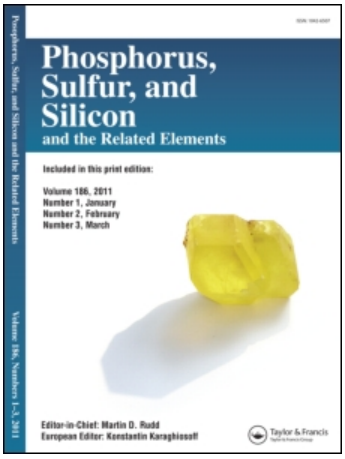

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Addition of Perfluoroalkylethane Thiols to Bis(Propargyl) Polyoxyethylene Ethers: Synthesis of Tetrakis-(*F*-Alkylethanethio) Polyoxyethylene Ethers

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Tetrakis-(F-alkylethanethio) polyoxyethylene ethers ***2*** *were prepared by free radical addition of perfluoroalkylethane thiols to bis(propargyl) polyoxyethylene ethers* ***1****. The reaction was carried out using AIBN as initiator. These new compounds may have important properties that would be advantageous for their application in industrial and biomedical areas.*

Keywords Perfluoroalkylethane thiol; polyoxyethylene dipropargyl ethers; tetrakis-(*F*-alkylethanethio) polyoxyethylene ethers

INTRODUCTION

In 1962, Brace reported that long chain alkanoic acids bearing perfluoroalkyl terminal segments had unique surface active and wettability properties.[1] Since then, many works describing the potentialities of perfluoroalkyl compounds have been carried out. Hybrid anionic surfactants containing fluorocarbon and hydrocarbon chains have been shown to form supermolecular assembies,[2,3] and some of them have potential biological applications.[4] Hybrid bolamphiphiles have been recently synthesized to study the "Flip-Flop" behavior of spin label in vesicles.[5] On the other hand, mercaptans show excellent properties as intermediates for the synthesis of a large range of elaborated molecules, such as surfactants,[6] polymers for fire retardants,[7] liquid crystals,[8] biocides,[9] blood substitutes,[10] and so on.

More recently, Brace et al.[11] have prepared new highly surface active *F*-alkyl amphiphiles by radical addition of perfluoroalkyl iodide to

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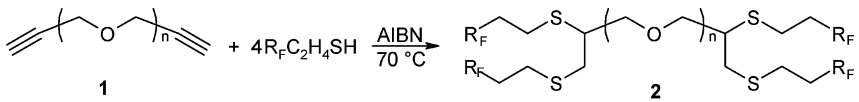
some succinic anhydride derivatives. The synthesis of bis(propargyl) polyoxyethylene ethers, compounds not well-known, easy to prepare, and can undergo four addition reactions on the both triple bonds, has been previously reported.[12,13] In a previous work, we have shown that the free radical addition of perfluoroalkylethane thiols to bis(allyl) polyoxyethylene ethers afforded bis(*F*-alkylethanethio) polyoxyethylene ethers.[14]

In view of these results, we present herein the synthesis of new tetrakis-(*F*-alkylethanethio) polyoxyethylene ether derivatives **2a–d** by radical addition of perfluoroalkylethane thiols to bis(propargyl) polyoxyethylene ethers **1**. All compounds were fully characterized by their spectroscopic ^{1}H, ^{13}C, ^{19}F NMR, and HRMS data. These new tetrakis-adducts containing two terminal *F*-alkylethanethio groups bridged by a polyoxyethylene chain may be useful as models for further studies.

