



# International Journal of Medicine and Pharmaceutical Research

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## Research Article

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### A Simple UV Spectroscopic Method for the Determination of Rufinamide in Bulk and Tablets

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#### ABSTRACT

A simple, accurate and precise Zero order spectroscopy method was developed and validated for the estimation of Rufinamide in bulk and pharmaceutical dosage forms. Dimethyl sulfoxide and distilled water was used as a diluent to dissolve rufinamide. The drug mixture was sonicated for 5 mins for the enhanced solubility. The maximum absorption was found to be at 262.0 nm, which was selected for the further analysis of rufinamide in bulk and its tablet dosage forms. The proposed method was validated according to ICH guidelines. The method showed high sensitivity with linearity range from 100 to 600 µg/mL ( $r^2=0.999$ ). The limit of detection and limit of quantitation for estimation of rufinamide was found to be 3.44 µg/ml and 10.45 µg/ml, respectively. Recovery of rufinamide was found to be in the range of 99.09% – 100.89%. The reports expressed that the proposed method was found to be simple, precise, accurate and rapid for the estimation of rufinamide in bulk and tablet dosage form using UV spectroscopy.

**Keywords:** Rufinamide, Dimethyl Sulfoxide, Distilled Water, UV Spectroscopy, ICH Guidelines.

#### ARTICLE INFO

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**Article History:** Received 05 March 2015, Accepted 15 April 2015, Available Online 10 June 2015

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PAPER-QR CODE

**Citation:** S. Muneer, et al. A Simple UV Spectroscopic Method for the Determination of Rufinamide in Bulk and Tablets. *Int. J. Med. Pharm. Res.*, 2015, 3(3): 1041-1044.

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#### 1. Introduction

Rufinamide (RFN) is chemically 1-(2,6-difluorobenzyl)-1H-1,2,3-triazole-4-carboxamide (Figure 1) belongs to International Journal of Medicine and Pharmaceutical Research

triazole derivative used as an anticonvulsant drug [1]. It is used in combination with other medication and therapy to

treat Lennox–Gastaut syndrome and various other seizure disorders. It is presumed to involve stabilization of the sodium channel inactive state, effectively keeping these ion channels closed. Although the direct mechanism of action may be different, several other antiepileptic agents also stabilize a sodium channel inactive state. It is also being investigated as a treatment for anxiety disorders.

From the literature survey, it was found that RFN estimated by analytical methods such as spectrophotometric methods, Reversed-phase high-performance liquid chromatographic (Rp-HPLC) method [3-7]. The aim of this study was to develop and validate a simple UV method, to quantify RFN in pure form and pharmaceutical formulation (tablets). The proposed method was validated according to ICH

## 2. Materials and Methods

### Instruments and reagents

An analytically pure sample of RFN was procured as gift sample from Ranbaxy laboratories Ahmedabad, India. Analytical grade DMSO and distilled water was used as solvent for dilution. A double beam UV-Vis spectrophotometer (UV-1800, Shimadzu, Japan) connected to computer loaded with spectra manager software UV Probe was employed with spectral bandwidth of 1nm and wavelength accuracy of  $\pm 0.3$  nm with a pair of 10 mm matched quartz cells. For scanning, the wavelength range selected was from 400 nm to 200 nm with medium scanning speed. All experiments were performed at room temperature ( $25 \pm 1$ )°C. Rufinamide tablet formulation [Banzel containing 100, 200 and 400 mg of drug content, Eisai Pharmaceuticals, India] was procured from a local pharmacy. DMSO and water was used as a diluent to dissolve RFN.

### Preparation of working standard drug solution

50 mg of standard RFN was weighed accurately and transferred to 50 ml volumetric flask. It was dissolved properly and diluted up to the mark with diluent DMSO to obtain final concentration of 1000  $\mu\text{g/ml}$ . 400  $\mu\text{g/ml}$  solution was prepared from the stock solution with distilled water which was used as working standard.

### Analysis of marketed formulations

For the estimation of Rufinamide in tablets formulations, 20 tablets were weighed and triturate to fine powder. Tablet powder equivalent to 50 mg of RFN was weighed and transfer into 50 ml volumetric flask than dissolved in diluent DMSO. It was kept for sonication for 5 min; this was filtered through Whatman filter paper No. 41 and then final dilution was made with diluent to get the final stock solution of 1000  $\mu\text{g/ml}$ . From this stock solution, 400  $\mu\text{g/ml}$  of the sample solution was prepared with distilled water and analysed.

## 3. Results and Discussion

RFN has the zero order absorbance overlay spectra maxima (figure 2) at 262.0 nm. The polynomial regression data for the calibration plots showed good linear relationship in the concentration range of 100-600  $\mu\text{g/ml}$  with correlation coefficient ( $r^2$ ) was found to be higher than 0.999 and the linearity curve was shown in figure 3. Recovery studies International Journal of Medicine and Pharmaceutical Research

guidelines [8, 9]. The validated method was applied to the analysis of tablets containing rufinamide.

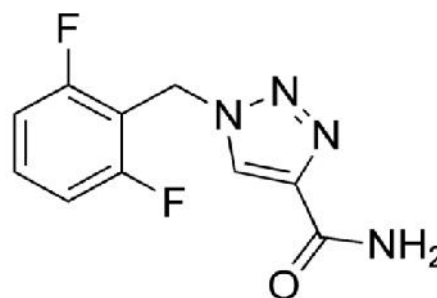


Figure 1: Chemical structure of Rufinamide

### Validation

Method validation was performed in terms of specificity and selectivity, precision and accuracy, linearity, LOD & LOQ.

### Linearity and range

Calibration standards of RFN, covering the range 100-600  $\mu\text{g/ml}$  were prepared with the suitable dilution made from RFN stock solution. The calibration curves were obtained by plotting the intensity of absorbance against of concentration of RFN. The slope and intercept of the calibration line were determined by linear regression using the least squares method.

