IGM Publication
Journal of Medical Science and Clinical Research
Volume||1||Issue||2||Pages99-107||2013
Website: www.jmscr.igmpublication.org ISSN (e): 2347-176X

Developement Of Validated UV Spectrophotometric Method For Estimation of Sumatriptan Succinate In Bulk and Pharmaceutical Dosage Forms

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ABSTRACT

The present research work discussed the development of a UV spectrometric method for validation of Sumatriptan Succinate. It is simple, fast, accurate and cost efficient and reproducible spectrophotometric method, developed for the estimation of Sumatriptan Succinate as a pure API. The wavelength (λ max) was found to be 229 nm by using acetonitrile as a solvent for the Sumatriptan Succinate. The linearity for this drug at the selected wavelength lies between 2-20 µg/ml. Beer's law was obeyed in this concentration range with correlation coefficient of 0.999. The accuracy and precision of the method were determined and validated according to ICH guidelines. The method has good reproducibility with % RSD less than one. Thus proposed method can successfully applied for Sumatriptan Succinate in routine analysis work.

Keywords: *Sumatriptan Succinate, API, spectrophotometric method, ICH guidelines.*

INTRODUCTION

Sumatriptan Succinate (SUMA) is chemically 3-[2-(dimethylamino)ethyl]-N-methyl-indole-5-methanesulfonamide succinate is a selective 5-hydroxytryptne1 receptor subtype. Migraine attack is a troublesome physiological condition associated with throbbing, intense headache in one-half of the head. During an attack, the blood vessels in the brain dilate and then draw together with stimulation of nerve endings near the affected blood vessels. These changes to the blood vessels and stimulation of nerves are probably what cause the pain.[1,2,3]

Sub-Q used for acute treatment of cluster headache episodes [4-12],

It is having Empirical formula-$C_{14}H_{21}N_3O_2S{\bullet}C_4H_6O_4$and Molecular weight - 413.5

Sumatriptan Succinate is a white to off-white powder that is readily soluble in water [13]

Analysis is an important component in the formulation development of any drug molecule. It becomes essential to develop a simple, sensitive, accurate, precise, reproducible method for the estimation of drug sample. Our main concern is development and validation of UV spectrometric method as per ICH guideline.[14,15]

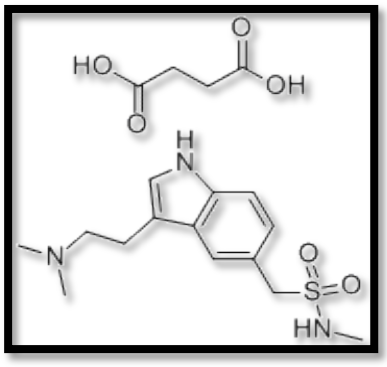

Figure no 1- Structure of Sumatriptan Succinate

MATERIALS AND METHODS:

Instrument:
Instrument used were UV-Visible double beam spectrophotometer Shimadzu UV1800 with one cm matched quartz cells. The glass wares used in each procedure were soaked overnight in a mixture of chromic acid and sulphuric acid rinsed thoroughly with double distilled water and dried in hot air oven prior to use. The absorption spectra of reference and test solution were carried out in a one cm quartz cell over the range of 200-400 nm.

Material:
Pure samples:

Sumatriptan Succinate was kindly supplied by Emcure Pharamaceuticals Pune, India.

Reagents and Chemicals:

Acetonitrile (Loba-Chemie.Pvt.Ltd., Mumbai, India), other reagents & chemicals used were of analytical grade.

Preparation of standard stock solution:

100 mg of Sumatriptan Succinate was accurately weighed and dissolved in small amount of acetonitrile in 100 ml volumetric flask and then the volume was adjusted with acetonitrile resultant solution gives the concentration of 1mg/ml ie.1000 µg/ml (stock –I solution). From this 10 ml solution was taken and then diluted up to 100 ml with same solvent in a volumetric flask and then the concentration of this stock was 100µg/ml (II stock solution).

Determination of absorbance maxima (λmax):

The stock solution was further diluted this solution was then scanned at wavelength of 200 to 400 nm against blank. The wavelength of maximum absorbance of ART was found at 229 nm which is used for preparation of calibration curve. **(Figure no. 2)**

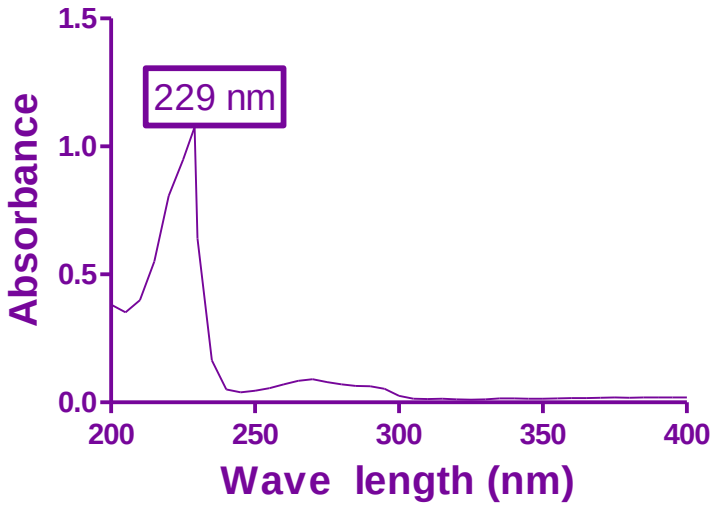

Figure no. 2: Determination of λ max of Sumatriptan Succinate

Preparation of Calibration Curve:

