Analytical Method Development and Validation of Canagliflozin: Review

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Abstract: Analytical method development and validation are the continuous and inter-dependent task associated with the research and development, quality control and quality assurance departments. Analytical procedures play a critical role in equivalence and risk assessment, management. It helps in establishment of product-specific acceptance criteria and stability of results. Validation should demonstrate that the analytical procedure is suitable for its intended purpose. Design of experiment is a powerful tool for the method characterization and validation. Analytical professionals should be comfortable to use it to characterize and optimize the analytical method. An effective analytical method development and its validation can provide significant improvements in precision and a reduction in bias errors. It can further help to avoid costly and time consuming exercises.

Literature survey reveals that few UV-Spectroscopy, HPLC, HPTLC methods have been reported for the estimation of Canagliflozin. The published methods were validated for various parameters as per ICH guidelines. Statistical analysis provided that the published methods were reproducible and selective for the estimation of the Canagliflozin in pure and pharmaceutical dosage form.

Introduction:

Canagliflozin (C24H25FO5S) is a white to off white solid with melting range of 95-105°C[1] is chemically named as (2S,3R,4R,5S,6R)-2-[3-[[5-(4-fluorophenyl)thiophen-2-yl]-methyl]-4-methylphenyl]tetrahydro-6-hydroxymethyl-2H-pyran-3,4,5-triol (Figure-1). It is soluble in many organic solvents (methanol, Dimethyl sulfoxide) but insoluble in aqueous media. Canagliflozin is the first Sodium-glucose co-transporter 2 (SGLT-2) inhibitor which was used for the treatment of patients with type 2 diabetes. Canagliflozin reduces reabsorption of filtered glucose by inhibiting Sodium-glucose co-transporter 2 (SGLT2) and lowers the renal threshold for glucose (RTG) and thereby increases urinary glucose excretion.

Fig. 1: Structure of Canagliflozin

Literature survey:

1. Ishpreet Kaur et al., have developed a simple, sensitive, precise, rapid and cost effective method for determination of Canagliflozin in bulk and pharmaceutical formulations as per ICH Guidelines. Methods: A simple double beam UV Spectrophotometric method has been developed and validated with different parameters such as Linearity, Precision, Repeatability, Limit of Detection (LOD), Limit of Quantification (LOQ), Accuracy, Robustness and Ruggedness.

2. Nareddy Preethi Reddy et al., have developed a simultaneous estimation of Metformin and Canagliflozin in pharmaceutical dosage forms. Chromatography was carried out on an ODS
250 mm x 4.6 mm, 5 μm particle size with an isocratic mobile phase composed of Buffer, Acetonitrile and methanol at a flow rate of 1mL/min. The column temperature was maintained at 30°C and the detection was carried out using a PDA detector at 212 nm. Validation parameters such as system suitability, linearity, precision, accuracy, specificity, limit of detection (LOD), limit of quantification (LOQ), Stability of sample and standard stock solutions and robustness were studied as reported in the International Conference on Harmonization guidelines. The retention times for Metformin and Canagliflozin were 2.783 min and 3.781 min respectively. The percentage recoveries of Metformin and Canagliflozin were 100.1% and 100.2% respectively. The relative standard deviation for assay of tablets found to be less than 2%. The method is fast, accurate, precise and sensitive hence it can be employed for routine quality control of tablets containing both drugs in quality control laboratories and pharmaceutical industries.

3. P.B. Dudhe et al., have developed a simple, specific, sensitive, precise, selective and accurate reverse phase high performance liquid chromatographic method was developed for the determination of canagliflozin in human plasma as per US-FDA guidelines. Plasma samples were extracted by protein precipitation method using methanol as extracting solvent. The chromatographic separation was performed with WATERS EA874 (250 × 4.6 mm, 5 mm) column and mobile phase composed of 36.46 mM Acetate buffer: acetonitrile: methanol (30:50:20, v/v), pH 4.5 adjusted with acetic acid at a flow rate of 1.0 ml/min. Canagliflozin was detected at 290 nm with retention time of 5.1 min. Linearity was found to be 0.9929 over the range of 33.33 – 233.33 ng/ml and percentage recoveries were found to be 94.68 - 103.76 %. The validation was successfully performed by means of accuracy and precision, selectivity and specificity, linearity, recovery and stability under various conditions. This developed method can be successfully employed for the determination of Canagliflozin in human plasma.

4. S. Sreenivasulu et al., have developed a simple reverse phase liquid chromatographic method with ultraviolet detector was developed for the accurate determination of Canagliflozin using Phenomenex Gemini-NX C18 Column (250×4.6 mm, 5 μm particle size). The mobile phase used for the determination was Acetonitrile: 1-octanesulphonic acid in a ratio of 70:30 v/v at a flow rate of 1 mL per min. Canagliflozin was eluted at 3.4 ± 0.5 min and detected at 245 nm. The method is linear over the concentration range of 10-100 μg/mL with correlation co-efficient r = 0.9997. The plate count and tailing factor was found 5398 and 1.05 respectively. The developed method was extensively validated with different parameters such as Linearity, Precision, Accuracy, Robustness, Ruggedness, Limit of Detection (LOD) and Limit of quantification (LOQ) and proved to be robust. The validated method is definite, meticulous and reproducible and can be used for routine analysis of Canagliflozin in bulk form.

5. Vinutha Kommineni et al., have developed a new stability indicating RP HPLC method has been developed and validated for simultaneous estimation of Metformin Hydrochloride and Canagliflozin in bulk and dosage forms. The method involves separation on Kromasil C18 column (250mm x 4.6mm x5μm particle size). The optimized mobile phase consists of 0.1% OPA (pH 2.8) and Acetonitrile (45:55v/v) with a flow rate of 1ml/min and UV detection at 254nm. Retention time was 2.112 min for Metformin Hydrochloride, 2.671 min for Canagliflozin. RPHPLC method for the simultaneous estimation of Metformin Hydrochloride and Canagliflozin in their combine dosage form was developed and validated as per the ICH guidelines. Linearity was observed in the range of 25150μg/ml for Metformin Hydrochloride and 2.515μg/ml for Canagliflozin with correlation coefficients (r2=0.999). The percentage recoveries of Metformin Hydrochloride and Canagliflozin were in the range of 98.2101.4% which was with in the acceptance criteria. The percentage RSD was NMT 2% which proved the precision of the developed method. When applied for tablet assay, drug content was within 98.55-101.4% of labeled content. Forced degradation studies indicated the suitability of the method for stability studies.

Conclusion:

Literature survey suggested that various Spectroscopy, RP- HPLC, RP-LC methods were developed and reported. The published methods were validated for various parameters as per ICH guidelines. Statistical analysis proved that the published methods were reproducible and selective. Thus it can be concluded that the reported and published methods can be successfully applied for the estimation of the Canagliflozin in pure and pharmaceutical dosage form.

References:

2. Ishpreet Kaur, Sharad Wakode, Harsharan Pal Singh. Development and Validation of


