ABSTRACT

The present work was aimed at developing new validated simultaneous RP – HPLC methods and UV Spectroscopic methods for selected API’s. The following are the formulations containing the selected API’s.

Janumet - sitagliptin and metformin hydrochloride
Juvisync - sitagliptin and simvastatin
Tizaran – diclofenac potassium and tizanidine hydrochloride

All the above mentioned formulations containing the selected API’s were subjected to simultaneous estimation by RP- HPLC.

The separations were carried out using Agilent C8 Column (25 x 4.6 mm, 5µ) as stationary phase. Methanol: water in varying ratios was used as mobile phase with 1ml/min flow rate for all the three Hplc methods. For the simultaneous estimation of metformin hydrochloride and sitagliptin, the detection wavelength was selected as 267nm. The retention times of metformin hydrochloride and sitagliptin were found to be 3.0min and 7.0min respectively. For the simultaneous estimation of sitagliptin and simvastatin, the detection wavelength was at 253nm. The retention times of sitagliptin and simvastatin were found to be 3.22min and 15.760 min respectively. For the simultaneous estimation of diclofenac potassium and tizanidine hydrochloride, the detection wavelength was at 235nm. Retention times of diclofenac potassium and tizanidine hydrochloride were found to be 5.9min and 2.1min respectively.
All the above mentioned formulations containing the selected API’s were subjected to simultaneous estimation by UV Spectrophotometer.

UV Spectroscopic method for the simultaneous estimation of metformin hydrochloride and sitagliptin was based on solving simultaneous equations with 232nm and 266nm as two analytical wavelengths. Distilled water was used as the common solvent. Linearity was found in the concentration range of 10 to 50µg/ml for metformin hydrochloride and 20 to 80µg/ml for and sitagliptin. Simultaneous estimation of sitagliptin and simvastatin was done by extraction method with 0.1NHCl and methanol: water (40:60) as individual solvents. λmax of sitagliptin and simvastatin was found to be at 246nm and 232nm respectively. Linearity was found in the concentration range of 10 to 50µg/ml and 6 to 20µg/ml for sitagliptin and simvastatin respectively. Simultaneous estimation of diclofenac potassium and tizanidine hydrochloride uses simultaneous equations method with 240nm and 225nm as two analytical wavelengths. Distilled water was used as the common solvent. Linearity was found to be in the concentration range of 2 to 30µg/ml for diclofenac potassium and 4 to 20µg/ml for tizanidine hydrochloride.

All the methods were validated for the parameters like Linearity, Precision, Recovery, Assay, Solution stability, Robustness, and Specificity in accordance with the International Conference on Harmonization (ICH) guide lines for method development and validation.