ELSEVIER

Journal of Fluorine Chemistry 74 (1995) 167–170

JOURNAL OF FLUORINE CHEMISTRY

Synthesis and reaction of β,β-di(trifluoroacetyl)ethylene derivatives, $(CF_3CO)_2C{=}CR_1R_2$

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Received 25 August 1994; accepted 14 May 1995

Abstract

Reaction of 1,1,1,5,5,5-hexafluoro-2,4-pentanedione with aromatic aldehydes, alkyl formate and *N,N*-dialkyl amides in acetic acid anhydride gave β,β-di(trifluoroacetyl)ethylene derivatives, $(CF_3CO)_2C{=}CR_1R_2$, which reacted readily with some nucleophiles containing reactive hydrogen such as ArSH, $HP(O)(OR)_2$ giving the corresponding addition product. When treated with 3,4-dihydro-2*H*-pyran, a cycloaddition product was formed.

Keywords: Condensation; Hexafluoro-2,4-pentanedione; Carbonyl compounds; Di(trifluoroacetyl)ethylene derivatives; Nucleophilic addition; NMR/IR spectroscopy

1. Introduction

In recent years much research has been carried out on the reactions of 1,1,1,5,5,5-hexafluoro-2,4-pentanedione, $CF_3COCH_2COCF_3$ (**1**), which is now commercially available. In this work, compound **1** was mainly used to prepare heterocycles containing the trifluoroacetyl or trifluoromethyl group by the reactions with hydrazine, aminothiol, aminopyrazol and some aniline derivatives [1–6]. However, other chemical transformations of **1** have rarely been studied. Recently, Pashkevich et al. reported that treatment of **1** with aldehydes under basic conditions gave α,β-unsaturated ketones [7], thus:

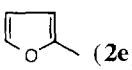
$$CF_3COCH_2COCF_3 + RCHO \xrightarrow{B} CF_3COCH{=}CHR$$

In this paper, we wish to report the condensation reactions of **1** with some carbonyl compounds such as ArCHO, HCOOR and $RCONR_2$. From these reactions, β,β-di(trifluoroacetyl)ethylene derivatives, $(CF_3CO)_2C{=}CR_1R_2$ (**3**), were prepared. The polar carbon–carbon double bond caused by the electron-withdrawing group CF_3CO– was easily saturated by some nucleophiles containing reactive hydrogen, NuH.

2. Results and discussion

In our previous work, 1-aryl-2,2-di(perfluoroalkanesulfonyl)alkenes, $(R_FSO_2)_2C{=}CHAr$, were prepared from the reactions of $(R_FSO_2)_2CH_2$ with the corresponding aromatic aldehydes [8,9]. As an extension of this work, we found that this synthetic method can also be applied to prepare 1-aryl-2,2-di(trifluoroacetyl)alkenes, $(CF_3CO)_2C{=}CHAr$ (**3**). Heating a 1:1 mixture of **1** and the aromatic aldehydes in Ac_2O gave **3** in good yield:

$$\underset{(\mathbf{2})}{\mathbf{1} + ArCHO} \xrightarrow[75\%\text{–}87\%]{Ac_2O,\ 80\ ^\circ C,\ 8\ h} \underset{(\mathbf{3a\text{–}e})}{(CF_3CO)_2C{=}CHAr}$$

Ar = C_6H_5 (**2a**); 4-$CH_3C_6H_4$ (**2b**); 4-$CH_3OC_6H_4$ (**2c**); 4-ClC_6H_4 (**2d**);  (**2e**)

The products **3** are yellowish liquids which are purified by vacuum distillation. The structure of all these new compounds are fully supported by their spectroscopic data and microanalyses. The ^{19}F NMR and ^{13}C NMR spectra indicate that the two trifluoroacetyl groups in compounds **3** are chemically unequal. For example, the ^{19}F NMR and ^{13}C NMR of compound **3c** are δ_F −5.5; 0.0 ppm (TFA as an external standard and upfield as positive); δ_C 186.8 (q); 178.8 (q); 116.6 (q); 115.6 (q) ppm for two different CF_3CO groups. They are chemically nonequivalent: one is *trans* to hydrogen and the other is *trans* to the aryl group. The large chemical shifts between the two carbonyl carbons ($\Delta\delta = 8$ ppm) may be due to the carbons *cis* to the aryl group lying in the shielding cone of the aromatic ring. The CF_3CO group, which is on the same side as the hydrogen atom, has upfield chemical shifts (i.e.

