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Ruthenium-catalyzed [2 + 2+ 2] cycloaddition of three different alkynes

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Abstract

A chemoselective [2 + 2 + 2] cycloaddition of an internal alkyne, a terminal alkyne and dimethyl acetylenedicarboxylate was efficiently catalyzed by Cp*RuCl(cod) to give trisubstituted *o*-phthalates in good yield. It is critical to control the molar ratio of the three substrates to achieve high chemoselectivity, and regioselectivity of the products is sensitively influenced by the bulkiness of substituents on the internal alkyne.
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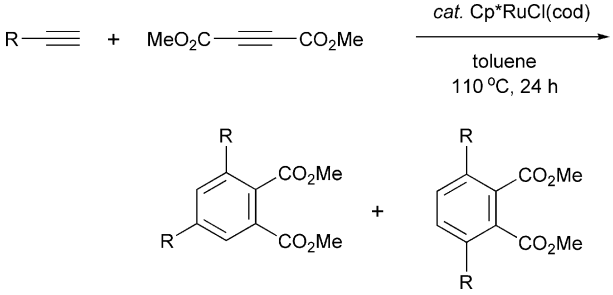
Keywords: Ruthenium; [2 + 2 + 2] Cycloaddition; Alkynes; *o*-Phthalates; Molar ratio

1. Introduction

Transition metal complex-catalyzed cyclotrimerization of alkynes is a powerful tool for aromatic ring construction [1–7]. Although intensive studies have been done to date, there are quite few reports for the selective [2 + 2 + 2] cycloaddition of *three different alkynes*, due to difficult control of the chemo- and regio selectivity [4]. Early-transition metals such as zirconium [8–10] and titanium [11,12] are known to mediate the selective cycloaddition, in which the chemoselectivity is well regulated by stepwise addition of the different alkynes. As for catalytic reactions using late-transition metals, a palladium-catalyzed unique arene formation from two different alkynes and a diyne has been reported by Yamamoto and co-workers, which mechanism is actually not simple [2 + 2 + 2] cycloaddition but dimerization of the alkynes and successive [4 + 2] benzannulation with the diyne [13]. Nickel(0)–zinc(II) phenoxide system is effective for the catalytic [2 + 2 + 2] cycloaddition proceeding under mild conditions, while the chemo- and regio-selectivity are still problematic [14]. Recently, a ruthenium-catalyzed reaction has also been reported, where in situ-generation of diynes from alkynylboronates and propargyl alcohol is elegantly applied [15].

Previously we reported the ruthenium-catalyzed highly chemoselective intermolecular [2 + 2 + 2] cycloaddition of 2 equiv of terminal alkynes with DMAD (dimethyl acetylenedicarboxylate) to afford *o*-phthalates in high yield (Eq. (1)) [16]. In the course of our further investigations, we found the same catalyst system could be applied to the three-component reaction and successfully achieved the chemoselective [2 + 2 + 2] cycloaddition of three different alkynes, by controlling the molar ratio of the substrates. Here we describe the reaction details and discuss the mechanism.



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2. Experimental

2.1. General

All reactions were performed under an argon atmosphere. GLC analyses were performed on a Shimadzu GC-14B gas chromatograph with a glass column (3.2 mm i.d. × 3.0 m) packed with Silicone OV-17 (2% on Chromosorb W (AW-DMCS), 60–80 mesh). NMR spectra were recorded on JEOL AL-300 (FT, 300 MHz (^{1}H), 75 MHz (^{13}C)) spectrometer. Chemical shift values (δ) were expressed relative to $SiMe_4$ as an internal standard. ^{13}C NMR Inadequate measurements were performed on a Bruker Dual Cryoprobe at Bruker BioSpin K.K. IR spectra were recorded on a Nicolet Impact 410 FT-IR spectrometer. Elemental analyses were performed at the Microanalytical Center of Kyoto University.

2.2. Materials

$Cp^*RuCl(cod)$ was synthesized as described in the literature [17]. Substrates were commercially available and were used without further purification. All solvents were distilled under argon over appropriate drying reagents (sodium, calcium hydride, sodium-benzophenone or calcium chloride).

