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Reactions of trichloromethanesulfonyl chloride and carbon tetrachloride with silyl enol ethers catalyzed by a ruthenium(II) phosphine complex

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Abstract

The reactions of trichloromethanesulfonyl chloride with trimethylsilyl enol ethers of acetophenones in the presence of a ruthenium(II) phosphine complex gave 1-aryl-3,3-dichloropropen-1-one together with α-chloroacetophenones. The product ratio depended on the substituent on the aromatic ring of the silyl enol ether. The reactions of carbon tetrachloride with the silyl enol ethers under similar conditions afforded 1-aryl-3,3-dichloropropen-1-one in good yield. © 1998 Elsevier Science S.A.

Keywords: Silyl enol ether; Ruthenium(II) complex; Catalytic reaction

1. Introduction

Previously, we reported that the ruthenium(II) phosphine complex catalyzed reactions of alkane- and arenesulfonyl chlorides with alkenes afforded the corresponding 1:1 adduct in high yield [1–3], on the other hand, the reactions of trichloromethane- and perfluoroalkanesulfonyl chlorides with alkenes gave 1:1 adduct with extrusion of sulfur dioxide [4,5]. Moreover, we recently reported that the reactions of alkane- and arenesulfonyl chlorides with silyl enol ethers in the presence of the ruthenium(II) complex gave the corresponding β-keto sulfones in high yield [6]. In the course of our studies on the ruthenium(II) catalyzed reactions of various sulfonyl chlorides with alkenes, we found, in this study, that the reactions of trichloromethanesulfonyl chloride with trimethylsilyl enol ethers of acetophenones gave 1-aryl-3,3-dichloro-propen-1-one and α-chloroacetophenone. The reactions of carbon tetrachloride with silyl enol ethers in the presence of the ruthenium(II) complex were also studied and found to give selectively 1-aryl-3,3-dichloropropen-1-one in high yield. The results are described herein.

2. Results and discussion

When a solution of trichloromethanesulfonyl chloride (2.0 mmol), 1-trimethylsiloxy-1-phenylethene (**1a**) (4.0 mmol), and dichlorotris(triphenylphosphine) ruthenium(II) (0.02 mmol) in benzene (4.0 cm^3) was degassed and heated at 120°C for 7 h, the reaction proceeded smoothly to afford 3,3-dichloro-1-phenylpropen-1-one (**2a**) in 25% yield together with phenacyl chloride (**3a**). No such 1:1 adduct as 1-chloro-2-(trichloromethanesulfonyl)-1-trimethylsiloxy-1-phenylethane (**4**) and 1,3,3,3-tetrachloro-1-trimethylsiloxy-1-phenylpropane (**5**) or 3,3,3-trichloro-1-phenylpropan-1-one (**6**), which is a β-elimination product of trimethylchlorosilane from the adduct **5**, was found in the reaction mixture. It is of interest that compound **6** was not found in the reaction mixture since the product **2a** should form via the dehydrochlorination of compound **6** once formed.

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This means that the dehydrochlorination proceeds very rapidly under the reaction conditions.

$$CCl_3SO_2Cl + \underset{\mathbf{1a}}{PhC(OSiMe_3)=CH_2} \xrightarrow[C_6H_6,120^\circ C,7h]{RuCl_2(PPh_3)_3} \underset{\mathbf{2a}}{PhCCH=CCl_2} + \underset{\mathbf{3a}}{PhCH_2Cl}$$

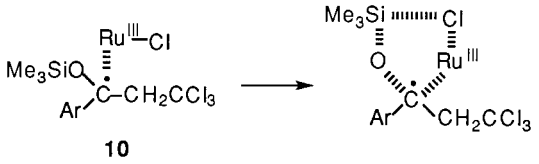
$$\underset{4}{PhCClCH_2SO_2CCl_3} \quad \underset{5}{PhCClCH_2CCl_3} \quad \underset{6}{PhCCH_2CCl_3}$$

