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Development and Validation of UV Spectroscopic Method for Estimation of Guaifenesin In Tablet Dosage Form

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ABSTRACT

To develop and validate simple, rapid, linear, accurate, precise and economical UV Spectroscopic method for estimation of Guaifenesin in tablet dosage form. The drug is freely soluble in analytical grade Methanol. The drug was identified in terms of solubility studies and on the basis of melting point done on melting point apparatus of Equiptronics. It showed absorption maxima were determined in analytical grade Methanol. The drug obeyed the Beer's law and showed good correlation of concentration with absorption which reflect in linearity. The UV spectroscopic method was developed for estimation of Guaifenesin in tablet dosage form and also validated as per ICH guidelines. The drug is freely soluble in analytical grade Methanol, moderately soluble in Benzene and soluble in Chloroform, Glycerol. So, the analytical grade Methanol is used as a diluent in method. The melting point of Guaifenesin was found to be 78-79°C (uncorrected). It showed absorption maxima 269 nm in analytical grade Methanol. On the basis of absorption spectrum the working concentration was set on 10µg/ml (PPM). The linearity was observed between 6-14 µg/ml (PPM). The results of analysis were validated by recovery studies. The recovery was found to be 98.75, 101 and 99.17% for three levels respectively. The % RSD for precision was found to be 0.97%. A simple, rapid, linear, accurate, precise and economical UV Spectroscopic method has been developed for estimation of Guaifenesin in tablet dosage form. The method could be considered for the determination of Guaifenesin in quality control laboratories.

Keywords: Guaifenesin, UV Spectrophotometer, Melting Point, Assay Method, Validation, Accuracy, Linearity, Ruggedness, Precision.

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INTRODUCTION

Guaifenesin (GUA) is chemically known as (+)-3-(2-methoxy phenoxy)- propane-1,2-diol. Its empirical formula is $C_{10}H_{14}O_4$ which corresponds to molecular weight of 198.21 [1]. It is used to relieve the chest congestion. It controls the symptom but does not treat the cause of the symptom. It is an expectorant and thinning the mucus and clears the airways [2]. Guaifenesin is a glyceryl guaiacolate with expectorant effects. Guaifenesin increases respiratory tract mucus secretions, acts as an irritant to gastric vagal receptors and recruits efferent parasympathetic reflexes that cause glandular exocytosis [3]. This agent reduces the viscosity of mucus secretion by reducing adhesiveness and surface tension as well as increasing hydration of mucus. Guaifenesin promotes the efficiency of the mucociliary mechanism important in removing accumulated secretions from the upper and lower airway [4].

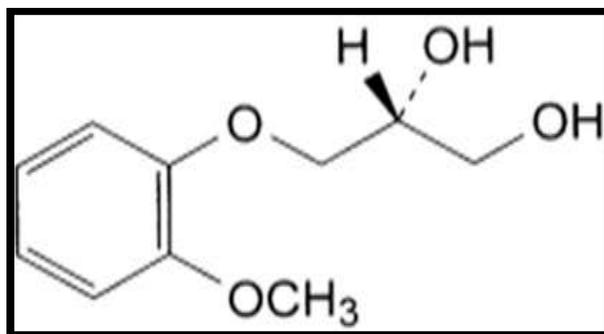


Figure 1: Chemical Structure of Guaifenesin

From literature review it's found that one method was reported on derivative spectrophotometry for simultaneous estimation of Guaifenesin with degrading product on UV for impurity in Guaifenesin [4]. Lot of work was done on UV method development for Guaifenesin in combination with other drugs [5, 6]. There is also method reported HPLC, RP-HPLC, TLC of Guaifenesin with other drug on HPLC [7-11]. There is also method reported Bioanalytical LCMS method of Guaifenesin on human plasma [12]. But very few methods were reported on estimation of Guaifenesin in tablet dosage form for UV spectroscopic method. This indicates that so far no UV method exists for the estimation and determination of Guaifenesin in tablet dosage forms. The aim of the study was to develop a simple, precise, linear, economic and accurate UV method for determination of Guaifenesin in tablet dosage forms.