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SSDI 0022-1139(95)03296-7

δ_F −76.8 ppm; δ_c 178.8; 115.6 ppm). Recently, the hydrocarbon analogues $Y_2C{=}CHAr$ [Y = RCO, ROC(O)] have been prepared by a two-step reaction [10]. In our case, compounds **3** are obtained more conveniently.

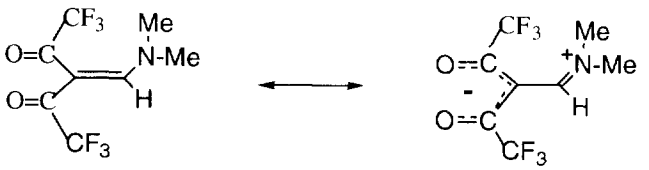
Extension of this reaction to other carbonyl compounds such as *N,N*-dialkyl amides and diethyl formate $HCONR_2$, CH_3CONR_2, HCOOEt are successful. Under the same reaction conditions, several *β,β*-di(trifluoroacetyl)ethylene derivatives are prepared:

$$\underset{(\mathbf{4})}{1 + R\text{–}C(O)\text{–}Y} \xrightarrow[64\%\text{–}78\%]{Ac_2O,\ 80\ ^\circ C,\ 8\ h} \underset{(\mathbf{5a\text{–}d})}{(CF_3CO)_2C{=}CRY}$$

R = H, Y = NMe_2 (**4a**); R = H, Y = NEt_2 (**4b**); R = CH_3, Y = NMe_2 (**4c**); R = H, Y = OEt (**4d**)

The compounds **5b** and **5d** were first reported by Schreiber [11] and Hojo et al. [12]. They were synthesized by treatment of $(CF_3CO)_2O$ with Et_3N and $EtOCH{=}CH_2$, respectively.

In contrast to compound **3**, the NMR spectra of **5** show that the two CF_3CO groups are apparently equivalent due to the electron donation of the $-NR_2$ or –OR group:



A **B**

The true nature of the bonding and the *π*-electron distribution is probably closer to canonical form **B** than it is to canonical form **A**. This phenomenon is known in other 'push-pull' alkenes, although the present case shows the effect to an extreme extent.

The ^{13}C NMR spectra of compounds **3** and **5** appear to support this. For example, the chemical shift of $(CF_3CO)_2C{=}$ in compound **3c** is 115.6 ppm and it is only 101.0 ppm in compound **5a**.

Attempts to prepare di(trifluoroacetyl)ketene dimethyl acetal, $(CF_3CO)_2C{=}C(OMe)_2$, by similar treatment of **1** with $(MeO)_2C{=}O$ failed.

All these results are summarized in Table 1.

Recently we found that the dialkyl phosphite $HP(O)(OR)_2$ (**6**) is readily added to polar double bonds. For example, both $(R_FSO_2)_2C{=}CHAr$ and $R_FSO_2N{=}CHAr$ reacted smoothly with **6** at room temperature. Similarly, treatment of **6** with **3** gave an addition product in high yield. Thiophenol reacted with **3** in the same way:

$$\underset{(\mathbf{3c})}{(CF_3CO)_2C{=}CHAr} + NuH \xrightarrow{CH_3CN} (CF_3CO)_2CHCHAr\text{-}Nu$$

Ar = 4-$MeOC_6H_4$; Nu = $P(O)(OCH_3)_2$ (**7**), C_6H_5S (**8**)

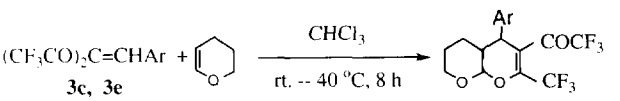
Table 1
Preparations of compounds **3** and **5**

Entry No.	Reactant **2** or **4**	Product **3** or **5**	B.p. (°C/Torr)	Yield (%) [a]
1	**2a**	**3a**	78/2	76
2	**2b**	**3b**	94/2	80
3	**2c**	**3c**	120/2	85
4	**2d**	**3d**	98–100/2	75
5	**2e**	**3e**	58–60/2	87
6	**4a**	**5a**	80/3	72
7	**4b**	**5b** [b]	40–42/3	64
8	**4c**	**5c**	83/3	78
9	**4d**	**5d** [b]	61–63/3	70

[a] Isolated yield based on **1**.
[b] Known compounds; cf. Refs. [11] and [12].

