

An Overview of Various Methods and Pyrazolone Bases, Including Chalcones and Schiff Bases

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ABSTRACT

This research focuses on developing novel nitrogen-containing heterocyclic compounds and evaluating their biochemical activities. Advanced synthetic methodologies, including metal-catalyzed cyclization and multicomponent reactions, were used to synthesize these compounds. The synthetic routes were optimized to enhance yield, purity, and sustainability, incorporating green chemistry principles. The biological evaluation revealed promising biochemical activities, including antimicrobial, anti-inflammatory, and anticancer properties. Some compounds showed potent inhibitory effects against key enzymes and cellular pathways associated with these conditions, suggesting their potential as therapeutic agents. Structure-activity relationship (SAR) studies were conducted to identify molecular features contributing to these biochemical activities, guiding the optimization of lead compounds. Computer modeling and docking investigations were used to predict interactions between these compounds and biological targets, which were validated through experimental assays. The findings highlight the potential of nitrogen-containing heterocyclic compounds in developing new therapeutics, contributing to organic chemistry and enhancing drug design with enhanced efficacy and reduced side effects.

Keywords: Chalcones; Synthesis; Reactions; Biological applications, Schiff bases, pyrazole.

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INTRODUCTION

The pharmaceutical industry, medicinal chemistry, and organic chemistry have all taken an interest in these nitrogen-containing heterocyclic molecules due to their unique characteristics and applications. Furthermore, the nitrogen heterocycle that is rich in protons may readily accept or donate protons and produce many weak interactions.

In medical chemistry, nitrogen compounds play a significant role due to the intermolecular forces, which include hydrogen bonding, dipole-dipole interactions, hydrophobic effects, van der Waals forces, and π -stacking interactions. Because of how easily they dissolve in water, they form strong binding interactions with enzymes and receptors. Because of this, their offspring have extensive bioactivities, their structural properties are helpful. Their synthetic worth and the amount of synthetic study on them have led to, heterocyclic molecules are becoming more common.

Most disciplines, including medical chemistry and biochemistry, use heterocyclic compounds. "Heteros" means "different" in Greek, therefore heterocyclic. A heterocyclic compound is any organic molecule with one or more hetero atoms, such as nitrogen, oxygen, sulfur, or other atoms. Heterocyclic compounds are the most diverse organic family. Heterocyclic chemistry, a prominent subfield of organic chemistry, studies the synthesis, properties, and uses of heterocycles.

With at least one nitrogen in their ring architecture, most nitrogen-containing heterocycles have significant biological and pharmacological effects. Hydrogen bonding, dipole-dipole interaction, π -stacking interaction, and hydrophobic contact are some of the intermolecular interactions that specific enzymes or receptors respond to when they interact with particular small molecule drugs having an N-heterocycle structure. Over half of all medications authorized in 2014 were N-heterocycles, and in 2018, the FDA has registered over 80% of the top 200 small molecule pharmaceuticals by sales.

An active ligation site allows for the creation of functional derivatives, which is an additional benefit of N-heterocyclic structures. N-amidation is often used in drug manufacturing. Nearly two-thirds of the small-molecule drugs registered with the FDA include amide bond groups. Acid-catalyzed Schmidt reactions, Ritter reactions, Schotten-Baumann reactions, Passerini reactions, Ugi reactions, and Beckmann rearrangements were still utilised for the N-amidation of heterocyclic compounds. The target was a low epimerization rate, a high conversion rate, and few byproducts. Greener, more efficient catalyst-based technologies were created. For instance, ruthenium catalyst may couple amines and alcohols without pH-dependent substances, bases, or stoichiometric oxidants, with hydrogen being the only product. Most chemical procedures for preparing structurally complicated compounds produce large quantities of byproducts,

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resulting in poor atomic economy. Chemically preparing 1 kilogram in the treatment of HIV infection Without include the solvents used in synthesis and purification, the 45 kg of basic ingredients needed to make Fuzeon—a molecule with an amide bond—is excessive. Novel enzymatic techniques with improved selectivity and efficiency have been rapidly developed. In order to anticipate possible enzymatic resources, this work analysed present chemical and enzymatic approaches to N-amidating nitrogen-containing heterocyclic molecules for in silico analysis.

