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Synthesis and Characterization of 5-{[5-(2-aminophenyl)-1,3,4-thiadiazol-2-yl] diazenyl} quinolin-8-ol.

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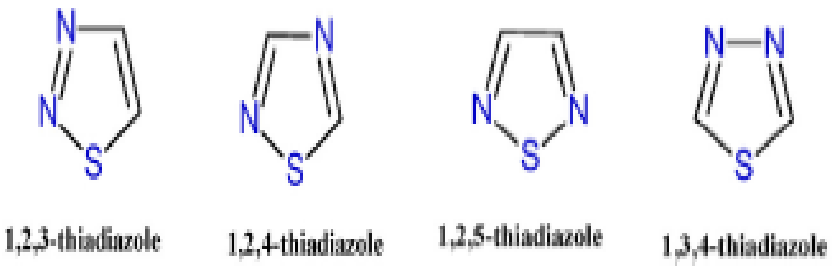
Abstract- ***This study reports the synthesis, Characterization and application of new azo-linked compound, Synthesis and Characterization of 5-{[5-(2-aminophenyl)-1,3,4-thiadiazol-2-yl]diazenyl}quinolin-8-ol, for textile applications. The compound was synthesized via diazotization and coupling reactions, with its structure confirmed by FT-IR, 1H-NMR, and 13 C-NMR spectroscopy, The dyes fastness properties were assessed on Terrycloth, nylon, and Cotton Mesh under different conditions: neutral, alkaline (NaOH, and acidic (HCl)environments, Terrycloth exhibited excellent fastness properties across all tests, with minimal color fading. Nylon showed moderate resistance in neutral and acidic environments but was less stable under alkaline conditions. Cotton Mesh the lowest fastness, particularly under alkaline conditions, due to its cellulose-based structure, making it more prone to dye leaching. These results indicate the suitability of Terrycloth and Nylon for high-performance textile applications requiring color durability.***

Keywords: Thiadiazole, Azo-dyes, Textile application, Heterocyclic compound.

I. INTRODUCTION

A heterocyclic compound are cyclic organic molecule that contain at least one hetero atom such as nitrogen, Sulfur or Oxygen as a part of the ring structure. The biological role of many heterocyclic compound are to form the building blocks of DNA and RNA bases (adenine, cytosine),Vitamins (thiamine), and natural alkaloids (caffeine, nicotine)[1]. Heterocyclic compounds play and impotrtant role in medicines[2], over half of all U.S. FDA-approved drugs contain a nitrogen heterocyclic [3]. Many heterocyclic compounds are beneficial in industries. Used to make synthetic dyes[4], Plastics[5], agrochemicals[6], and powerful industrial solvents like THF [7]. Thiadiazoles is a heterocyclic compound. Thiadiazole compounds are five-membered ring systems containing one sulfur and two nitrogen atoms. The thiadiazole rings are -electron deficient and hence do not react readily with electrophiles at nitrogen or at carbon[8]. Thiadiazole were known to displayed various of pharmacological activity. Thiadiazole were utilized as antibacterial[9], antifungal[10], and anti-cancer[11], activities in addition to and anti-inflammatory activities[12]. There are four isomer of thiadiazole are occurs in nature such as 1,3,4-thiadiazole, 1,2,3-thiadiazole, 1,2,4-thiadiazole and 1,2,5- thiadiazole.



Due to their pharmacological activities such as antimicrobial, antiviral, anti-helicobacter, anti -tubercular, anticonvulsant, antiulcer , anti-inflammatory , hypoglycemic , ant diabetic , and anticancer, etc. Heterocyclic systems containing a 1,3,4-thiadiazole is a great importance for medicinal chemistry, a large number of thiadiazole have been patented in the medical field for treatment of a wide

