CHAPTER-3: EXPERIMENTAL SECTION

The reaction of substituted benzoyl chloride with glycine in alkaline solution to prepared substituted hippuric acid (3a and 3b). The 5-oxazolone - azlactones series 4a-j and 7a-j synthesis reactions were performed by the hippuric acid with a substituted aldehyde in the existence of sodium acetate by Perkin condensation.

The carbonyl compounds such as aldehyde and ketones interactions lead to synthesis oxazolone compound. The ring closer reaction of oxazolone compounds 4a-j and 7a-j yielding 4-(substitutedbenzylidene)-2-(substitutedphenyl)-1,3-oxazol-5(4H)-one, where R a-j = -H, 2-NO₂, 2-Cl, 4-Cl, 4-methoxy, 3, 4-(OCH₃)₂, 2-methoxy, 4-methyl, 3-Br, 3,4,5-(OCH₃)₂. The reaction carried out in acetic anhydride at 110°C for 4hr. Subsequent adding ethanol. Solidify after overnight standing, filtration, water washing, cold ethanol washing, and recrystallization delivered 67-76% yield.

Oxygen element in Oxazole-5-one compounds replaced by alkyl aniline in existence of acetic acid to yield 4-(substitutedbenzylidene)-2-(substitutedphenyl)-1-(substitutedphenyl)-4H-imidazol-5-ones. The reaction was refluxed for > 4 hour and the mixture poured over crushed ice and the precipitate of compound recovered by filtration and followed by cold ethanol and cold water washing. The crude material crystallized from ethanol with 67-76% yield. Imidazole-5-one compounds could be synthesized by using a pyridine solvent. Pyridine itself hazardous and toxic reagent and work up procedure has to performed in a well-ventilated fume hood to prevent unpleasant pyridine smell. Microwave synthesis also attempted in the regular microwave and preferred conventional method due to limited resources of the microwave reactor.

The cyanuric chloride reaction with 6-Bromo-1,3- benzothiazol-2-amine in acetone were taken in to RBF at RT. and treat with sodium hydroxide solution followed by work up to get substituted finish product s-triazine -3-Cl-C₆H₄ , 4-
A mixture of Oxazole-5-one and phenylhydrazine in existence of potassium acetate and acetic acid with refluxing lead to yield the substituted compound 1,2,4-Triazines.

### 3.1 SYNTHESIS AND EVALUATION OF OXAZOLONE

#### 3.1.1 SYNTHESIS OF OXAZOLONE

Synthesis of 4-(substitutedbenzylidene)-2-(substitutedphenyl)-1,3-oxazol-5(4H)-one:

REACTION SCHEME 1
Preparation of substituted Hippuric acid (3a and 3b):
A substituted benzoyl chloride (1.2 mol) was added dropwise to a glycine (1.0 mol) in 10 % NaOH solution while stirring at 10°C. The reaction mixture stirs until each portion of chloride has been reacted. The reaction was exothermic and maintain a temperature of 20 °C. The clear yellow reaction mixture was added to ice and set pH: 2.0 with concentrated hydrochloric acid while stirring. The precipitate of compound recovered by filtration and followed by cold water washing. The final product was obtained after recrystallization from ethyl acetate and hexane. Yield: 80%

The reaction development was concluded on TLC aluminum sheet silica-gel 60 F245 using Ethanol: Chloroform: Acetic acid (4.5: 4.5:1) as irritate and was checked in UV light chamber. The solid dissolved in chloroform for check TLC.

Two substituted hippuric acid synthesized:

3-methyl hippuric acid (3a):
The molecular formula: C_{10}H_{11}NO_{3}
Formula Weight: 193.2 g/mol
M.P.: 137-139 °C
Yield: 80%

4-Chloro hippuric acid (3b):
The molecular formula: C_9H_8ClNO_3
Formula Weight: 213.62 g/mol
M.P: 144-146 °C,
Yield: 80%

Preparation of Oxazole-5-ones derivatives [4a-j, 7a-j]:
In a round bottom flask, a mixture of substituted hippuric acid (3a-b) (0.03mole), substituted benzaldehyde (0.03 mole), acetic anhydride (0.03 mole) and sodium acetate (0.03 mole) was taken. The reaction mixture was heated to 50-60 °C in an oil bath with constant shaking. As soon as the reaction mixture has been liquefying, the reaction mixture heated to 110 °C for 2-3 hours while stirring. Then ethanol (15gm) was added slowly while cooling and the reaction mixture was kept without stirring for 12 to 20 hours. The precipitate of compound recovered by filtration and followed by cold ethanol
Synthesis and Characterization of s-Triazine and Imidazole Heterocycles
and cold water washing. The crude material crystallized from ethanol or
toluene or chloroform: hexane (4a-j, 7a-j). NMR, IR, GCMS and elemental
analysis were performed of the compounds. The reaction progress was
checked on TLC aluminum sheet silica-gel 60 F_{254} using Ethanol: Chloroform:
Acetonitrile (4.5: 4.5:1) as irrigate and was checked in UV light chamber. The
solid dissolved in chloroform for check TLC.

