Stoichiometric formation of conjugated dienyl ketones from 2,3-dienes with kentenes at a ruthenium(0) centre

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Paper

Stoichiometric formation of conjugated dienyl ketones from 1,3-dienes with ketenes at a ruthenium(0) centre[†]

**Masafumi Hirano,*[a,b] Takuya Okamoto,[a] Nobuyuki Komine[a,b] and Sanshiro Komiya[§a]**

A stoichiometric reaction of [Ru(η^4-2,3-dimethyl-1,3-butadiene)(η^4-1,5-COD)(NCMe)] (**1a**) with diphenylketene at room temperature produces a new zero-valent complex [Ru(η^4-4,5-dimethyl-1,1-diphenylhexa-3,5-diene-2-one)(η^4-1,5-COD)(NCMe)] (**2aa**) in 92% yield. This is the first example of a conjugated dienyl ketone formation from 1,3-dienes with ketene. Similarly, the treatment of **1a** with
10 ethylphenylketene produces the decarbonylated coupling product (*Z*)-5-alkyl-2,3-dimethyl-5-phenylhepta-1,4-diene (**5**) in 77% yields.

Introduction

The cross-dimerisation between unsaturated compounds is one of the attractive methods for production of highly functionalised
15 organic molecules with high atom- and step-economy.[1] We have documented stoichiometric and catalytic cross-dimerisations between 1,3-diene and substituted alkenes,[2] homo- and cross-dimerisations between 1,3-dienes[3] by ruthenium(0) complex, that goes through the oxidative coupling mechanism (Scheme 1).

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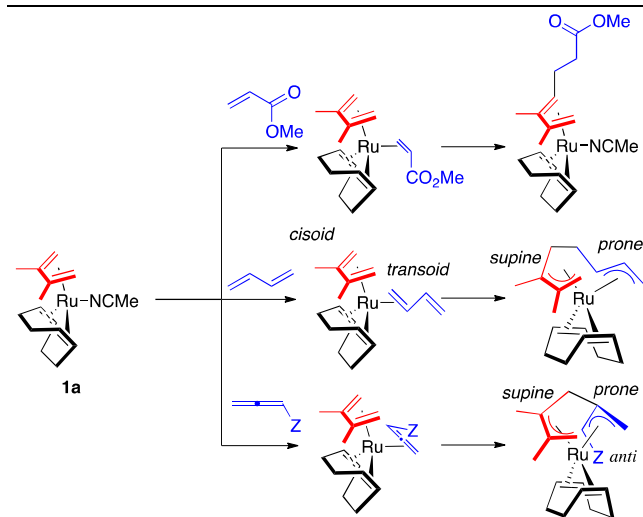
Scheme 1 Coupling reactions of 1,3-diene at Ru(0).

When a 1,3-diene complex of ruthenium(0) having a labile
25 acetonitrile ligand [Ru(η^4-1,3-diene)(η^4-1,5-COD)(NCMe)] (**1**)[4] was treated with mono-substituted allenes (1,2-dienes), bis(η^3-allylic)ruthenium(II) compounds having a new C–C bond were obtained as a single diastereomer having an *anti* configuration of the substituent Z, regardless of the electronic properties.[5] This

30 result means the Ru(0) centre distinguishes two different orthogonal π-planes and the prostereogenic faces of allenes, based on the steric congestion, where the Ru(0) centre favours the less congested site. Another feature of this reaction is the terminal carbon of 1,3-diene attacks the central *sp* carbon in allenes. This
35 probably arises from the nucleophilic nature of the coordinating 1,3-diene at the highly Lewis basic ruthenium(0).[2a]

Now we are interested in the reaction of **1** with ketenes as a series related to allenes. Although ketene is one of the powerful building blocks in modern synthetic chemistry,[6] the reaction
40 patterns with 1,3-dienes are rather limited except for tethered dienylketenes.[7] To our best knowledge, the reactions of ketenes with 1,3-dienes occur by two typical pathways; *i.e.* formal [2 + 2] cycloadditions by the C=C bond in ketenes giving four-membered cyclobutanones with cyclic 1,3-denes, and [4 + 2]
45 cycloadditions by the C=O bond giving dihydropyran with open-chain 1,3-dienes (Scheme 2).[8,9] The linear cross-dimerisation is unprecedented to date.
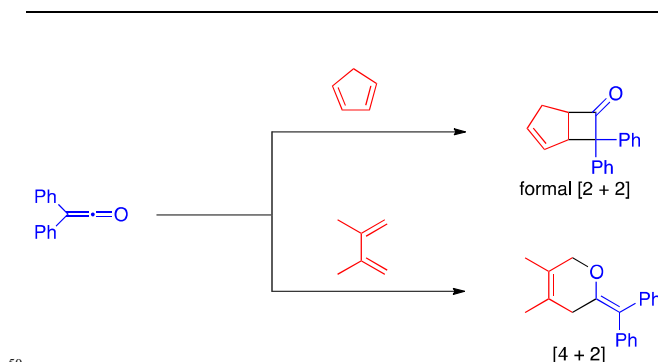
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Scheme 2 General reactions of ketene with 1,3-diene

