Radical Cyclization of Fluorinated 1,3-Dicarbonyl Compounds with Dienes Using Manganese(III) Acetate and Synthesis of Fluoroacylated 4,5-Dihydrofurans

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Radical cyclizations of fluorinated 1,3-dicarbonyl compounds with dienes mediated by $Mn(OAc)_3$ afforded 4,5-dihydrofurans containing difluoroacetyl, trifluoroacetyl, or heptafluorobutanoyl groups in good-to-excellent yields. Additionally, 2-(difluoromethyl)-4,5-dihydrofurans and a 4,7-dihydrooxepin derivative were obtained as unexpected products in the reaction of 4,4-difluoro-1-phenylbutane-1,3-dione with 1,3-diphenylbuta-1,3-diene. The radical cyclization of symmetrical dienes such as 2,3-dimethylbuta-1,3-diene and 1,4-diphenylbuta-1,3-diene with 1,3-diketones furnished the corresponding products in low yields. However, treatment of 1-phenylbuta-1,3-diene with 1,3-dicarbonyl compounds afforded 4,5-dihydrofurans containing fluoroacyl groups. The radical cyclizations with 3-methyl-1-phenylbuta-1,3-diene and 1,3-diphenylbuta-1,3-diene led to 4,5-dihydrofurans in good yields, since Me and Ph groups at C(3) of these dienes increase the stability of the radical intermediate.

Introduction. – The use of F-atom and fluoroalkyl groups in the synthesis of organic compounds is developing due to their unique electronic effects and biological properties. Thus, fluorinated organic compounds containing the difluoromethyl and trifluoromethyl groups have received considerable attention in the medicinal and agricultural chemistry [1]. Several difluoromethyl and trifluoromethyl ketones have been found to exhibit enzyme-inhibition properties [2] and antimicrobial activities [3]. Moreover, some trifluoromethyl ketones have been found to act as cytotoxic agents against human tumor cell [4].

Traditionally, methods for the synthesis of F-containing organic compounds are direct fluorination and fluoroalkylation [5]. One possible approach for the synthesis of trifluoromethyl ketones containing dihydrofurans would be the cyclization of suitable fluorinated 1,3-dicarbonyl compounds with an unsaturated system mediated by transition metal salts (Mn^{3+}, Ce^{4+}, Ag^{+}, *etc.*). Among these metal salts, $Mn(OAc)_3$ [6] and $(NH_4)_2Ce(NO_3)_6$ [6h][7] are very important radical oxidants in the organic synthesis for the construction of C–C bonds.

Dihydrofurans are a significant class of compounds, since they show a wide range of biological activities and form the basic structure of many natural compounds [8]. Our research group has been studying the synthesis of dihydrofuran derivatives by the radical cyclization of various 1,3-dicarbonyl compounds and 3-oxopropanenitriles with alkenes, alkynes, unsaturated amides, and dienes [9].

Previously, we described the synthesis of 4,5-dihydro-3-(trifluoroacetyl)furans and 3-[4,5-dihydrofuran-2(3*H*)-ylidene]-1,1,1-trifluoroacetones by the treatments of trifluoromethyl 1,3-diketones with alkenes [9e]. In this study, we investigated the radical cyclization of various di-, tri-, and heptafluorinated 1,3-diketones **1a**–**1g** with conjugated dienes **2a**–**2e** using $Mn(OAc)_3$. As a result of these reactions, we obtained 4,5-dihydro-5-(2-phenylethenyl)furans containing difluoroacetyl, trifluoroacetyl, and heptafluorobutanoyl residues.

Results and Discussions. – $Mn(OAc)_3 \cdot 2\ H_2O$ was prepared in a bipolar packed-bed reactor by an electrochemical method (see *Exper. Part*). All fluorinated 1,3-diketones **1a**–**1g**, 2,3-dimethylbuta-1,3-diene (**2a**), and 1,4-diphenylbuta-1,3-diene (**2b**) are commercial products. The other conjugated dienes, 1-phenylbuta-1,3-diene (**2c**) [10], 3-methyl-1-phenylbuta-1,3-diene (**2d**) [11], and 1,3-diphenylbuta-1,3-diene (**2e**) [12] were synthesized as described in literature. All radical cyclizations were performed at a 1.2 : 1 : 2.5 molar ratio (**1**/**2**/$Mn(OAc)_3$) under a N_2 atmosphere in AcOH. All compounds were purified by column chromatography or preparative TLC and characterized by IR, ^{1}H-, ^{13}C-, and ^{19}F-NMR spectroscopy, mass spectrometry, and microanalysis.

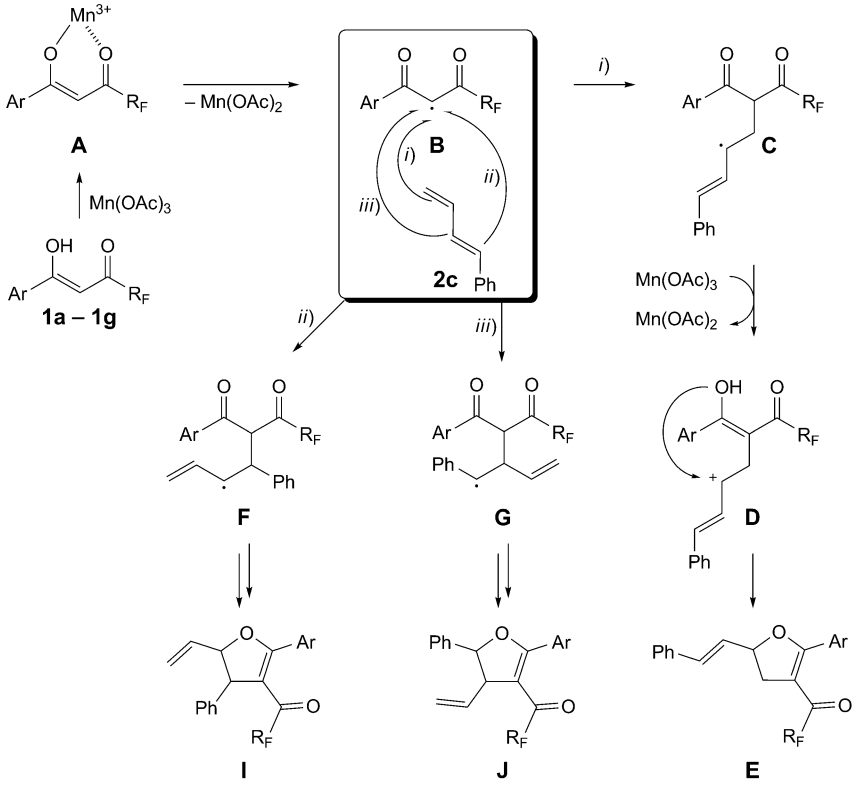
The mechanism proposed for the radical cyclization of conjugated dienes with fluorinated 1,3-diketones is presented in *Scheme 1*. According to this mechanism, the reaction of enol forms of the 1,3-diketones **1** with $Mn(OAc)_3$ produce an Mn^{III}-enolate complex **A**. While Mn^{3+} is reduced to Mn^{2+}, a C-radical **B** is formed. Then, addition of diene **2c** to the radical forms an allylic radical intermediate **C** by pathway *i*. Radical **C** is oxidized to the carbocation **D** by 1 equiv. of $Mn(OAc)_3$, followed by cyclization of **D** to give 4,5-dihydro-5-(2-phenylvinyl)furan **E**. Similarly, the allylic and benzylic radical intermediates **F** and **G** may be formed *via* pathways *ii* and *iii*, respectively. These intermediates can form 4,5-dihydrofurans **I** and **J** as a result of the same two steps as mentioned above.

The ^{1}H-NMR spectra of the isolated compounds showed signals of two olefinic H-atoms in (*E*)-position ($J = 16$) and of two diastereotopic H–C(4) as a *doublet* of *doublet* ($J = 14.4$ and 8.4), complying with structure **E**. Thus, we concluded that products **I** and **J** were not formed, and 4,5-dihydro-5-(2-phenylvinyl)furans **E** are the only products. Although both intermediate products **C** and **F** are allylic radicals, only the cyclization of **C** was observed. The explanation is that the radical **B** added to the diene to form the more stable allylic radical, which is conjugated with the Ph group.

