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RESEAECH ARTICLE

Development and Validation of UV-Spectroscopic Method for Estimation of Formoterol in Bulk and Pharmaceutical Dosage Form

K. Surendra*, P. Penchalamma, G. Vikas, C. Dileep Reddy, S. Chaitanya

Rao's College of Pharmacy, Chemudugunta, Venkatachalam, Nellore, Andhra Pradesh-524320

ABSTRACT

The aim of present research work method development and validation for the quantification of Formoterol in bulk and its pharmaceutical dosage form by using UV spectroscopy. The solvent employed for this method was distilled water and absorption maximum was found to be 287nm. The developed method was shown linearity in the range between 5-30µg/ml. The correlation co-efficient was found to be 0.9998. In precision studies the %RSD was found to be ≤ 2. The accuracy was performed by spiking standard drug at 50%, 100% and 150% of the test concentration and the values obtained were within the limit. All the results were satisfactory the developed method was linear, accurate and reproducible.

Keywords: Formoterol, UV Spectroscopy, Distilled water, Assay, Validation.

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Corresponding Author
K. Surendra
Rao's College of Pharmacy, Chemudugunta,
Venkatachalam, Nellore,
Andhra Pradesh-524320
MS-ID: IJCPS4167

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CONTENTS

1. Introduction

Formoterol chemically known as N-[2-hydroxy-5-(1-hydroxy-2-{[1-(4-methoxyphenyl) propan-2-yl] amino} ethyl) phenyl] formamide. It is a long-acting (12 hours) beta2-agonist used in the management of asthma and/or chronic obstructive pulmonary disease (COPD). Inhaled formoterol works like other beta2-agonists, causing bronchodilatation through relaxation of the smooth muscle in the airway so as to treat the exacerbation of asthma. Literature review reveals that there is no analytical method reported for the analysis of Formoterol by estimation by

Uv–Visible Spectrophotometer. Spectrophotometer and Spectroscopy are the reported analytical methods for compounds either individually or in combination with other dosage form. Hence, it was felt that, there is a need of new Spectrophotometer method development for the estimation of Formoterol in pharmaceutical dosage form.

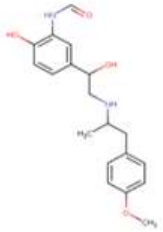

Fig 1: Chemical structure of Formoterol

2. Materials and Methods

Material: Formoterol was a gift sample from Dr. Reddys Lab, Hyderabad. All chemicals(distilled water, methanol) and reagents used were of analytical grade and purchased from Qualigens Fine Chemicals, Mumbai, India.

Apparatus:

A Labindia Uv–Visible spectrophotometer (UV-T60-India) was used for all absorbance measurements with matched quartz cells.

Method Development

Preparation of standard stock solution:

Accurately weighed 10 mg of Formoterol was transferred to a 100 ml volumetric flask, dissolved in 20 ml distilled water by shaking manually for 10 min. The volume was adjusted with the same up to the mark to give the final strength, i.e.100 µg/ml.

Selection of wavelength for analysis of Formoterol

Appropriate volume 0.5 ml of standard stock solution of Formoterol was transferred into a 10 ml volumetric flask, diluted to a mark with distilled water to give concentration of 5 µg/ml(and also 10, 15µg/ml). The resulting solution was scanned in the UV range (200–400 nm). In spectrum Formoterol showed absorbance maximum at 287 nm.

Validation of the method

The method was validated in terms of linearity, accuracy, precision, and ruggedness.

Linearity:

Different aliquots of Formoterol in the range 0.5–3 ml were transferred into series of 10 ml volumetric flasks, and the volume was made up to the mark with distilled water to get concentrations 5,10,15,20,25, 30 µg/ml, respectively. The solutions were scanned on a spectrophotometer in the UV range 200–400 nm. The spectrum was recorded at 287 nm. The calibration plot was constructed as concentration vs. absorbance.

Accuracy:

To the preanalysed sample solutions, a known amount of standard stock solution was added at different levels, i.e. 50%, 100%, and 150%. The solutions were reanalyzed by the proposed method.

Precision:

Precision of the method was studied as intraday and interday variations. Intraday precision was determined by analyzing the 10, 15 and 20 µg/ml of Formoterol solutions for three times in the same day. Interday precision was determined by analyzing the 10, 15, and 20 µg/ml of Formoterol solutions daily for 3 days over the period of week.

