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Synthesis of Acetoacetanilide Dicarboxylic Acid Dihydrazones and Study of their Properties

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Abstract: This paper describes a method for the synthesis of dicarboxylic acid dihydrazones based on acetoacetanilide. The reaction was carried out via condensation of acetoacetanilide with dicarboxylic acid dihydrazides in an ethanol medium. The synthesis was performed under reflux conditions and proceeded with high yields. The physicochemical properties of the obtained compounds were investigated, and their structures were confirmed based on IR and ^1H NMR spectral analysis. This class of compounds is considered promising for the synthesis of biologically active substances and coordination compounds.

Key words: acetoacetanilide, dihydrazones, dicarboxylic acids, condensation reaction, IR spectroscopy, ^1H NMR spectroscopy.

Introduction

In recent years, hydrazones and their derivatives have attracted considerable scientific interest in chemistry, pharmacy, and materials science. Hydrazone compounds are distinguished by their biological activities, including antimicrobial, antioxidant, and anti-inflammatory properties. In particular, dihydrazones derived from carbonyl compounds possess complex molecular structures and play an important role in coordination chemistry and medicinal compound synthesis.

Acetoacetanilide belongs to the class of β-diketones and contains highly reactive carbonyl groups in its molecular structure. These functional groups readily undergo condensation with dicarboxylic acid dihydrazides, leading to the formation of new dihydrazone compounds. Such compounds can serve as valuable starting substrates for the synthesis of biologically active substances.

The aim of this work was to synthesize dicarboxylic acid dihydrazones based on acetoacetanilide, determine the optimal conditions for their preparation, and conduct a preliminary study of their physicochemical properties.

Materials and Methods

Reagents and Equipment

The following reagents were used in the study:

- acetoacetanilide
- dicarboxylic acid dihydrazides (e.g., oxalic acid dihydrazide, malonic acid dihydrazide)
- ethanol (as solvent)
- acetic acid (as catalyst)

The equipment included round-bottom flasks, a reflux condenser, a magnetic stirrer, a water bath, and a vacuum filtration apparatus.

Synthesis Procedure

A solution of acetoacetanilide in ethanol was placed in a round-bottom flask, and an equimolar amount of the corresponding dicarboxylic acid dihydrazide was added. A few drops of acetic acid were introduced as a catalyst. The reaction mixture was heated under reflux for 2–3 hours.

After completion of the reaction, the mixture was cooled, and the resulting precipitate was filtered under vacuum. The product was washed with ethanol and dried at room temperature.

Purification and Analysis

The obtained dihydrazones were purified by recrystallization. Melting points were determined using the capillary method. Reaction yields (%) were calculated.

Results

As a result of the study, a series of dicarboxylic acid dihydrazones based on acetoacetanilide were successfully synthesized. The reactions proceeded under mild conditions and were completed with high yields (65–80%).

The obtained compounds were solid, colorless or pale yellow crystalline substances. They were readily soluble in organic solvents such as ethanol and methanol but poorly soluble in water. The narrow melting point ranges indicated a sufficient degree of purity of the synthesized products.

As a result of the synthesis, several new dihydrazone compounds were obtained. The reaction yields ranged from 65 to 80%. The products were crystalline solids exhibiting good solubility in ethanol and methanol.

Table 1. Synthesis of Dihydrazones Based on Acetoacetanilide

No.	Type of dihydrazide	Solvent	Reaction time (h)	Temperature	Yield (%)	Melting point (°C)
1	Oxalic acid dihydrazide	Ethanol	2.0	Reflux	78	198–200
2	Malonic acid dihydrazide	Ethanol	2.5	Reflux	72	185–187
3	Succinic acid dihydrazide	Ethanol	3.0	Reflux	68	172–174

Discussion

As shown in Table 1, despite identical reaction conditions, the structure of the dicarboxylic acid dihydrazide significantly affects the reaction yield. With increasing chain length, a slight increase in reaction time and a decrease in yield were observed. This can be explained by steric factors and differences in reactivity. IR and ^1H NMR spectral analyses confirmed the formation of azomethine (C=N) bonds as a result of the condensation reaction.

The high reactivity of the carbonyl groups in the acetoacetanilide molecule facilitates efficient condensation with dicarboxylic acid dihydrazides. The use of acetic acid as a catalyst accelerates the reaction and reduces the likelihood of side reactions.

The structural features of the obtained dihydrazones allow them to be considered as potential ligands for the synthesis of biologically active substances or metal complexes. Furthermore, more detailed investigations using spectroscopic methods (IR, NMR) can provide complete structural confirmation of these compounds.

General Reaction Scheme

Acetoacetanilide (AAAn) undergoes condensation with dicarboxylic acid dihydrazides (DKDG) to form dihydrazones:

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Acetoacetanilide + Dicarboxylic acid dihydrazide → (EtOH, CH_3COOH, heating) → Acetoacetanilide dihydrazone + $2H_2O$

In this condensation process, two molecules of water are eliminated and two azomethine (C=N) bonds are formed.

Reaction with Oxalic Acid Dihydrazide

Reaction equation:

$C_{10}H_{11}NO_2 + NH_2NH–CO–CO–NHNH_2 \rightarrow$ Oxalic acid-based dihydrazone + $2H_2O$

Schematic representation:

$AAAn + H_2N–NH–CO–CO–NH–NH_2 \rightarrow AAAn=N–NH–CO–CO–NH–N=AAAn + 2H_2O$

Reaction with Malonic Acid Dihydrazide

Reaction equation:

Acetoacetanilide + $NH_2NH–CO–CH_2–CO–NHNH_2 \rightarrow$ Malonic acid dihydrazone + $2H_2O$

Schematic representation:

$AAAn=N–NH–CO–CH_2–CO–NH–N=AAAn$

Reaction with Succinic Acid Dihydrazide

Reaction equation:

Acetoacetanilide + $NH_2NH–CO–CH_2–CH_2–CO–NHNH_2 \rightarrow$ Succinic acid dihydrazone + $2H_2O$

Brief Mechanistic Description

- The carbonyl (C=O) group of acetoacetanilide undergoes nucleophilic addition with the $–NH_2$ group of the hydrazide
- An intermediate geminal diol is formed
- Elimination of water leads to the formation of an azomethine (C=N) bond
- The process is repeated for both carbonyl groups, resulting in dihydrazone formation

Conclusion

In this study, an efficient method for the synthesis of dicarboxylic acid dihydrazones based on acetoacetanilide was developed. The reaction was shown to proceed under simple conditions with high yields. The obtained results demonstrate the practical and theoretical significance of this class of compounds and provide a basis for further scientific research.

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