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Communication

Molecular diversity of triphenylphosphine promoted reaction of electron-deficient alkynes and arylidene Meldrum acid (*N,N'*-dimethylbarbituric acid)

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ABSTRACT

The three-component reaction of triphenylphosphine, dimethyl hex-2-en-4-ynedioate and arylidene *N,N'*-dimethylbarbituric acids in dry methylene dichloride at room temperature afforded *trans*-1,3-disubstituted 7,9-diazaspiro[4.5]dec-1-enes in good yields and with high diastereoselectivity. However, the similar three-component reaction with arylidene Meldrum acids resulted in a mixtures of *cis*/*trans*-1,2-disubstituted 7,9-dioxaspiro[4.5]dec-1-enes. Additionally, the three-component reaction of triphenylphosphine, dimethyl but-2-ynedioate and arylidene Meldrum acids gave polysubstituted 5-(triphenyl-λ^5-phosphanylidene)cyclopenta-1,3-dienes. A plausible reaction mechanism was proposed for the formation of various products with different regioselectivity and diastereoselectivity.

In 1995, Lu and coworkers firstly reported a phosphine-catalyzed [3 + 2] annulation reaction to construct monocyclopentene derivatives by using 2,3-butadienoates with electron-deficient olefins [1]. From then on, the nucleophilic phosphine-catalyzed annulation reactions have emerged as one of the most powerful tools for the construction of diverse carbocyclic and heterocyclic systems [2–15]. In the conventional phosphine-catalyzed reactions, the initial nucleophilic attack of phosphines to electron-deficient allenes, alkynes, and the modified allylic carbonates generated active zwitterionic intermediates, followed by subsequent addition to other electrophiles in a multiple step manner to assembly of versatile five-membered carbo- and heterocycles [16–33]. With the explosive development of phosphine catalyzed domino reactions, electron-deficient alkynes such as dialkyl but-2-ynedioates, alkyl propionates and benzoylacetylenes have been widely used to give active zwitterionic intermediates in many domino [3 + 2] cycloaddition reactions [33–47]. In recent years, another electron-deficient alkynes, dialkyl hex-2-en-4-ynedioates, have emerged as new reactive substrates in the phosphine-catalyzed reaction [47–52]. Dialkyl hex-2-en-4-ynedioates can be easily prepared in nearly quantitative yields from base-catalyzed dimerization of alkyl propiolates under mild reaction conditions [53–57]. Chuang and coworkers established several phosphine-catalyzed cycloaddition of dialkyl hex-2-en-4-ynedioates with various electrophiles containing C=O, C=C and C=N double bonds [58–61]. Inspired by these pioneering works and in continuation of our interest in developing new efficient synthetic protocols for the heterocyclic spiro compounds, we have also developed several domino reactions by employing nucleophilic triphenylphosphine addition to electron-deficient alkynes as key protocol for the efficient construction of diverse polycyclic and spiro compounds [62–67]. The initially formed active delocalized zwitterionic intermediates, which were generated from addition of phosphine to dialkyl hex-2-en-4-ynedioates, showed versatile reactivity of both 1,3-dipole and 1,5-dipole in the domino reaction depending on the chemical structures of the substrates and reaction conditions. In order to further demonstrate the potential synthetic values of the nucleophilic phosphine catalyzed annulation reaction, herein we wish to report interesting regioselectivity and diastereoselectivity of triphenylphosphine promoted domino reaction of dimethyl hex-2-en-4-ynedioate and dimethyl but-2-ynedioate with two α,α'-diactivated cyclic alkenes such as arylidene *N,N'*-dimethylbarbituric acids and Meldrum acids.

