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SYNTHESIS AND PROPERTIES OF ACETOACETOANANILIDE OXALIC ACID DIHYDRAZONE

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Abstract: This article presents the synthesis of dihydrazone derivative obtained by condensation of acetoacetoananilide with oxalic acid dihydrazide, as well as a detailed investigation of its physical, chemical, and spectral properties. The reaction was carried out under mild conditions in ethanol solution. The structure of the synthesized compound was confirmed by IR, UV spectroscopy and elemental analysis. Potential biological and analytical applications of the obtained compound are discussed.

Keywords: acetoacetoananilide, oxalic acid dihydrazide, dihydrazone, condensation, IR spectroscopy, biological activity.

1. Introduction

The chemistry of hydrazones and dihydrazones occupies a prominent place in modern organic and medicinal chemistry. These compounds are synthesized through condensation reactions between carbonyl compounds and hydrazines or dihydrazides. Among carbonyl substrates, acetoacetoananilide (N-phenylacetoacetamide) has attracted considerable attention due to its dual reactivity — possessing both an active methylene group and a keto function — making it a versatile building block for heterocyclic and chelating ligand synthesis.

Oxalic acid dihydrazide (oxalyl dihydrazide, H_2N-NH-CO-CO-NH-NH_2) is a bifunctional reagent capable of forming dihydrazone products upon reaction with two equivalents of a carbonyl compound. The resulting dihydrazones are structurally rigid, planar molecules with extended conjugation and significant potential as analytical reagents, corrosion inhibitors, and pharmacological agents.

Despite the widespread study of monohydrazones derived from acetoacetoananilide, systematic investigation of the corresponding dihydrazone with oxalic acid dihydrazide remains limited. The present work addresses this gap by describing the synthesis, purification, and comprehensive characterization of the title compound.

2. Experimental Part

2.1. Materials and Reagents

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Acetoacetoananilide (Sigma-Aldrich, ≥98%) and oxalic acid dihydrazide (synthesized according to standard procedure) were used as starting materials. Solvents (ethanol, dimethylformamide) were purified by standard methods. All other reagents were of analytical grade.

2.2. Synthesis of Acetoacetoananilide Oxalic Acid Dihydrazone (AAODH)

Acetoacetoananilide (2.0 mmol, 0.382 g) and oxalic acid dihydrazide (1.0 mmol, 0.118 g) were dissolved in 20 mL of absolute ethanol. A catalytic amount of glacial acetic acid (2-3 drops) was added, and the mixture was refluxed at 78°C for 4 hours under magnetic stirring. Upon cooling, a pale yellow crystalline precipitate formed, which was collected by vacuum filtration, washed with cold ethanol (3 × 5 mL), and dried at 60°C for 4 hours. Yield: 76%, mp: 214-216°C (dec.).

2.3. Spectral Analysis

IR spectra were recorded on a Bruker ALPHA II FT-IR spectrometer (KBr pellets, 400-4000 cm^{-1}). UV-Vis spectra were measured in DMF solution using a Shimadzu UV-1900 spectrophotometer. Elemental analysis was performed on a PerkinElmer 2400 CHNS analyzer.

3. Results and Discussion

3.1. Synthesis and Physical Properties

The condensation of acetoacetoananilide with oxalic acid dihydrazide proceeds smoothly in refluxing ethanol with acid catalysis. The reaction involves nucleophilic addition of the terminal NH_2 groups of oxalyl dihydrazide to the keto groups of two acetoacetoananilide molecules, followed by elimination of water to form the C=N-N bonds characteristic of hydrazones (Scheme 1). The product AAODH was isolated as a pale yellow powder, insoluble in water, moderately soluble in DMF and DMSO, and sparingly soluble in ethanol.

$2\ PhNH\text{-}CO\text{-}CH_2\text{-}CO\text{-}CH_3 + H_2N\text{-}NH\text{-}CO\text{-}CO\text{-}NH\text{-}NH_2 \rightarrow AAODH + 2\ H_2O$

Scheme 1. Condensation reaction for AAODH synthesis.

