The use of HPLC and capillary zone electrophoresis for the study of inorganic complexes in solution

Olga V Shuvaeva1,3, Artem A Zhdanov2, Tamara Romanova1,3 and Victoria V Volchek1
1Nikolaev Institute of Inorganic Chemistry - SB RAS, Russia
2Boreskov Institute of Catalysis - SB RAS, Russia
3Novosibirsk State University, Russia

It is known that the chemistry of polyoxometalates (POM) as well as of polynuclear complexes (PNC) is one of the fastest growing areas of modern inorganic chemistry. These substances are widely used in many areas of modern science as catalysts, pharmacology and medicine, in the fields of micro- and nanotechnology. Frequently as a result of their synthesis, the composition of solid phase and solution are substantially different. The main problems of these compounds study is their instability in solution resulting in the presence of the different equilibrium chemical forms. On the other hand they demonstrate the different trends in the changing of their structure, depending on the medium conditions. There are a number of problems in the study of such compounds. Firstly, it is the presence of variety of chemical forms of indefinite composition presenting in solution; secondly, their limited stability which complicates the choice of separation conditions. Third, similarity in composition and structure of this type of complexes often appears in resembling UV-VIS absorption spectra, creating difficulties for identification. Finally, the lack of individual compounds also complicates the identification of speciation patterns. The observation of such processes requires special direct techniques and approaches, so the data interpretation is obstructed. In frame of the present work the separation possibilities of POM on the base of molybdenum, vanadium and phosphorus, and hydroxo-complexes of rhodium with the similar structure and composition using capillary electrophoresis (CE) and HPLC were estimated. The data obtained are in consistency with each other and in some cases confirmed using NMR spectroscopy. Thus, the approaches for the study of the mixtures formed in self-assembly reactions of POM and in the process of rhodium polynuclear forms generation were developed. It was shown that capillary electrophoresis and HPLC may be successfully applied for the study of the state of inorganic complexes in solution. However, the most appropriate approach for the study of these compounds is the use of complex of techniques such as HPLC, CE and spectroscopic methods such as NMR, et al. The use of two different modes of pre-capillary and in-capillary was also examined and compared. As a result the best-compromise conditions for the separation of the mixtures containing the reactants, intermediates and the reaction to achieve the best effectiveness, symmetry, and peak areas were optimized. It was shown that in-capillary mode is more informative than pre-capillary one for the study of the complex compounds formation process.

olga@niic.nsc.ru