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Analytical method development and validation: A review

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Abstract
Analytical method development and validation are continuous and interconnected activities conducted throughout the drug development process. The prime purpose of the analytical method development and validation is to prove that proposed analytical method is accurate, specific, precise and robust in the pharmaceutical industry for analysis of a drug moiety. Development of a method is essential for discovery, development, and evaluation of medicines in the pharmaceutical formulation. validation is necessary for proving that an analytical technique is appropriate and suitable for the meant use and this is often a very important requirement for analytical purpose. The review focus on concept need stages in analytical method development and validation.

Keywords: analytical method development, validation, accurate, specific, precise

Introduction
Analytical Chemistry is the branch of Science that uses advance technologies in determining the composition by analytical technique. We can achieve both qualitative as well as quantitative results. Analytical method could be spectral, chromatographic, electrochemical, hyphenated or miscellaneous. Analytical method development is the process of selecting an accurate assay procedure to determine the composition of a formulation. [1-4]

Rapid increase in pharmaceutical industries and production of drug in various parts of the world has risen the demand for new analytical techniques in the pharmaceutical industries. As aresult, analytical method development has become the basic activity of analysis. Recent development in analytical methods has been resulted from the advancement of analytical instruments. The improvement of the analytical method development and analytical instruments have reduced the time and cost of analysis, increased precision and accuracy. Analytical techniques are developed and validated for active pharmaceutical ingredients (API), excipients, drug products, degradation products and related substances, residual solvents, etc.

Analytical approach improvement and validation perform important functions in the discovery, improvement, and manufacturing of medications. The main aim of an analytical measure is to get consistent, realistic, and correct information. Validated analytical strategies play a significant role in achieving this goal. Outcomes from methodology validation may be used to choose the standard, reliability, and consistency of analytical results, that is associated as an integral part of any sensible analytical practice. [1-5]

Analytical method development
Analytical method development is the creation of a set of experimental conditions to perform analytical procedures in chemical samples. Developed analytical methods can be used to identify, separate, quantify, and learn more about the chemical components in drug products intended for commercial manufacturing.

Need for method development
- To identify,
- To separate,
- To quantify, and
- To learn more

about the chemical components in drug products intended for commercial manufacturing.

Life cycle of the analytical method
information. Validated analytical strategies play a significant role in achieving this goal. Outcomes from methodology validation may be used to choose the standard, reliability, and consistency of analytical results, that is associated as an integral part of any sensible analytical practice. [1-5]

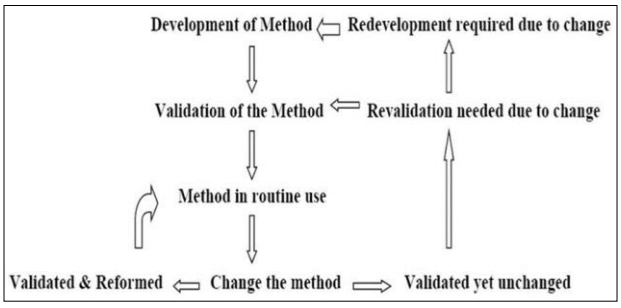

Fig 1: The life cycle of an analytical method [6,7]

Basic criteria for new method development for drug analysis
1. Basic criteria for new method development for drug analysis.
2. The drug or drug combination may not be official in any pharmacopoeias
3. A proper analytical procedure for the drug may not be available in the literature due to patent regulations.
4. Analytical methods may not be available for the drug in the form of a formulation due to the interference caused by the formulation excipients.

5. Analytical methods for the quantitation of the drug in biological fluids may not be available.
6. Analytical methods for a drug in combination with other drugs may not be available.
7. The existing analytical procedures may require expensive reagents and solvents.
8. It may also involve cumbersome extraction and separation procedures and these may not be reliable. [8, 9]

Steps involved in analytical method development

There are various steps in analytical method development which are as follows

- Purpose of Analytical Method Development.
- Highlighting of Steps.
- Analyte standard characterization.
- Requirement of the Method.
- Review of Literature and existing Methodology.
- Choosing an Analytical Method.
- Instrumental setup and initial studies.
- Optimization of Method.
- Documentation of analytical figures of merit.
- Evaluation of method development with actual samples.
- Estimation of percent recovery of real samples and demonstration of quantitative sample analysis.

