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# Rhodium-catalyzed addition of arylboron compounds to nitriles, ketones, and imines

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**Abstract**

The rhodium-catalyzed addition reactions of sodium tetraphenylborate and arylboronic acids to nitriles, ketones, and imines were examined. The reaction of nitriles could be carried out efficiently in the presence of a catalyst system of $[RhCl(cod)]_2$–dppp and $H_2O$ to give the corresponding monoarylated products selectively. Although unactivated ketones and imines are known to be poor electrophiles for rhodium-catalyzed arylation, the phenylation of them with use of sodium tetraphenylborate proceeded smoothly in the presence of $[RhCl(cod)]_2$ and Rh(acac)(cod) as catalysts, respectively. The addition of $NH_4Cl$ was found to be crucial to effectively conduct the reaction of ketones and imines.


*Keywords:* Rhodium catalyst; Arylation; Arylboron compounds; Nitriles; Ketones; Imines

## 1. Introduction

The rhodium-catalyzed nucleophilic addition reactions of organoboron and -stannane reagents to carbon-heteroatom multiple bonds are now recognized to be highly useful tools for C–C bond formation [1–6]. In these reactions, the mild, weakly nucleophilic organometallic reagents are effectively activated under rhodium catalysis to react readily with aldehydes and activated imines possessing electron-withdrawing groups, such as sulfonyl substituents, on their nitrogen (Scheme 1). However, in contrast to the additions toward the reactive electrophiles, those to ketones [4,7,8], unactivated imines [3,4,9], and nitriles [10,11] have been considered to be sluggish.

Meanwhile, in the course of our study of rhodium-catalyzed arylation reactions [12–15], we observed that treatment of benzonitrile with sodium tetraphenylborate can bring about the efficient addition of the phenylboron reagent to the C–N triple bond to produce the corresponding imine, and the product successively undergoes *ortho*-phenylation via C–H bond cleavage under the conditions employed (path A in Scheme 2) [16]. This is a rare example of rhodium-catalyzed intermolecular nucleophilic addition to nitriles [10,11]. We report herein our new findings that the reaction of nitriles to yield monophenylation products can be performed selectively under appropriate conditions with suppressing the *ortho*-arylation (path B in Scheme 2).

Moreover, a number of ketones and unactivated imines have also been found to undergo phenylation by the rhodium catalysis.

## 2. Results and discussion

As reported previously, treatment of sodium tetraphenylborate (**1**) (0.5 mmol) with benzonitrile (**2a**) (2 mmol) in the presence of $[RhCl(cod)]_2$ (cod = 1,5-cyclooctadiene, 0.005 mmol) and dppp (1,3-bis(diphenylphosphino)propane, 0.01 mmol) in *o*-xylene at 120 °C for 44 h under nitrogen gave benzophenone imine (**3a**) in 21% yield, along with di- (0.09 mmol, 36% based on the amount of **1** used) and triphenylated products (0.02 mmol, 11%) (Scheme 2 and Entry 1 in Table 1) [16]. To our surprise, addition of

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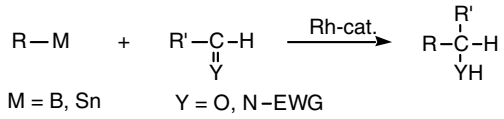
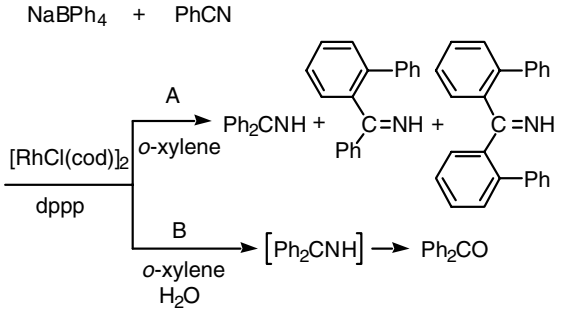


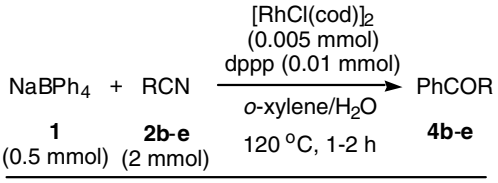

Scheme 1. Addition of organoboron and -stannane reagents to aldehydes and activated imines.




