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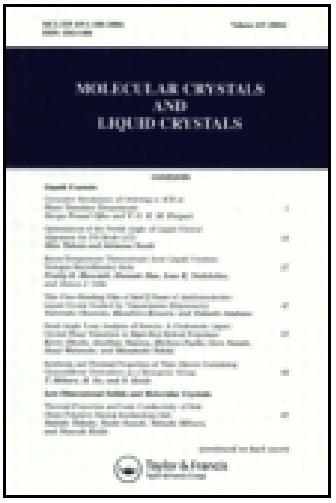

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Mesomorphism Behavior of Two Series of Chiral Compounds with Semiperfluorocarbon Chains

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Mesomorphism Behavior of Two Series of Chiral Compounds with Semiperfluorocarbon Chains

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Thermotropic liquid crystals with fluorocarbon chains have attracted much attention, because they are feasible to form smectic phases. Herein, two series of chiral compounds with semiperfluorocarbon chains were designed and synthesized. These compounds were characterized by IR, ^{1}H NMR, ^{19}F NMR, mass spectroscopy, and elemental analysis. Phase transition behavior was investigated by differential scanning calorimetry and polarized optical microscopy. Members of the two-ring series do not exhibit liquid crystalline phases. However, the three-ring analogues exhibit an enantiotropic SmA phase. When the length of alkyl chains is sufficient, an enantiotropic SmB phase is also identified.

Keywords: Chirality; liquid crystals; perfluorocarbon

Introduction

Liquid crystals with perfluoroalkyl and semiperfluoroalkyl chains attracted much attention during the last decades [1–23]. Due to fluorophilic associations, smectic mesophases are feasible to be formed. For diblock $H(CH_2)_n(CF_2)_mF$ and triblock $F(CF_2)_m(CH_2)n(CF_2)_mF$ molecules, they can melt into smectic B (SmB) or G mesophase [1, 2]. Generally, the thermostability of the smectic phases increased with increasing the length of the fluorocarbon chains [21]. It was also found that a change of the terminal hydrogen in the fluorocarbon chain to chlorine could increase the clearing points of the liquid crystals [19–21]. Particularly, molecules with semiperfluorocarbon chains exhibit tilted smectic phases and some of them are inherently ferroelectric [23].

Since liquid crystals with smectic C* (SmC*) phase are potentially applied in ferroelectric liquid crystal display mixtures, many kinds of chiral liquid crystals were designed and synthesized. For the liquid crystals with both semifluoroalkyl chains and chiral centers, they can also melt into SmC* phase [20–22]. Herein, base on previous results, two series of chiral molecules with semifluoroalkyl chains were synthesized [22]. Only one series of them exhibit enantiotropic smectic A (SmA) and SmB phases.

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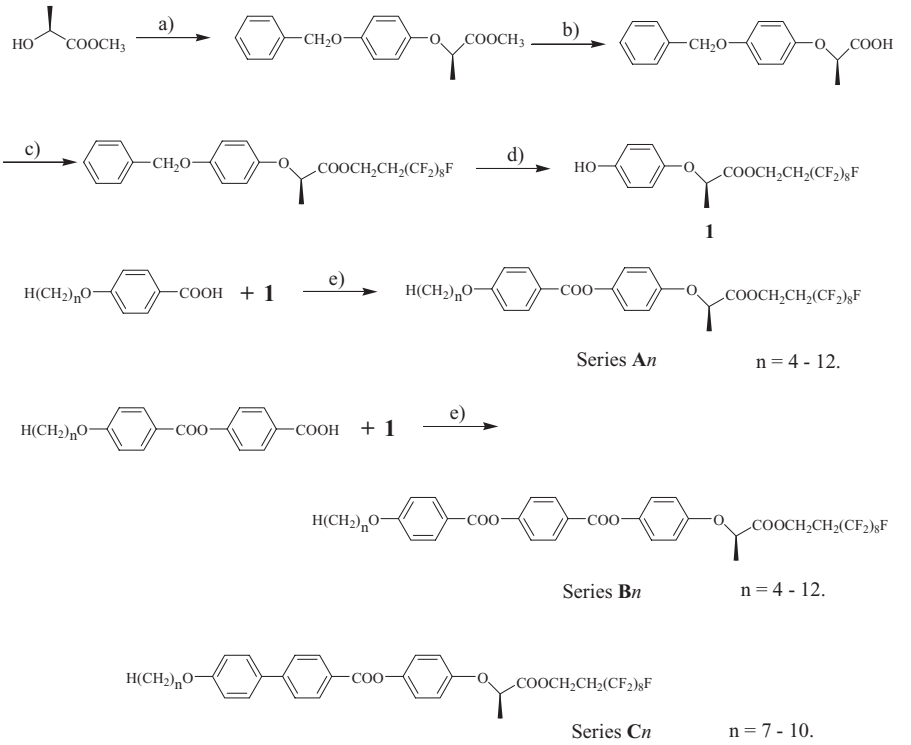
Results and Discussion