According to our previously established reaction conditions for the triphenylphosphine catalyzed domino reactions of electron-deficient alkynes [65], a mixture of triphenylphosphine **1**, dimethyl hex-2-en-4-ynedioate **2** and benzylidene *N,N'*-dimethylbarbituric acid **3** in methylene dichloride was stirred at 25 °C for 6 h. After workup, the expected 6,8,10-trioxo-7,9-diazaspiro[4.5]dec-1-enes **4a** was obtained 68% yield. The scaffold of triphenylphosphine was

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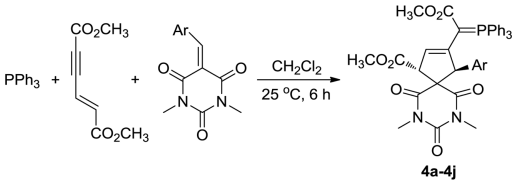
Table 1
Synthesis of 6,8,10-trioxo-7,9-diazaspiro[4.5]dec-1-enes **4a-4j**.[a]



Entry	Compd	Ar	Yield (%)[b]
1	**4a**	C_6H_5	68
2	**4b**	*p*-$CH_3C_6H_4$	81
3	**4c**	*m*-$CH_3C_6H_4$	71
4	**4d**	*p*-$CH_3OC_6H_4$	79
5	**4e**	*o*-$CH_3OC_6H_4$	85
6	**4f**	*p*-$(CH_3)_3CC_6H_4$	69
7	**4g**	*m*-ClC_6H_4	69
8	**4h**	*o*-BrC_6H_4	82
0	**4i**	*p*-$NO_2C_6H_4$	82
10	**4 j**	*o*-$NO_2C_6H_4$	89

[a] Reaction conditions: triphenylphosphine (0.5 mmol), dimethyl hex-2-en-4-ynedioate (0.7 mmol), arylidene *N,N'*-dimethylbarbituric acid (0.5 mmol), CH_2Cl_2 (20.0 mL), 25 °C, 6 h.
[b] Isolated yields.

retained in the final product as a triphenyl-λ^5-phosphanylidene unit, which is a common phenomenon for the triphenylphosphine promoted domino reactions of electron-deficient alkynes. Then, various arylidene *N,N'*-dimethylbarbituric acids were employed in the reaction. The results are summarized in Table 1. All reactions proceeded smoothly to give the functionalized 6,8,10-trioxo-7,9-diazaspiro[4.5]dec-1-enes **4a-4j** in high yields. Although there are two chiral carbon atoms in the products **4a-4j**, TLC monitoring showed that only one diastereoisomer was predominately formed in the reaction. The structures of the obtained spiro compounds **4a-4j** were fully characterized by IR, HRMS, ^{1}H, ^{13}C and ^{31}P NMR spectra. In the ^{1}H NMR spectrum of the compound **4a**, the three protons on the ring of cyclopentene display a singlet at δ 4.73 and two doublets at δ 4.12, δ 3.84 with coupling constant J =7.6 Hz. The two methyl groups showed two singlets at δ 3.43, δ 3.05 and two *N*-methyl groups showed two singlets at δ 2.84, δ 2.72. The single crystal structures of the compounds **4b** (Fig. 1) and **4f** (Fig. S1 in Supporting information) were successfully determined by X-ray diffraction method, crystallographic data **4b** (CCDC No. 1907398), **4f** (CCDC No. 1907399) have been deposited at the Cambridge Crystallographic Data base Centre. We were pleased to find that the two single crystals have same relative configuration. From the Fig. 1, it can be seen that a cyclopentene ring was formed. The 2-methoxy-2-oxo-1-(triphenyl-λ^5-phosphanylidene)ethyl group

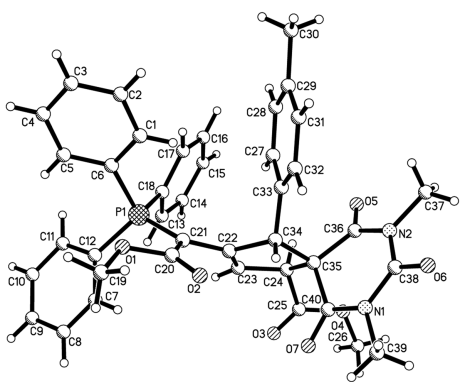
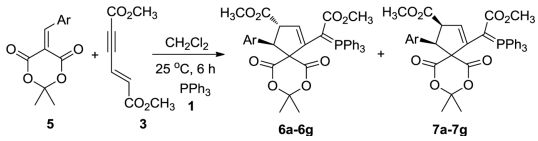


Fig. 1. Molecular structure of the compound **4b**.

