# Synthesis of Coumarins Catalyzed by Heterobimetallic Co/Rh Nanoparticles

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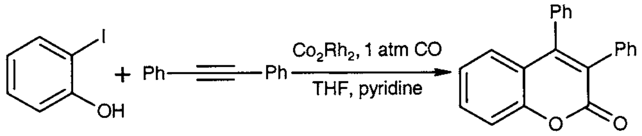
**Abstract:** Described here is the use of cobalt/rhodium ($Co_2Rh_2$) heterobimetallic nanoparticles in the catalytic synthesis of coumarins via cyclocarbonylation of alkynes with 2-iodophenol under 1 atm of carbon monoxide and reaction of phenol with propiolic esters.

**Key words:** coumarins, nanoparticles, heterobimetallic, cyclocarbonylation, catalysis

Coumarin and its derivatives occur widely in nature, particularly in plants, with the majority of them showing a wide range of biological activities.[1] Recently, their synthesis has attracted a lot of attention, not only because of their pharmacological activity, but also because of their applications as laser dyes, fluorescent labels, emission layers in organic light-emitting diodes, and as optical brighteners.[2] As a result, many useful methods for the synthesis of coumarins have been extensively developed,[3] including the Perkin reaction,[4] the Knoevenagel reaction,[5] and the Pechmann reaction.[6] Recently, several studies[7] on transition metal-catalyzed reactions have reported methods for the synthesis of coumarins. In particular, palladium-catalyzed reactions of phenols[7d–f] have been widely used. They involve inter- and intramolecular reactions of 2-iodo-phenol or its *O*-acyl derivatives in the presence of CO, the intramolecular reaction of (2-hydroxyphenyl)alkenes or -alkynes, reactions of phenols and ethyl propioloates, or reactions of 2-iodophenol and alkynes in the presence of CO.

Recently, the chemistry of transition-metal nanoparticles has been developing rapidly[8] and it has broadened to include many catalytic reactions, such as oxidation,[9] hydrogenation,[10] coupling reactions,[11] the Pauson–Khand reaction,[12] and some photocatalytic reactions.[13] Very recently, we found that cobalt–rhodium nanoparticles ($Co_2Rh_2$) derived from $Co_2Rh_2(CO)_{12}$ could be successfully utilized as catalysts in Pauson–Khand-type reactions and silylcarbocyclization.[14] While we were investigating the use of $Co_2Rh_2$, we discovered a $Co_2Rh_2$-catalyzed method of synthesis for coumarins. Here we report the use of cobalt–rhodium heterobimetallic nanoparticles in the catalytic synthesis of coumarins via the cyclocarbonylation of alkynes with 2-iodophenol under 1 atm of carbon monoxide and the reaction of phenol with propiolic esters. The cobalt–rhodium nanoparticles are easily recovered and can be reused at least five times without loss of catalytic activity.

As mentioned above, the palladium-catalyzed cyclocarbonylation of alkynes with 2-iodophenol has been well established. Thus, the use of $Co_2Rh_2$ nanoparticles was considered to be somewhat unusual at first. However, our preliminary experiment showed that $Co_2Rh_2$ nanoparticles could be used to catalyze this reaction (Scheme 1). Therefore, we carried out an investigation to find an optimized set of reaction conditions. The reaction parameters such as bases and CO sources were screened (Table 1). $Mo(CO)_6$ turned out to be a good CO source in the presence of pyridine and DBU (entry 2).[15] However, α,β-unsaturated aldehydes such as acrolein were not active as a CO source.[14b] Pyridine was better than triethylamine (entries 1 and 3). The final optimized reaction conditions for the reaction of 2-iodophenol, diphenylacetylene, and carbon monoxide have thus been determined to be 1.0 mmol of 2-iodophenol, 2.5 equivalents of diphenylacetylene, 3 mol% $Co_2Rh_2$, 1.2 equivalents of pyridine in 5 mL of THF, the reaction carried our at 120 °C for 18 hours under 1 atm of CO. The best yield obtained was 87%. The reusability of $Co_2Rh_2$ was also tested (entries 4–7). As shown in Table 1, the catalyst could be reused at least five times without loss of catalytic activity.



