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A novel ruthenium carbene dimer that is active in the metathesis of internal alkenes; the crystal structure of $Ru_2(=CHPh)_2(CF_3CO_2)_2(\mu\text{-}CF_3CO_2)_2(PCy_3)_2(\mu\text{-}H_2O)$

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Abstract

The reaction of $Ru(=CHPh)Cl_2(PCy_3)_2$ (**1**) with two equivalents of CF_3CO_2Ag gives $Ru_2(=CHPh)_2(CF_3CO_2)_2$-$(\mu\text{-}CF_3CO_2)_2(PCy_3)_2(\mu\text{-}H_2O)$ (**2**) in high yield. The crystal structure of **2** reveals that the ruthenium atoms are bridged by two trifluoroacetate groups and one water molecule, and that the ruthenium coordination is distorted octahedral. Complex **2** is active in the metathesis of internal alkenes. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Ruthenium; Carbene complex; Metathesis; Methyl oleate; *Trans*-4-decene

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1. Introduction

The development of well-defined ruthenium carbene complexes of the general formula $Ru(=CHR)Cl_2(PR'_3)_2$ has opened up new prospects for the alkene metathesis reaction [1]. The most active among them, $Ru(=CHPh)Cl_2(PCy_3)_2$ (**1**), is now widely used as a catalyst in organic syntheses, in particular for ring-closing metathesis reactions [2,3]. However, due to certain limitations of **1**, such as its moderate stability to oxygen and high temperatures and its lack of stereoselectivity, there have been considerable efforts to develop the next generation of metathesis catalysts [4–7].

Our research focuses on the preparation of analogues of **1** that are suitable for immobilisation on a solid support. Since immobilisation of $Ru(=CH\text{–}CH=CPh_2)$-$Cl_2(PPh_3)_2$ on phosphine-functionalized polystyrenes was only moderately successful [8], we assumed that introduction of carboxylic groups around the Ru(II) centre would be more useful for this purpose. Therefore, we investigated the reaction of **1** with CF_3CO_2Ag, which is well known to abstract halogens from transition metal complexes.

2. Experimental

All manipulations were performed under an atmosphere of purified nitrogen using standard Schlenk tube techniques. All solvents were purified by standard procedures (hexane and THF were distilled from sodium benzophenone ketyl). Compound **1** was prepared according to Ref. [9]. CF_3CO_2Ag (Merck) was used as received; alkenes (Fluka) were purified by passing through activated alumina and distillation. NMR spectra were measured with a Bruker AMX-300 spectrometer (at room temperature (r.t.), unless stated otherwise). The mass spectrum was measured with a Jeol JMS SX/SX102A mass spectrometer.

2.1. *Synthesis of $Ru_2(=CHPh)_2(CF_3CO_2)_2(\mu\text{-}CF_3CO_2)_2$-$(PCy_3)_2(\mu\text{-}H_2O)$ (**2**)*

A solution of 58.8 mg of CF_3CO_2Ag (0.266 mmol) in 4 ml of THF was added to a solution of **1** (109.6 mg,

