Synthesis and Liquid Chromatographic Determination of Optical Purity of Naphthyl Propionate Liquid Crystals†

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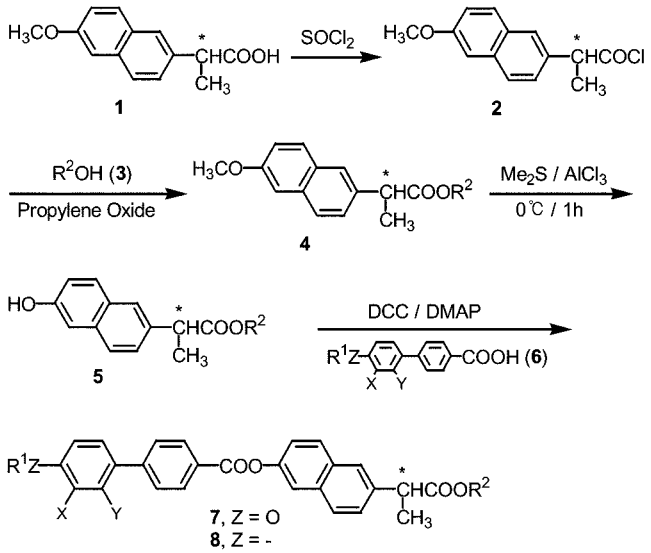
Most ferro- and antiferroelectric liquid crystals are chiral and their mesomorphic phase structures and electro-optical properties are largely dependent on the optical purity.[1,2] Thus, for the chiral liquid crystals the occurrence of chemical or thermal racemization has to be checked throughout the synthetic sequence and the investigation of the mesomorphic and electro-optical properties.

However, most research papers on the chiral liquid crystals did not mention the enantiomeric excess of the materials. They were just assumed to be the same as those of the starting materials or of various intermediates without any investigation. It is very important to establish convenient and accurate means of determining the enantiomeric composition in liquid crystal chemistry. Among various techniques, liquid chromatographic separation of enantiomers on the chiral stationary phases (CSPs) might be the choice because this technique is known to be the most simple and convenient means of determining the enantiomeric composition.[3] In this study, we show that a commercial HPLC chiral column, (*S*,*S*)-Whelk-O1, can resolve various types of naphthyl propionate liquid crystals.



Scheme 1

Naphthyl propionate ferroelectric liquid crystals with an ester linkage between naphthalene and biphenyl rings were prepared from (*S*)-naproxen **1** as depicted in Scheme 1.[4]

Since the hydrogen atom on the chiral carbon in the naproxen derivatives **2**, **4**, **5**, **7** and **8** is acidic enough due to the presence of α-naphthalene ring and either carboxyl or carboalkoxy group, racemization *via* enolization in the compounds can take place routinely under both acidic and basic reaction conditions. Thus one has to confirm the integrity of chiral center in the intermediate and final products by the HPLC analysis using chiral column.

The integrity of chiral center in the reaction products **4** prepared from (*S*)-naproxen chloride (**2**) with alcohol in the presence of propylene oxide,[5] was confirmed by the HPLC. Racemic naproxen was obtained from (*S*)-naproxen (**1**) by deprotonating the acidic hydrogen with lithium diisopropylamide (LDA) followed by acidification and base-catalyzed racemization in aprotic polar solvents.[6] Thus obtained racemic naproxen was converted to the racemic *n*-butyl ester **4a** (R^2=C_4H_9) according to Scheme 1. HPLC resolution of racemic ester **4a** on a chiral column showed a reasonable separation factor [Figure 1(a)]. Figure 1(b) shows that **4a** prepared from (*S*)-naproxen was optically pure (enantiomeric excess: > 99% ee); racemization did not occur at all during the formation of **4a** from (*S*)-naproxen.

Demethylation of methoxy group of the esters **4** by dimethylsulfide-$AlCl_3$ produced naphthols **5** in good yields with no actual racemization [Figure 1(c)]. Demethylation of **4a** with boron tribromide gave **5a** in much lower yield (50.3%).

Esterifications of (*S*)-naproxen (**1**) with alcohols were achieved by using DCC and DMAP as described in the literatures[7-11] racemizations occurred in some extent (Scheme 2). Contrary to the above results, the racemization was not almost observed in the esterification of **5** with **6** using DCC and DMAP to give optically pure **7a** (99% ee) [Figure 1(d)].

