Reactions of 2,3-epoxypolyfluoroalkanes with triethylamine

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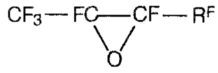
The reactions of 2,3-epoxyperfluoro- and 2,3-epoxy-ω-hydropolyfluoroalkanes with excess triethylamine at elevated temperatures yield secondary alcohols, which are the reduction products of intermediate isomeric ketones. Ring-opening occurs preferentially from the side of the less bulky trifluoromethyl group in all compounds except 2,3-epoxy-6-hydroundecafluorohexane.

Key words: 2,3-epoxypolyfluoroalkanes; triethylamine; polyfluorinated ketones and secondary α-monohydroalcohols.

It has been shown previously[1,2] that terminal fluoroolefin oxides are rearranged into perfluoroacyl fluorides when treated with catalytic amounts of triethylamine (TEA). When these reactions were carried out with greater amounts of TEA, they afforded unsaturated ketones, since TEA reacted with the acyl fluorides that formed.[3]

In the present work we studied the reaction of internal polufluorinated α-oxides (**1**—**5**)[4] with TEA.



1—**5**

$R^F = C_3H_7$ (**1**), C_5H_{11} (**2**), $H(CF_2)_3$ (**3**)

$H(CF_2)_6$ (**4**), $H(CF_2)_7$ (**5**)

It turned out that these compounds, unlike terminal polyfluoroepoxides,[1—3] change insignificantly in the presence of catalytic amounts of TEA, both at room temperature and at elevated temperatures. The reaction of α-oxides **1**—**5** with TEA could be carried out only with a greater amount of TEA under drastic conditions (sealed tubes, 130—140 °C). As a result, 3-hydropolyfluoro-3-alkanols (**6a**—**e**) and 2-hydropolyfluoro-2-alkanols (**7a**—**e**) were isolated (complete conversion of internal polyfluoroepoxides was observed with an excess of TEA, Scheme 1).

Mechanisms for the reaction of terminal fluoroolefin oxides with TEA have been suggested.[2,5] It is believed[5] that the reaction occurs as nucleophilic substitution of a fluorine atom at the less shielded (terminal) C atom followed by opening of the oxide ring by the fluoride ion formed. However, in the case of internal α-oxides **1**—**5**, this reaction mechanism should result in trifluoromethyl ketones (**8a**—**e**, pathway *b*) and, eventually, give secondary alcohols containing a trifluoromethyl substituent, **7a**—**e**. The other mechanism of the reaction of terminal fluoroepoxides with TEA[2] conforms better to our results (Table 1). According to this mechanism, TEA attacks hexafluoropropylene oxide at the central C atom, and the resulting betaine undergoes rearrangement to give an acyl fluoride.

Based on our data and data from the literature,[2] the scheme of the reaction of 2,3-epoxypolyfluoroalkanes **1**—**5** with TEA can be represented as follows (see Scheme 1).

Initially, TEA attacks epoxides at the C(2) and C(3) atoms (preferentially, at the less shielded C(2) atom (pathway *a*) in the case of compounds **1**, **2**, **4**, and **5**); this is followed by ring-opening and the formation of pentafluoroethyl (**9a**—**e**) and trifluoromethyl (**8a**—**e**) polyfluoroalkyl ketones. The latter, like acyl fluorides,[6] react with TEA to give adducts (**10a**—**e**, **11a**—**e**) that dissociate to give radical ion complexes. The radical cation of TEA reduces ketones **8a**—**e** and **9a**—**e** to alcohols **6a**—**e**, **7a**—**e** and is transformed to vinyldiethylamine, which is unstable[7] and, obviously, polymerizes under the reaction conditions.

Table 1 presents the molar ratios of the products formed in the reactions of oxides **1**—**5** in the presence of a 1.5-fold excess of TEA, the overall yields, the boiling points of the mixtures, and IR spectral data.

The structures of the compounds obtained, **6a**—**e** and **7a**—**e**, were confirmed by ^{19}F and ^{1}H NMR and IR spectroscopy (Tables 1—4). Alcohols **6a** and **7a**—**d** were obtained in the individual state by independent syntheses from polyfluoro-2-alkanones **9a** and **8a**—**d** (see Tables 2—4) (Scheme 2).

