Structural Diversity for Phosphine Complexes of Stibenium and Stibinidenium Cations

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Supporting Information

Experimental

General: Reactions were carried out in an Innovative Technologies glovebox under atmosphere of dry N_2. CH_2Cl_2 and pentane were purified on an MBraun solvent purification system and stored over 4 Å molecular sieves. MeCN was purchased from Aldrich, distilled from CaH_2 under an atmosphere of argon and stored over 4 Å molecular sieves. Et_2O was distilled from sodium/benzophenone under an atmosphere of argon and stored over 4 Å molecular sieves. Deuterated solvents were purchased from Aldrich and dried using 4 Å molecular sieves. The diphosphines dmpm, dppm, dmpe and dppe were obtained from Aldrich and used as received. $AlCl_3$ and $SbCl_3$ were purchased from Aldrich and sublimed before use. $Me_3SiOSO_2CF_3$ and PMe_3 were purchased from Aldrich and distilled prior to use.

NMR spectra were obtained at room temperature, unless otherwise stated, on a Bruker AVANCE 500 ^{1}H (500.13 MHz, 11.7 T) and Bruker AVANCE 300 ^{1}H (300.15 MHz, 7.02 T). Chemical shifts (δ) are reported in ppm. ^{13}C (125.76 MHz) chemical shifts are referenced to δ_{TMS}

= 0.00 ppm, ^{31}P (202.46 MHz, 121.56 MHz) chemical shifts are referenced to $\delta_{H3PO4\ (85\%)}$ = 0.00 ppm. ^{19}F (282.44 MHz) chemical shifts are referenced to $\delta_{CF3C6H3\ (0.5\%)}$ = 0.00 ppm. Spectra were obtained on aliquots of reaction mixture in appropriate deuterated solvent in a 5 mm tube. The tubes were capped and sealed with Teflon and Parafilm prior to removal from the inert atmosphere.

Raman spectra were obtained in sealed capillaries under N_2 at room temperature. Peaks are reported in wavenumbers (cm^{-1}) with ranked intensities in parentheses, where a value of 100 is indicative of the most intense peak in the spectrum. Melting points were recorded on an Electrothermal apparatus in sealed capillary tubes under N_2. Elemental analyses of selected samples were performed by Canadian Microanalytical Services Ltd. Delta, British Columbia, Canada.

Isolation and characterization of [dmpmSbCl$_2$] **1a** [SO$_3$CF$_3$]

SbCl$_3$ (0.228 g, 1.00 mmol) and TMSOTf (0.223 g, 1.00 mmol) were combined in CH$_2$Cl$_2$ (7 mL) and stirred for 30 minutes to yield a clear and colourless solution. A solution of dmpm (0.136 g, 1.00 mmol) in CH$_2$Cl$_2$ (7 mL) was added drop-wise over 30 seconds. A white precipitate was immediately observed. The reaction mixture was stirred overnight to obtain a light yellow suspension. Removal of volatiles under dynamic vacuum at room temperature over 4 h yielded a light yellow powder, which was washed with CH$_2$Cl$_2$ (3 x 1 mL) and MeCN (3 x 1 mL), and spectroscopically assigned as [dmpmSbCl$_2$] **1a** [SO$_3$CF$_3$]; Yield: 0.400 g, 84 %; mp 166 °C dec.; Raman: 83.8 (62), 114.2 (46), 151.8 (23), 169.2 (30), 196.2 (34), 216.9 (35), 255.5 (46), 276.7 (49), 310.9 (58), 338.4 (16), 350.9 (16), 364.9 (35), 511 (2), 573.2 (6), 615.6 (5), 656.1 (10), 694.7 (7), 725.1 (4), 743.9 (13), 761.2 (28), 1025.9 (45), 1162.3 (4), 1224.5 (8), 1298.8 (4), 1407.3 (9), 2770.7 (1), 2856 (25), 2910.5 (100), 2918.2 (36), 2972.7 (16), 3000.7 (15.3), 3012.2 (10).; ^{1}H NMR (CD$_3$CN, 300 MHz, 293 K): 2.13(*m*, 12H), 4.05 (*t*, 2H, $^2J_{PH}$ = 12 Hz); ^{19}F NMR (CD_3CN, 282.44 MHz, 293 K): -79.3 (*s*); $^{31}P\{^1H\}$ NMR (CD_3CN, 121.56 MHz, 293 K): 1.4 (*s*).

