



Nickel-catalyzed hydroalkylation of 1,3-dienes with malonates using a homoallyl carbonate as the 1,3-diene and hydride source

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ARTICLE INFO

Article history:

Received 18 December 2020

Revised 3 February 2021

Accepted 9 February 2021

Available online 12 February 2021

Keywords:

Nickel Catalysis

Hydroalkylation

1,3-Dienes

Malonates

Homoallyl carbonates

ABSTRACT

The use of a malonate nucleophile in the transition metal-catalyzed hydroalkylation of 1,3-dienes remains immature. Herein, we report the nickel-catalyzed hydroalkylation of 1,3-dienes with malonates using a homoallyl carbonate as the 1,3-diene and hydride source. A broad range of homoallyl carbonates and malonate derivatives were well tolerated under a Ni/DPEphos catalyst system, providing the corresponding 1,2-hydroalkylation products in 40–94% yields with excellent regioselectivity (32 examples). We also suggested the possible reaction mechanism for the nickel-catalyzed hydroalkylation of *in situ* generated 1,3-dienes with malonates.

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The catalytic hydroalkylation of unsaturated hydrocarbons by means of a transition metal catalyst presents an attractive strategy for the formation of carbon–carbon bond in an efficient and atom economical manner [1]. Due to the presence of an alkene group on the resulting alkylation products, the catalytic hydroalkylation of 1,3-dienes offers a rationale route for the manipulation of complex organic molecules [2]. Therefore, this type of reaction has received continuous interests from the synthetic community and a wide variety of C–pronucleophiles, such as active methylene compounds [3], simple carbonyl compounds [4], oxazolones [5], hydrazones [6], and others [7,1b], were attempted to employ in the hydroalkylation of 1,3-dienes using nickel and palladium as the representative catalysts. However, attentions have been sporadically paid to the use of a malonate nucleophile, which would lead to a valuable synthetic intermediate in organic synthesis [8]. Hata and co-workers reported the palladium-catalyzed hydroalkylation of 1,3-butadiene with malonates, even though telomerization and 1,2- and 1,4-additions occurred during the catalysis (Scheme 1a) [9]. After the pioneering work, Moberg, Jolly, and Hartwig independently disclosed the nickel- and palladium-catalyzed hydroalkylation of 1,3-dienes, such as 1,3-butadiene, 2,3-dimethylbutadiene, 1,3-cyclohexadiene, and 1,3-cyclopentadiene, with a limited number of malonates as a carbon nucleophile [10]. Recently, Malcolmson and co-workers reported the palladium-cat-

alyzed hydroalkylation of a series 1,3-dienes with activated C–pronucleophiles, providing the hydroalkylation products in up to 95% yield with high enantioselectivity (Scheme 1b) [11]. However, only Meldrum's acid derivatives can be utilized as a malonate analogue in this palladium catalysis. Given these research precedents, the transition metal-catalyzed hydroalkylation of 1,3-dienes with malonates is still immature in terms of the generality of 1,3-dienes as well as malonates.

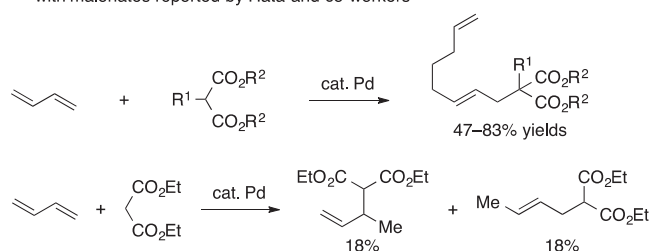
We have recently succeeded in the development of the nickel-catalyzed hydroarylation of *in situ* generated 1,3-dienes with arylboronic acids using a secondary homoallyl carbonate as a surrogate for the 1,3-diene and hydride source [12]. This method features the use of homoallyl carbonates, which is readily prepared by the allylation of aldehydes [13] followed by the formation of carbonate esters, as both 1,3-dienes and hydride sources. To address the above-mentioned issue in the transition metal-catalyzed hydroalkylation of 1,3-dienes, we sought to investigate the hydroalkylation of 1,3-dienes with malonates based on the methodology we developed. Herein, we report the nickel-catalyzed hydroalkylation of *in situ* generated 1,3-dienes with a malonate using a secondary homoallyl carbonate as the 1,3-diene and hydride source (Scheme 1c).

