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Note

C–Si bond cleavage of trihalomethyltrimethylsilane by alkoxo- and aryloxogold or -copper complexes

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Abstract

The C–Si bond cleavage of trihalomethyltrimethylsilane Me_3SiCX_3 (X = F, Cl) proceeds smoothly by alkoxo- and aryloxogold(I or III) or -copper(I) complexes Au(OR)L (OR = $OCH(CF_3)_2$, OPh), L = PCy_3, PPh_3, PMe_2Ph, PMe_3), *cis*-$AuMe_2(OPh)L$ (L = $PMePh_2$, PEt_3, PMe_2Ph, PMe_3), $Cu(OR)(PPh_3)_3$ (OR = $OCH(CF_3)_2$, OPh) to give trihalomethylgold or -copper complexes $Au(CX_3)L$, *cis*-$AuMe_2(CF_3)L$, or $Cu(CF_3)L_3$ with liberation of the corresponding silyl ether Me_3SiOR. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Alkoxogold complexes; Alkoxocopper complexes; C–Si bond cleavage; Trihalomethyltrimethylsilane; (Trihalomethyl)gold complexes

1. Introduction

Electrophilic substitution at C–Si bond by E^+–Nu^- giving C–E and Si–Nu is a well-encountered important reaction in organic synthesis, which frequently requires Lewis acid as an activator [1]. Late transition metal complexes are also intrinsically capable of cleaving these bonds to give new organometallic species, when the metal is nucleophilic enough to activate these bonds [2–5]. We reported previously highly basic and nucleophilic fluoroalkoxo- and aryloxogold(I or III) complexes with tertiary phoshpine ligand [6,7], showing hydrogen bonds between alkoxide and free alcohol, hydrogen abstraction from active methylene compounds and transition metal hydrides [8], and ring opening reaction of thiiranes [9] by the alkoxogold complexes. These complexes also act as catalysts for the Knöevenagel reaction [10]. In these reactions, gold is considered to assist these hydrogen abstraction processes by coordination. We now report carbon–silicon bond cleavage of trihalomethyltrimethylsilanes by these alkoxo- and aryloxogold or -copper complexes giving (trihalomethyl)gold (or copper) complexes.

2. Experimental

2.1. General

All manipulations were carried out under nitrogen or argon using standard Schlenk and vacuum-line techniques. All solvents were distilled from appropriate drying reagents under nitrogen or distilled under vacuum prior to use. Alkoxo- and aryloxogold complexes were prepared according to the method of our previous paper [6,7]. Alkoxo- and aryloxocopper complexes were prepared by literature methods [11,12]. NMR spectra were measured on a JEOL LA-300 (^1H, 300.4 MHz; ^{19}F, 298.5 MHz; ^{31}P, 121.6 MHz) spectrometer. Chemical shifts (ppm) are referred to internal TMS at 0.0 ppm, external C_6H_5F at -113.0 ppm and external 85% H_3PO_4 in D_2O at 0.0 ppm for ^1H, $^{19}F\{^1H\}$ and $^{31}P\{^1H\}$ NMR, respectively. IR spectra were recorded on a JASCO FT/IR-410 spectrometer, using KBr disks. Elemental analyses were performed by Perkin–Elmer 2400

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series II CHN analyzer. Melting points were measured by a Yamato MP-21, and the values were uncorrected.

2.2. Reactions of $Au(OR)L$ ($L = PCy_3$, $OR = OCH(CF_3)_2$ (**1a**); $OR = OPh$ (**1b**); $L = PPh_3$, $OR = OCH(CF_3)_2$ (**1c**); $OR = OPh$; $L = PMe_2Ph$, $OR = OCH(CF_3)_2$ (**1d**); $L = PMe_3$, $OR = OCH(CF_3)_2$ (**1e**)) with Me_3SiCF_3

