SYNTHESIS OF AZINE

Submitted in partial fulfilment of the requirements for the award of Bachelor of Science in Chemistry

by

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **PRASANNA DEVI.V (Reg.No. 39030019), MUTHULAKSHMI.K (Reg.No. 39030015)** and **SANGEETHA.J (Reg.No.39030028)** who carried out the project entitled **"SYNTHESIS AND CHARACTERIZATION OF AZINE"** under my supervision from December 2021 to April 2022.

Guide

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Submitted for Viva voce Examination held on_____

Internal Examiner **External Examiner**

DECLARATION

We **PRASANNA DEVI.V (Reg.No. 39030019), MUTHULAKSHMI.K (Reg.No. 39030015), B.BHARATHI(Reg.No. 39030005)** and **SANGEETHA.J (Reg.No.39030028)** hereby declare that the project Report entitled **"SYNTHESIS AND CHARACTERIZATION OF AZINE"** done by us under the guidance of **Dr.J.KARTHIKEYAN Ph.D.,** Associate Professor, Department of Chemistry, Sathyabama Institute of science and Technology is submitted in partial fulfilment of the requirements for the award of Bachelor of Science degree in Chemistry.

DATE:

PLACE: **SIGNATURE OF THE CANDIDATES**

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I am pleased to acknowledge by sincere thanks to Board of Management of SATHYABAMA for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I would like to express my sincere and deep sense of gratitude to my project guide **Dr.J. KARTHIKEYAN** for his valuable guidance, suggestions and constant encouragement which paved way for the successful completion of my project.

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CONTENTS

CHAPTER 1

INTRODUCTION

1.1 STRUCTURE OF AZINES

Azines are a functional class of organic compounds with the connectivity RR'C=N.N=CRR'. These compounds are the product of the condensation hydrazine with ketones and aldehyde ,although in practice .They are often made by alternative. Ketazines are azines derived from ketones an aldazines are a azines derived from aldehyde.

Azines are a functional class of organic compounds with the connectivity RR'C=NN=CRR'. These compounds are the product of the condensation of hydrazine with ketones and aldehydes, although in practice they are often made by alternative routes. Ketazines are azines derived from ketones,[1] and aldazines are azines derived from aldehydes.[2] The generic formula of an azine. For an aldazine, R2 = H.

1.2 APPLICATION OF AZHINES

Ketazines are also important intermediates in the industrial production of hydrazine hydrate by the peroxide process.[3] In the presence of an oxidant, ammonia and ketones react to give hydrazine via ketazine:

$$Me(Et)C=O + 2\ NH_3 + H_2O_2 \rightarrow Me(Et)C=NN=C(Et)Me + 2\ H_2O$$

The ketazine can be hydrolyzed to the hydrazine and regenerate the ketone:

$$Me(Et)C=NN=C(Et)Me + 2\ H_2O \rightarrow 2\ Me(Et)C=O + N_2H_4$$Ketazines have been also used as sources of hydrazine produced in situ, for example in the production of herbicide precursor 1,2,4-triazole

1.3 USES OF AZINES

The coordination chemistry of azines (as ligands) has also been studied. Acetone is used to derivatize hydrazine into acetone azine for analysis by gas chromatography. This method is used to determine trace levels of hydrazine in drinking water and pharmaceuticals.

1.4 SYNTHESIS OF AZINE

Azines have been conventionally synthesised by the condensation of hydrazine with ketones and aldehydes, and many alternate routes are also available.

1.5 REACTIONS OF AZHINES

Azines characteristically undergo hydrolysis to hydrazines. The reaction proceeds by the intermediacy of a hydrazone:

$R2C=N-N=CR2 + H2O \rightarrow R2C=N-NH2 + R2C=O$

$R2C=N-NH2 + H2O \rightarrow N2H4 + R2C=O$

Azines have been used as precursors to hydrazones.

