

Green Synthesis of N'-Substituted-N''-Substituted Formamidino-2-Imino-4-Thiobiurates

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Abstract: Fruit juices act as a biocatalyst in different chemical transformation. Pineapple fruit juice is extensively used for condensation reactions due to their powerful and selective natural acid biocatalytic property in organic synthesis and allows mild and highly selective transformation and synthesis in facile and environmentally friendly manner. Moreover, fruits are inexpensive and easily available in the market and its juice can be extracted easily which can be used as biocatalyst in the organic transformations.

Hence one-pot three-component condensation of 1,3-diformamidinothiocarbamide with various aldehydes in presence of pineapple juice as a biocatalyst respectively to synthesize a novel series of N'-substituted-N''-substitutedformamidino-2-imino-4-thiobiurates, which is heither to unknown.

Keywords: thiocarbamide, various aldehyde, Pineapple juice.

INTRODUCTION

Green chemistry is the utilization of set of principles that will help to reduce the use of hazardous substances during manufactured of chemical products¹. Green chemistry aims to not only protect the environment by cleaning up but also inventing new chemical processes. It is a rapidly developing and an important area in the chemical science which is tremendous application in synthetic chemistry. Aromatic substituted aldehydes with a hydroxyl group have initially the researcher's interest because it having two attacking sites for further reaction²⁻⁶. The antitumor activity relationship of a series of Schiff bases derived from various substituted aromatic amines and aldehydes it has been shown that azomethines from salicylaldehydes gave the basic correlation⁷⁻⁸. Schiff bases of salicylaldehydes have been shown various activities like as a plant growth regulators⁹, antimicrobial¹⁰, antimycotic¹¹.

Schiff base $-N=CH-$ (imine) characterized by the elucidating the mechanism of transformation and racemisation reaction in biological system¹³⁻¹⁴. The lone pair of electron in nitrogen sp^2 hybridized in the azomethine group has extensive chemical and biological importance¹⁵⁻²¹. Synthesis of Schiff bases have been describes in variant condition using sulphuric acid and glacial acetic acid²². Schiff bases having significant biological activities like anticancer²³, anti-tumor²⁴, anti-inflammatory²⁵, insecticidal²⁶, antibacterial²⁷. Last decades improvement in the synthetic of procedure of metal complexes interacts with Schiff are investigated and it has also played a key role in the synthesis of metal complexes. In the field of co-ordination chemistry Schiff bases is played vital role as chelating agents.

The main objective of the research work is that to synthesize a novel series of N'-substituted-N''-substituted formamidino-2-imino-4-thiobiurates (Va-f) also investigate and set up a new reaction condition which reduces the time span of such reactions and at the same time it was also thought to increase yield of product by maintaining the purity of them. It was observed during the studies pineapple juice was the best solvent which curtails the time span and also maintains green chemistry parameters. This work is useful to incoming researcher in organic chemistry. The formation of product is as depicted below

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During the work it was observed that pineapple juices is the best solvent which increase the yield of product as well as curtail time span of reaction by maintaining the purity of products, the results obtained during work is depicted in Table No.1

