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Regio- and Stereoselective Homodimerization of Monosubstituted Acetylenes in the Presence of the Second Generation Grubbs Catalyst

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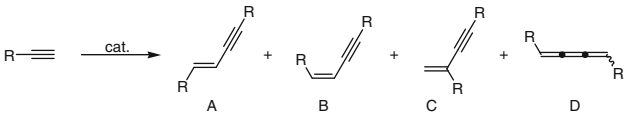
Abstract Silyl- and phenylacetylenes undergo efficient homodimerization in the presence of a second generation Grubbs catalyst. The reaction permits fully regio- and stereoselective synthesis of disubstituted 1,3-enynes. The other commonly used ruthenium-based olefin metathesis catalysts remain inactive in the reaction.

Keywords Homogeneous catalysis · Acetylenes · Dimerization · Ruthenium · Alkylidene complexes

1 Introduction

Conjugated 1,3-enynes are important building blocks widely used in synthetic organic chemistry. This class of molecules has been investigated for their antimicrobial activity [1] and more recently for their photophysical properties [2–4]. Moreover, conjugated 1,3-enyne skeleton has been found in natural products [5, 6]. Homodimerization of monosubstituted acetylenes is a convenient method for the preparation of 1,3-enynes allowing their synthesis in an atom economical manner. Many examples of effective run of the process in the presence of transition metal complexes have been described. Catalytic activity in the process has been reported to be shown by complexes of such metals as palladium [4, 7–13], rhodium [14–19], ruthenium [20–29], nickel [30], iridium [31–33], osmium [34, 35], iron [36] and the f-block metals [37–39]. However, a highly selective synthesis of conjugated enynes by dimerization is still a challenging process. Depending on the type of catalyst and the nature of substituent at the triple bond, the reaction may produce different products, or a mixture thereof (Eq. 1).



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From among a number of ruthenium-based catalytic systems used in alkyne dimerization, the application of ruthenium-based olefin metathesis catalysts has also been mentioned. The thermolysed first generation Grubbs catalyst $[RuCl_2(PCy_3)_2(=CHPh)]$ (**1**) exhibits activity in dimerization of phenylacetylene giving mixture of isomers [40] or preference for the formation of E-isomer of head to head dimer [41]. Moreover, complex **1** catalyzes the dimerization of arylethynes to the corresponding 1,4-substituted 1,3-enynes with pronounced Z-selectivity in aqueous environment in the presence of sodium dodecyl sulphate [42]. Formation of a product of phenylacetylene homodimerization was observed in the study of hydrosilylation of phenylacetylene with trisubstituted silanes in the presence of a second generation Grubbs catalyst [43]. Moreover, Ozawa has reported highly Z-selective dimerization of arylacetylenes with silylacetylenes catalyzed by ruthenium vinylidene precursors [44].

We now report on a highly efficient, regio- and stereoselective dimerization of terminal alkynes in the presence of

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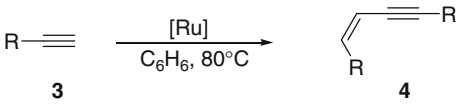
commercially available ruthenium alkylidene complex—the second generation Grubbs catalysts [$RuCl_2(H_2IMes)(PCy_3)$(=CHPh)] (**2**).

2 Results and Discussion

Addition of a second generation Grubbs catalyst (**2**) to a benzene solution of *tert*-butyldimethylsilylacetylene at 80 °C resulted in an immediate change in colour of the solution from red to green. Monitoring of the reaction mixture by GC demonstrated gradual formation of a single product, which was identified by GC–MS as a product of acetylene dimerization (Eq. 1). The product was isolated by liquid chromatography and subjected to analysis by NMR spectroscopy. NMR spectrum revealed formation of Z-isomer of head to head dimer (**4**) (Eq. 2). A similar reaction course was also observed for other silylacetylenes (Eq. 2).