Literature Review

Abdelaal, Reda. (2023)

Heterocyclic compounds play a significant role in pharmacological and medical studies because of the wide range of biological effects they may cause. Here we take a look at the biological significance, structural diversity, and synthesis of a number of heterocyclic molecules. The chemistry of these compounds is investigated with a focus on the most recent developments in synthetic methodologies. The article delves further into the therapeutic potential of heterocyclic motifs by discussing their effects on medication optimization and design. To help with drug development, we also look at how heterocyclic compounds interact with enzymes and receptors, two biological targets. It is clear that heterocyclic chemistry has the ability to address both current and future health problems due to its involvement in drug optimization and development. Researchers in medicinal chemistry, pharmacology, and medication development may find valuable insights from this review, which concludes by discussing the biological functions and chemistry of chosen

heterocyclic compounds. To understand the possibilities of heterocyclic molecules as drugs, synthetic approaches, structural analysis, and strategic evaluations are useful.

Gajjar, Bhoomika et.al. (2023)

Novel therapeutic drug sources in medicinal chemistry include nitrogen-based heterocycle modulators. Roughly three quarters of all FDA-approved drugs include heterocyclic nitrogen compounds. Researchers are developing new heterocyclic compounds with unique properties and potential medical applications. We need heterocyclic compounds based on nitrogen for new medicinal medications. It has been investigated the physiological effects of many new nitrogen-based heterocycles. The novel nitrogen-based heterocycles, their biological actions, and potential medicinal applications are discussed in this article.

Saber, Mohammed. (2023). Naturally occurring N-heterocyclic compounds possess a wealth of pharmacologically active molecules that exert their anti-cancer actions via several antiproliferative mechanisms. This study reviews articles published between 2019 and 2021 that discuss the possible anticancer effects of N-heterocyclic derivatives, their structure-activity relationships, and the mechanisms of action.

Gulati, Susheel et.al. (2023)

Sustainable chemical processes for Bioactive heterocyclic molecule synthesis have recently emerged as an area of emphasis for organic chemists. scientists out of worries about the environment. As a result, synthetic chemists saw the creation of safe synthesis techniques as an important next step. Many individuals all around the globe have died

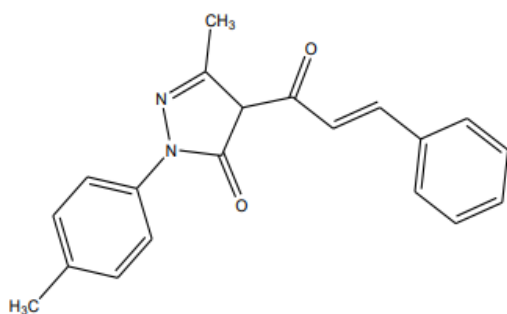


Figure 1: 6-methyl-4-(3-phenyl-acryloyl)-2-p-tolyl-2,4-dihydro-pyrazol-3-one is the compound in question (Compound- 2a)

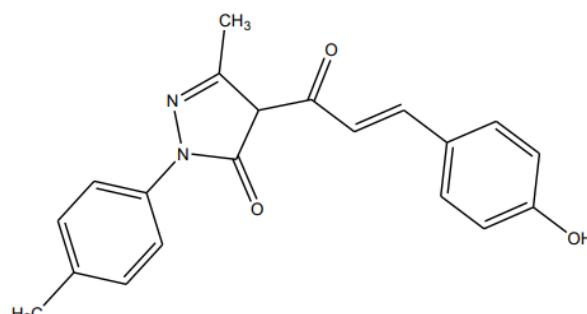


Figure 2: 4-[3-(4-hydroxy-phenyl)-acryloyl]-5-methyl-2-p-tolyl-2,4-dihydro-pyrazol-3-one is the chemical formula (Compound- 2b)

