

Research Article

ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF HPTLC METHOD FOR SIMULTANEOUS ESTIMATION OF SUMATRIPTAN SUCCINATE AND NAPROXEN SODIUM IN PHARMACEUTICAL DOSAGE FORM**Smruti P. Parekh^{1*}, Zarna R. Dedania², Ronak Dedania², S. M. Vijendraswamy²**¹M. Pharm Student, ²Assistant professor, Bhagwan Mahavir College of Pharmacy, Bharthana, Surat, Gujarat, India.

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ABSTRACT

The HPTLC method is based on separation of the two drugs followed by the densitometric measurements of their spots at 277 nm. The separation was carried out on Silica gel 60GF254 using mobile phase Ethylacetate: Methanol: Chloroform: Glacial acetic acid (60:20:19:1 v/v/v/v). The linearity range lies between 250-1500 ng/spot for Sumatriptan Succinate and 1000-6000 ng/spot for Naproxen Sodium with correlation coefficients of 0.997 and 0.996 respectively. The R_f value for Sumatriptan Succinate is 0.49 ± 0.02 and for Naproxen Sodium is 0.28 ± 0.02 . Recoveries of Sumatriptan Succinate and Naproxen Sodium was in the range of 99.54-100.50% and 99.75-100.07% respectively. LOD value for Sumatriptan Succinate was 39.85 ng/spot and for Naproxen Sodium was 80.35 ng/spot. LOQ value for Sumatriptan Succinate was 120.77 ng/spot and for Naproxen Sodium was 243.5 ng/spot. The developed method was validated as per ICH Guidelines.

KEYWORDS: Sumatriptan Succinate, Naproxen Sodium, HPTLC, Validation.**INTRODUCTION**

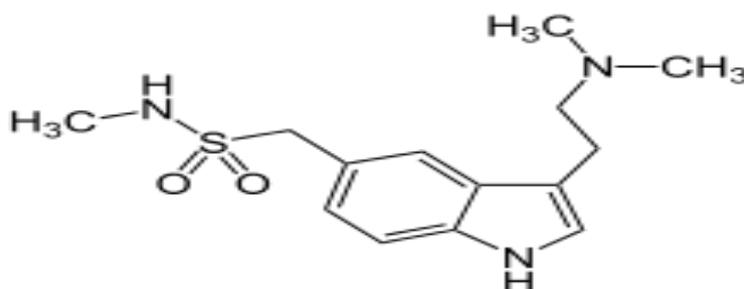
Sumatriptan Succinate^[1-5] is a synthetic drug belonging to the triptan class and 1-[3-(2-dimethylaminoethyl)-1*H*-indol-5-yl]-*N*-methylmethanesulfonamide. Selective 5-hydroxytryptamine₁ (5-HT₁) receptor subtype agonist act as antimigraine. Naproxen Sodium^[1-5] is a nonsteroidal anti-inflammatory drug (NSAID) and (2*S*)-2-(6-methoxynaphthalen-2-yl) propanoic acid. It is arylacetic acid group act as pain relief than used as antimigraine. Most of antimigraine drugs are not available in combined dosage form.

Spectroscopical method and chromatographical method like HPLC, HPTLC^{[6-}

8] are considered to be most suitable for the simultaneous estimation of drug present in a multi component dosage form.

The present paper describes a simple, accurate and precise method for simultaneous estimation of Sumatriptan Succinate and Naproxen Sodium in combined pharmaceutical dosage form. HPTLC method for Sumatriptan Succinate is published^[9]. Some article on HPLC for Sumatriptan Succinate^[10-12] and Naproxen Sodium^[13,14] were published.

The proposed method is optimized and validated as per the International Conference on Harmonization (ICH) guidelines^[15].

**Figure 1: Sumatriptan Succinate**

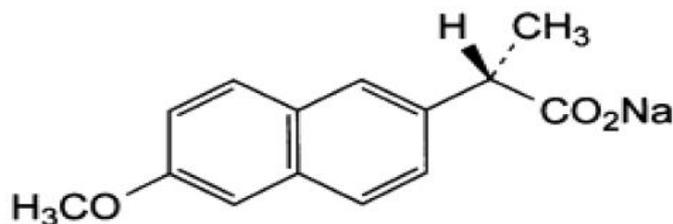


Figure 2: Naproxen Sodium

MATERIALS AND METHODS

Chemicals and reagents

Sumatriptan Succinate and Naproxen Sodium working standards were procured from Sun Pharmaceutical and Divi's Laboratory respectively, and the tested pharmaceutical formulations (Headset Sumatriptan Succinate (119mg) and Naproxen Sodium (500 mg) tablet) were procured from commercial pharmacy. All reagents used were of suitable analytical grade.

