

Available online at www.sciencedirect.com



Journal of Chromatography A, 990 (2003) 63-73

JOURNAL OF CHROMATOGRAPHY A

www.elsevier.com/locate/chroma

Electrophoretic enantiomer separations at high pH using the new, single-isomer octakis(2,3-dimethyl-6-O-sulfo)- γ -cyclodextrin as chiral resolving agent

M. Brent Busby, Omar Maldonado, Gyula Vigh*

Department of Chemistry, Texas A&M University, College Station, TX, 77842-3012, USA

Abstract

The latest, single-isomer, sulfated γ -cyclodextrin, the sodium salt of octakis(2,3-dimethyl-6-*O*-sulfo)- γ -cyclodextrin that is stable in basic media was used to separate the enantiomers of neutral, weak acid and weak base analytes by capillary electrophoresis in high pH aqueous background electrolytes. The effective mobilities and separation selectivities were found to follow trends similar to those observed earlier in acidic aqueous background electrolytes. Octakis(2,3-dimethyl-6-*O*-sulfo)- γ -cyclodextrin proved to interact with all three analyte types less strongly than other single-isomer sulfated cyclodextrins do under comparable conditions.

© 2002 Elsevier Science B.V. All rights reserved.

Keywords: Enantiomer separation; Cyclodextrins; Sulfated cyclodextrins; Octakis(2,3-dimethyl-6-O-sulfo)-y-cyclodextrin

1. Introduction

Reviews in the latest special issue of Electrophoresis dedicated to the capillary electrophoretic (CE) separation of enantiomers [1,2] show that single-isomer sulfated cyclodextrins (CDs) play a unique role among the most frequently used chiral resolving agents because, in addition to affording excellent separations, they facilitate fundamental physicochemical studies. With the recent synthesis [3] of the sodium salt of octakis(2,3-dimethyl-6-Osulfo)- γ -cyclodextrin (ODMS), there are now two families of single-isomer, sulfated cyclodextrins with

E-mail address: vigh@mail.chem.tamu.edu (G. Vigh).

isomeric purities greater than 95 mol%, a β -CD family and a γ -CD family, whose primary hydroxy groups on the 6-C atoms of the respective glucopyranose subunits have been completely sulfated, while their secondary hydroxy groups on the 2- and 3-carbon atoms were kept unchanged [4,5] or modified with acetyl groups [6,7] and methyl groups [3,8]. All of these single-isomer, sulfated CDs have been successfully used [3-9] in acidic background electrolytes (BGEs). However, due to their base stability, the single-isomer, sulfated CDs that carry hydroxy and methyl groups can also be used in high pH BGEs to probe additional separation selectivities [3,5,10-12]. This paper presents our first results with the latest, base-stable, single-isomer, sulfated CD, ODMS, in pH 9.5 aqueous BGEs for the CE separation of the enantiomers of neutral, weak acid and weak base analytes.

^{*}Corresponding author. Tel.: +1-979-845-2456; fax: +1-979-845-4719.

^{0021-9673/02/\$ –} see front matter @ 2002 Elsevier Science B.V. All rights reserved. doi:10.1016/S0021-9673(02)01797-1

2. Experimental

The chemicals used for the preparation of the BGEs, ethanolamine and methanesulfonic acid (MSA), were purchased from Aldrich (Milwaukee, WI, USA), while ODMS was synthesized and analytically characterized in our laboratory [3] and is now commercially available [13] from Antek (Houston, TX, USA). All chiral analytes listed in Fig. 1 were obtained from either Aldrich, Sigma (St. Louis, MO, USA), Wiley Organics (Coshocton, OH, USA) or Research Diagnostics (Rockdale, MD, USA). Dimethylsulfoxide (DMSO) was purchased from EM Science (Gibbstown, NJ, USA). All solutions were prepared with Milli-Q water (Millipore, Milford, MA, USA). The stock buffer was 20 mM ethanolamine titrated to pH 9.5 with MSA. The stock buffer was used to dissolve the calculated amounts of ODMS to obtain the 5-40 mM ODMS BGEs. Samples were dissolved in the respective BGEs at an approximate concentration of 0.5 mM.

