Asian Journal of Chemistry Vol. 16, Nos. 3–4 (2004), 1956–1958

NOTE

Comparative Study of UV Spectrophotometric Methods for the Determination of Ethamsylate in Bulk and Pharmaceutical Formulations

R. NAGESHWARA RAO*, P. NAGARAJU, C. SRINIVASULU,
P. JITENDRA KUMAR, V. SURESH and D. SIREESHA
Department of Pharmaceutical Chemistry, College of Pharmaceutical Sciences
Berhampur, At/PO Mohuda, Distt Ganjam-760 002, India

Two simple, sensitive and reproducible UV spectrophotometric methods (A and B) for the determination of ethamsylate (EMS) in bulk samples and pharmaceutical formulations are described. EMS was taken in methanol for method A and in 0.1 N HCl for method B. The maximum absorbance was observed at 302 nm for method A and 302 nm for method B. Regression analysis of Beer's law plots showed good concentration ranges 2–10 μg/mL for method A and 5–15 μg/mL for method B respectively. There is no interference from any common pharmaceutical excipients.

Key Words: UV spectrophotometric methods, Ethamsylate.

Ethamsylate (EMS)[1–3] is used under the category of haemostatics. It is also used in prevention and treatment of capillary haemorrhages associated with haematemeris, haedmoptysis, menorrhagia and post-abortion and post-partum haemorrhages. Chemically EMS[4] is known as 2,5-dihydroxy benzene sulphuric acid compound with diethyl amine (1 : 1). A very few physico-chemical methods have appeared in literature for the determination of EMS in pharmaceutical formulations. No UV spectrophotometric methods have been reported for the assay of EMS in literature. The authors have made some attempts in developing UV spectrophotometric methods and succeeded in developing two methods based on the solubility of the drug in methanol (method A) and in 0.1 N HCl (method B). These methods are successfully extended to pharmaceutical formulations containing EMS. EMS exhibits absorption maximum at 302 nm in methanol and Beer's law is obeyed in the concentration range 2–10 μg/mL. EMS exhibits absorption maximum at 302 nm in 0.1 N HCl and Beer's law is obeyed in the concentration range 5–15 μg/mL.

Spectral and absorbance measurements were made on Elico SL-159 UV-Vis spectrophotometer with 1 cm matched quartz cells.

All the chemicals and reagents were of analytical grade and the solutions were prepared in triply distilled water. 0.1 N HCl was prepared according to I.P.

Preparation of standard drug solution

For method A: 1 mg/mL Stock solution of EMS was prepared by dissolving 50 mg in 50 mL of methanol. The working standard solution was prepared by further dilution of the stock solution with methanol to obtain 100 μg/mL.

For method B: 1 mg/mL Stock solution of EMS was prepared by dissolving 50 mg in 50 mL of 0.1 N HCl. The working standard solution was prepared by further dilution of the stock soluiton with 0.1 N HCl to obtain 100 μg/mL.

For pharmaceutical formulations

Method A: The powder of 20 tablets was taken, pulverized and the weight equivalent to 100 mg of EMS was dissolved in methanol and filtered, and the filtrate was diluted to 100 mL with methanol.

Method B: The powder of 20 tablets was taken, pulverized and the weight equivalent to 100 mg of EMS was dissolved in 0.1 N HCl and filtered, and the filtrate was diluted to 100 mL with 0.1 N HCl.

Recommended procedures

Method A: To a series of 10 mL volumetric flasks, aliquot samples of EMS ranging from 0.2–1 mL (1 mL containing 100 μg) were transferred. Then the final volume was brought to 10 mL with methanol and the absorbance was measured at 302 nm against methanol as blank. The amount of EMS present in the sample solution was computed from its calibration curve.

Method B: To a series of 10 mL volumetric flasks, aliquot samples of EMS ranging from 0.5–1.5 mL (1 mL containing 100 μg) were transferred. Then the final volume was brought to 10 mL with 0.1 N HCl and the absorbance was measured at 302 nm against 0.1 N HCl as blank. The amount of EMS present in the sample solution was computed from its calibration curve.

The optical characteristics such as Beer's law limits, Sandell's sensitivity, molar extinction coefficient for each method are given in Table-1. The precision of each method was found by measuring absorbances of five replicate samples containing known amounts of drug and the results obtained are incorporated in Table-1. Regression analysis using the method of least squares was made to evaluate the slope (a), intercept (b) and correlation coefficient for each method and are presented in Table-1. Commercial formulations containing EMS were successfully analyzed by the proposed methods. The similarity of the results is obvious evidence that during the application of these methods, the addition and excipients that are usually present in pharmaceutical formulations do not interfere in the assay of proposed methods. As an additional check of accuracy of the proposed methods, recovery experiments were perfo med by adding a fixed amount of the drug to the preanalyzed formulations. The amount of drug found and the % recovery were calculated and presented in Table-2.

TABLE-1
OPTICAL CHARACTERSTICS AND PRECISION OF THE PROPOSED METHODS

Parameters	Ethamsylate in methanol	Ethamsylate in 1.0 N HCl
λ_{max} (nm)	302	302
Beer's law limit (μg/mL)	2–10	5–15
Sandell's sensitivity (μg/cm^2/0.001 absorbance unit)	0.0192	0.1950
Molar extinction coefficient (L/mol/cm)	1.229×10^4	1.08×10^4
(%) Relative standard deviation	0.5260	0.5260
(%) Range of error:		
0.05 confidence limits	0.2040	0.2050
0.01 confidence limits	0.3590	0.3600
Correlation coeficient	0.9999	0.9999
Regression equation (Y*):		
Slope (a)	0.0640	0.0540
Intercept (b)	0.0011	0.0019

Y* = b + aC, where "C" is concentration in μg/mL and Y is the absorbance unit

TABLE-2
ESTIMATION OF EMS IN PHARMACEUTICAL FORMULATIONS

Sample	Labelled amount (mg)	Amount found (mg) in methanol **(Method A)**	(%) Recovery	Amount found (mg) in 0.1 N HCl **(Method B)**	(%) Recovery
EMS tablets					
1	500	498.5	99.70	496.5	99.3
2	500	497.3	99.46	495.4	99.1

Conclusions

The proposed methods are most economic, sensitive and accurate and have the advantage of wider range under Beer's law limits. The decreasing order of sensitivity between the proposed methods are **A > B**. The proposed methods are simple, selective and can be used in the routine determinations of EMS in bulk samples and formulations with reasonable precision and accuracy.

REFERENCES

1. The Merk Index, 11th Edn., p. 587 (1989).
2. Martindale, The Extra Pharmacopoeia, 31st Edn., 763, 3 (1996).
3. *Drug Today* (Jan–Mar, 2003).
4. www.Rxlist.com

(*Received*: 23 February 2004; *Accepted*: 10 June 2004) AJC-3479