t) under the exposure of microwave irradiation (MWI: 210-350W; 80-280s), taking tetra hydro furan (10 mL) as the solvent. After completion of reaction, the reaction mixture was poured on a petri plate and solvent was evaporated to dryness. The sticky crude product was re-precipitated using acetonitrile or methanol. Recrystallization was done with acetonitrile or methanol to obtain the pure product.
3.2.1.4. Purification of the synthesized derivatives

The intermediates from first and second steps were purified by recrystallization and crystal washing methods using methanol as solvent. However, the final step derivatives were purified by recrystallization followed by column chromatography. For this, silica gel (200-400 mesh) was used as stationary phase to pack the column by wet packing method. Chloroform and methanol were taken as mobile phase, in varying polarity (9.9:0.1 to 8:2), to elute the desired compound. Fractions with similar TLC pattern were mixed and dried on a rotatory evaporator to obtain the pure product.

3.2.2. Characterization of the synthesized derivatives

3.2.2.1. Characterization of chalcone derivatives (1a-1t)

The intermediates from step-I was characterized by general physicochemical methods such as- % yield determination, solubility studies, thin layer chromatographic analysis, melting point determination, and mass spectrometric techniques.

3.2.2.2. Characterization of the 3,5-disubstituted-2-pyrazoline derivatives (2a-2t)

The second step derivatives were characterized by general physicochemical methods (such as- % yield determination, solubility studies, thin layer chromatographic analysis, melting point determination and elemental analysis) along with modern spectral techniques (such as- IR spectroscopy and mass spectrometry).

3.2.2.3. Characterization of the 1,3,5-trisubstituted-2-pyrazoline derivatives (3a-3t, 4a-4t and 5a-5t)

The third step derivatives were characterized by complete physicochemical methods (such as- color and state, % yield determination, solubility studies, thin layer chromatographic analysis, melting point determination and elemental analysis) along with modern spectral techniques (such as- IR, $^1$H-NMR, $^{13}$C-NMR and mass spectroscopy).

3.3. Results and discussion

 Appropriately substituted aromatic/ heteroaromatic aldehydes and ketones were reacted in the basic medium to form the substituted chalcones (1a-1t), through Claisen-Schmidt condensation in the first step of the reaction. This was followed by
heterocyclization of substituted chalcones to 2-pyrazoline derivatives (2a-2t), using hydrazine hydrate in excess. Further, the N1 position of the 2-pyrazoline intermediates was substituted by 4-nitrobenzenesulfonylchloride (3a-3t), 4-methoxy-benzenesulfonylchloride (4a-4t) and 4-chlorobenzenesulfonylchloride (5a-5t) in the final step of the reaction using both conventional and microwave assisted procedures, as given in Schemes 3.3, 3.4 and 3.5. The progress of the reactions was monitored by thin layer chromatography (TLC) on pre-coated silica gel G plates, using iodine vapors and UV light as the visualizing agents.

Intermediates from the first step (1a-1t) were characterized by % yield determination, solubility studies, thin layer chromatographic analysis and melting point determinations. The formation of compounds was further confirmed by mass spectrometry and the data for the same is given in Table 3.1.

The second step derivatives were characterized by physicochemical and spectral techniques and the values were found to be in accordance with the proposed derivatives. The physicochemical characterization details are provided in Table 3.2. Spectral analysis data expounds that IR spectra of the synthesized compounds afforded absorption bands in the regions corresponding to C=N stretching (1509-1612 cm⁻¹), N-H stretching (3456-3105 cm⁻¹) and C-H deformation (1428-1357 cm⁻¹). ¹H-NMR spectra of the compounds exhibited the presence of two non-equivalent protons of a methylene group (Hₐ/Hₖ) at δ 2.92–3.38 ppm and 3.70–3.93, coupled with each other and in turn with the vicinal methine proton (Hₓ) at δ 6.68–7.04. All the other aliphatic, aromatic and heteroaromatic protons were also observed at their expected ppm values. Details of spectral characterization for the second step derivatives are as follows-

4-[5-(3,4-Dimethoxy-phenyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (2a)

IR (cm⁻¹): N-H str (3278), C-H Ar (3044), C=N str (1597), C-H deform (1429). Anal. Calcd. for C_{17}H_{18}N_{2}O_{3}: C, 68.44; H, 6.08; N, 9.39. Found: C, 68.13; H, 6.55; N, 10.30.

4-[5-(4-Chloro-phenyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (2b)

IR (cm⁻¹): N-H str (3359), C-H Ar (3046), C=N str (1572), C-H deform (1461). Anal. Calcd. for C_{15}H_{13}ClN_{2}O: C, 66.06; H, 4.80; N, 10.27. Found: C, 65.97; H, 5.03; N, 10.28.

4-(5-Furan-2-yl-4,5-dihydro-1H-pyrazol-3-yl)-phenol (2c)
IR (cm\(^{-1}\)): N-H str (3283), C-H Ar (3112), C=N str (1600), C-H deform (1385). Anal. Calcd. for C\(_{13}\)H\(_{12}\)N\(_2\)O\(_2\): C, 68.41; H, 5.30; N, 12.27. Found: C, 68.55; H, 5.33; N, 12.24.

4-(5-Thiophen-2-yl-4,5-dihydro-1H-pyrazol-3-yl)-phenol (2d)

IR (cm\(^{-1}\)): N-H str (3452), C-H Ar (3089), C=N str (1532), C-H deform (1411). Anal. Calcd. for C\(_{13}\)H\(_{12}\)N\(_2\)OS: C, 63.91; H, 4.95; N, 11.47. Found: C, 63.79; H, 5.03; N, 11.52.

3,5-Bis-(4-chloro-phenyl)-4,5-dihydro-1H-pyrazole (2f)

IR (cm\(^{-1}\)): N-H str (3277), C-H Ar (2991), C=N str (1540), C-H deform (1387). Anal. Calcd. for C\(_{15}\)H\(_{12}\)Cl\(_2\)N\(_2\): C, 61.87; H, 4.15; N, 9.62. Found: C, 61.70; H, 4.56; N, 9.41.

3-(4-Chloro-phenyl)-5-furan-2-yl-4,5-dihydro-1H-pyrazole (2g)

IR (cm\(^{-1}\)): N-H str (3315), C-H Ar (3100), C=N str (1586), C-H deform (1397). Anal. Calcd. for C\(_{13}\)H\(_{11}\)ClN\(_2\)O: C, 63.29; H, 4.49; N, 11.36. Found: C, 63.13; H, 4.52; N, 11.44.

3-(4-Chloro-phenyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazole (2h)

IR (cm\(^{-1}\)): N-H str (3382), C-H Ar (3056), C=N str (1625), C-H deform (1419). Anal. Calcd. for C\(_{17}\)H\(_{14}\)N\(_2\)O: C, 77.84; H, 5.38; N, 10.68. Found: C, 77.72; H, 5.14; N, 10.54.

5-(3,4-Dimethoxy-phenyl)-3-naphthalen-1-yl-4,5-dihydro-1H-pyrazole (2i)

IR (cm\(^{-1}\)): N-H str (3345), C-H Ar (3056), C=N str (1625), C-H deform (1419). Anal. Calcd. for C\(_{21}\)H\(_{20}\)N\(_2\)O\(_2\): C, 75.88; H, 6.06; N, 8.43. Found: C, 75.70; H, 5.91; N, 8.40.

5-(4-Chloro-phenyl)-3-naphthalen-1-yl-4,5-dihydro-1H-pyrazole (2j)

IR (cm\(^{-1}\)): N-H str (3326), C-H Ar (2994), C=N str (1635), C-H deform (1421). Anal. Calcd. for C\(_{19}\)H\(_{15}\)ClN\(_2\): C, 74.38; H, 4.93; N, 9.13. Found: C, 74.40; H, 4.96; N, 8.99.