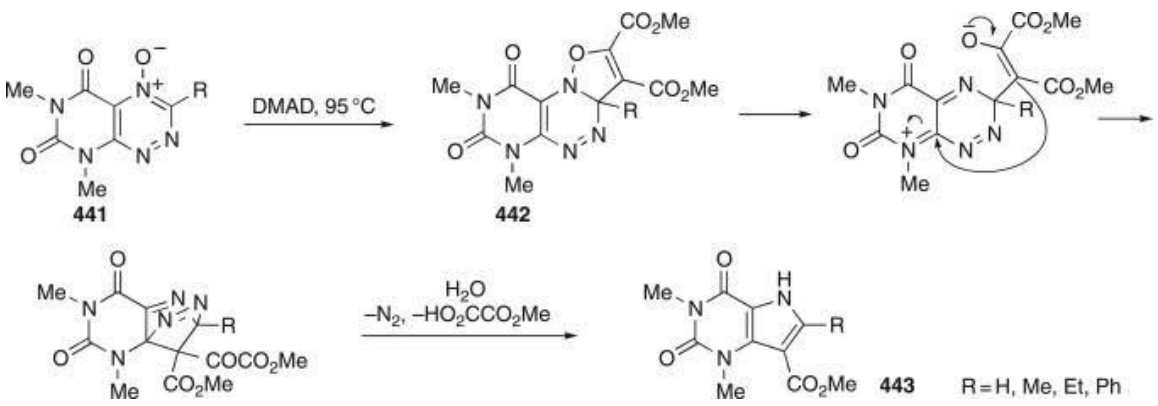
$R2C=N-N=CR2 + N2H4 \rightarrow 2\ R2C=N-NH2$

They are also precursors to diazo compounds.

Acetone azine chem.png

The coordination chemistry of azines (as ligands) has also been studied.

Acetone is used to derivative hydrazine into acetone azine for analysis by gas chromatography. This method is used to determine trace levels of hydrazine in drinking water[14] and pharmaceuticals.



1.6 PREPARATION OF AZINES

Usual method of industrial production is the peroxide process, starting from the ketone, ammonia, and hydrogen peroxide.

Pechiney-Ugine-Kuhlmann process.png In the laboratory, azines are typically prepared by condensation of hydrazine with two equivalents of a carbonyl.

Azines are also produced when chalcone reacts with a hydrazone to produce 3,5diphenyl-1Hpyrazole,[5] in a conversion also carried out with hydrazine hydrate.[6][7]

CHAPTER 2
LITERATURE SURVEY

GREEN SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL EVALUATION OF NOVEL

Azine As Anti-Bacterial Agents

BIOLOGICAL USES

It is known as a major pigment found in the eyes, hair, and skin of animals and humans which has protec-tive roles against the harmful effects of ultraviolet (UV) irradiation, oxidative stress, DNA damage, and malignant transformation [1, 2]. Despite the key features of melanin, excessive production, and hyperpigmentation of mela-nin cause dermatological disorders such as melasma, ephelides, chloasma, freckles, melanoderma, and senile lentigines .

E excess melanin synthesis can also induce inflammation such as eczema, irritant and allergic eczema contact dermatitis, which may be attributed to critical and emotionally distressing difficulty [4]. Moreover, there is some evidence about the correlation between neuromelanin and the pathogenesis of Parkinson's dis-ease [5]. Also in the agricultural industry, overproduction of melanin in fruits and vegetables causes food browning and decline in product quality [6].Tyrosinase (Polyphenol oxidase, a copper-containing enzyme, PPO, E.C.1.14.18.1) is a critical rate-limiting enzyme in the melanogenesis pathway.

Resveratrol (3,5,4′-trihydroxy-trans-stilbene) (A) is a natural antioxidant with numerous beneficial effects on human health such as neuroprotective, cardioprotective, antiinflammatory, and anti-cancer effects which prompted the use of resveratrol as a therapeutic agent [38, 39]. Resveratrol is also known as a powerful tyrosinase inhibitor with an IC50 value of 26.63 μM with no marked toxicity (main concern at doses of ≥ 0.5 g/day for long-term use) .

