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Review Article

Development and Validation of New Analytical Methods for Assay of Bioactive Molecules in Various Samples: A Comparative Perspective on Different Regulatory Authorities

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ABSTRACT

The methodical process of determining if an analytical method is appropriate for its intended use is known as analytical method validation. For regulatory submissions or internal use, the technique's developer or user typically produces proof of the method's specificity, linearity, accuracy, precision, detection limit, quantitation limit, ruggedness, and robustness. The quality of the data shown here is directly impacted by the iterative process of technique development and validation. When creating quality and safety compliance data throughout drug product development and post-approval, validated analytical methodologies for qualitative or quantitative testing of drug molecules are crucial. Key elements of the cycle of developing and validating analytical techniques are attempted to be explained in the current study. Additionally, it aims to analyze and compile standards published by different organizations for analytical techniques used to examine pharmaceutical formulations and pharmacological substances in their pure form.

INTRODUCTION

The process of demonstrating that analytical procedures are appropriate for their intended use and that they support the identity, quality, purity, and potency of the drug substances and drug products is known as "establishing a documented proof, which provides a high degree of assurance that a specific process will consistently produce a

desired result at its prearranged specifications and quality characteristics," according to ICH Q2 (R1). When a novel method is created or when established methods are applied in several labs and by different analysts, method validation is necessary. Accuracy, precision, specificity, linearity, range, ruggedness, robustness, detection of limit, and quantitation limit are among the analytical parameters that can be verified,

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according to USP. Accuracy, precision, specificity, detection of limit, quantitation limit, linearity, range, system adaptability, and robustness are among the analytical parameters that can be verified, according to ICH. Accuracy, precision, specificity/selectivity, detection of limit, quantitation limit, linearity, range, system suitability, repeatability, sample solution stability, and robustness are among the analytical parameters that can be verified, according to the FDA. European rules state that the following analytical parameters can be verified: linearity, range, accuracy, precision, specificity, detection of limit, and quantitation limit. This review explains about a thorough comparison of regulatory bodies and their standards/procedures for the validation of analytical methods in pharmaceuticals and related industries. The FDA (USA), EMA (Europe), ICH, WHO, and USP are the main international organizations that establish criteria for the validation of analytical methods; hence they are the focus of the comparison.

Types of analytical procedures to be validated

The following types analytical procedures to be validated.[3]

A) Identification tests

To confirm the identity of an analyte in a sample, identification tests are employed. This is often accomplished by comparing a sample's characteristics (such as its spectrum, chromatographic behavior, chemical reactivity, etc.) to those of a reference standard.

B) Quantitative and limit testing for controlling impurities

One way to test for impurities in a sample is to use a quantitative test to restrict the impurity. A

quantitative test requires different validation settings than a limit test.

C) Measurements of the active ingredient in drug substance or drug product samples

This kind uses assay methods to quantify the analyte in a particular sample. The assay is a quantitative assessment of the drug substance's primary component or components.

2. METHOD VALIDATION'S GOALS AND BENEFITS

Objectives

- a. To get accurate, dependable, and consistent data;
- b. To show that it is appropriate for the purpose for which it was designed.
- c. To act as a basis for recorded production and process control protocols meant to ensure pharmaceutical products' identity, potency, quality, and purity.
- d. To preserve the effectiveness, safety, and quality of the final product;
- e. To oversee each step of the production process;
- e. To produce the best analytical results

Advantages

- It boosts confidence for both developers and users.
- Produces high-quality items.
- Improve product efficiency, reduce rejects, and extend equipment life to Lower costs.
- Optimizes processes or methods.
- Assists with process optimization, technology transfer, product validation, and greater staff knowledge.
- Eliminating testing repetitions improves time management.

CORE VALIDATION PARAMETERS:



Understanding the qualities or parameters involved in the validation process is crucial. The different performance metrics, which are categorized as follows

- Accuracy
- Precision
 - Repeatability
 - Intermediate precision
 - Reproducibility
- Specificity/Selectivity
- Limit of Detection (LOD)
- Limit of Quantitation (LOQ)
- Linearity
- Range
- Robustness
- Ruggedness
- System suitability testing.

a) Accuracy

Closeness of test results obtained by the method to true value" is one way to describe the accuracy of an analytical method. For example, assess the analytical method's accuracy. The assay of a known quantity of analyte in the linearity range is used to express it as a percentage of recovery.

Determination methods

Application of analytical method to an analyte of known concentration

Applying the analytical method to an analyte of known purity (such as a reference standard) and comparing the method's results with those from a different, previously validated procedure are two ways to assess its accuracy.