Similarly, the reactions of trichloromethanesulfonyl chloride with several silyl enol ethers (**1b–f**) of substituted acetophenones were carried out in the presence of the ruthenium(II) complex, and the results are summarized in Table 1. The reaction of trichloromethanesulfonyl chloride with 1-(4′-nitrophenyl)-1-trimethylsiloxyethene (**1f**) selectively gave 3,3-dichloro-1-(4′-nitrophenyl)propen-1-one (**2f**) in 83% yield. On the other hand, the reactions of trichloromethanesulfonyl chloride with 1-(4′-methoxyphenyl)-1-trimethylsiloxyethene (**1b**) and 1-(4′-tolyl)-1-trimethylsiloxyethene (**1c**) afforded 4-methoxyphenacyl chloride (**3b**) and 4-methylphenacyl chloride (**3c**) in 80 and 90% yields, respectively, and no corresponding 1-aryl-3,3-dichloropropen-1-one (**2**) was found. The results indicate that the silyl enol ether possessing a strong electron-withdrawing group selectively gives product **2**, whereas one possessing an electron-donating group selectively affords chlorinated compound **3**.

$$CCl_3SO_2Cl + \underset{\mathbf{1}}{ArC(OSiMe_3)=CH_2} \xrightarrow[C_6H_6,120^\circ C,7h]{RuCl_2(PPh_3)_3} \underset{\mathbf{2}}{ArCCH(=O)} = CCl_2 + \underset{\mathbf{3}}{ArCCH_2Cl(=O)}$$

A plausible reaction mechanism for the ruthenium(II) catalyzed is given in Scheme 1. The redox-transfer reaction between trichloromethanesulfonyl chloride and the ruthenium(II) catalyst affords anion radical **7**, which cleaves homolytically to give trichloromethanesulfonyl radical **8** and Ru^{III}–Cl. The sulfonyl radical **8** releases sulfur dioxide, giving trichloromethyl radical **9**, which adds to the carbon–carbon double bond of silyl enol ether **1**. The resulting carbon radical **10** affords 1-aryl-3,3,3-trichloropropan-1-one (**11**) and trimethylchlorosilane, and the ruthenium(II) catalyst is regenerated.

The radicals **8**, **9**, and **10** are considered to be confined in the coordination sphere of the ruthenium catalyst [7]. Because of this, the formation of compound **11** and trimethylchlorosilane from radical **10** is considered to proceed via a five-membered transition state without forming adduct 1-aryl-1,3,3,3-tetrachloro-1-trimethylsiloxypropane (**5**). The dehydrochlorination of **11** giving **2** is considered to proceed by a catalytic reaction of the ruthenium(II) complex, as reported for similar reactions [8].



10

$$\rightarrow \underset{\mathbf{11}}{ArC(=O)CH_2CCl_3} + Me_3SiCl + Ru^{II}$$

$$\underset{\mathbf{11}}{ArC(=O)CH_2CCl_3} \xrightarrow{RuCl_2(PPh_3)_3} \underset{\mathbf{2}}{ArC(=O)CH=CCl_2} + HCl$$

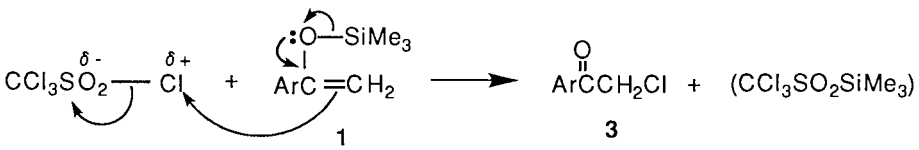
The chlorinated compounds **3** is considered to form by a non-catalytic reaction as follows. The chlorine and the sulfur atoms of trichloromethanesulfonyl chloride are polarized to $\delta+$ and $\delta-$, respectively, in contrast to ordinary alkane- and arenesulfonyl chlorides, since trichloromethyl group is strongly electron-withdrawing. Therefore, nucleophilic attack may occur on the positively charged chlorine atom of trichloromethanesulfonyl chloride by π-electrons of the carbon–carbon double bond in the silyl enol ether possessing an electron-donating group, to give phenacyl chloride derivatives. However, such a reaction does not occur when the