As a typical example, the reaction of diphenylketene with 2,3-dimethyl-1,3-butadiene at 40 °C thermally gives corresponding
55 dihydropyran by [4 + 2] cyclisation in 97% yield.[10] As a very

unusual reaction, Yasuda and coworkers reported the reaction of [Cp$_2$Zr(1,3-diene)] with ketenes, where the C=O bond in ketene inserted into a Zr–C bond to give oxazirconacycles (Scheme 3).[11] This is probably arising from the strong contribution of the LX$_2$

5 ($\sigma_2\pi$) extreme[12] of the diene moiety at Zr(II).
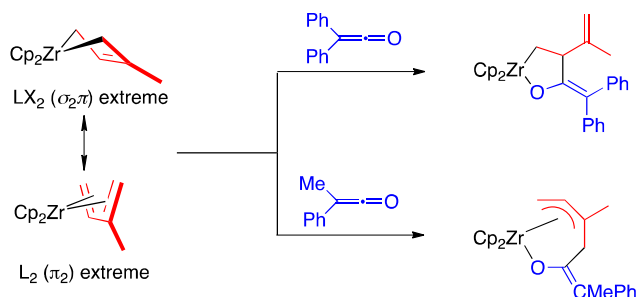
Scheme 3 Reaction of a diene complex of Zr with ketenes.

In this paper, we disclose the first example of the stoichiometric
10 linear cross-dimerisation between 1,3-dienes and ketenes to give the conjugated dienyl ketone complexes.[13]

Results and discussion

Treatment of [Ru(η^4-2,3-dimethyl-1,3-butadiene)(η^4-1,5-
15 COD)(NCMe)] (**1a**)[4] with diphenylketene in benzene at room temperature produced a new ruthenium(0) complex having a dienyl ketone, the coupling product, **2aa** in 92% yield. The single crystals suitable for the X-ray analysis were obtained by the recrystallisation from cold Et$_2$O as pure orange prisms in 25%
20 yield (Scheme 4).
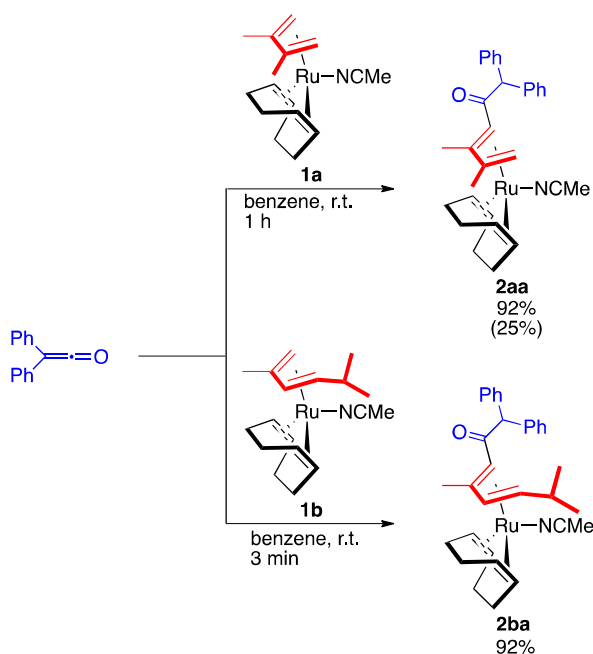
25 **Scheme 4** Reaction of 1,3-diene complexes of Ru(0) with

diphenylketene.[14]