The spiked-placebo recovery technique

This method involves adding a known quantity of pure active substances to a formulation blank,

which is a sample that contains all other ingredients but the active. The resulting mixture is then assayed, and the results are compared to expected results.

The conventional method of adding

This method involves assaying a given sample and then adding a known quantity of an active ingredient to the tested sample. This sample is then assayed once more. The disparity between the two assays' results is contrasted with the anticipated outcomes.

Recommended Data

According to the ICH document, a minimum of nine determinations per three concentration levels should be used to measure accuracy.

Acceptance criteria

The mean value should be within 15% of the supposed value except at LOQ, where it should not deviate by more than 20%. The deviation of the mean from the nominal value serves as the measure of accuracy.

b) Precision

"Closeness of agreement between a series of measurements obtained from multiple sampling of the same standardized sample under the prescribed conditions" is one way to characterize the precision of an analytical procedure. should be examined with true, homogenous samples. stated as SD/RSD

$$\%RSD = \frac{\text{Standard deviation}}{\text{Mean}} \times 100$$

Repeatability

It conveys precision over a brief period of time under the same operating conditions. i.e., the



analyst uses the same tools and techniques to analyze replicas.

Intermediate precision

It expresses the precision with in laboratories variations' different days, different analyst, and different equipment's etc. It is not necessary to study effects individually.

Reproducibility precision

It conveys the accuracy between laboratories (two-way investigations, typically applied to method standardization) for the inclusion of processes in pharmacopoeias. For example. An examination of precision is part of the validation process for assay and quantitative impurity determination procedures.

Recommended Data

For any kind of precision study, the standard deviation, relative standard deviation, and confidence interval should be provided.

Acceptance criteria

The precision determine date concentration level should not exceed 15% of the coefficient of variation (CV) except for the LLOQ, where it should not exceed 20% of the CV.

c) Specificity

According to ICH, an assay's specificity is its capacity to measure the analyte precisely and precisely in the presence of additional elements that might be anticipated to be present in the sample medium. In general, the phrase "specific" describes a technique that yields a response for just one analyte.

In particular, the ICH paper is divided into three areas.

Tests for identification

to confirm an analyte's identity.

Tests for purity

to guarantee that every analytical method carried out permits an accurate declaration of an analyte's impurity content, such as heavy metals, related compounds, etc.

Assay

to deliver a precise outcome that enables an accurate assessment of an analyte's strength or content in a sample?

d) Selectivity

selectivity of the technique to identify the analyte in the presence of elements that could be anticipated to be present in the sample matrix. It is, in essence, the capacity of a separative approach to resolve various compounds. It is a measurement of two peaks' relative method locations. It is a technique that offers answers for several chemical entities that might or might not be isolated from one another. The test results obtained on the analyte with and without the inclusion of potentially interfering material are compared to ascertain it.

e) Limit of detection

The lowest concentration of an analyte in a sample that can be identified—though not necessarily quantified—under specified experimental conditions is known as the analytical procedure's limit of detection. In essence, it shows whether the sample is over or below a particular threshold. The type of instrument as well as the analysis method will determine the LOD.

The basis for measurement is

- Visual assessment.
- The ratio of signal to noise.
- The response's standard deviation and slope

Ratio of signal to noise

This method is limited to analytical procedures that exhibit baseline noise. It determines the lowest concentration at which the analyte may be identified by comparing measured signals from samples with known low analyte concentrations with those of blank samples. Generally, a signal-to-noise ratio of 2:1 or 3:1 is acceptable.

LOD is $3.3\sigma/s$.

where s is the calibration curve's slope and σ is the intercept's standard deviation.

f) Quantitation limit

The LOQ, which varies depending on the type of method used and the sample's characteristics, is the lowest amount of analyte in a sample that may be quantitatively determined that may be quantified with an acceptable level of accuracy and precision under the state working parameters of the method. It is typically employed to identify contaminants or degradation products.

Visual inspection and signal to noise ratio are the foundations of measurement. The slope and the response's standard deviation.

Visual assessment

Analyzing samples with known analyte concentrations and determining the lowest level at which the analyte can be identified yields the LOQ. Both instrumental and non-instrumental procedures can make use of it.

