ANALYTICAL METHOD DEVELOPMENT AND VALIDATION OF FIRST ORDER DERIVATIVE DETERMINATION METHOD FOR SIMULTANEOUS ESTIMATION OF SUMATRIPTAN SUCCINATE AND NAPROXEN SODIUM IN PHARMACEUTICAL DOSAGE FORM

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ABSTRACT

Simple, Precise and Accurate methods are described for the direct determination of Sumatriptan Succinate and Naproxen Sodium in combined dosage form without prior separation. The UV Spectrophotometric (First order derivative) method selected wavelength were 226.50 nm and 230 nm for determination of Sumatriptan Succinate and Naproxen Sodium respectively. Linearity range lies between 0.5-2.5 μg/ml for Sumatriptan Succinate and 2-10 μg/ml for Naproxen Sodium. Correlation coefficients of Sumatriptan Succinate and Naproxen Sodium were 0.998 and 0.997 respectively. %Recoveries of Sumatriptan Succinate and Naproxen Sodium was in the range of 98.05-102.63% and 99.34-102.86% respectively. LOD value for Sumatriptan Succinate was 0.09 μg/ml and for Naproxen Sodium was 0.97 μg/ml. LOQ value for Sumatriptan Succinate was 0.28 μg/ml and for Naproxen Sodium was 1.98 μg/ml. The developed method was validated as per ICH Guidelines.

KEYWORDS: Sumatriptan Succinate, Naproxen Sodium, UV Spectrophotometric method, Validation.

INTRODUCTION

Sumatriptan Succinate is a synthetic drug belonging to the triptan class and 1-[3-(2-dimethylaminoethyl)-1H-indol-5-yl]-N-methyl-methanesulfonamide. Selective 5-hydroxytryptamine1 (5-HT1) receptor subtype agonist act as antimigraine. Naproxen Sodium is a nonsteroidal anti-inflammatory drug (NSAID) and (2S)-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 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. Some
published methods for Sumatriptan Succinate like UV-visible spectrophotometer\textsuperscript{6,7}, HPTLC\textsuperscript{8}, HPLC\textsuperscript{9,10,11} were obtained same as method for Naproxen Sodium like UV-visible spectrophotometer\textsuperscript{12,13,14}, HPLC\textsuperscript{15,16} also present. The proposed method is optimized and validated as per the International Conference on Harmonization (ICH) guidelines\textsuperscript{17}.  

\textbf{MATERIALS AND METHODS}

\textbf{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.

\textbf{Spectrophotometric Conditions:}
Double beam UV-visible spectrophotometer (Shimadzu-1800) having two matched quartz cells with 1 cm light path loaded with UV probe 2.32 software. In Spectrum mode, Scan speed is Medium at Wavelength range in 400-200 nm Absorbance scale 0.00A – 4.00A at 226.50 and 230 nm as distilled water as blank.

\textbf{Preparation of Standard Solution:}
Accurately weighed quantity of Sumatriptan Succinate and Naproxen Sodium 50 mg was transferred into separate 50 ml volumetric flask, dissolved in distilled water. The flask was shaken and the volume was made up to the mark with distilled water. 1 ml of this solution
diluted up to 10 ml i.e. 100 μg/ml of each solution was prepared by diluting 10 ml of standard stock solution to 100 ml with distilled water. Further 5 ml was transferred in 50 ml volumetric flask and distilled water was added up to the mark to give a solution containing 10 μg/ml.

**Preparation of Calibration curve:**
Appropriate volume of aliquots from Sumatriptan Succinate working standard solution (10 μg/ml) were transferred to different volumetric flask of 10 ml capacity. The volume with adjust to mark with distilled water to obtain the concentration 0.5, 1, 1.5, 2 and 2.5 μg/ml Sumatriptan Succinate. The volume with adjust to mark with distilled water to obtain the concentration 2, 4, 6, 8 and 10 μg/ml 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. Distilled water was added to adjust level up to mark and sonicated for 15 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/µl Sumatriptan Succinate and 1000 ng/µl Naproxen Sodium. 1 µl of this solution is used for the estimation. From this solution 1 ml was transferred to volumetric flask of 10 ml capacity and adjust the mark with distilled water. From this solution 0.6 ml was transferred to volumetric flask of 10 ml capacity. Volume was made up to the mark to a give solution contain 1.4 μg/ml of Sumatriptan Succinate and 6 μg/ml of Naproxen Sodium.

