Research Article CODEN: IJPRNK ISSN: 2277-8713
Venkata S Rao Somisetty, IJPRBS, 2013; Volume 2(6): 57-68 IJPRBS

INTERNATIONAL JOURNAL OF PHARMACEUTICAL RESEARCH AND BIO-SCIENCE

NEW ANALYTICAL SPECTROPHOTOMETRIC METHODS DEVELOPMENT AND VALIDATION OF RISEDRONATE SODIUM HEMI PENTA HYDRATE IN BULK AND IN TABLET DOSAGE FORM

VENKATA S RAO SOMISETTY[1], DR. D. DHACHINAMOORTHI[1], D. LAVANYA[1], P. PADMALATHA[1]
1. Department of Pharmaceutical Analysis, QIS college of Pharmacy, ongole-523272, Andhrapradesh, India.

Accepted Date: 08/10/2013; Published Date: 27/12/2013

Abstract: Three simple and sensitive spectrophotometric methods (A, B and C) for the determination of Risedronate Sodium Hemi Penta Hydrate (RSHPH) in pure and pharmaceutical formulations are described. In method A, 0.05 M Hydrochloric acid was used as solvent and shows absorbance maximum at 262 nm. In method B, 0.1 N NaOH was used as solvent and shows absorbance maximum at 262 nm. Method C is a Derivative Spectrophotometric Method 0.05 M Hydrochloric acid was used as solvent and shows absorbance maximum at 272 nm. In method A linearity was found to be in the range of 5 - 60 µg/ml. In method B linearity was 10 – 100 µg/ml and for method C linearity was 5 – 60 µg/ml; for method A (Y=0.019312088x+0.001688645; r2=0.9998) for method B (Y=0.010772121x-0.002545455; r2=0.9997), and for method C (Y=0.001535238x+0.000174603; r2=0.9999), respectively. The proposed methods were successfully applied for the determination of RSHPH in pharmaceutical formulations.

Keywords: Risedronate Sodium Hemi Penta Hydrate (RSHPH), UV-Spectrophotometric methods, Tablets.

PAPER-QR CODE

Corresponding Author: MR. VENKATA S RAO SOMISETTY

Access Online On:

www.ijprbs.com

How to Cite This Article:

Venkata S Rao Somisetty, IJPRBS, 2013; Volume 2(6): 57-68

INTRODUCTION

Risedronate Sodium Hemi-pentahydrate chemically known [1-hydroxy -2-(3-pyridinyl) ethylidene] bis [phosponic acid] monosodium salt (Figure 1 (a)). Has an affinity for hydroxyapatite crystals in bone and acts as an antiresorptive agent. At the cellular level, Risedronate Sodium Hemi-pentahydrate inhibits osteoclasts. The osteoclasts adhere normally to the bone surface, but show evidence of reduced active resorption (e.g. lack of ruffled border). Literature survey reveals that there are only few methods reported for quantitative analysis of Risedronate Sodium Hemi Penta Hydrate and its metabolites in human plasma and urine samples. The aim of this study was to develop rapid, economical, precise and accurate methods for the determination of Risedronate Sodium Hemi-pentahydrate in pharmaceutical formulations. The method described is quite suitable for the routine analysis of pharmaceutical formulations.

MATERIALS AND METHODS

T60 UV-Visible Spectrophotometer with 1 cm matched quartz cells were used for all spectral measurements. Digital Balance: BL-220H, Shimadzu was used.

Reagents: NaOH, Hydrochloric acid, Double distilled water.

Procedure

From the solubility studies, 0.05 M Hydrochloric acid was selected as solvents for Method A, Method C and 0.1 N NaOH was selected as solvent for method B. UV spectroscopical studies of Risedronate Sodium Hemi Penta Hydrate (RSHPH) in bulk drug and tablet dosage form. The λmax was determined in Hydrochloric acid and NaOH.

Method A

Standard stock solution of Risedronate Sodium Hemi Penta Hydrate (RSHPH) (1000 µg/ml) was prepared in 0.05 M Hydrochloric acid. It was further diluted to obtain 10, 20, 30, 40, 50 and 60 µg/ml with 0.05 M Hydrochloric acid. The absorbance was measured at 262 nm against 0.05 M Hydrochloric acid as blank. The calibration curve was plotted in the concentration range of 10 to 60 µg/ml of RSHPH in 0.05 M Hydrochloric acid. The sample solution was also treated in the similar manner. The amount of drug in the tablet sample was computed from Beer-Lambert plot.

