

Electron Transfer Initiated Free Radical Additions of Perfluoroalkyl Iodides and Diiodides to Alkenes

Jürgen O. Metzger^{*a}, Ralf Mahler^a, and Andreas Schmidt^b

Fachbereich Chemie der Universität Oldenburg^a,
Carl-von-Ossietzky-Straße 9–11, D-26111 Oldenburg

Technische Hochschule Darmstadt, Institut für Anorganische Chemie^b,
Petersenstraße 18, D-64287 Darmstadt

Received September 1, 1995

Key Words: Perfluoroalkylated carboxylic acids and alcohols / Perfluoroalkyl radicals / Radical addition

Perfluoroalkyl iodides were added in very good yields to 1,2-dialkyl substituted double bonds of e.g. petroselinic acid initiated by lead/copper(II) acetate in methanol and by copper without solvent, respectively. Using the new initiator system

lead/copper(II) acetate, we also obtained good yields in the additions of perfluoroalkyl iodides to allyl alcohol and ω -unsaturated alcohols and of perfluoroalkyl 1, ω -diiodides to methyl 10-undecenoate.

Recently, we showed that linear and branched perfluoroalkylated carboxylic acids are easily available by tin(II) chloride/silver(I) acetate-initiated free radical additions of perfluoroalkyl iodides to unsaturated carboxylic acids followed by reduction of the respective addition products with tributylstannane^[1]. Quantitative yields were obtained with ω -unsaturated carboxylic acids such as methyl 10-undecenoate. However, only moderate yields of 25–50% were obtained in the addition of perfluoroalkyl iodides to 1,2-dialkyl substituted double bonds e.g. of oleic acid and petroselinic acid, respectively.

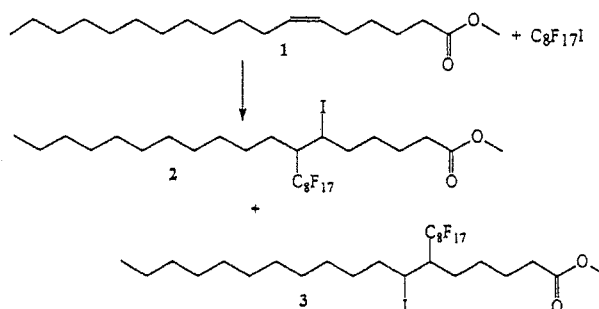
We report now that lead powder together with a catalytic amount of $\text{Cu}(\text{OAc})_2$ in methanol at room temperature is an excellent reagent for the electron transfer initiated additions of perfluoroalkyl iodides to alkenes affording very good yields with alkenes containing 1,2-dialkyl substituted double bonds e.g. methyl petroselinate and with unsaturated alcohols. The addition of perfluoroalkyl 1, ω -diiodides to methyl 10-undecenoate was performed as well.

Results and Discussion

Treatment of methyl petroselinate (1) with perfluorooctyl iodide in the presence of lead powder and a catalytic amount of copper(II) acetate (molar ratio $1/\text{C}_8\text{F}_{17}\text{I}/\text{Pb}/\text{Cu}(\text{OAc})_2 = 1:1.57:1.21:0.01$) in methanol at room temperature for 4 days gave an equimolar mixture of the regioisomeric addition products 2 and 3 in 79% yield (Scheme 1).

Improved yields of the addition products 2 and 3 compared to literature procedures^[1] could also be obtained when the reaction was initiated by metallic copper^[2,3]. The procedure is very simple: the alkene 1, perfluorooctyl iodide, and commercial copper powder were mixed without solvent and without any pretreatment and heated at 130 °C for 8 h. The yield of the addition products 2, 3 was 85%.

Scheme 1

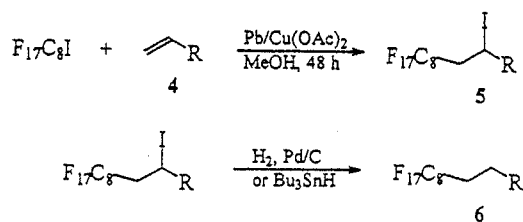


Perfluorooctyl iodide was added smoothly to unsaturated alcohols 4a–c. The reaction was initiated by $\text{Pb}/\text{Cu}(\text{OAc})_2$. The reaction time was 48 h at room temperature and methanol was used as solvent. The addition products 5 were obtained in good yields (Table 1). The comparable addition of perfluorooctyl iodide to 4a with copper as initiator gave lower yields of 5a and unseparable byproducts. The reduction of the addition products 5b, c was carried out with H_2 and Pd/C. However, the addition product 5a was reduced to alcohol 6a with Bu_3SnH because reduction with H_2 and Pd/C would lead to the formation of unseparable byproducts.

