Transesterification of dialkyl carbonates with fluorine-containing alcohols*

A. M. Semenova,[a]★ M. G. Pervova,[a] M. A. Ezhikova,[a] M. I. Kodess,[a,b] A. Ya. Zapevalov,[a] and A. V. Pestov[a,b]

[a]*I. Ya. Postovsky Institute of Organic Synthesis, Ural Branch of the Russian Academy of Sciences,*
22/20 ul. Sof'i Kovalevskoy, 620108 Ekaterinburg, Russian Federation.
E-mail: sam@ios.uran.ru
[b]*Ural Federal University named after the First President of Russia, B. N. Yeltsin,*
19 ul. Mira, 620002 Ekaterinburg, Russian Federation

Reactions of transesterification of dimethyl, diethyl and dibutyl carbonates with 2,2,3,3-tetrafluoroethyl-, 2,2,3,3,4,4,5,5-octafluorobutyl- and 2,2,3,3,4,4,5,5,6,6,7,7-dodecafluorohexylcarbinols were investigated in the presence of various catalysts. It was found that the conversion of starting dialkyl carbonate increases upon increasing the length of fluorine-containing radical in the presence of sodium alkoxide. It was shown that the maximum conversion of dimethyl carbonate (90%) was achieved in its reaction with 2,2,3,3,4,4,5,5-octafluoropentan-1-ol upon using catalysts such as K_2CO_3, NaOH, and sodium alkoxide. To achieve similar values of the conversion, $Ti(OEt)_4$ and $Ti(OiPr)_4$ should be used in the cases of diethyl and dibutyl carbonates, respectively.

Key words: transesterification, polyfluorinated alcohols, telomer alcohols, dialkyl carbonates.

An introduction of fluorine atoms or fluorinated functional groups into hydrocarbon-containing compounds allows obtaining derivatives possessing unique physical, biological, and chemical properties.[1—5] Fluor-containing esters of carbonic acid, *viz*. fluorinated dialkyl carbonates, are underexplored compounds, but have recently attracted significant interest due to the opportunity to use them as solvents both in chemical sources of electrical energy and for carrying out specific chemical reactions. The presence of fluorine atoms in organic carbonates maintains a high dielectric constant and low viscosity,[6] which improves the solubility of alkali metal salts upon preparation of an electrolyte, as compared to hydrocarbon analogs, and also leads to a wide temperature range of their practical use.[7—10] Polyfluorinated carbonates are also employed as valuable highly reactive intermediates for the preparation of various derivatives, *e.g.*, carbamates[11,12] and nonsymmetric aliphatic ureas.[13]

The traditional method for the synthesis of dialkyl carbonates, including fluorine-containing ones, is a reaction of alcohols with phosgene.[14] Another method based on the reaction of 2,2,3,3-tetrafluoropropan-1-ol with CCl_4 in the presence of $AlCl_3$ was reported for the preparation of polyfluorinated carbonates.[15] The synthesis of polyfluorinated carbonates in methanol is covered by a patent,[16] whereas there are no any analytical nor spectral data characterizing the composition and structure of the products obtained. Toxic, explosive, and aggressive reaction media causing a negative effect on the environment were used in all those cases. A method for the preparation of dialkyl carbonates using diphenyl carbonate in the presence of amines allows obtaining only non-fluorinated dialkyl carbonates.[17]

The present work was aimed at the one-step transesterification reaction of dimethyl (**1a**), diethyl (**1b**), and dibutyl (**1c**) carbonates with 2,2,3,3-tetrafluoroethyl- (**2a**), 2,2,3,3,4,4,5,5-octafluorobutyl- (**2b**), and 2,2,3,3,4,4,5,5,6,6,7,7-dodecafluorohexylcarbinol (**2c**) in the presence of various catalysts in order to develop methods for the preparation of fluorine-containing dialkyl carbonates.