Ratio of signal to noise

This method is limited to analytical procedures that exhibit baseline noise. It determines the lowest concentration at which the analyte may be identified by comparing measured signals from samples with known low analyte concentrations with those of blank samples. It is generally agreed that the signal to noise ratio is 10:1. LOQ is stated as

$$LOQ = 10\sigma/s$$

Where σ is the standard deviation of the intercept and s is the slope of the calibration curve

g) linearity

The method's ability to produce test findings that are exactly proportionate to the analyte concentration within a specified range is known as linearity. Over the course of the analytical process, a linear relationship should be assessed. By diluting a standard stock solution, it can be directly determined on the drug ingredient. Plotting a graph of concentration (on the x-axis) vs mean response (only on the Y-axis) should be used to visually assess linearity. Determine the correlation coefficient, Y-intercept, and regression equation. The degree of linearity may be estimated mathematically using data from the regression line itself. It is advised to use at least five concentrations to determine linearity

h) Range

The period between the highest and lowest analyte concentration in the sample for which it has been shown that the analytical technique has an appropriate degree of precision, accuracy, and linearity is known as the range of analytical procedure. often obtained from linearity studies, and the precise range depends on how the process is intended to be used.



The minimum defined ranges listed below must to be taken into account:

- Drug material or finished product assay: 80–120% of the test concentration.
- 70 to 130% of the test concentration is the content homogeneity.
- Dissolution testing: within the designated range +/-20%;

I) Robustness

In order to give an idea of the method's variability in typical laboratory conditions, it measures the analytical method's ability to stay unaffected by minor but intentional changes in process. The stability of analytical solutions and extraction time are two examples of common differences.

Examples of common deviations in liquid chromatography include:

- The impact of pH changes in a mobile phase;
- The impact of composition changes in a mobile phase;
- An alternative column
- Temperature;
- Flow rate.

j) Ruggedness

Degree of repeatability of test results achieved by examining the same material under range of regular test settings such as various. i.e., the

approach is unaffected by environmental factors. The method's ruggedness can be directly measured by comparing the reproducibility of test findings to the assay's precision. This includes analysts, instruments, days, reagents, columns, and TLC plates.

Comparison of Analytical Method Validation Guidelines

1. **FDA:** provides comprehensive guidelines for analytical and bio analytical methods. Verification. Accepts alternative ways if scientifically supported. Promotes the life cycle approach.
2. **EMA:** More focused on bio analytical techniques; less thorough for general analytical techniques.
3. **ICH:** The most well recognized and standardized framework. Updated rules Q2 (R2) and Q14 present risk-based and contemporary lifecycle methodologies.
4. **WHO:** Harmonized with ICH, but with a focus on accessibility and public health, this organization offers a globally relevant strategy for nations with limited resources
5. With a heavy emphasis on system appropriateness and performance-based approaches, the **USP** focuses more on compendial procedures.

Aspect	FDA (USA)	EMA (Europe)	ICH (International)	WHO	USP (United States Pharmacopeia)
Key Guideline	FDA Guidance for Industry: Analytical Procedures and Methods Validation	EMA Guideline on Bio analytical Method Validation	ICH Q2(R1) (and soon Q2(R2), Q14)	WHO TRS 1025 Annex 3	USP <1225> and USP <621>



Scope	Drug substances and products (including NDAs/ ANDAs)	Bio analytical methods for human clinical studies	Analytical procedures for drug substances and products (chemical and biological)	Pharmaceuticals for international markets	Compendial and non-compendial analytical methods
Validation Parameters	Accuracy, precision, specificity, detection limit, quantitation limit, linearity, range, robustness	Focuses on bio analytical: accuracy, precision, selectivity, sensitivity, reproducibility, stability	Same as FDA (based on ICH Q2R1): Accuracy, precision, specificity, detection limit, etc.	Similar to ICH and FDA – includes robustness, ruggedness, reproducibility	Same as ICH but includes statistical methods and system suitability tests
Bio analytical Method Focus	Yes – separate FDA guidance for bio analytical methods (e.g., for PK studies)	Primary focus of EMA guideline	ICH M10 covers bio analytical methods	Covered in WHO guidelines for bio logical	Covered to a lesser extent
System Suitability	Emphasized	Required	Required	Required	Strong emphasis, includes detailed criteria
Lifecycle Approach	Supported, especially in newer guidance	Less explicit	Emphasized in ICH Q14 (Analytical Procedure Development)	Some mention, but not as fully developed	Emerging focus via <1220> Analytical Procedure Lifecycle
Documentation Requirements	Detailed method SOPs, validation reports, raw data	Detailed documentation required	Requires comprehensive validation protocol and report	Requires a full validation report	Emphasis on documentation, raw data, and system suitability
Statistical Treatment	Basic statistics, confidence intervals, %RSD	Requires statistical evaluation for bio analytical methods	Encouraged but not overly prescriptive	Encouraged	Strongly integrated, especially for intermediate precision and ruggedness
Bridging/ Transfer Requirements	Requires method transfer validation	Method transfer considered part of validation	Addressed in Q14 as part of method lifecycle	Explicitly mentioned	Covered under USP <1224>

Validation Philosophy

Authority and Validation Philosophy

FDA: Validation Philosophy emphasizes reproducibility and scientific validity. promotes a lifecycle approach, but "point-in-time" validation has historically been the main focus.



