RP-LC METHOD FOR THE DETERMINATION OF VILDAGLIPTIN IN TABLET FORMULATION

Barden, Amanda T.[1,*]; Salamon, Bárbara[1]; Steppe, Martin[1]

[1]Laboratório de Controle de Qualidade, Faculdade de Farmácia, UFRGS.
***Mestrando – Início: 2009/1**

Introduction: Vildagliptin (VLG), (2*S*)-1-[*N*-(3-hydroxy-1-adamantyl) glycyl] pyrrolidine-2, is a potent and selective dipeptidyl peptidase-4 (DPP-4) inhibitor that improves glycaemic control in patients with type 2 diabetes mellitus by increasing α- and β-cell responsiveness to glucose.[1] DPP4 inhibitors represent a new class of antidiabetic agents that improve glycemic control by preventing glucagon-like peptide-1 (GLP-1) and glucose-dependent insulinotropic polypeptide (GIP) degradation.[2] The pharmaceutical product is commercially available, but at the moment, there are no methods published for the quantitative analysis of VLG as active pharmaceutical ingredient or finished product.
Objective: To develop and validate a simple, fast, accurate and stability-indicating reversed-phase liquid chromatography (RP-LC) method for the determination of VLG in solid pharmaceutical dosage form.
Materials and Methods: The reference standard (purity of 99.5%) was purchased of Sequoia Researched Products (United Kingdom) and the commercial tablets of Galvus® (Novartis Biociências S. A., SP, Brazil) containing 50 mg of VLG were obtained from commercial sources within their shelf life period. LC grade acetonitrile was obtained from Tedia (Fairfield, OH, USA). All chemicals used were of pharmaceutical or special analytical grade. Purified water was obtained by a Millipore® Direct-Q 3UV with pump (Molsheim, AL, France). The LC system was operated isocratically, at room temperature, using a mobile-phase consisted of acetonitrile and a solution of triethylamine 0.3% adjusted to pH 7.0 with phosphoric acid (15:85; v/v) run at a flow-rate of 1.0 mL min^{-1}, and using photodiode array (PDA) detection at 207 nm.
Results and Discussion: The method was validated in accordance to ICH guidelines acceptance criteria for specificity, linearity, precision, accuracy, robustness and system suitability. Stress studies were carried out and no interference of the degradation products was observed. There was no interference of the excipients in the determination of VLG. The chromatographic separation was obtained within 6 min and was linear in the range of 20-80 μg mL^{-1} (r^2 = 0.9999). Limit of detection and limit of quantitation were 0.63 μg mL^{-1} and 2.82 μg mL^{-1}, respectively. Basic degradation was determined how first-order kinetics, with constant k of 0.00443 min^{-1} and $t_{90\%}$ of 23.93 min.
Conclusions: The proposed method was successfully applied for the quantitative analysis of VLG in tablet dosage form, contributing to improve the quality control and for studies of stability of pharmaceutical tablets containing this drug.

References:
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2. M. M. Jost *et al.*, *Biochem. Pharmacol.* **77**, 228 (2009).
Acknowldgements: Financial support from CNPq/Brazil and master scholarship from CAPES/Brazil.