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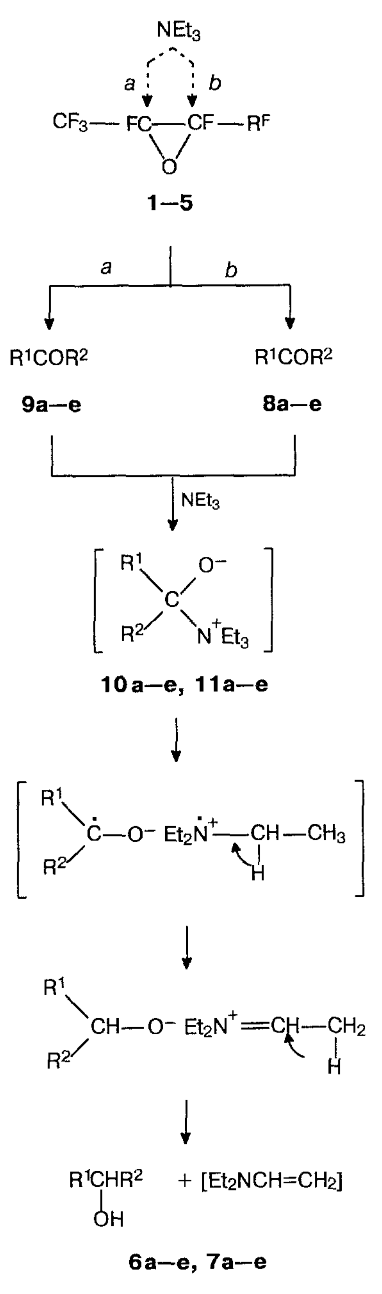
Scheme 1



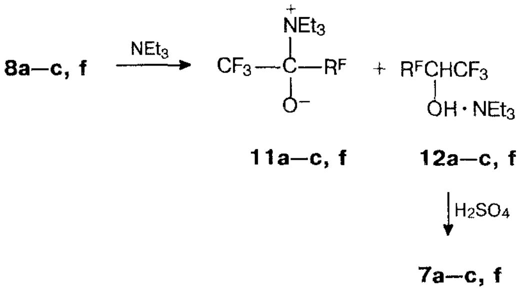
6a, 9a, 10a: $R^1 = C_3F_7$, $R^2 = C_2F_5$
6b, 9b, 10b: $R^1 = C_5F_{11}$, $R^2 = C_2F_5$
6c, 9c, 10c: $R^1 = H(CF_2)_3$, $R^2 = C_2F_5$
6d, 9d, 10d: $R^1 = H(CF_2)_5$, $R^2 = C_2F_5$
6e, 9e, 10e: $R^1 = H(CF_2)_7$, $R^2 = C_2F_5$
7a, 8a, 11a: $R^1 = C_4F_9$, $R^2 = CF_3$
7b, 8b, 11b: $R^1 = C_6F_{13}$, $R^2 = CF_3$
7c, 8c, 11c: $R^1 = H(CF_2)_4$, $R^2 = CF_3$
7d, 8d, 11d: $R^1 = H(CF_2)_6$, $R^2 = CF_3$
7e, 8e, 11e: $R^1 = H(CF_2)_8$, $R^2 = CF_3$

Scheme 2

$$\mathbf{9a,\ 8a–d} \xrightarrow[Et_2O]{LiAlH_4} \mathbf{6a,\ 7a–d}$$

The reaction mechanism considered is consistent with the fact that compounds **1—5** do not react with catalytic amounts of TEA, which is consumed in the reduction of the intermediate ketones **9a—e** and **8a—e**. This mechanism is also supported by reactions of compounds **8a—c** and decafluoro-2-pentanone with TEA (Scheme 3).

Scheme 3



7a, 8a, 11a, 12a: $R^F = C_4F_9$
7b, 8b, 11b, 12b: $R^F = C_6F_{11}$
7c, 8c, 11c, 12c: $R^F = H(CF_2)_4$
7f, 8f, 11f, 12f: $R^F = C_3F_7$

It is noteworthy that IR and ^{19}F NMR spectroscopy showed that, along with the triethylammonium salts of alcohols **12a—c,f**, the reaction mixtures produced by the exothermic reactions of ketones **8a—c,f** with excess TEA contain adducts **11a—c,f**, which, when the reaction mixture is heated slightly, are transformed to alcohols **7a—c,f**, again in the form of triethylammonium salts **12a—c,f** (see Scheme 3).