Molecular formula: C ₂₀ H ₁₈ N ₂ O ₂		Elemental analysis		
Molecular weight: 318 gm/mol			%C	% H % N
Melting point: 141-143 °C (uncorrected)		Calculated	75.45	5.70 8.80
Yield: 82%		Found	75.50	5.72 8.79
IR features around Cm ⁻¹		¹ H-NMR spectral features (δ-ppm)		
2893 Aromatic C-H stretching		7.10-8.0 (9H,m, Ar-H)		
1669 C=O		6.88, 7.61 (2H,d, CH=CH)		
1663,1593 α, β- unsaturated ketones		3.2 (1H,s,Pyrazolone)		
1605 C=N		1.92 (3H,s,CH ₃)		
1537 C=C		2.4 (3H,s,CH ₃)		

Molecular formula: C ₂₀ H ₁₈ N ₂ O ₃		Elemental analysis		
Molecular weight: 334 gm/mol			%C	% H % N
Melting point: 150-152 °C (uncorrected)		Calculate	71.84	5.43 8.38
Yield: 78%		Found	71.8	5.4 8.3
IR features around Cm ⁻¹		¹ H-NMR spectral features (δ-ppm)		
3337 -OH (phenolic)		7.0-7.60 (8H, m, Ar-H)		
2896 Aromatic C-H stretching		6.91, 7.64 (2H, d, CH=CH)		
1662 C=O		3.40 (1H, s, Pyrazolone)		
1658,1590 α, β- unsaturated ketones		1.94 (3H, s, CH ₃)		
1606 C=N		2.35 (3H, s, CH ₃)		
1542 C=C Ar		4.20 (1H, singlet, OH)		

from microbial diseases since the beginning of time. Cancer, an incurable disease that poses a threat to everyone worldwide regardless of their socioeconomic status, has currently no cure. The development and synthesis of novel chemical classes is necessary for the prevention of various diseases. Differentiating precursors for agrochemical and medicinal synthesis are heterocycles having N- or O-moieties. The biological applications and environmentally friendly methods of synthesizing heterocyclic compounds are the subject of this review study.

Sharma, Neha & Gupta, Monika. (2023)

Novel therapeutic drug sources in medicinal chemistry include nitrogen-based heterocycle analogues. In terms of therapeutic medications approved by the FDA, heterocyclic nitrogen compounds constitute over 75%. The number of novel nitrogen-based medications is expected to increase in the next decade. The number of newly designed nitrogen heterocycles is enormous. Newer N-heterocyclic compounds with applications in physiological and medicinal chemistry are appearing. This section provides an overview of novel nitrogen-containing heterocycles along with their individual biological roles.

Pyrazolone Bases and Methods for General Characterization Bases on Schiff and Chalcones Elemental Analysis

There are very few components that make up most organic molecules. Hydrogen, carbon, oxygen, nitrogen, sulfur, chlorine, and so on are among the most crucial.

Determining the elemental composition of an organic compound's molecular structure is the primary goal of elementary quantitative organic analysis.

Introduction of Spectroscopy

When it comes to studying the structures of systems that are important to chemicals, spectroscopy is the most important and promising method. Spectroscopy is a wonderful tool for

extracting structural and other physicochemical features of molecules, since it explores the phenomena caused by the interaction between matter and electromagnetic radiation. Biological atoms emit electromagnetic radiations when their electric and magnetic dipoles oscillate. Matter absorbs or emits energy in definite quantities termed quanta, which is the most essential consequence of electromagnetic interaction. distinct molecular energy levels have distinct shapes, and this energy gap of individual atoms and molecules may be measured using spectroscopic techniques.

The molecular structure may be elucidated with the use of several spectroscopy techniques and quantum chemical approaches. The visible and ultraviolet (UV) regions of an electronic spectrum are produced by changes in the energy levels of individual electrons. It reveals details about bonding and molecular orbitals. Organic chemical bond and functional group information is revealed via vibrational transitions that take place in the infrared part of the electromagnetic spectrum. Radioactive decays of nuclear spins may provide light on what is around hydrogen atoms chemically, and how many organic molecules include carbon and hydrogen atoms

Spectroscopy has been a great asset in the structural study of biological molecules, polymers, minerals, inorganic and organometallic compounds, and both simple and complex molecules.

Infrared Spectroscopy

Organic chemical identification using infrared spectroscopy is commonplace. The main focus of infrared spectroscopy is the sample's ability to absorb infrared energy. We have prepared 4 derivatives. The molecular vibrations caused by the absorption of infrared light give birth to densely packed absorption bands. Qualitative and quantitative analysis may be done using the IR approach in conjunction with intensity measurements.