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0.133 mmol) in 80 ml of hexane at 0°C in 15 min. Formation of a white precipitate and a change of colour from purple to light-green followed. The mixture was stirred for another 30 min at 0°C and then it was filtered. The green filtrate was evaporated to dryness to yield a green solid (92.4 mg, 98%). Crystalline **2** was obtained from hexane at -22°C (73.9 mg, 76%). M.p. 130°C (dec.). ^{1}H-NMR (CD_2Cl_2): δ (ppm) 20.68 (d, $^3J_{PH} = 5.7$ Hz, 2H, Ru=CH), 11.75 (s, 2H, H_2O), 8.15 (d, $^3J_{HH} = 7.5$ Hz, 4H, *o*-H of Ph), 7.76 (t, $^3J_{HH} = 7.5$ Hz, 2H, *p*-H of Ph), 7.44 (t, $^3J_{HH} = 7.8$ Hz, 4H, *m*-H of Ph), 2.03–0.84 (m, 74H, PCy_3 and C_6H_{14}). ^{13}C-NMR (CD_2Cl_2, -20°C): δ (ppm) 327.1 (d, $^2J_{PC} = 16$ Hz, Ru=CH), 172.7 (q, $^2J_{CF} = 38$ Hz, $\mathit{C}O_2CF_3$), 169.2 (q, $^2J_{CF} = 37$ Hz, $\mathit{C}O_2CF_3$), 158.5 (s, *ipso*-C of Ph), 134.7, 133.4, 133.3 (all s, Ph), 116.6 (q, $^1J_{CF} = 283$ Hz, $CO_2\mathit{C}F_3$), 35.04 (s, *ipso*-C of PCy_3), 30.85 (d, $J_{PC} = 9.8$ Hz), 29.31 (bs), 26.12 (s), all PCy_3. ^{31}P-NMR (CD_2Cl_2): δ (ppm) 43.97 (s). ^{19}F-NMR (CD_2Cl_2): δ (ppm) -75.38 (s), -76.14 (s). IR (KBr): (cm^{-1}) ν(CH) 2932, 2854; ν_{asym}(COO) 1690, 1656; ν_{sym}(COO) 1448; 1266, 1202, 1147, $\nu(CF_3)$ 726. MALDI-TOF MS: *m*/*z* (rel. intensity) 1168 ($[Ru_2(=CHPh)_2(CF_3CO_2)_2(PCy_3)_2]^+$, 9%), 1055 ($[Ru_2(=CHPh)_2(CF_3CO_2)(PCy_3)_2]^+$, 7%), 667 (100%), 371 (61%), 281 ($[HPCy_3]^+$, 100%). Anal. Calc. for $C_{58}H_{80}F_{12}O_9P_2Ru_2 \cdot 0.5C_6H_{14}$: C, 50.3; H, 6.02%. Found: C, 50.4; H, 6.11%.

The solid remaining on the filter was washed with CH_2Cl_2 (5 × 2 ml) to produce a pale-yellow solution and an insoluble residue (AgCl). An off-white solid was obtained after solvent removal. It was purified by crystallisation from CH_2Cl_2–hexane and identified as $AgClPCy_3$ (46.9 mg, 84% yield). ^{1}H-NMR ($CDCl_3$): δ (ppm) 2.0–1.6 (m, 18H), 1.5–1.1 (m, 15H). ^{13}C-NMR ($CDCl_3$): δ (ppm) 31.60 (2d, $J_{PC} = 17$ Hz), 31.02 (s), 26.88 (d, $J_{PC} = 12$ Hz), 25.57 (s). ^{31}P-NMR (CD_2Cl_2): δ (ppm) 43.28 (2d, $^1J_{AgP} = 599$ and 691 Hz). Anal. Calc. for $C_{18}H_{33}AgClP$: C, 51.0; H, 7.85%. Found: C, 50.6; H, 7.65%. Molecular weight determination in CH_2Cl_2 showed the complex to be monomeric.

2.2. X-ray structure determination

Crystals suitable for X-ray study were grown from *n*-hexane at -22°C. Crystal data and details of refinement are shown in Table 1.

2.3. Metathesis experiments

The glassware used in these experiments was dried for several hours at 120°C and cooled to the reaction temperature under nitrogen. In a typical experiment, *trans*-4-decene (0.15 ml, 0.79 mmol) was added to a solution of complex **2** (2.1 mg, 1.44×10^{-3} mmol) in an appropriate solvent (0.48 ml). The resulting solution was vigorously stirred. The progress of the metathesis reaction was monitored by sampling through a septum at suitable time intervals. The catalyst in these samples was immediately quenched with an excess of ethyl vinyl ether[1]. The samples were analysed by GC (Carlo Erba 8000^{Top}) on a DB-5 (J&W Scientific) column. *Trans*/*cis* ratios were measured on a CP-Sil 88 column (Chrompack).