Table 1
Reactions of trichloromethanesulfonyl chloride with silyl enol ethers (**1**) in the presence of a ruthenium(II) complex[a]

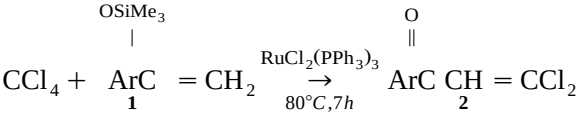
Ar in **1**		Products, Yield[b]/%			
1a	Ph	**2a**	25	**3a**	34
1b	*p*-$MeOC_6H_4$	**2b**	0	**3b**	80
1c	*p*-MeC_6H_4	**2c**	0	**3c**	90
1d	*p*-FC_6H_4	**2d**	21	**3d**	58
1e	*p*-ClC_6H_4	**2e**	41	**3e**	49
1f	*p*-$NO_2C_6H_4$	**2f**	83	**3f**	0

[a] The reactions were carried out at 120°C for 7 h in a degassed sealed tube containing trichloromethanesulfonyl chloride (2.0 mmol), a silyl enol ether (4.0 mmol), dichlorotris(triphenylphosphine)ruthenium(II) (0.02 mmol) in benzene (4.0 cm^3). [b] Isolated yield.

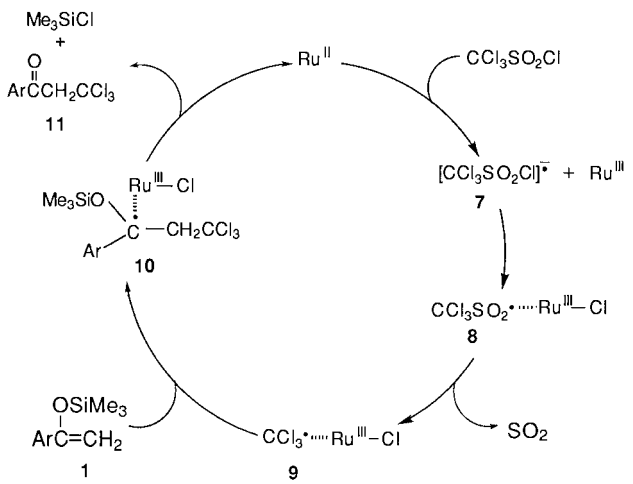
aromatic nucleus of silyl enol ether possesses a strong electron-withdrawing group such as nitro group, i.e.; in this case the π-electron of the carbon–carbon double bond in the silyl enol ether is not nucleophilic enough to attack the chlorine atom of trichloromethanesulfonyl chloride. We have found a similar chlorination of silyl enol ethers possessing electron-donating group by trifluoromethanesulfonyl chloride [9].



Then, the reactions of carbon tetrachloride with silyl enol ethers **1a–f** were studied in the presence of the ruthenium(II) complex since Nagai et al. reported that the similar reactions of carbon tetrachloride with 1-alkene afforded the corresponding 1:1 adduct in high yield [10–13]. When a solution of carbon tetrachloride (6.0 mmol), 1-trimethyl-siloxy-1-phenylethene **1a** (2.0 mmol), and dichlorotris(triphenylphosphine) ruthenium(II) (0.02 mmol) was degassed and heated at 80°C for 7 h, the reaction proceeded smoothly and 3,3-dichloro-1-phenylpropen-1-one **2a** was obtained in 61% yield. The reactions of carbon tetrachloride with other silyl enol ethers **1b–f** were also carried out under similar conditions, and the results are summarized in Table 2. In all cases, 1-aryl-3,3-dichloropropen-1-one **2** formed as a sole product in good yield, and in this case no phenacyl chloride derivative **3** was found in the reaction mixture.