Table 2
Synthesis of *cis/trans*-6,10-dioxo-7,9-dioxaspiro[4.5]dec-1-enes.[a]



Entry	Ar	Compd.	Yield (%)[b]	Compd	Yield (%)[b]
1	C_6H_5	**6a**	57	**7a**	trace
2	*p*-$CH_3OC_6H_4$	**6b**	42	**7b**	33
3	*o*-$CH_3OC_6H_4$	**6c**	trace	**7c**	48
4	*m*-FC_6H_4	**6d**	43	**7d**	37
5	*p*-ClC_6H_4	**6e**	47	**7e**	32
6	*p*-BrC_6H_4	**6f**	50	**7f**	31
7	*p*-$NO_2C_6H_4$	**6g**	46	**7g**	35

[a] Reaction conditions: triphenylphosphine (0.5 mmol), dimethyl hex-2-en-4-ynedioate (0.7 mmol), arylidene Meldrum acid (0.5 mmol), CH_2Cl_2 (20.0 mL), 25 °C, 6h.
[b] Isolated yields.

connected on the cyclic C=C double bond. The aryl group and the methyl ester unit exist on *trans*-1,3-positions. Therefore, we have assigned all spiro compounds **4a-4j** have *trans*-1,3-configuration on the basis of the spectroscopy and single crystal structures. It should be pointed out that the spiro compounds **4a-4j** have the similar structural pattern to that of our previously reported reaction of triphenylphosphine, dimethyl hex-2-en-4-ynedioate and 2-arylidene-1,3-indanediones [65].

In order to demonstrate the synthetic values of this domino reaction, various arylidene Meldrum acids **5** were also tested in the three-component reaction under same reaction conditions. The results are summarized in Table 2. Although the similar spiro compounds were obtained in this reaction, the reactions with arylidene Meldrum acids gave very different regioselectivity and diastereoselectivity to that of the above mentioned reaction with arylidene *N,N'*-dimethylbarbituric acids. At first, different from the formation of spiro compounds with aryl group and methoxycarbonyl group at 1,3-positions in the reaction of arylidene *N,N'*-dimethylbarbituric acids, the spiro compounds with aryl group and 3-methoxycarbonyl group at 1,2-positions were obtained in the reaction containing arylidene Meldrum acids. Secondly, the reaction usually gave *trans*-2,3-disubstituted 6,10-dioxo-7,9-dioxaspiro[4.5]dec-1-enes **6a-6g** as main products and *cis*-2,3-6,10-dioxo-7,9-dioxaspiro[4.5]dec-1-enes **7a-7g** as minor products. In the reaction of benzylidene Meldrum acid, the expected *cis*-isomer **7a** was not obtained due to too lower yield. However, only *cis*-isomer **7c** was obtained in the reaction of *o*-methoxybenzylidene Meldrum acid, while the expected *trans*-isomer **6c** was not successfully isolated. It should be pointed out that a mixture of *cis/trans*-isomers with similar molecular ratios were also obtained when the three-component reaction was carried out in other solvent such as dimethoxyethane, tetrahydrofuran and acetonitrile, The structures of the obtained *cis/trans*-isomers **6a-6g** and **7a-7g** were fully characterized by IR, HRMS, ^{1}H, ^{13}C and ^{31}P NMR spectroscopies. The ^{1}H NMR spectra of the *cis/trans*-isomers give very different patterns of the characteristic absorptions. For example, the ^{1}H NMR spectrum of **6b** shows a singlet at δ 4.40 and a mixed peak at δ 4.12-4.03 for the three protons in cyclopentene ring. The three methoxy group give three singlets at δ 3.27, δ 2.96, δ 2.27, the one methyl groups in propylene unit shows a normal peak at δ 1.79, while the sign of another methyl group shifts to the high magnetic field at δ 0.60 due to effect of ring current of phenyl group. In the ^{1}H NMR spectrum of **7b**, the three protons on the ring of cyclopentene display a singlet at δ 4.77, and two doublets at δ 4.20, δ 3.96. The three methoxy

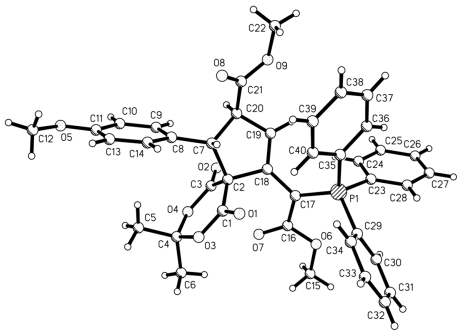


Fig. 2. Single crystal structures of the *trans*-isomer **6b**.

