Synthesis characterization of some Schiff bases derived from vanillin containing 1,3,4-thiadiazole moiety

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Abstract---The aim of this paper, thy schiff bases 1, 3, 4- Thaidiazol organic Compounds with high pharm. acological efficacy, wich play akay important vole in the tratment of urinary track infections, as anti-inflammatory, antimicrobial, anti-bacterial, analgesic, pain relief treatment of diabetics, attiveral, and anti-concer in this research, Vanillen has been used as abioimportant precursor. the Vanillen together, thiosimicarbizid is reated in the Presonce of ethanol to yield the substituted thiesimicarbazid compounds which are used in the preparation of several heterogeneous ving compounds include substituents (1,3,4-thiad; Zol-2-yl) thiesimcarbazide reacts with Sodium acetate in the presence of a lithium steak and the substituted thiosimi carbazide Compounds are also Irapped 4 (5-amino-1; 3; 4-thiadizol-2-yl) 2-methox phenol substituted by its reaction with phosphors Penta Sulfide- the vehicles prepared were diagnozed by the physical and spectral by IR methods of the nuclear magnetic resonance spectrum (H-NMR)

Keywords---hyterocyclic, thadia Zole, thio semi carbaza.

Introduction

Vanillen is an organic compound (C₈H₈O₃) with a fromula and functional groups of dehyde ether phenol is major component of Vanilla grain extract that Crystallizes into white pins, it is found in the pods of the Vanilla plant and is used as an alternative to Vanilla Plant and is used as an alternative to Vanilla material from Vanilla (3-hydroxy -4-methoxy-ban zaldehyde) and has other names, Vanilla dehyde and it is found in Voasted Coffee and Chinesered Pine, Sometimes Vanilla is used as a synthetic, instead of natural Vanilla extract, as aflavoring agent in Food, beverages and pharmaceuticals, with a distinctive oder and sweet taste, and Vanilla is used as an analytical reagent and intermediate in the Synthesis of preparation Such as methyl dopa antiky pertensive, in addition
to sickle cell anaemia, it is an anti Fungal and is used to cover the odour of certain combinations such as Caffeine and Polythazide diuretics and antihypertensive tablest it is native to mexice, but now grows. in the tropics, Madagas car is now the largest Producer of vaniilan (B-D-gly coside), has no taste odor after harvest\(^{(1)}\), Adistin cive Flavor develops after the processing process. and lasts a whole moth, But-in general, it is as follows:

Vanilla is used in skin care products such as skin, Skin and lip moisturizers an acne-a cne therapy Vanilla ahels to purge the skin and reduce the appearance of pengetbaskip and food blisters Vanilla also Containse a good source of Vitamin (B6) an vitamin (Bl2) and pan the nic acid it also helps to slow the onstet of signs of aging such as wrings and is used in Comsmetics for Fragrance\(^{(2)}\). Also to treat burns and wounds, Vanilla extract, which is focused directly on open. burns and wounds, should not be applied to hair when blending Vanilla with other ingredients such as almond oil and castor oil and applying for 15 minutes to hair that makes it soft, single and Shining, Cough, nausea, and depressio depression\(^{(3)}\)

**Experimental**

The melting pont of the prepared vehicles and all of my company's materials was measured (Fluka) and (BDH), the infrared spectrum of some models Was measured using (Frans form FTIR -84005) and the (H-NMR) has been measured for a number of the prepared using (HNVMR), Bruker Analyische messte chink GmbH j400 MHZ). and the remote has been use chem Bio 3D ultrall the digital scale, a sublimatory a heater, and a hydrant were, to pull out gases and Vapors.
Synthesis of 4-substituted 2-(4-hydroxy-3 methoxy benzyldene) thiosemicarbazide)

ThiosemiCarbuzed (0.1 mi) and Crystalline & Sodium acetate (0.03 M) were taken RB Flask, (8-10M1) water an (0.3gm) of aldehyde was added. Slowly continuous stirring added The mixture, was turbid so added methanal abs. until clear. Solution obtained shake Mixture For few minutes and allowed to stand thiosemicarbazone precipitated from the Cold Solution filler off the precipitate and recrystallize with ethandiabs(6). Other compounds were prepared Similary and their character, action date are recorder in Table (1)

Table(1) Some physical constant for thosemicar bacide Compounds (E₁, E₂)