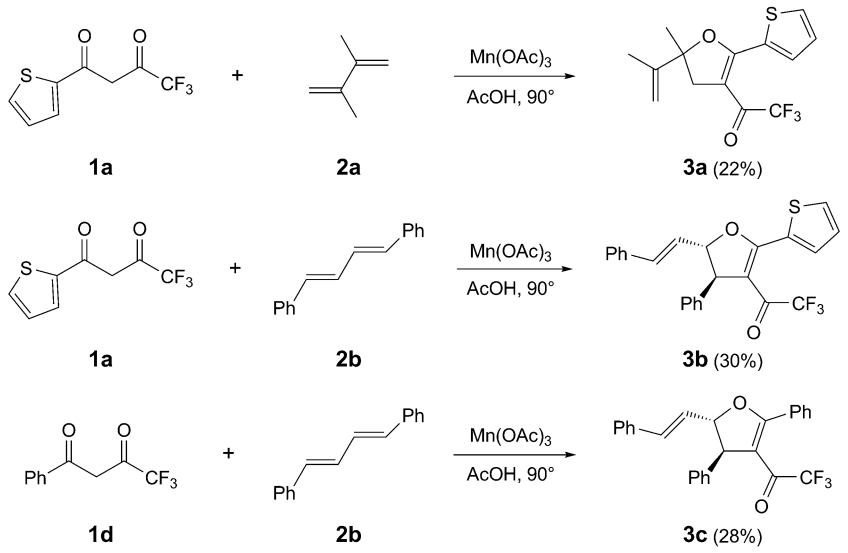
Radical cyclization of 4,4,4-trifluoro-1-(thiophen-2-yl)butane-1,3-dione (**1a**) with the symmetric 2,3-dimethylbuta-1,3-diene (**2a**) gave **3a** in 22% yield (*Scheme 2*). Similarly, treatment of 1,4-diphenylbuta-1,3-diene (**2b**) with **1a** and **1d** produced 4,5-dihydro-5-(2-phenylvinyl)furans **3b** (30%) and **3c** (28%), respectively. Since the coupling constants between H–C(4) and H–C(5) of **3b** and **3c**, J_{trans}, are in the range of 3.5–4.0, the 2-phenylethenyl and Ph groups are in *trans*-position. Coupling constants of *cis* H-atoms in similar structures are reported as $J_{cis} = 8-9$ [13] and $J_{cis} = 9.6-10$ [9g].

Radical cyclizations of thiophen-2-yl-substituted 1,3-dicarbonyl compounds containing a CF_3 group (*i.e.*, **1a**) and heptafluoropropyl group (*i.e.*, **1b**) with 1-phenylbuta-1,3-diene (**2c**) as a terminal diene gave **3d** and **3e** in moderate yields, respectively (*Table*, *Entries 1* and *2*). The reactions of **1c**–**1e** with **2c** gave similar results. 3-(2,2-

Scheme 1. *Mechanism of the Radical Cyclization of* **1a**–**1g** *with Dienes*

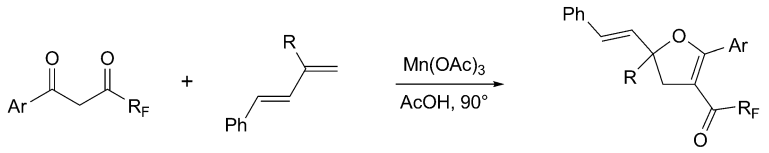


Scheme 2. *Radical Cyclization of* **1a** *and* **1c** *with Symmetric Dienes* **2a** *and* **2b**



difluoroacetyl)-4,5-dihydrofurans **3i** and **3j** were obtained *via* the radical cyclization of 4,4-difluoro-1-(furan-2-yl)buta-1,3-dione (**1f**) and 4,4-difluoro-1-phenylbutane-1,3-dione (**1g**), respectively, with **2c**. The ^{1}H-NMR spectra of 4,5-dihydrofurans **3d** and **3f**–**3h** containing a CF_3 group exhibit signals of one of the two H–C(4) as *dd* ($J = 14.4$ and 8.8 Hz) and of the other one as *ddq* ($J = 14.4$, 9.6, and $^5J(H,F) = 0.8$ Hz). The ^{13}C-NMR spectra of these compounds show signals for CF_3 as *quadruplet* ($^1J(C,F) = 287$), for C=O as *quadruplet* ($^2J(C,F) = 53$), and for C(4) as *quadruplet* ($^4J(C,F) = 2.4–3.1$ Hz). The ^{1}H-NMR spectra of **3i** and **3j** exhibit a *triplet* ($^1J(H,F) = 54$) for the CF_2H groups, and the ^{19}F-NMR spectra of these compounds show a *doublet* ($^1J(F,H) = 54.5$) for each F-atom.

Table. *Reactions of* **1a**–**1g** *and Buta-1,3-diene Derivatives* **2c**–**2e**



1a – 1g **2c – 2e** **3d – 3u**

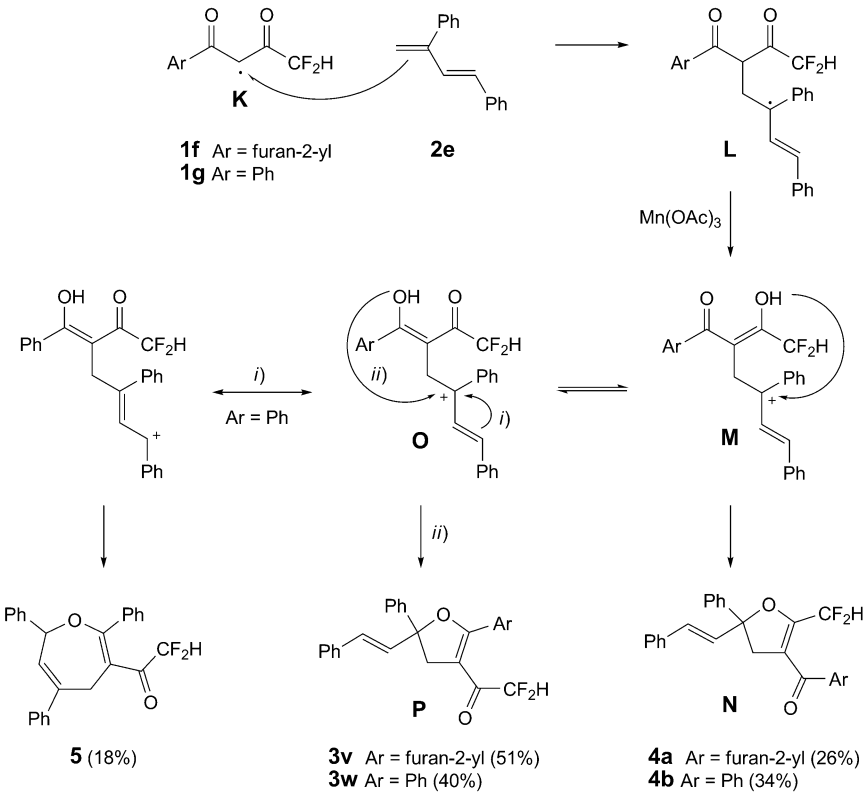
Entry	Ar	R_F	**1**	R	**2**	**3**	Yield [%][a]
1	Thiophen-2-yl	CF_3	**1a**	H	**2c**	**3d**	54
2	Thiophen-2-yl	$CF_3CF_2CF_2$	**1b**	H	**2c**	**3e**	57
3	Furan-2-yl	CF_3	**1c**	H	**2c**	**3f**	51
4	Ph	CF_3	**1d**	H	**2c**	**3g**	52
5	Naphthalen-2-yl	CF_3	**1e**	H	**2c**	**3h**	41
6	Furan-2-yl	CF_2H	**1f**	H	**2c**	**3i**	46
7	Ph	CF_2H	**1g**	H	**2c**	**3j**	48
8	Thiophen-2-yl	CF_3	**1a**	Me	**2d**	**3k**	72
9	Thiophen-2-yl	$CF_3CF_2CF_2$	**1b**	Me	**2d**	**3l**	74
10	Furan-2-yl	CF_3	**1c**	Me	**2d**	**3m**	67
11	Ph	CF_3	**1d**	Me	**2d**	**3n**	70
12	Naphthalen-2-yl	CF_3	**1e**	Me	**2d**	**3o**	53
13	Furan-2-yl	CF_2H	**1f**	Me	**2d**	**3p**	58
14	Ph	CF_2H	**1g**	Me	**2d**	**3q**	60
15	Thiophen-2-yl	CF_3	**1a**	Ph	**2e**	**3r**	84
16	Thiophen-2-yl	$CF_3CF_2CF_2$	**1b**	Ph	**2e**	**3s**	88
17	Furan-2-yl	CF_3	**1c**	Ph	**2e**	**3t**	77
18	Ph	CF_3	**1d**	Ph	**2e**	**3u**	75

[a]) Yield of isolated product based on the amount of diene.

The radical cyclizations of 3-methyl-1-phenylbuta-1,3-diene (**2d**) with 1,3-diketones **1a**–**1g** formed 4,5-dihydrofurans in 53–74% yields. However, reactions of diene **2e** containing a Ph group at C(2) with **1a**–**1d** afforded fluoroacylated 4,5-dihydrofurans in higher yields (*Entries 15–18*). The best result was obtained in the reaction of **1b** with **2e** forming 3-(heptafluorobutanoyl)-4,5-dihydrofuran **3s** in 88% yield. On the other hand, the reactions of **2e** with **1f** and **1g** produced 2-(difluoromethyl)-4,5-dihydrofurans **4a** and **4b** along with 3-(2,2-difluoroacetyl-4,5-dihydrofurans **3v** and **3w**, respectively. Additionally, the reaction of **2e** with **1g** also afforded 2,2-difluoro-1-(4,7-

dihydro-2,5,7-triphenyloxepin-3-yl)ethanone (**5**) as an unexpected product. The proposed mechanism for the formation of these products is shown in *Scheme 3*.

