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Review Article

A review on specific and sensitive analytical method development and validation

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ABSTRACT

A specific and sensitive analytical method development and validation plays an important role for building drug discovery, product development and manufacturing of pharmaceuticals. The main purpose of this review article is to check the report of specific and sensitive analytical method development and validation parameters involved in different pharmaceuticals manufacturing. Development of sensitive analytical method is extremely much significant because it ensures product efficacy and product quality. The sensitive analytical method development and validation is the process of proving that the analytical method meets the analytical acceptance criteria and it can use to measure the concentration of target molecule at its specification limit. The aim of method development is to produce a reproducible specific and sensitive analytical method in a cost effective manner. The parameters of validation involved are introduced as specificity, method precision, accuracy, linearity, limit of detection (LOD), limit of quantitation (LOQ), ruggedness, robustness and system precision of the target molecule.

1. Introduction

Specific and sensitive analytical method development is the prerequisite of method validation. It involves analytical chemistry that deals with quantification and qualification of target molecules.[1] Specific and sensitive analytical method development is required for noncompendial method. Based on molecular nature and physiological properties of target molecule, critical quality attributes of the method selected. After the selection of analytical method, method validation conducted. Based on method validation report, analytical test procedure has updated. Specific and sensitive analytical method development is the continuous process of solving problems finding at any site of stage of product development.[2] The analytical chemistry and both quantification and qualification of target molecule conducted based on current good manufacturing practices (cGMP) and USFDA (Food and drug administration, USA) regulations.[3]

2. Discussion

2.1. Sensitive & specific analytical method development

Analytical method development introduces a procedure for unknown compound identified and quantified from sample matrix. When no detection technique available in compendia, specific molecule method development needs to initiate based on molecular nature, pH, pKa and other physicochemical properties. Based on method development, method validation conducted for the target molecule. However, any problem finds at the time of routine use of that analytical method, method development initiates and revalidated.[4]

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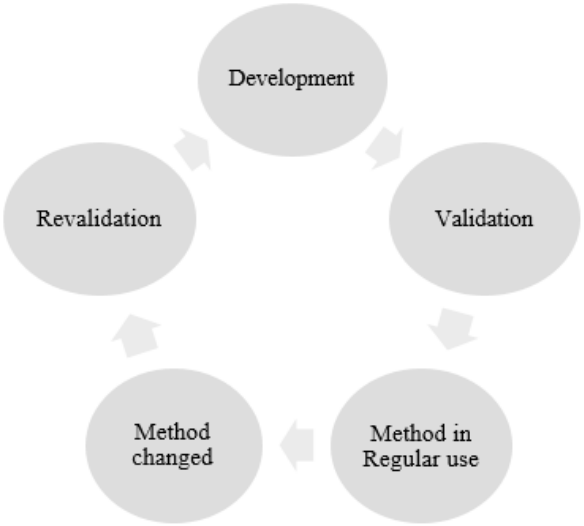

Fig. 1: Flow chart for life cycle of analytical method development

3. Analytical Method Validation

The goal of analytical method validation is to produce evidence that the method is suitable for its intended purpose. Validation involves Accuracy, Specificity, Method precision, Intermediate precision, Linearity, Limit of detection (LOD), Limit of quantitation (LOQ) and Robustness.

3.1. Accuracy

The accuracy of an analytical procedure is the closeness of agreement between the value which is accepted either as conventional true value or an accepted reference value and the value found. The accuracy of method can confirm by analyzing 9 determinations covering three different concentration of triplicate sample.

Accuracy of prochlorperazine maleate (PRO) and betahistine hydrochloride (BET) was confirmed by the addition of standard solution among three different concentration for triplicate samples (50%, 100%, and 120%). A defined amount of drug was added to the test sample and percentage recovery calculated. When this method was used for accuracy, the recovery was found to be 99.5% for betahistine hydrochloride and 99.7% for prochlorperazine maleate.[5]

4. Method Precision (Repeatability)

The method precision is studied by preparing six different sample solutions of 100% concentration.

The method precision of prochlorperazine maleate (PRO) and betahistine hydrochloride (BET) was confirmed by the addition of standard solution by repeated injection (n = 6) of BET (15μg/ml) and PRO (15μg/ml). The % RSD found for six repeated injection of standard solution is 0.227% for prochlorperazine maleate (PRO) and 0.219% for betahistine hydrochloride (BET).[5]

4.1. Limit of detection and limit of quantitation

Limit of detection is the lowest amount required to detect the analyte by specific and sensitive analytical method and it can be calculated based on calibration curve

LOD = 3.3 x /S

Where,

= Standard deviation of the response obtained from calibration curve

S = Slope of calibration curve

Limit of quantitation is the lowest amount required to quantify the analyte by specific and sensitive analytical method and it can be calculated based on calibration curve

LOQ = 10 x /S

Where,

= Standard deviation of the response obtained from calibration curve

S = Slope of calibration curve

4.2. Linearity study

Linearity study performs by preparing at least five different concentrations by dilution of the standard stock solution.

The linearity study of paracetamol was performed by preparing 5 different concentrations among (6.25, 12.5, 25, 50, and 100μg/mL) and found the correlation coefficient between concentration 6.25 to 100 μg/mL is 0.999.[6]

4.3. Intermediate precision

Intermediate precision study performs by a typical variation from method precision (Instrument, different day, different analyst, different column etc.)

4.4. Specificity

Specificity confirms whether the method is specific and sensitive for the target analyte or not. In specificity, there should be no interference at the elution zone of the target analyte for blank, placebo or other degradants.

Specificity was conducted to identify the elution zone of each drug in a mixture and in the sample. The elution zone of standard drugs individually was determined, and it was found to be 3.750 min and 1.533 min for nitazoxanide and elution zone of both drugs in the standard mix was found to be 3.760 min for nitazoxanide and 1.542 min for ofloxacin respectively.[7]

5. Conclusion

This article provides information regarding method development to method validation. Method validation is an integral part of pharmaceutical manufacturing company

for the support of production. It confirms product quality. So, specific and sensitive analytical method is a must for pharmaceutical product quality assurance.

6. Source of Funding

None.

7. Conflict of Interest

None.

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