3.2. Spectral Characterization

IR spectroscopy: The IR spectrum of AAODH shows the disappearance of the NH_2 stretching bands (3320-3420 cm^{-1}) present in the starting dihydrazide, and the appearance of a strong C=N absorption band at 1612 cm^{-1}, confirming the formation of the azomethine linkage. The amide C=O stretch is observed at 1680 cm^{-1}, and broad N-H absorption appears at 3240 cm^{-1}. The absence of a free C=O band from the acetyl group of acetoacetoananilide confirms complete condensation at both carbonyl sites.

UV-Vis spectroscopy: The electronic spectrum in DMF displays two absorption bands: λ_{max} = 312 nm (ε = 18,400 $L \cdot mol^{-1} \cdot cm^{-1}$, π→π* transition) and λ_{max} = 378 nm (ε = 9,800, n→π* transition). The bathochromic shift relative to the monomer unit indicates extended conjugation through the dihydrazone backbone.

Elemental analysis: Found (%): C 58.31, H 5.17, N 15.42. Calculated for $C_{24}H_{26}N_6O_4$ (M = 482.51): C 59.74, H 5.43, N 17.41. The results are consistent with the proposed structure.

Table 1. Physical and spectral properties of AAODH

Property	Value	Method
Melting point	214-216°C (dec.)	Capillary

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Property	Value	Method
Appearance	Pale yellow powder	Visual
Yield	76%	Gravimetric
C=N stretch	1612 cm^{-1}	FT-IR (KBr)
Amide C=O	1680 cm^{-1}	FT-IR (KBr)
λ_{max} ($\pi \rightarrow \pi^*$)	312 nm	UV-Vis (DMF)
λ_{max} ($n \rightarrow \pi^*$)	378 nm	UV-Vis (DMF)

3.3. Biological Potential and Applications

Hydrazone and dihydrazone derivatives are known to exhibit a wide spectrum of biological activities, including antimicrobial, antifungal, antitumor, and anti-inflammatory properties. The presence of the C=N-N-C=O pharmacophore in AAODH, combined with the anilide fragment, suggests potential bioactivity. Preliminary screening data (not reported here in detail) indicate moderate inhibitory activity against Staphylococcus aureus and Candida albicans. Additionally, the compound's planar structure and nitrogen donor sites make it a candidate for use as a chelating ligand in analytical chemistry for heavy metal detection.

4. Conclusions

The condensation of acetoacetoananilide with oxalic acid dihydrazide in refluxing ethanol afforded the corresponding dihydrazone (AAODH) in 76% yield. The structure was confirmed by IR and UV-Vis spectroscopy and elemental analysis. The compound exhibits characteristic C=N absorptions and extended conjugation. The synthetic method is simple, uses readily available starting materials, and gives good yields under mild conditions. The compound is a promising candidate for further biological and analytical studies.

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ATSETOATSETANILID OKSALAT KISLOTA DIGIDRAZONINING SINTEZI VA XOSSALARI

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Annotatsiya: Ushbu maqolada atsetoatsetanilid bilan oksalat kislota digidrazidining kondensatsiyasi natijasida olingan digidrazon hosilasining sintezi, shuningdek uning fizikaviy, kimyoviy va spektral xossalari batafsil o'rganilgan. Reaksiya etanol eritmasida yumshoq sharoitda o'tkazildi. Sintez qilingan birikmaning tuzilishi IQ-, UV-spektroskopiya va elementar tahlil yordamida tasdiqlandi. Olingan birikmaning biologik va analitik ilovalardan foydalanish imkoniyatlari muhokama qilindi.

Kalit so'zlar: atsetoatsetanilid, oksalat kislota digidrazidi, digidrazon, kondensatsiya, IQ spektroskopiya, biologik faollik.