4a: 4-benzylidene-2-(3-methyl-phenyl)-1,3-oxazol-5(4H)-one
The molecular formula: C_{17}H_{13}NO_{2} Formula Weight: 263.29 gm/mol, MP:132-
137°C.
Compound 4a to 4j and 7a to 7j were synthesized by a similar method. The
physical data of oxazole-5-ones derivatives are recorded in the following data

3.1.2 PHYSICAL DATA OF OXAZOLONE:
3.1.2.1 PHYSICAL DATA OF OXAZOLONE TABLE NO. 1
Physical constant of 4-(substitutedbenzyldiene)-2-(3-methylphenyl)-1,3-
oxazol-5(4H)-ones.

The product 4a where -R_1=-H, M.F = C_{17}H_{13}NO_{2}, M.W.= 263.29, yield: 72%,
MP: 132-137 °C, C % Found / Required = 77.57/77.55 and N % Found /
Required = 5.36/5.32. The product 4b where -R_1= 2-NO_2, M.F = C_{17}H_{12}N_{2}O_{4},
M.W.= 308.28, yield: 70%, MP: 166-168 ºC, C % Found / Required =
66.29/66.23 and N % Found / Required = 9.15/9.09. The product 4c where -
R_1= 2-Cl, M.F = C_{17}H_{12}NO_2, M.W.= 297.73, yield: 67%, MP: 142-145 ºC, C %
Found / Required = 68.59/68.58 and N % Found / Required = 4.75/4.70. The
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Product 4d where -R₁= 4-Cl, M.F = C_{17}H_{12}NO₂, M.W. = 297.73, yield: 75%, MP: 180-183 °C, C % Found / Required = 68.65/68.58 and N % Found / Required = 4.75/4.70. The product 4e where -R₁= 4-methoxy, M.F = C_{18}H_{15}NO₃, M.W. = 293.31, yield: 77%, MP: 166-173 °C, C % Found / Required = 73.79/73.71 and N % Found / Required = 4.85/4.78.

The product 4f where -R₁= 3, 4-(OCH₃)₂, M.F = C_{19}H_{17}NO₄, M.W. = 323.34, yield got 67%, %, MP: 146-149-208 °C, C % Found / Required = 70.65/70.58 and N % Found / Required = 4.39/4.33. The product 4g where -R₁= 2-methoxy, M.F = C_{18}H_{15}NO₃, M.W. = 293.31, yield got 69%, %, MP: 162-166 °C, C % Found / Required = 73.79/73.71 and N % Found / Required = 4.85/4.78. The product 4h where -R₁= 4-methyl, M.F = C_{18}H_{15}NO₂, M.W. = 277.31, yield: 68%, %, MP: 157-161 °C, C % Found / Required = 77.99/77.96 and N % Found / Required = 5.16/5.05. The product 4i where -R₁= 3-Br, M.F = C_{17}H_{12}BrNO₂, M.W. = 342.18, yield: 72%, %, MP: 147-150 °C, C % Found / Required = 59.75/59.64 and N % Found / Required = 4.11/4.09. The product 4j where -R₁= -3,4,5-(OCH₃)₃, M.F = C_{20}H_{19}NO₅, M.W. = 353.36, yield: 71%, MP: 133-136 °C, C % Found / Required = 68.05/67.98 and N % Found / Required = 3.99/3.96.

3.1.2.2 Physical Data of Oxazolone Table No. 2

Physical constant of 4-(substitutedbenzylidene)-2-(4-chlorophenyl)-1,3-oxazol-5(4H)-ones.