Isolation and characterization of [dppmSbCl_2] **1b** [AlCl_4]

$SbCl_3$ (1.140 g, 5.00 mmol) and $AlCl_3$ (0.666 g, 5.00 mmol) were combined in CH_2Cl_2 (45 mL) and stirred for 30 minutes to yield a yellow suspension. A solution of dppm (1.922 g, 5.00 mmol) in CH_2Cl_2 (15 mL) was added drop-wise over one minute and stirred for an additional 40 minutes. The resulting bright-yellow reaction mixture was clear and exhibited one signal in its $^{31}P\{^1H\}$ NMR spectrum (5 ppm). Removal of volatiles under dynamic vacuum at room-temperature over 16 hours yielded a bright-yellow powder (3.844 g), which was re-dissolved in CH_2Cl_2 (12 mL), filtered, layered with pentane (3 mL) and stored in the freezer at -30 °C. Large, yellow block shaped crystals were obtained after 3 days, isolated by decantation and washed with cold pentane (3 x 5 mL) and identified as [dppmSbCl_2] **1b** [AlCl_4]; Yield: 2.913 g, 78 %; mp 115 °C dec.; Elemental Analysis Calcd. (Found): C 40.26 (39.52), H 2.97 (2.89); Raman: 83.8 (81), 116.6 (40), 136.9 (25), 176.4 (50), 205.8 (14), 218.4 (7), 268 (8), 301.3 (72), 329.2 (19), 334.5 (28), 346.1 (17), 407.3 (5), 420.4 (3), 434.8 (2), 452.2 (3), 464.2 (2), 491.7 (5), 493.2 (6), 527.4 (8), 614.2 (9), 671.5 (4), 693.2 (2), 740.9 (2), 748.7 (3), 983 (3), 998.4 (89.9), 1024.9 (37), 1034.1 (6), 1.87.6 (17), 1095.8 (22), 1158.9 (2), 1164.3 (6), 1188.8 (6), 1331.6 (2), 1435.7 (6), 1479.6 (4), 1573.6 (13), 1582.3 (100), 2918.2 (10), 2964.5 (5), 3001.1 (1), 3057.1 (45), 3061.9 (32), 3140.5 (1), 3157.3 (1).; ^{1}H NMR (CD_2Cl_2, 500 MHz, 293 K): 4.31 (*t*, 2H, $^2J_{PH}$ = 9.9 Hz), 7.50 (*t*, 8H, $^3J_{PH}$ = 7.4 Hz), 7.55-7.63 (*m*, 12H); $^{13}C\{^1H\}$ NMR (CD_2Cl_2, 282.44 MHz, 293 K): 23.5 (*t*, $^1J_{PC}$ = 10.4 Hz), 125.0 (*t*, $^1J_{PC}$ = 17.9 Hz), 130.4 (*t*, $^2J_{PC}$ = 5.1 Hz), 133.7 (*s*), 133.8 (*m*); $^{31}P\{^1H\}$ NMR (CD_2Cl_2, 202.46 MHz, 293 K): 5.2 (*s*).

Isolation and characterization of [dmpeSbCl_2] **2a** [SO_3CF_3]

