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Trifluoromethylated cyclopropanes and epoxides from CuI-mediated transformations of α-trifluoromethyl-diazophosphonate

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Abstract

An efficient method for the preparation of α-(trifluoromethyl)-cyclopropylphosphonates and α-(trifluoromethyl)-1,2-epoxyphosphonates has been developed. The method is based on metal-catalyzed reaction of diethyl 1-diazo-2,2,2-trifluoroethylphosphonate (**1**) with alkenes or carbonyl compounds, respectively. It established an essential advantage of CuI-catalysis over $Rh_2(OAc)_4$ in cyclopropanation reactions. An inexpensive copper iodide has also exhibited a good catalytic activity in the synthesis of epoxyphosphonates (**3**).

Keywords: Diazophosphonate; Catalysis; Carbene; Cyclopropanation; Epoxides

1. Introduction

Phosphorus-containing small cycles are very important building blocks in organic and medicinal chemistry, mainly as a result of high degree of ring strain and rigidity of the system [1] as well as due to the ability of phosphorus moiety to specific coordination with metal-dependant enzymes. Many of their derivatives, especially cyclopropyl- and 1,2-epoxypropylphosphonates, have attracted great attention due to a broad spectrum of their biological properties including antiviral, anticancer, antibiotic, antibacterial, pesticidal, insecticidal and enzyme inhibitory activities [2]. A great deal of effort is presently being devoted to development of methods, which make simpler the preparation of these molecules with high selectivity. A number of methodologies for the construction of cyclopropyl and epoxide units have been already reported. Among them, metal-mediated cyclopropanation of olefins and epoxidation of aldehydes or ketones with diazo compounds as carbenoid precursors is a highly useful method in terms of its simplicity and mild reaction conditions [3].

On top of this, the introduction of fluorine or fluoroalkyl substituents into biologically active compounds has become an important tool in drug discovery process [4]. Special attention is paid to trifluoromethyl containing compounds due to unique properties of the trifluoromethyl group, such as high electronegativity, electron density, steric hindrance and hydrophobic character [5] that can profoundly improve the pharmaco-kinetic properties of the potential drugs.

The transition-metal-catalyzed decomposition of α-diazocarbonyl compounds has became a standard method in organic synthesis. Rhodium(II) complexes have been proven to be the most versatile catalysts for mild decomposition of α-diazocarbonyl compounds and are now widely used in different chemical transformations such as X–H insertion (X = C, N, O, S), cyclopropanation, cycloadditions to nitriles and carbonyl compounds as well as ylide formation and their subsequent rearrangements [6]. Despite the fact, that α-diazophosphonates have became much less attention in such transformations the usage of Rh(II)-based catalysis for their decomposition is also preferable [7].

Recently we have developed an efficient pathway to the novel α-CF_3-containing α-diazophosphonate **1** based on commercially available ethyl trifluorohemiacetal and have shown its synthetic utility in rhodium(II)-catalyzed OH- and NH-insertion reactions with various hydroxy and amino compounds to afford the fluorinated hydroxy- and aminophosphonate derivatives in good yields [8] (Scheme 1). This method demonstrates a possibility for simultaneous introduction of trifluoromethyl and dialkyl

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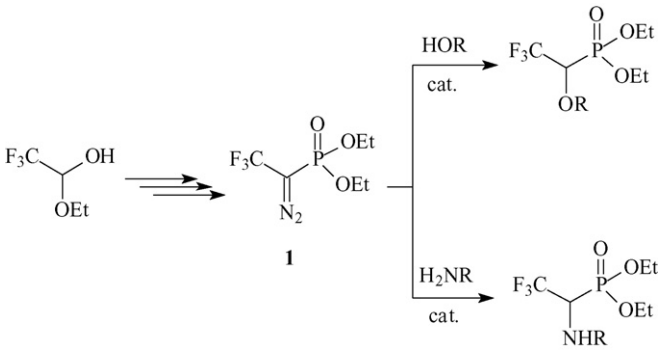


Scheme 1.

phosphonate groups into appropriate molecule *via* corresponding carbene generated *in situ*.