The synthesis of the target molecules containing a highly fluorinated tail chain (the 1,1,2,2-tetrahydroperfluorodecanyl group) is outlined in the scheme according to the literatures [21,22]. The one hydroxyl group of hydroquinone was protected with benzyl bromide and the protected phenol occurred Mitsunbobu coupling reaction with L-methyl lactate. Then the methyl ester was hydrolyzed to give the carboxylic acid, which was esterified with the fluorinated alcohol using dicyclohexylcarbodiimide (DCC) as dehydrating agent and N,N-dimethylaminopyridine (DMAP) as catalyst in dry tetrahydrofuran. The benzyl ether produced was converted to the corresponding phenol ester by removal of the benzyl protecting group which was achieved by stirring it at room temperature in an H_2 atmosphere in the presence of palladium on active carbon in ethyl acetate. The *para*-alkoxy benzoic acid was prepared with the previously prepared protected phenol; it was esterified with the previously prepared ester phenol **1** using the DCC/DMAP system. Details of synthetic procedure for series **A***n* is shown in the scheme. Series **B***n* were also prepared through similar procedure .

The phase transition temperatures of all the compounds were determined by DSC with a heating rate of 5.0°C min^{-1}. The mesomorphic textures were observed on polarizing optical textures for determining the types of mesophases. The transition temperatures of the series **A***n* and **B***n* shown in the Table 1 are the maxima of the transition peaks on each



Scheme 1. Synthetic procedure of series **A***n* and **B***n*, and molecular structures of series **C***n*. (a) $C_6H_5CH_2OC_6H_4OH$, DEAD/PPh_3, CH_2Cl_2, 0°C; (b) NaOH, C_2H_5OH/H_2O; (c) $HOCH_2CH_2(CF_2)_8F$, DCC/DMAP, THF, 0°C to r.t.; (d) Pd/C, ethyl acetate; (e) DCC/DMAP, THF, 0°C to r.t.

Table 1. Transition temperatures of the compounds **A***n* and **B***n* (Cr = crystal; CrX = crystal X; SmA = smectic A phase; SmB = smectic B phase; I = isotropic liquid; Recr = recrystallization)

Compound	*n*	Transition temperatures/°C
A*4*	4	Cr 96.8, **I** 90.3 Recr
A*5*	5	Cr 100.3, **I** 95.5 Recr
A*6*	6	Cr 97.5, **I** 92.7 Recr
A*7*	7	Cr 98.5, **I** 93.4 Recr
A*8*	8	Cr 98.9, **I** 92.3 Recr
A*9*	9	Cr 97.5, **I** 94.0 Recr
A*10*	10	Cr 99.6, **I** 93.6 Recr
A*11*	11	Cr 98.8, **I** 93.8 Recr
A*12*	12	Cr 99.4, **I** 95.7 Recr
B*4*	4	Cr 113.0, SmA 200.3, **I** 198.5, SmA 91.1 Recr
B*5*	5	Cr 112.5, SmA 196.1, **I** 193.8, SmA 98.6 Recr
B*6*	6	Cr 113.2, SmA 203.5, **I** 201.2, SmA 100.0 Recr
B*7*	7	Cr 114.4, SmA 198.4, **I** 197.0, SmA 105.3 Recr
B*8*	8	Cr 120.0, SmA 195.5, **I** 194.1, SmA 104.9 Recr
B*9*	9	Cr 112.1, SmB 121.0, SmA 186.8, **I** 185.6, SmA 120.0, SmB 105.0 Recr
B*10*	10	Cr 115.6, SmB 136.8, SmA 184.5, **I** 182.8, SmA 136.5, SmB 107.2 Recr
B*11*	11	Cr 113.6, CrX 119.8, SmB 141.9, SmA 178.6, **I** 176.0, SmA 141.2, SmB 110.4 Recr
B*12*	12	Cr 117.8, SmB 145.6, SmA 176.5, **I** 175.4, SmA 145.3, SmB 111.8 Recr

DSC trace. For the series **A***n*, no mesophases were identified. The melting points of them are about 100°C. Generally, molecules with even number of methylene units (*n*) exhibit higher melting points than those with odd ones, except **A***5*. Figure 1 plots the transition temperatures of the series **B***n* as a function of the number of methylene units (*n*) in the

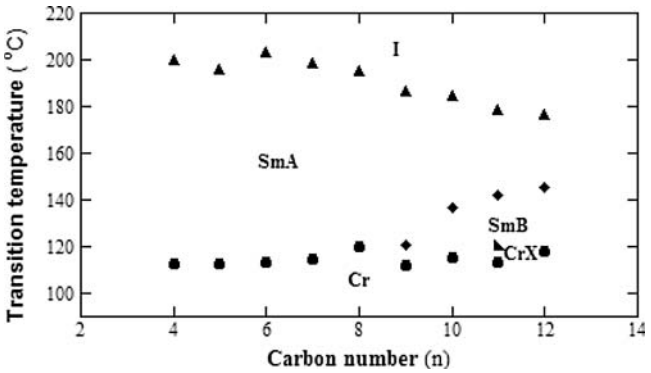


Figure 1. Transition behavior of series **B***n*: dependence of the transition temperatures on the number (*n*) of methylene units of the alkoxy chain.

