



# Highly stereoselective synthesis of *trans*-4-trifluoromethylsulfonyl-2,3-dihydrofurans from arsonium ylides and (*E*)- $\alpha$ -trifluoromethylsulfonyl- $\alpha,\beta$ -unsaturated ketones

Zhenqi Chen <sup>a,†</sup>, Jiapeng Zhang <sup>a,†</sup>, Jie Chen <sup>a</sup>, Hongmei Deng <sup>d</sup>, Min Shao <sup>d</sup>, Hui Zhang <sup>a,\*</sup>, Weiguo Cao <sup>a,b,c,\*</sup>

<sup>a</sup> Department of Chemistry, Shanghai University, Shanghai 200444, PR China

<sup>b</sup> State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, PR China

<sup>c</sup> Key Laboratory of Organofluorine Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, PR China

<sup>d</sup> Instrumental Analysis and Research Center of Shanghai University, Shanghai 200444, PR China

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## ABSTRACT

The reactions of arsonium bromides with (*E*)- $\alpha$ -trifluoromethylsulfonyl- $\alpha,\beta$ -unsaturated ketones in the presence of  $\text{Cs}_2\text{CO}_3$  or  $\text{K}_2\text{CO}_3$  proceeded smoothly under refluxing condition in dichloromethane (DCM) to give the corresponding trifluoromethylated *trans*-2,3-dihydrofurans in good to excellent yields with high stereoselectivity.

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## 1. Introduction

Dihydrofurans are an important heterocyclic scaffold, used for the construction of a variety of naturally occurring substances that generally possess a diversity of biological activities.<sup>1,2</sup> Moreover, some dihydrofuran derivatives substituted with polyfluoroalkanesulfonyl groups have attracted considerable interest recently because the polyfluoroalkanesulfonyl groups,  $R_f\text{SO}_2$ , are one of the strongest electron-withdrawing groups, which can activate R—C—H bonds and adjacent olefins or function as nucleophilic leaving groups having an electron pair to form sulfinate anions.<sup>3</sup> Due to their manifold reactivities, polyfluoroalkanesulfonyl groups are of special interest in organic chemistry, especially in organofluorine chemistry.  $\alpha$ -Polyfluoroalkanesulfonyl acetate esters are moderately active methylene compounds that are widely used in the synthesis of heterocycles and unsaturated sulfonyl esters.<sup>4</sup>

For these reasons, the development of new and efficient methods for synthesis of substituted dihydrofurans remains an area of keen interest.<sup>5</sup> To date, a number of methods have been devised for the synthesis of dihydrofuran derivatives containing sulfonyl groups, especially polyfluoroalkanesulfonyl groups.<sup>6</sup>

Although it is well known that the introduction of polyfluoroalkanesulfonyl groups into organic molecules can bring about some remarkable changes in their properties, methods for preparing dihydrofurans with the polyfluoroalkanesulfonyl group still remain very limited.<sup>3,4,7</sup>

In 1996, Moorhoff first reported the synthesis of dihydrofurans by the reaction of arsonium ylides with  $\alpha,\beta$ -unsaturated ketones.<sup>8</sup> During the same period, our group reported a similar reaction, but with higher chemoselectivity and formation of a single stereoisomer.<sup>9</sup> Subsequently, a series of approaches to dihydrofurans have been published by our group including the stereoselective synthesis of dihydrofurans substituted with a sulfonyl moiety.<sup>10</sup> Yang et al. have utilized an ammonium ylide instead of an arsonium one and found that the reaction gave high chemical yield, but produced a mixture of the cis and trans isomers.<sup>11</sup>

As part of our ongoing efforts in developing synthetic approaches for the synthesis of novel fluorinated heterocycles with potential biological applications,<sup>12</sup> we built upon the reports by Zhu's group

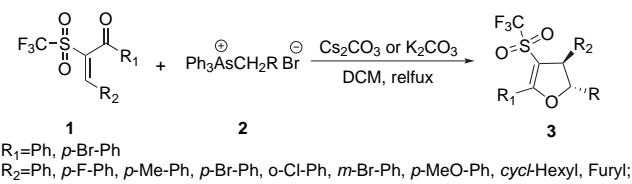
\* Corresponding authors. Fax: +86 21 6613 4856; e-mail addresses: wgciao@staff.shu.edu.cn (H. Zhang), yehao7171@shu.edu.cn (W. Cao).

† With equal contribution to this work.

on the synthesis of the tetrasubstituted *trans*-2,3-dihydrofurans.<sup>6c</sup> It was found that although the reaction between  $\beta$ -poly-fluoroalkanesulfones, such as  $R_FSO_2CH_2COR$  ( $R_F=ClC_4F_8$ ,  $R=Ph$  or  $CH_3$ ) and aromatic aldehydes gave the unexpected formation of tetrasubstituted *trans*-2,3-dihydrofurans instead of the (*E*)- $\alpha$ -trifluoromethylsulfonyl- $\alpha,\beta$ -unsaturated ketones under Knoevenagel condensation reaction conditions, the four substituted groups of products could not have various selections conveniently because of the coherent Knoevenagel reaction and Michael addition composing the reaction together.<sup>6c</sup> On considering the high electronegativity of polyfluoroalkanesulfonyl groups, here we report a convenient and highly stereoselective synthesis of trifluoromethylated *trans*-2,3-dihydrofurans via an [1+4] reaction of arsonium ylides with trifluoromethylated  $\alpha,\beta$ -unsaturated ketones.

## 2. Results and discussion

The substituted *trans*-2,3-dihydrofurans containing trifluoromethyl group were obtained via an [1+4] reaction of arsonium ylides with trifluoromethylated- $\alpha,\beta$ -unsaturated ketones in good to excellent yields with high stereoselectivity (Scheme 1). The structures of the products were confirmed by IR,  $^1H$  NMR,  $^{13}C$  NMR,  $^{19}F$  NMR, MS, and X-ray diffraction analysis as well.



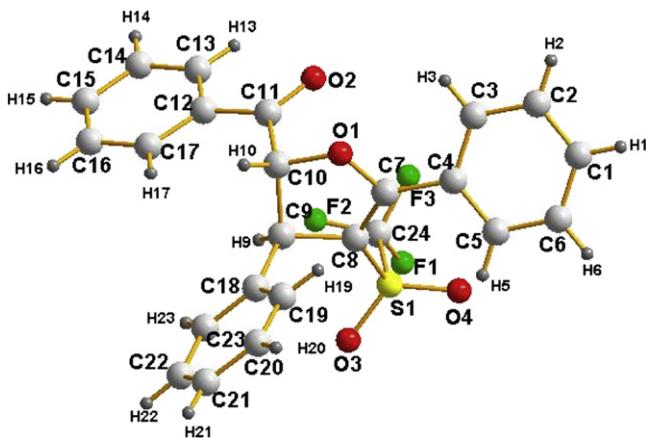
**Scheme 1.** Preparation of *trans*-4-trifluoromethylsulfonyl-2,3-dihydrofurans.

