

Abstract

Simultaneous determination of two active components in powders and tablets using NIR spectroscopy

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Introduction

A procedure for the simultaneous quantitation of metoprolol tartrate (MTP) and hydrochlorthiazide (HTZ) in powders and tablets using NIR spectroscopy was established and validated. A fast and accurate method for collecting the reference values was proposed.

Materials and Methods

Powder samples (n = 130) composed of microcrystalline cellulose, MTP and HTZ were accurately weighed in 4 ml glass vials using an analytical balance. Powder samples in the vials were mixed in a tumbler mixer and scanned in the same vials in diffuse reflectance measurement mode on an FT-NIR spectrometer over the spectral region of 5000-10000 cm⁻¹ at intervals of 4 cm⁻¹. Spectra were pretreated with first derivative and PLS1 regression was applied for correlating the spectral data with the MTP and HTZ reference values which were obtained by weighing each component individually. Two-thirds (2/3) of the samples were used for calibration and one third (1/3) for internal validation. After scanning the powders and building the prediction models, the die of the tablet press simulator-Presster[®] was filled manually with the powder mixtures from the glass vials and tablets were manufactured. They were scanned and the modeling was done. Prediction models were externally validated on the independent test sets.

Results and Discussion

The content of MTP and HTZ was accurately predicted by the models in both powder and tablet samples. The root mean square error of prediction (RMSEP) of MTP for powder samples was 1.9 mg and for tablet samples 2.1 mg (100 mg target value). HTZ predictions showed RMSEP of 1.6 mg and 1.7 mg for the powder and tablet samples respectively (25 mg target value).

Conclusion

Metoprolol tartrate and hydrochlorthiazide were simultaneously determined in powder and tablet samples with satisfactory accuracy. A gravimetric reference method was shown to be accurate, fast and convenient for feasibility studies.