Donor-Stabilized Cations and Imine Transfer from N-Silylphosphoranimines

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Supporting Information

Experimental Section

All reactions and manipulations were carried out under an atmosphere of prepurified nitrogen or argon (Air Products) using common Schlenk techniques or an inert atmosphere glove box (M-Braun). Solvents were dried and collected using a Grubbs-type solvent purification system manufactured by M-Braun.[1] ^{1}H and $^{31}P\{^{1}H\}$ NMR spectra were obtained on a Varian Gemini 300 spectrometer (300.1 and 121.5 MHz) and were referenced either to protic impurities in the solvent (^{1}H) or externally to 85 % H_3PO_4 ($^{31}P\{^{1}H\}$) in $CDCl_3$, CD_2Cl_2 or D_2O (insert). $^{13}C\{^{1}H\}$ and $^{29}Si\{^{1}H\}$ NMR spectra were obtained on a Varian Unity 400 spectrometer (100.5 and 79.4 MHz) and were both referenced externally to $SiMe_4$ in $CDCl_3$. Mass spectra were obtained with the use of a VG-250S mass spectrometer using a 70 eV electron impact ionization source. Melting points (uncorrected) were obtained in 0.5 mm (o.d.) glass capillaries which were flame sealed under nitrogen. Elemental analyses were performed at the University of Toronto using a Perkin-Elmer 2400 Series CHN Analyzer. 4-Dimethylaminopyridine (DMAP) was obtained from Aldrich and used as received. The silver salts Ag[OTf], Ag[BF_4] and Ag[SbF_6] were also obtained from Aldrich and were dried under dynamic vacuum at 100 °C for 24 h prior to use. Triphenylphosphine (BDH Chemicals) and tetra-n-octylammonium bromide (Aldrich) were dried *in vacuo* for 24 h prior to use. [Ph_3P=N=PPh_3]Cl (Aldrich) was recrystallized from CH_2Cl_2 and dried *in vacuo* at 100 °C for 16 h before use. Tri-n-butylphosphine (Aldrich) was vacuum distilled and stored under an atmosphere of nitrogen. Cl_3P=$NSiMe_3$ (**1**) was prepared according to a literature procedure.[2]

Xray Structure Determination of [3]OTf. Data were collected on a Nonius Kappa-CCD diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). A combination of 1° ϕ and ω (with κ offsets) scans were used to collect sufficient data. The data frames were integrated and scaled using the Denzo-SMN package.[3] The structures were solved and refined with the SHELXTL-PC v6.12 software package.[4] Refinement was by full-matrix least squares on F^2 using data (including negative intensities) with hydrogen atoms bonded to carbon atoms included in calculated positions and treated as riding atoms.

Preparation of [DMAP•Cl_2P=$NSiMe_3$]OSO_2CF_3, [3]OTf. To a mixture of DMAP (1.06 g, 8.67 mmol) and Ag[OSO_2CF_3] (2.21 g, 8.63 mmol) in 20 mL of CH_2Cl_2 was added dropwise a 3 mL solution of Cl_3P=$NSiMe_3$ (1.94 g, 8.63 mmol) in CH_2Cl_2 at 25 °C (in the absence of light). A white precipitate formed immediately and the reaction was stirred for 1 h. The reaction mixture was then filtered and slow evaporation of the filtrate under nitrogen (16 h) afforded large colorless needles of **[3]OTf** (3.26 g, 82 %). Attempts to generate a stable phosphoranimine cation by the treatment of **1** with Ag[OTf] in the presence of pyridine, bipyridine or without base, led to the formation of poly(dichlorophosphazene) [^{31}P NMR ($CDCl_3$): δ = -18.0 ppm (s)] and Me_3SiOTf [^{1}H NMR ($CDCl_3$): δ = 0.51 ppm (s); ^{19}F NMR ($CDCl_3$): δ = -78.0 ppm (s)].

