



1,3-Alternate 25,27-dibenzoiloxo-26,28-bis-[3-propyloxy]-calix[4]arene-bonded silica gel as a new type of HPLC stationary phase

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Abstract

A new 1,3-alternate 25,27-dibenzoiloxo-26,28-bis-[3-propyloxy]-calix[4]arene-bonded silica gel stationary phase (*1,3-Alt* CalixBn) has been prepared and used for the separation of aromatic positional isomers by high-performance liquid chromatography (HPLC). The effect of organic modifier content and pH of the mobile phase on retention and selectivity of these compounds were studied. Application examples were provided for separation of purine and pyrimidine bases and non-steroidal anti-inflammatory drugs.

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1. Introduction

The main goal of investigation of high-performance liquid chromatography (HPLC) is searching more efficient stationary phases and optimisation of the separation process. Reversed-phase chromatography (RPC) is the first choice for most regular samples. RPC is typically more convenient and rugged than other types of liquid chromatography (LC) and is more likely to result in a satisfactory final separation. Useful changes in selectivity and sample retention can be achieved by selecting appropriate type of stationary phase. There are many contemporary commercially available reversed-phase columns, e.g. RP-C₁₈, RP-C₈, RP-Phenyl and others. Recently, calixarenes also have attracted attention of many researchers because they are able to form reversible complexes with neutral as well as charged molecules [1–3]. In last years, the potential of this class of macrocyclic compounds has been shown for several applications in gas chromatography [4–7], capillary electrophoresis [8–11], solid-phase extraction [12] and overall high-performance liquid chro-

matography. Calix[*n*]arene-bonded (*n* = 4, 5, 6, 8) silica gel high-performance liquid chromatography stationary phases were used for separation of many inorganic and organic compounds, e.g. metal ions [13,14]; aromatic positional isomers [15–21]; uracil derivatives and estradiol epimers [17]; proline-containing peptides [22]; water soluble vitamins [23]; PAHs [18,20,24,25]; amino acid esters [25]; quinolones and sulphonamides [26]; nucleosides [16,27–29]; barbituric acids; benzoxepin and thioxanthene derivatives [25,30,31]. Several previous works have shown that calixarene-bonded stationary phases in a cone conformation are excellent in reversed-phase chromatography and exhibit promising application in HPLC. Recently, we have reported synthesis of new 25,27-dipropoxy-26,28-bis-[3-propyloxy]-calix[4]arene-bonded silica gel stationary phase in 1,3-alternate conformation (*1,3-Alt* CalixPr) and we have confirmed that this phase possess high selectivity toward selected positional aromatic isomers [32]. In this paper, we described the synthesis of new 1,3-alternate 25,27-dibenzoiloxo-26,28-bis-[3-propyloxy]-calix[4]arene-bonded silica gel stationary phase (*1,3-Alt* CalixBn) and its application to resolution of compounds of very similar structure (e.g. aromatic positional isomers), purine and pyrimidine bases as well as

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