Scheme 3. *Reaction Mechanism of the Radical Cyclization of* **1f** *and* **1g** *with* **2e**



Reaction of **2e** with the radical **K**, obtained from treatment of 1,3-dicarbonyl compounds with $Mn(OAc)_3$, forms intermediate radical **L**. This product is oxidized to carbocation **M** with $Mn(OAc)_3$, and 2-(difluoromethyl)-4,5-dihydrofurans **N** (*i.e.,* **4a** and **4b**) are formed by subsequent intramolecular cyclization. 3-(Difluoroacetyl)-4,5-dihydrofurans **P** (*i.e.*, **3v** and **3w**) are obtained by intramolecular cyclization of carbocation **O**, which is the tautomeric enol form of **M**. The unexpected product **5** is obtained by the cyclization of the other mesomeric structure of **O**. The structures of compounds of type **N** and **P** were deduced on the basis of the chemical shifts and coupling constants of the C=O and C(2) atoms in the ^{13}C-NMR spectra. While the spectrum of **N** shows a *singlet* for C=O, it appears in **P** at 181 – 183 ppm as *triplet* (J = 24). Similarly, whereas the ^{13}C-NMR spectrum of **N** exhibits a *triplet* for C(2) at 156 – 159 ppm (J = 22), that of **P** shows it as a *singlet*. On the other hand, the ^{13}C-NMR spectrum of **5** shows a *dd* for C=O at 198.6 (^2J(C,F) = 29.7, 20.6), confirming that the CF_2H group is adjacent to C=O. In addition, the ^{1}H-NMR spectrum of this compound exhibits only one olefinic H-atom singlet at 6.12 ppm, revealing that the structure of **5**

is not consistent with any of the dihydrofurans shown in *Scheme 1*. Moreover, the signals of diastereotopic H-atom at C(4) appear as an *AB* system with $J = 16.8$. According to these results, we concluded that compound **5** is a 4,7-dihydro-2,5,7-triphenyloxepin derivative.

Conclusions. – In conclusion, radical cyclizations of available fluorinated 1,3-diketones with conjugated dienes were performed, leading to di-, tri-, and heptafluoro-acylated 4,5-dihydrofurans. Radical cyclizations of symmetric dienes with 1,3-diketones afforded dihydrofurans in low yields, whereas the dienes conjugated with a Ph group gave 4,5-dihydrofurans in higher yields. With dienes **2d** and **2e** having Me and Ph groups at C(3), respectively, remarkable increases in yields were observed. This can be explained by increased stability of intermediate radicals or cations by Me and Ph groups.

Experimental Part

General. All solvents were dried by standard methods. Column chromatography (CC): silica gel (SiO_2, 230–400 mesh; *Merck*). TLC: anal. aluminium plates coated with SiO_2 *60* $F_{254-366nm}$ (0.2 mm, *Merck*). M.p.: *Electrothermal*; uncorrected. IR Spectra: *Mattson 1000* FT-IR instrument; KBr pellets, in the 400–4000-cm^{-1} range with 4-cm^{-1} resolution; $\tilde{\nu}$ in cm^{-1}. ^{1}H-, ^{19}F- and ^{13}C-NMR spectra: *Mercury-400* high performance digital FT-NMR insrument; in $CDCl_3$; δ in ppm rel. to Me_4Si as internal standard, J in Hz. LC/ESI-MS: *Waters 2695 Alliance Micromass ZQ* instrument; in m/z.

Electrochemical Preparation of Mn(OAc)$_3$. $Mn(OAc)_3 \cdot 2\,H_2O$ was obtained in an optimized bipolar packed-bed reactor by the electrochemical method described in [14]. However, to obtain $Mn(OAc)_3$, this reactor was modified to a semi-pilot scale. Therefore, a reactor consisting of a glass tube (inner diameter, 52 mm) having 40 rows of graphite rings inside was designed. Each horizontal row in the reactor had seven graphite rings which were 14 mm long, 16 mm in outer diameter, and 10 mm in inner diameter. Each horizontal row was insulated from each other by placing a *Teflon* net with 1-mm thickness beetween the rows. Applying a potential to the reactor through two graphite rods, 10 cm in lenght and 6 mm in diameter, placed top and bottom of the reactor, $Mn(OAc)_2$ was oxidized to $Mn(OAc)_3$ by electrolysis. For this, a soln. containing 100 mM $Mn(OAc)_2$ and 400 mM AcONa was prepared from 95% AcOH. During applying 180 V cell potential supplied by 500 V/1A dc power unit to the reactor, this colorless soln. was added to the reactor continuously with a dosage pump with 2.5 l/h flow rate. The red-brown soln. collected from the bottom of the reactor was recycled through the reactor under the same condition. The obtained soln. was kept in a closed glass container for 3–4 d, until $Mn(OAc)_3$ completely precipitated, and the color of the soln. disappeared. The red-brown precipitate was filtered through a sintered funnel (diameter, 120 mm) with porosity 4 and washed with glacial AcOH (2×100 ml) and than Et_2O (2×100 ml). The obtained solid was dried under vacuum in a desiccator containing P_2O_5 for one week. Yield: 120 g (90%).

General Procedure for Synthesis of 4,5-Dihydrofurans. A soln. of $Mn(OAc)_3$ (0.67 g, 2.5 mmol) in 10 ml of glacial AcOH was heated under N_2 at 80°, until it dissolved. After the soln. was cooled to 60°, a soln. of 1,3-ketone (1.2 mmol) and diene (1 mmol) in AcOH was added. Then, the temp. was raised to 90°. The reaction was completed when the dark brown color of the soln. converted to yellow (30–60 min), and disappearance of the diene was monitored by TLC. Then, H_2O was added, and the mixture was extracted with $CHCl_3$ (3×20 ml). The combined org. phases were neutralized with sat. $NaHCO_3$, dried (anh. Na_2SO_4), and evaporated. Crude products were purified by CC on SiO_2 or prep. TLC (20×20 cm plates, 2 mm thickness) with hexane/AcOEt 6:1 as eluant.

1-[4,5-Dihydro-5-methyl-5-(1-methylethenyl)-2-(thiophen-2-yl)furan-3-yl]-2,2,2-trifluoroethanone (**3a**). Yield: 0.066 g (22%). Pale yellow oil. IR: 1674 (C=O), 1600 (C=C), 1185, 845. ^{1}H-NMR: 1.64 (*s*, Me); 1.87 (*t*, $J = 0.56$, Me); 3.14 (*dd*, $J = 14.6$, 0.9, 1 H, CH_2(4)); 3.35 (*dd*, $J = 14.6$, 0.5, 1 H, CH_2(4)); 4.94

(t, J = 1.3, 1 H, CH_2=); 5.11 (s, 1 H, CH_2=); 7.20 (dd, J = 5.0, 4.0, 1 H); 7.68 (dd, J = 5.0, 1.2, 1 H); 8.56 (dd, J = 4.0, 1.2, 1 H). ^{19}F-NMR: −77.3 (s, CF_3). ^{13}C-NMR: 18.5 (Me); 25.8 (Me); 40.4 (q, ^4J(C,F) = 3.3, C(4)); 91.25 (C(5)); 102.9 (C(3)); 110.8 (C=); 117.2 (q, ^1J(C,F) = 289.5, CF_3); 127.7 (C=); 131.2, 133.4, 134.9, 146.1 (4 C(thiophene)); 165.6 (C(2)); 174.4 (q, ^2J(C,F) = 33.9, C=O). LC/ESI-MS: 303 (100, $[M+H]^+$). Anal. calc. for $C_{14}H_{13}F_3O_2S$ (302.31): C 55.62, H 4.33, S 10.61; found: C 55.59, H 4.56, S 10.37.

*1-{4,5-Dihydro-4-phenyl-5-[(*E*)-2-phenylethenyl]-2-(thiophen-2-yl)furan-3-yl}-2,2,2-trifluoroethanone* (**3b**). Yield: 0.142 g (30%). Yellow oil. IR: 3061, 1686 (C=O), 1650 (C=C), 1550, 1135, 874, 750, 694. ^{1}H-NMR: 4.87 (d, J = 3.5, H–C(4)); 5.46 (dd, J = 7.4, 3.5, H–C(5)); 6.66 (dd, J = 15.7, 7.5, 1 H); 6.96 (d, J = 15.7, 1 H); 7.49–7.72 (m, 11 H); 8.35 (dd, J = 5.0, 1.1, 1 H); 8.87 (dd, J = 3.9, 1.1, 1 H). ^{19}F-NMR: −74.4 (s, CF_3). ^{13}C-NMR: 54.7 (d, ^4J(C,F) = 2.4, C(4)); 90.3 (C(5)); 93.3 (C(olefin)); 108.5 (C(3)); 119.5 (q, ^1J(C,F) = 291.4, CF_3); 124.3; 126.6; 128.1; 128.2; 128.8; 129.2; 129.8; 129.9; 130.0; 130.3; 131.8; 135.0; 135.4; 136.7; 137.0; 143.6; 168.2 (C(2)); 177.2 (q, ^2J(C,F) = 34.8, C=O). LC/ESI-MS: 427 (100, $[M+H]^+$). Anal. calc. for $C_{24}H_{17}F_3O_2S$ (426.45): C 67.59, H 4.02, S 7.52; found: C 67.74, H 3.86, S 7.71.

