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VISIBLE SPECTROPHOTOMETRIC ESTIMATION OF ACEBROPHYLLINE IN BULK AND CAPSULE FORMULATION

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ABSTRACT

Two simple, sensitive and accurate spectrophotometric methods have been developed for the estimation of Acebrophylline in bulk and in pharmaceutical formulations (Method A & Method B). Method A is based on the formation of orange red colored chromgen with ferric chloride in the presence of 2,22 -bipyridyl which obeys beer's law in the concentration range of 10-100 µg/ml exhibiting maximum absorption at 522nm. Method B is based on the diazotization of Acebrophylline with nitrous acid. The diazonium salt formed was then coupled with â-naphthol which shows maximum absorption at 456nm (yellow colored chromogen). It obeys Beer's law in the concentration ranging from 50-300 µg/ml. The methods were extended to capsule formulation and there was no interference from excipients and diluents. These methods have been statistically validated and are found to be precise and accurate.

Keywords: *Acebrophylline (ACE), Ferric chloride, 2,22 -bipyridyl, diazotization, â-naphthol.*

INTRODUCTION

Acebrophylline is chemically 4-[(2-amino-3,5-dibromophenyl)methylamino]cyclohexan-1-ol, compound with 2-(1,3-dimethyl-2,6-dioxopurin-7-yl)acetic acid (Fig.1) and is used in the treatment of chronic obstructive pulmonary disease and bronchial asthma[1-4]. No method of estimation for acebrophylline in bulk and formulation has been reported so far. All the measurements were made using Shimadzu UV Visible Spectrophotometer with 1mm matched quartz cells.

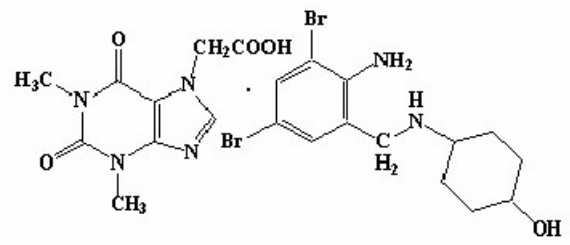

Fig.1 : *Acebrophylline*

EXPERIMENTAL

Reagents

Ferric chloride (0.03M), 2,22 -bipyridyl (0.02M), Hydrochloric acid (1M), Sodium nitrite (3%W/V), Sodium hydroxide (1M), â-naphthol (0.2%W/V). All the reagents used were of analytical grade.

Preparation of standard stock solution

An accurately weighed amount of 100mg of ACE was taken in a 100ml volumetric flask and dissolved in 25ml of distilled water and then made up to volume with distilled water.

Preparation of sample solution

Twenty capsules were weighed accurately and then emptied. The empty shells were reweighed and the powder was mixed uniformly. The powder equivalent to 100mg of acebrophylline was taken in a 100ml volumetric flask and dissolved in 25ml of distilled water and then made up to volume with the same distilled water. The solution was then filtered, the first few ml of the filtrate was discarded and remaining solution was used for the analysis.

ASSAY PROCEDURE

METHOD A

Aliquots of standard stock solution, 0.5-2.5ml were transferred to a series of 25ml volumetric flasks. To each flask 2ml of ferric chloride and 3ml of 2,22 -bipyridyl were added and kept in a water bath for 15min and cooled to room temperature. The volume was then made up with distilled water and the absorbance of the orange red colored chromogen was measured at 522nm against the reagent blank[5]. The amount of ACE was computed from the calibration curve obtained by plotting concentration versus absorbance.

METHOD B

Aliquots of standard stock solution, 1.25-7.5ml were transferred to a series of 25ml volumetric flasks. To each flask 1ml of 1M Hydrochloric acid, 2ml of 3% Sodium nitrite, 3ml of 1M Sodium hydroxide followed by 2ml of 0.2% â-naphthol were added. The volume was then made up with distilled water and the absorbance was measured at 456 nm against the reagent blank[6-7]. The amount of ACE was computed from the calibration curve obtained by plotting concentration versus absorbance.

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SAMPLE ANALYSIS

Pharmaceutical formulation of ACE was successfully analyzed by the proposed methods. Appropriate aliquots were subjected to the above methods and the amount of ACE was determined from the calibration curves.

RESULTS AND DISCUSSION

The optical characteristics such as RSD, regression equation, correlation coefficient, slope and intercept for the two methods were calculated and the results are summarized in Table 1. To evaluate the validity and reproducibility of the methods, recovery studies were carried out by adding a known amount of pure drug to previously analyzed capsules powder sample and re-analyzed. The results obtained are presented in Table 2. Interference studies revealed that the excipients and additives did not interfere. Hence these methods are most economic, simple, sensitive and accurate and can be used for the routine determination of ACE in pharmaceutical preparations.

Table 1: *Optical Characterestics of Acebrophylline*

PARAMETERS	METHOD A	METHOD B
λ_{max} (nm) Beer's law limits (µg/ml)	522 10-180	455 60-380
Molar absorptivity (l.mol^{-1}.cm^{-1})	6.67×10^{4}	1.492×10^{4}
Sandell's sensitivity (µg.cm^{-2}/0.001 abs unit)	0.00924	0.04131
Slope (m) Intercept (c)	0.010128 0.003048	0.00236 0.00514
Regression equation (y = mx + c)	0.010128x+0.003048	0.00236x+0.00514
Correlation coefficient (r)	0.9999	0.9999
Relative standard deviation	0.00951	0.00216

Table 2: *Assay and Recovery of Acebrophylline and its Formulation*

FORMULATION	LABEL CLAIM (mg)	AMOUNT FOUND BY PROPOSED METHOD* (mg)		% RECOVERY BY THE PROPOSED METHOD	
		METHOD A	METHOD B	METHOD A	METHOD B
Capsule	100	99.79	100.49	99.5%	100.08%

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