5-Furan-2-yl-3-naphthalen-1-yl-4,5-dihydro-1H-pyrazole (2k)

IR (cm\(^{-1}\)): N-H str (3281), C-H Ar (2958), C=N str (1623), C-H deform (1437). Anal. Calcd. for C\(_{17}\)H\(_{14}\)N\(_2\)O: C, 77.84; H, 5.38; N, 10.68. Found: C, 77.72; H, 5.14; N, 10.54.

3-Naphthalen-1-yl-5-thiophen-2-yl-4,5-dihydro-1H-pyrazole (2l)

IR (cm\(^{-1}\)): N-H str (3292), C-H Ar (3047), C=N str (1560), C-H deform (1353). Anal. Calcd. for C\(_{17}\)H\(_{14}\)N\(_2\)S: C, 73.35; H, 5.07; N, 10.06. Found: C, 73.10; H, 5.02; N, 10.37.
2-[5-(3,4-Dimethoxy-phenyl)-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (2q)

IR (cm\(^{-1}\)): N-H str (3321), C-H Ar (2955), C=N str (1647), C-H deform (1473). Anal. Calcd. for C\(_{16}H_{17}N_3O_2\): C, 67.83; H, 6.05; N, 14.83. Found: C, 68.05; H, 5.95; N, 14.11.

2-[5-(4-Chloro-phenyl)-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (2r)

IR (cm\(^{-1}\)): N-H str (3400), C-H Ar (3119), C=N str (1635), C-H deform (1374). Anal. Calcd. for C\(_{14}H_{12}ClN_3\): C, 65.25; H, 4.69; N, 16.30. Found: C, 65.02; H, 4.98; N, 16.42.

2-(5-Thiophen-2-yl-4,5-dihydro-1H-pyrazol-3-yl)-pyridine (2t)

IR (cm\(^{-1}\)): N-H str (3430), C-H Ar (3136), C=N str (1615), C-H deform (1381). Anal. Calcd. for C\(_{12}H_{11}N_3S\): C, 62.86; H, 4.84; N, 18.33. Found: C, 62.90; H, 4.98; N, 18.42.

The final step derivatives were also characterized by various physicochemical and spectral methods and the values were found to be in accordance with the proposed derivatives. The physicochemical characterization details are provided in Table 3.2. However, spectral analysis data are as follows-

4-[5-(3,4-Dimethoxy-phenyl)-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (3a)

IR (cm\(^{-1}\)): N-H str (3220), C-H Ar (2861), C=N str (1637), C-H deform (1516), sym., asym S(=O)\(_2\) str (1160, 1350). Anal. Calcd. for C\(_{23}H_{21}N_3O_7S\): C, 57.14; H, 4.38; N, 8.69. Found: C, 56.83; H, 4.77; N, 9.01.

4-[5-(4-Chloro-phenyl)-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (3b)

IR (cm\(^{-1}\)): N-H str (3440), C-H Ar (2861), C=N str (1516), C-H deform (1437), sym., asym S(=O)\(_2\) str (1169, 1351). Anal. Calcd. for C\(_{21}H_{16}ClN_3O_5S\): C, 56.38; H, 3.61; N, 6.26. Found: C, 55.88; H, 4.10; N, 6.02.

4-[5-Furan-2-yl-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (3c)

IR (cm\(^{-1}\)): N-H str (3486), C-H Ar (3185), C=N str (1641), C-H deform (1525), sym., asym S(=O)\(_2\) str (1165, 1347). Anal. Calcd. for C\(_{19}H_{15}N_3O_6S\): C, 55.20; H, 3.66; N, 10.16. Found: C, 54.92; H, 4.17; N, 9.85.
4-[1-(4-Nitro-benzenesulfonyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazol-3-yl]-phenol (3d)

IR (cm⁻¹): N-H str (3268), C-H Ar (2876), C=N str (1566), C-H deform (1434), sym., asym S(=O)₂ str (1212, 1352). ¹H NMR (DMSO, δ ppm): 2.48-2.51 (d, J_ab: 17.12 Hz, J_ax: 3.41 Hz, 1H, H_a), 3.57-3.93 (dd, J_ab: 4.01 Hz, J_bx: 16.23 Hz, 1H, H_b), 5.47 (s, 1H, Ar-OH), 6.44-6.51 (dd, J_ax: 3.28 Hz, J_bx: 17.46 Hz, 1H, H_x), 6.86-7.13 (m, 4H, Ar), 7.35-7.85 (m, 3H, Ar), 8.77-8.20 (m, 4H, Ar). ¹³C NMR (DMSO, ppm): 38.43 (CH₂ pyrazoline), 41.59 (CH pyrazoline), 115.8 (4CH benzene), 126.8-128.2 (2CH benzene), 130.2 (2CH benzene), 137.5 (C furan), 161.3 (C pyrazoline). Anal. Calcd. for C₁₉H₁₅N₃O₅S₂: C, 53.14; H, 3.52; N, 9.78. Found: C, 52.70; H, 3.46; N, 10.05.

3,5-Bis-(4-chloro-phenyl)-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazole (3f)

IR (cm⁻¹): N-H str (3356), C-H Ar (2992), C=N str (1522), C-H deform (1457), sym., asym S(=O)₂ str (1275, 1353). Anal. Calcd. for C₂₁H₁₅Cl₂N₃O₄S: C, 52.95; H, 3.17; N, 8.82. Found: C, 53.10; H, 3.55; N, 9.33.

3-(4-Chloro-phenyl)-5-furan-2-yl-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazole (3g)

IR (cm⁻¹): N-H str (3315), C-H Ar (3101), C=N str (1588), C-H deform (1399), sym., asym S(=O)₂ str (1166, 1345). Anal. Calcd. for C₁₉H₁₄ClN₃O₄S: C, 52.84; H, 3.27; N, 9.73. Found: C, 53.20; H, 3.50; N, 9.15.

3-(4-Chloro-phenyl)-1-(4-nitro-benzenesulfonyl)-3-thiophen-2-yl-4,5-dihydro-1H-pyrazole (3h)


5-(3,4-Dimethoxy-phenyl)-3-naphthalen-1-yl-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazole (3i)
IR (cm\(^{-1}\)): N-H str (3345), C-H Ar (2916), C=N str (1594), C-H deform (1456), sym., asym S(=O)\(_2\) str (1167, 1350). Anal. Calcd. for C\(_{27}\)H\(_{23}\)N\(_3\)O\(_6\)S: C, 62.66; H, 4.48; N, 8.12. Found: C, 63.20; H, 4.89; N, 7.80.

5-(4-Chloro-phenyl)-3-naphthalen-1-yl-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazole (3f)

IR (cm\(^{-1}\)): N-H str (3260), C-H Ar (2992), C=N str (1565), C-H deform (1514), sym., asym S(=O)\(_2\) str (1169, 1345). Anal. Calcd. for C\(_{25}\)H\(_{18}\)ClN\(_3\)O\(_4\)S: C, 61.04; H, 3.69; N, 8.54. Found: C, 60.70; H, 4.00; N, 8.80.

5-Furan-2-yl-3-naphthalen-1-yl-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazole (3k)

IR (cm\(^{-1}\)): N-H str (3312), C-H Ar (3109), C=N str (1598), C-H deform (1523), sym., asym S(=O)\(_2\) str (1170, 1351). Anal. Calcd. for C\(_{23}\)H\(_{17}\)N\(_3\)O\(_4\)S: C, 61.74; H, 3.83; N, 9.39. Found: C, 62.10; H, 4.18; N, 9.73.

