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Functional group transformation of perfluoroadamantane derivatives

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Abstract

An adamantane skeleton, having rigid structure, has been used for a thermally stable and/or anti-etching material. It is expected to be a material having good optical properties, if fluorine atoms are introduced into the skeleton. Thus, the investigation concerning the functional group transformation of perfluoroadamantanecarboxylic acid derivatives was performed to develop the methods for introduction of perfluoroadamantyl group into molecules. Their unique reactivities are also discussed.

Keywords: Adamantane; Perfluorinated compound

1. Introduction

Adamantane have been attracted for about a century in academic field, since it shows unusual chemical and physical properties derived from its highly symmetric caged structure [1]. Thus, several polymers containing adamantane skeletons have been synthesized, and some of them have showed good thermal stability and transparency [2–8]. Moreover, such the polymers are recently expected to use as highly functional materials for state-of-the-art industry. For example, they have been investigated for a couple of decades as material for opto-electronics [9,10] due to their good optical property and thermal stability, or as material for photolithography [11–13] due to their anti-etching property and UV transparency.

It is expected to be a material having better thermal stability, transparency and so on, if fluorine atoms are introduced into the skeleton. However, only a few highly fluorinated adamantane derivatives are known. Adcock et al. fluorinated several adamantane derivatives using their aerosol fluorination technique to obtain perfluorinated adamantyl halides, hydrides and alcohols [14–17]. A research group in Idemitsu Industries presented monomers containing polyfluoroadamantyl group and their polymers [18–20].

Recently, our research group demonstrated that adamantyl and/or adamantanemethyl perfluoroalkanoates were easily fluorinated in liquid phase and then eliminated [21,22] to obtain perfluoroadamantanols and/or perfluoroadamantanecarbonyl fluorides, respectively [23]. These results opened up the way for the industrial scale preparation of perfluorinated adamantane derivatives.

Herein we report the functional group transformation of perfluoroadamantanecarboxylic acid derivatives. To apply these derivatives for industrial materials, it is necessary to know the reactivity of the functional groups, and to change the functional groups into desired forms. After that, they can be introduced in the materials. Throughout the investigation, they showed unique reactivates, which are discussed hereinafter.

2. Results and discussion

Pentadecafluoro-1-adamantanecarbonyl fluoride (**1**) was used as the starting material in this work. Compound **1** was prepared by the method according to the patent [23].

2.1. Ester

The methyl ester **2** was obtained easily by the reaction of **1** with methanol in the presence of sodium fluoride as a base (Scheme 1).

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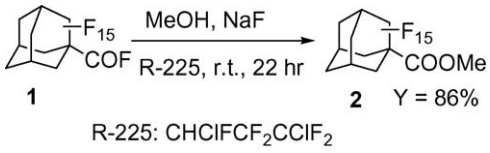
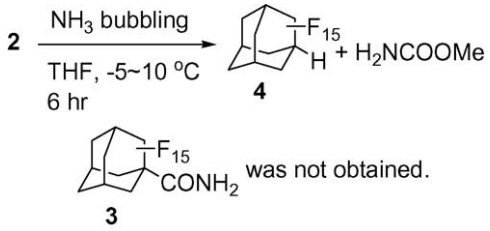
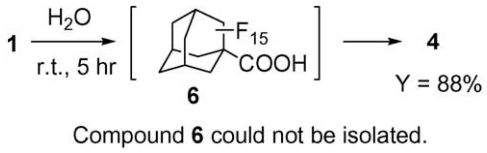


Scheme 1. Synthesis of **2**.



Scheme 2. Reaction of **2** with NH_3.



Scheme 4. Synthesis of **4**.

Table 1
Reaction of **4**: **4** + reagent $\xrightarrow{KOH/DMSO}$ F_{15}–X

Entry	Reagent	Condition	–X	Yield (%)
1	36–38% formalin	75 °C, 6 h	$–CH_2OH$ (**5**)	73
2	CH_2=$CHCH_2Br$	70 °C, 6 h	–CH=CHMe (**7**)	74

The attempt to obtain the amide **3** by the reaction of **2** with ammonia in THF was unsuccessful. Instead, the hydrofluorocarbon **4** and methyl carbamate were obtained (Scheme 2). It is suggested that perfluoroadamantyl group is better leaving group than methoxy group in this case.