2.3. The synthetic procedure of the products

A mixture of DMAD (4.0 mmol), 1-octyne (24 mmol), 3-hexyne (160 mmol) and $Cp^*RuCl(cod)$ (0.20 mmol) in toluene (50 mL) was stirred at 110 °C for 24 h. After removal of the solvent, silica gel column chromatography (hexane/ethyl acetate = 20/1) and subsequent Kugelrohr distillation afforded **1a** in 24% isolated yield (0.32 g, 0.97 mmol). Compounds **1b-g** and **3d-e** were also prepared in a similar manner described above.

2.4. Dimethyl 3,4-diethyl-5-n-hexyl-o-phthalate (*1a*)

Pale yellow liquid; IR (cm^{-1}, neat) 2955, 2925, 1734, 1270. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.64 (s, 1H), 3.90 (s, 3H), 3.83 (s, 3H), 2.68 (m, 2H), 2.61 (m, 2H), 1.56 (m, 2H), 1.29 (m, 8H), 1.14 (m, 6H), 0.86 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.3, 166.1, 145.5, 142.1, 139.1, 133.3, 128.6, 124.7, 52.3, 52.2, 33.0, 32.6, 31.7, 29.4, 23.8, 22.7, 21.9, 15.9, 15.2, 14.2. Anal. Calcd. for $C_{20}H_{30}O_4$: C 71.82, H 9.04. Found: C 71.87, H 8.89.

2.5. Dimethyl 3,4-diethyl-5-n-octyl-o-phthalate (*1b*)

Pale yellow liquid; IR (cm^{-1}, neat) 2930, 2849, 1734, 1463. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.67 (s, 1H), 3.93 (s, 3H), 3.86 (s, 3H), 2.72 (m, 2H), 2.64 (m, 2H), 1.56 (m, 2H), 1.28 (m, 10H), 1.17 (m, 6H), 0.89 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.6, 166.4, 145.8, 142.4, 139.4, 133.7, 128.9, 125.0, 52.3, 52.2, 33.0, 32.0, 31.5, 29.9, 29.5, 29.3, 23.8, 22.7, 21.9, 15.9, 15.2, 14.2. Anal. Calcd. for $C_{22}H_{34}O_4$: C 72.89, H 9.45. Found: C 72.94, H 9.45.

2.6. Dimethyl 3,4-diethyl-5-n-decyl-o-phthalate (*1c*)

Pale yellow liquid; IR (cm^{-1}, neat) 2918, 2852, 1739, 1466. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.59 (s, 1H), 3.88 (s, 3H), 3.80 (s, 3H), 2.63 (m, 2H), 2.52 (m, 2H), 1.50 (m, 2H), 1.19 (m, 16H), 1.10 (m, 6H), 0.82 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.4, 166.2, 145.6, 142.2, 139.1, 133.3, 128.7, 124.7, 52.4, 52.3, 33.0, 32.6, 29.9, 29.7, 29.7, 29.6, 29.6, 29.4, 23.9, 22.8, 22.0, 16.0, 15.2, 14.3.

2.7. Dimethyl 3,4-dipropyl-5-n-octyl-o-phthalate (*1d*)

Pale yellow liquid; IR (cm^{-1}, neat) 2955, 2926, 1731. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.58 (s, 1H), 3.85 (s, 3H), 3.78 (s, 3H), 2.54 (m, 8H), 1.50 (m, 2H), 1.20 (m, 12H), 0.95 (m, 6H), 0.82 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.5, 166.2, 144.5, 142.3, 138.1, 133.3, 128.6, 124.7, 52.4, 52.3, 33.2, 32.0, 31.5, 29.9, 29.5, 29.4, 25.1, 24.4, 22.8, 22.8, 22.8, 15.0, 15.0, 14.2. Anal. Calcd. for $C_{24}H_{38}O_4$: C 73.81, H 9.81. Found: C 74.10, H 9.62.

