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Carbon–hydrogen bond activation of chloroalkanes by a rhodium trispyrazolylborate complex

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Abstract

The trispyrazolylborate complex fragment [Tp′Rh(CNneopentyl)], generated photochemically from the carbodiimide complex, reacts with chloro-substituted alkanes to give primary C–H oxidative addition products. Little reactivity is seen of the carbon–chlorine bond except when chlorine is in the β-position of the alkyl group.
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Keywords: Trispyrazolylborate; C–H activation; Rhodium

1. Introduction

The activation of C–H bonds of alkanes and arenes by coordinatively unsaturated metal complexes is a well-studied area of organometallic chemistry [1]. A number of complexes shown to be successful in alkane activation are Cp or Cp* metal complexes [2–4]. More recently, however, the analogous trispyrazolylborate complexes [5] have been shown to also activate a variety of C–H bonds in aliphatic and aromatic hydrocarbons [6–11]. Despite these many investigations, there are only a few examples that look at the activation of hydrocarbons that already contain reactive functional groups ([12], for examples of C–H activation in aryl halides, see [13]), perhaps because of a belief that the functional group would prove more reactive than the C–H bond. In this report, the reactions of a number of monochloroalkanes with the reactive fragment [Tp′Rh(CNneopentyl)] are investigated.

2. Experimental

2.1. General

All operations were performed under a nitrogen atmosphere, either in a Vacuum Atmospheres Corporation Glove Box or on a high vacuum line using modified Schlenk techniques. Benzene-d_6 was purchased from the Cambridge Isotope Labs and distilled under vacuum from dark purple solutions of benzophenone ketyl and stored in ampules with Teflon-sealed vacuum line adaptors. 1-Chloropentane, 2-chloropentane, and 2-chloropropane were purchased from Aldrich Chemical Co., distilled and dried over 4A molecular sieves. 3-chloropentane was prepared according to a published procedure [14]. Preparation of Tp′Rh(L)(η^2-PhN=C=NCH$_2$C(CH$_3$)$_3$) (**1**), Tp′Rh(L)Cl$_2$ (**2**), and neopentylisocyanide (L), have been previously reported [15–17]. All NMR spectra were recorded on a Bruker AMX400 or Bruker AVANCE 400 spectrometer. Chemical shifts are reported in ppm relative to tetramethylsilane and referenced using chemical shifts of residual solvent resonances (C_6D_5H, δ 7.15).

2.2. Preparation of Tp′Rh(L)HCl (3)

5.7 mg (0.010 mmol) of Tp′Rh(CNCH$_2$C(CH$_3$)$_3$)Cl$_2$ was dissolved in 0.75 ml of C_6D_6. To this solution was added 1.7 mg (0.0076 mmol) of Cp_2ZrH_2 in increments of 3 equal amounts with stirring for 20 min in between additions. Intermediate ^{1}H NMR spectra showed the conversion of **2** into **3**. The reaction mixture was stirred an additional 30 min, after which a ^{1}H NMR spectrum showed the formation of **3**. For **3**, ^{1}H NMR

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(C_6D_6): δ Rh–H: −13.40 (d, $J = 11.5$ Hz, 1H); Tp′Me: 2.074 (s, 3H), 2.120 (s, 3H), 2.207 (s, 3H), 2.286 (s, 3H), 2.862 (s, 3H), 2.869 (s, 3H); TpCH: 5.499 (s, 2H), 5.803 (s, 1H); CNR: 0.655 (s, 9H), 2.554 (s, 2H). For Tp′Rh(L)Cl_2 (**2**), ^{1}H NMR (C_6D_6): 2.033 (s, 6H), 2.106 (s, 3H), 2.735 (s, 6H), 3.241 (s, 3H), 5.491 (s, 2H), 5.522 (s, 1H), 2.688 (s, 2H), 0.793 (s, 9H).

