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Ruthenium-catalysed synthesis of *o*-phthalates by highly chemoselective intermolecular [2 + 2 + 2] cycloaddition of terminal alkynes and dimethyl acetylenedicarboxylate

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Abstract

A highly chemoselective intermolecular [2 + 2 + 2] cycloaddition of 2 eq. of terminal alkynes with dimethyl acetylenedicarboxylate, which enables the straightforward synthesis of dialkylated *o*-phthalates, was successfully accomplished using a ruthenium catalyst, Cp*RuCl(cod) (Cp*: pentamethylcyclopentadienyl, cod: 1,5-cyclooctadiene). The co-cyclotrimerisation of alkynes and acetylenedicarboxylates usually affords 1:2 adducts (1,2,3,4-benzenetetracarboxylates), however, in the present reaction 2:1 adducts (*o*-phthalates) are the major products unprecedentedly.

Keywords: Dimethyl acetylenedicarboxylate; [2 + 2 + 2] Cycloaddition; Terminal alkyne; *o*-Phthalates; Cp*RuCl(cod)

1. Introduction

Aromatic ring construction by transition-metal complex-catalysed cyclotrimerisation of alkynes is a well-known process [1,2]. The selective arene formation from three different alkynes has been achieved by the use of stoichiometric zirconium [3] or titanium [4] reagents. As for the catalytic co-cyclotrimerisation of different alkynes, diynes have been frequently used as a substrate which couples with another alkyne chemo- and regioselectively (i.e. half-intermolecular [2 + 2 + 2] cycloaddition) [1,5–8]. On the other hand, in the completely intermolecular catalytic reaction, the co-cyclotrimerisation of two different alkynes is even surprisingly rare [1,9,10] because it normally affords a complex mixture of aromatic compounds due to difficult control of the cross-coupling selectivity, and, therefore, it still remains to be explored. Here we disclose the first transition-metal complex-catalysed synthesis of *o*-phthalates by highly chemoselective intermolecular [2 + 2 + 2] cycloaddition of 2 eq. of terminal alkynes and dimethyl acetylenedicarboxylate (DMAD).

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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. IR spectra were recorded on a Nicolet Impact 410 FT-IR spectrometer. Elemental analyses were performed at the Microanalytical Center of Kyoto University. HR-MS spectra were recorded on JEOL SX102A spectrometers with *m*-nitrobenzyl alcohol (*m*-NBA) as a matrix.

2.2. Materials

CpRuCl(cod) [11], Cp*RuCl(cod) (Cp*: pentamethylcyclopentadienyl, cod: 1,5-cyclooctadiene) [12], $RuCl_2(PPh_3)_3$ [13], $RuHCl(CO)(PPh_3)_3$ [14], Ru(cod)(cot) [15], Ru(cot)(dmfm)$_2$ [16], $RhH(CO)(PPh_3)_3$ [17] and Pd(dba)$_2$ [18]

were synthesised as described in the literature. Other complexes and 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. Synthesis of dimethyl 3,5-di-n-alkyl-o-phthalates (1) and dimethyl 3,6-di-n-alkyl-o-phthalates (2)

2.3.1. Dimethyl 3,5-di-n-hexyl-o-phthalate (1a) and dimethyl 3,6-di-n-hexyl-o-phthalate (2a)

A mixture of 1-octyne (5.0 mmol), DMAD (1.0 mmol) and Cp*RuCl(cod) (0.050 mmol) in toluene (30 ml) was stirred at 110 °C for 24 h under Ar. Kugelrohr distillation (180 °C, 2.0 mmHg) of the reaction mixture, and subsequent silica gel column chromatography (hexane/ethyl acetate = 25/1) afforded pure **1a** and **2a** together with a mixture of **1a** and **2a**, in the total isolated yield of 59% (**1a**:*2*a = 1.3:1). Compounds **1b**–**d** and **2b**–**d** were also prepared in a similar manner described above.