3. Results and discussion

3.1. Synthesis

Grubbs et al. [11] substituted chlorine ligands in $Ru(=CH{-}CH{=}CPh_2)Cl_2(PPh_3)_2$ with trifluoroacetate groups by reacting a CH_2Cl_2 solution of this complex with a THF solution of CF_3CO_2Ag. However, when we applied the same procedure to complex **1** complete decomposition of the carbene ligand resulted. Therefore, we turned our attention to a solvent in which **1** shows the lowest catalytic activity for metathesis and is more stable, viz. hexane [12].

Table 1
Crystal data and structure refinement for **2**

Formula	$C_{58}H_{80}F_{12}O_9P_2Ru_2 \cdot 0.5C_6H_{14}$ [a]
M_r	1456.39
Temperature (K)	150(2)
Diffractometer	Nonius KappaCCD
Radiation	Mo–K$_\alpha$ ($\lambda = 0.71073$ Å)
Crystal system	Triclinic
Space group	$P\bar{1}$
a (Å)	12.2484(5)
b (Å)	16.9499(9)
c (Å)	17.2405(8)
α (°)	97.750(2)
β (°)	96.165(3)
γ (°)	96.146(3)
V (Å^3)	3499.1(3)
Z	2
ρ (g cm^{-3})	1.382
μ (mm^{-1})	0.558
Crystal size (mm^3)	0.20 × 0.20 × 0.08
$(\sin \nu/\lambda)_{max}$ (Å^{-1})	0.595
Reflections collected/unique	15002/11071 [$R_{int} = 0.0348$]
Parameters/restraints	778/61
S	1.035
Final *R* indices [$I > 2\sigma(I)$]	$R_1 = 0.0505$, $wR_2 = 0.1262$
R indices (all data)	$R_1 = 0.0670$, $wR_2 = 0.1369$
Structure solution	SIR-97 [13]
Structure refinement	SHELXL-97 [14] (refinement on F^2)
Structure drawings and checking	PLATON [15]

[a] The positions of the hexane solvent molecules are only partially occupied.

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[1] $Ru(=CH{-}CH{=}CPh_2)X_2(PR_3)_2$ (X = Cl, CF_3CO_2, R = Ph, Cy) reacts with ethyl vinyl ether to yield metathesis-inactive ethoxy-substituted carbene species [9,10].

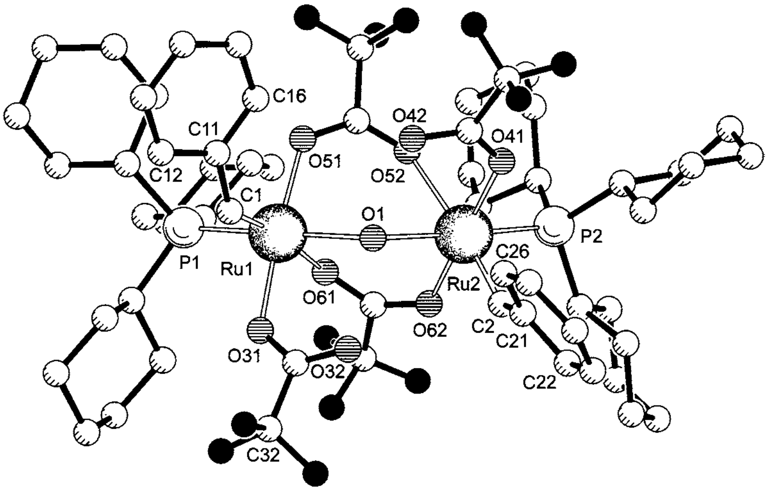

Fig. 1. Molecular plot of **2**. Hydrogen atoms and solvent molecules are omitted for clarity. Only one conformation of the disordered CF_3 group at C(32) is shown.