Different solvents were first examined by using the reaction of (*E*)-1, 3-diphenyl-2-trifluoromethylsulfonylprop-2-en-1-one **1A** (1.0 equiv) with cyanomethyltriphenylarsonium bromide **2a** (1.2 equiv) as a model with  $K_2CO_3$  (2.0 equiv) as base. The reaction completed in  $CH_2Cl_2$  within 5 h at room temperature giving the corresponding *trans*-2,3-dihydrofuran **3Aa** in 71% yield (Table 1, entry 1). When the reaction was carried out under refluxing condition, it completed within 3 h, giving corresponding products **3Aa** in 89% yield (Table 1, entry 2). Both  $CHCl_3$  and DMSO were proved to be good solvents (87% yield in  $CHCl_3$ , refluxing condition; 85% yield in DMSO, 80 °C, Table 1, entries 3 and 4), while in THF, the yield was only 78% (Table 1, entry 5). The reaction yield remained unchanged when the amount of  $K_2CO_3$  was increased. However, decreased the amount of base lowered the yield.

Thus, the optimal reaction conditions were 1.0 equiv of **1**, 1.2 equiv of **2**, 2.0 equiv of  $K_2CO_3$  in  $CH_2Cl_2$  at refluxing temperature (40 °C) for 3 h.

Under the above reaction conditions, the scope and generality of the process were explored. The corresponding trifluoromethylated *trans*-2,3-dihydrofurans **3A** were obtained in good to excellent yields (Table 1, entries 6–12).

The five-membered heterocyclic structure of **3Ab** (Fig. 1<sup>13</sup>) was established by single-crystal X-ray diffraction analysis indicating a *trans* configuration at C-2 and C-3 positions. Moreover, the coupling constant of two adjacent protons was 3.3 Hz in  $^1H$  NMR of **3Ab**, which further confirmed the *trans* configuration,<sup>6c,12</sup> while the coupling constant between the two protons in the *cis*-isomers (*cis*-**3Ad**, *cis*-**3Ag**, *cis*-**3Bd** and *cis*-**3Bg**) was found to be over 9.0 Hz.<sup>12d</sup>



**Figure 1.** X-ray diffraction of **3Ab**.

It is noteworthy that the yield of *trans*-**3Ad** and *trans*-**3Ag** was rather low under these reaction conditions due to the formation of the mixtures of isomers. But when we replaced  $K_2CO_3$  with  $Cs_2CO_3$ , the reaction was completed in  $CH_2Cl_2$  under refluxing condition within 1.5 h, giving the corresponding *trans*-**3Ad** in 81% yield and *cis*-**3Ad** in 8% (Table 2, entry 1). The other reactions were also examined again with  $Cs_2CO_3$  as base (Table 2, entries 2–7). After changing the base, not only the stereoselectivity of some reactions were improved, but also the reaction time was shortened. The distinguishing feature of the reaction is its high stereoselectivity: only one isomer containing stronger electron-withdrawing substituent at C-4 position of the *trans*-2,3-dihydrofurans is formed in most reactions, which could be explained by our previous reported result.<sup>12</sup>

**Table 1**

Reaction of (*E*)-1,3-diphenyl-2-trifluoromethylsulfonylprop-2-en-1-one **1A** (1.0 equiv) with arsonium bromides **2** (1.2 equiv) in the presence of  $K_2CO_3$  (2.0 equiv)

Entry	<b>1</b>	$R_1$	$R_2$	<b>2</b>	$R$	Product <b>3</b>	Solvent	Temperature (°C)	Time (h)	Yield <sup>a</sup> (%)
1	<b>1A</b>	Ph	Ph	<b>2a</b>	CN	<b>3Aa</b>	$CH_2Cl_2$	rt	5	71
2	<b>1A</b>	Ph	Ph	<b>2a</b>	CN	<b>3Aa</b>	$CH_2Cl_2$	Reflux	3	89
3	<b>1A</b>	Ph	Ph	<b>2a</b>	CN	<b>3Aa</b>	$CHCl_3$	Reflux	4	87
4	<b>1A</b>	Ph	Ph	<b>2a</b>	CN	<b>3Aa</b>	DMSO	80 °C	4	85
5	<b>1A</b>	Ph	Ph	<b>2a</b>	CN	<b>3Aa</b>	THF	Reflux	5	78
6	<b>1A</b>	Ph	Ph	<b>2b</b>	COPh	<b>3Ab</b>	$CH_2Cl_2$	Reflux	2.5	89
7	<b>1A</b>	Ph	Ph	<b>2c</b>	$CO_2Me$	<b>3Ac</b>	$CH_2Cl_2$	Reflux	2.5	90
8	<b>1A</b>	Ph	Ph	<b>2d</b>	Ph	<i>trans</i> - <b>3Ad</b>	$CH_2Cl_2$	Reflux	2	50
	<b>1A</b>	Ph	Ph	<b>2d</b>	Ph	<i>cis</i> - <b>3Ad</b>	$CH_2Cl_2$	Reflux	2	32
9	<b>1A</b>	Ph	Ph	<b>2e</b>	$CO_2Me$	<b>3Ae</b>	$CH_2Cl_2$	Reflux	2.5	86
10	<b>1A</b>	Ph	Ph	<b>2f</b>	Vinyl	<b>3Af</b>	$CH_2Cl_2$	Reflux	3	84
11	<b>1A</b>	Ph	Ph	<b>2g</b>	<i>p</i> -Cl-Ph	<i>trans</i> - <b>3Ag</b>	$CH_2Cl_2$	Reflux	2	48
	<b>1A</b>	Ph	Ph	<b>2g</b>	<i>p</i> -Cl-Ph	<i>cis</i> - <b>3Ag</b>	$CH_2Cl_2$	Reflux	2	34

<sup>a</sup> Isolated yield.

**Table 2**Reaction of (*E*)-1,3-diphenyl-2-trifluoromethylsulfonylprop-2-en-1-one **1A** (1.0 equiv) with arsonium bromides **2** (1.2 equiv) in the presence of  $\text{Cs}_2\text{CO}_3$  (2.0 equiv)

Entry	<b>1</b>	$R_1$	$R_2$	<b>2</b>	R	Product <b>3</b>	Solvent	Temperature (°C)	Time (h)	Yield <sup>a</sup> (%)
1	<b>1A</b>	Ph	Ph	<b>2a</b>	CN	<b>3Aa</b>	$\text{CH}_2\text{Cl}_2$	Reflux	3	90
2	<b>1A</b>	Ph	Ph	<b>2b</b>	COPh	<b>3Ab</b>	$\text{CH}_2\text{Cl}_2$	Reflux	2	92
3	<b>1A</b>	Ph	Ph	<b>2c</b>	$\text{CO}_2\text{Me}$	<b>3Ac</b>	$\text{CH}_2\text{Cl}_2$	Reflux	2	93
4	<b>1A</b>	Ph	Ph	<b>2d</b>	Ph	<i>trans</i> - <b>3Ad</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1.5	81
	<b>1A</b>	Ph	Ph	<b>2d</b>	Ph	<i>cis</i> - <b>3Ad</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1.5	8
5	<b>1A</b>	Ph	Ph	<b>2e</b>	$\text{COMe}$	<b>3Ae</b>	$\text{CH}_2\text{Cl}_2$	Reflux	2	89
6	<b>1A</b>	Ph	Ph	<b>2f</b>	Vinyl	<b>3Af</b>	$\text{CH}_2\text{Cl}_2$	Reflux	2.5	86
7	<b>1A</b>	Ph	Ph	<b>2g</b>	<i>p</i> -Cl-Ph	<i>trans</i> - <b>3Ag</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1.5	82
	<b>1A</b>	Ph	Ph	<b>2g</b>	<i>p</i> -Cl-Ph	<i>cis</i> - <b>3Ag</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1.5	6

<sup>a</sup> Isolated yield.

