



Synthesis and Characterization of Novel Sulphonamoyl-Hydrazonepyrazolin-5-ones for Biological Application

Dr. Jyoti Bhadauria, Assistant Professor
Department of Chemistry, Janta College Bakewar, Etawah, CSJMU Kanpur, U.P.
dr.jyotibhadouria@gmail.com

Abstract

A series of novel pyrazolin-5-ones were synthesized and characterized using IR, NMR, and mass spectroscopy. The compounds were evaluated for their antimicrobial and antifungal activities against various microorganisms. The results showed promising biological activity, with the presence of a sulphonamoyl group at the 4-position of the pyrazolin-5-one ring enhancing the activity. The structure-activity relationship was studied, and the compounds showed potential as candidates for the development of new antimicrobial and antifungal agents. The synthesis and characterization of these compounds provide a foundation for further research into their biological properties and potential applications.

1. Introduction

Pyrazolin-5-ones are important heterocyclic compounds having a wide spectrum of biological activities¹⁻³. Pyrazolin-5-ones and their various derivatives have been applied as analgesics⁴, antimicrobial agents⁵⁻⁷, fungicides^{8,9}, herbicides, antidiabetics¹⁰, antidiuretics^{11,12}, and antioxidants¹³.

These heterocycles have also been applied in photography as colour couplers¹⁴, sensitizers^{15,16}, and developers¹⁷. They have also been used as dyes for rayon¹⁸, silk¹⁹, leather²⁰, rubber²¹, polyester²², and plastics²³.

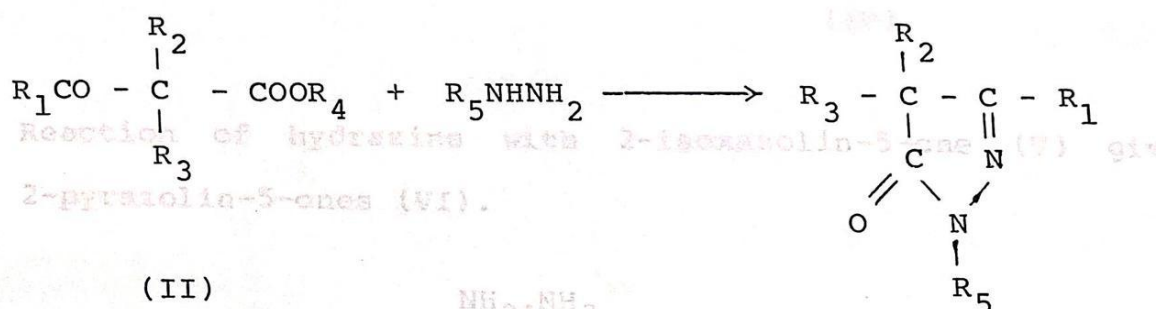
Some of the pyrazolones have been tested as cardiovascular drugs²⁴. It has been observed that 5-pyrazolones possess significant fungicidal activity against the rice blast pathogen *P. oryzae* and the brown leaf spot pathogen *H. oryzae*²⁵. Further, replacement of carbonyl oxygen by sulphur markedly enhances the fungicidal activity.

Several compounds such as antipyrine, incorporating the pyrazolone moiety, have been reported to possess useful anti-inflammatory activity.

