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Simultaneous determination of aspirin, clopidogrel bisulphate and atorvastatin calcium in capsule dosage form by RP-HPLC

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A simple, rapid, reliable and precise reversed phase high performance liquid chromatographic method has been developed and validated for the simultaneous estimation of aspirin (ASP), clopidogrel bisulphate (CLP) and atorvastatin calcium (ATV) from capsule dosage form. Chromatography was carried out at 10°C on a 50 x 2.5 mm i.d., 5 µm Equisil ODS column with isocratic mobile phase 0.1% orthophosphoric acid and acetonitrile (50:50% v/v) at a flow rate of 1.0 mL/min. The detection was carried out with UV-visible detector at 245 nm. The retention times were about 1.09, 1.24 and 2.99 min for ASP, CLP and ATV, respectively. The total runtime was less than 4 min. The method was validated according to ICH guidelines and the acceptance criteria for accuracy, precision, linearity, specificity and system suitability were met in all cases. The method was linear in the range of 12-48 µg/mL for ASP, 12-48 µg/mL for CLP and 3-12 µg/mL for ATV. Limit of detection obtained were 0.03µg/mL for ASP, 0.06µg/mL for CLP and 0.07µg/mL for ATV. The percentage recoveries were found to be 98% for ASP, 96% for CLP and 93.3% for ATV. The proposed method can be used for the estimation of these drugs in combined dosage forms.

LC-MS method for the simultaneous measurement of niacin and nicotinuric acid in human plasma and its appplications in comparitive bio-availability studies

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A sensitive, specific, accurate, and reproducible HPLC/MS-method for the simultaneous quantitative determination of Niacin and its metabolite Nicotinuric acid in human plasma using Nevirapine as an internal standard is developed and validated according to USFDA guidelines. Analytes and the internal standard are separated by Liquid-Liquid extraction.

In LC MS/MS Method the extraction procedure is short and simple, it consumes small amount of solvent and biological fluid for extraction and short turnaround time when compared to earlier methods.

The chromatographic separation is achieved within 3 minutes by an isocratic mobile phase containing 0.1% formic acid in water and acetonitrile (20:80 v/v), flowing through HYPERSIL BDS,150×4.6mm, 5.0µm analytical column, at a flow rate of 1.0 mL min^{-1} .

The calibration curves are linear in the measured range between 100.1 ng/mL and 20009.7 ng/mL plasma for Niacin and 10.2ng/mL to 3000.6ng/mL for Nicotinuric acid. The overall precision and accuracy for all concentrations of quality controls and standards is better than 15%. No indications are found for possible instabilities of Niacin and its metabolite upon carrying out various stability studies such as room temperature stock solution stability, Refrigerated stock solution stability, Bench top stability, Auto sampler stability, Freeze thaw stability, Reinjection stability and Wet extraction stability in plasma. The recovery was good and also the Precision and accuracy for dilution integrity and partial volume analysis are found to be within the acceptance criteria.

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