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A highly selective and sensitive Tb^{3+}-acetylacetone photo probe for the assessment of acetazolamide in pharmaceutical and serum samples

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ABSTRACT

A novel, simple, sensitive and selective spectrofluorimetric method was developed for the determination of Acetazolamide in pharmaceutical tablets and serum samples using photo probe Tb^{3+}-ACAC. The Acetazolamide can remarkably quench the luminescence intensity of Tb^{3+}-ACAC complex in DMSO at pH 6.8 and $\lambda_{ex} = 350$ nm. The quenching of luminescence intensity of Tb^{3+}-ACAC complex especially the electrical band at $\lambda_{em} = 545$ nm is used for the assessment of Acetazolamide in the pharmaceutical tablet and serum samples. The dynamic range found for the determination of Acetazolamide concentration is 4.49×10^{-9}–1.28×10^{-7} mol L^{-1}, and the limit of detection (LOD) and limit of quantification (LOQ) are (4.0×10^{-9} and 1.21×10^{-8}) mol L^{-1}, respectively.

1. Introduction

Acetazolamide Fig. 1 is a carbonic anhydrase inhibitor, chemically named according to the IUPAC system as 5-acetamido-1, 3, 4-thiadiazole-2- sulphonamide; N-(5- sulphamoyl-1, 3, 4, thiadiazole-2-yl) acetamide. It acts on the central nervous system to retard the abnormal, paroxysmal, and excessive discharge from CNS neurons [1] used clinically in the management of glaucoma [2]. It is also used, either alone or in association with other antiepileptics, for the treatment of various forms of epilepsy, and it is the most frequently used drug for the prophylaxis of high-altitude disorders [3]. The pharmacokinetics of acetazolamide is well documented in healthy subjects. The adsorption of drug is fast, reaching peak plasma concentrations approximately 1–3 h after oral administration. About 80% of the drug is excreted by tubular secretion of the anionic species, and 70–90% of the administered dose is recovered unchanged within 24 h [4]. Various methods were reported for its determination in pharmaceutical preparations and biological fluids including: spectrophotometry [5–7], a potentiometric and spectrophotometric study on acid–base equilibria in ethanol aqueous solution of acetazolamide and related compounds [8], HNMR [9], electrochemical methods: such as joint determination of acetazolamide in human serum by differential pulse polarography [10], reductive amperometric determination of acetazolamide at a sessile mercury drop electrode using flow injection analysis [11]. Determination of acetazolamide in biological fluids by sol–gel optical biosensor [12], polarography [13], enzymatic assay [14], electron-capture GLC [15], molecular imprinted polymer (MIP) [16], carbon paste electrode modified with gold nanoparticle [17] and high-performance liquid chromatography [18–25], a high performance liquid chromatographic assay for the quantitation of acetazolamide in both tablet and injection form [26], Liquid chromatography/tandem mass spectrometry (LC-MS/MS) [27]. Luminescent optical sensors lanthanide complexes have more advantages over the present ones; optical sensor has high stability and durability. The sensor can provide constant signal response for 2 years which is a 24-fold better stability compared to the life time warranted for the chromatographic and colorimetric methods [18–26]. Sensor is stable over all measurements which prevent the source of error in the measurement process and it gives a low standard deviation values. In this work, Acetazolamide was determined by spectrofluorimetric method by using photo probe of [Tb^{3+}-ACAC]. The method depend on the quenching of the fluorescence intensity of photo probe [Tb^{3+}-ACAC] by different concentrations of acetazolamide.

2. Experimental

2.1. Materials

Pure standard Acetazolamide supplied by the National Organization for Drug control and Research (Giza, Egypt). Pharmaceutical preparation of Cidamex tablets containing 250 mg of Acetazolamide produced by, Chemical industrial development CID, Giza, A.R.E is purchased from local market.

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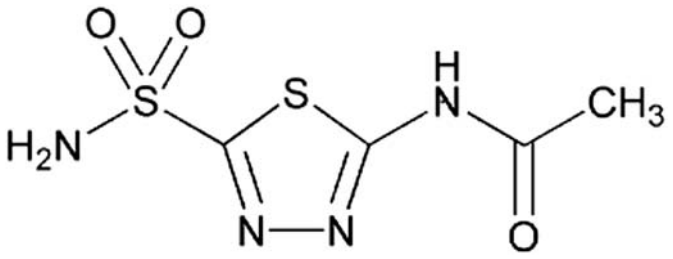

Fig. 1. Structure of acetazolamide: *N*-(5-sulfamoyl-1,3,4-thiadiazol-2-yl)- acetamide.

2.2. Reagents and Solutions

(10^{-2} mol L^{-1}) was freshly prepared by dissolving 0.0555 g of Acetazolamide in 25 mL DMSO. More diluted solution (1.0×10^{-4} mol L^{-1}) was prepared by appropriate dilution with DMSO. Stock and working solutions are stored at 2–8 °C when are not in use.

A stock solution of (1.0×10^{-2} mol L^{-1}) Terbium (III) nitrate $Tb(NO_3)_3 \cdot 5H_2O$ was prepared by dissolving 0.0109 g $Tb(NO_3)_3 \cdot 5H_2O$ (delivered from Aldrich - 99.99%) in ethanol then transferred to a 25 mL volumetric flask, and completed to the mark with absolute ethanol.

The working solution of $Tb(NO_3)_3$ of 1.0×10^{-3} mol L^{-1} was obtained by appropriate dilution with DMSO. A stock solution of acetylacetone (1.0×10^{-2} mol L^{-1}) was prepared by transferring of 0.11 mL of acetylacetone to 100 mL volumetric flask, then completed to the mark with absolute ethanol. The working solution of acetylacetone of 1.0×10^{-3} mol L^{-1} was obtained by appropriate dilution with DMSO.