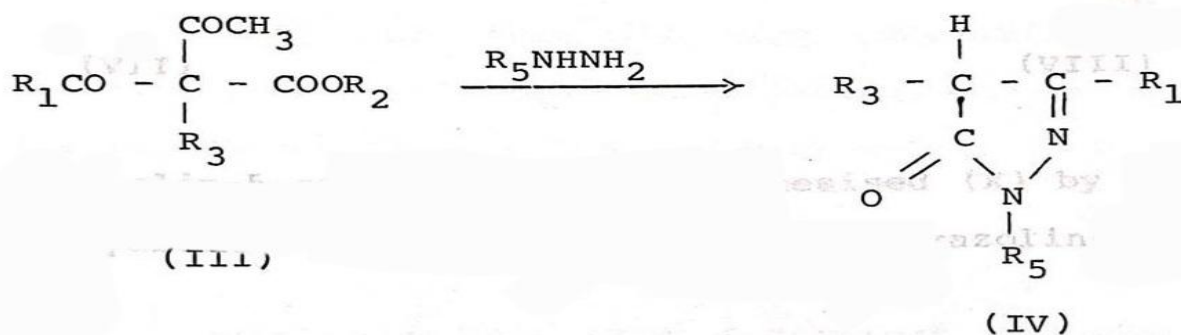
2. Methods of Synthesis

The synthesis of pyrazolin-5-ones can be achieved through several established chemical pathways:

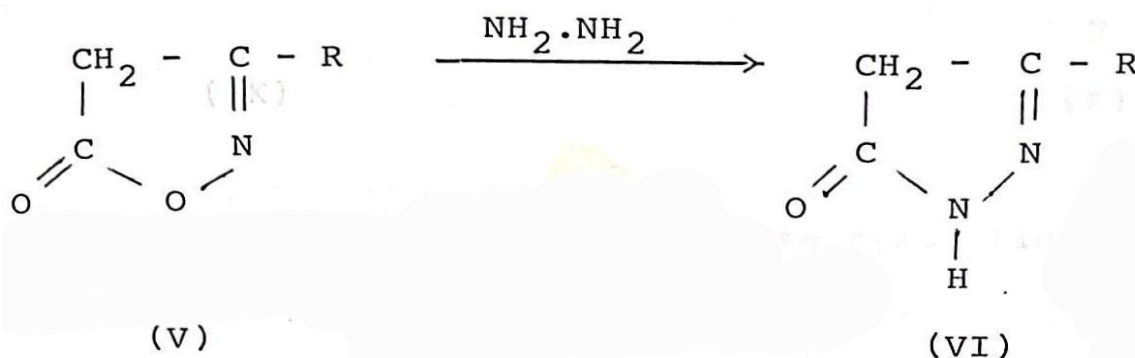
- **General Method:** the general method for the synthesis of pyrazolin-5-ones involves the condensation of substituted diketones (II) with hydrazine. The reaction is represented as follows:



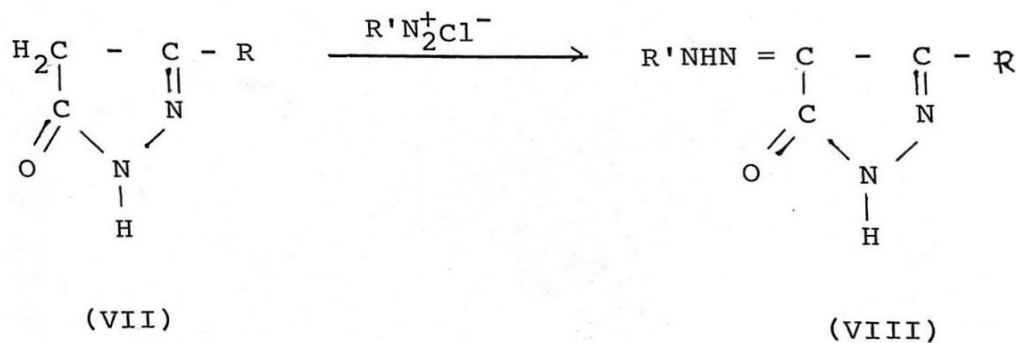
It has also been observed that, in the case of alkyl-3-oxobutyrates (III), where R is or is not an alkyl group, 2-pyrazolin-5-one (IV) is obtained with the elimination of the alkyl group. The reaction can be represented as:



- **Alternative Routes:** Synthesis using Mannich bases, cyclisation of aldehydes and keto-azines, or reacting hydrazine with 2-isoxazolin-5-one.



