**ANALYTICAL METHOD DEVELOPMENT AND VALIDATION FOR SIMULTANEOUS DETERMINATION OF SUMATRIPTAN AND NAPROXEN BY RP-HPLC**

**TABLES AND FIGURES:**

**Table -1**

**Linearity:-**

**Linearity data of Naproxen**

|  |  |  |  |
| --- | --- | --- | --- |
| **S.No** | **Linearity Level** | **Concentration** | **Area** |
| 1 | I | 60ppm | 1189032 |
| 2 | II | 70ppm | 1497165 |
| 3 | III | 80ppm |  1851004 |
| 4 | IV | 90ppm | 2186380 |
| 5 | V | 100ppm | 2548658 |
| Correlation Coefficient | 0.999 |

**Table -2**

**Linearity data of Sumatriptan:-**

|  |  |  |  |
| --- | --- | --- | --- |
| **S.No** | **Linearity Level** | **Concentration** | **Area** |
| 1 | I | 60ppm | 1219297 |
| 2 | II | 70ppm |  1531332 |
| 3 | III | 80ppm |  1856868 |
| 4 | IV | 90ppm | 2252060 |
| 5 | V |  100ppm | 2560402 |
| Correlation Coefficient | 0.999 |

|  |  |  |
| --- | --- | --- |
| **Parmeters** | **Results observed****Sumatriptan** | **Results observed****Naproxen** |
| Slope | 34029.38 | 34898.55 |
| Intercept | -838359 | -945575 |
| Correlation | 0.999168 | 0.999901 |

**Table -3**

Linearity parameters

**Table -4**

**Assay:-Sumatriptan and Naproxen**

|  |  |  |
| --- | --- | --- |
| **S.no** | **Naproxen** | **Sumatriptan** |
| **Standard Rt** | **Sample Rt** | **Standard area** | **Sample area** | **Standard Rt** | **Sample Rt** | **Standard area** | **Sample area** |
| 01 | 2.248 | 2.24 | 1523510 | 1521502 | 5.871 | 5.865 | 1566643 | 1561262 |
| 02 | 2.249 | 2.250 | 1531670 | 1529265 | 5.875 | 5.863 | 1573899 | 1564536 |
| MEAN |  |  | 1532594 | 1525384 |  |  | 1570271 | 1562899 |
| STD |  |  | 1527590 | 5488.8 |  |  | 1570271 | 2315.2 |
| %RSD |  |  | 0.377 | 0.36 |  |  | 0.327 | 0.15 |

**Table -5**

**Accuracy:-**

Accuracy data of Sumatriptan

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **%Concentration****(at specification Level)** | **Area** | **Amount Added****(mg)** | **Amount Found****(mg)** | **% Recovery** | **Mean Recovery** |
| 50% | 984243 | 6.15 | 6.23 | 101.4% | 101.0% |
| 100% | 1567396 | 10.0 | 9.98 | 99.8 |
| 150% | 2497228 | 15.5 | 15.79 | 101.9% |

**Table -6**

**Accuracy data of Naproxen:-**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **%Concentration****(at specification Level)** | **Area** | **Amount Added****(mg)** | **Amount Found****(mg)** | **% Recovery** | **Mean Recovery** |
| 50% | 958196 | 6.15 | 6.25 | 101.7% | 101.0% |
| 100% | 1532695 | 10.0 | 9.96 |  99.5%  |
| 150% | 2425792 | 15.6 | 15.8 | 101.7% |

**Table -7**

**Precision:**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **SI.No** | **Injection number (80 mcg/ml)** | **Retention Time of****Naproxen** | **Retention Time of****Sumatriptan** | **Area of****Naproxen** | **Area of****Sumatriptan** |
| 1 | Injection-1 | 2.412 | 6.374 | 1549491 | 1579612 |
| 2 | Injection-2 | 2.259 | 5.900 | 1530248 | 1574521 |
| 3 | Injection-3 | 2.259 | 5.884 | 1530713 | 1566564 |
| 4 | Injection-4 | 2.261 | 5.863 | 1527834 | 1570849 |
| 5 | Injection-5 | 2.257 | 5.857 | 1537667 | 1567180 |
|  | AVG |  |  | 1535191 | 1571745 |
|  | STD |  |  | 8792.8 | 5433.0 |
|  | %RSD |  |  | 0.57 | 0.35 |

