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Nucleophilic displacements of 2-perfluoroalkyl-1-iodoethanes: improved synthesis of fluorine-containing malonic esters

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Abstract

The replacement of iodide in 2-perfluoroalkyl-1-iodoethanes by anions derived from dialkyl malonate affords α-(2-perfluoroalkyl)ethyl malonic esters in high yields with negligible elimination and dialkylation, using bases such as K_2CO_3 or NaH in THF. The physical properties of the products are described.

Keywords: 2-Perfluoroalkyl-1-iodoethane; Dialkyl malonate; Dialkyl-α-(2-perfluoroalkylethyl)malonic esters; Dialkyl-α,α-di(2-perfluoroalkylethyl)malonic esters

1. Introduction

2-Perfluoroalkyl-1-iodoethanes are useful intermediates in the synthesis of monomers bearing a perfluoroalkyl 'tail' such as alcohols [1–7], azides [8,9], amines [10,11], isocyanates [12], isothiocyanates [13], isocyanide dichlorides [14], oxiranes [15–16], nitriles [17], thiols [18,19]. However, the main drawback of these electrophiles as alkylating agents in the presence of basic and nucleophilic reagents is that often the major reaction is a bimolecular elimination (E_2), leading to the formation of olefins $R_FCH = CH_2$. Such facile elimination is common among alkyl halides that have an electron-attracting (acid-strengthening) group at the beta carbon atom. Further, it was reported that the replacement of halogen atom in $R_FCH_2CH_2I$ by nucleophilic reagents predominates over the usual elimination to olefin when the reagent is strongly nucleophilic but only weakly basic. So, reagents such as RS^- [8], NCS^- [9], N_3^- [9,11] and phosphorus nucleophiles [20–22] react very well in displacements, and in addition, increasing the number of methylene groups increases the rate of substitution and decreases the rate of elimination [23].

Previously, Smeltz [24] had claimed that the preparation of malonic esters having polyfluoroalkyl substituents was effected by alkylation of dialkyl malonates with 2-perfluoroalkyl-1-iodoethane. However, the reaction requires the use of sodium *t*-butoxide in *t*-butanol, and elimination is an important competing side reaction as also are dialkylation and *trans*-esterification. We have examined the scope and limitations of this reaction, and we report our findings herein.

2. Results and discussion

The general procedure for alkylation of dialkyl malonic esters with the different possible products is summarized in Scheme 1.

When sodium alkoxides are used as base in the corresponding alcohol as solvent (methanol for dimethylmalonate or ethanol for diethylmalonate), the reaction leads to a mixture of fluorinated products depending on the stoichiometry of the reagents. For instance, treatment of one equivalent of diethyl malonate **1a** with one equivalent of sodium in ethanol and one equivalent of 2-perfluorohexyl-1-iodoethane **2b** affords a multicomponent mixture which gives the olefin **3b** as the major fraction (46%), monoalkylated diethylmalonic ester **4c** (35%), and dialkylated diethylmalonic ester **5c** (5%). Moreover, a *trans*-esterification reaction is also observed when an alcohol different to that corresponding to the malonic ester is used. Thus, using a mixture of sodium *t*-butoxide in *t*-butanol and methanol with diethyl malonate, Smeltz obtained a mixture of methyl and ethyl fluoroalkylated malonic esters [24].

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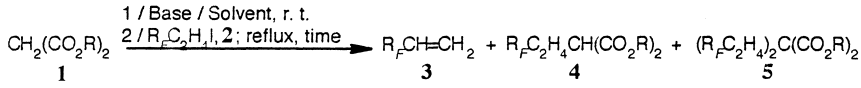


1a R = Et; **1b** R = Me

2a $R_F = C_4F_9$; **2b** $R_F = C_6F_{13}$; **2c** $R_F = C_8F_{17}$

3a $R_F = C_4F_9$; **3b** $R_F = C_6F_{13}$; **3c** $R_F = C_8F_{17}$

4a, **5a** $R_F = C_4F_9$, R = Et; **4b**, **5b** $R_F = C_4F_9$, R = Me; **4c**, **5c** $R_F = C_6F_{13}$, R= Et;

4d, **5d** $R_F = C_6F_{13}$, R = Me; **4e**, **5e** $R_F = C_8F_{17}$, R = Et; **4f**, **5f** $R_F = C_8F_{17}$, R= Me

Scheme 1.