The specific rotation of compound **7e** (R^1=$C_{10}H_{21}$, R^2=C_4H_9, X=Y=H) prepared by us showed $[\alpha]_D^{27.5}$ = +17.06, which is much higher than the reported value of $[\alpha]_D^{23}$ = +4.93.[10] This proves that the compounds prepared in this study are of better optical purity for investigating physical

†This paper is dedicated to Professor Yong Hae Kim for his distinguished achievements in chemistry.

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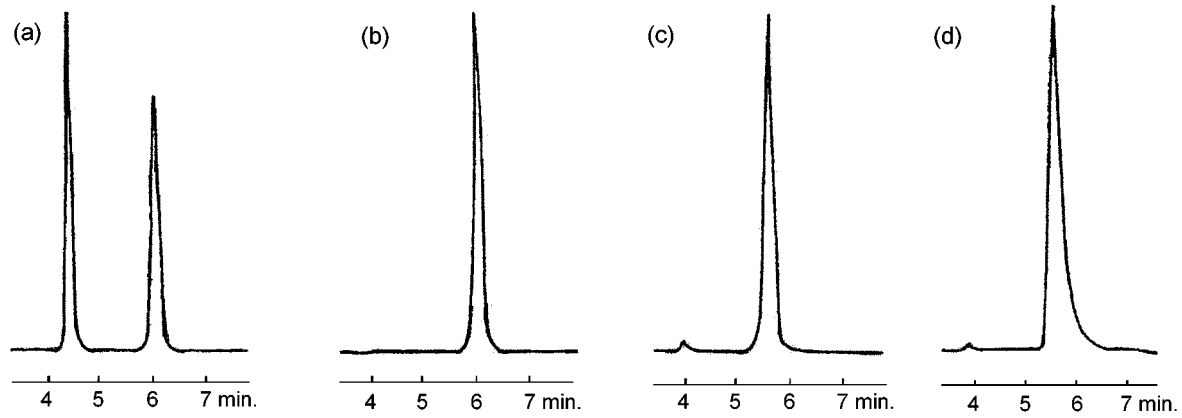
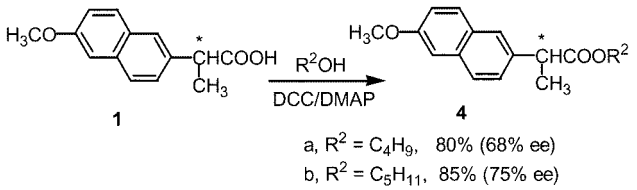


Figure 1. Chromatograms for resolving ester **4a** of (a) racemic **4a**, (b) **4a**, (c) **5a**, and (d) **7a** prepared from (*S*)-naproxen through Scheme 1



Scheme 2

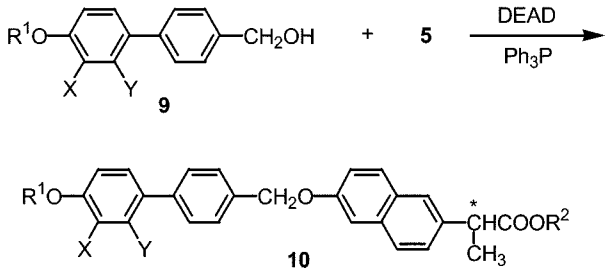
Table 1. Liquid chromatographic resolution of naphthyl propionate liquid crystals **7** and **8** on (*S,S*)-Whelk-Ol chiral column[a]

Liquid Crystal	R^1	R^2	X	Y	k_1[b]	k_2[b]	α[c]
7a	C_8H_{17}	C_4H_9	H	H	3.17	4.91	1.55
7b	C_8H_{17}	C_5H_{11}	H	H	3.12	4.79	1.54
7c	C_8H_{17}	C_6H_{13}	H	H	3.03	4.19	1.38
7d	C_9H_{19}	C_4H_9	H	H	4.27	5.33	1.06
7e	$C_{10}H_{21}$	C_4H_9	H	H	3.45	4.84	1.40
7f	C_8H_{17}	C_4H_9	F	H	4.14	5.79	1.40
7g	C_8H_{17}	C_5H_{11}	F	H	3.21	5.20	1.62
7h	C_8H_{17}	C_4H_9	F	F	3.46	4.71	1.36
7i	C_8H_{17}	C_5H_{11}	F	F	3.02	3.97	1.31
8a	C_8H_{17}	C_4H_9	H	H	2.29	3.37	1.47
8b	C_8H_{17}	C_5H_{11}	H	H	2.26	3.15	1.39
8c	C_8H_{17}	C_6H_{13}	H	H	2.00	2.95	1.48

[a]See the Experimental part for the chromatographic condition. [b]Retention factor of the first and second eluted enantiomers. [c]Separation factor.

properties. The results of chromatographic resolution are summarized in Table 1.