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nonfluorinated chain. All of these compounds exhibit smectic mesophases. The melting points of them are about 110°C–120°C. When the number of methylene units (*n*) is smaller than 9, only enantiotropic SmA phase is identified. When it is larger, both enantiotropic SmA and SmB phases are identified. Generally, the clearing points of them decrease with increasing alkoxy chain length.

It has been reported by us that the series **C***n* exhibited both enantiotropic SmA and SmC* phases [22]. However, the series **A***n* are not liquid crystals. The phenyl group drives the clear points of the series **C***n* about 100°C higher than the melting points of the series **A***n*. Compare the series **B***n* with **C***n*, there are no significant difference among their clearing points. However, series **B***n* only exhibit enanotiotropic SmA and SmB phases. The bridged –COO– group seems play an important role in the organization of the molecules. Although the –COO– group decreases the stiffness of the molecules, it can increase the polarity and the length/diameter ratio of them. Series **B***n* were proposed applied in the liquid crystal display mixtures.

In summary, two series of chiral compounds with semifluorocarbon chains have been synthesized. One series of them exhibit enantiotropic SmA and SmB phases. Generally, the increase of the alkoxy chains' length can decrease their clearing points. The addition of phenyl rings in the core increases the clearing points; the addition of the –COO– group in the core plays an important role in the organization of the molecules.

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Experimental

Characterization

The structures of the final products and intermediates were determined by a variety of spectral methods. FT-IR spectra were taken on a Nicolet 6700 spectrometer, using KBr pellets of the solids. ^{1}H NMR spectra, with TMS as internal NMR standard, were recorded on a Varian NMR spectrometer (300 or 400 MHz); ^{19}F NMR spectra, with trifluoroacetic acid (TFA) as external standard, were recorded on a Varian NMR spectrometer (300 or 400 MHz). For the ^{19}F NMR spectra spectrum the high field was negative. Mass spectra (MS) were measured with MicroMass TOF-MS spectroscope. Field-emission electron microscopy (FESEM) images were taken on a Hitachi S-4700. Elemental analyses were performed on a Perkin Elmer series II CHNS/O analyzer 2400. The phase transition temperatures of the target compounds were measured by optical microscopy using a Leica DMRX polarization microscope fitted with a Linkam LTS350 hot stage and CI94 temperature controller and differential scanning calorimetry (DSC, TA Q-200 calorimeter with a data system and heating and cooling rate of 5°C min^{-1}). The transition temperatures reported in this paper were the peak values of the transition on DSC traces. Phase identification was made by comparing the observed textures with those reported in the literature.

Synthesis

All of the final compounds were purified by column chromatography on silica gel using petroleum ether (b.p. 60°C–90°C)/ethyl acetate (20:1) as eluant and then recrystallized from acetone/methanol. The following were structural characterization of target molecules.

Synthesis of Compound A4. A typical synthetic procedure for Sereis **A***n* and **B***n* is shown as following. 4-*n*-Butoxybenzoic acid (200 mg, 1.03 mmol), compound **1** (647 mg, 1.03 mmol), *N*,*N*′-dicyclohexylcarbodiimide (213 mg, 1.03 mmol), catalytic DMAP and dry

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THF (10 ml) were stirred under N_2 at room temperature for 48 h. The mixture was filtered and the residue was washed with THF. The collected filtrates were evaporated on a rotary evaporator. The residue was purified by flash chromatography and recrystallized from acetone/methanol to give 269 mg of white solid yield 32.5%. M.p. 96.8°C. IR (KBr, cm^{-1}): 2941, 2874, 1753, 1729, 1607, 1510, 1467, and 1198. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ (ppm) 0.99 (t, $J = 7.4$ Hz, 3H, $–CH_2CH_3$), 1.43–1.85 (m, 4H, aliphatic hydrogens). 1.63 (d, $J = 6.9$ Hz, 3H, $–CHCH_3$), 2.48 (tt, $J_1 = 18$ Hz, $J_2 = 6.3$ Hz, 2H, $–CH_2R_f$), 4.04 (t, $J = 6.5$ Hz, 2H, $–CH_2CH_2R_f$), 4.47 (t, $J = 6.5$ Hz, 2H, $–OCH_2$), 4.77 (q, $J = 6.7$ Hz, 1H, $–CHCH_3$), 6.90 (d, $J = 9.0$ Hz, 2H, ArH), 6.95 (d, $J = 9.0$ Hz, 2H, ArH), 7.10 (d, $J = 9.0$ Hz, 2H, ArH), 8.11 (d, $J = 9.0$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$, TFA): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 804 (M^++1), 313 ($C_4H_9O–C_6H_4–COO–C_6H_4–O–C^+H–CH_3$, 4.0), 177 ($C_4H_9–C_6H_4–CO^+$, 100), 121 ($HO–C_6H_4–CO^+$, 30.0). Elemental analysis: calculated for $C_{30}H_{25}O_6F_{17}$, C 44.79, H 3.13%; found, C 45.28, H 3.02%.

