

Colorimetric Determination of Nicotinamide with Dichlorodicyano Benzoquinone

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Abstract: A Simple and Spectrophotometric method has been developed for the determination of nicotinamide with 2, 3- dichloro -5, 6-dicyano-1, 4- benzoquinone (DDQ). The method is based on charge transfer reaction between nicotinamide and DDQ. The Stoichiometric relationship between nicotinamide and DDQ shows a 2:1 mole ratio. A shift in absorption maxima was noted at 468nm. Beers law was evident between the concentrations of 10 μ g/ml - 90 μ g/ml with molar absorptivity of 0.37 x10³ L mol⁻¹cm⁻¹ and Sandell's sensitivity of 1.0 per 0.001 absorbance unit. The proposed method has been successfully applied in the determination of nicotinamide in pure and commercial forms.

Keywords: Nicotinamide, DDQ, Charge transfer complex, Colorimetry.

Introduction

Nicotinamide (3 -pyridine carbamide) is a water soluble vitamin which plays a role in the controlling of aging process and gene silencing. It is obtained through synthesis in the body or as a dietary source and supplement¹. Nicotinamide can be used in the treatment and prevention of pellagra in humans². A high performance liquid chromatographic method has been reported for the determination of nicotinamide in tablet formulation³. Other reported methods include: non - aqueous titrimetry method⁴, Spectrophotometry⁵. DDQ and other pi - acceptors have been successfully utilized in the determination of a variety of electron donating basic compounds^{6 - 8}. The aim of the present investigation is to optimize the colorimetric conditions for the determination of nicotinamide in pure and commercial form.

Experimental

Instrument

Absorption measurements were made on 752W UV-Visible Grating Spectrophotometer with 1cm silica cell.

Materials and Methods

All chemicals and reagents were of analytical grade and were used as such. Nicotinamide tablet was procured from Contract pharmaceutical, New York. Pure Nicotinamide was supplied by BDH, England and DDQ (98%) was supplied by Sigma - Aldrich, England.

Determination of optimum conditions for Nicotinamide-DDQ complexation 2 ml solution of DDQ

(0.001M) was made in methanol and the wavelength of maximum absorption was determined. 2ml of nicotinamide (0.001M) was made in methanol and the wavelength of absorption was determined. A colour was developed by mixing 2ml of DDQ (1×10^{-3} M) solution in methanol with 2ml of nicotinamide (1×10^{-3} M) solution in methanol and the wavelength of maximum absorption for the resultant solution was determined. Another mixture with a 2:1 ratio of the drug and reagent were prepared and left for 5mins at 50°C before analysis at 468nm against a blank.

Procedure for calibration curve

Serial volumes of 0.04, 0.08, 0.12 0.36ml in 0.04 steps of the standard nicotinamide solution (0.002g/ml) were mixed with sufficient methanol, similar volumes of DDQ solution (0.001g/ml) in 0.04 steps were added. The content were mixed and left at 50°C for 5mins before analysis at 468nm.

Assay determination of nicotinamide

Two tablets equivalent to 40mg of nicotinamide were grinded. An amount equivalent to 0.02g was weighed, dissolved with some solvent and stirred to extract the active ingredient, it was filtered before making up to 10ml to give a concentration of 0.002g/ml. Analysis was carried out at the detected optimum condition at 468nm.

Results and Discussion

Table 1 Optimum conditions, statistical data and regression equation of nicotinamide complex

Parameters	Data
Absorption peaks of the drug(nm)	320 and 370
Absorption peak of DDQ(nm)	350
Absorption maxima(nm)	468
Stoichiometric ratio	2:1
Beer's law limits (μ g/ml)	10-90
Molar Absorptivity ($L \text{ mol}^{-1} \text{ cm}^{-1}$)	0.37×10^3
Maximum time of complexation(mins)	5
Maximum temperature for complex formation($^{\circ}\text{C}$)	50
Sand ell's Sensitivity($\mu\text{gcm}^{-2}/0.001\text{A}$)	1.0
Regression equation ^a ($Y = a + bx$)	$Y = 0.002x - 0.019$
Slope a	0.002
Intercept b	0.019
LOQ	2.89
LOD	0.95
Correlation coefficient(r^2)	0.954