$SbCl_3$ (0.456 g, 2.00 mmol) and TMSOTf (0.446 g, 2.00 mmol) were combined in CH_2Cl_2 (7 mL) and stirred for 30 minutes to yield a clear and colourless solution. A solution of dmpe (0.300 g, 2.00 mmol) in CH_2Cl_2 (7 mL) was added drop-wise over 30 seconds. A mixture of brown and white precipitate formed immediately and the reaction was slightly exothermic. The reaction mixture was stirred for 3 h to yield a dark brown powder under a clear and colourless supernatant. The $^{31}P\{^1H\}$ NMR spectrum of this supernatant exhibited no signals. Removal of volatiles under dynamic vacuum at room temperature over 4 h yielded a brown powder (1.104 g) and the $^{31}P\{^1H\}$ NMR spectrum of this powder in MeCN exhibited one signal (34 ppm). The powder was dissolved in a 1:1 mixture by volume of MeCN and Et_2O and placed in the freezer at -30 °C. Small colorless block shaped crystal were obtained after one week, were isolated by decantation, washed with a cold mixture of MeCN/Et_2O (3 x 1 mL), and characterization as [dmpe$SbCl_2$] **2a** [SO_3CF_3]; Yield (total of three crops over 2 weeks): 0.691 g, 76 %; mp 175-220 °C darkens, 220-230 °C dec.; Elemental Analysis Calcd. (Found): C 17.09 (17.11), H 3.28 (3.02); Raman: decomposition in laser; ^1H NMR (CD_3CN, 500 MHz, 293 K): 2.05 (*d*, 12H, $^2J_{PH} = 14.3$ Hz), 2.72 (*m*, 4H); $^{13}C\{^1H\}$ NMR (CD_3CN, 125.76 MHz, 293 K): 8.9 (*d*, $^1J_{PC} = 36.8$ Hz), 25.1 (*d*, $^1J_{PC} = 32.7$ Hz), 118.4 (*s*); ^{19}F NMR (CD_3CN, 282.44 MHz, 293 K): -79.0 (*s*); $^{31}P\{^1H\}$ NMR (CD_3CN, 202.46 MHz, 293 K): 34.1 (*s*).

Isolation and characterization of [dppe$SbCl_2$] **2b** [$AlCl_4$]•CH_2Cl_2

$SbCl_3$ (0.228 g, 1.00 mmol) and $AlCl_3$ (0.134 g, 1.00 mmol) were combined in CH_2Cl_2 (7 mL) and stirred for 30 minutes to yield a yellow suspension. A solution of dppe (0.399 g, 1.00 mmol) in CH_2Cl_2 (7 mL) was added drop-wise over 30 seconds. The resulting clear and light-yellow solution was stirred for an additional 20 minutes to give a clear orange solution, the $^{31}P\{^1H\}$ NMR spectrum of which exhibited one signal (17 ppm). Removal of volatiles under dynamic vacuum at room-temperature over 4 hours yielded an orange-brown powder (0.736 g), which was recrystallized in CH_2Cl_2 (12 mL) at -30

^{o}C to give a small amount of light-orange crystals after two weeks that were characterized as [dppeSbCl$_2$] **2b** [AlCl$_4$]•CH$_2$Cl$_2$; Yield: 0.135 g, 18%; mp 104 ^{o}C dec.; Raman: decomposition in laser; ^{1}H NMR (CD$_2$Cl$_2$, 500 MHz, 293 K): 3.41 (*m*, 4H), 7.59-7.71 (*m*, 20H); ^{13}C{^{1}H} NMR (CD$_2$Cl$_2$, 125.76 MHz, 293 K): 130.6 (*t*, $^{1}J_{PC}$ = 5.4 Hz), 133.8 (*m*), 133.9 (*s*); ^{31}P{^{1}H} NMR (CD$_2$Cl$_2$, 202.46 MHz, 293 K): 17.5 (*s*). While it was not possible to recover the bulk of the material in this fashion, the NMR spectra of the bulk powder and the crystals were identical.

Isolation and characterization of [(Me$_3$P)$_2$SbCl$_2$] **4** [SO$_3$CF$_3$]