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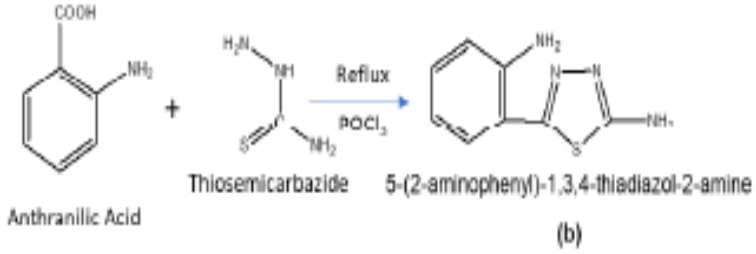
variety of diseases and some of them have become commercial product and some of them derivatives used as a against viral infections such as human viral infection, pathogens, human cytomegalovirus (HCMV), hepatitis viruses , etc. A vast number of study prove that heterocyclic compounds possesses the biological activity such as including antibacterial , anti-micro bacterial , antifungal , anti-depressive .A large number of studies are devoted to the biological activity of these compounds, including antibacterial , anti- mycobacterial , antifungal , anti-depressive , being notable. Azo dyes are organic compounds bearing the functional group R-N=N-R". In which R&R" are usually aryl. Azo dye, any of a large class of synthetic organic dyes that contain nitrogen as the azo group –N=N- as part of their molecular structures. More than half the commercial dyes belong to this class. Azo dyes are the most widely used class of coloring materials because of their massive applications in various fields of science and technology and industry including photosensitive, photodynamic therapy, electro photographic system applications and are predominant organic photo-conductive materials, such as textile, food, cosmetics and paper printing, dyeing textile leather, biomedical studies, advanced application in organic synthesis, high technology areas such as laser, liquid crystalline displays, electro-optical devise and ink-jet printer. Due to above said fact the synthesis of 1,3,4-thiadiazole derivatives and investigation, characterization and study of their biological activities has accelerated in last decades

II. METHOD AND SYNTHESIS:

The synthesis of 5-{[5-(2-aminophenyl)-1,3,4-thiadiazol-2-yl]diazenyl}quinolin-8-ol achieved through a series of steps starting from basic materials like Anthranilic acid, thiosemicarbazide, and appropriate reagents for diazotization and coupling reactions.

Step 1: Reflux Cyclization from Anthranilic Acid -
Anthranilic acid (0.01 mol) and thiosemicarbazide (0.01 mol) were mixed in $POCl_3$ (25 mL) and the reaction flask was equipped with a reflux condenser. The mixture was refluxed for 7 hours at 100°C, leading to intramolecular cyclization and formation of 2-amino-5-(2-aminophenyl)-1,3,4-thiadiazole. The mixture was cooled and quenched with ice, followed by neutralization with saturated Na_2CO_3. The solid was filtered, washed, and dried.



(b)

Step 2: Coupling with 8-Hydroxyquinoline-
The diazotization of the thiadiazole intermediate was carried out at 0–5°C using NS acid. The freshly formed diazonium salt was then slowly added to a chilled ethanolic solution of 8-hydroxyquinoline (0.01 mol) adjusted to pH 5. The product separated gradually as a deep yellow-orange solid, which was washed with cold ethanol and recrystallized.

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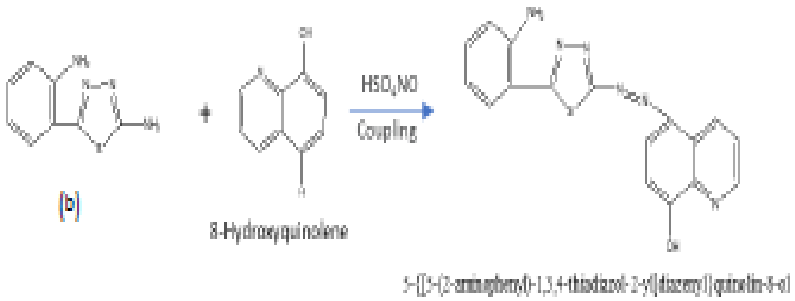

M.W: 348.38 g.mol-4
Color- Orange

Sr. No.	Name of Elements	Percentage (%)
1	Carbon (C)	58.61
2	Hydrogen (H)	3.4
3	Nitrogen (N)	24.12

^{1}H NMR spectrum-
The ^{1}H NMR spectrum of 5-{[5-(2-aminophenyl)-1,3,4-thiadiazol-2-yl]diazenyl}quinolin-8-ol was recorded in DMSO d_6 at 400 MHz. The spectrum reveals signals consistent with the expected proton environments of the molecule:

δ 9.01 (s, 2H) – attributed to the aromatic protons of the quinoline ring, significantly deshielded due to the adjacent nitrogen atom.