Similarly, the reduction of **2** to obtain the methylol **5** was not successful. Several reductants such as lithium aluminum hydride, diisobutylaluminum hydride, and sodium borohydride were applied for the reaction. However, the main product was not **5** in all the cases (Scheme 3).

2.2. Hydride

Compound **4** was formed instead of the carboxylic acid **6** in the case of the reaction of **1** with water (Scheme 4). It seems that **6** readily loses carbon dioxide at ambient temperature. Cheburkov and Knunyants reported that perfluoropivaloyl fluoride reacted similarly [24], though Campbell et al. stated that perfluoro-1-bicyclo[2,2,1]heptanecarboxylic acid was synthesized [25].

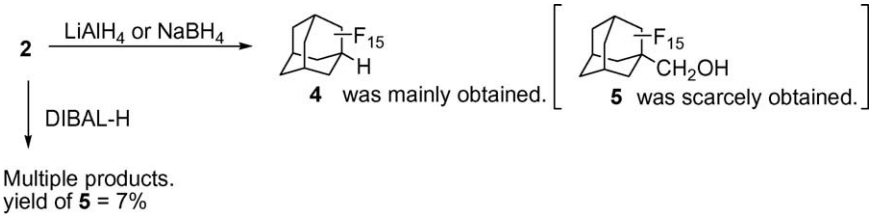
Comparing with other cases, the decarboxylation of linear perfluoroalkanoic acids was reported to occur at above 400 °C in gas phase to produce perfluoroolefines (dehydrofluorination occurred simultaneously) [26]. LaZerte et al. reported that their potassium salts were decarboxylated above 170 °C in ethylene glycol [27]. Perfluorocyclohexanecarboxylic acid was reported to be distillated at 170 °C (740 mmHg), and released carbon dioxide and hydrogen fluoride in gas phase at 550 °C to produce perfluorocyclohexene [28]. According to Howell et al., tetrafluoro-2-perfluoroalkoxypropanoic acids released carbon dioxide at 220, and 160 °C in the case of their ammonium salts [29]. It is supposed that both the steric repulsion between carbonyl group and perfluoroadamantyl group, and high elimination ability of perfluoroadamantyl group causes the decarboxylation easily.

Compound **4** seemed to have enough acidity to be deprotonated. Compound **4** was already prepared and lithiated by Adcock and Luo [15]. They also prepared 1-iodoperfluoroadamantane from **4**. Further, Stephens, Tatlow et al. prepared 1*H*-perfluorobicyclo[2,2,1]heptane and 1*H*-perfluorobicyclo[2,2,2]octane, and transformed them into several derivatives [25,30–33]. Their pH values were measured as 20.5 and 18.3, respectively [34]. It was reported that the hydrogen of 1*H*-perfluorobicyclo[2,2,1]heptane was subtracted by potassium hydroxide in aqueous polar solvent, and the formed carboanion behaved as nucleophile [33]. Thus, we applied the reaction condition to the reaction of **4** with formalin, and successfully obtained **5**. Compound **4** also reacted with allyl bromide to give **7**, the double bond migrated product (Table 1) [35].

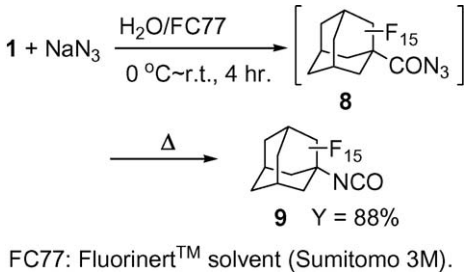
2.3. Isocyanate

Commonly, isocyanates are useful substrates because of their high reactivity. The isocyanate **9** was prepared using Curtius rearrangement of the azide **8** (Scheme 5) [36].

Further, compound **9** was reacted with nucleophiles under mild conditions to obtain the amine **10**, the carbamate **11** and the urea **12** (Table 2). Compound **10** has less nucleophilicity than common amines so that it did not react with acetyl chloride (NEt_3/CH_2Cl_2, r.t., 22 h) to form the amide.



Scheme 3. Reaction of **2** with reductants.



Scheme 5. Synthesis of **9**.

Table 2
Reaction of **9** with nucleophiles: **9** + nucleophile → F_{15}–NHY

Entry	Nuleophile	Condition	–Y	Yield (%)
1	H_2O	THF, r.t. 5.5 h	–H (**10**)	90
3	$PhNH_2$	R-225[a], r.t. 5.5 h	–CONHPh (**12**)	82
2	EtOH	R-225[a], r.t. 5 h	–COOEt (**11**)	88

[a] 1,3-Dichloro-1,1,2,2,3-pentafluoropropane.