Naphthyl propionates having an ethereal linkage **10** were prepared from Mitsunobu reaction of naphthols **5** with 4-(4-alkoxyphenyl)benzyl alcohols **9** (Scheme 3). Although the yields were low, optically pure products **10** were obtained. Their optical purites were estimated in the same manner using the same HPLC chiral column, (*S,S*)-Whelk-O1 (Table 2). The yields of the ether **10** were improved (> 72%) in the reactions of naphthol **5** with 4-(4-alkoxyphenyl)benzyl chloride in the presence of potassium carbonate, however, complete racemizations were occurred.



Scheme 3

Thus obtained naphthyl propionate ferroelectric liquid crystals having an oxymethylene linking group **10** exhibited an enantiotropic mesogenic phases with the phase sequence: Cry-Sc*-S_A-Iso. But the compound **10a** with short alkyl chain as terminal group does not show the chiral smectic C phase. The naphthyl propionates with a fluorinated biphenyl group **10h-10l** have lower transition temperature than the nonfluorinated compounds **10a-10g**. Table 3 summarizes the mesomorphic transition temperatures and enthalpies for the

Table 2. Liquid chromatographic resolution of naphthyl propionate liquid crystals **10** on (*S,S*)-Whelk-Ol chiral column[a]

Liquid Crystal	R^1	R^2	X	Y	k_1[b]	k_2[b]	α[c]
10a	C_8H_{17}	C_4H_9	H	H	1.76	2.81	1.60
10b	C_8H_{17}	C_5H_{11}	H	H	1.60	2.61	1.63
10c	C_8H_{17}	C_6H_{13}	H	H	1.40	2.31	1.65
10d	C_8H_{17}	C_7H_{15}	H	H	1.28	2.15	1.68
10e	C_9H_{19}	C_4H_9	H	H	1.68	2.69	1.60
10f	C_9H_{19}	C_5H_{11}	H	H	1.46	2.37	1.62
10g	C_9H_{19}	C_6H_{13}	H	H	1.35	2.26	1.67
10h	C_8H_{17}	C_5H_{11}	F	H	1.77	2.86	1.62
10i	C_9H_{19}	C_5H_{11}	F	H	1.70	2.77	1.63
10j	$C_{10}H_{21}$	C_5H_{11}	F	H	1.68	2.72	1.62
10k	$C_{10}H_{21}$	C_4H_9	F	H	1.85	2.93	1.58
10l	$C_{10}H_{21}$	C_6H_{13}	F	H	1.49	2.50	1.68

[a]See the Experimental part for the chromatographic condition. [b]Retention factor of the first and second eluted enantiomers. [c]Separation factor.

Table 3. Transition temperatures (°C) and enthalpies (ΔH/kJmol^{-1}) (*in italics*) of naphthyl propionates **10** on cooling

Liquid Crystal	I		S_A		S_C*		Cr	mp
10a	·	116.0	·		–	82.2	·	107.5
		4.71				*25.2*		
10b	·	116.9	·	106.2	.	66.8	·	100.9
		6.33		*0.11*		*25.2*		
10c	·	113.2	·	97.9	·	75.5	·	98.7
		6.76		*0.04*		*23.9*		
10d	·	115.7	·	96.3	·	77.1	·	96.7
		6.81		*1.32*		*19.8*		
10e	·	118.1	·	113.3	·	85.1	·	112.7
		6.15		*0.39*		*20.3*		
10f	·	112.8	·	105.3	·	79.2	·	96.5
		5.86		*0.26*		*20.7*		
10g	·	111.2	·	101.7	·	74.2	·	99.2
		6.25		*0.18*		*19.2*		
10h	·	88.4	·	73.2	·	41.3	·	82.7
		4.70		*0.11*		*21.7*		
10i	·	87.0	·	74.9	·	43.4	·	75.1
		4.75		*0.11*		*23.4*		
10j	·	88.7	·	78.1	·	47.5	·	75.3
		5.19		*0.10*		*26.4*		
10k	·	91.3	·	82.2	·	52.3	·	71.9
		4.96		*0.14*		*26.7*		
10l	·	88.6	·	75.9	·	47.2	·	66.2
		5.03		*0.12*		*28.2*		

Cr = crystalline phase; S_C* = chiral smectic C phase; S_A = smectic A phase; I = isotropic liquid phase.

alkyl (*S*)-2-{6-[4-(4'-alkyloxybiphenyl)benzyloxy]-2-naphthyl}propionates **10**. Exchanging of central linkage group from ester (compounds **7** and **8**) to ether (compound **10**) in the naphthyl propionate liquid crystals, the temperature range of the S_A phase became narrower and the N* phase was not appeared.