For **1a**: IR (cm^{-1}, neat) 2953, 2927, 2856, 1736, 1608. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.63 (s, 1H), 7.27 (s, 1H), 3.91 (s, 3H), 3.87 (s, 3H), 2.60 (m, 4H), 1.59 (m, 2H), 1.58 (m, 2H), 1.29 (m, 12H), 0.88 (t, $J = 6.8$ Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 169.0, 165.7, 144.0, 139.8, 133.1, 131.9, 127.6, 126.9, 51.8, 51.7, 35.2, 33.0, 31.3, 31.3, 31.0, 30.8, 28.8, 28.5, 22.3, 22.2, 13.7 (2C). Anal. Calcd for $C_{22}H_{34}O_4$: C 72.89, H 9.45. Found: C 72.62, H 9.75.

For **2a**: IR (cm^{-1}, neat) 2954, 2928, 2857, 1731, 1610. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.49 (s, 2H), 3.89 (s, 6H), 2.64 (t, $J = 7.8$ Hz, 4H), 1.57 (m, 4H), 1.34 (m, 12H), 0.89 (t, $J = 8.0$ Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 168.0 (2C), 144.1 (2C), 129.5 (2C), 129.1 (2C), 52.4 (2C), 32.6 (2C), 31.6 (2C), 30.9 (2C), 29.2 (2C), 22.7 (2C), 14.2 (2C). Anal. Calcd for $C_{22}H_{34}O_4$: C 72.89, H 9.45. Found: C 72.65, H 9.47. The structures of **1a** and **2a** were also confirmed by ^{13}C NMR inadequate measurements.

2.3.2. Dimethyl 3,5-di-n-butyl-o-phthalate (1b) and dimethyl 3,6-di-n-butyl-o-phthalate (2b)

For **1b**: Colourless liquid. IR (cm^{-1}, neat) 2949, 2928, 2872, 1737, 1610. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.64 (s, 1H), 7.22 (s, 1H), 3.92 (s, 3H), 3.87 (s, 3H), 2.63 (m, 4H), 1.61 (m, 4H), 1.35 (m, 4H), 0.88 (t, $J = 7.2$ Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 169.7, 166.3, 143.9, 140.2, 133.5, 132.1, 127.8, 127.4, 52.4, 52.3, 35.3, 33.6, 33.4, 33.0, 22.6, 22.3, 14.0 (2C). Anal. Calcd for $C_{18}H_{26}O_4$: C 70.56, H 8.55. Found: C 70.43, H 8.75.

For **2b**: Colourless liquid. IR (cm^{-1}, neat) 2949, 2867, 1729, 1610, 1564. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.49 (s, 2H), 3.89 (s, 6H), 2.65 (t, $J = 8.0$ Hz, 4H), 1.57 (quintet, $J = 8.0$ Hz, 4H), 1.40 (m, 4H), 0.86 (t, $J = 8.0$ Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 168.0 (2C), 144.0 (2C), 129.5 (2C), 129.0 (2C), 52.4 (2C), 33.0 (2C), 32.2 (2C), 22.7 (2C), 14.0 (2C). Anal. Calcd for $C_{18}H_{26}O_4$: C 70.56, H 8.55. Found: C 70.77, H 8.85.

2.3.3. Dimethyl 3,5-di-n-heptyl-o-phthalate (1c) and dimethyl 3,6-di-n-heptyl-o-phthalate (2c)

For **1c**: Colourless liquid. IR (cm^{-1}, neat) 2953, 2927, 2856, 1735, 1610. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.63 (s, 1H), 7.21 (s, 1H), 3.91 (s, 3H), 3.87 (s, 3H), 2.62 (t, $J = 8.0$ Hz, 2H), 2.58 (t, $J = 8.0$ Hz, 2H), 1.58 (m, 4H), 1.29 (m, 16H), 0.88 (t, $J = 7.6$ Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 169.7, 166.3, 143.9, 140.3, 133.5, 132.1, 127.8, 127.4, 52.4, 52.3, 35.6, 33.4, 31.8, 31.8, 31.4, 31.2, 29.5, 29.3, 29.2, 29.1, 22.7 (2C), 14.2 (2C). HR-MS (FAB–*m*-NBA): *m*/*z* 391.2860 $(M + H)^+$, calcd for $C_{24}H_{39}O_4$ 391.2848.