*1-{4,5-Dihydro-2,4-diphenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2,2-trifluoroethanone* (**3c**). Yield: 0.118 g (28%). Yellow oil. IR: 3128, 1677 (C=O), 1655 (C=C), 1566, 1526, 1246, 1113, 693, 736. ^{1}H-NMR: 4.55 (d, J = 4.0, H–C(4)); 5.22 (dd, J = 7.2, 3.6, H–C(5)); 6.45 (dd, J = 16.0, 7.6, 1 H); 6.96 (d, J = 15.6, 1 H); 7.30–7.60 (m, 13 H); 7.89 (m, 2 H). ^{19}F-NMR: −73.7 (s, CF_3). ^{13}C-NMR: 55.0, 92.9 C(5); 110.1 (C(olefin)); 118.1 (q, ^1J(C,F) = 290.0, CF_3); 125.5; 127.2; 127.3; 127.9; 128.4; 128.9; 129.0; 129.3; 130.0; 132.4; 134.3; 135.7; 142.2; 173.3 (C(2)); 177.2 (q, ^2J(C,F) = 35.9, C=O). LC/ESI-MS: 421 (100, $[M+H]^+$). Anal. calc. for $C_{26}H_{19}F_3O_2$ (420.42): C 74.28, H 4.56; found: C 73.93, H 4.28.

*1-{4,5-Dihydro-5-[(*E*)-2-phenylethenyl]-2-(thiophen-2-yl)furan-3-yl}-2,2,2-trifluoroethanone* (**3d**). Yield: 0.189 g (54%). Yellow oil. IR: 3122, 1670 (C=O), 1662 (C=C), 1567, 1528, 1243, 1032, 750, 693. ^{1}H-NMR: 3.20 (dd, J = 14.8, 8.4, 1 H, CH_2(4)); 3.55 (ddd, J = 14.4, 9.6, 0.8, 1 H, CH_2(4)); 5.46 (ddd, J = 16.4, 8.0, 1.6, H–C(5)); 6.34 (dd, J = 16.0, 8.0, 1 H); 6.76 (d, J = 16.0, 1 H); 7.20 (dd, J = 5.2, 4.0, 1 H); 7.32–7.46 (m, 5 H); 7.68 (dd, J = 4.8, 1.2, 1 H); 8.57 (dd, J = 4.4, 1.2, 1 H). ^{19}F-NMR: −84.0 (s, CF_3). ^{13}C-NMR: 35.8 (d, ^4J(C,F) = 3.1, C(4)); 84.6 (C(5)); 103.4 (C(3)); 117.5 (q, ^1J(C,F) = 289.6, CF_3); 126.4; 127.1; 128.1; 128.8; 129.0; 131.1; 133.9; 134.2; 135.4; 135.8; 166.8 (C(2)); 174.8 (q, ^2J(C,F) = 34.3, C=O). LC/ESI-MS: 351 (100, $[M+H]^+$). Anal. calc. for $C_{18}H_{13}F_3O_2S$ (350.36): C 61.71, H 3.74, S 9.15; found: C 61.87, H 3.44, S 8,92.

*1-{4,5-Dihydro-5-[(*E*)-2-phenylethenyl]-2-(thiophen-2-yl)furan-3-yl}-2,2,3,3,4,4,4-heptafluorobutan-1-one* (**3e**). Yield: 0.256 g (57%). Yellow oil. IR: 1675 (C=O), 1661, 1596 (C=C), 1221, 1051, 749, 690. ^{1}H-NMR: 3.22 (dd, J = 15.2, 8.8, 1 H, CH_2(4)); 3.56 (dd, J = 14.8, 9.6, 1 H, CH_2(4)); 5.44 (dd, J = 17.2, 8.4, H–C(5)); 6.33 (dd, J = 16.4, 8.0, 1 H); 6.73 (d, J = 16.0, 1 H); 7.18 (td, J = 4.8, 0.8, 1 H); 7.30 (dd, J = 8.0, 1.2, 1 H); 7.34 (t, J = 7.2, 2 H); 7.42 (d, J = 8.0, 2 H); 7.66 (d, J = 5.2, 1 H); 8.5 (d, J = 4.4, 1 H). ^{19}F-NMR: −80.66 (t, J = 9.4, CF_3); −118.06 (m, CCF_3, CF'_2); −126.5 (t, J = not determined, $CF_3CF_2CF_2$). ^{13}C-NMR: 35.6 (t, ^4J(C,F) = 2.4, C(4)); 84.4 (C(5)); 104.9 (C(3)); 109.1 (tqt, ^1J(C,F) = 265.2, ^2J(C,F) = 38.1, ^3J(C,F) = not determined, CF_3CF_2); 110.4 (tt, ^1J(C,F) = 265.9, ^2J(C,F) = 31.3, $CF_3CF_2CF_2$); 117.7 (qt, ^1J(C,F) = 285.7, ^2J(C,F) = 33.5, CF_3); 126.0; 126.9; 127.8; 128.6; 128.7; 130.9; 133.9; 134.2; 135.4; 135.6; 167.2 (C(2)); 176.8 (t, ^2J(C,F) = 24.4, C=O). LC/ESI-MS: 451 (100, $[M+H]^+$). Anal. calc. for $C_{20}H_{13}F_7O_2S$ (450.37): C 53.34, H 2.91, S 7.12; found: C 53.56, H 2.68, S 7.25.

*1-{4,5-Dihydro-5-[(*E*)-2-phenylethenyl][2,2′-bifuran]-3-yl}-2,2,2-trifluoroethanone* (**3f**). Yield: 0.171 g (51%). Light yellow solid. M.p. 101–103°. IR: 3028, 1665 (C=O), 1566 (C=C), 1532, 1263, 1063, 749, 693. ^{1}H-NMR: 3.20 (dd, J = 14.4, 8.8, 1 H, CH_2(4)); 3.55 (dd, J = 14.4, 9.6, 1 H, CH_2(4)); 5.50 (ddd, J = 16.4, 8.8, 0.8, H–C(5)); 6.36 (dd, J = 15.6, 8.0, 1 H); 6.61 (dd, J = 4.0, 1.6, 1 H); 6.75 (d, J = 15.6, 1 H); 7.30–7.43 (m, 5 H); 7.65 (t, J = 0.8, 1 H); 7.68 (d, J = 3.6, 1 H). ^{19}F-NMR: −74.3 (s, CF_3). ^{13}C-NMR: 35.2 (d, ^4J(C,F) = 3.1, C(4)); 85.3 (C(5)); 103.8 (C(3)); 112.9; 117.3 (q, ^1J(C,F) = 289.6, CF_3); 122.5; 126.0; 127.1; 128.8; 129.0; 131.1; 134.8; 135.8; 144.4; 146.8; 161.9; 170.1 (C(2)); 174.2 (q, ^2J(C,F) = 34.3, C=O). LC/ESI-MS: 335 (100, $[M+H]^+$). Anal. calc. for $C_{18}H_{13}F_3O_3$ (334.29): C 64.67, H 3.92; found: C 64.40, H 4.18.

*1-{4,5-Dihydro-2-phenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2,2-trifluoroethanone* (**3g**). Yield: 0.179 g (52%). Light-yellow solid. M.p. 64–66°. IR: 3029, 1697 (C=O), 1670 (C=C), 1534, 1200, 1133, 751, 692. ^{1}H-NMR: 3.23 (dd, J = 14.4, 8.4, 1 H, CH_2(4)); 3.56 (ddq, J = 14.8, 10.0, 0.8, 1 H, CH_2(4)); 5.52 (ddd, J = 18.0, 8.8, 0.8, H–C(5)); 6.40 (dd, J = 16.0, 8.0, 1 H); 6.76 (d, J = 15.6, 1 H); 7.32–7.53 (m, 8 H);

7.87 (*m*, 2 H). ^{19}F-NMR: −76.4 (*s*, CF_3). ^{13}C-NMR: 36.0 (*d*, ^4J(C,F) = 3.0, C(4)); 84.9 (C(5)); 105.1 (C(3)); 117.2 (*q*, ^1J(C,F) = 289.5, CF_3); 126.4; 127.1; 128.2; 128.8; 129.0; 129.2; 129.8; 123.3; 134.4; 135.8; 173.7 (C(2)); 175.4 (*q*, ^2J(C,F) = 34.3, C=O). LC/ESI-MS: 345 (100, $[M+H]^+$). Anal. calc. for $C_{20}H_{15}F_3O_2$ (344.33): C 69.76, H 4.39; found: C 69.47, H 4.54.