To a THF (6.5 ml) solution of $Au[OCH(CF_3)_2](PCy_3)$ (**1a**) (103 mg, 0.160 mmol) was added Me_3SiCF_3 (0.50 M THF solution, 0.320 ml, 0.16 mmol). The solution was stirred at −40°C for 1 h and then at room temperature (r.t.) for 3 h. After the reaction, GLC analysis showed the formation of $Me_3SiOCH(CF_3)_2$ 0.125 mmol (78%) [13]. All volatile matters were removed in vacuo to give white powder, which was washed with cold MeOH, and dried in vacuo. Recrystallization from MeOH gave white crystals of $Au(CF_3)(PCy_3)$ (**2a**). Yield 20.8 mg (23%). ^1H NMR of the reaction mixture in C_6D_6 revealed quantitative formation of **2a**. Compound **2a** was also prepared from $Au(OPh)(PCy_3)$ (**1b**) (75.0 mg, 0.136 mmol) and Me_3SiCF_3 (0.50 M THF solution, 0.260 ml, 0.13 mmol). Yield 24.0 mg (32%). Me_3SiOPh 0.121 mmol (89%). ^1H NMR (C_6D_6): δ 0.9–1.5 (m, P*Cy*). $^{19}F\{^1H\}$ NMR (C_6D_6): δ −29.1 (d, $J_{F-P} = 41$ Hz, C*F*$_3$). $^{31}P\{^1H\}$ NMR (C_6D_6): δ 53.5 (q, $J_{P-F} = 41$ Hz, *P*Cy). IR (KBr): 2998, 2903, 1421, 1189, 1120, 992 cm^{-1}. M.p. (dec.): 147–148°C. *Anal.* Calc. for $C_{19}H_{33}AuF_3P$: C, 41.76; H, 6.09. Found: C, 41.58; H, 6.02%.

Reactions of other alkoxo- and aryloxogold(I) complexes with Me_3SiCF_3 were carried out analogously. The yields, spectroscopic and physical data of the resulting complexes are summarized below.

$Au(CF_3)(PPh_3)$ (**2b**) [14] was prepared from $Au[OCH(CF_3)_2](PPh_3)$ (**1c**) (10.3 mg, 0.164 mmol) and Me_3SiCF_3 (0.50 M THF solution, 0.640 ml, 0.32 mmol). Yield 18.4 mg (21%). $Me_3SiOCH(CF_3)_2$ 0.118 mmol (72%). *Anal.* Calc. for $C_{19}H_{15}AuF_3P$: C, 43.20; H, 2.86. Found: C, 43.56; H, 2.52%. Compound **2b** was also prepared from $Au(OPh)(PPh_3)$ (**1d**) (103.0 mg, 0.186 mmol) and Me_3SiCF_3 (0.50 M THF solution, 0.750 ml, 0.37 mmol). Yield 23.0 mg (23%). Me_3SiOPh 0.125 mmol (67%). This complex was characterized spectroscopically. ^1H NMR (C_6D_6): δ 6.9–7.2 (m, P*Ph*$_3$). $^{31}P\{^1H\}$ NMR (C_6D_6): δ 38.6 (q, $J = 46$ Hz, *P*Ph$_3$).

$Au(CF_3)(PMe_2Ph)$ (**2c**) was prepared from $Au[OCH(CF_3)_2](PMe_2Ph)$ (**1e**) (32.5 mg, 0.0647 mmol) and Me_3SiCF_3 (0.50 M THF solution, 0.130 ml, 0.065 mmol). Yield 5.4 mg (21%). $Me_3SiOCH(CF_3)_2$ 0.0606 mmol (94%). *Anal.* Calc. for $C_9H_{11}AuF_3P$: C, 26.75; H, 2.74. Found: C, 27.03; H, 2.50%.

$Au(CF_3)(PMe_3)$ (**2d**) was prepared from $Au[OCH(CF_3)_2](PMe_3)$ (**1f**) (12.1 mg, 0.0275 mmol) and Me_3SiCF_3 (0.50 M THF solution, 0.0550 ml, 0.028 mmol). Yield 8.2 mg (87%). $Me_3SiOCH(CF_3)_2$ 0.020 mmol (71%). This complex was characterized spectroscopically. ^1H NMR (C_6D_6): δ 0.56 (d, $J = 10$ Hz, PMe$_3$). $^{19}F\{^1H\}$ NMR (C_6D_6): δ −29 (br s, C*F*$_3$). $^{31}P\{^1H\}$ NMR (C_6D_6): δ −1.8 (br s, *P*Me$_3$).