δ 8.04 (s, 2H) – assigned to protons on the thiadiazole-linked phenyl ring, influenced by the electron-withdrawing azo and thiadiazole groups.

δ 7.908–7.279 (m, 2H) – corresponds to aromatic protons of the quinoline and diazenyl linkage.

δ 7.275 (s, 2H) – likely due to aromatic protons on the aminophenyl group adjacent to the diazenyl moiety.

δ 7.237–6.81 (m, 2H) – attributed to overlapping aromatic signals, including remaining protons of the aminophenyl ring.

δ 6.16–6.01 (br s, 2H) – corresponds to the –NH_2 group, appearing as a broad singlet due to hydrogen bonding and exchange effects in DMSO.
^{1}H NMR (400 MHz, DMSO d_6) δ (ppm):
9.01 (s, 2H, aromatic H)
8.04 (s, 2H, aromatic H)
7.908–7.279 (m, 2H, aromatic H)
7.275 (s, 2H, aromatic H)

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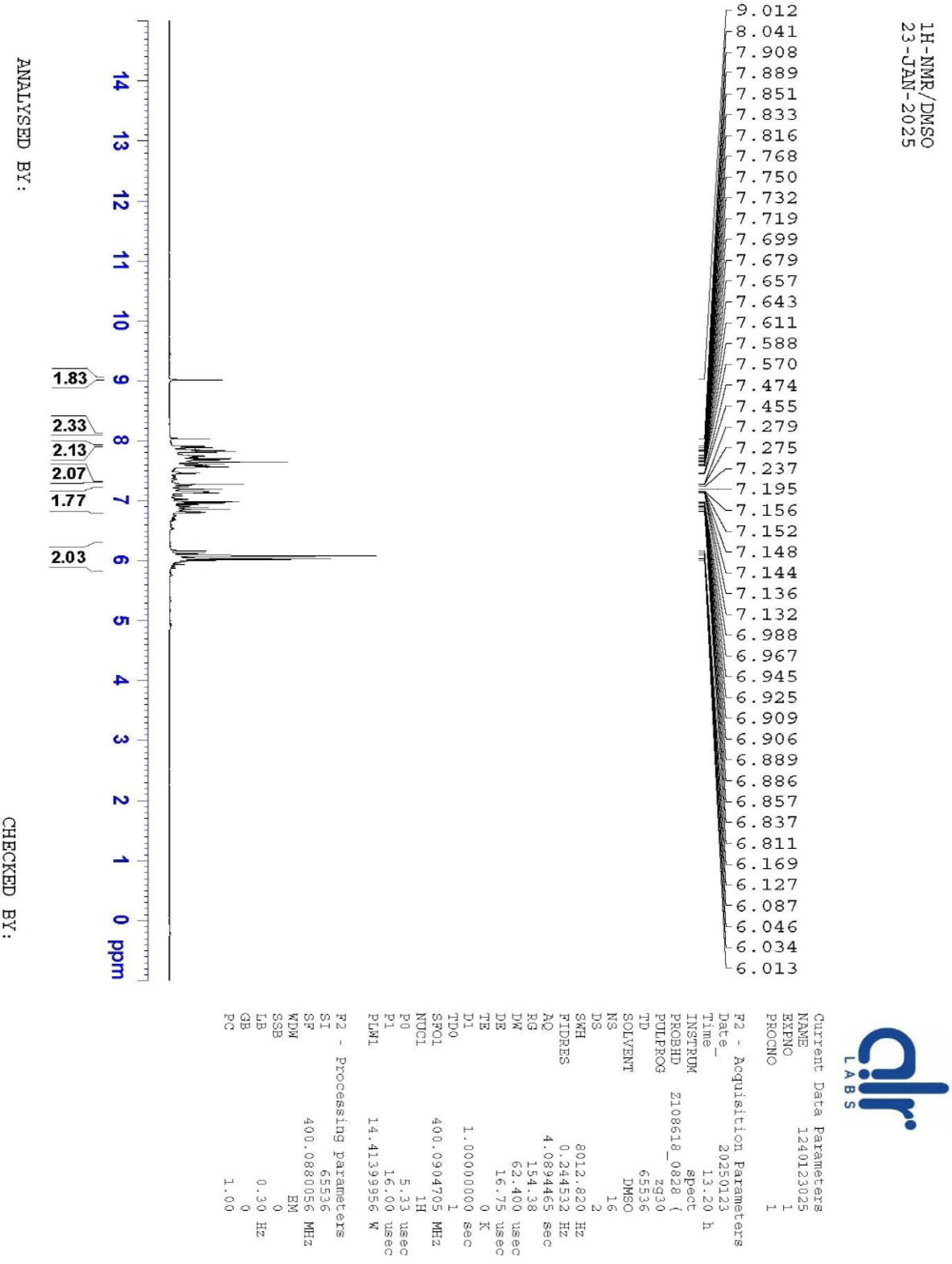

