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Method Development and Validation for the Estimation of Sodium Metal Content in Pantoprazole Sodium by Atomic Absorption Spectrophotometer

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Abstract: *A validated method was developed for determination of sodium metal content in pantoprazole sodium by using atomic absorption spectrophotometer (AAS). The method was developed on atomic absorption spectrophotometer with 0.2nm slit width with sodium hallow cathode lamp. The read time was set at 5.0 second with 589.0 nm wavelength. The system performance was evaluated by performing the system suitability parameters. The limit of detection (LOD) and limit of quantification (LOQ) were found to be 0.012 ppm and 0.036 ppm, respectively. The percentages of recovery for low, medium and high spiked concentration levels of sodium metal in pantoprazole sodium were found to be 98.26%, 99.59% and 93.28%, respectively. With the developed method the sodium metal content in pantoprazole sodium bulk sample was found to be 5.72 % which compliance the United States Pharmacopoeia standard.*

Keywords: *Pantoprazole Sodium, Sodium metal, AAS, Validation.*

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I. Introduction

Pantoprazole sodium is an official drug in USP [1] belongs to a category of proton pump inhibitor and is used in treating various acid related disorders [2-3]. From the literature review [4-7], it was found that there is no reported method for the estimation of Sodium metal content in pantoprazole sodium. Abnormal sodium metal levels in body leads to serious ill effects. In the present study, a validated method was developed by using atomic absorption spectrophotometer (AAS) to determine sodium metal content in pantoprazole sodium bulk drug.

II. Materials and Methods

Reagents

Pantoprazole sodium was obtained as a gift sample from Varun herbals, Hyderabad. Sodium metal standard and potassium chloride were purchased from Merck. The Milli Q Water was used.

Preparation of potassium chloride Solution (0.1%):

1.0 gram of potassium chloride was weighed and transferred into a 1000mL volumetric flask and made up to volume with Milli Q water.

Sodium metal Standard Stock Solution Preparation:

Sodium chloride (2.542 gm) was weighed and transferred into a 1000mL volumetric flask and made up to the volume with Milli Q water which was used as stock solution (1000 ppm). Sodium metal standard stock solution (5 mL) was transferred into a 250 mL volumetric flask and made up to the volume with water. To this solution (5mL) was transferred to 200mL volumetric flask and made the volume up to the mark with 0.1% potassium chloride solution which was used as stock solution A.

Sample Preparation

Sample Stock Solution Preparation

Pantoprazole sodium Sample (200 mg) was taken in a dry 250 ml volumetric flask. It was dissolved and made up to the volume with Milli Q water. From the above solution, 10 mL was taken into a 100 mL volumetric flask and made up to the mark with Milli Q water.

Sample Solution Preparation

Sample stock solution (10 mL) was taken into 100 mL volumetric flask and made up to the mark with 0.1 % potassium chloride solution.

Blank Solution

Potassium chloride solution (0.1 %) was used as blank solution.

Instrumental Conditions:

An AA-6300 atomic absorption spectrometer equipped with fully integrated atomizers of Shimadzu make was used for the analysis. The system was operated from an interfaced computer running Wizaard software. The following are the optimal operating conditions for flame atomization of Sodium metal are presented in table 1.

Table 1: Optimal Operating Conditions for Flame Atomization of Sodium Metal

Element	Sodium Metal
Wavelength	589.0 Nm
Read Time	5 Sec
Lamp Current	12 Ma
Recommended Flame	Air-Acetylene
Fuel Gas Flow	2.0 L/Min
Support Gas Flow	17.0 L/Min
Slit Width	0.2 Nm
Signal Type	Atomic Absorption
Atomization Site	Burner Head
Equation	Linear Through Zero
Flame Type	Air-Acetylene
Pre Spray Time	3 Sec
Integration Time	5 Sec

III. Results and Discussion

Specificity

Specificity is the ability to assess the analyte in sample with presents of unexpected other elements which interfere the results of analyte in sample. The results for standard deviation of absorption values (n=6) for sodium metal working standards was within range of acceptance criteria and no interferences were observed. The results were presented in table 2.

Table 2: Specificity

Sodium Metal Concentration (PPM)	**Wavelength (nm)**	**Mean Absorbance (N=6)**
Blank Solution	285.2	0.0020
Standard Solution	285.2	0.3532
Sample Solution	285.2	0.3582

System Suitability

System suitability is a measure to ensure the performance of the system. The results obtained for standard deviation of absorption values (n=3) for sodium metal working standards were presented in Table 3 and was within range of acceptance criteria (correlation coefficient should be NLT 0.99).

