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Synthesis of stable arsonium and sulfur ylides from perfluoroalkanesulfonyl diazocarbonyl compounds and their X-ray diffraction analysis

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Abstract

A series of stable fluorine-containing arsonium ylides and sulfur ylides were simply synthesized from perfluoroalkanesulfonyl diazocarbonyl compounds in the presence of rhodium catalyst. The ylide products are fairly stable due to the strong electron-withdrawing properties of perfluoroalkanesulfonyl group and carbonyl group. They are fully confirmed by spectral methods and the structures of **3ba** and **3cd** are also established by X-ray single crystal diffraction analysis. Several interesting features about the ylide structures and some weak fluorine interaction between them are described here.

Keywords: Perfluoroalkanesulfonyl; Ylides; Diazocarbonyl compound; X-ray diffraction analysis; Weak fluorine interaction

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1. Introduction

It is known that both direct and indirect routes for the formation of ylide from diazocarbonyl compounds have been well developed [1]. Thermal, photochemical and catalytic methods have been used, but catalytic methodologies offer the greatest advantages. Metal carbenes derived from α-diazocarbonyl compounds are highly electrophilic. They readily react with an available Lewis base to effect ylide formation [2].

The most common reactions of catalytically generated ylide are [2,3]-sigmatropic rearrangement of allyl-substituted ylide intermediates; [1,2] insertion or Stevens rearrangement; β-hydride elimination; dipolar cycloaddition. These processes, which can proceed intermolecularly or intramolecularly, have shown great versatility in the synthesis of natural products [3]. On the other hand, these ylide carbenes also can be isolated as stable reaction products if two electron-withdrawing groups stabilize the ylide carbon and their structures do not facilitate subsequent rapid reactions.

Per(poly)fluoroalkanesulfonyl group, R_fSO_2, is one of the strongest electron-withdrawing groups which can active α-C–H bonds and adjacent olefins or function as nucleofugic leaving group having an electron pair to form sulfinate anions depending upon their environment [4]. Due to their various reactivities, per(poly)fluoroalkanesulfonyl groups are of special interest in organic chemistry, especially in organofluorine chemistry.

In the last decade, weak fluorine interaction, aryl-perfluoroary-, C–F···H, F···F, F···O and F···π_F interactions, gained an increasing interest. They are interactions between electron–neutral atoms or structures, which are not sensitive to the solvent, pH value. They are ubiquitous in the fluorine-containing molecules. Herein, how to make use of these ubiquitous weak interactions remained a great challenge to chemists [5].

In this paper, we describe a simple synthesis of stable perfluoroalkanesulfonyl-containing ylides from perfluoroalkanesulfonyl diazocarbonyl compounds by catalytic methods. Furthermore, some interesting structural features and weak fluorine interactions of the stable ylide **3ba** and **3cd** are also reported here.

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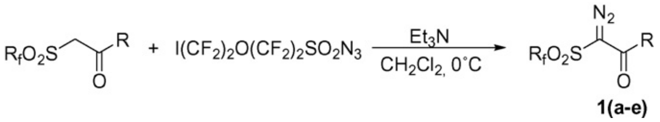
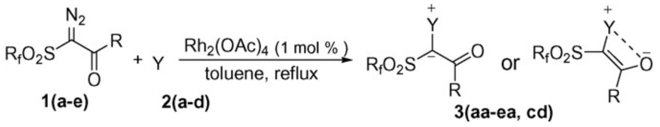


Scheme 1.



Scheme 2.

2. Results and discussion

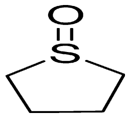
Diazocarbonyl compound **1** can be conveniently prepared by the diazo group transfer reactions with polyfluoroalkanesulfonyl azide as diazo transfer reagent (Scheme 1) [6].