3-Naphthalen-1-yl-1-(4-nitro-benzenesulfonyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazole (3l)

IR (cm\(^{-1}\)): N-H str (3292), C-H Ar (3047), C=N str (1580), C-H deform (1513), sym., asym S(=O)\(_2\) str (1176, 1352). \(^1\)H NMR (DMSO, \(\delta\) ppm): 2.48-2.51 (dd, \(J_{ab}: 17.12\) Hz, \(J_{ax}: 3.41\) Hz, 1H, H\(_a\)), 3.50-3.56 (dd, \(J_{ab}: 4.01\) Hz, \(J_{bx}: 16.23\) Hz, 1H, H\(_b\)), 3.84-3.93 (dd, \(J_{ax}: 3.28\) Hz, \(J_{bx}: 17.46\) Hz, 1H, H\(_x\)). 13C NMR (DMSO, ppm): 38.6 (CH\(_2\) pyrazoline), 40.7 (C pyrazoline), 114.7-119.0 (4CH benzene), 127.5-129.6 (7CH naphthalene), 134.3-137.0 (3CH thiophene), 152.5 (C thiophene), 156.7 (C benzene), 157.6 (C pyrazoline). Anal. Calcd. for C\(_{23}\)H\(_{17}\)N\(_3\)O\(_4\)S\(_2\): C, 59.60; H, 3.70; N, 9.07. Found: C, 60.27; H, 3.25; N, 9.83.

2-[5-(3,4-Dimethoxy-phenyl)-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (3q)

IR (cm\(^{-1}\)): N-H str (3406), C-H Ar (3093), C=N str (1608), C-H deform (1513), sym., asym S(=O)\(_2\) str (1174, 1344). Anal. Calcd. for C\(_{22}\)H\(_{20}\)N\(_4\)O\(_6\)S: C, 56.40; H, 4.30; N, 11.96. Found: C, 55.79; H, 4.72; N, 12.33.
2-[5-(4-Chloro-phenyl)-1-(4-nitro-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (3r)


2-[1-(4-Nitro-benzenesulfonyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (3t)


4-[5-(3,4-Dimethoxy-phenyl)-1-(4-methoxy-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (4a)


4-[5-(4-Chloro-phenyl)-1-(4-methoxy-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (4b)


4-[5-Furan-2-yl-1-(4-methoxy-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (4c)

IR (cm⁻¹): N-H str (322), C-H Ar (3114), C=N str (1602), C-H deform (1403), sym., asym S(=O)₂ str (1156, 1351). ¹H NMR (DMSO, δ ppm): 1.74-1.83 (dd, Jₘₙ: 17.12 Hz, Jₘₐₚ: 3.41 Hz, 1H, Hₐ), 2.40-2.52 (dd, Jₘₜₙ: 4.01 Hz, Jₙₘₚₚ: 16.23 Hz, 1H, Hₐ), 3.23-3.32 (dd, Jₘₐₚ: 3.28 Hz, Jₙₚₙₚ: 17.46 Hz, 1H, Hₐ), 3.52-3.61 (m, 3H), 5.18 (s, 1H, Ar-OH), 7.25-7.94 (m, 8H, Ar), 8.02-8.43 (m, 4H, Ar). ¹³C NMR (DMSO, ppm): 21.06 (CH₃), 38.6 (CH₂ pyrazoline), 40.06 (C pyrazoline), 124.7-134.5 (12CH benzene), 141.4 (2C benzene),
147.4 (C pyrazoline). Anal. Calcd. for C₂₀H₁₈N₂O₅S: C, 60.29; H, 4.55; N, 7.03. Found: C, 59.95; H, 4.82; N, 6.66.

4-[1-(4-Methoxy-benzenesulfonfyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazol-3-yl]-phenol (4d)

IR (cm⁻¹): N-H str (3392), C-H Ar (3089), C=N str (1609), C-H deform (1463), sym., asym S(=O)₂ str (1160, 1353). Anal. Calcd. for C₂₀H₁₈N₂O₄S: C, 57.95; H, 4.38; N, 6.76. Found: C, 58.27; H, 4.73; N, 6.44.

3,5-Bis-(4-chloro-phenyl)-1-(4-methoxy-benzenesulfonfyl)-4,5-dihydro-1H-pyrazole (4f)


3-(4-Chloro-phenyl)-5-furan-2-yl-1-(4-methoxy-benzenesulfonfyl)-4,5-dihydro-1H-pyrazole (4g)

IR (cm⁻¹): N-H str (3383), C-H Ar (3000), C=N str (1591), C-H deform (1494), sym., asym S(=O)₂ str (1115, 1352). Anal. Calcd. for C₂₀H₁₇ClN₂O₄S: C, 57.62; H, 4.11; N, 6.72. Found: C, 57.05; H, 4.54; N, 6.28.

3-(4-Chloro-phenyl)-1-(4-methoxy-benzenesulfonfyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazole (4h)


5-(3,4-Dimethoxy-phenyl)-1-(4-methoxy-benzenesulfonfyl)-3-naphthalen-1-yl-4,5-dihydro-1H-pyrazole (4i)

IR (cm⁻¹): N-H str (3505), C-H Ar (2835), C=N str (1592), C-H deform (1456), sym., asym S(=O)₂ str (1153, 1355). Anal. Calcd. for C₂₈H₂₆N₂O₅S: C, 66.91; H, 5.21; N, 5.57. Found: C, 67.01; H, 5.29; N, 6.11.

5-(4-Chloro-phenyl)-1-(4-methoxy-benzenesulfonfyl)-3-naphthalen-1-yl-4,5-dihydro-1H-pyrazole (4j)

5-Furan-2-yl-1-(4-methoxy-benzenesulfonyl)-3-naphthalen-1-yl-4,5-dihydro-1H-pyrazole (4k)


1-(4-Methoxy-benzenesulfonyl)-3-naphthalen-1-yl-5-thiophen-2-yl-4,5-dihydro-1H-pyrazole (4l)

IR (cm⁻¹): N-H str (3224), C-H Ar (2940), C=N str (1513), C-H deform (1463), sym., asym S(=O)₂ str (1144, 1359). Anal. Calcd. for C₂₄H₂₀N₂O₃S₂: C, 64.26; H, 4.49; N, 6.25. Found: C, 63.90; H, 4.26; N, 6.01.

2-[5-(3,4-Dimethoxy-phenyl)-1-(4-methoxy-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (4q)

IR (cm⁻¹): N-H str (3224), C-H Ar (2940), C=N str (1513), C-H deform (1463), sym., asym S(=O)₂ str (1144, 1359). Anal. Calcd. for C₂₃H₂₃N₂O₅S: C, 60.91; H, 5.11; N, 9.27. Found: C, 61.22; H, 5.40; N, 9.55.

2-[5-(4-Chloro-phenyl)-1-(4-methoxy-benzenesulfonyl)-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (4r)

2-[1-(4-Methoxy-benzenesulfonyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (4t)

IR (cm⁻¹): N-H str (3430), C-H Ar (3069), C≡N str (1583), C-H deform (1468), sym., asym S(=O)₂ str (1150, 1372). Anal. Calcd. for C₁₉H₁₇N₃O₃S: C, 57.12; H, 4.29; N, 10.52. Found: C, 58.23; H, 4.45; N, 9.80.

