International Journal of Scientific Research in Science and Technology
Available online at : www.ijsrst.com
Print ISSN: 2395-6011 | Online ISSN: 2395-602X
doi : https://doi.org/10.32628/IJSRST52411162

A Review on Analytical Method Validation

Ms. Nilam Bavdhane*[1], Dr. Vijaya Barge[2]
[1] Bachelor of Pharmacy, PDEA's Shankarrao Ursal College of Pharmaceutical Science's and Research Centre, Kharadi, Pune-14, Maharashtra, India.
[2]Vice principal, pharmaceutical quality assurance department, PDEA's Shankarrao Ursal College of Pharmaceutical Science's and Research Centre, Kharadi, Pune-14, Maharashtra, India

ARTICLEINFO

Article History:
Accepted: 02 Feb 2024
Published: 09 Feb 2024

Publication Issue :
Volume 11, Issue 1
January-February-2024

Page Number :
432-444

ABSTRACT

The process of finding new drugs, releasing them onto the market, and developing them further to get a marketing approval all depend critically on the development of sound analytical method(s). Aims to achieve; this study reviews the development, optimization, and validation of the drug's technique. Product from the formulation's early stages of development to the product's commercial batch. Approach development for the interested party in process tests, the sample, or the final product manufacturing of the therapeutic product and to offer useful methods for figuring out selectivity, specificity, detection limit, quantitation limit, linearity, range, accuracy, precision, stability of the recovery solution, liquid chromatographic techniques' resilience and robustness to enable routine, in-process, and analysis of stability.

Keywords : Analytical Method Development, Validation Parameters, Acceptance Criteria

I. INTRODUCTION

The reliability of an analytical finding's dependability is extremely important in guiding the formulation of scientist in the process of development stage and impurity profile in the dissolution data and stability study as well as regular analysis. The significance of validation lies in generating consistent and reliable for routine analysis as well as stability analysis. This is particularly valid in relation to quality control and accreditation, which are now concerns about growing significance of analysis in dissolution and the impurity profile in recent years. Therefore, this topic should be broadly discussed on an international level in order to arrive at an agreement regarding the number of validation experiments and on acceptance criteria for validation parameters of the analytical methods[1,31-35].

NEED OF ANALYTICAL METHOD VALIDATION:

When analysing the registration batch and accelerated stability testing samples in the laboratory,

it is imperative to utilise analytical methods that have been thoroughly validated and well-characterized in order to produce reliable results. Furthermore, it's critical to stress that every analytical method has unique qualities that differ from between analytes. In these cases, particular validation criteria need to be created for each analyte. Moreover, the suitability of additionally, the technique could be impacted by the final goal of the research. After a sample Analysis for a particular study is carried out at multiple sites and a commercial batch for individuals consumption, it's essential to confirm the analytical technique(s) in accordance with ICH guidelines and to give accurate validation data for various sites, various parameters, and to determine the intra- and inter-laboratory reliability[2,81,82].

ANALYTICAL METHOD DEVELOPMENT AND VALIDATION:

The process by which a particular analytical method is to be developed for drug products from the stage of in process to the final product's and minivalidation must be completed prior to beginning analyses of routine samples, investigation samples and stability.

Development of analytical methods and finalizing the method consist of:

1) Creating a uniform working standard based on reference standards.
2) Improving the chromatographic environment, standard and sample solution concentrations and the sample extraction process.
3) Verification of analytical methods, or minivalidation must be completed prior to analysis (standard samples) analyses such as dissolution and assay and associated material under development laboratories, etc.
4) Before beginning the validation process, the acceptable Findings from the mini validation should indicate formulation needs to be completed[3,76-78].

DEFINATION OF VALIDATION:

It is accepted that during the course of a typical drug product development program, a defined analytical method will undergo many modifications because composition changes, lower strength may be added or percentage of coating material may change on the formulation. Because of the changes the analytical method may be modified and if modified it should be verified so it requires different levels of validation. Two different levels/types of method validations, complete validation and partial validation or mini, validation, are defined and characterized as follows.

1) Complete validation:

Complete validation is necessary before executing clinical batch or registration batch of drug product. If any modification in the formulation or if any impurity found in the stability study the existing method to be modified and validated again, and the parameters are checked as per ICH guidelines.

2) Mini validation:

Mini validations is required for all the test methods like Assay, Related substance, UOD and Blend Uniformity for analysing the routine samples prior starting the complete validation some parameters to be checked as per ICH guidelines[4,38-40].