The enantiomer separations were obtained with a P/ACE 5000 CE system (Beckman-Coulter, Fullerton, CA, USA). Its variable wavelength UV detector was operated at 214 nm, its thermostat at 20 °C. All separations were carried out on uncoated, fusedsilica capillaries with an I.D. of 25 µm, an O.D. of 150 μ m, a total length, L, of 46 cm, and an injectorto-detector length, L_d, of 39 cm (Polymicro Technologies, Phoenix, AZ, USA). The samples were pressure injected in triplicate with 1 p.s.i. nitrogen for 1 s (1 p.s.i.=6894.76 Pa). The external mobility marker method [14] indicated that DMSO did not appreciably complex with ODMS. Thus, DMSO could be used as electroosmotic flow (EOF) marker and was co-injected with each sample to determine the actual electroosmotic flow mobility, μ_{EOF} .

In order to obtain meaningful effective electrophoretic mobility values, all measurements were made within the linear region of the respective Ohm's plots: power dissipation was maintained by varying the applied potential (U_{appl}) between 7 and 12 kV. The effective mobilities of the enantiomers $(\mu_1^{eff} \text{ and } \mu_2^{eff})$ were obtained as $\mu_1^{eff} = \mu_1^{obs} - \mu_{EOF}$, the separation selectivities, α , as $\alpha = \mu_1^{eff} / \mu_2^{eff}$ (where subscript 2 arbitrarily refers to the enantiomer whose effective mobility in the 5 mM ODMS BGE was smaller), and the normalized electroosmotic flow mobility values, β , as $\beta = \mu_{EOF}/\mu_2^{eff}$ [15]. Typical μ_{EOF} values decreased from $+40 \cdot 10^{-5}$ cm² V⁻¹ s⁻¹ to $+18 \cdot 10^{-5}$ cm² V⁻¹ s⁻¹ as the ODMS concentration of the BGE was increased from 5 to 40 m*M*.

3. Results and discussion

ODMS was used for the CE separation of the enantiomers of 65 neutral, weak acid and weak base analytes whose structures are shown in Fig. 1. The effective mobilities, μ (in 10⁻⁵ cm² V⁻¹ s⁻¹ units), the separation selectivities, α , the measured peak resolution values, R_s , the corresponding dimensionless EOF mobility values, β , and the injector-to-detector effective potential drop values, U ($U = U_{appl}L_d/L_t$, in kV units), for the neutral, weak acid and weak base enantiomers are listed in Tables 1–3, respectively. When a mobility value could not be measured (due to interference by a system peak or DMSO), the tables list NA (not available).

For the neutral analytes tested, the anionic effective mobilities increased very slightly, in the 0 to -2.6 mobility unit range, as the ODMS concentration was increased from 5 to 40 m*M* (Table 1). This indicates that interactions between typical neutral analytes and ODMS are much weaker than what were observed with the other single-isomer 6-*O*-sulfo CDs [3–12].

For the weak acid analytes tested, the anionic effective mobilities are higher at low ODMS concentrations and become lower as the ODMS concentration is increased. This phenomenon is due to the interplay of their increasing complexation with ODMS and the mobility suppressing effects of the increasing ionic strength of the BGE [16]. Separation selectivities were generally closer to unity than those obtained at low pH for the same analytes [3] indicating that weak acid enantiomers and ODMS follow mostly desionoselective separation patterns [15]. The only exception is fenoprofen, for which $\alpha = 1.02$, and baseline resolution could be obtained at 40 mM ODMS.