For **2c**: Colourless liquid. IR (cm^{-1}, neat) 2954, 2928, 2856, 1747, 1595, 1564. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.49 (s, 2H), 3.89 (s, 6H), 2.64 (t, $J = 8.0$ Hz, 4H), 1.57 (m, 4H), 1.30 (m, 16H), 0.89 (t, $J = 8.0$ Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 168.1 (2C), 144.1 (2C), 129.5 (2C), 129.1 (2C), 52.5 (2C), 32.6 (2C), 31.8 (2C), 31.0 (2C), 29.7 (2C), 29.2 (2C), 22.7 (2C), 14.2 (2C). HR-MS (FAB–*m*-NBA): *m*/*z* 391.2833 $(M + H)^+$, calcd for $C_{24}H_{39}O_4$ 391.2848.

2.3.4. Dimethyl 3,5-di-n-octyl-o-phthalate (1d) and dimethyl 3,6-di-n-octyl-o-phthalate (2d)

For **1d**: Colourless liquid. IR (cm^{-1}, neat) 2926, 2855, 1736, 1465, 1436. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.63 (s, 1H), 7.22 (s, 1H), 3.92 (s, 3H), 3.87 (s, 3H), 2.62 (t, $J = 8.0$ Hz, 2H), 2.58 (t, $J = 8.0$ Hz, 2H), 1.58 (m, 4H), 1.30 (m, 10H), 1.27 (m, 10H), 0.88 (t, $J = 6.8$ Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 169.7, 166.3, 143.9, 140.2, 133.6, 132.1, 127.7, 127.4, 52.4, 52.3, 35.6, 33.4, 31.9 (2C), 31.4, 31.3, 29.6, 29.5, 29.4, 29.3, 29.3, 29.2, 22.8 (2C), 14.2 (2C). HR-MS (FAB–*m*-NBA): *m*/*z* 419.3161 $(M + H)^+$, calcd for $C_{26}H_{43}O_4$ 419.3161.

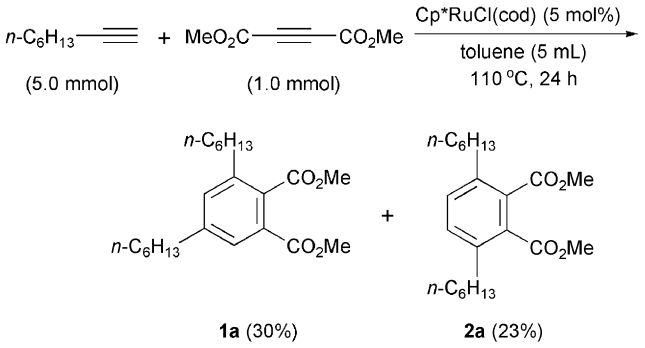
For **2d**: Colourless liquid. IR (cm^{-1}, neat) 2926, 2855, 1731, 1466, 1434. ^1H NMR ($CDCl_3$, 300 MHz) δ 7.49 (s, 2H), 3.89 (s, 6H), 2.63 (t, $J = 8.0$ Hz, 4H), 1.57 (m, 4H), 1.28 (m, 20H), 0.80 (t, $J = 8.0$ Hz, 6H). ^{13}C NMR ($CDCl_3$, 75 MHz) δ 168.0 (2C), 144.0 (2C), 129.4 (2C), 129.0 (2C), 52.4 (2C), 32.6 (2C), 31.9 (2C), 30.9 (2C), 29.7 (2C), 29.5 (2C), 29.3 (2C), 22.7 (2C), 14.2 (2C). HR-MS (FAB–*m*-NBA): *m*/*z* 419.3178 $(M + H)^+$, calcd for $C_{26}H_{43}O_4$ 419.3161.

