mm^{-3}; 1175 data measured to a 2θ max. of 115°; $R = 0.0412$, $wR2 = 0.1077$ for all 1079 unique reflections.

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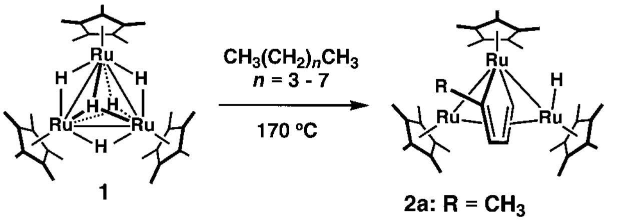
[20] X-ray intensity data for **4**–**7** were measured on a Bruker diffractometer with $Cu_{K\alpha}$ radiation ($\lambda = 1.54178$ Å) at $T = 293$ K. Structures were solved by direct methods, aided by program XS, and refined with the full-matrix least-squares program XL, from SHELXTL. (G. Sheldrick, SHELXTL96, *Acta Crystallogr. Sect. A* **1990**, *46*, 467). The XL program minimizes F^2 differences, and uses all data; that is, weak data are not considered "unobserved", and are included in the R-factors reported. Crystallographic data (excluding structural factors) for the structures reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-127760 (**4**), -127761 (**5**), -127762 (**6**), and -127763 (**7**). Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk)

Intermolecular Activation of *n*-Alkanes by a Trinuclear Ruthenium Pentahydride Complex—Formation of *closo*-Ruthenacyclopentadiene Complexes**

Akiko Inagaki, Toshifumi Takemori, Masako Tanaka, and Hiroharu Suzuki*

Activation of the carbon–hydrogen bonds of alkanes is of special importance because of its potential applicability to the functionalization of alkanes. The selective activation of alkane C-H bonds under mild conditions has, therefore, been one of the most challenging targets in recent organometallic chemistry and has been intensively studied using mononuclear hydride and carbonyl complexes as precursors to the active species.[1] Over the past ten years we have tackled the activation of organic substrates on multimetallic sites and demonstrated that a metal–polyhydride cluster effectively activates an organic substrate in a unique manner as a result of the cooperative action of the metal centers.[2] We report herein an unprecedented successive activation of alkane C-H bonds that is mediated by a trinuclear pentahydride complex of ruthenium $[\{(C_5Me_5)Ru\}_3(\mu\text{-}H)_3(\mu_3\text{-}H)_2]$ (**1**).

This novel reaction was discovered during the course of a study on the stability of **1** in solution at high temperature. We have already confirmed that complex **1** is thermally stable for 14 days at 200 °C in a sterically bulky solvent such as 1,3,5-trimethylcyclohexane, and does not decompose into mono- or bimetallic fragments. Heating a solution of **1** in hexane at 170 °C for 120 h resulted in the formation of a novel trinuclear *closo*-ruthenacyclopentadiene complex **2b** ($R = C_2H_5$) in a reasonable yield [Eq. (1)].



(1)

1

2a: R = CH_3
2b: R = CH_2CH_3
2c: R = $CH_2CH_2CH_3$
2d: R = $CH_2CH_2CH_2CH_3$
2e: R = $CH_2CH_2CH_2CH_2CH_3$

Complex **1** reacts with *n*-alkanes such as pentane, heptane, octane, and nonane in a similar manner and leads to the formation of **2a** ($R = CH_3$), **2c** ($R = CH_2CH_2CH_3$), **2d** ($R = CH_2CH_2CH_2CH_3$), and **2e** ($R = CH_2CH_2CH_2CH_2CH_3$), respectively, in good yields.[3] The structure of these compounds was inferred from spectroscopic data and then confirmed by an X-ray diffraction study on a single crystal of **2b** (Figure 1).