However, heating **3** with phenol or ethanol did not give the corresponding nucleophilic addition products. When **3** were treated with the alcohol containing some water, the hydrolysis products $(CF_3CO)_2CH_2$ and ArCHO were formed readily.

Diels–Alder reactions between 2-aryl-1-(perfluoroalkanesulfonyl) acrylonitriles, $R_FSO_2C(CN){=}CHAr$, and dienes have been reported by Hanack et al. [13]. Astonishingly, in our case push-pull dienophiles such as **3b**, **3c** and **5** did not react with cyclodienes or isopentadiene. However, compound **3** reacted readily with 3,4-dihydro-2*H*-pyran giving a bicyclic addition product:



3c, **3e**

Ar: 4 - $CH_3OC_6H_4$ (**9**)

(**10**)

It is obvious that in this reaction 3,4-dihydro-2*H*-pyran acted as a dienophile [12]. Other alkenes such as cyclopentene or cyclohexene failed to form similar cycloaddition products.

A complete study on the condensation reaction of **1** with carbonyl compounds and the use of these condensation products is under investigation.

In summary, we have synthesized a new and reactive class of ethylene derivatives, their polar C=C double bonds being subject to nucleophilic addition.

3. Experimental details

Melting points were measured on a Thiele apparatus and all were uncorrected. Boiling points were uncorrected. ^1H NMR and ^{19}F NMR spectra were recorded on a Varian 360L instrument with Me_4Si and TFA as an internal and external standard, respectively, and the ^{19}F NMR spectra were converted to δ_{CFCl_3}. ^{13}C NMR and ^{31}P NMR spectra were recorded on a Bruker AM-300 instrument with TMS and H_3PO_4 (85%) as external standards, respectively. IR spectra

were obtained with an IR-440 Shimadizu spectrophotometer. Low-resolution mass spectra were obtained on a Finnigan GC–MS 4021 instrument. Elemental analyses were performed by this Institute. 1,1,1,5,5,5-Hexafluoro-2,4-pentanedione was commercially available from PCR Co. Other reagents and solvents were dried before use.

3.1. General procedure for the preparation of 3

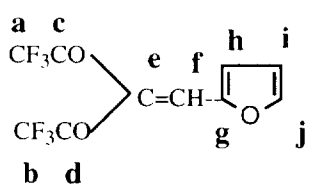
A mixture of **1** (2.08 g, 10 mmol), **2b** (1.20 g, 10 mmol) and acetic acid anhydride (10 ml) in a 25 ml flask, equipped with a reflex condenser, drying tube and magnetic stirring bar, was heated at 80 °C for 8 h. Ac_2O and the AcOH formed were removed by distillation, the residue being distilled under vacuum to give $(CF_3CO)_2C{=}CHC_6H_4CH_3$-4 (**3b**) (2.48 g, 80%).

$(CF_3CO)_2C{=}CHC_6H_5$ (**3a**): IR (film) ν_{max} (cm^{-1}): 1754, 1693 (C=O); 1600 (C=C). ^1H NMR δ: 7.40 (s, C=CH); 6.51–7.15 (ArH, 5H) ppm. ^{19}F NMR δ: −71.3 (s, CF_3); −76.8 (s, CF_3) ppm. MS (*m/z*, %): 297 (3.09, M^+H); 296 (24.20, M^+); 69 (100.00, CF_3^+). Analysis: Calc. for $C_{12}H_6O_2F_6$: C, 48.65; H, 2.03; F, 38.51%. Found: C, 48.78; H, 2.15; F, 38.62%.

$(CF_3CO)_2C{=}CHC_6H_4CH_3$-4 (**3b**): IR (film) ν_{max} (cm^{-1}): 1750, 1690 (C=O); 1600, 1570 (C=C). ^1H NMR δ: 7.46 (s, C=CH); 6.43–7.03 (AA′BB′, 4H); 1.73 (s, CH_3) ppm. ^{19}F NMR δ: −71.3 (s, CF_3); −76.8 (s, CF_3) ppm. MS (*m/z*, %): 310 (41.38, M^+); 119 (100.00, 4-$CH_3C_6H_4CO^+$). Analysis: Calc. for $C_{13}H_8O_2F_6$: C, 50.32; H, 2.58; F, 36.77%. Found: C, 50.11; H, 2.85; F, 36.90%.