Complex **2aa** was characterised by spectroscopic methods and elemental analysis but most unambiguously by the X-ray analysis
30 (Figure 1).
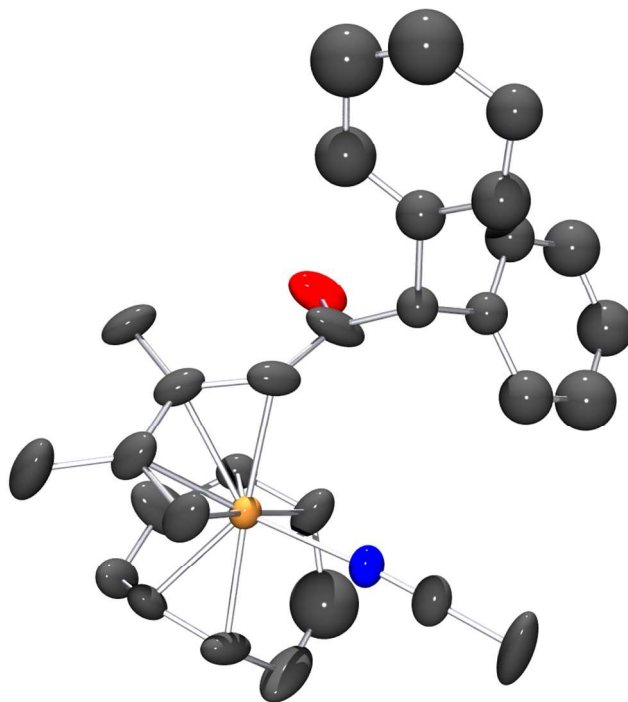
35 **Fig. 1** Molecular structure of **2aa**. The disordered diphenylmethyl fragment and all hydrogen atoms are omitted for clarity. Ellipsoids represent 50% probability.

40 Like allenes,[5] the central carbon in ketene reacts with a terminal carbon in 2,3-dimethylbutadiene. Unlike allenes, this reaction produces a ruthenium(0) complex of the coupling product. This is the first example for the linear cross-dimerisation between 1,3-dienes and ketenes giving conjugated dienyl ketones, that is also
45 formally regarded as an insertion of the C=C bond in ketene into the terminal C–H bond in 1,3-diene. As shown in Fig. 1, the coupling product, (*E*)-4,5-dimethyl-1,1-diphenylhexa-3,5-diene-2-one, coordinates to the ruthenium(0) centre as a *cisoid*-diene ligand and the diphenylmethylcarbonyl moiety locates in the *exo*
50 position. This compound was also characterised by ^{1}H NMR, ^{13}C{^{1}H} NMR, DEPT-135, COSY, and HETCOR. The IR spectrum in KBr showed intensive bands at 1636 and 2360 cm^{-1}, assignable to the stretching vibrations of the carbonyl and nitrile groups.

55 The acetonitrile ligand in **2aa** was readily replaced by PPh$_3$ at room temperature in 84% yield, and iodolysis of **2aa** gave (*E*)-4,5-dimethyl-1,1-diphenyhexa-3,5-diene-2-one (**4aa**) in 71% yield, which was confirmed by ^{1}H NMR, COSY and NOESY experiments, and GC-MS (Scheme 5).
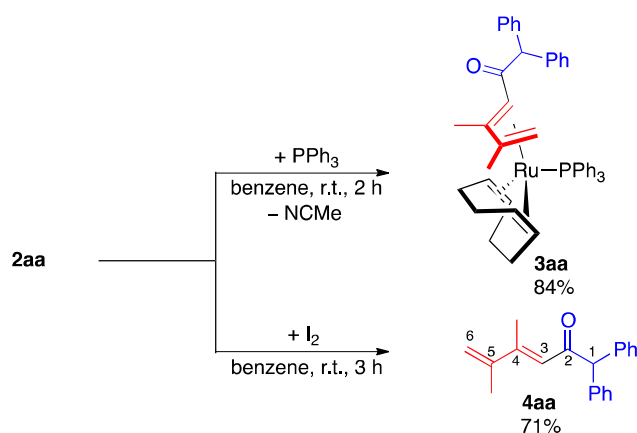
Scheme 5 Reactions of the dienyl ketone complex of Ru(0).

The *E*-form of **4aa** is consistent with this fragment in **2aa** observed by the X-ray. Heating of **2aa** at 50 °C, and reactions of **2aa** with 2,3-dimethyl-1,3-butadiene, electron-rich and –deficient alkenes, and diphenylketene produced complex mixtures. The treatment of 2,5-dimethylhexa-1,3-diene complex **1b** with diphenylketene also produced a similar zero-valent complex **2ba** in 92% yield (Scheme 4). However, reactions using the 1,3-butadiene, 1,3-pentadiene, and isoprene complexes gave complex mixtures.