SbCl$_3$ (0.228 g, 1.00 mmol) and TMSOTf (0.222 g, 1.00 mmol) were combined in CH$_2$Cl$_2$ (4 mL) and stirred for 30 minutes to yield a clear and colourless solution. This was added drop-wise to a stirring solution of PMe$_3$ (0.228 g, 3.00 mmol) in CH$_2$Cl$_2$ (4 mL) over 30 seconds to produce a cloudy white suspension. The reaction mixture was allowed to stir for 1 hour yielding a fine white powder and a clear and colourless supernatant, the ^{31}P{^{1}H} NMR spectrum of which exhibited two signals (5.8 ppm, 85% and -2.2 ppm, 15%). Removal of solvent under dynamic vacuum at room temperature over 4 hours yielded a fine white powder (0.515 g), which was recrystallized in MeCN (10 mL) at -30 ^{o}C. Highly-reflective colourless block shaped crystals were obtained after 5 days, isolated by decantation, washed with cold MeCN (3 x 3 mL), and characterized as [(Me$_3$P)$_2$SbCl$_2$] **4** [SO$_3$CF$_3$]; Yield: 0.401 g, 80%; mp: 135 ^{o}C dec.; Elemental Analysis Calcd. (Found): C 17.02 (16.97), H 3.67 (3.41); Raman: 84.8 (37), 115.2 (13), 123.4 (35), 140.7 (43), 184.6 (9), 208.7 (17), 212.1 (14), 250.2 (88), 272.4 (12), 317.7 (7), 350.4 (30), 520.6 (1), 576.1 (10), 674.4 (48), 757.8 (35), 765.6 (26), 1024.9 (85), 1226.9 (5), 1246.2 (3), 1415.9 (8), 1418.3 (8), 1433.3 (4), 2920.6 (100), 2989.6 (18), 2996.3 (31), 3006.9 (22); ^{1}H NMR (CD$_3$CN, 500 MHz, 293 K): 2.08 (*d*, 18H, $^{2}J_{PH}$ = 14.1 Hz); ^{13}C{^{1}H} NMR (CD$_3$CN, 125.76 MHz, 293 K): 11.6 (*d*, $^{1}J_{PC}$ = 34.4 Hz), 118.5 (*s*); ^{19}F NMR (CD$_3$CN, 282.44 MHz, 293 K): -79.1 (*s*); ^{31}P{^{1}H} NMR (CD$_3$CN, 202.46 MHz, 293 K): 6.2 (*s*).

Isolation and characterization of [dppmSbCl] **6b** $[AlCl_4]_2$•CH_2Cl_2•C_5H_{12}

$SbCl_3$ (0.228 g, 1.00 mmol) and $AlCl_3$ (0.532 g, 4.00 mmol) were combined in CH_2Cl_2 (8 mL) and stirred for 12 hours to obtain a light yellow suspension. A solution of dppm (0.384 g, 1.00 mmol) in CH_2Cl_2 (5 mL) was added at once to immediately yield a yellow, cloudy solution. The reaction mixture was allowed to stir for 30 minutes to give a light yellow solution, the $^{31}P\{^1H\}$ NMR spectrum of which exhibited one signal (-3 ppm). The slightly cloudy solution was filtered, concentrated to 4 mL under vacuum, layered with pentane (1 mL) and placed in the freezer at -30 °C. A small amount of shiny transparent crystals formed below an orange oil after two weeks and were identified as **6b**$[AlCl_4]_2$•CH_2Cl_2•C_5H_{12} by X-ray crystallography. The orange oil was washed repeatedly with a 1:10 mixture of Et_2O and hexane and under vacuum gave a light yellow powder characterized as **6b**$[AlCl_4]_2$•Et_2O, Yield: 0.784 g, 85 %; mp 100-104 °C; Elemental Analysis Calcd. (Found): C 36.54 (36.04), H 3.38 (3.22); ^1H NMR (CD_2Cl_2, 500 MHz, 293 K): 3.79 (*t*, 2H, $^2J_{PH} = 7.4$ Hz), 7.35 (*m*, 8H), 7.44 (*m*, 4H), 7.59 (*m*, 8H); $^{31}P\{^1H\}$ NMR (CD_2Cl_2, 202.46 MHz, 293 K): -2.3 (*s*).

Isolation and characterization of [dppeSbCl] **7b** $[Al_2Cl_7]_2$

$SbCl_3$ (0.456 g, 2.00 mmol) and $AlCl_3$ (1.068 g, 8.00 mmol) were combined in CH_2Cl_2 (8 mL) and stirred for 40 minutes to obtain a light yellow suspension. A solution of dppe (0.797 g, 2.00 mmol) in CH_2Cl_2 (5 mL) was added at once to immediately yield a golden-yellow, clear solution. The reaction mixture was allowed to stir at room temperature for 1.5 hours and the resulting clear and yellow solution had a $^{31}P\{^1H\}$ NMR spectrum which exhibited one signal (49 ppm). The reaction mixture was filtered, concentrated to 5 mL under vacuum, layered with pentane (2 mL) and placed in the freezer at -30 °C giving an oil containing a small amount of light orange block crystals that were identified as **7b**$[AlCl_4]_2$ by X-ray crystallography. The orange oil was washed repeatedly with a 1:10 mixture of

