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DIFFERENCE SPECTROPHOTOMETRIC METHOD FOR ESTIMATION OF SALMETEROL XINOFOATE IN BULK DRUG

Shembade S. H.*[1], Landage S. S.[2] and Tamboli A. M.[3]

Department of Pharmaceutical Chemistry, Sahyadri College of Pharmacy, Methwade Sangola, Solapur, Maharashtra, India. 413307.

***Corresponding Author: Shembade S. H.**

Department of Pharmaceutical Chemistry, Sahyadri College of Pharmacy, Methwade Sangola, Solapur, Maharashtra, India. 413307.

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ABSTRACT

In this research we aimed to develop a simple, precise, economical, accurate and selective method for estimation of Salmeterol xinofoate in bulk drug by using difference spectrophotometric method. Salmeterol xinofoate standard solution was scanned in the UV range (200-400nm) in a double beam UV Spectrophotometer. The spectrophotometric detection was carried out at an absorption maximum of 254nm by using Acetonitrile: Methanol (50:50) as a solvent. The accuracy, precision and other parameters of the method are determined and validated according to ICH guidelines. Thus the proposed method can be applied for estimation of Salmeterol xinofoate in pharmaceutical dosage form by difference spectrophotometric method.

KEYWORDS: Salmeterol xinofoate, Double beam UV Spectrophotometer, Difference Spectrophotometric Method.

INTRODUCTION

Salmeterol xinofoate chemically known as a (RS)-2-(hydroxymethyl)-4-{1-hydroxy-2-[6-(4-phenylbutoxy) hexylamino] ethyl} phenol.[1] Salmeterol xinofoate is a selective adrenergic beta-2 receptor which is used as a bronchodilator. It is used in treatment of asthma and chronic obstructive pulmonary disease. Inhaled Salmeterol work like other beta 2-agonist causing bronchodilation by relaxing smooth muscle in the airway so as to treat the exacerbation of asthma.[2] Spectrophotometric techniques have been used for the determination of Salmeterol xinofoate in its dosage form[3] Salmeterol xinofoate was determined by several method spectrophotometry[4-6], HPTLC[7], HPLC[8-10] and electrophoresis.[11] Salmeterol xinofoate dissociates into solution to yield Salmeterol base and hydroxynaphthoate and having poor aqueous solubility.[12] There are 3 main types of drugs are available for the anti-inflammatory effect such as corticosteroids, decongestants and antihistamines.[13] The chemical formula of Salmeterol xinofoate is $C_{36}H_{45}NO_7$ and molar mass is 603.756g/mol. It is official in I.P. It is freely soluble in methanol.[14]

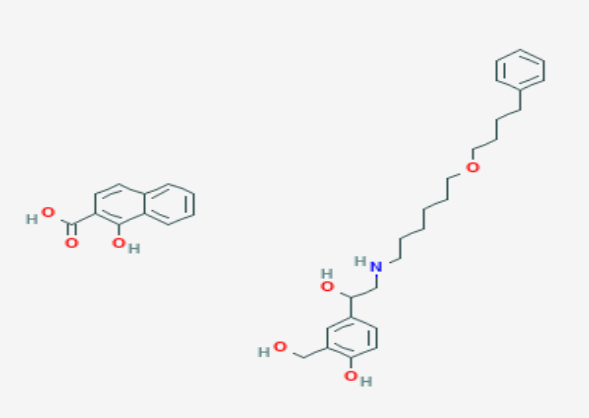

Fig. 1: Chemical structure of Salmeterol Xinofoate.

Molecular formula: - $C_{36}H_{45}NO_7$
Molecular weight: - 603.756g/mol

MATERIALS AND METHODS

Instrument

For weighing the sample calibrated weighing balance is used. A Shimadzu 1800 UV Visible double beam Spectrophotometer with 1cm quartz cell is used for the measurements. All the glassware is well calibrated.

Chemicals

Salmeterol xinofoate pure drug was gifted by Vamsi Labs Ltd, Solapur.

UV SPECTROPHOTOMETRIC METHOD

Selection of solvent

Salmeterol xinofoate is freely soluble in Acetonitrile and water in the ratio (50:50) so, acetonitrile and water is used as a solvent. Also 1N HCL and 1N NaOH is used.

Preparation of standard stock solution

Standard stock solution of Salmeterol xinofoate was prepared by dissolving 10 mg of sample in 100 ml of volumetric flask containing Acetonitrile: Methanol and dissolve properly. Then it is diluted with 1N NaOH and 1N HCl separately to get a series of dilution ranging from 5-25 µg/ml and then absorbance recorded at 214nm and 212nm respectively against blank. Calibration curve was obtained by plotting the concentration Vs difference in absorbance.