In summary, various types of optically pure naphthyl propionate liquid crystals were prepared from (*S*)-naproxen in a four step reaction sequence. We found that a commercial HPLC chiral column, (*S*,*S*)-Whelk-O1, successfully resolves the (*S*)-naproxen-based liquid crystals. This technique is very simple and effective in monitoring the enantiomeric excess of the intermediate and final products without any structural modification at all.

Experimental Section

^{1}H-NMR spectra were recorded on Varian Gemini-200 (200 MHz) and Varian Inova (500 MHz) spectrometer using chloroform as an internal standard. The latter instrument was also used for recording ^{13}C-NMR spectra in $CDCl_3$ (solvent and internal reference) and ^{19}F-NMR spectra in $CDCl_3$ (trifluoroacetic acid as an internal reference). Elemental analyses were performed at the National Center for Inter-University Research Facilities, Seoul National University. High mass spectra were taken on a Jeol JMS-700 spectrometer. Phase transition temperature and phase appearance of final products were measured by using polarizing microscope (Olympus BH-2) with a hot stage and a controller (Mettler FP 90). Transition temperature and enthalpy were determined by differential scanning calorimetry (DSC) using a Perkin Elmer DSC 7 calorimeter. HPLC analyses were performed with an instrument consisted of Waters model 510 pump, a Rheodyne model 7125 injector with a 20 μL sample loop, a Youngin model 710 absorbance detector with a 254 nm UV filter and a Youngin D520B computing integrator. A mixed solvent of isopropyl alcohol and hexanes (80 : 20, v/v) was used as a mobile phase with a flow rate of 2.0 mL/min at room temperature. The column void volume was determined by injecting 1,3,5-tri-*tert*-butylbenzene, a presumed unretained solute.[12] The chiral column (*S*,*S*) Whelk-O1, used in this study is commercially available from Regis Technologies, Inc. (Morton Grove, Illinois, USA). Optical rotations were determinated as a solution in chloroform using a JASCO DIP-1000 digital polarimeter with a path length of 1 dm. Concentrations are quoted in g/100 mL. (*S*)-Naproxen (**1**, > 98% ee) was purchased from Aldrich Chemical Co., and used without further purification.

Synthesis of Alkyl (*S*)-2-{6[4-(4'-Alkyloxyphenyl)benzyloxy]-2-naphthyl}propionate 10. A typical procedure for the synthesis of **10a** is following. To a THF (5 mL) solution of 4-(4'-octyloxyphenyl)benzyl alcohol **7a** (0.20 g, 0.59 mmol) was added triphenyl phosphine (0.17 g, 0.65 mmol) and diethyl azodicarboxylate (DEAD, 0.11 g, 0.64 mmol) and stirred at room temperature for 1 h. To the mixture butyl (*S*)-2-(6-hydroxy-2-naphthyl)propionate **5a** (0.17 g, 0.62 mmol) in THF (2 mL) was added and stirred for 24 hrs at room temperature. The precipitated triphenylphosphine oxide and diethyl hydrazinedicarboxylate were removed by filtration. The filtrate was concentrated *in vacuo* and the residue was chromatographed on silica gel (CH_2Cl_2) to give 0.091 g (25%) of **10a**. Compounds **10b-10l** were similarly prepared.

10a: ^{1}H-NMR δ 0.81-1.79 (m, 25H), 3.84 (q, 1H, J = 7.0 Hz), 3.94-4.10 (m, 4H), 5.18 (s, 2H), 6.95-7.74 (m, 14H); ^{13}C-NMR δ 13.6, 14.1, 18.5, 19.0, 22.6, 26.1, 29.2, 29.3, 29.4, 30.6, 31.8, 45.5, 64.6, 68.1, 69.9, 107.0, 114.8, 119.3, 125.9, 126.3, 126.9, 127.1, 128.0, 128.1, 129.1, 129.3, 133.1, 133.6, 135.1, 136.0, 140.7, 158.7, 158.8, 174.7; $[\alpha]_D^{22}$ = −5.5 (C 0.29); Anal. Calc. for $C_{38}H_{46}O_4$: C 80.53, H 8.18: found C 80.38, H 8.22.

