Organic Chemistry

Synthesis of fluorine-containing alkyl 2-cyano-3-oxocarboxylates. Reactions with nucleophiles

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Fluorine-containing alkyl 2-cyano-3-oxocarboxylates were synthesized for the first time by the reaction of ethyl cyanoacetate with fluorine-substituted carboxylic acid halides or alkyl carboxylates. The reactions with hydrazine hydrate, phenylhydrazine, and o-phenylenediamine give hydrazinium (ammonium) enolates or products of acid decomposition.

Key words: alkyl perfluoro-2-cyano-3-oxocarboxylates, acylation, condensation, hydrazine hydrate, phenylhydrazine, o-phenylenediamine, salt formation, acid decomposition.
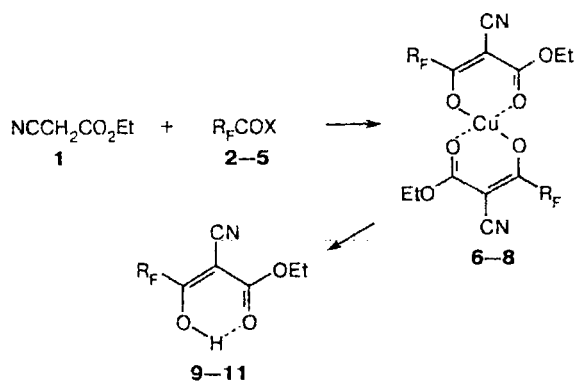
Ethyl 2-cyanoacetoacetate can be obtained by the reactions of sodium ethyl acetoacetate with ClCN[1] or of ethyl cyanoacetate (1) with ketene.[2,3] The most common and convenient method for the synthesis of this 2-cyano-3-oxo ester and its nonfluorinated analogs is acylation of ester 1 with the corresponding acid chlorides[4,5] and anhydrides.[6] Ethyl 2-cyanoacetoacetate reacts with NH_3, $EtNH_2$,[1] and semicarbazide[4] to give azomethines in the form of β-aminocrotonic acid derivatives. Literature data on the synthesis of fluorine-containing alkyl 2-cyano-3-oxocarboxylates and their reactivity are lacking.

The goal of the present investigation is to obtain new esters of fluorinated 3-oxocarboxylic acids. Among the representatives of this class and their derivatives, many substances were found to possess useful properties for practical purposes. Ethyl perfluoro-2-cyano-3-oxocarboxylates and their copper chelates were synthesized for the first time, and their reactions with N-nucleophiles (hydrazine hydrate, phenylhydrazine, and o-phenylenediamine) were studied.

One of the methods of the synthesis of fluorinated alkyl 2-cyano-3-oxocarboxylates is the Claisen condensation of ester 1 with ethyl fluorocarboxylates (2 and 3)

in the presence of Na (Scheme 1). The corresponding esters (9 and 10) are isolated and purified *via* copper chelates (6 and 7) (Table 1).

Scheme 1

$NCCH_2CO_2Et$ + R_FCOX ⟶

1   **2—5**

6—8

9—11

X = OEt (**2, 3**), F (**4**), Cl (**5**)
R_F = CF_3 (**2, 6, 9**), C_4F_9 (**3, 4, 7, 10**), C_6F_5 (**5, 8, 11**)