2.3. Activation of 1-chloropentane: Tp′Rh(L)(H)($CH_2CH_2CH_2CH_2CH_2Cl$) (*4*)

A bright yellow solution of 9.7 mg of **1** in 0.75 ml of 1-chloropentane was irradiated with $\lambda > 345$ nm for 20 min at −20 °C. The solution changes to a tan color upon photolysis. The volatiles were removed under vacuum at 0 °C, leaving a brown oily solid. The resulting solid was dissolved in C_6D_6 and a ^{1}H NMR spectrum was taken immediately showing a single C–H bond activated product, **4**. ^{1}H NMR (C_6D_6): δ Rh–H: −14.92 (d, $J = 24.5$ Hz, 1H); Tp′Me: 2.197 (s, 3H), 2.211 (s, 3H), 2.295 (s, 3H), 2.374 (s, 3H), 2.544 (s, 3H), 2.566 (s, 3H); Tp′CH: 5.656 (s, 1H), 5.661 (s, 1H), 5.843 (s, 1H); CNR: 0.653 (s, 9H), 2.647 (s, 2H); CH_2Cl: δ 3.219 (t, $J = 6.8$ Hz, CH_2Cl). The remaining CH_2 resonances for the alkyl group were highly split and obscured, δ 1.0–2.3.

2.4. Activation of 3-chloropentane: Tp′Rh(L)(H)($CH_2CH_2CHClCH_2CH_3$) (*5*)

The activation of 3-chloropentane was identical to that of 1-chloropentane except that 7.1 mg of **1** was dissolved in 0.75 ml of 3-chloropentane. The ^{1}H NMR spectrum in C_6D_6 shows evidence for two products assigned as diastereomers in a 1:1 ratio. For Tp′Rh(L) (H)(CH_2CH_2-$CHClCH_2CH_3$) (**5**) (two diastereomers present), ^{1}H NMR (C_6D_6): δ Rh–H: 14.92 (d, $J = 24$ Hz, 2H); Tp′Me: 2.184 (s, 3H), 2.197 (s, 6H), 2.203 (s, 3H), 2.282 (s, 3H), 2.287 (s, 3H), 2.364 (s, 6H), 2.519 (s, 3H), 2.532 (s, 3H), 2.606 (s, 6H); Tp′CH: 5.626 (s, 1H), 5.645 (s, 1H), 5.654 (s, 2H), 5.821 (s, 1H), 5.839 (s, 1H); CNR: 0.652 (s, 9H), 0.667 (s, 9H), 2.638 (s, 2H), 2.651 (s, 2H); CH_2CH_3: 1.010 (t, $J = 7.3$ Hz, 3H), 1.017 (t, $J = 7.3$ Hz, 3H); CHCl: 3.93 (m, 1H), 4.00 (m, 1H); the remaining CH_2 resonances for the alkyl group were highly split and obscured.

2.5. Activation of 2-chloropentane: Tp′Rh(L)(H)($CH_2CH_2CH_2CHClCH_3$) (*6*) and Tp′Rh(L)HCl (*3*)

The procedure was identical to that for activation of 1-chloropentane except that 10.5 mg of **1** was dissolved in 0.75 ml of 2-chloropentane and irradiated at −20 °C. The volatiles were removed under vacuum, and examination by GC showed the presence of 1-pentene. The non-volatiles were dissolved in C_6D_6. Both Tp′Rh(L) HCl (**3**) and a CH activated product **6** were observed by ^{1}H NMR spectroscopy in a 1:2.5 ratio. The C–H activation product **6** was assigned as the terminal 4-chloropentyl derivative (two diastereomers present). For **6** (two diastereomers present), ^{1}H NMR (C_6D_6): δ Rh–H: 14.915 (d, $J = 24.8$ Hz, 1H), −14.909 (d, $J = 24.8$ Hz, 1H), Tp′Me: 2.194 (s, 3H), 2.201 (s, 3H), 2.208 (s, 6H), 2.286 (s, 3H), 2.293 (s, 3H), 2.378 (s, 6H), 2.537 (s, 3H), 2.560 (s, 3H), 2.578 (s, 3H), 2.611 (s, 3H); Tp′CH: 5.659 (s, 4H), 5.835 (s, 1H), 5.845 (s, 1H); CNR: 2.691 (s, 2H), 2.731 (s, 2H), 0.677 (s, 9H), 0.680 (s, 9H); $CHClCH_3$: 1.082 (d, $J = 6.1$ Hz, 3H), 1.031 (d, $J = 6.1$ Hz, 3H), 3.98 (m, 1H), 4.06 (m, 1H); the remaining CH_2 resonances for the alkyl group were highly split and obscured.