Synthesis of Compound A5. M.p. 100.3°C. IR (KBr, cm^{-1}): 2942, 2871, 1753, 1729, 1607, 1511, 1468, and 1198. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ (ppm) 0.96 (t, $J = 7.1$ Hz, 3H, $–CH_2CH_3$), 1.40–1.88 (m, 6H, aliphatic hydrogens). 1.64 (d, $J = 6.9$ Hz, 3H, $–CHCH_3$), 2.49 (tt, $J_1 = 18.0$ Hz, $J_2 = 6.5$ Hz, 2H, $–CH_2R_f$), 4.05 (t, $J = 6.6$ Hz, 2H, $–CH_2CH_2R_f$), 4.49 (t, $J = 6.5$ Hz, 2H, $–OCH_2$), 4.78 (q, $J = 6.6$ Hz, 1H, $–CHCH_3$), 6.91 (d, $J = 9.3$ Hz, 2H, ArH), 6.97 (d, $J = 9.0$ Hz, 2H, ArH), 7.11 (d, $J = 9.0$ Hz, 2H, ArH), 8.12 (d, $J = 8.7$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 818 (M^++1), 337 ($C_5H_{13}O–C_6H_4–COO–C_6H_4–O–C^+H–CH_3$, 4.0), 191 ($C_5H_{13}–C_6H_4–CO^+$, 100), 121 ($HO–C_6H_4$-$CO^+$, 15.8). Elemental analysis: calculated for $C_{31}H_{27}O_6F_{17}$, C 45.49, H 3.32%; found, C 45.53, H 3.17%.

Synthesis of Compound A6. M.p. 97.5°C. IR (KBr, cm^{-1}): 2941, 2869, 1753, 1729, 1608, 1511, 1468, and 1198. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ (ppm) 0.93 (t, $J = 7.1$ Hz, 3H, $–CH_2CH_3$), 1.23–1.88(m, 8H, aliphatic hydrogens). 1.65 (d, $J = 6.9$ Hz, 3H, $–CHCH_3$), 2.50 (tt, $J_1 = 18.3$ Hz, $J_2 = 6.5$ Hz, 2H, $–CH_2R_f$), 4.05 (t, $J = 6.6$ Hz, 2H, $–CH_2CH_2R_f$), 4.49 (t, $J = 6.3$ Hz, 2H, $–OCH_2$), 4.79 (q, $J = 6.8$Hz, 1H, $–CHCH_3$), 6.92 (d, $J = 9.0$ Hz, 2H, ArH), 6.97 (d, $J = 9.0$ Hz, 2H, ArH), 7.17 (d, $J = 9.0$ Hz, 2H, ArH), 8.13 (d, $J = 8.7$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 832 (M^++1), 341 ($C_6H_{13}O–C_6H_4–COO–C_6H_4–O–C^+H–CH_3$, 4.0), 205 ($C_6H_{13}–C_6H_4–CO^+$, 100), 121 ($HO–C_6H_4–CO^+$, 34.2). Elemental analysis: calculated for $C_{32}H_{29}O_6F_{17}$, C 46.16, H 3.51%; found, C 45.77, H 3.37%.

Synthesis of Compound A7. M.p. 98.5°C. IR (KBr, cm^{-1}): 2939, 2866, 1753, 1731, 1607, 1512, 1468, and 1198. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ (ppm) 0.91 (t, $J = 6.8$ Hz, 3H, $–CH_2CH_3$), 1.33–1.88 (m, 10H, aliphatic hydrogens). 1.65 (d, $J = 6.6$ Hz, 3H, $–CHCH_3$), 2.50 (tt, $J_1 = 18.3$ Hz, $J_2 = 6.2$ Hz, 2H, $–CH_2R_f$), 4.05 (t, $J = 6.6$ Hz, 2H, $–CH_2CH_2R_f$), 4.49 (t, $J = 6.5$ Hz, 2H, $–OCH_2$), 4.79 (q, $J = 6.8$ Hz, 1H, $–CHCH_3$), 6.92 (d, $J = 9.0$ Hz, 2H, ArH), 6.97 (d, $J = 9.0$ Hz, 2H, ArH), 7.12 (d, $J = 9.0$ Hz, 2H, ArH), 8.13 (d, $J = 9.0$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 846 (M^++1), 355 ($C_7H_{15}O–C_6H_4–COO–C_6H_4–O–C^+H–CH_3$, 15.8),

219 (C_7H_{15}–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 57.5). Elemental analysis: calculated for $C_{33}H_{31}O_6F_{17}$, C 46.82, H 3.69%; found, C 46.35, H 3.52%.

Synthesis of Compound A8. M.p. 98.9°C. IR (KBr, cm^{-1}): 2925, 2855, 1753, 1731, 1607, 1512, 1468, and 1198. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ (ppm) 0.89 (t, $J = 5.1$ Hz, 3H, –CH_2CH_3), 1.30–1.85 (m, 12H, aliphatic hydrogens). 1.63 (d, $J = 5.1$ Hz, 3H, –$CHCH_3$), 2.48 (tt, $J_1 = 13.5$ Hz, $J_2 = 4.7$ Hz, 2H, –CH_2R_f), 4.04 (t, $J = 4.8$ Hz, 2H, –$CH_2CH_2R_f$), 4.48 (t, $J = 4.7$ Hz, 2H, –OCH_2), 4.77 (q, $J = 5.1$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 6.6$ Hz, 2H, ArH), 6.96 (d, $J = 6.9$ Hz, 2H, ArH), 7.11 (d, $J = 6.6$ Hz, 2H, ArH), 8.11 (d, $J = 6.6$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 860 (M^++1), 369 ($C_8H_{17}O$–C_6H_4–COO–C_6H_4–O–C^+H–CH_3, 2.1), 233 (C_8H_{17}–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 30.0). Elemental analysis: calculated for $C_{34}H_{33}O_6F_{17}$, C 47.45, H 3.87%; found, C 46.88, H 3.92%.