In an attempt to avoid the formation of side products, potassium carbonate in tetrahydrofuran was used instead of metal alkoxides in alcohols. Under these conditions mono-alkylation is the main reaction, and only low percentages of dialkylation and elimination products are observed in GC. It must be pointed out that using potassium carbonate, the reaction is not complete after 27 h, and 20% of unchanged starting 2-perfluoroalkyl-1-iodoethanes are always recovered. Increasing the time of the reaction does not decrease the ratio of halides but favors the elimination reaction. Thus, to increase the yields of mono-alkylation reaction, we ran the reactions with 1/2/2 M stoichiometric amounts of halides, base and dialkyl malonates, respectively. Adding an excess of potassium carbonate and an excess of the starting active methylene compound to the reaction mixture allows complete conversion of the halide. Thus, although the reaction times are long, better results were obtained for mono-alkylated compounds, and neither elimination nor dialkylation is observed (see Scheme 1 and Table 1).

In an effort to make the reaction proceed faster, sodium hydride is substituted for potassium carbonate, using two equivalents of dialkyl malonate with one equivalent of sodium hydride and one equivalent of 2-perfluoroalkyl-1-iodo ethane. This decreases the time of reaction from 27 to 18 h and increases the yield to 90%; negligible elimination and dialkylation reactions are observed (Table 1).

As mentioned above, dialkyl malonates **1** were mono-substituted successfully by the poorly reactive polyfluoroalkyl halides, indicating that anions derived from malonic esters react very well in this nucleophilic displacement. In addition, among the base–solvent combinations used for performing such perfluoroalkylations, it appears that weak bases such as potassium carbonate and aprotic solvent such as THF or strong bases such as sodium hydride in THF should be used in order to obtain an appreciable concentration of anion derived from the active methylene compound **1**.

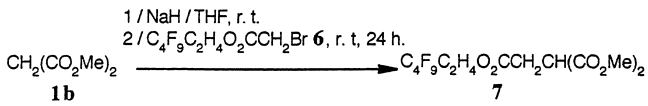
The present malonic ester synthesis of polyfluoroakyl malonic esters compounds may be of more general use.

Table 1
Fluorinated products obtained from the malonic synthesis under different experimental conditions

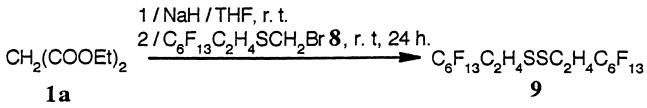
Malonic ester	Halides	Concentration **1a** and **b** ($M \times 5 \times 10^{-2}$)	Equivalent base	Solvent	Time (h)	Reaction mixture composition (GC %)[a]	Products (yield %)[b]
1a	**2b**	1	1EtONa	EtOH	28	**3b** (53) + **4c** (39) + **5c** (8)	**3b** (46) **4c** (35) **5c** (5)
1a	**2a**	2	1NaH	THF	18	**3a** (3) + **4a** (90) + **5a** (2)	**4a** (85)
1a	**2a**	2	$2K_2CO_3$	THF	27	–	**4a** (75)
1b	**2a**	2	1NaH	THF	18	**3a** (3) + **4b** (91) + **5b** (2)	**4b** (85)
1b	**2a**	1	$2K_2CO_3$	THF	27	–	**4b** (70)
1a	**2b**	1	$2K_2CO_3$	THF	27	**3b** (5) + **4c** (59) + **5c** (3)	**4c** (52)
1a	**2b**	2	$2K_2CO_3$	THF	27	–	**4c** (75)
1a	**2b**	2	1NaH	THF	18	**3b** (3) + **4c** (94) + **5c** (2)	**4c** (90)
1b	**2b**	2	1NaH	THF	18	**3b** (3) + **4d** (92) + **5d** (2)	**4d** (87)
1b	**2b**	2	$2K_2CO_3$	THF	27	–	**4d** (70)
1a	**2c**	2	1NaH	THF	18	**3c** (3) + **4e** (95) + **5e** (2)	**4e** (90)
1a	**2c**	2	$2K_2CO_3$	THF	27	–	**4e** (80)
1b	**2c**	2	1NaH	THF	18	**3c** (3) + **4f** (92) + **5f** (2)	**4f** (85)
1b	**2c**	2	$2K_2CO_3$	THF	27	–	**4f** (75)
1b	**6**	2	1NaH	THF	24	–	**7** (85)
1a	**8**	2	1NaH	THF	24	–	**9** (70)

[a] Determined in crude mixture of products; not calibrated.

[b] Isolated yield.



Scheme 2.



Scheme 3.

Thus, the mono-alkylated compound **7** is prepared under the same conditions in good yield (85%) (Scheme 2).