The product 7a where -R₁= -H, M.F = C_{16}H_{10}ClNO₂, M.W. = 283.7, yield: 76%, MP: 180-183 °C, C % Found / Required = 67.76/67.74 and N % Found
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The product 7b where \(-R_1=\ 2-\text{NO}_2, \text{M.F} = \text{C}_{19}\text{H}_9\text{ClN}_2\text{O}_4, \text{M.W} = 328.70\), yield: 74\%, MP: 228-233 °C, C % Found / Required = 58.49/58.46 and N % Found / Required = 5.54/5.52. The product 7c where \(-R_1=\ 2-\text{Cl}, \text{M.F} = \text{C}_{16}\text{H}_9\text{Cl}_2\text{NO}_2, \text{M.W} = 318.15\), yield: 73\%, MP: 226-230 °C, C % Found / Required = 60.44/60.40 and N % Found / Required = 4.44/4.40.

The product 7d where \(-R_1=\ 4-\text{Cl}, \text{M.F} = \text{C}_{16}\text{H}_9\text{Cl}_2\text{NO}_2, \text{M.W} = 318.15\), yield: 67\%, MP: 226-230 °C, C % Found / Required = 60.44/60.40 and N % Found / Required = 4.44/4.40. The product 7e where \(-R_1=\ 4-\text{methoxy}, \text{M.F} = \text{C}_{17}\text{H}_{12}\text{ClNO}_3, \text{M.W} = 313.73\), yield: 69\%, MP: 178-182 °C, C % Found / Required = 64.09/64.08 and N % Found / Required = 4.49/4.46.

The product 7f where \(-R_1=\ 3, 4-(\text{OCH}_3)_2, \text{M.F} = \text{C}_{18}\text{H}_14\text{ClNO}_4, \text{M.W} = 343.76\), yield got 71\%, MP: 205-208 °C, C % Found / Required = 62.92/62.89 and N % Found / Required = 4.09/4.07. The product 7g where \(-R_1=\ 2-\text{methoxy}, \text{M.F} = \text{C}_{17}\text{H}_{12}\text{ClNO}_3, \text{M.W} = 313.73\), yield got 73\%, MP: 178-182 °C, C % Found / Required = 65.12/65.12 and N % Found / Required = 4.49/4.46.

The product 7h where \(-R_1=\ 4-\text{methyl}, \text{M.F} = \text{C}_{17}\text{H}_{12}\text{ClNO}_2, \text{M.W} = 297.73\), yield: 69\%, MP: 201-205 °C, C % Found / Required = 68.62/68.58 and N % Found / Required = 4.72/4.70. The product 7i where \(-R_1=\ 3-\text{Br}, \text{M.F} = \text{C}_{16}\text{H}_9\text{BrClNO}_2, \text{M.W} = 362.60\), yield: 67\%, MP: 181-185 °C, C % Found / Required = 53.05/53.00 and N % Found / Required = 3.89/3.86.

The product 7j where \(-R_1=\ -3,4,5-(\text{OCH}_3)_3, \text{M.F} = \text{C}_{19}\text{H}_16\text{ClNO}_5, \text{M.W} = 373.78\), yield: 70\%, MP: 186-190 °C, C % Found / Required = 61.06/61.05 and N % Found / Required = 3.76/3.75.
Synthesis and Characterization of s-Triazine and Imidazole Heterocycles

3.2 SYNTHESIS AND EVALUATION OF IMIDAZOLONE

3.2.1 SYNTHESIS IMIDAZOLONE

4-(substitutedbenzylidene)-2-(substitutedphenyl)-1-(substitutedphenyl)-4H-imidazol-5-ones:

**REACTION SCHEME-2**

Preparation of Imidazole-5-ones derivatives [5a-j, 6a-j, 8a-j, 9a-j]:
A mixture of Oxazole-5-one (0.0035 moles), alkyl aniline (0.007 moles) and acetic acid (15 gm) added to 100 mL of the round bottom flask. The reaction mixture was refluxed in an oil bath at 120 °C gently for 5-12 hr. following completion of reaction, the mixture poured over crushed ice and the precipitate of imidazole-5-one. The precipitate of compound recovered by filtration and followed by cold ethanol and cold water washing. The crude material crystallized from ethanol. NMR, IR, GCMS and elemental analysis were performed of the compounds. The reaction progress was monitored on TLC aluminum sheet silica gel 60 F245 using toluene: Ethylacetate (7.5:2.5) as
Synthesis and Characterization of s-Triazine and Imidazole Heterocycles

Irrigate and was checked in a UV light chamber. The solid dissolved in chloroform for check TLC. Compound 5a-j, 6a-j, 8a-j, 9 a-j were synthesized by similar method. The physical data of imidazol-5-ones derivatives recorded in Table 3, 4, 5 & 6.

