Colorimetric Determination Of Thioctic Acid With 2, 2[1]-Bipyridine And Ferric Chloride

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Abstract

The oxidative coupling of the proposed method is simple, rapid and sensitive with reasonable precision and accuracy. The precision of the method was found by analyzing a set of eight solutions, each containing a final concentration value approximately in the middle of the Beer's law range. The percent relative standard deviation in this method is presented in table-2. The accuracy of the method was determined by taking different known amounts (with in Beer's law limits) of the drug and analyzing them by proposed method. The results are given in table – 3. In the determination of Thioctic acid the excipients usually present in formulations (glucose, starch, sodium hexaphosphate and some vitamins) and the other antioxidants and antidiabetics did not interfere.

Keywords: Thioctic acid, Spectrophotometer, 2,2[1]-bipyridine, Ferric chloride,

Introduction

A very few physico-chemical methods have appeared in the literature for the determination of Thioctic Acid in bulk and pharmaceutical formulations. The literature suggested and reported only a few chromatographic techniques like Gas chromatographic methods with flame ionization detection and flame photometric detection[4], High-performance liquid chromatographic methods[5] with ultraviolet, fluorescence and electrochemical detection, spectrophotometric using palladium(II) chloride[9], Reversed phase liquid chromatography, LC-MS/MS with atmospheric pressure chemical ionization and electro spray ionization interfaces, Tandem mass spectrometry, Electrophoresis, Extractive spectrophotometry and potentiometric techniques[3]. The analytically important functional groups of Thioctic acid[1] are not fully exploited for designing suitable spectrophotometric methods for the determination of Thioctic acid[6]. Hence the need arises to develop certain sensitive, precise, accurate and flexible visible spectrophotometric methods, which prompted the authors to choose Thioctic acid[7] for the investigation based on various chemical reactions, by exploiting various functional groups from the structure.

These methods are based on the reaction of Thioctic acid with 2,2[1]-bipyridine and compounds like ferric chloride and to produce colored species of reasonable stability, paving the possibility for spectrophotometric determination of α -LA in its bulk from and pharmaceutical formulations.

Materials And Methods

Preparation of reagents:

I) Thioctic acid (0.25mg/ml): The stock solution (0.25mg/ml) of Lipoic Acid was prepared by dissolving 100mg of the drug in 400ml of distilled water to get a clear solution. A portion of this stock solution was diluted to get the working standard solutions of concentration 100 μg/ml.

2) 2, 2[1]-Bipyridine*:* 0.01M aqueous Solution.

3) Ferric chloride solution (0.7%):700mg of analytical grade ferric chloride in 100ml of 0.5N HCL.
4) Orthophosphoric Acid: 0.2M

All the other chemical reagents were of analytical grade

Instrumentation: A systronics double beam UV visible spectrophotometer 2201 with 1cm matched quartz cells was used for all spectral and absorbance measurements. A systronics digital P^H meter was used for all P^H instruments.

Absorption spectra : The absorption spectrum of the colored species obtained by reaction of the Thioctic Acid with 2,2-bipyridine -$FeCl_3$ was scanned over the wavelength region 360 – 600 nm against a reagent blank and the data is graphically represented in Figure-5.1. The absorption curves show a maximum at 520nm against the corresponding reagent blank.

Procedure : An aliquotes of standard Thioctic acid solution (100µg/ml) ranging from 0.4 to 2.0ml are taken into a series of 10ml graduated test tubes and aqueous solutions of 2, 2^1-Bipyridine (1.0ml), $FeCl_3$ (1ml) were added. And the resulting solution is heated for 15 minutes at 100^0c and finally 2ml of orthophosphoric acid is added. The volume was made upto 10ml with distilled water and the obsorbance of the orange colored chromogen was measured at 520nm against the reagent blank. The amount of Thioctic Acid was computed from the Beer-Lambert's plot.

For dosage forms: A quantity equivalent to 10mg of Thioctic Acid was dissolve in 100ml of water and filtered if necessary, so as to get 100µg/ml the recommended procedure was them followed.

Results And Discussion

Comparison of the results incorporated in Tables 1 – 4 reveal that the proposed method is simple, rapid and sensitive with reasonable precision and accuracy.

Chemistry involved: In this method the oxidative coupling of Thioctic acid with in the presence of Fe (III).Under the reaction conditions MBTH on oxidation with Fe(III) loses two electrons and one portion forming an electrophilic attack on the most nucleophilic site of Thioctic acid.

Table-1. Optical characteristics

Concentration range(µg/ml) (or) Beer's law limit	4.0 to 20.0
* Regression equation	A=0.1369-0.0023C
Correlation coefficient	0.9989
Molar absorptivity (1-mole $^{-1}$ cm^{-1})	$2.55*10^4$
Sandell's sensitivity (µg / cm^2 / 0.001 absorbance unit)	0.0083
Optimum photometric range (µg/ml)	5.2 to 16.8

*Found in this work; it must be determined independently by users of the method.

Table 2: Precision Of The Method

Compound	%RSD**	%Range of errors confidence limit	
		0.05 level	0.01 level
Thioctic Acid	1.42	±1.1873	±1.7566

** For six replicates.

Table 3: Accuracy Of The Method

Amount of Thioctic acid		% Error
Taken	Found	
450	443.0	1.55

TABLE 4.4
Assay Of Formulations And % Recovery Data*

Sample	Labeled amount [mg]	**Amount found(mg) in method		*Recovery proposed method
		Proposed	Reported	
Tablet	150	148.2	147.2	98.8
Capsule	150	147.4	146.9	98.3
Capsule	300	296.3	290.1	98.8
Capsule	450	443.7	440.1	98.6

** After adding 5 mg of drug.
** mean of duplicate determination

Conclusion

The proposed method is simple, rapid and sensitive with reasonable precision and accuracy and it is useful for the determination of Thioctic acid in bulk samples, pharmaceutical preparations and biological fluids.

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