2.3. Apparatus

The absorption spectra were recorded with a double beam PerkinElmer Lambda 25 UV–Visible spectrophotometer fitted with a tungsten halogen lamp for operation in the visible range and a deuterium lamp for operation in the UV range. All luminescence measurements were recorded with a Meslo-PN (222-263000) Thermo Scientific Lumina fluorescence Spectrometer in the range (190–900 nm). The pH was measured using a pHs - Jenway 3330 research pH meter. The separation of protein from samples was carried out by centrifuging of sample for 15 min and 4000 rpm.

2.4. General Procedure

2.4.1. Preparation of Lanthanide Complex Tb^{3+}-ACAC Solution

The Tb^{3+}-ACAC complex was prepared according to the previous reported methods [28–45] in which, to 10 mL measuring flasks, solutions were added in the following order: 1.0 mL of 1.0×10^{-3} mol L^{-1} Tb $(NO_3)_3$ solution and 3 mL of 1.0×10^{-3} mol L^{-1} Acetyl Acetone solution to give 1.0×10^{-4} mol L^{-1} of $Tb(NO_3)_3$ and 3.0×10^{-4} mol L^{-1} of ACAC. The mixture was diluted to the mark with DMSO. The above procedure was used for the subsequent measurements of absorption, emission spectra and effect of pH and solvents.

The luminescence intensity was measured at $\lambda_{ex}/\lambda_{em} = 350/545$ nm.

2.4.2. Calibration Curve

After the preparation of the different standard solutions of Acetazolamide in DMSO as described above, the photo probe Tb^{3+}-ACAC was mixed with each standard solution of Acetazolamide in the cell of the spectrofluorimetric device, then the luminescence spectrum was measured at the selected excitation wavelength $\lambda_{ex} = 350$ nm.

2.5. Determination of Acetazolamide in Pharmaceutical Preparations

Five tablets of pharmaceutical formulation (Cidamex) were carefully weighed and ground to finely divided powders. Accurate weights equivalent to 1.5 mg was dissolved in 50 mL DMSO and mixed well and filtered up using 12 mm filter papers. The concentration of the drug was determined by using different concentrations from the corresponding calibration graph.

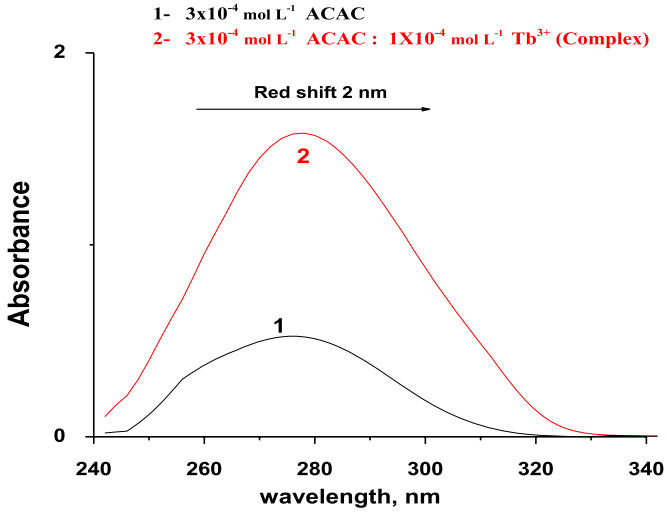

Fig. 2. The absorption spectrum of 3.0×10^{-4} mol L^{-1} of ACAC and Tb^{3+}-ACAC complex.

2.6. Determination of Acetazolamide in Serum Solution

3 mL of trichloro acetic acid was added to 1.0 mL blood of a real health volunteers and the solution was centrifuged for 15 min at 4000 rpm to remove proteins, then 100 μL of the serum was added to 0.1 mL of Tb^{3+}-ACAC solution 10 mL measuring flask and complete to the mark with DMSO and the pH was adjusted to 6.8. The luminescence intensity of the test solution was measured before and after addition of Tb^{3+}-ACAC photo probe. The change in the luminescence intensity was used for determination of Acetazolamide in serum sample.

3. Result and Discussion

3.1. Absorption Spectra

The absorption spectrum of 3.0×10^{-4} mol L^{-1} of ACAC shows a π → π* transition band at 276 nm, Fig. 2. Upon addition of 1.0×10^{-4} mol L^{-1} of Tb^{3+} ion into ACAC, a red shift was observed in the band by 2 nm and the absorbance is also enhanced which indicates that acetyl acetone can form a complex with Tb^{3+} ion.

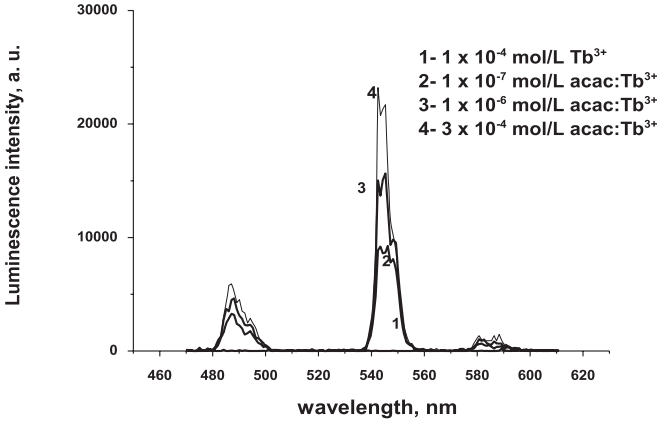

Fig. 3. The luminescence emission spectra of Tb^{3+} in different concentrations of ACAC.