There are some examples in the literature of the addition of perfluoroalkyl 1, ω -diiodides to alkenes^[4]. We could show that perfluoroalkyl diiodides can be added to methyl 10-undecenoate (7) to give the mono- and the diaddition product, respectively, using the initiator system $\text{Pb}/\text{Cu}(\text{OAc})_2$, depending on the ratio of the reactants.

The bisaddition product 8 was obtained when a threefold excess of methyl 10-undecenoate (7) was used. The isolated yield of product 8 was 65%. Catalytic hydrogenation gave the reduction product 9 in 95% yield. Use of an excess of the perfluoroalkyl diiodides gave the mono addition prod-

Scheme 2



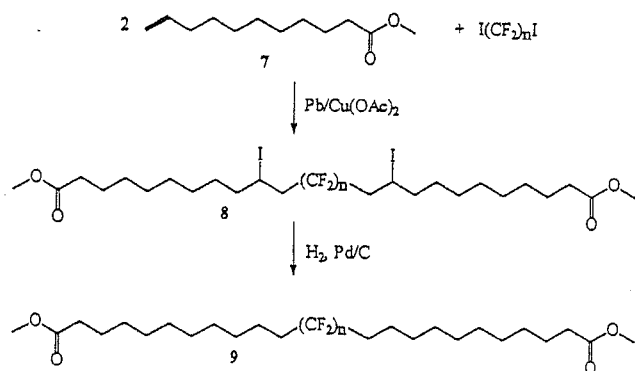
4-6	a	b	c
R	CH ₂ OH	(CH ₂) ₂ OH	(CH ₂) ₄ OH

Table 1. Free radical additions of perfluorooctyl iodide to unsaturated alcohols 4 initiated by Pb/Cu(OAc)₂ and reduction of the addition products 5 to give perfluoroalkylated alcohols 6

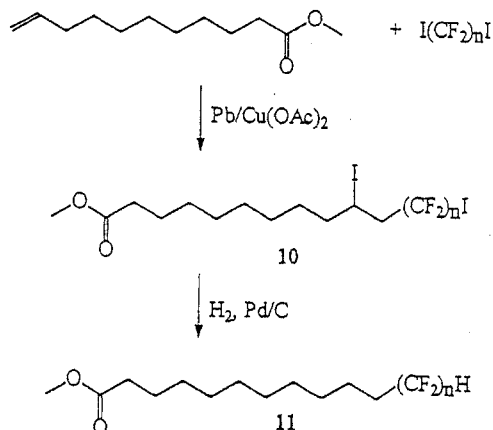
Alcohol	Product	Yield [%] ^[a]	Product	Yield [%] ^[b]
4a	5a	67	6a	80 ^[c]
4b	5b	85	6b	75 ^[d]
4c	5c	90	6c	76 ^[d]

^[a] Isolated yield of product 5 based on 4. - ^[b] Isolated yield of 6 based on 5. - ^[c] Reduction with Bu₃SnH. - ^[d] Catalytic hydrogenation with Pd/C.

Scheme 3



Scheme 4



uct 10 in 56% yield. Catalytic hydrogenation of 10 afforded the reduction product 11 in 60% yield.

The free radical addition reaction is initiated by electron transfer from the copper-lead couple and copper, respectively, as discussed in ref.^[1-3].

This work was supported by the *Bundesministerium für Ernährung, Landwirtschaft und Forsten* (Förderkennzeichen 93NR066-F). We also thank the *Henkel KGaA*, the *Hoechst AG*, *Schering*, *Atochem*, and *Unichema* for providing chemicals and Dr. *K. von Werner*, *Hoechst AG*, for providing perfluoroalkyl 1,ω-diiodides.