2.6. Activation of 2-chloropropane

The photolysis of 2-chloropropane was identical to that of 1-chloropentane except that 5.1 mg of **1** was dissolved in 0.75 ml of 2-chloropropane and irradiated at −20 °C for 20 min. Hydridochloride **3** was observed as well as the dichloride **2** in roughly a 3:1 ratio.

2.7. Activation of 1,1,1-trichloroethane

The photolysis of 1,1,1-trichloroethane was identical to that of 1-chloropentane except that 4.9 mg of **1** was dissolved in 0.75 ml of 1,1,1-trichloroethane and irradiated at −20 °C for 20 min. The only product observed was the dichloride **2**. A control experiment demonstrated that hydridochloride **3** reacts rapidly with 1,1,1-trichloroethane to give **2**.

2.8. Activation and quench of 1-chloropentane: Tp′Rh(L)(Cl)($CH_2CH_2CH_2CH_2CH_2Cl$) (*4-Cl*)

A bright yellow solution of 10 mg of **1** was dissolved in 0.75 ml of 1-chloropentane. The solution was cooled to −20 °C and photolyzed for 20 min. The resulting tan solution was quenched in vacuo with CCl_4 and kept at −20 °C for 2 h. Solvent was removed yielding a yellow-brown solid. A ^{1}H NMR spectrum shows Tp′Rh(L)(Cl)($CH_2CH_2CH_2CH_2CH_2Cl$) (**4-Cl**) and **2** in a 4:1 ratio, respectively. For **4-Cl**, ^{1}H NMR (C_6D_6): δ Tp′Me: 2.092 (s, 3H), 2.156 (s, 3H), 2.220 (s, 3H), 2.345 (s, 3H), 2.775 (s, 3H), 2.949 (s, 3H); Tp′CH: 5.574 (s, 1H,), 5.627 (s, 1H), 5.704 (s, 1H); CNR: 0.712 (s, 9H), 2.632 ('d' of AB quartet, $J = 3.8$ Hz, 2H); $RhCH_2$: 3.34 (m, 1H), 3.16 (m, 1H); CH_2Cl: 3.157 (t, $J = 6.8$ Hz, 2H), the three remaining CH_2 resonances for the alkyl group were highly split and obscured, δ 1.3–2.0.

2.9. Activation and quench of 3-chloropentane: Tp′Rh(L)(Cl)($CH_2CH_2CHClCH_2CH_3$) (*5-Cl*)

Activation and quench was identical to that of 1-chloropentane except that 9.7 mg of **1** was photolyzed in 0.75 ml of 3-chloropentane. Quenching in vacuo with

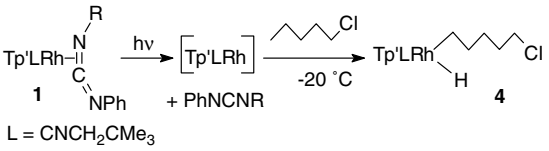
CCl_4 and removal of volatiles afforded a yellow-brown solid. A ^{1}H NMR spectrum shows two products assigned as diastereomers in a 1:1 ratio and about 20% **2**. For **5-Cl**, ^{1}H NMR (C_6D_6): δ Tp′Me: 2.057 (s, 3H), 2.080 (s, 3H), 2.152 (s, 3H), 2.165 (s, 3H), 2.206 (s, 3H), 2.210 (s, 3H), 2.442 (s, 3H), 2.493 (s, 3H), 2.770 (s, 3H), 2.775 (s, 3H), 2.879 (s, 3H), 2.954 (s, 3H); Tp′CH: 5.550 (s, 1H), 5.559 (s, 1H), 5.586 (s, 1H), 5.619 (s, 1H), 5.695 (s, 2H), CNR: 0.703 (s, 9H), 0.710 (s, 9H), 2.601 (s, 2H), 2.628 (s, 2H). CH_2CH_3: 1.063 (t, $J = 7.3$ Hz, 3H), 0.874 (t, $J = 7.5$ Hz, 3H); CHCl: 4.12 (br m, 2H); Rh–CH_2: 3.2–3.5 (br m, 4H), the remaining CH_2 resonances for the alkyl group were highly split and obscured.