EMA: strong regulatory emphasis on bioanalytical techniques; very prescriptive with quality control (QC) standards and statistical validation.

ICH: encourages consistent, risk-based, and empirically supported validation. Lifecycle-based validation is the focus of recent revisions (Q14, Q2(R2)).

WHO: focuses on practicality and viability for environments with limited resources while adhering to ICH. contains instructions for method transfer and revalidation.

USP: emphasizes technique performance and system adaptability while concentrating on broad chapters and compendial norms. Lifecycle principles introduced in <1220>.

Type of Analytical Procedures Covered

Types of Authority Methods

FDA: Assay, identification, dissolution, contaminants, stability-indicating techniques, and bioanalytical techniques (PK/PD investigations).

EMA: The majority of drug development techniques are bioanalytical (e.g., plasma concentrations), with sporadic chemical techniques.

ICH: both biological and chemical compounds. Validation is covered in Q2 (R1/R2), whereas Q14 concentrates on the creation of analytical techniques.

WHO: Herbal, chemical, and biological therapies. strong focus on method durability and transferability across other labs.

USP: both non-compendial and compendial (monograph) techniques. Validation and verification of USP addresses <1225> and <1226>.

3. DETAILED COMPARISON OF VALIDATION CHARACTERISTICS

Parameter	FDA	EMA	ICH Q2 (R1/R2)	WHO	USP <1225> / <1226>
Accuracy	Required	Required (with QC samples)	Required	Required	Required
Precision	Repeatability, Intermediate	Required; also, within/ between runs	Repeatability, Intermediate	Required	Detailed statistical treatment
Specificity	Required	Referred to as "selectivity"	Required	Required	Required
Linearity	Required	Required	Required	Required	Required
LOD/LOQ	Required (LOD optional if not relevant)	Required (lower limit of quantitation)	Required	Required	Required
Range	Based on intended purpose	Based on concentration levels	Based on range of application	Required	Required
Robustness	Encouraged	Required (via system suitability)	Required	Required	Required
Ruggedness	Sometimes referred to as intermediate precision	Not explicitly defined	Not originally defined, but included in updates	Included in revalidation & transfer	Distinctly considered



System Suitability	Strong emphasis	Mandatory for bio analytical runs	Recommended in Q2; mandated in Q14	Required	Core to method performance
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4. Bio analytical Method Validation

Feature	FDA	EMA	ICH M10 (2022)	WHO	USP
Matrix effects	Must be evaluated	Must be evaluated	Required	Required	Briefly touched upon
Stability studies	Long- and short-term required	Extensive stability testing	Required	Required	Not directly covered
Cross-validation	Required for bridging studies	Mandatory when methods/labs differ	Required	Required	Not emphasized
Dilution integrity	Required	Required	Required	Required	Not typically addressed
Partial validation	Allowed under certain conditions	Clearly defined	Defined under M10	Required	Not covered

5. Analytical Procedure Lifecycle (APL)

Phase	FDA	EMA	ICH Q14/Q2(R2)	WHO	USP <1220>
Analytical Development	Encouraged (recent trend)	Not well defined	Core part of Q14	Mentioned	Clearly defined
Method Validation	Required before submission	Required	Required	Required	Required
Method Transfer	Validation needed	Emphasis on reproducibility	Covered in Q14 lifecycle	Defined	Covered in USP <1224>
Ongoing Verification	Lifecycle approach emerging	Not clearly mandated	Required as part of lifecycle	Encouraged	Mandated in <1220>

6. GLOBAL HARMONIZATION AND IMPLICATION

The original Q2 (R1) is modernized by ICH Q2(R2) (Analytical Validation), which incorporates a clear classification of procedures (e.g., identification, test, impurity).

ICH Q14 (Analytical Development): Describes methodical development using testing, performance-based testing, method control plan, and risk assessment.

