

UV-Spectrophotometric Estimation of Ibuprofen Lysine: Method Development and Validation as per ICH Guidelines

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ABSTRACT:

Ibuprofen lysine is the lysine salt of ibuprofen, a non-steroidal anti-inflammatory drug used for its analgesic, anti-inflammatory and antipyretic action. It is prescribed for back pain, osteoarthritis, rheumatoid arthritis, dysmenorrhea, gout and patent ductus arteriosus. Since the molecule contains an aromatic ring and functional groups that absorb in the ultraviolet region, this work was carried out to develop and validate a UV-spectrophotometric method for estimating Ibuprofen lysine in bulk drug and in tablet formulation, following ICH Q2(R1) guidelines.

Water was chosen as the solvent, as the drug dissolved readily in it and gave a clear absorption spectrum. A stock solution of 100 µg/mL was prepared, from which working standards of 2–10 µg/mL were made. Scanning between 200 and 400 nm gave a maximum absorbance (λ_{max}) at 222 nm, which was used for further analysis. The calibration curve was linear over the 2–10 µg/mL range. Precision was checked on the same day and across six different days, giving %RSD values of 0.271 and 0.369, both comfortably within the ICH limit of 2%. Recovery studies at 80%, 100% and 120% levels gave a mean recovery of 100.14%, confirming that the method is accurate. The limit of detection and limit of quantification were calculated as 1.926 µg/mL and 5.836 µg/mL, showing the method is sensitive enough for low concentrations. When applied to a marketed brand (RAPID), the assay gave 99.95% of the labelled amount with a %RSD of 0.66.

Keywords: Ibuprofen lysine; UV-Visible spectrophotometry; Method validation; Linearity; ICH Q2(R1) guidelines

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INTRODUCTION:

Ibuprofen lysine is the lysine salt form of ibuprofen used in pharmaceutical formulations. The IUPAC name of ibuprofen lysine is 2,6-diaminohexanoic acid 2-[4-(2-methyl propyl) phenyl] propanoic acid. It has a molecular formula of C₁₉H₃₂N₂O₄ and a molecular weight reported around 334.45–352.48 g/mol. The drug appears as a white to off-white crystalline powder with a melting point above 155°C and is soluble in water. Structurally, the compound contains an aromatic phenyl ring and functional groups capable of electronic transitions in the ultraviolet region, where π -electrons and non-bonding electrons absorb UV radiation forming the basis of spectroscopic analysis [1].

Pharmacologically, ibuprofen lysine belongs to the class of non-steroidal anti-inflammatory drugs (NSAIDs) and exhibits analgesic, anti-inflammatory and antipyretic effects by inhibiting prostaglandin synthesis through cyclo-oxygenase inhibition. The drug is used for conditions such as back pain, headache, osteoarthritis, rheumatoid arthritis, gout and dysmenorrhea, and is also indicated in patent

ductus arteriosus. Several analytical techniques including chromatography and spectroscopic methods are used for drug analysis. Among them, UV-Visible spectroscopy is widely applied due to its simplicity and rapid analysis; therefore, the present study aims to develop and validate a UV spectrophotometric method for the estimation of ibuprofen lysine in pharmaceutical dosage forms [2].

MATERIALS AND METHOD

Selection of Solvent:

The water was selected as solvent for IBUPROFEN LYSINE. Because the spectrum was clearly visible by using water as a solvent. It is insoluble in Ethanol and soluble in Chloroform, NAOH, Water. So, we have selected the solvent (Water) to better solubility by even taking 10mg of Ibuprofen Lysine [3].

Preparation of Stock Solution:

Accurately weighted quantity of Ibuprofen lysine (10mg) which transferred into 100ml of volumetric flask. Then dissolved with water and then diluted up to the mark with same solvent (stock solution

100µg/ml) A training set of standard mixtures containing 2-10 µg/ml Ibuprofen lysine was made from stock solution. A calibration curve was prepared by plotting the concentration vs absorbance at their respective max.

Result And Discussion:

Determination of λ max:

Dilute the stock solution with water and measure the absorbance at the UV-region from 200- 400 nm. The max was obtained as 222 nm [4,5].

Method validation:

The validation parameter was performed in compliance with the ICH Q2B recommended conditions. The parameter is carried out by linearity, accuracy, precision and LOD& LOQ [6-10].

