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Trace Analysis of Genotoxic Impurity Pyridine in Sitagliptin Drug Material Using UHPLC-MS

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Abstract : Background: Pyridine is a reactive base that might be used in preparing sitagliptin. International Agency for Research on Cancer classifies pyridine in group 2B; this classification means that pyridine is possibly carcinogenic to humans. Therefore, pyridine should be monitored at the allowed limit in sitagliptin pharmaceutical ingredients. Objective: The aim of this study was to develop a novel ultra high performance liquid chromatography mass spectrometry (UHPLC-MS) method to estimate the quantity of pyridine impurity in sitagliptin pharmaceutical ingredients. Methods: The separation was performed on C8 shim-pack (150 mm X 4.6 mm, 5 µm) in reversed phase mode using a mobile phase of water-methanol-acetonitrile containing 4 mM ammonium acetate in gradient mode. Pyridine was detected by mass spectrometer using selected ionization monitoring mode at m/z = 80. The flow rate of the method was 0.75 mL/min. Results: The method showed excellent sensitivity with a quantitation limit of 1.5 ppm of pyridine relative to sitagliptin. The linearity of the method was excellent at the range of 1.5-22.5 ppm with a correlation coefficient of 0.9996. Recoveries values were between 93.59-103.55%. Conclusions: The results showed good linearity, precision, accuracy, sensitivity, selectivity, and robustness. The studied method was applied to test three batches of sitagliptin raw materials. Highlights: This method is useful for monitoring pyridine in sitagliptin during its synthesis and testing sitagliptin raw materials before using them in the production of pharmaceutical products.

Keywords : genotoxic impurity, pyridine, sitagliptin, UHPLC -MS
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