2.3. Reactions of *cis*-$AuMe_2(OPh)L$ ($L = PMePh_2$ (**3f**), PEt_3 (**3g**), PMe_2Ph (**3h**), PMe_3 (**3i**)) with Me_3SiCF_3

To *cis*-$AuMe_2(OPh)(PMe_2Ph)$ (**3h**) (59.1 mg, 0.101 mmol) was added Me_3SiCF_3 (0.50 M THF solution, 0.640 ml, 0.32 mmol) in THF (5 ml). The solution was stirred at r.t. for 3 h. After the stirring, Me_3SiOPh (0.0972 mmol, 96%) was detected by GLC. After removal of volatile matters in vacuo, the residual solid was washed with cold hexane to give white powder of *cis*-$AuMe_2(CF_3)(PMe_2Ph)$ (**4h**) [15]. Yield 40.1 mg (91%). ^1H NMR (C_6D_6): δ 0.36 (d, $J = 9$ Hz, 3H, Au–*Me trans* to P), 1.23 (d, $J = 10$ Hz, 6H, P*Me*$_2$Ph), 1.66 (qui, $J = 8$ Hz, 3H, Au–*Me cis* to P), 6.8–7.4 (m, 5H, PMe$_2$*Ph*). $^{31}P\{^1H\}$ NMR (C_6D_6): δ 4.4 (q, $J = 6$ Hz, *P*Me$_2$Ph). *Anal.* Calc. for $C_{11}H_{17}AuF_3P$: C, 30.43; H, 3.95. Found: C, 30.79; H, 3.74%.

Reactions of other aryloxogold(III) complexes with various phosphine ligands were carried out analogously. The yields of the resulting complexes are summarized below.

cis-$AuMe_2(CF_3)(PMePh_2)$ (**4f**) was prepared from the reaction of *cis*-$AuMe_2(OPh)(PMePh_2)$ (**3f**) (12.3 mg, 0.0143 mmol) with Me_3SiCF_3 (0.33 M THF solution, 0.0440 ml, 0.015 mmol). ^1H NMR yield 0.0142 mmol (100%). Me_3SiOPh 0.0140 mmol (98%).

cis-$AuMe_2(CF_3)(PEt_3)$ (**4g**) was prepared from the reaction of *cis*-$AuMe_2(OPh)(PEt_3)$ (**3g**) (11.6 mg, 0.0144 mmol) with Me_3SiCF_3 (0.33 M THF solution, 0.0440 ml, 0.015 mmol). ^1H NMR yield 0.0142 mmol (100%). Me_3SiOPh 0.0144 mmol (100%)

cis-$AuMe_2(CF_3)(PMe_3)$ (**4i**) [15] was prepared from the reaction of *cis*-$AuMe_2(OPh)(PMe_3)$ (**3i**) (115 mg, 0.290 mmol) with Me_3SiCF_3 (0.50 M THF solution, 1.75 ml, 0.87 mmol). Yield 84.2 mg (78%). Me_3SiOPh 0.262 mmol (90%). ^1H NMR (C_6D_6): δ 0.50 (d, $J = 9$ Hz, 3H, Au–*Me trans* to P), 1.60 (d, $J = 11$ Hz, 9H, P*Me*$_3$). The methyl group locating *cis* to the PMe$_3$ ligand was obscure due to overlapping to the incorporated THF. $^{19}F\{^1H\}$ NMR (C_6D_6): δ −32.2 (br s, C*F*$_3$). $^{31}P\{^1H\}$ (C_6D_6): δ −3.8 (q, $J = 6$ Hz, *P*Me$_3$).