REASONS FOR VALIDATION:

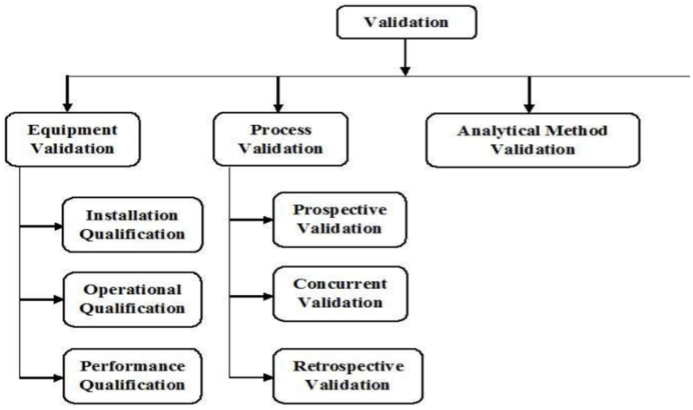
- It is a mandatory condition for enrolment of any pharmaceutical item or pesticide plan.
- It supports to accomplish the scope of legitimate/reference technique" endorsed by administrative offices.
- It ensures a high caliber of the outcomes.
- It improves the money-related main concern of the research facility.
- It is an obligatory necessity for accreditation of the research center by ISO 17025 rules.
- It helps in arriving at the confession of the drugs by worldwide organizations[5-6,56-58].

IMPORTANCE OF VALIDATION:

1) Assurance of quality.
2) Minimal batch failure.
3) Decreased number of rejections.

4) Enhanced effectiveness and output.
5) Higher production.
6) Less testing both during and after completion items[7,48-55].

Validations are of different types which are given below:



EQUIPMENT VALIDATION:

Equipment Validation is a detailed process of confirming that an instrument is installed correctly, that it is operating efficiently, and that it is performing without error[8,59].

PROCESS VALIDATION:

"Process validation" is establishing documented evidence which provides a high degree of assurance that specific processes consistently produce a product meeting its predetermined specifications and quality attributes"[9,60].

ANALYTICAL METHOD VALIDATION:

Validation of Analytical Methodology, to proceed with the chemical evaluation, validation pertaining to the analytical method is a fundamental requirement. The process of carrying out different assessments meant to determine whether a way of analysis provides a reasonable and expected explanation and demonstrates their capacity to provide profitable and acceptable measurement in accordance with the rules. In accordance with the rules and regulations, the approach should offer beneficial information that guarantees the product's quality. To ascertain such, the material is subjected to multiple testing outcomes. A thoroughly validated approach ought to meet every requirement. The analytical method's validation ought to consist of testing the excipients and ought to concentrate on standard testing circumstances. All of these conditions demonstrate that the approval of analytical method is specific to the product [59-62].

The objective of Analytical Method Validation:

1) When there are changes in the formulation or if changes are done in the concentration, further validation is not required if and only if the method validation of the analytical method is performed.
2) It decreases the risk of regulatory non-compliance.
3) Critical parameters of the process can be fully understood due to the analytical method.
4) Minimization of interference on accuracy and precision.
5) It is used in authorization of product and marketing licenses for new products which are non-pharmacopeia[10, 11].

CLEANING VALIDATION:

The product must be free from contamination which can only be influenced by the validation of the cleaning process. Removal of unwanted substances from the facilities and equipment used during the process be guaranteed by the technique of the cleaning. The unwanted contamination should be less than that of a regulatory requirement. In the drug factory, cleaning validation is fundamentally a process. The validation of the cleaning process can be done by different analytical processes. The swab test is the most common test for checking the cleanliness of the equipment. The validation of the cleaning process ought to likewise clarify the development of acknowledgment measures. The correct method for the sampling should be followed. Free from microbial contamination and chemical contamination are vital requirements. The impurities should be less than the detection limit[12,62].

ANALYTICAL METHOD VALIDATION:

For creating trustworthy analytical data from the competent laboratory, a proper standard method should be set up. It can only be possible from the validation of the analytical method. The total information about chemicals should be studied for the set-up of the method and its validation. Reproducible data should be given by the analytical procedure even when performed by the different analysts in various lab centers utilizing distinctive reagents, different instruments, and equipment. For the validation of the analytical method, certain parameters should be followed such as linearity, accuracy, precision, specificity, and reproducibility of the result of the sample. The number of medications presented for consumers has been increasing every day at a higher rate. These medications might contain a fresh element that is not yet seen in the market or there might be small basic alterations or modifications in the structure from the current medication[13, 14].

The following list of essential steps includes drug analysis method validation[63-70]:

1) The assay may be challenging to carry out using the analytical method for biological fluids.
2) The formulation contains a large number of excipients that may cause interference. Because of the patient, access to the full body of literature regarding the drug's analytical techniques is not possible.
3) The current analytical procedures may not be appropriate due to their requirement for expensive reagents and solvents. It may also involve challenging extraction and separation techniques that are inappropriate.
4) The medication or drug combination is not included in any pharmacopeia. Validation of analytical procedure is required by law and must be carried out.
5) The guidelines for the validation of the analytical method have been established by the ICH guidelines [Q2 (R1)].

Analytical technique types that need validation

For the following test, the analytical methods need to be validated:

1) Identity assessments.
2) Impurity analysis for limit testing and quantification.
3) A quantification of the active pharmaceutical ingredient.