3. Results and discussion

3.1. Catalytic [2 + 2 + 2] cycloaddition of 1-octyne and DMAD

A reaction of an excess of 1-octyne with DMAD was examined in toluene under reflux for 24 h in the presence of a catalytic amount of Cp*RuCl(cod) (Eq. (1)). As a result, an isomeric mixture of dialkylated *o*-phthalates **1a** and **2a** was obtained as main products in 30 and 23% yield, respectively, along with the formation of by-products, hexamethyl mellitate (for Ru-catalysed homocyclotrimerisation of acetylenedicarboxylates, see [19])

and tetramethyl 5-hexyl-1,2,3,4-benzenetetracarboxylate. 4,5-Dihexyl-*o*-phthalate was not obtained at all. Although the co-cyclotrimerisation of alkynes and acetylenedicarboxylates usually affords 1:2 adducts (1,2,3,4-benzenetetracarboxylates) [9], in this reaction 2:1 adducts (*o*-phthalates) are the major products unprecedentedly.



(1)

Several catalysts and solvents were surveyed for the [2 + 2 + 2] cycloaddition of 1-octyne and DMAD at 110 °C for 24 h (Table 1). The use of cyclohexane and diglyme afforded **1a** and **2a** in moderate yields (runs 2 and 3); however, polar solvents such as DMF and propionitrile were not suitable for this reaction (runs 4 and 5). A less sterically hindered complex, CpRuCl(cod), gave lower yields of the products compared with Cp*RuCl(cod) (run 6). Others such as $RuCl_2(PPh_3)_3$, $RuHCl(CO)(PPh_3)_3$, $RuCl_3{\cdot}3H_2O$ and zero-valent ruthenium complexes as well as rhodium and palladium complexes were also found to be inefficient (runs 7–14).

Table 1
Effects of catalysts and solvents on catalytic [2 + 2 + 2] cycloaddition of 1-octyne and DMAD[a]

Run	Catalyst	Solvent	Yield (%)[b]		
			1a	**2a**	Total
1	Cp*RuCl(cod)	Toluene	30	23	53
2	Cp*RuCl(cod)	Cyclohexane	23	12	35
3	Cp*RuCl(cod)	Diglyme	15	14	29
4	Cp*RuCl(cod)	DMF	1	3	4
5[c]	Cp*RuCl(cod)	Propionitrile	2	5	7
6	CpRuCl(cod)	Toluene	17	18	35
7	$RuCl_2(PPh_3)_3$	Toluene	9	14	23
8	$RuHCl(CO)(PPh_3)_3$	Toluene	6	8	14
9	$RuCl_3{\cdot}3H_2O$	Toluene	8	11	19
10	$Ru_3(CO)_{12}$	Toluene	5	6	11
11	Ru(cod)(cot)	Toluene	3	3	6
12	Ru(cot)(dmfm)$_2$	Toluene	5	7	12
13	$RhH(CO)(PPh_3)_3$	Toluene	6	3	9
14	Pd(dba)$_2$	Toluene	3	6	9

[a] 1-Octyne (5.0 mmol), DMAD (1.0 mmol), catalyst (0.050 mmol as a Ru atom), and solvent (5.0 ml) at 110 °C for 24 h.
[b] Determined by GLC.
[c] 1-Octyne (3.0 mmol) was used.

