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"ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF RIFAXIMIN: REVIEW"

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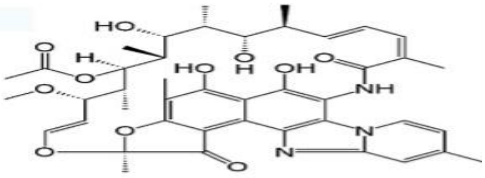
ABSTRACT

Analytical method development and validation are the continuous and interdependent task associated with the research and development, quality control and quality assurance departments. Analytical procedures play a vital role in equivalence and risk assessment, management. It helps in establishment of product specific acceptance criteria and stability of results. Validation demonstrates that the analytical procedure is suitable for its intended purpose. Design of experiment is a powerful tool for the method characterization and its validation. Analytical methods for the estimation of rifaximin in bulk and Pharmaceutical dosage forms developed by HPLC, HPTLC and UV visible spectroscopic techniques. The present methods were accomplish the validation parameters according to ICH guidelines like accuracy, precision, linearity, range, robustness, ruggedness, limit of detection and limit of quantitation etc. The published methods were specific, sensitive, reproducible, precise, rapid and simple.

Key words: Rifaximin, Literature Survey, Method Development, Validation, ICH Guidelines.

1. INTRODUCTION

Rifaximin is used as an antibiotic for the treatment of traveler's diarrhea and hepatic encephalopathy but not used for the systemic bacterial infection and for children below age of 12 years. Rifaximin binds to the beta-subunit of bacterial DNA dependent RNA polymerase and prevents catalysis of polymerization of deoxyribonucleotides into a DNA strand, thereby inhibiting bacterial RNA synthesis. In vitro studies of rifaximin have demonstrated broad-spectrum coverage including gram-positive, gram-negative and anaerobic bacteria as well as a limited risk of bacterial resistance[1].



Chemical structural of Rifaximin is (2S,16Z,18E,20S,21S,22R,23R,24R,25S, 26S,27S,28E)-5,6,21,23,23,25-penta hydroxy-27-methoxy-

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2,4,11,16,20,22,24,26-octamethyl-2,7-(epoxypentadeca-[1,11,13]trienimino)benzofuro[4,5-e] pyrido[1,2-a]-benzimida-zole-1,15(2H)-dione,25-acetate.

2. REVIEW LITERATURE

Krishna V. Prajapati[2] *et al.,* The derivative spectrophotometric method was based on the determination of both the drugs at their respective zero crossing point (ZCP). The first order derivative spectra were obtained in 0.01 N NaOH and the determinations were made at 329.20 nm (ZCP of RIFA) for MESA and 292.80 nm (ZCP of MESA) for RIFA. The linearity was obtained in the concentration range of succinate 10-50 µg/ml for MESA and 10-50 µg/ml for RIFA. The limit of determination was 0.321 µg/ml and 0.301 µg/ml for MESA and RIFA, respectively. The limit of quantification was 0.974 µg/ml and 0.912 µg/ml for MESA and RIFA, respectively. The mean recovery was 100.20 and 99.52% for MESA and RIFA, respectively.

Ana Carolina Kogawa[3] *et al*., The method was validated, showing linearity, selectivity, precision, accuracy, and robustness. It was linear over the concentration range of 10–30mg L−1 with correlation coefficients greater than 0.9999 and limits of detection and quantification of 1.39 and 4.22mg L−1, respectively.

T Sudha[4] *et al*., Two simple, sensitive, accurate and rapid spectrophotometric methods were developed for the estimation of Rifaximin in pure and tablet dosage forms. Method is a colorimetric method based on the oxidation coupling reaction of Rifaximin with ferric chloride and 3-methyl 1,2 benzothiazoline hydrazine hydrochloride (MBTH) reagent giving green coloured chromogen which shows maximum absorbance 637 nm against blank method B is ultra violet method which uses alkaline Borate Buffer (PH - 12) and maximum absorbance was found to be 296 nm. Rifaximin Beers – Lamberts law in the concentration range of 5-25 µg /ml in both method A and B. sandells sensitivity, molar extinction coefficient, slop, intercept, LOD and LOQ were determined for the both methods. The percentage recovery was found to be 100.03 ± 1.2928 in method A and B respectively.

B. R Challa[5] *et al*., The present study was aimed at developing a simple, sensitive, and specific liquid chromatography–tandem mass spectrometry method for the quantification of Rifaximin in human plasma using Rifaximin D6 as internal standard. Chromatographic separation was performed on Zorbax SB C18, 4.6 x 75 mm, 3.5 µm column with an isocratic mobile phase composed of 10 mM ammonium format (pH 4.0) and acetonitrile in the ratio of (20:80 v/v), at a flow-rate of 0.3 mL/min. Rifaximin and Rifaximin D6 were detected with proton adducts at m/z 786.4→ 754.4 and 792.5→ 760.5 in multiple reaction monitoring positive mode respectively. The acidified samples were subjected to liquid–liquid extraction using a mixture of methyl t-butyl ether-dichloromethane (75: 25) followed by centrifugation, nitrogen-aided evaporation and reconstitution. The method was validated over a linear concentration range of 20 20000 µg/mL with correlation coefficient of more than 0.9995.