Results and Discussion

To develop methods for the production of organic carbonates based on 2,2,3,3-tetrafluoropropan-1-ol (**2a**) as modern "green" reagents and solvents,[18] the transesterification reaction of dimethyl carbonate[19] and titanium(IV) tetraalkoxides[20] has been investigated in details. It was impossible under those conditions to obtain carbonates containing more than four carbon atoms in a fluorine-containing hydrocarbon radical. A subsequent two-step transesterification of titanium(IV) alkoxides based on polyfluoroalkan-1-ol allowed obtaining a mixture of alkylpolyfluoroalkyl carbonate and bispolyfluoroalkyl carbonate in the yields of 50—80%. Taking into account the opportunity to efficiently utilize 2,2,3,3-tetrafluoropropan-1-ol (n = 1) (**2a**), 2,2,3,3,4,4,5,5-octafluoropentan-1-ol

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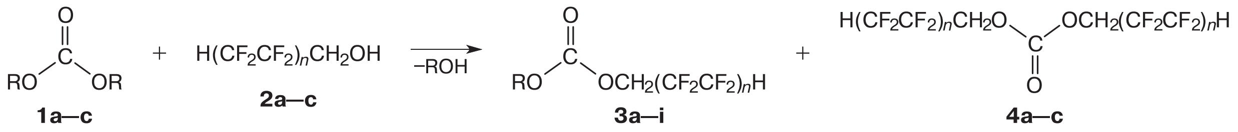
* Dedicated to Academician of the Russian Academy of Sciences V. N. Charushin on the occasion of his 70th birthday.

Published in Russian in *Izvestiya Akademii Nauk. Seriya Khimicheskaya,* No. 5, pp. 933—936, May, 2021.

Scheme 1



1: R = Me (**a**), Et (**b**), Bu (**c**); **2, 4:** *n* = 1 (**a**), 2 (**b**), 3 (**c**);
3: R = Me, *n* = 1 (**a**), 2 (**b**), 3 (**c**); R = Et, *n* = 1 (**d**), 2 (**e**), 3 (**f**); R = Bu, *n* = 1 (**g**), 2 (**h**), 3 (**i**)

(*n* = 2) (**2b**), and 2,2,3,3,4,4,5,5,6,6,6,7,7-dodecafluoroheptan-1-ol (*n* = 3) (**2c**) in the synthesis of fluorine-containing dialkyl carbonates *via* a one-step transesterification, we performed herein a competitive comparison of the catalytic activity of various Lewis acids and bases, depending on the structure of the starting reagents (Scheme 1).

Obtained products **3a—i** and **4a—c** were separated using a fractional distillation, and the ratio between the reaction products was estimated using gas-liquid chromatography (GLC) and ^{1}H NMR spectroscopy. According to the acquired data (Table 1), the reaction of dialkyl carbonates **1a—c** with 2,2,3,3,4,4,5,5-octafluoropentan-1-ol (**2b**) proceeds to give a mixture of alkyl-2,2,3,3,4,4,5,5-octafluoropentyl carbonate (**3b,e,h**) and bis(2,2,3,3,4,4,5,5-octafluoropentyl) carbonate (**4b**) (see Scheme 1). In the case of dimethyl carbonate **1a**, hydroxides and alkoxides ensure a high conversion of alcohol into the carbonate, while sodium compounds exhibited an increased activity compared to potassium compounds. It should be noted that Na_2CO_3 exhibited the lowest activity not only in the comparison with K_2CO_3, but also among all the studied catalysts. Titanium(IV) ethoxide is an efficient Lewis acid, including the transesterification reaction,[21,22] but did not show any activity in our case. However, in the case of transesterification of diethyl (**1b**) and dibutyl (**1c**) carbonates with fluorine-substituted carbinols, titanium(IV) alkoxide exhibited a catalytic activity. Thus, the conversion of diethyl carbonate (**1b**) in its reaction with 2,2,3,3,4,4,5,5-octafluoropentan-1-ol (**2b**) in the presence of tetraethoxytitanium reached 89%, while that in the case of dibutyl carbonate was 63%. Using titanium tetraisopropoxide allowed us to increase the conversion up to 88%.