4-[1-(4-Chloro-benzenesulfonyl)-5-(3,4-dimethoxy-phenyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (5a)


4-[1-(4-Chloro-benzenesulfonyl)-5-(4-chloro-phenyl)-4,5-dihydro-1H-pyrazol-3-yl]-phenol (5b)

IR (cm⁻¹): N-H str (3359), C-H Ar (3238), C≡N str (1606), C-H deform (1516), sym., asym S(=O)₂ str (1165, 1351). ¹H NMR (DMSO, δ ppm): 1.98-1.99 (dd, J_ab: 17.12 Hz, J_ax: 3.41 Hz, 1H, H_a), 3.40-3.48 (dd, J_ab: 4.01 Hz, J_bx: 16.23 Hz, 1H, H_b), 3.70-3.80 (dd, J_ax: 3.28 Hz, J_bx: 17.46 Hz, 1H, H_a), 5.39-5.45 (s, 1H, Ar-OH), 6.88-7.89 (m, 12H, Ar). ¹³C NMR (DMSO, ppm): 38.6 (CH₂ pyrazoline), 43.2 (C pyrazoline), 115.6-140.3 (12CH benzene), 158.3-160.1 (3C benzene), 161.2 (C pyrazoline). Anal. Calcd. for C₂₃H₁₆Cl₂N₂O₅S: C, 56.38; H, 3.61; N, 6.26. Found: C, 57.03; H, 4.15; N, 6.44.

4-[1-(4-Chloro-benzenesulfonyl)-5-furan-2-yl-4,5-dihydro-1H-pyrazol-3-yl]-phenol (5c)

IR (cm⁻¹): N-H str (3472), C-H Ar (3239), C≡N str (1602), C-H deform (1434), sym., asym S(=O)₂ str (1159, 1348). Anal. Calcd. for C₁₉H₁₅ClN₂O₃S: C, 56.65; H, 3.75; N, 6.95. Found: C, 57.65; H, 4.00; N, 6.73.

4-[1-(4-Chloro-benzenesulfonyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazol-3-yl]-phenol (5d)
Chapter 3

Synthesis and Characterization

IR (cm$^{-1}$): N-H str (3270), C-H Ar (3010), C=N str (1615), C-H deform (1463), sym., asym S(=O)$_2$ str (1189, 1382). Anal. Calcd. for C$_{19}$H$_{15}$ClN$_2$O$_3$S$_2$: C, 54.47; H, 3.61; N, 6.69. Found: C, 55.13; H, 3.42; N, 6.57.

1-(4-Chloro-benzenesulfonyl)-3,5-bis-(4-chloro-phenyl)-4,5-dihydro-1H-pyrazole (5f)

IR (cm$^{-1}$): N-H str (3315), C-H Ar (3100), C=N str (1616), C-H deform (1465), sym., asym S(=O)$_2$ str (1164, 1389). Anal. Calcd. for C$_{21}$H$_{15}$ClN$_2$O$_2$: C, 54.15; H, 3.25; N, 6.01. Found: C, 54.93; H, 3.82; N, 5.60.

1-(4-Chloro-benzenesulfonyl)-3-(4-chloro-phenyl)-5-furan-2-yl-4,5-dihydro-1H-pyrazole (5g)

IR (cm$^{-1}$): N-H str (3345), C-H Ar (3056), C=N str (1577), C-H deform (1462), sym., asym S(=O)$_2$ str (1165, 1393). Anal. Calcd. for C$_{27}$H$_{23}$ClN$_2$O$_4$: C, 63.96; H, 4.57; N, 5.53. Found: C, 64.07; H, 4.61; N, 5.20.

1-(4-Chloro-benzenesulfonyl)-5-(3,4-dimethoxy-phenyl)-3-naphthalen-1-yl-4,5-dihydro-1H-pyrazole (5i)

IR (cm$^{-1}$): N-H str (3266), C-H Ar (3011), C=N str (1570), C-H deform (1495), sym., asym S(=O)$_2$ str (1166, 1362). Anal. Calcd. for C$_{25}$H$_{18}$Cl$_2$N$_2$O$_2$: C, 62.37; H, 3.77; N, 5.82. Found: C, 63.19; H, 3.80; N, 5.80.
1-(4-Chloro-benzenesulfonyl)-5-furan-2-yl-3-naphthalen-1-yl-4,5-dihydro-1H-pyrazole (5k)

IR (cm\(^{-1}\)): N-H str (3290), C-H Ar (3050), C=N str (1635), C-H deform (1469), sym., asym S(=O)\(_2\) str (1166, 1355). \(^1\)H NMR (DMSO, δ ppm): 2.48-2.51 (dd, J\(_{ab}\): 17.12 Hz, J\(_{ax}\): 3.41 Hz, 1H, H\(_a\)), 3.04-3.89 (dd, J\(_{ab}\): 4.01 Hz, J\(_{bx}\): 16.23 Hz, 1H, H\(_b\)), 4.88-5.21 (dd, J\(_{ax}\): 3.28 Hz, J\(_{bx}\): 17.46 Hz, 1H, H\(_x\)), 6.80-6.93 (d, 2H, Ar), 7.43-7.54 (m, 4H, Ar), 7.66-7.85 (m, 7H, Ar). \(^13\)C NMR (DMSO, ppm): 50.336 (CH\(_2\)pyrazoline), 77.449 (C\(_H\)pyrazoline), 124.4-126.9 (2CH furan), 129.0-131.3 (7CH naphthalene), 133.7-138.2 (4CH benzene), 159.4 (C pyrazoline). Anal. Calcd. for C\(_{23}\)H\(_{17}\)ClN\(_2\)O\(_3\)S: C, 63.23; H, 3.92; N, 6.41. Found: C, 62.96; H, 4.27; N, 6.40.

1-(4-Chloro-benzenesulfonyl)-3-naphthalen-1-yl-5-thiophen-2-yl-4,5-dihydro-1H-pyrazole (5l)

IR (cm\(^{-1}\)): N-H str (3292), C-H Ar (3047), C=N str (1581), C-H deform (1509), sym., asym S(=O)\(_2\) str (1177, 1322). Anal. Calcd. for C\(_{23}\)H\(_{17}\)ClN\(_2\)O\(_2\)S\(_2\): C, 60.98; H, 3.78; N, 6.18. Found: C, 61.16; H, 3.56; N, 6.24.

2-[1-(4-Chloro-benzenesulfonyl)-5-(3,4-dimethoxy-phenyl)-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (5q)

IR (cm\(^{-1}\)): N-H str (3321), C-H Ar (3084), C=N str (1624), C-H deform (1466), sym., asym S(=O)\(_2\) str (1170, 1337). Anal. Calcd. for C\(_{23}\)H\(_{20}\)ClN\(_3\)O\(_4\)S: C, 57.70; H, 4.40; N, 9.18. Found: C, 57.24; H, 4.55; N, 9.34.

2-[1-(4-Chloro-benzenesulfonyl)-5-(4-chloro-phenyl)-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (5r)

IR (cm\(^{-1}\)): N-H str (3421), C-H Ar (3091), C=N str (1616), C-H deform (1471), sym., asym S(=O)\(_2\) str (1170, 1383). Anal. Calcd. for C\(_{23}\)H\(_{15}\)ClN\(_3\)O\(_2\)S: C, 55.56; H, 3.50; N, 9.72. Found: C, 56.70; H, 4.05; N, 9.38.

