

solvent) was injected rapidly into a conical test tube containing aqueous solution of MR. Influence variables such as volume of chloroform and methanol, pH and ionic strength, extraction time and centrifugation time and speed were investigated. Then significant variables were optimized by using a Box-Behnken design (BBD) [5] combined with desirability function (DF). The optimized conditions (extractant solvent: 100 μL of chloroform, disperser solvent: 1.3 mL of ethanol, pH: 4 and 4 w/v NaCl) resulted in a linear calibration graph in the range of 0.005–100 mg mL^{-1} of MR in initial solution with $R^2 = 0.995$ ($n = 5$). The Limits of detection and quantification were 0.001 and 0.008 mg mL^{-1} , respectively. Finally, the DLLME method was applied for determination of MR in different water samples and relative standard deviation (RSD) for spiked 0.1 and 0.2 mg mL^{-1} of MR in water samples were less than 4% ($n = 5$).

Keywords: Methyl red, Dispersive liquid–liquid microextraction, Box-Behnken design, Desirability function

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Study mephnamic acid delivery in use hydrogel nanocomposite based chitosan

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A hydrogel nanocomposite was prepared base on chitosan via graft acrylic acid (AA) and maleic acid (MA) for the controlled delivery of mephnamic acid. Powdered sample of hydrogel nanocomposite (0.01 g) with 250–350 μm particle sizes, was accurately weighted and immersed in basic solution of mephnamic acid (0.1 g dissolved in 100 cc distilled water) at 25 $^{\circ}\text{C}$ for 72 h. Hydrogel nanocomposite loaded with drug dried under vacuum at 50 $^{\circ}\text{C}$. Drug release from the sample was studied in simulated gastric (pH 1.6) and intestinal (pH 7.4). At regular intervals of time, aliquot sample was with drawn and analyzed for drug by uv-vis spectrophotometry at the fixed wavelength (λ_{max}) of 235 nm. The drug release data showed: that was release was higher at pH 7.4 than at pH 1.6. A maximum release (65%) of mephnamic acid was absorbed at pH 7.4, whereas 7.9% was released at pH 1.6.

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Preconcentration and determination the traces of ciprofloxacin by solid phase microextraction with functionalized silica with nanomagnetic Fe_3O_4 by UV-Vis spectrophotometry

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In the present age the pharmaceutical application continues to increase and this results in the environmental pollution. As a result, pharmaceutical control is a common technique in many labs. This project is on the improvement of the procedure to determine a small amount of ciprofloxacin in bio-environmental sample [1]. Regarding the fact that each method requires preliminary measures to preconcentration and prepare analyte prior to measurement and recognition. In recent study, preconcentration of samples containing trace of ciprofloxacin (as antibiotic) based on solid phase microextraction method using functionalized silica with nanomagnetic particle Fe_3O_4 was proposed which acted as solid phase adsorbent [2]. In addition UV-Vis spectrophotometry was carried out to determine the amount of ciprofloxacin [3]. The spectral region between 250 and 400 nm was tested that the wavelength of maximum absorption (λ_{max}) was 275 nm. Results show using this procedure increased the concentration factor by 68/38. Also, the optimum conditions of various parameters such as the kind (nature) of organic solvent, the pH of solution, amount of nanomagnetic particles, time, the volume of aqous and organic phases, the rate of centrifugation, temperature and salt effect were investigated. By plotting the calibration curve of analysis, under the optimum conditions, the dynamic range for was obtained between 0/1 – 0/4 ppm, LOD and (RSD%) were 0/0060 ppm and 1/878 respectively. This method was successfully applied to analyze ciprofloxacin in hospital waste water.

Key words: Ciprofloxacin, nanomagnetic, solid phase microextraction, spectrophotometry

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The theoretical and experimental ^1H NMR study of some beta-diketones (A DFT calculations)

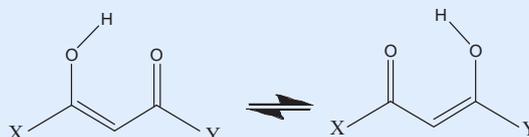
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Proton having a small mass and radius shows various characteristic phenomena such as hydrogen bonding. Recently, many researches are devoted to very strong hydrogen bonds, because of their important role in biochemical reactions, and enzyme catalysis as transition state [1]. It is well known that the cis-enol form of β -diketones is characterized by a strong intramolecular hydrogen bond [2]. The NMR and vibrational spectroscopy techniques have been intensively

used to study the hydrogen bond strength and also keto–enol equilibrium in these compounds [3].