1) Identification Tests: An identification test is scheduled to determine the identity of a chemical or ingredient. It can be completed using a variety of analytical techniques. Analysis of a range of characteristics, including spectral analysis, properties of a chromatogram, and interactions with other substances. In this test, the sample and the reference standard are compared.

2) Analysis of the impurities for its quantification and its limit test: It is possible to quantify and identify impurities. Impurities are present in almost all raw materials. Complete elimination of the impurities is an extremely challenging task. Therefore, the regulatory body has established specific guidelines for the limit in the presence of impurities. This test shows the percentage purity of the ingredients. Observing the different parameters: Validation is crucial criterion for quantification analysis, but it is less important in limit tests.

3) Analysis of API for quantification: The most important aspect of the analytical test is the quantification of API or other chemicals. It shows that the drug product contains the API accurately and that it functions as intended. In relation to this archive, the assay is the measurement of a pharmaceutical ingredient that is active in the product qualitatively. The quantification of API ought to adhere to a specific process with identical parameters of confirmation. Similarly, dissolution, which also addresses API release, ought to adhere to the same recommendations for the validation.

Steps in Method Validation[70-75]:

1) Develop a validation protocol or operating procedure for the Validation.
2) Define the application, purpose and scope of the method.
3) Define the performance parameters and acceptance criteria.
4) Define validation experiments.
5) Verify relevant performance characteristics of equipment.
6) Qualify materials, e.g. standards and reagents.
7) Perform pre-validation experiments.
8) Adjust method parameters or/and acceptance criteria if necessary.
9) Perform full internal (and external) validation experiments.
10) Develop SOPs for executing the method in the routine.
11) Define criteria for revalidation.
12) Define type and frequency of system suitability tests and/or analytical quality control (AQC) checks for the routine.
13) Document validation experiments and results in the validation[15].

ADVANTAGES:

1) It builds a degree of confidence, not only for the developer but also to the user.
2) Produces quality products.
3) Reduce the product cost by increasing efficacy, few reject and longer equipment life.
4) Helps in optimization of process or method.
5) Helps in process improvement, technology transfer related products validation and increased employee awareness.
6) It eliminates testing repetitions and leads to better time management in the end[19].

ANALYTICAL METHOD VALIDATION CHARACTERISTICS:

An ICH guideline has set certain criteria for the validation of the analytical method.

The parameters are listed below[16-19]:

1) Specificity.
2) Accuracy.
3) Precision.
   a. Repeatability.
   b. Intermediate Precision.
   c. Reproducibility.
4) Limit of Detection.
5) Limit of Quantification.
6) Linearity.
7) Range.
8) Robustness.
9) Ruggedness.
10) System Suitability.

Besides, revalidation may be essential for the following conditions:

1) Alteration in the process of product manufacturing.
2) Alteration in the ingredients in the final product of the drug.
3) Alteration in the steps of the analytical method (ICH harmonized tripartite guideline, 2005).

The level of revalidation requires relies upon the alteration type. Validation is required for more other alteration.

Indication of sign:

-ve = not essential parameter to performed.

+ve =essential parameter to performed.

(1) = the test of intermediate precision is not necessary to performed if reproducibility test id performed.

(2) Another supporting method can cover the specificity if specificity is unable to perform.

(3) For certain condition it might be required (ICH harmonized tripartite guideline, 2005).

Characteristics	Test of Identification	Kinds of Analytical Procedures		
		Impurities test		Assay
		Quantification	Limit	Test of dissolution (measurement only) Content/Potency
Accuracy	-ve	+ve	-ve	+ve
Precision • Repeatability • Intermediate Precision	 -ve -ve	 +ve +ve(1)	 -ve -ve	 +ve +ve
Specificity	+ve	+ve	+ve	+ve
Limit of Detection	-ve	-ve(3)	+ve	-ve
Limit of Quantification	-ve	+ve	-ve	-ve
Linearity	-ve	+ve	-ve	+ve
Range	-ve	+ve	-ve	+ve

SPECIFICITY (Selectivity) [20,85]:

ICH defines specificity of an assay is the ability to measure accurately and specifically the analyte in the presence of other components that may be expected to present in the sample medium. The term specific generally refers to a method that produces a response for a single analyte only.

Identification: It assures the identification of the ingredient.

Purity Tests: The total removal of the impurities is almost impossible. So certain limits are set for impurities. Impurities can be present in the form of content of residual solvent, heavy metals, related substances, etc. The test of such substances can be done by purity test.

Assay (Content or Potency): It refers to the quantitative determination of the API. API shows the potency of the drug. (ICH harmonized tripartite guideline, 2005).

ACCURACY (Recovery) [21,46-47]:

The accuracy of an analytical procedure expresses the closeness of agreement between the value, which is accepted either as conventional true value or an accepted reference value and the value found, i.e. analytical result. The accuracy of an analytical method is indicated by the recovery of analytical results. It is done by either spiking or taking linear conc. of samples over the range of 80% to 120% of the target concentration with triplicate samples in each concentration. Trueness is another term of accuracy.