The reaction of **1** with CF_3CO_2Ag in hexane proceeded within minutes and yielded a green solid after a simple work-up. The ^{31}P-NMR spectrum of this product showed only one peak at 43.97 ppm. The presence of a carbene proton in the new compound was clearly established by ^{1}H-NMR as a doublet at 20.68 ppm. Integration of cyclohexyl protons versus the carbene proton showed that the reaction took a somewhat unexpected course as only one tricyclohexylphosphine ligand remained in the product[2]. The presence of two different trifluoroacetate groups was also concluded from the spectroscopic data (^{13}C and ^{19}F-NMR, IR). However, it was not possible to determine the mode of coordination of these groups. Therefore, we undertook an X-ray structure determination of our product.

3.2. X-ray structure

A perspective view of the molecule together with the atom numbering scheme is shown in Fig. 1. Selected bond lengths and bond angles are listed in Table 2.

The two carbene units are in *cis* position to each other with the phosphine ligands located on the other side of the molecule. The two Ru=C bond lengths are identical (1.859(5) and 1.858(5) Å) and comparable to those of Ru(=CH-*p*-$C_6H_4Cl)Cl_2(PCy_3)_2$ (1.839(3) Å) [9] and [Ru(=CHPh)Tp(PCy_3)(H_2O)][BF_4] (1.878(4) Å) [7] (Tp = tris(pyrazolyl)borate). The two Ru–P distances are slightly different (2.3625(13) and 2.3345(14) Å) and significantly shorter than the shorter Ru–P bond in Ru(=CH-*p*-$C_6H_4Cl)Cl_2(PCy_3)_2$ (2.397(1) Å). In contrast to **1**, where the carbene ligand is perpendicular to the P–Ru–P plane, the corresponding angles between carbene ligands and P–Ru–C(carbene) planes (i.e. C1–Ru1–P1/C11–C12–C16 and C2–Ru2–P2/C21–C22–C26) are 68 and 55°, respectively. Accordingly, the carbene protons of **2** are observed as a doublet with $^3J_{PH} = 5.7$ Hz in the NMR spectrum.

The bridging unit is similar to those of closely related ruthenium dimers [16–18]. The water molecule is coordinated in the *cis* position relative to both carbene ligands. It is connected by two strong hydrogen bonds with non-coordinated oxygen atoms of monodentate trifluoroacetate groups (O···O distances of 2.546(5) and 2.510(5) Å). In agreement with the solid-state structure

Table 2
Selected bond lengths (Å) and angles (°) for **2** with estimated S.D. in parentheses

Bond lengths (Å)			
Ru(1)–C(1)	1.859(5)	Ru(2)–C(2)	1.858(5)
Ru(1)–P(1)	2.3625(13)	Ru(2)–P(2)	2.3345(14)
Ru(1)–O(1)	2.183(3)	Ru(2)–O(1)	2.221(3)
Ru(1)–O(31)	2.113(3)	Ru(2)–O(41)	2.099(3)
Ru(1)–O(51)	2.075(3)	Ru(2)–O(52)	2.272(3)
Ru(1)–O(61)	2.228(4)	Ru(2)–O(62)	2.080(3)
Bond angles (°)			
C(1)–Ru(1)–P(1)	89.90(16)	C(2)–Ru(2)–P(2)	89.84(16)
C(1)–Ru(1)–O(1)	98.58(18)	C(2)–Ru(2)–O(1)	94.35(18)
P(1)–Ru(1)–O(1)	171.27(10)	P(2)–Ru(2)–O(1)	175.22(10)
C(1)–Ru(1)–O(31)	86.70(19)	C(2)–Ru(2)–O(41)	97.22(19)
C(1)–Ru(1)–O(51)	94.90(19)	C(2)–Ru(2)–O(52)	172.57(18)
C(1)–Ru(1)–O(61)	171.39(19)	C(2)–Ru(2)–O(62)	84.71(18)
O(31)–Ru(1)–O(51)	175.29(13)	O(41)–Ru(2)–O(62)	176.35(14)
O(61)–Ru(1)–O(51)	92.62(14)	O(62)–Ru(2)–O(52)	96.01(13)
Ru(1)–O(1)–Ru(2)	122.47(16)		

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[2] The other PCy_3 ligand was isolated and characterised as a 1:1 complex with AgCl (see Experimental).

the protons of the aqua ligand are found at 11.75 ppm in a CD_2Cl_2 solution.