Table 1. Main characteristics of the compounds synthesized

Com- pound	M.p. /°C (sol- vent)	Yield (%)	Found / Calculated (%)				Molecular formula	IR, ν/cm^{-1}	NMR (DMSO-d$_6$), δ (J/Hz)	
			S	H	F	N			^{1}H	^{19}F
6	>200 (MeOH)	70	<u>35.10</u> 35.05	<u>2.08</u> 2.10	<u>23.85</u> 23.76	<u>5.76</u> 5.84	(C$_7$H$_5$F$_3$NO$_3$)$_2$Cu	1635 (CO$_2$Et); 1530 (C=C); 2210 (CN)		
7	191 (MeOH)	48 (*A*) 66 (*B*)	<u>30.2</u> 30.8	<u>1.15</u> 1.29	<u>43.67</u> 43.85	<u>3.21</u> 3.59	(C$_{10}$H$_5$F$_9$NO$_3$)$_2$Cu	1635 (CO$_2$Et); 1525 (C=C); 2230 (CN)		
8	196	53	<u>42.65</u> 42.65	<u>1.43</u> 1.49	<u>27.85</u> 28.10	<u>4.15</u> 4.14	(C$_{12}$H$_5$F$_5$NO$_3$)$_2$Cu	1620 (CO$_2$Et); 1510—1480 (C=C); 2220 (CN)		
9	—*	95	<u>40.29</u> 40.20	<u>2.89</u> 2.89	<u>27.42</u> 27.25	<u>6.82</u> 6.70	C$_7$H$_6$F$_3$NO$_3$	2700 (OH); 2230 (CN); 1675 (CO$_2$Et); 1620 (C=C)	1.42 (t, 3 H, OCH$_2$<u>Me</u>, J = 7.1); 4.47 (q, 2 H, O<u>CH</u>$_2$Me, J = 7.1); 13.40 (br.s, 1 H, OH)	91.46 (s, CF$_3$)
10	—*	92	<u>33.30</u> 33.44	<u>1.59</u> 1.68	<u>47.95</u> 47.61	<u>3.79</u> 3.90	C$_{10}$H$_6$F$_9$NO$_3$	2700 (OH); 2230 (CN); 1670 (CO$_2$Et); 1610 (C=C)	1.43 (t, 3 H, OCH$_2$<u>Me</u>, J = 7.1); 4.48 (q, 2 H, O<u>CH</u>$_2$Me, J = 7.1); 13.80 (br.s, 1 H, OH)	
11	30—31 (CHCl$_3$)	94	<u>47.03</u> 46.92	<u>2.14</u> 1.97	<u>30.59</u> 30.92	<u>4.53</u> 4.56	C$_{12}$H$_6$F$_5$NO$_3$	2650 (OH); 2230 (CN); 1655 (CO$_2$Et); 1600 (C=C$_{enol}$); 1520—1460 (C=C$_{arom}$)	1.44 (t, 3 H, OCH$_2$<u>Me</u>, J = 7.1); 4.47 (q, 2 H, O<u>CH</u>$_2$Me, J = 7.1); 14.45 (br.s, 1 H, OH)	24.73 (m, 2 F); 15.14 (m, 1 F); 8.87 (m, 2 F)
12	129—130 (CHCl$_3$— MeOH)	96	<u>34.83</u> 34.86	<u>4.03</u> 4.18	<u>16.88</u> 17.42	<u>23.04</u> 23.63	C$_7$H$_{10}$F$_3$N$_3$O$_3$	3300—3180, 2700, 1580 (NH); 2195 (CN); 1690 (CO$_2$Et); 1530 (C=C)	1.14 (t, 3 H, OCH$_2$<u>Me</u>, J = 7.3); 3.96 (q, 2 H, O<u>CH</u>$_2$Me, J = 7.3); 6.8 (br.s, 5 H, NH)	
14	—*	77	<u>41.06</u> 41.13	<u>3.24</u> 3.02	<u>36.68</u> 36.59	<u>8.90</u> 8.99	C$_{16}$H$_{14}$F$_9$N$_3$O$_3$	3270, 2720, 1600 (NH); 2210 (CN); 1680 (CO$_2$Et); 1570—1500 (C=C)	1.09 (t, 3 H, OCH$_2$<u>Me</u>, J = 7.1); 3.98 (q, 2 H, O<u>CH</u>$_2$Me, J = 7.1); 7.03 (m, 5 H, Ph); 9.9 (br.s, 2 H, NH)	82.09 (m, 3 F, CF$_3$); 9.34 (m, 2 F, CF$_2$); 40.91 (m, 2 F, CF$_2$); 37.73 (m, 2 F, CF$_2$)
15	125—126 (CHCl$_3$)	72	<u>52.08</u> 52.06	<u>3.36</u> 3.40	<u>10.14</u> 10.12	<u>22.90</u> 22.87	C$_{18}$H$_{14}$F$_5$N$_3$O$_3$	3210, 2670, 1600 (NH); 2215 (CN); 1660 (CO$_2$Et); 1550, 1500 (C=C)	1.04 (t, 3 H, OCH$_2$<u>Me</u>, J = 7.1); 3.86 (q, 2 H, O<u>CH</u>$_2$Me, J = 7.1); 7.15 (m, 5 H, Ph); 9.99 (br.s, 5 H, Ph)	17.42 (m, 2 F); 3.64 (m, 1 F); 1.23 (m, 2 F)
16	128—129	77	<u>49.34</u> 49.31	<u>4.57</u> 4.45	<u>18.30</u> 17.96	<u>13.22</u> 13.24	C$_{13}$H$_{14}$F$_3$N$_3$O$_3$	3340—3070, 2590, 1580 (NH); 2180 (CN); 1690 (CO$_2$Et); 1630, 1550, 1490 (C=C)	1.14 (t, 3 H, OCH$_2$<u>Me</u>, J = 7.1); 3.96 (q, 2 H, O<u>CH</u>$_2$Me, J = 7.1); 7.6 (br.s, 5 H, NH); 6.93 (m, 4 H, C$_6$H$_4$)	
17	144—145	67	<u>40.81</u> 41.13	<u>2.72</u> 3.02	<u>36.83</u> 36.59	<u>8.98</u> 8.99	C$_{16}$H$_{14}$F$_9$N$_3$O$_3$	3380, 3310, 3090, 2660, 2590, 1570 (NH); 2210 (CN); 1660 (CO$_2$Et); 1625, 1500, 1480 (C=C)	1.18 (t, 3 H, OCH$_2$<u>Me</u>, J = 7.0); 3.87 (q, 2 H, O<u>CH</u>$_2$Me, J = 7.0); 7.0 (br.s, 5 H, NH); 6.9 (m, 4 H, C$_6$H$_4$)	

