Validation of chromatographic method for quantification of Budesonide in HPMC phthalate Microparticles

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PURPOSE The purpose of this study was to develop and validate a chromatographic method for quantification of budesonide in hydroxylpropylmethylcellulose phthalate (HPMCph) microparticles **METHODS** Budesonide standard was dispersed in a mixture (60:40) of acetonitrile and water (pH 3,2) yielding concentrations from 50.0 to 500.0 µg/ml. The absorbance was measured by high-pressure liquid chromatography (λ = 242 nm). Six replicates were carried out to estimate the inherent variability. Linearity and range were studied by preparing the calibration curves, which were constructed in six replicates at six concentration levels. The detection (LOD) and the quantification (LOQ) limits were calculated based on the standard deviation (SD) and the slope of the calibration graphs. Similarly, precision and accuracy were determined by analyzing three different concentrations of the sample in triplicate. The precision was verified by repeatability and intermediate precision studies. Repeatability studies were performed by analyses of three different concentrations in triplicate on the same day. Accuracy was determined for both intra- and inter-day (two different working days) variations by analyzing the samples in triplicate. The results were analyzed using linear regression and ANOVA and the main validation parameters of the method were also determined, according to ICH guidelines. **RESULTS** The calibration curve (y = 79,304x – 682,32) showed linear response with correlation coefficient "r" 0.9992. The detection limit (LOD) was 5.45 µg/ml and quantification limit (LOQ) was 16.50 µg/ml. The residues analysis demonstrated that the regression was highly significative. The relative standard deviation or coefficient of variation percentage for the budesonide concentrations analyzed demonstrated reproducibility. In order to determine the accuracy and the precision (repeatability and intermediate precision - different days) of the method, stock solutions in three different concentrations were analyzed in triplicate. The mean recovery (% ± SD) obtained was 99.26 ± 2.73 and the precision results showed mean RSD < 5%. **CONCLUSION** The validation of the analytical method showed linearity, selectivity, accuracy and precision for quantification of budesonide. **ACKNOWLEDGMENTS** The authors acknowledge the Brazilian funding agencies CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior).