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Method Validation and Method Development by using Analytical Method- HPLC- Review on Current Research

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ABSTRACT:

Analytical method development is should be validated to give proper data for regulatory submission. High performance liquid chromatography (HPLC) is very useful in the drug discovery, manufacturing process, and development of pharmaceutical materials. It is also useful to separate manufacturing drugs from drug substance impurities, for detection and quantify the synthetic drugs and it reduce rest impurities at the time of the separation. It is also need to ensure that safety and quality of drugs. Validation is the method of use to the performance characteristic and its limitation of method and its identification of the influence that may replace these characteristics. This review described the planning and the problems pertinent to designed High performance liquid chromatography (HPLC) development of method and method validation. The force degradation studies useful in method validation and method development of stability indicating analytical method.

KEY WORDS: Method development, HPLC, validation, force degradation.

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INTRODUCTION [1-5]:

Analytical chemistry is an science and art of recognized the various substance use in specify matrix and determination of its constitute. Analytical chemistry is derived in two parts. 1) Qualitative analysis and 2) Quantitative analysis. Qualitative analysis is useful in identify the drug components and Quantitative analysis is useful in determination of material quantity. The principle of HPLC is a technique to separate the mixture of compound into its components on the basis of their molecular composition and molecular structure. It involves a solid or liquid supported on a solid (stationary phase) and a liquid or a gas (mobile phase). The mobile phase flows through the stationary phase and carry the components of the mixture with it. HPLC is very highly improved analytical method of column liquid chromatography. It is force through under high pressure of up to 400 to 450 atmosphere. It done it more faster. All chromatographic separations is under HPLC operate the similar basic principle. HPLC is much versatile than gas chromatography because of 1) It is not limited to volatile and thermally stable samples 2) Wider the choice of Stationary phase and mobile phase. The reason was include versatility, simplicity, and the scope of the reversed phase method and it is important in handle compound of divers polarity and mass of molecule.

Different types of HPLC [6-10]

There is four different types of HPLC following. That depend on stationary phase method.

1) **Normal Phase HPLC**
2) **Reverse Phase HPLC**
3) **Size- Exclusion HPLC**
4) **Ion- Exchange HPLC**

Application of HPLC [11-16]

Here is different application of HPLC they are use in different areas following.

- **Pharmaceutical application**
  1) In control of stability of drugs.
  2) Dissolution study of tablets.
  3). Pharmaceutical Q.C lab.
- **Environment application**
  1) Identification and detection of acidic and phenolic compounds present in drinking water.
  2) Bio monitoring of pollutants.
- **Forensics application**
  1) Identification of steroids present in blood, urine.
  2) Quantification of biological drug sample.
  3) Forensics analysis of textiles dye.
  4) Detection of cocaine and other drugs present in blood and urine.
- **Application in Clinical tests.**
  1) Detection of endogenous neuropeptides in extracellular fluid of brain.
  2) Analysis of antibiotics and Urine in blood.
- **Flavors and Food**
  1) Preservative Analysis
  2) Analysis of sugar in fruit juices.
  3) Measurement of Quality control of water and soft drinks.

Method Development [17-20]

The broad variety of instrument, column, eluant , and operational parameters involved make the HPLC method development seem complex. The main purpose of the method development of HPLC is try and separate quantify the major active drug and drug impurities, all present synthetic intermediated and any degradants.

Method Development steps include in HPLC

The method is influenced by the nature of the analysis and generally follow the below four steps.

1) Selection of the HPLC method and initial system.
2) Selection of Initial Conditions.
3) Method Validation
4) Method Optimization

Method Validation [21-22]

The analytical performance characteristics that may be tested during the validation method are below

- Accuracy
- Recovery
- Linearity
- Intermediate precision
- Detection of limit
- Quantification limit
- Range
- Specificity
- Force degradation study
- Robustness
- Solution stability study
- System suitability study

Research studies on Method validation and method development

Pradnya N. et al (2016) see the main purpose of RP-HPLC method is useful for the simultaneous separation and determination of MET and GLM. It has distinct merits over other existing methods with respect to sensitivity, time consuming and minimum detection limits. All the analysts were well resolved and separated in a short chromatographic run time. The development method is a stability-indicating method, which depend on the use of regular working procedure and can be simply used in routine analysis of pharmaceutical dosage form and stability samples of MET and GLM, with good accuracy, precision, selectivity, and recovery.

Naresh Chandra Joshi et al (2016) in this article author used the new, basic, delicate, exact & powerful superior dainty layer chromatographic (HPTLC) strategy was created for synchronous estimation of TENO & EMTRI in pharmaceutical measurement shapes. After improvement of plate, Camas TLC scanner III (examining speed 20 mm sec-1) was utilized for densitometry checking with Win Cats programming (opening

miniaturized scale, 6 x 0.45 mm). Investigation of plate in absorbance mode at 274 nm was done for both medications all through trial. Framework was found to give smaller spots for TENO & EMTRI with RF (Retardation variable) estimation of (0.41±0.01) and (0.56±0.01) separately. Data for adjustment plots demonstrated great direct association with r2 = 0.9985 and 0.9979 in fixation scope of 100-600ng/ml for both medications. Recent method was accepted by Conference on Harmonization (ICH) rules.

Varshney Neha et al (2016) had described the study of HPLC method enables quantitative determination of related compounds of Oxcarbazepine API. UV detection at 256nm was measure to be suitable without any interference. The result of linearity, precision, specificity and ruggedness were within limits. Making of samples is easy and efficient. From the results of related substances of Oxcarbazepine analysis it can be concluded that the proposed HPLC method is precise, linear and robust that can be applicable for continuous analysis.

