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METHYL GALLATE AS THE FRAMEWORK FOR THE CONSTRUCTION OF FLUOROUS BUILDING BLOCKS

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Methyl gallate was used as the framework for the construction of the building blocks 3–5 with three fluorous ponytails. Fluorous gallate 3 was prepared from methyl gallate in three different pathways. The first is based on perfluorohexyl iodide addition to the corresponding allylderivative 1 and reduction of C-I bond in adduct 2; other ways are based on direct O-alkylation of methyl gallate by 3-(perfluorohexyl)propyl triflate and 3-(perfluorohexyl) propyl iodide, respectively. Reduction of polyfluoroalkylated gallate 3 provided benzyl alcohol 4, which can be easily converted to corresponding benzyl bromide 5.

Keywords: Dendron; fluorinated gallate; fluorous building blocks; methyl gallate; polyfluoroalkylation

INTRODUCTION

Many synthetic chemists interested in chemistry of highly fluorinated compounds utilize various polyfluorinated building blocks for the construction of sophisticated target structures. Unfortunately, required polyfluorinated units are not commercially available and therefore in all cases must be synthesized. Polyfluorinated blocks based on the gallate unit have been already used for the constructions of highly fluorinated complex structures by some research groups.^[1–11] The advantages of the gallate as framework for the construction of polyfluorinated building blocks are the following: first, there is a possibility to connect three polyfluorinated chains. Second, connection of polyfluorinated chains via an ether bond to the gallate is sufficiently stable for the next synthetic transformations, and third, the ester group of the gallate can further easily modified into other reactive functional group.

The most frequently used polyfluorinated gallate has three polyfluorinated chains, which are each separated from the aromatic part by butylene-oxo spacers.^[1-6] In 2008, we described utilization of the gallate with a three-carbon

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spacer,^[11] but no mention of its preparation and utilization have been published yet. Therefore, we decided to deeply explore possibilities of preparation of the polyfluorinated gallate with the three-carbon spacer and try to judge which pathway is most suitable regarding yields and synthetic workability. A further criterion was price of polyfluoroalkylating agents: perfluorohexyl iodide is approximately 10 times cheaper than polyfluorinated alkanol (precursor for the preparation of corresponding triflate) or 3-(perfluorohexyl)propyl iodide, respectively.

RESULTS AND DISCUSSION

There exist two general possibilities for substituting gallate with polyfluorinated chains. The first one is a simple alkylation by means of polyfluorinated alkylating agent as a iodide or triflate. The second possibility is based on radical addition of perfluoroalkyl iodide on corresponding allyl gallate derivative. Our synthetic strategy to reach the final fluorous gallate **3** (Scheme 1) was devided into individual three synthetic pathways. The first one is based on an addition–reduction method, the second utilizes 3-(perfluoroalkyl)propyl triflate, and the third pathway uses 3-(perfluoroalkyl)propyl iodide.

The first pathway is based on perfluorohexyl iodide addition to the corresponding allylderivative 1 and following reduction of C-I bond of adduct 2. First, methyl gallate was transformed into trially derivative 1 by reaction with twofold molar excess of allyl bromide in the presence of potassium carbonate in acetonitrile.^[12,13] The reaction afforded methyl ester of 3,4,5-tris(allyloxy)benzoic acid 1 in quantitative yield (Scheme 1). In the next step, radical addition of perfluorohexyl iodide to the double bond of allyl group was used for the connection of the fluorinated chains. There are exist many approaches for the radical initiation.^[14] We used three different radical initiators: sodium dithionite,^[15,16] azobisisobutyronitrile (AIBN),^[14,16] and triethyl borane^[17,18] (Scheme 1). While using sodium dithionite in the acetonitrile-water system, yields of additions were greater (84%) in the case of using of triethyl borane in hexane or AIBN without any solvent, yields were lower; (79% and 64% respectively). In the case of using Et_3B and AIBN, the conversion of triallyl derivative to desired tris(polyfluoroalkyl) derivative was not complete, and the reaction mixture included tris- and bis(polyfluoroalkyl) derivatives, even if the reaction time was prolonged or greater excess of perfluorohexyl iodide was used. Other systems for initiating the radical addition of $C_6F_{13}I$ [Pd(PPh)₄, Ph₃P, dibenzoyl peroxide, irradiation] failed.