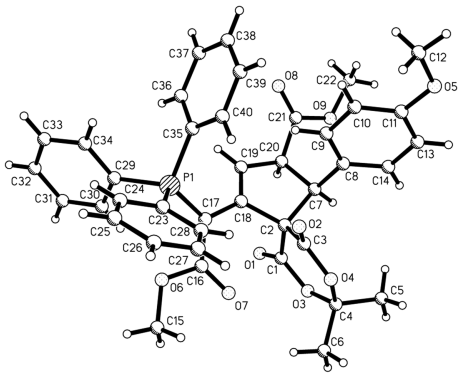
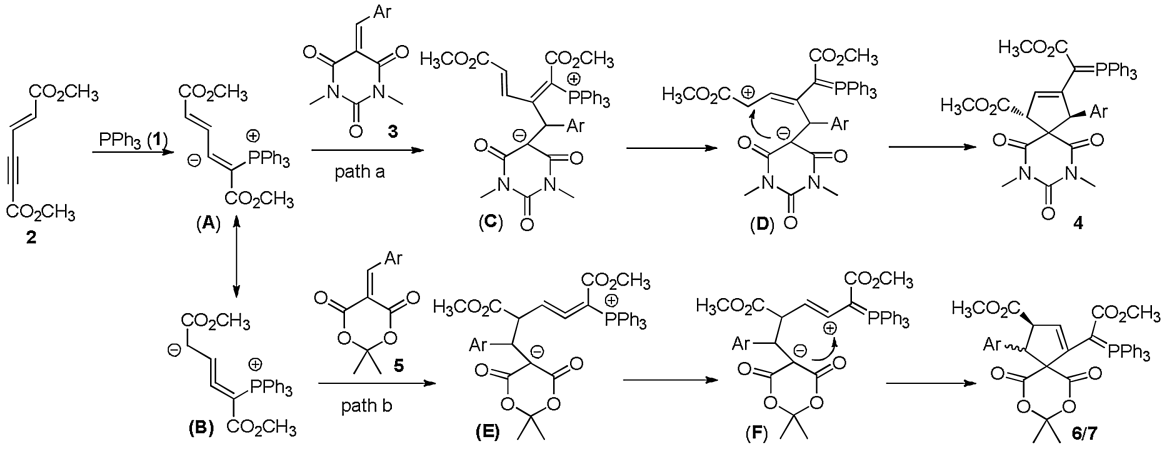


Fig. 3. Single crystal structure of the *cis*-isomer **7b**.

group display three singlets at δ 2.98, δ 2.85, δ 2.25 and two methyl groups show two singlets at 1.88 and δ 1.80. The single crystal structures of the three-couples of *cis*/*trans*-isomers **6b** (Fig. 2), **7b** (Fig. 3), **6f, 7f, 6g** and **7g** (Figs. S2-S5 in Supporting information) were successfully determined by X-ray diffraction method. Crystallographic data **6b** (CCDC No. 1907400), **6f** (CCDC No. 1907401), **6g** (CCDC No. 1907402), **7b** (CCDC No. 1907403), **7f** (CCDC No. 1907404), **7g** (CCDC No. 1907405) have been deposited at the Cambridge Crystallographic Data base Centre. From the Fig. 1, it is clearly observed that the 2-aryl group and 3-methoxycarbonyl group exist in *trans*-configuration in spiro compound **6b**. The one methyl group actually stands on the upper position of phenyl ring, which causes its absorption shifting to a high magnetic field in ^{1}H NMR spectrum. On the other hand, the 2-aryl group and 3-methoxycarbonyl group exist on *cis*-configuration in spiro compound **7b**. The two methyl groups do not exist on the top of phenyl ring. Thus, the two methyl groups give the normal absorption peaks in ^{1}H NMR spectrum.

