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Development and Validation of UV Spectrophotometric Method for Estimation Ibandronate sodium in Pharmaceutical Formulation

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ABSTRACT

A simple, accurate, precise, rapid spectrophotometric method for estimation of Ibandronate sodium in pharmaceutical formulation. Ibandronate sodium is one off the nitrogen carrying bisphosphonate. It prevents osteoclast-conciliate bone resorption, Paget's disease, postmenopausal osteoporosis. The maximum wavelength (λmax) of ibandronate sodium is 218nm. Linearity was observed in the concentration range 2-100µg/ml. The coefficient of variation value was found to be 0.3499. Amount of drug estimated from tablet formulation were in precise with label claim. The method was statistically validated as per ICH guidelines and can be successively applied for analysis for tablet formulation. The proposed method is economical and sensitive for estimation of ibandronate sodium in pharmaceutical formulation.

Keywords-Ibandronate sodium, ICH guidelines, Bisphosphonate, pharmaceutical formulation.

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INTRODUCTION

Bisphosphonates are a class of drugs that prevent the loss of bone density, used to treat osteoporosis and similar diseases. They are the most commonly prescribed drugs used to treat osteoporosis. They are called bisphosphonates because they have two phosphonates ($PO(OH)_2$) groups. They are thus also called diphosphonates. **Ibandronate sodium** is one of the nitrogen carrying bisphosphonate.[13] According to IUPAC nomenclature it is 3-(N-methyl-N-pentyl) amino-1-hydroxypropane-1,1-diphosphonic acid, sodium salt, monohydrate with the molecular formula $C_9H_{22}NO_7P_2Na.H_2O$ and molecular weight of 359.23. It prevents osteoclast-conciliate bone resorption.[16] It is precious for the cure of hypercalcemia of malignancy, Paget's disease, postmenopausal osteoporosis, and corticosteroid-induced osteoporosis metastatic bone disease. The activity of ibandronate on bone tissue is depending on its resemblance for hydroxyapatite, which is fraction of the mineral matrix of bone. In postmenopausal women, it decreases the high rate of bone mass, leading to, a net gain in bone mass.[15] For quantification of impurity and assay of ibandronate sodium, there are so many analytical methods have been determined. The aim of our study was to develop an easy responsive accurate and precise method for determination of ibandronate sodium in pharmaceutical formulations and bulk drugs using UV spectrophotometer.

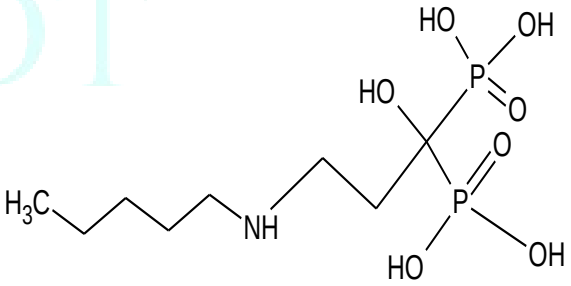

Fig.1 Structure of Ibandronate sodium

MATERIALS AND METHODS

Ibandronate sodium drug (Batch No. IBS/07/11) was obtained from JPN pharma Pvt Ltd, Mumbai. Shimadzu UV Visible Spectro- photometer (UV-1800) with asynchronized pair of 10 mm quartz cells were used for experimental reason.

1. Selection of Solvent

0.1N NaOH was selected as the suitable solvent for estimation of Ibandronate sodium after several trails.

2. Standard Stock Solution

An accurately weighed quantity of Ibandronate sodium(10.0mg) was dissolved in 0.1N NaOH in volumetric flask(10.0ml). The volume was made up to mark with 0.1N NaOH. Appropriate dilutions were made from resulting solution with NaOH so as to get a concentration of 10 µg/ml.

3. Selection of Maximum wavelength (λ max)

Solution was scanned within the wavelength region of 200-400nm against NaOH as blank using UV spectrophotometer. At 218 nm of Ibandronate sodium maximum absorption in absorption curve.

