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$Rh_2(OAc)_4$ 催化的 Wittig-Type 烯基化反应: 一种合成烷基、芳基取代的亚烷基丙二酸酯的简便方法

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摘要 发展了一种羰基化合物与重氮丙二酸二甲酯在三苯基砷和催化量的 $Rh_2(OAc)_4$ 作用下发生一锅反应, 合成芳基、烷基取代的亚烷基丙二酸酯的新方法. 该方法的底物范围很广, 包括芳香醛和脂肪醛以及酮等羰基化合物, 都能够顺利地进行反应, 得到中等至优良的收率(59%～99%). 在 0.5 mol%的铑催化剂作用下, 能够实现克级反应. 初步的机理研究表明, 这一烯烃化反应是通过铑-卡宾转化为砷叶立德的途径实现的.

关键词 重氮化合物; 亚烷基丙二酸酯; 铑; 三苯基胂; Wittig 反应

$Rh_2(OAc)_4$ Catalyzed Wittig-Type Olefination: A Facile Access to Alkylidene and Arylidene Malonates

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Abstract In the presence of triphenylarsine and catalytic amount of $Rh_2(OAc)_4$, one pot reactions with carbonyl compounds and dimethyl diazomalonate give the corresponding alkylidene and arylidene malonates in moderate to excellent yields (59%～99%). The scope of the carbonyl compounds covered a broad range, including aromatic and aliphatic aldehydes and ketone. The reaction was easily scaled up to gram scale with 0.5 mol% rhodium catalyst loading. The preliminary mechanistic studies showed that the olefination proceeded via a rhodium-carbene transformed arsonium ylide pathway.

Keywords diazo compounds; alkylidene malonates; rhodium; triphenylarsine; Wittig reaction

1 Introduction

α,*β*-Unsaturated malonates are a series of very useful synthetic intermediates in which the C=C double bond is activated by conjugation with two electron-withdrawing groups, and are widely employed in Michael addition reaction and other transformations.[1] Although the arylidene malonates could be easily obtained through Knoevenagel condensation,[2] as to the alkylidene malonates, undesired side reactions are usually occurred under the weak basic condensation conditions. An alternative strategy to construct the C=C double bond is Wittig reaction.[3] However, the synthesis of traditional ylide also requires a basic condition.[4] Recently, electrophilic metal carbenes have been used for the preparation of ylides under neutral conditions, in order to avoid potential problem caused by basic

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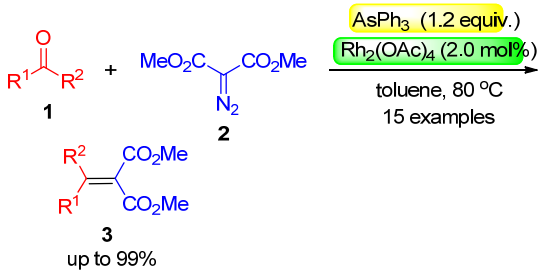
condition involved in traditional ylide chemistry.[5,6] In 1990, Huang and coworkers developed the stibonium ylide involved Wittig-type olefination of aldehydes and ketones with dimethyl diazomalonate catalyzed by Cu(I) to give the desired *α*,*β*-unsatuated diesters in good to excellent yields.[7] In 1993, they expand this reaction to telluronium ylide, and a variety of aromatic and aliphatic aldehydes were easily transformed to the corresponding *α*,*β*-unsaturated malonates.[8] However, the antimonides and tellurides were unstable and easily decomposed to toxic antimonides and tellurides, which greatly limited their further application. Our group has long been engaged in field of ylide chemistry.[9] We noticed that the triphenylarsine is much stable and less toxic than antimonides and tellurides. Herein, we report a practical new method that in a Rh(II) catalyzed Wittig-Type reaction, with triphenylarsine, the aldehydes and dimethyl diazomalonate give the corresponding alkylidene and arylidene malonates in 59%～99% yield.