For weak bases, the effective mobility patterns are similar to those observed in the low pH BGEs [3] indicating that pH 9.5 is not high enough to render most of the weak base analytes tested neutral. The



N02: 2-Phenylbutanol



N10: Ethylmandelate

N15: Methylmandelate



N19: Pantolactone



N20: 1-Phenylethylpropionate



N21: 1-Phenybutanol



N22: 3-Phenylbutyraldehyde



N26: 2-Phenyl-2-pentanol



N36: 1-Indanol



N24: Styrene glycol



N30: trans-2-Phenyl-1-cyclohexanol



ÓН

N25: 1-Phenylpentanol

N34: 1-Phenylpropanol



N38: 2-Phenyl-2-butanol



N39: 2-Phenylpropionaldehyde



N40: 2,3-Epoxypropyl-4-methoxyphenylether

Fig. 1. Structures of the neutral, weak acid and weak base enantiomers used in this study.



A02: trans-2-Phenyl-1-cyclopropane carboxylic acid







A04: Carprofen





A03:
a-Methyl-a-phenylsuccinimide



A09: Ethosuximide

A16: 5-(4-methylphenyl)-5-phenylhydantoin



A27: Ketoprofen

A31: Naproxen

ЭH

ЮH

→ → → OH

A23: Ibuprofen



A28: Mandelic acid

F OH

A26: Flurbiprofen



A30: 2-phenyl-3-methylvaleric acid (R,R),(R,S),(S,R),(S,S)



A36: 2-Phenylpropionic acid

Fig. 1. (continued)

weak bases tested here can be divided into two major groups: the first is the group of weakly binding weak bases whose effective mobilities remain cationic over the entire 5-40 mM ODMS concentration range, the second is the group of strongly binding weak bases whose effective mobilities become anionic at relatively low ODMS concentrations and pass (or approach) shallow anionic mobility maxima as the ODMS concentration is increased. This effective mobility pattern is again the result of the interplay between the increasing degree of complexation and the differential depression of the mobilities of the



B02: Alprenolol

B09: Atropine



B04: Halostachine

B10:Bupivacaine

CF





B06: Phenylethanolamine



B08:Atenolol

B11: Bupropion

B13: 4-Chloroamphetamine



B22: Ketamine

B14: Chlophedianol

.OH







B20: Hemicholinium-15





B25: Mepenzolate

B26: Metanephrine



B23: Ketotifen



B28: Methoxyphenamine

B30: Metaproterenol

Fig. 1. (continued)

free and complexed forms of the weak base analyte by the increasing ionic strength of the BGEs. Favorable separation selectivities were found only for 14 of the 36 weak bases tested, fewer than in the low pH BGEs [3].

However, diltiazem could only be separated in the high pH BGE, indicating that the diltiazem-ODMS

system follows a desionoselective separation pattern [15].

Representative electropherograms obtained in the high pH BGEs for some of the analytes are shown in Figs. 2 and 3. The numbers next to the compound codes (Fig. 1) indicate the ODMS concentrations and the applied potentials used.







B33: (1-Napthyl)ethylamine



B34: Norephedrine (1R,2S) & (1S,2R)





B36: Oxyphencyclimine



B41: Pindolol

B37: Oxprenolol





B38: Piperoxan



B39: Phenylglycinonitrile



B42: Propranolol

B43: Propafenone

B45: Salbutamol

B46: Scopolamine



B47: Terbutaline





B57: Diltiazem

ОH



B58: Tetrahydrozoline



B51: Tolperisone

B60: Ephedrine (1R,2S) & (1S,2R) B61: Synephrine

НΟ

Fig. 1. (continued)

4. Conclusions

The use of the new, single-isomer, base-stable,

sulfated γ -CD, the sodium salt of octakis(2,3-dimethyl-6-O-sulfo)-y-cyclodextrin has been studied in high pH BGEs. Interactions between ODMS and