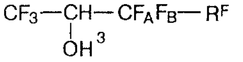
The IR spectra of the reaction mixtures produced from compounds **8a—c,f** and TEA no longer contain the band of the ketone carbonyl group ν(C=O) at 1780 cm^{-1}; instead, intense absorption appears at 1640—1645 cm^{-1} (Table 5), which is characteristic of carboxylate ions[8,9] (the alkoxy anion of triethylammonium salts **12a—c,f** manifests itself in the IR spectra as an absorption band of lower intensity in the 1670—1700 cm^{-1} region).

The ^{19}F NMR spectra (acetone-d_6, C_6F_6, δ) of compounds **11a—c,f** contain signals of the CF_3 groups (87.1—87.2, 82.7—82.9) and CF_2 groups (36.9—46.8). The fluorine atoms of the β-CF_2 groups are not equivalent and appear as AB systems. The signals of an adduct were observed most distinctly in the ^{19}F NMR spectrum of **11f**: $C(1)F_3(O^-)C(N^+Et_3)–C(3)F_2CF_AF_BC(2)F_3$: 87.1 (tt, $C(1)F_3$, J = 12.8 Hz, 3.7 Hz); 82.9 (t, $C(2)F_3$,

Table 1. Composition and physicochemical parameters of the products of the reaction of 2,3-epoxypolyfluoroalkanes **1—5** with TEA at the molar ratio α-oxide : TEA = 1 : 1.5

Starting α-oxide	Composition of reaction products (molar ratio)	Overall yield (%)	B.p. of the mixture/ °C	IR, ν(OH)/ cm^{-1}
1	**6a, 7a** (62 : 38)	32	84—91	3450, 3600, 3640
2	**6b, 7b** (68 : 32)	36	125—137	3450, 3600, 3630
3	**6c, 7c** (45 : 55)	31	118—122	3360, 3600,
4	**6d, 7d** (61 : 39)	35	160—168	3400, 3610, 3640
5	**6e, 7e** (71 : 29)	30	182—186	3350, 3500, 3600, 3630

Table 2. ^{19}F and ^{1}H NMR spectra of 2-hydrodecafluoro-2-pentanol **7f**, 2-hydrododecafluoro-2-hexanol **7a**, 2-hydrohexadecafluoro-2-octanol **7b**, 2,6-dihydroundecafluoro-2-hexanol **7c**, 2,8-dihydropentadecafluoro-2-octanol **7d**, and 2,10-dihydroheptadecafluoro-2-decanol **7e**



