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RP-HPLC method development and validation for rufinamide quantification in tablet dosage forms

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Abstract

For the measurement of Rufinamide in pharmaceutical dosage forms, a straightforward, accurate, quick, and repeatable stability indicating RP-HPLC method was created and verified. A C18 (ODS) acetonitrile: water:triethylamine buffer was used for the chromatography. pH 4.6: The mobile phase was methanol (70:20:10 v/v), and the flow rate was set at 0.8 ml/min. The analytical Tech. UV-Visible detector SpD-10AVP was used to do the detection at 292 nm. In the concentration range of 10–50 ug/ml, the calibration curve that was obtained was linear. It was discovered that the limits of detection and quantification were 3.09 and 1.056 ug/ml, respectively.

Introduction

The antiepileptic medication now on the market is structurally unrelated to rufinamide, a triazole derivative. It is almost completely insoluble in water, very slightly soluble in ethanol and acetonitrile, and marginally soluble in methanol and tetrahydrofuran. 2.
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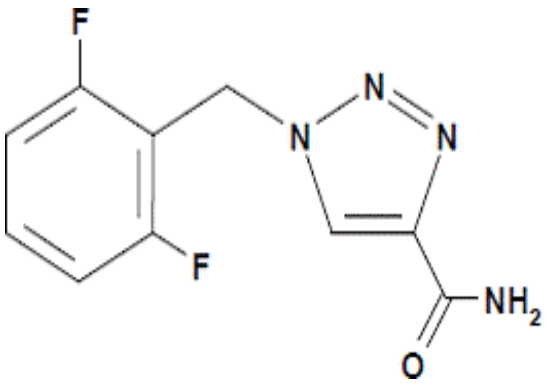


Fig. 1: Chemical Structure of Rufinamide

Numerous techniques for estimating rufinamide in pharmaceutical formulations have been documented, according to a review of the literature. Present study concerns the development of HPLC method3-4 employing simple mobile phase which is sensitive and rapid for quantification of Rufinamide in tablet dosage forms as well as subsequent ICH-guideline-compliant validation of the created approach. Sonication of the sample at ambient temperature treatment with a little amount of powder sample at ambient temperature was one of the method's key features and originality.

Experimental

For the suggested procedure, all of the reagents were HPLC-grade solvents. We bought water and acetonitrile from SD Fine Chemicals in Mumbai, India. We purchased a rufinamide sample from Dr. Reddy's Labs Pvt. in Hyderabad, India. The commercial tablet

formulation of rufinamide (Glenmark Pharma Pvt. Ltd.) was purchased at the neighborhood market. The 400 mg of rufinamide in the tablet dosage forms that were obtained were intended for oral consumption.

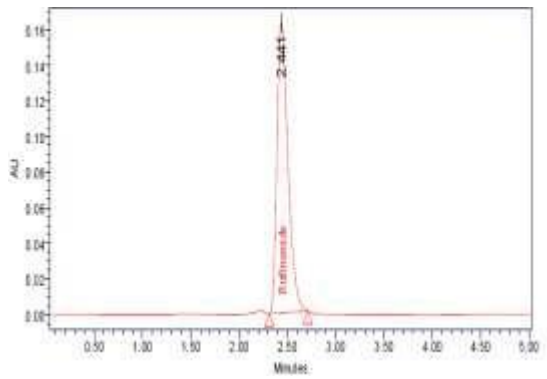
Instrumentation and analytical conditions

For this separation process, Waters-2695 advanced equipment was utilized, with the mobile phase connected to a 2487 quaternary pump. At 27 1 c, the analytical column C18 was utilized. Acetonitrile is present in the mobile phase and temperature: Methanol (70:20:10)% v/v was utilized at a flow rate of 1 milliliter per minute in triethylamine buffer pH 4.6. Before being used, the freshly made diluents were sonicated for five minutes to degas them. Rufinamide's highest absorbance was discovered at 243 nm.