RESULTS AND DISUSSION

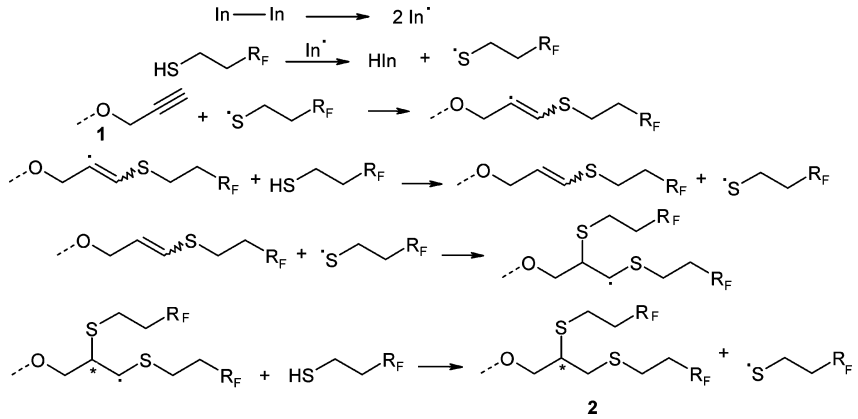
The radical addition of an excess of *F*-alkylethane thiols to bis(propargyl) polyoxyethylene ethers in the presence of AIBN as initiator led exclusively to the saturated addition products **2** (Scheme 1).



SCHEME 1

When the reaction was carried out with stoichiometric amounts, a complex mixture was obtained. The formation of this mixture may be due to competitive addition reactions with the alkenes formed (*Z* and *E* 1-thioenol isomers) and starting alkyne. This indicates that each of the reaction sites in the dipropargyl ether reacts independently as if they behave like two different molecules, as a result of their separation by the polyoxyethylene chain in the molecule.

The *E*/*Z* isomers were identified in the reaction mixture by NMR spectroscopy. For instance, H_a and H_b protons of the O-CH_2-CH_b=CH_a-S moiety constitute an AB system in which the B nucleus is coupled to the CH_2 protons. The NMR spectrum exhibits for the H_a proton in the ethylenic region two signals centred at 6.23 ppm and 6.08 ppm assigned to the *E* and *Z* isomers respectively, showing coupling



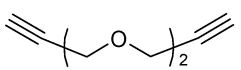
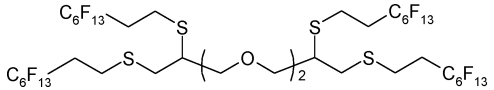
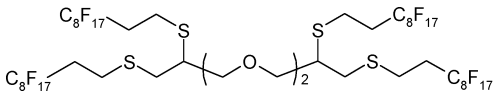
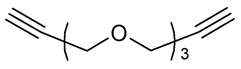
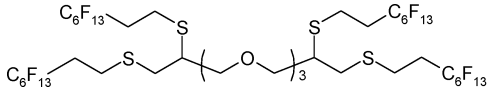
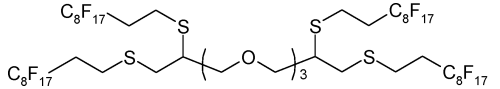
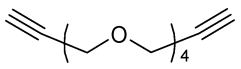
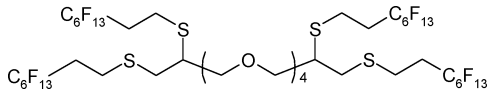
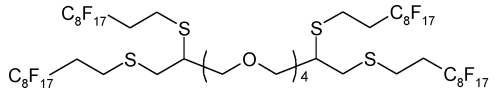
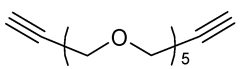
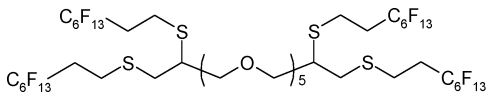
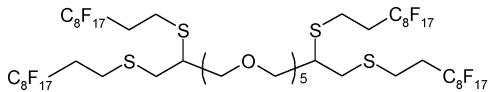
SCHEME 2

constants of $^3J_{AB}(E) = 15.06$ Hz and $^3J_{AB}(Z) = 9.57$ Hz and relative ratios of 60/40 (E/Z).

