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Short Commentary Open Access

A View on Analytical Method Validation of Drugs

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Short Commentary

After the development of an analytical procedure, it is must important to assure that the procedure will consistently produce the intended a precise result with high degree of accuracy. The method should give a specific result that may not be affected by external matters. This creates a requirement to validate the analytical procedures. Validation is a scientific study which provides high degree of assurance that a specific process will consistently produce the product or service or result. Validation of an analytical procedure is the process by which it is established, by laboratory studies, that the performance characteristics of the procedure meet the requirements for the intended analytical applications. Method validation provides an assurance of reliability during normal use, and is sometime referred to as "the process for providing documented evidence that the method does what it is intended to do."

The main objective of the validation is to demonstrate that the analytical method is suitable for its intended purpose, is accurate, specific and precise over the specified range that an analyte will be analyzed. It gives the judgment of quality, reliability and consistency of analytical result. It is received considerable attention from the regulating bodies as well as the consumer committee. Method validation is also an important requirement of national and international regulatory bodies for new product marketing and clinical trial.

Analytical method validation need to be performed for:

- New analytical methods before using in routine analysis
- For transfer of analytical method
- For current methods when any changes are made to the procedure, composition of the drug product and synthesis of the drugs substances.

When analytical method is developed, it must be validated to show its reliability and consistency. After validation, the method is transferred to the laboratory for use in routine analysis. The receiving laboratory should conduct the verification of the analytical method with its own facilities to demonstrate the laboratory's ability to perform the method successfully. If the verification procedure is not successful, a new alternate method is developed and validated. Verification procedures require only selected validation characteristics to show the suitability for use of the analytical method.

The method validation demonstrates high degree of assurance for the suitability of an analytical procedure. The data obtained from the validation process should be accurate and reliable. Thus, various elements of analytical data quality are important in laboratory for validation:

- Instrument Qualification and calibration
- Reference Standards
- Suitability of Reagents and Chemicals
- Qualified personnel
- System Suitability

These ensure the complete expectations of the tests in validation process.

Validation Characteristics

The major analyses in drugs are: Identification, Impurity and Assay. The analytical method for the determination of these parameters have vital role in the drug quality. These methods should be validated to show that they can give the result for what they are intended. The validation program can be performed for these analytical methods:

- Identification tests
- Quantitative tests for impurities content
- Limit tests for the control of impurities
- Quantitative tests of the active components in samples of drug substance or drug product or other selected components in the drug product

The validation procedures consists of some characteristics parameters that makes the method acceptable with addition of statistical tools. The suitability of an analytical method for the intended purpose is given by validation characteristics. Some of the common typical validation characteristics which may be considered are listed below:

- Accuracy
- Precision
- Specificity
- Detection Limit
- Quantitation Limit
- Linearity
- Range
- Robustness

The requirement of validation characteristics depends on the type of analytical method. The validation characteristics are to be evaluated on the basis of the type of analytical procedures (Table 1).

The validation characteristics are performed to ensure the quality of drug products. The analytical data obtained can be treated and interpreted for the scientific acceptance. Some statistical tools are also used to interpret the analytical results of the validation characteristics. The validation of analytical methods not only requires the performance of characteristics parameter but also the statistical treatments of the analytical data. Some of the statistical tools may be helpful in the interpretation of analytical data. Many descriptive statistics, such as the mean and standard deviation, are in common use. Other statistical

tools, such as calculating confidence interval, outlier tests, etc. can be performed using several different, scientifically valid approaches. The acceptance of the variation of the analytical data is determined by these statistical treatments.

Characteristics	Type of Analytical Procedures			
	Identification	Impurities		Quantitative Tests
		Quantitative	Limit	
Accuracy	Not evaluated	Evaluated	Not evaluated	Evaluated
Precision	Not evaluated	Evaluated	Not evaluated	Evaluated
Specificity	Evaluated	Evaluated	Evaluated	Evaluated
Detection Limit	Not evaluated	Not evaluated	Evaluated	Not evaluated
Quantitation Limit	Not evaluated	Evaluated	Not evaluated	Not evaluated
Linearity	Not evaluated	Evaluated	Not evaluated	Evaluated
Range	Not evaluated	Evaluated	Not evaluated	Evaluated

Table 1: Evaluation of validation characteristics.

Successful validation of analytical method requires prior identification of steps and approach of validation process. It requires ability and availability of the resources and proper estimation of time duration. Proper planning and its execution to the validation process improve it economically, physically and environmentally. Its correct implementation ensures consistency of validation project. The results and data obtained from the validation process are summarized and documented. The calculations and statistical interpretation of the analytical data may be performed using Microsoft Excel. It requires the involvement of top to low level staff of the laboratory.

Validation protocol

Method validation should have a protocol before its initiation. Protocol is an approved written document of the method validation process. It is a step by step guide for preparing resources and performing method validation. It includes the methodology and the details of the resources required in the validation process. It includes the step by step process of the validation process and the acceptance criteria. Each validation process in the protocol is followed by blank worksheet for the entry of the observation and calculation/results obtained during the validation process. Any changes made during validation are also included in the protocol.

A method validation protocol should contain following sections:

- Purpose of study
- Details of the study
- Method of Analysis
- Resources and their information
- Validation Steps with worksheet and Acceptance Criteria
- Remarks
- Conclusions

We have some of the guidelines from different organizations. The main objective of these guidelines is to achieve valid analytical method. These guidelines regulate the validation procedure with some requirements for the acceptance of the analytical method for the analysis of a drug material or a drug product. These guidelines are important to assure that the results obtained from the analytical method ensure the quality and safety of the products.

Some of the validation guidelines are:

- ICH Guidelines, Validation of Analytical Procedures: Text and Methodology Q2(R1), 2005
- U.S. FDA – Guidance for Industry (draft): Analytical Procedures and Methods Validation: Chemistry, Manufacturing, and Controls and Documentation, 2000
- ISO/IEC 17025 - General requirements for the competence of testing and calibration laboratories, 2005
- USP Chapter <1225> Validation of Compendial Methods Validation of Analytical Procedures, British Pharmacopeia.