# Resolution of DL-Pantolactone with Ethyldiamine Bridged Dimer Permethy β -Cyclodextrin as GC Stationary Phase

Dan Ni TAO[1], Xue Yan SHI[2], Jun Ling GU[1], Ruo Nong FU[1*]

[1] College of Chemical Engineering and Materials Science, Beijing Institute of Technology, Beijing 100081
[2] Department of Application Chemistry, Chinese Agriculture University, Beijing 100094

**Abstract:** Ethyldiamine bridged dimer permethy- β -CD and other cyclodextrins were used as GC stationary phase to resolute DL-pantolactone. It is found that this CD dimer has a good selectivity for DL-pantolactone.

**Keywords:** Cyclodextrin dimer, gas chromatography, stationary phase.

Cyclodextrin dimers (CD dimers) are synthesized by linking two single cyclodextrins with a difunctional spacer. They have been used as enzymatic models for catalytic purpose in the last 20 years, but this separation behaviors as GC stationary phase have been investigated recently[1]. Ethyldiamine bridged dimer permethyl- β -CD is synthesized only by linking two single cyclodextrins with an ethyldiamine molecular, which is first used as GC stationary phase. DL-Pantolactone, DL-dihydro-3-hydroxy-4,4-dimethyl-2(3H)-furanone, was found in the degradation product of pantothenic acid from liver by Willimas in 1940. It is an important intermediate in the synthesis of pantothenic acid, D-pantothenyl alcohol and D-pantothenic acid calcium salt, which are usually used in biochemical study[2]. So far the chiral resolution of DL-pantolactone by gas chromatography has not been reported. In this work, the colume coated by ethyldiamine bridged dimer permethyl- β -CD exhibited good selectivity for DL-pantolactone .

## Column Preparation

Fused-silica capillary tubes (10 m×0.25 mm I.D.,Yong Nian Optical Fibre factory, Hebei province, China) were roughened by depositing sodium chloride onto their inner wall, the columns were then statically coated at 35℃ with 0.5% (w/v) solution of the stationary phase in dichloromethane. Following with conditioning under a slow nitrogen flow at 40℃, 80℃, 120℃, 160℃ for 1h each and finally at 200℃ for 5h.

## Column Evalution

A Model 3700 gas chromatography (Beijing Analytical Instrument Factory, Beijing,

China) equipped with a capillary split injection system and flame-ionization detector (FID) was used. Carrier gas was high purity nitrogen. The injection split ratio was 72:1. The injector and detector temperature were 250℃ and 260℃ respectively.

## Results and Discussion

Six columns coated by various CDs stationary phases were chosen. **Table 1** lists the relative retention (α) and resolution (R) values of DL-pantolactone on them. **Figure 1** and **Figure 2** show that DL-pantolactone was separated by ethyldiamine bridged dimer permethyl-β-CD and heptakis (2,6-di-o-benzyl-3-o-allyl)-β-CD bonded polysiloxane. Ethyldiamine bridged dimer permethyl-β-CD linking two single cyclodextrins with ethyldiamine spacer gave better selectivity for DL-pantolactone than the rest.

**Table 1** The relative retention (α)and resolution (R) values of resolving DL-pantolactone

| Stationary phase | T/°C | R | α |
|---|---|---|---|
| Ethyldiamine bridged dimer permethyl-β-CD | 140 | 0.943 | 1.09 |
| Heptakis (2,6-di-o-benzyl-3-o-allyl)-β-CD bonded polysiloxane | 120 | 0.510 | 1.05 |
| Mono (2,6-di-o-pentyl-3-o-allyl)-hexakis (2,6-di-o-pentyl-3-o-methyl)-β-CD | 120 | 0.000 | 1.00 |
| Mono (2,6-di-o-pentyl-3-o-propanyl-3')-hexakis (2,6-di-o-penntyl-3-o-methyl)-β-CD | 120 | 0.000 | 1.00 |
| 2,6-di-o-pentyl-3-o-benzyl-β-CD | 120 | 0.000 | 1.00 |
| Tri (2,6-di-o-pentyl-3-o-allyl)-hexakis (2-o-pentyl-2-o-allyl-6-o-penylcarbamate)-β-CD | 120 | 0.000 | 1.00 |

**Figure 1** chromatogram of DL-pantolactone on column 1 at 140°C

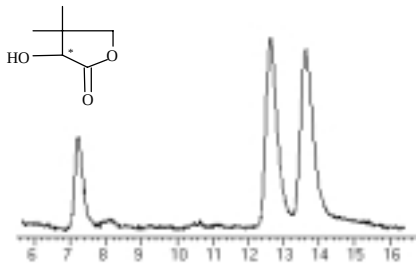
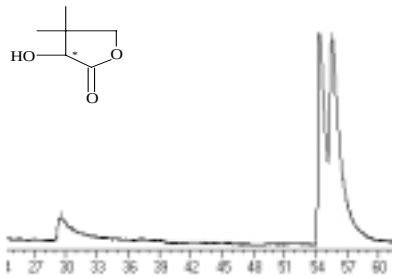



**Figure 2** Chromatogram of DL- pantolactone column 2 at 120℃.




## Reference

1. X. Y. Shi, Y. Q. Zhang, J. H. Han, R. N. Fu, *Chromatographia*, **2000**, *52*, 200.
2. The Merk Index, 9th Eds., M. Windholz, Eds., Merk&Co., Inc., Rahway, N. J. **1976**, pp 2435, 6811

Received 20 November, 2000