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A Recent Research Review- Development and Validation with liquid chromatography

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ABSTRACT:

Nowadays many different methods of HPLC are uses for Development of Drugs. HPLC are used to able separate, quantify and detection of drug and drug related substance that can form on manufacturing or storage of drugs. Different type of Chromatographic parameters are evaluated in order for optimize the method. A mobile phase, column, temperature of column, gradient and wavelength that are use for stability and comparability of drug and drug substance and also impurities of drugs. HPLC validation method is give all information regarding like Accuracy, precision, specificity, linearity, range and limit of detection, limit of quantification, robustness and system suitability testing as per guidelines of ICH.

KEY WORDS: HPLC, Method Development, Method validation

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INTRODUCTION [1-4]:

High Performance Liquid Chromatography (HPLC) is derived from the classical column chromatography. HPLC is one of the very important tools of analytical chemistry in modern era. In the modern pharmaceutical industry, high-performance liquid chromatography is the big and important analytical tool in all stages of drug discovery, development, and production. HPLC is the techniques of choice for checking peak purity of new chemical compounds, monitoring reaction changes is in synthetic procedures or scale up, measuring and evaluated new formulations and carrying out quality control / assurance of the final drug formulation or product. The main purpose of HPLC method is to separate, quantify the main drug, any reaction impurities present in chemical compounds. Number of drugs now manufacturing and place in market every year. This new drugs have different structural modification. So the objective of this analytical method is to obtain consistent, accurate data and reliable. This analytical validation method are more useful in achieving this goal. The principle of HPLC is the solution of sample is injected into a column of porous material (stationary phase) and liquid phase (mobile phase) is pumped at high pressure through the column. The separation principle is followed the adsorption of solute on stationary phase based on its affinity towards the stationary phase. The successful use of HPLC is the possible problem requires the right combination of variety of operating conditions like the type of column packing and mobile phase,

column length and diameter, mobile phase flow rate , column temperature and sample size. Today reversed-phase chromatography is the more commonly used separation technique in HPLC because of it broad application range. It is estimated that over 60% to 90% of all HPLC separations are carried out in the reversed phase chromatography. The reasons behind in it simplicity, versatility and scope of the reversed-phase method as it is able to handle compounds of a diverse polarity and molecular mass.

Characterization the new method for new drugs characterization. [5-9]

- The drug and drug combination may not be involved in any pharmacopeia.
- A selected analytical procedure for the drug may not be present in the literature due to patent rules and regulations.
- Analytical methods may not be available for the drug in the form of a manufacturing due to the interference caused by the formulation ingredients of drugs.
- Analytical methods for the quantization of the drug in biological fluids may not be available in procedure.
- Combination drug's procedure is not available in analytical procedure.
- The existing analytical methods may require costly reagents and solvents. That may also involve cumbersome extraction and separation procedures and these may not be reliable.

Method Development of HPLC [10-16]

The High performance liquid Chromatography method development have no properly methods for new manufactured products or drugs. Alternative methods for old products are decrease the time and cost for good precision and ruggedness. When alternative method proposed is intended to replace the existing procedure comparative laboratory data including advantages or disadvantages are made available. The main purpose of the HPLC-method is to try & separate, quantify the main active drug, and reaction impurities, all available synthetic materials and drug compounds.

5 Steps are important in method development: [17-20]

1. Understand the physiochemical criteria of drug molecule.
2. Set up the condition of HPLC.
3. Preparation the sample solution for method development of HPLC.
4. Validation of method.
5. Method optimization

Method Validation of HPLC [21-24]

Validation of an analytical process that is established by laboratory studies, that the performance characteristics of the method attach the requirements for the intended analytical uses. Validation is required for any new or existing method to confirm that it is capable of giving reproducible and reliable results, when used by different operators employing with the same equipment in the same or different laboratories. All analytical methods that are used to intend for analysis of any drugs samples will required to be validated. The validation of analytical methods is done as per the United state pharmacopeia guidelines.

Parameters of Validation

Whenever the methods and the conditions are change than have to recommend by FDA, USP, and ICH as below.

- Specificity
- Precision
- Linearity and Range
- Accuracy
- LOD (limit of detection)
- LOQ (limit of quantification)
- Stability of Solution
- System suitability
- Robustness
- Recovery

Research studies of Method development and Validation

Nalini Kanta Sahoo et al (2016) had developed the RP-HPLC process indicating assay of AMX in bulk and in pharmaceutical dosage forms is established. This method is easy, reliable, linear, accurate, sensitive and recoverable as well as low cost for the effective quantitative analysis of AMX in bulk and tablet formulations. The method was completely validated showing satisfactory data for all the method validation parameters tested and method is free from interference

of the other API and additives used in the formulations. So the method is suitable for use of the routine quality control analysis of AMX in API or in pharmaceutical dosage forms.

Harrizul Rivai et al (2016) In this article, TLC technique was developed and validated for the analysis of mefenamic acid in pharmaceutical solid dosage form. The proposed method is simple, accurate and highly selective for mefenamic acid. The satisfactory sensitivity and simplicity make the methods suitable for regular analysis of mefenamic acid in Q.C laboratories.

