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# A NOVEL METHOD DEVELOPMENT FOR THE ESTIMATION OF IRBESARTAN IN TABLETS BY USING REVERSE PHASE LIQUID CHROMATOGRAPHY

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## ABSTRACT

A simple, sensitive, accurate method was developed for the estimation of Irbesartan in tablets by RP-HPLC technique. Chromatographic conditions used are stationary phase standard ODS (150 mm x 4.6 mm, 5µ) column, mobile phase was orthophosphate buffer: methanol in the ratio of (50:50,v/v) and flow rate was maintained at 1 ml/min, detection wave length was 258 nm, column temperature was set to 30$^{o}$C and diluent was mobile phase conditions were finalized as optimized method. System suitability parameters were studied by injecting the standard six times and results were well under the acceptance criteria. Linearity study was carried out between 37.5 to 225 µg/ml levels, $R^2$ value was found to be as 0.999. Precision was found to be 1.3 for repeatability and 0.8 for intermediate precision. LOD and LOQ are 0.205 µg/ml and 0.623 µg/ml, respectively. By using above method assay of marketed formulation was carried out 99.89% was present.

**KEYWORDS:** RP-HPLC, validation, irbesartan and estimation.

## INTRODUCTION

Irbesartan is a non peptide angiotensin II antagonist with antihypertensive activity[1] and the chemical name is 2-butyl-3-({4-[2-(2H-1,2,3,4-tetrazol-5-yl)phenyl]phenyl}methyl)-1,3-diazaspiro[4.4] non-1-en-4-one. Irbesartan was used for the treatment of hypertension and microalbuminuria or proteinuria.[2] Irbesartan selectively and competitively blocks the binding of angiotensin II to the angiotensin I receptor and angiotensin II stimulates

aldosterone synthesis and secretion by adrenal cortex, which decreases the excretion of sodium and increases the excretion of potassium. The existing available literature reveals that very few analytical methods are available for the estimation by UV[3-5] and HPLC[6] using different compositions of aetonitrile, methanol and water. All validation parameters reached the acceptable limits by performing the method as per the ICH guidelines.[7] So there is a need for a suitable method for routine analysis of Irbesartan in pure and formulations by using the mixture of mobile phases. Hence an attempt is made to develop a validated, simple, precise and accurate analytical method for estimation of Irbesartan by RP-HPLC and extend it for their determination in formulation.

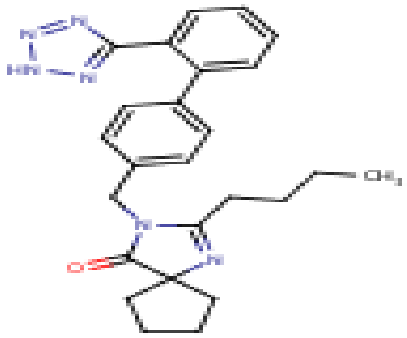

**Fig. 1: Molecular structure of Irbesartan.**

## MATERIALS AND METHODS

### Reagents and chemicals

Working standards of Irbesartan RS (Purity 100.1) was provided by ORTIN laboratories. The formulated local brand tablets were used for analysis and these are procured from local market. LC-grade water and methanol were procured from Merck (Germany). All other chemical reagents were of analytical grade.

### Preparation of reference solutions

Accurately weighed and transferred 15 mg Irbesartan working standard into a 10 ml clean dry volumetric flask, add 7 ml of diluent, sonicated for 30 minutes and make up to the final volume with diluents. From the above stock solution, 1 ml was pipetted out in to a 10ml volumetric flask and then make up to the final volume with diluent.

### Preparation of sample solutions

20 tablets were weighed and calculate the average weight of each tablet then the weight equivalent to 1 tablet was transferred into a 100 ml volumetric flask, 5 ml of diluent added

and sonicated for 30 min, further the volume made up with diluent and filtered. From the filtered solution 1ml was pipette out into a 10 ml volumetric flask and made up to 10 ml with diluent.

## RESULTS AND DISCUSSION

### Method development

The STD ODS (150 mm x 4.6 mm, 5 µm) column was used for detection at a wavelength of 258 nm with mobile phase is orthophosphate buffer: methanol taken in the ratio (50:50, v/v) a gradient elution mode. The contents of the mobile phase was degassed, with a helium sparge for 15 min and filtered through vacuum filtration and pumped from the respective solvent reservoirs to the column at a flow rate of 1 ml/min. The column temperature was maintained at 30$^{o}$c and run time is 10 min and the injection volume of sample was 10 µl. The retention time for Irbesartan was found to be 2.330 min shown in Figure.2.

### Method of validation

The proposed method was validated with respect to linearity, precision, accuracy, limit of quantitation (LOQ), limit of detection (LOD) and robustness.

### Linearity

To establish the linearity of analytical method, a series of dilution in the range from 37.5-225 µg/ml for Irbesartan was prepared. All the solutions were filtered through 0.22 µm membrane filter prior to use and injected in chromatograph. A calibration curves were plotted between the mean peak areas vs respective concentrations. The corresponding linear regression equation was y=130406x+37606 with square of correlation coefficient $R^2$ of 0.999 for Irbesartan was shown in Figure.3 and Table.1.

### Precision

The precision of the instruments was checked by repeatedly injecting (n=6) solutions of Irbesartan (100 µg/ml). The intra-day and inter-day precision of the proposed methods were determined by the corresponding responses 6 times on the same day and on 2 different days of Irbesartan (100 µg/ml).