2.4. Reactions of $Au(OR)L$ ($L = PCy_3$, $OR = OCH(CF_3)_2$ (**1a**); $L = PPh_3$, $OR = OCH(CF_3)_2$ (**1c**); $OR = OPh$ (**1d**)) with Me_3SiCCl_3

To a benzene (5 ml) solution of $Au[OCH(CF_3)_2](PCy_3)$ (**1a**) (101.0 mg, 0.157 mmol) was added Me_3SiCCl_3 (89.2 mg, 0.464 mmol). The solution was stirred at r.t. for 3 days and the solvent was removed in vacuo to give white powder, which was washed with hexane and dried in vacuo. Recrystallization of the powder from toluene gave white crystals of $Au(CCl_3)(PCy_3)$ (**5a**). Yield 65.5 mg (70%) [16]. $Me_3SiOCH(CF_3)_2$ 0.109 mmol (69%). ^1H NMR (C_6D_6): δ 0.7–1.7 (m, P*Cy*$_3$). $^{31}P\{^1H\}$ (C_6D_6): δ 50.9 (s, *P*Cy$_3$).

Reaction of **1c** or **1d** with Me_3SiCCl_3 was carried out analogously. The yields, spectroscopic data, and physical data of the resulting complexes are summarized below.

Reaction of $Au[OCH(CF_3)_2](PPh_3)$ (**1c**) (49.2 mg, 0.0763 mmol) with Me_3SiCCl_3 (17.8 mg, 0.0925 mmol) gave $Au(CCl_3)(PPh_3)$ (**5b**) [16]. Yield 8.5 mg (19%). $Me_3SiOCH(CF_3)_2$ 0.0258 mmol (34%). ^1H NMR (C_6D_6): δ 6.9–7.2 (m, P*Ph*$_3$). $^{31}P\{^1H\}$ NMR (C_6D_6): δ 33.7 (s, *P*Ph$_3$).

Reaction of $Au(OPh)(PPh_3)_3$ (**1d**) (9.5 mg, 0.056 mmol) with Me_3SiCCl_3 (13.9 mg, 0.0723 mmol) also gave **5b**. Yield 9.5 mg (28%). Me_3SiOPh 0.032 mmol (57%).

2.5. Reactions of $Cu(OR)(PPh_3)_3$ ($OR = OCH(CF_3)_2$ (6a), OPh (6b)) with Me_3SiCF_3

To a THF (5 ml) solution of $Cu[OCH(CF_3)_3](PPh_3)_3$ (**6a**) (100 mg, 0.0983 mmol) was added Me_3SiCF_3 (0.31 M THF solution, 0.330 ml, 0.10 mmol). The solution was stirred at 50°C for 12 h. $Me_3SiOCH(CF_3)_2$ 0.0966 mmol (98%) was detected in solution. Removal of volatile matters in vacuo gave white powder, which was washed with cold hexane and dried in vacuo. Recrystallization of the white powder from THF gave white crystals of $Cu(CF_3)(PPh_3)_3$ (**7**). Yield 104 mg (92%). Compound **7** was also prepared from the reaction of $Cu(OPh)(PPh_3)_3$ (**6b**) (104 mg, 0.113 mmol) with Me_3SiCF_3 (0.31 M THF solution, 0.550 ml, 0.17 mmol). Yield 30.5 mg (30%). Me_3SiOPh 0.0935 mmol (83%). ^1H NMR (C_6D_6): δ 6.52–7.89 (m, P*Ph*). $^{19}F\{^1H\}$ NMR (C_6D_6): δ −23.3 (br s, C*F*$_3$). $^{31}P\{^1H\}$(C_6D_6): δ −1.59 (br s, *P*Ph$_3$). IR (KBr): 2998, 2903, 1421, 1189, 1052, 882 cm^{-1}. M.p. (dec.): 181–182°C. *Anal.* Calc. for $C_{55}H_{45}CuF_3P_3$: C, 71.85; H, 4.93. Found: C, 71.82; H, 5.04%.

