

Quantitative determination of sitagliptin by gas chromatography using ethyl chloroformate as a derivatizing reagent in pure and pharmaceutical preparation

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A novel gas liquid chromatographic (GC) method has been developed for the quantitative estimation of Sitagliptin in bulk drug and pharmaceutical dosage forms. Ethyl chloroformate (ECF) was used as a precolumn derivatizing reagent. GC separation was carried out on an Rtx-5 capillary column (cross bond 5% diphenyl/ 95% dimethyl polysiloxane) with a length of 30 meters and an internal diameter of 0.25 mm with flame ionization detector. The elution was carried out at an initial temperature of 80°C for 4 minutes and temperature increased at the rate of 100°C/min up to 180°C for 5 min. Column pressure was programmed as 29.8 Kpa for 3.5 minutes and pressure increased at rate of 20Kpa/min up to 120Kpa for 6 minutes. The linear calibration ranges for LEV was observed between 2- 10 ng/ml. The method was subsequently applied to the determination of LEV in pharmaceutical preparations. The relative standard deviation (RSD) was found to be 0.27%. The recovery studies were done and the percentage recovery of LEV was found to be 98.36%.

Biography

Sruthi is a student of JSS College of Pharmacy, JSS University, Mysore. She has completed her B.Pharm from Raghu College of Pharmacy, Vishakhapatnam. Presently she is pursuing M.Pharm Degree in the branch of Pharmaceutical analysis. Her current area of research is on analytical method development of novel drugs using HPLC and GC.

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RP-HPLC method development and validation for simultaneous estimation of lafutidine and domperidone

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A simple, precise, and reproducible RP-HPLC method has been developed for the simultaneous determination of Lafutidine (LAF) and Domperidone (DOM). RP-HPLC separation of two drugs was achieved on a Thermo BDS Hypersil C18 column (250 mm × 4.6 mm, 5 μm) using UV detection at 273 nm. The optimized mobile phase consisted of methanol: acetonitrile: 0.05M phosphate buffer (20:20:60v/v/v) using 1 ml/min flow rate. The column temperature was 30°C and injection volume was 20 μL. The two drugs were satisfactorily resolved with retention time values of 4.0 ± 0.02 and 7.9 ± 0.02 mins for Lafutidine and Domperidone respectively. The LOD for Lafutidine and Domperidone were found to be 0.02 μg/ml and 0.11 μg/ml, respectively, while LOQ were 0.06 μg/ml and 0.34 μg/ml, respectively. The method was validated for linearity, accuracy, precision, robustness, and specificity, as per ICH guidelines. Insignificant differences in peak areas and less variability in retention time values as indicated by low values of % R.S.D were observed, which proves that the method was robust. The method was found to be linear over concentration ranges of 10-20 μg/ml ($r^2 = 0.998 \pm 0.008$) and 30-60 μg/ml ($r^2 = 0.999 \pm 0.002$) for Lafutidine and Domperidone respectively. Recovery studies revealed average recoveries of 100.24% for Lafutidine and 99.68 % for Domperidone, by using standard addition method.

RP-HPLC method was successfully applied to the analysis of two drugs in a pharmaceutical capsule formulation. The method proved to be simple and rapid for routine simultaneous estimation of LAF and DOM in the bulk drug and in a capsule formulation.

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