Precision of Sumatriptan and Naproxen

**Table -8**

**Robustness:-**

Robustness of Sumatriptan

|  |  |  |
| --- | --- | --- |
| **Proposed variations** | **USP Plate Count** | **USP Tailing** |
| **Variation in mobile phase composition** |  10% less | 2396.9 |  1.8 |
| \*Actual | 2183.4 |  1.6 |
| 10% more |  2218.9 | 1.5 |
| **Variation in flow rate** | **0.6ml/min** | 2228.3 |  1.6 |
| **0.8ml/min** | 2183.4 | 1.6 |
| **1.0ml/min** |  2142.7 | 1.6 |

**Table -9**

 Robustness of Naproxen

|  |  |  |
| --- | --- | --- |
| **Proposed variations** | **USP Plate Count** | **USP Tailing** |
| **Variation in mobile phase composition** |  10% less | 2963.1 | 1.2 |
| \*Actual | 2203.7 |  1.3 |
| 10% more |  2268.8 |  1.3 |
| **Variation in flow rate** | **0.6ml/min** | 2519.4 |  1.3 |
| **0.8ml/min** | 2203.7 | 1.3 |
| **1.0ml/min** |  2474.4 |  1.3 |

**Table -10**

**Ruggedness:**

Ruggedness of Sumatriptan and Naproxen

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
|  | **Retention Time of Naproxen** | **Retention Time of Sumatriptan** | **Area of** **Naproxen** | **Area of Sumatriptan** |
| **Standard(80mcg)** | 2.293 | 5.951 | 1976857 | 1990948 |
| **Analyst(1)(80mcg)** | 2.291 | 5.955 | 1971778 | 1993690 |
| **Analyst(2)(80mcg)** | 2.290 | 5.944 | 1970279 | 1995386 |
| **Analyst(3)(80mcg)** | 2.290 | 5.935 | 1979007 | 1992472 |
| **Analyst(4)(80mcg)** | 2.286 | 5.923 | 1970631 | 1993363 |
| AVG |  |  | 1973711 | 1993172 |
| STD |  |  | 3966.9 | 1631.1 |
| %RSD |  |  | 0.20 | 0.08 |

**Figure 1:**

**chromatogram of Naproxen**



**Figure 2:**

**chromatogram of Sumatriptan**

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**Figure 3:**

**Linearity:-**

**Linearity data of Naproxen**

**Figure 4:**

**Linearity data of Sumatriptan:-**

**Figure 5:**

**Assay: - Sumatriptan and Naproxen**

**Standard**

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**Sample**



**Figure 6:**

**Accuracy:-**

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**Accuracy 50%**

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**Accuracy 100%**

****

**Accuracy 150%**

**Figure 7:**

**PRECISSION:-**

Injection 1

****

Injection 2

****

Injection 3

****

Injection 4

****

Injection 5

****

**Figure 8:**

**System suitability:-**



|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | Name | Retenction Time (min) | Area (µv\*sec) | Height(µv) | USP Plate count | USP Tailing |
| 1 | Naproxen | 2.249 | 1531670 | 147652 | 2187.1 | 1.6 |
| 2 | Sumatriptan | 5.875 | 1573899 | 84946 | 2196.7 | 1.3 |

**Figure 9:**

**Robustness**

**More organic solvent**



**Less organic solvent**



**More flow**



**Less flow**



**Figure 10:**

**Ruggedness:-**

Injection 1

****

Injection 2

****

Injection 3

****

Injection 4

****

Injection 5

**Figure 11:**

**Limit of detection:-**

**Sumatriptan**



**Naproxen**



**Figure 12:**

**Limit of quantification:-**

**Sumatriptan**



**Naproxen**



**Figure 13:**

Structure of Sumatriptan



**Figure 14:**

Structure of Naproxen

