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Development and validation of new RP-HPLC method for the estimation of furazolidone in bulk and solid dosage form

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ABSTRACT

Method is A Rapid, precise, accurate, specific and simple HPLC method developed for estimation of Furazolidone. A High performance liquid chromatograph using Phenomenex-Luna RP-18(2), 250×4.6mm, 5 µm column, with mobile phase having a composition of Methanol: Acetonitrile [90:10 (v/v)] was used. The flow rate of 1.0 ml min^{-1} and effluent was detected at 259 nm. The retention time of furazolidone was 2.770 minutes. Linearity was observed in concentration range of 2-18µg/ml. Atorvastain was used as internal standard (IS), the retention time was found to be 2.196 minutes. The conc of internal standard used was 8 µg/ml in each dilution The mean recoveries were found in the range of 99-100 %. The proposed method was validated for various ICH parameters like linearity, limit of detection, accuracy, precision, ruggedness, robustness, and system suitability.

KEYWORDS

Furazolidone;
Reversed phase;
HPLC estimation;
Validation.

INTRODUCTION

Furazolidone chemically 3-(5-nitrofurfurylidene amino) oxazolidon-2 one belongs to the class of antibacterial and antiprotozoal[1]. Furazolidone is active against the protozoan *Giardia lamblia* (*Giardia intestinalis)* and against a range of bacteria in vitro including *staphylococci, enterococci, Escherichia coli, Salmonella spp., Shigella spp.*, and *Vibrio cholerae*. Furazolidone is bactericidal and appears to act by interfering with bacterial enzyme systems. Resistance is reported to be limited. It is used in the treatment of giardiasis, trichomoniasis, cholera and other vibrio infections[1,2]. It has been suggested for other bacterial gastrointestinal infections but antibacterial therapy with Furazolidone is regarded as unnecessary in mild & self-limiting gastro-enteritis[1].

MATERIALS AND METHODS

Reagents and chemicals

Acetonitrile HPLC grade, Methanol HPLC grade, Standard Furazolidone solution, Double Distilled water

Application of the proposed procedure for the determination of Furazolidone in tablets

Brand name: Furoxone (100mg),
Company name: Glaxo Smith Kline

Preparation of mobile phase (90:10 v/v)

Procedure: 450 ml of methanol was mixed with 50 ml of acetonitrile (90:10) filtered through nylon membrane filter paper and sonicated for 15 min.

Standard solution of furazolidone solution

Accurately 10 mg of pure furazolidone was dis-

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solved in small volume of mobile phase (2.0-5.0 ml) then mix with mobile phase up to 10 ml. 1ml of above stock solution was pipetted to 100 ml volumetric flask with the mobile phase. This gave 100µg/ml of drug concentration.

Procedure

Aliquotes of 0.2 ml, 0.4 ml, 0.6 ml, 1.0 ml, 1.4 ml and 1.8 ml of 100µg/ml of furazolidone solution were pipetted into each of six 10 ml volumetric flasks and 1.0 ml of internal standard solution of 80µg/ml was pipetted into each of above 10 ml volumetric flask and then the volume was made up to 10 ml with mobile phase.20 µl of each solution was injected and chromatograms were recorded.

TABLE 1

Sl. no	Conc. of FZ in (µg/ml)	Peak response
01	02	00.33
02	04	00.58
03	06	00.87
04	10	01.34
05	14	01.91
06	18	02.41

TABLE 2

% of std. addition	Total drug conc in µg/ml	Total conc found in µg/ml	% Recovery ± SD*
75	07	07. 01	100.19±1.207
100	08	07.97	99.66±1.477
125	09	09.01	100.14±0.4045

*Average of five readings

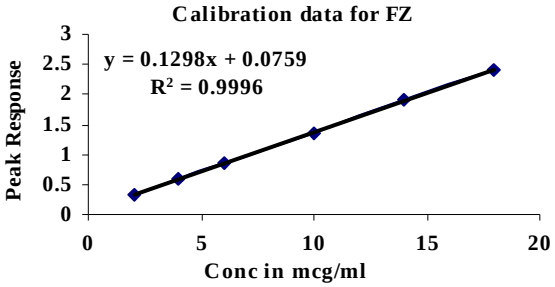

Figure 1: Calibration curve for Furazolidone

RESULTS AND DISCUSSION

Accuracy

Accuracy was found out by recovery study from prepared solution (five replicates) at three levels of standard addition, from 75%, 100% and 125% of the label claim. Aliquots of 0.4 ml of sample drug (FZ) solution of 100 µg/ml were pipetted into each of three volumetric flasks. To this 0.3 ml, 0.4 ml and 0.5 ml of standard drug (FZ) solution of 100 µg/ml was added to each volumetric flask respectively. The volume was made up to 10 ml with mobile phase. 20 µl of each solution was injected and chromatograms were recorded.