Unexpectedly, the treatment of **1a** with alkylphenylketenes gave decarbonylated cross-dimers (Scheme 6).
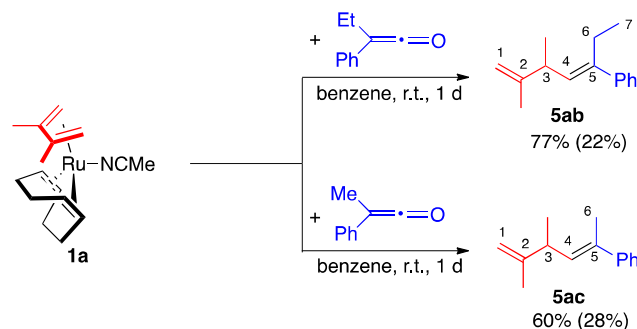

Scheme 6 Reactions of 1,3-diene complex of Ru(0) with alkylphenylketene.

Although fate of the corresponding ruthenium fragment is not clear because of low solubility of the precipitate containing the metal fragment in the solvents tried, the IR spectrum of the precipitate has an intense band in the range 1947-1938 cm⁻¹, suggesting a carbonyl complex. Similar decarbonylative couplings were reported for the reactions of diphenylketene with norbornene,[15,16] methyl acrylate[16] and terminal alkynes.[17]

In view of all these studies, ketenes are proposed to behave like allenes to give **2** (Scheme 7). In the first step, ketene initiates facile displacement of the acetonitrile ligand in **1a**. Since allenes

always coordinate to Ru(0) through the less-hindered π-plane regardless of the electronic properties,[5] ketenes probably favour to coordinate through the C=O bond from the steric point of view. Like allenes, the oxidative coupling reaction occurs to give **B** followed by isomerisation to **C** and then successive β-hydride elimination and reductive elimination yield **2**.
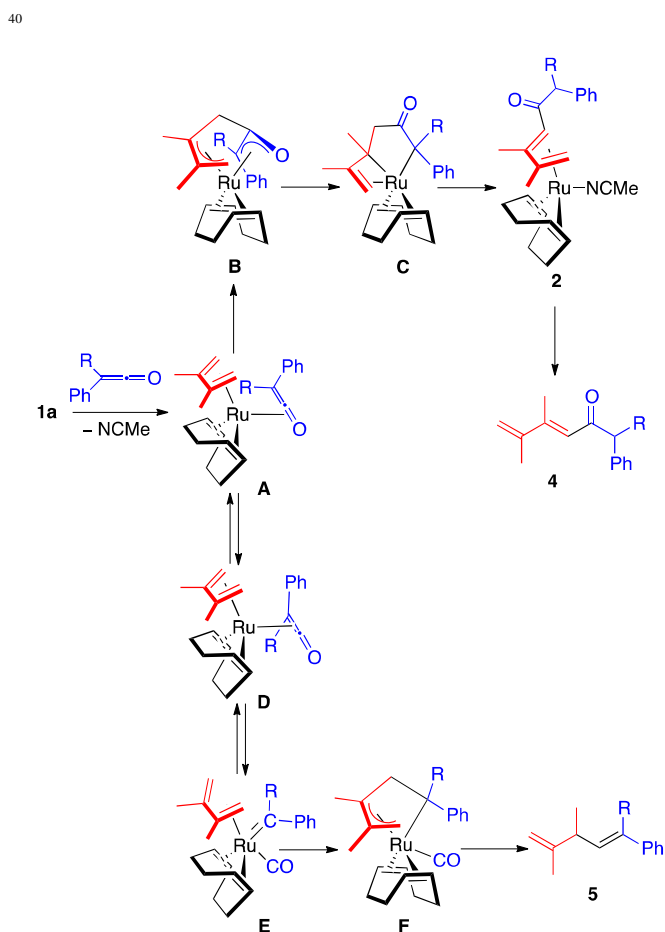
Scheme 7 Proposed reaction pathways