The stable perfluoroalkanesulfonyl-containing ylides **3** were easily prepared as shown in Scheme 2. Treatment of diazocarbonyl compound **1** (0.5 mmol) with triphenylarsine, sulfide or sulfoxide **2** (0.5 mmol) in the presence of $Rh_2(OAc)_4$ (1 mol%) afforded stable ylides **3** in 27–96% overall yields. The results are summarized in Table 1.

As indicated in Table 1, because of two strong withdrawing groups, all of the triphenylarine, dimethylsulfide, dimethylsulfoxide and tetramethylene sulfoxide can afford the stable ylides. The yields varied greatly depending on the power of electron withdrawing of R_f and R. For instance, comparing entries 1 (R = Me) and 2 (R = Ph), it is clear that **1b** gives a markedly higher conversion to the coupled product than **1a**. We speculated that the strength of the electron-withdrawing and conjugate effect of phenyl was stronger than methyl.

These ylides **3** are quite stable and can be stored indefinitely at room temperature. For example, the ylide **3ba** can be heated to melting without any decomposition. Key to the success of the synthesis of **3** seems to be the presence of two highly electron-withdrawing perfluoroalkylsulfonyl group and carbonyl group which stabilize the ylide carbanions through delocalization of the negative charge. The structures of products **3** were determined by IR and NMR (^{1}H, ^{19}F) spectroscopy, mass spectrometry and elemental analysis. Infrared spectroscopy demonstrated significant lowering of the carbonyl frequency, consistent with the presence of an electron-rich center (ylidic carbon) in the α-position [7].

Table 1
Reaction results of perfluoroalkanesulfonyl diazocarbonyl compounds **1** with **2**

Entry	1	R_f	R	2 (Y)	Product	Yield (%)[a]	m.p. (°C)
1	**a**	ClC_4F_8	CH_3	Ph_3As	**3aa**	28	205–207
2	**b**	ClC_4F_8	Ph	Ph_3As	**3ba**	96	196–198
3	**c**	CF_3	CH_3	Ph_3As	**3ca**	65	156–158
4	**d**	CF_3	Ph	Ph_3As	**3da**	60	197–199
5	**e**	CF_3	OC_2H_5	Ph_3As	**3ea**	32	151–153
6	**c**	CF_3	CH_3	CH_3SCH_3	**3cb**	42	67–69
7	**c**	CF_3	CH_3	$Me_2S(O)$	**3cc**	27	144–146
8	**c**	CF_3	CH_3		**3cd**	40	148–150

[a] Isolate yield based on **1**.

The ylides **3** were soluble in chloroform, dichloromethane and ethyl acetate, but not in the less polar solvent hexane. Solid state structure of **3ba**, obtained by recrystallization from hexane-ethyl acetate at room temperature, illustrates an ylide structure (Fig. 1). Analysis of the Fig. 1 showed several interesting features: the four atoms As1, C1, C2, O1 are coplanar with the distance deviation of 0.172 Å from the plane of As1, C1 and C2 and with the sums of the C1-centered bond angles $\sum$°C1 = 358.8°, indicating an sp^{2} hybridization of the ylide carbanion C1. The distance of As1–C1 (1.888 (6) Å) is considerably shorter than those of As1–C9 (1.915 (6) Å), As1–C15 (1.921 (6) Å), As1–C21 (1.924 (6) Å). These results suggest little double-bond character for the ylidic bond as well as a negligibly small electrostatic attraction between the oppositely charged As1 and C1. The As1–C1–C2 bond angle is 110.2 (5)°. The distance of As1···O1 is 2.756 Å, which is shorter than that of the sum of van der Waals radii (3.37 Å). Interestingly, analysis of the X-ray crystal structure of **3ba**, we found that the bond length of the C1–C2 is 1.432 (9) Å, which is longer than the standard double bond of C=C (1.34 Å), but shorter than the single bong of C–C (1.53 Å). And the bond length of the C2–O1 is 1.241 (8) Å, which is longer than the standard double bond of C=O (1.22 Å), but shorter than the single bong of C–O (1.43 Å). In addition to the intramolecular short As1···O1 (2.756 Å) contact, two nearly vertical C2–O1···H* secondary bonding to one of the phenyl of an adjacent molecule was found with O···H distance of 2.502 and 2.698 Å, which links individual molecules of **3ba** into infinite zigzag chains (Fig. 2). In addition, the packing structure of **3ba** is further stabilized by a nearly linear C–F···H* weak