Table 2
Effect of the amount of toluene on Cp*RuCl(cod)-catalysed [2 + 2 + 2] cycloaddition of 1-octyne and DMAD[a]

Run	Amount of toluene (ml)	Yield (%)[b]		
		1a	**2a**	Total
1	5.0	30	23	53
2	10	37	29	66
3	30	47	39	86
4	50	43	38	81

[a] 1-Octyne (5.0 mmol), DMAD (1.0 mmol), Cp*RuCl(cod) (0.050 mmol), and toluene at 110 °C for 24 h.
[b] GLC yields.

Table 3
Cp*RuCl(cod)-catalysed [2 + 2 + 2] cycloaddition of several terminal alkynes and DMAD[a]

Run	R	Yield (%)[b]		
		1	**2**	Total
1	*n*-C_4H_9	35	26	61 (57)
2	*n*-C_6H_{13}	47	39	86 (59)
3	*n*-C_7H_{15}	49	39	88 (66)
4	*n*-C_8H_{17}	55	44	99 (62)

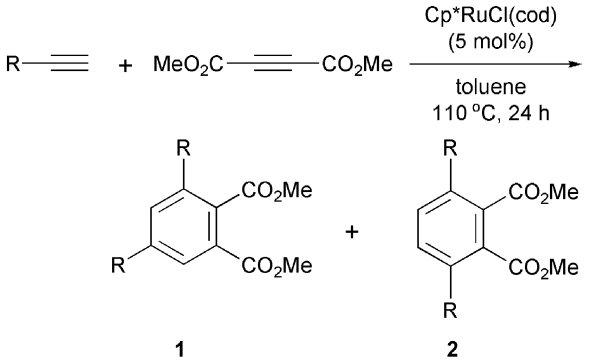
[a] Alkyne (5.0 mmol), DMAD (1.0 mmol), Cp*RuCl(cod) (0.050 mmol), and toluene (30 ml) at 110 °C for 24 h.
[b] GLC yields. Isolated yields are in parentheses.

3.2. Effect of the concentration of the reaction solution

Significant improvement of the yield was attained when the amount of toluene was increased (Table 2). The best result was obtained by the use of 30 ml of toluene for 1.0 mmol of DMAD (run 3). Formation of by-products, hexamethyl mellitate and tetramethyl 5-hexyl-1,2,3,4-benzenetetracarboxylate, was effectively suppressed by this dilution method. The diluting effect is not yet clear, while aggregation of the catalytically active species may contribute to the formation of by-products under high concentration.

3.3. Catalytic [2 + 2 + 2] cycloaddition of several terminal alkynes and DMAD

Under the optimised reaction conditions, Cp*RuCl(cod)-catalysed [2 + 2 + 2] cycloaddition of several terminal alkynes and DMAD was examined (Eq. (2), Table 3). Linear alkyl-substituted alkynes were applicable to afford the corresponding *o*-phthalates in high to excellent yields. Formation of **1** was slightly predominant over **2** (**1**:***2*** = 1.2–1.3:1), reducing the steric hindrance between the alkyl substituents and the ester groups during the cycloaddition reaction. On the other hand, bulky terminal alkynes and aromatic terminal alkynes, such as *tert*-butylacetylene, trimethylsilylacetylene and phenylacetylene, as well as internal alkynes mainly gave 1:2 cycloadducts of the alkyne and DMAD, and desired 2:1 cycloadducts were not obtained at all.

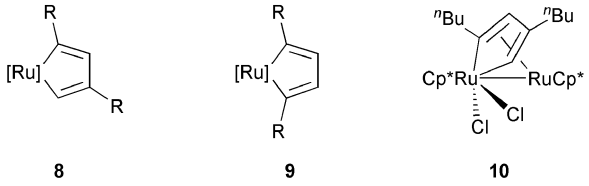


(2)