When it comes to revealing the structure of mysterious substances, this method is now dominating the market, even over other scientific techniques, such as electron spin resonance and X-ray diffraction.

Infrared Activity

Changing the dipole moment is what makes a standard vibration mode infrared active. as the vibration progresses. The electric vector associated with radiation may interact with the alternating electric field produced up by the dipole moment's ongoing variation during a molecule's oscillation. Because of the way it spins or vibrates, the molecule changes its electrical dipole moment and amplitude of vibration, which allows it to absorb infrared light.

Selection Rule for IR Spectra

An integral is what determines the infrared spectrum selection rule in quantum mechanics.

$$[\mu]_{v'v''} = \int \psi_{v'}^* (Q_a) \mu \psi_{v''} (Q_a) dQ_a \dots (3.1)$$

The symbol μ here represents the dipole moment in the electrical ground condition. In order to determine the activity of the normal coordinate Q_a , we use Quantum vibrational numbers of pre- and post-transition states as by v' and v , respectively, and the vibrational eigen function is denoted as ψ . In the x, y, and z dimensions, the dipole moment may be broken divided into three sections, that is,

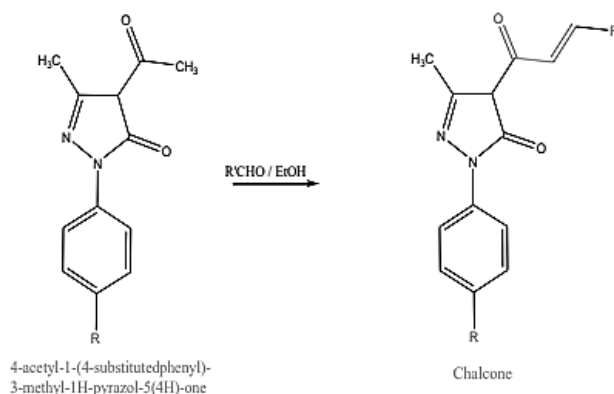


Figure 3: shows the process of chalcone production

Where R = -CH₃ & -Cl Where R' = a. phenyl

- b. 4 - hydroxy phenyl
- c. 4 - Nitro phenyl
- d. 4 - methoxy phenyl
- e. 2 - methyl phenyl
- f. 4-chloro phenyl
- g. 4 - bromo phenyl
- h. 4-methyl phen

$$[\mu x]v'v'' = \int \psi v'(Qa) \mu x \psi v''(Qa) dQa \quad \dots (3.2)$$

$$[\mu y]v'v'' = \int \psi v'(Qa) \mu y \psi v''(Qa) dQa \quad \dots (3.3)$$

$$[\mu z]v'v'' = \int \psi v'(Qa) \mu z \psi v''(Qa) dQa \quad \dots (3.4)$$

A non-zero A part Regarding the dipole moment's derivatives concerning the normal coordinate obtained at the equilibrium site determines the infrared activity of the vibrations. The shaking is inert if and only if all of the integrals are zero.

Overarching Comments on the Experimental Methods

All compounds that have been reported have crystallized. Every solvent that was utilized was thereafter dried and distilled. The compounds' purity was verified using TLC. With cell the use of silica gel in the chromatographic process. Thermofinigen 1101flash elemental analyzer was used to record the amounts of carbon, hydrogen, nitrogen, and sulphur in each chemical.

The Nicolet 760D Pellets obtained using a spectrophotometer were used in order to record the infrared spectra of KBR.

The Bruker NMR We recorded the ¹H-NMR spectra using a spectrophotometer.

A single sample from each series was subjected to MS analysis using a CH₃CN solvent on an MSD Trap 01046 apparatus.

MATERIALS

This class of compounds includes the compound 4-acetyl-1-(4-methylphenyl) (The IUPAC name - 1-(4-methylphenyl) ethan-1-one), Triple-methylpyrazol-5(4H)-1H-pyrazol (IUPAC name - 3-methyl-1H-pyrazol-5(4H)-one), The compounds 4-acetyl-1-(4-chlorophenyl) (IUPAC name - 1-[4-(4-chlorophenyl) phenyl] ethenone) and -one pyrazolones. The compounds were synthesized using the previously described procedure. Benzaldehyde compounds of different kinds were obtained from the neighborhood

store. We used analytical grade chemicals for everything else.