Experimental

Melting points (uncorrected): Leitz Laborlux 12. - *n_D*: Zeiss-Abbé-Refraktometer. - Elemental analyses: Carlo Erba, Modell 1104. - ¹H and ¹³C NMR: Bruker AM 300 (300 MHz); solvent CDCl₃; internal standard tetramethylsilane. - MS: Finnigan MAT 212 and Finnigan MAT 95 (for GC-MS coupled with Varian 3700). - Analytical GC: Carlo Erba GC 6000 Vega Series 2 with FID detector and Spectra Physics Data Jet Integrator, fused silica capillary column DB 1 30 m. - Medium-pressure liquid chromatography (MPLC): Merck silica gel 60, 0.04–0.063 mm. - Thin-layer chromatography (TLC): Merck plates, silica gel 60 F; detection by treatment with a solution of 15% H₂SO₄ followed by heating at 200°C. - Solvents were purified and dried in the usual way. - Petroselinic acid (81.3% petroselinic acid, 3.3% palmitic acid, 0.4% stearic acid, 13.5% C_{18:2}), Henkel KGaA, methyl 10-undecenoate, Elf Atochem, perfluorooctyl iodide, Hoechst AG, were used without further purification. Perfluoroalkyl 1,ω-diiodides were used as a mixture of C₄F₈I₂ = 53.8%, C₆F₁₂I₂ = 13.9%, C₈F₁₆I₂ = 15.0%, C₁₀F₂₀I₂ = 6.9% and higher oligomers. Lead powder (Merck, >140 mesh), copper powder (Merck, >230 mesh).

Methyl 6(7)-Iodo-7(6)-perfluorooctyldecanoate (2, 3): A mixture of 3.50 g (17 mmol) of lead powder and 0.2 g (0.2 mmol) of copper(II) acetate in 100 ml of dry methanol was stirred under nitrogen for 30 min. 5.00 g (14 mmol) of methyl petroselinate and 12.04 g (22 mmol) of perfluorooctyl iodide were slowly added. The reaction mixture was stirred for 4 d under nitrogen at room temp. The solvent was removed, the residue dissolved in diethyl ether and the solution filtered. Concentration of the filtrate yielded 11.71 g of crude product. After flash chromatography (petroleum ether/diethyl ether, 9:1) 9.08 g (79%) of 2, 3 was obtained. The ¹H- and ¹³C-NMR data correspond in all respects with those of the compound described in ref.^[1].

A mixture of 0.70 g (17 mmol) of copper powder, 2.50 g (7 mmol) of methyl petroselinate, and 6.02 g (11 mmol) of perfluorooctyl iodide was stirred for 8 h under nitrogen at 130°C. The reaction mixture was subsequently dissolved in diethyl ether and the solution filtered. Concentration yielded 8.51 g of crude product. After flash chromatography (petroleum ether/diethyl ether, 9:1) 5.38 g (85%) of a mixture of 2 and 3 (~1:1) was isolated.

rac-4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11-Heptadecafluoro-2-iodoundecanol (5a): A mixture of 26.75 g (129 mmol) of lead powder and 3.44 g (17 mmol) of Cu(OAc)₂ in 100 ml of dry methanol was stirred under nitrogen for 30 min. 70.5 g (129 mmol) of perfluorooctyl iodide and 5.87 ml (86 mmol) of allyl alcohol (4a) were slowly added. The reaction mixture was stirred for 48 h at room temp. The solvent was removed, the residue was dissolved in diethyl ether and the solution filtered. The solvent was removed and crystallization of the residue from methanol/water gave 35.0 g (67%) of 5a. - M.p.: 54°C. - ¹H NMR (CDCl₃): δ = 4.45 (m, 1H, 2-

H), 3.88–3.77 (m, 2H, 1-H), 3.18–2.71 (m, 2H, 3-H), 2.10 (t, $J = 6.4$ Hz, 1H, OH). – ^{13}C NMR (CDCl_3): $\delta = 61.5$ (C-1), 37.5 (t, C-3), 21.7 (C-2). – CI MS (isobutane), m/z : 477 [$\text{MH}^+ - \text{HI}$]. – MS (70 eV), m/z : 604 [M^+] (0.1), 477 (12), 457 (6), 131 (11), 109 (17), 91 (10), 77 (13), 69 (56), 43 (100). – $\text{C}_{11}\text{H}_{17}\text{F}_{17}\text{IO}$ (604.0): calcd. C 21.87, H 1.00; found C 22.21, H 0.96.