Method B

Standard stock solution of Risedronate Sodium Hemi Penta Hydrate (RSHPH) (1000 µg/ml) was prepared in 0.1 N NaOH. It was further diluted to obtain 20, 40, 60, 80, 100 µg/ml with 0.1 N

NaOH. The absorbance was measured at 262 nm against 0.1 N NaOH as blank. The calibration curve was plotted in the concentration range of 20 to 100 µg/ml of RSHPH in 0.1 N NaOH.The amount of RSHPH present in the tablet sample solution was computed from its calibration curve.

Method C

Standard stock solution of Risedronate Sodium Hemi Penta Hydrate (RSHPH) (1000 µg/ml) was prepared in 0.05 M Hydrochloric acid. It was further diluted to obtain 10, 20, 30, 40 and 50µg/ml with 0.05 M Hydrochloric acid. The Zero order spectrum was derivatised into first order derivative spectrum The absorbance was measured at 272 nm against 0.05 M Hydrochloric acid as blank. The calibration curve was plotted in the concentration range of 10 to 60 µg/ml of RSHPH in 0.05 M Hydrochloric acid. The sample solution was also treated in the similar manner. The amount of drug in the tablet sample was computed from Beer-Lambert plot.

Preparation of sample solution

Tablets containing Risedronate Sodium Hemi Penta Hydrate (RSHPH) were successfully analyzed by the proposed methods: Twenty tablets of RSHPH were accurately weighed and powdered. Tablet powder equivalent to 100 mg of RSHPH was dissolved in 50 ml of 0.05 M Hydrochloric acid and sonicated for 15 minutes, filtered and washed with 0.05 M Hydrochloric acid, the filtrate and washings were combined and the final volume was made to 100 ml with 0.05 M Hydrochloric acid. The solution was suitably diluted and analyzed as given under the assay procedure for bulk samples for Method A and Method C. Same procedure was followed by using 0.1 N NaOH as solvent for Method B.

RESULTS AND DISCUSSION

The UV spectrum of Risedronate Sodium Hemi Penta Hydrate (RSHPH) in 0.05 M Hydrochloric acid for method A and Method C and 0.1 N NaOH for Method B (Fig 2, Fig 3 and Fig 4) has showed maximum absorbance at 262 nm for Method A Method B and 272 nm for Method C respectively. The optical characteristics such as absorption maxima, Beer's law limits, molar absorptivity and Sandell's sensitivity are presented in Table 1. The regression analysis was made, slope (m), intercept (b) and correlation coefficient (r2) obtained from different concentrations and the results are summarized in Table 1. Tablets containing RSHPH were successfully analyzed by the proposed methods. The results are represented in Table 2.

The percentage recoveries thus obtained were given in Table 4. None of the excipients usually employed in the formulation of tablets interfered in the analysis of Risedronate Sodium Hemi Penta Hydrate, by the proposed methods. The precision of the methods were studied as

intra-day, inter-day and repeatability the reslts are summarized in Table 3. The % RSD values less than 2 indicate the methods are accurate and precise.

Fig 1 Structure of Risedronate Sodium Hemi Penta Hydrate

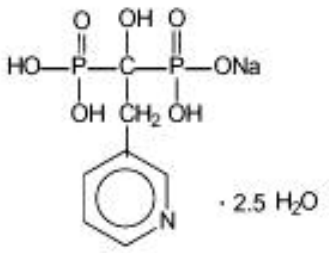

Fig 2 Uv Spectrum Of Risedronate Sodium Hemi Penta Hydrate

In 0.05 M Hydrochloric Acid

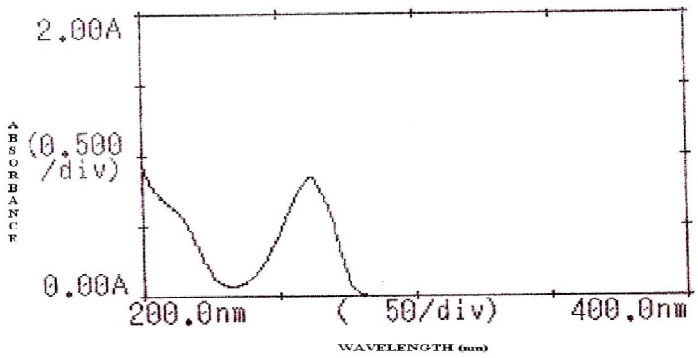

Fig 3 Uv Spectrum Of Risedronate Sodium Hemi Penta Hydrate In 0.1N Sodium Hydroxide