Oozeki et al. Reported resveratrol base structure as strong tyrosinase inhibitors with an IC50 value of 0.37 μM for the most potent compound (compound B, Fig. 2). Their results showed that the symmetry bibenzyl skeleton could easily bind to the active site of the tyrosinase and improved the inhibitory potency [41]. Furthermore, incorporation of an azo group into the linker while keeping bi-aryl structure gave azo-resveratrol derivatives. Particularly, compound C showed IC50 = 36.28 ± 0.72 μM in a dosedependent manner, comparable to that of resveratrol .

SYNTHESIS AND BIOLOGICAL EVALUATION OF SOME

Novel Pyrazoline Derivatives Derived from azine:

In view of the various activities reported for compounds possessing benzofuran [1-2] and pyrazoline moiety [3-5], in the present study we attempted to synthesize benzofuran pyrazolines, as they appeared to be highly promising. Since the compounds are mainly targeted for antiinflammatory activity, an acidic group is introduced at the 5^{th} position on the pyrazoline ring in the form of m (or) p-phenoxyacetic acid. Benzofuran chalcones (2a and 2b) were synthesized by reacting 2-acetyl benzofuran (1) with m (or) pJournal of Natural Products.

DRUGS FOR PARASITICAL INFECTIONS

Parasitic infections, starvation, insufficient shelter, and lack of clean water sources are the greatest barriers to health in our world’s growing population. Several of the parasitic infections that are very common throughout the world (e.g., ascariasis) frequently occur with mild, obscure symptoms or none at all. It is common for a host (especially of an intestinal parasite) to be asymptomatic, which is technically more of a mutualistic coexistence than true parasitism. The parasitic infections thought to be the most prevalent worldwide include toxoplasmosis, ascariasis, hookworm disease, and trichomoniasis.

CHAPTER 3

AIM AND SCOPE

3.1 AIM

The Aim of the project to Synthesize and characterization of Azines

3.2 SCOPE

Scope of building-block azines amenable to fluoroalkylation

a. Scope of trifluoromethylation using 10.

b. Scope of difluoromethylation using 11. aRun at 60 °C. bNaHCO3 (3 equiv.); H2O (10 equiv.); THF; RT used for coupling.

c. Run at 80 °C.

d. Yield determined by ^{1}H NMR using 1,3,5-trimethoxybenzene as an internal standard.

e. K2CO3 (1 equiv.), H2O:THF 1:1; RT used for coupling.

f. HCl (1 equiv.); TBAF (1 equiv.); 60 °C used for coupling.

g. A 17:1 regiomeric mixture of 4-position to 2-position. boc, tert-butoxycarbonyl; Bn, benzyl; n-Bu, normal butyl; Et, ethyl; n-Pr, normal propyl.

CHAPTER 3

MATERIALS AND METHODS

4.1 EXPERIMENTAL METHODS

The Azines was synthesized by taking 0.49 g of 4-(bis (2-chloroethyl) amino Benzaldehyde in 250 mL round bottom flask, add 10ml of ethanol, stir until it is Completely dissolved. Then, add 0.1 mL of . 4 amino benz hydrazide. Add 1 mL of 10% NaOH solution.

Which acts as a catalyst. The mixture was kept stirring for 2 hours using Magnetic Stirrer. After Precipitation, transfer the mixture into a clean Beaker. Add ice cubes to it.

Filtered it using Whatman Filter Paper. Then, wash it several times with distilled water.

Filter it and allowed to dry. To Crystallize the compound, 5 mL of 4 amino benz hydride and 0.5 mL Of Dimethyl Formamide is added in a test tube. Then, dissolve the compound till the Saturation point. Keep the test tube in the stand without any disturbance for 3 to 4 days.