2.10. Activation and quench of 2-chloropentane

The exact procedure was followed as for 1-chloropentane except that 8.0 mg of **1** was dissolved in 0.75 ml of 2-chloropentane. Removal of the solvent leaves a yellow-brown solid. ^{1}H NMR spectroscopy shows three products: Tp′Rh(L)(Cl)($CH_2CH_2CH_2CHClCH_3$) (**6-Cl**) (two isomers), and Tp′(L)Cl_2 (**2**) in a 1:1:1 ratio. For **6-Cl** (two diastereomers present), ^{1}H NMR (C_6D_6): δ Tp′Me: 2.068 (s, 3H), 2.080 (s, 3H), 2.155 (s, 3H), 2.167 (s, 3H), 2.218 (s, 6H), 2.407 (s, 3H), 2.426 (s, 3H), 2.779 (s, 3H), 2.785 (s, 3H), 2.946 (s, 3H), 2.980 (s, 3H); Tp′CH: 5.703 (s, 2H), 5.644 (s, 1H), 5.611 (s, 1H), 5.579 (s, 2H); CNR: 0.726 (s, 9H), 0.755 (s, 9H), 2.654 (s, 2H), 2.675 (s, 2H); $CHClCH_3$: 1.270 (d, $J = 6.6$ Hz, 3H), 1.248 (d, $J = 6.5$ Hz, 3H), 2.86 (m, 1H), 3.92 (m, 1H); $RhCH_2$: 3.29 (m, 2H). The remaining CH_2 resonances for the alkyl group were highly split and obscured.

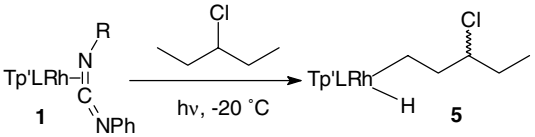
3. Results and discussion

The fragment [Tp′Rh(L)] (L = neopentyl isocyanide), generated cleanly by irradiation of the carbodiimide complex Tp′Rh(L)(PhN=C=Nneopentyl) (**1**), is known to react selectively with the terminal C–H bonds of linear alkanes [15–17]. Importantly, activation of the internal secondary C–H bonds has been demonstrated *not* to occur. In looking at the reaction of this fragment with chloroalkanes, it was expected that the reactive C–Cl bond would undergo oxidative addition to the Rh^I fragment to give Tp′Rh(L)(*n*-alkyl)Cl in light of the ubiquitous reactivity of four-coordinate d^8 Rh(I) and Ir(I) complexes with alkyl halides. It was found, however, that irradiation of **1** in neat 1-chloropentane gives a single product that displays a hydride resonance in the ^{1}H NMR spectrum at δ −14.92 (d, $J = 24.5$ Hz), indicating that a selective C–H bond activation had occurred (Eq. (1)). The presence of six Tp′ methyl and three Tp′ methine resonances was diagnostic for an asymmetric Tp′Rh(L)XY product. The observation of a triplet at δ 3.219 ($J = 6.8$ Hz) indicated the presence of a terminal CH_2CH_2Cl group, and the product was assigned as Tp′Rh(L)($CH_2CH_2CH_2CH_2CH_2Cl$)H (**4**). No evidence for activation of the C–Cl bond or other positions on the hydrocarbon was seen. The hydride product could be converted to its stable chloride derivative (**4-Cl**) by treatment with CCl_4. **4-Cl** displayed a similar set of resonances in the ^{1}H NMR spectrum. Neither **4** nor **4-Cl** could be crystallized suitably for structure determination. **4Cl** displays characteristic multiplet resonances for the α-CH_2Rh protons at δ 3.2 [18]. The alkyl hydride **4** appears to have a stability towards reductive elimination of chloropentane comparable to that of the *n*-pentyl hydride complex studied earlier ($\tau_{1/2} = 2$ h at 22 °C) [15–17].