To further explore the generality of this improved methodology, we subjected the less reactive (*E*)-3-furyl-2-trifluoromethylsulfonyl-1-phenylprop-2-en-1-one to the reaction. In  $\text{CH}_2\text{Cl}_2$ , the reaction of (*E*)-3-furyl-2-trifluoromethylsulfonyl-1-phenylprop-2-en-1-one **1B** with cyanomethyltriphenylarsonium bromide **2a** in the presence of  $\text{Cs}_2\text{CO}_3$  gave **3Ba** with relatively lower yield (85%). However, it only took 1.5 h to complete the reaction. It was found that several other arsonium ylides could also react with (*E*)-3-furyl-2-trifluoromethylsulfonyl-1-phenylprop-2-en-1-one **1B** to give the corresponding *trans*-2,3-dihydrofurans **3B** as the only stereoisomer in good to excellent yields (Table 3).

intermediate **I**. An intramolecular nucleophilic attack of the oxygen enolate to the pendent arsonium-containing carbon in the trifluoromethylsulfonyl-stabilized enolate intermediate **I** containing chiral carbon atoms **C**<sub>2</sub> and **C**<sub>3</sub> should result in the two isomers **3** and **3'**. Two possible scenarios occurred when the enolate oxygen attacked **C**<sub>2</sub> from the backside of leaving group ( $\text{Ph}_3\text{As}$ ), such as **II** and **III** as shown in Scheme 2. Intermediate **II** was relatively more stable than **III** owing to its relief of the repulsion of two large groups ( $R_2$  and R). The preferred reactive conformer **II** would generate *trans*-2,3-dihydrofuran **3**. Alternatively, when arsonium ylides, derived from **2d** (benzyltriphenylarsonium bromide) and **2g**

**Table 3**Reaction of (*E*)-3-furyl-2-trifluoromethylsulfonyl-1-phenylprop-2-en-1-one **1B** (1.0 equiv) with arsonium bromides **2** (1.2 equiv) in the presence of  $\text{Cs}_2\text{CO}_3$  (2.0 equiv)

Entry	<b>1</b>	$R_1$	$R_2$	<b>2</b>	R	Product <b>3</b>	Solvent	Temperature (°C)	Time (h)	Yield <sup>a</sup> (%)
1	<b>1B</b>	Ph	Furyl	<b>2a</b>	CN	<b>3Ba</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1.5	85
2	<b>1B</b>	Ph	Furyl	<b>2b</b>	COPh	<b>3Bb</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1	87
3	<b>1B</b>	Ph	Furyl	<b>2c</b>	$\text{CO}_2\text{Me}$	<b>3Bc</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1	88
4	<b>1B</b>	Ph	Furyl	<b>2d</b>	Ph	<i>trans</i> - <b>3Bd</b>	$\text{CH}_2\text{Cl}_2$	Reflux	0.5	77
	<b>1B</b>	Ph	Furyl	<b>2d</b>	Ph	<i>cis</i> - <b>3Bd</b>	$\text{CH}_2\text{Cl}_2$	Reflux	0.5	7
5	<b>1B</b>	Ph	Furyl	<b>2e</b>	$\text{COMe}$	<b>3Be</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1	84
6	<b>1B</b>	Ph	Furyl	<b>2f</b>	Vinyl	<b>3Bf</b>	$\text{CH}_2\text{Cl}_2$	Reflux	1.5	82
7	<b>1B</b>	Ph	Furyl	<b>2g</b>	<i>p</i> -Cl-Ph	<i>trans</i> - <b>3Bg</b>	$\text{CH}_2\text{Cl}_2$	Reflux	0.5	78
	<b>1B</b>	Ph	Furyl	<b>2g</b>	<i>p</i> -Cl-Ph	<i>cis</i> - <b>3Bg</b>	$\text{CH}_2\text{Cl}_2$	Reflux	0.5	5

<sup>a</sup> Isolated yield.

Furthermore, this method also has the generality in the reactions of different (*E*)- $\alpha$ -trifluoromethylsulfonyl- $\alpha,\beta$ -unsaturated ketones with arsonium ylide derived from **2a** to give the corresponding *trans*-2,3-dihydrofurans **3Ca**–**3Ja** with the similar stereoselectivity in good to excellent yields (Table 4).

(*p*-chlorobenzyltriphenylarsonium bromide), reacted with  $\alpha,\beta$ -unsaturated ketones, *cis*-2,3-dihydrofuran **3'** was formed as minor products together with the major product *trans*-2,3-dihydrofuran **3**.

The exact reason why the reactions of ylides derived from **2d** (benzyltriphenylarsonium bromide) and **2g** (*p*-chlorobenzyl-

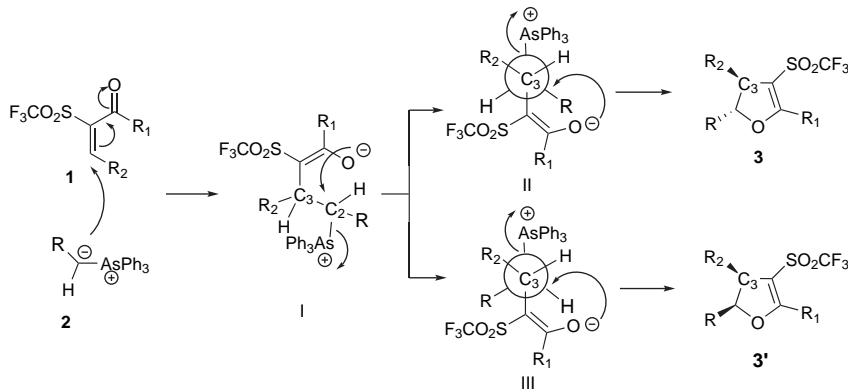
**Table 4**Reaction of (*E*)- $\alpha$ -trifluoromethylsulfonyl- $\alpha,\beta$ -unsaturated ketones **1** (1.0 equiv) with arsonium bromide **2a** (1.2 equiv) in the presence of  $\text{Cs}_2\text{CO}_3$  (2.0 equiv)