Table 1. Transesterification of dialkyl carbonates (**1a—c**) with 2,2,3,3,4,4,5,5-octafluoropentan-1-ol (**2b**) in the presence of various catalysts

Catalyst	Carbonate	Molar ratio **2b** : carbonate	Conversion of **2** (%)	Products	Ratio of products
NaOH	**1a**	1 : 0.85	89	**3b** + **4b**	1 : 32
KOH	**1a**	1 : 0.85	71	**3b** + **4b**	1 : 2.3
$Me_4N^+OH^-$	**1a**	1 : 0.85	85	**3b** + **4b**	1 : 7.1
NaOR	**1a**	1 : 0.85	90	**3b** + **4b**	1 : 2
KOR	**1a**	1 : 0.85	71	**3b** + **4b**	1 : 2
Na_2CO_3	**1a**	1 : 0.85	43	**3b** + **4b**	1 : 2
K_2CO_3	**1a**	1 : 0.85	89	**3b** + **4b**	1 : 26
$Ti(OEt)_4$	**1a**	1 : 0.85	0	**3b** + **4b**	—
NaOR	**1b**	1 : 0.85	79	**3e** + **4b**	2.8 : 1
$Ti(OEt)_4$	**1b**	1 : 0.85	89	**3e** + **4b**	1 : 1
$Ti(OEt)_4$	**1b**	1 : 0.68	53	**3e** + **4b**	1 : 0.14
NaOR	**1b**	1 : 0.5	83	**3e** + **4b**	2 : 1
$Ti(OEt)_4$	**1b**	1 : 0.5	86	**3e** + **4b**	1 : 1.4
$Ti(OBu)_4$	**1c**	1 : 0.85	70	**3h** + **4b**	2.3 : 0
NaOR	**1c**	1 : 0.5	50	**3h** + **4b**	1 : 0
Imidazole	**1c**	1 : 0.5	47	**3h** + **4b**	0.9 : 0
$Ti(OEt)_4$	**1c**	1 : 0.5	63	**3h** + **4b**	1.7 : 0
$Ti(OPr^i)_4$	**1c**	1 : 0.5	88	**3h** + **4b**	1 : 2
$Ti(OBu)_4$	**1c**	1 : 0.5	43	**3h** + **4b**	0.8 : 0
$Al(secBuO)_3$	**1c**	1 : 0.5	70	**3h** + **4b**	2.3 : 0
$Sn(Bu^tO)_3$	**1c**	1 : 0.5	47	**3h** + **4b**	0.9 : 0
$BF_3 \cdot Et_2O$	**1c**	1 : 0.5	47	**3h** + **4b**	0.9 : 0
$SnCl_2 \cdot$ 1,4-dioxane	**1c**	1 : 0.5	65	**3h** + **4b**	12 : 1
$SnCl_4 \cdot$ THF	**1c**	1 : 0.5	50	**3h** + **4b**	1 : 0

Table 2. Transesterification of dialkyl carbonates (**1a—c**) with fluorine-containing alcohols (**2a—c**) in the presence of sodium alkoxide*

Alcohol **2**	Carbonate	Conversion of **2** (%)	Products	Ratio of products
2a	**1a**	25	**3a** + **4a**	2.1 : 1
2a	**1b**	18	**3d** + **4a**	9.3 : 1
2a	**1c**	6	**3g** + **4c**	1 : 0
2b	**1a**	90	**3b** + **4b**	1 : 2
2b	**1b**	79	**3e** + **4b**	2.8 : 1
2b	**1c**	79	**3h** + **4b**	2 : 1
2c	**1a**	65	**3c** + **4c**	1 : 3.2
2c	**1b**	66	**3f** + **4c**	1 : 2
2c	**1c**	78	**3i** + **4c**	2.9 : 1

* The molar ratio of starting reagents was alcohol **2a—c** : dialkyl carbonate **1a—c** = 1 : 0.85.