3 **4** 2

R = $SiMe_2{}^tBu$ (a), $SiEt_3$ (b), Si^iPr_3 (c)

High yields and complete regio- and stereoselectivity was observed for triethylsilylacetylene and tri*iso*-propylsilylacetylene (Table 1). From among the silylacetylenes tested, trimethylsilylacetylene was the only one for which non-selective reaction occurred. In this case, the reaction leads to a mixture of head to head (**4**) and head to tail (**5**) dimers (Eq. 3).

Table 1 Homodimerization of silylacetylenes in the presence of the second generation Grubbs catalyst as compared with other Ru catalysts

Silylacetylene SiR_3=	Cat.	Temp [°C]	Conversion [%]	Yield of 4 [%]
$SiMe_2$t-Bu	**1**	80	0	0
$SiMe_2$t-Bu	**2**	80	89	85 (80)
$SiMe_2$t-Bu	**6**	80	<5	0
$SiMe_2$t-Bu	**7**	80	<5	0
$SiMe_2$t-Bu	**8**	80	<5	0
$SiMe_2$t-Bu	**9**	80	0	0
$Si(i\text{-}Pr)_3$	**2**	110[a]	77	75 (69)
$SiEt_3$	**2**	80	84	83 (70)
$SiMe_3$	**2**	80[b]	88	85[c] (80)

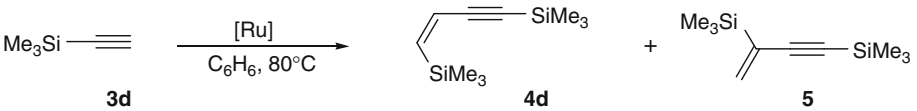
Reaction conditions: [Ru] 5 mol% in relation to alkyne, benzene, 80 °C, 24 h

[a] Toluene

[b] Closed system

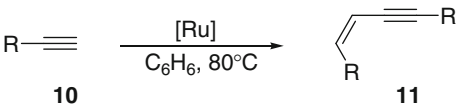
[c] Total yield, mixture of isomers [**4d**]:[**5**] = 1:3.3

In order to determine the scope of the reaction, a number of other monosubstituted acetylenes were tested. It was found that in the presence of ruthenium alkylidene complex **2** selective dimerization was also observed when phenylacetylene and its derivatives substituted in position 4 were used as reaction substrates. Catalytic tests performed for selected substituted phenylacetylenes showed complete regio- and stereoselective course of the reaction. For each arylacetylene tested the exclusive formation of isomer **11** was observed (Eq. 4 ; Table 2).



3d **4d** **5** 3

For each reaction, the resulting dimer was isolated by liquid chromatography and spectroscopically characterized. Identification of the product structures was made on the basis of ^{1}H NMR and MS spectra and by comparison of the spectra recorded with those reported in literature. For dimerization of trimethylsilylacetylene a mixture of isomers **4d** and **5** was isolated and analyzed by spectroscopic methods. Structure of product **5** was confirmed by ^{1}H and ^{13}C NMR and DEPT (see "Experimental Section"). Interestingly, the reaction was observed only in the presence of catalyst **2**. The other commonly used ruthenium alkylidene complexes (**1**, **6**–**9** in Fig. 1) remained inactive in the reaction.