Entry	<b>1</b>	$R_1$	$R_2$	<b>2</b>	R	Product <b>3</b>	Solvent	Temperature (°C)	Time (h)	Yield <sup>a</sup> (%)
1	<b>1C</b>	Ph	<i>o</i> -Cl-Ph	<b>2a</b>	CN	<b>3Ca</b>	$\text{CH}_2\text{Cl}_2$	Reflux	3	85
2	<b>1D</b>	Ph	<i>m</i> -Br-Ph	<b>2a</b>	CN	<b>3Da</b>	$\text{CH}_2\text{Cl}_2$	Reflux	3	84
3	<b>1E</b>	Ph	<i>p</i> -Br-Ph	<b>2a</b>	CN	<b>3Ea</b>	$\text{CH}_2\text{Cl}_2$	Reflux	3	87
4	<b>1F</b>	Ph	<i>p</i> -CH <sub>3</sub> O-Ph	<b>2a</b>	CN	<b>3Fa</b>	$\text{CH}_2\text{Cl}_2$	Reflux	2	86
5	<b>1G</b>	Ph	<i>p</i> -F-Ph	<b>2a</b>	CN	<b>3Ga</b>	$\text{CH}_2\text{Cl}_2$	Reflux	2.5	90
6	<b>1H</b>	Ph	<i>p</i> -CH <sub>3</sub> -Ph	<b>2a</b>	CN	<b>3Ha</b>	$\text{CH}_2\text{Cl}_2$	Reflux	2	87
7	<b>1I</b>	Ph	cycl-C <sub>6</sub> H <sub>12</sub>	<b>2a</b>	CN	<b>3Ia</b>	$\text{CH}_2\text{Cl}_2$	Reflux	3	85
8	<b>1J</b>	<i>p</i> -Br-Ph	Ph	<b>2a</b>	CN	<b>3Ja</b>	$\text{CH}_2\text{Cl}_2$	Reflux	3	90

<sup>a</sup> Isolated yield.

On the basis of the previous reports,<sup>12</sup> the proposed reaction mechanism shown in Scheme 2 might account for the stereoselective formation of the *trans*-substituted dihydrofuran products. Initial attack of the  $\alpha,\beta$ -unsaturated ketone **1** via the carbanion derived from arsonium bromide and base produced an

triphenylarsonium bromide) with **1** would generate both *cis* and *trans* isomers are still unknown. One reasonable explanation is that these two ylides have relatively higher reactivity than others in which there are no electron-withdrawing groups in their structures to stabilize carbon anion. When they were added into the reaction,



Scheme 2. Mechanism for the formation of product 3 or 3'.

the reaction will be faster and the reaction time will also be shorter than others, which can be confirmed by the data in Table 2 and 3. So it is also easy to generate both intermediates II and III to obtain both cis and trans isomers together.

### 3. Conclusions

In conclusion, we have developed a convenient and highly stereoselective method for the synthesis of trifluoromethylated *trans*-2,3-dihydrofurans via an [1+4] reaction of arsonium ylides with trifluoromethylated  $\alpha,\beta$ -unsaturated ketones. Compared to previous methods, this technique is simple, experimentally convenient and proceeds smoothly under mild conditions in the presence of  $\text{Cs}_2\text{CO}_3$  or  $\text{K}_2\text{CO}_3$ . The corresponding trifluoromethylated *trans*-2,3-dihydrofurans were obtained in good to excellent yields with high stereoselectivity. Efforts to explore further applications of these products are currently in progress in our laboratory.

## 4. Experimental

### 4.1. General information

All reagents and solvents were purchased from commercial sources and used without further purification, except that (*E*)- $\alpha$ -trifluoroalkanesulfonyl- $\alpha,\beta$ -unsaturated ketones **1** and arsonium ylides **2** were prepared according to the reported literatures.<sup>7,14,15</sup> Melting points were uncorrected.  $^1\text{H}$ ,  $^{19}\text{F}$ , and  $^{13}\text{C}$  NMR spectra were recorded on a 500 MHz spectrometer. All chemical shifts are reported in parts per million downfield (positive) of the standard:  $\text{C}_6\text{F}_6$  for  $^{19}\text{F}$ , TMS for  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra. IR spectra were obtained on a FT-IR spectrometer. Elemental analysis was performed on an elemental analysis instrument. MS (ESI) was run on a mass spectrometer and MS (EI) on a mass spectrometer. X-ray analysis was performed on an X-ray spectrometer. Preparative TLC on silica gel was performed by using self-coated GF<sub>254</sub> plates, which were activated immediately before use.

### 4.2. General procedure for preparation of compound 3

To the solution of (*E*)- $\alpha$ -trifluoromethylsulfonyl- $\alpha,\beta$ -unsaturated ketones<sup>14a</sup> **1** (1.0 mmol) in  $\text{CH}_2\text{Cl}_2$  (10 mL), arsonium bromides **2** (1.2 mmol), and  $\text{Cs}_2\text{CO}_3$  (2 mmol) were added and the mixture was stirred at refluxing temperature for 2 h. After the completion of the reaction (monitored by TLC), the products were purified by preparative TLC [eluent: petroleum ether (60–90 °C)–ethyl acetate] to give pure product **3**.

**4.2.1. 2-Cyano-3,5-diphenyl-4-trifluoromethylsulfonyl-*trans*-2,3-dihydrofuran (3Aa).** Oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.85 (1H, d,  $J=3.1$  Hz, CH), 5.34 (1H, d,  $J=3.1$  Hz, CH), 7.31–7.88 (10H, m, ArH).  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.08 (s, 3F).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  55.48, 75.42, 106.58 (C<sub>3</sub>, d,  $^3J_{\text{F}-\text{C}}=2.1$  Hz), 115.42, 119.45 (CF<sub>3</sub>, q,  $^1J_{\text{F}-\text{C}}=324.3$  Hz), 125.33, 127.07, 128.50, 129.42, 129.67, 130.39, 133.69, 137.61, 172.09. MS (EI)  $m/z$  (%): 379 (4), 310 (7), 119 (5), 105 (9), 86 (65), 84 (100), 77 (5), 51 (6). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3444, 3070, 2925, 1701, 1611, 1369. Anal. Calcd for  $\text{C}_{18}\text{H}_{12}\text{F}_3\text{NO}_3\text{S}$ : C, 56.99; H, 3.19; N, 3.69. Found: C, 56.82; H, 3.36; N, 3.77.

**4.2.2. 2-Benzoyl-3,5-diphenyl-4-trifluoromethylsulfonyl-*trans*-2,3-dihydrofuran (3Ab).** A white solid: mp: 139.4–140.0 °C;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.64 (1H, d,  $J=3.3$  Hz, CH), 6.05 (1H, d,  $J=3.3$  Hz, CH), 7.41–8.02 (15H, m, ArH).  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.51 (s, 3F).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  53.22, 89.74, 105.06 (C<sub>3</sub>, d,  $^3J_{\text{F}-\text{C}}=1.5$  Hz), 120.01 (CF<sub>3</sub>, q,  $^1J_{\text{F}-\text{C}}=324.9$  Hz), 126.68, 127.46, 128.18, 128.89, 129.29, 129.54, 130.54, 132.43, 132.96, 134.70, 139.95, 174.86, 191.53. MS (EI)  $m/z$  (%): 458 (1), 389 (5), 340 (79), 191 (20), 105 (100), 77 (38). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3421, 3063, 2921, 1757, 1612, 1364. Anal. Calcd for  $\text{C}_{24}\text{H}_{17}\text{F}_3\text{O}_4\text{S}$ : C, 62.88; H, 3.74. Found: C, 62.78; H, 3.96.