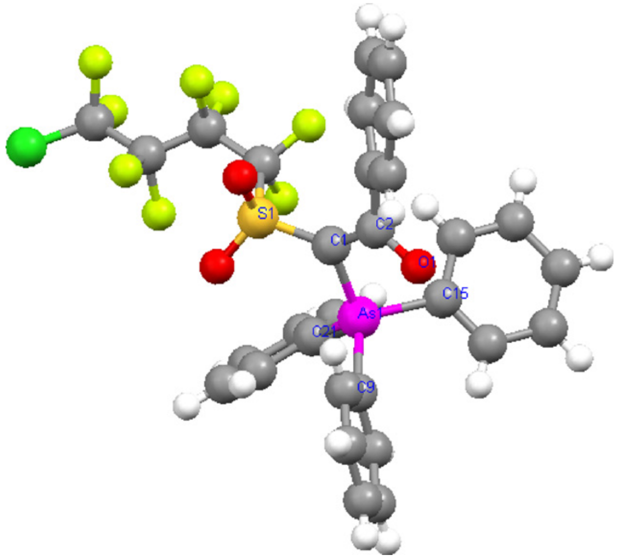


Fig. 1. The molecular structure of compound **3ba**.

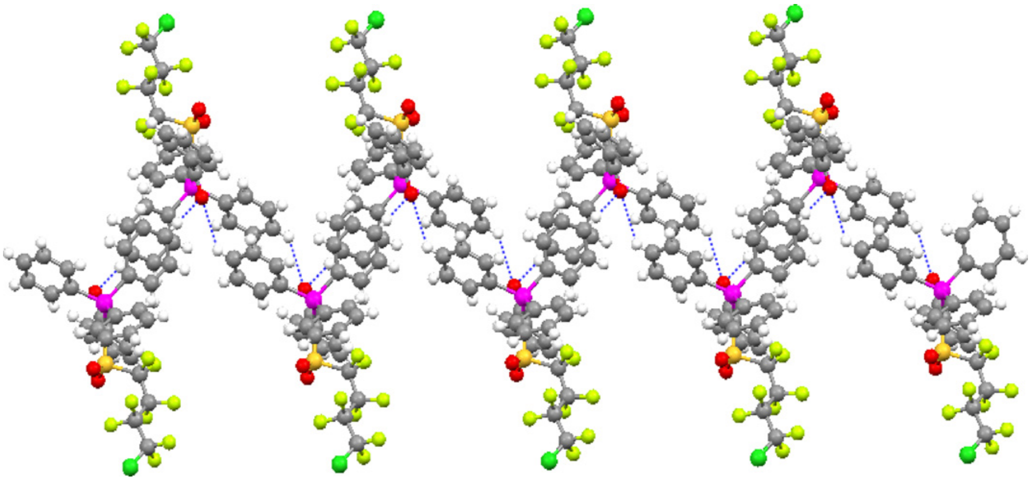


Fig. 2. Packing map of compound **3ba** by O···H bands.

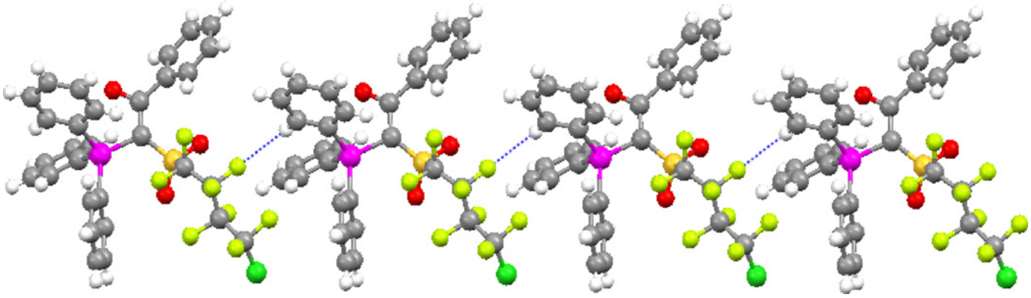


Fig. 3. Packing map of compound **3ba** by F···H bands.