The reaction mechanism for the ruthenium catalyzed reaction of carbon tetrachloride with silyl enol ether will be quite similar to that for trichloromethanesulfonyl chloride with silyl enol ether shown in Scheme 1. The redox-transfer reaction between carbon tetrachloride and the ruthenium(II) catalyst affords anion radical **12** of carbon tetrachloride, which cleaves homolytically to give trichloromethyl radical **9** and Ru^{III}–Cl. Note that the same radical is formed by the interaction between trichloromethanesulfonyl chloride and the ruthenium(II) complex (Scheme 1), and therefore the subsequent reaction with silyl enol ether proceeds similarly in the case of trichloromethanesulfonyl chloride. The reason why the phenacyl chloride derivative **3** did not form in the case of the reactions of carbon tetrachloride is considered as follows. The chlorine atom of carbon tetrachloride is not so positive enough to be attacked nucleophilically by π-electron of carbon–carbon bond of silyl enol ether, and in this case the catalytic reaction by the ruthenium(II) complex proceeds preferentially.



Scheme 1.

3. Experimental details

M.p.s. were determined on a Yamato MP21 apparatus and are uncorrected. IR spectra were determined on a JASCO A-100 IR spectrophotometer with samples either neat liquids or KBr disks. ^{1}H or ^{13}C NMR spectra were determined on a JEOL JNM-EX 400 and a JEOL JNM-LA 500 FT NMR spectrometers at 400 or 100 and 500 or 125 MHz, respectively, using Me_4Si as an internal standard. Mass spectra were measured on a JEOL JMS-AX 500 spectrometer by electron impact (EI) at 70 eV. Gas–liquid chromatography (GLC) was performed using a Hitachi G-3000 gas chromatography with OV-1 (10%) 25 m capillary column. Gel-permeation chromatography (GPC) was performed using a JAI LC-08 and JAI LC-908 liquid chromatography with two JAIGEL-1H columns (20 mm × 600 mm) with chloroform as eluent.

All solvents were distilled and stored under nitrogen. *p*-Methoxyacetophenone and *p*-fluoroacetophenone from Wako Chemicals, *p*-methylacetophenone, *p*-chloroacetophenone, and *p*-nitroacetophenone from Tokyo Kasei Chemicals, and trimethylchlorosilane from Shin-Etsu Chemicals were used without further purification

Table 2
Reactions of carbon tetrachloride with silyl enol ethers (**1**) in the presence of a ruthenium(II) complex[a]

Ar in **1**		Product, Yield[b]/%	
1a	Ph	**2a**	61
1b	*p*-$MeOC_6H_4$	**2b**	71
1c	*p*-MeC_6H_4	**2c**	63
1d	*p*-FC_6H_4	**2d**	65
1e	*p*-ClC_6H_4	**2e**	65
1f	*p*-$NO_2C_6H_4$	**2f**	70

[a] The reaction was carried out at 80°C for 7 h in a degassed sealed tube containing carbon tetrachloride (6.0 mmol), silyl enol ether (2.0 mmol), and dichlorotris(triphenylphosphine)ruthenium(II) (0.02 mmol). [b] Isolated yield.

for the preparation of corresponding silyl enol ether. 1-Trimethylsiloxy-1-phenylethene (**1a**) of Aldrich Chemicals and trichloromethanesulfonyl chloride of Tokyo Kasei Chemicals were used without further purification. Dichlorotris(triphenyl-phosphine)ruthenium(II) was prepared by the method described in the literature [14–16].

