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The Optical Resolution of Chiral Tetrahedrone-type Clusters Containing SCoFeM (M=Mo or W) Using High Performance Liquid Chromatography Chiral Stationary Phase

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Abstract: Amylose tris (phenylcarbamate) chiral stationary phase (ATPC-CSP) was prepared and used for optical resolution of clusters **1** and **2**. n-Hexane/2-propanol (99/1; v/v) were found to be the most suitable mobile phase on ATPC-CSP.

Keywords: Optical resolution, amylose tris (phenylcarbamate) chiral stationary phase, chiral tetrahedrone –type cluster.

Optical resolution using high performance liquid chromatography chiral stationary phases has been widely used in many modern research fields such as asymmetric synthesis, asymmetric catalytic and separation of racemic pharmaceuticals. In the past years, various chiral stationary phases have been developed and have been commercially available[1]. Among them, polysaccharide based phases are the widely used CSPs. A wide variety of enantiomeric compounds including chiral aromatic alcohols, enantiomeric amides, pyriproxyfen, amino alcohols, *etc.* have been separated on these CSPs. Optical resolution of chiral tetrahedrone-type cluster using ATPC-CSP has been less reported. The chiral tetrahedrone-type clusters have been attracting intensive attention because of their potential applications in catalysis as well as the novelty and versatility of their reactions and structures. However, the optical resolution of chiral clusters has an important problem for the further study of the catalyzed properties of these chiral clusters[2].

The aim of this study is to present a direct separation of the novel chiral tetrahedrone-type clusters on amylose-tris (phenylcarbamate) chiral stationary phase (ATPC-CSP). ATPC-CSP was prepared according to previous studies[3] with small modification. The structures of clusters **1** and **2** are shown in **Figure 1**.

The optical resolution of clusters **1** and **2** on ATPC-CSP was obtained using n-hexane as the mobile phase with 2-propanol as mobile phase modifier. The retention factors (k'), enantioselectivity factor (α) and resolution (R_s) of all the solutes decreased on increasing 2-propanol concentration from 1% to 10% in mobile phase composition.

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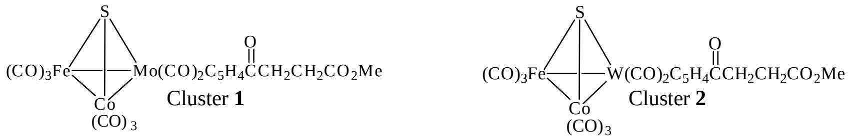
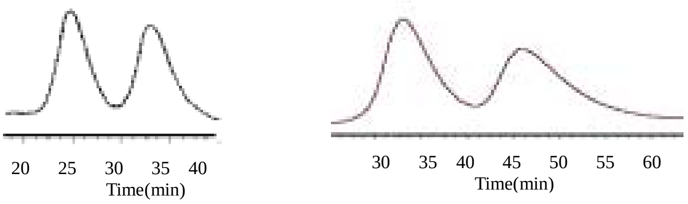
Figure1 The structures of chiral tetrahedrone clusters



Cluster **1** Cluster **2**

For cluster **1** the parameters $(k'\alpha, R_s)$ were (13.5, 1.35, 1.05), (10.33, 1.12, 0.54), (4.10, 1.08, 0.28) and (2.39, 1.00, 0.00) with mobile phase n-hexane/ 2-propanol (v/v) 99/1, 97/3, 95/5 and 90/10, respectively, at a flow rate of 1.0 mL/min. For cluster **2** the parameters $(k'\alpha, R_s)$ were (19.22, 1.33, 0.84), (15.07, 1.12, 0.67), (7.79, 1.10, 0.48) and (3.28, 1.10, 0.32) with mobile phase n-hexane/ 2-propanol (v/v) 99/1, 97/3, 95/5 and 90/10, respectively, at a flow rate of 1.0 mL/min. It suggested that the steric fit in the cavity of helical structure of the amylose was a primary contribution for chiral separation, and the hydrogen bonding interaction between C=O function groups of clusters and –NH group on ATPC-CSP had significant impact on optical resolution for chiral clusters. The chromatograms of cluster **1** and cluster **2** were shown **Figure 2**

Figure 2 The Chromatograms of chiral tetrahedrone clusters



Cluster **1** Cluster **2**

Mobile phase: n-hexane:2-propanol=99:1(v/v), flow rate:1.0 mL/min, detector length:254 nm, 0.02A.U.F.

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