$\overset{1}{CF_3}—\underset{OH^3}{\overset{2}{CH}}—\overset{4}{CF_AF_B}—R^F$

Compound	R^F	NMR, δ	J/Hz
7f	5 6 $CF_AF_BCF_3$	89.9 (dddt, F-1); 44.9 (m, F$_A$-4); 37.9 (m, F$_B$-4); 39.3 (dm, F$_A$-5); 36.6 (dm, F$_B$-5); 82.8 (dd, F-6); 4.81 (ddq, H-2); 6.62 (s, H-3)	$J_{6,4B} = J_{4B,5A} = 8.5$, $J_{6,4A} = 12.2$, $J_{1,2} = J_{4A,5B} = 7.3$, $J_{1,4A} = 14.6$, $J_{2,4B} = 18.1$, $J_{1,5} = 2.4$, $J_{1,4B} = 9.8$, $J_{4A,4B} = 285.3$, $J_{5A,5B} = 291.1$
7a	5 6 7 $CF_2CF_2CF_3$	90.3 (dddt, F-1); 44.6 m (F$_A$-4); 38.4 (m, F$_B$-4); 41.7 (m, F-5); 38.7 (m, F-6); 82.8 (tt, F-7); 4.66 (ddq, H-2); 6.68 (s, H-3)	$J_{1,2} = 6.7$, $J_{1,4B} = 8.5$, $J_{1,4A} = 13.4$, $J_{1,5} = 2.4$, $J_{2,4B} = 17.6$, $J_{2,4A} = 6.3$, $J_{6,7} = 2.6$, $J_{5,7} = 10.2$, $J_{4A,4B} = 286.3$
7b	5 6 7 8 9 $CF_2CF_2CF_2CF_2CF_3$	90.0 (dddt, F-1); 45.3 (m, F$_A$-4); 39.0 (m, F$_B$-4); 42.7 (m, F-5, F-6); 41.7 (m, F-7); 38.2 (m, F-8); 82.9 (tt, F-9); 4.93 (ddq, H-2); 6.27 (s, H-3)	$J_{1,2} = 7.3$, $J_{1,4A} = 14.6$, $J_{1,4B} = 9.8$, $J_{1,5} = 2.3$, $J_{2,4A} = 6.3$, $J_{2,4B} = 18.3$, $J_{8,9} = 2.4$, $J_{7,9} = 10.4$, $J_{4A,4B} = 290.5$
7c	5 6 7 8 $CF_2CF_2CF_2H$	90.1 (dddt, F-1); 44.5 (q, F$_A$-4); 38.1 (m, F$_B$-4); 39.9 (m, F-5); 33.9 (m, F-6); 26.1 (dm, F-7); 4.85 (ddq, H-2); 6.85 (s, H-3); 6.40 (tt, F-8)	$J_{1,2} = 7.3$, $J_{1,2A} = 14.6$, $J_{1,2B} = 9.8$, $J_{1,3} = 2.4$, $J_{2,4A} = 6.1$, $J_{2,4B} = 15.2$, $J_{6,8} = 5.5$, $J_{7,8} = 52.2$, $J_{4A,4B} = 286.2$
7d	5 6 7 8 9 10 $CF_2CF_2CF_2CF_2CF_2H$	90.1 (ddd, F-1); 45.0 (m, F$_A$-4); 38.7 (m, F$_B$-4); 42.5 (m, F-5, F-6); 40.9 (m, F-7); 34.4 (m, F-8); 26.1 (dm, F-9); 4.79 (ddq, H-2); 6.72 (s, H-3); 6.37 (tt, H-10)	$J_{1,2} = 6.8$, $J_{2,4A} = 6.1$, $J_{1,2B} = 8.8$, $J_{1,5} = 2.0$, $J_{2,4B} = 18.3$, $J_{1,4A} = 14.6$, $J_{8,10} = 5.2$, $J_{9,10} = 51.2$, $J_{4A,4B} = 290.0$
7e	5 6 7 8 9 10 11 12 $CF_2CF_2CF_2CF_2CF_2CF_2CF_2H$	90.2 (ddd, F-1); 45.6 (m, F$_A$-4); 38.6 (m, F$_B$-4); 42.6 (m, F-5—F-7); 41.0 (m, F-8, F-9); 34.7 (m, F-10); 25.9 (dm, F-11); 5.13 (ddq, H-2); 7.77 (s, H-3); 6.75 (tt, H-12)	$J_{1,2} = 7.3$, $J_{1,4A} = 14.6$, $J_{1,4B} = 8.5$, $J_{4A,2} = 6.1$

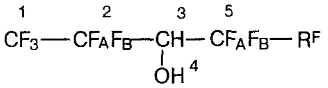
$J = 11.0$ Hz); 45.7 (qq, $J = 12.8$ Hz, 11.0 Hz); 40.5 (m, F$_A$); 42.8 (m, F$_B$).

It follows from the data in Table 1 that the opening of the epoxide ring of compounds **1**, **2**, **4**, **5** on treatment with TEA occurs in both possible directions, preferably, from the side of the less bulky trifluoromethyl group (pathway *a*). This is in accordance with the data reported in Ref. 10 concerning the reaction of 2,3-epoxyperfluoroalkanes with other nucleophiles, in particular, with the fluoride ion. However, in the case of 2,3-epoxy-6-hydroundecafluorohexane **3** the fraction of isomeric alcohol **7c** (pathway *b*) increases to 55 % (accordingly, the fraction of alcohol **6c**, pathway *a*, decreases). Probably, this is due to the presence of an

Table 3. Physicochemical characteristics of polyfluorinated secondary alcohols **6a**, **7a—d**