Brent
Busby
et
al.
~
У.
Chromatogr.
Α
066
(2003)
63

Table 1

Analyte	0 mM ODMS U = 10.2 kV	5 mM C U = 8.5 L	DMS kV			$\begin{array}{l} 10 \text{ m}M \text{ ODMS} \\ U = 6.8 \text{ kV} \end{array}$				20 mM ODMS $U = 6.8 kV$				30 mM U = 5.9	ODMS kV		40 mM ODMS $U = 7.6 kV$				
	μ	μ	α	β	R _s	μ	α	β	R_s	μ	α	β	R_s	μ	α	β	R _s	μ	α	β	R_s
N02	0	-0.3	1.0	-151	0	-0.7	1.00	-60	0.0	-1.0	1.18	-30	0.6	-1.5	1.19	-13	1.3	-1.8	1.19	-10	1.9
N10	0	NA				-0.4	1.00	-103	0.0	-0.6	1.00	-47	0.0	-1.1	1.00	-18	0.0	-1.2	1.07	-15	< 0.5
N15	0	NA				NA				-0.6	1.00	-47	0.0	-1.0	1.00	-18	0.0	-1.2	1.00	-14	0.0
N19	0	NA				NA				NA				-0.3	1.00	-68	0.0	-0.4	1.00	-45	0.0
N20	0	NA				-0.7	1.00	-55	0.0	-1.2	1.00	-25	0.0	-2.1	1.00	-9	0.0	-2.1	1.00	-8	0.0
N21	0	-0.4	1.0	-127	0	-0.8	1.00	-52	0.0	-1.1	1.00	-25	0.0	-1.9	1.00	-9	0.0	-2.1	1.00	-8	0.0
N22	0	NA				NA				-0.9	1.00	-31	0.0	-1.5	1.05	-16	< 0.5	-1.6	1.07	-14	0.5
N24	0	NA				-0.4	1.00	-110	0.0	-0.5	1.00	-56	0.0	-0.9	1.00	-17	0.0	-1.0	1.00	-22	0.0
N25	0	-0.4	1.0	-118	0	-0.8	1.00	-48	0.0	-1.2	1.00	-24	0.0	-2.1	1.00	-12	0.0	-2.2	1.00	-10	0.0
N26	0	-0.3	1.0	-155	0	-0.7	1.00	-61	0.0	-1.0	1.00	-32	0.0	-1.6	1.00	-10	0.0	-1.7	1.00	-11	0.0
N30	0	NA				-0.8	1.32	-52	0.6	-1.2	1.34	-24	1.7	-2.2	1.35	-9	3.6	-2.3	1.34	-8	4.4
N34	0	-0.3	1.0	-150	0	-0.7	1.00	-63	0.0	-1.0	1.00	-29	0.0	-1.8	1.00	-9	0.0	-1.9	1.00	-10	0.0
N36	0	-0.4	1.0	-134	0	-0.7	1.00	-61	0.0	-1.0	1.00	-28	0.0	-1.7	1.00	-10	0.0	-1.9	1.00	-10	0.0
N38	0	-0.3	1.0	-134	0	-0.6	1.00	-61	0.0	-0.9	1.00	-28	0.0	-1.6	1.00	-10	0.0	-1.7	1.00	-10	0.0
N39	0	NA				-0.5	1.00	-71	0.0	-0.7	1.00	-32	0.0	-1.2	1.00	-12	0.0	-1.4	1.00	-11	0.0
N40	0	NA				-1.0	1.00	-87	0.0	-1.5	1.00	-41	0.0	-2.6	1.00	-17	0.0	-2.6	1.00	-14	0.0

Effective mobilities of the less mobile enantiomer (μ , in 10⁻⁵ cm² V⁻¹ s⁻¹ units), separation selectivities (α), measured peak resolution values (R_s), dimensionless EOF mobility values (β) and the injector-to-detector effective potential drop (U) in pH 9.5 ODMS BGEs for nonionic analytes