Determination methods:

Application of analytical method to an analyte of known concentration:

The accuracy may be determined by application of the analytical method to an analyte of known purity (example: reference standard) and also by comparing the results of the method with those obtained using an alternate procedure that has been already validated World.

Spiked – placebo recovery method:

In this method, a known amount of pure active constituents is added to formulation blank (sample that contains all other ingredients except the active) and then perform the assay of resulting mixture and compare the obtained results with predictable results.

Standard addition method:

In this method, perform the assay of given sample, then add a known amount of active constituent to that assayed sample. After that this sample is again assayed. The difference between the results of the two assays is compared with the expected results.

Recommended Data ICH document recommend that accuracy should be measured using a minimum of nine determinations per 3 concentration level.

The recovery is determined by the equation: $Recovery = Analytical\ Result/True\ Value\ X$ 100%

Limit: The recovery should be in the range of 98.0% to 102.0%.

PRECISION[22,43-45]:

Precision reflects the closeness of agreement of a series of measurements between the series measurement obtained from multiple sampling from the same sample under the same condition at the same time. Precision may be considered in three levels repeatability, intermediate precision and reproducibility.

Repeatability:

Repeatability expresses the precision under the same operating condition over a short interval of time. It is also termed intra-assay precision. A minimum of six replicate sample preparation of a same sample or homogenous sample prepared at the 100% test concentration.

Intermediate precision:

Intermediate precision reflects within-laboratory variations such as different days, different analysts, and different equipment's. Intermediate precision testing can consist of two different analysts, each preparing a six sample preparations, as per specified analytical method. The analysts execute their testing on different days using separate instruments and analytical columns.

Reproducibility:

Reproducibility expresses the precision of a method with in the laboratory variation like different days, different analyst and different equipment's etc. Each testing site can prepare a total of six sample preparations, as per the analytical method. Results are evaluated to ensure statistical equivalence among various testing sites. Acceptance criteria similar to those applied to intermediate precision also apply to reproducibility.

LIMIT OF DETECTION[23,42]:

The lowest concentration of an analyte in a sample that can be identified but may not necessarily be quantified under specified experimental conditions is known as the analytical procedure's limit of detection. It indicates whether the sample is above or below a certain level. LOD is not depend on the procedure of analysis but also on type of instrument.

Measurement is based on:

1) Visual evaluation.
2) Signal to noise ratio.
3) The standard deviation of the response and the slope.

Visual evaluation:

LOD is established by determining the lowest level at which the analyte can be detected and by analysis of samples with known analyte concentrations. Both instrumental and non-instrumental procedures can use it.

Signal to noise ratio:

This approach can only be applied to analytical procedure which shows baseline noise. It is performed by comparing measured signals from samples with known low concentration of analyte with those of blank samples and establishes the minimum concentration at which the analyte can be detected. Signal to noise ratio 2:1 or 3:1 is generally accepted.

The standard deviation of the response and the slope:

$LOD = 3.3\sigma / \mathbf{S}$

Where, σ = Standard deviation of the response. S = Slope of the calibration curve of the analyte from regression line.

LIMIT OF QUANTIFICTION[24,41]:

The lowest quantity of analyte in a sample that can be quantitatively determined and measured with a respectable degree of accuracy and precision under the specified operating conditions of the method is known as the limit of quantification, or LOQ. LOQ may change based on the nature of sample and the technique used. Typically, it is employed to determine impurities or degradation products.

Measurement is based on:

1) Visual evaluation.
2) Signal to noise ratio.
3) The standard deviation of the response and the slope.

Visual evaluation:

LOD is established by determining the lowest level at which the analyte can be detected and by analysis of samples with known analyte concentrations. Both instrumental and non-instrumental procedures can use it.

Signal to noise ratio:

This approach can only be applied to analytical procedure which shows baseline noise. It is performed by comparing measured signals from samples with known low concentration of analyte with those of blank samples and establishes the minimum concentration at which the analyte can be detected.

Signal to noise ratio 10:1 is generally accepted.

The standard deviation of the response and the slope:

LOD = 10σ/ **S**

Where, σ = Standard deviation of the response. S = Slope of the calibration curve of the analyte from regression line.

LINEARITY[25,78,85-86]:

The ability of a method to produce test results that are exactly proportionate to the analyte concentration within a specified range is known as linearity. It is important to assess a linear relationship throughout the analytical process. It can be directly determined on the drug material by diluting a reference stock solution. Visual examination of a plot showing the concentration (on the x-axis) versus the mean response (on the Y-axis) is the best method for determining linearity. Determine the correlation coefficient, Y-intercept, and regression equation. Mathematical estimates of the degree of linearity may be obtained using data from the regression line itself. It is recommended to use at least five concentrations in order to determine linearity.