3.1. General procedure for the preparation of silyl enol ether [17]

To a solution of aryl methyl ketone (80 mmol) in DMF (32 ml) was added triethylamine (19.5 g, 192 mmol) and then trimethylchlorosilane (10.4 g, 96 mmol) under nitrogen, and the solution was refluxed with stirring for 48 h. The organic component was extracted with pentane (80 ml), and the extract was washed with a saturated potassium bicarbonate solution (3 times), with water (80 cm^3, 3 times), with 1.5 N hydrochloric acid (80 cm^3, 2 times), with water (80 cm^3), with saturated potassium bicarbonate solution (80 cm^3, 2 times), with water (80 cm^3, 2 times), and with brime (80 cm^3). The solvent was removed under reduced pressure, and the residual oil of silyl enol ether was purified by distillation. Yields and boiling points of silyl enol ethers are as follows: 1-trimethylsiloxy-1-(4′-methoxyphenyl)ethene (**1b**) yield 50%, bp 88°C/0.50 mmHg; 1-trimethylsiloxy-1-(4′-tolyl)ethene (**1c**) yield 67%, bp 59–60°C/0.45 mmHg; 1-trimethylsiloxy-1-(4′-fluorophenyl)ethene (**1d**) yield 59%, bp 43–46°C/0.50 mmHg; 1-trimethylsiloxy-1-(4′-chlorophenyl)ethene (**1e**) yield 42%, 54–56°C/0.8 mmHg; 1-trimethylsiloxy-1-(4′-nitrophenyl)ethene (**1f**) yield 34%, bp 95–97°C/0.45 mmHg.

3.2. General procedure for the reaction of trichloromethanesulfonyl chloride with silyl enol ethers

A solution containing trichloromethanesulfonyl chloride (2.0 mmol), silyl enol ether **1** (4.0 mmol), and dichlorotris(triphenylphosphine)ruthenium(II) (0.02 mmol) in dry benzene (4.0 cm^3) was degassed by a freeze–pump–thaw cycle, sealed in an ampoule, and heated at 120°C for 7 h. The reaction mixture was subjected to column chromatography on Merck 7734 silica–gel 60 with hexane-dichloromethane (2:1) or hexane-benzene (1:1) as eluent. The product was further purified by recrystallization from dichloromethane-hexane and identified by IR, NMR and MS spectroscopy.

3.3. General procedure for the reaction of carbon tetrachloride with silyl enol ether

A solution containing carbon tetrachloride (961 mg, 6.0 mmol), silyl enol ether **1** (2.0 mmol), and dichlorotris(triphenylphosphine)ruthenium(II) (0.02 mmol) was degassed by a freeze–pump–thaw cycle, sealed in an ampoule, and heated at 80°C for 7 h. The reaction mixture was subjected to column chromatography on Merck 7734 silica-gel 60 with hexane-dichloromethane (2:1) or hexane-benzene (1:1) as eluent. The product was further purified by recrystallization from dichloromethane-hexane and identified by IR, NMR and MS spectroscopy. The physical and spectral data for the compounds obtained are as follows.

3,3-Dichloro-1-phenylpropen-1-one [18] (**2a**): colorless oil; IR (neat) 3060, 1670, 1570, 1220, and 1160 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.28 (1H, s), 7.50 (2H, t, $J = 7.5$ Hz), 7.61 (1H, t, $J = 7.5$ Hz), and 7.93 (2H, d, $J = 7.5$ Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 124.0, 128.5, 128.9, 133.7, 135.6, 136.9, and 186.6; MS (m/z) 200 (M^+, ^{35}Cl), 172, 105, and 77.

3,3-Dichloro-1-(4′-methoxyphenyl)propen-1-one (**2b**): pale yellow oil; ^1H NMR (400 MHz, $CDCl_3$) δ 3.89 (3H, s), 6.96 (2H, d, $J = 9.0$ Hz), 7.21 (1H, s), and 7.91 (2H, d, $J = 9.0$ Hz); MS (m/z) 230 (M^+), 202, 135, and 92.

3,3-Dichloro-1-(4′-tolyl)propen-1-one (**2c**): pale yellow oil; ^1H NMR (500 MHz, $CDCl_3$) δ 2.43 (3H, s), 7.24 (1H, s), 7.29 (2H, d, $J = 8.3$ Hz), and 7.83 (2H, d, $J = 8.3$ Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.8, 124.3, 128.7, 129.6, 134.4, 134.9, 144.8, and 186.4; MS (m/z) 214 (M^+), 186, 119, and 91.