2.6. Solvent effect for the reaction of $Au(OPh)(PPh_3)$ (1d) or cis-$AuMe_2(OPh)(PMe_3)$ (3i) with Me_3SiCF_3

The initial rates for the reactions of **1d** (10.0 mg, 0.0181 mmol) with Me_3SiCF_3 (0.33 M, 0.0550 ml, 0.0182 mmol) at 30°C in toluene-d_8, THF-d_8, and DMSO-d_6 (0.545 ml) obtained from ^1H NMR were 6.0×10^{-6}, 6.2×10^{-6}, and 5.8×10^{-6} M s^{-1}, respectively. Compound **2b** and Me_3SiOPh were formed quantitatively for all reactions.

The initial rates in toluene-d_8, THF-d_8, and DMSO-d_6 (0.520 ml) for the reactions of **3i** (10.0 mg, 0.0252 mmol) with Me_3SiCF_3 (0.33 M, 0.0800 ml, 0.0264 mmol) at 30°C were 4.3×10^{-6}, 4.0×10^{-6}, or 4.3×10^{-6} M s^{-1}, respectively. Compound **4i** and Me_3SiOPh were formed quantitatively for all reactions.

3. Results and discussion

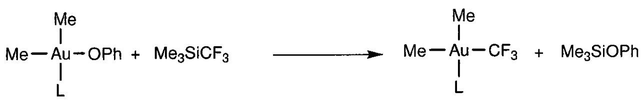
Reaction of alkoxogold(I) complex $Au[OCH(CF_3)_2]$-(PCy_3) (**1a**) with trifluoromethyltrimethylsilane Me_3Si-CF_3 in THF resulted in the C–Si bond cleavage giving trifluoromethylgold(I) complex $Au(CF_3)(PCy_3)$ (**2a**) quantitatively with concomitant formation of silyl ether $Me_3SiOCH(CF_3)_2$ [13] at r.t. within 2 h. $^{31}P\{^1H\}$ NMR spectrum of **2a** shows a quartet signal assignable to PCy_3 ligand at 53.5 ppm due to coupling to three F nuclei ($J_{P-F} = 41$ Hz) of the CF_3 group. Consistently, the $^{19}F\{^1H\}$ NMR spectrum shows a doublet at −29.0 ppm coupled to a P nucleus ($J_{F-P} = 41$ Hz). The IR spectrum of **2a** shows two intensive bands due to C–F stretching vibration at 992 and 1120 cm^{-1}. These spectroscopic data show the formation of **2a** by the sp^3 C–Si bond cleavage (Eq. (1)).

$$\underset{\mathbf{1}}{Au(OR)L} + Me_3SiCF_3 \longrightarrow \underset{\mathbf{2}}{Au(CF_3)L} + Me_3SiOR \qquad (1)$$

1a: L = PCy$_3$, R = OCH(CF$_3$)$_2$
1b: L = PCy$_3$, R = OPh
1c: L = PPh$_3$, R = OCH(CF$_3$)$_2$
1d: L = PPh$_3$, R = OPh
1e: L = PMe$_2$Ph, R = OCH(CF$_3$)$_2$
1f: L = PMe$_3$, R = CH(CF$_3$)$_2$

2a: L = PCy$_3$
2b: L = PPh$_3$
2c: L = PMe$_2$Ph
2d: L = PMe$_3$

Treatments of other alkoxo- or aryloxogold(I) complexes having a triphenylphosphine, dimethylphenylphosphine, or trimethylphosphine ligand Au(OR)L (L = PCy$_3$, OR = OPh (**1b**), L = PPh$_3$, OR = OCH(CF$_3$)$_2$ (**1c**), OPh (**1d**); L = PMe$_2$Ph, OR = OCH(CF$_3$)$_2$ (**1e**); L = PMe$_3$, OR = OCH(CF$_3$)$_2$ (**1f**)) with Me_3SiCF_3 also resulted in the sp^3 C–Si bond cleavage to form the corresponding trifluoromethylgold(I) complex Au(CF$_3$)L (L = PPh$_3$ (**2b**), PMe$_2$Ph (**2c**), PMe$_3$ (**2d**)) [14] (Eq. (2)).



$$Me-\overset{Me}{\underset{L}{Au}}-OPh + Me_3SiCF_3 \longrightarrow Me-\overset{Me}{\underset{L}{Au}}-CF_3 + Me_3SiOPh$$