Synthesis of Compound A9. M.p. 97.5°C. IR (KBr, cm^{-1}): 2924, 2854, 1753, 1731, 1607, 1512, 1468, and 1199. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ (ppm) 0.89 (t, $J = 6.6$ Hz, 3H, –CH_2CH_3), 1.25–1.86 (m, 14H, aliphatic hydrogens). 1.63 (d, $J = 3.5$ Hz, 3H, –$CHCH_3$), 2.48 (tt, $J_1 = 18.3$ Hz, $J_2 = 6.3$ Hz, 2H, –CH_2R_f), 4.03 (t, $J = 6.5$ Hz, 2H, –$CH_2CH_2R_f$), 4.48 (t, $J = 6.5$ Hz, 2H, –OCH_2), 4.77 (q, $J = 6.8$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 9.3$ Hz, 2H, ArH), 6.96 (d, $J = 9.0$ Hz, 2H, ArH), 7.11 (d, $J = 9.0$ Hz, 2H, ArH), 8.11 (d, $J = 9.0$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 874 (M^++1), 383 ($C_9H_{19}O$–C_6H_4–COO–C_6H_4–O–C^+H–CH_3, 2.5), 247 ($C_9H_{19}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 33.3). Elemental analysis: calculated for $C_{35}H_{35}O_6F_{17}$, C 48.06, H 4.03%; found, C 47.68, H 3.75%.

Synthesis of Compound A10. M.p. 99.6°C. IR (KBr, cm^{-1}): 2923, 2853, 1753, 1732, 1607, 1512, 1471, and 1199. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.89 (t, $J = 6.6$ Hz, 3H, –CH_2CH_3), 1.23–1.85 (m, 16H, aliphatic hydrogens). 1.64 (d, $J = 6.8$ Hz, 3H, –$CHCH_3$), 2.48 (tt, $J_1 = 18.0$ Hz, $J_2 = 6.4$ Hz, 2H, –CH_2R_f), 4.04 (t, $J = 6.6$ Hz, 2H, –$CH_2CH_2R_f$), 4.48 (t, $J = 6.2$ Hz, 2H, –OCH_2), 4.77 (q, $J = 6.7$Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 9.2$ Hz, 2H, ArH), 6.96 (d, $J = 8.8$ Hz, 2H, ArH), 7.10 (d, $J = 9.2$ Hz, 2H, ArH), 8.11 (d, $J = 8.8$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 888 (M^++1), 397 ($C_{10}H_{21}O$–C_6H_4–COO–C_6H_4–O–C^+H–CH_3,2.5), 261 ($C_{10}H_{21}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4-CO^+, 27.5). Elemental analysis: calculated for $C_{36}H_{37}O_6F_{17}$, C 48.66, H 4.20%; found, C 48.29, H 3.73%.

Synthesis of Compound A11. M.p. 98.8°C. IR (KBr, cm^{-1}): 2922, 2853, 1753, 1734, 1607, 1512, 1468, and 1200. ^{1}H NMR (300 MHz, $CDCl_3$, TMS): δ (ppm) 0.88 (t, $J = 6.8$ Hz, 3H, –CH_2CH_3), 1.27–1.86 (m, 18H, aliphatic hydrogens). 1.63 (d, $J = 6.9$ Hz, 3H, –$CHCH_3$), 2.48 (tt, $J_1 = 18.3$ Hz, $J_2 = 6.5$ Hz, 2H, –CH_2R_f), 4.03 (t, $J = 6.6$ Hz, 2H, –$CH_2CH_2R_f$), 4.48 (t, $J = 6.3$ Hz, 2H, –OCH_2), 4.77 (q, $J = 6.8$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 9.3$ Hz, 2H, ArH), 6.95 (d, $J = 9.0$ Hz, 2H, ArH), 7.10 (d, $J = 9.0$ Hz, 2H, ArH), 8.11 (d, $J = 9.0$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 902 (M^++1), 411 ($C_{11}H_{23}O$–C_6H_4–COO–C_6H_4–O–C^+H–CH_3,

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2.5), 275 ($C_{11}H_{23}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 10.8). Elemental analysis: calculated for $C_{37}H_{39}O_6F_{17}$, C 49.23, H 4.35%; found, C 48.98, H 4.19%.