The most informative data regarding the structure of these complexes comes from the NMR study.[4] Whereas resonance signals for the triply bridging carbons in the ruthenacycle, C1 and C4, appear at $\delta = 118.0$ (d, $J_{CH} = 156$ Hz) and 144.5 (s), respectively, those for the doubly bridging carbon atoms, C2 and C3, are observed at the relatively high-field values of $\delta = 66.3$ (d, $J_{CH} = 178$ Hz) and 62.7 (d, $J_{CH} = 174$ Hz) in the ^{13}C NMR spectrum. The ^1H NMR spectrum of **2b** reveals, discounting the signals for the C_5Me_5 and R group, three resonances at $\delta = 4.35$ (d, $J = 4.4$ Hz, H1), 4.30 (dd, $J = 4.4$, 1.6 Hz, H2), and 3.88 (br, $w_{1/2} = 3.3$ Hz, H3) that are assignable to hydrogen atoms directly bonded to the ruthenacyclopentadiene framework. The ^1H-^{13}C COSY spectrum shows these ^1H resonances consistently correlate with the ^{13}C signals for the ring carbon atoms of the ruthenacyclopentadiene moiety. The signals of the C_5Me_5 groups appear to be inequivalent in both the ^1H and ^{13}C NMR spectra. The inequivalency of the chemical shifts of the resonances for the three C_5Me_5 groups suggests that a hydride ligand does not lie on the pseudosymmetry plane that is coplanar to the ruthenacycle. These resonances are characteristic of the *closo*-

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Supporting information for this article is available on the WWW under http://www.wiley-vch.de/home/angewandte/ or from the author.

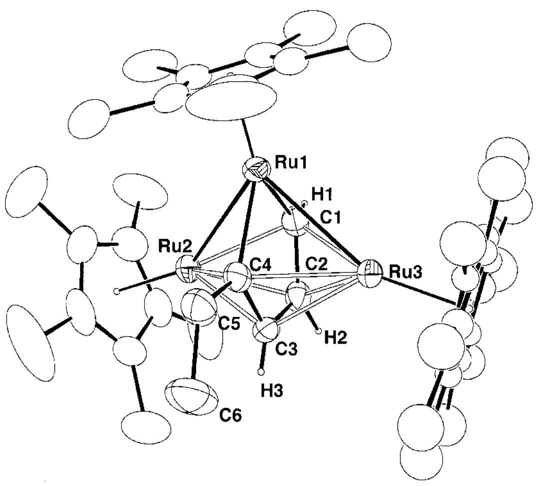


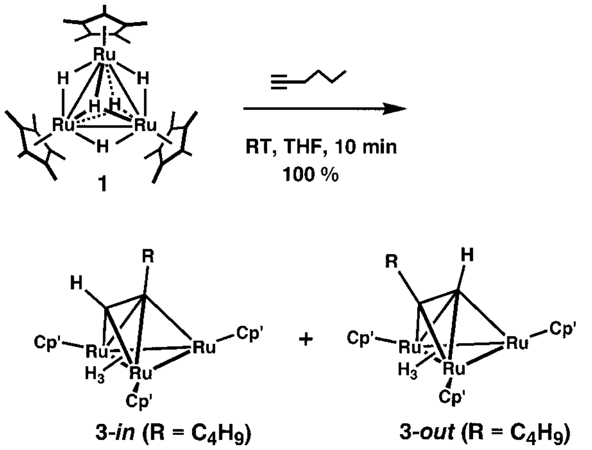
Figure 1. Molecular structure of **2b** (thermal ellipsoids at the 30% probability level). Selected bond lengths [Å] and angles [°]: Ru(1)-Ru(2) 2.7878(10), Ru(1)-Ru(3) 2.7741(10), Ru(1)-C(1) 2.062(9), Ru(1)-C(4) 2.057(7), Ru(2)-C(1) 2.249(8), Ru(2)-C(2) 2.148(7), Ru(2)-C(3) 2.135(7), Ru(2)-C(4) 2.283(7), Ru(3)-C(1) 2.349(8), Ru(3)-C(2) 2.169(8), Ru(3)-C(3) 2.198(7), Ru(3)-C(4) 2.408(7), C(1)-C(2) 1.442(11), C(2)-C(3) 1.449(12), C(3)-C(4) 1.455(11), C(4)-C(5) 1.514(12), C(5)-C(6) 1.492(12); Ru(2)-Ru(1)-Ru(3) 83.72(3), C(1)-Ru(1)-C(4) 78.9(3), Ru(2)-Ru(1)-C(1) 52.7(2), Ru(2)-Ru(1)-C(4) 53.7(2), Ru(3)-Ru(1)-C(1) 55.8(2), Ru(3)-Ru(1)-C(4) 57.5(2), Ru(1)-C(1)-C(2) 117.4(6), C(1)-C(2)-C(3) 112.5(7), C(2)-C(3)-C(4) 115.1(6), C(3)-C(4)-Ru(1) 115.7(6), C(3)-C(4)-C(5) 119.4(7), C(4)-C(5)-C(6) 120.1(9).

ruthenacyclopentadienes **2a**–**2e**. On the basis of the similarity of the ^{1}H and ^{13}C chemical shifts and the coupling pattern, complexes **2a**, **2c**, **2d**, and **2e** were unambiguously identified as the analogous *closo*-ruthenacyclopentadienes.