**4.2.3. 2-Methoxycarbonyl-3,5-diphenyl-4-trifluoromethylsulfonyl-*trans*-2,3-dihydrofuran (3Ac).** Oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  3.92 (s, 3H), 4.66 (1H, d,  $J=2.9$  Hz, CH), 5.15 (1H, d,  $J=2.9$  Hz, CH), 7.36–7.93 (10H, m, ArH).  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.60 (s, 3F).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  53.28, 53.95, 86.19, 105.01 (C<sub>3</sub>, d,  $^3J_{\text{F}-\text{C}}=1.5$  Hz), 120.04 (CF<sub>3</sub>, q,  $^1J_{\text{F}-\text{C}}=326.5$  Hz), 127.05, 128.20, 128.58, 129.34, 129.67, 130.45, 133.08, 139.83, 168.90, 174.24. MS (EI)  $m/z$  (%): 412 (3), 343 (11), 279 (20), 191 (11), 105 (20), 84 (100), 77 (9). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3060, 2922, 1731, 1609, 1366. Anal. Calcd for  $\text{C}_{19}\text{H}_{15}\text{F}_3\text{O}_5\text{S}$ : C, 55.34; H, 3.67. Found: C, 55.29; H, 3.78.

**4.2.4. 2,3,5-Triphenyl-4-trifluoromethylsulfonyl-*trans*-2,3-dihydrofuran (trans-3Ad).** Oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.55 (1H, d,  $J=4.8$  Hz, CH), 5.72 (1H, d,  $J=4.8$  Hz, CH), 7.35–7.90 (15H, m, ArH).  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.18 (s, 3F).  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  51.60, 87.85, 101.81 (C<sub>3</sub>, d,  $^3J_{\text{F}-\text{C}}=1.5$  Hz), 115.42, 119.96 (CF<sub>3</sub>, q,  $^1J_{\text{F}-\text{C}}=325.6$  Hz), 126.48, 126.82, 127.50, 128.20, 129.47, 129.81, 132.72, 137.05, 140.12, 174.30. MS (EI)  $m/z$  (%): 430 (1), 297 (2), 191 (6), 122 (66), 105 (100), 77 (60), 57 (26). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3442, 3063, 2921, 1730, 1607, 1365. Anal. Calcd for  $\text{C}_{23}\text{H}_{17}\text{F}_3\text{O}_3\text{S}$ : C, 64.18; H, 3.98. Found: C, 64.32; H, 3.92.

**4.2.5. 2,3,5-Triphenyl-4-trifluoromethylsulfonyl-*cis*-2,3-dihydrofuran (cis-3Ad).** Oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.76 (1H, d,  $J=9.1$  Hz, CH), 6.28 (1H, d,  $J=9.1$  Hz, CH), 6.96–7.96 (15H, m, ArH).

<sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.48 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  52.77, 83.56, 101.90 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.5$  Hz), 120.11 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=325.5$  Hz), 126.59, 126.93, 127.47, 127.90, 128.31, 128.77, 129.60, 130.08, 136.04, 137.88, 174.42. MS (EI)  $m/z$  (%): 430 (13), 361 (2), 297 (19), 191 (7), 120 (45), 105 (100), 77 (61), 51 (19). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3441, 3065, 2921, 1728, 1606, 1351. Anal. Calcd for  $\text{C}_{23}\text{H}_{17}\text{F}_3\text{O}_3\text{S}$ : C, 64.18; H, 3.98. Found: C, 64.02; H, 3.83.

**4.2.6. 2-Acetyl-3,5-diphenyl-4-trifluoromethylsulfonyl-trans-2,3-dihydrofuran (3Ae).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  2.43 (s, 3H), 4.69 (1H, d,  $J=3.8$  Hz, CH), 5.10 (1H, d,  $J=3.8$  Hz, CH), 7.38–7.93 (10H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.41 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  29.93, 53.48, 86.39, 105.21 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.5$  Hz), 120.24 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=325.1$  Hz), 126.62, 127.25, 128.40, 129.54, 130.65, 133.28, 140.03, 174.44, 197.66. MS (EI)  $m/z$  (%): 396 (2), 353 (4), 286 (100), 263 (33), 191 (19), 105 (39), 77 (23), 57 (18). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3439, 3065, 2921, 1731, 1610, 1366. Anal. Calcd for  $\text{C}_{19}\text{H}_{15}\text{F}_3\text{O}_4\text{S}$ : C, 57.57; H, 3.81. Found: C, 57.75; H, 3.98.

**4.2.7. 2-Vinyl-3,5-diphenyl-4-trifluoromethylsulfonyl-trans-2,3-dihydrofuran (3Af).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.34 (1H, d,  $J=4.0$  Hz, CH), 5.21 (1H, dd,  $^1J=6.5$  Hz,  $^2J=4.0$  Hz, CH), 5.44 (1H, d,  $J=10.5$  Hz, =CH), 5.49 (1H, d,  $J=17.5$  Hz, =CH), 6.12 (1H, ddd,  $^1J=17.5$  Hz,  $^2J=10.5$  Hz,  $^3J=6.5$  Hz, =CH), 7.34–7.86 (10H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.34 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  55.21, 86.14, 105.32 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.6$  Hz), 118.68, 119.98 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=325.5$  Hz), 126.98, 127.39, 127.96, 128.52, 128.84, 129.91, 133.03, 141.22, 172.71. MS (EI)  $m/z$  (%): 380 (14), 311 (7), 289 (12), 247 (16), 191 (56), 105 (100), 77 (44). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3443, 3023, 2961, 1728, 1605, 1355. Anal. Calcd for  $\text{C}_{19}\text{H}_{15}\text{F}_3\text{O}_3\text{S}$ : C, 59.99; H, 3.97. Found: C, 60.17; H, 3.85.

**4.2.8. 2-(4-Chlorophenyl)-3,5-diphenyl-4-trifluoromethylsulfonyl-trans-2,3-dihydrofuran (trans-3Ag).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.50 (1H, d,  $J=4.8$  Hz, CH), 5.68 (1H, d,  $J=4.8$  Hz, CH), 7.30–7.88 (14H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.14 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  52.88, 82.79, 101.83 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.4$  Hz), 120.20 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=324.9$  Hz), 125.61, 127.47, 128.30, 128.75, 129.09, 129.47, 130.02, 132.92, 135.72, 137.54, 174.69. MS (EI)  $m/z$  (%): 464 (1), 340 (29), 207 (13), 178 (10), 105 (100), 77 (42), 51 (10). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3441, 3065, 2921, 1714, 1696, 1351. Anal. Calcd for  $\text{C}_{23}\text{H}_{16}\text{ClF}_3\text{O}_3\text{S}$ : C, 59.42; H, 3.47. Found: C, 59.68; H, 3.76.