For the reduction of C-I bond in adduct **2**, we used a method successfully applied previously on threefold polyfluoroalkylated iododerivatives,^[16] hydrogenation on a palladium catalyst under atmospheric pressure. Reaction runs in a methanol–ethyl acetate mixture in the presence of sodium acetate (Scheme 1). The reduction afforded polyfluorinated gallate **3** in a yield of 96%. Owerall yield of this three-step pathway leading to fluorous gallate **3** was around 90%.

Next, two pathways were based on direct *O*-alkylation of the gallate. The second way to polyfluorinated gallate **3** is based on the reaction of methyl gallate with 3-(perfluorohexyl)propyl triflate (Scheme 1). We have already published this approach.^[11,19] In the original work,^[11,19] the triflate afforded polyfluorinated gallate **3** in 80% yield. We focused our effort on increasing the yield. The original method,



Scheme 1. Three approaches to polyfluoroalkylated gallate.

which we recently published,^[11] utilizes tetrahydrofuran (THF) as a solvent for the alkylation. We tried to replace the THF by another solvent or mixtures of solvents. The selection of the solvent is strongly limited by solubility of methyl gallate. Herein we report an improved method for the preparation of this compound as follows: methyl gallate was transformed to sodium salt by reaction with sodium hydride in dimethylformamide (DMF) and subsequently reacted with solution of 3-(perfluoro-hexyl)propyl triflate in DMF. Desired product **3** was obtained in the yield of 91% (lit.^[11] 80%). On the other hand, when sodium salt of methyl gallate was generated in DMF and polyfluoroalkylated triflate was added in THF, the yield was less, 85%.

The third way to polyfluorinated gallate **3** is based on alkylation of methyl gallate by 3-(perfluorohexyl)propyl iodide. Several examples of polyfluoroalkylation of phenolic –OH group by polyfluoroalkylated iodides have already been published, using Cs₂CO₃ in DMF,^[20] K₂CO₃ in MeCN,^[21,22] or K₂CO₃ in DMF.^[2] 3-(Perfluoroalkyl)propyl iodides have been also used for direct polyfluoroalkylation of N–,^[23,24] C–,^[8,25] and S-nucleophiles.^[26] For polyfluoroalkylation, we used potassium carbonate as a base in DMF (Scheme 1). The reaction was carried out with the excess of polyfluorinated iodide (1.5 molar equivalent per phenolic –OH group). Under these conditions, the conversion of methyl gallate was complete, and the reaction provided tris(polyfluoroalkylated) gallate **3** in quantitative yield. Part of the excess of 3-(perfluorohexyl)propyl iodide was recovered by column chromatography and reused. The same results were obtained when sodium hydride in DMF was used. When using less then 1.2 molar equivalents of 3-(perfluorohexyl)propyl iodide per phenolic –OH group, the conversion of methyl gallate to target tris(polyfluoroalkyl) derivative was not complete.

Further utilization of the polyfluoroalkylated methyl-gallate **3** as a building block depends on the reactive functional group, which was generated from ester, because the ester group in itself is not suitable for connection to the substrate. On the other hand, the ester function of the gallate can be easily transformed into another more reactive benzylic function as an alcohol or an bromide. Thus, in the



Scheme 2. Preparation of fluorous benzyl alcohol 4 and benzyl bromide 5.

first step, we reduced the ester group, providing corresponding benzyl alcohol 4. Recently we reported^[11] this preparation using lithium aluminium hydride in diethyl ether $(-78^{\circ}C \text{ to rt}, 1 \text{ h})$ with the yield of 93%. Herein we presented an improved method for the reduction using lithium aluminium hydride in anhydrous THF (0°C to rt, overnight) with the greater yield, 98% (Scheme 2).

In the next step, polyfluoroalkylated benzyl alcohol **4** was transformed into corresponding benzyl bromide **5** by the reaction with phosphorus tribromide in diethyl ether.^[27] The bromination afforded a corresponding bromide in almost quantitative yield (98%) (Scheme 2).