**Scheme 1**

**Table 1** Optimization of the Reaction Conditions

| Entry | Catalyst | CO source | Base | Yield (%)[a] |
|---|---|---|---|---|
| 1 | Pd nanoparticles | 1 atm CO | Pyridine | 79 |
| 2 | $Co_2Rh_2$ | 1 atm CO | Pyridine | 87 |
| 3 | $Co_2Rh_2$ | $Mo(CO)_6$ | Pyridine | 73 |
| 4 | $Co_2Rh_2$ | 1 atm CO | $Et_3N$ | 43 |
| 5 | $Co_2Rh_2$[b] | 1 atm CO | Pyridine | 85 |
| 6 | $Co_2Rh_2$[c] | 1 atm CO | Pyridine | 83 |
| 7 | $Co_2Rh_2$[d] | 1 atm CO | Pyridine | 85 |
| 8 | $Co_2Rh_2$[e] | 1 atm CO | Pyridine | 86 |

[a] Isolated yields.
[b] Recovered from the reaction in entry 2.
[c] Recovered from the reaction in entry 5.
[d] Recovered from the reaction in entry 6.
[e] Recovered from the reaction in entry 7.

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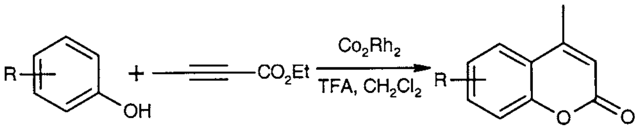
The scope and limitations of this annulation reaction have been studied by allowing several internal alkynes to react with 2-halophenol under our optimized reaction conditions. The results of this study are summarized in Table 2. The yields were higher than those obtained by the Pd(OAc)$_2$-catalyzed carbonylative annulation of internal alkynes.[3] When the halide was a bromide (entry 2), the yield was 65%. In the case of 2-chlorophenol (entry 3), the yield was only 13%. The order of reactivity, ArI > ArBr > ArCl, is consistent with the strength of the Ar-X bond. Treatment of 2-iodophenol with a symmetric alkyne, 3-hexyne and 4-octyne (entries 4 and 5), gave the product in 69% and 72% yields, respectively. When an unsymmetric alkyne, 1-phenyl-1-propyne (entry 6), was used, two isomeric products were obtained in 67% and 13% yields, respectively. The regioselectivity (5.1:1) was higher than that (3.8:1) obtained by the Pd(OAc)$_2$-catalyzed carbonylative annulation of internal alkynes.[3]

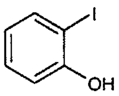
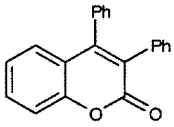
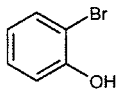
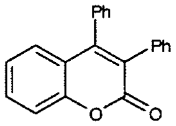
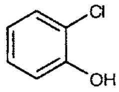
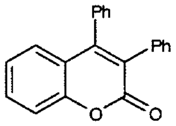
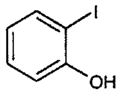
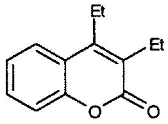
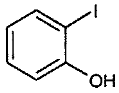
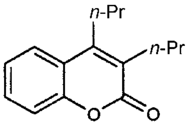
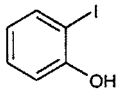
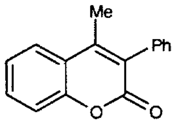
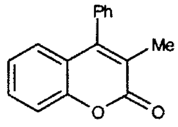
Several years ago, Trost reported[3] the palladium-catalyzed synthesis of coumarins from phenols and alkynoates under mild reaction conditions. Very recently, Kitamura et al. also reported[3] a palladium-catalyzed synthesis of coumarins from phenols and propiolic esters. Their results encouraged us to study the reaction between phenol and alkynoates. Thus, we investigated the Co$_2$Rh$_2$-catalyzed synthesis of coumarins from the reaction of various phenols with alkynoates under mild reaction conditions (Scheme 2 and Table 3).