**4.2.9. 2-(4-Chlorophenyl)-3,5-diphenyl-4-trifluoromethylsulfonyl-cis-2,3-dihydrofuran (cis-3Ag).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.74 (1H, d,  $J=9.1$  Hz, CH), 6.23 (1H, d,  $J=9.1$  Hz, CH), 6.90–7.95 (14H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.45 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  51.59, 87.04, 101.65 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.5$  Hz), 120.02 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=325.1$  Hz), 126.34, 127.04, 127.75, 128.16, 128.54, 128.88, 129.86, 132.60, 135.14, 138.60, 140.22, 174.51. MS (EI)  $m/z$  (%): 464 (2), 340 (34), 207 (14), 178 (10), 105 (100), 77 (44), 51 (13). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3437, 3064, 2920, 1722, 1608, 1363. Anal. Calcd for  $\text{C}_{23}\text{H}_{16}\text{ClF}_3\text{O}_3\text{S}$ : C, 59.42; H, 3.47. Found: C, 59.22; H, 3.59.

**4.2.10. 2-Cyano-3-furyl-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (3Ba).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  5.03 (1H, d,  $J=3.8$  Hz, CH), 5.49 (1H, d,  $J=3.8$  Hz, CH), 6.41–6.47 (2H, m, ArH), 7.45–7.81 (6H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.38 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  48.96, 72.18, 103.45 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.6$  Hz), 110.03, 111.20, 115.04, 119.90 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=324.3$  Hz), 128.42, 130.24, 133.61, 143.86, 148.11, 172.86. MS (EI)  $m/z$  (%): 369 (56), 342 (3), 300 (37), 217 (29), 181 (41), 152 (34), 105 (100), 77 (64). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3442, 3068, 2926, 1731, 1609,

1370. Anal. Calcd for  $\text{C}_{16}\text{H}_{10}\text{F}_3\text{NO}_4\text{S}$ : C, 52.03; H, 2.73; N, 3.79. Found: C, 51.83; H, 2.96; N, 3.54.

**4.2.11. 2-Benzoyl-3-furyl-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (3Bb).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.92 (1H, d,  $J=3.7$  Hz, CH), 6.18 (1H, d,  $J=3.7$  Hz, CH), 6.45–6.48 (2H, m, ArH), 7.35–8.03 (11H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.70 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  54.41, 88.67, 104.58 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.5$  Hz), 108.02, 110.73, 119.96 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=324.8$  Hz), 127.69, 128.10, 128.47, 129.06, 130.35, 133.16, 134.56, 143.11, 151.04, 174.47, 192.24. MS (EI)  $m/z$  (%): 448 (4), 379 (2), 343 (2), 315 (16), 181 (100), 105 (65), 77 (56), 51 (46). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3441, 3063, 2957, 1707, 1665, 1384. Anal. Calcd for  $\text{C}_{22}\text{H}_{15}\text{F}_3\text{O}_5\text{S}$ : C, 58.93; H, 3.37. Found: C, 58.67; H, 3.12.

**4.2.12. 2-Methoxycarbonyl-3-furyl-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (3Bc).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  3.91 (s, 3H), 4.87 (1H, d,  $J=3.7$  Hz, CH), 6.14 (1H, d,  $J=3.7$  Hz, CH), 6.41–6.45 (2H, m, ArH), 7.42–7.99 (6H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.73 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  48.26, 52.74, 86.76, 102.40 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.5$  Hz), 108.22, 110.68, 120.12 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=324.8$  Hz), 127.50, 128.68, 130.28, 143.76, 151.39, 170.07, 174.32. MS (EI)  $m/z$  (%): 402 (13), 343 (18), 333 (8), 269 (30), 221 (16), 181 (100), 105 (33), 77 (53). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3429, 3066, 2957, 1760, 1608, 1366. Anal. Calcd for  $\text{C}_{17}\text{H}_{13}\text{F}_3\text{O}_6\text{S}$ : C, 50.75; H, 3.26. Found: C, 50.89; H, 3.08.

**4.2.13. 2,5-Diphenyl-3-furyl-4-trifluoromethylsulfonyl-trans-2,3-dihydrofuran (trans-3Bd).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.74 (1H, d,  $J=5.8$  Hz, CH), 5.89 (1H, d,  $J=5.8$  Hz, CH), 6.36–6.43 (2H, m, ArH), 7.39–7.88 (11H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.59 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  51.67, 88.78, 101.73 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.4$  Hz), 108.48, 110.88, 120.10 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=324.4$  Hz), 125.13, 127.10, 128.22, 129.31, 129.37, 129.92, 132.67, 138.68, 143.14, 151.80, 174.60. MS (EI)  $m/z$  (%): 420 (25), 287 (15), 256 (24), 181 (100), 177 (83), 105 (65), 84 (89). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3440, 3059, 2929, 1809, 1604, 1363. Anal. Calcd for  $\text{C}_{21}\text{H}_{15}\text{F}_3\text{O}_4\text{S}$ : C, 60.00; H, 3.60. Found: C, 60.29; H, 3.28.

**4.2.14. 2,5-Diphenyl-3-furyl-4-trifluoromethylsulfonyl-cis-2,3-dihydrofuran (cis-3Bd).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.75 (1H, d,  $J=9.1$  Hz, CH), 5.79 (1H, d,  $J=9.1$  Hz, CH), 6.12–6.22 (2H, m, ArH), 7.12–7.92 (11H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.51 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  52.64, 84.53, 101.91 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.5$  Hz), 110.53, 111.05, 120.27 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=324.5$  Hz), 125.30, 127.27, 128.39, 129.48, 129.54, 130.09, 132.84, 136.53, 143.31, 146.86, 174.77. MS (EI)  $m/z$  (%): 420 (14), 287 (35), 256 (15), 191 (49), 177 (62), 149 (59), 105 (100), 61 (68). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3443, 3065, 2924, 1731, 1609, 1363. Anal. Calcd for  $\text{C}_{21}\text{H}_{15}\text{F}_3\text{O}_4\text{S}$ : C, 60.00; H, 3.60. Found: C, 59.79; H, 3.88.

**4.2.15. 2-Acetyl-3-furyl-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (3Be).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  2.36 (s, 3H), 4.58 (1H, d,  $J=4.4$  Hz, CH), 5.32 (1H, d,  $J=4.4$  Hz, CH), 6.38–6.42 (2H, m, ArH), 7.46–7.84 (6H, m, ArH). <sup>19</sup>F NMR ( $\text{CDCl}_3$ , 470 MHz, ppm):  $\delta$  –78.59 (s, 3F). <sup>13</sup>C NMR ( $\text{CDCl}_3$ , 125 MHz, ppm):  $\delta$  29.67, 48.91, 72.13, 103.39 ( $\text{C}_3$ , d,  $^3J_{\text{F-C}}=1.5$  Hz), 109.97, 111.15, 119.85 ( $\text{CF}_3$ , q,  $^1J_{\text{F-C}}=324.8$  Hz), 128.37, 130.19, 133.56, 143.81, 148.06, 172.81, 197.44. MS (EI)  $m/z$  (%): 386 (8), 343 (13), 281 (12), 253 (22), 181 (100), 105 (49), 77 (41), 51 (21). IR (KBr,  $\text{cm}^{-1}$ ):  $\nu$  3430, 3063, 2924, 1722, 1673, 1365. Anal. Calcd for  $\text{C}_{17}\text{H}_{13}\text{F}_3\text{O}_5\text{S}$ : C, 52.85; H, 3.39. Found: C, 52.71; H, 3.64.