Scheme 2. Reaction of sodium tetraphenylborate with benzonitrile.

$H_2O$ (0.5 mL) completely suppressed the *ortho*-phenylation [9] and significantly promoted the reaction to afford **3a** in 91% yield within 1 h (Entry 2). Under the conditions with $H_2O$, part of **3a** was hydrolyzed to benzophenone (**4a**). During the post-treatment for product isolation by column chromatography, the imine **3a** was completely hydrolyzed to give **4a** in 85% yield. Decreasing the amount of **2a** somewhat reduced the product yield (Entry 3). The omission of dppp (Entry 4) or the use of $PPh_3$ in place of it (Entry 5) resulted in low efficiency of the phenylation.

The reaction using other nitriles **2b–e** with **1** was next examined (Scheme 3). 4-Methylbenzonitrile (**2b**) underwent phenylation by **1** to give the corresponding ketone **4b** in 98% isolated yield. The reaction of 4-chlorobenzonitrile (**2c**) also proceeded efficiently, but in this case, *ortho*-phenylation could not be suppressed completely even in the presence of $H_2O$ to give a minor amount of diphenylated product (ca. 20%) other than monophenylated one **4c** (63%). Not only aromatic but also aliphatic nitriles underwent phenylation upon treatment with **1** under the




| 2 | Product, Yield (%)[a] |
|---|---|
| **2b**: R = 4-$CH_3C_6H_4$ | **4b**: R = 4-$CH_3C_6H_4$, 98 |
| **2c**: R = 4-$ClC_6H_4$ | **4c**: R = 4-$ClC_6H_4$, 63 |
| **2d**: R = $PhCH_2$ | **4d**: R = $PhCH_2$, 120 |
| **2e**: R = Et[b] | **4e**: R = Et, 73 |

[a] Isolated yield. [b] With **2e** (5 mmol).

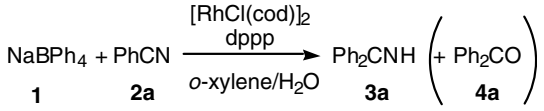
Scheme 3. Phenylation of nitriles.

same conditions. Thus, benzylcyanide (**2d**) and propionitrile (**2e**) reacted smoothly to afford benzyl phenyl ketone (**4d**) and propiophenone (**4e**) in 120% and 73% yields, respectively. In the former case, the yield exceeding 100% means that more than one phenyl group in **1** can be utilized.

Arylboronic acids could be used instead of borate **1** with employing an appropriate base for their activation. Thus, treatment of phenylboronic acid (**5a**) (0.5 mmol) with **2a** (2 mmol) in the presence of [RhCl(cod)]$_2$–dppp (0.005 mmol and 0.01 mmol, respectively) and CsF (2 mmol) as base in *o*-xylene at 120 °C for 2 h afforded **4a** in 54% yield (Scheme 4). In contrast to the case using **1**, addition of water significantly retarded the reaction. Under the same conditions, 4-methyl- (**5b**) and 4-chlorophenylboronic acids (**5c**) reacted with **2a** to give **4b** and **4c**, respectively. The reaction of **5c** was relatively slow, and a small amount of diarylated product (ca. 10%) was detected by GC–MS analysis as in the reaction of **1** with **2c** (Scheme 3).

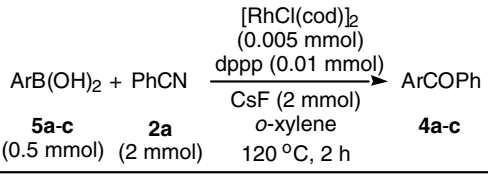
A plausible mechanism for the reaction of arylboron reagents **1** and **5** to nitriles **2** is illustrated in Scheme 5. The reaction proceeds via nucleophilic addition of an arylrhodium(I) intermediate, which is generated by transmetallation of Rh(I)X species with **1** or **5**, to **2**. The resulting imidorhodium species may undergo protonolysis by water,

Table 1
Reaction of sodium tetraphenylborate (**1**) with benzonitrile (**2a**)[a]




| Entry | L (mmol) | Time (h) | Yield of **3a** + **4a** (%)[b] |
|---|---|---|---|
| 1[c] | dppp (0.01) | 44 | 21[d] |
| 2 | dppp (0.01) | 1 | 91 (85) |
| 3[e] | dppp (0.01) | 1 | 76 |
| 4 | – | 1 | 32 |
| 5 | $PPh_3$ (0.02) | 2 | 41 |