**RESULTS AND DISCUSSION**
The present study was aimed at development of speedy and cost effective UV spectrophotometric (First order derivative) technique for determination of Sumatriptan Succinate and Naproxen Sodium in pharmaceutical dosage forms.

**Validation of the Method:**
1. **Linearity and Range:** Linearly was found in the range of 0.5-2.5 μg/ml for Sumatriptan Succinate and 2-10 μg/ml for Naproxen Sodium. The drug peak area was calculated for each concentration level and a graph was plotted of drug concentration against absorbance.

<table>
<thead>
<tr>
<th>Parameters</th>
<th>SUM</th>
<th>NAP</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linearity range (μg/ml)</td>
<td>0.5-2.5</td>
<td>2-10</td>
</tr>
<tr>
<td>Linearity equation</td>
<td>0.031x + 0.043</td>
<td>0.044x + 0.600</td>
</tr>
<tr>
<td>Co-relation coefficient</td>
<td>0.998</td>
<td>0.997</td>
</tr>
<tr>
<td>Slope</td>
<td>0.031</td>
<td>0.044</td>
</tr>
<tr>
<td>Intercept</td>
<td>0.043</td>
<td>0.600</td>
</tr>
</tbody>
</table>

- amplitude at this wavelength in mixture.
Fig 1: Overlain 1\textsuperscript{st} order derivative UV spectra of Sumatriptan Succinate (0.5-2.5 μg/ml)

Fig 2: Overlain 1\textsuperscript{st} order derivative UV spectra of Naproxen Sodium (2-10 μg/ml)

Fig 3: Overlain 1\textsuperscript{st} order derivative UV spectra of Sumatriptan Succinate (2.5 μg/ml) and Naproxen Sodium (10 μg/ml)
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 0.1175% for Sumatriptan Succinate and 0.2456% for Naproxen Sodium.

2.2 The data for intraday % R.S.D. was found to be 0.2769% for Sumatriptan Succinate and 0.2536% for Naproxen Sodium.

2.3 The data for interday % R.S.D was found to be 0.6550% for Sumatriptan Succinate and 0.7105% for Naproxen Sodium.

3. 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>
<thead>
<tr>
<th>Sr. No</th>
<th>Assay level</th>
<th>Tablet content taken (µg/ml)</th>
<th>Standard added (µg/ml)</th>
<th>Total drug recovered (µg/ml)</th>
<th>% Recovery ±S.D (n=3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>SUM NAP</td>
<td>11.9 50</td>
<td>SUM 49.56</td>
<td>21.45 90.34</td>
<td>101.63±1.22 101.2±1.50</td>
</tr>
<tr>
<td>2.</td>
<td>80% SUM NAP</td>
<td>11.9 50 9.5 40</td>
<td>23.89 100.56</td>
<td>101.43±1.65 101.2±1.56</td>
<td></td>
</tr>
<tr>
<td>3.</td>
<td>100% SUM NAP</td>
<td>11.9 50 11.9 50</td>
<td>26.19 110.56</td>
<td>100.04±1.10 101.83±0.53</td>
<td></td>
</tr>
<tr>
<td>4.</td>
<td>120% SUM NAP</td>
<td>11.9 50 14.2 60</td>
<td>29.19 117.56</td>
<td>100.3±1.20 101.8±0.53</td>
<td></td>
</tr>
</tbody>
</table>

Table 3: Assay result of marketed formulation by UV spectrophotometric (First order derivative) method

<table>
<thead>
<tr>
<th>Formulation</th>
<th>Tablet content taken eq. to (ng/spot)</th>
<th>Amount found (ng/spot)</th>
<th>Assay (%estimated) (n±3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Headset</td>
<td>SUM 119 500</td>
<td>SUM 118.20 501.49</td>
<td>SUM 99.32±0.56 100.3±0.92</td>
</tr>
</tbody>
</table>

4. LOD:
LOD was found to be 0.09 µg/ml for Sumatriptan Succinate and 0.97 µg/ml for Naproxen Sodium.
5. LOQ:
LOQ was found to be 0.28 µg/ml for Sumatriptan Succinate and 1.98 µg/ml for Naproxen Sodium.

CONCLUSION
Sumatriptan Succinate and Naproxen Sodium show maximum UV absorption at 226.50 and 230 nm. Hence, an appropriate method of estimation when these drugs are administered together is UV-visible spectrophotometer analysis. UV-visible spectrophotometer (First order derivative) 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.

ACKNOWLEDGEMENT
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REFERENCES
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