We initially examined the optimization of the reaction conditions using secondary homoallyl carbonate **1a** as the 1,3-diene and hydride source (Table 1). At the outset of the optimization study, dimethyl methylmalonate (**2a**) was subjected to a catalyst system consisting of 10 mol% Ni(cod)₂ and 20 mol% P(*p*-MeOC₆H₄)₃, which was proved to be effective for the previous

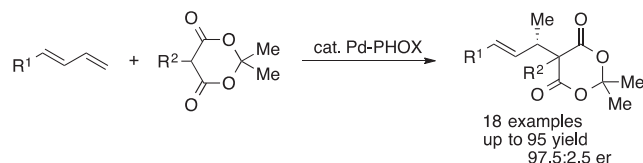
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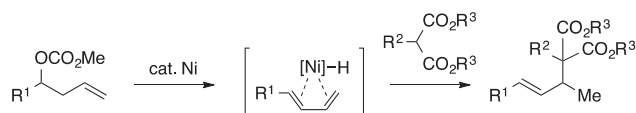
a) Pioneering examples for the catalytic hydroalkylation of 1,3-butadiene with malonates reported by Hata and co-workers



b) Pd-Catalyzed hydroalkylation of a series of 1,3-dienes with Meldrum's acid derivatives



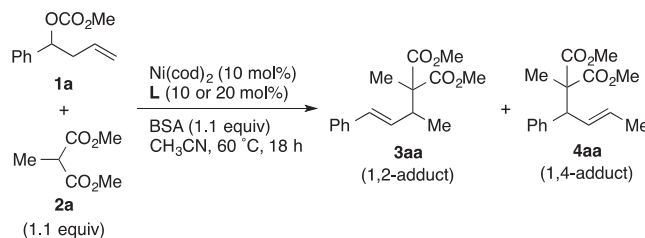
c) **This work:** Ni-Catalyzed hydroalkylation of *in situ* generated 1,3-dienes with malonates



Scheme 1. Transition metal-catalyzed hydroalkylation of 1,3-dienes with a malonate and its analogue.

hydroarylation reaction [12], in the presence of *N,O*-bis(trimethylsilyl)acetamide (BSA) as a base (Table 1, entry 1). The use of $P(p\text{-tol})_3$ and PPh_3 as the ligands afforded the comparable yield and regioselectivity (Entries 2 and 3). Electron-deficient and sterically congested monophosphine ligands were totally ineffective in this nickel catalysis (Entries 4 and 5). We next tested several bidentate phosphine ligands (Entries 6–14). The reaction with 10 mol% DPPE and DPPP failed to give desired hydroalkylation product **3aa** (Entries 6 and 7). Nickel catalyst systems derived from DPPB, DPPPent, DPPF exclusively produced **3aa** in 13–46% yields (Entries 8–10). When 10 mol% DPEphos was used as the ligand, hydroalkylation product **3aa** was obtained in 82% yield with excellent regioselectivity (Entry 11). Xantphos and NiXantphos also facilitated the hydroalkylation reaction to give **3aa** in 78% and 67% yield, respectively, even though the formation of small amount of 1,4-hydroalkylation product **4aa** was observed (Entries 12 and 13). BINAP was not beneficial as a bisphosphine ligand in the present catalyst system (Entry 14). Additionally, we also examined the reaction in the presence of bidentate nitrogen ligands such as 4,4'-di-*t*-Bu-bipyridine and 1,10-phenanthroline (Entries 15 and 16). However, the desired hydroalkylation reaction did not take place under the reaction conditions. The screening revealed that DPEphos is the suitable choice of the ligand in the present nickel catalysis. To achieve the improvement of the yield of **3aa**, we investigated further optimization of the reaction conditions using DPEphos as the optimal ligand. The reaction was conducted at 80 °C to give **3aa** in maintained yield (Entry 17). Fortunately, the

Table 1
Optimization of reaction conditions.^a



Entry	L	Yield ^b (%) of 3aa	Yield ^b (%) of 4aa	3aa:4aa ^c
1	$P(p\text{-MeOC}_6\text{H}_4)_3$	35	1	98:2
2	$P(p\text{-tol})_3$	41	1	98:2
3	PPh_3	44	1	98:2
4	$P(p\text{-CF}_3\text{C}_6\text{H}_4)_3$	<1	<1	–
5	$P(o\text{-tol})_3$	<1	<1	–
6	DPPE	<1	<1	–
7	DPPP	<1	<1	–
8	DPPB	46	<1	>99:1
9	DPPPent	32	<1	>99:1
10	DPPF	13	<1	>99:1
11	DPEphos	82	1	99:1
12	Xantphos	78	6	93:7
13	NiXantphos	67	5	93:7
14	<i>rac</i> -BINAP	<1	<1	–
15	4,4'-di- <i>t</i> -Bu-2,2'-bpy	<1	<1	–
16	1,10-Phenanthroline	<1	<1	–
17 ^d	DPEphos	83	1	99:1
18 ^e	DPEphos	97 (88) ^f	1	99:1

