New Stereoselective $C_{sp^2} - C_{sp^3}$ Coupling: Catalytic Iodomethylation of Acetylene with Methyl Iodide into *E*-1-Iodopropene

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Abstract—The catalytic addition of methyl iodide to acetylene yielding *E*-1-iodopropene has been discovered. The reaction competes with the formation of *E*, *E*-1, 4-diiodobuta-1, 3-diene. The key intermediate in these reactions is the methylvinyl derivative of Pt^{IV} resulting from acetylene iodoplatination by the Pt^{IV} methyl complex forming in the equilibrium process. The $C_{sp^2}-C_{sp^3}$ coupling product in the reductive elimination of the ligands is *E*-1-iodopropene. *E*, *E*-1, 4-diiodobuta-1, 3-diene is the product of the iodoplatination of the second acetylene molecule by this intermediate followed by the $C_{sp^2}-C_{sp^2}$ coupling of the vinyl ligands.

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INTRODUCTION

Catalytic C-C cross-coupling reactions of that make it possible to obtain complicated compounds from simpler ones with high efficiency have become an important tool of contemporary organic chemistry [1, 2]. Bis(organyl) metal complexes are key intermediates of coupling catalyzed by metal complexes [3]. We have found recently [4, 5] that the oxidative addition of methyl iodide to Pt^{II} iodide complexes followed by the iodoplatination of some substituted alkynes in acetone solution leads to the formation of methylvinyl derivatives of Pt^{IV} , which are more stable to C–C coupling. Evident reasons for the stability of the earlier synthesized methylvinyl complexes of Pt^{IV} are the steric hindrance from the bulky substituents in the vinyl ligands to the reaction and the bidentate character [5] of some of the ligands. In this work, it is shown that the catalytic $C_{sp^2} - C_{sp^3}$ coupling occurs in the MeI-Pt^{II}-NaI-acetone system to form E-1-iodopropene when an unsubstituted alkyne (acetylene) is used. The results of our kinetic and mechanistic study of the discovered reaction are presented.

EXPERIMENTAL

Commercial NaI (reagent grade), methyl iodide (Aldrich), and acetone- d_6 (Merck) were used without additional purification to prepare solutions. The salt Na₂PtCl₄ was synthesized according to a known procedure [6]. The platinum iodide complexes were syn-

thesized in situ by the substitution of the chloride ligands in the presence of excess NaI in acetone- d_6 solution. Na₂PtCl₄ was dissolved in a 1.5 M solution of NaI in acetone- d_6 (0.6 mL), CH₃I was added, and the mixture was left standing for 24 h. The resulting sodium chloride precipitate was separated by centrifugation, the filtrate was transferred to an NMR ampule, and an aliquot of a solution of acetylene and NaI (1.5 mol/L) in acetone- d_6 was added.

The reactions were carried out at room temperature and were monitored as the variation of the intensity of the corresponding signals in the ¹H NMR spectrum. The concentrations of acetylene, methyl iodide, and products were determined relative to the internal standard (benzene). The equilibrium concentration of MePt^{IV}was calculated using the equilibrium constant for the oxidative addition of MeI to the Pt^{II} iodide complexes, which is 0.017 L/mol [4].

The NMR parameters of the reaction products and intermediates are listed in Table 1. ¹H and ¹³CNMR spectra were recorded on Bruker AVANCE 500, Bruker AVANCE II 600, and Bruker AVANCE II 400 spectrometers (Bruker, Germany).

RESULTS

Reaction Kinetics

When Pt^{II} and MeI are in excess over acetylene (at the initial concentration ratios of $[Pt^{II}]_0$: $[C_2H_2]_0$: $[MeI] \approx 1 : 0.1 : 15$) in acetone-d₆ and when there is a large excess of NaI excess, acetylene consumption is