Harmonization Trends:

ICH Q14 and Q2(R2) are being followed by the FDA, EMA, PMDA (Japan), and WHO. The general chapters (<1220>, <1225>) are being updated by USP to incorporate Life cycle concepts.

As a result, analytical packages are accepted globally and submissions are simplified.

SUMMARY

Authority	Strengths	Gaps
FDA	Strong scientific framework, adaptable, lifecycle-supportive	Lacks formal lifecycle in older guidance
EMA	Detailed bio analytical focus, strict standards	Less guidance for general chemical methods
ICH	Harmonized, modern, globally accepted	Implementation varies by region
WHO	Practical, global health-focused, includes transferability	Less depth in advanced statistical tools
USP	Robust performance criteria, compendial integrity	

CONCLUSION

The efficient development and validation of analytical procedures is critical to pharmaceutical development and regulatory compliance. Analytical methods must be validated in order to ensure their efficacy. Recommendations from various organizations and regulatory bodies vary on a variety of topics. Even if ICH standards have resolved the problems between Europe, the USA, and Japan, organizations like Europe still differ on several matters. There should be an effort to create a similar platform for approval criteria and to establish uniform validation requirements worldwide. When it comes to the quality and safety of medications, method validation serves as a link between science, regulation, and patient safety.

When taken as a whole, these recommendations show a complementary framework in which meeting international quality standards in drug research and manufacturing requires harmonization and flexibility. A deep comprehension of these regulatory viewpoints improves the robustness, reproducibility, and international acceptability of analytical techniques in addition to strengthening compliance. In the end, method validation is a crucial instrument for promoting innovation in the pharmaceutical sector, ensuring patient safety, and assisting with regulatory approval.

REFERENCES

1. ICH Harmonized tripartite guidelines, Validation of Analytical Procedures': Text and Methodology, Q2 (R1), current step 4 version; 27 Oct 1994.
2. Naga G. R., Vignesh K., Analytical Method Validation: An Updated Review, IJAPBC-Vol.1 (1), Jan- Mar, 2012.
3. Ravichandran, Shalini s, and Harish Rajak, Validation of analytical Methods-Strategies and Importance. Vol.2, 3 May 2010.
4. Shah, Kumar's, Upmanyu.N and Mishra.P, Review Article on, "Evaluation of an Analytical Method" IJPCR, Vol.1 issue 1, 2012
5. Eugenie Webster (Khlebnikova) , Statistical Analysis in Analytical Method Validation, IVT Dec 16, 2013
6. Reddy V.P, Rajan T.V.S, Kumar A. N, A review on analytical method validation. Int J Rev Life Sci 1: 141-144.
7. Chinmaya K. S, Muvvala S., Nalini k. S., Validation of Analytical Methods: A Review, International Journal of Chromatography and Separation Techniques, Volume 2018; Issue 01, 19 January, 2018
8. Jaha S. M., Validation in Pharmaceutical Industry: Cleaning Validation - A Brief, Vol. 5, Issue 1, January 2017
9. Chandran S , Singh RP. Comparison of various international guidelines for analytical method validation. Die Pharmazie -An