4,4,5,5,6,6,7,7,8,8,9,9,10,10,11,11,11,11-Heptadecafluoroundecanol (6a): 25.0 g (41 mmol) of 5a and 14.55 g (50 mmol) of Bu_3SnH were dissolved in 100 ml toluene. The solution was heated at reflux for 8 h. The solvent was removed and the residue purified by crystallization from petroleum ether to afford 15.84 g (80%) of 6a. – M.p. 42°C (46.5°C^[5]). – ^1H NMR (CDCl_3): $\delta = 3.75$ (td, $J = 6.4$, 5.1 Hz, 2H, 1-H), 2.01–1.84 (m, 2H, 2-H), 2.22 (tt, $J = 18.5$, 8.5 Hz, 2H, 3-H), 1.42 (t, $J = 5.1$ Hz, 1H, OH). – ^{13}C NMR (CDCl_3): $\delta = 61.5$ (C-1), 27.6 (t, C-3), 23.4 (C-2). – CI MS (isobutane), m/z : 461 [$\text{MH}^+ - \text{H}_2\text{O}$]. – MS (70 eV), m/z (%): 478 [M^+] (3), 441 (8), 395 (12), 169 (16), 109 (50), 91 (77), 69 (100), 60 (44), 48 (72). – $\text{C}_{11}\text{H}_7\text{OF}_{17}$ (478.1): calcd. C 27.63, H 1.48; found C 27.45, H 1.43.

rac-5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,12-Heptadecafluoro-3-iodo-dodecanol (5b): A mixture of 14.50 g (75 mmol) of lead powder and 1.00 g (5 mmol) of $\text{Cu}(\text{OAc})_2$ in 100 ml of methanol was stirred under nitrogen for 30 min. Then 41.04 g (75 mmol) of perfluorooctyl iodide and 4.15 g (58 mmol) of 3-buten-1-ol (4b) were slowly added. The reaction mixture was stirred for 48 h at room temp. The solvent was removed, the residue dissolved in diethyl ether and the solution was filtered. The solvent was removed and crystallization of the residue from methanol/water gave 30.32 g (85%) of 5b. – M.p.: 83°C. – ^1H NMR (CDCl_3): $\delta = 4.57$ –4.50 (m, 1H, 3-H), 3.89 (m, 1H, 1-H), 3.79 (m, 1H, 1-H), 3.05–2.80 (m, 2H, 4-H), 2.08–1.99 (m, 2H, 2-H), 1.6–1.8 (m, 1H, OH). – ^{13}C NMR (CDCl_3): $\delta = 62.5$ (C-1), 42.3 (C-3), 42.0 (t, C-4), 16.6 (C-2). – CI MS (isobutane), m/z : 601 [$\text{MH}^+ - \text{H}_2\text{O}$]. – MS (70 eV), m/z (%): 618 [M^+] (0.2), 491 (12), 473 (8), 441 (10), 131 (14), 119 (12), 91 (18), 69 (64), 58 (66), 42 (100). – $\text{C}_{12}\text{H}_8\text{F}_{17}\text{IO}$ (618.1): calcd. C 23.32, H 1.30; found C 23.56, H 1.28.

5,5,6,6,7,7,8,8,9,9,10,10,11,11,12,12,12-Heptadecafluorododecanol (6b): 30.00 g (49 mmol) of 5b was dissolved in 200 ml of methanol. To the solution were added 4.20 g (42 mmol) of sodium hydrogen carbonate and 1.00 g of Pd/C (10%) and the mixture was hydrogenated for 24 h at room temp. at a hydrogen pressure of 2.8 bar. After filtration the solvent was evaporated from the filtrate and the residue was subjected to kugelrohr distillation at 50–150°C (0.1 mbar) affording 17.93 g (75%) of 6b. – M.p.: 40°C. – ^1H NMR (CDCl_3): $\delta = 3.70$ (t, $J = 5.7$ Hz, 2H, 1-H), 2.11 (tt, $J = 18.5$, 8.3 Hz, 2H, 4-H), 1.76–1.58 (m, 4H, 2-, 3-H). – ^{13}C NMR (CDCl_3): $\delta = 62.2$ (C-1), 31.9 (C-3), 30.7 (t, C-3), 16.8 (C-2). – CI MS (isobutane), m/z : 475 [$\text{MH}^+ - \text{H}_2\text{O}$]. – MS (70 eV), m/z (%): 454 (16), 135 (20), 131 (20), 119 (18), 77 (50), 69 (92), 56 (100). – $\text{C}_{12}\text{H}_9\text{OF}_{17}$ (492.2): calcd. C 29.28, H 1.84; found C 29.25, H 1.78.