Overall reactivity of the starting carbonates decreases[23] in the series of **1a**, **1b**, and **1c**, which results in a change in the selectivity of formation of fluorine-substituted carbonates **3a—i** and **4a—c** regardless of the alcohol used. The comparison of reactivity of fluorinated alcohols under the conditions of considered transesterification reaction revealed that in the case of carbonates **1a** and **1b**, the maximum reactivity was achived for 2,2,3,3,4,4,5,5-octafluoropentan-1-ol (**2b**) (Table 2). In the case of dibutyl carbonate **1c**, the reactivity of polyfluorinated alcohols **2b** and **2c** was the same.

In summary, the present work revealed that fluorine-containing alcohols can efficiently undergo the transesterification reaction in the presence of Lewis both acids and bases. The conversion of dialkyl carbonates increases with the length of fluorine-containing radical and depends on the composition of carbonate. The maximum conversion of dimethyl carbonate **1a** in the reaction with 2,2,3,3,4,4,5,5-octafluoropentan-1-ol **2b** was achieved in the cases of using K_2CO_3, NaOH, and sodium alkoxide. Titanium(IV) alkoxides maintain a comparable conversion of diethyl and dibutyl carbonates only in the cases of 2,2,3,3,4,4,5,5-octafluoropentan-1-ol and 2,2,3,3,4,4,5,5,6,6,7,7-dodecafluoroheptan-1-ol.

Experimental

2,2,3,3-Tetrafluoropropan-1-ol (98%), 2,2,3,3,4,4,5,5-octafluoropentan-1-ol (98%), and 2,2,3,3,4,4,5,5,6,6,7,7-dodecafluoroheptan-1-ol (98%, HaloPolymer Perm, Russia) and other reagents (AlfaAesar) were used as received without any additional purification.

^1H (400 MHz), ^{19}F (376 MHz), and ^{13}C (126 MHz) NMR spectra were recorded using Bruker AVANCE-500 and DRX-400 spectrometers for solutions in $CDCl_3$ and DMSO-d_6. The ^1H and ^{19}F chemical shifts are given relative to the internal standards $SiMe_4$ and C_6F_6, respectively, while those of ^{13}C are reported relative to the solvent residual signal ($\delta_C = 39.5$). IR spectra were recorded on a Nicolet 6700 spectrometer equipped with an ATR module with a diamond crystal for the range of 400—4000 cm^{-1}. Elemental analysis was performed on a Perkin Elmer CHN PE 2400 automatic analyzer. Chromatographic analysis was carried out using a Shimadzu GC2010 gas—liquid chromatograph equipped with a flame ionization detector and a ZB-5 quartz capillary column (length of 30 m, diameter of 0.25 mm, film thickness of 0.25 μm); nebulizer gas N_2, split flow 1 : 30. The initial column temperature was 40 °C (isotherm 3 min), the temperature was then increased at the rate of 10 °C min^{-1} up to 280 °C (isotherm 15 min), and temperatures of the evaporator and detector were 250 and 300 °C, respectively.

Transesterification (general procedure). A catalyst (1.5 mmol) was added to a mixture of hydropolyfluoroalkan-1-ol (0.1 mol) and dialkyl carbonate (0.085 mol), and the mixture was heated up to reflux. The reaction mass was fractionally distilled at the temperatures >110 °C in the case of 2,2,3,3-tetrafluoropropan-1-ol, >140 °C in the case of 2,2,3,3,4,4,5,5-octafluoropentan-1-ol (98%), and >170 °C for 2,2,3,3,4,4,5,5,6,6,7,7-dodecafluoroheptan-1-ol. The composition of obtained products was analyzed using GLC and ^1H NMR spectroscopy.

Sodium and potassium alkoxides were generated *in situ* by adding certain amounts of metallic sodium or potassium *tert*-butoxide, respectively.