MATERIALS AND METHOD

Instruments :

Shimadzu double beam UV-visible spectrophotometer 1700 Ultra with matched pair

Quartz cells corresponding to 1 cm path length and spectral bandwidth of 1 nm, Bath sonicator and citizen weighing balance.

Melting point apparatus of Equiptronics were used.

Materials:

Guaifenesin is a gift sample obtained from M/S Serin formulations, Hyderabad. Guaifenesin tablets were procured from local pharmacy. Methanol used was of analytical grade. Freshly prepared solutions were employed.

Method development:

Determination of λ max (10 PPM)

100 mg weighed amount of Guaifenesin was dissolved into 100 ml of volumetric flask with analytical grade Methanol. Pipette out 1 ml and added in 100 ml of volumetric flask dissolved and diluted up to the mark with analytical grade Methanol. This solution was subjected to scanning between 200-400 nm and absorption maximum was determined [14, 15, 16].

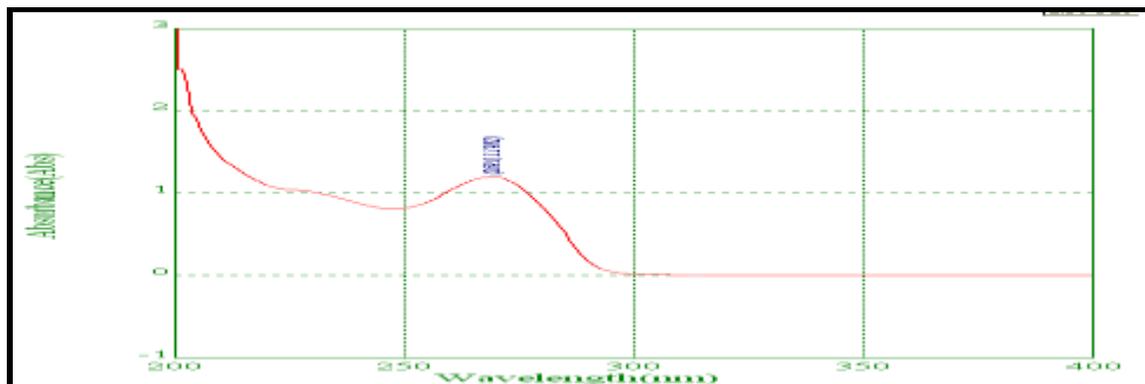


Figure 2: Calibration Curve

A. Preparation of Working concentration

Preparation of Standard stock solution:

Standard stock was prepared by dissolving 100 mg of Guaifenesin in 100 ml of analytical grade Methanol to get concentration of 1000 $\mu\text{g/ml}$ (PPM).

Preparation of Standard solution:

Pipette out 1 ml from standard stock solution and diluted up to 100 ml with analytical grade Methanol to get concentration of 10 $\mu\text{g/ml}$ (PPM).

B. Preparation of Working concentration

Preparation of Standard stock solution:

Standard stock was prepared by dissolving 100 mg of Guaifenesin in 100 ml of analytical grade Methanol to get concentration of 1000 $\mu\text{g/ml}$ (PPM).

Preparation of Standard solution:

Pipette out 1 ml from standard stock solution and diluted up to 100 ml with analytical grade Methanol to get concentration of 10 µg/ml (PPM).

C. Procedure for UV reading**Blank Solution:** (For Auto zero)

Fill the cuvette with analytical grade Methanol. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

Standard Solution:

Fill the cuvette with standard solution. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

Sample Solution:

Fill the cuvette with sample solution. Wipe it with tissue paper properly then placed inside the chamber. Note down the reading.