3.4. Reaction mechanism

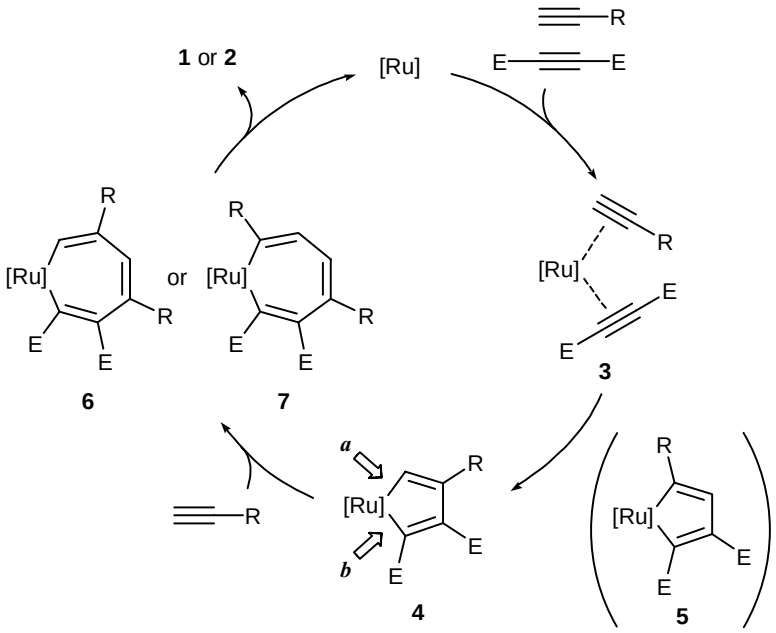
A possible reaction mechanism is shown in Scheme 1. First, the terminal alkyne and DMAD coordinate to an unsaturated ruthenium center ([Ru] = Cp*RuCl) to form 18-electron species **3** and subsequent oxidative cyclisation gives ruthenacyclopentadiene **4**. The regioisomer **5** might be also considered as an intermediate at this stage; however, steric hindrance between the Cp* and R groups should disturb the formation of **5**. Besides, formation of **2** and no formation of 4,5-dialkyl-*o*-phthalate from **5** cannot be explained. There are two directions *a* and *b*, from which a third alkyne can insert to a ruthenium-carbon bond in **4**. Insertion from the direction *a* seems to be more favourable, since the insertion from *a* is less hindered than from *b* and the ruthenium–carbon bond bearing an ester group is made stronger by π-back donation from ruthenium to the π* orbital of α-carbon. Either ruthenacycloheptatriene **6** or **7** is formed depending on the insertion direction of the third alkyne from *a*, and reductive elimination from **6** or **7** gives the corresponding *o*-phthalate **1** or **2**, with regeneration of the catalytically active species. Although the insertion mechanism seems to be reasonable, Diels–Alder mechanism also can explain the formation of the products.

As an alternative pathway, formation of two isomers of ruthenacyclopentadiene (**8** and **9**) from two moles of the terminal alkyne and subsequent insertion of DMAD can be considered. However, it is reported that the reaction of Cp*RuCl(tmeda) with 1-hexyne gave ruthenacyclopentadiene **10** regioselectively, which has two *n*-butyl groups at α and β′ positions, and a species derived from α,α′-dialkyl substituted ruthenacycle **9** was not obtained at all [20]. If the [2 + 2 + 2] cycloaddition proceeds via the oxidative cyclization of two terminal alkynes as a first step, only the ruthenacycle **8** would be formed according to the result described above, and **1** would be obtained as a sole product. This assumption is not consistent with the regioisomer distribution of the present reaction (**1**:***2*** = 1.2–1.3:1), therefore the mechanism via **8** and/or **9** is unlikely.



4. Conclusion

We have developed a novel ruthenium-catalysed synthesis of *o*-phthalates by [2 + 2 + 2] cycloaddition of terminal



Scheme 1. Possible reaction mechanism.

alkynes and DMAD. Although the regioselectivity and the scope of the reaction are not satisfactory so far, the high chemoselectivity is remarkable since it is difficult to control whenever monoynes are used as substrates. Further applications taking advantage of the highly chemoselective coupling step are expected.

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