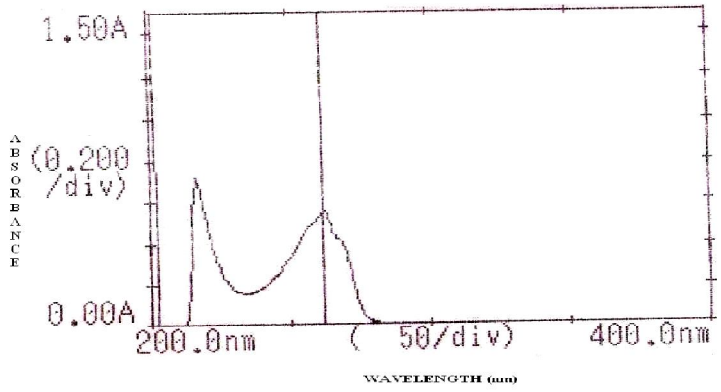

Fig 4 Derivative Spectrum Of Risedronate Sodium Hemi Penta Hydrate In 0.05 M Hydrochloric Acid

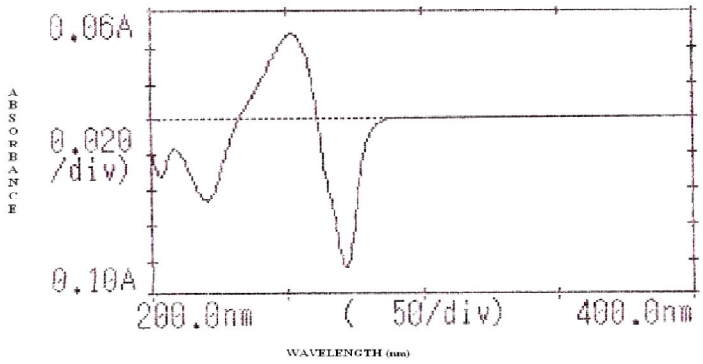

Fig 5 Calibration Curve Of Risedronate Sodium Hemi Penta Hydrate By Uv Spectroscopy Using 0.05 M Hydrochloric Acid At 262nm

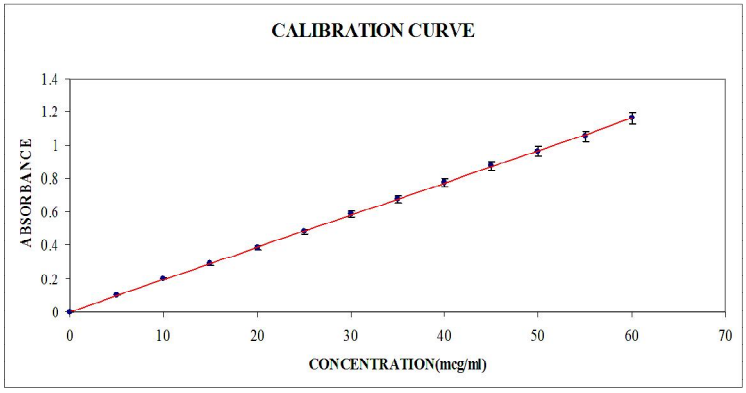

Fig 6 Calibration Curve Of Risedronate Sodium Hemi Penta Hydrate By Uv Spectroscopy Using 0.1 N Sodium Hydroxide At 262nm

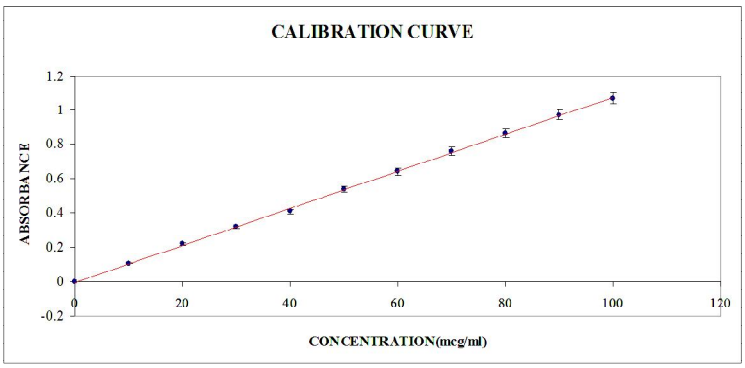

Fig 7 Calibration Curve Of Risedronate Sodium Hemi Penta Hydrate By Derivative Spectroscopy Using 0.05 Hydrochloricacid At 272nm