For the decarbonylation of ketenes, Miyashita and coworkers documented equilibria of coordinating ketenes among κ^2-*C,O*, η^2-*C,C* and the (carbene)(carbonyl) complexes of Ni(0)[18] and Pt(0)[19] (Scheme 8). On the other hand, Mitsudo and coworkers reported insertion of a C=C bond of ketene into a Pd–C bond followed by decarbonylation reaction (Scheme 8).[20] Although the formation mechanism for **5** remains unclear, we favour the (carbene)(carbonyl) mechanism because all our results support the terminal carbon of coordinating 1,3-diene to attack the central carbon of ketenes and allenes without exceptions. The second collateral evidence for this process is that the reaction of a carbonyl complex of ruthenium(0) with carbene is reported to give a ketene,[21] that can be regarded as a reverse reaction of the present one. On the basis of these considerations, we can propose equilibria among **A**, **D** and **E** in Scheme 7, and subsequent process yields the decarbonylated coupling product **5**. The reason of why alkyl ketene leads to the decarbonylation process is not clear at present. However, because the conversion into carbene/carbonyl is generally caused by strong back-donation to

the η^2-C,C-ketene, one of the reasons may be due to facile coordinational mode change from κ^2-C,O to the η^2-C,C for sterically less hindered ketenes.[22] In an attempt to probe the possibility that a (carbene)(diene)ruthenium(0) species (like **E**) serves as a precursor to **5**, 1 or 2 equiv of ethyl diazoacetate was added to a toluene solution of **1a** at room temperature.[23] Although gas evolution was observed from the mixture immediately, the coupling product was not observed in the product mixtures.

Miyashita

Mitsudo
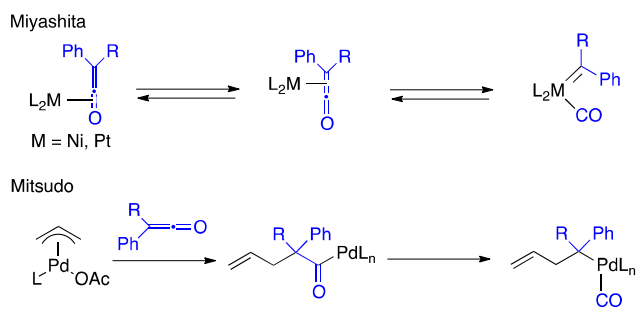
Scheme 8 Decarbonylation mechanisms

Conclusions

In summary, we have shown the first linear coupling reaction between coordinating 1,3-dienes and diphenylketenes to give conjugated dienyl ketones at a ruthenium(0) centre. A well-known decarbonylative coupling reaction also occurred when alkylphenylketene was used in this reaction. The key reaction is the oxidative coupling reaction between 1,3-diene and the C=C bond in ketene at highly Lewis basic ruthenium(0).

Experimental

General procedures

All procedures described in this paper were carried out under a nitrogen or argon atmosphere by use of Schlenk and vacuum line techniques. Benzene, toluene, hexane, THF, and Et$_2$O were dried and purified using a Glass Contour Ultimate Solvent Systems. Benzene-d_6 was dried over sodium wire and stored under vacuum, and it was used after vacuum distillation prior to use. [Ru(η^4-1,3-diene)(η^4-1,5-diene)(NCMe)] (**1**) was prepared according to the literature methods.[4] Diphenylketene,[16] methylphenylketene[24] and ethylphenylketene[24] were prepared by the literature methods and purified by distillation under reduced pressure. ^{1}H, ^{13}C and ^{31}P$\{^1$H$\}$ NMR spectra were measured on a JEOL EXC400P (400 MHz for ^{1}H) NMR spectrometer. The IR spectra were measured on a JASCO FT/IR4100 Fourier-Transform spectrometer. GC and GC-MS were performed on a Shimadzu GC14B (FID) and a Shimadzu QP2010 (EI) analysers, respectively, equipped a ZB-WAX-Plus column (0.25 mmϕ x 30 m). The elemental analyses were performed on a Perkin-Elmer 2400 Series II CHN analyser.

