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Original article

A rapid hydrophilic interaction liquid chromatographic determination of glimepiride in pharmaceutical formulations

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ABSTRACT

Glimepiride is one of the most widely prescribed antidiabetic drugs and contains both hydrophobic and hydrophilic functional groups in its molecules, and thus could be analyzed by either reversed-phase high performance liquid chromatography (HPLC) or hydrophilic interaction liquid chromatography (HILIC). In the literature, however, only reversed-phase HPLC has been reported. In this study, a simple, rapid and accurate hydrophilic interaction liquid chromatographic method was developed for the determination of glimepiride in pharmaceutical formulations. The analytical method comprised a fast ultrasound-assisted extraction with acetonitrile as a solvent followed by HILIC separation and quantification using a Waters Spherisorb S_5NH_2 hydrophilic column with a mobile phase consisting of acetonitrile and aqueous acetate buffer (5.0 mM). The retention time of glimepiride increased slightly with decrease of mobile phase pH value from 6.8 to 5.8 and of acetonitrile content from 60% to 40%, indicating that both hydrophilic, ionic, and hydrophobic interactions were involved in the HILIC retention and elution mechanisms. Quantitation was carried out with a mobile phase of 40% acetonitrile and 60% aqueous acetate buffer (5.0 mM) at pH 6.3, by relating the peak area of glimepiride to that of the internal standard, with a detection limit of 15.0 μg/L. UV light absorption responses at 228 nm were linear over a wide concentration range from 50.0 μg/L to 6.00 mg/L. The recoveries of the standard added to pharmaceutical tablet samples were 99.4–103.0% for glimepiride, and the relative standard deviation for the analyte was less than 1.0%. This method has been successfully applied to determine the glimepiride contents in pharmaceutical formulations.

1. Introduction

Glimepiride, 1-[[p-[2-(3-ethyl-4-methyl-2-oxo-3-pyrroline-1-carboxamido)ethyl] phenyl] sulfonyl]-3-(trans-4-methylcyclohexyl) urea (Fig. 1), is a sulfonylurea derivative and one of the most widely prescribed oral drugs for the treatment of non-insulin dependent type II diabetes mellitus (T2DM) (Davis, 2004; Harper et al., 2013). Glimepiride acts as an insulin secretagogue. It stimulates the secretion of insulin by pancreatic β-cells and increases sensitivity of intracellular insulin receptors, which lowers blood glucose level (Kabadi and Kabadi, 2004; Shukla et al., 2004). Glimepiride is usually used after diet and exercise measures fail to achieve appropriate control of blood glucose level. Like all other sulfonylurea medicines, glimepiride is normally given to diabetic patients over a long period of time. The overdose of glimepiride can cause hypoglycemia and other side effects, such as gastrointestinal tract disturbance, allergic reactions, liver dysfunction, chest pain, irregular heartbeat, endocrine disruption, and hemolytic anemia (Adachi and Yanai, 2015; Chounta et al., 2005; Harper et al., 2013; Papathanassiou et al., 2009). Thus, the amount of glimepiride in pharmaceutical dosage formulations administrated to the patients is critical in achieving high level of efficacy and safety of the anti-diabetic medication. Therefore, an accurate, simple and fast analytical method for monitoring glimepiride in pharmaceutical formulations is needed for the quality control.

Many analytical methods have been reported for the determination of glimepiride. Altinöz and Tekeli (2001) used a simple derivative UV spectrophotometric method for the determination of glimepiride in pharmaceutical tablets. Fahim et al. (2014)

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described a transmission Fourier transform infrared spectroscopy (FTIR) technique for analysis of metformin and glimepiride in drug samples. Badawy et al. (2010) performed quantitative measurement of glimepiride, and three other anti-diabetic drugs, rosiglitazone, pioglitazone and glyburide, using cyclic voltammetry and differential pulse voltammetry. Several research groups developed and validated analytical methods based on high performance liquid chromatographic (HPLC) and high performance thin layer liquid chromatography (HPTLC) for the determination of glimepiride individually or with some other anti-diabetic drugs (Jain et al., 2008; Kovaříková et al., 2004; Ni et al., 2014; Dash et al., 2016; Sane et al., 2004a, b; Shaodong et al., 2010). Among all reported analytical methods, HPLC has been the most commonly used method with high selectivity and accuracy, especially for complex biological samples. However, all previously reported HPLC methods for the analysis of glimepiride were based on reversed phase separation; and no hydrophilic interaction (aqueous normal phase) liquid chromatographic (HILIC) technique has been reported on the analysis of glimepiride, a drug containing both hydrophilic and hydrophobic functional groups, in the literature. In contrast to reversed phase HPLC, which employs a nonpolar stationary phase (SP) and a polar mobile phase (MP), HILIC uses a polar hydrophilic (normal) SP and an aqueous-polar organic solvent MP (Chen and Zuo, 2007; Jiao and Zuo, 2009; Zuo et al., 2002; Zuo et al., 2014). Hence, HILIC provides a different elution order and selectivity from reversed phase HPLC and has been increasingly applied to the separation and determination of polar pharmaceutical drugs and metabolites in recent years (Qin et al., 2008; Dejaegher and Heyden, 2010; Ares and Bernal, 2012; Zuo et al., 2011, 2014, 2015). In this study, an accurate, simple and rapid HILIC method has been developed for the determination of glimepiride in pharmaceutical formulations.