It is noteworthy that, although the bromine atom of α-bromo alkyl acetates is known to be very labile, no trace of dialkylated product, $(C_4F_9C_2H_4O_2CCH_2)_2C(CO_2Me)_2$, is observed. An attempt to involve the (2-perfluorohexylethylthio)bromomethane, **8**, as a reagent possessing a labile bromine atom failed, and led to a disulfide, **9** (Scheme 3).

These results are in accordance with those reported in the literature, showing that (2-perfluoroalkylethylthio)methyl bromides are particularly sensitive to bases and nucleophiles [25].

3. Conclusion

We have established conditions under which active methylene compounds are mono-polyfluoroalkylated. From this study, it is clear that mono-functionalization of malonic esters by polyfluoroalkyl halides is considerably less difficult to achieve than was previously claimed [24], requiring quite different conditions from those used to mono-functionalize malonic esters by alkyl halides. By further reactions, these compounds could be used as precursors in the synthesis of surfactant agents, and this is presently under investigation.

4. Experimental section

The structure of all the compounds prepared was elucidated on the basis of IR, NMR and mass spectroscopic data. Additional evidence for **4a–f** and **7** were obtained from microanalysis.

4.1. General

The starting 2-perfluorobutylethyl-α-bromoacetate [26], and (2-perfluorohexylethylthio)bromomethane [25,27] are prepared following standard literature procedures. THF was distilled over NaH immediately prior to use. All reactions were conducted under an atmosphere of dry N_2 — IR: Perkin-Elmer 1420 — NMR Bruker WH 200 (200 and 188 MHz for ^1H and ^{19}F, respectively). For ^1H NMR, TMS as an internal standard. For ^{19}F NMR, $CFCl_3$ as an internal standard. The solvent for NMR measurement was $CDCl_3$ — MS: Finnigan Mat INCOS 500 E GC/MS system (70 eV).

4.2. Alkylation of dialkyl malonate with 2-perfluoroalkyl-1-iodo ethane in presence of NaH

To a suspension of 50×10^{-3} mol of NaH (80% in oil) in dry 100 ml of THF, 0.1 mol of dialkyl malonate was added dropwise over 30 min, and after cessation of H_2 evolution the mixture was further stirred at room temperature for 20 min. To this solution, 50×10^{-3} mol of 2-perfluoroalkyl-1-iodo ethane was added dropwise, and the mixture was refluxed for 18 h. The solvent was evaporated; then the residue was dissolved in 100 ml of Et_2O. The organic phase was washed with 30 ml of H_2O, and dried (Na_2SO_4). The Et_2O layer is then concentrated under reduced pressure, and the resulting oil was purified by distillation under reduced pressure to yield products **4a–f**.

4a (85%), Colorless oil, bp 65°C/0.2 Torr — IR (ν cm^{-1}): 1753 (C=O), 1738 (C=O), 1300–1100 (CF) — ^1H NMR (δ ppm; J Hz): 1.3 (t, 6H, $2 \times OCH_2\underline{CH_3}$; $J = 7$), 2.21 (m, 4H, $\underline{CH_2CH_2}C_4F_9$), 3.45 (t, 1H, CH; $J = 7$), 4.3 (q, 4H, $2 \times O\underline{CH_2}CH_3$; $J = 7$) — ^{19}F NMR (δ ppm): 81.5 (3F, CF_3), 115.3 (2F, CF_2), 124.8 (2F, CF_2), 126.5 (2F, CF_2) — MS (*m/z*, species, %): 406, M^+, 2; 55, peak of base, 100 — Anal. $C_{13}H_{15}F_9O_4$ (406.2): calcd. C, 38.44; H, 3.72; F, 42.09; found C, 38.56; H, 3.63; F, 42.29%.

4b (85%), Colorless oil, bp 61°C/0.3 Torr — IR (ν cm^{-1}): 1755 (C=O), 1740 (C=O), 1300–1100 (CF) — ^1H NMR (δ ppm; J Hz): 2.19 (m, 4H, $\underline{CH_2CH_2}C_4F_9$), 3.45 (t, 1H, CH; $J = 7$), 3.77 (s, 6H, $2 \times OCH_3$) — ^{19}F NMR (δ ppm): 81.5 (3F, CF_3), 115.2 (2F, CF_2), 124.9 (2F, CF_2), 126.5 (2F, CF_2) — MS (70 eV); *m/z* (%): 378 (2) [M^+], 55 (100) peak of base — Anal. $C_{11}H_{11}F_9O_4$ (378.2): calcd. C, 34.93; H, 2.93; F, 45.21; found C, 35.08; H, 2.88; F, 45.46%.