Chromatographic Conditions

The instrument used for the estimation was CamagLinomat V semi automatic sample applicator, Camag TLC scanner III, WinCATs software for interpretation of the data, Hamilton syringe and Camag twin trough chamber. The Mobile phase was Ethylacetate: methanol: chloroform: Glacial acetic acid (60:20:19:1). Chamber saturation time was 20min. Detection was performed at 277nm.

Preparation of Standard Solution

250 mg of Sumatriptan Succinate and 500 mg of Naproxen Sodium were dissolved and diluted with methanol upto 100ml. (2500 μ g/ml of Sumatriptan Succinate and 5000 μ g/ml of Naproxen Sodium).

Preparation of Calibration curve

The working standard stock solution containing the mixture in the ratio of (1:4) of Sumatriptan Succinate and Naproxen Sodium. From 250 ng/ μ l Sumatriptan Succinate solution applied 1, 2, 3, 4, 5 and 6 ng/ μ l to the plate for the calibration curve were obtained 250-1500 ng/spot of Sumatriptan Succinate. From 1000 ng/ μ l Naproxen Sodium solution applied 1, 2, 3, 4, 5 and 6 ng/ μ l to the plate for the calibration curve were obtained 1000-6000 ng/spot of Naproxen Sodium.

Preparation of Sample Solution

20 tablets (Headset) were weighed and powdered. Powder equivalent to 23.8 mg of Sumatriptan Succinate and 100 mg of Naproxen Sodium transferred into 100ml volumetric flask. Methanol was added to adjust level up to mark and sonicated for 10 min. The solution was filtered through whatman filter paper no. 42. First few ml of filtrate was discarded. 1 ml of this solution diluted for 10ml to give 238 ng/ μ l Sumatriptan Succinate and 1000 ng/ μ l Naproxen Sodium. 1 μ l of this solution is used for the estimation.

RESULTS AND DISCUSSION

The present study was aimed at development of speedy and cost effective HPTLC technique for determination of Sumatriptan Succinate and Naproxen Sodium in pharmaceutical dosage forms.

Various blends of solvent systems in varying proportions were tried as mobile phase. However, mobile phase consisting ethyl acetate: methanol: chloroform: glacial acetic acid (60:20:19:1) was found to be more suitable with R_f values of 0.49 ± 0.02 and 0.28 ± 0.02 for Sumatriptan Succinate and Naproxen Sodium, respectively with saturation time of 20 minutes. The selection of wave length was based on maximum absorbance for optimum sensitivity. The drugs showed good linearity in the range of 250-1500 ng/spot for Sumatriptan Succinate and 1000-6000 ng/spot for Naproxen Sodium with coefficient of correlation value 0.997 and 0.996, respectively. From the recovery studies, the accuracy results were 99.54-100.50% for Sumatriptan Succinate and 99.75-100.07% for Naproxen Sodium and were found to be highly accurate.

Validation of the Method

1. Linearity and Range: Linearity was found in the range of 250-1500 ng/spot for Sumatriptan

Succinate and 1000-6000 ng/spot for Naproxen Sodium. The drug peak area was calculated for each concentration level and a graph was

plotted of drug concentration against the peak area.

Table 1: Calibration parameters

Parameters	SUM	NAP
Linearity range (ng/spot)	250-1500	1000-6000
Linearity equation	$y = 5.962x + 3683$	$y = 0.767x + 1244$
Co-relation coefficient	0.997	0.996
Slope	5.962	0.767
Intercept	3683	1244