2-[1-(4-Chloro-benzenesulfonyl)-5-thiophen-2-yl-4,5-dihydro-1H-pyrazol-3-yl]-pyridine (5t)
IR (cm\(^{-1}\)): N-H str (3387), C-H Ar (3083), C=N str (1612), C-H deform (1468), sym., asym S(=O)\(_2\) str (1167, 1389). Anal. Calcd. for C\(_{18}\)H\(_{14}\)ClN\(_3\)O\(_2\)S\(_2\): C, 53.53; H, 3.49; N, 10.40. Found: C, 52.14; H, 3.73; N, 9.80.
Table 3.1: Characterization data of first step chalcone derivatives (1a-1t)

<table>
<thead>
<tr>
<th>S. N</th>
<th>Comp.</th>
<th>Color and State</th>
<th>% Yield</th>
<th>Solubility</th>
<th>R_f Value</th>
<th>Melting Range (°C)</th>
<th>MS (m/z)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>1a</td>
<td>Yellow solid</td>
<td>81</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.76</td>
<td>178-180</td>
<td>259.0 (M^+)</td>
</tr>
<tr>
<td>2.</td>
<td>1b</td>
<td>Light yellow solid</td>
<td>68</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.93</td>
<td>170-174</td>
<td>285.15 (M^+)</td>
</tr>
<tr>
<td>3.</td>
<td>1c</td>
<td>Yellow solid</td>
<td>63</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.68</td>
<td>140-142</td>
<td>215.09 (M^+)</td>
</tr>
<tr>
<td>4.</td>
<td>1d</td>
<td>Yellowish brown solid</td>
<td>44</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.67</td>
<td>156-158</td>
<td>285.15 (M^+)</td>
</tr>
<tr>
<td>5.</td>
<td>1e</td>
<td>Light yellow solid</td>
<td>37</td>
<td>Hot methanol, acetone</td>
<td>0.86</td>
<td>94-96</td>
<td>303.11 (M^+)</td>
</tr>
<tr>
<td>6.</td>
<td>1f</td>
<td>Colorless solid</td>
<td>85</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.87</td>
<td>128-130</td>
<td>262.37 (M^+)</td>
</tr>
<tr>
<td>7.</td>
<td>1g</td>
<td>Yellowish brown solid</td>
<td>70</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.78</td>
<td>62-64</td>
<td>288.32 (M^+)</td>
</tr>
<tr>
<td>8.</td>
<td>1h</td>
<td>Yellowish brown shiny solid</td>
<td>55</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.90</td>
<td>96-98</td>
<td>249.04 (M^+)</td>
</tr>
<tr>
<td>9.</td>
<td>1i</td>
<td>Light yellow shiny solid</td>
<td>81</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.75</td>
<td>78-80</td>
<td>319.18 (M^+)</td>
</tr>
<tr>
<td>10.</td>
<td>1j</td>
<td>Shiny yellow solid</td>
<td>85</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.82</td>
<td>74-76</td>
<td>293.11 (M^+)</td>
</tr>
<tr>
<td>11.</td>
<td>1k</td>
<td>Brown color semisolid</td>
<td>90</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.80</td>
<td>---</td>
<td>249.20 (M^+)</td>
</tr>
<tr>
<td>12.</td>
<td>1l</td>
<td>Brownish yellow solid</td>
<td>79</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.78</td>
<td>72-74</td>
<td>265.20 (M^+)</td>
</tr>
<tr>
<td>13.</td>
<td>1m</td>
<td>Cream yellow solid</td>
<td>62</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.88</td>
<td>102-104</td>
<td>259.13 (M^+)</td>
</tr>
<tr>
<td>14.</td>
<td>1n</td>
<td>Cream color solid</td>
<td>46</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.86</td>
<td>120-122</td>
<td>283.27 (M^+)</td>
</tr>
<tr>
<td>15.</td>
<td>1o</td>
<td>Dark yellowish brown solid</td>
<td>53</td>
<td>Hot methanol</td>
<td>0.82</td>
<td>74-76</td>
<td>249.09 (M^+)</td>
</tr>
<tr>
<td>16.</td>
<td>1p</td>
<td>Dirty brown solid</td>
<td>26</td>
<td>Acetone</td>
<td>0.60</td>
<td>60-62</td>
<td>205.05 (M^+)</td>
</tr>
<tr>
<td></td>
<td>1q</td>
<td>Light yellow solid</td>
<td>67</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.88</td>
<td>96-98</td>
<td>270.15 (M^+)</td>
</tr>
<tr>
<td>---</td>
<td>----</td>
<td>---------------------</td>
<td>-----</td>
<td>----------------------------------</td>
<td>------</td>
<td>------</td>
<td>---------------</td>
</tr>
<tr>
<td>18</td>
<td>1r</td>
<td>Cream color solid</td>
<td>56</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.73</td>
<td>60-62</td>
<td>244.07 (M^+)</td>
</tr>
<tr>
<td>19</td>
<td>1s</td>
<td>Dirty greenish brown solid</td>
<td>34</td>
<td>Chloroform, acetone</td>
<td>0.84</td>
<td>56-58</td>
<td>200.09 (M^+)</td>
</tr>
<tr>
<td>20</td>
<td>1t</td>
<td>Greenish brown solid</td>
<td>71</td>
<td>Hot methanol, chloroform, acetone</td>
<td>0.85</td>
<td>64-66</td>
<td>216-07 (M^+)</td>
</tr>
</tbody>
</table>

*Rf Values were calculated taking solvent system, Chloroform (9.5) : Methanol (0.5).*
Table 3.2: Comparative study of physicochemical properties of synthesized 3,5-disubstituted -2-pyrazoline derivatives (2a-2t) and 1,3,5-trisubstituted -2-pyrazoline derivatives (3a-3t, 4a-4t, and 5a-5t)