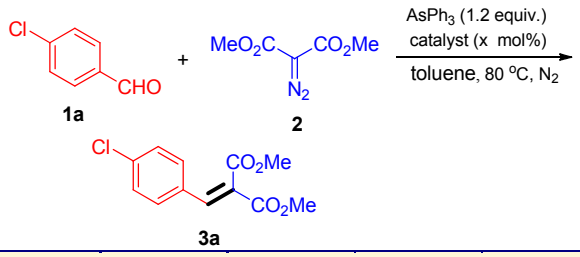


Scheme 1 Rh(II) catalyzed Wittig-type olefinations between carbonyl compounds and diazomalonate

2 Results and discussion

Initially, we found 4-chloro-benzaldehyde (**1a**) could react with dimethyl diazomalonate (**2**) in the presence of $AsPh_3$ and 10 mol% CuI to afford Wittig-type olefination product **3a** in 34% yield (Entry 1, Table 1). Further screening of other copper salts the yield could be improved to 75% (Entries 2～4). Some other metal salts such as Fe(TCP)Cl and Co(TCP) could also promote this reaction but only gave poor to moderate yields (Entries 5, 6). Rhodium compounds were proved much more effective than copper salts in the decomposition of dimethyl diazomalonate,[10] thus, we turned to employ $Rh_2(OAc)_4$ as catalyst, and to our delight, with 2.0 mol% $Rh_2(OAc)_4$, the reaction proceeded very efficient, leading to the olefination product in 89% yield (Entry 7). The ratio of **1a**/**2** was also investigated. As shown in Table 1, when 1.5 equiv. of **2** was used, the best yield was obtained (Entry 7). Changing the ratio of **1a**/**2** to 1.0/1.2 resulted in a dramatic decline of the yield (Entry 8). When 1.5 equiv of **1a** was used, only moderate yield of **3a** was afforded (Entry 9). The concentration of the substrate also affected the yield. When 1 mL of toluene was used as solvent, only 72% yield of **3a** was obtained, together with a number of undesired byproducts (Entry 10). Thus, the optimal reaction condition was developed as that **1a** (1.0 equiv.) reacted with **2** (1.5 equiv.) in the presence of $AsPh_3$ (1.2 equiv.) and 2.0 mol% $Rh_2(OAc)_4$ in toluene (2 mL) at 80 ℃ (Entry 7).

Table 1 Optimization of the reaction conditions[a]



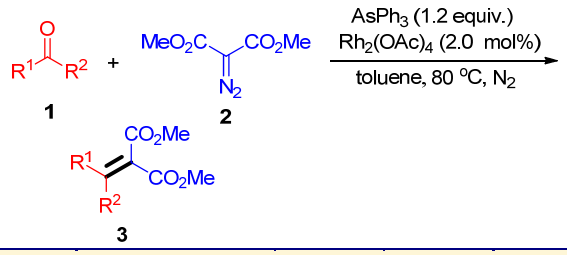
Entry	Catalysts	*x*/mol%	**1a**/**2**/equiv.	Yield[b]/%
1	CuI	10	1.0/1.5	34
2	$CuSbF_6$	10	1.0/1.5	49
3	$CuPF_6$	10	1.0/1.5	62
4	CuOTf	10	1.0/1.5	75
5	Fe(TCP)Cl	10	1.0/1.5	25
6	Co(TCP)	10	1.0/1.5	47
7	$Rh_2(OAc)_4$	2	1.0/1.5	89
8	$Rh_2(OAc)_4$	2	1.0/1.2	75
9	$Rh_2(OAc)_4$	2	1.5/1.0	50
10[c]	$Rh_2(OAc)_4$	2	1.0/1.5	72

[a] $AsPh_3$ (1.2 equiv., 0.48 mmol), toluene (2 mL), 80 ℃; **2** was slowly added within 8 h via a syringe pump. [b] Isolated yield. [c] *C*=0.4 mol/L.

To explore the generality of this reaction, a variety of aldehydes were investigated under the above optimal conditions. As shown in Table 2, aromatic aldehyde substituted by 2-NO_2 group afforded the *α*,*β*-unsaturated diester **3b** in 69% yield, which is very difficult to obtained through Knoevenagel condensation pathway (Entry 2). Aromatic aldehydes bearing electron-donating group, such as 4-Me led to the corresponding **3c** in 65% yield (Entry 3). The substrate with bromo substituent was studied, and 85% yield was obtained (Entry 5). 2-Furaldehyde also worked well to give the desired ester **3f** in 99% yield (Entry 6). Remarkably, both cinnamyl and dihydrocinnamyl aldehydes were also suitable substrates, resulting the *α*,*β*-unsaturated diesters **3g** and **3h** in 75% and 86% yields, respectively (Entries 7, 8). Other aliphatic aldehydes were tolerated in this reaction, affording the corresponding alkylidene malonates in good to moderate yields. For examples, the acyclic aldehydes **1i** and **1j** gave **3i** and **3j** in 80% and 59% yields, respectively (Entries 9, 10). Furthermore, cyclic aldehydes were also suitable substrates, leading to the cyclopentyl and cyclohexyl substituted unsaturated diesters in 71% and 61% yields (Entries 11, 12). Aldehyde bearing functional groups, such as OBn, could also obtain good yield (Entry 13). The terephthalaldehyde was employed as substrate and bis-olefination product was obtained in 20% yield (Entry 14). Notably, when ketone **1l** was employed as substrate, the reaction worked smoothly, producing the tetrasubstituted olefin **3o** in 76% yield (Entry 15).