CONCLUSIONS

Gallate **3** with three fluorous ponytails was prepared from methyl gallate in three different pathways. The most economically advantageous pathway appeared to be a three-step reaction sequence including perfluorohexyl iodide addition, which afforded final product **3** in an overall yield of 90%. In terms of ease of the synthesis of **3**, using commercially available 3-(perfluorohexyl)propyl iodide as polyfluoro-alkylating agent appeared as the better choice. It is surprising that the third pathway, which utilized the most reactive polyfluoroalkylating agent, polyfluorinated triflate, afforded desired gallate **3** in less yield than corresponding iodide, probably due to

side elimination of the triflate under basic conditions. Polyfluoroalkylated gallate 3 can be easily converted into polyfluoroalkylated benzyl alcohol 4 and benzyl bromide 5, respectively. Compounds 3–5 can be used as prospective building blocks in dendrimer and supramolecular chemistry or as the fluorous tags or reagents in organic synthesis, including fluorous separation techniques.

EXPERIMENTAL

General Comments and Chemicals

NMR spectra were recorded on a Varian Gemini 300 HC (FT, ¹H at 300 MHz, ¹³C at 75 MHz, ¹⁹F at 281 MHz) instrument using TMS and CFCl₃ as the internal standards. Chemical shifts are quoted in parts per million (δ scale; s singlet, bs broad singlet, d doublet, t triplet, q quadruplet, m multiplet), coupling constants *J*, in hertz, and solvents were CDCl₃ and CD₃COCD₃.

All chemicals were purchased from Sigma-Aldrich; silica gel (60–100 μ m) was from Merck. Solvents were purchased from Penta and dried according to standard procedures.

Chemistry

Preparation of 3-(perfluorohexyl)propyl triflate. Solution of triflic anhydride (42.81 g, 152 mmol) in DCM (250 mL) was cooled to -30° C (dry ice–ethanol bath), and a solution of 3-(perfluorohexyl)propanol (52.16 g; 138 mmol) and pyridine (10.92 g; 138 mmol) in DCM (250 mL) was added dropwise while stirring at -30° C. The reaction mixture was stirred for an additional 30 min at -30° C and slowly warmed up to rt (45 min). Insoluble salts were filtered and washed with pentane (150 mL). Volatile compounds were removed under reduced pressure, and the crude product was distilled under high vacuum (bp=71–76°C/2 Torr) yielded 62.05 g (88%) of colorless liquid.

¹H NMR (CDCl₃, δ ppm): 2.36–2.09 (m, 4H, CH₂CH₂R_F), 4.60 (t, 2H, ${}^{3}J_{\text{HH}} = 5.72$ Hz, CH₂OTf); ¹³C NMR (CDCl₃, δ ppm): 20.9 (t, 1C, ${}^{3}J_{\text{CF}} = 4$ Hz), 27.1 (t, 1C, ${}^{2}J_{\text{CF}} = 22$ Hz), 75.3 (s, 1C), 118.7 (q, 1C, ${}^{1}J_{\text{CF}} = 319$ Hz), 105.0–125.0 (m, 6C); ¹⁹F NMR (CDCl₃, δ ppm): –75.3 (s, 3F), –81.5 (t, 3F, ${}^{3}J_{\text{FF}} = 10.6$ Hz), –114.9 (m, 2F), –122.4 (m, 2F), –123.4 (m, 2F), –124.0 (m, 2F), –126.7 (m, 2F). Anal. calcd. for C₁₀H₆F₁₆O₃S: C, 23.54; H, 1.19. Found: C, 23.67; H, 1.07.

Preparation of methyl ester of 3,4,5-tris(allyloxy)benzoic acid (1). Methyl gallate (3655 mg; 19.8 mmol), K_2CO_3 (12,300 mg; 89 mmol), allyl bromide (14,300 mg; 118.2 mmol; 20 mL), and anhydrous acetonitrile (150 mL) were stirred at reflux for 1 h. Then volatile compounds were removed under reduced pressure. The pasty residue was suspended in toluene (200 mL), and insoluble salts were filtered and washed with additional portions of toluene (3×100 mL). Toluene was removed under reduced pressure; the crude product was passed through a short column of silica gel (eluent: diethyl ether). Product **1** was obtained as a yellowish honey, which solidified after several days in refrigerator (6034 mg; 100%) (lit.^[5] using K₂CO₃ in DMF: 83%).