<table>
<thead>
<tr>
<th>Comps.</th>
<th>Structure</th>
<th>Molecular Formula</th>
<th>Color and State</th>
<th>Solubility</th>
<th>Rf Value</th>
<th>Melting Range (°C)</th>
<th>Conventional Synthesis (Refluxing)</th>
<th>Microwave Assisted Synthesis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Reaction Time (h) % Yield</td>
<td>Microwave Power (W)</td>
</tr>
<tr>
<td>2a</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{17}H_{18}N_{2}O_{3}</td>
<td>Colorless crystalline solid</td>
<td>Hot methanol, chloroform, acetone, DMSO</td>
<td>0.41</td>
<td>178-180</td>
<td>4.5</td>
<td>54.7</td>
</tr>
<tr>
<td>2b</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{15}H_{13}ClN_{2}O</td>
<td>Colorless crystalline solid</td>
<td>Hot methanol, chloroform, acetone, DMSO</td>
<td>0.38</td>
<td>210-212</td>
<td>4.5</td>
<td>67.2</td>
</tr>
<tr>
<td>2c</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{13}H_{12}N_{2}O_{2}</td>
<td>Cream colored crystalline solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.48</td>
<td>148-150</td>
<td>5.0</td>
<td>60.5</td>
</tr>
<tr>
<td>2d</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{13}H_{12}N_{2}OS</td>
<td>Colorless crystalline solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.31</td>
<td>88-91</td>
<td>6.0</td>
<td>78.8</td>
</tr>
<tr>
<td>2f</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{15}H_{12}Cl_{2}N_{2}</td>
<td>Colorless crystalline solid</td>
<td>Hot methanol, Chloroform, acetone, DMSO</td>
<td>0.83</td>
<td>118-120</td>
<td>8.0</td>
<td>68.9</td>
</tr>
<tr>
<td>2g</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{13}H_{11}ClN_{2}O</td>
<td>Brownish black colored amorphous solid</td>
<td>Hot methanol, Chloroform, acetone, DMSO</td>
<td>0.96</td>
<td>80-82</td>
<td>5.0</td>
<td>57.4</td>
</tr>
<tr>
<td>2h</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{13}H_{11}ClN_{2}S</td>
<td>Brown colored amorphous solid</td>
<td>Hot methanol, chloroform, acetone, DMSO</td>
<td>0.88</td>
<td>90-92</td>
<td>4.5</td>
<td>59.6</td>
</tr>
<tr>
<td>2i</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{21}H_{20}N_{2}O_{2}</td>
<td>Cream yellow colored crystalline solid</td>
<td>Hot methanol, Chloroform, acetone, DMSO</td>
<td>0.91</td>
<td>112-114</td>
<td>6.0</td>
<td>63.09</td>
</tr>
<tr>
<td>2j</td>
<td><img src="image" alt="Structure" /></td>
<td>C_{19}H_{16}ClN_{2}</td>
<td>Cream White colored crystalline solid</td>
<td>Methanol, chloroform, acetone, DMSO</td>
<td>0.50</td>
<td>118-120</td>
<td>5.0</td>
<td>86.7</td>
</tr>
<tr>
<td></td>
<td>Chemical Structure</td>
<td>Chemical Formula</td>
<td>Physical Properties</td>
<td>Melting Point</td>
<td>Boiling Point</td>
<td>CAS Registry Number</td>
<td>Molecular Weight</td>
<td></td>
</tr>
<tr>
<td>---</td>
<td>-------------------</td>
<td>------------------</td>
<td>---------------------</td>
<td>---------------</td>
<td>--------------</td>
<td>---------------------</td>
<td>------------------</td>
<td></td>
</tr>
<tr>
<td>2k</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C$<em>{17}$H$</em>{13}$N$_2$O</td>
<td>Light brown colored crystalline solid</td>
<td>0.90</td>
<td>88-90</td>
<td>4.5</td>
<td>71.3</td>
<td>240-350</td>
</tr>
<tr>
<td>2l</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C$<em>{17}$H$</em>{13}$N$_2$S</td>
<td>Cream yellow colored crystalline solid</td>
<td>0.97</td>
<td>98-100</td>
<td>4.5</td>
<td>73.5</td>
<td>280-350</td>
</tr>
<tr>
<td>2q</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C$<em>{16}$H$</em>{13}$N$_3$O$_2$</td>
<td>Brown colored crystalline solid</td>
<td>0.65</td>
<td>110-113</td>
<td>3.0</td>
<td>93.8</td>
<td>280</td>
</tr>
<tr>
<td>2r</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C$<em>{14}$H$</em>{12}$ClN$_3$</td>
<td>Light orange colored crystalline solid</td>
<td>0.72</td>
<td>170-172</td>
<td>3.0</td>
<td>48.2</td>
<td>280-350</td>
</tr>
<tr>
<td>2t</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C$<em>{12}$H$</em>{11}$N$_3$S</td>
<td>Brown colored crystalline solid</td>
<td>0.54</td>
<td>82-84</td>
<td>4.0</td>
<td>51.6</td>
<td>280-350</td>
</tr>
<tr>
<td></td>
<td>Chemical Structure</td>
<td>Chemical Formula</td>
<td>Color and State</td>
<td>Solvents</td>
<td>Log P</td>
<td>Melting point (°C)</td>
<td>Boiling point (°C)</td>
<td>Tp (°C)</td>
</tr>
<tr>
<td>---</td>
<td>--------------------</td>
<td>------------------</td>
<td>-----------------</td>
<td>----------</td>
<td>-------</td>
<td>-------------------</td>
<td>-------------------</td>
<td>---------</td>
</tr>
<tr>
<td><strong>3a</strong></td>
<td><img src="image" alt="3a Structure" /></td>
<td>C$<em>{23}$H$</em>{21}$N$_{3}$O$_7$S</td>
<td>Brown color solid</td>
<td>Methanol, chloroform, acetone, DMSO</td>
<td>0.74</td>
<td>116-118</td>
<td></td>
<td>1.0</td>
</tr>
<tr>
<td><strong>3b</strong></td>
<td><img src="image" alt="3b Structure" /></td>
<td>C$<em>{21}$H$</em>{18}$ClN$_{3}$O$_5$S</td>
<td>Yellow color crystalline solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.66</td>
<td>168-170</td>
<td></td>
<td>1.0</td>
</tr>
<tr>
<td><strong>3c</strong></td>
<td><img src="image" alt="3c Structure" /></td>
<td>C$<em>{19}$H$</em>{15}$N$_{3}$O$_6$S</td>
<td>Black color solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.78</td>
<td>182-184</td>
<td></td>
<td>2.0</td>
</tr>
<tr>
<td><strong>3d</strong></td>
<td><img src="image" alt="3d Structure" /></td>
<td>C$<em>{19}$H$</em>{15}$N$_{3}$O$_5$S$_2$</td>
<td>Cream yellow solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.70</td>
<td>140-142</td>
<td></td>
<td>2.0</td>
</tr>
<tr>
<td><strong>3f</strong></td>
<td><img src="image" alt="3f Structure" /></td>
<td>C$<em>{21}$H$</em>{18}$ClN$_{3}$O$_5$S</td>
<td>Yellowish brown solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.66</td>
<td>150-152</td>
<td></td>
<td>2.0</td>
</tr>
</tbody>
</table>
### 3g

| 3g | ![Chemical Structure](image1) | C_{19}H_{14}ClN_{3}O_{5}S | Black color solid | DMSO | 0.58 | 196-198 | 3.0 | 61.2 | 240-350 | 210 | 81.1 |

### 3h

| 3h | ![Chemical Structure](image2) | C_{19}H_{14}ClN_{3}O_{5}S_{2} | Yellowish grey color solid | Methanol, acetone, DMSO | 0.69 | 152-154 | 3.0 | 38.5 | 210-350 | 185 | 77.6 |

### 3i

| 3i | ![Chemical Structure](image3) | C_{27}H_{23}N_{3}O_{6}S | Cream yellow color solid | Methanol, acetone, DMSO | 0.75 | 168-170 | 1.0 | 23.8 | 280 | 110 | 70.2 |