Linearity:

Aliquots were prepared from the stock solution 2 to 10µg/ml. The sample was scanned in UV-Visible Spectrophotometer using water as a blank [6].

Table 1. Linearity of Ibuprofen lysine.

concentration	absorbance
2	0.106
4	0.209
6	0.308
8	0.412
10	0.511
Average	0.309

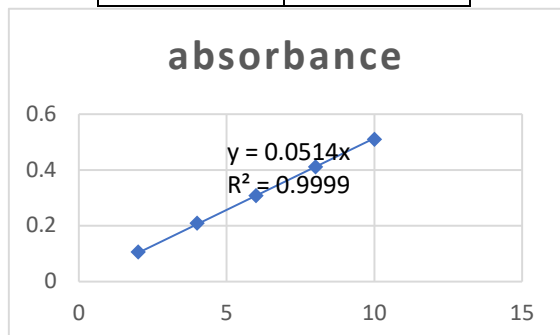


Figure 1. Calibration curve Ibuprofen lysine in water.

Precision:

To assess the intra-day and inter-day precision. 6µg/ml solution was analysed six times at a short interval of time in one day for intra-day precision and perform the same type of 6µg/ml solution was analysed at six separate days for inter-day precision [11,12,13].

Table 2. Precision table.

Concentration (µg/ml)	Absorbance (nm)
6	0.308
6	0.310
6	0.307
6	0.309
6	0.308
<i>Average</i>	0.308
<i>%RSD</i>	0.369

Intra-Day

Concentration (µg/ml)	Absorbance (nm)
6	0.308
6	0.309
6	0.307
6	0.307
6	0.308
<i>Average</i>	0.307
<i>%RSD</i>	0.271

Inter-Day

Accuracy:

The accuracy of the method was determined and suggested technique's recovery study was resolved by the standard addition method at. The stock solution standard and the sample were prepared. Pipette out [14,15].

Table 3. Result of accuracy.

Nam e of drug	Lev el of reco very	Concen tration (µg/ml)	Absor bance	Am ount reco very (µg/ ml)	% Reco very
Ibup rofen lysin e	80%	4.8	0.245	4.77	99.3 7
	100 %	6.0	0.310	6.03	100. 50
	120 %	7.2	0.372	7.24	100. 55
Mean Recovery = 100.14%					

The sensitivity of Ibuprofen lysine by proposed method was Ibuprofen lysine by method was

estimated by limit of detection (LOD) and limit of quantification (LOQ). The LOD and LOQ were calculated using following formula [16,17].

Sensitivity

The sensitivity of luliconazole by proposed method was estimated by limit of detection (LOD) and limit of quantification (LOQ). The LOD and LOQ were calculated using following formula

$$\text{LOD}=3.3 \text{ S.D/Slope}$$

$$\text{LOQ}=10 \text{ S.D/Slope}$$

Where 'S. D' is standard deviation of the response and 'S' is the slope of the corresponding calibration curve of analyte [6,7].

The linearity equation was found to be $y= 0.0514$. The LOD and LOQ of Ibuprofen lysine was found to be 1.926 and 5.836 respectively.

Assay:

The drug content present in the formulation was analysed and determined by statistical method [18,19]

The absorbance of the sample was recorded as 212 nm and showed in the table. The percentage purity of Ibuprofen lysine was found to be and it was presented

The %RSD value was found to be

Brand name	Label claim	Amount estimated	% Assay
RAPID (Ibuprofen lysine)	342	340.8	99.65
	342	341.6	99.88
	342	341.1	100.32
	Average	341.83	99.95
S.D		1.15	
%RSD		0.66	

CONCLUSION:

A simple, accurate, and precise UV-spectrophotometric method was developed and validated for the estimation of Ibuprofen lysine in bulk and pharmaceutical dosage form, using water as solvent, with detection at 222 nm. The method showed good linearity over 2–10 µg/mL, high precision (intra-day %RSD 0.271, inter-day %RSD 0.369 — both well within the ICH acceptance limit of $\leq 2\%$), and good accuracy (mean recovery 100.14%). LOD and LOQ were found to be 1.926

µg/mL and 5.836 µg/mL respectively, indicating adequate sensitivity. Assay of the marketed formulation (RAPID) gave 99.95% of label claim with %RSD 0.66. Given these validation parameters comply with ICH Q2(R1) guidelines, the method is suitable for routine quality-control analysis of Ibuprofen lysine in pharmaceutical formulations.

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