**4.2.16. 2-Vinyl-3-furyl-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (3Bf).** Oil; <sup>1</sup>H NMR ( $\text{CDCl}_3$ , 500 MHz, ppm):  $\delta$  4.48 (1H, d,  $J=4.8$  Hz, CH), 5.32 (1H, dd,  $^1J=6.5$  Hz,  $^2J=4.8$  Hz, CH), 5.42 (1H, d,

$J=10.5$  Hz, =CH), 5.49 (1H, d,  $J=17.5$  Hz, =CH), 6.06 (1H, ddd,  $^1J_F=17.5$  Hz,  $^2J_F=10.5$  Hz,  $^3J_F=6.5$  Hz, =CH), 6.33–6.38 (2H, m, ArH), 7.43–7.79 (6H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.69 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  48.96, 88.10, 102.80 ( $C_3$ , d,  $^3J_{F-C}=1.7$  Hz), 108.18, 110.75, 118.99, 120.05 ( $CF_3$ , q,  $^1J_{F-C}=325.7$  Hz), 128.09, 129.91, 132.60, 133.54, 142.92, 151.60, 174.38. MS (EI)  $m/z$  (%): 370 (21), 301 (7), 289 (12), 181 (100), 152 (22), 105 (54), 77 (28). IR (KBr,  $cm^{-1}$ ):  $\nu$  3428, 2957, 2922, 1697, 1609, 1364. Anal. Calcd for  $C_{17}H_{13}F_3O_4S$ : C, 55.13; H 3.54. Found: C, 54.95; H, 3.82.

**4.2.17. 2-(4-Chlorophenyl)-3-furyl-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (*trans*-**3Bg**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  4.68 (1H, d,  $J=5.9$  Hz, CH), 5.86 (1H, d,  $J=5.9$  Hz, CH), 6.35–6.42 (2H, m, ArH), 7.32–7.85 (10H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.56 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  51.66, 87.91, 101.86 ( $C_3$ , d,  $^3J_{F-C}=1.4$  Hz), 108.67, 110.91, 120.05 ( $CF_3$ , q,  $^1J_{F-C}=324.3$  Hz), 126.54, 128.26, 129.53, 129.87, 132.78, 135.31, 137.08, 143.24, 151.35, 174.36. MS (EI)  $m/z$  (%): 454 (1), 386 (1), 321 (5), 225 (6), 181 (7), 105 (16), 84 (100), 51 (5). IR (KBr,  $cm^{-1}$ ):  $\nu$  3427, 3059, 2925, 1699, 1612, 1360. Anal. Calcd for  $C_{21}H_{14}ClF_3O_4S$ : C, 55.45; H, 3.10. Found: C, 55.66; H, 3.22.

**4.2.18. 2-(4-Chlorophenyl)-3-furyl-4-trifluoromethylsulfonyl-5-phenyl-cis-2,3-dihydrofuran (*cis*-**3Bg**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  4.76 (1H, d,  $J=9.2$  Hz, CH), 5.96 (1H, d,  $J=9.2$  Hz, CH), 6.18–6.23 (2H, m, ArH), 7.13–7.93 (10H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.45 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  52.65, 83.64, 105.56 ( $C_3$ , d,  $^3J_{F-C}=1.8$  Hz), 110.39, 110.67, 120.07 ( $CF_3$ , q,  $^1J_{F-C}=324.3$  Hz), 127.62, 128.26, 128.58, 129.67, 130.31, 133.10, 134.91, 143.28, 146.30, 174.42. MS (EI)  $m/z$  (%): 454 (1), 386 (1), 321 (4), 225 (6), 181 (7), 105 (16), 84 (100), 77 (6). IR (KBr,  $cm^{-1}$ ):  $\nu$  3441, 3066, 2924, 1731, 1609, 1364. Anal. Calcd for  $C_{21}H_{14}ClF_3O_4S$ : C, 55.45; H, 3.10. Found: C, 55.52; H, 3.21.

**4.2.19. 2-Cyano-3-(2-chlorophenyl)-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (**3Ca**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  5.28 (1H, d,  $J=3.4$  Hz, CH), 5.75 (1H, d,  $J=3.4$  Hz, CH), 7.05–7.85 (9H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.90 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  53.48, 74.70, 105.70 ( $C_3$ , d,  $^3J_{F-C}=1.5$  Hz), 114.73, 119.89 ( $CF_3$ , q,  $^1J_{F-C}=325.1$  Hz), 127.01, 127.60, 128.30, 128.71, 128.96, 129.62, 130.32, 133.64, 138.76, 172.84. MS (EI)  $m/z$  (%): 413 (4), 386 (1), 344 (7), 226 (55), 149 (59), 105 (100), 77 (57), 57 (35). IR (KBr,  $cm^{-1}$ ):  $\nu$  3437, 3068, 2922, 1819, 1671, 1369. Anal. Calcd for  $C_{18}H_{11}ClF_3NO_3S$ : C, 52.25; H, 2.68; N, 3.38. Found: C, 52.31; H, 2.43; N, 3.17.

**4.2.20. 2-Cyano-3-(3-bromophenyl)-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (**3Da**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  4.68 (1H, d,  $J=3.6$  Hz, CH), 5.51 (1H, d,  $J=3.6$  Hz, CH), 7.33–7.91 (9H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.81 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  55.53, 73.42, 106.67 ( $C_3$ , d,  $^3J_{F-C}=1.7$  Hz), 115.48, 120 ( $CF_3$ , q,  $^1J_{F-C}=325.5$  Hz), 125.20, 127.11, 128.25, 128.95, 129.48, 129.98, 130.50, 131.43, 142.04, 172.15. MS (EI)  $m/z$  (%): 459 (27), 389 (26), 325 (25), 270 (51), 105 (100), 77 (49), 51 (36). IR (KBr,  $cm^{-1}$ ):  $\nu$  3440, 3065, 2922, 1972, 1700, 1364. Anal. Calcd for  $C_{18}H_{11}BrF_3NO_3S$ : C, 47.18; H, 2.42; N, 3.06. Found: C, 47.09; H, 2.45; N, 3.29.

**4.2.21. 2-Cyano-3-(4-bromophenyl)-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (**3Ea**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  4.70 (1H, d,  $J=3.4$  Hz, CH), 5.65 (1H, d,  $J=3.4$  Hz, CH), 7.34–7.86 (9H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.77 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  55.33, 75.27, 106.45 ( $C_3$ , d,  $^3J_{F-C}=1.8$  Hz), 115.28, 119.80 ( $CF_3$ , q,  $^1J_{F-C}=325.7$  Hz), 124.25, 128.35, 128.76, 129.78, 130.21, 134.57, 140.12, 171.95. MS (EI)  $m/z$  (%): 459 (36), 389 (21), 325 (24), 270 (45), 188 (40), 105 (100), 77 (54), 51 (57). IR (KBr,  $cm^{-1}$ ):  $\nu$  3441,

2957, 2923, 1671, 1586, 1366. Anal. Calcd for  $C_{18}H_{11}BrF_3NO_3S$ : C, 47.18; H, 2.42; N, 3.06. Found: C, 46.97; H, 2.34; N, 3.12.