Reactions

[Ru(η^4-2,3-dimethylbuta-1,3-diene)(η^4-1,5-diene)(NCMe)] (1a) with diphenylketene. (method A): Complex **1a** (99.4 mg, 0.299 mmol) was placed in a 25 ml Schlenk tube into which benzene (6 ml) was introduced. Then diphenylketene (55 μl, 0.301 mmol) was added to the solution by a hypodermic syringe and the reaction mixture was stirred at room temperature for 3.5 h. Removal of all volatile matters gave yellow powder, which was recrystallised from cold Et$_2$O to give [Ru(η^4-CH$_2$CMeCMeCHC(O)CHPh$_2$)(η^4-1,5-diene)(NCMe)] (**2aa**) in 25% yield (38.2 mg, 0.0725 mmol). ^{1}H NMR (400 MHz, benzene-d_6, r.t.): δ 0.81 (s, 1H, *endo*-6-C*H*H), 0.99 (s, 3H, NC*Me*), 1.45 (s, 1H, *endo*-3-C*H*), 1.63 (s, 1H, *exo*-6-CH*H*), 1.82 (s, 3H, 4-C*Me*), 1.88-1.94 (m, 2H, COD), 2.03-2.06 (m, 2H, COD), 2.25-2.32 (m, 2H, COD), 2.39-2.47 (m, 3H, COD), 2.50 (s, 3H, 5-C*Me*), 3.21-3.26 (m, 1H, COD), 3.46-3.50 (m, 1H, COD), 3.96-4.00 (m, 1H, COD), 4.93 (s, 1H, 1-C*H*), 7.03-7.07 (m, 2H, *Ph*), 7.14-7.17 (m, 4H, *Ph*), 7.50-7.53 (m, 4H, *Ph*). ^{13}C$\{^1$H$\}$ NMR (100.5 MHz, benzene-d_6): δ 2.5 (s, NC*Me*), 14.4 (s, 5-C*Me*), 19.7 (s, 4-C*Me*), 30.8 (s, COD), 31.0 (s, COD), 31.6 (s, COD), 33.8 (s, COD), 42.2 (s, 6-*C*H$_2$), 56.2 (s, 3-*C*H), 64.9 (s, 1-*C*H), 73.4 (s, COD), 77.4 (s, COD), 79.5 (s, COD), 80.8 (s, COD), 98.6 (s, 4- or 5-*C*Me), 102.9 (s, 5- or 4-*C*Me), 126.5 (s, Ph), 126.7 (s, Ph), 128.8 (s, Ph), 129.2 (s, Ph), 129.7 (s, Ph), 141.5 (s, Ph), 142.0 (s, Ph), 201.6 (s, *C*O). IR: (KBr, cm^{-1}): 3061 (w), 3019 (w), 2983 (w), 2918 (m), 2872 (m), 2825 (m), 2360 (m), 1942 (br), 1636 (vs), 1398 (s), 1107 (m), 1026 (m), 700 (s). EA: Anal. Calcd for C$_{30}$H$_{35}$NORu: C, 68.41; 6.70; N, 2.66. Found: C, 68.79; H, 7.03; N, 2.58. (method B): Complex **1a** (12.2 mg, 0.0367 mmol) and triphenylmethane (11.3 mg, 0.0462 mmol) as an internal standard were placed in an NMR tube and benzene-d_6 (600 μl) was introduced into the NMR tube. After addition of diphenylketene (6.8 μl, 0.037 mmol) at room temperature, the NMR spectrum showed formation of **2aa** in 92% yield.

[Ru(η^4-2,5-dimethylhexa-1,3-diene)(η^4-1,5-diene)(NCMe)] (1b) with diphenylketene. Similar to the reaction of **1a** with diphenylketene, complex **1b** (14.8 mg, 0.041 mmol) was reacted with diphenylketene (7.5 μl, 0.041 mmol) in benzene-d_6 (600 μl) at room temperature, and the yield of [Ru(η^4-MeCHMeCHCHCMeCHC(O)CHPh$_2$)(η^4-1,5-diene)(NCMe)] (**2ba**) was estimated on the basis of triphenylmethane (8.0 mg, 0.033 mmol) as an internal standard. Yield : 92%. ^{1}H NMR (400 MHz, benzene-d_6, r.t.): δ 0.95 (d, J = 4.0 Hz, 3H, 8-C*Me*), 1.08 (s, 3H, NC*Me*), 1.10-1.12 (m, 3H, 8-C*Me*), 1.10-1.12 (m, 1H, 6-C*H*), 1.19-1.26 (m, 1H, 7-C*H*), 1.33 (s, 1H, 3-C*H*), 1.85-2.10 (m, 4H, COD), 2.17-2.21 (m, 2H, COD), 2.34-2.41 (m, 2H, COD), 2.63 (s, 3H, 4-C*Me*), 2.92-2.96 (m, 1H, COD), 3.14-3.19 (m, 2H, COD), 3.91-3.96 (m, 1H, COD), 4.55 (d, J = 8.6 Hz, 1H, 5-C*H*), 4.89 (s, 1H, 1-C*H*), 6.99-7.15 (m, 6H, *Ph*), 7.48-7.52 (m, 4H, *Ph*).

