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A Good GC Stationary Phase for Separation of Xylene Isomers

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Abstract: An excellent separation of xylene isomers is reported using 2, 6-O-di-pentyl-3-O-acetyl-β-CD as capillary stationary phase. The optimum conditions for the separation are also discussed.

Keywords: Xylene isomers separation; capillary stationary phase; optimum conditions for separation.

The separation of xylene isomers using GC is a much difficult task for a long time, on the other hand they are very important industrial materials, so finding a good stationary phase has been an interesting work. An excellent separation of xylene isomers was obtained by using 2, 6-di-O-pentyl-3-O-acetyl-β-cyclodextrin synthesized in our lab as the capillary stationary phase. The chromatograms and separation factor (α) are better than those reported in the literature. We hope this will provide a good method for the separation of xylene isomers for practical use.

Preparation of Heptatakis (2, 6-di-O-pentyl-3-O-acetyl-β-cyclodextrin) : β-cyclodextrin was subjected to pentylation with sodium hydroxide and n-pentyl bromide in DMSO according to the method of Ciucanu and Kerek[1]. The resulted heptakis (2, 6-di-O-pentyl) -β-cyclodextrin was further acetylated with acetic anhydride and dry pyridine by heating the solution at 80℃ for 3 hours. After removing the solvent in vacuum the residue was purified by column chromatography on silica gel.

Table I gives the chromatographic data at different conditions.

Table I shows that in thetemperaturerange of 90℃ to 110℃ changes of temperature and carrier gas velocity have little effect on the separation factor (α) . But when the temperature was out of this range, either lower α values or worse peak shapes appeared. It indicates that the optimum temperature range for xylene isomers separation is 90-110℃.

Other stationary phases listed in **Table II** either give smaller α values than those in **Table I** or could not separate three xylene isomers completely.

Table I. Capacity factor（k）and separate factor (α) for xylem isomers

Temp. (℃)	Velocity of carrier gas (cm/s)	k	α *	Temp. (℃)	Velocity of carrier gas (cm/s)	k	α
		m 5.22				m 1.52	
90	17.86	p 6.07	1.16	110	18.82	p 2.82	1.16
		o 6.91	1.14			o 3.19	1.13
		m 6.06				m 2.30	
	16.20	p 7.01	1.16		15.77	p 2.67	1.16
		o 7.99	1.14			o 3.04	1.14
		m 3.24				m 2.15	
100	14.23	p 3.76	1.16	120	17.16	p 2.46	1.15
		o 4.28	1.14			o 2.75	1.12
		m 2.97	1.16			m 2.61	
	19.23	p 3.46	1.14		22.03	p 2.96	1.13
		o 3.95				o 3.32	1.12

- The values listed are for two adjacent eluting isomers.

Table II. Related data reported in literature

Ref.	Stationary phase	Temp. (℃)	k	α	Ref.	Stationary phase	Temp. (℃)	k	α
	Crown-ether		m 2.00			Peralkylated β-CD		p 1.517	
4	bonded β-CD	80	p 2.00	1.00	3		80	m 1.558	1.03
			o 2.67	1.33				o 1.817	1.17
			m 2.46					m /	
4	Resorararenes[4]	80	p 2.46	1.00	2	Heptakis (2, 6-O-di-benzyl-3-O-acetyl) - β-CD	60	p /	1.00
			o 3.17	1.29				o /	1.19
	Metal complex		m 0.80						
4	liquid crystalline*	45	p 1.33	1.66					
			o 1.44	1.19					

*A baseline separation for m- and p- xylene isomers was not obtained.

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