*1-{4,5-Dihydro-2-(naphthalen-2-yl)-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2,2-trifluoroethanone* (**3h**). Yield: 0.162 g (41%). Pale yellow oil. IR: 3060, 1683 (C=O), 1663 (C=C), 1554, 1492, 1178, 1096, 746, 691. ^{1}H-NMR: 3.27 (*dd*, *J* = 14.4, 8.4, 1 H, CH_2(4)); 3.60 (*ddq*, *J* = 14.4, 9.6, 0.8, 1 H, CH_2(4)); 5.55 (*ddd*, *J* = 17.2, 9.2, 1.2, H–C(5)); 6.43 (*dd*, *J* = 16.0, 8.0, 1 H); 6.77 (*d*, *J* = 16.0, 1 H); 7.33 (*tt*, *J* = 7.2, 1.6, 1 H); 7.39 (*t*, *J* = 7.6, 2 H); 7.47 (*dt*, *J* = 6.8, 1.6, 2 H); 7.52–7.61 (*m*, 2 H); 7.87 (*dd*, *J* = 4.8, 1.6, 2 H); 7.95 (*d*, *J* = 8.0, 1 H); 8.51 (*s*, 1 H). ^{19}F-NMR: −76.35 (*s*, CF_3). ^{13}C-NMR: 35.8 (*q*, ^4J(C,F) = 3.0, C(4)); 84.7 (C(5)); 105.0 (C(3)); 120.0 (*q*, ^1J(C,F) = 289.6, CF_3); 125.6; 126.1; 126.6; 126.8; 127.5; 127.7; 128.1; 128.6; 128.7; 129.2; 131.1; 132.3; 134.2; 135.0; 135.6; 173.5 (C(2)); 175.4 (*q*, ^2J(C,F) = 34.3, C=O). LC/ESI-MS: 395 (100, $[M+H]^+$). Anal. calc. for $C_{24}H_{17}F_3O_2$ (394.39): C 73.09, H 4.34; found: C 73.36, H 4.58.

*1-{4,5-Dihydro-5-[(*E*)-2-phenylethenyl][2,2′-bifuran]-3-yl}-2,2-difluoroethanone* (**3i**). Yield: 0.145 g (46%). Pale yellow oil. IR: 3029, 1661, 1524, 1209, 749, 719, 692. ^{1}H-NMR: 3.19 (*dd*, *J* = 14.8, 8.4, 1 H, CH_2(4)); 3.53 (*dd*, *J* = 14.8, 10.0, 1 H, CH_2(4)); 5.46 (*ddd*, *J* = 17.6, 8.4, 1.2, H–C(5)); 6.0 (*t*, ^2J(H,F) = 54.0, 1 H); 6.34 (*dd*, *J* = 15.6, 7.2, 1 H); 6.59 (*dd*, *J* = 4.0, 2.0, 1 H); 6.74 (*d*, *J* = 16.0, 1 H); 7.28 (*tt*, *J* = 7.6, 1.2, 1 H); 7.33 (*td*, *J* = 7.2, 1.6, 2 H); 7.41 (*dt*, *J* = 7.2, 1.2, 2 H); 7.62 (*d*, *J* = 1.2, 1 H); 8.15 (*d*, *J* = 4.0, 1 H). ^{19}F-NMR: −125.6 (*d*, ^2J(F,H) = 53.0, 1 F); −125.7 (*d*, ^2J(F,H) = 53.0, 1 F). ^{13}C-NMR: 35.2 (*t*, ^4J(C,F) = 3.8, C(4)); 84.6 (C(5)); 105.6 (C(3)); 111.4 (*t*, ^1J(C,F) = 250.7, CHF_2); 112.5; 120.5; 126.2; 126.8; 128.5; 128.7; 134.2; 133.7; 144.3; 146.0; 159.0 (C(2)); 181.7 (*t*, ^2J(C,F) = 24.4, C=O). LC/ESI-MS: 317 (100, $[M+H]^+$). Anal. calc. for $C_{18}H_{14}F_2O_3$ (316.30): C 68.35, H 4.46; found: C 68.67, H 4.23.

*1-{4,5-Dihydro-2-phenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2-difluoroethanone* (**3j**). Yield: 0.157 g (48%). Pale-yellow oil. IR: 3058, 1736 (C=O), 1673 (C=C), 1595, 747, 695. ^{1}H-NMR: 3.17 (*dd*, *J* = 14.8, 8.8, 1 H, CH_2(4)); 3.50 (*dd*, *J* = 14.8, 10.4, 1 H, CH_2(4)); 5.47 (*dd*, *J* = 18.0, 8.0, H–C(5)); 5.74 (*t*, ^2J(H,F) = 54.0, 1 H); 6.36 (*dd*, *J* = 16.0, 7.6, 1 H); 6.73 (*d*, *J* = 16.0, 1 H); 7.30 (*t*, *J* = 7.2, 1 H); 7.35 (*t*, *J* = 6.8, 2 H); 7.42–7.48 (*m*, 4 H); 7.52 (*t*, *J* = 7.2, 1 H); 7.71 (*dd*, *J* = 7.2, 1.2, 2 H). ^{19}F-NMR: −125.1 (*d*, ^2J(F,H) = 54.5, 1 F); −125.25 (*d*, ^2J(F,H) = 54.5, 1 F). ^{13}C-NMR: 36.0 (C(4)); 84.4 (C(5)); 103.4 (C(3)); 109.2 (*t*, ^1J(C,F) = 249.9, CHF_2); 109.0; 126.5; 126.8; 128.3; 128.5; 128.7; 129.3; 129.5; 131.8; 133.8; 135.7; 170.7 (C(2)); 183.1 (*t*, ^2J(C,F) = 24.4, C=O). LC/ESI-MS: 327 (100, $[M+H]^+$). Anal. calc. for $C_{20}H_{16}F_2O_2$ (326.34): C 73.61, H 4.94; found: C 73.50, H 4.84.

*1-{4,5-Dihydro-5-methyl-5-[(*E*)-2-phenylethenyl]-2-(thiophen-2-yl)furan-3-yl}-2,2,2-trifluoroethanone* (**3k**). Yield: 0.262 g (72%). Pale yellow solid. M.p. 79–81°. IR: 3028, 1683 (C=O), 1653 (C=C), 1637, 1243, 748, 694. ^{1}H-NMR: 1.75 (*s*, Me); 3.28 (*dd*, *J* = 14.4, 1 H, CH_2(4)); 3.44 (*dd*, *J* = 14.4, 1 H, CH_2(4)); 6.40 (*d*, *J* = 16.0, 1 H); 6.70 (*d*, *J* = 16.4, 1 H); 7.22 (*td*, *J* = 4.8, 0.8, 1 H); 7.28 (*d*, *J* = 7.2, 1 H); 7.35 (*t*, *J* = 7.6, 2 H); 7.42 (*d*, *J* = 7.2, 2 H); 7.70 (*dd*, *J* = 5.2, 0.8, 1 H); 8.60 (*dd*, *J* = 3.2, 0.8, 1 H). ^{19}F-NMR: −76.4 (*s*, CF_3). ^{13}C-NMR: 26.6; 41.79; 89.3 (C(5)); 103.1 (C(3)); 117.5 (*q*, ^1J(C,F) = 289.6, CF_3); 127.0; 128.0; 128.4; 128.6; 128.9; 129.5; 131.3; 133.8; 135.4; 136.1; 165.8 (C(2)); 174.5 (*q*, ^2J(C,F) = 33.5, C=O). LC/ESI-MS: 365 (100, $[M+H]^+$). Anal. calc. for $C_{19}H_{15}F_3O_2S$ (364.38): C 62.63, H 4.15, S 8.80; found: C 62.71, H 4.46, S 8.63.

*1-{4,5-Dihydro-5-methyl-5-[(*E*)-2-phenylethenyl]-2-(thiophen-2-yl)furan-3-yl}-2,2,3,3,4,4,4-heptafluorobutan-1-one* (**3l**). Yield: 0.343 g (74%). Yellow oil. IR: 1650 (C=O), 1615 (C=C), 1586, 1062, 747, 692. ^{1}H-NMR: 1.77 (*s*, Me); 3.26 (*d*, *J* = 14.8, 1 H, CH_2(4)); 3.42 (*d*, *J* = 14.8, 1 H, CH_2(4)); 6.36 (*d*, *J* = 16.0, 1 H); 6.66 (*d*, *J* = 16.0, 1 H); 7.18 (*t*, *J* = 4.8, 1 H); 7.25 (*t*, *J* = 7.6, 1 H); 7.32 (*t*, *J* = 7.2, 2 H); 7.38 (*dd*, *J* = 7.2, 1.6, 2 H); 7.66 (*d*, *J* = 5.2, 1 H); 8.52 (*d*, *J* = 3.6, 1 H). ^{19}F-NMR: −80,6 (*t*, *J* = 9.0, CF_3); −118.06 (*q*, *J* = 9.4, CF_3CF_2); −126.5 (*t*, *J* = not determined, $CF_3CF_2CF_2$). ^{13}C-NMR: 26.2; 41.6 (*t*, ^4J(C,F) = 6.1, C(4)); 89.1 (*t*, ^5J(C,F) = 2.3, C(5)); 104.7 (C(3)); 109.1 (*tqt*, ^1J(C,F) = 265.1, ^2J(C,F) = 38.1, ^3J(C,F) = not determined, CF_3CF_2); 110.4 (*tt*, ^1J(C,F) = 266.7, ^2J(C,F) = 31.3, $CF_3CF_2CF_2$); 117.7 (*qt*, ^1J(C,F) = 285.8, ^2J(C,F) = 33.5, CF_3); 126.7; 127.8; 128.2; 128.7; 129.4; 131.1; 131.2; 133.7; 135.3; 135.9; 166.5 (C(2)); 177.0 (*q*, ^2J(C,F) = 24.4, C=O). LC/ESI-MS: 465 (100, $[M+H]^+$). Anal. calc. for $C_{21}H_{15}F_7O_2S$ (464.40): C 54.31, H 3.26, S 6.90; found: C 54.60, H 3.42, S 7.17.