3. Conclusion

In summary, a series of perfluoroadamantane derivatives have been synthesized using the acid fluoride **1** as starting material (Scheme 6). Compound **1** and its derivatives showed somewhat unusual chemical properties because of the electron-withdrawing property, the stability and the steric bulkiness of perfluoroadamantyl group. Thus, unique methods were often required for the functional group transformation. It is expected that the methods described in this report will be used for the development of high performance materials containing perfluoroadamantane moiety.

4. Experimental

4.1. General

The boiling point and the melting point of **2** were not corrected. IR spectra were recorded on a Nicolet Impact 410 spectrometer. ^{1}H NMR (internal TMS) and ^{19}F NMR (internal $CFCl_3$) were taken on a JEOL AL300 spectrometer. HRMS were taken at the analytical science division of our research center. Commercially obtained materials were used as received unless otherwise noted. All reactions sensitive to oxygen and/or moisture were conducted under nitrogen atmosphere with magnetic stirring. Compound **1** was purified by sublimation *in vacuo* (heated up to 110 °C at 30 mmHg) before use.

4.2. Pentadecafluoro-1-adamantanecarboxylic acid methyl ester (*2*) [107376-45-4]

To a stirred mixture of 3.90 g (122 mmol) of dry methanol and 6.35 g (151 mmol) of sodium fluoride in 40 mL of 1,3-dichloro-1,1,2,2,3-pentafluoropropane (R-225) at 10 °C was added the solution of 45.2 g (100 mmol) of **1** in 20 mL of R-225 dropwise for 50 min. The reaction was exothermic and the internal temperature raised up to 29 °C. The mixture was stirred for 22 h at room temperature. The mixture was filtered and the solvent was evaporated. Distillation *in vacuo* provided 40.0 g of **2** (86.3 mmol, $Y = 86\%$).

2: mp 26–28 °C; bp 81–84 °C/10 mmHg; ^{1}H NMR (300.4 MHz, $CDCl_3$, δ): 4.05 cm^{-1} (s); ^{19}F NMR (282.7 MHz, $CDCl_3$, δ): −111.03 cm^{-1} (m, 6F), −121.12 cm^{-1} (m, 6F), −219.24 cm^{-1} (m, 3F); IR (neat): 2969.5, 1776.1, 1280.0, 974.2, 964.5 cm^{-1}.

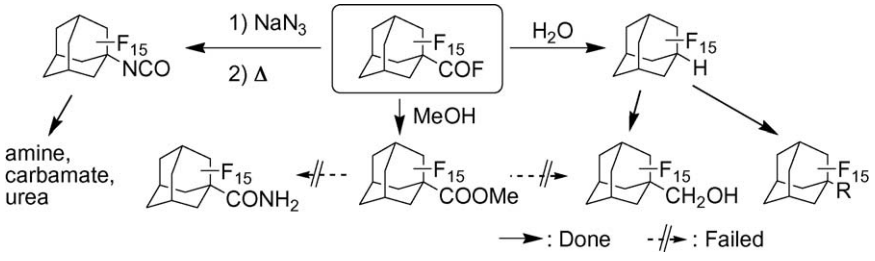
4.3. 1H-pentadecafluoroadamantane (*4*) [54767-15-6]

To a stirred mixture of 27.5 g (61.3 mmol) of **1** and 3.78 g (90.0 mmol) of sodium fluoride in 100 mL of acetone under 5 °C was added 1.14 g (63.3 mmol) of water dropwise for 10 min. The reaction was exothermic and the internal temperature raised up to 29 °C. The mixture was stirred for 5 h at 20 °C. The mixture was filtered and the filtrate was dried over $MgSO_4$, filtered, and freed of solvent *in vacuo*. Sublimation at 50 mmHg gave 22.0 g of **4** (54.2 mmol, $Y = 88\%$) as a white solid.

4: sublimed away above 90 °C/50 mmHg; ^{1}H NMR (300.4 MHz, $CDCl_3$, δ): 3.91 cm^{-1} (dec, $J = 5.6$ Hz); ^{19}F NMR (282.7 MHz, $CDCl_3$, δ): −108.73 cm^{-1} (m, 6F), −120.33 cm^{-1} (dm, $J = 276.2$ Hz, 3F), −121.72 cm^{-1} (dm, $J = 276.2$ Hz, 3F), −219.43 cm^{-1} (m, 3F); IR (KBr): 2921.2, 2850.6, 1239.5 cm^{-1}.

