www.ijarp.com ISSN 2456-9992 Page: 01-07
International Journal Advanced Research Publications
Research Article
Volume: 02
Issue: 01

VALIDATED QUANTITATIVE ESTIMATION OF DEXAMETHASONE AND INDOMETHACIN IN COMBINE DOSAGE FORM BY UV SPECTROSCOPY

Akash Kushwah*, Dr. Avinash Krishnarao Kondalkar, Narottam Singh, Muraree Lal, Shankar

Department of Pharmacy, Sun institute of Pharmaceutical Education and Research (SIPER), Lahar Bhind,(M.P), India 477445.

Article Received: 15 December 2025, Article Revised: 03 January 2026, Published on: 23 January 2026

***Corresponding Author: Akash Kushwah**
Department of Pharmacy, Sun institute of Pharmaceutical Education and Research (SIPER), Lahar Bhind,(M.P), India 477445.
DOI: https://doi-doi.org/101555/ijarp.5235

ABSTRACT:

The present study was undertaken to identify, characterize, develop, and validate a UV spectrophotometric method for the simultaneous estimation of Dexamethasone (DMS) and Indomethacin (IMC) using the simultaneous equation method. The wavelength selection study revealed λmax values of 242.0 nm for Dexamethasone and 266.0 nm for Indomethacin, which were selected for quantitative analysis. Linearity studies demonstrated a direct proportional relationship between absorbance and concentration over the range of 5–25 µg/ml for DMS and 10–50 µg/ml for IMC. The calibration curves exhibited excellent linearity with correlation coefficients (r^2) of 0.999 for both drugs. The slopes were found to be 0.049 for DMS and 0.009 for IMC, indicating good sensitivity of the method. Overlay spectra confirmed the presence of suitable absorbance characteristics for simultaneous estimation using the simultaneous equation method. Mixed standard studies showed accurate quantification, with percentage concentration found ranging from 96.00–98.00% for DMS and 99.84–99.92% for IMC, confirming the suitability of the developed method for combined analysis. Accuracy studies conducted at 80%, 100%, and 120% recovery levels demonstrated satisfactory recovery values. For Dexamethasone, mean recoveries were 95.20%, 96.62%, and 95.08%, with %RSD values below 2%. For Indomethacin, mean recoveries were 99.05%, 99.06%, and 99.83%, with %RSD values well below 1%, indicating excellent accuracy.

KEYWORDS: Validation, Calibration Curve, Simultaneous Equation Methods, Estimation, Dexamethasone, linearity.

INTRODUCTION:

Validation

The concept of validation was formally introduced in the USA, in early 1979, its scope, since then has got expanded to include a wide range of function related to the pharmaceutical development & manufacturing. Analytical data are used to screen potential drug candidates aid in the development of drug synthesis, formulation studies monitor the stability of bulk pharmaceuticals and formulated products, and test final products for release. The quality of analytical data is a key factor in the success of a drug development program.

So the process of method development and validation has direct impact on the quality of pharmaceutical products. Method validation is an integral part of the method development. It is the process of demonstrating that analytical procedures are suitable for their intended use and that they should support the identity, quality, purity & potency of the drug substances and drug products. Simply method development is the process of proving that an analytical method is acceptable for its intended purpose. Method validation, however, is generally a one-time process performed after the method has been developed to demonstrate that the method is scientifically sound and that is serves the intended analytical purpose. For pharmaceutical method, guidelines from United States pharmacopoeia (USP) ICH, and food & drug administration (FDA) provide a frame work for performing such validation (Eurachem, 1998; Huber, 1998).

General Concept of Validation

The general concept of validation is that, the valid method:

- Provides useful analytical data in a specific situation
- Is suitable for its intended use
- Meets predetermined requirement (specifications) of the analytical problem.
- Has established level performance like accuracy, consistency, reliability etc.

Literature Review:

Sharma *et al.*, (2024) aimed to develop and validate an accessible, accurate, fast, and commercial UV spectrophotometric approach for assessing Pioglitazone in bulk and

polymeric nanoparticles. In the estimation method that involves a pH 7.4 phosphate buffer, the maximum absorbance of Pioglitazone is reported to be 269 nm. With a correlation coefficient of 0.999, the drug exhibits linearity in the concentration series of 5–60 µg mL^{-1}. The proposed technique is validated as per ICH guidelines for linearity, accuracy, precision, and ruggedness.