S. Sumakala[6] *et al*., A RP-HPLC method was developed and validated as per ICH guidelines for the determination of Rifaximin in bulk and formulation. The samples were analyzed by high-performance liquid chromatography (HPLC). Chromatographic separation

was achieved on an inertial C18 (250 x 4.6 mm, packed with 5µ) column using an aqueous sodium acetate Buffer:Acetonitrile (60:40 v/v pH adjusted to 5.0 by using NaOH) with flow rate 1.0ml/min at ambient temperature and the retention time was about 3.489 minutes. UV detection was found at 293nm. Linearity was observed over the concentration range of 20-80 µg/ml (R2 = 0.9997). The LOD and LOQ were found to be 0.7129µg/ml and 2.160µg/ml respectively.

Bikshal Babu Kasimala[7] *et al*., A simple, selective, linear, precise and accurate RP-HPLC method was developed and validated for rapid assay of Rifaximin in tablet dosage form. Isocratic elution at a flow rate of 1ml min-1 was employed on a symmetry C18 column at ambient temperature. The mobile phase consisted of Acetonitrile: Ammonium Acetate 85:15 (v/v). The UV detection wavelength was at 236nm.Linearity was observed in concentration range of 5-50ppm. The retention time for Rifaximin was 4.3 min. The method was validated as per the ICH guidelines.

M. Mathrusri Annapurna[8] *et al*., An isocratic reverse-phase high-performance liquid chromatographic method was developed and validated for the determination of Rifaximin. Linearity was observed in the concentration range of 1.0–300µg mL-1 (R2 = 0.9998). The LOQ was found to be 0.891 µg mL-1 and the LOD was found to be 0.289 µg mL-1. Rifaximin was subjected to stress conditions of degradation in aqueous solutions including acidic, alkaline, oxidation and thermal degradation. Rifaximin is more sensitive towards oxidation in comparison to other degradations. The method was validated as per ICH guidelines. The RSD for intra-day (0.24-0.55) and inter-day (0.38-0.78) precision were found to be less than 1%.

A. Narendra[9] *et al*., An isocratic reversed-phase high-performance liquid chromatographic method was developed and validated for the determination of Rifaximin. The samples were analyzed by high-performance liquid chromatography (HPLC). Chromatographic separation was achieved on a C18 column using an aqueous tetra butyl ammonium hydrogen sulphate: methanol (10:90, v/v), with flow rate 1.0 mL/min (UV detection at 454 nm). Linearity was observed over the concentration range of 1.0–200 µg/mL with regression equation 15407x +6677 (R2 = 0.9999). The LOQ was found to be 0.786µg/mL and the LOD was found to be 0.238µg/mL. Rifaximin was subjected to stress conditions of degradation in aqueous solutions including acidic, alkaline, oxidation, photolysis and thermal degradation. The RSD for intra-day (0.24-0.55) and inter-day (0.38-0.78) precision were found to be less than 1%.

T sudha[10] *et al*., A simple, selective and rapid reverse phase high performance liquid chromatographic (RP-HPLC) method for the analysis of Rifaximin in bulk and in tablet dosage form has been developed and validated. Sample was resolved on a Luna Phenomenax, C18 (150mm X 4.6 mm i.d., particle size 5µ) column. The mobile phase consisted of methanol l:10M phosphate buffer (70:30 v/v pH adjusted to 3.0 by using orthophosphoric acid was delivered at a flow rate of 1.2 ml/min at ambient temperature and the retention time was about 5.12 minutes. Studies were performed on an HPLC system equipped with a UV/Visible detector at 293nm.

M. Mathrusri Annapurna[11] *et al*., An isocratic reversed-phase high-performance liquid chromatographic

method was developed and validated for the determination of Rifaximin. Chromatographic separation was achieved on a C18 column using an aqueous tetra butyl ammonium hydrogen sulphate (10 mM) (pH 3.37): acetonitrile (40:60, v/v), with flow rate 1.2 mL/min (UV detection at 441 nm). Linearity was observed in the concentration range of 0.1–200 µg/mL (R2 = 0.9999). The limit of quantitation was found to be 0.0794 µg/mL and the limit of detection was found to be 0.0241 µg/mL.

3. CONCLUSION

Literature survey suggested that various HPLC, UV, and few HPTLC methods were developed and reported. The published methods were validated for various parameters as per ICH guidelines. Statistical analysis proved that the published methods were reproducible and selective. Thus it can be concluded that the reported and published methods can be successfully applied for the estimation of the rifaximin in pure and pharmaceutical dosage form.

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