Data for **[3]OTf**: $^{31}P\{^{1}H\}$ NMR ($CDCl_3$): δ = -39.8 ppm (s). ^{1}H NMR ($CDCl_3$): δ = 0.30 (s, $SiMe_3$, 9H), 3.51 (s, NMe_2, 6H), 7.31 (dd, J = 2.7 and 8.4 Hz, *ortho-* ArH, 2H) and 8.46 ppm (dd, J = 7.4 and 10.7 Hz, *meta*-ArH, 2H). $^{13}C\{^{1}H\}$ NMR ($CDCl_3$): δ = 1.7 (d, $^{3}J_{CP}$ = 5.0 Hz, $SiMe_3$), 41.5 (s, NMe_2), 108.9 (d, $^{2}J_{CP}$ = 8.0 Hz, *ortho*-C (DMAP)), 120.5 (q, $^{1}J_{CF}$ = 166.1 Hz, OTf), 138.7 (d, $^{3}J_{CP}$ = 5.0 Hz, *meta*-C (DMAP)) and 157.8 ppm (s, *para*-C (DMAP)). ^{19}F NMR ($CDCl_3$): δ = -78.2 ppm (s, OTf). $^{29}Si\{^{1}H\}$ NMR ($CDCl_3$): δ = 2.5 ppm (d, $^{2}J_{SiP}$ = 11.9 Hz). mp (°C): 98-102 (dec). EI-MS (70 eV, *m/z*, %): 312 (M^+ - OTf, 2), 208 (Cl_3P=$NSiMe_3$ - Me, 1), 189 (Cl_2P=$NSiMe_3$, 4), 155 (ClP=$NSiMe_3$, 8), 121 ($DMAP^+$, 100), 69 (CF_3^+, 64). Anal. Calcd. for $C_{11}H_{19}Cl_2F_3N_3O_3PSSi$ (460.3): %C: 28.70; %H: 4.16; %N: 9.13. Found: %C: 28.50; %H: 3.98; %N: 9.05.

Preparation of [DMAP•Cl_2P=$NSiMe_3$]Cl, [3]Cl. To a solution of Cl_3P=$NSiMe_3$ (93 mg, 0.41 mmol) in 1 mL of CH_2Cl_2 was added dropwise a 0.5 mL solution of DMAP (47 mg, 0.38 mmol) in CH_2Cl_2. The resulting colorless solution was stirred for 16 h and the volatiles (including any excess Cl_3P=$NSiMe_3$) were removed *in vacuo* to give a white solid (110 mg, 82 %).
$^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = -39.2 ppm (s). ^1H NMR ($CDCl_3$): δ = 0.08 (s, $SiMe_3$, 9H), 3.46 (s, NMe_2, 6H), 7.65 (dd, *ortho*-ArH, 2H, J = 3.0 and 8.1 Hz) and 8.37 ppm (dd, *meta*-ArH, 2H, J = 8.4 and 10.1 Hz). $^{13}C\{^1H\}$ NMR ($CDCl_3$): δ = 1.7 (d, $^3J_{CP}$ = 4.5 Hz, $SiMe_3$), 42.1 (s, NMe_2), 109.7 (d, $^2J_{CP}$ = 8.6 Hz, *ortho*-C (DMAP)), 138.6 (d, $^3J_{CP}$ = 5.9 Hz, *meta*-C (DMAP)) and 157.8 ppm (s, *para*-C (DMAP)). $^{29}Si\{^1H\}$ NMR ($CDCl_3$): δ = 2.4 ppm (d, $^2J_{SiP}$ = 11.1 Hz).

To investigate the equilibrium nature of **[3]Cl**, sequential amounts of chloride ion, as [Ph_3P=N=PPh_3]Cl, was added to a freshly prepared solution of **[3]Cl** in $CDCl_3$ (see Table 1). One equiv. of bromide ion, [$^nOct_4N$]Br, did not react with the **[3]**$^+$ cation.