*1-{4,5-Dihydro-5-methyl-5-[(*E*)-2-phenylethenyl][2,2′-bifuran]-3-yl}-2,2,2-trifluoroethanone* (**3m**). Yield: 0.233 g (67%). Pale yellow solid. M.p. 92–94°. IR: 3030, 1673 (C=O), 1666, 1569 (C=C), 1536,

1126, 743, 731, 688. ^{1}H-NMR: 1.77 (*s*, Me); 3.25 (*d*, *J* = 14.4, 1 H, CH_2(4)); 4.20 (*d*, *J* = 14.8, 1 H, CH_2(4)); 6,41 (*d*, *J* = 16.0, 1 H); 6.62 (*dd*, *J* = 3.6, 1.6, 1 H); 6.70 (*d*, *J* = 16.0, 1 H); 7.27 (*t*, *J* = 7.6, 1 H); 7.33 (*t*, *J* = 8.0, 2 H); 7.42 (*d*, *J* = 7.2, 2 H); 7.67 (*d*, *J* = 1.6, 1 H); 8.43 (*d*, *J* = 3.6, 1 H). ^{19}F-NMR: −74.0 (*s*, 3 F). ^{13}C-NMR: 26.6; 41.2 (*d*, ^4J(C,F) = 3.1, C(4)); 90.1 (C(5)); 103.5 (C(3)); 112.9; 117.4 (*q*, ^1J(C,F) = 289.5, CF_3); 122.5; 127.0; 128.5; 128.9; 129.8; 131.0; 136.1; 144.6; 146.7; 161.0 (C(2)); 174.0 (*q*, ^2J(C,F) = 33.6, C=O). LC/ESI-MS: 349 (100, $[M+H]^+$). Anal. calc. for $C_{19}H_{15}F_3O_3$ (348.32): C 65.52, H 4.34; found: C 65.21, H 4.56.

*1-{4,5-Dihydro-5-methyl-2-phenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2,2-trifluoroethanone* (**3n**). Yield: 0.251 g (70%). Colorless solid. M.p. 87–89°. IR: 3027, 1656 (C=O), 1605 (C=C), 1536, 1049, 747, 694. ^{1}H-NMR: 1.77 (*s*, Me); 3.25 (*d*, *J* = 14.4, 1 H, CH_2(4)); 3.43 (*d*, *J* = 14.8, 1 H, CH_2(4)); 6.42 (*d*, *J* = 16.4, 1 H); 6.72 (*d*, *J* = 16.0, 1 H); 7.30 (*t*, *J* = 7.2, 1 H); 7.37 (*t*, *J* = 7.2, 2 H); 7.45–7.49 (*m*, 4 H); 7.52 (*tt*, *J* = 7.2, 1.2, 1 H); 7.87 (*dd*, *J* = 6.8, 1.2, 2 H). ^{19}F-NMR: −76.2 (*s*, CF_3). ^{13}C-NMR: 26.7; 42.0; 89.6 (C(5)); 104.7 (C(3)); 117.1 (*q*, ^1J(C,F) = 290.3, CF_3); 127.0; 128.2; 128.5; 129.0; 129.6; 129.8; 131.4; 132.2; 136.1; 172.7 (C(2)); 175.4 (*q*, ^2J(C,F) = 34.3, C=O). LC/ESI-MS: 359 (100, $[M+H]^+$). Anal. calc. for $C_{21}H_{17}F_3O_2$ (358.35): C 70.38, H 4.78; found: C 70.45, H 4.54.

*1-{4,5-Dihydro-5-methyl-2-(naphthalen-2-yl)-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2,2-trifluoroethanone* (**3o**). Yield: 0.216 g (53%). Pale-yellow oil. IR: 3029, 2954, 1623 (C=O), 1600 (C=C), 1203, 748, 688. ^{1}H-NMR: 1.76 (*s*, Me); 3.25 (*d*, *J* = 14.8, 1 H, CH_2(4)); 3.43 (*d*, *J* = 14.4, 1 H, CH_2(4)); 6.42 (*d*, *J* = 16.0, 1 H); 6.70 (*d*, *J* = 16.0, 1 H); 7.25 (*t*, *J* = 7.6, 1 H); 7.33 (*t*, *J* = 7.2, 2 H); 7.40 (*d*, *J* = 7.2, 2 H); 7.52 (*m*, 2 H); 7.83 (*d*, *J* = 8.0, 2 H); 7.85, (*s*, 1 H); 7.91 (*d*, *J* = 8.0, 1 H); 8.48 (*s*, 1 H). ^{19}F-NMR: −76.2 (*s*, CF_3). ^{13}C-NMR: 26.4; 41.8 (*d*, ^4J(C,F) = 3.0, C(4)); 89.4 (C(5)); 104.6 (C(3)); 117.0 (*q*, ^1J(C,F) = 290.4, CF_3); 125.6; 126.5; 126.6; 126.7; 127.5; 127.7; 128.0; 128.2; 128.7; 129.2; 129.4; 130.9; 131.1; 132.3; 134.9; 135.9; 172.5 (C(2)); 175.8–175.8 (*q*, ^2J(C,F) = 33.6, C=O). LC/ESI-MS: 409 (100, $[M+H]^+$). Anal. calc. for $C_{25}H_{19}F_3O_2$ (408.41): C 73.52, H 4.69; found: C 73.58, H 4.83.

*1-{4,5-Dihydro-5-methyl-5-[(*E*)-2-phenylethenyl][2,2′-bifuran]-3-yl}-2,2-difluoroethanone* (**3p**). Yield: 0.192 g (58%). Pale-yellow solid. M.p. 67–69°. IR: 2948, 1623 (C=O), 1600 (C=C), 1227, 968, 756, 691. ^{1}H-NMR: 1.73 (*s*, Me); 3.23 (*d*, *J* = 14.4, 1 H, CH_2(4)); 3.40 (*d*, *J* = 14.8, 1 H, CH_2(4)); 6.0 (*t*, ^2J(H,F) = 54.4, CHF_2); 6.40 (*d*, *J* = 16.4, 1 H); 6.60 (*t*, *J* = 1.6, 1 H); 6.67 (*d*, *J* = 16.0, 1 H); 7.25 (*t*, *J* = 8.0, 1 H); 7.32 (*t*, *J* = 7.2, 1 H); 7.64 (*s*, 1 H); 8.12 (*d*, *J* = 3.6, 1 H). ^{19}F-NMR: −125.6 (*d*, ^2J(F,H) = 54.1, 2 F). ^{13}C-NMR: 26.5; 41.1 (*d*, ^4J(C,F) = 3.1, C(4)); 89.3 (C(5)); 105.3 (C(3)); 111.4 (*t*, ^1J(C,F) = 251.5, CHF_2); 112.5; 120.4; 126.7; 128.1; 128.6; 129.3; 131.2; 136.0; 144.5; 146.0; 158.2 (C(2)); 181.8 (*t*, ^2J(C,F) = 24.4, C=O). LC/ESI-MS: 331 (100, $[M+H]^+$). Anal. calc. for $C_{19}H_{16}F_2O_3$ (330.32): C 69.08, H 4.88; found: C 70.15, H 4.59.

*1-{4,5-Dihydro-5-methyl-2-phenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2-difluoroethanone* (**3q**). Yield: 0.204 g (60%). Pale-yellow oil. IR: 3027, 2978, 1691 (C=O), 1643 (C=C), 1235, 966, 748, 693. ^{1}H-NMR: 1.71 (*s*, Me); 3.18 (*d*, *J* = 14.8, 1 H, CH_2(4)); 3.36 (*d*, *J* = 14.8, 1 H, CH_2(4)); 5.7 (*t*, ^2J(H,F) = 54.0, 1 H, CHF_2); 6.40 (*d*, *J* = 16.0, 1 H); 6.67 (*d*, *J* = 16.0, 1 H); 7.25 (*tt*, *J* = 7.6, 1.2, 1 H); 7.32 (*t*, *J* = 7.2, 1 H); 7.40 (*dd*, *J* = 6.8, 1.6, 2 H); 7.45 (*t*, *J* = 8.0, 2 H); 7.50 (*tt*, *J* = 7.2, 1.2, 1 H); 7.71 (*dd*, *J* = 7.2, 1.6, 2 H). ^{19}F-NMR: −125.1 (*d*, ^2J(F,H) = 54.0, 1 F); −125.12 (*d*, ^2J(F,H) = 54.0, 1 F). ^{13}C-NMR: 26.6; 42.0; 89.2 (C(5)); 109.1 (*t*, ^1J(C,F) = 249.1, CHF_2); 108.8; 126.7; 128.1; 128.3; 128.7; 129.1; 129.2; 129.8; 131.4; 131.7; 136.0; 169.7 (C(2)); 183.2 (*t*, ^2J(C,F) = 24.4, C=O). LC/ESI-MS: 341 (100, $[M+H]^+$). Anal. calc. for $C_{21}H_{18}F_2O_2$ (340.36): C 74.10, H 5.33; found: C 74.21, H 5.47.