^a Reaction conditions: **1a** (0.25 mmol), **2a** (0.275 mmol), $Ni(cod)_2$ (0.025 mmol), **L** (0.05 mmol (20 mol%) for monophosphine ligands and 0.025 mmol (10 mol%) for bidentate ligands), *N,O*-bis(trimethylsilyl)acetamide (BSA) (0.275 mmol) in CH_3CN (0.25 M) at 60 °C for 18 h.

^b The yields were determined by 1H NMR analyses of the crude reaction mixture using dibromomethane as an internal standard.

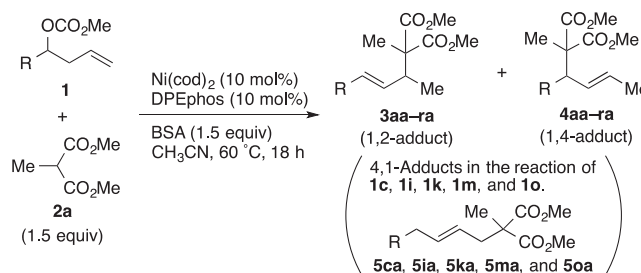
^c Determined by 1H NMR analyses of the crude reaction mixture.

^d The reaction was performed at 80 °C.

^e With 1.5 equiv of **2a** and BSA.

^f Isolated yield.

Table 2
Scope of secondary homoallyl carbonates.^a



Entry	1	R	3	Yield ^b (%)	3:4 ^c
1 ^d	1a	Ph	3aa	94	>99:1
2	1b	4-MeC ₆ H ₄	3ba	87	99:1
3 ^e	1c	4-MeOC ₆ H ₄	3ca	87 ^f	99:1
4	1d	4-CF ₃ C ₆ H ₄	3da	83	>99:1
5	1e	4-(CO ₂ Me)C ₆ H ₄	3ea	92	>99:1
6	1f	4-ClC ₆ H ₄	3fa	70	99:1
7	1g	4-FC ₆ H ₄	3ga	81	99:1
8	1h	3-MeC ₆ H ₄	3ha	87	>99:1
9 ^e	1i	piperonyl	3ia	87 ^f	>99:1
10	1j	3-CNC ₆ H ₄	3ja	86	>99:1
11 ^e	1k	2-MeOC ₆ H ₄	3ka	85 ^f	>99:1
12 ^g	1l	2-CF ₃ C ₆ H ₄	3la	51	>99:1
13 ^e	1m	6-MeO-2-naphthyl	3ma	89 ^f	99:1
14 ^g	1n	3-pyridyl	3na	55	>99:1
15 ^e	1o	3-thienyl	3oa	80 ^f	97:3
16 ^g	1p	2-benzofuranyl	3pa	49	95:5
17	1q	cyclohexyl	3qa	77	>99:1
18	1r	phenethyl	3ra	76	95:5

^a Reaction conditions: **1** (0.25 mmol), **2a** (0.375 mmol), $\text{Ni}(\text{cod})_2$ (0.025 mmol), DPEphos (0.025 mmol), BSA (0.375 mmol) in CH_3CN (0.25 M) at 60°C for 18 h.

^b Combined isolated yield of **3** and **4**.

^c Determined by ^1H NMR analyses of the crude reaction mixture.

^d 3 mmol scale.

^e 4,1-Hydroalkylation product **5** was observed in the ^1H NMR analyses of crude reaction mixture (**5ca**: 1% yield, **5ia**: 2% yield, **5ka**: 1% yield, **5ma**: 6% yield, **5oa**: 6% yield).

^f Combined isolated yield of **3**, **4**, and 4,1-hydroalkylation products **5**.

^g 20 mol% $\text{Ni}(\text{cod})_2$ and 20 mol% DPEphos were used.

desired product **3aa** was obtained in 97% NMR yield (88% isolated yield) with excellent regioselectivity, when 1.5 equivalents of **2a** and BSA were employed in the reaction system (Entry 18).

We investigated the scope of secondary homoallyl carbonates **1** with dimethyl methylmalonate (**2a**) as the model nucleophile under the optimized reaction conditions (Table 2). The reaction of homoallyl carbonate **1a** with **2a** could be conducted on 3 mmol scale without loss of the catalytic performance, providing **3aa** in 94% yield with >99:1 regioselectivity (Table 2, entry 1).