CHAPTER 5

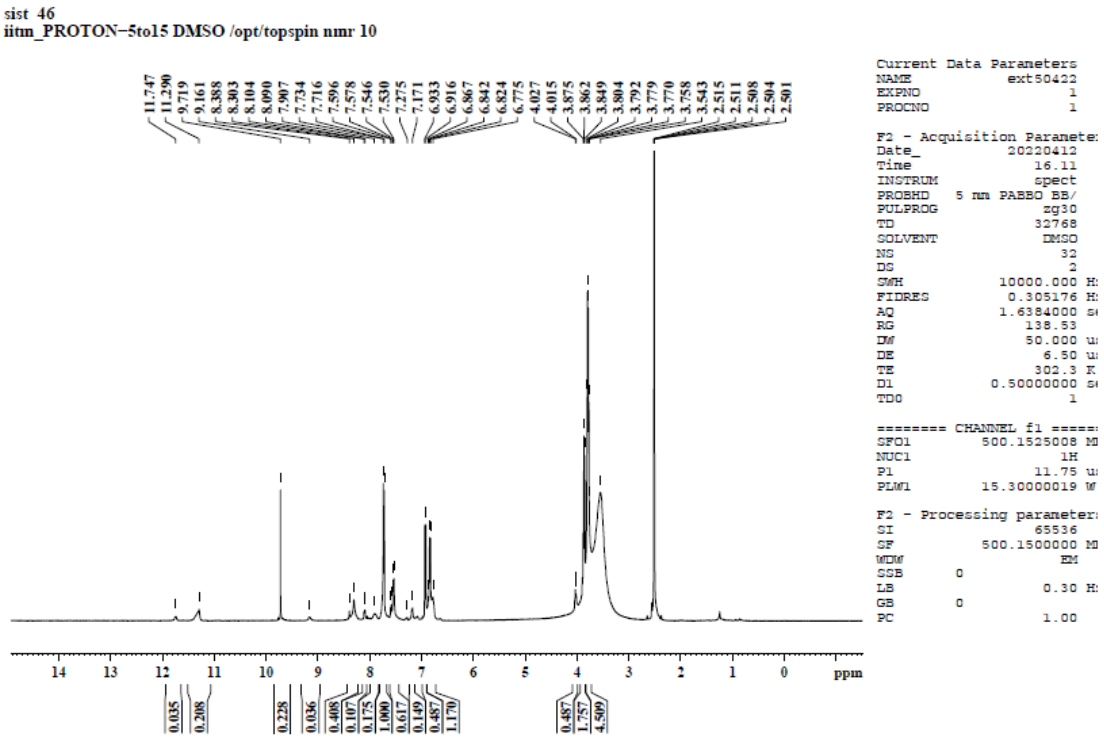
RESULTS AND DISCUSSION

NRM SPECTRAL STUDIES

5.1 H1 NMR SPECTRAL STUDIES

Nuclear Magnetic Resonance (NMR) spectroscopy is an analytical chemistry technique Used in quality control and research for determining the content and purity of a sample As well as its molecular structure.

1H NMR spectroscopy is used more often than 13C NMR, partly because proton spectra are much easier to obtain than carbon spectra. The 13C isotope is only present in about 1% of carbon atoms, and that makes it difficult to detect. The 1H isotope is almost 99% Abundant, which helps make it easier to observe. Another advantage is that 1H NMR spectroscopy gives more information than 13C



CHAPTER 6

SUMMARY AND CONCLUSIONS

4 bis (2 – chloroethyl)amino benzaldehyde

4 Amino benzahydrazine

Ethanol Con. Hcl. have been synthesized and characterized by
(NMR Spectroscopic) technique.

It is the Hydrolysis to hydrazines.

The reaction proceeds by the intermediary of hydrazine. To get the color dye.

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