100 mg Sumatriptan Succinate of was accurately weighed and dissolved in small amount of acetonitrile in 100 ml volumetric flask and then the volume was adjusted with acetonitrile, the resultant solution gives the concentration of 1mg/ml ie.1000 µg/ml (stock –I solution). From this 10 ml solution was taken and then diluted up to 100 ml with the same solvent in a volumetric flask and then the concentration of this stock will be 100µg/ml (II stock solution). From this II stock solution, 2, 4,6,8,10,12,14,16,18,and 20 ml solutions were pippetted and volume was made to 100 ml using acetonitrile as a solvent to get concentrations 2, 4,6,8,10,12,14,16,18,and 20 respectively. The absorbance of these solutions was measured at 229 nm. The

standard calibration curve was obtained for data of concentration v/s absorbance; standard calibration curve data reported in **(Table no.1, Figure no.3)**

Table 1: Calibration data for the method development.

Sr .No.	Concentration (µg/ml)	Absorbance at 229nm ± standard deviation
1	2	0.226±0.0026
2	4	0.386±0.00360
3	6	0.581±0.00556
4	8	0.768±0.00503
5	10	0.958±0.0036
6	12	1.165±0.0020
7	14	1.326±0.00305
8	16	1.555±0.00360
9	18	1.745±0.0055
10	20	1.931±0.002636

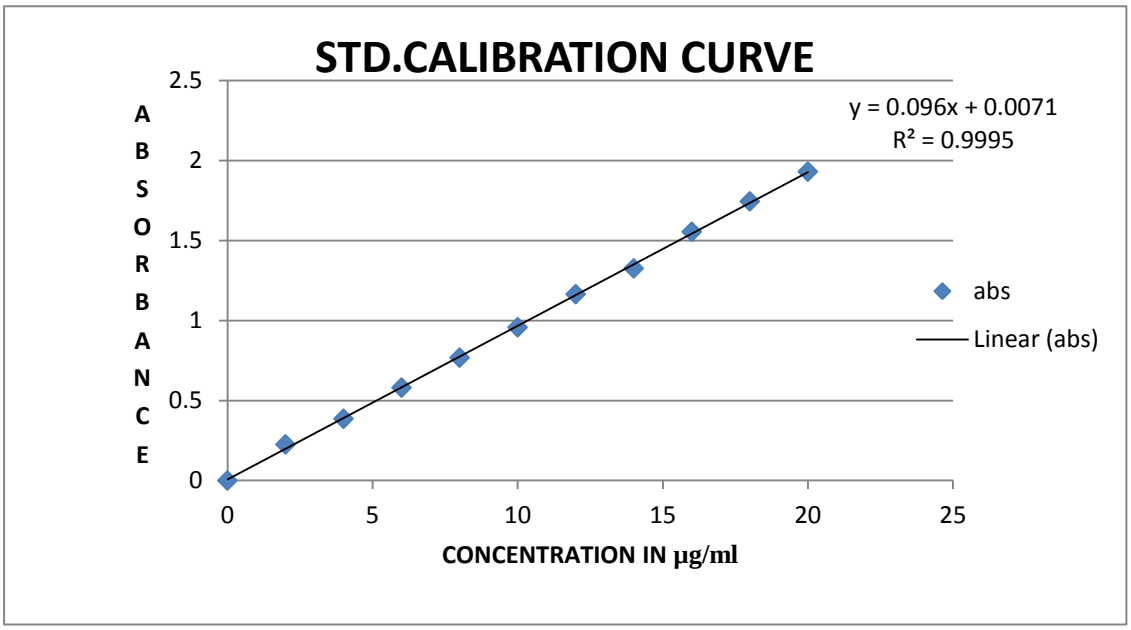

Figure. 3: Calibration Curve for Sumatriptan Succinate

METHOD VALIDATION:

Linearity and Range:

The linearity of the response of the drug was obtained at 2 to 20 µg/ml concentrations. The calibration curve was obtained by plotting the absorbance versus the concentration data and was treated by linear regression analysis (Table no. 3). The equation of the calibration curve for Sumatriptan Succinate obtained was y = 0.096x + 0.007, the calibration curve was found to be linear in the a forementioned concentrations (The correlation coefficient (R^2) of determination was 0.999).