The mechanism of the reaction corresponds to the addition of a first radical to the terminal carbon of the triple bond on the either sides of the molecule to form the 1-thioenol, which is confirmed by the existence of ethylenic proton signals H_a and H_b of the O-CH_2-CH_b=CH_a-S-moiety in the Z and E isomers as mentioned above. A second radical addition corresponds to the attack of sulfur radical on the adjacent carbon atom to give the 1,2-disulfur products **2**. The ^{1}H NMR spectra of the latter showed a multiplet at ~3 ppm assigned to the proton directly linked to an asymmetric carbon. Whereas in the case of the ionic addition of thiol on thioenol ethers which gave the 1,1-disulfur product[15], the proton linked to the disulfur carbon is more shifted to low fields (~4.8 ppm). These reactions are in good agreement with those reported in the literature,[16] and show the formation of the most stable radical product that determines the orientation of these radical additions (Scheme 2).

It is worth noting that compounds **2** are obtained as two meso-threo diastereoisomers which are, however, indiscernable by NMR techniques. The separation of the two asymmetric centers of the molecule by the polyoxyethylene ether chain renders the interaction between these two sites negligible in a manner that they can be considered as two independent asymmetric centres and NMR spectroscopy is therefore expected to be unable to distinguish between the two diastereoisomers.[17]

TABLE I Tetrakis-(*F*-alkylethanethio) Polyoxyethylene Ethers 2

Dipropargylic ether **1**		Tetrakis-(*F*-alkylthio) ethers **2**		t (h)	Y (%)
	**1a**		**2a**	40	73
			**2a'**	42	75
	**1b**		**2b**	48	81
			**2b'**	54	82
	**1c**		**2c**	59	85
			**2c'**	60	86
	**1d**		**2d**	60	85
			**2d'**	62	87

CONCLUSION

Highly-fluorinated tetrakis compounds were synthesized by the radical addition of *F*-alkylethane thiol on the bis(propargyl) polyoxyethylene ethers. The reaction is regiospecific and lead to bis(1,2-disulfur) **2**.

The coexistence of four *F*-alkylated chains and four sulfur atoms linked to a modulable polyoxyethylene chain may be the origin of the interesting applications associating the well-known proprieties of highly fluorinated compounds[18,19] to those related to the existence of sulfur atoms.

EXPERIMENTAL

^{1}H, ^{13}C and ^{19}F NMR spectra were recorded on a Brüker AC 300 spectrometer at 300, 75, and 282 MHz, respectively. TMS is used as standard reference for ^{1}H and ^{13}C spectra and $CFCl_3$ for the ^{19}F ones. HRMS spectra in C. I. mode were carried out on a MAT 95 SBE spectrometer. The silica gel used is of the Merck 7734 type.

Preparation of Ethers 2—General Procedure

Into a 25 mL three-necked round-bottomed flask equipped with a refrigerant and a drying tube of $CaCl_2$, under nitrogen atmosphere, 5.0 mmol of ether **1**, 22.0 mmol of *F*-alkylethane thiol and 0.3 g of AIBN was introduced. The mixture was heated at 70°C. The crude product was purified on silica gel column using ethanol, then chloroform as eluents and isolated as viscous oils.

2a

^{1}H NMR [$CDCl_3$, δ (ppm/TMS)]: 2.36–2.53 (m, 8H, 4CH_2-CF_2), 2.78–2.90 (m, 12H, 6CH_2-S), 3.05 (m, 2H, 2*CH*-S), 3.65 (m, 4H, 2CH_2-CH_2-O), 3.69 (m, 4H, 2CH-CH_2-O); ^{13}C NMR: 69.03 (s, 2C, $\underline{C}H_2$-O), 70.33 (s, 2C, O-$\underline{C}H_2$-CH), 45.99 (s, 2C, $\underline{C}$H-S), 34.15 (s, 2C, CH-$\underline{C}H_2$-S), 22.13 (s, 2C, $\underline{C}H_2$-S), 31.63–32.66 (m, 4C, $\underline{C}H_2CF_2$), 21.95 (s, 2C, $\underline{C}H_2$-S); ^{19}F NMR [$CDCl_3$, δ (ppm/$CFCl_3$)]: −115.61 (m, 8F, 4$CF_{2\alpha}$), −123.12 (m, 8F, 4$CF_{2\beta}$), −124.63 (m, 8F, 4$CF_{2\gamma}$), −124.12 (m, 8F, 4$CF_{2\delta}$), −127.47 (m, 8F, 4$CF_{2\omega}$), −82.16 (m, 12F, 4CF_3, $^3J_{FF} = 9.75$); HRSM: Found: 1658.02973, Calcd.: 1658.02984, Δ (umm) = −0.11.