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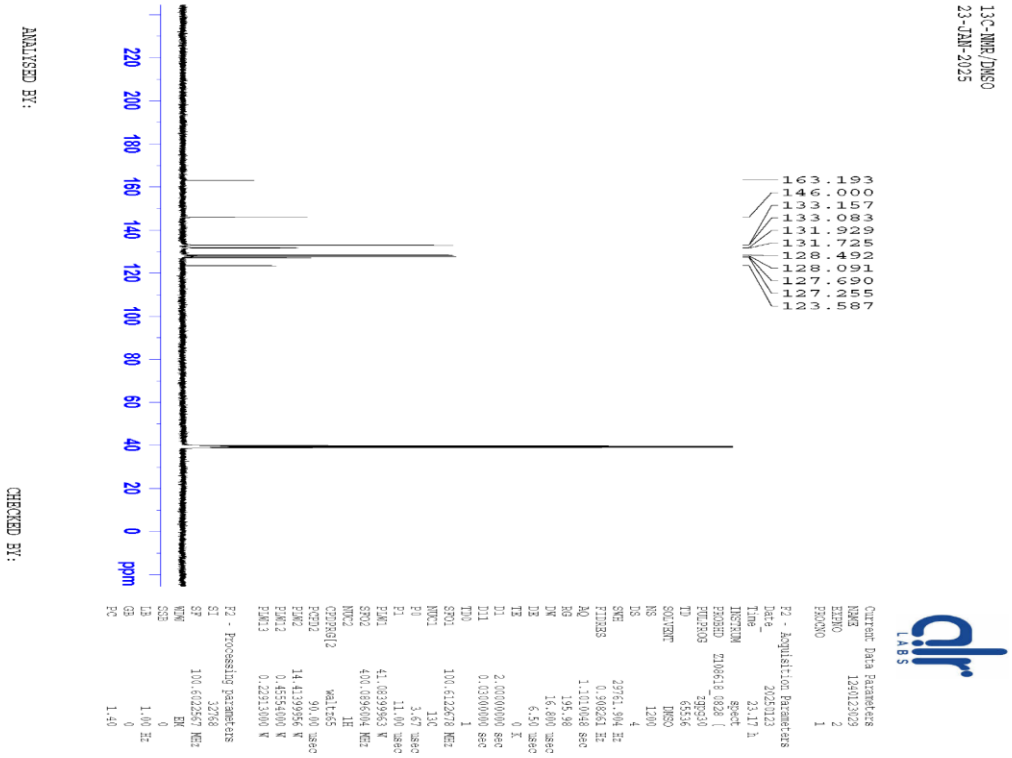
13C NMR (Fig. 4.49):

- Quinolin-8-ol has an OH at C8 → carbon usually around 160–165 ppm . Diazenyl linkage (–N=N–) pulls adjacent carbons downfield (around 145–150 ppm). Thiadiazole ring carbons show up around 130–135 ppm . The 2-aminophenyl ring contributes aromatic carbons ~127–132 ppm, slightly shifted due to NH_2 group.