Acetyl pyrazolone, also known as the compound in question is This compound is known as 4-acetyl-1-(4-chlorophenyl)-3-methyl-1H-pyrazol-5(4H)-one. Synthesis was performed on the chemicals. using the previously described procedure. We got the different aniline derivatives from the neighborhood market. We used analytical grade chemicals for everything else.

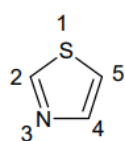
Purification of methyl-4-acetyl-1-(4-substitutedphenyl)-3-methyl compound(2a-h) and (3a-h) Chalcones that are Derived from -1H-pyrazol-5(4H)-one

Combining The compound 4-acetyl-1-(4-methylphenyl)-3-methyl The compound -1H-pyrazol-5(4H)-one (0.01mol) with a mixture of the reaction was carried out in RBF using 0.01mol of substituted aldehydes in EtOH as the media. After that, For the next day, the mixture was allowed to sit at room temperature and be stirred. adding 5 40 millilitres of a potassium hydroxide solution. We were able to monitor the reaction's progress using TLC. The addition of HCl halted immediately after the reaction, the liquid was placed into a container of ice water. to cool. Isolation of the solid from the ethanol by filtering, drying, and recrystallization. The chalcones that form as a consequence are called 2a-h. All of the chalcones 3a-h follows the same method of preparation (fig. 3.1).

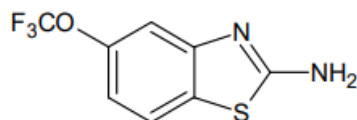
New 2,4-Disubstituted Thiazoles: Synthesis, Evaluation of Antimicrobial Antioxidant Activities

Chemically, thiazole (S-4.1) is a heterocyclic ring-shaped molecule having five carbon atoms. (C₃H₃NS) of the atomic numbers of three carbons, one sulphur, and one nitrogen. Thiazole has a pyridine-like odour and is a flammable liquid that ranges in colour from clear to light yellow. Its thiazole group is essential to thiamine and epothilone.

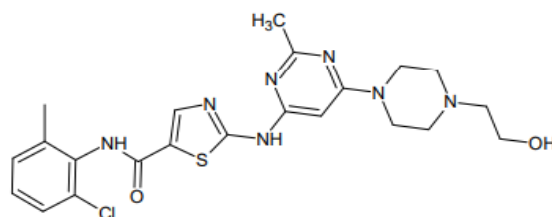
Thiazoles are often found in innovative, structurally varied natural compounds with several biological functions.



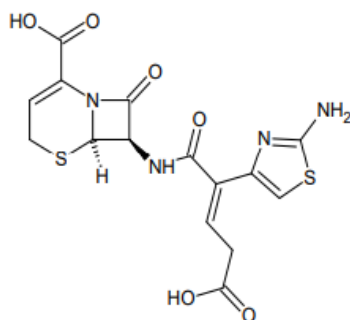
S-4.1: Thiazole



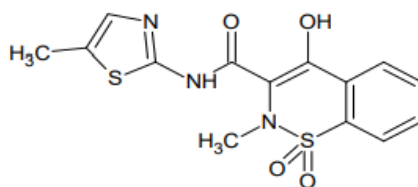
S-4.2: Riluzole (IUPAC name - 6-(trifluoromethoxy)-1,3-benzothiazol-2-amine) (Anticonvulsant)



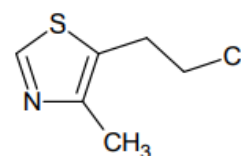
S-4.3: Dasatinib is an inhibitor of tyrosine kinases



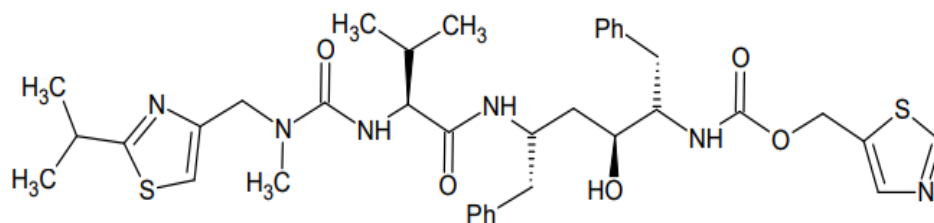
S-4.4: (Cephalosporin antibiotic) Cefibuten