intermolecular interactions. The F···H* distance is 2.607 Å and the C–F···H* angle is 178.89°, which links individual molecules of **3ba** into infinite linear chains (Fig. 3).

Likewise, we can seen from the crystal structure of sulfur ylide **3cd** (Fig. 4), the four atoms S1, C5, C6, O2 are coplanar with the distance deviation of 0.050 Å from the plane of S1, C5 and C6 and with the sums of the C5–centered bond angles $\sum^{\circ}$C5 = 357.1°, indicating an sp^2 hybridization of the ylide carbanion C5. The distance of S1–C5 is 1.715 (3) Å, which is between the standard single bong of S–C (1.81 Å) and double bond of S=C (1.61 Å). These results suggest little double-bond character for the ylidic bond as well as a negligibly small electrostatic attraction between the oppositely charged S1 and C5. The S1–C5–C6 bond angle is 114.62 (18)°. The distance of S1···O2 is 2.800 Å, which is shorter than that of the sum of van der Waals radii (3.32 Å). Interestingly, analysis of the X-ray crystal structure of **3cd**, we found that the bond length of the C5–C6 is 1.455 (9) Å, which is longer than the standard double bond of C=C (1.34 Å), but shorter than the single bong of C–C (1.53 Å). And the bond length of the C6–O2 is 1.226 (3) Å, which is longer than the standard double bond of C=O (1.22 Å), but shorter than the single bong of C–O (1.43 Å). In addition to the intramolecular short S1···O2 (2.800 Å) contact, two nearly parallel C6–O2···H* secondary bonding to one of the five-membered of an adjacent molecule were found with the distances of 2.356 Å (O2···H4B) and 2.698 Å (O4···H1A), which links individual molecules of **3cd** into infinite zigzag chains (Fig. 5). Some selected bond lengths and angles of compounds **3ba** and **3cd** are summarized in Table 2.

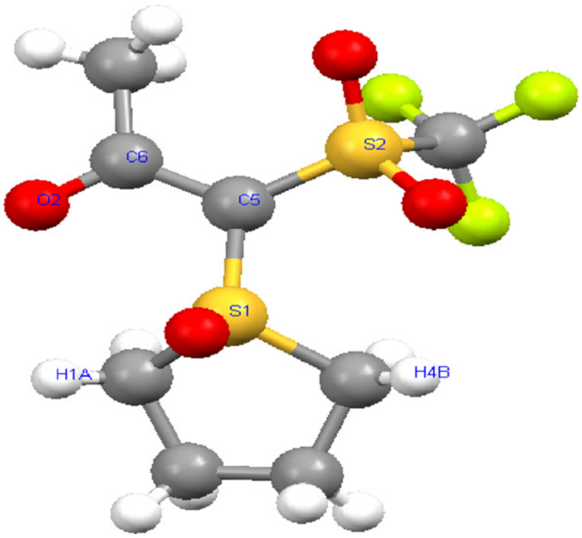


Fig. 4. The molecular structure of compound **3cd**.