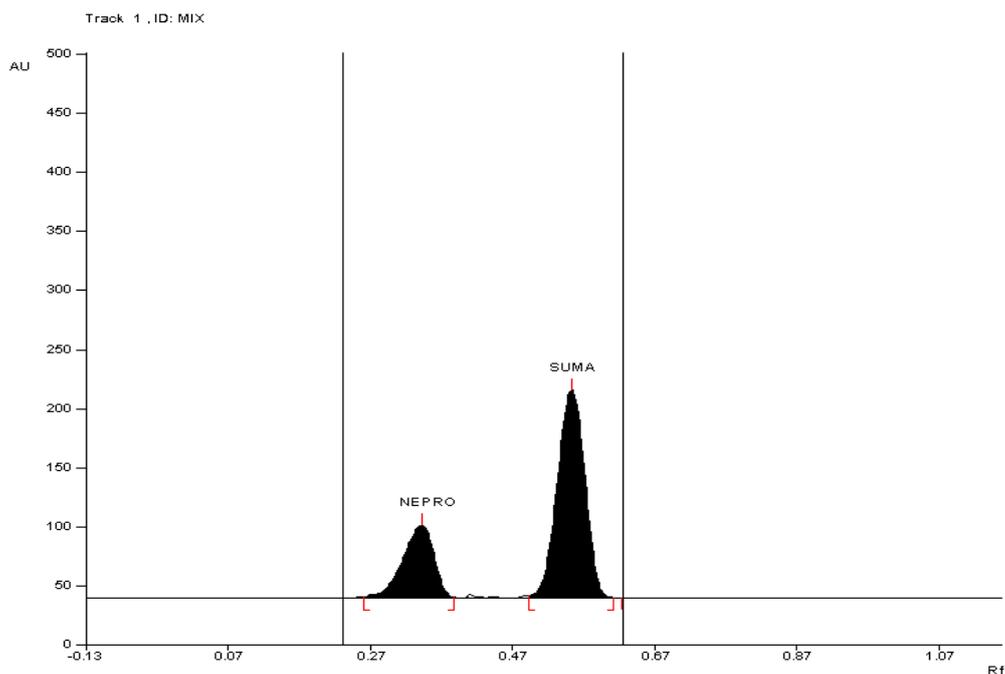


Figure 3: Chromatogram of mixed standard solution containing 250 ng/spot of Sumatriptan Succinate and 1000 ng/spot of Naproxen Sodium

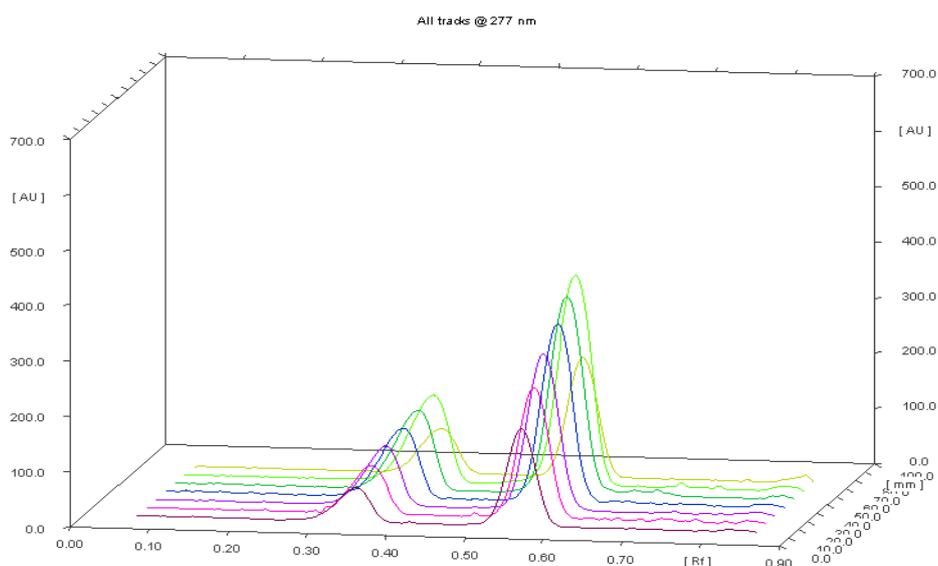


Figure 4: 3D view of all tracks of Sumatriptan Succinate and Naproxen Sodium

2. Precision: The precision expressed as standard deviation or relative standard deviation. Combined dosage form was analyzed at three levels of concentration of the assay for three times in a day. Peak Area of the solutions was measured.

2.1 The data for Repeatability % R.S.D. was found to be 0.2209% for Sumatriptan Succinate and 0.2911% for Naproxen Sodium.

2.2 The data for intraday % R.S.D. was found to be 0.5099% for Sumatriptan Succinate and 0.6960% for Naproxen Sodium.

2.3 The data for interday % R.S.D. was found to be 0.5661% for Sumatriptan Succinate and 0.8989% for Naproxen Sodium.

3. Specificity: Specificity is carried out by taking peak purity of standard and sample of each drug and standard and sample peak spectra were overlain to check specificity of each individual drug peak.