Compound	¹ H NMR: δ (ppm) and J (Hz)	$^{13}C{^{1}H} NMR (\delta, ppm)$	
Complex 1 $\begin{bmatrix} 3 \\ CH_{3/} \\ I \\ $	8.18 (d, $J(H-H) = 13$ with satellites ¹⁹⁵ Pt, ² $J(Pt-H) = 51.5$; 1H; H ¹), 5.61 (d, $J(H-H) = 13$ with satellites ¹⁹⁵ Pt, ³ $J(Pt-H) = 39.1$; 1H; H ²), 3.16 (s with satellites ¹⁹⁵ Pt, ² $J(Pt-H) = 76.6$; 3H; H ³)	_	
Complex 2 $ \begin{array}{c} $	8.01 (d, $J(H-H) = 13$ with satellites ¹⁹⁵ Pt, ² $J(Pt-H) = 40.3$; 1H; H ¹), 5.39 (d; $J(H-H) = 13$ with satellites ¹⁹⁵ Pt, ³ $J(Pt-H) = 44.9$; 1H; H ²), 2.08 (s with satellites ¹⁹⁵ Pt, ² $J(Pt-H) = 77.5$; 6H; H ³ and H ⁴)	_	
<i>E</i> -1-iodopropene $\stackrel{e^2}{H} \xrightarrow{I}$ $\stackrel{H}{\to} \stackrel{H}{\to} \stackrel{H}{\to}$	6.22 (d, $J(H-H) = 13$; 1H; H ^{e1}), 6.53 (m, 1H; H ^{e2}), 1.73 (dd, $J(H-H) = 6.8$; $J(H-H) = 1.6$; 3H; H ^{e3})	75.3 (C ^{e1}), 141.1 (C ^{e2}), 18.4 (C ^{e3})	
<i>E</i> -but-2-ene $H \xrightarrow{b1} \\ CH_3$ H_3C H_{b2} H_1	5.41 (m, 2H, H ^{b1}); 1.59 (m, 6H, H ^{b2})	125.2 (C ^{b1}), 17.3 (C ^{b2})	
<i>E,E</i> -1,4-diiodobuta-1,3-diene $I \xrightarrow{d2} H \\ H \xrightarrow{d1} H \\ H \\ d1 \xrightarrow{d2} I$	6.96 (m, 2H, H ^{d1}); 7.23 (m, 2H, H ^{d2})	82.3 (C ^{d1}), 145.9 (C ^{d2})	
<i>E,E</i> -1-iodopenta-1,3-diene H_3C H_4 H_1	6.43 (d, $J(H-H) = 14.3$, 1H, H ^{t1}); 7.05 (dd, $J(H-H) = 14.3$; 10.3; 1H, H ^{t2}); 6.15 (m, 1H, H ^{t3}); 5.84 (m, 1H, H ^{t4}); 1.69 (m, 3H, H ^{t5})	76.6 (C ^{t1}), 145.1 (C ^{t2}), 131.5 (C ^{t3}), 130.4 (C ^{t4}), 21.1 (C ^{t5})	
<i>E,E</i> -hexa-2,4-diene H_3C H_{p1} H_{p1} H_{p2} H_{p3}	5.55 (m, 2H, H ^{p1}); 6.01 (m, 2H, H ^{p2}); 1.68 (m, 6H, H ^{p3})	125.7 (C ^{p1}), 131.6 (C ^{p2}), 13.3 (C ^{p3})	

Table 1. NMR parameters for the products and intermediates of the reaction between acetylene and methyl iodide in the C_2H_2 -MeI-Pt^{II}-NaI-acetone system

accompanied by the parallel formation of the crosscoupling product of acetylene and methyl iodide, namely, *E*-1-iodopropene as the major product (ip, ~70% yield based on reacted acetylene) and a minor amount of *E*,*E*-1,4-diiodobuta-1,3-diene (dibd, ~29% yield based on reacted acetylene)



The presence of both Pt^{II} and methyl iodide is necessary for these reactions to take place: reactions (I) do not occur in the absence of at least one of the reactants. In addition to the formation of ip and dibd, the formation of the intermediate methylvinyl derivative of Pt^{IV} (complex 1) (Fig. 1) and the evolution of traces of methane were detected by ¹H NMR spectroscopy.

The acetylene disappearance kinetics obeys a firstorder equation. The apparent pseudo-first-order rate constant of acetylene disappearance, $k_{app} = -\frac{d \ln[C_2H_2]}{dt}$, increases with an increasing concentration of Pt^{II} or methyl iodide (Table 2). The dibd yield increases, the ip yield decreases, and k_{app} increases approximately two times as the initial acety-





Fig. 1. Typical kinetics of (*I*) acetylene disappearance, (*2*) *E*-1-iodopropene accumulation, (*3*) *E*,*E*-1,4-diiodobuta-1,3-diene accumulation, and (*4*) complex **1** accumulation. The initial platinum concentration is $[Pt^{II}]_0 = 0.05 \text{ mol/L}$.

lene concentration increases and the other conditions remain unchanged (Table 2, entries 4-6). In this case, the formation of another organoplatinum intermediate (complex 2) was detected by ¹H NMR spectros-copy.