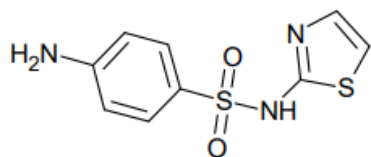
S-4.5: Meloxicam (Anti-inflammatory)



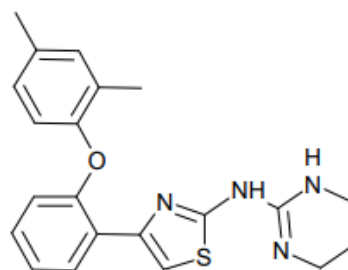
S-4.6: Sedative Clomethiazole



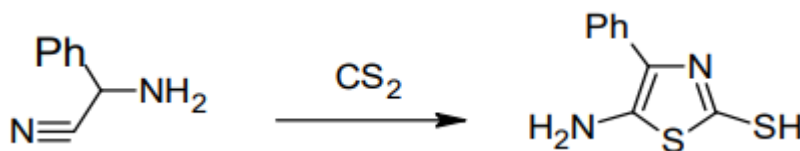
S-4.7: Ritonavir (Antiviral)



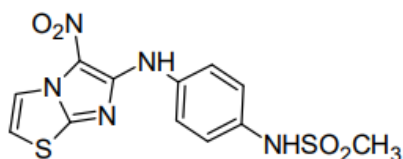
S-4.8: Sulfathiazole (Antimicrobial)



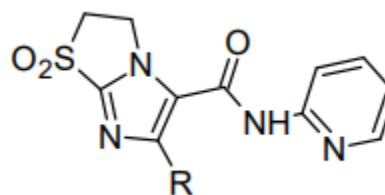
S-4.9: Abafungin (Antifungal)



S-4.10: Thiazole synthesis



S-4.11: Produced compounds


 S-4.12: 2,3-dihydroimidazo[2,1-b]; (R = CH₃, Cl)

Thiazole-containing compounds' existence in peptides, capacity to bind to proteins, DNA, and RNA, and anticancer, antiviral, and antibiotic properties have inspired many synthetic investigations and novel uses.

As seen by the abundance of medications available, several natural and synthetic compounds containing thiazole ring systems show activity against viruses, cancers, bacteria, fungi, seizures, Parkinson's disease, and inflammation. The following are examples of marketed drugs that include thiazole:

Production of 6-anilinoimidazo[2,1-b] thiazoles and tested their HeLa cell line cytotoxicity. Only S-4.11 was active among the produced compounds.

Andreani et al. (1995) synthesized and evaluated Thiazole-5-carboxamides that are 2,3-dihydroimidazo[2,1-b] (S-4.12). In relation to a test for fungi, 2-aminopyridine amides with methyl and chloro substituents on imidazole rings were the most active.

Turan-Zitouni et al. (2003) developed and examined (S-4.13) During dehalogenation, for example, the reaction generates substantial amounts of simple thiazoles but low amounts of specific modified thiazoles due to the Sulphur atom's strong nucleophilicity in thioamides and thioureas.

Pharmacology

Effectiveness Against Microbes

Newly The "well-plate" method was used to test the antibiotic activity of the manufactured substances P25–38 in a lab setting in Mueller-Hinton Agar. as described by measuring inhibition zone diameter (mm). The Microbiology Department of Kuvempu University in Shimoga, India, provides these cultures. At $\pm 25^\circ\text{C}$, all fungal strains were kept on potato dextrose agar (PDA).

Investigations on Behaviour and Acute Toxicity

The Kuvempu University Department of Biochemistry in Shimoga, India, conducted acute toxicity investigations. The current research employed Veterinary College in Bangalore, Karnataka, India, supplied the 20-25 g Swiss albino mice. In cages, the animals were kept between 45 and 55 percent relative humidity and $25 \pm 2^\circ\text{C}$ in temperature. They experienced a 12-hour cycle of light and shade and given normal feed and water. The housing conditions for the animals were standard. Prior to being used, all animals were given a week to acclimatise.