* Oil.

The acylation of ester **1** with fluorocarboxylic acid halides was also used to synthesize the target fluorine-containing alkyl 2-cyano-3-oxocarboxylates. Thus the reaction of perfluoropentanoyl fluoride **4** with ester **1** in the presence of Et_3N in anhydrous benzene yielded ester **10**, which was isolated *via* copper chelate **7** (see Table 1, Scheme 1). Analogously, ester **11** and its copper chelate (**8**) were obtained by acylating ethyl cyanoacetate with pentafluorobenzoyl chloride (**5**) in the presence of $Mg(OEt)_2$. The use of Et_3N as a catalyst in this reaction did not result in the expected product.

Treatment of chelates **6—8** with gaseous HCl yields free ligands **9—11**.

Compounds **9—11**, like all 1,3-dicarbonyl compounds, exist in both the enol and ketone forms (keto-enol tautomerism). However, the ^{19}F and ^1H NMR spectra of compounds **9—11** (see Table 1) exhibit one set of signals suggesting the presence of only one tautomer in each case. This is enol, which is evidenced by a broadened low-field ^1H singlet for the OH group and no signal for the methine proton. Analysis of IR spectra (see Table 1) also confirms the enol structure of fluorine-containing 2-cyano-3-oxocarboxylic acid esters.

Thus, we established that, in contrast to nonsubstituted esters of fluorinated 3-oxocarboxylic acids where the degree of enolization depends on the structure of a fluorinated substituent,[7] 2-cyano esters **9—11** exist exclusively in the form of enols, irrespective of the nature of the fluorinated radical. It is noteworthy that the degree of enolization of ethyl 2-cyanoacetoacetate, a nonfluorinated analog of compound **9**, is 93%.[2] All the aforesaid correlates with the well known fact that the introduction of electron-withdrawing groups into α- and γ-positions of 1,3-dicarbonyl compounds sharply increases the content of the enol form.[7]

^1H NMR signals for the enol protons in compounds **9—11** are shifted downfield (δ 13.40—14.45) as compared to those in nonsubstituted 3-oxo esters (δ 11.92—11.90[8]). This paramagnetic shift can be explained by the effect of an electron-withdrawing substituent in position 2 (see Ref. 2).

The C=O and C=C absorption bands in the IR spectra of compounds **9—11** are shifted to the lower-frequency region by 15—40 cm^{-1} as compared to those in the IR spectra of fluorinated 3-oxo esters,[8] which is due to the stabilization of (and, accordingly, the strengthening of the bonds in) the chelate ring because of the effect of the electron-withdrawing α-cyano group.[9] The IR spectra of esters **9—11** recorded in $CHCl_3$ and in a thin film are virtually alike, which attests to a minor contribution of intermolecular interactions between the enol OH groups.
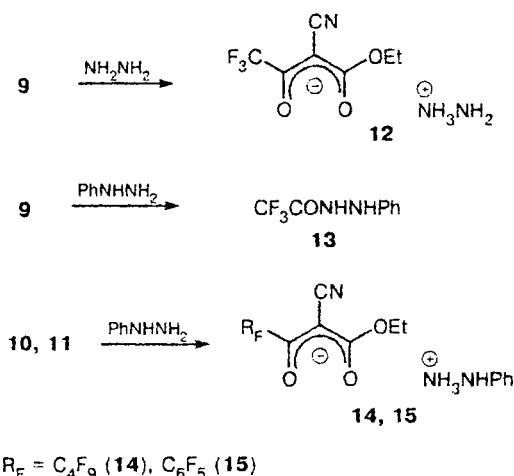
Thus, the intramolecular hydrogen bonds in fluorine-containing 2-cyano-3-oxocarboxylates are stronger than those in the corresponding nonsubstituted 3-oxo esters.