### 3j

<p>| 3j | <img src="image4" alt="Chemical Structure" /> | C_{25}H_{18}ClN_{3}O_{4}S | Yellowish brown solid | Acetonitrile, DMSO | 0.54 | 140-142 | 1.5 | 78.2 | 280-350 | 180 | 92.5 |
| 3k | <img src="image" alt="Chemical Structure" /> | C\textsubscript{23}H\textsubscript{17}N\textsubscript{2}O\textsubscript{5}S | Black color solid | Acetonitrile, acetone, DMSO | 0.80 | 132-134 | 2.5 | 73.8 | 280-350 | 210 | 95.1 |
| 3l | <img src="image" alt="Chemical Structure" /> | C\textsubscript{23}H\textsubscript{17}N\textsubscript{2}O\textsubscript{5}S\textsubscript{2} | Brown color solid | Methanol, acetone, DMSO | 0.74 | 158-160 | 2.0 | 68.2 | 210-280 | 190 | 83.4 |
| 3q | <img src="image" alt="Chemical Structure" /> | C\textsubscript{22}H\textsubscript{20}N\textsubscript{4}O\textsubscript{6}S | Yellowish brown solid | Methanol, acetone, DMSO | 0.62 | 132-134 | 3.0 | 55.9 | 210-280 | 200 | 63.2 |
| 3r | <img src="image" alt="Chemical Structure" /> | C\textsubscript{20}H\textsubscript{13}ClN\textsubscript{3}O\textsubscript{4}S | Brown color solid | Methanol, acetone, DMSO | 0.60 | 180-182 | 3.0 | 48.3 | 240-350 | 175 | 80.4 |</p>
<table>
<thead>
<tr>
<th></th>
<th>Structure</th>
<th>Molecular Formula</th>
<th>Description</th>
<th>Solvent(s)</th>
<th>Yield</th>
<th>M.p.</th>
<th>Combustion Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>3t</td>
<td><img src="image" alt="3t Structure" /></td>
<td>C_{18}H_{14}N_{2}O_{2}S_{2}</td>
<td>Brown color sticky solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.85</td>
<td>2.5</td>
<td>40.7</td>
</tr>
<tr>
<td>4a</td>
<td><img src="image" alt="4a Structure" /></td>
<td>C_{24}H_{24}N_{2}O_{6}S</td>
<td>Red color solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.77</td>
<td>122-124</td>
<td>3.0</td>
</tr>
<tr>
<td>4b</td>
<td><img src="image" alt="4b Structure" /></td>
<td>C_{22}H_{19}ClN_{2}O_{4}S</td>
<td>Yellow color solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.73</td>
<td>188-190</td>
<td>2.5</td>
</tr>
<tr>
<td>4c</td>
<td><img src="image" alt="4c Structure" /></td>
<td>C_{20}H_{18}N_{2}O_{5}S</td>
<td>Brown color solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.68</td>
<td>174-176</td>
<td>3.0</td>
</tr>
<tr>
<td>4d</td>
<td><img src="image" alt="4d Structure" /></td>
<td>C_{20}H_{18}N_{2}O_{4}S_{2}</td>
<td>Yellowish brown solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.60</td>
<td>150-152</td>
<td>2.0</td>
</tr>
<tr>
<td>4f</td>
<td><img src="image_url" alt="Chemical Structure" /></td>
<td>C_{22}H_{18}Cl_{2}N_{2}O_{5}S</td>
<td>Yellowish color solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.61</td>
<td>144-146</td>
<td>4.0</td>
</tr>
<tr>
<td>4g</td>
<td><img src="image_url" alt="Chemical Structure" /></td>
<td>C_{20}H_{17}ClN_{2}O_{4}S</td>
<td>Black color sticky solid</td>
<td>Acetonitrile, DMSO</td>
<td>0.70</td>
<td>Sticky material</td>
<td>3.5</td>
</tr>
<tr>
<td>4h</td>
<td><img src="image_url" alt="Chemical Structure" /></td>
<td>C_{20}H_{17}ClN_{2}O_{3}S_{2}</td>
<td>Brown color solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.66</td>
<td>192-194</td>
<td>2.0</td>
</tr>
<tr>
<td>4i</td>
<td><img src="image_url" alt="Chemical Structure" /></td>
<td>C_{28}H_{26}N_{2}O_{5}S</td>
<td>Cream color solid</td>
<td>Acetonitrile, DMSO</td>
<td>0.58</td>
<td>178-180</td>
<td>2.5</td>
</tr>
<tr>
<td>4j</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C_{26}H_{21}ClN_{2}O_{3}S</td>
<td>Grey color solid</td>
<td>DMSO</td>
<td>0.60</td>
<td>196-198</td>
<td>3.0</td>
</tr>
<tr>
<td>-----</td>
<td>-----------------------------</td>
<td>-----------------------------</td>
<td>------------------</td>
<td>--------</td>
<td>------</td>
<td>---------</td>
<td>-----</td>
</tr>
<tr>
<td>4k</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C_{24}H_{26}N_{2}O_{4}S</td>
<td>Black color solid</td>
<td>DMSO</td>
<td>0.54</td>
<td>120-122</td>
<td>3.0</td>
</tr>
<tr>
<td>4l</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C_{24}H_{26}N_{2}O_{4}S_{2}</td>
<td>Creamy yellow solid</td>
<td>DMSO</td>
<td>0.58</td>
<td>148-150</td>
<td>2.0</td>
</tr>
<tr>
<td>4q</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C_{23}H_{23}N_{2}O_{5}S</td>
<td>Light brown solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.70</td>
<td>190-192</td>
<td>3.0</td>
</tr>
<tr>
<td></td>
<td>Molecular Structure</td>
<td>Chemical Formula</td>
<td>Physical and Chemical Properties</td>
<td>Melting Point (°C)</td>
<td>Boiling Point (°C)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>---</td>
<td>-------------------</td>
<td>------------------</td>
<td>-------------------------------</td>
<td>-------------------</td>
<td>-------------------</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4r</td>
<td><img src="https://example.com/image1" alt="Image" /></td>
<td>C$<em>{21}$H$</em>{18}$ClN$_3$O$_3$S</td>
<td>Brownish yellow solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.78</td>
<td>200-202</td>
<td>2.5</td>
</tr>
<tr>
<td>4t</td>
<td><img src="https://example.com/image2" alt="Image" /></td>
<td>C$<em>{19}$H$</em>{17}$N$_3$O$_3$S$_2$</td>
<td>Brown color solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.60</td>
<td>164-166</td>
<td>2.0</td>
</tr>
<tr>
<td>5a</td>
<td><img src="https://example.com/image3" alt="Image" /></td>
<td>C$<em>{23}$H$</em>{21}$ClN$_2$O$_3$S</td>
<td>Cream yellow color amorphous solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.67</td>
<td>206-208</td>
<td>2.5</td>
</tr>
<tr>
<td>5b</td>
<td><img src="https://example.com/image4" alt="Image" /></td>
<td>C$<em>{21}$H$</em>{16}$Cl$_2$N$_2$O$_3$S</td>
<td>Colorless crystalline solid</td>
<td>Methanol, DMSO, chloroform, acetone</td>
<td>0.92</td>
<td>198-200</td>
<td>2.0</td>
</tr>
<tr>
<td>5c</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C\textsubscript{19}H\textsubscript{13}ClN\textsubscript{2}O\textsubscript{2}S</td>
<td>Light brown color amorphous solid</td>
<td>Methanol, acetonitrile, chloroform, acetone, DMSO</td>
<td>0.88</td>
<td>162-164</td>
<td>4.5</td>
</tr>
<tr>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>5d</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C\textsubscript{19}H\textsubscript{13}ClN\textsubscript{2}O\textsubscript{3}S\textsubscript{2}</td>
<td>Brown color amorphous solid</td>
<td>Methanol, acetonitrile, acetone, DMSO</td>
<td>0.75</td>
<td>148-150</td>
<td>3.5</td>
</tr>
<tr>
<td>5f</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C\textsubscript{21}H\textsubscript{13}ClN\textsubscript{2}O\textsubscript{2}S</td>
<td>Grey color amorphous solid</td>
<td>Methanol, acetonitrile, chloroform, acetone, DMSO</td>
<td>0.52</td>
<td>185-187</td>
<td>2.0</td>
</tr>
<tr>
<td>5g</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C\textsubscript{19}H\textsubscript{14}ClN\textsubscript{2}O\textsubscript{3}S</td>
<td>Black color amorphous solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.80</td>
<td>120-122</td>
<td>4.0</td>
</tr>
<tr>
<td>5h</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C\textsubscript{19}H\textsubscript{14}ClN\textsubscript{2}O\textsubscript{3}S\textsubscript{2}</td>
<td>Yellowish brown color amorphous solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.66</td>
<td>193-195</td>
<td>2.5</td>
</tr>
<tr>
<td>5i</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td><strong>C_{27}H_{25}ClN_{2}O_{2}S</strong></td>
<td>Orange brown color</td>
<td>Amorphous solid</td>
<td>Methanol, acetone, DMSO</td>
<td>0.92</td>
<td>168-170</td>
</tr>
<tr>
<td>5j</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td><strong>C_{25}H_{18}Cl_{2}N_{2}O_{2}S</strong></td>
<td>Cream color</td>
<td>Amorphous solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.58</td>
<td>178-180</td>
</tr>
<tr>
<td>5k</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td><strong>C_{23}H_{17}ClN_{2}O_{2}S</strong></td>
<td>Black color</td>
<td>Amorphous solid</td>
<td>Acetone, DMSO, acetonitrile</td>
<td>0.77</td>
<td>218-220</td>
</tr>
<tr>
<td>5l</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td><strong>C_{23}H_{17}ClN_{2}O_{2}S_{2}</strong></td>
<td>Black color</td>
<td>Amorphous solid</td>
<td>Acetonitrile, acetone, DMSO</td>
<td>0.64</td>
<td>112-114</td>
</tr>
<tr>
<td>5q</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C_{22}H_{30}ClN_{3}O_{2}S</td>
<td>Brown color amorphous solid</td>
<td>Methanol, acetonitrile, chloroform, acetone, DMSO</td>
<td>0.90</td>
<td>180-182</td>
<td>2.5</td>
</tr>
<tr>
<td>----</td>
<td>----------------------------</td>
<td>---------------------------</td>
<td>-----------------------------</td>
<td>--------------------------------------------------</td>
<td>------</td>
<td>--------</td>
<td>-----</td>
</tr>
<tr>
<td>5r</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C_{20}H_{18}Cl_{2}N_{3}O_{2}S</td>
<td>Yellowish brown color amorphous solid</td>
<td>Methanol, acetonitrile, chloroform, acetone, DMSO</td>
<td>0.78</td>
<td>116-118</td>
<td>2.0</td>
</tr>
<tr>
<td>5t</td>
<td><img src="image" alt="Chemical Structure" /></td>
<td>C_{18}H_{14}ClN_{3}O_{2}S_{2}</td>
<td>Brown color amorphous solid</td>
<td>Methanol, acetonitrile, acetone, DMSO</td>
<td>0.60</td>
<td>138-140</td>
<td>3.0</td>
</tr>
</tbody>
</table>
(a) IR Spectrum of compound 3a