RANGE[26,86]:

The range of an analytical procedure is the range of analyte concentrations in the sample, both upper and lower, for which it has been shown that the analytical procedure has an appropriate degree of precision, accuracy, and linearity. Usually obtained from linearity studies, and the precise range depends on how the procedure is suggested to be used.

The following minimum specified ranges should be considered:

1. Assay of a drug substance or a finished (drug) product: 80 to 120 % of the test concentration.
2. Content uniformity: 70 to 130 % of the test concentration.
3. Dissolution testing: +/-20 % over the specified range.

ROBUSTNESS[27,87-88]:

It is the measure of the capacity of the analytical method to remain unaffected by small but deliberate changes in procedure to provide an indication about variability of the method during normal laboratory conditions.

Examples of typical variations are: Stability of analytical solutions; Extraction time.

In the case of liquid chromatography, examples of typical variations are:

1) Influence of variations of pH in a mobile phase;
2) Influence of variations in mobile phase composition;
3) Different columns (different lots and/or suppliers);
4) Temperature; Flow rate.

In the case of gas-chromatography, examples of typical variations are:

1) Different columns (different lots and/or suppliers);
2) Temperature; flow rate.

RUGGEDNESS[28]:

Ruggedness is measure of reproducibility test results under the variation in conditions normally expected from laboratory to laboratory and from analyst to analyst. The Ruggedness of an analytical method is degree of reproducibility of test results obtained by the analysis of the same samples under a variety of conditions, such as; different laboratories, analysts, instruments, reagents, temperature, time etc.

SYSTEM SUITABILITY TESTING[29]:

System suitability testing is an integral part of many analytical procedures. The tests are based on the concept that the equipment, electronics, analytical operations and samples to be analyzed constitute an integral system that can be evaluated as such. System suitability test parameters to be established for a particular procedure depend on the type of procedure being validated. See Pharmacopoeias for additional information.

II. CONCLUSION

Analytical method validation and method transfer data playing a fundamental role in pharmaceutical industry for releasing the commercial batch and long term stability data therefore, the data must be produced to acceptable scientific standards. For this reason and the need to satisfy regulatory authority requirements, all analytical methods should be properly validated and documented. The aim of this article is to provide simple to use approaches with a correct scientific background to improve the quality of the analytical method development and validation process. This article gives an idea about number of sample preparation, procedure and acceptance criteria for all analytical method validation parameters in wider range. Applications of analytical method and method transfer are also taken into consideration in this article. These various essential development and validation characteristics for analytical methodology have been discussed with a view to improving the standard and acceptance in this area of research.

III. REFERENCES

[1]. Thompson M, Ellison SLR and Wood R. Harmonised Guidelines for single Laboratory Validation of Method of Analysis. Pure Appl Chem. 2008; 74:835-55.

[2]. Wood R. How to Validate Analytical Methods. Trends Analyt Chem. 2005;54:149-58

[3]. McDowall RD. The Role of Laboratory information Management systems LIMS in Analytical Method Validation. Anal Chim Acta. 2077; 54:149-58.

[4]. Puluido A, Ruusanches I, Boquc R and Rius FX. Uncertainty of results ID routine Qualitative Analysis in Analytical Chemistry. J Pharm Biomed Anal. 2005;22:647-54

[5]. Boggula, N., Kasturi, J., Reddy, P., & Bakshi, V. (2019). Non-steroidal antiinflammatory drugs: an overview. Journal of Drug Delivery & Therapeutics, 9(1-s),442-448

[6]. Behera, S. (2012). UV-Visible Spectrophotometric Method Development and Validation of Assay of Paracetamol Tablet Formulation. Journal of Analytical And Bioanalytical Techniques

[7]. Lavanya G, Sunil M, Eswarudu MM, Eswaraiah MC, Harisudha K, Spandana BN. Analytical method validation: an updated review. International Journal of Pharmaceutical Sciences and Research. 2013 Apr 1;4(4):1280

[8]. Verma P, Madhav NS, KR Gupta V. A review article on pharmaceutical validation and process controls. Pharma Innovation 2012; 1:51-60.

[9]. Borisagar, S. L., Patel, H. U., Patel, A. N., & Patel, C. N. (2017). A Validated HPLC Method for Estimation of Lornoxicam and Paracetamol in their Combined Tablet Dosage Form. Internation Journal of Pharmacetical Sciences and Research, 1683-1686.

[10]. Nash R. A. and Wachter A. H Pharmaceutical Process Validation an International Third Edition. Revised and Expanded, Marcel Dekkar, Inc., New York 2003; 129, P. 760-792

[11]. Bhardwaj SK, Dwivedi K, Agarwal DD. A review: HPLC method development and validation. Int J Anal Bioanal Chem 2015; 5:76-1.