EXPERIMENTAL

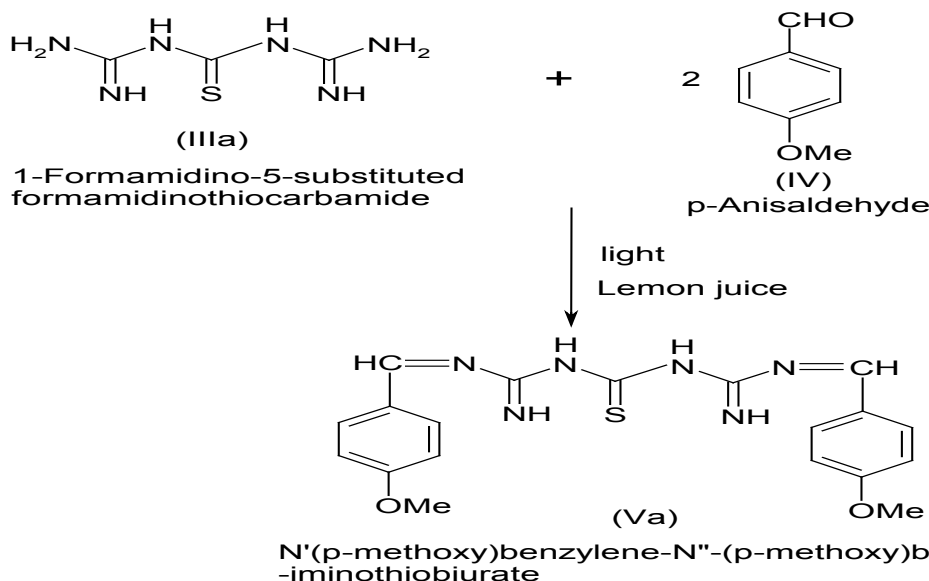
The melting point of the all synthesized compounds was recorded using hot paraffin bath. The carbon and hydrogen analysis were carried out on Carlo-Ebra1106 analyzer. Nitrogen estimation was carried out on Colman-N-analyzer-29. IR spectra were recorded on Perkin Elmer Spectrometer in range 4000-400 cm^{-1} in KBr pellets. PMR spectra were recorded on Bruker400F spectrometer with TMS as internal standard using CDCl_3 and DMSO-d_6 as solvent. The purity of compound was checked on silica Gel-G Pellets by TLC with layer thickness of 0.3mm. All chemicals used were AR-grade.

RESULT AND DISCUSSION

Synthesis of Synthesis of N-(p-methoxy)benzylene-N''-(p-methoxy)benzyleneform- amidino-2-imino-4-thiobiurate in pineapple juice (Vb)

A mixture of 1,3-diformamidinothiocarbamide (0.1M) (IIIa), anisaldehyde (0.2M) (IVa) and freshly extracted pineapple juice (20 ml) was taken in round bottom flask. It was tightly sealed and the reaction mixture was kept in sun light for 52 hours. Then the reaction mixture was poured on ice cubes with vigorous stirring, iveroy crystals were obtained these were washed several times with water. Recrystallised from aqueous ethanol.Yield 96%, melting point 265°C.

Reaction



Properties

It is iveroy crystalline solid having melting point 168°C. It gave positive test gave for nitrogen and sulphur. It was desulpurized by alkaline plumbite solution. It formed picrate having melting point 181°C.

Elemental Analysis

C[(found 56.20%) calculated 57.14], H[(found 4.40%) calculated 05.76], N[(found 21.5%) calculated 21.5], S[(found 07.08%) calculated 08.02].

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IR spectrum (cm-1)

The IR spectrum was carried out in KBr-pellets: 3405.50 (N-H stretching), 2858.10 (Ar-CH stretching), 1689.20(C=S stretching), 1591.40(C=NH imino group), 1477.60(N-C=S stretching), 1197.60(C-N stretching).

NMR Spectrum

The PMR spectrum of compound was carried out in CDCl₃ and DMSO-d₆ and reproduced on PMR. The PMR spectrum of compound was carried out in CDCl₃ and DMSO-d₆ and reproduced on PMR Plate No. This spectrum distinctly displayed the signals due to Ar-H protons at δ 10.1833-8.1600 ppm, NH proton at δ 3.3679 ppm, imino (=CH) proton at δ 2.5586 ppm.

Similarly, N-benzylene-N''-benzyleneformamidino-2-imino-4-thiobiurate (Vc), N-(m-nitro)benzylene-N''-(m-nitro)benzyleneformamidino-2-imino-4-thiobiurate (Vd) and N-methyl-N''-methylformamidino-2-imino-4-thiobiurate (Ve) were synthesized by the interaction of 1,3-diformamidinothiocarbamide (0.1M) (IIIa) with benzaldehyde, (0.2M) (IVc), m-nitrobenzaldehyde(0.2M) (IVd) and aldehyde (0.2M) (IVe) and sulphuric acid (0.1 drop) pineapple juice, respectively and enlisted in Table-II

Table-II

Sr. No.	N-substituted-N''-substituted formami-dino-2-imino-4-thiobiurate	Juice	Yield %	M. P.
1	N-benzylene-N''-benzylene -----	Pineapple	98	241
2	N-(m-nitro)benzylene-N''-(m-nitro) -----	Pineapple	96	201
3	N-methyl-N''methyl-----	Pineapple	94	230

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