¹H NMR (CDCl₃, δ ppm): 3.85 (s, 3H, CH₃), 4.59 (m, 6H, OCH₂), 5.12–5.46 (m, 6H, CH₂), 5.95–6.14 (m, 3H, =CH–), 7.26 (s, 2H, ArH); ¹³C NMR (CDCl₃, δ

ppm): 52.0 (s, 1C), 69.8 (s, 2C), 73.9 (s, 1C), 108.6 (s, 1C), 117.5 (s, 2C), 117.7 (s, 1C), 124.9 (s, 1C), 132.8 (s, 2C), 134.1 (s, 1C), 141.7 (s, 1C), 152.1 (s, 2C), 166.5 (s, 1C). Anal. calcd. for $C_{17}H_{20}O_5$: C, 67.09; H, 6.62. Found: C, 67.00; H, 6.71.

Addition of perfluorohexyl iodide to allyl derivative 1

Via sodium dithionite. Allyl derivative 1 (152 mg; 0.5 mmol) and perfluorohexyl iodide (1338 mg; 3 mmol) were dissolved in acetonitrile (12 mL), and water (4 mL) was added, followed by $Na_2S_2O_4$ (653 mg; 3 mmol) and $NaHCO_3$ (252 mg; 3 mmol). The reaction mixture was vigorously stirred at rt overnight. Then water (100 mL) was added and extracted with diethyl ether (5 × 50 mL). Combined organic fractions were then extracted with brine (50 mL) and dried over Na_2SO_4 . After filtration, volatile compounds were removed under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: toluene). Product 2 was obtained as a yellowish wax (686 mg; 84%).

Via AIBN. Allyl derivative 1 (152 mg; 0.5 mmol), perfluorohexyl iodide (1338 mg; 3 mmol), and AIBN [30 mg; three portions $(3 \times 10 \text{ mg})$ were added over an hour] were heated under argon atmosphere at 85°C for 6 h. Then excess of perfluorohexyl iodide was removed under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: toluene). Product **2** was obtained as a yellowish wax (523 mg; 64%).

Via Et_3B . Allyl derivative 1 (99 mg; 0.32 mmol), perfluorohexyl iodide (562 mg; 1.26 mmol), and hexane (0.5 mL) were mixed. Triethyl borane (1 M in hexanes; 0.2 mL; 0.2 mmol) were added, and the reaction mixture was stirred under an argon atmosphere at rt overnight. The reaction mixture was directly introduced into a column with silica gel and eluted with toluene. Product **2** was obtained as a white wax (417 mg; 79%).

¹H NMR (CDCl₃, δ ppm): 2.55–3.50 (m, 6H, CH₂R_F), 3.92 (s, 3H, CH₃), 4.20–4.44 (m, 6H, $3 \times \text{OCH}_2$), 4.45–4.63 (m, 3H, CHI), 7.30 (s, 2H, ArH); ¹³C NMR (CDCl₃, δ ppm): 12.1 (s, 3C), 37.8 (t, 3C, ³J_{CF}=21 Hz), 52.5 (s, 1C), 73.5 (s, 2C), 77.6 (s, 1C), 105.0–125.0 (m, 18C), 108.7 (s, 2C), 126.2 (s, 1C), 140.5 (s, 1C), 151.2 (s, 2C), 165.9 (s, 1C); ¹⁹F NMR (CDCl₃, δ ppm): -81.7 (m, 9 F), -115.1 (m, 6 F), -122.5 (m, 6 F), -123.5 (m, 6 F), -124.0 (m, 6 F), -126.6 (m, 6 F). Anal. calcd. for C₃₅H₂₀F₃₉I₃O₅: C, 25.60; H, 1.23. Found: C, 25.44; H, 1.31.

Reduction of C-I bond in 2 produced methyl gallate 3. Iodo derivative 2 (613 mg; 0.373 mmol) and sodium acetate (305 mg; 3.73 mmol) were dissolved in MeOH (20 mL) and ethyl acetate (20 mL). Pd/C [10%; 400 mg; previously suspended in MeOH (20 mL)] was added, and the black suspension was stirred under a hydrogen atmosphere overnight. The Reaction mixture was filtered: Pd/C was washed with methanol (40 mL) and ethyl acetate (40 mL). Solvents were removed under reduced pressure. Solid residue was suspended in petroleum ether (150 mL), and insoluble salts were filtered and washed with additional portions of petroleum ether (3×50 mL). Petroleum ether was removed under reduced pressure; product 3 was obtained as a white wax (453 mg; 96%).