*1-{4,5-Dihydro-5-phenyl-5-[(*E*)-2-phenylethenyl]-2-(thiophen-2-yl)furan-3-yl}-2,2,2-trifluoroethanone* (**3r**). Yield: 0.358 g (84%). Yellow oil. IR: 1649 (C=O), 1623 (C=C), 1218, 1028, 750, 690. ^{1}H-NMR: 3.72 (*d*, *J* = 14.8, 1 H, CH_2(4)); 3.81 (*d*, *J* = 14.8, 1 H, CH_2(4)); 6.52 (*d*, *J* = 16.0, 1 H); 6.58 (*d*, *J* = 16.0, 1 H); 7.23–7.26 (*m*, 2 H); 7.30 (*tt*, *J* = 7.6, 1.6, 2 H); 7.35–7.37 (*m*, 3 H); 7.42 (*t*, *J* = 7.2, 2 H); 7.51 (*dt*, *J* = 7.6, 1.6, 2 H); 7.71 (*dd*, *J* = 5.2, 1.2, 1 H); 8.65 (*dd*, *J* = 4.0, 1.2, 1 H). ^{19}F-NMR: −76.0 (*s*, CF_3). ^{13}C-NMR: 41.79 (*q*, ^4J(C,F) = 3.1, C(4)); 91.9 (C(5)); 103.0 (C(3)); 117.2 (*q*, ^1J(C,F) = 289.6, CF_3); 125.2; 126.9; 128.0; 128.2; 128.3; 128.6; 128.7; 130.5; 130.7; 131.0; 133.7; 135.2; 135.7; 142.4; 165.2 (C(2)); 174.4 (*q*, ^2J(C,F) = 34.3, C=O). LC/ESI-MS: 427 (100, $[M+H]^+$). Anal. calc. for $C_{24}H_{17}F_3O_2S$ (426.45): C 67.59, H 4.02, S 7.52; found: C 67.71, H 3.80, S 7.48.

*1-{4,5-Dihydro-5-phenyl-5-[(*E*)-2-phenylethenyl]-2-(thiophen-2-yl)furan-3-yl}-2,2,3,3,4,4,4-heptafluorobutan-1-one* (**3s**). Yield: 0.463 g (88%). Yellow oil. IR: 3025, 1650 (C=O), 1631 (C=C), 741, 690.

^{1}H-NMR: 3.74 (*dt*, *J* = 15.2, 1.6, 1 H, CH_2(4)); 3.83 (*dt*, *J* = 14.8, 1.6, 1 H, CH_2(4)); 6.53 (*d*, *J* = 16.0, 1 H); 6.58 (*d*, *J* = 16.0, 1 H); 7.21–7.25 (*m*, 2 H); 7.29 (*tt*, *J* = 7.2, 1.6, 2 H); 7.34–7.37 (*m*, 3 H); 7.41 (*tt*, *J* = 7.2, 1.6, 2 H); 7.51 (*dt*, *J* = 7.2, 1.6, 2 H); 7.70 (*dd*, *J* = 5.2, 1.2, 1 H); 8.61 (*dd*, *J* = 4.0, 1.2, 1 H). ^{19}F-NMR: −80.6 (*t*, *J* = 9.4, CF_3); −118.0 (*q*, *J* = 9.3, CF_3CF_2); −126.7 (*t*, *J* = not determined, $CF_3CF_2CF_2$). ^{13}C-NMR: 42.0 (*q*, ^4J(C,F) = 6.1, C(4)); 91.9 (C(5)); 104.8 (C(3)); 108.8 (*tqt*, ^1J(C,F) = 265.2, ^2J(C,F) = 38.9, ^3J(C,F) = not determined, CF_3CF_2); 110.4 (*tt*, ^1J(C,F) = 266.7, ^2J(C,F) = 31.2, $CF_3CF_2CF_2$); 117.6 (*qt*, ^1J(C,F) = 286.5, ^2J(C,F) = 34.2, CF_3); 125.2; 126.9; 128.0; 128.2; 128.4; 128.7; 128.8; 130.7; 131.0; 134.0; 135.4; 135.7; 142.3; 165.9 (C(2)); 176.7 (*t*, ^2J(C,F) = 25.1, C=O). LC/ESI-MS: 527 (100, $[M+H]^+$). Anal. calc. for $C_{26}H_{17}F_7O_2S$ (526.47): C 59.32, H 3.25, S 6.09; found: C 59.54, H 3.51, S 7.12.

*1-{4,5-Dihydro-5-phenyl-5-[(*E*)-2-phenylethenyl][2,2′-bifuran]-3-yl}-2,2,2-trifluoroethanone* (**3t**). Yield: 0.316 g (77%). Pale yellow solid. M.p. 97–99°. IR: 3026, 1648 (C=O), 1623 (C=C), 1221, 740, 695. ^{1}H-NMR: 3.68 (*d*, *J* = 14.4, 1 H, CH_2(4)); 3.79 (*d*, *J* = 14.4, 1 H, CH_2(4)); 6.54 (*d*, *J* = 16.0, 1 H); 6.60 (*d*, *J* = 16.0, 1 H); 6.63 (*dd*, *J* = 4.0, 1.6, 1 H); 7.23 (*t*, *J* = 7.2, 2 H); 7.29 (*tt*, *J* = 6.8, 2.0, 2 H); 7.35 (*t*, *J* = 8.0, 2 H); 7.41 (*t*, *J* = 7.6, 2 H); 7.51 (*dt*, *J* = 7.2, 1.6, 2 H); 7.00 (*d*, *J* = 1.2, 1 H); 8.41 (*d*, *J* = 3.6, 1 H). ^{19}F-NMR: −76.4 (*s*, CF_3). ^{13}C-NMR: 41.8 (*q*, ^4J(C,F) = 3.1, C(4)); 92.8 (*d*, ^5J(C,F) = 1.5, C(5)); 103.5 (C(3)); 113.0; 117.4 (*q*, ^1J(C,F) = 289.5, CF_3); 122.6; 125.5; 127.2; 128.4; 128.6; 128.9; 129.0; 130.8; 131.0; 135.9; 142.5; 144.5; 146.9; 160.8 (C(2)); 174.1 (*q*, ^2J(C,F) = 34.3, C=O). LC/ESI-MS: 411 (100, $[M+H]^+$). Anal. calc. for $C_{24}H_{17}F_3O_3$ (410.38): C 70.24, H 4.18; found: C 69.95, H 4.29.

*1-{4,5-Dihydro-2,5-diphenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2,2-trifluoroethanone* (**3u**). Yield: 0.315 g (75%). Pale yellow oil. IR: 1667 (C=O), 1620 (C=C), 1592, 731, 697. ^{1}H-NMR: 3.69 (*d*, *J* = 14.8, 1 H, CH_2(4)); 3.76 (*d*, *J* = 14.8, 1 H, CH_2(4)); 6.54 (*s*, 2 H); 7.22–7.50 (*m*, 13 H); 7.90 (*d*, *J* = 6.8, 2 H). ^{19}F-NMR: −76.2 (*s*, CF_3). ^{13}C-NMR: 42.1 (*q*, ^4J(C,F) = 3.1, C(4)); 92.2 (C(5)); 104.7 (C(3)); 117.0 (*q*, ^1J(C,F) = 290.4, CF_3); 125.2; 126.7; 128.0; 128.2; 128.4; 128.6; 128.7; 129.0; 129.7; 130.5; 130.9; 132.1; 135.7; 142.5; 172.0 (C(2)); 175.0 (*q*, ^2J(C,F) = 34.3, C=O). LC/ESI-MS: 421 (100, $[M+H]^+$). Anal. calc. for $C_{26}H_{19}F_3O_2$ (420.42): C 74.28, H 4.56; found: C 74.13, H 4.30.