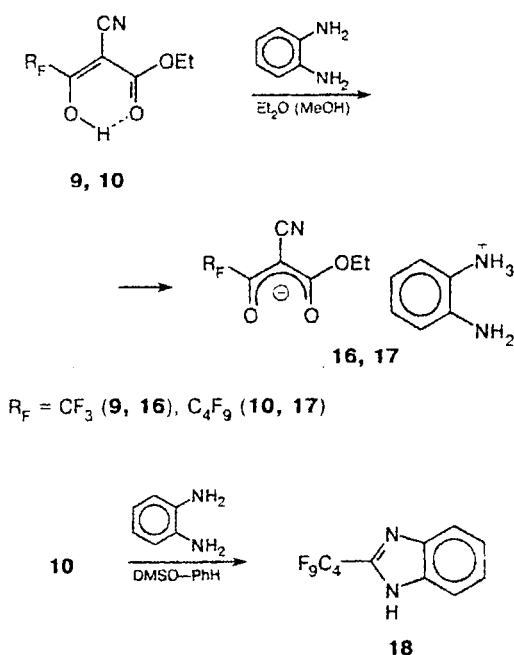Ester **9** reacts with hydrazine hydrate in Et_2O or MeOH to give salt **12** (Scheme 2, Table 1), while oxo

ester **11** does not react with hydrazine hydrate. Under more drastic conditions (refluxing in benzene, AcOH, fusion), the reactions of compounds **9** and **11** with hydrazine hydrate yield a mixture of products that are difficult to separate.

The results of reactions of oxo esters **9—11** with $PhNHNH_2$ depend on the structure of a fluorinated substituent. For example, 2-cyanotrifluoroacetoacetate **9**, when refluxed with $PhNHNH_2$ in Et_2O, undergoes acid decomposition with elimination of trifluoroacetic

Scheme 2

$R_F = C_4F_9$ (**14**), C_6F_5 (**15**)

Scheme 3

9, 10

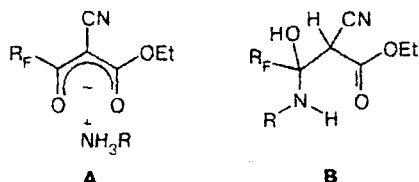**16, 17**

$R_F = CF_3$ (**9, 16**), C_4F_9 (**10, 17**)

18

acid, which gives phenylhydrazide 13 under the reaction conditions (see Scheme 2, Table 1) identical with an authentic sample.[10,11] 3-Oxo esters 10 and 11 react with PhNHNH$_2$ in Et$_2$O or MeOH to form salts 14 and 15 (see Scheme 2, Table 1).

The acid decomposition observed in the reaction of oxo ester 9 with phenylhydrazine may be due to the easier elimination of a fluorocarboxylic acid containing the shorter (as compared to nonafluorobutyl and pentafluorophenyl analogs) trifluoromethyl substituent, which correlates well with the literature data.[12—14]

The pathways of reactions of oxo esters 9 and 10 with o-phenylenediamine depend on the reaction conditions. Thus refluxing in Et$_2$O or MeOH yields salts 16 and 17 (Scheme 3, Table 1), while heating oxo ester 10 with o-phenylenediamine in a benzene—DMSO mixture results in an acid decomposition product, namely, benzoimidazole 18 (see Scheme 3), whose physicochemical characteristics correspond to the published data.[14]

Oxo ester 11, even under mild conditions (Et$_2$O or MeOH, 20 °C), reacts with o-phenylenediamine to give a mixture of products which are difficult to identify.