3.2.2 PHYSICAL DATA OF IMIDAZOLONE:

3.2.2.1 PHYSICAL DATA OF IMIDAZOLONE TABLE NO3: Physical constant of 4-(substitutedbenzylidene) -2-(3-methylphenyl) -1-(4-methylphenyl) -4H-imidazol-5-ones. (5a-j)

The product 5a where \(-R_1= -H, M.F = C_{24}H_{20}N_2O, M.W. = 352.42,\) yield: 68%, MP: 208-212°C, C % Found / Required = 81.82/81.89 and N % Found / Required = 7.96/7.95. The product 5b where \(-R_1= 2-NO_2, M.F = C_{24}H_{19}N_3O_3, M.W. = 397.42,\) yield: 72%, MP: 165-169 °C, C % Found / Required =72.56/72.53 and N % Found / Required = 10.59/10.57. The product 5c where \(-R_1= 2-Cl, M.F = C_{24}H_{19}ClN_2O, M.W. = 386.87,\) yield: 74%, MP: 182-186 °C, C % Found / Required =74.55/74.51 and N % Found / Required = 7.26/7.24.

The product 5d where \(-R_1= 4-Cl, M.F = C_{24}H_{19}ClN_2O, M.W. = 386.87,\) yield: 71 %, MP: 188-192 °C, C % Found / Required = 74.55/74.51 and N % Found / Required = 7.26/7.24. The product 5e where \(-R_1= 4-methoxy, M.F.= C_{25}H_{22}N_2O_2, M.W. = 382.45,\) yield: 67%, MP: 168-172°C, C % Found / Required = 78.56/78.51 and N % Found / Required =7.36/7.32. The product 5f where \(-R_1= 3, 4-(OCH3)_2, M.F = C_{26}H_{24}N_2O_3, M.W. = 412.48,\) yield: 69%, MP: 194-197°C, C % Found / Required = 75.73 /75.71 and N % Found /
Synthesis and Characterization of s-Triazine and Imidazole Heterocycles

Required = 6.81/6.79. The product 5g where -R₁ = 2-methoxy, M.F = C₂₅H₂₂N₂O₂, M.W. = 382.45, yield: 73 %, MP: 186-190 °C, C % Found / Required = 78.56/78.51 and N % Found / Required = 7.36/7.32. The product 5h where -R₁ = 4-methyl, M.F = C₂₅H₂₂N₂O₂, M.W. = 366.45, yield: 68 %, MP: 188-191 °C, C % Found / Required = 81.95/81.94 and N % Found / Required = 7.66/7.64. The product 5i where -R₁ = 3-Br, M.F = C₂₄H₁₉BrN₂O₂, M.W. = 431.32, yield: 69 %, MP: 168-172 °C, C % Found / Required = 66.85/66.83 and N % Found / Required = 6.51/6.49. The product 5j where -R₁ = -3,4,5-(OCH₃)₃, M.F = C₂₇H₂₆N₂O₄, M.W. = 442.50, yield: 74 %, MP: 212-217 °C, C % Found / Required = 73.32/73.28 and N % Found / Required = 6.36/6.33

3.2.2.2 PHYSICAL DATA OF IMIDAZOLONE TABLE NO. 4:
Physical constant of 4-(substituted benzylidene)-2-(3-methylphenyl)-1-(3,4,5-trimethylphenyl) -4H-imidazol-5-ones (6a to 6j):

Synthesis and Characterization of s-Triazine and Imidazole Heterocycles

The product 6f where \(-R_1= 3,4-(OCH_3)_2\), M.F = C_{28}H_{28}N_{2}O_{3}, M.W. = 440.53, yield: 71%, MP: 166-170°C, C % Found / Required = 76.36/76.34 and N % Found / Required = 6.39/6.6.

The product 6g where \(-R_1= 2\text{-methoxy}, M.F = C_{27}H_{26}N_{2}O_{2}, M.W. = 410.50, yield: 74%, MP: 156-159°C, C % Found / Required = 79.02/79.00 and N % Found / Required = 6.85/6.82. The product 6h where \(-R_1= 4\text{-methyl}, M.F = C_{27}H_{26}N_{2}O, M.W. = 394.50, yield: 71%, MP: 158-161°C, C % Found / Required = 82.22/82.20 and N % Found / Required = 7.12/7.10. The product 6i where \(-R_1= 3\text{-Br}, M.F = C_{26}H_{23}BrN_{2}O, M.W. = 459.37, yield: 69%, MP: 180-185 °C, C % Found / Required = 67.99/67.98 and N % Found / Required = 6.12/6.10. The product 6j where \(-R_1= -3,4,5-(OCH_3)_3, M.F = C_{29}H_{30}N_{2}O_{4}, M.W. = 470.55, yield: 72%, MP: 263-267 °C, C % Found / Required = 74.03/74.02 and N % Found / Required = 5.98/5.94.