Definitive proof for the proposed structure was provided by an X-ray study on the single crystal of **2b**.[5] Figure 1 clearly establishes the structural identity of **2b** as a trinuclear *closo*-ruthenacyclopentadiene in which the ruthenacycle bisects the Ru2-Ru3 vector.[6] The Ru1-Ru2 and Ru1-Ru3 distances are almost identical and the values of 2.7878(10) and 2.7741(10) Å, respectively, lies within the range of those observed for a Ru-Ru single bond. The Ru-Ru bond lengths indicate that the hydride in **2b** is not a bridging one but a terminal one. The hydride is most likely to be located in a space between two C_5Me_5 ligands bound to Ru1 and Ru3, although the position of a hydrogen atom bonded to a ruthenium atom could not be determined by the difference Fourier synthesis. The Ru1-C1 and Ru1-C4 bond lengths of 2.062(9) and 2.057(7), respectively, correspond to the Ru-C σ-bond. The four-carbon fragment from C1 to C4 is π-bonded to Ru2. On the opposite side of the RuC_4 plane, Ru3 is π-bonded to C2 and C3. The values of 2.349(8) and 2.408(7) Å for Ru3-C1 and Ru3-C4, respectively, are significantly longer than that of the Ru-C π-bond.

It is clear that the carbon framework of 2-alkylruthenacyclopentadiene in **2** originates from the *n*-alkane used as the solvent. In this reaction, six C-H bonds of the alkanes are successively cleaved on the trimetallic site, and these results strongly suggest the applicability of transition metal cluster complexes, especially polyhydridometal clusters, to the functionalization of alkanes.

Signals for some of the intermediates were observed by monitoring the reaction by ^{1}H NMR spectroscopy, and these intermediates finally converged into thermodynamically stable products **2a**–**2e**. When the reaction of **1** with hexane was monitored at 170 °C by means of ^{1}H NMR spectroscopy an equilibrated mixture of μ_3-(⊥)-hexyne complexes, $\{(C_5Me_5)Ru\}_3(\mu\text{-H})_3\{\mu_3\text{-}(\perp)\text{-HCCC}_4H_9\}$ (**3**-*in*), and $\{(C_5Me_5)Ru\}_3(\mu\text{-H})_3\{\mu_3\text{-}(\perp)\text{-C}_4H_9\text{CCH}\}$ (**3**-*out*), were detected in a complex mixture of intermediates.[7] An attempt to isolate the equilibrated mixture of **3** (**3**-*in* + **3**-*out*) from the reaction mixture was unsuccessful, and so we could not get direct evidence that compound **3** was converted into **2b**. We, therefore, confirmed the intermediacy of **3** using an authentic complex independently synthesized by treatment of **1** with 1-hexyne [Eq. (2)].[8] We employed 1,3,5-trimethylcyclohexane as the solvent in the thermolysis of **3** since it was inactive toward the ruthenium clusters noted in this manuscript, probably as a result of its bulkiness. As anticipated, thermolysis of **3** in 1,3,5-trimethylcyclohexane at 170 °C led to the formation of **2b** [65% yield; Eq. (3)]. Thermolysis of the authentic μ_3-(⊥)- pentyne, μ_3-(⊥)-heptyne, and μ_3-(⊥)-octyne complexes $[\{(C_5Me_5)Ru\}_3(\mu\text{-H})_3\{\mu_3\text{-}(\perp)\text{-HCCR}\}]$ and $[\{(C_5Me_5)Ru\}_3(\mu\text{-H})_3\{\mu_3\text{-}(\perp)\text{-RCCH}\}]$ ($R = C_3H_7$, C_5H_{11}, C_6H_{13}), also led to the formation of **2** ($R = C_3H_7$: 67%, $R = C_5H_{11}$: 53%, $R = C_6H_{13}$: 73%).