2.8. Dimethyl 3,4-dipentyl-5-n-octyl-o-phthalate (*1e*)

Pale yellow liquid; IR (cm^{-1}, neat) 2954, 2926, 1732. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.58 (s, 1H), 3.85 (s, 3H), 3.78 (s, 3H), 2.54 (m, 8H), 1.50 (m, 2H), 1.20 (m, 12H), 0.95 (m, 6H), 0.82 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.5, 166.2, 144.5, 142.3, 138.1, 133.3, 128.6, 124.7, 52.4, 52.3, 33.2, 32.0, 31.5, 29.9, 29.5, 29.4, 25.1, 24.4, 22.8, 22.8, 22.8, 15.0, 15.0, 14.2. Anal. Calcd. for $C_{28}H_{46}O_4$: C 75.29, H 10.38. Found: C 75.34, H 10.41.

2.9. Dimethyl 3,4-diethyl-5-phenyl-o-phthalate (*1f*)

Pale yellow liquid; IR (cm^{-1}, neat) 2970, 1746, 1731. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.70 (s, 1H), 7.44–7.35 (m, 3H), 7.28–7.25 (m, 2H), 3.98 (s, 3H), 3.84 (s, 3H), 2.71 (q, J = 7.8 Hz, 2H), 2.65 (q, J = 7.8 Hz, 2H), 1.24 (t, J = 7.8 Hz, 3H), 0.95 (t, J = 7.8 Hz, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.1, 165.9, 145.7, 143.5, 141.0, 139.5, 134.8, 129.6, 128.8 (2C), 128.0 (2C), 127.1, 124.4, 52.5, 52.3, 23.9, 22.8, 16.0, 15.3. MS (EI) m/z 326 (M^+). Anal. Calcd. for $C_{20}H_{22}O_4$: C, 73.60; H, 6.79. Found: C, 73.30; H, 6.86.

2.10. Dimethyl 3,4-diethyl-5-trimethylsilyl-o-phthalate (*1g*)

Pale yellow liquid; IR (cm^{-1}, neat) 2953, 1738, 1727, 1453, 1434, 1291. ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.96 (s, 1H), 3.91 (s, 3H), 3.84 (s, 3H), 2.81 (q, J = 7.8 Hz, 2H), 2.64 (q, J = 7.7 Hz, 2H), 1.16 (t, J = 7.8 Hz, 3H), 1.14 (t, J = 7.7 Hz, 3H), 0.32 (s, 9H). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 170.3, 166.5, 153.7, 140.7, 138.8, 136.6, 134.3, 124.3, 52.4, 52.3, 26.5, 23.2, 16.6, 16.0, 0.79 (3C). MS (EI) m/z 322 (M^+). Anal. Calcd. for $C_{17}H_{26}O_4Si$: C, 63.32; H, 8.13. Found: C, 63.19; H, 7.94.

2.11. Dimethyl 3,4-dipropyl-6-n-octyl-o-phthalate (**3d**)

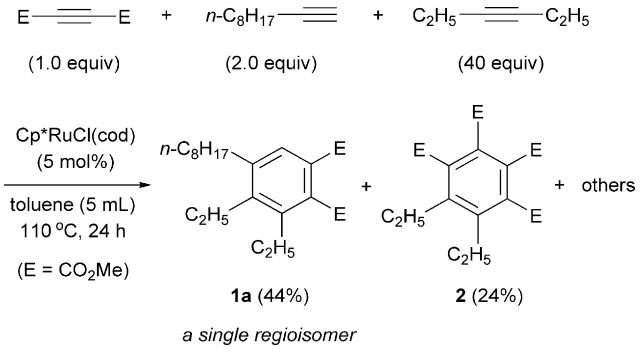
Pale yellow liquid; IR (cm^{-1}, neat) 2955, 2926, 1731. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.42 (s, 1H), 3.81 (s, 3H), 3.78 (s, 3H), 2.44 (m, 8H), 1.50 (m, 2H), 1.20 (m, 12H), 0.95 (m, 6H), 0.82 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.5, 168.1, 144.1, 144.0, 140.3, 133.6, 129.5, 129.1, 52.5, 52.3, 32.6, 31.4, 31.0, 29.8, 29.5, 29.3, 25.1, 24.4, 22.8, 22.8, 22.8, 15.0, 15.0, 14.2. Anal. Calcd. for $C_{24}H_{38}O_4$: C 73.81, H 9.81. Found: C 74.10, H 9.62.