Table 2

Effective mobilities of the less mobile enantiomer (μ , in 10⁻⁵ cm² V⁻¹ s⁻¹units), separation selectivities (α), measured peak resolution values (R_s), dimensionless EOF mobility values (β) and the injector-to-detector effective potential drop (U) in pH 9.5 ODMS BGEs for weak acid analytes

Analyte	0 mM ODMS U = 10.2 kV μ	5 mM O U = 8.5 I	10 mM U = 6.8 l	ODMS kV			20 mM ODMS $U=6.8 kV$				30 mM ODMS U=5.9 kV				40 mM ODMS $U = 7.6 \text{ kV}$						
		μ	α	β	R _s	μ	α	β	R_s	μ	α	β	R _s	μ	α	β	R_s	μ	α	β	R_s
A02	-20.3	-20.3	1.00	-2.4	0.0	-18.8	1.00	-2.2	0.0	-15.6	1.00	-2.0	0.0	-16.4	1.00	-1.7	0.0	-14.0	1.00	-1.6	0.0
A03	-16.3	-14.2	1.00	-2.8	0.0	-14.4	1.00	-2.8	0.0	-14.2	1.00	-2.1	0.0	-13.7	1.00	-2.0	0.0	-11.9	1.00	-1.8	0.0
A04	-15.9	-16.7	1.00	-2.9	0.0	-15.6	1.00	-2.6	0.0	-13.7	1.00	-2.2	0.0	-14.9	1.00	-1.8	0.0	-12.9	1.00	-1.7	0.0
A09	-8.4	-10.2	1.00	-4.0	0.0	-10.4	1.00	-3.9	0.0	-11.6	1.00	-2.6	0.0	-9.4	1.00	-2.8	0.0	-10.0	1.00	-1.9	0.0
A16	-12.9	-12.5	1.00	-3.2	0.0	-13.4	1.00	-3.0	0.0	-11.7	1.00	-2.6	0.0	-11.9	1.00	-2.3	0.0	-10.5	1.00	-1.8	0.0
A22	-16.8	-16.7	1.00	-2.9	0.0	-15.5	1.00	-2.6	0.0	-13.2	1.01	-2.3	0.5	-14.0	1.01	-1.9	1.1	-12.2	1.02	-1.8	1.6
A23	-16.9	-16.6	1.00	-2.9	0.0	-15.3	1.00	-2.7	0.0	-12.6	1.00	-2.4	0.0	-13.0	1.00	-1.9	0.0	-11.1	1.00	-1.9	0.0
A26	-17.1	-16.9	1.00	-2.9	0.0	-15.8	1.00	-2.6	0.0	-13.5	1.00	-2.2	0.0	-14.1	1.00	-1.6	0.0	-12.4	1.00	-1.7	0.0
A27	-16.3	-16.0	1.00	-3.0	0.0	-14.7	1.00	-2.8	0.0	-12.4	1.00	-2.4	0.0	-13.0	1.00	-2.1	0.0	-11.1	1.00	-1.9	0.0
A28	-22.5	-20.5	1.00	-2.2	0.0	-20.6	1.00	-2.0	0.0	-16.7	1.00	-1.8	0.0	-17.3	1.00	-1.5	0.0	-14.5	1.00	-1.6	0.0
A30	-17.7	-17.0	1.01	-2.8	0.6	-16.0	1.01	-2.5	0.6	-13.0	1.01	-2.3	0.6	-13.1	1.01	-1.6	1.1	-11.4	1.01	-1.9	1.0
A31	-17.4	-17.2	1.00	-2.8	0.0	-16.3	1.00	-2.5	0.0	-13.3	1.00	-2.2	0.0	-13.7	1.00	-1.6	0.0	-11.7	1.00	-1.8	0.0
A36	-22.0	-21.0	1.00	-2.3	0.0	-19.4	1.00	-2.1	0.0	-16.0	1.00	-1.8	0.0	-16.6	1.00	-1.4	0.0	-13.9	1.00	-1.6	0.0