4c (90%), Colorless oil, bp 81°C/0.2 Torr — IR (ν cm^{-1}): 1753 (C=O), 1738 (C=O), 1300–1100 (CF) — ^1H NMR (δ ppm; J Hz): 1.3 (t, 6H, $2 \times OCH_2\underline{CH_3}$; $J = 7$), 2.21 (m, 4H, $\underline{CH_2CH_2}C_6F_{13}$), 3.45 (t, 1H, CH; $J = 7$), 4.3 (q, 4H, $2 \times O\underline{CH_2}CH_3$; $J = 7$) — ^{19}F NMR (δ ppm): 81.2 (3F, CF_3), 115.1 (2F, CF_2), 122.4 (2F, CF_2), 123.4 (2F, CF_2), 123.9 (2F, CF_2), 126.6 (2F, CF_2) — MS (70 eV); *m/z* (%): 506 (2) [M^+], 55 (100) peak of base — Anal. $C_{15}H_{15}F_{13}O_4$ (506.2): calcd. C, 35.59; H, 2.98; F, 48.79; found C, 35.47; H, 2.92; F, 48.69%.

4d (87%), Colorless oil, bp 75°C/0.2 Torr — IR (ν cm^{-1}): 1753 (C=O), 1740 (C=O), 1300–1100 (CF) — ^1H NMR (δ ppm; J Hz): 2.19 (m, 4H, $\underline{CH_2CH_2}C_6F_{13}$), 3.45 (t, 1H, CH; $J = 7$), 3.77 (s, 6H, $2 \times OCH_3$) — ^{19}F NMR (δ ppm): 81.2 (3F, F_3), 115.1 (2F, CF_2), 122.4 (2F, CF_2), 123.3 (2F, CF_2), 123.8 (2F, CF_2) 126.6 (2F, CF_2) — MS (70 eV); *m/z* (%): 478 (2) [M^+], 55 (100) peak of base — Anal.

$C_{13}H_{11}F_{13}O_4$ (478.2): calcd. C, 32.65; H, 2.31; F, 51.64; found C, 32.82; H, 2.35; F, 51.78%.

4e (90%), Colorless oil, bp 90°C/0.1 Torr — IR (ν cm^{-1}): 1753 (C=O), 1740 (C=O), 1300–1100 (CF) — ^{1}H NMR (δ ppm; J Hz): 1.3 (t, 6H, $2 \times OCH_2\underline{CH_3}$; $J = 7$), 2.21 (m, 4H, $\underline{CH_2}\underline{CH_2}C_8F_{17}$), 3.45 (t, 1H, CH; $J = 7$), 4.3 (q, 4H, $2 \times O\underline{CH_2}CH_3$; $J = 7$) — ^{19}F NMR (δ ppm): 81.2 (3F, CF_3), 115.3 (2F, CF_2), 122.4 (4F, $2 \times CF_2$), 123.2 (2F, CF_2) 123.9 (2F, CF_2), 126.5 (2F, CF_2) — MS (70 eV); *m/z* (%): 606 (2) [M^+], 55 (100) peak of base — Anal. $C_{17}H_{15}F_{17}O_4$ (606.3): calcd. C, 33.67; H, 2.49; F, 53.26; found C, 33.84; H, 2.45; F, 53.38%.

4f (85%), Colorless oil, bp 72°C/0.1 Torr — IR (ν cm^{-1}): 1753 (C=O), 1740 (C=O), 1300–1100 (CF) — ^{1}H NMR (δ ppm; J Hz): 2.19 (m, 4H, $\underline{CH_2}\underline{CH_2}C_8F_{17}$), 3.45 (t, 1H, CH; $J = 7$), 3.77 (s, 6H, $2 \times OCH_3$) — ^{19}F NMR (δ ppm): 81.2 (3F, CF_3), 115.3 (2F, CF_2), 122.4 (4F, $2 \times CF_2$), 122.7 (2F, CF_2) 123.8 (2F, CF_2), 126.6 (2F, CF_2) — MS (70 eV); *m/z* (%): 578 (2) [M^+], 55 (100) peak of base — Anal. $C_{15}H_{11}F_{17}O_4$ (578.2): calcd. C, 31.15; H, 1.91; F, 55.85; found C, 31.31; H, 1.98; F, 55.78%.