2. Experimental

2.1. Chemicals

Glimepiride and 2,2′-azino-bis (3-ethylbenz-thiazoline-6-sulfo nic acid) diammonium salt (internal standard) were purchased from Sigma-Aldrich (St. Louis, MO, USA). HPLC grade acetonitrile (ACN) and acetic acid (95%) were supplied by Fisher Scientific (Fair Lawn, NJ, USA). Sodium hydroxide was obtained from CMS. Inc. (Houston, Texas, USA). Except where noted, all reagents were of analytical grade and all solutions were prepared using distilled-deionized water. The mobile phase solvents were degassed by vacuum filtration through 0.45 μm nylon membranes (Fisher Scientific, Fair Lawn, NJ, USA) before HPLC analysis.

2.2. Chromatographic conditions

An Alliance HPLC system (Waters Corporation, Milford, MA. USA) equipped with a Waters 2695 Separation Module, a Waters 486 Tunable UV-Visible Absorbance Detector and Empower 2 software was employed for analyses. The analytical column used was a Waters Spherisorb S_5NH_2 column (250 mm × 4.6 mm, 5 μm; Waters, Milford, MA). The detection of glimepiride was carried out by UV absorbance at 228 nm. The flow rate was 1.0 mL/min. The injection volume was 20 μL.

2.3. Standard solution preparation

Standard stock solution of glimepiride was prepared in acetonitrile with concentration of 250 mg/L. Internal standard stock solution was prepared by dissolving 2,2′-azino-bis (3-ethylbenz-thiazoline-6-sulfonic acid) diammonium salt in distilled-deionized water with concentration of 1000 mg/L. Working standard solutions were prepared by adding appropriate amount of standard stock solutions into mobile phase (40% ACN and 60% of 5.0 mM sodium acetate buffer solution, pH 6.3).

2.4. Optimization of mobile phase

Mobile phase consisted of acetonitrile and acetate buffer solution (5.0 mM) was tested for separation of the standard mixtures and samples. To examine the effect of pH value on the analyte retention, the pH value of mobile phase was varied from 5.8 to 6.8 while other mobile phase compositions remain constant (50:50 ACN and acetate buffer). The effect of organic solvent percentage on the retention was tested by changing the content of acetonitrile in mobile phase from 40% to 60% in intervals of 10%, while pH of mobile phase was maintained at 6.3.

2.5. Calibration curve

Standard working solutions (10.0 mL) of 0.00, 1.00, 2.00, 3.00, 4.00, 5.00, and 6.00 mg/L glimepiride were prepared by mixing the desired volume of standard stock solution with constant amount (25 μL) of internal standard stock solution. Calibration curves were constructed by linear regression of the peak area ratio of glimepiride standards to the internal standard versus the concentration of glimepiride. The accuracy (recovery) was tested at two concentration levels by spiking known amount (2.00 and 3.00 mg/L) of glimepiride stock solution into the ground samples and determining the amounts of standard recovered.

2.6. Sample preparation

Glimepiride tablets were obtained from Walgreen (1.0 mg pink tablet, Sanofi Pharmacy, Deerfield, IL, USA) and Sanofi (2.0 mg green tablet, Beijing, China), respectively. Tablets of each brand were weighed and finely grinded, a quantity of around 5.0 mg powder was accurately weighed and transferred into 8.00 mL of acetonitrile. To increase extraction efficiency, the mixture of tablet powder and acetonitrile was ultra-sonicated for 5 min and then centrifuged at 2500 rpm for 15 min. The supernatants were filtrated through 0.45 μm membrane filters. 4.0 mL of the filtrates were mixed with 6.0 mL aqueous acetate buffer solution (5.0 mM). The pH of final solution was adjusted to 6.30 by using 2.0 M NaOH. To minimize local acid-base concentration change during the pH adjustment, micro syringe was used for adding pH adjusting reagent. 25 μL of internal standard stock solution was added into prepared sample solutions before injection into HPLC.

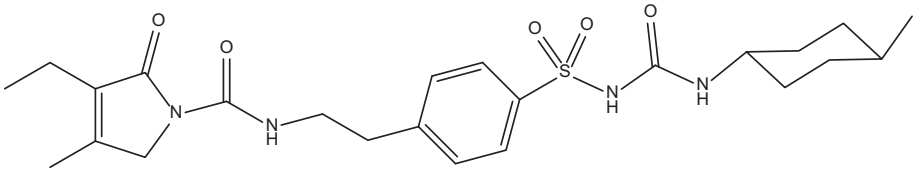

Figure 1. Chemical structure of glimepiride.