2a′

^{1}H NMR [$CDCl_3$, δ (ppm/TMS)]: 2.36–2.53 (m, 8H, 4CH_2=CF_2), 2.76–2.88 (m, 12H, 6CH_2-S), 3.01 (m, 2H, 2*CH*-S), 3.61–3.67 (m, 4H, 2CH_2-CH_2-O), 3.76 (m, 4H, 2CH-CH_2-O); ^{13}C NMR: 70.56–73.53 (s, 2C,

$\underline{C}H_2$-O), 70.26 (s, 2C, O-$\underline{C}H_2$-CH), 46.21 (s, 2C, $\underline{C}$H-S), 34.52 (s, 2C, CH-$\underline{C}H_2$-S), 22.21 (s, 2C, $\underline{C}H_2$-S), 31.75–32.65 (m, 4C, $\underline{C}H_2CF_2$), 22.13 (s, 2C, $\underline{C}H_2$-S); ^{19}F NMR [$CDCl_3$, δ (ppm/$CFCl_3$)]: −115.53 (m, 8F, $4CF_{2\alpha}$), −122.93 (m, 8F, $4CF_{2\beta}$), −124.51 (m, 8F, $4CF_{2\gamma}$), −123.99 (m, 8F, $4CF_{2\delta}$), −123.05 (m, 16F, $8CF_{2\varepsilon}$), −127.39 (m, 8F, $4CF_{2\omega}$), −82.14 (m, 12F, $4CF_3$, $^3J_{FF} = 9.74$); HRSM: Found: 2126.00452, Calcd.: 2126.00429, Δ (umm) = 0.23.

2b

^{1}H NMR [$CDCl_3$, δ (ppm/TMS)]: 2.35–2.52 (m, 8H, $4CH_2$-CF_2), 2.78–2.89 (m, 12H, $6CH_2$-S), 2.99 (m, 2H, 2*CH*-S), 3.60–3.65 (m, 8H, $4CH_2$-CH_2-O), 3.71 (m, 4H, 2CH-CH_2-O); ^{13}C NMR: 70.44–73.04 (s, 4C, $\underline{C}H_2$-O), 70.19 (s, 2C, O-$\underline{C}H_2$-CH), 46.13 (s, 2C, $\underline{C}$H-S), 34.68 (s, 2C, CH-$\underline{C}H_2$-S), 22.29 (s, 2C, $\underline{C}H_2$-S), 31.70–32.50 (m, 4C, $\underline{C}H_2CF_2$), 22.23 (s, 2C, $\underline{C}H_2$-S); ^{19}F NMR [$CDCl_3$, δ (ppm/$CFCl_3$)]: −115.57 (m, 8F, $4CF_{2\alpha}$), −123.18 (m, 8F, $4CF_{2\beta}$), −124.46 (m, 8F, $4CF_{2\gamma}$), −124.16 (m, 8F, $4CF_{2\delta}$), −127.53 (m, 8F, $4CF_{2\omega}$), −82.24 (m, 12F, $4CF_3$, $^3J_{FF} = 9.79$); HRSM: Found: 1702.05610, Calcd.: 1702.05605, Δ (umm) = 0.05.