As for the source of this water molecule, we first analysed the commercial CF_3CO_2Ag by weight loss on prolonged drying as well as by determination of silver content by direct titration. Both measurements showed that amounts of water in this solid were one order of magnitude too low to account for the formation of **2**. Therefore, we attribute the presence of water in **2** to the residual water in hexane used as a solvent for its preparation, which was determined at 40 ppm by coulometric Karl–Fischer titration (20 ppm of water is required to form **2**).

In common with other adducts of $Ru(II)_2$ carboxylates [19], the mass spectrum of **2** does not contain the molecular peak. The peak with the highest mass (at m/z 1168 with the molecular pattern consistent with the presence of two Ru atoms) corresponds to a fragment derived from **2** by abstraction of the water molecule and two (presumably monodentate) trifluoroacetate groups. The next observed fragment is formed by abstraction of another trifluoroacetate group. Further fragmentation pathways are not clear at the moment.

Compound **2** is well soluble in common organic solvents. The molecular weight determination of **2** in CH_2Cl_2 and C_6H_6 proves that the compound is also dimeric in solution. We have not seen any decomposition of **2** in hexane; it decomposes, however, within several hours in CH_2Cl_2 at r.t. Solid samples of complex **2** are handled in air and show no signs of decomposition on a bench-top for weeks.

3.3. Catalytic activity

We tested **2** for its metathesis activity with internal alkenes, e.g. methyl oleate (Eq. (1)).

$$2CH_3(CH_2)_7CH{=}CH(CH_2)_7CO_2CH_3 \overset{\mathbf{2}}{\rightleftharpoons} CH_3(CH_2)_7CH{=}CH(CH_2)_7CH_3 + CH_3CO_2(CH_2)_7CH{=}CH(CH_2)_7CO_2CH_3 \quad (1)$$

We first checked the activity of **2** in different solvents at r.t. It appeared that **2** was the most active in CH_2Cl_2; conversion data in CH_2Cl_2 are given in Table 3. The activity of **2** was significantly lower in 1,2-dichlorobenzene, THF and ethyl acetate, while almost no activity was observed in Et_2O and hexane.

The activity of **2** in the metathesis of methyl elaidate or *trans*-4-decene was nearly the same (Table 3, entries 1 and 4). This means that, similar to **1**, the Ru(II) centre in **2** is not reactive towards the ester functionality. The activity of **2** increased greatly when the temperature was raised to 40°C (Table 3, entry 3). The catalyst reacted significantly faster with a *cis*-substrate than with a *trans*-substrate (Table 3, entries 2 and 4). The conversions obtained with **2** are somewhat lower than with **1** under the same conditions[3]. Within the reaction times shown in Table 3 the selectivity for the primary metathesis products was very high as only traces of other products (< 0.1%) were detected by GC.

Table 3
Activity of **2** for the metathesis of internal alkenes [a]

Entry	Substrate	Temperature (°C)	Time (h)	Conversion (%)
1	*Trans*-4-decene	20	4	24
2	Methyl oleate [b]	20	4	36
3	Methyl oleate	40	1	41
4	Methyl elaidate [c]	20	4	20

[a] Reaction conditions: 3 mM solutions in CH_2Cl_2, molar ratio substrate:**2** = 550:1.

[b] Methyl *cis*-9-octadecenoate.

[c] Methyl *trans*-9-octadecenoate.

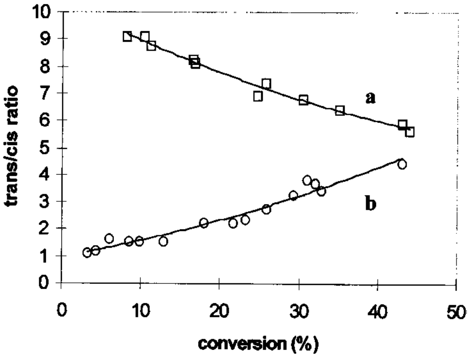

Fig. 2. *Trans/cis* composition of alkenes in the metathesis using complex **2** as a catalyst: (**a**) dimethyl 9-octadecene-1,18-dioate formed from methyl oleate; (**b**) 6-dodecene formed from *trans*-4-decene. All runs performed at 20°C with 3 mM solutions of **2** in CH_2Cl_2 and a substrate:**2** molar ratio of 550:1.