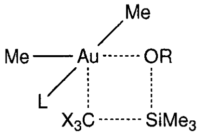
3 **4**

3f: L = PMePh$_2$
3g: L = PEt$_3$
3h: L = PMe$_2$Ph
3i: L = PMe$_3$

4f: L = PMePh$_2$
4g: L = PEt$_3$
4h: L = PMe$_2$Ph
4i: L = PMe$_3$

(2)

Unfortunately these alkoxogold(I) complexes did not cause other C–Si bond cleavage such as tetramethylsilane, phenyltrimethylsilane, vinyltrimethylsilane, nor allyltrimethylsilane.

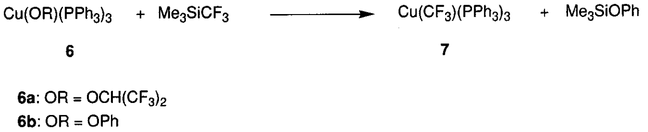


Scheme 1.

On the other hand, alkoxo- or aryloxogold(III) complexes with a triphenylphosphine or tricyclohexylphosphine ligand, *cis*-$AuMe_2[OCH(CF_3)_2]L$ (L = PPh_3 (**3a**); PCy_3 (**3b**)) or *cis*-$AuMe_2(OPh)L$ (P^iPr_3 (**3c**); PPh_3 (**3d**); PCy_3 (**3e**)), showed no reactivity toward Me_3SiCF_3 at all under similar reaction conditions. However, the analogous aryloxogold complex having a $PMePh_2$ (**3f**), PEt_3 (**3g**), PMe_2Ph (**3h**), or PMe_3 (**3i**) ligand reacted with Me_3SiCF_3 in benzene or THF in 1 h at r.t. to form *cis*-$AuMe_2(CF_3)L$ (L = $PMePh_2$ (**4f**), PEt_3 (**4g**), PMe_2Ph (**4h**), or PMe_3 (**4i**)) [15]. For gold(III) complexes, more compact phosphine seems to enhance the C–Si bond cleavage reaction. The observed large steric effect of ancillary phosphine ligand suggests that the gold center is also taking part in the rate determining step of the C–Si bond cleavage reaction. In addition, the rates of the reaction were approximately the same in solvents with different dielectric constants such as toluene, THF and DMSO, suggesting that the transition state of the reaction is not polarized. Therefore, the following four-center concerted intermediate has been proposed (Scheme 1).

Analogous reactions of **1c**, **1d** and **1e** with trimethyltrichlorosilane Me_3SiCCl_3 also proceeded to give $Au(CCl_3)L$ (L = PCy_3 (**5a**); PPh_3 (**5b**)) [16] and Me_3SiOR in benzene for 72 h at r.t. However, they decomposed gradually to AuClL in benzene at r.t., although the mechanism and the fate of CCl_2 moiety were not clear.

Similar reactions of alkoxo- and aryloxocopper complexes $Cu(OR)(PPh_3)_3$ (OR = $OCH(CF_3)_2$ (**6a**), OPh (**6b**)) with Me_3SiCF_3 remained unreacted completely at r.t. However, by heating the reaction mixture at 50°C gave $Cu(CF_3)(PPh_3)_3$ (**7**) and Me_3SiOR in 12 h (Eq. (3)).



$$\underset{\mathbf{6}}{Cu(OR)(PPh_3)_3} + Me_3SiCF_3 \longrightarrow \underset{\mathbf{7}}{Cu(CF_3)(PPh_3)_3} + Me_3SiOPh \qquad (3)$$

6a: OR = $OCH(CF_3)_2$
6b: OR = OPh

In summary, alkoxo- and aryloxogold (or -copper) smoothly cleave the C–Si bond of trihalomethyltrimethylsilanes to give (trihalomethyl)gold (or copper) complexes with the liberation of corresponding silyl ethers. The present results reflect the nucleophilicity of these Group 11 metal alkoxides and the oxophilicity of the Si atom.

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