3.2.2.3 PHYSICAL DATA OF IMIDAZOLONE TABLE NO. 5: Physical constant of 4-(substitutedbenzylimidene)-2-(4-chlorophenyl)-1-(4-methylphenyl)-4H-imidazol-5-ones (8a to 8j):

\[
\text{R}_1
\]

The product 8a where \(-R_1= -H, M.F = C_{23}H_{17}ClN_{2}O, M.W. = 372.84, yield: 75%, MP: 197-202°C, C % Found / Required = 74.12/74.09 and N % Found / Required = 7.53/7.51. The product 8b where \(-R_1= 2\text{-NO}_2, M.F = C_{23}H_{16}ClN_{3}O_{3}, M.W. = 417.84, yield: 73%, MP: 161-166°C, C % Found / Required = 66.13/66.11 and N % Found / Required = 10.08/10.06. The product 8c where \(-R_1= 2\text{-Cl}, M.F = C_{23}H_{16}Cl_{2}N_{2}O, M.W. = 407.29, yield: 71%, MP: 175-179°C, C % Found / Required = 67.85/67.83 and N % Found /
Synthesis and Characterization of s-Triazine and Imidazole Heterocycles


3.2.2.4. PHYSICAL OF IMIDAZOLONE DATA TABLE NO. 6:
Physical constant of 4-(substitutedbenzylidene)-2-(4-chlorophenyl)-1-(3,4,5-trimethylphenyl) -4H-imidazol-5-ones (9a to 9j):

The product 9a where -R1= -H, M.F = C25H21ClN2O, M.W.= 400.90, yield: 75 %, MP: 142-145°C, C % Found / Required = 74.92/74.90 and N % Found /
Synthesis and Characterization of s-Triazine and Imidazole Heterocycles


3.3 SYNTHESIS AND EVALUATION OF s-TRIAZINE

3.3.1 SYNTHESIS OF S-TRIAZINE:
\[ N_2^2, N_4^4 \text{-bis(6-bromobenzod} \text{thiazol-2-yl)-N}_6^6 \text{-ph-1,3,5-triazine-2,4,6-triamine} \text{Br} \]

**REACTION SCHEME-3**

\[ X - \text{NH}_2 + \text{Cl}^+ \text{S}^+ \text{K}^- \xrightarrow{\text{CH}_3 \text{COOH}} H_2N - \text{Br} \]

\[ \begin{align*}
\text{Cl} & \quad \text{N} \\
\text{Cl} & \quad \text{N} \\
\text{Cl} & \quad \text{N}
\end{align*} \]

\[ (I) \]

\[ \text{Acetone} \text{ Heat} \]

\[ \begin{align*}
\text{X} & \quad \text{Cl} \\
\text{II} & \quad \text{N} \\
\text{II} & \quad \text{N}
\end{align*} \]

\[ (II) \]

\[ \text{R-NH}_3 \text{ 1, 4- Dioxane} \text{ -HCl} \]

\[ (III) \]

\[ \begin{align*}
\text{X} & \quad \text{R} \\
\text{IV} & \quad \text{NH}_3 \\
\text{IV} & \quad \text{N}
\end{align*} \]

\[ (IV) \]

*Where R: -C_6H_5, -3-Cl-C_6H_4, -4-Cl-C_6H_4, -3-NO_2-C_6H_4, -4-NO_2-C_6H_4, -4-Br-C_6H_4, -4-F-C_6H_4, -2-C_6H_4N_2, -4-C_6H_4N_2, -N-CH_3-C_6H_4, -4-CH_3-C_6H_4, 4-OH-C_6H_4*