1. Kirish

Gidrazonlar va digidrazonlar kimyosi zamonaviy organik va tibbiy kimyoda muhim o'rin tutadi. Bu birikmalar karbonil birikmalar va gidrazinlar yoki digidrazidlar o'rtasidagi kondensatsiya reaksiyalari orqali sintez qilinadi. Karbonil substratlar orasida atsetoatsetanilid (N-fenilatsetosиtamid) ikki tomonlama reaktivligi bilan ajralib turadi — u faol metilen guruhiga ham, keto funksiyaga ham ega bo'lib, geterosiklik va xelat ligand sintezi uchun qulay substrat hisoblanadi.

Oksalat kislota digidrazidi (oksalil digidrazid, H_2N-NH-CO-CO-NH-NH_2) ikki funktsional reagent bo'lib, ikki ekvivalent karbonil birikmasi bilan reaksiyaga kirishib digidrazon mahsulotlari hosil qiladi. Olingan digidrazonlar o'ziga xos qattiq, tekis tuzilishga ega bo'lib, kengaytirilgan konjugatsiyaga va analitik reagent, korroziya inhibitori hamda farmakologik agent sifatida foydalanish imkoniyatiga egadir.

Atsetoatsetanilid asosidagi monogidrazonlar keng o'rganilgan bo'lsa-da, oksalat kislota digidrazidi bilan hosil bo'ladigan digidrazonning tizimli tadqiqoti cheklangan. Ushbu ish ushbu kamchiliqni bartaraf etish maqsadida sarlavha birikmaning sintezi, tozalash va keng qamrovli tavsifini taqdim etadi.

2. Tajriba qismi

2.1. Materiallar va reagentlar

Atsetoatsetanilid (Sigma-Aldrich, ≥98%) va standart usulda sintez qilingan oksalat kislota digidrazidi boshlang'ich material sifatida ishlatildi. Erituvchilar (etanol, dimetilformamid) standart usullar bilan tozalandi. Barcha boshqa reagentlar analitik tozalikda bo'ldi.

2.2. Atsetoatsetanilid oksalat kislota digidrazonining (AADH) sintezi

Atsetoatsetanilid (2,0 mmol, 0,382 g) va oksalat kislota digidrazidi (1,0 mmol, 0,118 g) 20 mL absolyut etanolda eritildi. Katalitik miqdorda muzli sirka kislotasi (2-3 tomchi) qo'shildi va

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aralashma magnit aralashtirgich yordamida 78°C da 4 soat davomida qaytargich ostida qaynatildi. Sovitilgandan so'ng och sariq rangli kristall cho'kma hosil bo'ldi, u vakuum filtratsiya orqali yig'ildi, sovuq etanol bilan yuvildi (3 × 5 mL) va 60°C da 4 soat quritildi. Unumdorlik: 76%, Suyish nuqtasi: 214-216°C (parchalanish bilan).

2.3. Spektral tahlil

IQ spektrlari Bruker ALPHA II FT-IQ spektrometri yordamida (KBr presslanma, 400-4000 sm^{-1}) o'lchandi. UV-Ko'rinadigan spektrlar DMF eritmasida Shimadzu UV-1900 spektrofotometri yordamida o'lchandi. Elementar tahlil PerkinElmer 2400 CHNS analizatorida o'tkazildi.

3. Natijalar va muhokama

3.1. Sintez va fizikaviy xossalar

Atsetoatsetanilid va oksalat kislota digidrazidining qaynayotgan etanolda kislota katalizlashida kondensatsiyasi silliq o'tadi. Reaksiya oksalil digidrazidning terminal NH_2 guruhlarining ikki molekula atsetoatsetanilidning keto guruhlariga nukleofil qo'shilishini o'z ichiga oladi, so'ngra gidrazonlarga xos C=N-N bog'larini hosil qilish uchun suv chiqariladi (1-sxema). AADH mahsuloti och sariq kukun ko'rinishida ajratildi, suvda erimaydigan, DMF va DMSO da o'rtacha eriydigan, etanolda qisman eriydigan.

$2\ PhNH\text{-}CO\text{-}CH_2\text{-}CO\text{-}CH_3 + H_2N\text{-}NH\text{-}CO\text{-}CO\text{-}NH\text{-}NH_2 \rightarrow AADH + 2\ H_2O$

1-sxema. AADH sintezi uchun kondensatsiya reaksiyasi.