Preparation and Decomposition of [DMAP•Cl_2P=$NSiMe_3$]BF_4, [3]BF_4. In the absence of light, a solution of Cl_3P=$NSiMe_3$ (0.28 g, 0.12 mmol) in 1 mL of dichloromethane was added to a mixture of DMAP (0.15 g, 0.12 mmol) and Ag[BF_4] (0.25 g, 0.13 mmol) in 2 mL of CH_2Cl_2. The reaction was stirred for 1 h to give a white suspension. The AgCl was filtered off and the volatiles were removed from the filtrate to give a white solid (0.31 g, 62 %) which was identified as **[3]BF_4** by NMR spectroscopy. $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = -39.8 (s) ppm. ^1H NMR ($CDCl_3$): δ = 0.25 (s, 9H, $SiMe_3$), 3.44 (s, 6H, NMe_2), 7.20 (br, 2H, *ortho* ArH) and 8.40 ppm (br, *meta* ArH). ^{19}F NMR ($CDCl_3$): δ = -151.4 ppm (pseudoquartet, BF_4^-).

Compound **[3]BF_4** gradually decomposed over the period of 3 days in $CDCl_3$ to give [Cl_2P=N]$_3$ and DMAP•BF_3 (see below).
Data for decomposition products: $^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = 20.0 ppm (s, [Cl_2P=N]$_3$). ^1H NMR ($CDCl_3$): δ = 3.20 (s, 6H, NMe_2), 6.66 (d, 2H, *ortho* ArH, J = 7.0 Hz) and 8.1 ppm (br, *meta* ArH). ^{19}F NMR ($CDCl_3$): δ = -151.6 ppm (m). ^{11}B NMR ($CDCl_3$): δ = -0.02 (br). Lit. for DMAP•BF_3:[5] ^1H NMR ($CDCl_3$): δ = 3.16 (s), 6.61 (d, J = 7.0 Hz) and 8.09 ppm (br). ^{19}F NMR ($CDCl_3$): δ = -152.7 ppm (m). ^{11}B NMR ($CDCl_3$): δ = 0.34 ppm (br).

In Situ Preparation of [DMAP•Cl_2P=$NSiMe_3$]SbF_6, [3]SbF_6. In the absence of light, a 0.5 mL solution of **1** (90 mg, 0.40 mmol) in $CDCl_3$ was added quickly to a stirred mixture of Ag[SbF_6] (138 mg, 0.40 mmol) and DMAP (49 mg, 0.40 mmol) in 1 mL of $CDCl_3$. The resulting grey suspension was stirred for 1 h and the AgCl was filtered off. Analysis of the resulting colorless filtrate by NMR indicated the formation of **[3]SbF_6** (95 % pure; trace of unreacted **1**, ^{31}P: -54.0 ppm (s), was present). Compound **[3]SbF_6** was stable in solution for up to two weeks without any noticeable sign of decomposition.
$^{31}P\{^1H\}$ NMR ($CDCl_3$): δ = -40.2 ppm (s). ^1H NMR ($CDCl_3$): δ = 0.24 (s, 9H, $SiMe_3$), 3.38 (s, 6H, NMe_2), 7.04 (dd, 2H, *ortho* ArH, J = 3.2 and 7.1 Hz) and 8.34 ppm (dd, *meta* ArH, J = 8.1 and 11.1 Hz). ^{19}F NMR ($CDCl_3$): δ = -110 (very broad) and –135 ppm (very broad) (SbF_6^-).

Preparation of Ph_3P=N-PCl_2 (7) from Ph_3P and Cl_3P=$NSiMe_3$. A solution of Ph_3P (0.34 g, 1.3 mmol) in 3 mL of CH_2Cl_2 was added to a solution of **1** (0.29 g, 1.3 mmol) in 2 mL of CH_2Cl_2. The reaction was stirred for 3 h and removal of the volatiles afforded a white solid which was characterized as the previously known phosphoranimine Ph_3P=N-PCl_2 (**7**)[6] (0.43 g, 89 %). Repeating the reaction in $CDCl_3$ also identified $ClSiMe_3$ as a byproduct [^1H NMR: δ = 0.45 ppm (s)].

Data for **7**: ^{31}P NMR ($CDCl_3$): δ = 165.6 (d, $^2J_{PP}$ = 75.0 Hz, -*P*Cl_2) and 15.8 ppm (d, $^2J_{PP}$ = 75.0 Hz, Ph_3*P*=N-). ^{1}H NMR ($CDCl_3$): δ = 7.5 – 8.0 ppm (m).