Compounds 12 and 14—17 are stable and do not undergo any transformations under prolonged heating *in vacuo* or under refluxing in toluene, even in the presence of TsOH. The structure of both salts (A) and the addition products of amine at the ketone carbonyl group of the oxo ester without elimination of water (B) may be assigned to them. Note that nonfluorinated alkyl 2-cyano-3-oxocarboxylates react with nucleophiles to give imines, while fluorinated compounds in such reactions yield addition products.[15—17] However, the ^{1}H NMR spectra of these compounds do not contain signals for the methine protons, which would be present in the case of structure B; in contrast, their IR spectra exhibit absorption bands at 3000—2700 cm^{-1} characteristic of an ammonium group, which is in favor of structure A. In addition, treatment of salt 14 with an aqueous solution of copper acetate gives copper chelate of the initial ligand 7. All of these data are in favor of the salt structure A for compounds 12 and 14—17.

A   B

Thus, there is a competition between salt formation and acid decomposition in the reactions of fluorinated alkyl 2-cyano-3-oxocarboxylates with amines, whereas 3-oxo esters and their α-substituted analogs react with diamines to give heterocyclic compounds.[7,15—17] It is known that the presence of electron-withdrawing α-substituents in fluorinated 3-oxo esters favors their acid decomposition. Thus esters of fluorine-containing 2-acyl-3-oxocarboxylic acids re-

act with NH$_2$NH$_2$, PhNH$_2$, NH$_2$CH$_2$CH$_2$NH$_2$, and o-phenylenediamine to undergo acid decomposition into derivatives of fluorocarboxylic acids, *viz.*, amides and 2-fluoroalkylbenzoimidazoles.[12,13] In the case of fluorinated alkyl 2-cyano-3-oxocarboxylates, salt formation becomes predominant, probably, because of a sharp increase in their acidity (pK_a of ethyl acetoacetate, ethyl 2-cyanoacetoacetate, and ethyl trifluoroacetoacetate are equal to 12.6, 6.0 (see Ref. 15), and 7.4 (see Ref. 7), respectively).

Thus, we showed in this work that the introduction of the electron-withdrawing cyano group into α-position of fluorinated 3-oxo esters basically changes the reactivity of these compounds.

Experimental

IR spectra were recorded on a Specord 75-IR spectrometer in the range 400—4000 cm^{-1} (thin film or Vaseline oil suspension). ^{1}H (80 MHz, relative to SiMe$_4$) and ^{19}F (75 MHz, relative to C$_6$F$_6$) NMR spectra were recorded on a Tesla BS-587 A spectrometer. Elemental analysis was performed with the use of a Carlo Erba CHNS-O EA 1108 analyzer. The properties of the compounds synthesized are presented in Table 1.

Bis(ethyl 2-cyano-4,4,4-trifluoro-3-hydroxybut-2-enoato)-copper (6). A solution of ethyl trifluoroacetate 2 (11.3 g, 0.1 mol) and ethyl cyanoacetate (1) (11.3 g, 0.1 mol) in 100 mL of anhydrous Et$_2$O was added rapidly to metallic sodium (2.5 g, 0.11 g-at.). After the exothermic reaction ceased, the reaction mixture was poured into a saturated aqueous solution of Cu(OAc)$_2$ (9.1 g, 0.05 mol). The precipitate that formed was filtered off and recrystallized from MeOH to give chelate 6 (16.8 g).

Bis(ethyl 2-cyano-4,4,5,5,6,6,7,7,7-nonafluoro-3-hydroxy-hept-2-enoato)copper (7). *A.* Similar reaction of ethyl perfluorovalerate 3 (29.2 g, 0.1 mol) with ester 1 (11.3 g, 0.1 mol) in the presence of metallic Na (2.5 g, 0.11 g-at.) afforded chelate 7 (18.7 g).

B. Perfluoropentanoyl fluoride 4 (26.6 g, 0.1 mol) was added dropwise with stirring at 0—10 °C to a solution of ester 1 (11.3 g, 0.1 mol) and Et$_3$N (11.1 g, 0.1 mol) in 60 mL of anhydrous benzene. The reaction mixture was stirred at 0—10 °C for 1 h, kept at 20 °C for 12 h, and then poured into a mixture of conc. H$_2$SO$_4$ (10 mL) and ice (100 g). The benzene layer was separated, and the products from the aqueous layer were extracted with benzene (2×50 mL). The organic phases were combined and washed with water (3×50 mL), and the solvent was removed. The residue was treated with a saturated aqueous solution of Cu(OAc)$_2$ (9.1 g, 0.05 mol) and recrystallized from MeOH to give product 7 (25.7 g).