Synthesis of Compound A12. M.p. 99.4°C. IR (KBr, cm^{-1}): 2922, 2853, 1733, 1734, 1607, 1512, 1468, and 1200. ^{1}H NMR (300 MHz, $CDCl_3$), δ (ppm) 0.88 (t, $J = 6.6$ Hz, 3H, –CH_2CH_3), 1.23–1.85 (m, 20H, aliphatic hydrogens). 1.63 (d, $J = 6.8$ Hz, 3H, –$CHCH_3$), 2.49 (tt, $J_1 = 18$ Hz, $J_2 = 6.4$ Hz 2H, –CH_2R_f), 4.04 (t, $J = 6.6$ Hz, 2H, –$CH_2CH_2R_f$), 4.48 (t, $J = 6.4$ Hz, 2H, –OCH_2), 4.77 (q, $J = 6.8$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 9.2$ Hz, 2H, ArH), 6.95 (d, $J = 8.8$ Hz, 2H, ArH), 7.10 (d, $J = 9.2$ Hz, 2H, ArH), 8.11 (d, $J = 8.8$ Hz, 2H, ArH). ^{19}F NMR (300 MHz, $CDCl_3$): δ (ppm) −48.56 (t, $J = 12.27$ Hz, 2F), −45.98 (s, 2F), −45.16 (s, 2F), (−44.39 to −44.12) (m, 6F), −36.01 (m, 2F), −3.16 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 916 (M^++1), 425 (–C_6H_4–COO–C_6H_4–O–C^+H–CH_3, 1.1), 289 ($C_{12}H_{25}$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 14.2). Elemental analysis: calculated for $C_{38}H_{41}O_6F_{17}$, C 49.79, H 4.51%; found, C 50.13, H 4.51%.

Synthesis of Compound B4. M.p. 113.0°C. IR (KBr, cm^{-1}): 2958, 2872, 1753, 1736, 1605, 1510, 1414, and 1198. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.98 (t, $J = 7.8$ Hz, 3H, –CH_2CH_3), 1.45–1.84 (m, 4H, aliphatic hydrogens), 1.62 (d, $J = 6.8$ Hz, 3H, –$CHCH_3$), 2.47 (tt, $J_1 = 18$ Hz, $J_2 = 6.4$ Hz, 2H, –CH_2R_f), 4.04 (t, $J = 6.4$ Hz, 2H, –$CH_2CH_2R_f$), 4.47 (t, $J = 6.2$ Hz, 2H, –OCH_2), 4.76 (q, $J = 6.5$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 9.2$ Hz, 2H, ArH), 6.97 (d, $J = 8.8$ Hz, 2H, ArH), 7.11 (d, $J = 8.8$ Hz, 2H, ArH), 7.34 (d, $J = 8.8$ Hz, 2H, ArH), 8.13 (d, $J = 8.8$ Hz, 2H, ArH), 8.24 (d, $J = 8.8$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F), (−43.81−−44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 924 (M^++1),297 (C_4H_9O–C_6H_4–COO–C_6H_4–CO^+, 20), 177 (C_4H_9O–C_6H_4–CO^+, 100), 121 (HO–C_6H_4-CO^+,15). Elemental analysis: calculated for $C_{37}H_{29}O_8F_{17}$, C 48.06, H 3.16%; found, C 47.30, H 3.30%.

Synthesis of Compound B5. M.p. 112.5°C. IR (KBr, cm^{-1}), 2937, 2871, 1753, 1736, 1606, 1511, 1415, and 1199. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.93 (t, $J = 6.8$ Hz, 3H, –CH_2CH_3), 1.34–1.87 (m, 6H, aliphatic hydrogens). 1.62 (d, $J = 6.8$ Hz, 3H, –$CHCH_3$), 2.47 (tt, $J_1 = 19.8$ Hz, $J_2 = 6.8$ Hz, 2H, –CH_2R_f), 4.04 (t, $J = 6.4$ Hz, 2H, –$CH_2CH_2R_f$), 4.47 (t, $J = 6.4$ Hz, 2H, –OCH_2), 4.76 (q, $J = 6.6$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 6.8$ Hz, 2H, ArH), 6.97 (d, $J = 8.0$ Hz, 2H, ArH), 7.11 (d, $J = 8.8$ Hz, 2H, ArH), 7.34 (d, $J = 8.4$ Hz, 2H, ArH), 8.13 (d, $J = 8.0$ Hz, 2H, ArH), 8.24 (d, $J = 8.0$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F), (−43.81 to −44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 938 (M^++1), 311 ($C_5H_{11}O$–C_6H_4–COO–C_6H_4–CO^+, 16), 191 ($C_5H_{11}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 20). Elemental analysis: calculated for $C_{38}H_{31}O_8F_{17}$, C 48.63, H 3.33%; found, C 48.73, H 4.57%.

Synthesis of Compound B6. M.p. 113.2°C. IR (KBr, cm^{-1}), 2938, 2872, 1752, 1736, 1606, 1511, 1416, and 1199. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.90 (t, $J = 6.8$ Hz, 3H, –CH_2CH_3), 1.26–1.86 (m, 8H, aliphatic hydrogens). 1.62 (d, $J = 6.8$ Hz, 3H, –$CHCH_3$), 2.47 (tt, $J_1 = 18.2$ Hz, $J_2 = 6.0$ Hz, 2H, –CH_2R_f), 4.04 (t, $J = 6.6$ Hz, 2H, –$CH_2CH_2R_f$), 4.47 (t, $J = 6.2$ Hz, 2H, –OCH_2), 4.76 (q, $J = 6.5$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 8.8$ Hz, 2H, ArH), 6.97 (d, $J = 8.8$ Hz, 2H, ArH), 7.11 (d, $J = 8.8$ Hz, 2H, ArH), 7.34 (d, $J = 8.8$ Hz, 2H, ArH), 8.13 (d, $J = 8.8$ Hz, 2H, ArH), 8.24 (d, $J = 8.8$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F),

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(−43.81 to −44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 952 (M^++1), 325 ($C_6H_{13}O$–C_6H_4–COO–C_6H_4–CO^+, 22), 205 ($C_6H_{13}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 30). Elemental analysis: calculated for $C_{39}H_{33}O_8F_{17}$, C 49.17, H 3.49%; found, C 48.61, H 3.55%.