2.12. Dimethyl 3,4-dipentyl-6-n-octyl-o-phthalate (**3e**)

Pale yellow liquid; IR (cm^{-1}, neat) 2954, 2926, 1732. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.58 (s, 1H), 3.85 (s, 3H), 3.78 (s, 3H), 2.54 (m, 8H), 1.50 (m, 2H), 1.20 (m, 12H), 0.95 (m, 6H), 0.82 (m, 3H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 170.5, 166.2, 144.5, 142.3, 138.1, 133.3, 128.6, 124.7, 52.4, 52.3, 33.2, 32.0, 31.5, 29.9, 29.5, 29.4, 25.1, 24.4, 22.8, 22.8, 22.8, 15.0, 15.0, 14.2. Anal. Calcd. for $C_{28}H_{46}O_4$: C 75.29, H 10.38. Found: C 75.34, H 10.41.

3. Results and discussion

A reaction of DMAD (1.0 equiv), 1-decyne (2.0 equiv) and 3-hexyne (40 equiv) was performed in toluene under reflux for 24 h in the presence of a catalytic amount (5 mol%) of $Cp^*RuCl(cod)$ (Eq. (2)). The reaction proceeded to afford a single regioisomer of three-component coupling products, dimethyl 3,4-diethyl-5-*n*-octyl-*o*-phthalate **1a**, in 44% yield based on DMAD, along with the formation of byproducts such as tetramethyl 5,6-diethyl-1,2,3,4-benzenetetracarboxylate **2** and other aromatic compounds. The structure of **1a** was confirmed by ^{13}C inadequate measurement, in addition to general spectroscopic analysis.



(2)

Several solvents were surveyed for the [2 + 2 + 2] cycloaddition of DMAD, 1-decyne and 3-hexyne at 110 °C for 24 h (Table 1). Hydrocarbon solvents afforded **1a** in moderate yields (runs 1–3); especially, cyclohexane was almost comparable to toluene. On the other hand, polar solvents such as DMF and pyridine were not effective (runs 4 and 5). Other solvents such as 1,2-dichloroethane, *i*-PrOH and *n*-BuOH gave **1a** in 26, 4 and 34% yield, respectively. The reaction was also conducted without a solvent; however, the yield of **1a** was low (19%) and **2** was formed in 20% yield.

Table 1
Solvent effects on catalytic [2 + 2 + 2] cycloaddition of three different alkynes

Run	Solvent	Yield (%)[a]	
		1a	**2**
1	Toluene	44	24
2	Mesitylene	31	26
3	Cyclohexane	44	28
4	DMF	19	13
5	Pyridine	trace	trace
6	1,2-Dichloroethane	26	22
7	*i*-PrOH	4	22
8	*n*-BuOH	34	20

DMAD (0.20 mmol), 1-decyne (0.40 mmol), 3-hexyne (8.0 mmol), $Cp^*RuCl(cod)$ (0.010 mmol), and solvent (5.0 mL) at 110 °C for 24 h.
[a] Determined by GLC.

Improvement of the product yield was attained by controlling the molar ratio of DMAD/1-decyne/3-hexyne. First, the ratio of 3-hexyne was varied from 10 to 60 equiv against to DMAD/1-decyne (fixed as 1/2) (Table 2, runs 1–4). At the ratio of 1/2/40, **1a** was formed in 44% yield along with **2** in 24% yield. Under the fixed ratio of DMAD/3-hexyne = 1/40, the amount of 1-decyne was gradually increased (runs 3, 5–8). The best result was obtained (57%) when the molar ratio was 1/6/40 (run 6). Although the formation of byproduct **2** was supressed by further increment of 1-decyne, the yield of **1a** was also decreased (runs 7 and 8).