D. Procedure for sample preparations

For analysis of commercial formulations; twenty tablets are taken weighed it and powdered. The powder equivalent to 100 mg of Guaifenesin was accurately weighed and transferred into the 100 ml of volumetric flask, added 60 ml analytical grade Methanol, the solution was sonicated for 20 min. After sonication cool the flask and diluted upto 100 ml with analytical grade Methanol. Filtered the solution through whatmann filter paper. Pipette out 1 ml of the above solution and diluted up to 100 ml with analytical grade Methanol. The absorbance was measured at 269 nm [17, 18, 19]. The absorbance was recorded:

Table 1: Absorbance of Dosage Form

Mckesson Pharmaceutical Limited (100 mg)		
Sr. no.	Sample	Absorbance
1	Blank	0.0001
2	Standard	0.4298
3	Sample	0.4225

Table 2: Dosage Form Specifications

Type	Company	M.D.	E.D.	Batch No.	Average weight (g)	Assay (%)
1	Mckesson Pharma LTD (400 mg)	05/2018	07/2021	F177667227	0.5027	98.3

E. Method of validation

The proposed method was developed by using linearity, accuracy, precision and ruggedness as per ICH guidelines, 1996 [17, 18, 19].

Linearity:

The linearity of the proposed assay was studied in the concentration range 6 - 14 PPM at 269nm. The calibration data showed a linear relationship between concentrations.

Table 3: Linearity Studies

Sr. no.	Sample Concentration	Absorbance
1	6 PPM	0.2614
2	8 PPM	0.3358
3	10 PPM	0.4265
4	12 PPM	0.5137
5	14 PPM	0.5965
Correlation coefficient		0.999

Accuracy:

To ensure the accuracy of the method, recovery study was performed by preparing 3 sample solutions of 80, 100 and 120% of working concentration and adding a known amount of active drug to each sample solution and dissolved in 100ml of volumetric flask with analytical grade Methanol and measuring the absorbance at 269nm.

Table 4: Accuracy Studies

Accuracy (%)	Qty weighed (mg)	Qty found (mg)	Recovery (98-102%)
80	8	7.9	98.75
100	10	10.1	101.0
120	12	1.19	99.17

Precision:

The precision of the method was demonstrated by inter-day and intra-day variation studies. Five sample solutions were made and the %RSD was calculated.

Table 5: Precision studies

Sr. No.	Sample Solution	Absorbance
1	Sample Solution 1	0.4221
2	Sample Solution 2	0.4278
3	Sample Solution 3	0.4164
4	Sample Solution 4	0.4212
5	Sample Solution 5	0.4234
MEAN		0.4222
SD		0.0041
% RSD		0.9727

Ruggedness:

Ruggedness is a measure of the reproducibility of a test result under normal, expected operating condition from instrument to instrument and from analyst to analyst.

Table 6: Results for Ruggedness Studies

Sr. No.	Analyst	Results	Mean	% Assay	% RSD
1	Analyst 1	0.4278 0.4215	0.4247	98.8	0.5748
2	Analyst 2	0.4207 0.4212	0.4210	98.0	

RESULTS AND DISCUSSION

Solubility of Guaifenesin

Solubility test was passed as per criteria.

Table 7: Results for solubility studies

Sr. no.	Title	Result
1	Analytical grade Methanol	Freely Soluble
2	Benzene	Moderately soluble
3	Chloroform, Glycerol	Soluble