Sensitivity:

The sensitivity of measurements of Formoterol by the use of the proposed method was estimated in terms of the limit of quantification (LOQ) and limit of detection (LOD). The LOQ and LOD were calculated using equation LOD = 3 × N/B and LOQ = 10 × N/B, where 'N' is standard deviation of the peak areas of the drugs (n = 3), taken as a measure of noise, and 'B' is the slope of the corresponding calibration curve.

Repeatability:

Repeatability was determined by analyzing 20 µg/ml concentration of Formoterol solution for six times.

Ruggedness:

Ruggedness of the proposed method is determined for 20 µg/ml concentration of Formoterol by analysis of aliquots from a homogenous slot by two analysts using same operational and environmental conditions.

3. Results and Discussions

Selection of wavelength for analysis of Formoterol:

During the development phase, the use of ethanol as the diluent resulted in preferable outcome in UV analysis. The pre-determined wavelength of maximum absorption (λmax) was 287 nm.

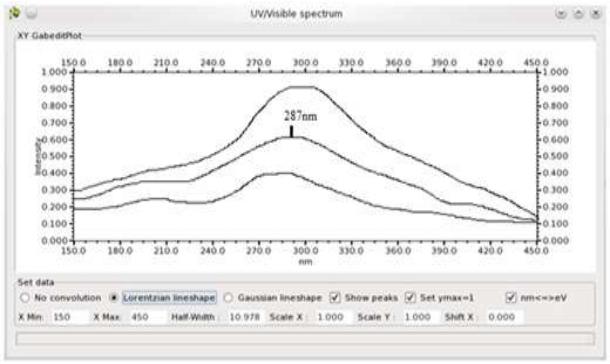

Fig 2: UV Spectrum

Linearity:

Formoterol showed good linearity in the range of 5-50µg/ml. The correlation coefficient was found to be 0.9998. The linearity data are shown in table 1 and figure 2.

Table 1: Linearity Results

Concentration (ug/ml)	absorbance(nm)
0	0
5	0.143
10	0.274
15	0.403
20	0.532
25	0.658
30	0.792

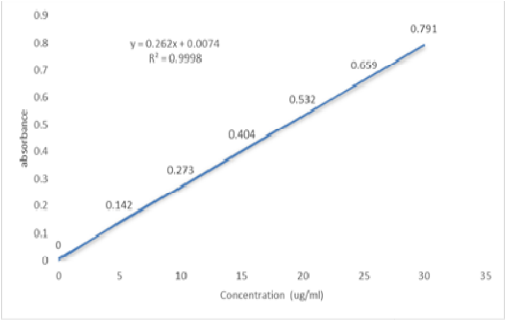

Fig 3: Linearity graph of Formoterol

Accuracy: The recovery studies with standard addition method at 50%, 100% and 150% levels of the test concentration showed good results with a percentage mean recovery was found be 99.65 %. The developed method was accurate. The results are shown in table 2.

Precision:

Both intra-day and inter-day precision was within the acceptable limit with% RSD less than 2%. So, the developed method was more precise and repeatable.

Sensitivity:

The linearity equation was found to be Y = 0.263X + 0.3 The LOQ and LOD for Formoterol were found to be 0.52 µg and 2.98 µg, respectively.

Ruggedness:

The peak area was measured for same concentration solutions, six times. The results are in the acceptable range for both the drugs. The result showed that the % RSD was less than 2%.

Table 2: Results of Accuracy

%Concentration (at specification Level)N=3	absorbance	Amount Added (mg)	Amount Found (mg)	% Recovery	Mean Recovery
50%	0.4215	2.5	2.497	99.93	99.68
100%	0.6214	5.0	4.990	99.07	
150%	0.9199	10	9.995	99.95	

Table 3: Precision Results

Concentration (µg/mL)	Intra-day precision			Inter-day precision		
	Absorbance measured	RSD (%)	Average (%)	Absorbance measured	RSD (%)	Average (%)
10	0.4112	0.140	98.97	0.4120	0.242	98.95
15	0.6148	0.095	98.60	0.6154	0.095	98.72
20	0.9212	0.123	98.78	0.8215	0.071	98.84

Table 4: Results for Ruggedness

Analyst	Concentration (µg/mL)	Absorbance measured (Mean ± SD)	Amount Found (%)	RSD (%)
I	20	0.8216±0.0016	98.97	0.03
II	20	0.8314±0.0012	99.13	0.02

4. Conclusion

This UV-spectrophotometric technique is quite simple, accurate, precise, reproducible, and sensitive. The UV method has been developed for quantification of Formoterol in tablet formulation. The validation procedure confirms that this is an appropriate method for their quantification in the formulation. It is also used in routine quality control of the formulations containing this entire compound.

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