### Accuracy

Recovery studies were performed to validate the accuracy of developed method. To the pre analysed sample solution, a definite concentration of standard drug was added and then its

recovery was analysed. The range of percent recovery for Irbesartan from 98.90 to 100.34% was established. Results presented in Table.2 indicated good accuracy and showed no interference from tablet excipients.

**Limit of Detection**

The limit of detection of an individual analytical procedure is the lowest amount of analyte in a sample which can be detected but not necessarily quantitated as an exact value. The limits of detection were calculated from the standard deviations and slopes of the responses using a signal-to-noise ratio. The LOD for Irbesartan was found to be 0.205 µg/ml.

**Limit of Quantitation**

The quantitation limit of an analytical procedure is the lowest amount of analyte in a sample, which can be quantitatively determined with suitable precision and accuracy. The limit of quantitation was calculated from the standard deviations and slopes of the responses using a signal-to-noise ratio. The LOQ for Irbesartan was found to be 0.623 µg/ml.

**Robustness**

As part of the robustness, deliberate change in the flow rate, mobile phase composition was made to evaluate the impact on the method. From the results regarding validation for method developed it is observed that the results were within the limits according to ICH hence the method developed was successfully shown in Table.3.

**Assay of pharmaceutical formulation**

The proposed validated method was successfully applied to determine Irbesartan in their tablet dosage form. The result obtained for Irbesartan was comparable with the corresponding labeled amounts shown in Table.4.

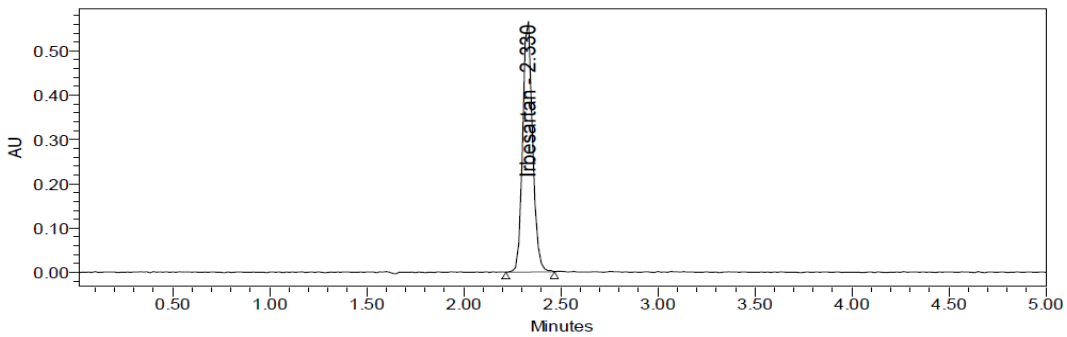


**Fig. 2: Standard chromatogram of Irbesartan.**

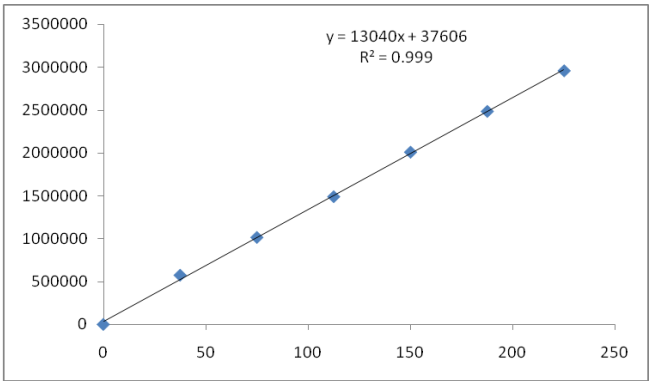


**Fig. 3: Linearity plot of Irbesartan.**

**Table 1: Linearity data of Irbesartan.**

| Concentration (ppm) | Area |
|---|---|
| 0 | 0 |
| 37.5 | 574321 |
| 75 | 1014843 |
| 112.5 | 1490146 |
| 150 | 2008914 |
| 187.5 | 2485191 |
| 225 | 2958651 |

**Table 2: Accuracy data of Irbesartan.**

| % Level | Amount Spiked (µg/mL) | Amount recovered (µg/mL) | % Recovery |
|---|---|---|---|
| 50% | 75 | 74.09 | 98.90 |
| | 75 | 75.53 | 100.07 |
| | 75 | 75.63 | 100.34 |
| 100% | 150 | 149.60 | 99.46 |
| | 150 | 148.00 | 98.66 |
| | 150 | 149.75 | 99.67 |
| 150% | 225 | 224.82 | 99.39 |
| | 225 | 224.00 | 99.55 |
| | 225 | 224.59 | 99.19 |

**Table 3: Robustness data of Irbesartan.**

| Parameter | % RSD |
|---|---|
| Flow Minus | 1.4 |
| Flow Plus | 0.9 |
| Mobile phase Minus | 0.1 |
| Mobile phase Plus | 0.4 |
| Temperature minus | 0.5 |
| Temperature plus | 0.2 |

**Table 4: Assay of Formulation.**

| Sample No | %Assay |
|---|---|
| 1 | 98.35 |
| 2 | 98.48 |
| 3. | 101.29 |
| 4. | 101.30 |
| 5. | 100.09 |
| 6. | 99.83 |
| AVG | 99.89 |
| STDEV | 1.2930 |
| %RSD | 1.29 |

## CONCLUSION

Thus the proposed estimation of Irbesartan in pure and tablet dosage form by RP-HPLC method was accurate, precise, linear, reliable, simple, economic and robust. The method has several advantages, including simple mobile phase, rapid analysis, simple sample preparation and improved selectivity as well as sensitivity. The method can be used for routine analysis of pharmaceutical formulation of Irbesartan in tablet formulation.

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