We have extended the synthetic utility of diazo compound **1** for the preparation of new family of fluorine-phosphorus-containing three-membered cycles based on the reactions of 1-trifluoromethyl-1-diethoxyphosphoryl carbene, formed *in situ* under metal-catalyzed conditions with unsaturated substrates such as olefins and aldehyde and now report our preliminary results of this research.

2. Results and discussions

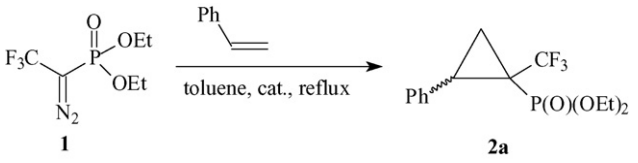
To make a scope and limitation of this methodology clear as well as to establish the most effective catalysts we studied the cyclopropanation of styrene with diazophosphonate **1** as model reaction (Scheme 2).

In our initial attempt, we examined rhodium(II) acetate as one of the most active catalyst normally used for the mild diazo decomposition. Thus, we found that the reaction takes place in toluene at 100 °C in the presence of 5 mol% of $Rh_2(OAc)_4$ and 0.5 equiv. excess of styrene and leads to completion for 3 h to afford the desired cyclopropane **2a** with moderate yield (Table 1, entry 1). Surprisingly, the usage of copper acetylacetonate (20 mol%) significantly improved the yield of **2a** (Table 1, entry 2). In this case the reaction completion was achieved by refluxing in toluene. Monitoring of the reaction was accomplished using ^{19}F NMR-spectroscopy and TLC-analysis. In both cases cyclopropane **2a** was isolated as a mixture of *cis*,*trans*-stereoisomers in *ca*. 1:1.5 ratio, which can be separated by column chromatography.

Table 1
Cyclopropanation of styrene with diethyl 1-diazo-2,2,2-trifluoroethylphosphonate **1**[a]

Entry	Catalyst	mol%	Yield (%)[b]
1	$Rh_2(OAc)_4$	5	46 (42[c])
2	$Cu(Acac)_2$	20	63
3	$Cu(PhCOCHCOCF_3)_2$	10	56
4	$CuBr_2$	20	18
5	**CuI**	**10**	**85**
6	Cu powder	10	70
7	CuI × 2,2′-bpy	10	53
8	CuI × phenanthroline	10	38

[a] Conditions: diethyl 1-diazo-2,2,2-trifluoroethylphosphonate (0.5 mmol), styrene (0.75 mmol), catalyst, toluene, reflux 3 h.
[b] Determined by ^{19}F NMR using CF_3CO_2H as internal standard.
[c] Isolated yield.

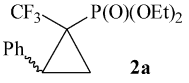
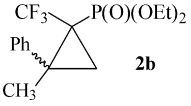
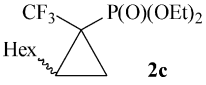
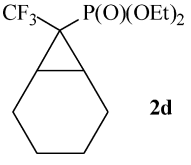
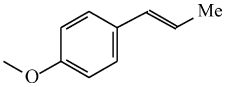
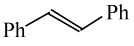
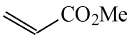


Scheme 2.

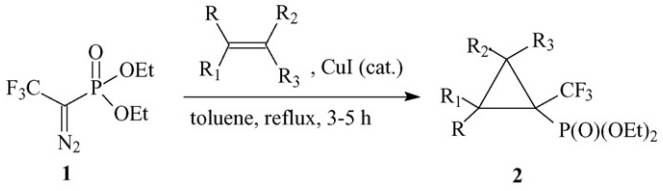
Further screening of inexpensive Cu-based catalysts has allowed us to find more active ones in the series performing the reaction under the same conditions. Even in the case of copper powder the yield of **2a** was 70% (Table 1, entry 6), but superior catalyst has proved to be copper iodide, which improved the yield up to 85% (Table 1, entry 5). In all cases a ratio of isomers was in a range from 1:1 to 1:2.