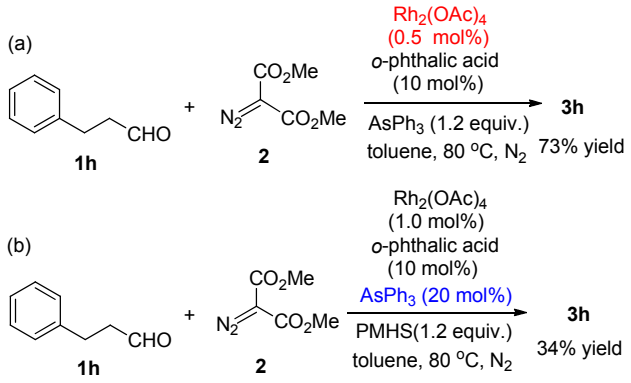
In order to make the reaction more practical, the scale-up reaction was carried out under lowered catalyst loading. When dihydrocinnamyl aldehyde was employed

Table 2 Substrate scope[a]



Entry	R^1	R^2	3	Yield[b]/%
1	4-ClC_6H_4	H	**3a**	89
2	2-$NO_2C_6H_4$	H	**3b**	69
3	4-MeC_6H_4	H	**3c**	65
4	4-$MeOC_6H_4$	H	**3d**	44
5	4-BrC_6H_4	H	**3e**	85
6	2-Furyl	H	**3f**	99
7[c,d]	$C_6H_5CH{=}CH$	H	**3g**	75
8[c,d]	$C_6H_5CH_2CH_2$	H	**3h**	86
9[c]	$CH_3(CH_2)_6$	H	**3i**	80
10[c,d]	$CH_3(CH_2)_4$	H	**3j**	59
11[c]	Cyclopentyl	H	**3k**	71
12[c,d]	Cyclohexyl	H	**3l**	61
13[c,d]	$BnO(CH_2)_3$	H	**3m**	67
14	OHC–C$_6$H$_4$–CHO		**3n**	20
15	Ph	CF_3	**3o**	76

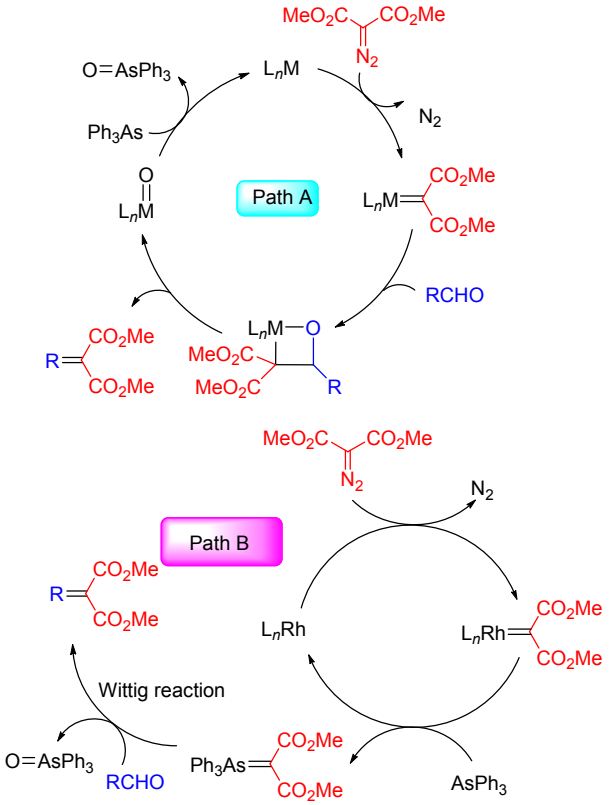
[a] **1** (0.4 mmol), **2** (0.6 mmol), $AsPh_3$ (0.48 mmol), toluene (2 mL), 80 ℃; **2** was slowly added within 8 h via a syringe pump. [b] Isolated yield. [c] **2** (0.8 mmol), with 10 mol% of *o*-phthalic acid as additive. [d] Toluene (4 mL).