4.3. Alkylation of diethyl malonate with 2-perfluorohexyl-1-iodo ethane in presence of EtONa

To 1.15 g (50×10^{-3} mol) of clean sodium cut into small pieces, 10 ml of dry ethanol was added slowly. When all the sodium was dissolved, 8 g (50×10^{-3} mol) of diethyl malonate was added dropwise over 10 min, and the mixture was stirred at 40°C for 15 min. Then, a solution of 23.7 g (50×10^{-3} mol) of 2-perfluorohexyl-1-iodo ethane in 20 ml of dry ethanol was added slowly and heated for another 28 h at 75°C. After cooling, the mixture was extracted with Et_2O and concentrated. The residue was analyzed by GC to show **3b** (53%), **4c** (39%) and **5c** (8%), and distilled to give pure **3b** identified by comparison (GC) with an authentic sample, yield 7.9 g (46%) and 8.8 g of pure **4c** (35%) and 2.1 g of pure **5c** (5%), colorless oil, bp 126°C/0.2 Torr — IR (ν cm^{-1}): 1753 (C=O), 1738 (C=O), 1300–1100 (CF) — ^{1}H NMR (δ ppm; J Hz): 1.3 (t, 6H, $2 \times OCH_2\underline{CH_3}$; $J = 7$), 2.20 (m, 8H, $\underline{CH_2}\underline{CH_2}C_6F_{13}$), 4.3 (q, 4H, $2 \times O\underline{CH_2}CH_3$; $J = 7$) — ^{19}F NMR (δ ppm): 81.1 (6F, CF_3), 115.1 (4F, CF_2), 122.4 (4F, CF_2), 123.3 (4F, CF_2), 123.9 (4F, CF_2), 126.5 (4F, CF_2) — MS (70 eV); *m/z* (%): 852 (2) [M^+], 55 (100) peak of base.

4.4. Alkylation of dialkyl malonate with 2-perfluoroalkyl-1-iodo ethane in presence of K_2CO_3

To a mixture of 50×10^{-3} mol of 2-perfluoroalkyl-1-iodo ethane and of 0.1 mol of dry potassium carbonate, a solution of 0.1 mol of dialkyl malonate in dry 30 ml of THF was added, and the mixture was stirred at 75°C for 27 h. The solvent was evaporated; then the residue was dissolved in 100 ml of Et_2O. The solution was washed with 2×30 ml of H_2O, and dried (Na_2SO_4). The Et_2O layer was then concentrated under reduced pressure and the resulting oil was purified by distillation to yield products **4a–f** according to Table 1.

*4.5. Dimethyl(2-perfluorobutylethoxycarbonyl)methylmalonate (**7**)*

To a suspension of 1.5 g (50×10^{-3} mol) of NaH (80% in oil) in dry 100 ml of THF, 13.2 g (0.1 mol) of dimethyl malonate was added dropwise over 30 min, and after cessation of H_2 evolution the mixture was further stirred at room temperature for 20 min. To this solution were added dropwise 19.2 g (50×10^{-3} mol) of 2-perfluorobutylethyl-α-bromoacetate, and the mixture was stirred at room temperature for 24 h. The solvent was evaporated; then the residue was dissolved in 100 ml of Et_2O. The solution was washed with 40 ml of H_2O, and dried (Na_2SO_4). Removal of solvent gives the crude product which is purified by distillation under vacuum; yield 18.5 g of **7** (85%), colorless oil, bp 85°C/0.01 Torr — IR (ν cm^{-1}): 1743 (C=O), 1300–1100 (CF) — ^{1}H NMR (δ ppm; J Hz): 2.5 (m, 2H, $\underline{CH_2}C_4F_9$), 2.9 (d, 2H, CH_2; $J = 7$), 3.77 (s, 6H, $2 \times OCH_3$), 3.9 (t, 1H, CH; $J = 7$), 4.4 (t, 2H, OCH_2; $J = 6.5$) — ^{19}F NMR (δ ppm): 81.5 (3F, CF_3), 114.4 (2F, CF_2), 121.5 (2F, CF_2), 126.5 (2F, CF_2) — MS (70 eV); *m/z* (%): 436 (2) [M^+], 55 (100) peak of base — Anal. $C_{13}H_{13}F_9O_6$ (436.2): calcd. C, 35.79; H, 3.00; F, 39.20; found C, 35.65; H, 2.94; F, 39.45%.

*4.6. Bis(2-perfluorohexylethyl)disulfide (**9**)*

Preparation of **9** was carried out as for **7** using (2-perfluorohexylethylthio)methylbromide instead of (2-perfluorobutylethyl) α-bromoacetate and **9** is obtained as an oil crystallized at room temperature; mp 35–36°C, litt [28] 36–37°C.

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