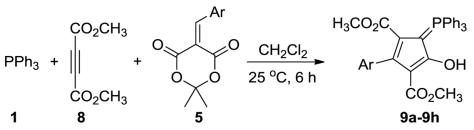
For explaining the formation of two kinds of spiro compounds in the three-component reaction, a plausible reaction mechanism is rationally proposed in Scheme 1 based on the present experiments and the previuosly reported similar reactions [62–66]. At first, addition of triphenylphosphine to C≡C triple bond of dimethyl hex-2-en-4-ynedioate gave the active dipolar intermediate **A**, which has a resonance hybride **B** *via* allylic immigration. Both active dipolar intermediate **A** and **B** could take part in the sequential annulation reaction. On path a, the addition of dipolar spice **A** to arylidene *N,N'*-dimethylbarbituric acid resulted in a adduct intermediate **C**. Then, intemediate **C** in turn converted to a triphenylphosphanylidene intermediate **D** by allylic arrangement of carbanion. Finally, the intramolecular coupling of the positive charge with the negative charge in intermediate **D** gave the final 6,8,10-trioxo-7,9-diazaspiro[4.5]dec-1-enes **4**. Because all three sequential reaction was in the retro-equilibrium reactions, it is nature to find that the stable *trans*-isomer was predominatedly formed as the main product in the domino reaction. On path b. the addition of active dipolar spice **B** to arylidene Meldrum acid resulted in a adduct intermediate **E**, which also converted to a triphenylphosphanylidene intermediate **F** and sequentially afforded the 6,10-dioxo-7,9-dioxaspiro[4.5]dec-1-enes **6** and **7**. It might be due to the neighboring 2-aryl group and 3-methoxycarbonyl group has little steric hindance, both *cis/trans*-isomers **6** and **7** were formed in the sequential reaction process. At present the exact reason for the reaction proceeded *via* path a or path b are not clear, which would depend on the slightly different nucleophilic reactivity of arylidene Meldrum acids to that of arylidene *N,N'*-dimethylbarbituric acids.

For developing the molecular diversity of this three-component reaction, another common electron-deficient alkyne, dimethyl but-2-ynedioate **8**, was also employed in the reaction. The three-component reaction of triphenylphosphine, dimethyl but-2-ynedioate and arylidene Meldrum acids was also carried out in methylene dichloride at room temperature. Steading of formation of the expected spiro compound, the polysubstituted 5-(triphenyl-λ^5-phosphanylidene)cyclopentadienes **9a-9h** was obtained in satisfactory yields (Table 3). The structures of the compounds **9a-9h** were confirmed by determination of the three single crystals of **9b** (Fig. 4), **9d** and **9e** (Figs. S6 and S7 in Supporting information). Crystallographic data **9b** (CCDC No. 1907406), **9d** (CCDC No. 1907407), and **9e** (CCDC No. 1907408) have been deposited at the Cambridge Crystallographic Data base Centre. The reaction did not result in the formation of the expected spiro compounds, while the



Scheme 1. Proposed reaction mechanism for three-component reaction.

Table 3
Synthesis of (triphenyl-λ^5-phosphanylidene)cyclopentadienes **9a-9h**.[a]



Entry	Compd	Ar	Yield (%)[b]
1	**9a**	C_6H_5	79
2	**9b**	*p*-$CH_3C_6H_4$	72
3	**9c**	*o*-$CH_3OC_6H_4$	58
4	**9d**	*p*-$CH_3OC_6H_4$	70
5	**9e**	*p*-$NO_2C_6H_4$	66
6	**9f**	*p*-ClC_6H_4	55
7	**9g**	*p*-BrC_6H_4	49
8	**9h**	*m*-FC_6H_4	65

[a] Reaction conditions: triphenylphosphine (0.5 mmol), dimethyl but-2-ynedioate (0.5 mmol), arylidene Meldrum acid (0.5 mmol), CH_2Cl_2 (20.0 mL), 25 °C, 6 h.
[b] Isolated yields.