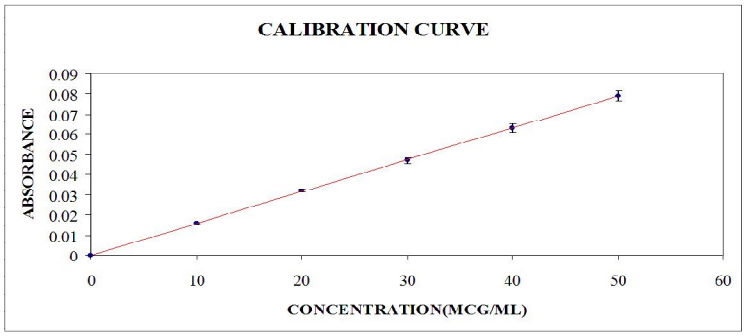

Table 1.

OPTICAL CHARACTERISTICS OF RISEDRONATE SODIUM HEMI PENTA HYDRATE (RSHPH)

Parameters	Method A	Method B	Method C
λ_{max}(nm)	262	262	272
Beer's law limit(µg/ml)	5-60	10-100	10-50
Sandell's sensitivity (µg/cm^2/0.001 AU)	0.051789305	0.092832942	0.651484643
Correlation Co-efficient (r)	0.9998	0.9997	0.9999
Regression equation (Y=mx+c)	Y=0.019312088x+0.001688645	Y=0.010772121x-0.002545455	Y=0.001535238x+0.000174603
Slope(m)	0.019312088	0.010772121	0.001535238
Intercept(c)	0.001688645	(-0.002545455)	0.000174603
LOD(µg/ml)	0.328081742	0.854535649	0.134759674
LOQ(µg/ml)	0.994187095	1.589501968	0.408362649
Standard error of mean of regression line	0.000791395	0.001219376	0.000046456

Table 2.

QUANTIFICATION OF FORMULATION (GEMFOS) BY DERIVATIVE SPECTROPHOTOMETRIC METHODS

Drug	Sample No.	Labeled amount (mg/tab)	Amount found (mg/tab)*	Percentage Obtained*	Average (%) ± S.D	% R.S.D.	S.E.
RSHPH	Method A						
	1.	35	34.65	99.01	99.11 ± 0.0601	0.06058	0.0245
	2.	35	34.72	99.22			
	3.	35	34.80	99.42			
	4.	35	34.65	99.01			
	5.	35	34.65	99.01			
	6.	35	34.65	99.01			
	Method B						
	1.	35	34.693	99.12			
	2.	35	34.888	99.68			
	3.	35	34.888	99.68	99.52 ± 0.1251	0.1257	0.0508
	4.	35	34.823	99.49			
	5.	35	34.693	99.12			
	6.	35	35.018	100.05			
	Method C						
	1.	35	35.17	100.48			
	2.	35	35.10	100.28			
	3.	35	35.33	100.94	100.60 ± 0.1010	0.100	0.0408
	4.	35	35.17	100.48			
	5.	35	35.17	100.48			
	6.	35	35.33	100.94			

Table 3.

INTRA DAY AND INTER DAY ANALYSIS OF FORMULATION (GEMFOS) BY DERIVATIVE SPECTROPHOTOMETRIC METHODS

Drug	Sample No.	Labeled amount (mg/tab)	Percentage obtained*		± S.D		% R.S.D.	
			Intra day	Inter day	Intra day	Inter day	Intra day	Inter day
RSHPH	Method A							
	1	35	98.91	98.15	0.2365	0.2486	0.2382	0.2521
	2	35	99.21	98.62				
	3	35	99.71	99.01				
	Mean		99.27	98.59				
	Method B							
	1	35	97.21	98.62	0.5660	0.4782	0.5785	0.4832
	2	35	98.02	98.79				
	3	35	98.30	99.52				
	Mean		97.84	98.97				
	Method C							
	1	35	97.62	100.60	0.6185	1.2434	0.6341	1.2522
	2	35	96.89	98.12				
	3	35	98.12	99.20				
	Mean		97.54	99.30				

Table 4.