Table 3: System Suitability

S.No	**Sodium Metal Concentration (µg/mL)**	**Average Absorbance**
1	Blank	-
2	0.1 Ppm Standard	0.0973
3	0.5 Ppm Standard	0.5137
4	1 Ppm Standard	1.0001
	Correlation	0.9998

Linearity

The concentration of standard solution is directly related and proportional to absorption in their lower and upper limits. The calibration curve was shown in fig. 1 and regression equation presented in table 4 and was within range of acceptance criteria (Correlation Coefficient should be NLT 0.99)..

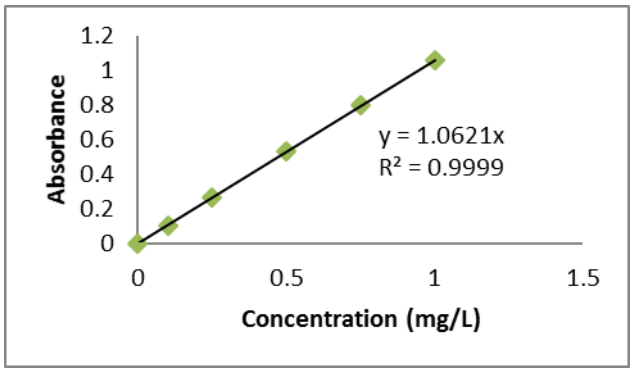

Fig 1: Calibration Curve

Table 4: Calibration Curve

S.No	Concentration (µg/ mL)	Absorbance	Correlation
1	Blank	-	1.0000
2	0.1 Ppm	0.1026	
3	0.25 Ppm	0.2632	
4	0.5 Ppm	0.5326	
5	0.75 Ppm	0.8011	
6	1.0 Ppm	1.0589	

System Precision:

System precision reported as percentage relative standard deviation (%RSD) by aspirating the 0.5 ppm sodium metal standard for 6 times. The results were presented in table 5 and were within range of acceptance criteria (%RSD should be NMT 5.0).

Table 5: System Precision

S.No	Sodium Metal Concentration (0.5 PPM)	Absorbance
1	Replicate-1	0.5340
2	Replicate-2	0.5324
3	Replicate-3	0.5373
4	Replicate-4	0.5376
5	Replicate-5	0.5339
6	Replicate-6	0.5375
Average		0.5354
%RSD		**0.43**

Method Precision

Method Precision was performed by aspirating 6 preparations of sample and single preparation of 0.5 ppm sodium metal standard. The results were presented in table 6 and were within range of acceptance criteria (%RSD should be NMT 5.0).

Table 6: Method Precision

S.No	Name	Weight of sample taken	Average Standard Absorbance (0.5 PPM)	Average Sample Absorbance	Sodium metal Content on Anhydrous basis w/w
1	Sample -1	200.3 mg	0.5358	0.4506	5.62
2	Sample -2	200.2 mg		0.4546	5.67
3	Sample -3	200.6 mg		0.4605	5.74
4	Sample -4	205.2 mg		0.4605	5.61

5	Sample -5	200.2 mg		0.4608	5.75
6	Sample -6	200.4 mg		0.4622	5.76
Average Sodium metal Content					**5.69**
SD					**0.067**
%RSD					**1.18**

LOD

LOD is the lowest amount of analyte that can be detected which is determined from linearity curve. The LOD was found to be 0.012 ppm

From Linearity Slope: 1.06514

From Linearity Steyx: 0.00386

Detection Limit (LOD) = 3.3 × Steyx/ Slope

= 3.3 × 0.00386/ 1.06514

= 0.012 ppm

LOQ

LOQ is the lowest amount of analyte that can be quantitatively determined from linearity curve. The LOQ was found to be 0.036 ppm

Quantification Limit (LOQ) = 10.0 × Steyx/ Slope

= 10.0 × 0.00386/ 1.06514

= 0.036 ppm

Accuracy

Accuracy is the closeness of the test results obtained by the method to the true value which was obtained by spiking 50, 100 and 150% of pantoprazole sodium working standard concentrations. Accuracy was indicated by % recovery as shown in Table 7 and were within range of acceptance criteria (% Recovery should be between 80% to 120%).

Table 7: Accuracy

S.No	Sample	% Recovery
1	50 % Spiked	98.26%
2	100 % Spiked	99.59%
3	150 % Spiked	93.28%

Sample Analysis

The sample was prepared and aspirated with the new method developed. The sodium metal content found in the given sample was 5.72 % which is within the limits specified in USP.

IV. Conclusion

In the present study, a validated method was developed to determine the content of sodium metal in pantoprazole sodium bulk drug. The allowed sodium metal content was present between 5.4 % to 6.6% on anhydrous basis by USP. The sample was analyzed by using the method developed and the sodium metal content was found to be 5.72 % which was in acceptable range.

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