Scheme 2 Gram scale synthesis and attempt for catalytic triphenylarsine reaction

as substrate, with 0.5 mol% of $Rh_2(OAc)_4$ as catalyst, the reaction gave the gram scale olefin product in 73% yield (Scheme 2a). Considering that Ph_3As could be regenerated through the reduction of $Ph_3As{=}O$ by employing polymethylhydrosiloxane (PMHS) as the reductive reagent, we tried the reaction by using 20 mol% Ph_3As in the presence of 1.2 equiv. of PMHS with slow addition of diazo compound. It was disappointing that in this case, the reaction proceeded very slowly, yielding the product **3h** in 34% yield after prolonging the reaction time (Scheme 2b).

For olefination of aldehydes with diazomalonate catalyzed by a transition metal complex, there are two possible pathways[5,11] as shown in Scheme 3. In path A, the diazo compound firstly decomposed in the presence of metal catalyst to form metal carbene. Then the metal carbene reacted with the aldehyde via a four membered ring transition state to give the olefin and release the oxidized metal complex. The Ph_3As served as a reductant to regenerate the metal catalyst. On the other hand, in path B, the *in situ* generated metal carbene reacted with Ph_3As to give the corresponding arsonium ylide, which was further transformed to olefin via a Wittig reaction mechanism.

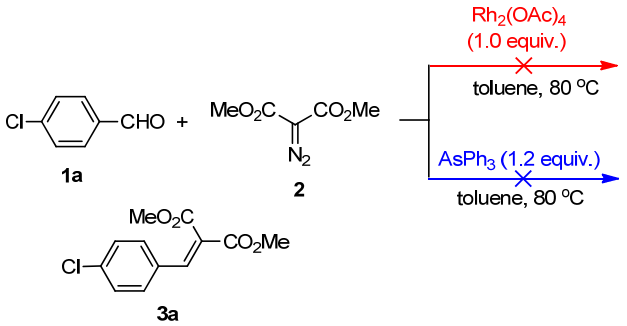


Scheme 3 Possible reaction pathways

In order to explain the mechanism of this reaction, two control experiments were conducted (Scheme 4). It was found that, in the absence of Ph_3As, the desired olefination product could not be detected in the reaction of **1a** with **2** even when a stoichiometric amount of $Rh_2(OAc)_4$ was used. This observation ruled out the possibility of path A. In addition, **3a** could also not be produced without $Rh_2(OAc)_4$, which suggests that the olefination reaction might undergo a rhodium-carbene transformed to arsonium ylide process (path B).

3 Conclusions

In summary, we have developed the triphenylarsine involved Wittig-type reaction of aromatic and aliphatic aldehydes with diazomalonate dimethyl ester **2** by using 2 mol% $Rh_2(OAc)_4$ as catalyst under neutral reaction condition. Many different aldehydes, including those with different types of substituents on the phenyl ring, heteroaro-



Scheme 4 Control experiments

matic, acyclic and cyclic aliphatic aldehydes, were suitable substrates, providing a variety of alkylidene and arylidene malonates in good to excellent yields. The reaction was practical that the procedure was quite simple (in one pot), and it was easily scaled up to gram scale with 0.5 mol% rhodium catalyst loading. The preliminary mechanistic studies showed that the olefination proceeded via rhodium-carbene transformed arsonium ylide route. The extension of this method to other carbonyl compounds and its applications are in progress in our laboratory.

4 Experimental section

4.1 Instruments and reagents

Unless stated otherwise, all reactions were carried out under an atmosphere of nitrogen using standard Schlenk techniques. All solvents and reagents were obtained from commercial sources and were purified according to standard procedures before use. The substrates used in the experiments were distilled. ^{1}H NMR spectra were recorded on a VARIAN Mercury 300 MHz or a VARIAN Mercury 400 MHz spectrometer in chloroform-*d*. All signals were reported with the internal TMS signal at δ 0.0 or chloroform signal at δ 7.26 as a standard. ^{13}C NMR spectra were recorded on a VARIAN Mercury 75 MHz or 100 MHz spectrometer in chloroform-*d*. All signals are reported with the internal chloroform signal at δ 77.0 as a standard. IR spectra were recorded on a Bruker-Tensor 27; Mass spectra were determined on an Agilent 6224 TOF LC/MS (ESI) mass spectrometer.