A range of other olefins with diazophosphonate **1** were then investigated under optimized reaction conditions. The results are summarized in Table 2. Terminal olefins such as styrene, α-methylstyrene and octyne-1 were converted into corresponding cyclopropanes **2a**–**2c** with good to excellent yields (Table 2, entries 1–3), whereas cyclohexene selected as convenient internal *cis*-olefine was transformed to the corresponding bicyclic phosphonate **2d** (Table 2, entry 4) with rather poor yield. Unfortunately, internal *trans*-olefins such as *p*-methox-

Table 2
Cyclopropanation of alkenes with diethyl 1-diazo-2,2,2-trifluoroethylphosphonate **1** using CuI as catalyst[a]

Entry	Alkene	Product	Yield (%)[b]
1	Styrene	 **2a**	85
2	α-Methylstyrene	 **2b**	98
3	Octene-1	 **2c**	60
4	Cyclohexene	 **2d**	20
5		–	0
6		–	0
7		–	0

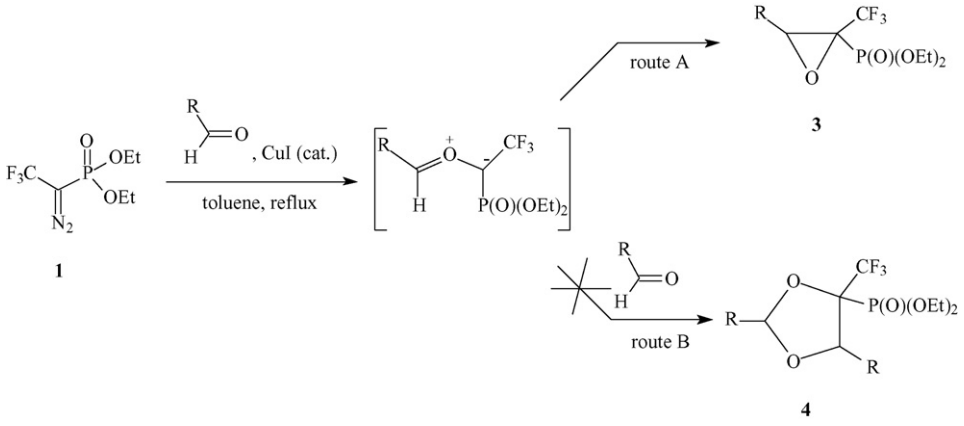
[a] Conditions: diethyl 1-diazo-2,2,2-trifluoroethylphosphonate (0.5 mmol), alkene (0.75 mmol), CuI (0.05 mmol), toluene, reflux 3–5 h.
[b] Isolated yield.



Scheme 3.

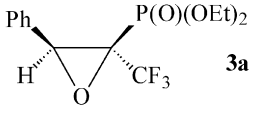
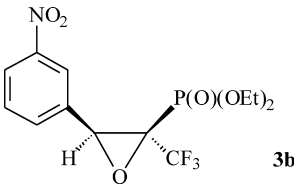
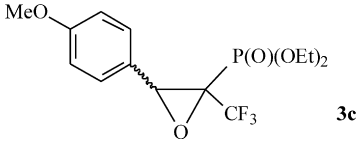
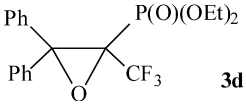
yphenyl-β-methylstyrene and stilbene and electron-poor methylacrylate have not reacted with **1** (Table 2, entries 5–7) even under more drastic conditions (140 °C in xylenes); in these cases the substrates were recovered from reaction mixtures (Scheme 3).

It is well known that oxygen ylides derived from diazo and carbonyl compounds under metal catalysis undergo proton transfer, 1,3-dipolar cycloaddition, or internal reactions that specific to the diazo compounds employed. At the same time there are very few examples of ring closure in initially formed carbonyl ylides to give the corresponding epoxides; 1,3-dioxalanes are usual products [9,10] in such transformations as a result of 1,3-dipolar cycloaddition with second molecule of carbonyl compound (Scheme 4, route B). We have investigated the interaction of α-diazo phosphonate **1** with some aromatic aldehydes and diphenylketone under CuI-catalyzed conditions and found that in all cases epoxides **3** are formed regioselectively. The reactions were performed in refluxed toluene in the presence of 10 mol% of copper iodide for 1 h to afford **3** in moderate to good yields independently of functional groups in aromatic ring (Scheme 4 and Table 3). To the best of our knowledge there is just one publication described the epoxidation of aldehydes with α-diazophosphonates [11].