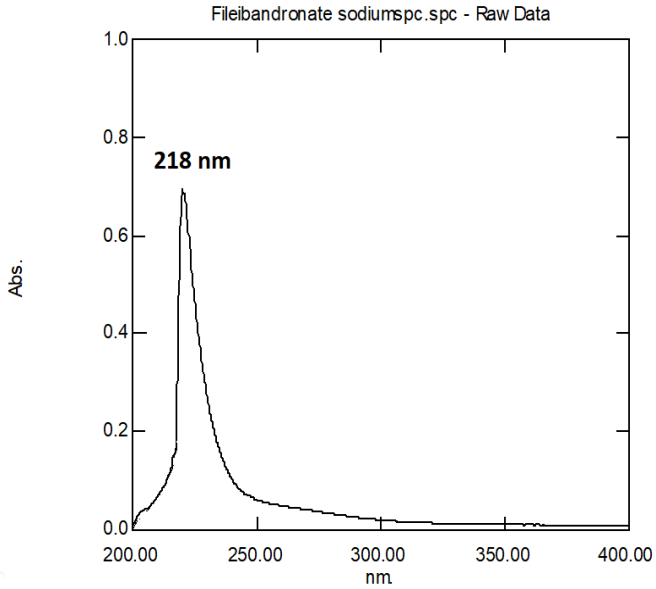

Fig no.2 UV Spectra of Ibandronate sodium

Determination of A(1%.1cm) value of Ibandronate sodium at 218nm A(1%,1cm) = Absorbance/ Concentration (g/100ml)

Table no.1 Result of A1%,1cm.

Sr.no	Concentration	Absorbance	A1%
1	10µg/ml	0.155nm	155
2	10µg/ml	0.153nm	153
3	10µg/ml	0.149nm	149
4	10µg/ml	0.152nm	152
5	10µg/ml	0.158nm	158

Table no.2 Statistical Data

Mean	153.4
Standard deviation	0.9992
Relative standard deviation	0.6513

Application of Proposed Method to Marketed Formulation

Twenty tablets were weighed, calculate average weight and powdered. An amount of tablet powdered equivalent to 10 mg of Ibandronate sodium was weighed accurately and transferred to a 10 ml of NaOH in volumetric flask and sonicated for 15 min and then diluted up to the mark with NaOH. The resultant solution was again diluted by 0.1N NaOH to get concentration 10 µg/ml. The absorbance was measured at selected wavelength 218nm and concentration of the sample present in marketed formulation was determined.

Average weight of tablet=0.601g

Table no.3 Result of assay of tablet

Sr.no	Amount of tablet powder taken(g)	Amount of drug estimated(g)	% labelled claim
1	0.0402g	0.149	99.33
2	0.0400g	0.148	98.66
3	0.0401g	0.151	100.66
4	0.0403g	0.146	97.33
5	0.0401g	0.147	98.00

Table no.4 Statistical Data

Mean	98.79
Standard deviation	1.1436
Relative standard deviation	1.1576

Method Validation

Validation is one of the important steps for analytical determination of any drug. There is a number of most significant validation parameters such as linearity and range, accuracy and precision, LOD, LOQ, recovery, ruggedness, robustness were assessed in developed method.

Linearity and Range

Standard solution was prepared by daily diluting stock solution with NaOH. To get calibration standard solutions 2,5,10,20,30,40,50,60,70,80,90,100 µg /ml of Ibandronate sodium to construct Beers law plot for pure drug. The range of drug to study the Beers law is 2-100µg/ml. A calibration curve was plotted as concentration verses absorbance.

Accuracy

Accuracy of the method was determined by replicate analysis of three sets of samples spiked with three different levels of Ibandronate sodium at level 80%, 100% and 120% and comparing the difference between the spiked value (theoretical value) and that actually found value

Precision

The precision of the method based on the study of ruggedness was carried out under different conditions.

Limit of detection (LOD) and Limit of quantification (LOQ)

The method is used based on standard deviation of the response and slope of calibration curve.