Analyte	$0 \text{ m}M \text{ ODMS}$ $U = 22.9 \text{ kV}$ μ	5 mM (U = 8.5	DDMS kV		10 mM U=6.8	ODMS kV			20 mM U = 6.8	ODMS kV			30 m <i>M</i> <i>U</i> =5.9		$\begin{array}{c} 40 \text{ m}M \text{ ODMS} \\ U = 7.6 \text{ kV} \end{array}$							
		μ	α	β	R_s	μ	α	β	R_s	μ	α	β	R_s	μ	α	β	R_s	μ	α	β	R_s	
B02	11.2	4.0	1.04	12	< 0.5	3.3	1.08	12	0.7	0.8	1.07	36	< 0.5	NA	1.14	10	1.2	-0.1	1.00	-207	0.0	
B04	14.5	7.1	1.00	6.9	0.0	5.5	1.00	7.0	0.0	3.2	1.00	9.0	0.0	3.1	1.00	9.1	0.0	2.8	1.00	6.6	0.0	
B06	6.8	4.2	1.09	11	0.6	2.2	1.12	18	0.5	1.5	1.14	20	< 0.5	1.2	1.18	23	0.7	1.1	1.18	17	0.6	
B08	10.0	4.1	1.00	12	0.0	3.5	1.00	11	0.0	1.2	1.00	24	0.0	0.9	1.00	30	0.0	0.7	1.00	27	0.0	M
B09	11.3	NA				3.5	1.00	11	0.0	2.6	1.00	11	0.0	2.5	1.00	9.2	0.0	1.8	1.00	10	0.0	В
B10	0.7	NA				NA				-0.8	1.00	-38	0.0	-1.3	1.00	-20	0.0	-1.5	1.00	-12	0.0	ren
B11	1.3	0.4	1.00	131	0.0	0.8	1.00	46	0.0	-0.8	1.00	-35	0.0	-1.5	1.00	-18	0.0	-1.7	1.00	-10	0.0	t E
B13	14.8	4.4	1.14	10	1.4	3.8	1.19	10	1.6	1.7	1.34	17	1.9	1.4	1.34	19	1.9	1.3	1.31	14	1.9	Bus
B14	5.9	2.7	1.05	17	< 0.5	1.2	1.27	32	0.9	0.7	1.63	40	1.9	-0.4	-1.04	-66	2.8	-1.0	0.10	-19	5.7	by
B18	10.7	-1.5	0.18	-30	2.5	-2.6	0.50	-15	2.5	-2.4	0.53	-12	4.3	-3.0	0.61	-7.6	4.6	-2.7	0.65	-6.6	3.4	et
B19	11.2	4.8	1.00	9.6	0.0	3.3	1.00	12	0.0	2.7	1.00	11	0.0	2.9	1.00	8.6	0.0	2.1	1.00	8.5	0.0	al
B20	18.2	8.3	1.00	5.6	0.0	6.3	1.00	6.3	0.0	4.5	1.00	6.3	0.0	4.8	1.00	5.2	0.0	3.8	1.00	4.7	0.0	~
B22	0.0	NA				-0.7	1.00	- 59	0.0	-1.0	1.00	-27	0.0	-1.7	1.00	-17	0.0	-2.0	1.00	-9.3	0.0	J.
B23	1.6	-1.3	1.00	-35	0.0	-2.9	0.85	-14	1.3	-3.9	0.80	-7.2	2.9	-5.5	0.80	-4.2	5.8	-6.0	0.77	-3.1	10.3	Q
B25	14.1	5.4	1.00	9.0	0.0	3.4	1.00	12	0.0	2.2	1.00	13	0.0	2.1	1.00	11	0.0	1.5	1.00	12	0.0	uro
B26	4.0	0.9	1.00	56	0.0	-1.1	1.00	-36	0.0	-1.4	1.00	-20	0.0	-2.8	1.00	-10	0.0	-2.5	1.00	-7.4	0.0	ma