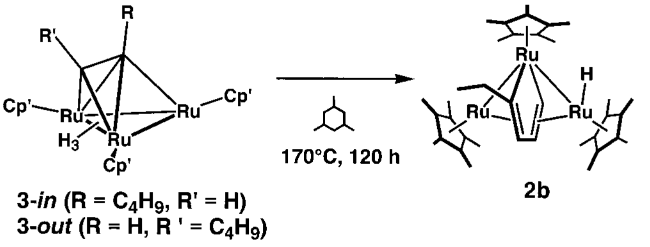


1

(2)

3-*in* (R = C_4H_9)

3-*out* (R = C_4H_9)



(3)

3-*in* (R = C_4H_9, R' = H)
3-*out* (R = H, R ' = C_4H_9)

2b

Although the reaction mechanism of successive C-H bond activation still remains unclear, we believe that alkane C-H bond activation on multimetallic sites is one of the promising approaches to the functionalization of alkanes.

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[3] Heptane (5 mL) and **1** (59.6 mg, 0.083 mmol) were added to a 50-mL glass autoclave. The reaction mixture was heated at 170 °C for 154 h, during which time the solution changed from reddish brown to purplish brown. Removal of the solvent under reduced pressure afforded crude product **2c** as a brown solid (ca. 70% purity based on ^{1}H NMR). The solid was dissolved in toluene and the product purified by column chromatography on neutral alumina. Removal of solvent under reduced pressure afforded 45.3 mg of **2c** as a brown solid (68% yield).

[4] **2b**: ^{1}H NMR (400 MHz, C_6D_6, 23 °C, TMS): $\delta = 4.35$ (d, $J = 4.4$ Hz, 1H; C1-H), 4.30 (dd, $J = 4.4$, 1.6 Hz, 1H; C2-H), 3.88 (brs, $w_{1/2} = 3.3$ Hz, 1H; C3-H), 2.23 (s, 15H; C_5Me_5), 1.85 (q, $J = 7.2$ Hz, 2H; C5-H), 1.82 (s, 15H; C_5Me_5), 1.65 (s, 15H; C_5Me_5), 0.71 (t, $J = 7.2$ Hz, 3H; C6-H), -10.50 (s, 1H; Ru-H); ^{13}C NMR (100 MHz, C_6D_6, 23 °C, TMS): $\delta = 144.5$ (s, C4), 118.0 (d, J(C,H) = 156 Hz; C1), 90.6 (s; C_5Me_5), 83.0 (s; C_5Me_5), 82.7 (s; C_5Me_5), 66.3 (d, J(C,H) = 178 Hz; C2), 62.7 (d, J(C,H) = 174 Hz; C2), 36.9 (t, J(C,H) = 125 Hz; C5), 19.2 (q, J(C,H) = 128 Hz; C6), 13.3 (q, J(C,H) = 125 Hz; C_5Me_5), 12.2 (q, J(C,H) = 125 Hz; C_5Me_5), 11.7 (q, J(C,H) = 126 Hz; C_5Me_5).

[5] X-ray structural determination of **2b:** Data were collected at 23 °C on a Rigaku AFC5R diffractometer with graphite monochromated $Mo_{K\alpha}$ radiation (ω scan $2\theta_{max} = 50.0°$). Laue symmetry revealed a monoclinic crystal system with the dimensions: $a = 38.477(8)$, $b = 8.635(2)$, $c = 20.712(4)$ Å, $\beta = 94.86(2)°$, and $V = 6856(2)$ Å^3. $\rho_{calcd} = 1.53$ g cm^{-3}, $Z = 8$, $M_r = 790.03$; space group $P2_1/n$ (No. 14). Of the 13326 reflections that were collected, 12946 were unique ($R_{int} = 0.048$). $\mu = 13.3$ cm^{-1}. Azimuthal scans of several reflections indicated no need for an absorption correction. The data were corrected for Lorentz and polarization effects. The structure was solved by the Patterson method (DIRDIF 94 PATTY) and non-hydrogen atoms other than disordered C_5Me_5 groups were refined on F^2 using the SHELXL-97 program systems. The unit cell contains two molecules and one of the C_5Me_5 groups in each molecule is thermally disordered (occupancy: 51.14:48.86 and 51.23:48.77, respectively). Hydrogen atoms H1–3 and H39–41 were located by sequential difference Fourier synthesis and the remaining hydrogen atoms were included in geometric positions. All hydrogen atoms and disordered carbon atoms of C_5Me_5 groups were refined with isotropic thermal parameters. The final structure for **2b** was refined to $R = 0.0545$ for 8447 observed reflections ($F_o > 4\sigma(F_o)$) and $wR_2 = 0.1615$ for 12006 data and 590 parameters. Crystallographic data for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-118781. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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[7] The reaction of **1** with *n*-hexane was monitored as follows: A 50-mL autoclave was charged with **1** (111.5 mg, 0.156 mmol) and *n*-hexane (10 mL) and the reaction mixture heated at 170 °C for 12 h. The reaction mixture was then cooled down to room temperature. After a portion (0.5 mL) of the reaction mixture had been evaporated, C_6D_6 (0.5 mL) was added to the residue and the ^{1}H NMR spectrum recorded. The rest of the reaction mixture was again heated at 170 °C. The ^{1}H NMR spectra of the reaction mixture were measured at intervals of 12 h.