Bis(ethyl 2-cyano-3-hydroxy-3-pentafluorophenylprop-2-enoato)copper (8). Magnesium cuttings (2.7 g, 0.11 g-at.) were mixed with 1,2-dibromoethane (0.3 mL) in 5 mL of anhydrous EtOH. After beginning of a vigorous reaction, anhydrous EtOH (7 mL) and 50 mL of anhydrous benzene were added. After the magnesium was consumed completely, ester 1 (11.3 g, 0.1 mol) was added dropwise, and the reaction mixture was stirred at 60 °C for 1 h. Then, a solution of pentafluorobenzoyl chloride 5 (22 g, 0.1 mol) in 50 mL of anhydrous benzene was added, and stirring was continued at 20 °C for 1 h and at 50 °C for 15 min. The reaction mixture was cooled to 5—10 °C, and a solution of conc. H$_2$SO$_4$ (5.4 mL) in 32 mL of water was added. The benzene layer was separated, the solvent was re-

moved, and the residue was treated with $Cu(OAc)_2$ to give product **8** (17.9 g).

Ethyl 2-cyano-4,4,4-trifluoro-3-hydroxybut-2-enoate (9). A flow of HCl was passed through a suspension of compound **6** (24 g, 0.1 mol) in Et_2O until the green color of the solution disappeared. The precipitate that formed was filtered off, and the filtrate was concentrated and chromatographed on Si_2O in $CHCl_3$ to give compound **9** (19.9 g).

Ethyl 2-cyano-4,4,5,5,6,6,7,7,7-nonafluoro-3-hydroxyhept-2-enoate (10) and ethyl 2-cyano-3-hydroxy-3-pentafluorophenylprop-2-enoate (11) were obtained analogously.

Hydrazinium ethyl 2-cyano-4,4,4-trifluoro-3-hydroxybut-2-enoate (12). $NH_2NH_2 \cdot H_2O$ (0.5 g, 10 mmol) was added to a solution of compound **9** (2.09 g, 10 mmol) in 20 mL of Et_2O. The salt that formed was filtered off and recrystallized from a $CHCl_3$—MeOH (10 : 1) mixture, yield 2.26 g.

N'-Phenyltrifluoroacetohydrazide (13). A mixture of compound **9** (2.09 g, 10 mmol) and $PhNHNH_2$ (1.08 g, 10 mmol) was refluxed for 3 h in 20 mL of Et_2O. The solvent was removed, and the residue was reprecipitated with hexane from benzene to give product **13** (1.43 g, 78%), m.p. 130—132 °C (*cf.* Ref. 10).

Phenylhydrazinium ethyl 2-cyano-4,4,5,5,6,6,7,7,7-nonafluoro-3-hydroxyhept-2-enoate (14). A mixture of compound **10** (3.9 g, 10 mmol) and phenylhydrazine (1.08 g, 10 mmol) was refluxed in 20 mL of Et_2O for 1 h. The ether was removed, and the residual oil was washed with $CHCl_3$ to give product **14** (3.6 g).

Phenylhydrazinium salt of ethyl 2-cyano-3-hydroxy-3-pentafluorophenylprop-2-enoate (15) was obtained similarly.

2-Aminoanilinium ethyl 2-cyano-4,4,4-trifluoro-3-hydroxybut-2-enoate (16). A mixture of compound **9** (2.09 g, 10 mmol) and o-phenylenediamine (1.08 g, 10 mmol) was refluxed in 20 mL of Et_2O for 1 h. The precipitate that formed was filtered off and washed with hot $CHCl_3$ to give salt **16** (3.04 g).

2-Aminoanilinium salt of ethyl 2-cyano-4,4,5,5,6,6,7,7,7-nonafluoro-3-hydroxyhept-2-enoate (17). A mixture of compound **10** (3.59 g, 10 mmol) and o-phenylenediamine (1.08 g, 10 mmol) was refluxed in MeOH for 1 h. The product was precipitated with water from MeOH, filtered off, and washed with hot $CHCl_3$ to give compound **17** (3.13 g).

2-Nonafluorobutylbenzoimidazole (18). A mixture of compound **10** (3.59 g, 10 mmol) and o-phenylenediamine (1.08 g, 10 mmol) was heated in a mixture of benzene (20 mL) and DMSO (4 mL) for 5 h. The benzene was removed, and the product was precipitated with water from DMSO and recrystallized to give product **18** (2.3 g, 61%), m.p. 178—179 °C (*cf.* Ref. 15).

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