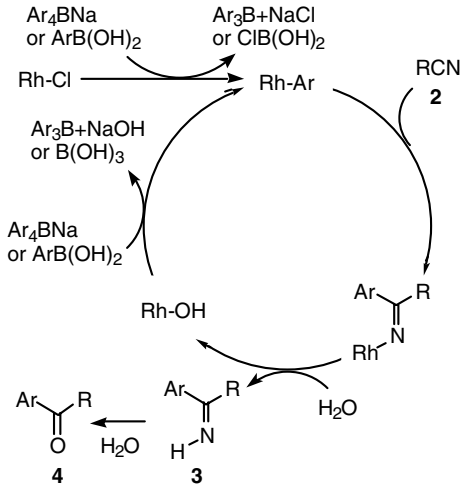
[a] Reaction conditions: **1** (0.5 mmol), **2a** (2 mmol), [RhCl(cod)]$_2$ (0.005 mmol) in *o*-xylene/$H_2O$ (9:1, 5 mL) at 120 °C under $N_2$.
[b] GC yield based on the amount of **1** used. Value in parentheses indicates isolated yield of **4a**.
[c] In *o*-xylene (5 mL).
[d] Di- and triphenylated products were also formed (0.09 and 0.02 mmol, respectively).
[e] With **2a** (1 mmol).




| 5 | Product, Yield (%)[a] |
|---|---|
| **5a**: Ar = Ph | **4a**: Ar = Ph, 54 |
| **5b**: Ar = 4-$MeC_6H_4$ | **4b**: Ar = 4-$MeC_6H_4$, 66 |
| **5c**: Ar = 4-$ClC_6H_4$ | **4c**: Ar = 4-$ClC_6H_4$, 16 |

[a] GC yield.

Scheme 4. Arylation of benzonitrile.




Scheme 5. Plausible mechanism.

which is either added or generated in situ by condensation of **5**, to give an imine **3** and a Rh(I)OH species. The latter undergoes transmetallation to regenerate the arylrhodium intermediate. Under the wet conditions, part of **3** once produced tends to be hydrolyzed to form **4**.

As described above, the rhodium-catalyzed arylation of ketones is known to be sluggish. Actually, it was recently reported that an attempted phenylation of acetophenone (**6a**) by trimethylphenylstannane failed to occur [6]. In the present system, however, **6a** was found to undergo phenylation. Thus, treatment of **6a** (1 mmol) with **1** (0.5 mmol) in the presence of $[RhCl(cod)]_2$ (0.005 mmol) and dppp (0.01 mmol) in *o*-xylene at 120 °C under nitrogen for 25 h gave 1,1-diphenylethanol (**7a**) in 47% yield (Entry 1, Table 2). An increase in the amount of **6a** added slightly decreased the yield of **7a** (Entry 2). In contrast to the case using **2a** as electrophile (Table 1), the omission of dppp did not affect the reaction efficiency (Entry 3). Other rhodium species examined as catalysts, Rh(acac)(cod) (acac = acetylacetonate), $[Rh(OH)(cod)]_2$, $[RhCl(nbd)]_2$ (nbd = norbornadiene), and $[RhCl(C_2H_4)]_2$ were less effective than $[RhCl(cod)]_2$ (Entries 4–7). Addition of water decreased the product yield (Entry 8). Fortunately, however, use of $NH_4Cl$ (1 mmol) as an additive significantly improved it up to 96% (Entry 9). The reaction using **5a** in place of **1** was less efficient (Entry 10).

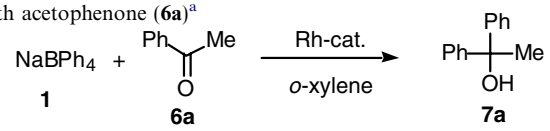
The reaction of ketones seems to proceed via a similar sequence to that for the reaction of nitriles (Scheme 3). Thus, a phenylrhodium intermediate generated by transmetallation is considered to react with a ketone to afford an alkoxyrhodium species, which releases an alcohol or alkoxide salt via protonolysis or transmetallation, depending on the reaction conditions employed. Although the exact role of the weak acid, $NH_4Cl$, is not clear at the present stage, it is possible that it promotes the nucleophilic addition step by protonation of the carbonyl oxygen, and/or acts as the effective source for the alcoholic hydrogen at the protonolysis step.