10b: ^{1}H-NMR δ 0.78-1.79 (m, 27H), 3.85 (q, 1H, J = 7.0 Hz), 3.96-4.10 (m, 4H), 5.19 (s, 2H), 6.95-7.75 (m, 14H); ^{13}C-NMR δ 13.8, 14.1, 18.5, 22.2, 22.6, 26.0, 27.9, 28.2, 29.2, 29.3, 29.4, 31.8, 45.5, 64.9, 68.1, 69.9, 107.0, 114.8, 119.3, 125.9, 126.3, 126.9, 127.1, 128.0, 128.1, 129.1, 129.3, 133.1, 133.6, 135.1, 136.0, 140.7, 156.7, 158.8, 174.7; $[\alpha]_D^{23}$ = −12.8 (C 0.24); Anal. Calc. for $C_{39}H_{46}O_4$: C 80.65, H 8.33: found C 80.57, H 8.33.

10c: ^{1}H-NMR δ 0.79-1.79 (m, 29H), 3.81 (q, 1H, J = 7.0 Hz), 3.94-4.07 (m, 4H), 5.18 (s, 2H), 6.92-7.32 (m, 14H); ^{13}C-NMR δ 13.9, 14.1, 18.5, 22.4, 22.6, 25.4, 26.1, 28.5,

29.2, 29.3, 29.4, 31.3, 31.8, 45.5, 64.9, 68.1, 69.9, 107.0, 114.8, 119.3, 125.9, 126.3, 126.9, 127.1, 128.0, 128.1, 129.1, 129.3, 133.1, 133.6, 135.1, 136.0, 140.7, 156.8, 158.8, 174.7; $[\alpha]_D^{24}$ = –13.5 (C 0.10); Anal. Calc. for $C_{40}H_{50}O_4$: C 80.77, H 8.47: found C 80.59, H 8.57.

10d: ^{1}H-NMR δ 0.82-1.79 (m, 31H), 3.84 (q, 1H, *J* = 7.0 Hz), 3.98-4.07 (m, 4H), 5.19 (s, 2H), 6.93-7.73 (m, 14H); ^{13}C-NMR δ 14.0, 14.1, 18.5, 22.5, 22.6, 25.7, 26.1, 28.5, 28.8, 29.2, 29.3, 29.7, 31.6, 31.8, 45.6, 64.9, 68.1, 69.9, 107.1, 114.8, 119.3, 125.9, 126.3, 126.9, 127.1, 128.0, 128.1, 129.1, 129.3, 133.1, 133.6, 135.1, 136.0, 140.7, 156.8, 158.8, 174.7; $[\alpha]_D^{26}$ = –13.2 (C 0.11); Anal. Calc. for $C_{41}H_{52}O_4$: C 80.88, H 8.61: found C 80.07, H 8.73.

10e: ^{1}H-NMR δ 0.82-1.83 (m, 27H), 3.84 (q, 1H, *J* = 7.0 Hz), 3.96-4.07 (m, 4H), 5.18 (s, 2H), 6.94-7.74 (m, 14H); ^{13}C-NMR δ 13.6, 14.1, 18.5, 19.0, 22.7, 26.1, 29.2, 29.3, 29.4, 29.5, 30.6, 31.9, 45.5, 64.6, 68.1, 69.9, 107.0, 114.9, 119.3, 125.9, 126.3, 126.9, 127.1, 128.0, 128.1, 129.1, 129.3, 133.1, 133.6, 135.1, 136.0, 140.7, 156.8, 158.8, 174.7; $[\alpha]_D^{28}$ = –12.0 (C 0.11); Anal. Calc. for $C_{39}H_{48}O_4$: C 80.65, H 8.33: found C 80.66, H 8.36.

