

# Development and Validation of UV-Spectroscopic Method for Estimation of Niacin in Bulk and Pharmaceutical Dosage Form

Indranil Chanda<sup>1</sup>, Ripunjoy Bordoloi<sup>2\*</sup>, Debarupa D. Chakraborty<sup>3</sup>, Prithviraj Chakraborty<sup>2</sup>, Smriti Rekha Chanda Das<sup>1</sup>

<sup>1</sup>Girijananda Chowdhury Institute of Pharmaceutical Science, Hathkhowapara, Azara, Guwahati-781017, Assam, India.

<sup>2</sup>Bengal College of Pharmaceutical Sciences and Research, B.R.B. Sarani, Bidhannagar, Durgapur-713212, West Bengal, India.

## ARTICLE INFO

### Article history:

Received on: 01/12/2016

Accepted on: 07/02/2017

Available online: 30/09/2017

### Key words:

Niacin, UV Spectrophotometric, Method development, validation, ICH guideline.

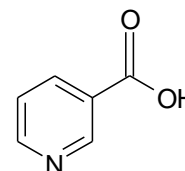
## ABSTRACT

A novel, simple, specific and economic UV Spectrophotometric method has been developed using ethanol as solvent to estimate niacin content in bulk and pharmaceutical dosage formulation. The  $\lambda_{\max}$  of niacin was found to be 262 nm. Linearity in the concentration range of 01-19 $\mu$ g/ml was found to be exhibiting good correlation coefficient ( $R^2=0.9991$ ). The developed method was validated statistically to demonstrate linearity, accuracy, precision, LOD and LOQ. The validation parameters were selected as per the ICH [Q2 (R1)] guideline. The results of the study proved the applicability of the present method in routine analysis of niacin in bulk as well as in the formulation.

## INTRODUCTION

Niacin, known as Vitamin B3 or nicotinic acid, is chemically pyridine-3-carboxylic acid (Fig. 1) official in IP (Indian Pharmacopoeia, 2007); which is a colorless, water-soluble solid. It has the ability to reduce low density lipoprotein cholesterol (LDL), very low-density lipoprotein cholesterol (VLDLC), and triglycerides (TG), and also effectively increase high density lipoprotein cholesterol (HDL) (Villines *et al*, 2012). Literature survey had revealed various analytical methods (RP-HPLC, HPTLC, UV-Spectroscopy, LC-MS/MS (Vasanthi *et al*, 2015; Narayankar *et al*, 2015; Pravish and Padmakar, 2010; Ranganath and Raja, 2014; Bratati *et al*, 2014; Dewani *et al*, 2015) for determination of Niacin in pharmaceutical formulations in combination with other drugs. In the present study, efforts were made in developing a simple, specific and economic

UV spectrophotometric method using ethanol as solvent to determine Niacin content in bulk and pharmaceutical dosage formulation and validate it as per the ICH guidelines (ICH, 2005).



NIACIN

**Fig 1:** Chemical Structure of Niacin hydro-2H-pyran-2-yl] ethyl]-3, 7-dimethyl-1, 2, 3, 7, 8, 8a-hexahydronaphthalen-1-yl 2, 2-dimethylbutanoate

## MATERIALS AND METHODS

### Instruments

UV-Visible double beam spectrophotometer (UV-1800, Shimadzu, Japan) with 1cm matched quartz cells, Micropipette of variable volumes (Microlit, India) and Digital balance (Denver Instrument, Germany) were used.

\* Corresponding Author

Ripunjoy Bordoloi, Research Scholar, Girijananda Chowdhury Institute of Pharmaceutical Science, Hathkhowapara, Azara, Guwahati-781017, Assam, India. Email: [ripun.bordoloi@gmail.com](mailto:ripun.bordoloi@gmail.com)

## Materials

Niacin API was procured from B. S. Trading, Howrah, West Bengal, having 99.98% w/w assay value and was therefore used without further purification. Analytical grade Methanol, Potassium dihydrogen phosphate, Hydrochloric acid, Sodium hydroxides were purchased from CDH (P) Ltd. New Delhi. Niacin tablets were purchased from local pharmacy shop of Guwahati, Assam.

## Determination of wavelength of maximum absorption ( $\lambda_{\max}$ )

A standard stock solution of Niacin (100  $\mu\text{g/ml}$ ) was prepared using ethanol as solvent and 0.2 ml was diluted to 10 ml with the same solvent to obtain 2  $\mu\text{g/ml}$  reference solutions. The reference solution was scanned in the wavelength region of 200-400 nm.