δ (ppm)	Probable Assignment
163.93	C–OH at C-8 of quinolin-8-ol (phenolic –OH carbon, deshielded due to H-bonding)
146.00	Quaternary carbon adjacent to azo linkage or N=N–C in the diazenyl bridge
133.15	Quaternary carbon in the thiadiazole ring or near N=N
133.08	Quaternary aromatic carbon (quinoline or phenyl)
131.92	Aromatic CH in quinoline ring
131.72	Aromatic CH in phenyl ring of 2-aminophenyl

Figure- 13C NMR spectra of 5-{[5-(2-aminophenyl)-1,3,4-thiadiazol-2-yl]diazenyl}quinolin-8-ol



FTIR Spectra-

This compound contains the following key functional groups: Azo linkage (–N=N–), Thiadiazole ring, Aromatic amine (–NH_2), Phenyl and quinoline aromatic ring, Hydroxyl group (–OH)

Key Observed Bands:

1. Broad absorption near 3400–3300 cm^{-1}. Assignment: O–H and N–H stretching vibrations
Interpretation: The presence of both hydroxyl (from quinolin-8-ol) and amino groups.

2. Peaks around 3100–3000 cm^{-1} Assignment: Aromatic C–H stretching
Interpretation: Confirms aromatic structure from both quinoline and phenyl rings.

3. Band around 1600–1550 cm^{-1}. Assignment: N=N azo stretching and aromatic C=C stretching
Interpretation: Confirms presence of azo linkage and aromaticity.

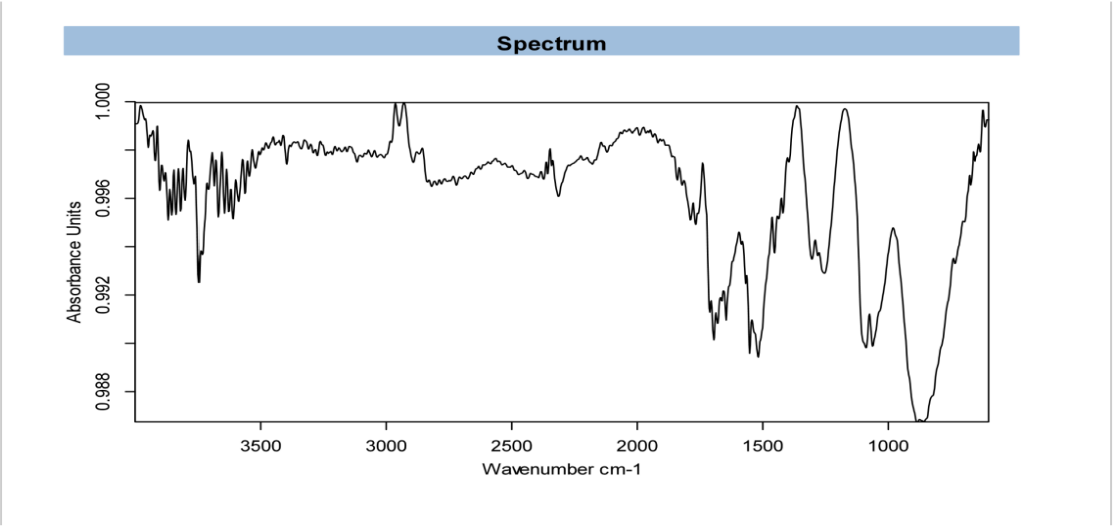
4. Medium band near 1470–1380 cm^{-1}. Assignment: C–N stretching (aromatic amine and heterocycle). Interpretation: Correlates with $–NH_2$ group and thiadiazole presence.

5. Sharp band near 1250–1100 cm^{-1}. Assignment: C–O (phenolic group) and C–N stretching Interpretation: Supports quinolinol ring (–OH at 8-position) and amino linkage.

6. Bands below 900–700 cm^{-1} Assignment: Aromatic C–H bending out-of-plane
Interpretation: Characteristic of substituted aromatic rings.

The FTIR spectrum strongly supports the successful synthesis of 5-{[5-(2-aminophenyl)-1,3,4-thiadiazol-2-yl]diazenyl}quinolin-8-ol. It shows: Broad O–H and N–H stretches, Sharp azo (N=N) vibration, Signals for aromatic and heteroaromatic rings (quinoline + thiadiazole)

Figure 4.29.- FTIR Spectra of 5-{[5-(2-Aminophenyl)-1,3,4-thiadiazol-2 yl]diazenyl}quinolin-8-ol



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