[Ru(η^4-CH$_2$CMeCMeCHC(O)CHPh$_2$)(η^4-1,5-diene)(NCMe)] (2aa) with PPh$_3$. Complex **2aa** (42.7 mg, 0.0811 mmol) was placed in a 25 ml Schlenk tube into which benzene (3 ml) and PPh$_3$ (22.1 mg, 0.0843 mmol) were added. The reaction mixture was stirred at room temperature for 2 h. Removal of all volatile matters yielded brown precipitate. Recrystallisation of the

precipitate from cold hexane/THF produced yellow-brown powders of [Ru(η^4-CH$_2$CMeCMeCHC(O)CHPh$_2$)(η^4-1,5-diene)(PPh$_3$)] (**3aa**) in 84% (55.4 mg, 0.0685 mmol). ^{1}H NMR (400 MHz, benzene-d_6, r.t.): δ 0.15 (d, J_{H-P} = 10.3 Hz, 1H, 3-C*H*), 0.18 (d, J_{H-P} = 14.9 Hz, 1H, *endo*-6-C*H*H), 0.37-0.42 (m, 1H, COD), 1.37-1.42 (m, 2H, COD), 1.55-1.70 (2H, COD), 1.77 (d, J_{H-P} = 2.3 Hz, 1H, *exo*-6-CH*H*), 1.91-1.95 (m, 1H, COD), 2.02 (d, J_{H-P} = 1.7 Hz, 3H, 5-C*Me*), 2.02-2.05 (m, 1H, COD), 2.10-2.21 (m, 1H, COD), 2.29-2.31 (m, 1H, COD), 2.40 (d, J_{H-P} = 2.8 Hz, 3H, 4-C*Me*), 3.01-3.07 (m, 1H, COD), 3.65-3.71 (m, 1H, COD), 4.14 (s, 1H, 1-C*H*), 4.23-4.26 (m, 1H, COD), 6.96-7.09 (m, 14H, *Ph*), 7.29 (t, J = 7.4 Hz, 2H, *Ph*), 7.36-7.42 (m, 2H, *Ph*), 7.55-7.59 (m, 5H, *Ph*), 7.76 (d, J = 7.4 Hz, 2H, *Ph*). ^{31}P{^{1}H} NMR (162 MHz, benzene-d_6, r.t.): δ 58.1 (s).

[Ru(η^4-CH$_2$CMeCMeCHC(O)CHPh$_2$)(η^4-1,5-diene)(NCMe)] (2aa) with idodide. Complex **2aa** (12.3 mg, 0.0234 mmol) was placed in an NMR tube into which benzene-d_6 (600 µl) and iodine (6.4 mg, 0.025 mmol) were added. After 3 h at room temperature, the supernatant liquid was transferred to another NMR tube and the resulting solid was washed with benzene-d_6. Into the combined benzene-d_6 triphenylmethane (10.107 mg, 0.04121 mmol) was added as an internal standard. The ^{1}H NMR spectrum and GC-MS showed formation of (*E*)-4,5-dimethyl-1,1-diphenylhexa-3,5-diene-2-one (**4aa**) in 71% yield. ^{1}H NMR (400 MHz, benzene-d_6, r.t.): δ 1.53 (s, 3H, 5-*Me*), 2.36 (s, 3H, 4-*Me*), 4.92 (s, 1H, 1-C*H*H), 5.06 (s, 1H, 1-C*H*), 5.15 (s, 1H, 6-CH*H*), 6.32 (s, 1H, 3-C*H*), 7.09-7.26 (m, 10H, *Ph*). GC-MS (IE): m/z = 276 (M$^+$).

[Ru(η^4-2,3-dimethylbuta-1,3-diene)(η^4-1,5-diene)(NCMe)] (1a) with ethylphenylketene. (method A): Complex **1a** (87.6 mg, 0.263 mmol) was dissolved in benzene (6 ml) in a Schlenk tube. Ethylphenylketene (60 µl, 0.393 mmol) was added into the solution by a hypodermic syringe and the reaction mixture was stirred at room temperature for one night. After removal of solvent, the product was extracted with benzene and the product was purified by silicagel column chromatography (hexane/Et$_2$O = 50/1) to give (*E*)-2,3-dimethyl-5-phenylhepta-1,4-diene (**5ab**) in 22% yield (15.2 mg, purity 75%, 0.0569 mmol). ^{1}H NMR (400 MHz, benzene-d_6, r.t.): δ 0.93 (t, J = 7.4 Hz, 3H, 7-*Me*), 1.05 (d, J = 6.9 Hz, 3H, 3-*Me*), 1.68 (s, 3H, 2-*Me*), 2.28 (qd, J = 7.4, 1.7 Hz, 2H, 6-C*H*$_2$), 2.92 (dq, J = 10.3, 6.8 Hz, 1H, 3-C*H*), 4.77 (s, 1H, 1-C*H*H), 4.82 (s, 1H, 1-CH*H*), 5.38 (dt, J = 9.8, 1.1 Hz, 1H, 4-C*H*), 7.03-7.25 (m, 5H, *Ph*). ^{13}C{^{1}H} NMR (100.5 MHz, benzene-d_6, r.t.): δ 13.3 (s, 7-*Me*), 20.2 (s, 3-*Me*), 21.3 (2-*Me*), 32.6 (s, 6-*Me*), 40.2 (s, 3-*C*H), 109.0 (s, 1-*C*H$_2$), 130.2 (s, 4-*C*H), 141.8 (s), 142.6 (s), 150.0 (s). GC-MS (EI): m/z = 200 (M$^+$). (method B): Complex **1a** (18.0 mg, 0.0541 mmol) and tripheylmethane (5.00 mg, 0.0205 mmol) was placed in an NMR tube into which benzene-d_6 was introduced (600 µl). Ethylphenylketene (15.0 µl, 0.0982 mmol) was added to the solution by a hypodermic syringe. The NMR spectra suggested formation of **5ab** in 77% yield.