Liu *et al.*, (2023) analysed quantitative amorphous form in indomethacin binary polymorphic mixtures using infrared spectroscopy analytical techniques combined with chemometrics methods. In this study, rapid and nondestructive methods for quantitative analysis of amorphous indomethacin (A-INDO) content in A-INDO and γ-indomethacin (γ-INDO) binary mixtures were established based on Attenuated Total Reflection Fourier Transform Infrared spectroscopy (ATR-FTIR) and Near-Infrared spectroscopy (NIR) combined with chemometrics methods. Partial least squares regression (PLSR) was used to establish quantitative analysis models of A-INDO content ranging from 0.000 % to 10% w/w %. A variety of spectral pretreatment methods were used to pretreat the spectral, reducing the influence of inconsistent particle size and uneven mixing, and highlighting the sample component information.

Shaikh *et al.*, (2023) developed and validated simple, rapid, accurate, economical and precise UV/VIS method Choices of a common solvent were essential so various solvent ranges including methanol, ethanol, acetonitrile, and 0.1 N HCl, and various concentrations ranges of various buffers were analyzed. Hence 0.1 N HCl was selected as a solvent for the proposed method. Caffeine and Pioglitazone HCl showed maximum absorbance at 273 and 220 nm respectively. Both drugs obey Beer Lambert's law in the concentration range of 3- 18 µg/mL for Caffeine and Pioglitazone HCl respectively. The LOD and LOQ were found to be 0.5476 µg/mL and 1.6594 µg/mL for Caffeine respectively. For Pioglitazone HCl the LOD and LOQ values were found to be 0.6111 µg/mL and 1.8519 µg/mL respectively. The method was quantitatively evaluated in terms of linearity, precision, precision, LOD, LOQ and recovery.

Tang *et al.*, (2023) developed and validated an LC-MS/MS method for the simultaneous determination of CLP, clopidogrel carboxylic acid (CLPCA), 2-oxo-clopidogrel (2-O-CLP), SV, and simvastatin hydroxy acid (SVA) in beagle plasma. Chromatographic separation was achieved on an Infinity Lab Poroshell 120 SB-C_8 column (2.1 × 100 mm, 2.7 µm) using methanol and 0.1% formic acid in water as the mobile phase at a flow rate of 0.3 mL/min in

gradient mode. The lower limits of quantification are 0.1, 0.8, 0.05, 0.05, and 0.05 ng/mL for CLP, CLPCA, 2-O-CLP, SV, and SVA, respectively. The selectivity, linearity, accuracy, precision, extraction recovery, matrix effect, and stability were validated within acceptable criteria. This method was successfully applied to the pharmacokinetic drug interaction study between CLP and SV, and its revealed that combined administration affected the metabolic rate of CLP, SV, and their metabolites.

Youssrey *et al.*, (2023) described sensitive and selective green chromatographic methods with UV detection for the simultaneous determination of aspirin, rosuvastatin and clopidogrel. The first proposed method is an RP-HPLC one, which was described and successfully validated for the simultaneous separation and determination of the three components on Prontosil Hyperchom C_{18} (250 × 4.6 mm, 5 µm) column using an isocratic elution. The drugs were applied on silica gel plates.

Experimental Work and Result:

Solubility

Solubility of the drug was determined by taking some quantity of drug (about 1-2 mg) in the test tube separately and added the 5 ml of the solvent (Water, methanol, 0.1 N HCl, acetonitrile, methanol (80%) and acetate buffer). Shake vigorously and kept for some time. Note the solubility of drug in various solvents (at room temperature) (Indian pharmacopeia, 2007).

Table 1: Solubility of drugs in different solvents.