*Where X: Both -Br or Both -Cl*

**Preparation of 6-Bromo-1,3-benzothiazol-2-amine (I):**

6-bromoaniline (0.01mol), acetic acid (15mL) and potassium thiocyanate (0.03mol) were taken in a flask and stirred 30 min at 20 °C. A solution of bromine (0.01mol) in acetic acid (10mL) was added portion wise, the reaction was exothermic and the temperature raised to 30 °C. The reaction mixture was stirred for 5 to 8 hours at RT. The reaction mixture was added slowly into ice cubes and stir the mixture for one hour and the solution was neutralized by 10% NaOH in water solution. The precipitate of compound recovered by filtration and followed by cold water washing. The final product was obtained after recrystallization from acetone in alcohol. The yield was 70%
Synthesis and Characterization of s-Triazine and Imidazole Heterocycles

(1) 6-Bromo-1,3-benzothiazol-2-amine

(la) 6-Cl-1,3-benzothiazol-2-amine:

Compound (la) was prepared through the above similar method and crystallized in alcohol. Yield 70%, M.P.: 199-201 °C. M.F: C₇H₅ClN₂S, Formula Weight: 184.65 gm/mol

Preparation of N², N⁴-bis(6-bromobenzo[d]thiazol-2-yl)-6-chloro-1,3,5-triazine-2,4-diamine (III):
Cyanuric chloride (II) (0.02 mol), 40 mL of acetone and 6-Bromo-1,3- benzothiazol-2-amine (I) (0.04 mol) were taken into RBF at RT. Sodium hydroxide 4% solution in water was added slowly at RT and the reaction mixture was kept stirring for 4 hours. The reaction mixture was added slowly into ice cubes and stir the mixture for 1 hour. The mixture was slowly acidified from pH 7 to pH 6.5 with diluted hydrochloric acid. The mixture was cooled at 0 to 5°C. The precipitate of compound recovered by filtration and followed by cold water washing. The final product was obtained after recrystallization from acetone. Yield 76%.

Preparation of N², N⁴-bis(6-bromobenzo[d]thiazol-2-yl)-N₆-ph-1,3,5-triazine-2,4,6-triamine (IV):
1, 4 dioxane (40mL) and Compound (III) (0.02 mol) were taken in to 250mL of RBF. Substituted Aniline (0.02 mol) was added slowly to the reaction mixture at RT. The solution was neutralized by 10% NaOH in water solution and adjust pH: 6.5 to 7. The reaction mixture was stirred at 65-70 °C for four hours. The reaction mixture was added slowly into ice cubes and stir the mixture for one hour. The mixture was slowly acidified from pH 7 to pH 6.5 with diluted hydrochloric acid. The mixture was cooled at 0 to 5°C. The precipitate of compound recovered by filtration and
followed by cold water washing. The final product was obtained after recrystallization from methanol. NMR, IR and elemental analysis were performed of the compounds. Yield: 65 %. Compound Br$_1$ to Br$_{12}$ and Cl$_1$ to Cl$_{12}$ was synthesized by an above similar method. The physical data of Br$_{1-12}$ and Cl$_{1-12}$ derivatives are recorded in Table 7&8.

### 3.3.2 PHYSICAL DATA OF s-TRIAZINE:

#### 3.3.2.1 PHYSICAL DATA OF s-TRIAZINE TABLE NO. 7:

**Physical constant of Br$_1$ to Br$_{12}$**

The product Br$_1$ where -$R_1$= -C$_6$H$_5$, M.F = C$_{23}$H$_{14}$Br$_2$N$_8$S$_2$, M.W. = 626.35, yield: 68%, MP: 225-230 °C, C % Found / Required = 44.05/44.10 and N % Found / Required = 17.85/17.89. The product Br$_2$ where -$R_1$= -3-Cl-C$_6$H$_4$, M.F = C$_{23}$H$_{13}$Br$_2$ClN$_8$S$_2$, M.W. = 660.79, yield: 56%, MP: 215-220 °C, C % Found / Required = 41.60/41.81 and N % Found / Required = 16.95/16.95. The product Br$_3$ where -$R_1$= -4-Cl-C$_6$H$_4$, M.F = C$_{23}$H$_{13}$Br$_2$ClN$_8$S$_2$, M.W. = 660.79, yield: 64%, MP: 190-195 °C, C % Found / Required = 41.71/41.81 and N % Found / Required = 16.92/16.96.