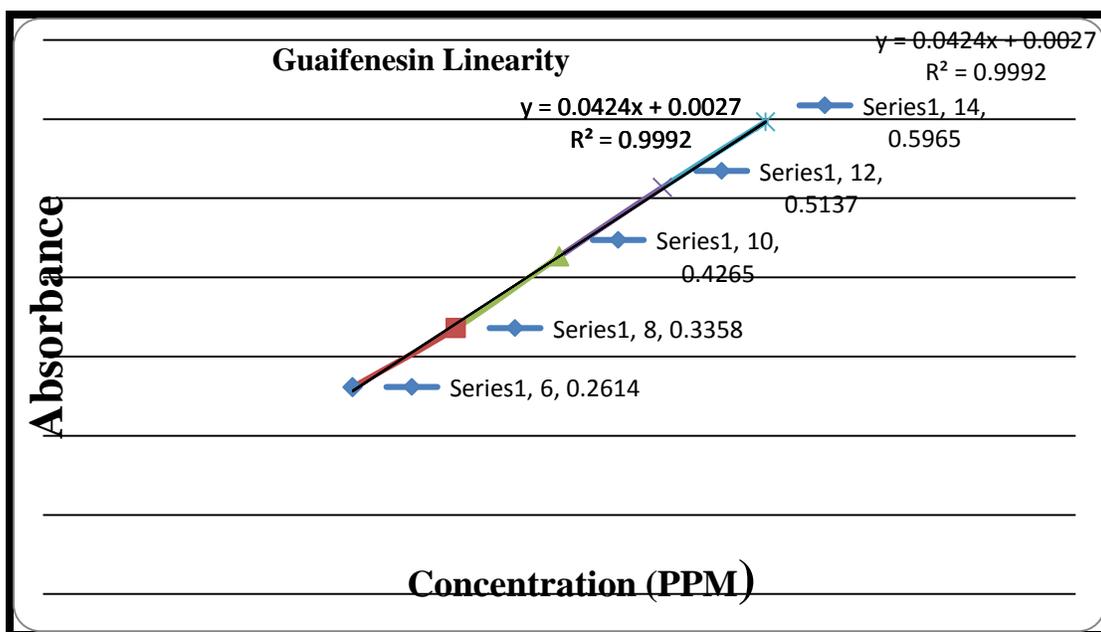
Melting point of Guaifenesin

The melting point of Guaifenesin was found to be 78-79°C (uncorrected).

Results for linearity for assay method of Guaifenesin

The linearity of method was determined at concentration level ranging from 6 to 14 µg/ml (PPM).

The correlation coefficient value was found to be (R^2) **0.999**.

**Figure3: Guaifenesin Standard Curve**

Results for accuracy for assay method of Guaifenesin

The accuracy of the method was determined by recovery experiments. The recovery studies were carried out and the percentage recovery were calculated and represented in Table - 4. The high percentage of recovery indicates that the proposed method is highly accurate. Accuracy results were found within acceptance criteria that are within 98-102% as per ICH Guidelines.

Results for precision for assay method of Guaifenesin

The % RSD for different sample of precision was found to be 0.9727 and it is within acceptance criteria as per ICH Guidelines represented in Table - 5.

Results for ruggedness for assay method of Guaifenesin

The %RSD for different sample of ruggedness was found to be 0.5748 and it is within acceptance criteria as per ICH Guidelines represented in Table - 6.

CONCLUSION

A method for the estimation of Guaifenesin in tablet form has been developed. From the spectrum of Guaifenesin, it was found that the maximum absorbance was 269 nm in analytical grade Methanol. A good linear relationship was observed in the concentration range of 6-14 µg/ml (PPM). The high percentage recovery indicates high accuracy of the method. This demonstrates that the developed spectroscopic method is simple, linear, accurate, rugged and precise for the estimation of Guaifenesin in solid dosage forms. Hence, the method could be considered for the determination of Guaifenesin in quality control laboratories.

ABBREVIATIONS

1. PPM - Parts per Million
2. HPLC - High Performance Liquid Chromatography
3. UV - Ultra violet
4. HBV - Hepatitis B virus
5. DNA - Deoxyribonucleic acid
6. HIV - Human Immunodeficiency Virus
7. ICH - International Council for Harmonization
8. RSD - Relative Standard Deviation
9. SD - Standard Deviation
10. Qty - Quantity
11. C - Celsius
12. M.D. - Manufacturing Date
13. E.D. - Expiry Date

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