10f: ^{1}H-NMR δ 0.78-1.82 (m, 29H), 3.82 (q, 1H, *J* = 7.0 Hz), 3.94-4.08 (m, 4H), 5.19 (s, 2H), 6.92-7.73 (m, 14H); ^{13}C-NMR δ 13.9, 14.1, 18.5, 22.2, 22.7, 26.0, 27.9, 28.2, 29.2, 29.3, 29.4, 29.5, 31.9, 45.5, 64.9, 68.1, 69.9, 107.0, 114.8, 119.3, 125.9, 126.3, 126.9, 127.1, 128.0, 128.1, 129.1, 129.3, 133.1, 133.6, 135.1, 136.0, 140.7, 156.8, 158.8, 174.7; $[\alpha]_D^{28}$ = –8.9 (C 0.30); Anal. Calc. for $C_{40}H_{50}O_4$: C 80.77, H 8.47: found C 80.59, H 8.46.

10g: ^{1}H-NMR δ 0.82-1.84 (m, 31H), 3.84 (q, 1H, *J* = 7.0 Hz), 3.96-4.09 (m, 4H), 5.18 (s, 2H), 6.94-7.75 (m, 14H); ^{13}C-NMR δ 13.9, 14.1, 18.5, 22.5, 22.7, 25.4, 26.1, 28.5, 29.3, 29.3, 29.4, 29.5, 31.3, 31.9, 45.6, 64.9, 68.1, 69.9, 107.1, 114.8, 119.3, 125.9, 126.3, 126.9, 127.1, 128.0, 128.1, 129.1, 129.3, 133.1, 133.6, 135.1, 136.0, 140.7, 156.8, 158.8, 174.7; $[\alpha]_D^{28}$ = –12.4 (C 0.13); Anal. Calc. for $C_{38}H_{46}O_4$: C 80.88, H 8.61: found C 81.20, H 8.71.

10h: ^{1}H-NMR δ 0.78-1.87 (m, 27H), 3.84 (q, 1H, *J* = 7.0 Hz), 4.02-4.09 (m, 4H), 5.20 (s, 2H), 6.97-7.75 (m, 13H); ^{19}F-NMR 134.63; MS; m/z 598.3466 [M^+ (100%), $C_{39}H_{47}O_4F_1$ requires 598.3458], 598 (M^+, 5), 483 (2), 313 (100), 201 (100), 200 (24), 71 (5%); $[\alpha]_D^{26}$ = +5.3 (C 0.19).

10i: ^{1}H-NMR δ 0.78-1.91 (m, 29H), 3.84 (q, 1H, *J* = 7.0 Hz), 4.02-4.09 (m, 4H), 5.20 (s, 2H), 6.97-7.75 (m, 13H); ^{19}F-NMR 134.61; MS; m/z 612.3610 [M^+ (100%), $C_{40}H_{49}O_4F_1$ requires 612.3615], 612 (M^+, 8), 497 (2), 327 (100), 201 (67), 200 (14), 71 (3%); $[\alpha]_D^{26}$ = +18.2 (C 0.18).

10j: ^{1}H-NMR δ 0.78-1.91 (m, 31H), 3.84 (q, 1H, *J* = 7.0 Hz), 4.02-4.09 (m, 4H), 5.20 (s, 2H), 6.97-7.75 (m, 13H); ^{19}F-NMR 134.61; MS; m/z 622.3778 [M^+ (100%), $C_{41}H_{51}O_4F_1$ requires 627.3771], 626 (M^+, 3), 511 (1), 341 (100), 201 (56), 200 (12), 71 (1%); $[\alpha]_D^{27}$ = +13.8 (C 0.20).

10k: ^{1}H-NMR δ 0.82-1.87 (m, 29H), 3.84 (q, 1H, *J* = 7.0 Hz), 4.03-4.12 (m, 4H), 5.19 (s, 2H), 6.97-7.75 (m, 13H); ^{19}F-NMR 134.67; MS; m/z 612.3619 [M^+ (100%), $C_{40}H_{49}O_4F_1$ requires 612.3615], 612 (M^+, 3), 511 (1), 341 (100), 201 (69), 200 (14), 57 (13%); $[\alpha]_D^{27}$ = +17.3 (C 0.21).

10l: ^{1}H-NMR δ 0.78-1.87 (m, 33H), 3.84 (q, 1H, *J* = 7.0 Hz), 4.03-4.09 (m, 4H), 5.20 (s, 2H), 6.97-7.75 (m, 13H); ^{19}F-NMR 134.63; MS; m/z 640.3926 [M^+ (100%), $C_{42}H_{53}O_4F_1$ requires 640.3928], 640 (M^+, 12), 511 (2), 341 (100), 201 (63), 200 (13%); $[\alpha]_D^{27}$ = +12.7 (C 0.27).

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