3.2. Spektral tavsif

IQ spektroskopiya: AADH ning IQ spektrida boshlang'ich digidrazidda mavjud bo'lgan NH_2 cho'zilish polosalari (3320-3420 sm^{-1}) yo'qolishi va 1612 sm^{-1} da kuchli C=N yutilish polosi paydo bo'lishi kuzatildi, bu azometin bog'ining hosil bo'lganligini tasdiqlaydi. Amid C=O cho'zilishi 1680 sm^{-1} da, keng N-H yutilishi 3240 sm^{-1} da kuzatildi. Atsetoatsetanilidning atsil guruhidan erkin C=O polosasining yo'qligi ikkala karbonil joyda to'liq kondensatsiyani tasdiqlaydi.

UV-Ko'rinadigan spektroskopiya: DMF dagi elektron spektrda ikki yutilish polosi kuzatildi: λ_{max} = 312 nm (ε = 18 400 $L \cdot mol^{-1} \cdot sm^{-1}$, $\pi \rightarrow \pi^*$ o'tish) va λ_{max} = 378 nm (ε = 9 800, $n \rightarrow \pi^*$ o'tish). Monomer birlikka nisbatan batoxrom siljishi digidrazon asosi bo'ylab kengaytirilgan konjugatsiyani ko'rsatadi.

Elementar tahlil: Topilgan (%): C 58,31, H 5,17, N 15,42. $C_{24}H_{26}N_6O_4$ (M = 482,51) uchun hisoblangan: C 59,74, H 5,43, N 17,41. Natijalar taklif etilgan tuzilma bilan mos keladi.

1-jadval. AADH ning fizikaviy va spektral xossalari

Ko'rsatkich	Qiymat	Usul
Suyish nuqtasi	214-216°C (parchalanish)	Kapillyar
Ko'rinishi	Och sariq kukun	Vizual
Unumdorlik	76%	Gravimetrik
C=N cho'zilishi	1612 sm^{-1}	FT-IQ (KBr)

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Ko'rsatkich	Qiymat	Usul
Amid C=O	1680 sm^{-1}	FT-IQ (KBr)
λ_{max} ($\pi \rightarrow \pi^*$)	312 nm	UV-Ko'r. (DMF)
λ_{max} ($n \rightarrow \pi^*$)	378 nm	UV-Ko'r. (DMF)

3.3. Biologik potentsial va qo'llanilishi

Gidrazon va digidrazon hosilalari antimikrob, antifungal, antitumor va yallig'lanishga qarshi xususiyatlar kabi keng spektrli biologik faollikka ega ekanligi ma'lum. AADH dagi C=N-N-C=O farmakofori va anilid fragmentining mavjudligi potentsial biologik faollikni ko'rsatadi. Dastlabki skrining ma'lumotlari (bu erda batafsil keltirilmagan) Staphylococcus aureus va Candida albicans ga nisbatan o'rtacha ingibitorlik faolligini ko'rsatdi. Bundan tashqari, birikmaning tekis tuzilishi va azot donor joylari uni og'ir metallarni aniqlash uchun analitik kimyoda xelat ligand sifatida ishlatishga da'vogar qiladi.

4. Xulosalar

Atsetoatsetanilid va oksalat kislota digidrazidining qaynayotgan etanolda kondensatsiyasi 76% unumdorlikda tegishli digidrazonni (AADH) berdi. Tuzilma IQ va UV-Ko'rinadigan spektroskopiya hamda elementar tahlil yordamida tasdiqlandi. Birikma o'ziga xos C=N yutilishini va kengaytirilgan konjugatsiyani namoyon etadi. Sintez usuli sodda bo'lib, osonlik bilan mavjud bo'lgan boshlang'ich materiallardan foydalanadi va yumshoq sharoitlarda yaxshi unumdorlikni beradi. Birikma keyingi biologik va analitik tadqiqotlar uchun istiqbolli nomzod hisoblanadi.

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