Solvent	**Dexamethasone**	**Indomethacin**
Water	Practically insoluble	Practically insoluble
0.1 N HCl	Slightly soluble	Practically insoluble
0.1 N NaOH	Slightly soluble	Soluble
Methanol	Freely soluble	Freely soluble
Methanol (80%)	Freely soluble	Freely soluble
Acetonitrile	Soluble	Soluble
Acetate Buffer	Slightly soluble	Slightly soluble

RESULTS OF METHOD DEVELOPMENT

Standard stock solutions were prepared by dissolving separately 100 mg of each drug in 80ml methanol in 100 ml volumetric flask. The flask was sonicated for about 10 min to solubilize the drug and the volume was made up to the mark 100ml with distilled water to get a concentration of 1000 µg/ml (Stock-A) for both drugs.

Table 2: Linearity of DMS At λ_{max} = 242.0 nm.

Standard Conc. (□g/ml)	Rep-1	Rep-2	Rep-3	Rep-4	Rep-5	Mean
5	0.254	0.254	0.253	0.252	0.253	0.2532
10	0.495	0.495	0.494	0.493	0.493	0.494
15	0.748	0.747	0.746	0.745	0.744	0.746
20	1.005	1.004	1.003	1.002	1.003	1.0034
25	1.232	1.231	1.232	1.231	1.312	1.2476
Correlation Coefficient (r^2)						0.999
Slope (m)						0.049
Intercept (c)						0.000

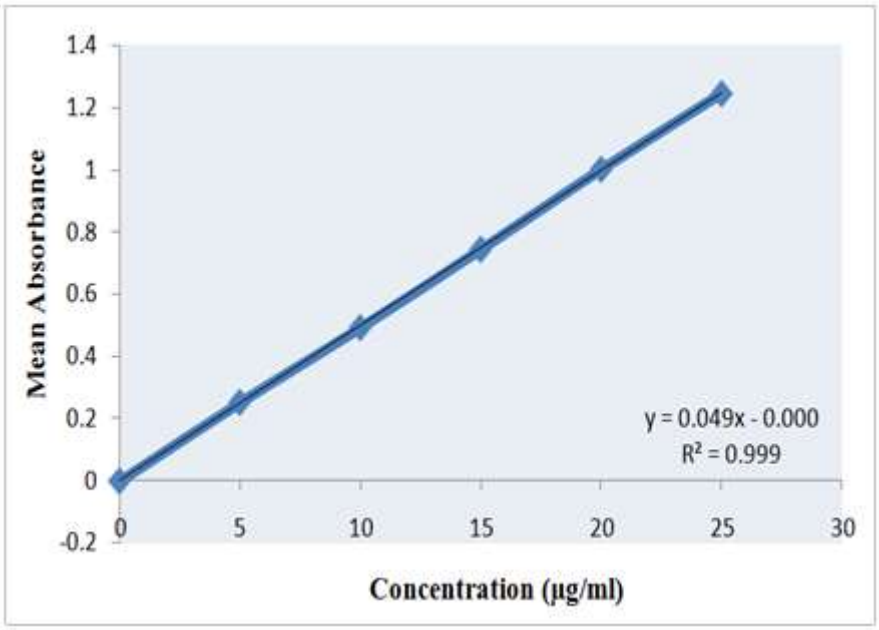

Figure 1: Calibration Curve of DMS.

CONCLUSION:

The present study was undertaken to identify, characterize, develop, and validate a UV spectrophotometric method for the simultaneous estimation of Dexamethasone (DMS) and Indomethacin (IMC) using the simultaneous equation method. Comprehensive preformulation, method development, and validation studies were carried out, and the results obtained are summarized below.

The physical evaluation of the drugs revealed that Dexamethasone appeared as a white, odorless, bitter powder, while Indomethacin was observed as a white to off-white, odorless, bitter powder, confirming their characteristic sensory properties. Solubility studies demonstrated that both drugs were practically insoluble in water, freely soluble in methanol and 80% methanol, and soluble in acetonitrile. Dexamethasone showed slight solubility in

0.1 N HCl and 0.1 N NaOH, whereas Indomethacin was soluble in 0.1 N NaOH but practically insoluble in 0.1 N HCl. Both drugs exhibited slight solubility in acetate buffer, supporting the selection of suitable solvents for spectrophotometric analysis.

