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Review Article

METHOD VALIDATION OF COMPENDIAL AND NON COMPENDIAL UV-VISIBLE SPECTROSCOPIC METHOD FOR DRUG SUBSTANCES AS PER USP AND ICH GUIDELINES

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Methods Validation is a critical quality attribute for the evaluation of any drug substance through an established method in the quality control laboratory. Method Validation is also the main regulatory requirement in pharmaceutical analysis with compliance as per the guidelines or chapter any pharmacopeia of the same scope. Method on UV spectrophotometer can be developed which is called non-compendial method and method which is provided from pharmacopoeia is compendial Method. Validation is establishing documented evidences, which provides a high degree of assurance that a specific process will consistently produce a product meeting its predetermined specifications and quality characteristics. Validation is considered a good manufacturing practice (GMP) activity; validation experiments must be properly documented and performed on qualified and calibrated instrumentation and equipment. At this stage, there should be documented evidence that the method is robust. The USP has published specific guidelines for method validation for compound evaluation. USP defines eight steps for validation which are Accuracy, Precision, Specificity, Limit of detection, Limit of Quantitation, Linearity and range, Ruggedness, Robustness. This review was written to assist chemists/analysts to perform for method validation on UV spectrophotometer. This review study may facilitate to academics and pharmaceutical industry personnel to know the analytical method validation of UV Spectrophotometer as per USP and ICH guidelines.

Keywords: Validation, UV Spectroscopy, USP, ICH, Regulatory, QC Lab

INTRODUCTION

Validation is establishing documented evidences that provide a high degree of assurance that a particular method can systematically produce a product meeting its preset specifications and quality characteristics. Validation is taken into account of good manufacturing practice (GMP) activity; validation experiments should be properly documented and performed on qualified and calibrated instrumentation and instrumentation. At this stage, there ought to be documented proof that the method is robust.[1]

Spectroscopy may be a branch of science addressing the study of interactions of electromagnetic radiation with matter. Spectroscopy is one in every of the foremost powerful tools offered for the study of atomic and molecular structure and is employed in analysis of a large vary of sample.[2]

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UV spectroscopy is sort of absorption chemical analysis in which light of ultra-violet region (200-400 nm.) is absorbed by the molecule

Fig. 1: UV Spectrophotometer[2]

Absorption of the ultra-violet radiations ends up in the excitation of the electrons from the ground state to higher energy. The energy of the ultra-violet radiation that's absorbed is up to the energy distinction between the bottom state and higher energy states.[3]

Validation is a vital a part of quality control and quality assurance. Various regulatory authorities offer particular emphasis on the validation of all the processes utilised within the industry. The analytical techniques talk to the approach of performing arts the analysis. All the investigatory procedures that are planned for examining any specimen need to be approved. [4]

Validation protocol can be a document that indicates the company's approach to validation of analytical procedures. It ensures consistent and economical execution of validation comes and jointly answers auditor throughout audits.

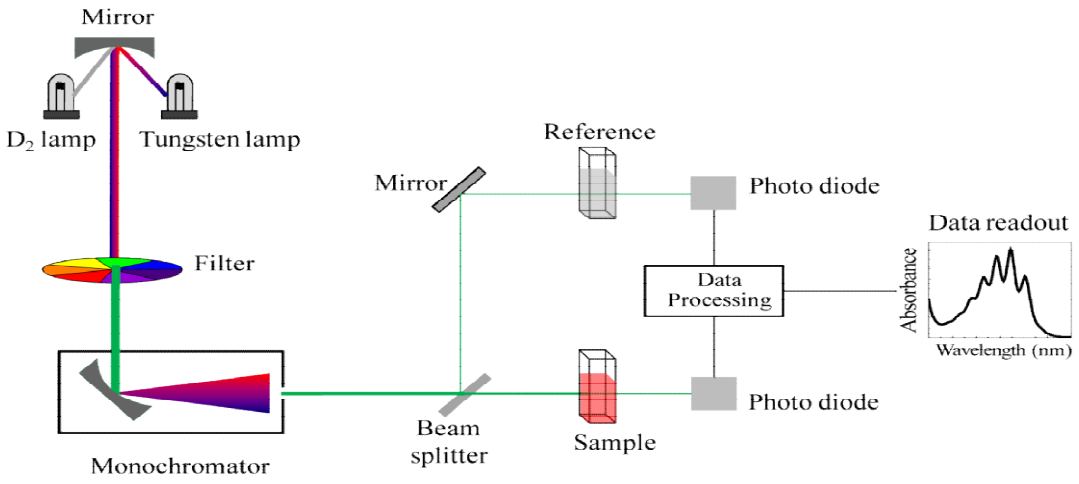

Fig. 2: Schematic Diagram of UV Spectroscopy[2]

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Method development by UV-Spectrophotometric methods:

The drug or drug combination might not be official in any pharmacopoeias. A correct analytical procedure for the drug might not be offered within the literature because of patent regulations; Analytical ways might not be offered for the drug within the kind of a formulation because of the interference caused by the formulation excipients. Analytical ways for the quantization of the drug in biological fluids might not be offered, Analytical methods for a drug together with different medication might not be offered the existing analytical procedures could need costly reagents and solvents.[5]

- Selection of the solvent.
- Selection of analytical wavelengths.
- Study of Beer-Lambert's Law.
- To perform analysis of ordinary laboratory mixture and tablet formulations by proposed technique.
- To validate the developed ways by mistreatment totally different statistical parameters.