In Situ Preparation of $^nBu_3P$=N-PCl_2 (8) from $^nBu_3P$ and Cl_3P=$NSiMe_3$. To a solution of **1** (0.52 g, 2.3 mmol) in 5 mL of CH_2Cl_2 was added $^nBu_3P$ (0.60 mL, 2.4 mmol) dropwise. Analysis of the resulting yellow solution after 2 h revealed the formation of PCl_3 (δ = 218.0 ppm (s); *ca.* 5 %), $^nBu_3P$=$NSiMe_3$ (δ = -8.8 ppm (s); *ca.* 5 %), $^nBu_3P$=N-PCl_2 (**8**) [δ = 158.0 (d, $^2J_{PP}$ = 85.0 Hz, -*P*Cl_2) and 40.7 ppm (d, $^2J_{PP}$ = 85.0 Hz, $^nBu_3$*P*=N-); 1:1 ratio; *ca*. 55 %] and an unidentified species at δ = 105.1 (s, *ca.* 25 %) and 35.1 ppm (s, *ca.* 10 %). Stirring the reaction for further 6 days gave an 80 % (*in situ*) yield of $^nBu_3P$=N-PCl_2[7] and unknown species (*ca.* 20 %) with ^{31}P NMR resonances at δ = 72 (br) and 35 ppm (s). Attempts to isolate pure **8** have yet to be successful.

References:

(1) Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.

(2) Wang, B.; Rivard, E.; Manners, I. *Inorg. Chem.* **2002**, *41*, 1690.

(3) Otwinowski, Z.; Minor, W. *Methods Enzymol.* **1997**, *276*, 367.

(4) Sheldrick, G. M. SHELXTL-Windows NT. V6.12, Bruker Analytical X-Ray Systems Inc., Madison, WI, 2001.

(5) Lesley, M. J. G.; Woodward, A.; Taylor, N. J.; Marder, T. M.; Cazenobe, I.; Ledoux, I.; Zyss, J.; Thornton, A.; Bruce, D. W.; Kakkar, A. K. *Chem. Mater.* **1998**, *10*, 1355.

(6) Riesel, L.; Friebe, R. *Z. Anorg. Allg. Chem.* **1981**, *474*, 105

(7) Riesel, L.; Friebe, R. *Z. Anorg. Allg. Chem.* **1991**, *604*, 85.

Table 1 Equilibrium between **[3]Cl** and free **1** and DMAP in the presence of added chloride ion[a]

Molar equiv. of chloride[c]	Mol % of **[3]Cl**[b]	Mol % of $Cl_3P=NSiMe_3$[b]
0	95	5
0.25	80	20
0.5	60	40
0.75	25	75
1.0	0	100

[a]As determined by ^{31}P NMR spectroscopy.[b] Initial concentration of **[3]Cl** (0.3 – 0.4 M); reaction time, 1.5 h.[c] Source of chloride: $[Ph_3P=N=PPh_3]Cl$.

Table 2. Crystal data and structure refinement for **[3]OTf**.

Identification code	k0384	
Empirical formula	C11 H19 Cl2 F3 N3 O3 P S Si	
Formula weight	460.31	
Temperature	150(1) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.0840(4) Å	α= 88.5630(19)°.
	b = 8.6430(4) Å	β= 86.5950(18)°.
	c = 14.6080(7) Å	γ = 85.038(2)°.
Volume	1014.84(8) Å^3	
Z	2	
Density (calculated)	1.506 Mg/m^3	
Absorption coefficient	0.602 mm^{-1}	
F(000)	472	
Crystal size	0.24 x 0.16 x 0.08 mm^3	
Theta range for data collection	2.77 to 27.54°.	
Index ranges	-10<=h<=10, -11<=k<=11, -18<=l<=18	
Reflections collected	13104	
Independent reflections	4659 [R(int) = 0.0986]	
Completeness to theta = 27.54°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.970 and 0.783	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4659 / 0 / 232	
Goodness-of-fit on F^2	0.987	
Final R indices [I>2sigma(I)]	R1 = 0.0550, wR2 = 0.1243	
R indices (all data)	R1 = 0.1090, wR2 = 0.1513	
Extinction coefficient	0.005(2)	
Largest diff. peak and hole	0.451 and -0.535 e.Å^{-3}	