We observed an interesting stereoselectivity of **2** for the formation of carbon–carbon double bonds. Initially, both metathesis products have predominantly the opposite configuration of their double bond than has the substrate. This is illustrated by analysis of the *trans/cis* composition of the two compounds depicted in Fig. 2. In the case of a *cis*-substrate (methyl oleate) the product dimethyl 9-octadecene-1,18-dioate (Fig. 2, curve **a**) is formed mostly as the *trans*-isomer. For the metathesis of *trans*-4-decene equimolar amounts of *cis*- and *trans*-6-octadecene were detected at 2% conversion (Fig. 2, curve **b**). The alkenes approached their *trans/cis*

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[3] The conversions after 4 h using **1** as a catalyst under the same reaction conditions as in Table 3 were 36 and 40% with *trans*-4-decene and with methyl oleate, respectively (see Ref. [12]).

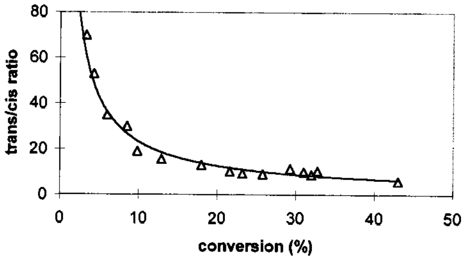

Fig. 3. *Trans*/*cis* isomerization of 4-decene during its metathesis using complex **2** as a catalyst (at 20°C, 3 mM solutions of **2** in CH_2Cl_2, *trans*-4-decene:**2** molar ratio of 550:1).

thermodynamic equilibrium value on prolonged reaction times due to secondary reactions (i.e. *trans*/*cis* isomerization via metathesis). At the same time the substrates were isomerized towards their *trans*/*cis* thermodynamic composition, as shown in Fig. 3 for 4-decene.

The effect of the reaction conditions (solvent, substrate and temperature) on the metathesis activity of **2** closely resembles that of **1**. Therefore, we assume for the mechanism of its catalytic activity the existence of analogous species as shown for **1**, i.e. with one phosphine bonded to the Ru(II) centre [20]. The enhanced stereoselectivity of **2** compared to that of **1**[4] suggests that these species are more sterically crowded and most likely dimeric. The detailed study of the crystal structure of **2** points to the bridging water molecule as the most likely ligand to be cleaved during the initiation step. This proposal is supported by our observation that the catalytic activity of **2** is enhanced when the presence of traces of water is avoided. When the glassware used for the metathesis experiments was left on a bench-top for several hours before the experiment was performed, the conversion after 4 h with *trans*-4-decene reached only 18%.

4. Conclusions

We have selectively converted $Ru(=CHPh)Cl_2(PCy_3)_2$ (**1**) into a novel carbene complex $Ru_2(=CHPh)_2(CF_3CO_2)_2(\mu\text{-}CF_3CO_2)_2(PCy_3)_2(\mu\text{-}H_2O)$ (**2**), which is active in the metathesis of internal alkenes. The new catalyst displays increased product stereoselectivity compared to **1**. The synthetic method applied for the preparation of **2** provides possibilities for introducing a large variety of ligands around the Ru(II) centre.

5. Supplementary material

Crystallographic data (excluding structure factors) for the structure in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 118783. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (fax: +44-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

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[4] The corresponding *trans*/*cis* ratios using **1** as a catalyst were, for instance, 4.9 at 7% conversion for dimethyl 9-octadecene-1,18-dioate and 3.8 at 3% conversion for 6-dodecene, respectively. For more data on *trans*/*cis* composition of alkenes in the metathesis using **1** see Ref. [12].

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