[12]. Dash, D. K., & Vadher, M. (2014). Analytical Method Development and Validation for Simultaneous Determination of Zaltoprofen and Paracetamol in their Combined Solid Dosage

[13].Gaikwad, R., Chadhari, F., Kande, T., & Barge, V. (2017). Development and Validation of Analytical Mehtod for Simultaneous Estimation of Paracetamol and Ibuprofen Using UV Visible Spectroscopy. Human Journal Research Article.

[14].Tangri Pranshu, Rawat Prakash Singh, Jakhmola Vikash: Validation: A Critical Parameter for Quality Control of Pharmaceuticals. Journal of Drug Delivery & Therapeutics 2012; 2(3): 34-40

[15].Ravichandran V, Shalini S, Sundram KM, Rajak H (2010) Validation of analytical methods strategies & importance. Int J Pharmacy and Pharm Sci 2: 18-22

[16].ICH Harmonized tripartite guidelines, Validation of Analytical Procedures': Text and Methodology, Q2 (R1), current step 4 version; 27 Oct 1994. [2]

[17].Naga G. R., Vignesh K., Analytical Method Validation: An Updated Review, IJAPBC- Vol.1 (1), Jan- Mar, 2012.

[18].Ravichandran.V, Shalini s, and Harish Rajak, Validation of analytical Methods-Strategies and Importance. Vol.2, 3 May 2010.

[19].Shah.K, Kumar.S, Upmanyu.N and Mishra.P, Review Article on, "Evaluation of an Analytical Method" IJPCR, Vol.1 issue 1, 2012

[20].Moharana AK, Banerjee M, Sahoo CK, Sahoo NK (2011) Development and validation of RP-HPLC method for Mesalamine. Asian J of Pharmaceutical and Clinical Reserch4:71-73

[21].Hokanson GC (1994) a life cycle approach to the validation of analytical methods during pharmaceutical product development part I: The initial validation process. Pharmaceutical Technology: 118-130.

[22].Sahoo NK, Sahu M, Veerachamy A, Lalitha BV, Moharana AK, et al. (2015) Quantification and validation of simvastatin and ezetimibe in bulk drugs and combined dosage form by reverse phase liquid chromatographic method (RPLC). Pharmaceutical Methods 6: 115-119

[23].Sahoo NK, Sahu M, Praneeth P, Manjeera PSK, Lavanya K (2013) Degradation motitoring method development and validation of spectrophotometric estimation of atrovastatin calcium in bulk and tablet formulation. J Pharm. Biomed Sci 33: 1551-1557.

[24].Patel NK, Subhaiah G, Shah H, Kundlik M, Shrivastav SP (2008) Rapid LC-ESI-MS-MS method for the simultaneous determination of clopidogrel and its carboxylic acid metabolite in human plasma. J Chromatogr Sci 46: 867-875

[25].Sahoo NK, Sahu M, Alagarsamy V, Srividya B, Sahoo CK (2015) Current status of two-dimensional gel validation of assay indicating method development of imatinib in bulk and its capsule dosage form by liquid chromatography. Ann Chromatogr Sep Tech 1: 1010

[26].Renger B, Jehle H, Fischer M, Funk W (1995) Validation of analytical procedures in pharmaceutical analytical chemistry: HPTLC assay of theophylline in an effervescent tablet. J Planar Chrom 8: 269-278.

[27].Vander HY, Nijhuis A, Verbeke JS, Vandegtnste BG, Massart DL (2009) Guidance for robustness/ruggedness test in method validation. J Pharm Biomed Anal 24: 723-53.

[28].Sahoo NK, Sahu M, Rao PS, Indira JN, Rani SN, et al. (2014) Validation of assay for bulk clopidogrel and for some tablet forms by reverse phase high performance liquid chromatography. J of Taibah University for Science 8: 331-336.

[29].Panda SS (2010) Ion pairing RP-HPLC method for simultaneous determination of aspirin and clopidogrel bisulphate in tablet and capsule dosage form. Int J PharmTech Res 2: 269-273

[30].David G Watson, High Performance Liquid Chromatography, Pharmaceutical Analysis-A Text Book for Pharmacy Students and Pharmaceutical Chemists, 1st Ed, 1999, 237-276.

[31].George Lunn, Methoxychlor, HPLC Methods for Recently Approved Pharmaceuticals, 1st Ed, 2005, 387.

[32].Guidance for Industry, Q2 (R1) Validation of

[33].Analytical Procedures: Methodology, U.S.Department of Health and Human Services, Food and Drug Administration, Centre for Drug Evaluation and Research (CDER), Centre for Biologics Evaluation and Research (CBER), International Conference on Harmonization, Geneva, November 2005.

[34].Indian Pharmacopoeia, Vol. I , II & III, Government of India, Ministry of Health and family Welfare, Controller of Publications, Delhi, 2007, Vol-II, 96-98.

[35].Indian Pharmacopoeia, Vol. I , II & III, Government of India, Ministry of Health and family Welfare, Controller of Publications, Delhi, 2007, Vol-II, 129-131.