Table 3. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **[3]OTf**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cl(1)	2350(1)	3626(1)	6608(1)	37(1)
Cl(2)	5498(1)	4335(1)	7610(1)	38(1)
P(1)	4565(1)	2644(1)	6939(1)	28(1)
Si(1)	6815(1)	2056(1)	5196(1)	29(1)
N(1)	4045(3)	1394(3)	7818(2)	25(1)
N(2)	3097(3)	-1681(3)	9967(2)	27(1)
N(3)	5660(4)	1814(4)	6219(2)	32(1)
C(1)	3033(4)	1869(4)	8581(2)	29(1)
C(2)	2718(4)	895(4)	9285(2)	27(1)
C(3)	3394(4)	-679(4)	9288(2)	24(1)
C(4)	4441(4)	-1141(4)	8492(2)	31(1)
C(5)	4741(4)	-121(4)	7808(2)	31(1)
C(7)	1982(5)	-1292(5)	10774(2)	35(1)
C(8)	3730(5)	-3335(4)	9891(3)	33(1)
C(9)	7726(5)	112(4)	4841(3)	39(1)
C(10)	5420(5)	2895(5)	4316(3)	40(1)
C(11)	8467(5)	3337(5)	5392(3)	40(1)
S(1)	9386(1)	-3591(1)	8633(1)	30(1)
F(1)	8649(3)	-1395(3)	7411(2)	62(1)
F(2)	11106(3)	-2527(3)	7206(2)	65(1)
F(3)	9035(4)	-3691(3)	6860(2)	69(1)
O(1)	10414(4)	-5033(3)	8570(2)	52(1)
O(2)	10004(3)	-2411(3)	9165(2)	42(1)
O(3)	7630(3)	-3736(3)	8780(2)	41(1)
C(12)	9543(5)	-2770(5)	7473(3)	41(1)

Table 4. Bond lengths [Å] and angles [°] for **[3]OTf**.

Cl(1)-P(1)	1.9947(12)
Cl(2)-P(1)	2.0040(13)
P(1)-N(3)	1.490(3)
P(1)-N(1)	1.713(3)
Si(1)-N(3)	1.733(3)
Si(1)-C(11)	1.846(4)
Si(1)-C(9)	1.849(4)
Si(1)-C(10)	1.854(4)
N(1)-C(5)	1.380(4)
N(1)-C(1)	1.392(4)
N(2)-C(3)	1.325(4)
N(2)-C(7)	1.468(4)
N(2)-C(8)	1.480(4)
C(1)-C(2)	1.339(5)
C(2)-C(3)	1.422(5)
C(3)-C(4)	1.440(5)
C(4)-C(5)	1.341(5)
S(1)-O(2)	1.438(3)
S(1)-O(1)	1.439(3)
S(1)-O(3)	1.439(3)
S(1)-C(12)	1.822(4)
F(1)-C(12)	1.341(4)
F(2)-C(12)	1.333(4)
F(3)-C(12)	1.318(5)
N(3)-P(1)-N(1)	110.73(15)
N(3)-P(1)-Cl(1)	118.92(13)
N(1)-P(1)-Cl(1)	102.36(10)
N(3)-P(1)-Cl(2)	117.90(13)
N(1)-P(1)-Cl(2)	101.91(11)
Cl(1)-P(1)-Cl(2)	102.68(6)
N(3)-Si(1)-C(11)	108.60(17)
N(3)-Si(1)-C(9)	107.37(17)
C(11)-Si(1)-C(9)	110.65(18)