Then the primary products undergo slower transformations: ip gives *E*-but-2-ene, and dibd produces E,E-1-iodopenta-1,3-diene, which then transforms into E,E-hexa-2,4-diene:

$$H \xrightarrow{I} H \xrightarrow{CH_{3I}} H \xrightarrow{CH_{3I}} H \xrightarrow{CH_{3}} H \xrightarrow{CH_{3}} H$$

$$H \xrightarrow{H} H \xrightarrow{H} H \xrightarrow{H} H \xrightarrow{H} H \xrightarrow{CH_{3I}} H \xrightarrow{H} H \xrightarrow{CH_{3I}} H \xrightarrow{H} H \xrightarrow{CH_{3I}} H \xrightarrow{H} H \xrightarrow{CH_{3I}} H \xrightarrow{H} H$$

$$H \xrightarrow{H} H \xrightarrow{H} H \xrightarrow{H} H \xrightarrow{H} H \xrightarrow{CH_{3I}} H \xrightarrow{H} H \xrightarrow$$

For example, 30 h after the beginning of the reaction, under the conditions indicated in Table 2 (entry 6), 90% of the acetylene is reacted, *E*-but-2-ene is accumulated in trace amounts, and the yields of *E*,*E*-1-iodopenta-1,3-diene, *E*,*E*-hexa-2,4-diene, and dibd are (19 ± 2) , (4 ± 1) , and (58 ± 6) %, respectively, based on reacted acetylene. Fot this reason, the yields of ip and dibd in Table 2 are extrapolated to the onset of the reaction, when the formation of secondary products can be neglected. The yield ratio η_{dibd}/η_{ip} increases linearly with an increasing initial acetylene concentration (Fig. 2).

At initial concentrations of $[Pt^{II}]_0 = 0.01$, $[C_2H_2]_0 = 0.32$, and $[MeI]_0 = 1.3 \text{ mol/L}$, the concentration of the resulting *E*-1-iodopropene in 20 days is 0.034 mol/L, which indicates the catalytic character of the reaction and corresponds to a turnover number of 3.4.

Identification of the Products and Intermediates of Reactions (I) and (II)

Complexes 1 and 2. The fairly large value of the spin–spin coupling constant J(H-H) (Table 1) indicates the *trans* arrangement of the vinyl protons H¹ and H² (for the numbering of atoms, see Table 1). The

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Entry	$[Pt^{II}]_0$	[MeI] ₀	$[C_2H_2]_0$	η_{ip}	η_{dibd}	$k = h^{-1}$
	mol/L		%		λ_{app}, Π	
1	0	1.3	0.13	0	0	0
2	0.05	0	0.1	0	0	_
3	0.015	0.68	0.13	18 ± 3	75 ± 8	0.010 ± 0.002
4	0.05	1.0	0.0056	70 ± 6	29 ± 3	0.048 ± 0.005
5	0.05	1.0	0.027	51 ± 5	56 ± 6	0.06 ± 0.01
6	0.05	1.0	0.1	26 ± 2	73 ± 6	0.07 ± 0.01
7	0.1	0.9	0.0016	81 ± 6	19 ± 4	0.055 ± 0.005
8	0.11	1.3	0.002	75 ± 6	18 ± 3	0.095 ± 0.01

Table 2. Apparent pseudo-first-order rate constant k_{app} and the yields of the products extrapolated to the onset of the reaction as a function of the initial concentrations of acetylene, methyl iodide, and Pt^{II}

positions of H¹ signals and the value of J(Pt-H) are characteristic of protons at the C $_{sp^2}$ atom bound to platinum through a σ bond [4, 5, 7]. The position of the H³ (complex 1) and H^{3,4} (complex 2) signals and the values of J(Pt-H) correspond to the methyl ligands of the Pt^{IV} derivatives [7]. For complex 1, the integral intensity ratio is H¹ : H² : H³ = 1 : 1 : 3, which corresponds to the organoplatinum derivative including the vinyl and methyl ligands. Due to their equivalence, the protons of methyl groups 3 and 4 of complex 2 in the ¹H NMR spectrum form a single signal, and, as a consequence, the ratio of signal intensities is H¹ : H² : H^{3,4} = 1 : 1 : 6. The concentrations of complexes 1 and 2 were insufficient for ¹³C and ¹⁹⁵Pt NMR experiments, so the structures of these complexes were deduced from their ¹H NMR spectra.