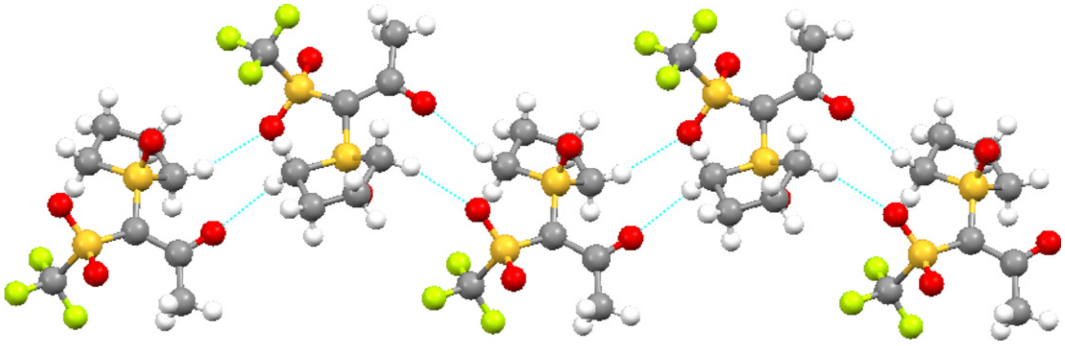


Fig. 5. Packing map of compound **3cd** by O···H bands.

Table 2
Selected bond lengths (Å) and angles (°) of compound **3ba** and **3cd**

Compound **3ba**		Compound **3cd**	
Bond lengths (Å)	Angles (°)	Bond lengths (Å)	Angles (°)
As1–C1: 1.888 (6)	As1–C1–S1: 121.1 (4)	S1–C5: 1.715 (3)	S1–C5–S2: 117.09 (14)
C1–C2: 1.432 (9)	S1–C1–C2: 127.4 (5)	C5–C6: 1.455 (3)	S2–C5–C6: 125.4 (2)
C2–O1: 1.241 (8)	C2–C1–As1: 110.2 (5)	C6–O2: 1.226 (3)	C6–C5–S1: 114.62 (18)
O1···As1: 2.756	$\sum$°C1 = 358.7°	O2···S1: 2.800	$\sum$°C5 = 357.1°
The distance deviation of O1 from the plane of As1, C1 and C2 = 0.172 Å		The distance deviation of O2 from the plane of S1, C5 and C6 = 0.050 Å	

3. Conclusion

In conclusion, stable perfluoroalkanesulfonyl-containing arsonium ylides and sulfur ylides have been readily prepared through the decomposition of perfluoroalkanesulfonyl diazocarbonyl compounds in the presence of rhodium catalyst. The X-ray crystal analyses of **3ba** and **3cd** clearly demonstrated the presence of weak fluorine interactions. The further chemical transformation and the weak fluorine interactions of these perfluoroalkanesulfonyl-containing ylides are the subject of ongoing investigations in our laboratory.

4. Experimental

Melting points were measured in a SGW® X-4 micromelting point apparatus and were uncorrected. ^{1}H and ^{19}F NMR spectra were recorded on a Bruker AM-300 spectrometer with Me_4Si and $CFCl_3$ (with upfield negative) as the internal and external standards, respectively. IR spectra were obtained with a Nicolet AV-360 spectrophotometer. Lower resolution mass spectrums were obtained on a Finnigan GC-MS 4021 instrument. The X-ray structural analysis was performed with a Rigaku/AFC 7R Diffractometer. Elemental analyses were performed by this institute.

*4.1. General procedure for the preparation of compound **1***

The mixture of $ClC_4F_8SO_2CH_2COCH_3$ (2.50 g, 7 mmol), $IC_2F_4OC_2F_4SO_2N_3$ (3.17 g, 7.1 mmol) in anhydrous CH_2Cl_2 (40 ml) was cooled in an ice-water bath and then Et_3N (1.0 ml, 0.72 mmol) was added dropwise over 20 min. After stirring for 10 min, TLC showed that the reaction was completed. The solvent was evaporated and the residue was purified by column chromatography on silica gel using ether-hexane as eluent to give **1a** (78%).

*4.2. 1-(4-Chloro-1,1,2,2,3,3,4,4-octafluorobutylsulfonyl)-1-diazopropan-2-one (**1a**)*

Pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.48 (CH_3, s). ^{19}F NMR (282 MHz, $CDCl_3$): δ −68.4 to −68.5 (2F, m), −112.2 to −112.3 (2F, m), −119.8 to −120.4 (4F, m).