*1-{4,5-Dihydro-5-phenyl-5-[(*E*)-2-phenylethenyl][2,2′-bifuran]-3-yl}-2,2-difluoroethanone* (**3v**). Yield: 0.200 g (51%). Pale-yellow oil. IR: 3082, 1667 (C=O), 1600 (C=C), 1597, 1030, 742, 693. ^{1}H-NMR: 3.68 (*d*, *J* = 15.2, 1 H, CH_2(4)); 3.80 (*d*, *J* = 15.2, 1 H, CH_2(4)); 6.0 (*t*, *J* = 54.4, CF_2H); 6.54 (*d*, *J* = 16.0, 1 H); 6.60 (*d*, *J* = 16.0, 1 H); 6.61 (*dd*, *J* = 3.2, 2.0, 1 H); 7.23 (*t*, *J* = 6.4, 2 H); 7.29 (*t*, *J* = 7.2, 2 H); 7.35 (*tt*, *J* = 7.2, 3.6, 2 H); 7.42 (*t*, *J* = 3.6, 1 H); 7.51 (*d*, *J* = 7.6, 2 H); 7.67 (*d*, *J* = 0.8, 1 H); 8.15 (*d*, *J* = 3.6, 1 H). ^{19}F-NMR: −125.5 (*d*, ^2J(F,H) = 54.5, 2 F). ^{13}C-NMR: 41.8 (*t*, ^4J(C,F) = 3.0, C(4)); 92.0 (C(5)); 105.4 (C(3)); 111.3 (*t*, ^1J(C,F) = 251.5, CF_2H); 112.5; 120.5; 125.3; 126.9; 128.0; 128.2; 128.6; 128.7; 130.9; 135.8; 142.6; 144.4; 146.1; 157.8 (C(2)); 181.7 (*t*, ^2J(C,F) = 24.4, C=O). LC/ESI-MS: 393 (100, $[M+H]^+$). Anal. calc. for $C_{24}H_{18}F_2O_3$ (392.40): C 73.46, H 4.62; found: C 73.30, H 4.84.

*1-{4,5-Dihydro-2,5-diphenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}-2,2-difluoroethanone* (**3w**). Yield: 0.161 g (40%). Pale-yellow oil. IR: 3026, 2943, 1620, 1244, 998, 749, 692. ^{1}H-NMR: 3.72 (*d*, *J* = 14.8, 1 H, CH_2(4)); 3.82 (*d*, *J* = 14.8, 1 H, CH_2(4)); 5.8 (*t*, *J* = 53.6, CF_2H); 6.60 (*d*, *J* = 16.0, 1 H); 6.61 (*d*, *J* = 16.0, 1 H); 7.28 (*t*, *J* = 7.2, 1 H); 7.34 (*d*, *J* = 8.0, 2 H); 7.38–7.43 (*m*, 4 H); 7.45 (*t*, *J* = 7.2, 2 H); 7.52–7.57 (*m*, 4 H); 7.83 (*dd*, *J* = 6.8, 1.6, 2 H). ^{19}F-NMR: −125.03 (*d*, ^2J(F,H) = 54.5, 1 F); −125.09 (*d*, ^2J(F,H) = 54.5, 1 F). ^{13}C-NMR: 42.6 (C(4)); 92.2 (C(5)); 109.1 (C(3)); 109.4 (*t*, ^1J(C,F) = 249.9, CHF_2); 125.5; 127.1; 128.3; 128.5; 128.6; 128.9; 129.0; 129.6; 130.5; 130.9; 131.5; 132.0; 136.0; 143.0; 169.6 (C(2)); 183.3 (*t*, ^2J(C,F) = 24.3, C=O). LC/ESI-MS: 403 (100, $[M+H]^+$). Anal. calc. for $C_{26}H_{20}F_2O_2$ (402.43): C 77.60, H 5.01; found: C 77.77, H 5.32.

*{2-(Difluoromethyl)-4,5-dihydro-5-phenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}(furan-2-yl)methanone* (**4a**). Yield: 0.102 g (26%). Pale-yellow oil. IR: 3027, 2974, 2927, 1650 (C=O), 1593 (C=C), 1239, 746, 693. ^{1}H-NMR: 3.82 (*dt*, *J* = 15.2, ^5J(H,F) = 4.4, 1 H, CH_2(4)); 3.92 (*dt*, *J* = 15.2, ^5J(H,F) = 4.8, 1 H, CH_2(4)); 6.51 (*d*, *J* = 16.0, 1 H); 6.62 (*d*, *J* = 16.0, 1 H); 6.56, (*dd*, *J* = 3.6, 1.6, 1 H); 7.19 (*t*, *J* = 52.8, CF_2H); 7.24–7.26 (*m*, 3 H); 7.30 (*tt*, *J* = 8.0, 1.6, 2 H); 7.35–7.40 (*m*, 2 H); 7.42 (*t*, *J* = 8.0, 2 H); 7.51 (*dt*, *J* = 7.2, 1.2, 2 H); 7.59 (*dd*, *J* = 2.0, 0.8, 1 H). ^{19}F-NMR: −125.07 (*dt*, ^2J(F,H) = 53.0, ^5J(F,H) = 4.4, 1 F); −125.17 (*dt*, ^2J(F,H) = 53.0, ^5J(F,H) = 4.4, 1 F). ^{13}C-NMR: 44.2 (C(4)); 93.0 (C(5)); 107.7 (*t*, ^1J(C,F) = 237.7, CHF_2); 112.9, 114.6 (*t*, ^3J(C,F) = 6.1, C(3)); 118.6; 125.4; 127.1; 128.2; 128.5; 128.8; 128.9; 130.3; 131.1; 136.0; 143.0; 146.4; 153.7; 159.0 (*t*, ^2J(C,F) = 22.1, C(2)); 176.4 (C=O). LC/ESI-MS: 393 (100, $[M+H]^+$). Anal. calc. for $C_{24}H_{18}F_2O_3$ (392.40): C 73.46, H 4.62; found: C 73.54, H 4.43.

*{2-(Difluoromethyl)-4,5-dihydro-5-phenyl-5-[(*E*)-2-phenylethenyl]furan-3-yl}(phenyl)methanone* (**4b**). Yield: 0.137 g (34%). Pale-yellow oil. IR: 3026, 2958, 1627 (C=O), 1605 (C=C), 1234, 748, 692. ^{1}H-NMR: 3.60 (*dt*, *J* = 15.2, ^5J(H,F) = 4.8, 1 H, CH_2(4)); 3.67 (*dt*, *J* = 15.2, ^5J(H,F) = 3.6, 1 H, CH_2(4)); 6.40 (*t*, *J* = 52.4, CF_2H); 6.45 (*d*, *J* = 16.0, 1 H); 6.13 (*d*, *J* = 16.0, 1 H); 7.25 (*t*, *J* = 7.2, 1 H); 7.31–7.51 (*m*, 11 H); 7.56 (*tt*, *J* = 6.8, 1.6, 1 H); 7.64 (*dt*, *J* = 6.8, 1.6, 2 H). ^{19}F-NMR: −124.1 (*dt*, ^2J(F,H) = 47.0, ^5J(F,H) = 3.4, 1 F); −124.1 (*dt*, ^2J(F,H) = 47.0, ^5J(F,H) = 3.4, 1 F). ^{13}C-NMR: 45.0 (C(4)); 92.5 (C(5)); 107.2 (*t*, ^1J(C,F) = 237.7, CHF_2); 117.1 (*t*, ^3J(C,F) = 5.4, C(3)); 125.3; 125.5; 127.1; 128.3; 128.3; 128.5; 128.6; 128.8; 128.9; 128.97; 129.0; 129.6; 130.2; 131.0; 131.5; 132.9; 136.0; 139.5; 142.8; 156.1 (*t*, ^2J(C,F) = 22.1, C(2)); 191.1 (C=O). LC/ESI-MS: 403 (100, $[M+H]^+$). Anal. calc. for $C_{26}H_{20}F_2O_2$ (402.43): C 77.60, H 5.01; found: C 77.81, H 4.82.

1-(4,7-Dihydro-2,5,7-triphenyloxepin-3-yl)-2,2-difluoroethanone (**5**). Yield: 0.072 g (18%). Pale-yellow oil. IR: 3058, 2916, 1690 (C=O), 1686 (C=C), 1073, 742, 693. ^{1}H-NMR: 3.12 (*d*, *J* = 16.8, 1 H, CH_2(4)); 4.46 (*dt*, *J* = 16.8, ^5J(H,F) = 2.4, 1 H, CH_2(4)); 4.48 (*t*, *J* = 53.6, CF_2H); 5.52 (*s*, H–C(7)); 6.12 (*s*, H–C(6)); 7.20–7.36 (*m*, 7 H); 7.43–7.50 (*m*, 4 H); 7.56 (*td*, *J* = 7.6, 0.8, 2 H); 7.82 (*d*, *J* = 7.6, 2 H). ^{19}F-NMR: −125.0 (*d*, ^2J(F,H) = 54.5, 1 F); −125.1 (*d*, ^2J(F,H) = 54.5, 1 F). ^{13}C-NMR: 40.5 (C(4)); 57.4 (C(7)); 73.4; 109.5 (*t*, ^1J(C,F) = 252.3, CHF_2); 126.1; 127.5; 128.2; 128.4; 128.7; 129.1; 129.13; 129.5; 130.5; 133.6; 134.8; 135.4; 138.2; 138.8; 193.3 (*d*, ^4J(C,F) = 2.3, C(2)); 198.6 (*dd*, ^2J(C,F) = 29.7, ^2J(C,F) = 20.6, C=O). LC/ESI-MS: 403 (100, $[M+H]^+$). Anal. calc. for $C_{26}H_{20}F_2O_2$ (402.43): C 77.60, H 5.01; found: C 77.46, H 5.23.

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