$(CF_3CO)_2C{=}CHC_6H_4OCH_3$-4 (**3c**): IR (film) ν_{max} (cm^{-1}): 1750, 1690 (C=O); 1590, 1560 (C=C). ^1H NMR δ: 7.43 (s, C=CH); 6.72–6.15 (AA′BB′, 4H); 3.16 (s, OCH_3) ppm. ^{19}F NMR δ: −71.3 (s, CF_3); −76.8 (s, CF_3) ppm. ^{13}C NMR δ: 55.9 (OCH_3); 165.1 (C=*C*H); 115.6 [$(CF_3CO)_2C$]; 124.0, 125.5, 134.3, 152.2 (C_6H_4); 178.8 [CF_3C(O)C=CH]; 186.8 [CF_3C(O)C=CH] ppm; $^2J_{C-F}$ = 40.5, 116.6 (CF_3), 115.6 (CF_3); $^1J_{C-F}$ = 292 Hz. MS (*m/z*, %): 327 (6.02, M^+H); 326 (37.60, M^+); 257 (100.00, $M^+ - CF_3$). Analysis: Calc. for $C_{13}H_8O_3F_6$: C, 47.85; H, 2.45; F, 34.97%. Found: C, 47.74; H, 2.52; F, 35.03%.

$(CF_3CO)_2C{=}CHC_6H_4Cl$-4 (**3d**): IR (film) ν_{max} (cm^{-1}): 1750, 1690 (C=O); 1600, 1575 (C=C). ^1H NMR δ: 7.40 (s, C=CH); 6.50–7.13 (ArH, 4H) ppm. ^{19}F NMR δ: −71.2 (s, CF_3); −76.8 (s, CF_3) ppm. MS (*m/z*, %): 332/330 (13.52/40.60, M^+); 263/261 (33.34/100.00, $M^+ - CF_3$). Analysis: Calc. for $C_{12}H_5ClO_2F_6$: C, 43.57; H, 1.51; F, 34.49%. Found: C, 43.65; H, 1.65; F, 34.64%.



(**3c**): IR (film) ν_{max} (cm^{-1}) 1750, 1690 (C=O); 1600 (C=C). ^1H NMR δ: 7.30 (s, C=CH); 7.13 (s, 1H); 6.67 (d, 1H); 6.10 (d, 1H) ppm. ^{19}F NMR δ: −71.3 (s, CF_3); −76.8 (s, CF_3) ppm. ^{13}C NMR δ: 185.04 (q, *d*); 178.30 (q, *c*, $^2J_{C-F}$ = 40.5 Hz); 150.72 (s, *f*); 148.23 (s, *g*); 134.78 (s, *j*); 126.14 (s, *i*); 123.42 (s, *e*); 115.29 (s, *h*); 116.74 (q, *b*); 115.83 (q, *a*, $^1J_{C-F}$ = 292.5 Hz) ppm. MS (*m/z*, %): 286 (34.85, M^+); 217 (100.00, $M^+ - CF_3$). Analysis: Calc. for $C_{10}H_4O_3F_6$: C, 41.96; H, 1.40; F, 39.86%. Found: C, 42.11; H, 1.55; F, 40.01%.

Similar treatment of **1** with **4(a–d)** gave **5(a–d)** respectively. Yields and boiling points are shown in Table 1. Compounds **5b** and **5d** are known compounds. Their NMR spectra are confirmed by the literature values [10,11].

$(CF_3CO)_2C{=}CHNMe_2$ (**5a**): IR (film) ν_{max} (cm^{-1}): 1770, 1690 (C=O); 1660, 1595 (C=C). ^1H NMR δ: 7.63 (s, C=CH); 3.33 (s, CH_3); 2.93 (s, CH_3) ppm. ^{19}F NMR δ: −72.3 (s, CF_3) ppm. ^{13}C NMR δ: 179.2 (q, CF_3C(O), $^2J_{C-F}$ = 36.0 Hz); 157.8 (s, *C*H=C); 115.3 (q, CF_3, $^1J_{C-F}$ = 292.5 Hz); 101.0 (s, *C*=CH); 48.5 (s, CH_3); 45.3 (s, CH_3) ppm. MS (*m/z*, %): 263 (25.8, M^+); 194 (100.00, $M^+ - CF_3$). Analysis: Calc. for $C_8H_7F_6NO_2$: C, 36.50; H, 2.66; F, 43.35; N, 5.32%. Found: C, 36.78; H, 2.53; F, 43.46; N, 5.26%.