<table>
<thead>
<tr>
<th>Comp.No</th>
<th>R</th>
<th>Molecular Formale</th>
<th>M.P (°C)</th>
<th>Yield (%)</th>
<th>Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>E₁</td>
<td>Ph</td>
<td>C₁₂H₁₂N₅O₄S</td>
<td>148</td>
<td>83</td>
<td>White</td>
</tr>
<tr>
<td>E₂</td>
<td>CH₂CH=CH₂</td>
<td>C₁₇H₁₈N₅O₂S</td>
<td>195</td>
<td>82</td>
<td>White</td>
</tr>
</tbody>
</table>

Synthesis of 4-(5-amino-1,3, 4-thiadiazol -2-yl) 2-methoxy Phenol)

Thios micarbazone (0.1M) and acetate Sodium(0.03m) were dissolved in (20-30ml) of glacial acetic acid taken inbround-bottom Flask equipped with a separating funnel for the addition of bromide. Bromine (0.6ml in 4ml glacial acid) was added slowly to it, white stirring magnetically After half an hour Stirring, the solution was Poured on crushed ice, the resulting Solid was separated, dried recrystallized from(5-6). Ethanol .abs, other compounds were prepared Similary and their characterization date are recorded in Table(2)

Table(2) Some Physical Constant for Compounds (E₃, E₄)

<table>
<thead>
<tr>
<th>Comp.No</th>
<th>R</th>
<th>Molecular Formale</th>
<th>M.P (°C)</th>
<th>Yield (%)</th>
<th>Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>E₃</td>
<td>CH₂=CH-CH₂</td>
<td>C₁₂H₁₃N₅O₂S</td>
<td>148</td>
<td>54</td>
<td>Dark yellow</td>
</tr>
<tr>
<td>E₄</td>
<td>Ph</td>
<td>C₁₇H₁₉N₅O₂S</td>
<td>195</td>
<td>82</td>
<td>White green</td>
</tr>
</tbody>
</table>

Synthesis of Sheff bass 2-methoxy-4-(5-(4- methyl benzyldene amino) - 1,3,4-thiadiazol-2-yl) phenol)

A solution of (0.1M) was prepared in 15ml Alcohol. bottom Flask Required aldehyde, (0.1m) dissolved in (10M1) alcohol, was then added to it. the mixture was fluxed for (6-7hr). the volume of alcohol, for was reduced to half by distillation under reduced Pressare the resulting solution was poured on Crushedice the precipitate which get separated was dried and recrystallized From ethanolabs(6-7). Other Compounds were prepared similary and their characterization date are recorded in Table (3)
Table (3) Some physical Constant for compounds (E₅, E₆)

<table>
<thead>
<tr>
<th>Comp.No</th>
<th>R</th>
<th>Molecular Formula</th>
<th>Yield (%)</th>
<th>M.P (°C)</th>
<th>Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>E₅</td>
<td>Ph</td>
<td>C₁₇H₁₉NOSCL</td>
<td>75</td>
<td>175</td>
<td>White</td>
</tr>
<tr>
<td>E₆</td>
<td>Ph</td>
<td>C₁₇H₁₉N₅O₂S</td>
<td>195</td>
<td>82</td>
<td>White green</td>
</tr>
</tbody>
</table>

Result and Discussion

Marly studies have shown that heterogeneous ving compounds are important in medicine as therapeutic sachst Substances, industrial dyes, and antibiotics and in Stimulants of in agriculture as triggers and growth
Synthesis of thiosemicarbazone (e₁)⁸⁻⁹.

ThioSimCarba Zid compounds are important becers because the Can be converted to this diazol derivatives when reacted with different reagent derived from the reaction of vanillin in the presence of ethanol abs. as shown by the allowing equation:

\[
\text{HO-CHO} + \text{NH}_2\text{NH-C-NH}_2 \rightarrow \text{HO-CH=N-N=C-NH}_2
\]

The frared spectros Copy if the spectrum shows Frequency bands at as have strong (C-H) due to the Frequency (3290), at the frequency. (C-C) the at bond (1157cm⁻¹), at the Frequency (C=N), (1249cm⁻¹), (C=S), (3070cm⁻¹) (N-H), HNMR (CDCl₃) (6-7.8) (4H, Ar-H), 3.8 (t, 3H of OCH₃), 5.8 (S, 1H, of OH), 8.2 (t, 3H of CH₃), 2.0 (t, 2H, NH₂)⁹⁻¹².