Ethyl(2,2,3,3-tetrafluoropropyl)carbonate 3d. The yield was 7.96 g (13%). Colorless liquid, b.p. 155—156 °C, n_D^{20} 1.340. IR, ν/cm^{-1}: 2990 (C—H), 1762 (C=O), 1271 (C—F), 1108 (C—O). ^1H NMR (DMSO-d_6), δ: 1.24 (t, 3 H, CH_3, J = 7.1 Hz); 4.20 (q, 2 H, $OC\underline{H}_2CH_3$, J = 7.1 Hz); 4.65 (t, 2 H, OCH_2CF_2, J = 14.2 Hz); 6.61 (tt, 1 H, CF_2H, J = 51.8 Hz, J = 5.2 Hz). ^{13}C NMR (DMSO-d_6), δ: 13.79 (CH_3); 62.38 (t, $O\underline{C}H_2CF_2$, J = 26.8 Hz); 64.78 ($O\underline{C}H_2CH_3$); 109.23 (tt, CF_2H, J = 248.0 Hz, J = 33.3 Hz); 114.27 (tt, $OCH_2\underline{C}F_2$, J = 249.4 Hz, J = 26.7 Hz); 153.62 (CO). ^{19}F NMR (DMSO-d_6), δ: 23.89 (dt, 2 F, CF_2H, J = 51.8 Hz, J = 5.2 Hz); 37.5 (tq, 2 F, OCH_2CF_2, J = 14.2 Hz, J = 5.2 Hz). Found (%): C, 35.10; H, 3.95; F, 37.03. $C_6H_8F_4O_3$. Calculated (%): C, 35.30; H, 3.95; F, 37.23.

Bis(2,2,3,3-tetrafluoropropyl)carbonate 4a. The yield was 17%. Colorless liquid, b.p. 181—182 °C, n_D^{20} 1.335. IR, ν/cm^{-1}: 2983 (C—H), 1778 (C=O), 1279 (C—F), 1108 (C—O). ^1H NMR (DMSO-d_6), δ: 4.77 (t, 4 H, OCH_2CF_2, J = 14.2 Hz); 6.63 (tt, 2 H, CF_2H, J = 51.8 Hz, J = 5.2 Hz). ^{13}C NMR (DMSO-d_6), δ: 63.41 (t, $O\underline{C}H_2CF_2$, J = 26.7 Hz); 109.22 (tt, CF_2H, J = 248.1 Hz, J = 33.2 Hz); 114.14 (tt, $OCH_2\underline{C}F_2$, J = 249.7 Hz, J = 26.9 Hz); 152.97 (CO). ^{19}F NMR (DMSO-d_6), δ: 23.95 (dt, 4 F, CF_2H, J = 51.8 Hz, J = 5.2 Hz); 37.55 (tk, 4 F, OCH_2CF_2, J = 14.2 Hz, J =5.2 Hz). Found (%): C, 28.95; H, 2.07; F, 52.21. $C_7H_6F_8O_3$. Calculated (%): C, 28.98; H, 2.08; F, 52.39.

Ethyl(2,2,3,3,4,4,5,5-octafluoropentyl)carbonate 3e. The yield was 15%. Colorless liquid, b.p. 193—195 °C, n_D^{20} 1.339. IR, ν/cm^{-1}: 2991 (C—H), 1764 (C=O), 1267 (C—F), 1127 (C—O). ^1H NMR ($CDCl_3$), δ: 1.34 (t, 3 H, CH_3, J = 7.1 Hz); 4.28 (q, 2 H, $OC\underline{H}_2CH_3$, J = 7.1 Hz); 4.63 (t, 2 H, OCH_2CF_2, J = 13.6 Hz); 6.06 (tt, 1 H, CF_2H, J = 51.9 Hz, J = 5.4 Hz). ^{13}C NMR (DMSO-d_6), δ: 13.85 (CH_3); 62.94 (t, OCH_2CF_2, J = 26.1 Hz); 64.86 (OCH_2CH_3); 109.24 (tt, CF_2H, J = 251.4 Hz, J = 30.9 Hz); 107.3—113.5 (m, 2 CF_2); 114.21 (tt, CH_2CF_2, J = 256.8 Hz, J = 31.2 Hz); 153.21 (CO). ^{19}F NMR ($CDCl_3$), δ: 24.68 (dm, 2 F, CF_2H, J = 51.9 Hz); 31.94 (m, 2 F, CF_2); 36.54 (m, 2 F, CF_2); 41.72 (m, 2 F, OCH_2CF_2). Found (%):