¹H NMR (CDCl₃, δ ppm): 1.96–2.20 (m, 6H, $CH_2CH_2R_F$), 2.20–2.48 (m, 6H, CH_2R_F), 3.90 (s, 3H, CH₃), 4.06 (t, 2H, ³ $J_{HH} = 6.0$ Hz, O⁴CH₂), 4.11 (t, 4H, O³CH₂)

and O⁵CH₂, ${}^{3}J_{HH} = 5.9$ Hz), 7.29 (s, 2H, ArH); ${}^{13}C$ NMR (CDCl₃, δ ppm): 20.6 (s, 2C), 21.4 (s, 1C), 27.8 (t, 3C, ${}^{3}J_{CF} = 22$ Hz), 52.3 (s, 1C), 67.6 (s, 2C), 71.9 (s, 1C), 105.0–125.0 (m, 18C), 108.3 (s, 2C), 125.6 (s, 1C), 141.5 (s, 1C), 152.2 (s, 2C), 166.4 (s, 1C); ${}^{19}F$ NMR (CDCl₃, δ ppm): -81.6 (t, 6F, ${}^{3}J_{FF} = 10.4$ Hz), -81.7 (t, 3F, ${}^{3}J_{FF} = 9.8$ Hz), -115.0 (m, 6F), -122.5 (m, 6F), -123.5 (m, 6F), -124.1 (m, 6F), -126.8 (m, 6F). Anal. calcd. for C₃₅H₂₃F₃₉O₅: C, 33.25; H, 1.83. Found: C, 33.43; H, 1.98.

Preparation of gallate 3 via direct o-polyfluoroalkylation by triflate. A solution of methyl gallate (291 mg; 1.58 mmol) in DMF (10 mL) at room temperature was added dropwise to suspension of sodium hydride (60% susp. in mineral oil; 215 mg; 5.4 mmol; previously washed with anhydrous hexane) in anhydrous DMF (10 mL). The slurry was stirred for an additional 30 min. The solution of 3-(perfluor-ohexyl)propyl triflate (2900 mg; 5.68 mmol) in DMF (20 mL) was added at room temperature, and the reaction mixture was then stirred at 70°C for 4 h. After cooling down, water (200 mL) was added, and the mixture was extracted with diethyl ether (5 × 50 mL). The organic fraction was then washed with brine (2 × 30 mL) and dried over MgSO₄. After filtration, volatile compounds were removed under reduced pressure. Crude product was purified by column chromatography on silica gel (eluent: DCM.) Product **3** was obtained as a white wax (1817 mg; 91%).

Preparation of gallate 3 via direct o-polyfluoroalkylation by iodide

Via potassium carbonate. Methyl gallate (194 mg; 1.05 mmol), 3-(perfluorohexyl)propyl iodide (2310 mg; 4.73 mmol), and K₂CO₃ (968 mg; 7 mmol) were stirred in anhydrous DMF (7 mL) at 60°C overnight. Then diethyl ether (40 mL) was added and filtered. Salts were washed with the next portion of diethyl ether (3×30 mL). After evaporation to dryness, the crude product was purified by column chromatography on silica gel; the column was washed with petroleum ether (unreacted polyfluorinated iodide obtained in this fraction can be reused), then diethyl ether. Product **3** was obtained as a pale yellow wax (1328 mg; 100%).

Via sodium hydride. To a suspension of sodium hydride (60% susp. in mineral oil; 145 mg; 3.6 mmol; previously washed with anhydrous hexane) in anhydrous DMF (10 mL), a solution of methyl gallate (185 mg; 1 mmol) in DMF (10 mL) was added dropwise at room temperature. The slurry was stirred for an additional 30 min. A solution of 3-(perfluorohexyl)propyl iodide (2198 mg; 4.5 mmol) in DMF (15 mL) was added at room temperature, and the reaction mixture was then stirred at 70°C for 4 h. After cooling down, water (150 mL) was added, and the mixture was extracted with diethyl ether (5×50 mL). The organic fraction was then washed with brine (2×30 mL) and dried over MgSO₄. After filtration, volatile compounds were removed under reduced pressure. The crude product was purified by column chromatography (as mentioned previously). Product **3** was obtained as a white wax (1260 mg; 99%).