Scheme 4.

Table 3
Reactions of carbonyl compounds with diethyl 1-diazo-2,2,2-trifluoroethylphosphonate (**1**) in the presence of CuI as catalyst[a]

Entry	R_1, R_2	Product	Yield (%)[b]
1	R_1 = Ph, R_2 = H	 **3a**	72
2	R_1 = 4-MeOC$_6$H$_4$, R_2 = H	 **3b**	71
3	R_1 = 3-NO$_2$C$_6$H$_4$, R_2 = H	 **3c**	90
4	R_1 = Ph, R_2 = Ph	 **3d**	41

[a] Conditions: diethyl 1-diazo-2,2,2-trifluoroethylphosphonate (0.5 mmol), carbonyl compound (0.75 mmol), CuI (0.05 mmol), toluene, reflux 2 h.
[b] Isolated yield.

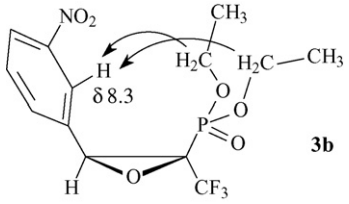


Chart 1.

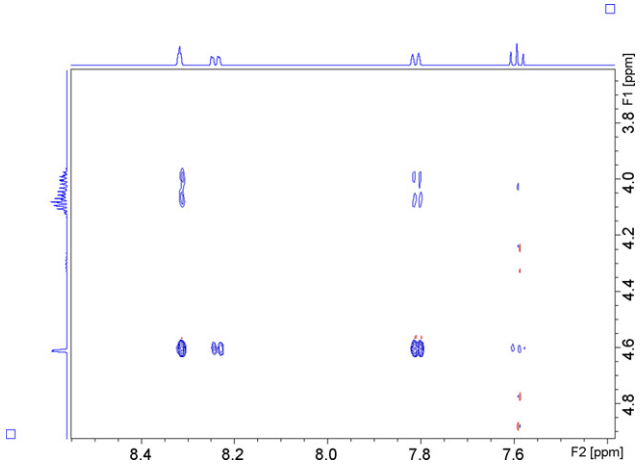


Fig. 1. 2D ROESY spectra of **3b**.

Interestingly, only *Z*-epoxides were formed in the cases of products **3a** and **3b**, whereas epoxide **3c** was obtained as inseparable mixture of isomers (*Z*/*E*, 3:1). The configuration assignment of **3b** was performed by means of 2D ROESY investigation. Thus, NOE correlations were found between multiplet signals of OCH_2 protons of diethoxyphosphoryl group (4.00–4.15 ppm) and triplet at 8.3 ppm corresponding to aromatic proton in *ortho*-position of benzene ring (Chart 1 and Fig. 1). This fact evidenced about steric closeness of these protons indicating the location of diethoxyphosphoryl and aromatic groups in *cis*-configuration.

3. Conclusion

In summary, we have developed an efficient method for the preparation of α-(trifluoromethyl)cyclopropylphosphonates and α-(trifluoromethyl)-1,2-epoxyphosphonates based on metal-catalyzed reaction of 1-diazo-2,2,2-trifluoroethylphosphonate **1** with alkenes or carbonyl compounds, respectively. It was found an essential advantage of CuI-catalysis over $Rh_2(OAc)_4$ in cyclopropanation reactions. An inexpensive copper iodide has also exhibited a good catalytic activity in the synthesis of epoxyphosphonates **3**.

4. Experimental

4.1. General methods

All solvents used in reactions were freshly distilled from appropriate drying agents before use. All other reagents were recrystallized or distilled as necessary. Reactions were performed under an atmosphere of dry nitrogen. Analytical TLC were performed with Merck silica gel 60 F_{254} plates. Visualization was accomplished by UV light or spraying by $Ce(SO_4)_2$, solution in 5% H_2SO_4. Flash chromatography was carried out using Merck silica gel 60 (230–400 mesh ASTM). All compounds were isolated as colorless oils. NMR spectra were obtained on a Bruker AV-300, AV-400, AV-600 spectrometers operating at 300, 400, 600 MHz, respectively (TMS) for ^1H; 100 and 150 MHz for ^{13}C; 288 and 376 MHz for ^{19}F ($CFCl_3$); 121.5 and 162 MHz for ^{31}P (H_3PO_4).