3,3-Dichloro-1-(4′-fluorophenyl)propen-1-one (**2d**): colorless needles; mp 29.0–29.9°C, IR (neat) 3060,

1670, 1600, 1570, 1220, 1100, and 920 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.17 (2H, dd, $J = 8.8$ and 8.8 Hz), 7.22 (1H, s), and 7.96 (2H, dd, $J = 8.8$ and 5.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 116.4, 124.1, 131.5, 133.6, 136.0, 166.4, and 185.5; MS (m/z) 218 (M^+, ^{35}Cl), 123, and 95. Anal. Calcd for $C_9H_5OFCl_2$: C, 49.35; H, 2.36. Found: C, 48.81; H, 2.11. HRMS: Calcd for $C_9H_5OFCl_2$, 217.9701. Found: m/z 217.9710.

3,3-Dichloro-1-(4′-chlorophenyl)propen-1-one (**2e**): colorless needles; mp 49.1–49.9°C (51–52°C [19,20]); IR (neat) 3080, 1660, 1580, and 1220 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.22 (1H, s), 7.47 (2H, d, $J = 8.8$ Hz), and 7.86 (2H, d, $J = 8.8$ Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 123.8, 129.5, 130.2, 135.5, 136.6, 140.6, and 185.7; MS (m/z) 234 (M^+, ^{35}Cl), 141, and 95.

3,3-Dichloro-1-(4′-nitrophenyl)propen-1-one (**2f**): pale yellow needles; mp 109.0–109.8°C (108–109°C [21,22]); IR (neat) 3100, 1680, 1600, 1560, 1520, 1350, and 1220 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.31 (1H, s), 8.09 (2H, d, $J = 8.8$ Hz), and 8.36 (2H, d, $J = 8.8$ Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 122.6, 123.7, 129.1, 138.2, 141.2, 150.2, and 184.5; MS (m/z) 245 (M^+, ^{35}Cl), 219, 164, and 151.

Phenacyl chloride (**3a**): mp 51.0–51.8°C (54°C [23]); ^1H NMR (400 MHz, $CDCl_3$) δ 4.67 (2H, s), 7.50 (2H, dd, $J = 7.3$ and 7.3 Hz), 7.62 (1H, t, $J = 7.3$ Hz), and 7.96 (2H, d, $J = 7.3$ Hz); MS (m/z) 154 (M^+), 105, and 77.

4-Methoxyphenacyl chloride (**3b**): colorless needles; mp 97.9–98.5°C (56–57°C [23]); IR (KBr) 3020, 3000, 2950, 1700, and 1600 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.88 (3H, s), 4.65 (2H, s), 6.96 (2H, d, $J = 5.3$ Hz), and 7.94 (2H, d, $J = 5.3$ Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 45.3, 55.5, 113.7, 113.8, 126.9, 130.6, and 189.3; MS (m/z) 184 (M^+), 135, and 77.

4-Methylphenacyl chloride (**3c**): mp 53.1–53.8°C (56–57°C [24]); ^1H NMR (400 MHz, $CDCl_3$) δ 2.43 (3H, s), 4.69 (2H, s), 7.30 (2H, d, $J = 8.0$ Hz), and 7.86 (2H, d, $J = 8.0$ Hz); MS (m/z) 168 (M^+), 119, 105, and 91.

4-Fluorophenacyl chloride (**3d**): mp 42.7–43.6°C; ^1H NMR (400 MHz, $CDCl_3$) δ 4.67 (2H, s), 7.18 (2H, dd, $J = 8.8$ and 8.8 Hz), and 8.01 (2H, dd, $J = 8.8$ and 5.1 Hz); MS (m/z) 172 (M^+), 137, 124, and 96. HRMS: Calcd for C_8H_6OFCl, 172.0091. Found: m/z 172.0075.

4-Chlorophenacyl chloride (**4e**): mp 96.8–97.6°C (102–103°C [24]); ^1H NMR (400 MHz, $CDCl_3$) δ 4.66 (2H, s), 7.48 (2H, d, $J = 8.6$ Hz), and 7.91 (2H, d, $J = 8.6$ Hz); MS (m/z) 188 (M^+, ^{35}Cl), 141, and 111.

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