Analytical Method Validation

Analytical method Validation may be outlined as (ICH) "Establishing documented proof that provides a high degree of assurance that a particular activity can systematically produce a desired result or product meeting its preset specifications and quality characteristics.[6]

Validation Parameters:

1. Accuracy

Table 1: Validation Parameters

Parameters		Compendial Method	Non-Compendial Method
Accuracy		Yes	Yes
Precision	Method Precision	Yes	Yes
	Intermediate Precision	Yes	Yes
Specificity		Yes	Yes
Limit of Detection		Yes	Yes
Limit of Quantification		Yes	Yes
Linearity / Range		Yes	Yes
Robustness		Not required	Yes

This is outlined as the closeness of agreement between a test result and also the accepted reference value (combination of random and systematic errors). The measure of the trueness is expressed by the bias, that is that the distinction between the expectation of the test results and an accepted reference value. The accuracy of a technique may be determined by performing recovery experiments, implementing standard addition calibration procedures, testing reference materials, etc. It's conjointly potential to compare the test results of a new method with those of an existing totally valid reference method through "cross validation" experiments. Accuracy is usually determined by recovery studies during which the analytes are spiked into a solution containing the matrix. [6]

2. Precision

A. Method Precision (Repeatability)

The repeatability of the analytical procedure is assessed by measuring the concentrations of six independently prepared sample solutions at 100% of the assay test concentration. Alternatively, it may be assessed by measuring the concentrations of 3 replicates of 3 separate sample solutions at totally different concentrations. The 3 concentrations ought to be close enough in order that the repeatability is constant across the concentration range. If this is often done, the repeatability at the 3 concentrations is pooled for comparison to the acceptance criteria. Six sample solutions containing the 100% target level of analyte will be prepared. Three replicates will be made from these sample solutions according to the final method procedure. [6]

B. Intermediate Precision (Ruggedness)

The effect of random events on the analytical precision of the method should be established. Typical variables embrace performing the analysis on completely different days, and/or having the method performed by 2 or more analysts. At a minimum, any combination of a minimum of 2 of those factors totalling six experiments can give an estimation of intermediate precision. Intermediate precision (within-laboratory variation) are demonstrated by 2 analysts, using UV-Visible spectrophotometer systems on completely different days. standard preparation, Sample Preparation are present

with three replicates. [6]

3. Specificity

Specificity is the ability to assess unequivocally analyse within the presence of parts which can be expected to be present." In UV-Vis measurements, specificity is ensured by the utilization of a reference standard where possible and is demonstrated by the lack of interference from alternative components present within the matrix. The specificity of this technique is investigated by analyzing the sample to demonstrate the absence of interference with the elution of analyte. 3 replicates of standard solution, 3 replicates of sample solution.[6]

4. Limit of Detection

The detection limit (DL) will be estimated by calculating the standard deviation of NLT six replicate measurements of a blank solution and multiplying by 3.3. as an alternative, the standard deviation will be determined from the error of the intercept from a calibration curve or by determining that the signal-to-noise is >3.3. The estimated DL should be confirmed by analyzing samples at the calculated concentration. Six Replicates of Blank solution which can show the absorbance. Calculate the standard Deviation of six Replicates and multiply by 3.3 for Limit of Detection. The Limit of Detection is established by variance obtained from six replicates of blank using following formula. [7]

LOD = Standard Deviation × 3.3

5. Limit of Quantitation

The Quantitation Limit (QL) will be estimated by calculating the standard deviation of NLT six replicate measurements of a blank solution and multiplying by ten. Alternatively, the standard deviation will be determined from the error of the intercept from a calibration curve or by decisive that the signal-to-noise is >10. Measure of a test solution prepared from a sample matrix spiked at the specified QL concentration should be performed to substantiate sufficient sensitivity and adequate precision. The ascertained signal -to-noise at the specified QL ought to be >10. Six Replicates of Blank solution which can show the absorbance. Calculate the standard Deviation of six Replicates and multiply by ten for Quantification Limit.

Table 2: Acceptance Criteria of Validation Parameters

Validation Parameter	Acceptance Criteria
Accuracy	98 % to 102 %
Method Precision	RSD 2 %
Intermediate Precision	RSD 2 %
Specificity	No interference
Limit of Detection	>2 times baseline
Limit of Quantitation	S/N ratio 10:1
Linearity/Range	$R^2 \geq 0.99$
Robustness	Same as method precision

The Limit of Quantification is established by variance obtained from six replicates of blank using following formula.[7]

LOQ = Standard Deviation × 10

6. Linearity and Range

Linearity:

A linear relationship between the analyte concentration and UV-Vis response should be demonstrated by preparation of NLT 5 standard solutions at concentrations encompassing the anticipated concentration of the check solution. The standard curve is then evaluated using acceptable statistical methods like a least-squares regression. Deviation from linearity results from either instrumental or sample factors, or both, and might be reduced to acceptable levels by reducing the analyte concentration and thereby the associated absorbance values. [8]

Range:

The operational range of an analytical instrument (and the analytical procedure as a whole) is that the interval between the higher and lower concentrations (amounts) of analyte within the sample (including these concentrations) that it's been demonstrated that the instrumental response perform contains a appropriate level of precision, accuracy, and linearity. [8]

7. Robustness

The reliability of an analytical measure is demonstrated by deliberate changes to experimental parameters. For UV-Vis this may include measure the stability of the analyte

underneath given storage conditions, varying pH, and adding possible interfering species, to list some examples. Robustness is set at the same time employing a appropriate design for the procedure. [9]

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Conflict of Interest

The authors declare that they have no conflict of interest.