

ABSTRACT

University	Charles University in Prague
Faculty	Faculty of Pharmacy in Hradec Králové
Department	Department of Pharmaceutical Chemistry and Drug Control
Candidate	Mgr. Peter Kalafut
Supervisor	Prof. RNDr. Jiří Klimeš, CSc.
Title of Doctoral Thesis	The possibilities of utilization of alternative stationary phases in pharmaceutical analysis.

The significance of metal-based HPLC stationary phases, mainly the zirconia, has arisen, recently. Zirconia-based phases came in the awareness of professional community mainly thanks to the versatile stability in different mobile phases and high temperatures as well as to the ability to provide very different selectivity as compared to common silica concurents.

The theoretical part of this doctoral thesis provides general overview of liquid chromatography and also describes the recent instrumentation. The main part is devoted to a bibliographic search dealing in detail with the properties of zirconia-based stationary phases and consequent applications in the analytical practice.

The fundamental of this thesis is represented by the experimental part, which studies the chromatographic behaviour of some chosen types of zirconia-based stationary phases and their potential use in HPLC analysis emphasizing especially on the pharmaceutical-analytical practice. The chromatographic behaviour is described from the viewpoint of the influence of changes in the mobile phase composition (the type and amount of organic solvent, the type, pH and concentration of a buffer) as well as the temperature on the resulting separation.

The experimental part of this doctoral thesis is divided into three individual sections. The title of the first experimental work is „An innovative approach to the analysis of 3-[4-(2-methylpropyl)phenyl]propanoic acid as an impurity of ibuprofen on a carbon-coated zirconia stationary phase“. Herein, a new HPLC method for the determination of the pharmacopoeial impurity F of ibuprofen was developed and validated showing the limit of detection at 2.3 % of

the admissible amount. The second paper is entitled „The influence of a carbon layer deposited on a zirconia surface on the retention of polar analytes in an organic rich mobile phase“. In this work the ability of hydrophobic Zr-CARB column to retain through multimodal retention mechanism some polar compounds under HILIC conditions was demonstrated. The title of the last experimental chapter is „The retention behavior of acidic, basic and neutral pharmaceuticals on the deactivated polybutadiene zirconia phase“. Based on the comparison with undeactivated polybutadiene zirconia phase, approx. 2.5-fold higher hydrophobicity of deactivated stationary phase was established and also small impact on the selectivity of a separation was observed.