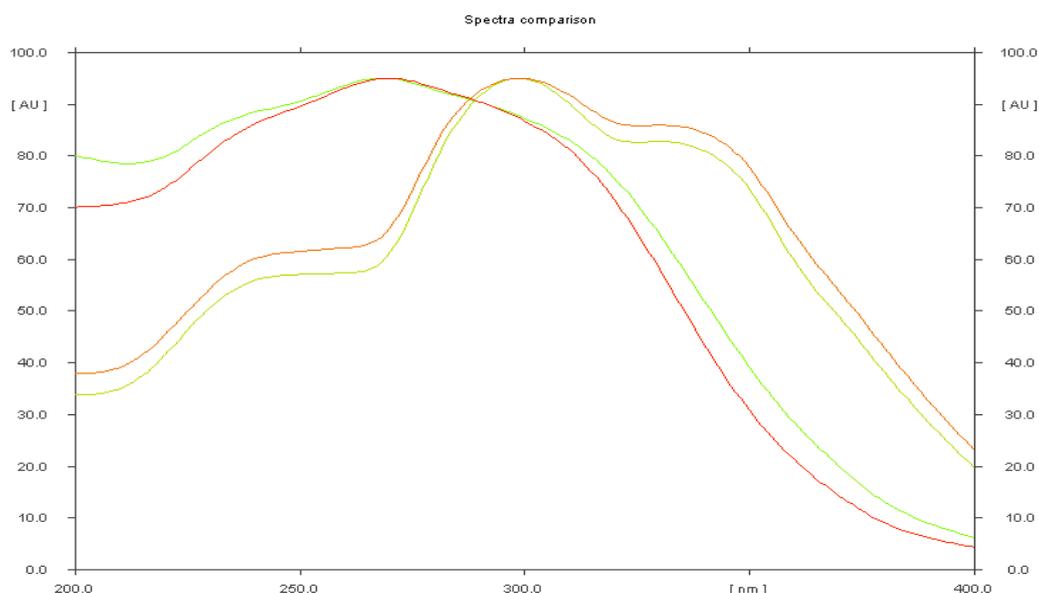


Figure 5: Peak purity graph of Sumatriptan Succinate and Naproxen Sodium

Table 2: Specificity data

Drug	Co-relation r (s,m)	Co-relation r (m,e)	Peak purity
SUM	0.999564	0.998809	Pass
NAP	0.999490	0.998730	Pass

4. Accuracy (Recovery study): The accuracy of the method was established using recovery technique i.e external standard addition method. The known amount of standard was

added at three different levels to preanalysed sample. Each determination was performed in triplicate.

Table 3: Accuracy Results

Sr. No.	Assay level	Tablet content taken (ng/spot)		Standard added (ng/spot)		Total drug recovered (ng/spot)		% Recovery \pm S.D (n=3)	
		SUM	NAP	SUM	NAP	SUM	NAP	SUM	NAP
1		500	2000	-	-	499.23	2000.4	-	-
2	80%	500	2000	250	1000	750.98	3001.6	100.50 \pm 0.46	100.17 \pm 0.5
3	100%	500	2000	500	2000	1000.9	4000.7	100.25 \pm 0.25	100.04 \pm 0.4
4	120%	500	2000	750	3000	1250.4	4999.7	100.1 \pm 0.23	99.99 \pm 0.25

Table 4: Assay result of marketed formulation by HPTLC method

Formulation	Tablet content taken eq. to (ng/spot)		Amount found (ng/spot)		Assay (%estimated) (n±3)	
	SUM	NAP	SUM	NAP	SUM	NAP
Headset (Label claim SUM=119 mg & NAP=500 mg)	119	500	118.20	501.49	99.32±0.56	100.3±0.92

5. LOD

LOD was found to be 39.85 ng/spot for Sumatriptan Succinate and 80.35 ng/spot for Naproxen Sodium.

6. LOQ

LOD was found to be 120.77 ng/spot for Sumatriptan Succinate and 243.5 ng/spot for Naproxen Sodium.

CONCLUSION

Sumatriptan Succinate and Naproxen Sodium show maximum UV absorption at 277 nm. Hence, an appropriate method of estimation when these drugs are administered together is chromatographic analysis. HPTLC determination of Sumatriptan Succinate and Naproxen Sodium shows no interference between two drugs and from the excipients it also shows the method is rapid, allowing a high sample throughput necessary for routine analysis with an added advantage of low solvent consumption. The method described herein is simple, rapid, selective method and well suited for quantitative estimation of Sumatriptan Succinate and Naproxen Sodium individually and from pharmaceutical preparations.

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