2b′

^{1}H NMR [$CDCl_3$, δ (ppm/TMS)]: 2.25–2.45 (m, 8H, $4CH_2$-CF_2), 2.70–2.79 (m, 12H, $6CH_2$-S), 2.89 (m, 2H, 2*CH*-S), 3.54–3.60 (m, 8H, $4CH_2$-CH_2-O), 3.63 (m, 4H, 2CH-CH_2-O); ^{13}C NMR: 70.30–72.98 (s, 4C, $\underline{C}H_2$-O), 70.08 (s, 2C, O-$\underline{C}H_2$-CH), 45.96 (s, 2C, $\underline{C}$H-S), 34.48 (s, 2C, CH-$\underline{C}H_2$-S), 22.02 (s, 2C, $\underline{C}H_2$-S), 31.52–32.41 (m, 4C, $\underline{C}H_2CF_2$), 22.16 (s, 2C, $\underline{C}H_2$-S); ^{19}F NMR [$CDCl_3$, δ (ppm/$CFCl_3$)] : −115.54 (m, 8F, $4CF_{2\alpha}$), −123.02 (m, 8F, $4CF_{2\beta}$), −124.49 (m, 8F, $4CF_{2\gamma}$), −124.09 (m, 8F, $4CF_{2\delta}$), −123.07 (m, 16F, $8CF_{2\varepsilon}$), −127.36 (m, 8F, $4CF_{2\omega}$), −82.15 (m, 12F, $4CF_3$, $^3J_{FF} = 9.66$); HRSM: Found: 2170.03036, Calcd.: 2170.03051, Δ (umm) = −0.15.

2c

^{1}H NMR [$CDCl_3$, δ (ppm/TMS)]: 2.35–2.51 (m, 8H, $4CH_2$-CF_2), 2.74–2.91 (m, 12H, $6CH_2$-S), 3.02 (m, 2H, 2*CH*-S), 3.60–3.67 (m, 12H, $6CH_2$-CH_2-O), 3.78 (m, 4H, 2CH-CH_2-O); ^{13}C NMR: 70.34–72.90 (s, 6C, $\underline{C}H_2$-O), 69.97 (s, 2C, O-$\underline{C}H_2$-CH), 46.02 (s, 2C, $\underline{C}$H-S), 34.39 (s, 2C, CH-$\underline{C}H_2$-S), 21.99 (s, 2C, $\underline{C}H_2$-S), 31.66–32.43 (m, 4C, $\underline{C}H_2CF_2$), 22.14 (s, 2C, CH_2-S); ^{19}F NMR [$CDCl_3$, δ (ppm/$CFCl_3$)]: −115,51 (m, 8F, $4CF_{2\alpha}$), −123.04 (m, 8F, $4CF_{2\beta}$), −124.53 (m, 8F, $4CF_{2\gamma}$), −124.11 (m, 8F, $4CF_{2\delta}$), −127.31 (m, 8F, $4CF_{2\omega}$), −82.19 (m, 12F, $4CF_3$, $^3J_{FF} = 10.23$); HRSM: Found: 1746.08218, Calcd.: 1746.08227, Δ (umm) = 0.05.

2c′

^{1}H NMR [$CDCl_3$, δ (ppm/TMS)]: 2.35–2.52 (m, 8H, 4CH_2-CF_2), 2.77–2.83 (m, 12H, 6CH_2-S), 3.00 (m, 2H, 2CH-S), 3.62–3.72 (m, 12H, 6CH_2-CH_2-O), 3.79 (m, 4H, 2CH-CH_2-O), ^{13}C NMR : 70.02–72.75 (s, 6C, $\underline{C}H_2$-O), 69.89 (s, 2C, O-$\underline{C}H_2$-CH), 45.91 (s, 2C, $\underline{C}$H-S), 34.12 (s, 2C, CH-$\underline{C}H_2$-S), 21.95 (s, 2C, $\underline{C}H_2$-S), 31.34–32.16 (m, 4C, $\underline{C}H_2CF_2$), 22.02 (s, 2C, $\underline{C}H_2$-S); ^{19}F NMR [$CDCl_3$, δ (ppm/$CFCl_3$)] : -115.48 (m, 8F, 4$CF_{2\alpha}$), -122.95 (m, 8F, 4$CF_{2\beta}$), -124.55 (m, 8F, 4x$CF_{2\gamma}$), -123.99 (m, 8F, 4$CF_{2\delta}$), -123.17 (m, 16F, 8$CF_{2\varepsilon}$), -127.45 (m, 8F, 4$CF_{2\omega}$), -82.27 (m, 12F, 4CF_3, $^3J_{FF} = 9.59$); HRSM: Found: 2214.05685, Calcd.: 2214.05672, Δ (umm) = 0.13.