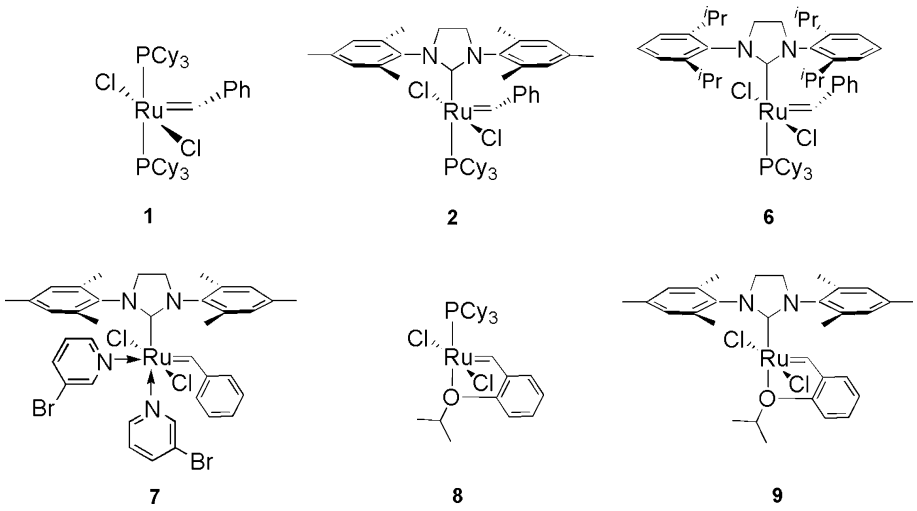


10 **11** 4

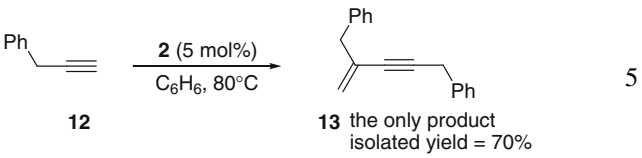
R= C_6H_5 (a), $C_6H_4CH_3$-4 (b), $C_6H_4CF_3$-4 (c)

Interestingly, dimerization of less sterically crowded 1-(prop-2-ynyl) benzene (**12**) in the presence of catalyst **2** leads to fully selective formation of head to tail dimer (**13**) (Eq. 5). Product **13** was isolated and its structure was proposed on the basis of spectroscopic characterization (^{1}H

Fig. 1 Olefin metathesis catalysts tested in dimerization of silyl- and arylacetylenes



and ^{13}C NMR, DEPT and MS). The presence of a geminal methylene group at the double bond was confirmed on the basis of DEPT sequence, which shows one CH_2 carbon in the olefinic region ($\delta = 121.3$ ppm).



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When hexyne or *tert*-butylacetylene was heated in the presence of catalyst **2**, both in the open system (under argon) and in the sealed glass vials (benzene, 80 °C, 24 h) no dimerization was observed.

There are two general mechanisms proposed for acetylene dimerization in the presence of ruthenium complexes (Scheme 1).

Bianchini mechanism (Scheme 1 path a) involves a formation of vinylidene complexes and migratory insertion of acetylide ligand to α-carbon of a ruthenium vinylidene intermediate [45–47]. The other mechanism, explaining formation of head to tail dimers involves direct insertion of the acetylene molecule coordinated to ruthenium into Ru–C bond in acetylide complex (Scheme 1, paths b and c). Protonation of enynyl species by the acidic proton of the terminal alkyne, produces enynes and regenerates the active catalyst. The insertion mechanism has been originally proposed by Trost for palladium complexes [8]. However, it has also been postulated for ruthenium complexes [48].

Although there are some examples in literature describing the activity of ruthenium-based olefin metathesis catalyst in non-metathesis transformations of acetylenes [49], the systems containing ruthenium alkylidene complexes and terminal alkynes have not yet been thoroughly investigated. In particular, there is no literature data on the mechanism of

Table 2 Homodimerization of phenylacetylenes in the presence of the second generation Grubbs catalyst as compared with other Ru catalysts

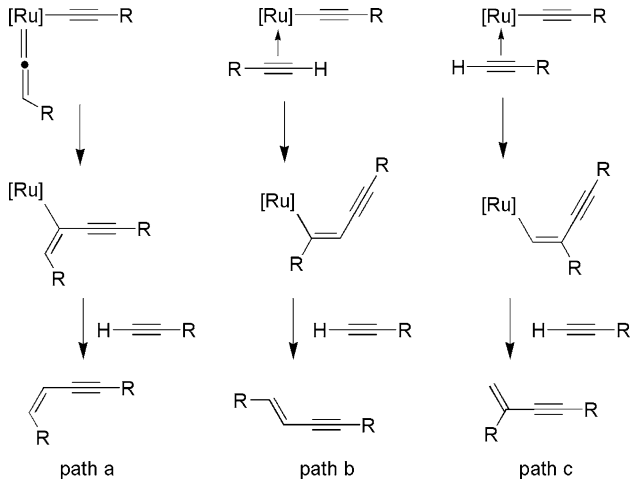
RC≡CH R=	Cat.	Conv. of 10 [%]	Yield (isolated) of 11 [%]
C_6H_5	**1**	<5	0
C_6H_5	**2**	65[a]	65[a]
		95	95 (90)
C_6H_5	**6**	75[a]	75[a]
		85	80
C_6H_5	**7**	0	0
C_6H_5	**8**	<5	0
C_6H_5	**9**	0	0
$C_6H_4CH_3$-4	**2**	98[a]	96[a]
		100	98 (80)
$C_6H_4CF_3$-4	**2**	78[a]	75[a]
		80	77 (71)