*4.3. 2-(4-Chloro-1,1,2,2,3,3,4,4-octafluorobutylsulfonyl)-2-diazo-1-phenylethanone (**1b**)*

White solid; m.p. 86–88 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.49–7.71 (5H, m). ^{19}F NMR (282 MHz, $CDCl_3$): δ −68.3 (2F, t, J = 12.4 Hz), −110.8 (2F, t, J = 14.4 Hz), −119.8 to −120.2 (4F, m).

*4.4. 1-Diazo-1-(trifluoromethylsulfonyl)propan-2-one (**1c**)*

White solid; m.p. 40–42 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.44 (CH_3, s). ^{19}F NMR (282 MHz, $CDCl_3$): δ −78.6 (CF_3, s).

*4.5. 2-Diazo-1-phenyl-2-(trifluoromethylsulfonyl)ethanone (**1d**)*

White solid; m.p. 37–38 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.62–7.70 (3H, m), 7.49–7.54 (2 H, m). ^{19}F NMR (282 MHz, $CDCl_3$): δ −77.1 (CF_3, s).

4.6. Ethyl 2-diazo-2-(trifluoromethylsulfonyl)acetate (*1e*)

Pale yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 4.38 (2H, q, $J = 7.2$ Hz), 1.36 (3H, t, $J = 7.2$ Hz). ^{19}F NMR (282 MHz, $CDCl_3$): δ −77.8 (CF_3, s).

4.7. General procedure for the synthesis of stable ylides from the decomposition of diazocompounds

A solution of diazocompound **1** (0.5 mmol) in toluene (2 ml) was added dropwise over a certain period (10–30 min) to a stirred solution of the $AsPh_3$ **2** (0.5 mmol) and $Rh_2(OAc)_4$ (2 mg, 1 mol%) in toluene (2 ml), and heated at reflux temperature under a nitrogen atmosphere. After heating for an additional 2–4 h, the reaction was completed (monitored by TLC). The solvent was evaporated under reduced pressure and the residue was purified by column chromatography on silica gel using ethyl acetate–hexane (V/V = 1:5) to afford the corresponding arsonium ylides. Preparation of sulfur ylides is similar as the above procedure.

4.8. 1-(4-Chloro-1,1,2,2,3,3,4,4-octafluoro-butane-1-sulfonyl)-1-(triphenyl-5-arsanylidene)-propan-2-one (*3aa*)

Colorless solid; m.p. 205–207 °C. IR (KBr) ν: 3648, 1583, 1484, 1440, 1343, 1286, 1194, 1157, 1081, 955, 742, 689 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 7.59–7.48 (m, 15H, Ar–H), 2.46 (s, 3H, CH_3). ^{19}F NMR ($CDCl_3$) δ: −67.8 (t, $J = 13$ Hz, 2F), −110.5 to −113.1 (m, 2F), −119.3 to −120.0 (m, 4F). MS (ESI): 661 (M$^+$). Anal. Calcd for $C_{25}H_{18}AsClF_8O_3S$: C; 45.44, H; 2.75%. Found: C; 45.46, H; 2.71%.

4.9. 2-(4-Chloro-1,1,2,2,3,3,4,4-octafluoro-butane-1-sulfonyl)-2-(triphenyl-5-arsanylidene) -1-phenyl-ethanone (*3ba*)

Colorless solid; m.p. 196–198 °C. IR (KBr) ν: 3064, 1546, 1485, 1441, 1347, 1204, 1156, 1108, 1080, 955, 774, 742, 689 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 7.70–7.36 (m, 20H, Ar–H). ^{19}F NMR ($CDCl_3$) δ: −67.9 (t, $J = 14$ Hz, 2F), −108.1 to −110.3 (m, 2F), −118.5 to −121.4 (m, 4F). MS (ESI): 723 (M$^+$ + 1). Anal. Calcd for $C_{30}H_{20}AsClF_8O_3S$: C; 49.84, H; 2.79%. Found: C; 49.88, H; 2.86%.