Madhuri Sharma et al (2016) In this article author describe that it is a simple, fast and sensitive Reverse Phase High Performance Liquid Chromatography method was developed for simultaneous estimation of Clindamycin Phosphate and Benzoyl Peroxide in gel formulation by using BDS Phenomanax Luna C18 (150X4.6) mm, 5µ and 20mM Ammonium acetate buffer pH 4.0: Methanol (45:55% v/v) as mobile phase at flow rate of 1.2 ml/min with detection wavelength of 210nm. Retention times for Clindamycin Phosphate and Benzoyl Peroxide were 4.49 min, 8.78 min respectively. The linearity of developed method was achieved in the range of 10.0-30.0 µg/ml for Clindamycin Phosphate and 25.0-75.1 µg/ml for Benzoyl Peroxide and limit of detection was found to be 0.32 µg/ml and 0.72 µg/ml and limit of quantification was found to be 0.98 µg/ml and 2.19 µg/ml for Clindamycin Phosphate and Benzoyl Peroxide respectively. The % Recovery of Clindamycin Phosphate was found to be (98.45 % -101.0%) and (99.8 % -99.38 %) for Benzoyl Peroxide. In a precision the repeatability % RSD was found to be 0.4 for Clindamycin Phosphate and 0.3 for Benzoyl Peroxide. Change in the ratio of mobile phase ±2.0 ml, Change in flow rate by ±0.2 ml/minute, Change in pH of mobile phase by ± 0.2 . Specificity of the method was ascertained by analysing standard drug and sample. No interfering peaks were found in the chromatogram by the proposed RP-HPLC method.

Kirsten Purschke et al (2016) had described that the one manual and one fully automated analysis method for THC, 11-OH-THC, and free, unconjugated THCCOOH in blood serum were developed and successfully validated according to GTFCh guidelines. Analysis values were concordant, and both methods ensure that only the free THC-COOH concentration is determined and the analysis value is not erroneously raise by THC-COOgluc coextraction and/or cleavage. Regarding LOQs, extraction efficiencies, and precision, both developed methods correspond well to methods from the literature. The LOQ for THC of 1 µg/L required for driving under the influence of cannabis cases in Germany can be reached, and the method can be employed in that context. The best for our knowledge is the first publication on a comprehensively automated classical liquid-liquid extraction workflow in the field of forensic toxicological analysis. Also, the employed analysis system including shaker, centrifuge, and evaporator modules is mentioned for the first time in the literature.

Orchid Ashraf et al (2016) This research paper reports a easy validated reversed phase high-performance liquid chromatography method which is rapid, precise, and highly specific for determination of baicalin. The separation was achieved using thermo scientific C18 ODS Hypersil column (mm) (250x4.6 mm, 5µm) with a mobile phase consisting of methanol to acetic acid (0.2%) at ratio 94:6, a flow rate of 1.0 ml per minute, and detection at a wavelength of 279 nm. The calibration curve was linear, having a correlation coefficient 0.9997 within range of 2.5–100 µg/ml. The retention time for baicalin was less than 4 minutes. Validation was done according to ICH guidelines with respect to accuracy, linearity, specificity, precision, limit of detection and limit of quantification and satisfactory results were we get.

Shashikant B. Landge et al (2015) in this article author said that this is the first method submitted in the literature for the separation and quantification of Apixaban and its process related and degradation related impurities on core shell column. The RP-HPLC method is rapid, specific, linear, sensitive, accurate, precise, and robust. Morethan, the developed method was found to be more selective and rapid with respect to less runtime and low back pressure as compared to conventional HPLC column method. Method is validated as per ICH requirements and based on solution stability study the auto sampler cooler temperature needs to be maintained at 8˚C during analysis. The developed method is stability indicating method which can be applicable for the analysis of everyday and stability samples of Apixaban drug substance and drug products.

C. L. SINGH et al (2015) had described that the developed method was found to be very simple, sensitive, accurate and economical. The reported UV-methods can be used for the analysis of BSF in simulated body fluids, in bulk and in marketed formulations. Thus proposed method

will be suitable for the analysis of besifloxacin hydrochloride.

Roopa Rani et al (2014) described in this article that the present communication reports a simple HPTLC method for fast analysis of contamination of dry fruits by aflatoxin B1 and B2 . Out of the four dry fruits, sample of maize corn and cashew nuts were found contaminated with aflatoxin B1 and B2. Using TLC-MS interface as an attachment confirmation of aflatoxin contamination has been performed.

Ishan R. Sharma et al (2014) had shown that this is a fast, rapid, precise and accurate high performance liquid chromatography method was developed for simultaneous estimation of enalapril and aliskiren in synthetic mixture. The separation was obtained using a mobile phase consisting of acetonitrile and water in ratio of 80:20 and adjusting pH 4.0 with ortho phosphoric acid (10%) using Phenomenex-luna C18 (250 × 4.6 mm, 5µm) column. The flow rate 1.0 mL min-1 and UV detection at 210 nm was employed. The retention time for enalapril and aliskiren was 2.63 min and 7.25 min respectively. Linearity for enalapril and aliskiren was search to be in the range of 2 to10 µg/mL and 15-75 µg/mL respectively. The method was validated as per the ICH guidelines and the results were within the acceptance criteria for precision, linearity, specificity, stability of solution and robustness.

Vinit Chavhan et al (2014) author has described that a simple and new RP-HPLCmethod have been developed for the simultaneous determination of Sitagliptin phosphate and Simvastatin in bulk and tablet dosage formand validated as per ICH guidelines. The results of the validation studies ensured that the proposed RP-HPLC method was also accurate, precise, specific, robust and sensitive. It possessed significant linearity, precision, high efficiency and resolution and no interference from the excipients. The proposed method was successfully applied and can be suggested for the quantitative analysis of Sitagliptin phosphate and Simvastatin in pharmaceutical formulations for QC, where economy and time areessential and to assure therapeutic efficacy.

CONCLUSION

Method development and method validation are interconnect their activites throughout the drug development. In this review described the general method development for the drug and drug combination were discussed. When developed the anylytical method for the drug compound by HPLC should have good practicle knowledge of chromatographic sepration and also know it done with sample and with varying experiment condition in order to achive optimum sepration. The last optimization will be done with the change the gradient slope and flow rate with the concentration of mobile phase modifiers.

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