rac-7,7,8,8,9,9,10,10,11,11,12,12,13,13,14,14,14-Heptadecafluoro-5-iodotetradecanol (5c): A mixture of 11.40 g (55 mmol) of lead powder and 1.00 g (5 mmol) of $\text{Cu}(\text{OAc})_2$ in 100 ml of methanol was stirred under nitrogen for 30 min. then 29.88 g (54 mmol) of perfluorooctyl iodide and 4.22 g (42 mmol) of 5-hexen-1-ol (4c) were slowly added. The reaction mixture was stirred for 48 h at room temp. and then filtered. The solvent was removed in a rotary evaporator, the residue was crystallized from methanol/water and 24.4 g (90%) of 5c was isolated. – M.p.: 24°C. – ^1H NMR (CDCl_3): $\delta = 4.34$ (tt, 1H, 5-H), 3.67 (t, $J = 6.3$ Hz, 2H, 1-H), 2.99–2.72 (m, 2H, 6-H), 1.90–1.79 (m, 2H, 4-H), 1.70–1.50 (m, 4H, 2-, 3-H). – ^{13}C NMR (CDCl_3): $\delta = 62.4$ (C-1), 41.7 (t, C-6),

40.0 (C-5), 31.5 (C-4), 26.0 (C-2), 20.4 (C-3). – CI MS (isobutane), m/z : 629 [$\text{MH}^+ - \text{H}_2\text{O}$]. – MS (70 eV), m/z (%): 519 [$\text{M}^+ - \text{I}$] (1.0), 501 (24), 481 (8), 473 (4), 131 (7), 119 (6), 91 (7), 81 (26), 69 (25), 43 (100). – $\text{C}_{14}\text{H}_{12}\text{F}_{17}\text{IO}$ (646.1): calcd. C 26.02, H 1.87; found C 26.25, H 1.84.

7,7,8,8,9,9,10,10,11,11,12,12,13,13,14,14,14-Heptadecafluorotetradecanol (4c): To a solution of 24.00 g (37 mmol) of 5c in 200 ml methanol were added 4.20 g (42 mmol) of sodium hydrogen carbonate and 1.00 g of Pd/C (10%) and the mixture was hydrogenated for 24 h at room temp. at a hydrogen pressure of 2.8 bar. After filtration the solvent was evaporated and the residue was subjected to kugelrohr distillation at 50–150°C (0.1 mbar) furnishing 14.73 g (76%) of 6c. – M.p.: 50°C. – ^1H NMR (CDCl_3): $\delta = 3.62$ (t, $J = 6.5$ Hz, 2H, 1-H), 2.05 (tt, $J = 18.5$, 8.4 Hz, 2H, 6-H), 1.72–1.31 (m, 8H, 2-, 3-, 4-, 5-H). – ^{13}C NMR (CDCl_3): $\delta = 62.7$ (C-1), 32.4 (C-2), 30.5 (t, C-6), 28.9 (C-4), 25.4 (C-3), 20.1 (C-5). – CI MS (isobutane), m/z : 503 [$\text{MH}^+ - \text{H}_2\text{O}$]. – MS (70 eV), m/z (%): 502 [$\text{M}^+ - \text{H}_2\text{O}$] (0.8), 474 (0.8), 169 (0.9), 131 (4), 119 (4), 75 (12), 69 (15), 55 (100), 43 (56). – $\text{C}_{14}\text{H}_{13}\text{OF}_{17}$ (520.2): calcd. C 32.32, H 2.56; found C 32.63, H 2.48.