### Specificity and selectivity

The interference from endogenous compounds was investigated by the analysis of tablets of various concentrations.

### Precision

The intra & inter-day precision was evaluated by analyzing six sample solutions ( $n = 6$ ), at the final concentration of analyses (400  $\mu\text{g/ml}$ ) of RFN. The RFN concentrations were determined and the relative standard deviations (RSD) were calculated.

### Accuracy

RFN reference standards were accurately weighed and added to a mixture of the tablets excipients, at three different concentration levels (200, 400 and 600  $\mu\text{g/ml}$  of rufinamide). At each level, samples were prepared in triplicate and the recovery percentage was determined.

### Detection and quantitation limits

Limit of detection LOD and limit of quantification LOQ were calculated by using the standard deviation from the precision and the slope of linearity.

were carried out at three different levels i.e. 50 %, 100 %, and 150 % by adding the pure drug to the previously analysed tablet powder sample. Percentage recovery for Rufinamide was determined by all the methods and they were found to be under acceptance criteria which are 99.20% to 101.69 % according to ICH guidelines [9]. The

results of accuracy were in table 2. The percentage recovery value indicates noninterference from excipients used in formulation. The precision was carried out as described in method and the results were presented in table 1. The values obtained in the repeatability (precision) shows that there is no significant difference in the precision values; hence the developed method can be used to analyze the

RFN in tablet formulation. The mean assay of the precision value is 99.33%. The LOD determined as the amount drug was found to be 3.44 µg/mL and the LOQ was determined as the lowest concentration was found to be 10.45 µg/mL in formulation. The summary of all the optical characterizes were shown in table 2.

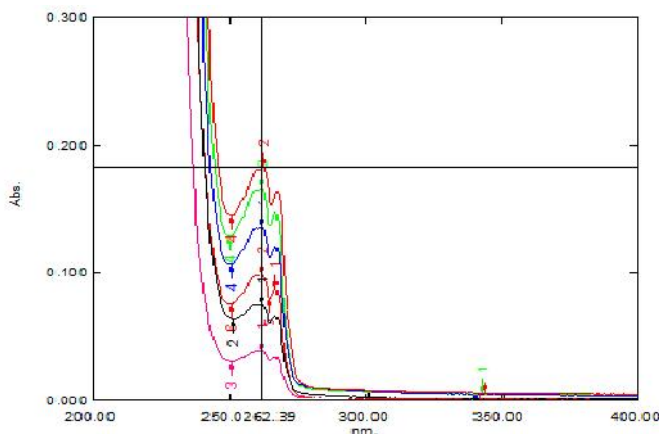


Figure 2: max curve of Rufinamide (Overlay spectra)

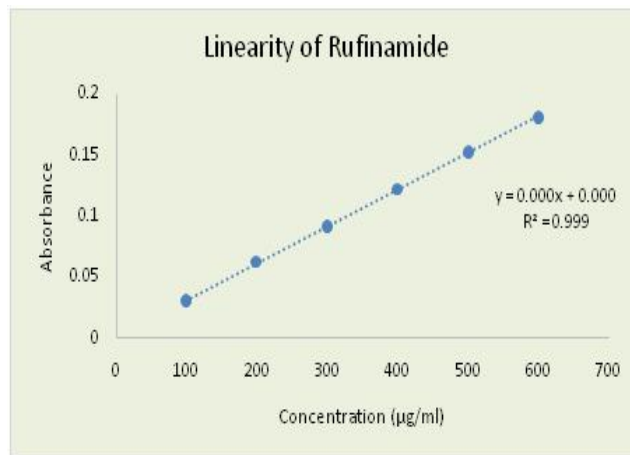


Figure 3: Linearity curve of Rufinamide

Table 1: Results of Precision

Sample No.	Sample Abs - 1	% Assay - 1
1	0.124	99.20
2	0.125	100.00
3	0.124	99.20
4	0.125	100.00
5	0.124	99.20
6	0.123	98.40
Average Assay:		99.33
STD		0.60
<b>% RSD</b>		<b>0.61</b>

Table 2: Summary of Optical characteristics and Other Parameters

S No.	Parameters	Results
1	Absorption Maxima (nm)	262
2	Beer's-Lambert's range (µg/ml)	100-600
3	Regression equation (y)*	Y = 0.0003 C + 0.00075
4	Slope (b)	0.0003
5	Intercept (a)	0.00075
6	Correlation coefficient (r <sup>2</sup> )	0.999
7	Intraday precision (% RSD)**	0.61
8	Interday precision (% RSD)**	0.74
9	Accuracy (% mean recovery)	100
10	Limit of detection (µg / ml)	3.44
11	Limit of quantification (µg / ml)	10.45
12	Assay of tablets (%Purity)	99.33

\*y = a + bx; when x is the concentration in mg/ml and y is absorbance unit, \*\*Average of six determinations.

#### 4. Conclusion

The most striking features of the method was its simplicity and rapidity, non- requiring consuming sample preparations such as extraction of solvents, heating, degassing which are needed for HPLC or other procedures. It can be concluded International Journal of Medicine and Pharmaceutical Research

that the proposed methods was fully validated and found to be simple, sensitive, accurate, precise, reproducible, rugged and robust and relatively inexpensive. So, the developed

method can be easily applied for the routine Quality Control analysis of RFN in pharmaceutical preparations.

## 5. Acknowledgement

We would like thank to Ranbaxy laboratories, Ahmedabad for providing reference sample of RFN to facilitate this work and also to the Principle Dr. Hindustan Abdul Ahad, Balaji College of Pharmacy, Anantapur for providing facilities to carry out this research work.

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