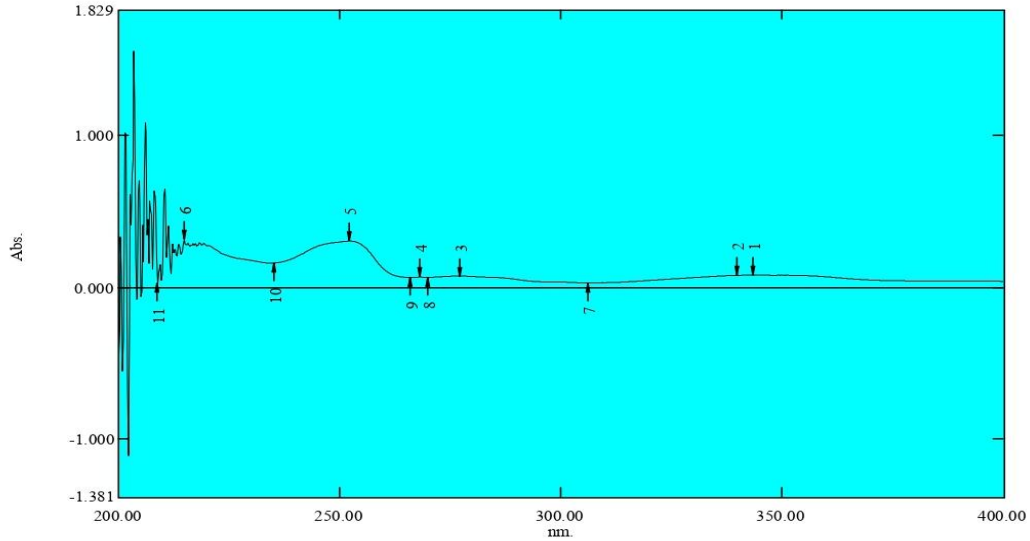

Fig. 2: 1N HCL with λmax 214 nm.

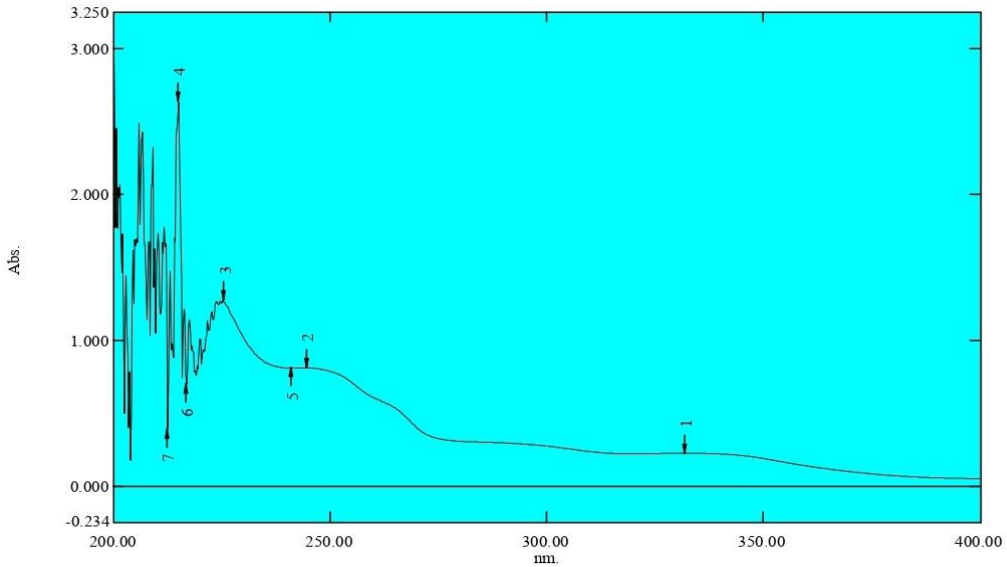

Fig. 3: 1N NaOH with λmax 215nm

Table 1: Linearity of Salmeterol by Difference Spectrophotometry.

Sr. No.	Concentration of Salmeterol xinofoate (µg/ml)	Absorbance at 214 nm (1N HCL)	Absorbance at 215nm (1N NaOH)	Difference in Absorbance
1	5	0.295	0.326	0.031
2	10	0.498	0.567	0.069
3	15	0.785	0.879	0.094
4	20	0.987	1.119	0.132
5	25	1.219	1.375	0.156

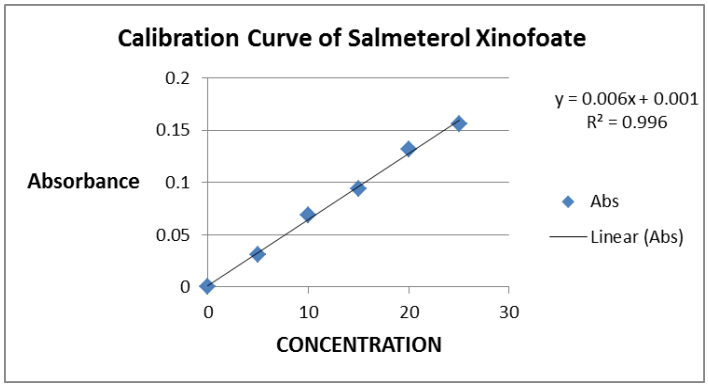

Fig. 4: Calibration Curve of Salmeterol Xinofoate.