Addition of Perfluoroalkyl 1, ω -Diiodides to Methyl 10-Undecenoate: A mixture of 5.20 g (25 mmol) of lead powder and 0.20 g (3 mmol) of $\text{Cu}(\text{OAc})_2$ in 100 ml methanol was stirred under nitrogen for 30 min. Then 5.00 g (10 mmol) of perfluoroalkyl 1, ω -diiodides and 10.00 g (34 mmol) of methyl 10-undecenoate were slowly added and the mixture was stirred for 48 h at room temp. It was subsequently concentrated, the residue was dissolved in diethyl ether and the solution was filtered. Flash chromatography (petroleum ether/diethyl ether, 9:1) of the residue obtained after evaporation of the solvent from the filtrate yielded 4.1 g (65%) of 8 (as a mixture of homologues)^[6]. – n_D^{20} : 1.4382. – ^1H NMR (CDCl_3): $\delta = 4.40$ –4.30 (m, 2H, 10-, 17-H), 3.67 (s, 6H, 2 \times OCH_3), 3.0–2.6 (m, 4H, H-11, -16), 2.30 (t, $J = 7.4$ Hz, 4H, 2-, 25-H), 1.98–1.20 (m, 28H, 3–9-, 18–24-H). – ^{13}C NMR (CDCl_3): $\delta = 175.1$ (C-1, -26), 52.2 (2 \times OCH_3), 42.6 (m, C-11, -16), 41.1 (C-10, -17), 34.9 (C-2, -25), 31.8–29.2 (10 C, C-4–8, -19–23), 25.7 (C-3, -24), 22.4–22.0 (C-9, -18). – CI MS (isobutane), m/z (%): 852 (16) [MH^+], 725 (100) [$\text{MH}^+ - \text{I}$]. – MS (70 eV), m/z (%): 723 [$\text{M}^+ - \text{I}$] (4), 691 (6), 659 (12), 566 (12), 533 (30), 98 (56), 74 (85), 69 (69), 56 (100)^[6].

Hydrogenation of the Addition Product 8: To a solution of 1.00 g (1.5 mmol) of 8 in 150 ml of methanol were added 0.30 g of sodium hydrogen carbonate and 0.20 g of Pd/C (10%). Hydrogenation of the mixture at a hydrogen pressure of 2.8 bar at room temp. for 24 h gave after filtration and concentration 0.60 g (95%) of 9 as a mixture of homologues^[6]. – M.p.: 25°C. – ^1H NMR (CDCl_3): $\delta = 3.67$ (s, 6H, 2 \times OCH_3), 2.30 (t, $J = 7.5$ Hz, 4H, 2-, 25-H), 2.20–1.8 (m, 4H, 11-, 16-H), 1.68–1.51 (m, 8H, 3-, 24-, 10-, 17-H), 1.98–1.20 (m, 24H, 4–9-, 18–23-H). – ^{13}C NMR (CDCl_3): $\delta = 174.3$ (C-1, -26), 51.4 (2 \times OCH_3), 31.1 (m, C-11, -16), 34.1 (C-2, -25), 31.8–29.2 (12 C, C-4–9, -18–23), 24.9 C-3, -24), 20.2 (C-10, -17). – CI MS (isobutane), m/z : 600 [MH^+]. – MS (70 eV), m/z (%): 568 [$\text{M}^+ - \text{OCH}_3$] (2.6), 526 (3.6), 494 (4.5), 159 (5), 112 (20), 98 (85), 74 (100), 69 (43), 56 (69)^[6].

Addition of 1, ω -Perfluoroalkyl Diiodides to Methyl 10-Undecenoate: A mixture of 2.60 g (13 mmol) of lead powder and 0.10 g (1.6 mmol) of $\text{Cu}(\text{OAc})_2$ in 100 ml of methanol was stirred under nitrogen for 30 min. Then 11 g (20.9 mmol) of 1, ω -perfluoroalkyl diiodides and 2.5 g (13 mmol) of methyl 10-undecenoate were slowly added. After 48 h the reaction mixture was concentrated, the resi-

