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Cleaning Method Development and its Validation for Quantification of Cinacalcet API Using UV Spectroscopy

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ABSTRACT

Simple, precise and cost effective cleaning method and validation by UV spectrophotometry has been developed for the estimation of Cinacalcet shows λmax at 279nm . The drug follows Beer-Lambert law in the concentration range of 2-10 µg/ml with correlation coefficient of 0.999. The method was validated by following analytical performance parameters suggested by the international conference on harmonization. All validation parameters were within the acceptable range. The developed method was successfully applied to estimate the amount of cinacalcet.

Keywords: cleaning method , Cinacalcet, UV spectrophotometry.

INTRODUCTION

Cinacalcet hydrochloride is an oral calcimimetic that is used to treat patients with parathyroid cancer and dialysis-dependent patients with end-stage renal disease (ESRD) in order to lower hypercalcaemia and treat secondary hyperparathyroidism (HPT)[1]. The first of a new class of medications known as calcimimetics, which function by making the parathyroid gland's calcium detecting receptors more sensitive,[2] is cinacalcet hydrochloride1, 2, 3. Cinacalcet hydrochloride is chemically represented as N-((1R)-1-(1-Naphthyl) ethyl)-3-(3-(trifluoromethyl) phenyl) propan-1-amine hydrochloride (Fig. 1).

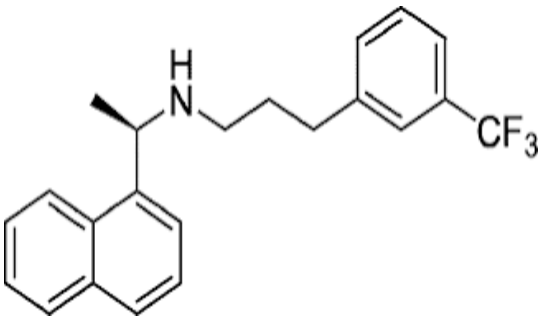

Figure 1: Cinacalcet hydrochloride's structure is displayed[3].

According to a review of the literature, many analytical techniques have been described for the measurement and identification of each drug separately in human plasma using tandem mass spectrometry and liquid chromatography[4-6]. There hasn't been a spectrophotometric approach for estimating cinacalcet hydrochloride using the two straightforward methods published in the literature. As a result, a straightforward, quick, accurate, and exact approach is created and verified for the estimation of cinacalcet hydrochloride in bulk and pharmaceutical formulation[7-10].

MATERIAL AND METHODS

Dr Reddy's provided samples of cinacalcet hydrochloride . 30 mg commercial PTH pills (Intas Pharma) were bought from a nearby market and utilised before the end of their shelf life. The other compounds that were employed were all analytical or pharmaceutical grade. Instruments For the purpose of measuring absorbance and spectrum, a LABINDIA double beam UV-visible spectrophotometer (Model: UV-3200) with a fixed bandwidth (1.5 nm) and a 1 cm quartz cell was employed. Furthermore, this study made use of an electronic balance, a micropipette, and a sonicator.

SAMPLE PREPARATION

Swab sampling

It is also know as direct surface sampling method. This method is based on the physical removal of residue left over on a piece of equipment .after it has been cleaned and dried. A swab wetted with a solvent is rubbed over a previously determined sample surface area to remove any potential residue, And thereafter extracted into a known volume of solvent in which the contaminant active ingredient residue is soluble. The amount of contaminant per swab is then determined by an analytical method of adequate sensitivityMethod: Standard stock solution preparation Cinacalcet hydrochloride (CIN) standard stock solutions were made by dissolving 100 mg of the medication in 100 millilitres of Acetonitrile, yielding a 1000 µg/ml standard stock solution. 1 ml pf the above solution is added on the ss plate and dried, then sample is swabbed and collected from ss plate by using swabstick and solution is make up with the suitable solvent (Acetonitrile). . The standard solution with a concentration of 100 µg/ml of CIN was obtained by further diluting this solution. Method A: Maximum Absorption Approach A spectrum mode scan of the Cinacalcet standard solution was performed from 400 nm to 200 nm in order to choose the analytical wavelength. The drug's spectra showed that the λmax of CIN was 279 nm, which was chosen for study. Standard stock solution was aliquoted, and a calibration curve was created at 279 nm in the concentration range of 2–10µg/ml.

At each sample's unique AUC range, a calibration curve was produced in the concentration range of 2–10 µg/ml. The medication complied with Beer-Lambert's law within the 2–10 µg/ml concentration range. Plotting absorbance against Cinacalcet hydrochloride concentration yielded the calibration curve. Table provides the values for the slope, intercept, and coefficient of correlation (r) for various techniques.. The calibration curve was used to determine the five concentrations of the sample solutions. Utilising the suggested techniques to calculate Cinacalcet in tablet dose form Twenty tablets were weighed in order to estimate the amount of drug in the tablet formulation. The weight equal to 100mg of Cinacalcet was then transferred to a 100 ml volumetric flask, ultrasonicated for 20 minutes, and the volume was adjusted with Acetonitrile. After that, the mixture was filtered using Whatmann filter paper (No. 42). The filtrate was further diluted according to protocol. In Method A, the absorbance of the sample at 279 nm in spectral mode was measured to determine the concentration of Cinacalcet. The calibration curve can be used to calculate the sample solution's concentration.

Verification of the created techniques. The techniques were validated in terms of selectivity, accuracy, linearity, and precision. Accuracy Three distinct recovery tests (50%, 100%, and 150%) were conducted using the standard addition method to determine the accuracy of the suggested approaches.