Validation

Validation is a concept developed in the United States in 1978. The concept of validation has been broaden over the years to achieve many activities like from analytical methods used to control quality of drug substances and drug products up to computerized systems for clinical trials, process control or labelling. Validation is best seen as a necessary and prime part of cGMP. [10]

The word validation means evaluation of validity or the act of proving effectiveness. Validation is a team work involving people from different branches of
plants. Method validation is a "process of establishing documented evidence" that provides a high level of guarantee that the product (equipment) will meet the requirements of the desired analytical applications

Importance of validation

- Assurance of quality
- Minimal batch failure
- Reduction in rejections
- Improved efficiency and productivity
- Increased output
- Reduced testing in process and in finished goods [12]

Types of validation

There are four types of validation

1. Equipment validation

a. Design Qualification
b. Installation Qualification
c. Operational Qualification
d. Performance Qualification

2. Process validation

a. Prospective validation
b. Retrospective validation
c. Concurrent validation
d. Revalidation

3. Analytical method validation

4. Cleaning validation [12]

Types of analytical procedures to be validated

- Identification tests
- Quantitative tests for impurities content
- Limit tests for the control of impurities
- Quantitative tests of the active moiety in samples [12]

Important stages in validation

The action identifying with validation studies can be categorized mainly into three stages:

Stage1

This includes pre-validation qualification stage which covers all exercises identifying with
product studies and improvement, formulation pilot batch testing, scale-up research, exchange of innovation to business scale groups, setting up stability conditions, and managing of in-process, finished pharmaceutical formulations, qualification of equipment, master documents, and process limit.[11]

Stage 2

This involves process validation phase. It is intended to check that every installed limit of the vitalprocess parameter issubstantial and that satisfactory products can be created even below the worst situations.[11]

Stage 3

It is also called as the validation maintenance stage, it requires constant review of all procedure related archives, including validation of the review reports, to guarantee that there have been no modifications, departure, failures, and alteration to the production procedure and that all standard operating procedures (SOPs), involving change control procedures, had been observed. At this phase, the approval team involving people representing all essential departments also guarantees that there have been no modifications/deviations that ought to have brought about requalification and revalidation.

Method validation

Definition

Analytical method validation is "A Documented evidence, which provides a high
degree of assurance that a specific process will consistently produce, a product meeting its pre-determined specifications and quality attributes.

Steps in method validation

1. Develop a validation protocol, an operating procedure or a valida-
   tion master plan for the validation.
2. Define the scope, purpose and applications of the method.
3. Define the performance parameters and its acceptance criteria.
4. Define validation experiments.
5. Verify related performance characteristics of equipment.
6. Qualify materials, ex. Standards and reagent.
7. Perform pre-validation experiments.

8. Adjust method parameters or/and acceptance criteria if required.
9. Perform full internal (and external) validation experiments.
10. Develop SOPs for implementing the method in the routine.
11. Define criteria for revalidation.
12. Define type and frequency of system suitability tests and/or Ana- lytical Quality Control (AQC) checks for the routine.
13. Document validation experiments and results in the validation

Parameters (components) of method validation

1. Accuracy
2. Precision
3. Linearity
4. Limit of detection
5. Limit of quantitation
6. Specificity
7. Range
8. Robustness

1. Accuracy

Accuracy is defined as the closeness of the test results to the true value.

2. Precision

Precision is defined as the measurement of closeness of agreement for
multiple measurements on the same sample.
The precision is expressed as the relative standard deviation.

%RSD = Standard deviation/Mean ×100

3. Linearity

Linearity is the ability of analytical procedure to obtain a response that is directly proportional to concentration (amount) of analyte in the sample.
Linearity is expressed as the confidence limit around the slope of the regression line.

4. Limit of Detection (LOD)

LOD is defined as lowest amount (con-centration) of analyte in a sample that can be detected or identified, not quantified. LOD is expressed as a concentration at a specified signal: noise ratio, usually 3:1.

LOD = 3.3 × S/ SD

5. Limit of Quantitation (LOQ)

LOQ is defined as lowest amount (concentration) of analyte is a sample that can be quantified. For LOQ, ICH has recommended a signal: noise ratio 10:1.

LOQ = 10 × S/SD

6. Specificity

Specificity is defined as the ability of an analytical method to measure the analyte clearly in the presence of other components.
This definition has following implications:
a. Identification
b. Purity tests
c. Assay

7. Range

The range of the method is the interval between upper level and lower level of analyte that have been determined with acceptable accuracy, precision and linearity. It is determined on either a linear or nonlinear response curve and expressed in the same unit as the test results are expressed.

8. Robustness

Robustness is defined as the measurement of capacity analytical procedure to remain unaffected by small variations in method parameters.[13]

Conclusion

This artical gives an idea about what is analytical method development, need, basic criteria and stages involved in analytical method development and validation, its process, steps involved in validation, parameters of validation. The primary objectives of development of analytical methods are for identification, purification and eventually to qualification of any necessary drug etc. The development of analytical methods helps in understanding the critical process parameters and to reduce their effects on precision and accuracy. Validation is a necessary technique in the Pharma sector and that used to ensure that quality work is done in the process which supports the development of medicine and products.

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