Under the optimized reaction conditions, $Cp^*RuCl(cod)$-catalyzed [2 + 2 + 2] cycloaddition of several terminal alkynes, internal alkynes and DMAD was examined (Eq. (3), Table 3). Linear alkyl-substituted terminal and internal alkynes were applicable to afford the corresponding 3,4,5- and 3,4,6-trialkylated *o*-phthalates **1** and **3** in moderate yields. Use of 3-hexyne (R^2 = Et) gave **1** regioselectivily (**1**:**3** = 100:0 in runs 1–3), and the ratio was independent of the chain length of R^1. However, internal alkynes

Table 2
Effect of molar ratio of DMAD/1-decyne/3-hexyne

Run	DMAD/1-decyne/3-hexyne	Yield (%)[a]	
		1a	**2**
1	1/2/10	34	18
2	1/2/20	38	21
3	1/2/40	44	24
4	1/2/60	39	26
5	1/4/40	53	24
6	1/6/40	57	20
7	1/8/40	43	9
8	1/10/40	40	trace

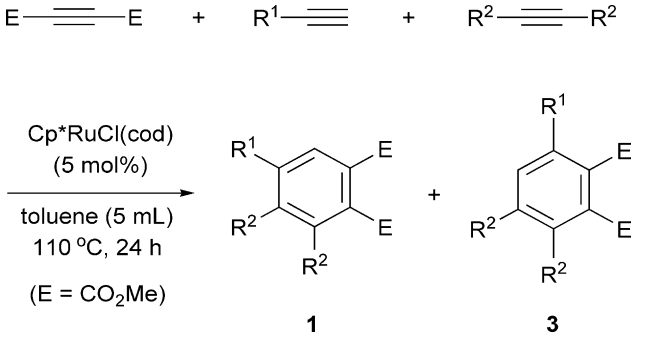
DMAD (0.20 mmol), $Cp^*RuCl(cod)$ (0.010 mmol), and toluene (5.0 mL) at 110 °C for 24 h.
[a] Determined by GLC.

Table 3
Catalytic [2 + 2 + 2] cycloaddition of several terminal alkynes, internal alkynes and DMAD

Run	R^1	R^2	Yield (%) (**1**:**3**)[a]
1	*n*-C_6H_{13}	C_2H_5	48 (100:0)
2	*n*-C_8H_{17}	C_2H_5	57 (100:0)
3	*n*-$C_{10}H_{21}$	C_2H_5	61 (100:0)
4	*n*-C_8H_{17}	*n*-C_3H_7	44 (65:35)
5	*n*-C_8H_{17}	*n*-C_5H_{11}	55 (28:72)
6	Ph	C_2H_5	35 (100:0)[b]
7	Me_3Si	C_2H_5	32 (100:0)[b]

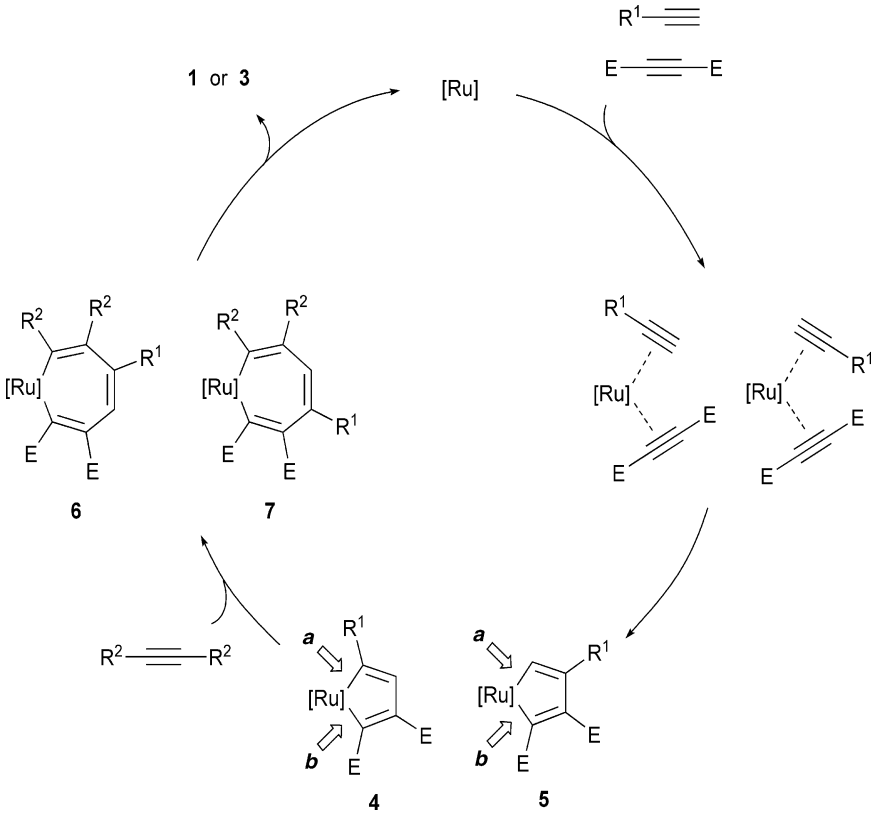
Terminal alkyne (24 mmol), internal alkyne (160 mmol), DMAD (4.0 mmol), $Cp^*RuCl(cod)$ (0.20 mmol), and toluene (100 mL) at 110 °C for 24 h.
[a] GLC yields. Ratios of **1**:**3** are in parentheses (determined by ^1H NMR).
[b] Isolated yields.