E-1-iodopropene. The ¹H NMR signal at 1.73 ppm is split into a doublet of doublets with J = 1.6 and 6.8 Hz. The signal at 6.53 ppm is a complicated multiplet, and the signal at 6.22 ppm is split into a doublet of quadruplets with J = 14.3 and 1.6 Hz. The spin—spin coupling between H^{e1}, H^{e2}, and H^{e3} shows itself as cross peaks in the 2D COSY spectrum, indicating that these protons belong to one molecule. The radiation damping of the signal at 1.73 ppm results in the degeneration of the two multiplets at 6.53 and 6.22 ppm into doublets with J = 14.3 Hz. The latter value is characteristic of the *trans* arrangement of the H^{e1} and H^{e2} protons. The radiation damping of the H^{e1} signal reduces the multiplicity of the H^{e2} signal: the doublet of quadruplets degenerates into a quadruplet.

E,E-1-Iodopenta-1,3-diene. In the 2D COSY spectrum, the t1 proton shows spin–spin coupling with the t2 proton; as a result, its signal is split into a doublet with a large coupling constant of J = 14.3 Hz, which is characteristic of the *trans* arrangement of H^{t1} and H^{t2}. The t2 proton is coupled with H^{t1} and H^{t3}, and, accordingly, its signal in the ¹H NMR spectrum is split into a doublet of doublets. The 2D COSY spectrum indicates spin–spin coupling between the H^{t3} proton and H^{t4} and between H^{t4} and H^{t5}.

E,E-Hexa-2,4-diene. Use of the 2D COSY technique makes it possible to reveal the spin-spin coupling between the p1 and p2 protons and between them and the p3 protons of the methyl groups. The radiation damping of the H^{p3} signal makes the multiplicity of the ¹H NMR spectrum similar to the multiplicity of the signal from dibd. The radiation damping of the H^{p1} signal makes the H^{p2} and H^{p3} signals degenerate into singlets. The radiation damping of the H^{p1} signal results in the degeneration of the H^{p1} signal into a quadruplet with J = 6 Hz and in the degeneration of the H^{p3} signal into a doublet with the same value of J. The ¹³C{¹H} NMR spectrum is in agreement with the literature [8].

The signals of *E*-but-2-ene and dibd were assigned according to earlier published data [9, 10].



Fig. 2. Ratio of the yields of dibd and ip extrapolated to the onset of the reaction as a function of the initial acetylene concentration.



Scheme 1. Stepwise mechanism of the formation of ip and dibd.

DISCUSSION

The observed effects can be explained in terms of the stepwise mechanism presented in Scheme 1. The oxidative addition of CH₃I to Pt^{II} is reversible and vields a Pt^{IV} methyl derivative [4] (step 1), which is the active form of the catalyst. The iodoplatination of the C=C bond of acetylene by the latter (step 2) results in the formation of an intermediate Pt^{IV} methylvinyl derivative (complex 1), whose decomposition via reductive elimination gives the $C_{sp^2} - C_{sp^3}$ cross-coupling product (ip) and regenerates the catalyst (step 3). The iodoplatination of the second acetylene molecule by complex 1 with the intermediate formation of the methyldivinyl derivative (step 4) followed by fast reductive elimination affords dibd. A similar iodoplatination of alkyne by the Pt^{IV} methylvinyl complex under the same conditions, accompanied by methyldivinyl derivative formation, was observed for methyl propionate [4].

In the framework of Scheme 1, in a large excess of methyl iodide and under the quasi-steady-state conditions with respect to intermediate complex 1, the acetylene disappearance kinetics is described by the equation

$$\frac{x}{\sqrt{\frac{\beta+2x}{\beta+2}}} = \exp(-at), \tag{1}$$

where $x = \frac{[C_2H_2]}{[C_2H_2]_0}$ is the fraction of acetylene that has

not reacted by the time t, $\beta = \frac{k_3}{k_4[C_2H_2]_0}$, and $a = k_2K_1[Pt^{II}][MeI]$. Equation (1) shows that the apparent

pseudo-first-order rate constant is $k_{app} \approx a$ and 2a in two limiting cases of $\beta \ge 2$ (low initial concentration of acetylene) and $\beta \ll 2$ (high initial concentration of acetylene), respectively. At medium values of the initial concentration, acetylene is consumed at the intermediate rate $\exp(-2at) < x < \exp(-at)$, but, in the strict sense, its disappearance kinetics does not obey a first-order equation. Nevertheless, this kinetics can be approximated [11] by a first-order equation with an apparent rate constant satisfying the inequality $a < k_{app} < 2a$. Thus, the experimentally observed twofold increase in \mathbf{k}_{app} as a result of an increase in the initial concentration of the substrate (Table 2, entries 4–6) can be explained in the framework of Scheme 1.