Synthesis of Compound B7. M.p. 114.4°C. IR (KBr, cm^{-1}), 2925, 2855, 1753, 1737, 1606, 1511, 1415, and 1197. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.88 (t, $J = 7.8$ Hz, 3H, –CH_2CH_3), 1.25–1.89 (m, 10H, aliphatic hydrogens). 1.62 (d, $J = 6.8$ Hz, 3H, –$CHCH_3$), 2.47 (tt, $J_1 = 18.2$ Hz, $J_2 = 7.6$ Hz, 2H, –CH_2R_f), 4.03 (t, $J = 6.2$ Hz, 2H, –$CH_2CH_2R_f$), 4.46 (t, $J = 6.6$ Hz, 2H, –OCH_2), 4.76 (q, $J = 6.6$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 8.0$ Hz, 2H, ArH), 6.97 (d, $J = 8.4$ Hz, 2H, ArH), 7.11 (d, $J = 8.4$ Hz, 2H, ArH), 7.34 (d, $J = 8.0$ Hz, 2H, ArH), 8.13 (d, $J = 8.4$ Hz, 2H, ArH), 8.23 (d, $J = 8.4$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F), (−43.81 to −44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 966 (M^++1),339 ($C_7H_{15}O$–C_6H_4–COO–C_6H_4–CO^+, 40), 219 ($C_7H_{15}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 64). Elemental analysis: calculated for $C_{40}H_{35}O_8F_{17}$, C 49.70, H 3.65%; found, C 49.60, H 3.58%.

Synthesis of Compound B8. M.p. 120.0°C. IR (KBr, cm^{-1}), 2924, 2854, 1753, 1736, 1606, 1511, 1415, and 1199. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.87 (t, $J = 5.8$ Hz, 3H, –CH_2CH_3), 1.15–1.86 (m, 12H, aliphatic hydrogens). 1.62 (d, $J = 6.4$ Hz, 3H, –$CHCH_3$), 2.48 (tt, $J_1 = 18$ Hz, $J_2 = 7.8$ Hz, 2H, –CH_2R_f), 4.03 (t, $J = 6.8$ Hz, 2H, –$CH_2CH_2R_f$), 4.46 (t, $J = 4.4$ Hz, 2H,–OCH_2), 4.76 (q, $J = 6.4$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 8.8$ Hz, 2H, ArH), 6.96 (d, $J = 8.8$ Hz, 2H, ArH), 7.10 (d, $J = 8.8$ Hz, 2H, ArH), 7.33 (d, $J = 8.8$ Hz, 2H, ArH), 8.13 (d, $J = 8.8$ Hz, 2H, ArH), 8.23 (d, $J = 8.8$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F), (−43.81 to −44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 980 (M^++1),353 ($C_8H_{17}O$–C_6H_4–COO–C_6H_4–CO^+, 12), 233 ($C_8H_{17}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 18). Elemental analysis: calculated for $C_{41}H_{37}O_8F_{17}$, C 50.21, H 3.80%; found, C 49.96%H 3.69%.

Synthesis of Compound B9. M.p. 112.1°C. IR (KBr, cm^{-1}), 2923, 2853, 1753, 1737, 1606, 1511, 1416, and 1198. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.89 (t, $J = 6.6$ Hz, 3H, –CH_2CH_3), 1.20–1.89 (m, 14H, aliphatic hydrogens). 1.64 (d, $J = 6.4$ Hz, 3H, –$CHCH_3$), 2.49 (tt, $J_1 = 18.6$ Hz, $J_2 = 6.2$ Hz, 2H, –CH_2R_f), 4.06 (t, $J = 6.6$ Hz, 2H, –$CH_2CH_2R_f$), 4.49 (t, $J = 6.2$ Hz, 2H, –OCH_2), 4.79 (q, $J = 6.6$ Hz, 1H, –$CHCH_3$), 6.92 (d, $J = 8.8$ Hz, 2H, ArH), 6.99 (d, $J = 8.8$ Hz, 2H, ArH), 7.13 (d, $J = 8.8$ Hz, 2H, ArH), 7.31 (d, $J = 8.8$ Hz, 2H, ArH), 8.15 (d, $J = 8.8$ Hz, 2H, ArH), 8.24 (d, $J = 8.8$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F), (−43.81 to −44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel.int.): 994 (M^++1), 367 ($C_9H_{19}O$–C_6H_4–COO–C_6H_4–CO^+, 13), 247 ($C_9H_{19}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 20). Elemental analysis: calculated for $C_{42}H_{39}O_8F_{17}$, C 50.71, H 3.95%; found, C 50.49, H 3.87%.