Reactions conditions: benzene, 80 °C, 24 h

[a] 5 h

decomposition of alkylidene complexes of ruthenium in the presence of monosubstituted acetylenes. The reaction pathways leading to formation of real catalyst are not always known. For vinylidene precursors of the type $[RuCl_2(P\textit{i}\text{-}Pr_3)_2(=C=CHPh)]$ a direct transformation to catalytically active σ-acetylide complex $[RuCl(C\equiv CPh)(P\textit{i}\text{-}Pr_3)_2]$ via abstraction of HCl in the presence of a base has been demonstrated [44, 50]. On the other hand, Verpoort has proposed the in situ formation of an active acetylide complex by interaction of thermolysed first generation Grubbs catalyst with phenylacetylene [40].

Our preliminary study of the equimolar reactions of catalyst **2** with phenylacetylene or *tert*-butyldimethylsilylacetylene or phenylacetylene performed in an NMR tube and monitored by ^1H NMR (benzene, 80 °C) revealed gradual decomposition of Grubbs catalyst (**2**). However, no formation of σ-acetylide complex was observed.



Scheme 1 Mechanisms of acetylene homodimerization in the presence of ruthenium complexes

Although at present a discussion on the mechanisms of dimerisation in the systems studied would be premature, the formation of head to tail dimers (**5** and **13**) in the reaction conditions (for certain systems of reagents) justifies the expectation that the reaction occurs according to the insertion mechanism.

In conclusion, we have demonstrated efficient homodimerization of monosubstituted silyl or arylacetylenes taking place in the presence of the second generation Grubbs catalyst and leading (with some exceptions) to regio- and stereoselective formation of Z-1,4-disubstituted 1,3-enynes.

3 Experimental Section

3.1 Materials and Methods

Unless mentioned otherwise, all operations were performed by using standard Schlenk techniques. ^{1}H- and ^{13}C NMR spectra were recorded on a Varian 400 operating at 402.6 and 101.2 MHz, respectively. ^{31}P NMR spectra were recorded on a Mercury 300 operating at 121.5 MHz. GC analyses were carried out on a Varian CP-3800 (column: Rtx-5 30 m I.D. 0.53 mm) equipped with TCD. Chemicals were obtained from the following sources: Acetylenes, ruthenium alkylidene complexes, dichloromethane, benzene-d_6, decane, dodecane and *n*-hexane were obtained from Aldrich. All solvents were dried prior to use over CaH_2 and stored under argon. CH_2Cl_2 was additionally passed through a column with alumina and after that it was degassed by repeated freeze–pump–thaw cycles.

3.2 Representative Synthesis (**4a**)

Schlenk vessel (20 mL) was charged under argon with 10 mL of dry benzene and 187 μL (1.00×10^{-3} mol) *tert*-butyldimethylsilylacetylene. Then, the solution was heated to 80 °C and the second generation Grubbs' catalyst 0.042 g (4.95×10^{-5} mol) was added. The reaction was carried out at the boiling point of benzene (80 °C) for 24 h. After completion of the reaction the resulting mixture was concentrated by evaporation of the solvent and the residue was purified using column chromatography (silica MN 60/hexane). (E)-1,4-Bis(*tert*-butyl-dimethylsilyl)but-1-en-3-yn (colorless oil) was obtained with 80 % of isolated yield.