^a n=5

^a Average of three determinations

^a Five independent determination

The optimum conditions for the assay method were established by studying the effect of temperature, time of the given complex. The results are shown in table 1. DDQ solution in methanol displayed absorption peak at 350nm. Nicotinamide displayed two absorption peaks at 320nm and 370nm. Mixing the solutions of nicotinamide and DDQ in methanol causes a bathochromic shift in the absorption Maxima with a new absorption peak at 468nm. Earlier reports had suggested charge transfer complex formation as reason for such new absorption peaks¹¹. The Stoichiometric ratios of the reactant were determined using Job's continuous method of variation¹². Also a 2:1 Stoichiometric ratio of nicotinamide and DDQ was observed which means that 2moles of nicotinamide reacted with 1mole of DDQ as shown in Eq 1. In addition, table 1 shows the effect of time on complex formation. Though the colour formation was instantaneous, maximum complexation was attained after 5mins. As absorbance is directly proportional to concentration, the increase in absorbance is an indication of increase in complex formation however maximum stability occurred at 50°C. Table 1 also shows the slope, intercept, correlation coefficient, sand ell's sensitivity, molar absorptivity, LOD, LOQ. The high molar absorptivity and the small value of sand ell's sensitivity of the resultant coloured complex indicate the sensitivity of the proposed method in the determination of the drug under investigation. Linear relationship (fig.1) was obtained between 10-90 μ g/ml with correlation coefficient of 0.954. The result shown in table 2

shows that the standard deviation was found to be small which indicates high precision of the proposed spectrophotometric method. The low values of limits of detection (LOD) and quantification (LOQ) indicate the possible application of DDQ reagent in the routine analysis of the drug under investigation. The percentage recovery of 94% indicates non interference from the excipients used in the formulations. The student's t test shows no significant differences were found between the calculated and theoretical values of the proposed method at 95% confidence level, this equally indicates good precision and accuracy of the proposed method. The quantitative results for the determination of nicotinamide by DDQ are summarized in table 2.

Table 2 Application of the proposed method for the assay of nicotinamide

Drug	Taken($\mu\text{g/ml}$)	Found ($\mu\text{g/ml}$)	Recovery(%)	RSD (%)	S.D	t-test	F-test
Nicotinamide	40	38	95	1.05	0.08	0.71	0.69
	60	57	95	0.98	0.11		
	70	65	93	1.01	0.14		

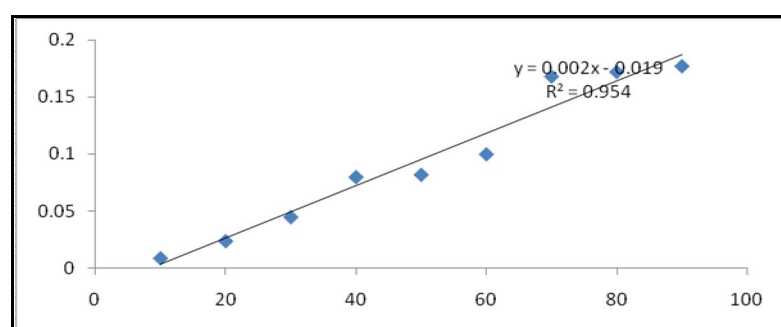


Figure 1: Beer's plot nicotinamide –DDQ Complex in Methanol medium



Conclusion

The proposed spectrophotometric method for the determination of nicotinamide is simple, rapid and economical as compared to other methods used previously. The proposed method has been validated and successfully applied for the quantitative determination of nicotinamide with good accuracy of 94% and can be used for the analysis of nicotinamide in pure and commercial forms.

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