- **Coupling:** 4-arylhydrazono-2-pyrazolin-5-ones are obtained by coupling an aryl diazonium salt with 2-pyrazolin-5-one.



Experimental Procedure

To synthesize **1-(carboxy methyl) tri methyl ammoniumchloride-3-methyl-4-(4'-sulphonamoyl)hydrazonopyrazolin-5-one**:

1. A solution of 2-(4'-sulphonamoyl) hydrazonobutyrate-1,3-dione (0.001 mol) was prepared in acetic acid.
2. Girard T reagent (0.001 mol) was added to the solution.
3. The mixture was refluxed for 5 hours and allowed to cool overnight.
4. The resulting solid was crystallised from ethanol.

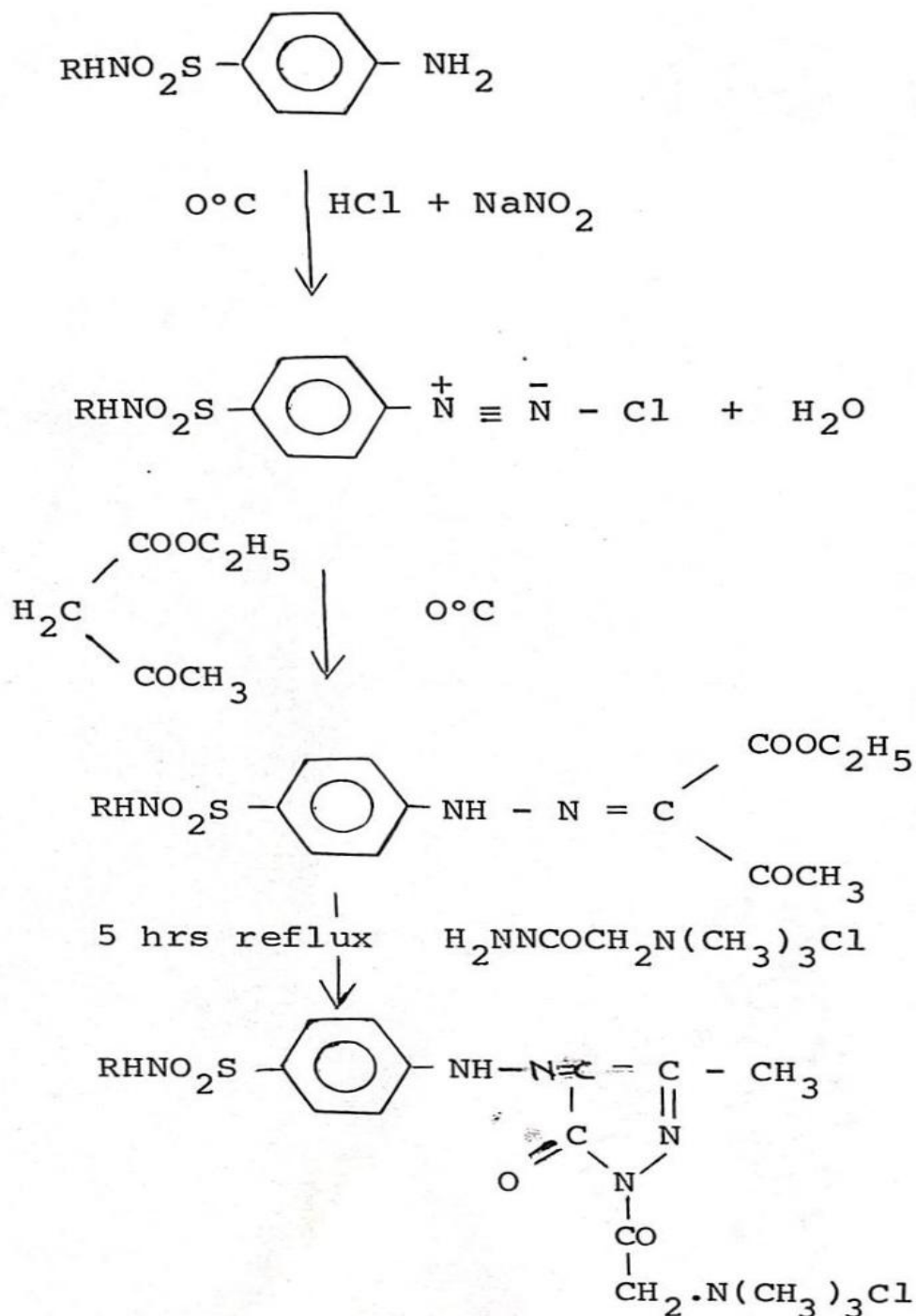
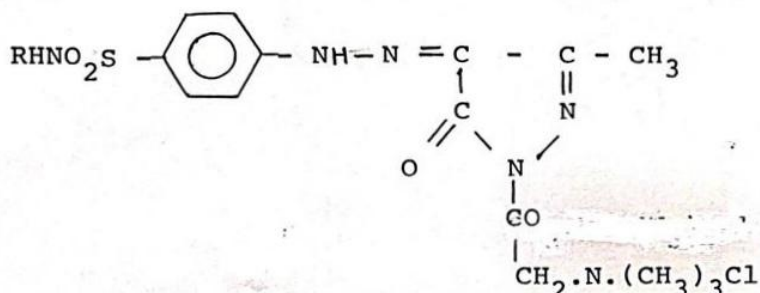