In the present work, we measure the theoretical and experimental (in several solvents at room temperature) ^1H NMR chemical shift for the symmetrical β -diketones, such as 2,4-pentanedione (AA) ($X = \text{CH}_3$, $Y = \text{CH}_3$), 1,3-diphenyl-1,3-propanedione (DBM) ($X = \text{Ph}$, $Y = \text{Ph}$), hexafluoro-acetylacetone (HFAA), ($X = \text{CF}_3$, $Y = \text{CF}_3$), and assymetrical compound like 1-Phenyl-1, 3-butanedione (BA) ($X = \text{Ph}$, $Y = \text{CH}_3$), 4,4-Dimethyl-1-phenylpentane-1,3-dione, (DMPD) ($X = \text{Ph}$, $Y = t\text{-butyl}$), and 1,1,1-trifluoro-2,4-pentanedione (TFAA) ($X = \text{CF}_3$, $Y = \text{CH}_3$), to investigate hydrogen bonding strength. Some chelated cis-enol form could be considered for the mentioned compounds. The theoretical results have been done by means of DFT calculations at B3LYP level with different basis sets and regression coefficient applied to find the best basis set(s) which is in more agreement to experimental data.



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Preconcentration of indium by dispersive liquid-liquid microextraction and its determination by spectrophotometry in real samples

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Indium is an important element in semiconductor industry and is used in high technology applications such as LCD production [1]. Indium enters into the composition of many rock – forming mineral and its overall abundance in the earth crust is estimated to be $0.11 \mu\text{g.g}^{-1}$ [2]. Different methods such as spectrophotometry [3-4], graphite furnace atomic absorption spectrometry[5-6] and polarography[7] has been used for determination of indium. However a very few works has been reported using microextraction techniques for indium so far. A highly sensitive method based on dispersive liquid – liquid microextraction was applied for preconcentration and determination of indium in real samples. In this study dithizone was used as a reagent to form a 1: 3 complex which upon dispersing process. The solution is centrifuged and the absorbance of the complex is measured at 510 nm by spectrophotometry using a micro cell. The effects of different parameters such as pH, volume and type of dispersive and extraction solvents, the rate and time of centrifuge and salt concentration on the extraction process were studied. At the optimum conditions, the LOD and RSD for determination were 2 ppb and 4.2% respectively. The calibration curve was linear in the range of 3.5 to 65 ng.ml^{-1} and the enrichment factor based on the slopes ratio after and before preconcentration was 85. The interference effects of different ions in indium determination was also investigated. This method was applied for determination of indium in real samples such as sea water, coal and ore.

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Supramolecular–based dispersive liquid–liquid microextraction for determination of copper in soil, water and vegetable samples

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Copper is an essential element whose role is complex in many body functions such as hemoglobin synthesis, normal function of the central nervous system, and oxidative phosphorylation [1]. It is an important nutrient for plants and animals and plays a significant role as a cofactor in at least 30 important enzymes [2]. Many different microextraction procedures combined with spectrometric determinations have been developed for copper[3] Supramolecular–based dispersive liquid–liquid microextraction was proposed for separation and preconcentration of trace quantities of copper as prior step to its determination by flame atomic absorption spectrometry for the first time. The copper ions are micro–extracted with coacervates composed of reverse micelles formed using decanoic acid and dispersed in tetrahydrofuran–water mixtures[4]. The method involves the partitioning of the metal chelates, produced from the reaction of Cu^{2+} with cupferron in acidic medium and combination of DLLME with coacervation–based microextraction. It combines the advantages of dispersive liquid–liquid microextraction with those based on coacervation and reverse micelles[5]. All the critical parameters affecting the analytical performance were studied. Under the optimum conditions, the enhancement factor was 30. The detection limit and precision (RSD) were $1.6 \mu\text{g L}^{-1}$ and 3.2% ($200 \mu\text{g L}^{-1}$) respectively. The accuracy of the developed method was evaluated by analyzing a certified reference material and applied successfully to the analysis of water and vegetables samples.

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