Stock solutions

A fresh standard stock solution of 1000 ug/ml of Rufinamide was made by precisely weighing 25 mg of Rufinamide into a 25 ml volumetric flask. Five working standards with concentrations ranging from 10 to 50 were obtained by further diluting the aforementioned solution with mobile phase in a 10-milliliter volumetric flask. of Rufinamide (ug/ml). Every solution was made in triplicate. The six concentrations of the 10–50 ug/ml working standard solutions were used to plot the calibration curve.

Tablet formulation assay



Twenty commercial tablets containing 400 mg of Rufinamide were weighed, and the mean mass of the pills was calculated. A precisely weighed amount of the tablet powder, equal to 25 mg of Rufinamide, was quantitatively transferred into a 25 ml volumetric flask along with roughly 20 ml of phosphate buffer pH 4.0 after the tablets had been ground into a fine powder in a glass mortar. A 1.5 ml aliquot was then transferred into a 10 ml volumetric flask. Following dilution, the predicted concentration of rufinamide was Procedure for validation

According to ICH guidelines8, the validation of the suggested method showed that it is appropriate for its quality control dosage forms. To test for linearity, standard plots were made using six concentrations of rufinamide produced in triplicate, ranging from 10 to 50 ug/ml. The calibration graph was created by plotting the peak area of Rufinamide against the concentration. Six duplicate injections of newly made Rufinamide test solution in the same apparatus at a concentration of 100% of the desired test concentration value on the same day were used to calculate repeatability. Three preparations are used for each of the three levels of rufinamide analysis in pure solutions. The percentage of rufinamide recovered in the samples was used to express the results.

Findings and Conversation

Optimization

Choosing the wavelength for detection: Rufinamide's UV spectra in acetonitrile: Triethylamine buffer pH4.6: Acetonitrile and phosphate buffer combinations with methanol (70:20:10 v/v) in the 200–400 nm range. It demonstrates that Rufinamide has its greatest absorbance at 292 nm. Therefore, the maximum amount of Rufinamide in the mobile phase was chosen as the ideal detection wavelength for Rufinamide quantification.

Conditions for Chromatography Optimal Approach Specifications: Ideal Situations:

Phase ratio of mobile devices: Triethylamine buffer in acetonitrile Methanol at pH 4.6 (70:20:10)

Column: 4.6 x 250 mm ODS C18 5µ Column temperature: 27ºC
Wavelength: 243 nanometers
Rate of flow: 0.8 ml/min Volume of injection: 10µl Run time: 5 minutes

Fig. 2: Rufinamide Optimization of chromatogram

Validation of methods

Range and Linearity: Three independent measurements were made at each concentration, and six point calibration graphs encompassing a concentration range of 10–50 ug/ml were created. Figure 3 illustrates the linear associations that were found between the Rufinamide peak area signal and the appropriate medication concentration. The linearity
values was (r^2) 0.9999 shown below

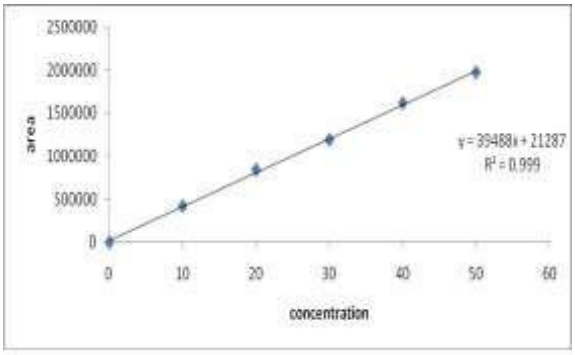


Fig. 3: Calibration Graph of Rufinamide

Precision

The analysis of commercial tablets containing 4 mg of Rufinamide was conducted using the established method. After the drug was extracted, the sample was examined six times using a similar process and the experimental section's assay sample preparation to demonstrate that the amount of rufinamide in tablet formulations matched the label claim's content requirements (95–105%). A research conducted by the same analyst on the same day and on three consecutive days (n 3) revealed an R.S.D. of 0.0455 and 0.03995% for the intermediate precision, respectively.