**Scheme 2**

The reaction conditions were adopted from the previous study.[3] Table 3 indicates that coumarins were obtained in high yields in all cases. This observation is somewhat different from the results obtained in Pd-catalyzed reactions.[3] For the Pd-catalyzed reactions, electron-rich phenols, such as 3-methoxyphenol, 4-methoxyphenol, 3,5-

**Table 2** Synthesis of Coumarins by the Co$_2$Rh$_2$-Catalyzed Carbonylative Annulation of Internal Alkynes[a]

| Entry | Phenol | Alkyne | Product | Yield (%) |
|---|---|---|---|---|
| 1 |  | Ph—≡—Ph |  | 87 |
| 2 |  | Ph—≡—Ph |  | 65 |
| 3 |  | Ph—≡—Ph |  | 13 |
| 4 |  | Et—≡—Et |  | 69 |
| 5 |  | Pr-n—≡—n-Pr |  | 72 |
| 6 |  | Ph—≡—Me |  | 67 |
| | | |  | 13 |

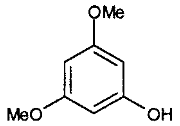
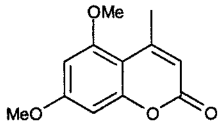
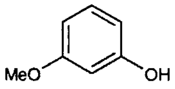
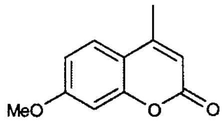
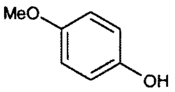
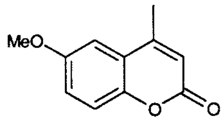
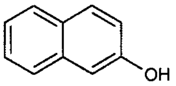
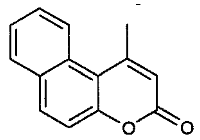
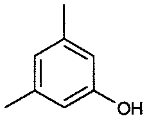
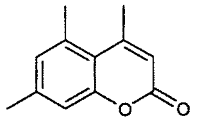
[a] Isolated yields. *Reaction conditions*: *o*-Iodophenol (0.11 g, 0.97 mmol), alkyne (0.445 g, 2.50 mmol), pyridine (0.1 mL, 1.20 mmol), Co$_2$Rh$_2$ (0.01 g, 0.029 mmol), THF (5 mL), 1 atm CO, 120 °C, and 18 h.

dimethoxyphenol, and 2-naphthol gave much higher yields than those of moderately activated phenols, such as 3,4- and 3,5-dimethylphenol. In addition to the carbonylative cycloaddition reaction, hydroarylation of coumarins was also observed for the Pd-catalyzed reaction under Kitamura's reaction conditions.[3] In the case of *m*-cresol, no reaction was observed under Trost's reaction conditions.[3] However, no hydroarylation was observed in the case of our reaction conditions either. But, under our reaction conditions, *m*-cresols produced a high yield.

Trost et al.[3] proposed that it is a Pd(0) species that acts as the actual catalyst. The reaction, they believed, was initiated by a hydropalladation, namely the formation of HPdX. However, Kitamura et al.[3] reported that a Pd(II) species catalyzed the hydroarylation of 2-alkynoates in TFA. They proposed the *ortho*-palladation of a phenol by a Pd(II) species, followed by hydroarylation of an alkynoate. This difference might originate in the differing reaction conditions. The precise mechanism of $Co_2Rh_2$ catalysis has not yet been revealed, but it is likely to be distinct from that of the palladium-catalyzed process.

In conclusion, we have demonstrated a simple method of synthesis for coumarins from a reaction of alkynes with 2-iodophenol under 1 atm of CO, and from a reaction of phenols with propiolic esters by using $Co_2Rh_2$ nanoparticles as efficient catalysts. The reaction does not need any additives or special conditions. The reusability of the catalyst and the simplicity of the experimental conditions are especially attractive and should encourage the use of this catalyst system among synthetic chemists and in industrial applications.