4.2. General procedure for cyclopropanation of olefins

A solution of diazo compound **1** (0.5 mmol) was added to a solution of alkene (0.75 mmol) and CuI (0.05 mmol, 10 mol%) or another catalyst in anhydrous toluene (2 mL). The reaction mixture was stirred under reflux for 3–4 h. After evaporation of the solvent under reduced pressure the crude product was purified by column chromatography on silica gel (EtOAc:hexanes, 1:3–1:2).

4.2.1. Diethyl 2-phenyl-1-(trifluoromethyl)cyclopropylphosphonate (*2a*)

Mixture of isomers in a ration of 1:1.5. Major isomer: ^1H NMR ($CDCl_3$, 400 MHz): δ 1.40 (t, 3H, J = 7.1 Hz), 1.46 (t, 3H, J = 7.1 Hz), 1.79 (m, 1H), 1.89 (m, 1H), 3.2 (m, 1H), 4.16–4.26 (m, 2 H), 4.27–4.37 (m, 2H), 7.28–7.32 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 12.6, 16.4 (dd, J_1 = 6.0 Hz, J_2 = 12.1 Hz), 25.4 (dq, $J_{C\text{-}P}$ = 182.5 Hz, $J_{C\text{-}F}$ = 33.2 Hz), 27.3, 63.2, 124.5 (dq, $J_{C\text{-}F}$ = 275.3 Hz, $J_{C\text{-}P}$ = 7.7 Hz), 127.5, 128.2, 129.4, 133.5. ^{31}P NMR ($CDCl_3$, 162 MHz): δ 24.84; ^{19}F NMR ($CDCl_3$, 282 MHz): δ 19.88. Minor isomer: ^1H NMR ($CDCl_3$, 400 MHz): δ 1.09 (t, 3H, J = 7.1 Hz), 1.24 (t, 3H, J = 7.1 Hz), 1.68 (m, 1H), 2.14 (m, 1H), 2.95 (d_{AB}, 1H, J_1 = 9.1 Hz, J_2 = 18.3 Hz), 3.60–3.80 (m, 2H), 3.84–3.92 (m, 1H), 3.97–4.07 (m, 1H), 7.24–7.40 (m, 5H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 13.3, 16.0 (dd, J_1 = 6.0 Hz, J_2 = 27.6 Hz), 25.4 (dq, $J_{C\text{-}P}$ = 182.5 Hz, $J_{C\text{-}F}$ = 33.2 Hz), 26.6, 62.5 (dd, J_1 = 40.9, J_2 = 6.6 Hz), 124.8 (dq, $J_{C\text{-}F}$ = 274.2 Hz, $J_{C\text{-}P}$ = 8.8 Hz), 127.4, 127.8, 129.9, 133.6. ^{31}P NMR ($CDCl_3$, 162 MHz): δ 17.58; ^{19}F NMR ($CDCl_3$, 282 MHz): δ 13.58 (d, J = 2.6 Hz). Anal. Calcd. for $C_{14}H_{18}F_3O_3P$: C, 52.18; H, 5.63. Found: C, 52.26; H, 5.74.

4.2.2. Diethyl 2-methyl-2-phenyl-1-(trifluoromethyl)cyclopropylphosphonate (*2b*)

Mixture of isomers in a ration of 1:1.5. Major isomer: ^1H NMR ($CDCl_3$, 400 MHz): δ 1.40 (t, 3H, J = 7.1 Hz), 1.46 (t, 3H, J = 7.1 Hz), 1.71 (s, 3H), 1.79 (d_{AB}, 0.5H, J_{AB} = 5.4 Hz), 1.83 (d_{AB}, 0.5H, J_{AB} = 5.4 Hz), 1.86–1.91 (m, 1H), 4.13–4.22 (m, 1H), 4.22–4.34 (m, 3H), 7.18–7.42 (m, 5H); ^{31}P NMR ($CDCl_3$, 162 MHz): δ 20.36; ^{19}F NMR ($CDCl_3$, 282 MHz): δ 21.55. Minor isomer: ^1H NMR ($CDCl_3$, 400 MHz): δ 1.11 (t, 3H, J = 7.1 Hz), 1.23 (t, 3H, J = 7.1 Hz), 1.33–1.42 (m, 1H), 1.60 (s, 3H), 2.08–2.18 (m, 1H), 3.52–3.63 (m, 1H), 3.82–4.00 (m, 2H), 4.13–4.33 (m, 1H), 7.15–7.50 (m, 5H); ^{31}P NMR