3.3 Spectroscopic Characterization

*3.3.1 (E)-1,4-bis(tert-butyldimethylsilyl)but-1-en-3-yne (**4a**) [51]*

^{1}H NMR (C_6D_6; δ (ppm)): 0.18 (s, 6H); 0.30 (s, 6H); 0.96 (s, 9H); 1.03 (s, 9H); 6.00 (d, J = 15.4 Hz, 1H); 6.34 (d, J = 15.4 Hz, 1H); ^{13}C NMR (C_6D_6; δ (ppm)): −5.16; −4.51; 16.92; 17.38; 26.35; 26.60; 96.39; 106.72; 126.36; 143.12; MS *m/z*, (related intensity): 53(13), 59(25), 67(12), 73(56), 77(13), 125(13), 149(12), 167(100), 168(18), 169(29), 195(13), 223(29, M^+).

*3.3.2 (E)-1,4-bis(triethylsilyl)but-1-en-3-yne (**4b**) [52]*

^{1}H NMR (C_6D_6; δ (ppm)): 0.66 (q, J = 7.9 Hz, 6H, –$C\underline{H}_2CH_3$); 0.83 (q, J = 7.9 Hz, 6H, –$C\underline{H}_2CH_3$); 1.06 (t, J = 5.9 Hz, 9H, –$CH_2C\underline{H}_3$); 1.08 (t, J = 5.9 Hz, 9H, –$CH_2C\underline{H}_3$); 5.96 (d, J = 15.3 Hz, 1H); 6.37 (d, J = 15.3 Hz, 1H); ^{13}C NMR (C_6D_6; δ (ppm)): 4.1; 4.7; 7.7; 7.8; 96.2; 106.9; 126.5; 142.5; MS *m/z* (related intensity): 56(17), 57(22), 59(100), 69(46), 83(46), 91(65), 97(26), 113(25), 120(36), 164(14), 195(18), 196(16), 223(52), 280(67, M^+).

*3.3.3 (E)-1,4-bis(triiso-propylsilyl)but-1-en-3-yne (**4c**) [51]*

^{1}H NMR (C_6D_6; δ (ppm)): 0.94–1.04 (m, 42H, $(CH_3)_2CH$); 5.88 (d, J = 15.8 Hz, 1H); 6.47 (d, J = 15.8 Hz, 1H); ^{13}C NMR (C_6D_6; δ (ppm)): 12.2; 12.4; 19.2; 19.6; 95.4; 108.3; 127.9; 141.0; MS *m/z* (related intensity): 54(36), 59(100), 60(15), 72(13), 83(24), 95(12), 109(12), 127(12), 128(18), 131(19), 139(19), 163(13), 237(21), 238(19), 240(15), 270(46), 280(52), 281(30), 282(12), 364(25, M^+).

*3.3.4 Mixture of (E)-1,4-bis(trimethylsilyl)but-1-en-3-yne (**4d**) and 2,4-bis(trimethylsilyl)but-1-en-3-yne (**5**) [53, 54]*

4d: ^{1}H NMR (C_6D_6; δ (ppm)): 0.10 (s, 18H); 6.07 (d, J = 15.1 Hz, 1H); 6.17 (d, J = 15.1 Hz, 1H); ^{13}C NMR (C_6D_6; δ (ppm)): 146.1; 124.4; 105.0; 98.5; −0.4; −1.2; MS *m/z* (related intensity): 73(15), 181(100), 182(18), 183(10); **5**: ^{1}H NMR ($CDCl_3$;δ (ppm)): 0.07 (s, 18H); 5.61 (d, J = 3.4 Hz, 1H); 6.03 (d, J = 3.4 Hz, 1H); ^{13}C NMR ($CDCl_3$;δ (ppm)): 134.8; 124.8; 105.0; 98.6; 1.2, −0.4; DEPT ($CDCl_3$; δ (ppm)): 105.0 (= CH_2); MS *m/z* (related intensity): 45(11), 73(35), 108(12), 155(52), 181(100).