The formula is

$$LOD=\frac{3.3\sigma}{S} \qquad LOQ=\frac{10\sigma}{S}$$

Signal to noise ratio (k)=3.3 and 10 for LOD and LOQ respectively

σ = standard deviation of the response ,
S = Slope of the calibration curve

RESULTS AND DISSCUSSION

Validation is one of the important steps for analytical determination of any drug. There is a number of most significant validation parameters such as linearity and range, accuracy and precision, LOD, LOQ, recovery, ruggedness, robustness were assessed in developed method.[20]

Linearity and Range

Standard solution was prepared by daily diluting stock solution with NaOH. To get calibration standard solutions 2,5,10,20,30,40,50,60,70,80,90,100 µg /ml of Ibandronate sodium to construct Beers law plot for pure drug. The range of drug to study the Beers law is 2-100µg/ml. A calibration curve was plotted as concentration verses absorbance.

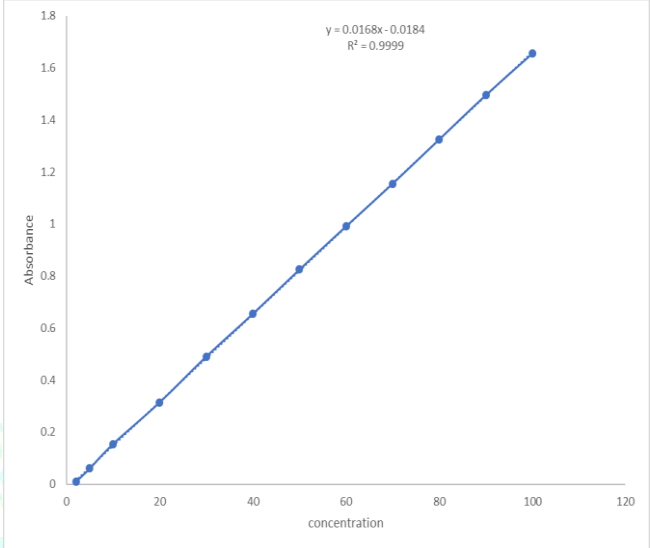

Fig. 3 Calibration curve of Ibandronate sodium

Accuracy

Accuracy of the method was determined by replicate analysis of three sets of samples spiked with three different levels of Ibandronate sodium at level 80%, 100% and 120% and comparing the difference between the spiked value (theoretical value) and that actually found value.

Table no.5 Results of recovery studies

Sr.no.	Amount of pure drug added	Amount of sample added	Amount of drug recovered	% Drug recovery
80 % recovery				
1	40	50	39.85	99.62
2	40	50	39.91	99.77
3	40	50	39.98	99.95
100 % recovery				
1	50	50	49.96	97.92
2	50	50	49.81	99.62
3	50	50	49.89	99.78
120% recovery				
1	60	50	58.91	98.18
2	60	50	59.49	99.15
3	60	50	59.98	99.96

Table.no 6 Statistical data

Mean	Standard deviation	Relative standard deviation
80 % recovery		
99.78	0.11545	0.11570
100 % recovery		
99.10	0.84063	0.84826
120 % recovery		
99.09	0.72341	0.73005

Precision

The precision of the method based on the study of ruggedness was carried out under different conditions.

Table no.7 Result of Intraday

Time	Amount of tablet powdered taken (g)	Amount of drug estimated (g)	% Drug estimated
0hr	0.0401	0.151	100.66
3hr	0.0400	0.150	100.00
6hr	0.0402	0.149	99.33

Table no.8 Statistical data

Drug	Mean	Standard Deviation	Relative standard deviation
Ibandronate sodium	99.33	0.8563	0.8620

Table no.9 Result of Interday

Day	Amount of tablet powdered taken (g)	Amount of drug estimated (g)	% labeled claim
Day1	0.0405	0.149	99.33
Day2	0.0406	0.148	98.66
Day3	0.0400	0.146	97.33

Table no.10 Statistical data

Drug	Mean	Standard Deviation	Relative standard deviation
Ibandronate sodium	98.44	0.8246	0.8376

Limit of detection (LOD) and Limit of quantification (LOQ)

The LOD and LOQ are given in Table no 12.

Table no.11 Result of LOD and LOQ

PARAMETERS	IBANDRONATE SODIUM
LOD(µg/ml)	0.56943
LOQ(µg/ml)	1.72551

CONCLUSION

Simple, accurate, precise, sensitive spectrometric method was done for the estimation of Ibandronate sodium in pharmaceutical formulation.

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