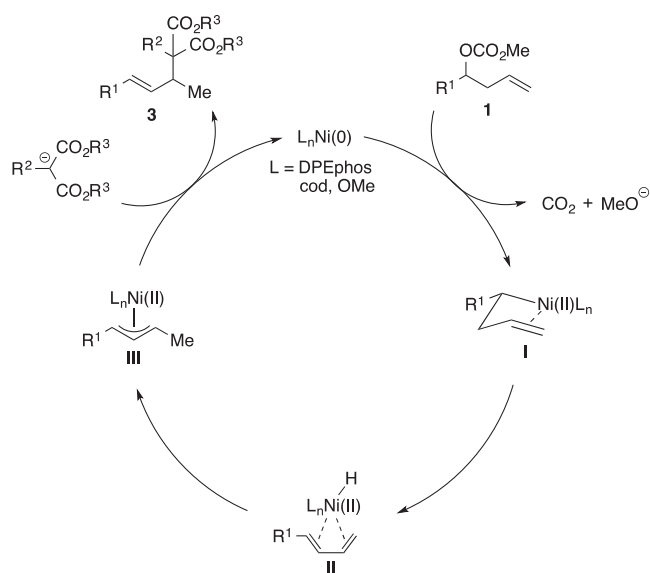
The introduction of electron-donating and -withdrawing substituents and halogen atoms at the para position on their aromatic rings was well tolerated to deliver the corresponding 1,2-hydroalkylation products **3ba–ga** in 70–92% yields with excellent regioselectivity (Entries 2–7). Desired hydroalkylation products **3ha–ja** having 3-methylphenyl, piperonyl, and 3-cyanophenyl groups could be obtained without significant influence to the yield and regioselectivity (Entries 8–10). Homoallyl carbonate **1k** containing a methoxy group at the ortho position on the aromatic ring reacted with **2a** to give desired product **3ka** in 85% yield (Entry 11). However, the introduction of a trifluoromethyl substituent at the ortho position (**1l**) affected the reaction efficiency, resulting in moderate yield of **3la** (Entry 12). When homoallyl carbonate **1m** bearing 6-MeO-2-naphthalene ring was subjected to the reaction system, desired product **3ma** was obtained in 89% yield with 99:1 regioselectivity (Entry 13). Homoallyl carbonates **1n–p** having heterocyclic rings, such as pyridine, thiophene, and benzofuran, underwent the reaction to give the corresponding hydroalkylation

products **3na–pa** in 49–80% yields, even though increasing the loading amount of the catalyst was required for the reaction of **1n** and **1p** (Entries 14–16). In contrast to the previous hydroarylation reaction using a combination of $\text{Ni}(\text{cod})_2$ and $\text{P}(p\text{-MeOC}_6\text{H}_4)_3$ as the catalyst [12], the present nickel catalyst system enabled the use of aliphatic substrates **1q** and **1r**, providing the hydroalkylation products **3qa** and **3ra** in 77% and 76% yield, respectively (Entries 17 and 18). During the scope of a series of homoallyl carbonates, we confirmed that the reactions of homoallyl carbonates bearing electron-rich aromatic rings, such as **1c**, **1i**, **1k**, **1m**, and **1o**, are prone to give the corresponding 4,1-hydroalkylation products **5ca**, **5ia**, **5ka**, **5ma**, and **5oa** in 1%, 2%, 1%, 6%, 6% NMR yields, respectively.

We next turned our attention to reveal the scalability of malonate derivatives **2** in the nickel-catalyzed hydroalkylation of *in situ* generated 1,3-dienes (Table 3). Diethyl methylmalonate (**2b**) and diisopropyl methylmalonate (**2c**) also engaged in the reaction to give the desired hydroalkylation products **3ab** and **3ac** in 82% and 83% yield, respectively (Table 3, Entries 1 and 2). The effect of substituents on the active methylene carbon of malonates was also surveyed (Entries 3–10). Diethyl malonate derivatives **2d–h** bearing a series of linear alkyl chains participated in the hydroalkylation reaction, providing the corresponding hydroalkylation products **3ad–ah** in 76–88% yields (Entries 3–7). It is worth mentioning that the functionalized linear alkyl chains such as cyanoethyl (**2f**), chloropropyl (**2g**), and 3-oxobutyl (**2h**) groups were well tolerated under the reaction conditions. When diethyl isobutylmalonate (**2i**)