Precision

The precision of the assay was determined by estimating samples containing Furazolidone at three different concentrations 6, 10 and 14mg/ml. The two peaks were well separated with the retention time 2.770 and 2.196 minute for Furazolidone and the internal standard respectively (Figure 2). The intra-day co-efficient

TABLE 3

Intra-day variation (n= 9)				Inter-day variation (n = 5)			
Concentration Used µg/ml	Quantity recovered µg/ml	Percent recovered	C.V. %	Concentration used µg/ ml	Quantity recovered µg/ml	Percent recovered	C.V. %
6	6.04±0.0100	100.68	1.162	6	6.12±0.0100	102.01	1.162
10	9.81±0.0100	98.15	0.740	10	10.04±0.00577	100.4	0.4184
14	13.97±0.0152	99.82	0.808	14	14.05±0.00577	100.37	0.3038

Values are mean ± SD, C.V. - Co-efficient of variation.

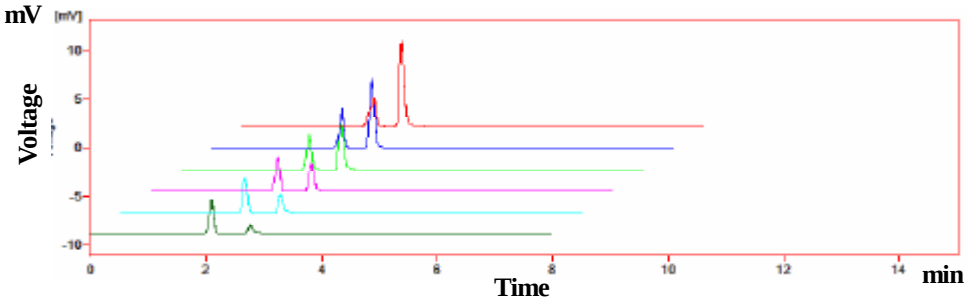

Figure 2: Showing overlay chromatogram of various concentrations of furazolidone with internal standard (Atorvastatin)

TABLE 4: Ruggedness report of FZ

Parameter	Result observed
Percentage area	99.96%
SD between set of analysis on the same date	± 0.04667
RSD between set of analysis on the same date	0.071 %
SD between set of analysis on the different date	± 1.371
RSD between set of analysis on the different date	2.05 %

of variation ranged between 0.7 to1.2 % the inter-day co-efficient of variation varied from 0.3-1.2% (TABLE 3).

Limit of detection (LOD)

Limit of detection was determined by the analysis of samples with known concentrations of analyte and by establishing the minimum level at which the analyte can be reliably detected. 20 µL each of various concentrations was injected in triplicates and the lowest concentration of drugs that can evoke a peak was recorded.

The Limit of detection for was found to be 0.5 µg mL^{-1}.

Ruggedness

The assay was performed in different condition, different analyst, and different dates. From the stock solution of 100µg/ml pipetted out 0.2 ml of Furazolidones to 10 ml volumetric flask, the volume was made up to 10 ml to get concentration of 2µg/ml. As the results in TABLE 4 are within the acceptance limit, the proposed method was found to be rugged.

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APPARATUS AND SOFTWARE

High performance liquid chromatograph, Shimadzu pumpLC-10AT VP equipped with universal injector (Hamilton 25 µL) SPD10A, UV-VIS detector SPD10A-10A VP (Shimadzu).Different kinds of equipments viz Analytical weighing balance (Shimadzu AX 200), Sonicator (model SONICA 2200MH), Water purification system, Vacuum pump (model XI 5522050 of Millipore), Millipore filtration kit for solvents and sample filtration were used through out the experiment. The Spinchrom CFR software was used for acquisition, evaluation and storage of chromatographic data.

CONCLUSION

The proposed RP-HPLC method is found to be accurate, precise, linear, stable, specific, and simple, for quantitative estimation of furazolidone in bulk and solid dosage form. Hence the present RP-HPLC method is suitable for routine assay of furazolidone in bulk and solid dosage form in the quality control laboratories.

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