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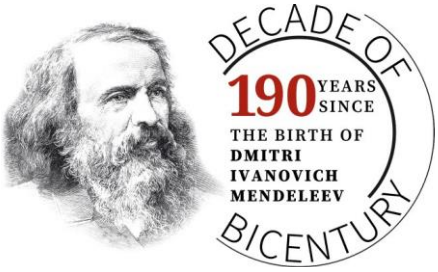

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STUDY OF THE RETENTION MECHANISM FOR ORGANIC AND INORGANIC IONS IN HILIC WITH SILICA- AND POLY(STYRENE-DIVINYLBENZENE)-BASED STATIONARY PHASES

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Hydrophilic interaction liquid chromatography (HILIC) is a modern promising method for the separation and determination of polar compounds. This method attracts analysts' attention due to its ability of simultaneous separation of positively and negatively charged analytes. It is necessary to understand the retention mechanism for various substances in order to select the optimal conditions for such analyses. The retention mechanism in HILIC is complex and still not fully understood. It is cosidered that adsorption, partitioning, and electrostatic interactions may contribute simultaneously to retention. In the case of mixed-mode stationary phases based on hydrophobic polymer substrate, additionally hydrophobic and π-π interactions take place [1]. Understanding the processes occurring in the chromatographic system makes it possible to develop the method. It allows to expand its theoretical basis, to find new approaches for characterization of adsorbents, and to control the separation efficiency and selectivity in HILIC mode.

In this work, a set of test ions was selected: p-toluenesulfonate, alkylsulfonates, chloride, nitrate, single- and double-charged inorganic cations. Their retention mechanism was studied for silica- and poly(styrene-divinylbenzene)-based stationary phases with predominant anion-exchange properties. A universal evaporative light-scattering detector was chosen to provide the required sensitivity. Mixtures of ammonium-acetate buffer solution, pH 4.7 and acetonitrile were used as mobile phases. The contribution of electrostatic and hydrophilic interactions to the retention of model analytes was evaluated by varying the concentration of eluting ion and the fraction of organic solvent in the mobile phase.

A decrease in the contribution of electrostatic interactions with decreasing hydrocarbon chain length was shown on silica phases for methane-, ethane-, propane-, and butanesulfonate anions. The opposite dependence was demonstrated for poly(styrene-divinylbenzene)-based adsorbents, with the lowest contribution observed for p-toluenesulfonate ion. This compound is used as a marker of anion exchange selectivity in the Tanaka HILIC test [2], but according to the data obtained and literature [1] it is not suitable for mixed-mode stationary phases due to the significant contribution of hydrophobic and π-π interactions into its retention. More hydrophilic anions like methanesulfonate may represent an alternative to this parameter.

Varying the eluting ion concentration in the mobile phase, it was shown that electrostatic repulsion governed the retention of single-charged inorganic cations (Li^+, Na^+, K^+) when using stationary phases of both types. Nevertheless, all the studied cations had reasonable retention factors on the anion-exchangers with the elution order opposite to that in cation-exchange chromatography due to partitioning. This process significantly prevailed over the repulsion of cations from the positively charged functional layer of the stationary phase [3]. As a result, it was possible to separate five monovalent inorganic ions both cations and anions using amino-modified stationary phases in a single run.

References

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