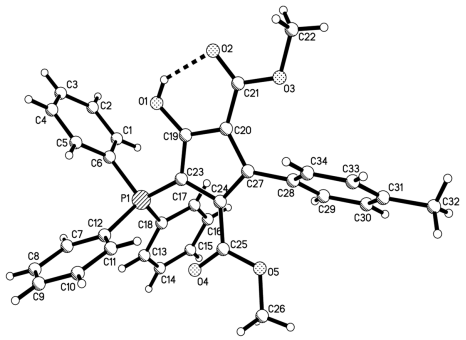
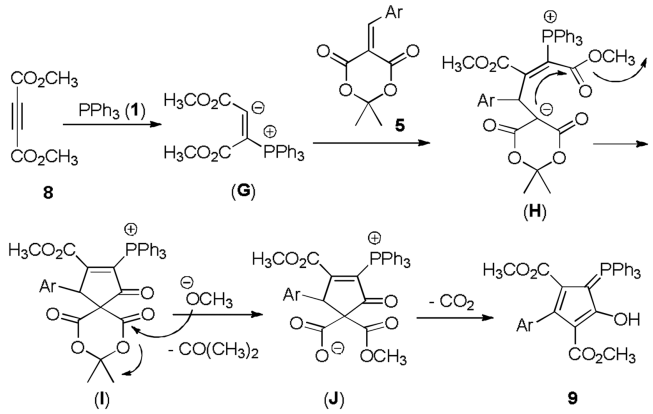


Fig. 4. Single crystal structure of the compound **9b**.

ring of Meldrum acid is opened in the reaction and a polysubstituted cyclopentadiene was constructed. From the single crystal structures, it can be seen that the ring of cyclopentadiene was in one plane and an H-bond exists between the hydroxyl group and the carbonyl group. It has been reported that triphenylphosphine promoted reaction of dimethyl but-2-ynedioate with arylidene *N,N'*-dimethylbarbituric acid afforded 2*H*-pyrano[2,3-*d*]pyrimidine derivatives and the highly substituted dimethyl 2-[(1,3-dimethyl-2,4,6-trioxotetrahydropyrimidin-5(2*H*)-ylidene)arylmethyl]-2-butenedioate under neutral conditions [68]. These results again showed that the nucleophilic phosphine-catalyzed reaction has very interesting molecular diversities.

For explaining the formation of the polysubstituted 5-(triphenyl-λ^5-phosphanylidene)cyclopentadienes **9a-9h**, a modified reaction mechanism was proposed in Scheme 2. At first, triphenylphosphine added to dimethyl but-2-ynedioate gave the active intermediate **G**, which in turn added to the benzylidene Meldrum acid to give the intermediate **H**. Then, the intramolecular substitution of carbanion to methoxide ion produced the cyclic intermediate **I**. The nucleophilic addition of the methoxide ion to the carbonyl group caused the ring-opening of Meldrum acid to form the intermediate J with elimination of acetone. Finally, the polysubstituted cyclopentadiene **9** was formed by decarboxylation process. The formation of the unstable spiro compound **I** with a triphenylphosphine cation might be the reason for the different results of dimethyl acetylenedicarboxylate to that of dimethyl hex-2-en-4-ynedioate in the three-component reaction.



Scheme 2. Proposed formation reaction for the compound **9a-9h**.

In summary, we have investigated the three-component reactions of triphenylphosphine, electron-deficient alkynes and active 1,1-diactivated alkenes such as arylidene Meldrum acids and *N,N'*-dimethylbarbituric acids. This reaction provided efficient synthetic protocols for the polysubstituted spiro compounds and cyclopentadiene derivatives. The most feature of this reaction is that the structurally similar arylidene Meldrum acids and *N,N'*-dimethylbarbituric acids displayed different regioselectivity and diastereoselectivity in the three-component reactions. It is interesting to find that triphenylphosphane not only acted as nucleophilic catalyst, but also as 5-(triphenyl-λ^5-phosphanylidene)ylidene scaffold retaining in the final product. The reaction has the advantages of using readily available reagents, mild reaction conditions, satisfactory yields, and molecular diversity with high regioselectivity and diastereoselectivity. The potential applications of this multicomponent reaction in synthetic and medicinal chemistry might be quite significant.

Declaration of competing interest

We declared there is no conflict of interest in this paper.

Acknowledgments

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:https://doi.org/10.1016/j.cclet.2019.10.042.

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