EXPERIMENTAL RESULTS AND DISCUSSION

Tab 1: Linearity study for cinacalcet by UV spectroscopy

CONCENTRATION(µg/ml)	Absorbance at 279nm
2	0.120
4	0.231
6	0.348
8	0.469
10	0.594

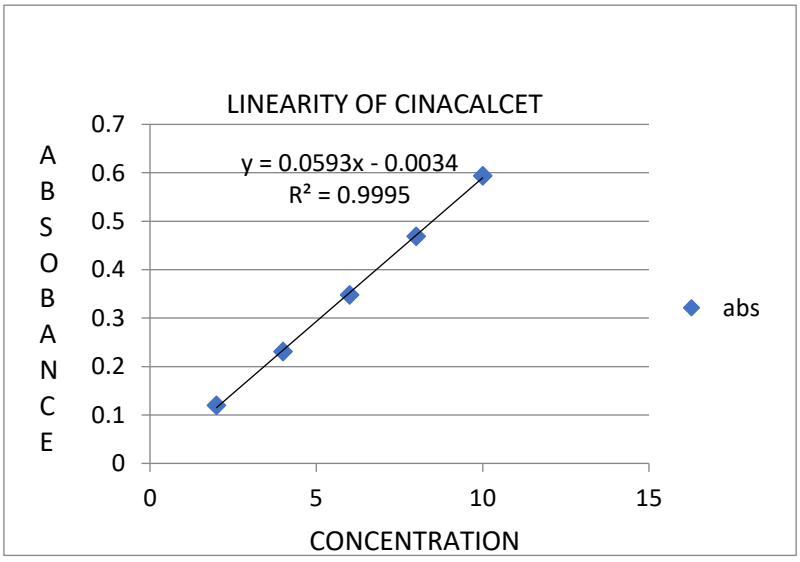

Fig 2 linearity curve of cinacalcet

Tab 2: Accuracy study for cinacalcet by UV spectroscopy

Spiked level(%)	Standard (µg/ml)	Sample(g/ml)	Absorbance	%Recovery	Mean	SD	%RSD
50	3	3	0.51	99.01	99.12	1	0.632
	3	3	0.345	98.55			
	3	3	0.481	99.79			
100	6	3	0.634	100.9	100.4	1.36	1.358
	6	3	0.729	99			
	6	3	0.670	101.4			
150	9	3	0.825	99.39	98.98	0.59	0.591
	9	3	0.83	98.31			
	9	3	0.79	0 79			

Tab 3: Precision study for cinacalcet by UV spectroscopy

S.NO	Concentration (µg/ml)	Absorbance
1	10	0.594
2	10	0.584
3	10	0.543
4	10	0.539
5	10	0.543
6	10	0.544
Mean		0.557
SD		0.024408
RSD		0.72926

Tab 4: Lod and Loq study for cinacalcet by UV spectroscopy

Parameters	Results
Wavelength	279nm
Linearity Range	2-12µg/ml
Slope	0.058
Intercept	0.05
Correlation Coefficient	0.999
LOD	0.300289
LOQ	0.909967

Tab 5: Robustness study for cinacalcet by UV spectroscopy

Concentration	Wavelength 278nm	Wavelength 279nm	Wavelength 280nm
	Absorbance	Absorbance	Absorbance
10ppm	0.536	0.543	0.534
10ppm	0.535	0.543	0.534
10ppm	O 536	0.544	0.535
Mean	0 535	0.543	0.534
SD	0.000577	0.000577	0.000577
RSD	0.035927	0.03542	0.036017

Tab 6: Ruggedness study for cinacalcet by UV spectroscopy

Instrument name-SHIMADZU			Instrument name-Lab India	
Day-1			Day-2	
Analyst -1	Analyst-2		Analyst-1	Analyst-2
Concentration	Absorbance	Absorbance	Absorbance	Absorbance
10ppm	0.593	0.591	0.401	0.3953
10ppm	0.594	0.594	0.404	0.3944

10ppm	0.592	0.584	0.403	0.3993
10ppm	0.594	0.583	0.405	0.3934
10ppm	0.589	0.587	0.403	0.3936
10ppm	0.584	0.585	0.402	0.3937
Mean	0.591	0.058733	0.403	0.39465
SD	0.003899	0.00432	0.001414	0.002305
%RSD	0.109947	0.122603	0.058487	0.097362

RESULT AND DISCUSSION

Cinacalcet hydrochloride in its pharmaceutical dose form can be easily and accurately analysed using the techniques covered in this article. The analysis focused on the Cinacalcet absorbance maxima at 279 nm. For approaches, linearity in the detector response was noted in the 2–10 µg/ml concentration range. In tablet analysis, the percent label claim for Cinacalcet was discovered to be between 99.87 % and 99.30% . High precision was indicated by the low standard deviation, percentage RSD, and standard error was calculated . Recovery studies were used to determine the accuracy of the suggested approaches, and the findings are expressed as a percentage of recovery. The percentage of Cinacalcet recovery was determined to be between 98.98% and 100.40 %. The recovery studies' findings demonstrated the high degree of accuracy of the suggested method . For the discovered Method , the percentage RSD for intraday assay precision was determined to be 0.729. Based on the results, it is determined that the suggested procedures are affordable, repeatable, accurate, and exact. They may be used for regular quality control of Cinacalcet hydrochloride in both its pharmaceutical dosage form and bulk drug form.

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