$(CF_3CO)_2C{=}C(CH_3)NMe_2$ (**5c**): IR (film ν_{max} (cm^{-1}): 1765, 1690 (C=O); 1658, 1590 (C=C). ^1H NMR δ: 2.80 (s, CH_3C=); 2.93 (s, CH_3N); 3.50 (s, CH_3N) ppm. ^{19}F NMR δ: −72.5 (s, CF_3) ppm. MS (*m/z*, %): 277 (10.33, M^+); 208 (100.00, $M^+ - CF_3$). Analysis Calc. for $C_9H_9F_6NO_2$: C, 38.99; H, 3.25; F, 41.16; N, 5.05%. Found: C, 40.08; H, 3.12; F, 41.08; N, 5.16%.

3.2. Reactions of 3 with dialkyl phosphite and thiophenol

A mixture of **3c** (1.6 g, 5 mmol), dimethyl phosphite (0.6 g, 5.5 mmol) and dry CH_3CN (5 ml) was stirred at room temperature for 4 h, then at 40 °C for another 2 h. After removing the solvent, the residue was distilled under vacuum to give **7** (2.0 g, 90%), b.p. 142–145 °C/2 Torr.

$(CF_3CO)_2CHCH(Ar)$–$P(O)(OCH_3)_2$ (**7**): IR (film) ν_{max} (cm^{-1}): 1780 (s, C=O); 1590, 1500 (ArH); 1250 (s, P=O); 1200–1110 (s, C–F); 1020 (m, P–O–C). ^1H NMR δ: 7.66 (AA′BB′, 2 ArH, $^2J_{HH}$ = 9 Hz); 7.06 (AA′BB′, 2 ArH); 6.02 [broad, $(CF_3CO)_2CH$]; 5.02 [d, C*H*P(O), $^2J_{PH}$ = 23 Hz]; 3.70 (d, $POCH_3$, $^2J_{PH}$ = 10 Hz); 3.50 (d, $POCH_3$); 2.67 (s, OCH_3) ppm. ^{19}F NMR δ: −77.1 (s, 2×CF_3) ppm. ^{31}P NMR δ: 11.3 [s, $P(O)(OMe)_2$] ppm. ^{13}C NMR δ: 184.97 (CF_3CO); 161.39, 134.37, 131.75, 131.21 (Ar); 114.92 [$(CF_3CO)_2CH$]; 98.71 (4-CH_3O–C_6H_4CH); 55.01 (4-*C*H_3O–C_6H_4); 42.37, 40.73 [$P(O)(OCH_3)_2$]; 123.61 (q, CF_3, $^2J_{C-F}$ = 40 Hz) ppm. MS (*m/z*, %): 436 (4.2, M^+); 367 (1.7, $M^+ - CF_3$); 339 (11.77, $M^+ - CF_3CO$); 307 (100.00, $M^+ - CF_3CO - 2O$); 293 (5.83, $M^+ - CF_3CO - CH_3 - OCH_3$); 229 (12.5, $M^+ - (CF_3CO)_2CH$); 213 (28.33, $ArCHP(OCH_3)_2^+$); 161 (39.17, $M^+ - CF_3CO - CF_3 - P(O)(OCH_3)_2$); 133 [10.83,

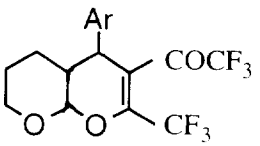
$M^+ - (CF_3CO)_2 - P(O)(OCH_3)_2]$; 69 (4.2, CF_3^+). Analysis: Calc. for $C_{15}H_{15}O_6F_6P$: C, 41.28; H, 3.44%. Found: C, 41.56; H, 3.81%.

Similar treatment of **3c** with C_6H_5SH gave compound **8**. Yield, 75%; m.p. 76 °C.

$(CF_3CO)_2CHCH(Ar)-SC_6H_5$ (**8**): IR (KBr) ν_{max} (cm^{-1}): 1780 (s, C=O); 1598, 1490 (ArH); 1210–1120 (s, C–F). ^1H NMR δ: 6.4–7.3 (ArH, 9H); 6.23 [s, $(CF_3CO)_2CH$]; 5.23 (s, ArC*H*); 2.9 (s, CH_3) ppm. ^{19}F NMR δ: −76.3 (s, $2\times CF_3$) ppm. MS (*m/z*, %): 368 (4.2, M^+H-CF_3); 353 (2.5, $M^+H-CF_3-CH_3$); 339 (2.5, M^+-CF_3CO); 313 (5.5, $M^+H-SC_6H_5-CH_3$); 285 (2.4, $M^+H-SC_6H_5-CH_3-CO$); 264 (2.5, $M^+H-C_6H_4-CF_3CO$); 239 (5.2, $M^+-CF_3CO-CF_3-OCH_3$); 236 (5.0, $M^+H-C_6H_4-CF_3CO-CO$); 185 (4.2, $M^+-(CF_3CO)_2$-CHCH$-OCH_3$); 109 (15.0, $SC_6H_5^+$); 97 (32.5, CF_3CO^+). Analysis: Calc. for $C_{19}H_{14}F_6SO_3$: C, 52.29; H, 3.21; F, 26.15%. Found: C, 52.48; H, 3.12; F, 26.43%.