Precision:

Precision of the method was analysed by repeatability, determined by analyzing 10 µg/ml of Sumatriptan Succinate for six times the results are reported in **(Table no. 2)**

Table no. 2: Data showing Repeatability of Absorbance's

Sr. No.	Conc. (µg/ml)	Wavelength (nm)	Absorbance	Mean± S.D.	%R.S.D
1		229	0.956		

2	10	229	0.964	0.958 ±0.0431	0.449
3		229	0.96		
4		229	0.962		
5		229	0.953		
6		229	0.955		

S.D. = Standard Deviation, R.S.D.= Relative Standard Deviation

Precision of the method was studied as intra-day and inter day variations. Intraday precision was determined by analyzing 4,8,12, µg/ml of Sumatriptan Succinate for three times within the day. Inter-day precision was determined by analyzing same concentration of solutions daily for three days, the results are reported in **(Table no. 4)**

Table no. 4: Results for Intra-day and Inter-day precision of Sumatriptan Succinate

Drug	**Conc. (µg/ml)**	**Intra-day Mean Abs**	**Absorbance ± S.D.**	**%RSD**	**Inter-day Mean Abs**	**Absorbance ± S.D.**	**%RSD**
sumatriptan succinate	04	0.380	±0.002	0.5263	0.391	±0.00207	0.5294
	08	0.756	±0.0030	0.3968	0.774	±0.002	0.2583
	12	1.152	±0.0017	0.1475	1.169	±0.0032	0.2737
Mean %RSD				0.3568			0.3538

S.D. = Standard Deviation, R.S.D.= Relative Standard Deviation

Limit of Detection And Quantitation (LOD and LOQ):

Determination of the detection and quantitation limits was performed based on the standard deviations of y-intercept and the slope of the least square line parameters as defined in the International Conference on Harmonization

(ICH) guidelines. [10] The LOD and LOQ were 0.068 and 0.2083 respectively

and data is reported in **(Table no.3)**

Table no 3: Validation parameters for sumatriptan succinate

Sr. No.	Parameters	Results
1	Absorption maxima (nm)	229 nm
2	Linearity range (µg/ml)	2-20 µg/ml
3	Standard Regression Equation	y = 0.096x + 0.007
4	Correlation coefficient (R^2)	R^2 = 0.999
5	Specificity	A 10 µg /ml solution of candidate drug in solvent accetonitrile at UV detection of 229 nm will show an absorbance value of 0.958
6	Accuracy (% Recovery)	99.79
7	Precision RSD Repeatability (n=6) Intra-day(n=3) Inter-day(n=3)	0.449 0.3568 0.3538
8	Molar Absorptivity	$4.054834*10^4$
9	LOD	0.068
10	LOQ	0.2083

n=no. of determinations, LOD=Limit of Detection, LOQ =Limit of Quantitaion, RSD= Relative Stanviationdard Deviation

Recovery Study:

To study the accuracy of proposed method, it was applied to analyse commercially available Sumatriptan Succinate tablet. Twenty tablets were weighed and powdered. The amount of tablet powder equivalent to 100 mg of Sumatriptan Succinate was weighed accurately and transfer to 100 ml volumetric flask then 10 ml of acetonitrile as a solvent was added and kept for 15-20 min with frequent shaking and volume was made up to mark with given solvent. The solution was then filtered through Whattman filter paper. This filtrate was diluted suitably with solvent to get the solution of 05µg/ml concentration. The absorbance was

measured against blank solution. The recovery experiment was performed at three different levels that are 80%, 100%, 120%. To the preanalyzed sample solution, a known amount of standard drug solution was added at three different levels and absorbance was recorded. The drug content of the preparation was calculated using standard calibration curve. Amount of drug estimated by this method is given in (**Table no. 5)**.

Table no. 5: Determination of Accuracy by Percentage Recovery Method.

Drug	Tablet amount (µg/ml)	Level of addition (%)	Amount added (µg/ml)	Amount recovered (µg/ml)	% Recovery	Average% Recovery
Sumatriptan Succinate	05	80	8	12.947	99.59	99.79
	05	100	10	14.979	99.86	
	05	120	12	16.989	99.93	

RESULTS AND DISCUSSION:

From dissolution point of view, attempt was made to dissolve the Sumatriptan Succinate in acetonitrile. The proposed method showed molar absorptivity $1.0596*10^4$ L/mol.cm. The calibration curve of Sumatriptan Succinate plotted at 229 nm (Figure no. 3) a linear relationship was obtained between 02-20 µg/ml. The accuracy of the method was determined by calculating mean percentage recovery it was found to be 99.79 % (**Table no.5)**. Further precision was calculated as repeatability, inter and intraday variations and %RSD was less than one **(Table no. 4)**. The LOD value was found to be 0.07352 and LOQ value was found to be 0.2228.

CONCLUSION:

The developed method was found to be simple, sensitive, accurate, precise, reproducible, and can be used for routine quality control analysis of Sumatriptan Succinate.

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