The product Br$_4$ where -$R_1$= -3-NO$_2$-C$_6$H$_4$, M.F = C$_{23}$H$_{13}$Br$_2$N$_9$O$_2$S$_2$, M.W. = 671.35, yield: 62%, MP: 134-137 °C, C % Found / Required = 41.10/41.15 and N % Found / Required = 18.75/18.78. The product Br$_5$ where -$R_1$= -4-NO$_2$-C$_6$H$_4$, M.F = C$_{23}$H$_{13}$Br$_2$N$_9$O$_2$S$_2$, M.W. = 671.35, yield: 67%, MP: 129-134 °C, C % Found / Required = 41.11/41.15 and N % Found / Required = 18.76/18.78.
Synthesis and Characterization of s-Triazine and Imidazole Heterocycles


The product Br10 where -R1= -N-methyl-C6H4, M.F = C24H16Br2N8S2, M.W. = 640.38, yield: 58 %, MP: 210-215 °C, C % Found / Required = 45.00/45.01 and N % Found / Required = 17.09/17.50. The product Br11 where -R1= -4-methyl-C6H4, M.F = C24H16Br2N8S2, M.W. = 640.38, yield: 58 %, MP: 245-250 °C, C % Found / Required = 44.96/45.01 and N % Found / Required = 17.46/17.50. The product Br12 where -R1= -4-OH-C6H4, M.F = C23H14Br2N8OS2, M.W. = 642.35, yield: 60 %, MP: 195-200 °C, C % Found / Required = 44.94/43.01 and N % Found / Required = 17.42/17.44

3.3.2.2 PHYSICAL DATA OF s-TRIAZINE TABLE NO. 8:

Physical constant of Cl 1 to Cl 12

Synthesis and Characterization of s-Triazine and Imidazole Heterocycles

The product \( \text{Cl}_4 \) where \(-\text{R}_1=\text{-}3\text{-NO}_2\text{-C}_6\text{H}_4\), \( \text{M.F} = \text{C}_{23}\text{H}_{13}\text{C}_2\text{N}_9\text{O}_2\text{S}_2 \), \( \text{M.W.} = 582.44 \), yield: \( 62\% \), MP: \( 170\text{-}175\ ^\circ\text{C} \), C % Found / Required = 47.41/47.43 and N % Found / Required = 21.52/21.64.

The product \( \text{Cl}_5 \) where \(-\text{R}_1=\text{-}4\text{-NO}_2\text{-C}_6\text{H}_4\), \( \text{M.F} = \text{C}_{23}\text{H}_{13}\text{C}_2\text{N}_9\text{O}_2\text{S}_2 \), \( \text{M.W.} = 582.44 \), yield: \( 60\% \), MP: \( 224\text{-}230\ ^\circ\text{C} \), C % Found / Required = 47.39/47.39 and N % Found / Required = 21.58/21.64. The product \( \text{Cl}_6 \) where \(-\text{R}_1=\text{-}4\text{-Br-C}_6\text{H}_4\), \( \text{M.F} = \text{C}_{23}\text{H}_{13}\text{BrCl}_2\text{N}_8\text{S}_2 \), \( \text{M.W.} = 616.34 \), yield: \( 58\% \), MP: \( 195\text{-}200\ ^\circ\text{C} \), C % Found / Required = 44.62/44.82 and N % Found / Required = 18.12/18.18.

The product \( \text{Cl}_7 \) where \(-\text{R}_1=\text{-}4\text{-F-C}_6\text{H}_4\), \( \text{M.F} = \text{C}_{23}\text{H}_{13}\text{C}_2\text{FN}_8\text{S}_2 \), \( \text{M.W.} = 555.44 \), yield: \( 63\% \), MP: \( 200\text{-}205\ ^\circ\text{C} \), C % Found / Required = 49.62/49.73 and N % Found / Required = 20.09/20.17. The product \( \text{Cl}_8 \) where \(-\text{R}_1=\text{-}2\text{-C}_5\text{H}_4\text{N}_2\), \( \text{M.F} = \text{C}_{22}\text{H}_{13}\text{C}_2\text{N}_8\text{S}_2 \), \( \text{M.W.} = 538.43 \), yield: \( 65\% \), MP: \( 225\text{-}229\ ^\circ\text{C} \), C % Found / Required = 49.02/49.07 and N % Found / Required = 23.29/23.41.