X-ray data of **3ba** $C_{30}H_{20}AsClF_8O_3S$: Mw = 722.89, CCDC no. 654257, triclinic, space group: P-1, $a = 10.6294$ (10) Å, $b = 11.6971$ (11) Å, $c = 12.4767$ (12) Å; $\alpha = 80.7060$ (10)°, $\beta = 85.4170$ (10)°, $\gamma = 75.1610$ (10)°; $V = 1478.6$ (2) Å^3, $Z = 2$, Dc = 1.624 g/cm^3, $F(0\,0\,0) = 724$. Radiation, Mo Kα ($\lambda = 0.71073$ Å). Crystal dimension, 0.30 mm × 0.20 mm × 0.20 mm.

Intensity data were collected at 273 (2) K with a Bruker P4 four-circle diffractometer with graphite monochromator and Mo Kα radiation ($\lambda = 0.71073$ Å). A total of 2815 independent reflection were measured in range $2.27° < \theta < 25.00°$. The structure was solved by directed methods and expanded using Fourier techniques. The nonhydrogen atoms were refined anisotropically, hydrogen atoms were included but not refined. The final cycle of fullmatrix least-square refinement was base on F^2. The final R and wR value were 0.0809 and 0.2084, respectively. All calculations were performed using the SHELX-97 program.

4.10. 1-(Trifluoromethanesulfonyl)-1-(triphenyl-λ^5-arsanylidene)-propan-2-one (*3ca*)

Colorless solid; m.p. 156–158 °C. IR (KBr) ν: 3064, 1589, 1485, 1439, 1340, 1284, 1204, 1176, 1147, 1081, 1052, 1000, 885, 740, 689 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 7.60–7.48 (m, 15H, Ar–H), 2.45 (s, 3H, CH_3). ^{19}F NMR ($CDCl_3$) δ: −79.8 (s, 3F). MS m/z (%): 495 (M$^+$ + 1, 18), 481 (18), 427 (78), 360 (39), 344 (95), 306 (12), 226 (56), 165 (30), 152 (100), 132 (6), 77 (29), 69 (15), 51 (40), 43 (91). Anal. Calcd for $C_{22}H_{18}AsF_3O_3S$: C; 53.45, H; 3.67%. Found: C; 53.61, H; 3.83%.

4.11. 2-(Trifluoromethanesulfonyl)-2-(triphenyl-λ^5-arsanylidene)-1-phenyl-ethanone (*3da*)

Colorless solid; m.p. 197–199 °C. IR (KBr) ν: 3060, 1582, 1535, 1441, 1352, 1204, 1178, 1163, 1118, 1081, 1018, 746, 691, 613 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 7.69–7.36 (m, 20H, Ar–H). ^{19}F NMR ($CDCl_3$) δ: −77.9 (s, 3F). MS (ESI): 557 (M$^+$ + 1). Anal. Calcd for $C_{27}H_{20}AsF_3O_3S$: C; 58.28, H; 3.62%. Found: C; 58.29, H; 3.58%.

4.12. 1-(Trifluoromethanesulfonyl)-1-(triphenyl-λ^5-arsanylidene)-acetic acid ethyl ester (*3ea*)

Colorless solid; m.p. 151–153 °C. IR (KBr) ν: 2979, 1630, 1481, 1444, 1371, 1334, 1281, 1206, 1178, 1069, 1035, 742, 691, 619 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 7.65–7.51 (m, 15H, Ar–H), 3.95 (q, $J = 7$Hz, 2H, CH_2), 0.91 (t, $J = 7$Hz, 3H). ^{19}F NMR ($CDCl_3$) δ: −79.2 (s, 3F). MS (ESI): 525 (M$^+$ + 1). Anal. Calcd for $C_{23}H_{20}AsF_3O_3S$: C; 52.68, H; 3.84%. Found: C; 52.37, H; 3.75%.

