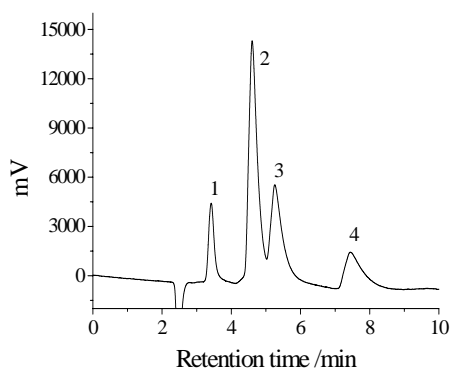


The present work describes the preparation and application of a new kind of stationary phase based on ionic liquid that is confined on silica gel. The ionic liquid used in this article is 1-allyl-3-hexyl imidazolium tetrafluoroborate ([AHIm]BF₄) that contains a long alkyl chains and an allyl group as the anchoring group. It was prepared according to the previous report⁷. The activated silica was modified with a silane-coupling agent, 3-mercaptopropyltrimethoxysilane (MPS). [AHIm]BF₄ reacted with MPS-modified silica in the presence of azodiisobutyronitrile (AIBN) as the initiator *via* the radical chain transfer addition^{8,9}. The reaction procedures are illustrated in **Scheme 1**. The obtained silica was packed into a 150×4.6 mm I.D. stainless steel column by the conventional slurry-packing procedure.

Figure 1 shows the representative chromatogram of ephedrines on ionic liquid modified silica. It can be seen that ephedrines can be efficiently separated. Comparatively, we have demonstrated that ephedrines were separated poorly on the C₁₈ column when using similar mobile phase as employed on the ionic liquid-based phase but without the addition of ion-pair agent¹⁰ or triethylamine¹¹. The figure also shows that the retention times are obviously decreased but the resolution was improved compared with that on C₁₈ column. Moreover, a low content (1%) of organic solvent (methanol) in the mobile phase is needed for effective separation.

Figure 1 Representative chromatogram of ephedrines



Chromatographic conditions: column, ionic liquid modified silica (150×4.6 mm I.D.); mobile phase, 1% methanol in 0.05 mol/L KH₂PO₄ at pH 3.0; flow-rate, 0.8 mL/min, detection, UV at 220 nm; injection volume, 20 μL. Peaks: 1. norephedrine, 2. ephedrine, 3. pseudoephedrine, 4. methylephedrine.

The separation mechanism seems to involve multiple interactions between the confined ionic liquid on the surface and solutes that are mainly electrostatic interaction, and ion-exchange interaction between phosphate buffer and basic solutes. Meanwhile, the confined ionic liquid effectively masked the free silanols on the silica surface, which decreased the nonspecific adsorption between the basic compounds and the free silanols.

In summary, the ionic liquid modified silica has special selectivity on the separation of ephedrines. The article provides a novel method for the modification of stationary phase that can be further studied.

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