($CDCl_3$, 162 MHz): δ 18.31; ^{19}F NMR ($CDCl_3$, 282 MHz): δ 13.0 (d, J = 2.6 Hz). Anal. Calcd. for $C_{15}H_{20}F_3O_3P$: C, 53.57; H, 5.99. Found: C, 53.96; H, 6.20.

4.2.3. Diethyl 2-hexyl-1-(trifluoromethyl)cyclopropylphosphonate (**2c**)

Mixture of isomers in a ration of 1:1.2. Major isomer: ^1H NMR ($CDCl_3$, 300 MHz): δ 0.93 (t, 3H, J = 7.1 Hz), 1.27–1.45 (m, 13H), 1.45–1.78 (m, 5H), 1.81–1.93 (m, 1H), 4.15–4.30 (m, 4H); ^{31}P NMR ($CDCl_3$, 162 MHz): δ 21.7; ^{19}F NMR ($CDCl_3$, 282 MHz): δ 21.2. Minor isomer: ^1H NMR ($CDCl_3$, 300 MHz): δ 0.95 (t, 6H, J = 7.1 Hz), 1.25–1.45 (m, 11H), 1.45–1.78 (m, 4H), 1.82–1.95 (m, 1H), 4.15–4.30 (m, 4H); ^{31}P NMR ($CDCl_3$, 121.5 MHz): δ 20.44; ^{19}F NMR ($CDCl_3$, 282 MHz): δ 13.65. Anal. Calcd. for $C_{14}H_{26}F_3O_3P$: C, 50.90; H, 7.93. Found: C, 50.76; H, 7.74.

4.2.4. Diethyl 7-(trifluoromethyl)bicyclo[4.1.0]heptan-7-ylphosphonate (**2d**)

^1H NMR ($CDCl_3$, 400 MHz): δ 0.84–0.93 (m, 1H), 1.25–1.42 (m, 10H), 1.63–1.72 (m, 1H), 1.90–2.09 (m, 3H), 2.30–2.39 (m, 1H), 4.15–4.30 (m, 4H); ^{31}P NMR ($CDCl_3$, 162 MHz): δ 23.1; ^{19}F NMR ($CDCl_3$, 376 MHz): δ 15.2. Analytically pure sample was not obtained.

4.3. General procedure for epoxidation of carbonyl compounds

A solution of diazo compound **1** (0.5 mmol) was added to a solution of aldehyde or ketone (0.55 mmol) and CuI (0.05 mmol, 10 mol%) in anhydrous toluene (2 mL). The reaction mixture was stirred under reflux for 1 h. After evaporation of the solvent under reduced pressure the crude product was purified by column chromatography on silica gel (EtOAc:hexanes, 1:3–1:2).

4.3.1. Diethyl (Z)-1,2-epoxy-2-phenyl-1-(trifluoromethyl)ethylphosphonate (**3a**)

^1H NMR ($CDCl_3$, 300 MHz): δ 1.22 (t, 6H, J = 7.1 Hz), 3.84–4.10 (m, 4H), 4.59 (s, 1H), 7.37–7.45 (m, 2H), 7.46–7.52 (m, 2H); ^{31}P NMR ($CDCl_3$, 121.5 MHz): δ 9.43 (q, J = 2.2 Hz), ^{19}F NMR ($CDCl_3$, 282 MHz): δ 6.91 (d, J = 2.2 Hz). Anal. Calcd. for $C_{13}H_{16}F_3O_4P$: C, 48.16; H, 4.97. Found: C, 47.91; H, 5.14.