Reduction of gallate 3 to benzyl alcohol 4. Polyfluoroalkylated methyl gallate **3** (4632 mg; 3.66 mmol) was dissolved in anhydrous THF (60 mL) and cooled to 0° C, and LiAlH₄ (1 M in THF; 11 mmol; 11 mL) was added dropwise. The reaction mixture was strirred at rt overnight. Then excess of LiAlH₄ was destroyed by water

(20 mL), and volatile compounds were evaporated. The residue was acidified with diluted HCl (1:5 v/v; 100 mL) and extracted with diethyl ether (3×100 mL). The organic fraction was washed with water (50 mL) and brine (50 mL) and dried over Na₂SO₄. Diethyl ether was removed under reduced pressure. Crude product was passed through the short column of silica gel (eluent: diethyl ether). Product **4** was obtained as yellowish wax (4463 mg; 98%).

¹H NMR (CDCl₃, δ ppm): 1.93–2.16 (m, 6H, $CH_2CH_2R_F$), 2.18–2.46 (m, 6H, CH₂R_F), 3.97 (t, 2H, ³J_{HH} = 5.9 Hz, O⁴CH₂), 4.05 (t, 4H, O³CH₂ and O⁵CH₂, ³J_{HH} = 5.9 Hz), 4.60 (s, 2H, CH_2OH), 6.60 (s, 2H, ArH); ¹³C NMR (CDCl₃, δ ppm): 20.7 (s, 2C), 21.4 (s, 1C), 27.8 (t, 2C, ³J_{CF} = 22 Hz), 27.9 (t, 1C, ³J_{CF} = 22 Hz), 65.2 (s, 1C), 67.5 (s, 2C), 71.8 (s, 1C), 105.0–125.0 (m, 18C), 105.5 (s, 2C), 136.8 (s, 1C), 137.0 (s, 1C), 152.6 (s, 2C); ¹⁹F NMR (CDCl₃, δ ppm): -81.5 (t, 6F, ³J_{FF} = 10.4 Hz), -81.6 (t, 3 F, ³J_{FF} = 9.8 Hz), -115.0 (m, 6 F), -122.5 (m, 6 F), -123.5 (m, 6 F), -124.1 (m, 6 F), -126.8 (m, 6 F). Anal. calcd. for C₃₄H₂₃F₃₉O₄: C, 33.03; H, 1.87. Found: C, 33.17; H, 1.95.

Preparation of benzyl bromide 5. Polyfluoroalkylated benzyl alcohol 4 (1694 mg; 1.37 mmol) was dissolved in anhydrous diethyl ether (25 mL) and PBr₃ (864 mg; 3.19 mmol; 0.3 mL) in diethyl ether (2 mL) was added dropwise. The reaction mixture was strirred at rt overnight. Then water (50 mL) was added and extracted with diethyl ether $(3 \times 50 \text{ mL})$. The organic fraction was washed with water (50 mL) and brine (50 mL) and dried over Na₂SO₄. Diethyl ether was removed under reduced pressure; the crude product was passed through the short column of silica gel (eluent: diethyl ether). Product **5** was obtained as yellowish wax (1737 mg; 98%).

¹H NMR (CD₃COCD₃, δ ppm): 1.98–2.22 (m, 6H, *CH*₂CH₂R_F), 2.40–2.64 (m, 6H, CH₂R_F), 4.10 (t, 2H, ³*J*_{HH} = 5.8 Hz, O⁴CH₂), 4.20 (t, 4H, O³CH₂ and O⁵CH₂, ³*J*_{HH} = 5.9 Hz), 4.60 (s, 2H, CH₂Br), 6.86 (s, 2H, ArH); ¹³C NMR (CD₃COCD₃, δ ppm): 21.4 (s, 2C), 22.2 (s, 1C), 28.4 (t, 2C, ³*J*_{CF} = 21 Hz), 28.6 (t, 1C, ³*J*_{CF} = 21 Hz), 34.9 (s, 1C), 68.3 (s, 2C), 72.2 (s, 1C), 105.0–125.0 (m, 18C), 109.0 (s, 2C), 135.0 (s, 1C), 138.5 (s, 1C), 153.5 (s, 2C); ¹⁹F NMR (CD₃COCD₃, δ ppm): -80.8 (t, 9 F, ³*J*_{FF} = 10.4 Hz), -113.9 (m, 6 F), -121.4 (m, 6 F), -122.5 (m, 6 F), -123.0 (m, 6 F), -125.8 (m, 6 F). Anal. calcd. for C₃₄H₂₂BrF₃₉O₃: C, 31.43; H, 1.71. Found: C, 31.34; H, 1.73.

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