(1)

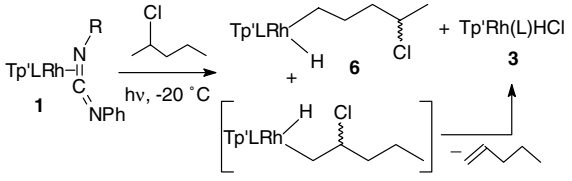
The irradiation of a solution of **1** in 3-chloropentane also leads to a C–H bond activation product, **5**. A single hydride resonance is observed just as for **4** at δ −14.92 (d, $J = 25$ Hz). However, examination of the Tp′ region of the spectrum shows evidence for *two* products. It was recognized that terminal activation of 3-chloropentane should yield two diastereomers of **5** due to the presence of chiral centers both at rhodium and at the γ-carbon of the pentyl chain (Eq. (2)). Confirmation of this possibility was obtained by the observation of two distinct triplets ($J = 7.3$ Hz) at δ 1.017 and 1.010 for the terminal methyl groups and two multiplets at δ 3.93 and 4.00 for the CHCl groups. Distinct *t*-butyl resonances for the isocyanide ligands were also observed. Once again, no activation of the C–Cl or other C–H bonds is seen. The hydride **5** could be converted to the stable chloride derivative **5-Cl**, but once again the product resisted attempts to produce X-ray quality crystals. Hydride complex **5** undergoes reductive elimination of 3-chloropentane over a period of several hours, similar to other alkyl hydrides of the type [15–17].



(2)

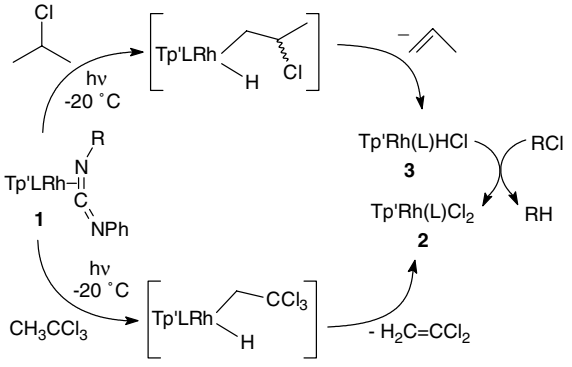
The photochemical reaction of **1** with 2-chloropentane was examined next. It was anticipated that two products would form resulting from the activation of either terminal methyl group, with each regioisomer existing as a pair of diastereomers. Upon irradiation of **1** in 2-chloropentane, two hydride resonances are seen inthe ^{1}H NMR spectrum in a 2.5:1 ratio. The minor resonance is

for the compound, Tp′Rh(L)HCl (**3**), not previously reported, but easily prepared by the titration of **2** with Cp_2ZrH_2. **3** displays a doublet at −13.40 with an unusually small Rh–H coupling constant ($J = 11.5$ Hz), along with characteristic Tp′ resonances. The second hydride belongs to a C–H bond activation product assigned as diasteriomers of $Tp'Rh(L)(CH_2CH_2CH_2CHClCH_3)H$ (**6**) (Eq. (3)). Two sets of Tp′ resonances are observed in a 1:1 ratio and two doublets are seen for the terminal methyl groups at δ 1.082 and 1.031. The CHCl methines appear as multiplets at δ 3.98 and 4.06. While no activation of the opposite end of the 2-pentylchloride is seen (position 1), it is proposed that this isomer did indeed form in the reaction, but that rapid β-chloride elimination leads to the formation of **3** and 1-pentene. 1-Pentene could be identified by GC examination of the volatiles removed from the initial solution. Treatment of **6** with CCl_4 leads to the formation of the chloro derivative, **6-Cl**. The observation of a 2.5:1 ratio of **6**:**3** therefore implies that a CH_3-CH_2-methyl group is 2.5× more reactive than a CH_3–CHCl–methyl group.