with longer alkyl chains gave a mixture of **1** and **3** (runs 4 and 5), and as shown in run 5, **3** was predominantly obtained over **1** when 6-dodecyne was applied. Phenylacetylene and trimethylsilylacetylene were also applicable as terminal alkynes to give the corresponding cocyclotrimers **1** regioselectivily in 35 and 32% (runs 6 and 7). In all cases, the formation of a certain amount of byproducts such as **2** still could not be suppressed. *t*-Butylacetylene did not afford the corresponding cotrimers, and instead, 5-*t*-butyl-1,2,3,4-benzenetetracarboxylate was obtained as a major product. Diphenylacetylene and bis(trimethylsilyl)acetylene in place of dialkylacetylenes did not give the desired cocyclotrimers.

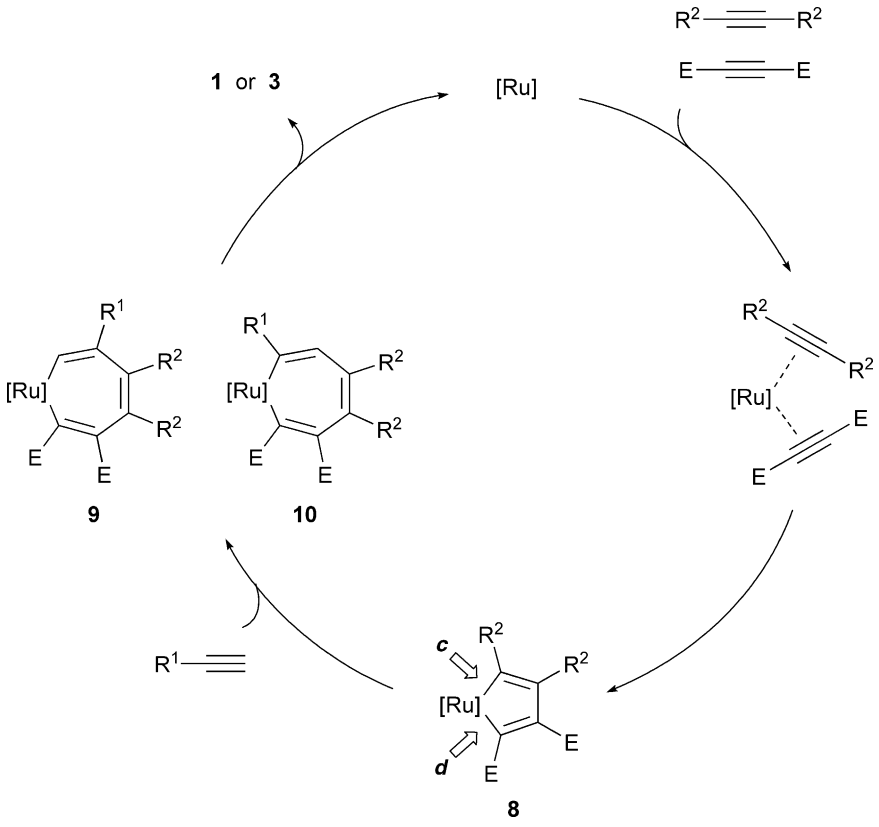


(3)