**4.2.22. 2-Cyano-3-(4-methoxyphenyl)-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (**3Fa**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  3.83 (s, 3H), 4.78 (1H, d,  $J=3.9$  Hz, CH), 5.78 (1H, d,  $J=3.9$  Hz, CH), 6.96–7.85 (9H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.07 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  55.30, 60.43, 74.97, 106.82 ( $C_3$ , d,  $^3J_{F-C}=1.6$  Hz), 112.82, 114.77, 119.79 ( $CF_3$ , q,  $^1J_{F-C}=324.2$  Hz), 125.32, 126.64, 128.50, 129.17, 129.44, 130.27, 133.60, 160.47, 171.20. MS (EI)  $m/z$  (%): 409 (52), 340 (11), 275 (34), 221 (33), 178 (13), 142 (98), 115 (100), 77 (52). IR (KBr,  $cm^{-1}$ ):  $\nu$  3432, 3065, 2922, 1721, 1610, 1365. Anal. Calcd for  $C_{19}H_{14}F_3NO_3S$ : C, 55.74; H, 3.45; N, 3.42. Found: C, 55.76; H, 3.32; N, 3.51.

**4.2.23. 2-Cyano-3-(4-fluorophenyl)-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (**3Ga**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  4.84 (1H, d,  $J=3.0$  Hz, CH), 5.31 (1H, d,  $J=3.0$  Hz, CH), 7.11–7.87 (9H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.04 (s, 3F), –111.39, –111.40, –111.41, –111.42, –111.43, –111.44, –111.45 (m, Ph–F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  54.83, 75.25, 106.56 ( $C_3$ , d,  $^3J_{F-C}=1.3$  Hz), 115.19, 116.73, 119.89 ( $CF_3$ , q,  $^1J_{F-C}=324.2$  Hz), 125.14, 128.16, 128.53, 128.64, 128.85, 128.95, 129.06, 130.36, 133.46, 133.80, 162.13, 164.11, 172.00. MS (EI)  $m/z$  (%): 397 (14), 328 (25), 209 (10), 147 (31), 135 (37), 120 (100), 105 (36), 77 (23). IR (KBr,  $cm^{-1}$ ):  $\nu$  3439, 3065, 2925, 1807, 1612, 1360. Anal. Calcd for  $C_{18}H_{11}F_4NO_3S$ : C, 54.41; H, 2.79; N, 3.53. Found: C, 54.52; H, 2.53; N, 3.48.

**4.2.24. 2-Cyano-3-(4-tolyl)-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (**3Ha**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  2.38 (s, 3H), 4.54 (1H, d,  $J=4.0$  Hz, CH), 5.10 (1H, d,  $J=4.0$  Hz, CH), 7.20–7.82 (9H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.94 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  24.70, 55.81, 75.34, 106.48 ( $C_3$ , d,  $^3J_{F-C}=1.5$  Hz), 114.22, 119.80 ( $CF_3$ , q,  $^1J_{F-C}=324.8$  Hz), 127.67, 127.96, 128.55, 128.97, 130.15, 136.11, 137.76, 172.63. MS (EI)  $m/z$  (%): 393 (4), 366 (6), 297 (7), 205 (35), 188 (15), 105 (100), 77 (39), 51 (19). IR (KBr,  $cm^{-1}$ ):  $\nu$  3458, 3056, 2923, 1640, 1363. Anal. Calcd for  $C_{19}H_{14}F_3NO_3S$ : C, 58.01; H, 3.59; N, 3.56. Found: C, 58.21; H, 3.37; N, 3.51.

**4.2.25. 2-Cyano-3-cyclohexyl-4-trifluoromethylsulfonyl-5-phenyl-trans-2,3-dihydrofuran (**3Ia**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  1.24–1.83 (11H, m,  $CH_2$ ), 3.49 (1H, dd,  $^1J=3.6$  Hz,  $^2J=1.7$  Hz, CH), 5.58 (1H, d,  $J=3.6$  Hz, CH), 7.46–7.97 (5H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.08 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  24.88, 25.79, 25.82, 28.68, 35.95, 72.13, 106.79 ( $C_3$ , d,  $^3J_{F-C}=1.5$  Hz), 114.98, 119.60 ( $CF_3$ , q,  $^1J_{F-C}=324.5$  Hz), 128.42, 129.83, 132.57, 172.81. MS (EI)  $m/z$  (%): 385 (10), 316 (4), 252 (26), 211 (19), 197 (61), 191 (26), 151 (31), 105 (100), 77 (51). IR (KBr,  $cm^{-1}$ ):  $\nu$  3428, 3062, 2954, 1724, 1563, 1364. Anal. Calcd for  $C_{18}H_{18}F_3NO_3S$ : C, 56.10; H, 4.71; N, 3.63. Found: C, 55.93; H, 4.97; N, 3.45.

**4.2.26. 2-Cyano-3-phenyl-4-trifluoromethylsulfonyl-5-(4-bromophenyl)-trans-2,3-dihydrofuran (**3Ja**).** Oil;  $^1H$  NMR ( $CDCl_3$ , 500 MHz, ppm):  $\delta$  4.70 (1H, d,  $J=3.5$  Hz, CH), 5.68 (1H, d,  $J=3.5$  Hz, CH), 7.43–7.82 (9H, m, ArH).  $^{19}F$  NMR ( $CDCl_3$ , 470 MHz, ppm):  $\delta$  –78.80 (s, 3F).  $^{13}C$  NMR ( $CDCl_3$ , 125 MHz, ppm):  $\delta$  54.26, 74.67, 102.59 ( $C_3$ , d,  $^3J_{F-C}=1.5$  Hz), 115.38, 119.84 ( $CF_3$ , q,  $^1J_{F-C}=324.7$  Hz), 124.79, 127.34, 128.03, 128.37, 128.75, 129.28, 132.36, 139.81, 173.31. MS (EI)  $m/z$  (%): 458 (20), 389 (10), 325 (13), 267 (17), 191 (58), 105 (100), 77 (54), 51 (26). IR (KBr,  $cm^{-1}$ ):  $\nu$  3430, 3063, 2953, 1720,

1364. Anal. Calcd for C<sub>18</sub>H<sub>11</sub>BrF<sub>3</sub>NO<sub>3</sub>S: C, 47.18; H, 2.42; N, 3.06. Found: C, 47.33; H, 2.47; N, 2.95.

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## Supplementary data

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- CCDC-770589 (**3Ab**) contains all crystallographic details of this publication and is available free of charge at [www.ccdc.cam.ac.uk/const/retrieving.html](http://www.ccdc.cam.ac.uk/const/retrieving.html) or can be ordered from the following address: Cambridge Crystallographic Data Centre, 12 Union Road, GB-Cambridge CB21EZ; fax: +44 1223 336 033; or [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk). Unit cell parameters: *a*: 10.189(4) Å; *b*: 11.037(4) Å; *c*: 19.179(7) Å;  $\alpha$ : 90.000;  $\beta$ : 90.000;  $\gamma$ : 90.000; space group: *P212121*.
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