**Table 3** $Co_2Rh_2$-Catalyzed Reaction of But-2-ynoic Acid Ethyl Ester with Phenols[a]

| Entry | Phenol | Product | Yield (%) |
|---|---|---|---|
| 1 |  |  | 87 |
| 2 |  |  | 89 |
| 3 |  |  | 85 |
| 4 |  |  | 79 |
| 5 |  |  | 83 |

[a] Isolated yields. *Reaction conditions*: phenol (0.158 mL, 1.430 mmol), $Co_2Rh_2$ (0.01 g, 0.029 mmol), TFA (0.5 mL), $CH_2Cl_2$ (5 mL), tetrolic acid ethyl ester (0.21 mL, 1.820 mmol), r.t., 12 h.

### General Remarks:

All the reactions were conducted under nitrogen using standard Schlenk-type flasks. Workup procedures were done in air. All the solvents were dried and distilled according to standard methods before use. The reagents were purchased from Aldrich Chemical Co. and Strem Chemical Co. and were used as received. $^1H$ NMR spectra were obtained with a Bruker 300 MHz or 500 MHz spectrometer. The elemental analyses were done at the National Center for Inter-University Research Facilities, Seoul National University. High resolution MS was done at Korea Basic Science Institute (Daegu).

### Preparation of $Co_2Rh_2$ Nanoparticles:

To a two-neck flask were added *o*-dichlorobenzene (24 mL), oleic acid (0.2 mL), and trioctylphosphine oxide (0.4 g). While the solution was heated at 180 °C, a solution of $Co_2Rh_2(CO)_{12}$ (1.0 g) in 6 mL of *o*-dichlorobenzene was injected into the flask. The resulting solution was heated at 180 ºC for 2 h. After the solution had cooled, the solution was concentrated to ca 10 mL by distillation. To the concentrated solution were added 200 mL of THF.

### Immobilization of $Co_2Rh_2$ Nanoparticles on Charcoal:

To the concentrated solution of $Co_2Rh_2$ (ca 10 mL), obtained by the above reaction, was added 25 mL of THF. After the solution had been well stirred for 10 min, flame-dried charcoal (2.0 g) was added to the solution. After the resulting solution was refluxed for 12 h, the precipitates were filtered and washed with $Et_2O$ (20 mL), $CH_2Cl_2$ (20 mL), acetone (20 mL), and MeOH. Vacuum drying gave a black solid.

### A Typical Procedure for the Synthesis of Coumarins by the $Co_2Rh_2$-Catalyzed Carbonylative Annulation of Internal Alkynes (Table 2):

*o*-Iodophenol (0.110 g, 0.97 mmol), diphenylacetylene (0.445 g, 2.50 mmol), pyridine (0.1 mL, 1.20 mmol), $Co_2Rh_2$ (0.010 g, 0.029 mmol), and THF (5 mL) were put into a stainless-steel bomb and charged with 1 atm CO. The bomb was heated at 120 °C for 18 h. After the solution had cooled, the solution was filtered and concentrated under reduced pressure. The residue was purified by column chromatography on a silica gel ($Et_2O$–hexane = 20:80) to give a 87% yield of coumarins.

### A Typical Procedure for the Synthesis of Coumarins by the $Co_2Rh_2$-Catalyzed Reaction of But-2-ynoic Acid Ethyl Ester with Various Phenols (Table 3):

To a cold mixture of $Co_2Rh_2$ (0.010 g, 0.029 mmol), 3-methoxyphenol (0.158 mL, 1.430 mmol), TFA (0.50 mL), and $CH_2Cl_2$ (5 mL) at 0 °C was added tetrolic acid ethyl ester (0.21 mL, 1.820 mmol). The mixture was stirred at r.t. for 12 h. The reaction mixture was filtered and the filtrate was then neutralized with an aq $NaHCO_3$ solution, extracted with $CH_2Cl_2$, dried over anhyd $MgSO_4$, and concentrated. Flash column chromatography on silica gel gave an 89% yield of the product.

### Recycling Experiment:

In order to recycle the catalyst, it was filtered from the reaction mixture and dried in vacuum. It could then be reused for further catalytic reactions.

## Acknowledgment

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