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Development of Stability Indicating Assay Method using HPTLC for Determination of Bilastine in Bulk and Tablet Dosage Form and Characterization of Degradant By LC-MS

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ABSTRACT:
Analytical chemistry is the science of obtaining, processing, and communicating information about the composition and structure of matter. Analytical chemistry of solution deals with reaction and equilibrium state of solutes in order to find appropriate conditions to characterize or quantify analysts.

Introduction To Analytical Chemistry
Qualitative analysis deals with the determination of identification of elements, ions or compounds present in sample.

I. INTRODUCTION

Qualitative analysis: Qualitative analysis deals with the determination of identification of elements, ions or compounds present in sample.
Quantitative analysis: Quantitative analysis deals with the determination of how much amount of one or more constituents are present in the sample.

AIM OF ANALYTICAL CHEMISTRY:

- Development of theory of analytical methods in every possible way.
- Improvement and scientific substantiation of the existing analytical methods.
- Scientific elaboration of new analytical methods which meet the requirement of the advancing science and modern production.

II. ANALYTICAL METHODS:

In the pharmaceutical industry, the quality of finished product i.e., drug product is very vital, as it involves life. The pharmaceutical analyst plays a very significant role in quality control of pharmaceuticals through rigid check on raw material used in manufacturing of formulations and on finished products. To perform this important task and to scope up with the requirement of analysis, a variety of analytical methods and techniques are available.

A) Chemical methods:
B) Instrumental methods:

A) Chemical methods

1. In these methods, volume and mass are used as means of detection.
2. Titrimetric methods like acid-base, oxidation-reduction, non- aqueous, complexometric and precipitation titrations. Gravimetric and thermo gravimetric methods.
3. Volumetric methods.

B) Instrumentalmethods

1. These methods are based upon the measurement of some physical properties as conductivity, electrode potential, light absorption or emission, mass-to-charge ratio and fluorescence of substance. There are many techniques available for the analysis of analyses.

III. VALIDATION

"The goal of validation is to establish documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting its predetermined specifications and quality attributes". Typical validation characteristics which should be considered are listed below as per ICH guidelines.

A) Accuracy. Precision.

- Repeatability.
- Intermediate Precision.
- Reproducibility.

B) Specificity.

- Limit of Detection.
- Limit of Quantitation.
- Linearity.

C) Range.
D) Robustness.

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IV. INTRODUCTION TO CHROMATOGRAPHY:

Chromatography (from Greek chroma, color and graphein to write) is the collective term for a set of laboratory techniques for the separation of mixtures. It involves passing a mixture dissolved in a "mobile phase" through a stationary phase, which separates the analyte to be measured from other molecules in the mixture based on differential partitioning between the mobile and stationary phases. Subtle differences in a compound's partition coefficient result in differential retention on the stationary phase and thus changing the separation. Chromatography may be preparative or analytical. The purpose of preparative chromatography is to separate the components of a mixture for further use (and is thus a form of purification).

HIGH PERFORMANCE THIN LAYER CHROMATOGRAPHY (HPTLC)

INTRODUCTION:

High-Performance Thin-Layer Chromatography (HPTLC) is a sophisticated instrumentation technique. It has been reported in many publications to provide excellent separation and qualitative and quantitative analysis of a wide range of compounds, such as herbal and botanical dietary supplements, nutraceuticals, traditional western medicines, traditional Chinese medicines, and Ayurvedic (Indian) medicines. Comparative studies have often found that HPTLC is superior to High-Performance Liquid Chromatography (HPLC) in terms of total cost and time required for analysis. HPTLC is an off- line process in which the various stages are carried out independently.

Factors influencing HPTLC separation and resolution of spots:

- Type of stationary phase (sorbent):
- Silica gels of different grades, aluminum oxide of different grades, florisil, kieselguhr G are commercially available sorbets for HPTLC. For quantitative analysis, use of HPTLC pre-coated plate is absolutely essential. The Precoated plates with different support material (glass, aluminum, plastic) with different layers are available in different format and thickness by various manufacturers.
- Layer thickness:
- Usually plates with sorbent thickness of 100 – 250 µm are used for
- qualitative and quantitative analysis.
- Mobile phase (solvent system):

Mobile phases should be chosen taking into consideration chemical properties of analytes and sorbent layer. Use of mobile phase containing more than three or four components should normally be avoided as it is often difficult to get reproducible ratios of different components.