Et_2O and hexane and under vacuum gave a light yellow powder characterized as **7b**$[Al_2Cl_7]_2$•Et_2O, Yield: 0.294 g, 12 %; mp 77-79 °C; Elemental Analysis: Calcd. (Found): C 29.20 (28.60), H 2.78 (2.24); Raman: 123 (19), 174.9 (45), 182.2 (24), 191.8 (14), 216.9 (3), 247.3 (2), 269.5 (9), 316.7 (33), 366.8 (13), 370.7 (48), 426.6 (8), 435.3 (13), 457.5 (4), 523.4 (7), 615.5 (11), 682.6 (4), 810.9 (2), 999.38 (100), 1027.8 (32), 1100.1 (24), 1168.1 (7), 1198.5 (1), 1440.5 (4), 1578.9 (16), 1583.7 (76), 2909.5 (11), 2926.4 (5), 2941.4 (12), 2960.6 (4), 3062.4 (23), 3068.6 (40); ^1H NMR (CD_2Cl_2,500 MHz, 293 K): 3.89 (*m*, 4H), 7.68-7.72 (*m*, 8H), 7.81-7.84 (*m*, 8H) , 7.95-7.99 (*m*, 4H); $^{13}C\{^1H\}$ NMR (CD_2Cl_2, 282.44 MHz, 293 K): 26.6 (*d*, $^1J_{PC}$ = 32 Hz), 116.0 (*d*, $^1J_{PC}$ = 59 Hz), 132.3(*s*), 133.9 (*m*), 137.6 (*s*); $^{31}P\{^1H\}$ NMR (CD_2Cl_2, 202.46 MHz, 293 K): 49.1 (*s*).

NMR Detection of $[(Me_3P)_2SbCl][OTf]_2$

$[(Me_3P)_2SbCl_2][OTf]$ (0.400 g, 0.80 mmol) was dissolved in MeCN (4 mL) to give a clear and colourless solution. A solution of excess TMSOTf (0.600 g, 2.70 mmol) in MeCN (4 mL) was added drop-wise over 30 seconds to give a clear and pale-yellow solution which was allowed to stir overnight at room temperature. The $^{31}P\{^1H\}$ NMR spectrum of the one-phase reaction mixture exhibited one signal (12.5 ppm) assigned to $[(Me_3P)_2SbCl][OTf]_2$.

NMR Detection of [dppmSbCl] **6b** $[OTf]_2$

$SbCl_3$ (0.228 g, 1.00 mmol) and TMSOTf (0.666 g, 3.00 mmol) were combined in CH_2Cl_2 (4 mL) and stirred for 40 minutes to yield a clear and colourless solution. This solution was added drop-wise to a solution of dppm (0.384 g, 1.00 mmol) in CH_2Cl_2 (4 mL) over 30 seconds to immediately give a clear and golden-yellow solution. Stirring for 15 minutes yielded a one-phase reaction mixture, the $^{31}P\{^1H\}$ NMR spectrum of which exhibited one signal (-2.6 ppm) assigned to $[dppmSbCl][OTf]_2$.

NMR Detection of [dmpeSbCl] **7a** $[OTf]_2$

$SbCl_3$ (0.228 g, 1.00 mmol) and TMSOTf (1.332 g, 6.00 mmol) were combined in MeCN (4 mL) and stirred for 15 minutes to yield a clear and colourless solution. This solution was added drop-wise to a stirring solution of dmpe (0.300 g, 2.00 mmol) in MeCN (4 mL) over 30 seconds. A yellow colour appeared immediately upon commencing addition, deepened to a dark-orange half-way through the addition, and returned to a clear and golden-yellow colour by the end of the addition. Stirring for 15 minutes yielded a one-phase reaction mixture, the $^{31}P\{^1H\}$ NMR spectrum of which exhibited one signal (59.7 ppm) assigned to [dmpeSbCl][OTf]$_2$.

Structures were solved by direct methods and refined using full matrix least squares on F2. (G. M. Sheldrick, Acta Crystallogr., Sect. A: Found. Crystallogr., 2008, A64, 112–122.)

Refinement details are summarized in Table S1.