Compound	Yield (%)	B.p. /°C	IR, ν(OH)/ cm^{-1}	Found (%) / Calculated C	H	F	Molecular formula
6a	82	98—99	3450, 3600, 3640	22.46 22.64	0.78 0.63	71.48 71.70	$C_6H_2F_{12}O$
7a	83	105—106	3450, 3610, 3650	22.92 22.64	0.56 0.63	71.56 71.70	$C_6H_2F_{12}O$
7b	76	139—140	3400, 3600, 3630	23.08 22.97	0.58 0.48	72.86 72.73	$C_8H_2F_{16}O$
7c	78	131—132	3480, 3605, 3640	23.85 24.00	1.31 1.00	69.62 69.67	$C_6H_3F_{11}O$
7d	80	160—162	3400, 3600, 3640	24.12 24.00	0.86 0.75	71.38 71.25	$C_8H_3F_{15}O$

Table 4. ^{19}F and ^{1}H NMR spectra of 3-hydrododecafluoro-3-hexanol **6a**, 3-hydrohexadecafluoro-3-octanol **6b**, 3,6-dihydroundecafluoro-3-hexanol **6c**, 3,8-dihydrotetradecafluoro-3-octanol **6d**, and 3,10-dihydroheptadecafluoro-3-decanol **6e**


$\overset{1}{CF_3}—\overset{2}{CF_AF_B}—\overset{3}{\underset{OH^4}{CH}}—\overset{5}{CF_AF_B}—R^F$

Compound	R^F	NMR, δ	J/Hz
6a	6 7 $CF_AF_BCF_3$	81.9 (t, F-1); 45.2 (m, F_A-2); 35.8 (dd, F_B-2); 45.6 (m, F_A-5); 39.6 (tq, F_B-5); 39.5 (m, F_A-6); 37.2 (m, F_B-6); 83.2 (dd, F-7); 5.11 (tt, H-3); 7.01 (s, H-4)	$J_{1,5}$ = 2.9, $J_{3,2B}$ = $J_{3,5B}$ = $J_{2B,5B}$ = 17.6, $J_{7,5B}$ = 8.8, $J_{7,5A}$ = 11.7, $J_{3,5A}$ = $J_{3,2A}$ = 5.0, $J_{2A,2B}$ = 278, $J_{5A,5B}$ = 289, $J_{6A,6B}$ = 292
6b	6 7 8 9 $CF_2CF_2CF_2CF_3$	81.7 (t, F-1); 45.2 (m, F_A-2); 35.1 (m, F_B-2); 46.0 (m, F_A-5); 39.3 (m, F_B-5); 38.1 (m, F-6); 41.7 (m, F-7); 42.7 (m, F-8); 82.8 (tt, F-9); 4.93 (tt, H-3); 6.59 (s, H-4)	$J_{1,5}$ = $J_{8,9}$ = 2.4, $J_{3,2A}$ = $J_{3,5A}$ = 4.3, $J_{3,2B}$ = $J_{3,5B}$ = 18.5, $J_{7,9}$ = 10.4, $J_{2A,2B}$ = 277, $J_{5A,5B}$ = 290.0
6c	6 7 8 $CF_AF_BCF_2H$	81.6 (t, F-1); 43.6 (dd, F_A-2); 36.1 (m, F_B-2); 45.0 (m, F_A-5); 38.1 (m, F_B-5); 34.5 (m, F_A-6); 30.8 (m, F_B-6); 26.6 (dm, F-7); 4.85 (tt, H-3); 6.85 (s, H-4); 6.45 (tt, H-8)	$J_{1,5}$ = 3.0, $J_{3,2B}$ = $J_{3,5B}$ = 18.5, $J_{3,2A}$ = $J_{3,5A}$ = 6.5, $J_{6,7}$ = 6.1, $J_{6,8}$ = 4.6, $J_{7,8}$ = 51.9, $J_{2A,2B}$ = 276, $J_{5A,5B}$ = 285.6, $J_{6A,6B}$ = 291.7
6d	6 7 8 9 10 $CF_2CF_2CF_2CF_2H$	81.9 (t, F-1); 45.3 (m, F_A-2); 35.7 (m, F_B-2); 45.0 (m, F_A-5); 39.7 (m, F_B-5); 42.5 (m, F-6); 40.9 (m, F-7); 34.4 (m, F-8); 26.1 (dm, F-9); 5.18 (tt, H-3); 7.29 (s, H-4); 6.79 (tt, H-10)	$J_{1,5}$ = 2.4, $J_{3,2A}$ = $J_{3,5A}$ = 5.0, $J_{3,2B}$ = $J_{3,5B}$ = $J_{2B,5B}$ =17.8, $J_{9,10}$ = 51.0, $J_{8,10}$ = 5.3, $J_{2A,2B}$ = 280.0, $J_{5A,5B}$ = 289.0
6e	6 7 8 9 10 11 12 $CF_2CF_2CF_2CF_2CF_2CF_2H$	81.9 (t, F-1); 45.1 (m, F_A-2); 35.4 (m, F_B-2); 45.5 (m, F_A-5); 39.4 (m, F_B-5); 42.6 (m, F-6—F-8); 41.0 (m, F-9); 34.7 (m, F-10); 25.9 (dm, F-11); 5.13 (tt, H-3); 7.77 (s, H-4); 6.78 (tt, H-12)	$J_{1,5}$ = 2.0, $J_{3,2B}$ = $J_{3,5B}$ = 17.5, $J_{11,12}$ = 51.0, $J_{2A,2B}$ = 277.5, $J_{5A,5B}$. = 290.0