Silvana Togneri MacMahon et al (2016) in this research article the author described that while IEC 80001-1 takes steps to address the risks associated with the placement of a medical device onto an IT network, HDOs may face challenges in understanding and implementing the requirements of the standard. The MedITNet framework has been developed using Action Design Research in order to assist HDOs in addressing these challenges. The use of ADR confirmed that the MedITNet Assessment Framework, involved the Assessment Method, provides a consistent, recover and tailorable approach to the assessment of the capability of risk management processes related to the management of medical IT networks. An assessment of these processes can highlight weaknesses therein and can be used as a foundation for an improvement of risk management processes. The application of ADR proof that the framework that was developed can be used in a specific context but is also suited for use across a range of HDO contexts. Effective risk management of medical IT networks ensures that the potential useful of networked medical devices are realized while ensuring the safety of the patient is safed, the effectiveness of the device is assured and the security of the data and system are preserved.

K. Siddappa et al (2016) described that the developed a highly selective RP-HPLC method using UV detection for the determination of BOS used as nonpeptidic dual endothelin receptor antagonist (ERA) without derivatization in pharmaceutical formulations. The developed method is fast since preparation of pharmaceutical samples prior to chromatography is relatively easy and the total chromatographic run time is 10 min. The RP-HPLC method has high repeatable and good reproducibility. For this benefits. it can be used for the determination from pharmaceutical formulations of BOS in regular quality control measurements.

Iztok Grabnar et al (2015) In this article the author study that a novel, simple, selective, fast and sensitive SEC method for quantitative analysis of heparin is introduced. The method does not inclued any laborious pre-analysis sample making. It was validated according to the ICH guidelines. The applicaion of L-arginine solution (pH 6.5, 1 mg mL–1) as a mobile phase allowed us to establish a method with a less limit of quantification due to a beer shaped heparin peak. The proposed method is also precise and accurate since all parameters met the validation criteria. It was successfully applied to measure intact heparin in commercial pharmaceutical heparin products and in the experimental formulation of heparin/chitosan nanocomplexes. We taught that this method is appropriate for routine quantitative analysis of heparin in pharmaceuticals preparation.

Subrahmanya Bhat K et al (2015) described that the study and data it is concluded that, the method is search to be specific. The method is also Stability indicating as proved by forced degradation studies. The method is search to be Precise. The method is find to be Linear in the specified range. The method is robust w.r.t. flow and temperature variations. System suitability is established and recorded. Since, this method can be applicable for everyday analysis.

Bharat Vala et al (2014) The author had developed RP-HPLC method for the quantification of GRS, MHB and PHB has various merits like less retention time, good peak symmetry and phenomenal linearity, highly sensitive, simple, precise and accurate. The mobile phase can be simply prepared and diluent is economical and readily available and it does not need sample preparation with sophisticated techniques or instruments. The drug employed in the study was stable up to 48 hours. This attributes the high quality of the method. The proposed method can be used for the regular analysis of GRS in oral suspension preparations and for everyday use in

quality control laboratories without interference of drug substance.

Ibrahim A. Aljuffali et al (2014) in this article author had developed method was validated in terms of accuracy, precisionand robustness. A excellent linear relationship was observed for GTX in the concentration ranges of 4–40 lg.mL. The correlation coefficient was search to be 0.9998. The inter-day and intra-day precision results were good enough to shown that the proposed method was precise and reproducible. making of samples was simple and efficient. UV detection at 293 nm was found to be suitable. The assay experiment showed that the contents of gatifloxacin estimated in the tablet dosage form, eye drops and SLN were free from the interference of excipients. This demonstrated that the developed HPLC method was easy, rapid as proved by short retention time, precise, accurate, sensitive and efficient and could be conveniently adopted for the routine quality control analysis of gatifloxacin from its pharmaceutical dosage forms and bulk drug. The results of forced degradation studies imply that the developed method is indicted the stability.

Terra Baker et al (2014) the author had Developing a method for the identification and separation of acetyl fentanyl and heroin is of growing importance due to the growing number of reports where acetyl fentanyl is being found in heroin. The development of this method increased in importance for drug analysts in Kentucky when acetyl fentanyl became a schedule I narcotic earlier this year. For more presently, acetyl fentanyl was given temporary placement as a schedule I controlled substance by the Drug Enforcement Administration. With the personally scheduling of acetyl fentanyl, it would be a good assumption that other fentanyl analogs will also be individually scheduled. The objective of this research project was to develop a method that could successfully separate acetyl fentanyl and heroin so that both could be positively identified in samples together. That's why this research project was successful in developing and validating a new method for the separation of acetyl fentanyl in heroin, more research could be done to further process and expand the method. In future studies, if the few remaining ions, 369 and some smaller ions, from heroin that carry over into the acetyl fentanyl and fentanyl spectra could be removed, it would be very beneficial. Also, if the source of the other extra ions present in the mass spectra, 279, 320, 429, and 503 could be determined that would be useful. Another topic for further study would be if the method developed in this research project could be applied to other drugs that have similar retention times and determining if the method developed would work to separate those as well.

Conclusion

The analytical method is described the simple techniques of HPLC method validation and method development of optimizing method. The analytical method proof of an analyst need data for a given analytical issues, sensitivity, accuracy, and range of analyst. Analytical process required to be validated before their introduction through the regular use. When the condition and process change which the method have need to be validated. Development of method include the series of simple steps. All the condition are optimized as need for the purpose of the separation and the method is validated as per guidelines of ICH. The validated data and method can be documented.

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