Table 3
Scope of malonates.^a

Entry	1	R ¹	R ²	3	Yield ^b (%)	3:4 ^c
1	2b	Me	Et	3ab	82	>99:1
2	2c	Me	<i>i</i> Pr	3ac	83	>99:1
3	2d	Et	Et	3ad	78	>99:1
4	2e	<i>n</i> Bu	Et	3ae	78	>99:1
5	2f	–CH ₂ CH ₂ CN	Et	3af	83	>99:1
6	2g	–CH ₂ CH ₂ CH ₂ Cl	Et	3ag	88	>99:1
7	2h	–CH ₂ CH ₂ COMe	Et	3ah	76	>99:1
8	2i	<i>i</i> Bu	Et	3ai	69	>99:1
9	2j	F	Et	3aj	59	98:2
10	2k	Ph	Et	3ak	40	>99:1
11	2l	H	Me	3al	77	>99:1
12	2m	H	Et	3am	78	>99:1
13	2n	H	Bn	3an	50	>99:1
14	2o	H	<i>t</i> Bu	3ao	76	>99:1

^a Reaction conditions: **1a** (0.25 mmol), **2** (0.375 mmol), Ni(cod)₂ (0.025 mmol), DPEphos (0.025 mmol), BSA (0.375 mmol) in CH₃CN (0.25 M) at 60 °C for 18 h.^b Combined isolated yield of **3** and **4**.^c Determined by ¹H NMR analyses of the crude reaction mixture.**Scheme 2.** Putative reaction mechanism.

was used as a nucleophile, hydroalkylation product **3ai** was obtained in 69% yield (Entry 8). Diethylmalonate analogues that bear not only alkyl substituents but also a fluorine atom (**2j**) and a phenyl group (**2k**) on the active methylene carbon were also compatible with the reaction to afford the hydroalkylation product **3aj** and **3ak** in 59% and 40% yield, respectively (Entries 9 and 10). The simple dialkyl malonates such as dimethyl malonate (**2l**) and diethyl malonate (**2m**) also underwent the reaction to give the desired hydroalkylation products **3al** and **3am** in satisfactory yields (Entries 11 and 12). Furthermore, the catalyst system also accommodated sterically congested dibenzyl malonate (**2n**) and

di-*tert*-butyl malonate (**2o**), producing the hydroalkylation products **3an** and **3ao** in 50% and 76% yield, respectively (Entries 13 and 14).

We show the putative reaction mechanism of the nickel-catalyzed hydroalkylation of *in situ* generated 1,3-dienes with malonates in Scheme 2. Given the Sigman's study [14] and our previous work [12], the alkene-directed oxidative addition of Ni(0) species to the C–O bond on homoallyl carbonates **1** would take place to generate the secondary alkyl nickel species **I**. Subsequent β-hydrogen elimination from the resultant alkyl nickel species **I** would produce the diene-coordinated Ni–H species **II** [15,16]. The Ni–H species **II** would undergo migratory insertion to generate π-allylnickel intermediate **III** [4a,b,6a,b,17]. As shown in Table 2, small amount of 4,1-hydroalkylation products were formed in some cases. We assume that the migratory insertion of the internal alkene moiety of the 1,3-dienes to the Ni–H species might be a cause of the formation of the side products. Finally, nucleophilic addition of the malonate anion would occur at the less hindered terminus of the unsymmetrical π-allylnickel species **III** to provide the desired hydroalkylation products **3** [18]. We observed the formation of 1,4-hydroalkylation products during the scope of homoallyl carbonates and malonate derivatives. These results would be attributed to the formation of the unsymmetrical π-allylnickel intermediate **III** and subsequent nucleophilic addition with a malonate nucleophile.

In conclusion, we have developed the nickel-catalyzed hydroalkylation of 1,3-dienes with malonate nucleophiles using readily accessible homoallyl carbonates as the 1,3-diene and hydride source. The Ni(cod)₂/DPEphos catalyst system found to be effective for the hydroalkylation of *in situ* generated 1,3-dienes with malonates in terms of the reaction yields and regioselectivity, providing a broad range of 1,2-hydroalkylation products. The nickel catalysis would proceed via regioselective addition of the malonate nucleophile to the unsymmetrical π-allylnickel intermediate generated from the diene-coordinated Ni–H species.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work was supported by a Grant-in-Aid for Scientific Research (C) from the Japan Society for the Promotion of Science (17K05794 and 20K05500).

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.tetlet.2021.152916>.

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