RECOVERY ANALYSIS OF FORMULATION (GEMFOS) BY DERIVATIVE SPECTROPHOTOMETRIC METHOD

Drug	Sample No.	Amount present (µg/ml)	Amount added (µg/ml)	Amount estimated * (µg/ml)	Amount recovered (µg/ml)	% Recovery *	±S.D	% R.S.D	S.E.
RSHPH	Method A								
	1.	2.3668	2.5	4.8545	2.4877	99.50	1.370	1.378	0.135
	2.	2.5222	7.5	10.1401	7.6179	101.57	46	174	982
	3.	2.3149	12.5	14.5450	12.2301	97.84			
	4.	2.4704	17.5	19.8827	17.4123	99.49			
	5.	2.5222	22.5	24.7540	22.2318	98.80			
					Mean	99.44			
	Method B								
	1.	20.08	5	25.00	4.920	98.41		1.131	0.111
	2.	19.01	10	29.07	10.06	100.6	1.117	742	428
	3.	19.90	15	34.64	14.74	98.26	618		
	4.	19.01	20	38.53	19.53	97.65			
	5.	19.01	25	43.72	24.71	98.84			
					Mean	98.75			
	Method C								
	1.	5.8387	5	10.871	5.0323	100.64	1.332	1.347	0.134
	2.	5.7080	10	15.511	9.8039	98.03	28	125	56
	3.	5.8387	15	20.675	14.8363	98.90			
	4.	5.7734	20	25.708	19.9346	99.67			
	5.	5.6427	25	29.956	24.3137	97.25			
					Mean	98.89			

CONCLUSION

Three methods are simple, rapid and accurate and precise and can be used for routine analysis of Risedronate Sodium Hemi Penta Hydrate from tablet formulations.

ACKNOWLEDGEMENT

The authors express their sincere thanks to the management, QIS College of pharmacy, ongole for providing the necessary facilities to carry out the research work.

REFERENCES

1. Anonymous. The Indian Pharmacopoeia Volume I and II. The Controller of Publication, New Delhi, **1996**.

2. Anonymous. The United States Pharmacopoeia. XXIII, 22nd Revision, United States of Pharmacopoeia Convention, Inc., Rockville, MD, USA, **1995**.

3. Alexeyev, V. N. Quantitative Chemical Semi Microanalysis. 1st Indian edn, CBS Publishers and Distributors, New Delhi, **2004**:16-17.

4. Beckett, A.H. and J. B.Stenlake, Practical Pharmaceutical Chemistry, 4th edition, CBS Publishers and Distributors, New Delhi, **2002,** 278-281, 296 - 301

5. Code Q2A, Text on Validation of Analytical Procedures, ICH Harmonized Tripartite Guidelines, Geneva, Switzerland, 27 October, **1994**: 1 - 5.

6. Code Q2B, Validation of Analytical Procedures; Methodology. ICH Harmonized Tripartite Guidelines, Geneva, Switzerland, 6th November, **1996**: 1 - 8.

7. Chatwal and Anand. Instrumental Methods of Chemicals Analysis. 1st edition, Himalaya Publishing House, Mumbai, **2000.**

8. Douglas, A. Skoog, Donald, M. West, James, F. Holler and Stanely, R. Crouch. Fundamentals of Analytical Chemistry. 8th edition, Thomson Asia Pvt. Limited, Singapore, **2006,** 784-811, 973-988.

9. Gary D. Christian. Analytical Chemistry. 6th edn, John Willey and Sons Ltd., New York, **2005**

10. Gupta, S.C. and Kapoor, V.K. Fundamentals of Mathematical Statistics. 9th edition, Sultan Chand and Sons, New Delhi, **1994**, 2.6, 3.2 – 3.28.

11. James, W. Munson. Pharmaceutical Analysis Modern Methods Part B. International Medical Book Distributors, Mumbai, **2001**, 16, 51, 76.

12. Kalsi, P.S. Spectroscopy of Organic Compounds. 6th edn., New age International Pvt. Ltd., New Delhi, **2007.**

13. Mendham. J, Denny.R.C, Barnes, J.D. and Thomas, M.J.K. Vogel's, Text book of Quantitative Chemical Analysis. 6th edition, Pearson Education Pvt. Ltd., New Delhi, **2002.**

14. P.D.Sethi / Dilip Charegaonkar, Identification of drugs in pharmaceutical formulations by Thin Layer Chromatography. 2nd edition; CBS publishers and distributors, 4596/1-A, 11 darya ganj, New delhi-110002, **1999,** 1-30, 32-52.