Melting point determination showed values of 262–264°C for Dexamethasone and 158–160°C for Indomethacin, which are in close agreement with reported literature values, indicating purity of the drugs. FTIR spectroscopy further confirmed the identity of both drugs, as the characteristic functional group peaks observed in the spectra matched the standard reference spectra, indicating no chemical alteration.

REFERENCE:

1. Lavanya G, Sunil M, Eswarudu MM, Eswaraiah MC, Harisudha K, Spandana BN. Analytical method validation: An updated review. Int J Pharm Sci Res. 2013; 4(4): 1280.
2. Rocha FS, Gomes AJ, Lunardi CN, Kaliaguine S, Patience GS. Experimental methods in chemical engineering: Ultraviolet visible spectroscopy-UV-Vis. Can J Chem Eng 2018;96:2512-7.
3. Van Den Broeke J, Langergraber G, Weingartner A. On-line and in-situ UV/vis spectroscopy for multi-parameter measurements: A brief review. Spectrosc Eur 2006;18:15-8
4. Uríčková V, Sádecká J. Determination of geographical origin of alcoholic beverages using ultraviolet, visible and infrared spectroscopy: A review. Spectrochim Acta A Mol Biomol Spectrosc 2015;148:131-7.
5. Venkatachalam S. Ultraviolet and visible spectroscopy studies of nanofillers and their polymer nanocomposites. In: Spectroscopy of Polymer Nanocomposites. Norwich, NY: William Andrew; 2016. p. 130-57.
6. Ríos-Reina R, Azcarate SM. How chemometrics revives the UV-Vis spectroscopy applications as an analytical sensor for spectralprint (nontargeted) analysis. Chemosensors 2022;11:8.
7. Rojas FS, Ojeda CB. Recent development in derivative ultraviolet/ visible absorption spectrophotometry: 2004-2008: A review. Anal Chim Acta 2009;635:22-44
8. Ojeda CB, Rojas FS. Recent developments in derivative ultraviolet/ visible absorption spectrophotometry. Anal Chim Acta 2004;518:1-24.
9. Plane JM, Saiz-Lopez A. UV-Visible differential optical absorption spectroscopy (DOAS). In: Analytical Techniques for Atmospheric Measurement. United States:

Wiley; 2006. p. 147-88.

10. Imam SS. The future of non-invasive ways to treat cancer. Int J Pharm Sci Res 2021;12:4684-96.
11. G. R. Chatwal, S. K. Anand Instrumental methods of chemical analysis, Himalaya Publishing House.1979.
12. Imam SS, Sharma S, Kumari D, Khan S, Pathak P, Katiyar D. An expedient approach to treat asthma through non-steroidal, natural transferosomes aerosol system. Innovare J Med Sci 2022;10:7-11.
13. Imam SS, Imam ST, Agarwal S, Kumar R, Ammar MY, Athar MW, et al. Lung cancer therapy using naturally occurring products and nanotechnology. Innovare J Med Sci 2022;10:1-5.
14. Imam ST, Imam SS. The cream which relieves the pain of menstrual cramps without interfering with the hormones or period cycle. Res J Pharm Technol 2023;16:1239-6.
15. Imam SS. Nanoparticles: The future of drug delivery. Int J Curr Pharm Sci 2023;15:8-15.
16. Imam SS, Mehdi S, Mansuri A. A comprehensive review on the pharmacological potential of red ginseng oil, tea tree oil and hemp seed oil. Eur J Biomed 2024;11:167-77.
17. Sharma DK, Pattnaik G, Behera A. Analytical Method Development and Validation of a UV-Spectrophotometric Technique for Pioglitazone HCL Estimation in Bulk and Polymeric Nanoparticles.In Macromolecular Symposia 2024 Apr (Vol. 413, No. 2, p. 2300167).
18. Mingdi Liu, Jichao Liu, Qiuhong Wang, Ping Song, Haichao Li, Yaru Qin, Songgu Wu, Junbo Gong, Quantitative analysis of amorphous form in indomethacin binary polymorphic mixtures using infrared spectroscopy analytical techniques combined with chemometrics methods, Arabian Journal of Chemistry, Volume 16, Issue 8, 2023,