Other aromatic ketones, 4-methyl- (**6b**) and 4-chloroacetophenone (**6c**), benzophenone (**6d**), and bis(4-chlorophenyl)ketone (**6e**), underwent phenylation upon treatment with **1** under the same conditions with those for the reaction of **6a** to give the corresponding alcohols in fair to good yields (Entries 1–4, Table 3). Aliphatic ketones such as cyclohexanone (**6f**) and 2-nonanone (**6g**) also reacted with **1** efficiently (Entries 5 and 6).

The phenylation system using $[RhCl(cod)]_2$–$NaBPh_4$ (**1**)-$NH_4Cl$ as catalyst, phenyl source, and promoter, respectively, was found to be effective for the reaction of unactivated aryl imines. When *N*-benzylideneaniline (**8a**) (1 mmol) was treated with **1** (0.5 mmol) in the presence of $[RhCl(cod)]_2$ (0.005 mmol) and $NH_4Cl$ (1 mmol) in *o*-xylene (5 mL) under $N_2$ at 160 °C (bath temperature) for 4 h, *N*-(diphenylmethyl)aniline (**9a**) was formed in 78% yield (Entry 1 in Table 4). It should be noted that this is a rare example for the rhodium-catalyzed arylation of poorly electrophilic imines. The phenylation of **8a** using trimethylphenylstannane under rhodium catalysis has been reported, as a sole precedent [3], to our knowledge, to produce **9a** with a moderate yield. Unexpectedly, the reaction efficiency was significantly improved by using Rh(acac)(cod) in place of $[RhCl(cod)]_2$. Thus, **9a** was obtained in 135% yield, which indicates the reaction of more than one phenyl group of **1** (Entry 2). Other Rh–acac complexes, $Rh(acac)(CO)_2$ and $Rh(acac)(C_2H_4)_2$, did not show any activity (Entries 3 and 4). The reaction also did not proceed at all without the addition of $NH_4Cl$ (Entry 5). Under the conditions using Rh(acac)(cod) and $NH_4Cl$, not only *N*-benzylidene-4-chloroaniline (**8b**) but also more electron-rich *N*-benzylidene-4-anisidine (**8c**) underwent phenylation efficiently (Entries 6 and 7). Other benzylideneanilines, *N*-(4-chlorobenzylidene)-4-chloroaniline (**8d**) and *N*-(4-methylbenzylidene)aniline (**8e**) also reacted with **1** under the same conditions to give the corresponding amines **9d** and **9e** in good yields (Entries 8 and 9).

In summary, we have demonstrated that the rhodium-catalyzed intermolecular phenylation of nitriles, ketones, and unactivated imines can be performed efficiently with use of sodium tetraphenylborate. Arylboronic acids can also be employed as aryl sources, albeit less effective than the borate. It has also been found that the addition of

Table 2
Reaction of sodium tetraphenylborate (**1**) with acetophenone (**6a**)[a]

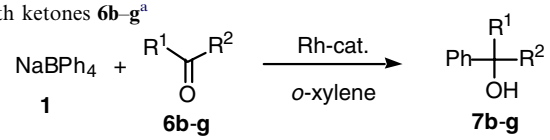



**1** **6a** **7a**

| Entry | Rh-cat. | Time (h) | Yield of **7a** (%)[b] |
|---|---|---|---|
| 1[c] | $[RhCl(cod)]_2$ | 25 | 47 |
| 2[c,d] | $[RhCl(cod)]_2$ | 25 | 40 |
| 3 | $[RhCl(cod)]_2$ | 25 | 44 |
| 4 | Rh(acac)(cod)[e] | 25 | 16 |
| 5 | $[Rh(OH)(cod)]_2$ | 25 | 32 |
| 6 | $[RhCl(nbd)]_2$ | 25 | 5 |
| 7 | $[RhCl(C_2H_4)_2]_2$ | 6[f] | 0 |
| 8[g] | $[RhCl(cod)]_2$ | 5[f] | 23 |
| 9[h] | $[RhCl(cod)]_2$ | 25 | 96 (93) |
| 10[i] | $[RhCl(cod)]_2$ | 2[f] | 37 |

