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EXPLORATION ON UPLC METHOD FOR THE ANALYSIS OF LOBEGLITAZONE SULPHATE AND GLIMEPIRIDE WITH THEIR STREE DEGRADATION STUDIES

Pharmaceutical Science

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ABSTRACT

Lobeglitazone Sulphate and Glimepiride are commonly prescribed drugs in the management of type 2 diabetes, often used together to improve glycemic control. The precise analysis of these compounds in pharmaceutical formulations is essential for ensuring drug quality, efficacy, and patient safety. By highlighting the advantages of UPLC over conventional chromatographic techniques, this review describes the procedure of method development in UPLC about the selection of mobile phase, column and detector wavelength. Validation and stability details are connected with validation parameters and degradation of drugs. At the outset this exploration underscores the importance of UPLC in ensuring the quality control and regulatory compliance of combination drug therapies, contributing to the safe and effective treatment of type 2 diabetes.

KEYWORDS

Lobeglitazone Sulphate , Glimepiride, UPLC method development, validation.

INTRODUCTION

Lobeglitazone sulphate and Glimepiride are two important oral antidiabetic agents commonly prescribed for the management of type-2 diabetes mellitus. Lobeglitazone sulphate is a thiazolidinedione (TZD) derivative, acts as a selective peroxisome proliferator-activated receptor gamma (PPAR-γ) agonist, enhancing insulin sensitivity in peripheral tissues and improving glycemic control. It offers advantages over earlier TZDs with reduced side effects such as fluid retention and weight gain. On the other hand, Glimepiride is a third-generation sulfonyl urea that stimulates pancreatic β-cells to release insulin, making it effective in reducing fasting and postprandial glucose levels. When used in combination, these drugs provide a complementary mechanism of action, targeting both insulin secretion and insulin resistance. The increasing use of these agents in fixed-dose combinations and their widespread clinical application necessitate the development of accurate, robust and efficient analytical methods to ensure pharmaceutical quality and regulatory compliance. Among modern analytical techniques, Ultra Performance Liquid Chromatography (UPLC) emerged as a powerful tool for the simultaneous determination of Lobeglitazone sulphate and Glimepiride. Compared to conventional HPLC, UPLC offers improved resolution, faster analysis times, and greater sensitivity, making it highly suitable for routine quality control and stability studies. This review highlights the analytical advancements in UPLC method development and validation for the estimation of Lobeglitazone Sulphate and Glimepiride in bulk and formulated products. It also discusses the use of stability-indicating methods and forced degradation studies, providing insights into the role of UPLC in ensuring the safety, efficacy, and stability of these antidiabetic agents.

MATERIALS AND METHODS

Chemicals

High-purity chemicals and reagents were used to assure method accuracy and reproducibility during the development and validation of the UPLC technique for Lobeglitazone Sulphate and Glimepiride. Methanol and UPLC-grade acetonitrile were employed as organic modifiers for sample preparation and mobile phase. Analytical grade potassium dihydrogen phosphate (KH_2PO_4) was used to make buffer solutions, and sodium hydroxide or diluted orthophosphoric acid were used to suitably alter the pH. For both standard and sample solutions, an acetonitrile and water mixture was used as the diluent.

Instrumentation

Lobeglitazone Sulphate and Glimepiride were subjected to an UPLC analysis using a Waters ACQUITY UPLC system, which was run by Empower software and included a photodiode array (PDA) detector and a binary solvent management. The separation was accomplished using a C18 reverse-phase column. The mobile phase was made up of phosphate buffer and acetonitrile in precisely the right amounts.

Chromatographic Conditions

Chromatographic separation of Lobeglitazone sulphate and glimepiride by UPLC is typically performed using a reverse-phase C18 column. The mobile phase generally consists of a mixture of acetonitrile and phosphate buffer, with the pH adjusted to about 3.0 using orthophosphoric acid. The flow rate is maintained around 0.3 to 0.5 mL/min, and detection is carried out using a UV detector set between 220 to 250 nm.

Mobile phase

Various combinations of acetonitrile and Phosphate buffer pH adjustment is provided to get optimum peak shape, retention, and analyte stability.

pH Range

Lobeglitazone sulphate and Glimepiride are analyzed by UPLC using acidic mobile phases. For Lobeglitazone Sulphatethe pH is adjusted between 3.0 to 4.0 and for Glimepiride between 3.0 to 4.5 using orthophosphoric acid. A C18 column is used for both drugs to achieve good separation. Maintaining pH below their pKa values help improve peak shape and stability.