2d

^{1}H NMR [$CDCl_3$, δ (ppm/TMS)]: 2.35–2.53 (m, 8H, 4CH_2-CF_2), 2.77–2.90 (m, 12H, 6CH_2-S), 3.03 (m, 2H, 2CH-S), 3.61–3.68 (m, 16H, 8CH_2-CH_2-O), 3.80 (m, 4H, 2CH-CH_2-O), ^{13}C NMR: 70.05–72.86 (s, 8C, $\underline{C}H_2$-O), 69.85 (s, 2C, O-$\underline{C}H_2$-CH), 45.88 (s, 2C, $\underline{C}$H-S), 34.09 (s, 2C, CH-$\underline{C}H_2$-S), 21.98 (s, 2C, $\underline{C}H_2$-S), 31.45–32.24 (m, 4C, $\underline{C}H_2CF_2$), 21.96 (m, 2C, $\underline{C}H_2$-S); ^{19}F NMR [$CDCl_3$, δ (ppm/$CFCl_3$)] : −115.47 (m, 8F, 4$CF_{2\alpha}$), −123.06 (m, 8F, 4$CF_{2\beta}$), -124.56 (m, 8F, 4$CF_{2\gamma}$), −124.06 (m, 8F, 4$CF_{2\delta}$), −127.36 (m, 8F, 4$CF_{2\omega}$), −82.09 (m, 12F, 4CF_3, $^3J_{FF} = 9.71$); HRSM: Found 1790.10865, Calcd.: 1790.10848, Δ (umm) = 0.17.

2d′

^{1}H NMR [$CDCl_3$, δ (ppm/TMS)]: 2.35–2.52 (m, 8H, 4CH_2-CF_2), 2.77–2.83 (m, 12H, 6CH_2-S), 3.03 (m, 2H, 2CH-S), 3.61–3.78 (m, 16H, 8CH_2-CH_2-O), 3.80 (m, 4H, 2CH-CH_2-O), ^{13}C NMR: 69.93–73.06 (s, 8C, $\underline{C}H_2$-O), 69.99 (s, 2C, O-$\underline{C}H_2$-CH), 45.94 (s, 2C,$\underline{C}$H-S), 34.52 (s, 2C, CH-$\underline{C}H_2$-S), 22.05 (s, 2C, $\underline{C}H_2$-S), 31.44–32.34 (m, 4C, $\underline{C}H_2CF_2$), 22.01 (s, 2C, $\underline{C}H_2$-S); ^{19}F NMR [$CDCl_3$, δ (ppm/$CFCl_3$)] : −115.51 (m, 8F, 4$CF_{2\alpha}$), −123.01 (m, 8F, 4$CF_{2\beta}$), -124.48 (m, 8F, 4$CF_{2\gamma}$), -123.98 (m, 8F, 4$CF_{2\delta}$), -123.01 (m, 16F, 8$CF_{2\varepsilon}$), −127.27 (m, 8F, 4$CF_{2\omega}$), −82.10 (m, 12F, 4CF_3, $^3J_{FF} = 9.73$); HRSM: Found: 2258.08313, Calcd.: 2258.08294, Δ (umm) = 0.19.

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