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Development and validation of spectrophotometric determination for niacin content in ginger

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Abstract
A novel, simple, specific and economic UV spectrophotometric method was developed using water as solvent to estimate niacin content in ginger. The λ-max of niacin was found to be 262 nm. Linearity in the concentration range of 5-30 µg/cm^3 was found to be exhibiting good correlation coefficient (R2=0.9955). The developed method was validated statistically to demonstrate linearity, accuracy, precision, LOD and LO. The results of the study proved the applicability of the present method in routine analysis of niacin in ginger.

Keywords: Niacin, UV Spectrophotometry, Method development, validation, ICH guideline

Introduction
Ginger is one of the medicinal plants known for its therapeutic benefits since ancient times, and is used in several forms fresh, dried, powder or oil. Ginger contains many minerals, vitamins and oils and antioxidants and so it has many medical and aesthetic benefits (Shady 2013) [10].
Niacin, or vitamin B-3, or nicotinic acid, is chemically pyridine-3-carboxylic acid, official in IP (Indian Pharmacopoeia 2007) which is a colorless, water- soluble solid. It has high stability towards light and heat. Niacin helps to maintain the structure of the blood cells and improves blood circulation. That's why niacin brings more blood flow to the scalp, bringing more oxygen and nutrients to the hair follicles. Literature survey has revealed various analytical methods for determination of niacin in pharmaceutical formulations in combination with other drugs (Narayankar 2015). In the present study, efforts will be made to develope and validate a simple, specific and economic UV spectrophotometric method using water as solvent to determine niacin content in ginger water extracts according to the ICH guidelines (2005).

Materials and methods
Materials
Niacin standard, distilled water, ginger powder, some plants extract, whattman filter paper no. 41.

Instrument
Spectrophotometric analysis was done using UV spectrophotometer. (Shimadzo, 1650PC, Japan) with 1cm matched quartz cells.

Methods
Determination of wavelength of maximum absorbance(λ-max)
A standard stock solution of niacin (100 µg/cm^3) was prepared using distilled water as solvent and 0.2cm^3 was diluted to 10cm^3 with the same solvent to obtain 2 µg/cm3 standard solution. The standard solution was scanned in the wavelength region of 200- 400 nm.

Assay of content of niacin in ginger
A method for the determination of niacin was developed and applied to analyze it in ginger and some plants extract. 0.5 g of ginger was powdered and dissolved into 25 cm3water by shaking for one hour for complete extraction of niacin. The solution was then filtered through Whattman filter paper no. 41. This filtrate was diluted suitably with distilled water. The absorbance of this solution was measured and the amount of niacin was read from the calibration curve.

Method validation
Validation of the developed method was done following the guidelines laid down in International Conference on Harmonization (ICH) guidelines (2005).The following parameters were evaluated:

Linearity and range
Linearity of any analytical method is its ability, within a given range, to get test results that are directly, or through a mathematical transformation, proportional to concentration of analyte. Six different concentrations (5- 30µg/cm^3) of niacin were scanned on UV spectrophotometer in UV-range (i.e., 200-400 nm). The spectrum was recorded. Least square regression analysis was done by constructing the calibration plot between concentration and absorbance (Jain 2011) [5].

Sensitivity
Sensitivity of the developed method was determined with respect to limit of detection (LOD) and limit of quantification (LOQ). A series of varying drug concentrations (5-30µg/ml) were analyzed to find LOD and LOQ. LOD is the lowest detectable amount of an analyte in a given sample that may or may not be quantified, under the stated experimental conditions, whereas LOQ is the lowest quantifiable amount of analyte in any sample. LOD and LOQ were computed by using standard deviation (σ) and slope value (s) obtained from calibration curve (Revathi 2014) [9].
Equations: LOD = 3.3 σ/s LOQ = 10 σ/s The LOD and LOQ were calculated according to the 3.3 σ/S and 10σ/S

criterions, respectively, where σ is the standard deviation of the y- intercept of the regression line and s is the slope of the calibration curve.

Accuracy

Accuracy is the percentage of analyte recovered by assay from known added amount. Solutions were prepared at levels 75%, 100% and 150% of 20 µg/cm^3 test concentration of the sample solution using standard working solution as per the test method and absorbance was noted down. The whole procedure was done in triplicate (Sethuraman 2013).

Precision

Precision of an analytical method is the degree of repeatability under the normal operation conditions. The precision was determined with standard quality control samples prepared in triplicate at same concentration covering the entire linearity range. The precision of assay was determined by intra-day and intermediate, i.e. inter-day, precision (comparing the assay conducted on 3 different days) and were recorded as % RSD for a statistically significant number of replicate measurements (Bhavar 2015) [1].

Repeatability

Repeatability analysis was performed by analyzing samples of same concentrations (six times) of standard niacin (0.8 µg/cm^3). From the resulting absorbance, SD (standard deviation) and RSD (relative standard deviation) were calculated.

Robustness

The robustness of any analytical method is the measure of its capacity to remain unaffected by small, but deliberate variations in method parameters. Robustness is an indicative of reliability of a method during normal usage. Robustness was tested by varying detection wavelength (±1 nm) of optimized conditions from the standard detection wavelength (262 nm) (Desai P 2013) [2].