VALIDATION OF METHODS

The developed method was validated as per ICH guidelines for following parameters.

1. Linearity

The absorbance of all solution was measured and graph was plotted concentration against absorbance. The correlation coefficient (r^2) of linear regression of Salmeterol xinofoate was calculated.

2. Accuracy

This study was carried out by the standard addition method by adding known amount of Salmeterol xinofoate to pre-analyzed sample at three different concentration levels that is 80%, 100%, 120% and percent recovery of Salmeterol xinofoate were calculated. Standard deviation % RSD was calculated by following equation.

% Recovery = Observed value / True value ×100

3. LOD and LOQ

Determination of detection and quantification limits was performed based on the standard deviation of the y-intercept and slope of least square line parameters. LOD and LOQ can be calculated by following equation.

$$LOD = 3.3\ \sigma/S \text{ and } LOQ = 10\ \sigma/S$$

Where, σ = standard deviation of response

S = slope of calibration curve

4. Precision

The precision of the method was determined in terms of repeatability and reproducibility. Intra-day precision and Inter-day precision. Intra-day precision was determined by analyzing the drug at each concentration for three times and Inter-day precision was determined as similarly but analysis is carried out daily for two days.

Table 2: Result of Accuracy.

Sr.No.	% Level	Spiked Concentration (µg/ml)	Amount recovered (µg/ml)	Absorbance	% Recovery	% RSD
1	80%	4.99	4.93	0.180	98.79	0.19
2	100%	9.98	9.94	0.271	99.59	0.68
3	120%	14.97	14.89	0.368	99.46	0.31

(Values are expressed as Mean ± S.D. of 3 reading)

Table 3: Result of LOD and LOQ of Salmeterol Xinofoate.

Drug	LOD (µg/ml)	LOQ (µg/ml)
Salmeterol Xinofoate	5.58 µg/ml	16.93 µg/ml

Table 4: Characteristics and Validation Parameters of Salmeterol xinofoate.

Parameters	Values	
	NaOH (215 nm)	HCL (214nm)
Beer's law limit (µg/ml)	10-25	10-25
λmax (nm)	244	252
Regression equation (Y=a + bc)	y=0.006x +0.001	
Correlation coefficient (r^2)	0.996	
Slope (b)	0.006	
Intercept (a)	0.001	
Recovery (%)	105.83±5.33	
SE of Intercept	0.004543	
SD of Intercept	0.010159	
LOD (µg/ml)	5.58 (µg/ml)	
LOQ (µg/ml)	16.93 (µg/ml)	

Table 5: Precision results % RSD.

Concentration (µg/ml)	Intraday precision %RSD		Interday precision %RSD	
	HCL	NaOH	HCL	NaOH
10	2.72	2.76	2.84	2.73
15	2.38	1.54	2.40	1.83

(Values are expressed as Mean ± S.D. OF 3 reading).

RESULT AND DISCUSSION

1. Linearity

The linearity for Salmeterol Xinofoate was found to be linear in the range 5-25µg/ml with R^2 = 0.0996 and straight line equation as y= 0.006x+0.001.
The linearity results are shown in table 1.

2. Accuracy

The accuracy of Salmeterol Xinofoate was determined at 80%, 100% and 120% of the standard solution and result was expressed in terms of % recoveries and % RSD. The % recovery was found to be 99.59- 99.46% and % RSD is below 2%.
The results of accuracy are shown in table 2.

3. LOD and LOQ

The limit of detection (LOD) and limit of quantification of Salmeterol xinofoate was found to be 5.58µg/ml and 16.93µg/ml respectively. The results are shown in table 3.

4. Precision

The results of precision are shown in table 4. The precision result showed good reproducibility with relative standard deviation (% RSD) below 2%. From this it is concluded that the method is highly precise. All statistical data prove validity of method and used for routine analysis of Salmeterol Xinofoate. The results are shown in table 5.

CONCLUSION

The UV Spectrophotometric method was developed and it is found to be simple, precise, highly reproducible and inexpensive. The proposed is method suitable for the determination of Salmeterol xinofoate in its dosage form. The advantage of this method is that low cost of reagent, simplicity of sample treatment and satisfactory results.

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