1. Size of developing chamber:

First, Stahl drew attention to importance of degree of saturation of atmosphere in the chamber and showed that it was necessary to adhere to certain conditions. He introduced the ratio Evaporation surface area: Chamber Volume as a characteristic. This ratio is 1:20 in usual rectangular chamber but 1:0.1 to 0.5 for narrow chamber.

2. Saturation of chamber (pre-equilibrium):

Chamber saturation has pronounced influence on the separation profile. When the plate is introduced into an unsaturated chamber, during the course of development, the solvent evaporates from the plate mainly at the solvent front. Therefore, larger quantity of the solvent shall be required for a given distance; hence resulting in increase in Rf values.

3. Sample volume to be spotted:

Substance zones which are too large from the beginning cause poor separation as during development spots do tend to become large and more diffused

AIM AND OBJECTIVE AIM:

To develop and validate Stability Indicating Assay Method for Bilastine using HPTLC.

OBJECTIVE:

- To develop HPTLC method for selected drug.
- To validate the proposed method as per ICH guideline.
- To study the FT-IR spectrum of selected drug
- To study the effect of forced degradation on bulk drug using various stress condition
- To separate, isolate and characterize the major degradant

DRUG PROFILE

BILASTINE

- Nature: Bilastine is a white solid powder. Structural Formula:

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- Figure 5.1: Structure of Bilastine
- IUPAC name: 2-[4-(2-{4-[1-(2-ethoxyethyl)-1H-1-3-benzodiazol-2-
- yl]piperidin-1-yl}ethyl)phenyl]-2-methylpropanoic acid
- Molecular formula: C28H37N3O3
- Molecular weight: 463.61g/mol.
- Melting point: 197-200°C
- Solubility: Freely soluble in chloroform, HCL 1N, NaoH 1N. Slightly soluble in Methanol, ethanol. Soluble in DMSO and DMF
- Category: Antiallergic, used in the treatment of allergic rhinitis and chronic urticaria.

DRUG PROFILE - Dosage forms available: Tablet form (20mg) Storage/stability: Store between 15°C -30°C.
Routes of administration: Oral route

V. EXPERIMENTAL WORK:

- Active Pharmaceutical Ingredients:
- Details of Bilastine bulk drug.
- Drug- Bilastine
- Quantity- 5 gm
- Instruments:
- UV-Vis Spectrophotometer:
- Model: Jasco-530 double beam UV –Vis spectrophotometer. Instruments:
- UV-Vis Spectrophotometer:
- Model: Jasco-530 double beam UV –Vis spectrophotometer
- FT-IR:
- Model: Jasco FT/IR-4100
- Detector: TGS.
- Scanning speed: Auto (2 mm/sec)

High Performance Thin Layer Chromatography (HPTLC):

- Sample applicator: CAMAG Linomat 5
- HPTLC Plate: Silica gel 60F 254 (E. Merck, Germany)
- Development chamber: Twin trough glass chamber, (10×10 cm
- CAMAG)
- Densitometric scanner: CAMAG TLC Scanner IV

VI. SUMMARY AND CONCLUSION:

High Performance Thin Layerchromatography (HPTLC)

A quick, precise and accurate method based on HPTLC has been developed for analysis of Bilastine. The method was developed and validated for the determination of Bilastine on precoated silica gel HPTLC plates using n-Hexane:methanol:iso propyl alcohol (1:5:4v/v/v) as a mobile phase with Densitometric detection at 277 nm. The method was validated for linearity, precision, accuracy and robustness. Linearity range for Bilastine was found 500-2500 ng/band Correlation coefficient was 0.997.The developed method was precise and robust, % RSD was found less than 2% And % recovery was found to be in range of 98.26- 101%. LOD and LOQ were 1.53 ng/band and 5.43 ng/band respectively.

Stress degradation studies were performed to evaluate the stability indicating properties and specificity of the method. Degradation study was carried out by exposing of working standard solution of Bilastine with acid (0.1N HCL at 800C), base (0.1 N NaOH at 80oC), hydrogen peroxide (3% H2O2), Distilled water (H2O) for 1 hours while one volumetric flask was exposed to (800 C) for 1 hour and one volumetric flask.

VII. FUTURE SCOPE

The Future scope of Stability Indicating assay method for Bilastine as bulk drug is as follows :

- The availability of this data will trigger similar data collection and analysis, and this may provide a platform for extensive research collaboration.
- The data allows other researchers working on related aspects to extend the statistical analysis and use it in any model evaluation and validation in relation with given weather parameters.
- This data will be useful to the researchers and scientific community working on method development and validation.
- Isolation ,identification and characterisation of degradation

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