4.4. (Pentadecafluoro-1-adamantane)methanol (*5*)

To a stirred solution of 2.03 g (5.00 mmol) of **4** in 50 mL of dimethyl sulfoxide at 20 °C was added the solution of 1.00 g (15.2 mmol) of potassium hydroxide in 10 mL of water. The internal temperature raised up to 45 °C. Formalin (36–38%,



Scheme 6.

20 mL) was successively added and the mixture was warmed to 76 °C and stirred for 6 h. The mixture was neutralized by 3 mL of 6*N*-HCl, and 150 mL of water was added. The mixture was extracted with R-225 (1 × 40 mL). The organic phase was washed with water twice, dried over $MgSO_4$, filtered, and the solvent was evaporated. Compound **5** (1.58 g, 3.62 mmol, $Y = 72\%$) was obtained as a white solid.

5: sublimed away above 62 °C; ^{1}H NMR (300.4 MHz, $CDCl_3$, δ): 1.91 cm^{-1} (br, 1H), 4.64 cm^{-1} (s, 2H); ^{19}F NMR (282.7 MHz, $CDCl_3$, δ): −113.90 cm^{-1} (m, 6F), −121.17 cm^{-1} (m, 6F), −219.34 cm^{-1} (m, 3F); IR (KBr): 3359.3, 2975.0, 2917.8, 1271.9, 1234.6, 1022.9, 984.7, 967.9, 905.9 cm^{-1}; HRMS (CI) *m/z* ($M + H^+$, $C_{11}H_4F_{15}O^+$) calcd. 437.0023, found 437.0029.

4.5. Pentadecafluoro-1-(1-propenyl)adamantane (7)

To a stirred solution of 2.03 g (5.00 mmol) of **4** in 50 mL of dimethyl sulfoxide at 20 °C was added the solution of 1.00 g (15.2 mmol) of potassium hydroxide in 10 mL of water. The internal temperature raised up to 40 °C. Allyl bromide (0.68 g, 5.6 mmol) was further added and the mixture was warmed to 70 °C and stirred for 5 h. The mixture was neutralized by 3 mL of 6*N*-HCl, and 150 mL of water was added. The mixture was extracted with R-225 (1 × 40 mL). The organic phase was washed with water twice, dried over $MgSO_4$, filtered, and the solvent was evaporated. Compound **7** (1.65 g, 3.70 mmol, $Y = 74\%$) was obtained as a white solid.

6: sublimed away above 69 °C; ^{1}H NMR (300.4 MHz, $CDCl_3$, δ): 1.95 cm^{-1} (dd, $J = 1.7$, 6.6 Hz, 3H), 5.46 cm^{-1} (d, $J = 16.5$ Hz, 1H), 6.57 cm^{-1} (dq, $J = 6.6$, 16.5 Hz, 1H); ^{19}F NMR (282.7 MHz, $CDCl_3$, δ): −113.68 cm^{-1} (m, 6F), −121.19 cm^{-1} (m, 6F), −219.36 (m, 3F); IR (KBr): 3004.9, 2870.9, 1668.4, 1273.3, 957.5, 900.4 cm^{-1}; HRMS (EI) *m/z* (M^+, $C_{13}H_5F_{15}^+$) calcd. 446.0152, found 446.0154.

4.6. Pentadecafluoro-1-isocyanatoadamantane (9)

In a 100-mL four-necked flask equipped with a thermometer, a dropping funnel and a reflux condenser, are placed a solution of 3.25 g (50.0 mmol) of previously activated sodium azide in 8 mL of water and 20 mL of FC77 (one of the Fluorinert™ solvent provided by Sumitomo 3M). Then a solution of 18.1 g (40.1 mmol) of **1** in 20 mL of FC77 (Sumitomo 3 M) was dropped in with vigorous stirring for 80 min. The internal temperature should not rise above 5 °C. Stirring was continued for 1 h with cooling, then for 4 h at room temperature. The organic layer was separated and dried over $MgSO_4$. The dried FC77 solution was placed in a flask with a reflux condenser, and allowed to stand at 80–100 °C till the gas had ceased to evolve (about 1 h). FC77 was removed *in vacuo* (*ca.* 20 mmHg) to afford 15.7 g of **9** (35.0 mmol, $Y = 88\%$) as a white solid.