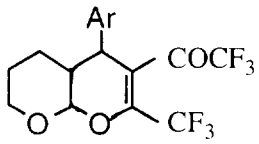
3.3. Cycloaddition reactions of 3 with 3,4-dihydro-2H-pyran

A mixture of **3c** (1.6 g, 5 mmol) and 3,4-dihydro-2*H*-pyran (5 ml) was stirred at room temperature for 24 h. After removing the excess 3,4-dihydro-2*H*-pyran, the residue was distilled under vacuum giving **9** (1.6 g, 77%); b.p. 140–143 °C/2 Torr.



(Ar = 4-$CH_3OC_6H_4$ (**9**): IR (film) ν_{max} (cm^{-1}): 1750 (s, C=O); 1655 (C=C); 1590, 1495 (ArH); 1210–1120 (s, C–F). ^1H NMR δ: 6.83–7.78 (AA′BB′, 4 ArH); 5.34 (s, –O–CH–O); 4.13–4.26 (m, C*H*Ar); 3.63 (s, OCH_2–); 3.00 (s, OCH_3); 1.93–2.40 (broad, 5H) ppm. ^{19}F NMR δ: −74.8 (s, CF_3); −66.8 (s, CF_3) ppm. MS (*m/z*, %): 410 (3.3, M^+); 392 (4.1, M^+H-F); 326 (5.1, $M^+-C_5H_8O$); 307 (2.5, $M^+-C_5H_8O-F$); 295 (6.6, M^+H-CF_3CO-F); 257 (25.6, $M^+-C_5H_8O-CF_3$); 207 (17.4, $M^+H-ArCH-C_5H_8O$), 166 (4.1, $CF_3CO-CF_3^+$); 137 (2.4, $M^+-Ar-CF_3CO-CF_3$); 107 (7.5, $C_6H_4OCH_3^+$); 84 (100.00, $C_5H_8O^+$); 69 (35.3, CF_3^+). Analysis: Calc. for $C_{18}H_{16}F_6O_4$: C, 52.68; H, 3.90; F, 27.80%. Found: C, 52.92; H, 3.68; F, 27.42%.

Treatment of **3e** with 3,4-dihydro-2*H*-pyran gave **10**. Yield, 70%; b.p. 120–125 °C/2 Torr.



Ar :

(**10**): IR (film) ν_{max} (cm^{-1}): 1760 (s, C=O); 1660, 1650 (C=C). ^1H NMR δ: 7.03 (s, 1H); 6.60 (d, 1H); 6.03 (d, 1H); 5.43 (s, 1H, O–CH–O); 5.30 (m, C*H*Ar); 3.60 (m, 2H, OCH_2); 2.43–1.23 (m, 5H) ppm. ^{19}F NMR δ: −75.3 (s, CF_3); −66.5 (s, CF_3) ppm. MS (*m/z*, %): 370 (5.9, M^+); 353 (4.74, M^+-H-O); 352 (28.42, M^+H-F); 296 (15.2, $M^+H-O-O-F-CO$): 286 (17.57, $M^+-C_5H_8O$); 267 (9.5, $M^+-F-C_5H_8O$); 217 (74.41, $M^+-CF_3-C_5H_8O$); 97 (2.8, CF_3CO^+); 84 (100.00, $C_5H_8O^+$); 69 (28.61, CF_3^+); 67 (4.1, $C_4H_8O^+$); 55 (19.8, $C_4H_7^+$). Analysis: Calc. for $C_{15}H_{12}F_6O_4$: C, 48.65; H, 3.24; F, 30.81%. Found: C, 48.43; H, 3.52; F, 30.66%.

Acknowledgement

The authors thank the National Natural Science Foundation of China (NNFSC No. 29472071) for financial support.

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