[8] An equilibrium mixture of regioisomers **3**-*in* and **3**-*out* is obtained quantitatively in the reaction of **1** with 1-hexyne. **3**-*in*: ^{1}H NMR (400 MHz, [D$_8$]THF, -80 °C, TMS): $\delta = 10.22$ (s, 1H; Ru-CH), 1.854 (s, 30H; C_5Me_5), 1.845 (s, 15H; C_5Me_5), 1.38 (m, 2H; CH_2), 1.00 (m, 2H; CH_2), 0.74 (t, $J = 8.0$ Hz, 3H; CH_3), 0.40 (m, 2H; CH_2), -6.95 (brs, $w_{1/2} = 11.0$ Hz, 2H; Ru-H), -25.03 (t, $J = 3.2$ Hz, 1H; Ru-H); ^{13}C NMR (100 MHz, [D$_8$]THF, -80 °C, TMS): $\delta = 180.7$ (d, J(C, H) = 177 Hz; Ru-CH), 90.7 (s; C_5Me_5), 85.4 (s; C_5Me_5), 67.2 (s; Ru-C), 37.8 (t, J(C,H) = 125 Hz; CH_2), 34.9 (t, J(C,H) = 125 Hz; CH_2), 25.1 (t, J(C,H) = 124 Hz; CH_2), 16.2 (q, J(C,H) = 128 Hz; CH_3), 13.7 (q, J(C,H) = 125 Hz; C_5Me_5), 13.6 (q, J(C,H) = 125 Hz; C_5Me_5); **3**-*out*: ^{1}H NMR (400 MHz, [D$_8$]THF, -80 °C, TMS): $\delta = 3.84$ (m, 2H; CH_2), 1.89 (s, 15H; C_5Me_5), 1.77 (s, 30H; C_5Me_5), 1.63 (m, 2H; CH_2), 1.62 (m, 2H; CH_2), 1.00 (t, $J = 6.2$ Hz, 3H; CH_3), -0.38 (brs, $w_{1/2} = 7.0$ Hz, 1H; Ru-CH), -11.78 (d, $J = 4.4$ Hz, 2H; Ru-H), -25.96 (dt, $J = 4.4$, 3.6 Hz, 1H; Ru-H); ^{13}C NMR (100 MHz, [D$_8$]THF, -80 °C, TMS): $\delta = 188.7$ (s, Ru-C), 91.9 (s; C_5Me_5), 85.8 (s; C_5Me_5), 64.6 (d, J(C, H) = 156 Hz; Ru-CH), 43.6 (t, J(C,H) = 125 Hz; CH_2), 37.0 (t, J(C,H) = 122 Hz; CH_2), 25.8 (CH_2; confirmed by ^{13}C-^{1}H HSC and ^{1}H-^{1}H COSY spectra), 16.2 (q, J(C,H) = 128 Hz; CH_3), 14.7 (q, J(C,H) = 125 Hz; C_5Me_5), 13.0 (q, J(C,H) = 126 Hz; C_5Me_5).

Two New Carbaalanes: Compounds with an Al_7C_5 and a $[B_{11}H_{11}]^{2-}$ Analogous Al_7C_4 Cluster**

Werner Uhl,* Frank Breher, Arne Lützen, and Wolfgang Saak

Some polyhedral borane or carbaborane cluster compounds are known from literature, which contain single aluminium atoms located on positions of high connectivity.[1]

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