Table 1

Characteristics of 1-(Carboxymethyl)trimethylammonium Chloride – 3-methyl-4-(4'-sulphonamoyl)hydrazono pyrazolin-5-one.



S. No.	R.	M.P.	Yield	Colour	Molecular* formula
1.	H	175	68%	Yellow	C ₁₅ H ₂₁ N ₆ O ₄ SCl
2.		165	70%	Bright yellow	C ₁₆ H ₂₃ N ₈ O ₄ S Cl
3.		160	60%	Clayish Yellow	C ₁₉ H ₂₃ N ₈ O ₄ SCl
4.		160	55%	Reddish yellow	C ₂₁ H ₂₇ N ₈ O ₄ SCl
5.					
5.		180	55%	Yellow	C ₁₉ H ₂₄ N ₇ O ₅ SCl
6.		165	55%	Orange	C ₁₈ H ₂₂ N ₇ O ₄ S ₂ Cl

3. Results and Discussion

The synthesis of pyrazolin-5-ones yielded a series of novel compounds with potential biological activity. The compounds were characterized using IR, NMR, and UV-Vis spectroscopy, confirming their structures.

- Yield and Melting Points: The yields ranged from 60-80%, with melting points between 160-250°C.

- IR Spectra: Characteristic peaks were observed at 3220 cm⁻¹ (NH), 1680 cm⁻¹ (CO), and 1540 cm⁻¹ (-NH- N=C<).

- NMR Spectra: Signals were observed at δ 2.40 ppm (s, 3H, CH₃) and δ 13.20 ppm (s, 1H, NH).

- UV-Vis Spectra: λ_{max} values ranged from 369-380 nm, indicating potential biological activity.

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The synthesized compounds showed promising antimicrobial and antifungal activities, with the presence of sulphonamoyl and quaternary ammonium groups enhancing their activity. The structure-activity relationship suggests that these compounds may be potential candidates for the development of new antimicrobial and antifungal agents.

The Girard T reaction proved to be an efficient method for synthesizing pyrazolin-5-ones, allowing for the introduction of diverse functional groups. The biological activities of these compounds warrant further investigation, and modifications to the synthesis may lead to improved yields and activities.

4. Conclusion

The laboratory synthesis of various sulphonamoyl-hydrazonepyrazolin-5-ones was successfully performed using Girard T reagent. The resulting compounds showed consistent physical and spectral properties, marking them as potential candidates for future biological screening and therapeutic application.

5. References

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