Before testing, 20-25 g albino Mice of both sexes were given water after a 24-hour fast. On trial day, animals were given various chemicals orally at increasing doses and at dosages of 250, 500, 1000, 2,000, 3,000, 4,000, and 5,000 mg/kg. We recorded the test drugs' acute harmful symptoms and aberrant behaviours. at 8, 12, and 24 hours after they had been observed continuously for 4 hours.

Antioxidant Studies (*In vitro*)

The Shimoga, India's Kuvempu University Department of Biochemistry, conducted antioxidant investigations.

Assay for DPPH Radical Scavenging

Divide the test sample and standard BHT (Butylated hydroxytoluene) into two tubes, each containing 100 µg/mL. Use methanol to bring the volume to 1 mL. After mixing and vortexing 2 mL of a new 0.1 mM DPPH solution, the combination spent 30 minutes in the dark. Stable DPPH• had an absorbance of 517 nm. The same method was used to make the DPPH control (no sample). Expressed as a percentage, the DPPH radical scavenging activity is equal to $(A_{\text{Control}} - A_{\text{Sample}} / A_{\text{Control}}) \times 100$. Absorbance of control (A_{Control}) and absorbance of sample (A_{Sample}) relative to standard BHT (A_{Sample}).

The Ability to Scavenge Nitric Oxide

The formation of nitrite ions may be shown by conducting Garrat's The Griess Illosvoy reaction involves the reaction of sodium nitroprusside in an aqueous solution at a pH that is typical of the human body. This reaction leads to the automatic generation of nitric oxide, as discovered in 1964. A modification was made to the Griess Illosvoy reagent by replacing 0.1% naphthyl ethylenediamine dihydrochloride for the original 5% 1-naphthylamine. The solution consisted of 0.33% sulphanilic acid dissolved in 20% glacial acetic acid. introduced into the 0.5 mL reaction mixture and left to undergo diazotization for a duration of 5 minutes. Diffused light was utilized to keep the mixture under observation for 30 minutes after adding 1 millilitre of naphthyl ethylenediamine dihydrochloride (0.1%). At 540 nm, the absorbance of the pink chromophore was evaluated in comparison to that of the control solution. Regarding the pursuit of food scraps and other abandoned goods purposes, test samples were contrasted with BHT. The formula to calculate the Nitric Oxide Scavenging Activity (%) is $(A_{\text{Control}} - A_{\text{Sample}} / A_{\text{Control}}) \times 100$. Absorbance of control (A_{Control}) and absorbance of sample (A_{Sample}) relative to standard BHT (A_{Sample}).

CONCLUSION

New pyrazole-ringed thiazole synthetic compounds have been developed to enhance their antioxidant and antibacterial capabilities. P₂₉ and P₃₈ showed superior antibacterial performance against all bacterial species at concentrations of 400 and 1000 µg/mL compared to Streptomycin. Fluconazole compounds P₂₉ and P₃₈ effectively inhibited the growth of all fungal strains at doses of 1000 and 500 microgrammes per millilitre. The C=N-NH- linker in the thiazole family enhances their antibacterial effect and serves as antioxidants in the Schiff base series. The 1,3,4-thiadiazole series' triazole ring's propyl chain and the pyrazole ring's p-chlorophenyl substituent are suitable for anti-inflammatory effects. These chemicals are essential for their enhanced biological activity. The study aims to understand the quantitative link between metal complexes' structure and activity on pyrazoline scaffolds used to treat tuberculosis. Chalcone was formed when imidazole-3-carboxaldehyde and 1-acetyl-2-hydroxynaphthalene performed a Claisen

Schmidt condensation in the presence of sodium hydroxide. The resulting ligands (L1-L3) were created, including zinc, nickel, copper, cobalt, and other ligand complexes. The complexes do not exhibit electrolytic characteristics, but their screening for anti-TB assays showed that copper complexes of pyrazoline derivatives were more effective than other metal complexes. Modifications will be made to enhance antimycobacterial action.

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