4.2 Experimental method

A mixture of $Rh_2(OAc)_4$ (0.008 mmol), Ph_3As (0.48 mmol) and *o*-phthalic acid (0.04 mmol) were subjected to toluene (1 mL) under an atmosphere of nitrogen. Then, the aldehydes (0.4 mmol) were added to the mixture of catalyst. The mixture was then add to 80 ℃, **2** (0.8 mmol) in 1 mL of anhydrous toluene was then added dropwise. The resulting suspension was allowed to stir at corresponding temperature. Upon disappearance of aldehydes as confirmed by thin-layer chromatography, the reaction was filtered through a glass funnel with a thin layer (20 mm) of silica gel (100～200 mesh) with CH_2Cl_2 (approx 150 mL). The filtrate was concentrated under reduced pressure. The residue was purified by flash chromatography (EtOAc/petroleum, V∶V=1/40) to afford the products.

Compounds **3a**～**3j** and **3l** are previously reported.[12]

Dimethyl 2-(cyclopentylmethylene)malonate (**3k**): Colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 6.94 (d, J=10.8 Hz, 1H), 3.83 (s, 3H), 3.78 (s, 3H), 2.82～2.73 (m, 1H), 1.94～1.85 (m, 2H), 1.76～1.64 (m, 4H), 1.45～1.33 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.1, 164.4, 154.8, 126.1, 52.2, 52.1, 40.4, 33.0, 25.5; IR (film) ν: 2953, 2869, 1725, 1641, 1248, 1222 cm^{-1}. HRMS (ESI) calcd for $C_{11}H_{17}O_4$ $(M+H)^+$ 213.1127, found 213.1118.

Dimethyl 2-(4-(benzyloxy)butylidene)malonate (**3m**): Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.35～7.29 (m, 5H), 7.06 (t, J=8.0 Hz, 1H), 4.49 (s, 2H), 3.80 (s, 3H), 3.78 (s, 3H), 3.49 (t, J=6.2 Hz, 2H), 2.45～2.40 (m, 2H), 1.84～1.79 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.8, 164.3, 149.9, 138.3, 128.4, 128.2, 127.61, 127.57, 72.9, 69.2, 52.3, 52.2, 28.4, 26.8; IR (film) ν: 2952, 2858, 1726, 1645, 1261, 1225 cm^{-1}. HRMS (ESI) calcd for $C_{16}H_{21}O_5$ $(M+H)^+$ 293.1389, found 293.1388.

Tetramethyl 2,2'-(1,4-phenylenebis(methanylylidene))-dimalonate (**3n**): White foam. m.p. 153.1～154.6 ℃; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.74 (s, 2H), 7.44 (s, 4H), 3.86 (s, 6H), 3.85 (s, 6H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.7, 164.2, 141.4, 134.8, 129.7, 126.9, 52.8; IR (neat) ν: 3013, 2955, 2924, 2853, 1724, 1646, 1621, 1551, 1533, 1514, 1434, 1415, 1374, 1338, 1301, 1259, 1220, 1193, 1143, 1063, 982, 967, 939, 841, 826, 790, 755, 736, 701, 596, 574, 516, 410 cm^{-1}; HRMS-ESI $[M+N]^+$ calcd for $C_{18}H_{19}O_8$ 363.1074, found 363.1074.

Dimethyl 2-(2,2,2-trifluoro-1-phenylethylidene)malonate (**3o**): Colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43～7.40 (m, 3H), 7.304～7.301 (m, 2H), 3.91 (s, 3H), 3.55 (s, 3H); ^{19}F NMR (282 MHz, $CDCl_3$) δ: −63.5 (s, 1F); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 163.494, 163.486, 162.5, 139.1, 138.8, 132.41, 132.37, 130.796, 130.785, 129.7, 128.5, 128.33, 128.32, 123.0, 120.2, 53.3, 52.9; IR (film) ν: 2958, 1742, 1236, 1211 cm^{-1}. HRMS (ESI) calcd for $C_{13}H_{15}F_3NO_4$ $(M+NH_4)^+$ 306.0953, found 306.0943

Supporting Information ^{1}H NMR and ^{13}C NMR spectra for all new compounds. The Supporting Information is available free of charge via the Internet at http://sioc-journal.cn/.

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