4.13. 1-(Trifluoromethanesulfonyl)-1-(dimethylsulfuranylidene)-propan-2-one (*3cb*)

Colorless solid; m.p. 67–69 °C. IR (KBr) ν: 3045, 2940, 1618, 1414, 1337, 1266, 1205, 1145, 1045, 967, 915, 756, 686, 639 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 3.06 (s, 6H), 2.38 (s, 3H, CH_3). ^{19}F NMR ($CDCl_3$) δ: −80.3 (s, 3F). MS m/z (%): 250 (M$^+$, 16), 235 (8), 181 (42), 117 (6), 102 (51), 69 (10), 62 (17), 43 (100). Anal. Calcd for $C_6H_9F_3O_3S_2$: C; 28.80, H; 3.62%. Found: C; 28.84, H; 3.70%.

4.14. 1-(Trifluoromethanesulfonyl)-1-(dimethyloxosulfuranylidene)-propan-2-one (*3cc*)

Colorless solid; m.p. 144–146 °C. IR (KBr) ν: 3026, 2935, 1605, 1353, 1283, 1215, 1174, 1150, 1061, 1017, 985, 699, 637 cm^{-1}. ^{1}H NMR ($CDCl_3$) δ: 3.67 (s, 6H), 2.42 (s, 3H, CH_3). ^{19}F NMR ($CDCl_3$) δ: −79.7 (s, 3F). MS m/z (%): 266 (M$^+$, 10), 251 (15), 197 (44), 155 (70), 79 (26), 69 (17), 63 (25), 43 (100).

Anal. Calcd for $C_6H_9F_3O_4S_2$: C; 27.07, H; 3.41%. Found: C; 27.49, H; 3.48%.

*4.15. 1-(Trifluoromethanesulfonyl)-1-(1-oxo-tetrahydro-1-λ^4-thiophen-1-ylidene)-propan-2-one (**3cd**)*

Colorless solid; m.p. 148–150 °C. IR (KBr) ν: 3045, 2976, 1628, 1352, 1263, 1197, 1134, 1047, 922, 762, 693, 609 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 4.05–3.90 (m, 2H), 3.85–3.73 (m, 2H), 2.57–2.47 (m, 2H), 2.44–2.31 (m, 5H). ^{19}F NMR ($CDCl_3$) δ: −90.1 (s, 3F). MS m/z (%): 292 (M^+, 14), 277 (9), 231 (34), 181 (61), 105 (12), 88 (5), 69 (15), 55 (100), 43 (80). Anal. Calcd for $C_8H_{11}F_3O_4S_2$: C; 32.87, H; 3.79%. Found: C; 32.87, H; 3.68%.

X-ray data of **3cd** $C_8H_{11}F_3O_4S_2$: Mw = 292.29, CCDC no. 666345, Monoclinic, space group: $P2(1)/c$, $a = 9.3534\ (14)$ Å, $b = 12.3263\ (18)$ Å, $c = 10.1592\ (15)$ Å; $\alpha = 90.00°$, $\beta = 96.144\ (5)°$, $\gamma = 90.00°$; $V = 1164.6\ (3)$ $Å^3$, $Z = 4$, Dc = 1.667 g/cm^3, $F(0\,0\,0) = 600$. Radiation, Mo Kα ($\lambda = 0.71073$ Å). Crystal dimension, 0.28 mm × 0.25 mm × 0.22 mm.

Intensity data were collected at 298 (2) K with a Bruker P4 four-circle diffractometer with graphite monochromator and Mo Kα radiation ($\lambda = 0.71073$ Å). A total of 2815 independent reflection were measured in range $2.74° < \theta < 27.55°$. The structure was solved by directed methods and expanded using Fourier techniques. The nonhydrogen atoms were refined anisotropically, hydrogen atoms were included but not refined. The final cycle of fullmatrix least-square refinement was base on F^2. The final R and wR value were 0.0430 and 0.1084, respectively. All calculations were performed using the SHELX-97 program.

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