## Linearity and range

Nineteen solutions (1-19  $\mu\text{g/ml}$ ) of different concentration were prepared from the standard stock solution of Niacin for linearity study. The absorbance of these solutions was observed against ethanol as blank at 262 nm and the obtained data was used for the linearity calibration curve.

## LOD and LOQ

Limit of detection (LOD) and Limit of quantitation (LOQ) for the assay was calculated using the following formula:

$\text{LOD} = 3.3 \times (\text{standard deviation of } y\text{-intercept of the regression line} / \text{slope of the calibration curve})$

$\text{LOQ} = 10 \times (\text{standard deviation of } y\text{-intercept of the regression line} / \text{slope of the calibration curve})$

## Assay of content of Niacin in tablet dosage form

The newly developed method was applied in order to analyze the Niacin in marketed tablet formulation. Niacin tablet powder equivalent to 100 mg of Niacin was dissolved into 100 ml ethanol by shaking to get the final concentration of 1mg/ml. The solution was then filtered through Whatman filter paper #41. This filtrate was diluted suitably with ethanol to get the solution concentration of 10  $\mu\text{g/ml}$ . The absorbance of this solution was measured and amount of Niacin was calculated from the calibration curve.

## Accuracy

Accuracy of the developed method was carried out by performing recovery study using standard addition method, in which standard drug was added at three different concentration (80%, 100% and subsequently by 120%) to the pre-analyzed formulation (10  $\mu\text{g/ml}$ ).

## Precision

Precision study of the method was performed by intra-day and inter-day variation study. The intraday precision and inter-day precision was ascertained by determining absorbance of 3 replicates of a fixed concentration of the drug (10  $\mu\text{g/ml}$ ) at three

different time period of the same day and on three different days. The result of the precision studies was expressed in terms of % RSD (percentage of Relative Standard Deviation).

## Solution Stability Study

To test the short term stability of Niacin solution, three different concentrations (2, 4 and 6  $\mu\text{g/ml}$ ) was prepared and analyzed at 10 hours.

## Ruggedness and Robustness

Ruggedness of the method was determined on carrying out the method by two different analysts and Robustness of the method was determined by measuring the absorbance of 10  $\mu\text{g/ml}$  solution of Niacin at 260 nm, 262 nm and 264 nm.

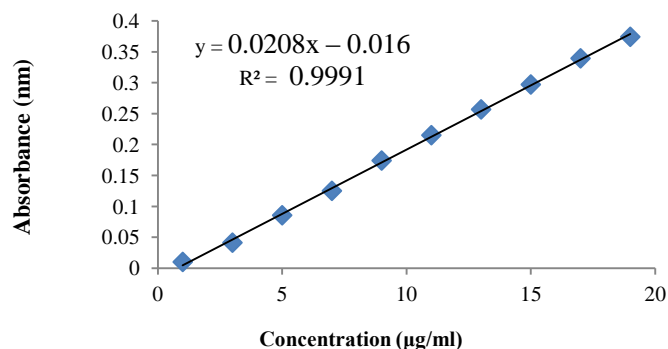
## RESULTS AND DISCUSSION

### Method Development

The  $\lambda_{\max}$  of Niacin in ethanol was found to be 262 nm. Niacin was found to be linear within the concentration range 01-19  $\mu\text{g/ml}$  and exhibited correlation coefficient of 0.9991 (Fig. 2). The result of regression analysis is given in Table 1.

**Table 1:** Result of regression analysis of Niacin.

Niacin	Beer's Range	Regression Equation	Regression coefficient ( $R^2$ )
Absorbance maxima method	01-19 $\mu\text{g/ml}$	$y = 0.0208x - 0.016$	0.9991



**Fig 2:** Linearity curve of Niacin at 262 nm.

## Validation

### LOD and LOQ

The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.64  $\mu\text{g/ml}$  and 1.94  $\mu\text{g/ml}$  respectively (Table 2) which indicates that the proposed UV method is sensitive.

**Table 2:** Result of LOD and LOQ.

Drug	LOD ( $\mu\text{g/ml}$ )	LOQ ( $\mu\text{g/ml}$ )
Niacin	0.64	1.94

### Assay of content of niacin in tablet dosage form

The assay results of the commercial formulations are shown in (Table 3). The developed method was in good agreement with the label claim.

