

SPECTROPHOTOMETRIC METHODS FOR THE DETERMINATION OF LIPOIC ACID BY USING 1,10-PHENANTHRENE 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 Lipoic acid the excipients usually present in formulations (glucose, starch, sodium hexa phosphate and some vitamins) and the other antioxidants and antidiabetics did not interfere.

Keywords: Lipoic acid, Spectrophotometer, 1,10-phenanthrene, Ferric chloride, o-phosphoric acid.

INTRODUCTION

A very few physico-chemical methods have appeared in the literature for the determination of Alpha-Lipoic Acid (α -LA) 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, High-performance liquid chromatographic methods with ultraviolet, fluorescence and electrochemical detection, spectrophotometric using palladium(II) chloride, 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. The analytically important functional groups of α -LA are not fully exploited for designing suitable spectrophotometric methods for the determination of α -LA. Hence the need arises to develop certain sensitive, precise,

accurate and flexible visible spectrophotometric methods, which prompted the authors to choose α -LA for the investigation based on various chemical reactions, by exploiting various functional groups from the structure.

These methods are based on the reaction of α -LA with 1,10-phenanthrene and compounds like ferric chloride and o-phosphoric acid to produce colored species of reasonable stability, paving the possibility for spectrophotometric determination of α -LA in its bulk form and pharmaceutical formulations.

MATERIALS AND METHODS

Preparation of reagents :

1) Lipoic 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 μ g/ml.

2) Ferric chloride (0.27%): 270mg of analytical grade FeCl_3 is dissolved in 100 ml of distilled water.

3) 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 Lipoic Acid with 1,10-phenanthrene, FeCl_3 , orthophosphoric acid 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 515nm. against the corresponding reagent blank.

Procedure : An aliquotes of standard lipoicacid solution (100 μ g/ml) ranging from 0.4 to 2.0ml are taken into a series of 10ml graduated test tubes and aqueous solutions of 1,10 phenanthrene (1.0-1.5ml), FeCl_3 (1.0 –1.5ml) and orthophosphoric acid (2.0-2.5ml) were added. The solution was kept aside for 10minites and solutions were stir occasionally. The

solutions were finally made up to the mark with distilled water and absorbance of the green colored solution was measured at 515nm against the corresponding reagent blank.

For dosage forms: A quantity equivalent to 10mg of Lipoic Acid was dissolve in 100ml of water and filtered if necessary, so as to get 100 μ g/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 oxidation of ferric ions to ferrous ions and then the ferrous ions react with 1,10-phenanthrene and finally with the Lipoic acid .resulting in a solution is then estimated using spectrophotometer.

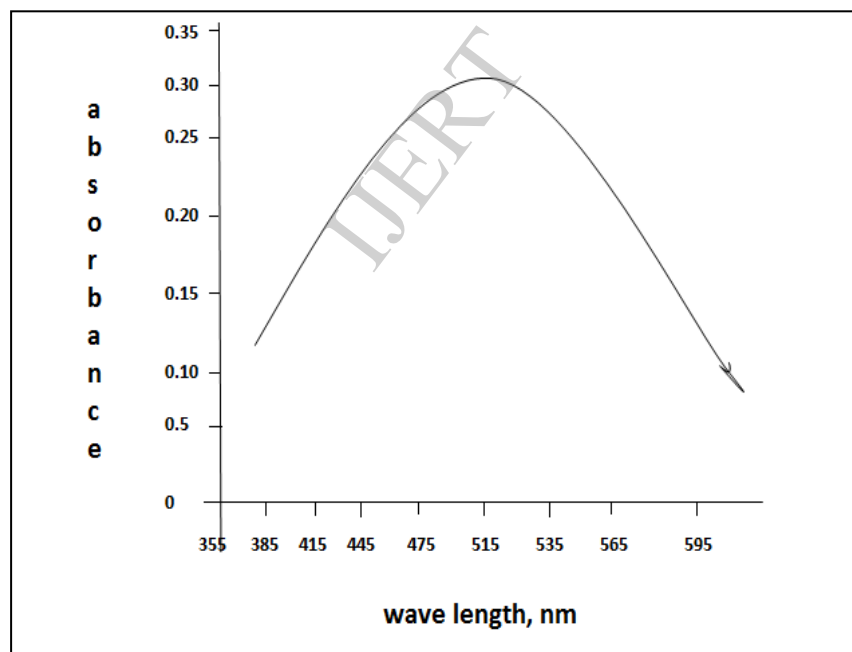


TABLE-1. OPTICAL CHARACTERISTICS

Concentration range($\mu\text{g/ml}$) (or) Beer's law limit	4 - 20
* Regression equation	$A = 1.605 - 0.0193C$
Correlation coefficient	998.7
Molar absorptivity ($1\text{-mole}^{-1} \text{cm}^{-1}$)	3.01×10^4
Sandell's sensitivity ($\mu\text{g} / \text{cm}^2 / 0.001$ absorbance unit)	0.0068
Optimum photometric range ($\mu\text{g/ml}$)	10.1 – 31.6

*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
Lipoic acid	1.02	1.087	1.608

TABLE 3: ACCURACY OF THE METHOD

Amount of Lipoic acid (μg)		% Error
Taken	Found	
450	439.7	2.28

TABLE-4. ASSAY OF FORMULATIONS AND % RECOVERY DATA*

Sample	Labeled amount	**Amount found(μg) in method		*Recovery proposed method
		Proposed	Reported	
tablet	150	148.2	147.2	1.2
Capsule	150	147.4	146.9	1.73
Capsule	300	296.3	290.1	1.23
Capsule	450	443.7	440.1	1.4

* Each result is an average of three determinations ** After adding 5 mg of drug.

CONCLUSION

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

REFERENCES

1. A review with 356 ref. On the U.V. Visible spectrophotometric analysis of antibiotics. M. Zoltan Dinya, J. Ferenc sztariscskai Drugs pharm. Sci., 27, 19(1986).
2. W.H.Unterman 'Colorimetric methods for determining penicillin's, streptomycin, tetracycline's (99 ref). Antibiotic, 7, 1112(1962).
3. Analytical profiles of drug substances', Ed. Klaus Florey, vol.9, 583(1980) and 4, (1975).

4. Review of various methods used in the determination of rifampicin, streptomycin.
R.codony saleedo, M. Marti pallares circ. Farm., 40 , 341(1982).
5. M. Llana, V.Gerona, J.De bolos, M.Castillo review of analytical methods for penicillin's cienc. Ind. Farm. 4, 13(1985).
6. K.C. Agrawal and B.N. Dutta 'assay methods far antibiotics' J. Instu. Chem. India, 33, 117(1961).
7. J. M. T. Hamilton miller. J.T. Smith and r. Knox J. Pharm. Pharmacol., 25, 81(1963).
8. M. D. Patter gill and D. E. Sands J. Chem. Edvc, 58, 244(1979).
9. AOL: <http://search.aol.com/cat.adp?id=168&layer=&from=subcats>
10. Family Village: <http://www.familyvillage.wisc.edu/specific.htm>
11. Google: <http://directory.google.com/Top/Health/ConditionsandJDiseases/>
12. Med Help International: <http://www.medhelp.Org/HealthT0pics/A.html>
13. Open Directory Project: http://dmoz.org/Health/Conditions_and_Diseases/