*3.3.5 (E)-1,4-diphenylbut-1-en-3-yne (**11a**) [55]*

^{1}H NMR ($CDCl_3$; δ (ppm)): 7.35–7.45 (m, 6H); 7.51–7.56 (m, 2H); 7.96–7.99 (m, 2H); 5.96 (d, J = 11.9 Hz, 1H); 6.75 (d, J = 11.9 Hz, 1H); ^{13}C NMR ($CDCl_3$; δ (ppm)): 88.2; 95.8; 107.3, 123.4; 128.1; 128.3; 128.4; 128.5; 128.7; 131.4; 136.5; 138.8; MS *m/z* (related intensity): 51(91), 101(100), 202(31), 203(67), 204(59, M^+).

*3.3.6 (E)-1,4-bis(4-methylphenyl)but-1-en-3-yne (**11b**) [55]*

^{1}H NMR ($CDCl_3$; δ (ppm)): 7.84 (d, J = 8.2 Hz, 2H, Ph); 7.40 (d, J = 8.1 Hz, 2H, Ph); 7.20 (d, J = 8.1 Hz, 2H, Ph); 7.16 (d, J = 8.2 Hz, 2H, Ph); 6.66 (d, J = 11.9 Hz, 1H); 5.86 (d, J = 11.9 Hz, 1H); 2.38 (s, 6H, CH_3); ^{13}C NMR ($CDCl_3$; δ (ppm)): 21.4; 21.5; 87.9; 95.9; 106.5; 120.5; 127.4; 128.7; 128.9; 129.1; 131.3; 133.9; 138.2; 138.4; MS *m/z* (related intensity): 115(11), 129(13), 132(24), 141(13), 142(14), 143(53), 144(13), 145(12), 156(10), 157(100), 158(20), 171(13), 202(11), 217(16), 232(43), 233(11).

*3.3.7 (E)-1,4-bis[4-(trifluoromethyl)phenyl]but-1-en-3-yne (**11c**) [38]*

^{1}H NMR ($CDCl_3$; δ (ppm)): 7.54–7.64 (m, 6H, Ph); 6.80 (d, J = 12.0 Hz, 1H); 6.06 (d, J = 12.0 Hz, 1H); ^{13}C NMR ($CDCl_3$; δ (ppm)): 89.6; 95.1; 109.4; 137.2; 130.2 (q, J = 33.2 Hz); 130.0; 128.8; 126.5; 125.4 (q, J = 4.0 Hz); 125.2 (q, J = 4.0 Hz); 122.7; 113.1; 138.1; 139.5; MS *m/z* (related intensity): 55(11), 67(30), 81(34), 82(14), 95(23), 96(16), 109(12), 128(10), 129(52), 141(17), 142(13), 143(55), 159(15), 173(21), 185(11), 191(32), 197(41), 198(32), 199(18), 210(23), 211(100), 212(31), 225(27).

*3.3.8 4-Methylene-1,5-diphenylpent-2-yne (**13**) [54]*

^{1}H NMR ($CDCl_3$; δ (ppm)): 3.51 (s, 2H, CH_2); 3.69 (s, 2H, CH_2); 5.24 (d, J = 1.8 Hz); 5.40 (d, J = 1.8 Hz); 7.08–7.96 (m, 10H, Ph); ^{13}C NMR ($CDCl_3$; δ (ppm)): 25.4; 43.8; 83.0; 88.0; 121.3; 126.3; 127.8; 128.4, 129.1; 131.1; 138.6; DEPT ($CDCl_3$; δ (ppm)): 121.3 (=CH_2); MS *m/z* (related intensity): 50(19), 51(21), 63(25), 65(38), 77(13), 78(10), 89(18), 91(72), 115(80), 116(37), 117(16), 128(38), 129(11), 139(21), 141(100), 142(11), 152(10), 153(25), 154(15), 202(23), 215(28), 216(22), 217(62), 218(13), 231(24), 232(10, M^+).

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