**Table 3:** Result of table assay (n=3).

Drug	Label Claim	Amount of Drug estimated (mg/tab)	Assay
Niacin	500 mg	495.97±0.4645	99.326±0.1929

### Accuracy

Results of recovery study were within the range of 99.15-99.66 % indicating that the developed method is an accurate method for determination of niacin. The results are summarized in Table 4.

**Table 4:** Statistical analysis for ACCURACY of the proposed method.

Sample	Concentration (µg/ml)		% Recovery	Statistical analysis
	Pure	Formulation		
S1 80%	8	10	99.65	Mean : 99.66 SD : 0.0981 %RSD 0.0984
S1 80%	8	10	99.54	
S1 80%	8	10	99.78	
S2 100%	10	10	99.54	Mean : 99.15 SD : 0.7354 %RSD 0.7417
S2 100%	10	10	98.12	
S2 100%	10	10	99.79	
S3 120%	12	10	99.43	Mean : 99.28 SD : 0.5167 %RSD 0.5204
S3 120%	12	10	98.59	
S3 120%	12	10	99.83	

**Table 5:** Statistical analysis for INTRADAY ASSAY of the proposed method.

Sl. No	Concentration (µg/ml)	Absorbance			Average % RSD
		Morning	Afternoon	Evening	
1	10	0.190	0.190	0.191	0.3677
2	10	0.190	0.191	0.192	
3	10	0.191	0.189	0.190	
% RSD		0.2632	0.4211	0.4188	

**Table 6:** Statistical analysis for INTERDAY ASSAY of the proposed method

Sl. No	Concentration (µg/ml)	Absorbance			Average % RSD
		Day1	Day2	Day3	
1	10	0.191	0.189	0.191	0.3672
2	10	0.190	0.190	0.191	
3	10	0.192	0.191	0.190	
% RSD		0.4188	0.4211	0.2618	

### Precision

The developed method was found to be precised as the average % RSD values for intraday and inter-day precision study was found to be 0.3677 % and 0.3672 % respectively (Table 5 and Table 6).

### Solution Stability Study

Result of short term stability study (Table 7) indicates towards the sample stability in solution for 10 hours which is within the acceptable range.

**Table 7:** Short term stability study.

Concentration (µg/ml)	Concentration found (at 10 hours)	
	Mean ± SD, (µg/ml)	
2	1.973± 0.0068	
4	3.870± 0.0690	
6	5.944± 0.0324	

### Ruggedness and Robustness

It was observed (Table 8 and Table 9) that there were no significant changes in the results, which demonstrated that the developed method is rugged and robust.

**Table 8:** Statistical analysis for RUGGEDNESS of the proposed method.

Sl.no	Analyst 1			Analyst 2		
	Concentration (µg/ml)	Absorbance	Statistical Analysis	Concentration (µg/ml)	Absorbance	Statistical Analysis
1	10	0.191	Mean-0.192 S.D-0.0005 %RSD-0.2604	10	0.191	Mean-0.190 S.D- 0.0008 % RSD -0.4211
2	10	0.192		10	0.190	

**Table 9:** Statistical analysis for ROBUSTNESS of the proposed method.

Sl. No.	260 nm	262 nm	264 nm
1	0.184	0.191	0.188
2	0.186	0.192	0.187
3	0.188	0.192	0.185
Mean	0.186	0.192	0.187
SD	0.0016	0.0005	0.0012
%RSD	0.8602	0.2604	0.6417

### CONCLUSION

The method proposed in the above study was found to be simple, specific, economic, precise and rapid for the determination of Niacin in bulk as well as in its dosage form. Sample recoveries in all formulations were in good agreement with their respective label claims without interference of excipients and additives. Being economic and precise, the developed method may be preferred as an alternative method for the routine analysis of the Niacin in bulk and pharmaceutical dosage form.

### ACKNOWLEDGEMENT

Authors are thankful to the authority of Girijananda Chowdhury Institute of Pharmaceutical Science, Guwahati for providing necessary facility for conducting this work.

**Financial support and sponsorship:** Nil.

**Conflict of interest:** The authors declare no conflict of interest.

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**How to cite this article:**

Chanda I, Bordoloi R, Chakraborty DD, Chakraborty P, Das SR. Development and Validation of Uv-Spectroscopic Method for Estimation of Niacin in Bulk and Pharmaceutical Dosage Form. *J App Pharm Sci*, 2017; 7 (09): 081-084.