[a] Reaction conditions: **1** (0.5 mmol), **6a** (1 mmol), Rh-cat. (0.005 mmol) in *o*-xylene (5 mL) at 120 °C under $N_2$.
[b] GC yield based on the amount of **1** used. Value in parentheses indicates isolated yield.
[c] With the addition of dppp (0.01 mmol).
[d] With **6a** (1.5 mmol).
[e] With Rh(acac)(cod) (0.01 mmol).
[f] The yield of **7a** did not increase even if the reaction time was elongated.
[g] In *o*-xylene/$H_2O$ (9:1, 5 mL).
[h] With the addition of $NH_4CI$ (1 mmol).
[i] **5a** (2 mmol) and CsF (2 mmol) were used in place of **1**.

Table 3
Reaction of sodium tetraphenylborate (**1**) with ketones **6b–g**[a]




**1** **6b-g** **7b-g**

| Entry | **6** | Product, yield (%)[b] |
|---|---|---|
| 1 | **6b**: $R^1$ = Me, $R^2$ = 4-$CH_3C_6H_4$ | **7b**, 74 (50) |
| 2 | **6c**: $R^1$ = Me, $R^2$ = 4-$ClC_6H_4$ | **7c**, 169 (129) |
| 3 | **6d**: $R^1 = R^2$ = Ph | **7d**, 61 (36) |
| 4 | **6e**: $R^1 = R^2$ = 4-$ClC_6H_4$ | **7e**, 103 (78) |
| 5 | **6f**: $R^1R^2$ = –$(CH_2)_5$– | **7f**, 139 (97) |
| 6 | **6g**: $R^1$ = Me, $R^2$ = *n*-$C_7H_{15}$ | **7g**, 55 (47) |

[a] Reaction conditions: **1** (0.5 mmol), **6** (1 mmol), $[RhCl(cod)]_2$ (0.005 mmol), $NH_4Cl$ (1 mmol) in *o*-xylene (5 mL) at 120 °C for 25 h under $N_2$.
[b] GC yield based on the amount of **1** used. Value in parentheses indicates isolated yield.

$H_2O$ and $NH_4Cl$ is crucial to smoothly conduct the reaction of nitriles and of the latter two substrates, respectively.

## 3. Experimental

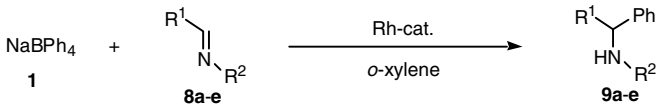
### 3.1. General

Reactions were carried out in a 20 mL two-neck flask under $N_2$. $[Rh(OH)(cod)]_2$ was prepared according to the published method [17]. $^1H$ and $^{13}C$ NMR spectra were recorded at 400 and 100 MHz, respectively, for $CDCl_3$ solutions. MS data were obtained by EI. GC analysis was carried out using a silicon OV-17 column (i.d. 2.6 mm × 1.5 m) or a CBP-1 capillary column (i.d. 0.25 mm × 25 m). GC–MS analysis was carried out using a CBP-1 capillary column (i.d. 0.25 mm × 25 m). Imines **8b–e** were prepared according to the published method [18]. Other reagents were commercially available. Products **4a** [19], **4b** [20], **4c** [20], **4d** [21], **4e** [22], **7a** [23], **7b** [24], **7c** [25], **7d** [26], **7e** [27], **7f** [28], **7g** [29], **9a** [30], **9b** [31], **9c** [31], **9d** [32], and **9e** [9] are known.