Table (4) spectval date for syn theses compounds (e₁-e₂)

<table>
<thead>
<tr>
<th>Comp.No</th>
<th>R</th>
<th>N-H</th>
<th>C-H</th>
<th>C-C</th>
<th>C=N</th>
<th>C=S</th>
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<tbody>
<tr>
<td>e₁</td>
<td>Ph</td>
<td>3090</td>
<td>3290</td>
<td>1157</td>
<td>1391</td>
<td>1249</td>
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<tr>
<td>e₂</td>
<td>CH₂CH=CH₂</td>
<td>3217</td>
<td>3100</td>
<td>1147</td>
<td>1595</td>
<td>1266</td>
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</tbody>
</table>

Synthesis of 4-(5-amino-1,3,4-thiadiazol-2y) 2-methoxy Phenol

Recation thios micarbazone with sodium acetate in ethanol-abs:-
The infrared spectral Copy the (IR) (3385cm⁻¹) (C-H); (1157cm⁻¹) (C-C), (1691cm⁻¹) (C=N), (1567cm⁻¹) (N=C), (949cm⁻¹) (C-S), (3120cm⁻¹) (OH), (1096cm⁻¹) (C-O), (1674cm⁻¹) (C=N), IHNMR (CDCl₃), 8 (6.7), (H, Ar-H), 8.8-7.3 (M, 4H, C₆H₅CH=N) 8.4 (S, 1H, OH), 8.8 (t, 3H, C₆H₅CHC=N), 3.3(t, 3 H, OCH₃)

Table (5) synthesis of 4- (5-amino-1,3,4-thiadiazol-2yl) 2-methoxy Phenol

<table>
<thead>
<tr>
<th>Comp.No</th>
<th>R</th>
<th>O-H</th>
<th>C-H</th>
<th>C-C</th>
<th>C=N</th>
<th>N=C</th>
<th>C=S</th>
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<tbody>
<tr>
<td>e3</td>
<td>Ph</td>
<td>3385</td>
<td>3387</td>
<td>1157</td>
<td>1691</td>
<td>1674</td>
<td>949</td>
</tr>
<tr>
<td>e4</td>
<td>CH₂=CH=CH₂</td>
<td>3290</td>
<td>3290</td>
<td>1246</td>
<td>1646</td>
<td>1670</td>
<td>930</td>
</tr>
</tbody>
</table>

Synthesis of Schiff bases of 2-methoxy-4-(5-(4- methoxy-4 (5-(4-methyl benzylidene eamino) -1, 3,4 thiadiazol-2-yl) phenol

The reaction Alcohol, Required and aldehyde and then added to it th Mixture:
The newly Synthesis Compound of Schiff bases were characterized by Using IR and HNMR spectroscopy, the Compounds showed Peaks at (3290-3271)(N-H) stretching (2935-2932 cm\(^{-1}\)) (C-H) Stretching (1170-1157) (C=N) stretching, (1631-1624), C=S Stretching / (1249-1216) cm\(^{-1}\) the NMR Spectrum of the Compound Showed, (6-7) (M,4H, Ar-H), (6-8-7.4) (M), (C\(_6\)H\(_5\)CH=N), (5-0 S,1H,OH) S8(S,H,C\(_6\) H\(_5\) CH=N)\(^{[13]}\).

Table (6) indicated the presence of aromatic proton

<table>
<thead>
<tr>
<th>Comp.No</th>
<th>R</th>
<th>N-H</th>
<th>C-H</th>
<th>C-C</th>
<th>C=N</th>
<th>C=S</th>
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<tbody>
<tr>
<td>e(_5)</td>
<td>Ph</td>
<td>3290</td>
<td>2935</td>
<td>1170</td>
<td>1691</td>
<td>1249</td>
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<tr>
<td>e(_6)</td>
<td>Ch(_2).CH=CH(_2)</td>
<td>3271</td>
<td>2932</td>
<td>1147</td>
<td>1624</td>
<td>1216</td>
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</tbody>
</table>

Conclusion

Atatal 6 compounds were synthesis with good gields All synthesis Compounds is exhibited analgesic anti-inflammatory and Antibacterial activity, The Compound (e\(_1\),e\(_2\),e\(_3\),e\(_4\),e\(_5\),e\(_6\)) were shown significant analgesic activity against swiss albino. Vats were compounds

References

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