intramolecular hydrogen bond and displacement of the electron density to the C(2) atom of the cycle in compound **3**.[11]

Thus, two consecutive reactions occur at elevated temperatures in the presence of excess TEA: rearrangement of α-oxides into isomeric ketones, which, like

Table 5. Characteristics of products obtained in the reactions of ketones **8a—c,f** with NEt_3 at a molar ratio ketone : TEA = 1 : 1.5

Starting ketone	Reaction products (molar ratio (%))	IR spectrum of the mixture of reaction products, ν/cm^{-1}	
		C—O$^-$	N^+HEt_3
8f	**12f, 11f** (45 : 55)	1645	2200—2750
8a	**12a, 11a** (51 : 49)	1640	2180—2770
8b	**12b, 11b** (51 : 49)	1640	2250—2760
8c	**12c, 11c** (62 : 38)	1640	2170—2770

hexafluoroacetone,[12] are then reduced by treatment with TEA to give secondary alcohols (in this case, the epoxides themselves are not hydrogen acceptors, unlike in their reactions with complex metal hydrides[13]).

Experimental

^{19}F NMR spectra were obtained on a Tesla BS-587A spectrometer (75.3 MHz) and ^1H NMR spectra were recorded on a Tesla BS-567A instrument (100 MHz) in acetone-d_6 using C_6F_6 and Me_4Si as the internal standards. IR spectra of the samples were recorded in thin layers on a Specord IR-75 spectrophotometer. GLC analyses were carried out on a LKhM-72 chromatograph using a catharometer as the detector, helium as the carrier gas, and a steel column (6000×4 mm) with 5 % SKTFT-100 on a Chromosorb G support.

Compounds **8a—d** and **9a** were obtained by a known procedure.[14]

The ratio of the reaction products was determined by comparing the integral intensities of their signals in the ^{19}F NMR spectra.

The reaction of α-oxides 1—5 with TEA. An α-oxide (0.02 mol) and TEA (0.03 mol) were placed in a Pyrex tube, which was sealed and kept for 16 h at 130—140 °C with intermittent shaking in a bath filled with Wood's alloy. The tube was then cooled and opened, 10 % HCl (10 mL) was added, and the liquid products were distilled off. The lower layer was separated, dried with $MgSO_4$, and distilled once more, and then the IR and ^{19}F and ^1H NMR spectra were recorded (see Tables 1—4).

Polyfluoroalkanols 6a, 7a—d. Ketone **9a, 8a—d** (0.06 mol) was added dropwise with stirring to a suspension of $LiAlH_4$ (0.06 mol) in dry Et_2O. The reaction mixture was stirred for an additional 0.5 h and decomposed with 10 % HCl. The ethereal layer was separated and the aqueous layer was extracted with ether. The combined extract was dried with $MgSO_4$, the ether was distilled off, and the residue was distilled from concentrated H_2SO_4. The yields, boiling points, elemental analysis data, and IR spectra of alcohols **6a, 7a—d** are presented in Table 3. The ^{19}F and ^1H NMR spectra are given in Tables 2 and 4.

The reaction of ketones 8a—c,f with TEA. Ketone **8a—c,f** was placed in a flask, then TEA was added dropwise with stirring (molar ratio ketone : TEA = 1 : 1.5), which caused an exothermic reaction. The reaction mixture was stirred for an additional 10 min, then IR and ^{19}F and ^1H NMR spectra were recorded (see Table 5).

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