According to Scheme 1, the ratio of the yields of dibd and ip extrapolated to the onset point of the reaction is given by the equation

$$\frac{\eta_{\text{dibd}}}{\eta_{\text{ip}}} \approx \frac{k_4}{k_3} [C_2 H_2]_0, \qquad (2)$$

which is consistent with the experimental results (Fig. 2) at $\frac{k_4}{k_3} = (29 \pm 2)$ L/mol. The value obtained makes it possible to estimate the lower limit of the ratio of the rate constants of $C_{sp^2}-C_{sp^2}$ and $C_{sp^2}-C_{sp^3}$ couplings (k_5/k_3). No formation of a noticeable concentration of the Pt^{IV} methyldivinyl derivative (complex **3**) was detected at the maximum acetylene concentration used in this work (0.13 mol/L). This means that, under the given conditions, the steady-state concentration of this derivative is substantially lower than the steady-state concentration of complex **1**. Therefore, 0.13 $k_4 \ll k_5$ and, hence, $k_5/k_3 \gg 4$. The validity of Eq. (2) indicates that ip forms predominantly in step 3. Therefore, reductive elimination during the decomposition of



Scheme 2. Possible transformation routes of the methyl derivative of Pt¹¹.

complex **3** with the formation of the $C_{sp^2}-C_{sp^2}$ coupling product occurs much more readily than of the elimination yielding the $C_{sp^2}-C_{sp^3}$ coupling product, which agrees with earlier published data [12, 13].

In the experiments where ip was the main product (Table 2, entries 4, 7, 8), $\beta \ge 2$ and, hence,

$$k_{\rm app} \approx k_2 [{\rm MePt}^{\rm IV}],$$
 (3)

where $[MePt^{IV}] = K_1[Pt^{II}][MeI]$ is the equilibrium concentration of the Pt^{IV} methyl complex. Indeed, the experiments demonstrate a linear dependence of the apparent pseudo-first-order rate constant on the equilibrium concentration of the Pt^{IV} methyl complex (Fig. 3). The validity of dependence (3) is consistent with the assumption that the Pt^{IV} methyl complex participates in step 2 of acetylene iodoplatination. The second-order rate constant determined from the slope of the plot of k_{app} versus the equilibrium concentration of the Pt^{IV} methyl complex is $k_2 = (0.012 \pm 0.001)$ L mol⁻¹ s⁻¹. This value is close in its order of magnitude to the rate constant of acetylene iodoplatination by PtI₆²⁻ in methanol solution at 40°C, which is 0.040 ± 0.003 L mol⁻¹ s⁻¹ [14]. The somewhat smaller value of k_2 obtained in the present work can be a consequence of the lower temperature of reaction (I), the solvent effect, and/or the lower electrophilicity of the methyl derivative of Pt^{IV} compared to that of PtI_6^{2-} .

Note that the acetylene iodoplatination steps (Scheme 1, steps 2, 4) involve the *trans (anti)* addition of platinum and iodine atoms to the triple bond of acetylene with the formation of *E*-iodovinyl derivatives of platinum(IV) only. The stereoselectivity of iodoplatination is usually attributed [15, 16] to the polarity of the solvent used: the *E*-conformers form in polar solvents, including acetone. The insertion of acetylene into the Pt–I bond with the formation of the *cis (syn)* addition product is low-sensitive to solvent polarity and can occur in low-polar and nonpolar solvents.

The subsequent transformations of the Pt^{II} methyl derivative formed in step 5 (Scheme 1) elucidate the routes of the formation of the experimentally observed secondary reaction products (Scheme 2).

The protolysis of MePt^{II} (step 1) under the action of water traces can be responsible for methane formation. The oxidative addition of the organoiodine compounds to MePt^{II} should afford bis(organyl) derivatives of Pt^{IV} (steps 3, 4). The iodoplatination of acetylene by the Pt^{IV} dimethyl complex (step 5) is for the cause of the formation of experimentally observed



Fig. 3. Apparent pseudo-first-order rate constant for acetylene disappearance as a function of the equilibrium $MePt^{IV}$ concentration.

complex **2**, which can decompose via $C_{sp^2}-C_{sp^3}$ coupling (step 6), regenerating MePt^{II} and providing an additional route for ip formation. More complicated $C_{sp^2}-C_{sp^3}$ coupling products can form via reductive elimination from the methylvinyl derivatives (step 7). The Pt^{II} methyl derivative can also be oxidized [17] with atmospheric oxygen to MePt^{IV} (step 2).

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