Possible reaction mechanisms (*paths A* and *B*) are shown in Schemes 1 and 2. In *path A*, DMAD and terminal alkyne coordinate to the ruthenium center ([Ru] = Cp^*RuCl) and subsequent oxidative cyclization gives ruthenacyclopentadiene **4** and **5**. There are two directions *a* and *b*, from which internal alkyne can insert to a ruthenium–carbon bond in **4** and **5**, respectively. Insertion from the direction *a* seems to be more favorable, since the ruthenium–carbon bond bearing an ester group is made stronger by π-back donation from ruthenium to the π^* orbital of α-carbon. Ruthenacycloheptatrienes **6** and **7** are formed by the insertion of an internal



Scheme 1. Possible reaction mechanism (*path A*).



Scheme 2. Possible reaction mechanism (*path B*).

alkyne to **4** and **5**, and reductive elimination from **6** and **7** gives the trialkylated *o*-phthalates **1** and **3** along with the regeneration of the catalytically active species. Although the insertion mechanism seems to be reasonable, Diels–Alder pathway from **4** and **5** also can explain the formation of the products. Recently, the mechanism via biscarbene intermediates has been proposed by Yamamoto and co-workers [18], and Kirchner et al. [19]. While this mechanism is also possible, we depicted here the present [2 + 2 + 2] cycloaddition using the classical mechanism because effects of substituents on acetylenes such as CO_2Me groups are not clarified completely.

An alternative mechanism, *path B*, is shown in Scheme 2. DMAD and an internal alkyne coordinate to ruthenium and oxidatively cyclizes to form ruthenacyclopentadiene **8** as a sole intermediate at this stage. There are two directions *c* and *d*, from which the terminal alkyne can insert to a ruthenium–carbon bond in **8**. Insertion from the direction *c* seems to be more favorable by the same reason described above. Either ruthenacycloheptatriene **9** or **10** is formed depending on the insertion direction of the terminal alkyne, and subsequent reductive elimination gives the trialkylated *o*-phthalates **1** and **3** along with the regeneration of the catalytically active species.

It seems to be reasonable to postulate that the initial oxidative cyclization step is almost irreversible, according to the DFT calculation of Ru(II)-catalyzed acetylene cyclotrimerization [18,19]. On the basis of this assumption, the ratio of metallacycles **4** and **5** formed in *path A* is kinetically determined and is independent of an internal alkyne. As a whole, the regioselectivity of products should mainly reflect the formation ratio of **4** to **5**, which is constant when the same terminal alkyne is used. However, as clearly shown in Table 3, the internal alkyne significantly influenced the regioselectivity of products (runs 2, 4 and 5). This is inconsistent with the discussion mentioned above. On the other hand, in *path B*, the regioselectivity depends on the insertion direction of a terminal alkyne in **8**. In this case it is plausible that substituents on the internal alkyne (R^2) influence the regioisomer ratio by steric hindrance between R^1 and R^2 at the insertion step, and thus, *path B* can explain the results shown in Table 3.

4. Conclusion

We have developed a ruthenium-catalyzed formation of polysubstituted *o*-phthalates by chemoselective [2 + 2 + 2] cycloaddition of three different alkynes. According to our observation, the present [2 + 2 + 2] cycloaddition would proceed via *path B*, where an internal alkyne reacts with DMAD first and the insertion of a terminal alkyne into the formed ruthenacycle intermediate is followed. Although some improvements are still required, the present methodology would contribute to overcome the problem of chemo- and

regio selectivity in the catalytic [2 + 2 + 2] cycloaddition of different alkynes lying in the long term.

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