[36].International Conference on Harmonization (ICH), Validation of analytical procedure: Text and methodology, Harmonized tripartite guideline, Q2 (R1), Geneva, 2005.

[37].Jay Br, Kelvin J, Pierre B, Understanding and Implementing Efficient Analytical Methods

[38].Development and Validation, 2003, Available from: http://www.pharmtech.comLloyd R, Snyder, Joseph J Kirkland, Joseph L Glajch, Practical HPLC Method Development, 2nd Ed., 1997, 179-184.

[39].Mendham J, Denny R C, Barns J D, Vogel's Text Book of Quantitative Chemical analysis, 6th Ed, 2001, 289, 297-305.

[40].Quality assurance of pharmaceuticals: A compendium of guidelines and related materials, Volume 2, 2nd updated edition, Good manufacturing practices and inspection, World Health Organization, 129-131.

[41].Ravisankar S, HPLC, Textbook of Pharmaceutical Analysis, 3rd Ed, 2006, 18(1)-18(15)

[42].Skoog DA, Holler FJ, Crouch SR, Principles of Instrumental Analysis, 6th Ed, Baba barkahanath printers, Haryana, 2007, 816-855.

[43].United States Pharmacopoeia 24, national formulary 19, section <1225> "Validation of compendial methods", US Pharmacopoeial convention, Rockville, 2000.

[44].United States Pharmacopoeia 27, section <621> "system suitability", US Pharmacopoeial convention, Rockville, 2004

[45].US FDA, Technical Review Guide: Validation of chromatographic Methods, 1993,726.

[46].W John Lough, Encyclopedia of Analytical Chemistry: Application, Theory and Instrumentation by R A Meyers, 1st Ed, 2000, 12173-12180.

[47].Willard H, Merritt L, Dean J A, Settle F A, Instrumental methods of analysis, 7th Ed, CBS Publishers & Distributor, New Delhi, 1986, 580-655.

[48].G. David Watson, Pharmaceutical Analysis (3rd Ed., Churchill Livingstone, London: Harcourt Publishers Limited, Essex CM 20 2JE, 2012A.

[49].H. Beckett, and J.B. Stenlake, Practical Pharmaceutical Chemistry (4th Ed., Vol. I & II. CBS Publishers and Distributors, New Delhi: 2007).

[50].T. Higuchi, and Brochman-Hansen, Pharmaceutical Analysis, (3rd edition, CBS Publishers and Distributors pvt. Ltd., New Delhi: 1997.

[51].G. Oliver, R. Gerrit, and VZ. Maxmilian, Leading Pharmaceutical Innovation, „Trends and drivers for Growth in the pharmaceutical industry, (2nd Ed., Springer, 2008)12-15.

[52].Br. Jay, J. Kelvin, and B. Pierre, Understanding and Implementing Efficient Analytical Methods Development and Validation, 2003.

[53].R.M. Christopher, and W.R. Thomas, Quality Systems approach to Pharmaceutical cGMP Development and validation of Analytical Methods, (1st Ed., 2005) 147-152.

[54].R. Lloyd Snyder, J. Joseph Kirkland and L. Joseph Glajah, Practical HPLC method development (2nd Ed., 1997) 179-184.

[55].B.K. Sharma, Instrumental method of chemical analysis (29th Ed., Meerut, Chromatography, HPLC, Goel Publishing House, 2013) 286-385.

[56].H.H. Willard, L.L. Merrit, J.A. Jr. Dean, and F.A. Jr. Settle, Instrumental Methods of Analysis (CBS Publishers, New Delhi: 1986).

[57].R.A. Day, and A.L, Underwood, Quantitative Analyses, (5th Ed., Prentice Hall, New Delhi: 1986).

[58].Macek and Karel, Pharmaceutical Applications of Thin Layer and Paper Chromatography, 62(6) 1972,1032.

[59].G. Ramana Rao, S.S.N. Murthy, and P. Khadgapathi, Gas Chromatography to Pharmaceutical Analysis, Eastern Pharmacist, 30(353), 1987, 35.

[60].G. Ramana Rao, S.S.N. Murthy, and P. Khadgapathi, High Performance Liquid Chromatography and its Role in Pharmaceutical Analysis, Eastern Pharmacist, 29(346), 1986, 53.

[61].C.S.P. Sastry, T.N.V. Prasad, and E.V. Rao, Recent applications of High Performance Liquid chromatography in Pharmaceutical analysis, Indian J. Pharm. Education, 21 (37),1987.

[62].Ravisankar P, Gowthami S, and Devala Rao G, A review on analytical method development, Indian journal of research in pharmacy and biotechnology, 2(3), 2014, 1183-1195.

[63].Ravisankar P, Rajyalakshmi G, Devadasu Ch, and Devala Rao G, Instant tips for right and effective approach to solve HPLC trouble shooting, Journal of chemical and pharmaceutical sciences. 7(3), 2014, 259-274.