Accuracy

The percentage recoveries of Rufinamide in the actual samples were used to express the accuracy data. (Table 4) In the actual sample, the average recovery of Rufinamide fell between 99.60 and100.05%. Mean % R.S.D. was 0.6069%, satisfying the acceptance criteria for the study.

Table 1: Accuracy study for Rufinamide (n 9)

S. No	Percentage	% recovery	Mean ± S.D	% RSD	S.E
1	50%	99.60	99.05± 0.6211	0.72 69	0.4196
2	100%	100.05			
3	150%	99.00			

* Mean of three observations

System suitability

All system suitability parameters were represented below Table 2.

Table 2: Summary for RP-HPLC Method

		Results obtained

Parameter	Acceptance criteria	For Rufinamide
System suitability	Theoretical Plates- NLT 2000	5342
	Tailing factor- NMT 2	1.09
	Retention time	2.41
Precision	%RSD- NLT 2	0.77
Linearity	Correlation Coefficient	0.9997
Accuracy	% Recovery	100.34%
Limit of detection		1.32µg/ml
LOQ		3.02µg/ml

The proposed method accurate, reproducibility and validation parameters for different parameters and let us to the conclusion that it could be used for the rapid and reliable determination of Rufinamide in tablet formulation.

Forced Degradation Studies

Sample Preparations to Perform Forced Degradation Studies

Acid degradation: Accurately weighed 10mg of equivalent weight of Rufinamide sample into a 10ml clean dry volumetric flaskand added about 3mL of 0.1N Hcl

Further pipetted 0.3 ml of above stock solution into a 10ml volumetric flask (it contains Rufinamide) and diluted up to the mark with diluent.

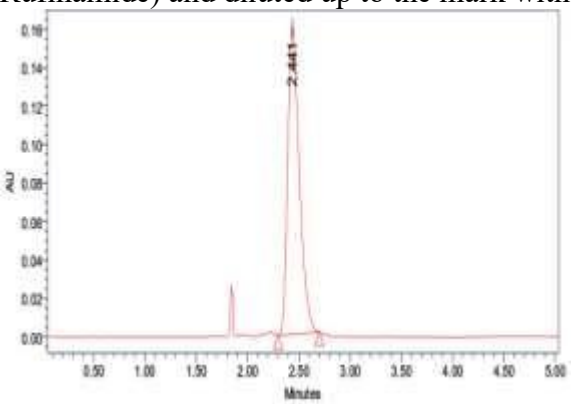


Fig. 4: Showing acid degradation for Rufinamide

Alkaline degradation

Accurately weighed 10 mg equivalent weight of Rufinamide sample into a 10mL clean dry volumetric flask and added about 1 mL of 0.1N NaOH.

Further pipetted 0.3ml of above stock solution into a 10ml volumetric flask (it contains Rufinamide) and diluted up to the mark with diluent.

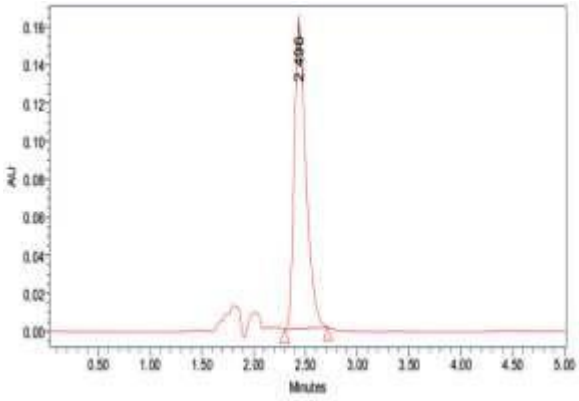


Fig. 5: Showing Alkaline degradation for Rufinamide

Peroxide degradation

Accurately weighed 10 mg equivalent weight of Rufinamide sample into a 10mL clean dry volumetric flask and added about 3mL of Hydrogen peroxide solution and kept side for 3hours and made the volume up to mark by using Diluent and sonicated to dissolve it completely.

Further pipetted 0.3ml of above stock solution into a 10ml volumetric flask (it contains Rufinamide) and diluted up to the mark with diluents Fig. 5.