### 3.2. Typical procedure for the rhodium-catalyzed reaction of sodium tetraphenylborate (*1*) with benzonitrile (*2a*) (Entry 2 in Table 1)

A mixture of **1** (0.5 mmol, 171 mg), **2a** (2 mmol, 206 mg), $[RhCl(cod)]_2$ (0.005 mmol, 2.5 mg), dppp (0.01 mmol, 4.1 mg), and dibenzyl (ca. 50 mg) as internal standard was stirred in *o*-xylene/$H_2O$ (9/1, 5 mL) at

Table 4
Reaction of sodium tetraphenylborate (**1**) with imines **8a–e**[a]




| Entry | **8** | Rh-cat. | Time (h) | Product, yield (%)[b] |
|---|---|---|---|---|
| 1 | **8a**: $R^1 = R^2 = Ph$ | $[RhCl(cod)]_2$[c] | 4 | **9a**, 78 |
| 2 | **8a**: $R^1 = R^2 = Ph$ | Rh(acac)(cod) | 5.5 | **9a**, 135 (85) |
| 3 | **8a**: $R^1 = R^2 = Ph$ | $Rh(acac)(CO)_2$ | 4 | – |
| 4 | **8a**: $R^1 = R^2 = Ph$ | $Rh(acac)(C_2H_4)_2$ | 4 | – |
| 5[d] | **8a**: $R^1 = R^2 = Ph$ | Rh(acac)(cod) | 6 | – |
| 6 | **8b**: $R^1 = Ph$, $R^2 = 4\text{-}ClC_6H_4$ | Rh(acac)(cod) | 3.5 | **9b**, 139 (108) |
| 7 | **8c**: $R^1 = Ph$, $R^2 = 4\text{-}CH_3OC_6H_4$ | Rh(acac)(cod) | 4 | **9c**, 103 (78) |
| 8 | **8d**: $R^1 = R^2 = 4\text{-}ClC_6H_4$ | Rh(acac)(cod) | 5 | **9d**, 121 (93) |
| 9 | **8e**: $R^1 = 4\text{-}CH_3C_6H_4$, $R^2 = Ph$ | Rh(acac)(cod) | 6 | **9e**, 121 (100) |
| 10[e] | **8a**: $R^1 = R^2 = Ph$ | Rh(acac)(cod) | 4 | **9a**, 26 |

[a] Reaction conditions: **1** (0.5 mmol), **8** (1 mmol), Rh-cat. (0.01 mmol), $NH_4Cl$ (1 mmol) in *o*-xylene (5 mL) at 160 °C under $N_2$.
[b] GC yield based on the amount of **1** used. Value in parentheses indicates isolated yield.
[c] With $[RhCl(cod)]_2$ (0.005 mmol).
[d] Without $NH_4Cl$.
[e] **5a** (0.5 mmol) and CsF (0.5 mmol) were used in place of **1**.

120 °C under $N_2$ for 1 h. After cooling, the reaction mixture was extracted with $Et_2O$ and dried over sodium sulfate. Product **4a** (77 mg, 85%) was isolated by column chromatography on silica gel using hexane–ethyl acetate as eluent.

### 3.3. Typical procedure for the rhodium-catalyzed reaction of sodium tetraphenylborate (*1*) with acetophenone (*6a*) (Entry 9 in Table 2)

In a flask was placed $NH_4Cl$ (1 mmol, 54 mg), which was then dried at 150 °C in vacuo for 2 h. Then, **1** (0.5 mmol, 171 mg), **6a** (1 mmol, 120 mg), $[RhCl(cod)]_2$ (0.005 mmol, 2.5 mg), 1-methylnaphthalene (ca. 50 mg) as internal standard, and *o*-xylene (5 mL) were added and the resulting mixture was stirred at 120 °C under $N_2$ for 25 h. After cooling, the reaction mixture was extracted with $Et_2O$ and dried over sodium sulfate. Product **7a** (92 mg, 93%) was isolated by thin-layer chromatography on silica gel using hexane–ethyl acetate as eluent.

### 3.4. Typical procedure for the rhodium-catalyzed reaction of sodium tetraphenylborate (*1*) with N-benzylideneaniline (*8a*) (Entry 2 in Table 4)

A mixture of **1** (0.5 mmol, 171 mg), **8a** (1 mmol, 181 mg), Rh(acac)(cod) (0.01 mmol, 3.1 mg), $NH_4Cl$ (1 mmol, 54 mg), and dibenzyl (ca. 50 mg) as internal standard was stirred in *o*-xylene (5 mL) at 160 °C under $N_2$ for 5.5 h. After cooling, the reaction mixture was extracted with $Et_2O$ and dried over sodium sulfate. Product **9a** (111 mg, 85%) was isolated by column chromatography on silica gel using hexane–ethyl acetate as eluent.

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