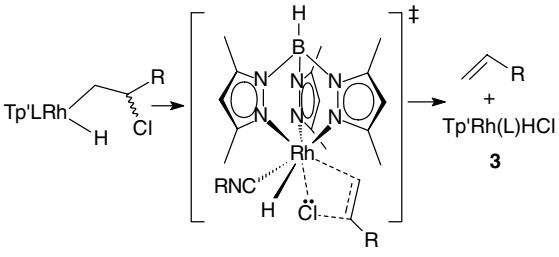
(3)

As further evidence for the occurrence of β-chloride elimination in β-chloro substituted alkyl hydrides, a solution of **1** in 2-chloropropane was irradiated. The ^1H NMR spectrum of this reaction shows the formation of **3** and **2** in a 3:1 ratio, but no C–H activation products (Eq. (4)). Propene was identified in the GC of the solution. Similarly, irradiation of a solution of **1** in 1,1,1-trichloroethane yields only **2**. In both of these reactions, the formation of **2** is believed to occur by reaction of **3** with chloroalkane, as confirmed by control experiments. In comparison, the earlier described reaction of **1** with 2-chloropentane showed formation of **3**, but not **2**, implying that **3** is less reactive with 2-chloropentane than 2-chloropropane.



(4)

One issue that remains to be addressed is the mechanism of β-chloride elimination to generate **3**. The intermediate alkyl hydride complexes in these reactions are formally d^6 octahedral Rh(III) species, which should be inert towards ligand loss. Yet β-elimination of the chloro group would be expected to require an adjacent vacant site, by analogy to the well-established need for a vacant site in β-hydride elimination from an alkyl group [19]. Either these intermediates are indeed labile (η^3-Tp′ → η^2-Tp′), which seems unlikely, or β-chloride elimination does not require an adjacent vacant site. Perhaps the presence of a pair of electrons on the chlorine permits this elimination by way of a direct attack at the metal to displace the incipient olefin that is formed, as indicated in Eq. (5). Such β-chloroalkyl compounds may indeed be inherently unstable towards elimination. A search of the literature showed *no examples* of stable rhodium compounds containing simple β-chloroalkyl ligands (for rhodium, β-chlorofluoroalkyls and β-chloroacetyls are known. For Rh and $IrCl_2(CF_2CF_2Cl)(CO)(PEt_3)_2$, see [20]; for $Rh(COCH_2Cl)Cl_2(CO)(PEt_3)_2$, see [21]; for $RhCl_2(PPh_3)_2$(COCHClMe), see [22]. For iridium, β-chloroalkyls are known. For $IrHCl(CO)(PPh_3)_2(CH_2CCl_2Ph)$, see [23]; for $Ir(PEt_2Ph)_2(CO)Cl_2(CH_2CHClMe)$, see [24]; for $[Re_7C(CO)_{21}Ir(C_8H_{14})(CO)(CH_2CH_2Cl)Cl]^{2-}$, see [25]), yet examples with α-chloroalkyl [26–28] and γ-chloroalkyl ligands were found [29,30]. Indeed the β-chloroalkyl group represents the 'missing link' in the polymerization of vinyl chloride to polyvinylchloride (PVC). Most attempts to polymerize vinylchloride via olefin insertion into a metal-alkyl have failed due to the tendency for β-chloride elimination to occur faster than insertion [31–33].



(5)

4. Conclusions

The reactive fragment [Tp′Rh(CNR)] is found to react with chlorinated alkanes to give terminal methyl group C–H activation products exclusively. If the alkane has chlorine substitution β to the methyl group, then activation is followed by rapid β-elimination to give Tp′Rh(L)HCl and the corresponding olefin. In no case is C–Cl oxidative addition observed.

Acknowledgements

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