Table S1 Crystal data for [dppmSbCl$_2$] **1b** [AlCl$_4$], [dmpeSbCl$_2$] **2a** [SO$_3$CF$_3$], [dppeSbCl$_2$] **2b** [AlCl$_4$]•CH$_2$Cl$_2$, [(Me$_3$P)$_2$SbCl$_2$] **4** [SO$_3$CF$_3$], [dppmSbCl] **6b** [AlCl$_4$]$_2$ and [dppeSbCl] **7b** [Al$_2$Cl$_7$]$_2$•CH$_2$Cl$_2$•C$_5$H$_{12}$.

	[dppmSbCl$_2$] **1b** [AlCl$_4$]	[dmpeSbCl$_2$] **2a** [OTf]	[dppeSbCl$_2$] **2b** [AlCl$_4$]•CH$_2$Cl$_2$	[(Me$_3$P)$_2$SbCl$_2$] **4**[OTf]
Empirical Formula	$C_{25}H_{22}AlCl_6P_2Sb$	$C_7H_{16}Cl_2F_3O_3P_2SSb$	$C_{27}H_{26}AlCl_8P_2Sb$	$C_7H_{18}Cl_2F_3O_3P_2SSb$
Formula Weight	745.80	491.85	844.75	493.86
Crystal System	triclinic	orthorhombic	triclinic	monoclinic
Space Group	$P\bar{1}$ (No. 2)	*P*212121 (No. 19)	$P\bar{1}$ (No. 2)	*C*2/*c* (No. 15)
***a* (Å)**	10.3131 (2)	11.1129 (3)	10.5270 (3)	25.4578 (11)
***b* (Å)**	11.7645 (3)	11.3226 (3)	12.8155 (3)	6.4594 (3)
***c* (Å)**	13.2776 (3)	13.0683 (4)	14.0046 (3)	22.9155 (10)
α (deg)	81.5321 (3)	90	74.3015 (3)	90
β (deg)	73.3679 (3)	90	71.3838 (3)	113.8940 (4)
γ (deg)	86.3619 (3)	90	87.5567 (3)	90
V (Å^3)	1526.32 (6)	1644.34 (8)	1721.72 (7)	3445.3 (3)
Dc (g cm^{-3})	1.623	1.987	1.629	1.904
Radiation, λ (Å)	Graphite-monochromatic Mo K*α* (0.71073)	Graphite-monochromatic Mo K*α* (0.71073)	Graphite-monochromatic Mo K*α* (0.71073)	Graphite-monochromatic Mo K*α* (0.71073)
Temp (K)	173	173	173	173
GoF	1.023	1.117	1.021	1.116
R1	0.0200	0.0131	0.0236	0.0156
***w*R2**	0.0480	0.0329	0.0582	0.0400

Table S1 continued. Crystal data for [dppmSbCl_2] **1b** [AlCl_4], [dmpeSbCl_2] **2a** [SO_3CF_3], [dppeSbCl_2] **2b** [AlCl_4]•CH_2Cl_2, [$(Me_3P)_2SbCl_2$] **4** [SO_3CF_3], [dppmSbCl] **6b** $[AlCl_4]_2$ and [dppeSbCl] **7b** $[Al_2Cl_7]_2$•CH_2Cl_2•C_5H_{12}.

	[dppmSbCl] **6b** $[AlCl_4]_2$•CH_2Cl_2•C_5H_{12}	[dppeSbCl_2] **7b** $[Al_2Cl_7]_2$
Empirical Formula	$C_{31}H_{36}Al_2Cl_9P_2Sb$	$C_{26}H_{24}Al_4Cl_{15}P_2Sb$
Formula Weight	1090.16	1159.81
Crystal System	Monoclinic	Triclinic
Space Group	$P2_1/n$	$P\bar{1}$ (No. 2)
***a* (Å)**	16.299 (2)	12.2162 (7)
***b* (Å)**	10.3844 (15)	12.2162 (7)
***c* (Å)**	25.251 (4)	17.8399 (11)
α (deg)	90	72.2279 (7)
β (deg)	98.272 (2)	73.7807 (7)
γ (deg)	90	63.2388 (7)
V (Å^3)	4229.3 (10)	2240.2 (2)
Dc (g cm^{-3})	1.712	1.719
Radiation, λ (Å)	Graphite-monochromatic Mo Kα (0.71073)	Graphite-monochromatic Mo Kα (0.71073)
Temp (K)	173	173
GoF	1.063	1.062
R1	0.0479	0.0262
***w*R2**	0.1257	0.0652