C, 31.60; H, 2.70; F, 50.03. $C_8H_8F_8O_3$. Calculated (%): C, 31.59; H, 2.65; F, 49.97.

Bis(2,2,3,3,4,4,5,5-octafluoropentyl)carbonate 4b. The yield was 18%. Colorless liquid, b.p. 234—235 °C, n_D^{20} 1.324. IR, ν/cm^{-1}: 2986 (C—H), 1780 (C=O), 1274 (C—F), 1124 (C—O). ^{1}H NMR (DMSO-d_6), δ: 5.00 (t, 4 H, OCH_2, J = 14.2 Hz); 7.07 (tt, 2 H, CF_2H, J = 50.2 Hz, J = 5.6 Hz). ^{13}C NMR (DMSO-d_6), δ: 63.07 (t, OCH_2, J = 26.2 Hz); 109.22 (tt, CF_2H, J = 251.4 Hz, J = 30.7 Hz); 107.8—113.0 (m, 2 CF_2); 114.27 (tt, $CH_2\underline{C}F_2$, J = 257.1 Hz, J = 31.0 Hz); 152.61 (CO). ^{19}F NMR (DMSO-d_6), δ: 24.30 (dm, 4 F, CF_2H, J = 50.2 Hz); 33.23 (m, 4 F, CF_2); 38.03 (m, 4 F, CF_2); 43.22 (m, 4 F, OCH_2CF_2). Found (%): C, 26.71; H, 1.14; F, 62.03. $C_7H_6F_8O_3$. Calculated (%): C, 26.96; H, 1.23; F, 62.02.

Bis(2,2,3,3,4,4,5,5,6,6,7,7-dodecafluoroheptyl)carbonate 4c. The yield was 21%. Colorless liquid, b.p. 300—301 °C, n_D^{20} 1.325. IR, ν/cm^{-1}: 2987 (C—H), 1781 (C=O), 1279 (C—F), 1140 (C—O). ^{1}H NMR (DMSO-d_6), δ: 5.03 (t, 4 H, OCH_2, J = 13.8 Hz), 7.14 (tt, 2 H, CF_2H, J = 50.2 Hz, J = 5.3 Hz). ^{13}C NMR (DMSO-d_6), δ: 63.06 (t, OCH_2, J = 26.8 Hz); 107.88 (tt, CF_2H, J = 252.0 Hz, J = 31.1 Hz); 108.0—116.6 (m, 5 CF_2); 152.50 (CO). ^{19}F NMR (DMSO-d_6), δ: 24.23 (dm, 4 F, CF_2H, J = 50.2 Hz); 33.64 (m, 4 F, CF_2); 39.57 (m, 8 F, CF_2); 40.55 (m, 4 F, CF_2); 43.49 (q, 4 F, OCH_2CF_2, J = 13.8 Hz). Found (%): C, 26.11; H, 0.80; F, 65.98. $C_{15}H_6F_{24}O_3$. Calculated (%): C, 26.10; H, 0.88; F, 66.06.

This work was carried out within the framework of the State Assignment to the I. Ya. Postovsky Institute of Organic Synthesis of the Ural Branch of the Russian Academy of Sciences (Topic Nos AAAA-A19-119012290116-9 and AAAA-A19-119012490006-1) using the equipment of the Center for Joint Use "Spectroscopy and Analysis of Organic Compounds".

This paper does not contain descriptions of studies on animals or humans.

The authors declare no competing interests.

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Received February 20, 2021;
in revised form March 29, 2021;
accepted March 30, 2021