due was dissolved in diethyl ether and the solution filtered. The solvent was evaporated and flash chromatography (petroleum ether/diethyl ether, 9:1) of the residue yielded 8.40 g (56%) of 10 as a mixture of homologs^[6]. — n_D^{25} : 1.4432. — ¹H NMR (CDCl₃): δ = 4.40–4.20 (m, 1H, 10-H), 3.64 (s, 3H, OCH₃), 3.0–2.6 (m, 2H, 11-H), 2.30 (t, J = 7.4 Hz, 2H, 2-H), 1.90–1.20 (m, 14H, 4–9-H). — ¹³C NMR (CDCl₃): δ = 174.3 (C-1), 51.4 (OCH₃), 41.6 (m, C-11), 40.3 (C-10), 34.1 (C-2), 29.5–28.4 (5 C, C-4, -5, -6, -7, -8), 24.9 (C-3), 21.2–21.0 (C-9). — MS (70 eV), m/z : 493 [M⁺ – HI – CH₃O] (0.3), 475 (0.3), 409 (0.4), 128 (14), 127 (10), 97 (28), 83 (30), 74 (50), 69 (85), 55 (100).

Hydrogenation of the Addition Product 10: To a solution of 1.00 g (1.4 mmol) of 10 in 150 ml of methanol were added 0.20 g of sodium hydrogen carbonate and 0.20 g of Pd/C(10%). Hydrogenation of the mixture at a hydrogen pressure of 2.8 bar at room temp. for 24 h gave after filtration and evaporation of the solvent 0.39 g (60%) of 11 as a mixture of homologs^[6]. — M.p.: 27°C. — ¹H NMR (CDCl₃): δ = 6.03 (tt, J = 52.0, 5.6 Hz, 1H, 17-H), 3.68 (s, 3H, OCH₃), 2.30 (t, J = 7.4 Hz, 2H, 2-H), 2.20–1.90 (m, 2H, 11-H), 1.68–1.52 (m, 4H, 3-, 11-H), 1.40–1.20 (m, 14H, 4–9-H). — ¹³C NMR (CDCl₃): δ = 174.3 (C-1), 51.4 (OCH₃), 34.1 (C-2), 30.7–31.0 (m, C-11), 29.3–29.1 (6 C, C-4, -5, -6, -7, -8, -9), 24.9 (C-3), 20.1 (C-10). — CI MS (isobutane), m/z : 501 [MH⁺]. — MS

(70 eV), m/z (%): 500 [M⁺] (6), 469 (7), 457 (3), 143 (10), 101 (14), 87 (90), 74 (100), 69 (25), 55 (32).

- [1] J. O. Metzger, U. Linker, *Liebigs Ann. Chem.* 1992, 209–216, and references cited therein.
 [2] Z.-Y. Yang, B. V. Nguyen, D. J. Burton, *Synlett* 1992, 141–142; Q.-Y. Chen, Y.-B. He, Z.-Y. Yang, *J. Fluorine Chem.* 1987, 37, 171–176; A. Miura, Y. Kobayashi, T. Taguchi, *Chem. Lett.* 1990, 1011–1014.
 [3] J. O. Metzger, R. Mahler, *Angew. Chem.* 1994, 107, 1012–1015; *Angew. Chem. Int. Ed. Engl.* 1994, 34, 902–904.
 [4] A. E. Feiring, *J. Org. Chem.* 1985, 50, 3269–3274; W. Bloechi, NL 6506069 1965, *Chem. Abstr.* 1966, 64, 17421c; W.-Y. Huang, L.-Q. Hu, W.-Z. Ge, *J. Fluorine Chem.* 1989, 43, 305–318; K. Baum, C. D. Bedford, R. J. Hunadi, *J. Org. Chem.* 1982, 47, 2251–2257; H. Urata, Y. Kinoshita, T. Asanuma, O. Kosukegawa, T. Fuchikami, *J. Org. Chem.* 1991, 56, 4996–4999; I. L. Knunyants, S. P. Khrlakyan, Yu. V. Zeifman, V. V. Shokina, *Bull. Acad. Sci. USSR Div. Chem. Sci. (Engl. Transl.)* 1964, 359–361; S. P. Khrlakyan, I. L. Knunyants, V. V. Shokina, *Bull. Acad. Sci. USSR Div. Chem. Sci. (Engl. Transl.)* 1965, 62–64.
 [5] F. Roehrscheid, Hoechst AG, DE 2163752, 1971, *Chem. Abstr.* 1973, 79, 78110.
 [6] Obtained yields were calculated on the basis of the average molecular weight of the perfluoroalkyl diiodides of 531.8. The mass spectroscopic data of the C₄F₈I₂ addition product are given.

[95241]