Synthesis of Compound B10. M.p. 115.6°C. IR (KBr, cm^{-1}), 2922, 2852, 1753, 1738, 1605, 1510, 1415, and 1198. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.87 (t, $J = 6.4$ Hz, 3H, –CH_2CH_3), 1.20–1.85 (m, 16H, aliphatic hydrogens). 1.62 (d, $J = 5.4$ Hz, 3H, –$CHCH_3$), 2.47 (tt, $J_1 = 19.6$ Hz, $J_2 = 6.4$ Hz, 2H, –CH_2R_f), 4.04 (t, $J = 6.4$ Hz, 2H, –$CH_2CH_2R_f$), 4.47 (t, $J = 6.4$ Hz, 2H, –OCH_2), 4.76 (q, $J = 6.9$ Hz, 1H, –$CHCH_3$),

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6.90 (d, $J = 8.4$ Hz, 2H, ArH), 6.97 (d, $J = 8.0$ Hz, 2H, ArH), 7.11 (d, $J = 8.0$ Hz, 2H, ArH), 7.33 (d, $J = 8.4$ Hz, 2H, ArH), 8.13 (d, $J = 8.0$ Hz, 2H, ArH), 8.24 (d, $J = 8.84$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F), (−43.81 to −44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 1008 (M^++1), 381 ($C_{10}H_{21}O$–C_6H_4–COO–C_6H_4–CO^+, 14), 261 ($C_{10}H_{21}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 30). Elemental analysis: calculated for $C_{43}H_{41}O_8F_{17}$, C 51.20, H 4.10%; found, C 50.25, H 4.36%.

Synthesis of Compound B11. M.p. 113.6°C. IR (KBr, cm^{-1}), 2922, 2852, 1753, 1738, 1605, 1510, 1415, and 1198. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.87 (t, $J =$ 6.8 Hz, 3H, –CH_2CH_3), 1.20–1.86 (m, 18H, aliphatic hydrogens). 1.62 (d, $J = 6.8$ Hz, 3H, –$CHCH_3$), 2.47 (tt, $J_1 = 18.6$ Hz, $J_2 = 6.6$ Hz, 2H, –CH_2R_f), 4.03 (t, $J = 6.6$ Hz, 2H, –$CH_2CH_2R_f$), 4.47 (t, $J = 6.4$ Hz, 2H, –OCH_2), 4.76 (q, $J = 6.8$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 8.8$ Hz, 2H, ArH), 6.97 (d, $J = 8.8$ Hz, 2H, ArH), 7.11 (d, $J = 9.2$ Hz, 2H, ArH), 7.34 (d, $J = 8.8$ Hz, 2H, ArH), 8.13 (d, $J = 8.8$ Hz, 2H, ArH), 8.24 (d, $J = 8.8$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F), (−43.81 to −44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 1022 (M^++1), 395 ($C_{11}H_{23}O$–C_6H_4–COO–C_6H_4–CO^+, 10), 275 ($C_{11}H_{23}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 18). Elemental analysis: calculated for $C_{44}H_{43}O_8F_{17}$, C 51.67, H 4.24%; found, C 50.99, H 4.57%.

Synthesis of Compound B12. M.p. 117.8°C. IR (KBr, cm^{-1}), 2921, 2851, 1755, 1738, 1605, 1510, 1415, and 1199. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ (ppm) 0.86 (t, $J =$ 6.6 Hz, 3H, –CH_2CH_3), 1.20–1.86 (m, 20H, aliphatic hydrogens). 1.62 (d, $J = 6.8$ Hz, 3H, –$CHCH_3$), 2.47 (tt, $J_1 = 18.2$ Hz, $J_2 = 6.0$ Hz, 2H, –CH_2R_f), 4.03 (t, $J = 6.6$ Hz, 2H, –$CH_2CH_2R_f$), 4.47 (t, $J = 6.2$ Hz, 2H, –OCH_2), 4.76 (q, $J = 6.8$ Hz, 1H, –$CHCH_3$), 6.90 (d, $J = 8.8$ Hz, 2H, ArH), 6.97 (d, $J = 8.8$ Hz, 2H, ArH), 7.11 (d, $J = 9.2$ Hz, 2H, ArH), 7.33 (d, $J = 8.8$ Hz, 2H, ArH), 8.13 (d, $J = 8.4$ Hz, 2H, ArH), 8.24 (d, $J = 8.8$ Hz, 2H, ArH). ^{19}F NMR (400 MHz, $CDCl_3$): δ (ppm) −48.28 (t, $J = 17.6$ Hz, 2F), −45.70 (s, 2F), −44.83 (s, 2F), (−43.81 to −44.12) (m, 6F), −35.71 (m, 2F), −2.91 (t, $J = 9.87$ Hz, 3F). MS m/z (rel. int.): 1036 (M^++1), 409 ($C_{12}H_{25}O$–C_6H_4–COO–C_6H_4–CO^+, 12), 289 ($C_{12}H_{25}O$–C_6H_4–CO^+, 100), 121 (HO–C_6H_4–CO^+, 30). Elemental analysis: calculated for $C_{45}H_{45}O_8F_{17}$, C 52.13, H 4.37%; found, C 51.17, H 4.35%.

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