9: sublimed away above 50 °C; ^{19}F NMR (282.7 MHz, $CDCl_3$, δ): −117.13 cm^{-1} (m, 6F), −121.06 cm^{-1} (m, 6F), −220.89 cm^{-1} (m, 3F); IR (KBr): 2276.4, 1277.3, 1244.1, 963.6 cm^{-1}; HRMS (EI) *m/z* (M^+, $C_{11}F_{15}NO^+$) calcd. 446.9740, found 446.9741.

4.7. Pentadecafluoro-1-adamantylamine (10)

To a stirred solution of 1.95 g (4.36 mmol) of **9** in 10 mL of THF at 0 °C was added 0.086 g (4.8 mmol) of water. The mixture was stirred for 5.5 h at room temperature. The solvent was evaporated to obtain 1.65 g of **10** (3.92 mmol, $Y = 90\%$) as a white solid.

10: sublimed away above 80 °C; ^{1}H NMR (300.4 MHz, $CDCl_3$, δ): 2.14 cm^{-1} (br); ^{19}F NMR (282.7 MHz, $CDCl_3$, δ): −120.14 cm^{-1} (m, 6F), −121.27 cm^{-1} (m, 6F), −221.11 cm^{-1} (m, 3F); IR (KBr): 3430.2, 1272.4, 961.3 cm^{-1}; HRMS (EI) *m/z* (M^+, $C_{10}H_2F_{15}N^+$) calcd. 420.9948, found 420.9951.

4.8. Ethyl N-(pentadecafluoro-1-adamantyl)carbamate (11)

To a stirred solution of 1.99 g (4.45 mmol) of **9** in 10 mL of R-225 at 0 °C was added dropwise 0.218 g (4.73 mmol) of dry ethanol. The mixture was stirred for 5 h at room temperature. The solvent was removed *in vacuo* to afford 1.94 g of **11** (3.93 mmol, $Y = 88\%$) as a white solid.

11: sublimed away above 75 °C; ^{1}H NMR (300.4 MHz, $CDCl_3$, δ): 1.32 cm^{-1} (t, $J = 7.1$ Hz, 3H), 4.24 cm^{-1} (q, $J = 7.1$ Hz, 2H), 5.24 cm^{-1} (br, 1H); ^{19}F NMR (282.7 MHz, $CDCl_3$, δ): −114.51 cm^{-1} (m, 6F), −121.12 (m, 6F), −220.67 cm^{-1} (m, 3F); IR (KBr): 3432.0, 3283.6, 30755, 2996.5, 1756.4, 1555.2, 1276.2, 992.6, 975.6, 958.8, 921.2 cm^{-1}; HRMS (CI) *m/z* ($M + H^+$, $C_{13}H_7F_{15}NO_2^+$) calcd. 494.0237, found 494.0249.

4.9. N-(Pentadecafluoro-1-adamantyl)-N′-phenylurea (12)

To a stirred solution of 2.01 g (4.50 mmol) of **9** in 10 mL of R-225 at 0 °C was added dropwise 0.419 g (4.50 mmol) of aniline. The mixture was stirred for 5 h at room temperature. The white solid was participated, which was collected by filtration and dried *in vacuo*. Compound **12** (1.98 g, 3.67 mmol, $Y = 82\%$) was obtained as a white solid.

12: sublimed away above 168 °C; ^{1}H NMR (300.4 MHz, acetone-d_6, δ): 3.79 cm^{-1} (s, 2H), 7.06 cm^{-1} (m, 1H), 7.31 cm^{-1} (m, 2H), 7.48 cm^{-1} (t, $J = 7.5$ Hz, 2H); ^{19}F NMR (282.7 MHz, $CDCl_3$, δ): −112.66 cm^{-1} (m, 6F), −120.37 cm^{-1} (m, 6F), −220.00 cm^{-1} (m, 3F); IR (KBr): 3345.6, 3103.7, 1692.9, 1564.8, 1501.8, 1447.8, 1273.8, 1028.8, 997.4, 976.2, 958.8, 938.4, 856.5 cm^{-1}; HRMS (EI) *m/z* (M^+, $C_{17}H_7F_{15}N_2O^+$) calcd. 540.0319, found 540.0316.

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