The Regularity of retention of Molybdenum Heteropoly Acids of Silicon and Phosphorus.

D.B. Dubovik, AV. Ivanov, P.N. Nesterenko, O.A. Shpigun.

Moscow State University, Department of Chemistry, 119899 Moscow, Vorobyevy Gory.

The chromatographic behavior of molybdenum heteropoly acids of silicon(MSA) and phosphorus (MPA) in form ion-associate with tetrabutiammonium bromide (TBAB) was researched in IP HPLC (Si- C_{18} , detection λ =310 nm). The using eluent contained: acetic buffer, sulphuric and perchloric acids (pH-regulators); TBAB (ion-pair reagent). The optimal conditions of determination silicon and phosphorus in combined presence are:

- 1. Eluent: 60% AcN; 0,1 M acetic buffer (pH=4.0), 0,8+1.2 mM TBAB
- 2. Detection: $\lambda=310$ nm.

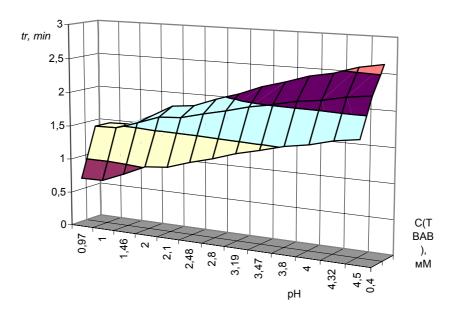


Fig.19. The influence pH and TBAB concentration on retention time of MSA. (eluent: 60% acetonitrile, 0.1 acetic buffer, HCl 3.94 g/l Na_2SO_4 , C_{Si} =1 mkg/ml).

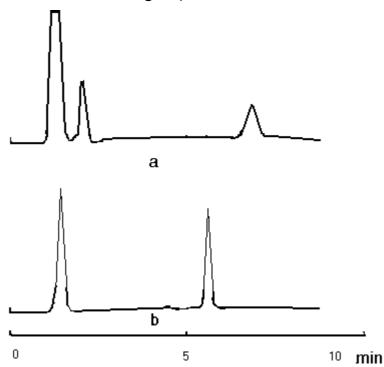


Fig.23. The chromatograms of MSA and MPA, formed in solution ($V_{a.p.}$ =25 ml C_{MSA} =3 mkg/ml, C_{MPA} =6 mkg/ml), (a) and syntesid (b) (C_{MSA} =3 mkg/ml, C_{MPA} =6 mkg/ml; eluent: 60% acetonitrile, H_2SO_4 3.94 g/l Na_2SO_4 , λ =310 nm; column - Mightysil RP-18 150×4.6 mm, \varnothing 5mkm). Conditions of concentrating: 100 ml water solution D=20+50, 1 M H_2SO_4 ;

 $2.5*10^{-4}$ M TBAB, C_{Mo} = $4.6*10^{-2}$ M, glass wool was used as sorbent (1 g); ion-associates GPA with TBAB were resorpted by AcN then water was added ($H_2O:AcN=1:1$). The volume of concentrate was $2\div4$ ml.