The product \( \text{Cl}_9 \) where \(-\text{R}_1=\text{-}4\text{-C}_5\text{H}_4\text{N}_2\), \( \text{M.F} = \text{C}_{22}\text{H}_{13}\text{C}_2\text{N}_8\text{S}_2 \), \( \text{M.W.} = 538.43 \), yield: \( 62\% \), MP: \( 235\text{-}240\ ^\circ\text{C} \), C % Found / Required = 49.03/49.07 and N % Found / Required = 23.31/23.41. The product \( \text{Cl}_{10} \) where \(-\text{R}_1=\text{-}N\text{-methyl-C}_6\text{H}_4\), \( \text{M.F} = \text{C}_{24}\text{H}_{16}\text{C}_2\text{N}_8\text{S}_2 \), \( \text{M.W.} = 551.47 \), yield: \( 58\% \), MP: \( 224\text{-}228\ ^\circ\text{C} \), C % Found / Required = 52.16/52.27 and N % Found / Required = 20.16/20.32.

The product \( \text{Cl}_{11} \) where \(-\text{R}_1=\text{-}4\text{-methyl-C}_6\text{H}_4\), \( \text{M.F} = \text{C}_{24}\text{H}_{16}\text{C}_2\text{N}_8\text{S}_2 \), \( \text{M.W.} = 551.47 \), yield: \( 62\% \), MP: \( 205\text{-}215\ ^\circ\text{C} \), C % Found / Required = 52.17/52.27 and N % Found / Required = 20.19/20.32. The product \( \text{Cl}_{12} \) where \(-\text{R}_1=\text{-}4\text{-OH-C}_6\text{H}_4\), \( \text{M.F} = \text{C}_{23}\text{H}_{14}\text{C}_2\text{N}_8\text{OS}_2 \), \( \text{M.W.} = 553.45 \), yield: \( 64\% \), MP: \( 205\text{-}210\ ^\circ\text{C} \), C % Found / Required = 49.83/49.91 and N % Found / Required = 20.16/20.25.
3.4 SYNTHESIS AND EVALUATION OF 1,2,4-TRIAZINE

3.4.1 SYNTHESIS 1,2,4-TRIAZINE

REACTION SCHEME-4: 1,2,4-triazine form 5-oxazolone

Preparation of 1,2,4-Triazine derivatives [10E, 10F, 10J, 11E, 11F, 11J]:
A mixture of Oxazole-5-one I (0.01 moles), phenylhydrazine (1.08gm, 0.01 moles) 0.2gm of potassium acetate and acetic acid (15 gm) added to 100 mL of the round bottom flask. The reaction mixture was refluxed in an oil bath at 120 °C gently for 5-12 hr. following completion of reaction, the mixture poured over crushed ice and the precipitate of 1,2,4-Triazine.
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The precipitate of compound recovered by filtration and followed by cold ethanol and cold water washing. The crude material crystallized from ethanol. NMR, IR, GCMS and elemental analysis were performed of the compounds.

The reaction progress was monitored on TLC aluminum sheet silica gel 60 F_{245} using toluene: Ethylacetate (7.5:2.5) as irrigate and was checked in a UV light chamber. The solid dissolved in chloroform for check TLC.

5-(substitutedbenzylidene)-2-phenyl-3-substitutedphenyl-1,2-dihydro-1,2,4-triazin-6(5H)-one

\[
\text{R} = \text{-CH}_3, \text{-Cl} \\
\text{R}_1 = \text{4-OCH}_3, \text{3-OCH}_3, \text{3,4,5-OCH}_3
\]

3.4.2 PHYSICAL DATA 1,2,4-TRIAZINE TABLE NO. 9:

Physical constant of 10f, 10j, 11f, and 11j compounds.

The product 10f where -R_1= 3, 4-(OCH)_2, M.F = C_{25}H_{23}N_{3}O_{3}, M.W. = 413.47, yield: 68 %, MP: 160-165 °C, C % Found / Required = 72.58/72.62 and N % Found / Required = 10.12/10.16. The product 10j where -R_1= -3,4,5-(OCH)_3, M.F = C_{26}H_{25}N_{3}O_{4}, M.W. = 443.49, yield: 65 %, MP: 220-225 °C, C % Found / Required = 70.38 / 70.41 and N % Found / Required = 9.43/9.47. The product 11f where -R_1= 3, 4-(OCH)_3, M.F = C_{24}H_{20}ClN_{3}O_{3}, M.W. = 433.89, yield: 62 %, MP: 225-230 °C, C % Found / Required = 66.40/66.44 and N % Found / Required = 9.64/9.68. The product 11j where -R_1= -3,4,5-(OCH)_3, M.F = C_{25}H_{22}ClN_{3}O_{4}, M.W. = 463.91, yield: 67 %, MP: 205-210 °C, C % Found / Required = 64.52/64.72 and N % Found / Required = 9.04/9.06.