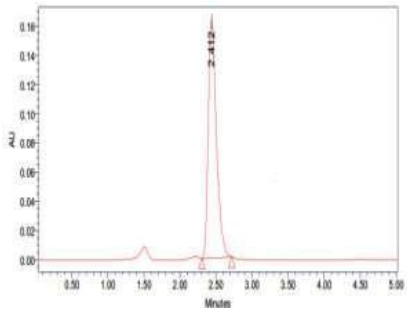


Fig. 5: Showing peroxide degradation for Rufinamide

Thermal degradation

Accurately weighed 10 mg equivalent weight of Rufinamide sample into a 10mL clean dry volumetric flask and exposed to heat at 80-90ºc for 3hours and then made the volume up to mark by using Diluent and sonicated to dissolve it completely.

Further pipetted 0.3ml of above stock solution into a 10ml volumetric flask (it contains Rufinamide) and diluted up to the mark with diluent. Fig. 6.

Photolytic degradation

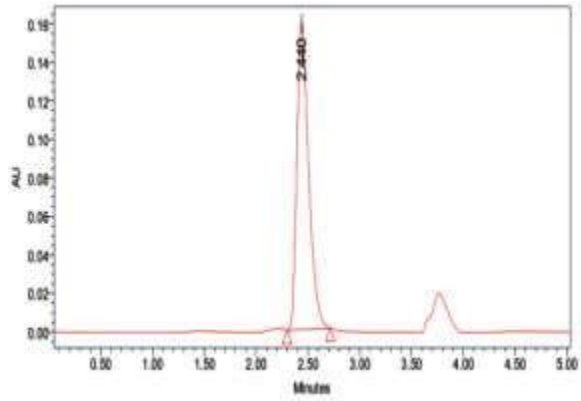


Fig. 6: Showing Thermal degradation for Rufinamide

Accurately weighed 10 mg equivalent weight of Rufinamide sample into a 10mL clean dry volumetric flask and exposed to sunlight for 3hours and made the volume up to mark by using Diluent and sonicated to dissolve it completely.

Further pipetted 0.3ml of above stock solution into a 10ml volumetric flask (it contains Rufinamide) and diluted up to the mark with diluent. Fig. 7

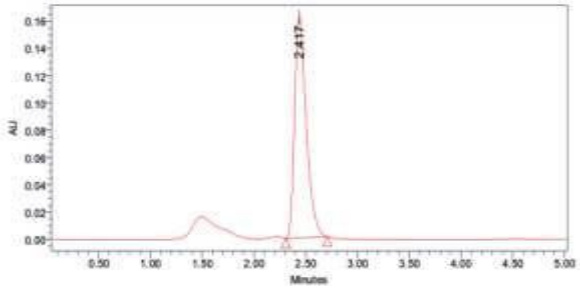


Fig. 7: Showing Photolytic degradation for Rufinamide

Table 3: Results for degradation studies

S. No	Type of degradation	Concentration of sample (µg/ml)	Area of sample	Assay content (% w/w)
1	Acid (0.1N HCl)	30 µg/ml	1206721	100%
2	Base (0.1N NaOH)	30 µg/ml	1214352	100%
3	Peroxide (3% H_2O_2)	30 µg/ml	1145269	94%
4	Thermal (at 60^0 c)	30 µg/ml	1195615	99%
5	Photolytic (sunlight)	30 µg/ml	1185917	98%

Summary and Conclusion

RP-HPLC method was developed for estimation of Rufinamide in bulk and Pharmaceutical dosage form. The separation was achieved on ODS C18 (4.6×250mm) 5µ containing the mobile phase mixture of Acetonitrile: Triethylamine buffer pH4.6: Methanol (70:20:10).

Conclusion

The suggested method for estimating rufinamide in bulk and in its pharmaceutical dosage form proved precise, quick, cost-effective, and specific. The method was determined to be accurate, the percentage RSD values were within 2, and the sample recoveries in each formulation were in good agreement with their individual Label Claims. Rufinamide may be routinely determined with this technology in both pharmaceutical dose forms and bulk.

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