(b) IR Spectrum of compound 3b
(c) IR Spectrum of compound 3c

(d) IR Spectrum of compound 3d
(e) IR Spectrum of compound 3f

(f) IR Spectrum of compound 3g
(g) IR Spectrum of compound 3h

(h) IR Spectrum of compound 3i
(i) IR Spectrum of compound 3j

(j) IR Spectrum of compound 3k
Chapter 3

Synthesis and Characterization

(k) IR Spectrum of compound 3l

(l) IR Spectrum of compound 3q
(m) IR Spectrum of compound 3r

(n) IR Spectrum of compound 3t
(o) IR Spectrum of compound 4a

(p) IR Spectrum of compound 4b
(q) IR Spectrum of compound 4c

(r) IR Spectrum of compound 4d
(s) IR Spectrum of compound 4f

(t) IR Spectrum of compound 4g
(u) IR Spectrum of compound 4h

(v) IR Spectrum of compound 4i
(w) IR Spectrum of compound 4j

(x) IR Spectrum of compound 4k
(y) IR Spectrum of compound 4l

(z) IR Spectrum of compound 4q
(aa) IR Spectrum of compound 4r

(bb) IR Spectrum of compound 4t
(cc) IR Spectrum of compound 5a

(dd) IR Spectrum of compound 5b
(ee) IR Spectrum of compound 5c

(ff) IR Spectrum of compound 5d
(gg) IR Spectrum of compound 5f

(hh) IR Spectrum of compound 5g
(ii) IR Spectrum of compound $5h$

(jj) IR Spectrum of compound $5i$
(kk) IR Spectrum of compound 5j

(II) IR Spectrum of compound 5k
(mm) IR Spectrum of compound 5l

(nn) IR Spectrum of compound 5q
Figure 3.2: IR Spectra of the synthesized derivatives (3a-3t, 4a-4t, and 5a-5t)
(a) Mass spectrum of compound 3a

(b) Mass spectrum of compound 3b
Chapter 3

Synthesis and Characterization

(c) Mass spectrum of compound 3c

(d) Mass spectrum of compound 3d
Chapter 3

Synthesis and Characterization

(e) Mass spectrum of compound 3f

(f) Mass spectrum of compound 3g
Chapter 3

Synthesis and Characterization

(g) Mass spectrum of compound 3h

(h) Mass spectrum of compound 3i
(i) Mass spectrum of compound 3j

(j) Mass spectrum of compound 3k
Chapter 3
Synthesis and Characterization

(k) Mass spectrum of compound 3l

(l) Mass spectrum of compound 3q
(m) Mass spectrum of compound 3r

(n) Mass spectrum of compound 3t
Chapter 3

Synthesis and Characterization

(o) Mass spectrum of compound 4a

(p) Mass spectrum of compound 4b
(q) Mass spectrum of compound 4c

(r) Mass spectrum of compound 4d
Mass spectrum of compound 4f

Mass spectrum of compound 4g
(u) Mass spectrum of compound 4h

(v) Mass spectrum of compound 4i
Chapter 3

Synthesis and Characterization

(w) Mass spectrum of compound 4j

(x) Mass spectrum of compound 4k
(y) Mass spectrum of compound 4l

(z) Mass spectrum of compound 4q
(aa) Mass spectrum of compound 4r

(bb) Mass spectrum of compound 4t
Chapter 3

Synthesis and Characterization

(ce) Mass spectrum of compound 5a

(dd) Mass spectrum of compound 5b
(ee) Mass spectrum of compound 5c

(ff) Mass spectrum of compound 5d
Chapter 3

Synthesis and Characterization

(gg) Mass spectrum of compound 5f

(hh) Mass spectrum of compound 5g
(ii) Mass spectrum of compound 5h

(ij) Mass spectrum of compound 5i
(kk) Mass spectrum of compound 5j

(ll) Mass spectrum of compound 5k
(mm) Mass spectrum of compound 5l

(nn) Mass spectrum of compound 5q
Figure 3.3: Mass spectra of the synthesized derivatives (3a-3t, 4a-4t, and 5a-5t)
(a) $^1$H-NMR Spectrum of compound 3d

(b) $^1$H-NMR Spectrum of compound 3l
(c) $^1$H-NMR Spectrum of compound 4c

(d) $^1$H-NMR Spectrum of compound 4l
Figure 3.4: $^1$H-NMR Spectra of the most active derivatives (3d, 3l, 4c, 4l, 5b, and 5k).
Chapter 3

Synthesis and Characterization

(a) $^{13}$C-NMR Spectrum of compound 3d

(b) $^{13}$C-NMR Spectrum of compound 3l
(c) $^{13}$C-NMR Spectrum of compound 4c

(d) $^{13}$C-NMR Spectrum of compound 4l
Figure 3.5: $^{13}$C-NMR Spectra of the most active derivatives (3d, 3l, 4c, 4l, 5b, and 5k)
3.4. Conclusion

A series of fourteen 3,5-disubstituted-2-pyrazoline derivatives (2a-2t) from the second step, and three series of fourteen each of 1,3,5-trisubstituted-2-pyrazoline derivatives (3a-3t, 4a-4t and 5a-5t) from the third step, were synthesized via Claisen-Schmidt condensation followed by heterocyclization and substitution with 4-substituted benzenesulfonylchloride derivatives. The synthesized compounds were characterized by various physicochemical and spectral methods and the results were in accordance with the proposed derivatives. The IR spectra of the synthesized compounds afforded absorption bands in the regions corresponding to C=N stretching (1509-1612 cm⁻¹), N-H stretching (3456-3105 cm⁻¹) and C-H deformation (1428-1357 cm⁻¹). In the third step derivatives, a characteristic peak of sulfonyl group was also observed in the IR spectra. ¹H-NMR spectra of the compounds exhibited the presence of two non-equivalent protons of a methylene group (H_a/H_b) at δ 2.92–3.38 ppm, 3.70–3.93 coupled with each other and in turn with the vicinal methine proton (H_x) at δ 6.68–7.04. All the other aliphatic, aromatic and heteroaromatic protons were also observed at their expected ppm values.