B28	17.9	10.4	1.00	4.7	0.0	8.0	1.00	4.9	0.0	5.9	1.00	4.7	0.0	5.7	1.00	4.9	0.0	4.8	1.00	3.8	0.0	tog
B30	0.0	-1.1	1.00	-45	0.0	-4.6	1.00	-8.6	0.0	-3.0	1.00	-9.5	0.0	-4.4	1.00	-5.4	0.0	-3.8	1.00	-4.9	0.0	
B31	8.4	3.9	1.00	12	0.0	1.6	1.00	25	0.0	1.3	1.00	22	0.0	0.7	1.00	32	0.0	0.6	1.00	32	0.0	Α
B33	8.2	0.7	1.00	61	0.0	NA				-1.0	0.87	-28	< 0.5	-2.4	0.94	-9.4	0.5	-2.7	0.96	-7.1	0.6	<i>66</i>
B34	6.9	2.6	1.00	16	0.0	2.3	1.00	17	0.0	2.4	1.00	11	0.0	1.5	1.00	15	0.0	1.1	1.00	18	0.0	00
B36	25.0	12.5	1.00	3.5	0.0	12.7	1.00	3.1	0.0	NA				2.3	1.00	10	0.0	0.7	1.00	28	0.0	200
B37	9.3	5.4	1.00	9.0	0.0	2.7	1.00	14	0.0	2.2	1.00	15	0.0	1.3	1.00	18	0.0	0.4	1.00	44	0.0	33
B38	2.3	0.8	1.00	62	0.0	-1.0	0.41	-38	1.7	-1.6	0.57	-20	2.4	-2.2	0.73	-11	2.5	-2.2	0.83	-8.5	2.2	6
B39	0.0	NA				-0.7	1.00	-60	0.0	-1.0	1.00	-29	0.0	-1.6	1.00	-15	0.0	-1.7	1.00	-11	0.0	7
B41	10.8	NA				-0.4	1.00	-88	0.0	-1.1	0.72	-25	1.4	-2.1	0.87	-11	1.3	-2.2	0.91	-8.5	1.2	ŝ
B42	9.7	-3.7	0.67	-13	3.3	-4.9	0.79	-8.0	3.9	-4.8	0.83	-5.8	4.0	-5.4	0.86	-4.2	4.1	-4.7	0.88	-4.0	3.9	
B43	6.9	1.9	1.00	21	0.0	0.4	1.00	91	0.0	-0.5	1.00	-56	0.0	-1.5	1.00	-15	0.0	-2.2	1.00	-8.6	0.0	
B45	6.0	2.6	1.00	18	0.0	0.8	1.21	49	0.5	0.6	1.35	45	0.6	NA				-1.0	0.87	-19	0.6	
B46	0.2	NA				NA				-0.4	1.00	-66	0.0	-0.8	1.00	-27	0.0	-1.0	1.00	-18	0.0	
B47	2.4	NA				-1.4	1.00	-29	0.0	-1.5	1.00	-19	0.0	-1.8	1.00	-12	0.0	-2.2	1.00	-8.4	0.0	
B51	5.4	1.2	1.38	41	1.0	1.6	1.37	25	1.5	1.5	1.46	23	1.8	0.4	2.12	57	2.0	-0.3	1.00	-56	0.0	
B57	0.4	NA				-0.8	1.00	-51	0.0	-1.5	0.87	-18	0.9	-2.2	0.87	-11	1.3	-2.4	0.88	-7.6	1.6	
B58	19.2	8.3	1.04	5.0	0.8	7.3	1.06	5.5	1.0	5.4	1.06	5.2	1.0	5.0	1.06	4.9	1.1	3.9	1.06	4.7	1.1	
B60	13.7	5.2	1.08	9.3	1.0	5.9	1.09	6.8	1.2	4.7	1.10	5.9	1.4	3.2	1.12	7.7	1.3	3.4	1.13	5.5	1.7	
B61	3.5	NA				0.7	1.00	58	0.0	0.3	1.00	105	0.0	-2.2	1.00	-11	0.0	-0.8	1.00	-22	0.0	