4.3.2. Diethyl (Z)-1,2-epoxy-2-(3-nitrophenyl)-1-(trifluoromethyl)ethylphosphonate (**3b**)

^1H NMR ($CDCl_3$, 300 MHz): δ 1.24 (t, 3H, J = 7.1 Hz), 1.29 (t, 3H, J = 7.1 Hz), 4.00–4.15 (m, 4H), 4.65 (s, 1H), 7.63 (t, 1H, J = 8.0 Hz), 7.85 (d, 1H, J = 8.0 Hz), 8.28 (d, 1H, J = 8.0 Hz), 8.36 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz): δ 16.1, 58.3 (dq, $J_{C\text{-}P}$ = 201.3 Hz, $J_{C\text{-}F}$ = 36.9 Hz), 58.79 (dd, J_1 = 3.6 Hz, J_2 = 7.66 Hz), 63.87, 63.99, 121.8 (dq, $J_{C\text{-}P}$ = 22.7 Hz, $J_{C\text{-}F}$ = 279.0 Hz), 122.3, 123.8, 129.0, 133.2, 133.3, 147.8. ^{31}P NMR ($CDCl_3$, 121.5 MHz): δ 8.61; ^{19}F NMR ($CDCl_3$, 282 MHz): δ 6.80. Anal. Calcd. for $C_{13}H_{15}F_3NO_6P$: C, 42.29; H, 4.09; N, 3.79. Found: C, 42.01; H, 3.98; N, 3.66.

4.3.3. Diethyl 1,2-epoxy-2-(4-methoxyphenyl)-1-(trifluoromethyl)ethylphosphonate (**3c**)

Inseparable mixture of *Z*/*E* isomers in 3:1 ratio. ^1H NMR ($CDCl_3$, 300 MHz): δ 1.24 (apt dd, 3H, J = 7.3 Hz, J = 7.1 Hz, *Z*), 1.47 (t, 3H, J = 7.1 Hz, *E*), 3.87 (s, 3H, *Z* + *E*), 3.80–4.15 (m, 3H, *Z* + *E*), 4.29–4.44 (m, 1H, *Z* + *E*), 4.54 (d, 1H, J = 2.3 Hz, *Z*), 4.72 (d, 1H, J = 1.9 Hz, *E*), 6.92–6.98 (m, 2H, *Z* + *E*), 7.29–7.44 (m, 2H, *Z* + *E*); ^{13}C NMR ($CDCl_3$, 100 MHz). Major isomer: δ 16.1, 55.2, 58.3 (dq, $J_{C\text{-}P}$ = 204.0 Hz, $J_{C\text{-}F}$ = 36.5 Hz), 59.9 (J = 3.3 Hz), 63.4, 113.4, 122.2 (qd, $J_{C\text{-}F}$ = 281.0 Hz, $J_{C\text{-}P}$ = 23.2 Hz), 122.9, 128.3, 160.0. ^{31}P NMR ($CDCl_3$, 121.5 MHz): δ 9.74 (q, J = 2.2 Hz, *Z*), 11.20 (q, J = 3.1 Hz, *E*); ^{19}F NMR ($CDCl_3$, 282 MHz): δ 6.92 (d, J = 2.2 Hz, *Z*), 14.6 (q, J = 2.9 Hz, *E*). Anal. Calcd. for $C_{14}H_{18}F_3O_5P$: C, 47.47; H, 5.12. Found: C, 47.32; H, 5.04.

4.3.4. Diethyl 1,2-epoxy-2,2-diphenyl-1-(trifluoromethyl)ethylphosphonate (**3d**)

^1H NMR ($CDCl_3$, 300 MHz): δ 1.14 (t, 3H, J = 7.1 Hz), 1.33 (t, 3H, J = 7.1 Hz), 3.68–3.81 (m, 1H), 3.90–4.08 (m, 2H), 4.09–4.20 (m, 1H), 7.25–7.38 (m, 6H), 7.48 (d, 2H, J = 8.0 Hz), 7.60 (d, 2H, J = 8.0 Hz); ^{31}P NMR ($CDCl_3$, 121.5 MHz): δ 9.71; ^{19}F NMR ($CDCl_3$, 282 MHz): δ 7.10. Anal. Calcd. for $C_{19}H_{20}F_3O_4P$: C, 57.00; H, 5.04. Found: C, 57.21; H, 4.88.

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