[Ru(η^4-2,3-dimethylbuta-1,3-diene)(η^4-1,5-diene)(NCMe)] (1a) with methylphenylketene. (method A): Similar to the reaction of **1a** with ethylphenylketene, treatment of **1a** (114.0 mg, 0.3429 mmol) with methylphenylketene (120.0 µl, 0.8808 mmol) produced (*E*)-2,3-dimethyl-5-phenylhexa-1,4-diene in 28% yield. ^{1}H NMR (400 MHz, chloroform-d_1, r.t.): δ 1.05 (d, J = 6.9 Hz,

3H, 3-*Me*), 1.67 (s, 3H, 2-*Me*), 2.03 (d, J = 1.7 Hz, 1H, 4-C*H*), 2.81 (dq, J = 10.3, 6.9 Hz, 1H, 3-C*H*), 4.69 (s, 2H, 1-C*H*$_2$), 5.36 (dq, J = 10.3, 1.7 Hz, 1H, 4-C*H*), 7.18-7.40 (m, 5H, *Ph*). GC-MS (EI): m/z = 186 (M$^+$). (method B): The reaction of **1a** (13.3 mg, 0.0400 mmol) with methylphenylketene (13.5 µL, 0.100 mmol) in benzene-d_6 produced **5ac** in 60% yield.

Crystallographic study

The single crystals suitable for the X-ray analysis were obtained by the recrystallisation of [Ru(η^4-CH$_2$CMeCMeCHC(O)CHPh$_2$)(η^4-1,5-diene)(NCMe)] (**2aa**) from cold Et$_2$O. A selected crystal was mounted on the top of glass capillary by use of Paraton-N oil. A Rigaku AFC-7R Mercury II diffractometer with graphite monochromated MoKα radiation (λ = 0.71075 Å) was used for data collection at 200.0 K. The collected data were solved by direct methods (SIR92), and refined by a full-matric least square procedure using SHELXL-97.[25] All non-hydrogen atoms were refined with anisotoropic displacement parameters. The diphenylmethyl group in **2aa** was disordered and one of the isomer was depicted in Figure 1 and the physical and crystallographic parameters are listed in Table 1. Hydrogen atoms were placed using the riding model. The structure was depicted by ORTEP-III and POV-Ray for Windows. The crystallographic data for the structure in this paper have been deposited to the CCDC, 12 Union Road, Cambridge CB21EZ, UK. Copies of the data can be obtained free of charge on quoting the depository number CCDC-1005883 (**2aa**) (http://www.ccdc.cam.ac.uk).

Table 1 Physical and crystallographic data for **2aa**

Experimental formula	C$_{30}$H$_{35}$NORu
Formula weight (g mol^{-1})	526.68
λ (Å)	0.71075
Temperature (K)	200
Crystal System	Monoclinic
Space group	$P2bc$
a (Å)	22.436(6)
b (Å)	8.563(2)
c (Å)	13.950(4)
β (deg)	104.135(4)
Volume (Å^3)	2598.8(12)
Z	4
Density (g cm^{-3})	1.346
Absorption coefficient (mm^{-1})	0.625
$F(000)$	1096.00
Reflection corrected	5921
Independent reflections	4533
R(int)	0.0370
Parameters	280
Goodness-of-fit	1.070
Final R ($_w$R)	0.0549 (0.1513)

Acknowledgements

Notes and references

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