- International Journal of Pharmaceutical Sciences. 2007 Jan 1;62 (1):4-14.
10. Kleindorfer GB, O'Neill L, Ganeshan R. Validation in simulation: Various positions in the philosophy of science. *Management Science*. 1998 Aug;44(8):1087-99.
 11. Riley CM, Rosanske TW. Development and validation of analytical methods. Elsevier; 1996 May 29.
 12. Feinberg M. Validation of analytical methods based on accuracy profiles. *Journal of Chromatography A*. 2007 Jul 27;1158(1-2):174-83.
 13. Kadian N, Raju KS, Rashid M, Malik MY, Taneja I, Wahajuddin M. Comparative assessment of bioanalytical method validation guidelines for pharmaceutical industry. *Journal of pharmaceutical and biomedical analysis*. 2016 Jul 15; 126:83-97.
 14. Burgess C, McDowall RD. Analytical Instrument Qualification and System Validation Lifecycle. *Method Validation in Pharmaceutical Analysis: A Guide to Best Practice*. 2025 Apr 7:35-50.
 15. Boisrobert CE, Keener L, Lelieveld HL. The global harmonization initiative. In *Ensuring Global Food Safety 2010* Jan 1 (pp. 71-90). Academic Press.
 16. Kost GJ, Hale KN. Global trends in critical values practices and their harmonization. *Clinical chemistry and laboratory medicine*. 2011 Feb 1;49(2):167-76.
 17. Riley CM, Rosanske TW. Development and validation of analytical metho29..T.A. Phazna Devi, Aravind Setti, S. Srikanth, original article on Method development and validation of paracetamol drug by RP-HPLC. 18 Feb 2013.
 18. Beckett A. Hand Stenlake J. B, In: *Practical Pharmaceutical Chemistry*, 3rd edn. Vol. II, CBS Publishers and Distributors, New Delhi; 1986.P. 131.
 19. G. Lavanya, M. Sunil, and M. Eswarudu*, Analytical Method Validation: an updated review. *IJPSR*,2013; Vol.4(4): 1280-1286.
 20. Prakash N., Sunita Arya, Gulbahar, A review article on analytical method validation, *JETIR* Volume9, Issue 2, February 2022.
 21. Basant L.1, Devesh K. 2*, Manish J., A review on analytical method validation and its regulatory perspectives, *Journal of Drug Delivery and Therapeutics*. 9(2):501-506, 2019.
 22. Ramole R., Mohini B.*, Ashish J., A Review: Analytical Method Development and Validation, *SysRevPharm*12(8): 450-454, 2021.
 23. Analytical Procedures and Method Validation (2000), Fed Reg 52: 776– 777.
 24. AOAC Peer Verified Methods Program (1993), Manual on policies and procedures, Arlington, VA.
 25. Bolton S (1997) *Pharmaceutical Statistics: Practical and Clinical Application*, 3rd edition, Marcel Dekker, New York, pp. 153, 216–269.
 26. Brown R, Caphart M, Faustino R, Frankewich E, Gibbs R, Lentzinger E, Lunn G, Rajagopalan R, Chiu Y, Sheinen E (2001) *Analytical Procedures and Method Validation: Highlights of FDA's draft guidance*, LCGC (www.chromatographyonline.com), 19: 74.
 27. General Chapter No. 1225, Validation of Compendial Methods, United States Pharmacopoeia XXIII, National Formulary, XVIII (1995) Rockville, MD, The United States Pharmacopoeial Convention, Inc, 1710. Green JM (1996) A practical guide to analytical method validation. *Anal Chem* 305A309A
 28. International Conference on Harmonization (ICH) of Technical Requirements for the Registration of Pharmaceuticals for Human



- use, Validation of Analytical Procedures, ICH Q2A (1996), Geneva.
29. International Conference on Harmonization (ICH) of Technical Requirements for the Registration of Pharmaceuticals for Human use, Validation of Analytical Procedures: Methodology, ICH Q2B (1996) Geneva.
 30. Karnes T, Shiu G, Shah VP (1991) Bioanalytical method validation. *Pharm Res* 8: 421–426
 31. Miller JC, Miller JN (1988) Basic statistics methods for analytical chemistry part 1- Statistics of repeated measures *Analyst* 113: 1351–1356.
 32. Miller JN (1991) Basic statistical methods for analytical chemistry, part 2-A Calibration and regression methods *Analyst* 116: 3–14
 33. Shah VP, Midha KK, Dighe S, Mc Gilveray IJ, Skelly JP, Yacobi A, Layloff T, Viswanathan CT, Cook CE, McDowall RD, Pittman KA, Spector S (1991) Analytical methods validation: Bioavailability, bioequivalence and pharmacokinetic studies. Conference report. *Eur J Drug Metab Pharmacokinet* 16: 249–255
 34. Thompson M, Ellison SLR, Wood R (2002) Guidelines for Single Laboratory Validation of Methods of Analysis. *Pure Appl Chem* 74: 835–855.
 35. US FDA Guidance of Industry-Bioanalytical Method Validation (2001), # 5600, Fishers Lane, Rockville, MD 20857.
 36. US FDA Technical Review Guide: Validation of Chromatographic Methods (1993) Center for Drug Evaluation and Research (CDER), Rockville, MD
 37. United States Pharmacopoeia (2003) United States Pharmacopeial Convention, 26th Edition, Rockville, MD.
 38. WHO Expert Committee report on specifications for pharmaceutical preparations (1992) 32nd Report. 32: 117–121.
 39. Willard HH, Meritt LL, Dean JA, Settle FA (1995) Instrumental Method of Analysis, CBS Publishers, New Delhi, 7th Edn., 2–17.
 40. Pasteelnic LA (1993) Analytical methods validation. In: Nash RA, Berry IR (eds.) *Pharmaceutical process validation*, 2nd ed., Marcel Dekker Inc.,

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