Effective mobilities of the less mobile enantiomer (μ , in 10⁻⁵ cm² V⁻¹s⁻¹units), separation selectivities (α), measured peak resolution values (R_s), dimensionless EOF mobility values (β) and the injector-to-detector effective potential drop (U) in pH 9.5 ODMS BGEs for weak base analytes

Table 3



Fig. 2. Typical electropherograms of neutral and weak acid analyte enantiomers. The numbers next to the compound codes (see Fig. 1) indicate the ODMS concentrations (m*M*) and applied potentials (kV) used. Capillary: 25 μ m I.D., 39/46 cm effective/total length, uncoated, fused-silica. Other conditions see Experimental.

many of the analytes studied were weaker than those observed with the other single-isomer, 6-O-sulfo CDs. Also, ODMS interacted with most of the analytes less strongly in the high pH BGE than it did in low pH BGEs. The enantiomers of several weak acids and one weak base were found to follow a

desionoselective separation pattern. Despite its stability in alkaline media, ODMS does not seem to be as widely applicable a chiral resolving agent in high pH BGEs as it is in low pH BGEs, the first such behavior observed for a member of the 6-*O*-sulfo CD family.



Fig. 3. Typical electropherograms of weak base analyte enantiomers. The numbers next to the compound codes (see Fig. 1) indicate the ODMS concentrations (mM) and applied potentials (kV) used. Capillary: 25 μ m I.D., 39/46 cm effective/total length, uncoated, fused-silica. Other conditions see Experimental.

Acknowledgements

Partial financial support of this work by the Texas Coordination Board of Higher Education ARP-TDT program (Project Number 010366-016 and 010366-0152-1999), ANTEK Instruments, Inc. (Houston, TX, USA), Beckman-Coulter (Fullerton, CA, USA) and the Texas A&M University Gradipore Chair in Separation Science Endowment is gratefully acknowledged.

References

[1] A. Amini, Electrophoresis 22 (2001) 3107.

- [2] A. Rizzi, Electrophoresis 22 (2001) 3079.
- [3] M.B. Busby, P. Lim, Gy. Vigh, Electrophoresis, in press.
- [4] J.B. Vincent, A.D. Sokolowski, T.V. Nguyen, Gy. Vigh, Anal. Chem. 69 (1997) 4226.
- [5] W. Zhu, Gy. Vigh, Electrophoresis 23 (2002) in press.
- [6] J.B. Vincent, D. Kirby, T.V. Nguyen, Gy. Vigh, Anal. Chem. 69 (1997) 4419.
- [7] W. Zhu, Gy. Vigh, Anal. Chem. 72 (2000) 310.
- [8] H. Cai, T.V. Nguyen, Gy. Vigh, Anal. Chem. 70 (1998) 580.
- [9] J.B. Vincent, Gy. Vigh, J. Chromatogr. A 817 (1998) 105.
- [10] H. Cai, Gy. Vigh, J. Microcol. Sep. 10 (1998) 293.
- [11] W. Zhu, Gy. Vigh, J. Microcol. Sep. 12 (2000) 167.
- [12] W. Zhu, Gy. Vigh, J. Chromatogr. A 987 (2003) 459.
- [13] Single Isomer Sulfated Cyclodextrins. Product Bulletin, Antek Instruments, Houston, TX, 2002.
- [14] B.A. Williams, Gy. Vigh, Anal. Chem. 69 (1997) 4445.
- [15] Y.Y. Rawjee, Gy. Vigh, Anal. Chem. 66 (1994) 619.
- [16] D.K. Maynard, Gy. Vigh, Electrophoresis 22 (2001) 3212.