[64].Jay Breaux, Kevin Jones, and Pierre Boulas, Development services analytical method development and validation „Pharmaceutical technology, 27(1), 2003, 6-13.

[65].E. Michael Swartz, and Iras Krull, Analytical method development and validation, CRC press, Marcel dekker, Inc., Madison Avenue, New York: 1997.

[66].K. Yuri, and LB. Rosario, HPLC for pharmaceutical scientists, (John Wiley and Sons, Inc., Hoboken, New Jersey, 2007) 92-98.

[67].G. P. Carr, and J. C. Wahlichs. „A practical approach to method validation in pharmaceutical analysis‟, J. Pharm, Biomed. Anal, 8, 1990, 613-618.

[68].United States Pharmacopoeia, 24, National Formulary 19, section „Validation of compendial methods‟. US Pharmacopoeial convention, Rockville, Validation of analytical procedures text and methodology Q2 (R1), November, 2000: 2005

[69].International conference on harmonization (ICH) of technical requirements for registration of pharmaceuticals for human use, Validation of analytical procedures: Text methodology, (Q2 (R1) Geneva, 2005) 6-13.

[70].Draft guidance analytical procedures and method validation, US food and drug administration, Centre for drugs and biologics, Department of Health and Human Services. http://www.fda.gov/cder/guidance/2396 dft.htm#111, 2000.

[71].Orr J.D, Krull I.S and Swartz M.E, Validation of impurity methods Part II, (LC North Am; 21, 2003) 1146-1152.

[72].ICH Q2 (R1), Validation of analytical procedures (definitions and terminology); 2005; pp. 9-10.

[73].D.L. Massart, B.G.M. Vandeginste, S.N. Deming, Y. Michotte and L. Kaufman, Chemometrics: A Text Book, Elsevier, And Amsterdam: 1988.

[74].M.M. Kiser and J.W. Dolan, Selecting the best curve fit LC-GC (Europe: 2004)138-143.

[75].R.E. Moore, Interval analysis (Englewood Cliffs, NJ: Prentice-Hall, 1966).

[76].Development and validation of HPLC method a review. International Re-search Jour- nal of Pharmaceutical and Ap-plied Sciences. 2012; 2(4):22-23.

[77]..N. Toomula, A. Kumar, S. D. Kumar, V.S. Bheemidi, Develop- ment and Validation of

Analytical Methods for Pharmaceuticals, J. Anal. Bioanal. Techniques. 2011; 2(5): 1-4.

[78].B. Nigovic, A. Moran, M. Sertic, Chromatography-The Most Versatile Method of Chemi-cal Analysis, Intech. 385-425 (2012).

[79].K. Kardani, N. Gurav, B. Solanki, P. Patel, B. Patel, RP-HPLC Method Development and Validation of Gallic acid in Polyherbal Tablet Formulation, Journal of Applied Pharmaceuti-cal Science. 2013; 3(5): 37-42.

[80].International Conference on Harmonization, Draft Guidance on specifications, Test procedures and acceptance criteria for new drug substances and products, Chemical Substances. Fed. Regist. 2000; 3(5): 83041-63.

[81].Putheti RR, Okigbo RN, Patil SC, Advanapu MS, Leburu R. Method development and validations, Characterization of critical elements in the development of pharma- ceuticals. Int J Health Res. 2008; 1:11-20.

[82].Wood R. How to Validate Analytical Methods. Trends Analyt Chem. 2005; 54: 149-58.

[83].Vessman J. Selectivity or specifi- city? Validation of analytical methods from the perspective of an analytical chemist in the pharmaceutical industry. J Pharm Biomed Analyt, 1996; 14:867-9.

[84].Hearn Perkin Elmer RA. www.standardbase.com., A Guide to Validation in HPLC Based on the Work of G. M. Holland.

[85].Lindner W, Wainer IW, Require- ments for initial assay validation and publication in J Chromatltography B. J Chromatogr. 2006; 707: 1.2.

[86].Validation of Compendial Proce- dures, Unit-ed State Pharmacopeia, USP 36 NF, 27 (2) (2010).

[87].Validation of Analytical Procedures: Text and Methodology, International Conferences on Harmonization, Draft Revised (2005), Q2 (R1).

[88].Boulanger B, Chiap P, Dewe W, Crommen J, Hubert P. An analysis of the SFSTP guide on validation of chromatographic Bioanalytical meth- odds: progress and limitations. J Pharm Biomed Anal. 2003; 32:753-65.

Cite this article as :

Ms. Nilam Bavdhane, Dr. Vijaya Barge, "A Review on Analytical Method Validation", International Journal of Scientific Research in Science and Technology (IJSRST), Online ISSN : 2395-602X, Print ISSN : 2395-6011, Volume 11 Issue 1, pp. 432-444, January-February 2024. Available at doi : https://doi.org/10.32628/IJSRST52411162
Journal URL : https://ijsrst.com/IJSRST52411162