PO-97 VALIDATION OF AN HPLC METHOD FOR THE DETERMINATION OF OMEPRAZOLE
S. Bozdağ, S. Çalış, M. Şumnu
Department of Pharmaceutical Technology, Faculty of Pharmacy, Hacettepe University, 06100 Ankara-Turkey

Omeprazole, is a substituted benzimidazole, has been widely used as a gastric acid secretion blocker and it degrades very rapidly in aqueous solutions at low pH values.

Validation is an important issue in pharmaceutical analysis and widely required in industrial product development and registration. This work describes the HPLC method used for quantitation of omeprazole and determination of related validation parameters.

The selective assay of omeprazole was carried out by HPLC according the to the modified method of Kobayashi et al. [1]. Precision, specificity and linearity tests were carried out for validation studies. Phosphate buffer was used in all of the concerning tests since omeprazole is known to be stable at pH 11 (300 days, at room temperature). Regarding the precision test, samples which consisted omeprazole (concentration range 100 to 1000 ng ml\(^{-1}\)) and phenacetine (internal standard, 400 ng ml\(^{-1}\)) were injected at same day (n = 6) and at different days (3 days) while the quantity of omeprazole was calculated from the ratio of peak area of the drug to the internal standard. Variation coefficients were obtained as 1.13 and 1.22% for intra-assay and inter-assay precision tests, respectively. As for the specificity test, a smooth peak was obtained by HPLC method used and no interferences were observed related to the excipients used in commercial products and in our formulations. Linearity was obtained with a correlation coefficient of 0.999 for a concentration range of 100 to 1000 ng ml\(^{-1}\) of omeprazole (internal standard, 400 ng ml\(^{-1}\)). As a result, it was concluded this method was quite reliable as it showed good repeatability, specificity and linearity.


PO-98 ANALYSIS OF DOXYCYCLINE BY CAPILLARY ELECTROPHORESIS. METHOD DEVELOPMENT AND VALIDATION
E. Castellanos Gil\(^1\), A. Van Schepdael\(^2\), E. Roets\(^2\), J. Hoogmartens\(^2\)
\(^1\)Centre de Quimica Farmaceutica, Calle 200 y 21, Atabey, Playa, P.O. Box 16042, Havana, Cuba; \(^2\)Laboratory for Pharmaceutical Chemistry and Drug Analysis, Faculty of Pharmaceutical Sciences, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium

An optimized capillary electrophoresis method for the analysis of doxycycline is described. The influence of methanol as organic modifier, buffer pH, buffer concentration, capillary length, column temperature, Triton X-100 and methyl-\(\beta\)-cyclodextrin was systematically investigated.

A central composite design was performed in order to optimize the method. The optimal separation conditions were: capillary, uncoated fused silica (40 cm total length, 32 cm effective length, ID 50 \(\mu\)m); background electrolyte, a solution of 145 mM sodium carbonate and 1 mM EDTA brought to pH 10.3-methanol (89:11, v/v); temperature, 15°C; voltage, 12 kV. The method is linear (\(r = 0.9991\)) and precise (day-to-day corrected peak area repeatability, \(n = 18\), RSD 2.4%). The limit of detection and quantitation are 0.0005 mg/ml (0.02%) and 0.0012 mg/ml (0.05%), respectively, relative to a 2.5 mg/ml solution. Six commercial samples were quantitatively analyzed. The results were compared with those established by the liquid chromatography method from the European Pharmacopoeia.

PO-99 DETERMINATION OF ASCORBIC ACID IN VEGETABLES AND SOFT DRINKS BY DERIVATIVE SPECTROPHOTOMETRY
S.M. Cetin, Z. Aydogmus
University of Istanbul, Faculty of Pharmacy, Department of Analytical Chemistry, 34452 Istanbul, Turkey

Ascorbic acid (AA) is widely distributed in plant material, e.g. fruits and vegetables. Many analytical methods have been described for its determination. The aim of the present method was to develop a simple and rapid second and third derivative spectrophotometric method for the determination of AA in garlic, green pepper, chestnut and soft drinks (trade-names: Capy and Capysun). The method is based on the measurement of the peak to peak amplitudes in the derivative spectra of the extracts. Ten percent trichloroacetic acid was found to be the most suitable solvent for the extraction. The extrema at 253.6 and 259 nm in the second derivative spectrum were used for the determination of AA in garlic while its determination in green pepper, chestnut and soft drinks (trade-names: Capy and Capysun) was based on the extrema at 257.5 and 261.6 nm in the third derivative spectrum. The calibration graphs were linear over the range of 2-10 \(\mu\)g/ml with a correlation coefficient of 0.9999. The relative standard deviation was 0.98-1.5%. Recoveries over 92% were found using the standard addition method.

PO-100 LIQUID CHROMATOGRAPHY OF TROLEANDOMYCIN
H.K. Chepkwony, E. Roets, J. Hoogmartens
Laboratorium voor Farmaceutische Chemie en Analyse van Geneesmiddelen, Faculteit Farmaceutische Wetenschappen, Katholieke, Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium

Troleandomycin is a semi-synthetic antibiotic obtained from oleandomycin by complete acetylation. In the French Pharmacopoeia the purity of the product is checked by thin layer chromatography and the activity is determined by microbiological assay.

No liquid chromatography (LC) method is described until