Results and discussion

The λ-max of niacin in distilled water was found to be 262 nm. The absorbance maximum of the drug was recorded by taking scan of the niacin sample solution in the UV region (200-400 nm) (Figur.1).

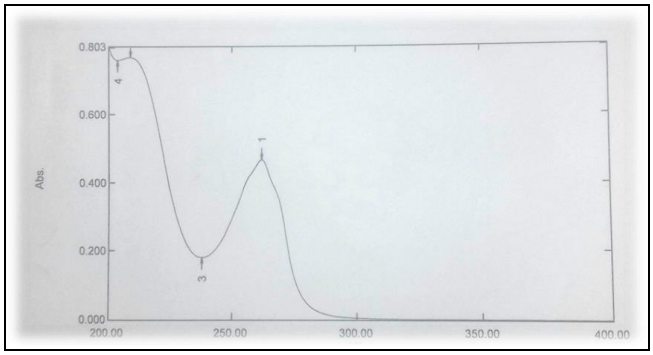

Fig 1: UV Spectrum of standard Niacin

Niacin was found to be linear within the concentration range 5-30 µg/ml and exhibited correlation coefficient of 0.995. The result of regression analysis is given in Table 1.

Validation parameters of developed analytical method

Linearity and range

Good linear correlation was observed between absorbance and concentration in the selected concentration range of 5-30 µg/cm^3. The regression equation was recorded to be y = 0.034x - 0.057. The correlation coefficient (R^2) of the standard curve was found to be 0.9955, (Figure 2). The results are tabulated in Table 1.

Table 1: Spectrophotometric data for calibration curve of niacin at 262 nm

Concentration (µg/ cm^3)	*Absorbance S.D. (nm)
5	0.133
10	0.256
15	0.465
20	0.603
25	0.818
30	0.961

*Each value is the average of three determinations

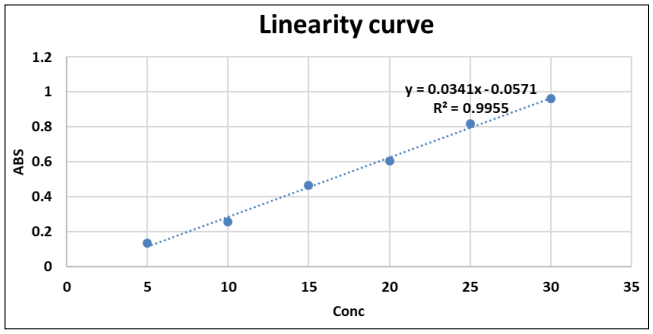

Fig 2: Linearity curve of niacin at 262 nm.

Sensitivity

Calculations of LOD and LOQ of method are based on the standard deviation of y-intercept of regression line (σ) and the slope (s) of the calibration curve at levels approximating the LOD and LOQ. LOD and LOQ were calculated according to the formulae: LOD = 3.3 σ/S = 2.996 µg/cm^3 LOQ = 10 σ/S = 9.08 µg/cm^3.

Accuracy/Recovery

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.

Precision

The samples were estimated similarly daily, for three consecutive days. The developed method was found to be precise as the average % RSD values for intraday and inter-day precision study was found to be 0.378%, 0.355%and 0.343% respectively. The results obtained from intra-day and inter- day precision are shown in Table 2.

Table 2: Results of intraday and inter day precision

S. no.	Conc. mg/ cm3	Abs Day 1	Abs Day 2	Abs Day 3
1	0.8	0.188	0.198	0.207
2	0.8	0.187	0.201	0.202
3	0.8	0.186	0.199	0.208
Mean		0.187	0.199	0.206
SD		0.0007	0.0007	0.0007
% RSD		0.378	0.355	0.343

Robustness

Robustness studies assume that the obtained results are insignificantly affected by small variations in any of the variables (Table 3), they ensured the reliability of the proposed method during routine analysis.

Table 3: Results of robustness studies

Sample NO	Conc. (µg/cm3)	Abs at 261nm	Abs at 262 nm	Abs at 263nm	Abs at 264nm
1	20	0.561	0.565	0.552	0.528
2	20	0.567	0.566	0.562	0.526
3	20	0.563	0.564	0.562	0.531
Mean		0.564	0.565	0.557	0.528
SD		0.0007	0.0007	0.0007	0.0007
RSD%		0.1254	0.125	0.1265	0.1339

Repeatability

The repeatability of the instrument was validated by taking the absorbance of six samples of the same concentration (0.8mg/ml). The mean absorbance was computed to be 0.253. The results are tabulated in Table 4.

Table 4: Results of repeatability studies of niacin at 262 nm.

Concentration (mg/ml)	Absorbance S.D.	Statistical analysis
0.8	0.257	Mean = 0.253
0.8	0.251	SD = 0.00089
0.8	0.251	%RSD = 0.3535
0.8	0.252	
0.8	0.254	
0.8	0.255	

Conclusion

The method proposed in the above study was found to be simple, specific, economic, precise and rapid for the determination of niacin in ginger. Sample recoveries in all formulations were in good agreement with their respective label claims without interference of excipients. Being economic and precise, the developed method could be preferred as an alternative method for the routine analysis of the niacin in ginger.

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