Column

Lobeglitazone Sulphate & Glimepiride on UPLC method use C18 reverse-phase columns with 3-5 min retention. Acidic mobile phase (pH 3.0) and acetonitrile ensure peak sharpness and analyte stability, solubility, reduces tailing and improves peak shape, with column choice critical for stability and validation.

Flow Rate

For both compounds 0.3 to 0.5 ml/min is used, which gives sharp well separated peaks.

Temperature

The temperature is adjusted in UPLC (Ultra Performance Liquid Chromatography) tests for Glimepiride and Lobeglitazone sulphate. Typically it is adjusted to 25°C to 40°C.

Detection

In UPLC analysis of Lobeglitazone Sulphate and Glimepiride, a UV detector is mostly used for Lobeglitazone Sulphate and Glimepiride at 220–250 nm. Sometimes a PDA (Photodiode Array) detector is also used to check peak purity.

Method Validation

Analytical technique validation is required by law to make sure the method is dependable, reproducible and appropriate for its intended use. For method validation, the ICH Q2 (R1) criteria are commonly adhered. The following are important metrics assessed during the validation process for both glimepiride and Lobeglitazone sulphate.

1. Specificity

specificity refers to the ability of a method to accurately measure only the intended analyte in the presence of other substances that may be

expected to be present in the sample. This includes components like impurities, degradation products, and matrix elements. Placebo and blank are injected separately to study the specificity. Chromatograms without any peaks indicates that the method is specific.

2. Linearity

In the context of analytical method validation, linearity refers to the ability of an analytical procedure to produce results that are directly proportional to the concentration of the analyte within a specified range. The method is considered linear if the correlation coefficient is ≥ 0.999 and minimum six concentration levels are used to prove the linearity. At each concentration level three injections readings are considered.

3. Accuracy

The accuracy expresses the closeness of agreement between the value which is accepted either as a conventional true value or an accepted reference value and the value found. This is determined by performing recovery studies at three concentration levels (e.g., 80%, 100%, and 120% of the target concentration). The percentage recovery should fall within the range of 98% to 102%, indicating the method's ability to measure the analyte correctly. At each concentration level three injections readings are considered.

4. Precision

By conducting repeat studies (at least six injections) of a sample and computing the relative standard deviation (RSD), precision is assessed. It is necessary to evaluate both intermediate precision (inter-assay precision) and repeatability (intra- assay precision). Both should produce RSD values below 2%, which would suggest high precision.

5. Limit of Detection (LOD) and Limit of Quantitation (LOQ)

The slope of the calibration curve and the response's standard deviation are used to calculate LOD and LOQ. The sensitivity of the approach is ensured by the fact that the LOD and LOQ for glimepiride and Lobeglitazone sulphate are normally found to be in the ratio of 1:3.

6. Robustness

Robustness is tested by making small deliberate changes to method parameters (e.g., flow rate, column temperature and mobile phase composition) and evaluating the impact on method performance. A robust method should not show significant variations under these small changes.

Forced Degradation Studies

Forced degradation studies involve subjecting the drug to extreme conditions (acidic, basic, oxidative, thermal, and photolytic) to accelerate degradation. The method should be able to separate the intact drug from its degradation products, ensuring that it is stability-indicating. For Lobeglitazone sulphate & Glimepiride degradation is typically observed under acidic, basic, thermal, photolytic and oxidative conditions.

Acid Degradation

Acid degradation is a type of forced degradation where a drug or chemical substance is exposed to acidic conditions (typically using hydrochloric acid, HCL) to study its stability, degradation pathways, and impurity profile.

Base Degradation:

Base degradation is when a drug or substance breaks down in alkaline conditions (using a strong base like sodium hydroxide, NaOH). This is done to study how the drug behaves in basic environments.

Oxidative Degradation:

Oxidative degradation refers to the breakdown or chemical change of a substance when exposed to oxygen or oxidizing agents. This can lead to the formation of degradation products due to the reaction of the drug with oxygen (air) or other oxidizing substances.

Photo Degradation:

Photo degradation refers to the breakdown or chemical change of a substance when exposed to light, particularly UV light. This type of degradation is important for understanding how a drug or chemical might degrade when exposed to light during storage or use.

Thermal Degradation:

Thermal degradation refers to the breakdown or chemical change of a substance when exposed to heat. This is commonly studied to understand how a compound or drug reacts under high-temperature conditions.

CONCLUSION

The development and validation of an RP- UPLC method for the quantification of Lobeglitazone sulphate and Glimepiride are crucial for ensuring the quality and safety of these antidiabetic drugs.

The study described the procedure for assessment of two drugs. The use of RP-UPLC for the analysis of these drugs offers numerous advantages, including faster analysis times, higher resolution, and reduced solvent consumption compared to traditional UPLC techniques.

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