N(3)-Si(1)-C(10)	109.16(16)
C(11)-Si(1)-C(10)	111.36(18)
C(9)-Si(1)-C(10)	109.60(19)
C(5)-N(1)-C(1)	117.9(3)
C(5)-N(1)-P(1)	119.3(2)
C(1)-N(1)-P(1)	122.6(2)
C(3)-N(2)-C(7)	123.3(3)
C(3)-N(2)-C(8)	120.5(3)
C(7)-N(2)-C(8)	115.8(3)
P(1)-N(3)-Si(1)	144.1(2)
C(2)-C(1)-N(1)	121.7(3)
C(1)-C(2)-C(3)	121.6(3)
N(2)-C(3)-C(2)	123.4(3)
N(2)-C(3)-C(4)	121.0(3)
C(2)-C(3)-C(4)	115.6(3)
C(5)-C(4)-C(3)	120.8(3)
C(4)-C(5)-N(1)	122.3(3)
O(2)-S(1)-O(1)	115.75(18)
O(2)-S(1)-O(3)	114.50(16)
O(1)-S(1)-O(3)	115.36(18)
O(2)-S(1)-C(12)	102.37(19)
O(1)-S(1)-C(12)	103.64(17)
O(3)-S(1)-C(12)	102.51(17)
F(3)-C(12)-F(2)	106.1(3)
F(3)-C(12)-F(1)	108.0(3)
F(2)-C(12)-F(1)	106.7(3)
F(3)-C(12)-S(1)	112.5(3)
F(2)-C(12)-S(1)	111.8(3)
F(1)-C(12)-S(1)	111.4(3)

Symmetry transformations used to generate equivalent atoms:

Table 5. Anisotropic displacement parameters ($Å^2 x 10^3$) for **[3]OTf**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cl(1)	32(1)	40(1)	38(1)	3(1)	-4(1)	6(1)
Cl(2)	49(1)	35(1)	33(1)	3(1)	-6(1)	-14(1)
P(1)	28(1)	28(1)	26(1)	3(1)	0(1)	-3(1)
Si(1)	26(1)	32(1)	28(1)	0(1)	3(1)	-4(1)
N(1)	29(2)	22(1)	25(2)	-4(1)	3(1)	0(1)
N(2)	29(2)	25(2)	27(2)	0(1)	1(1)	1(1)
N(3)	30(2)	35(2)	30(2)	3(1)	4(1)	-2(1)
C(1)	25(2)	28(2)	32(2)	1(2)	3(1)	1(2)
C(2)	27(2)	25(2)	28(2)	-3(2)	5(1)	-2(2)
C(3)	22(2)	26(2)	24(2)	0(1)	-1(1)	-3(1)
C(4)	29(2)	24(2)	36(2)	0(2)	5(2)	4(2)
C(5)	31(2)	27(2)	32(2)	-2(2)	6(2)	1(2)
C(7)	34(2)	42(2)	29(2)	6(2)	3(2)	-3(2)
C(8)	39(2)	23(2)	37(2)	3(2)	-1(2)	0(2)
C(9)	35(2)	37(2)	46(2)	-6(2)	6(2)	-4(2)
C(10)	41(2)	46(2)	32(2)	7(2)	2(2)	-9(2)
C(11)	35(2)	45(2)	41(2)	-4(2)	0(2)	-13(2)
S(1)	28(1)	31(1)	29(1)	-2(1)	3(1)	2(1)
F(1)	71(2)	57(2)	51(2)	21(1)	11(1)	18(1)
F(2)	46(2)	82(2)	61(2)	14(2)	27(1)	-5(1)
F(3)	88(2)	85(2)	34(1)	-12(1)	-2(1)	-14(2)
O(1)	59(2)	40(2)	51(2)	-1(1)	6(1)	25(2)
O(2)	39(2)	49(2)	40(2)	-12(1)	-3(1)	-9(1)
O(3)	29(1)	49(2)	45(2)	5(1)	6(1)	-8(1)
C(12)	37(2)	47(2)	36(2)	1(2)	8(2)	6(2)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for k0384.

	x	y	z	U(eq)
H(1A)	2552	2911	8603	34
H(2A)	2027	1266	9796	32
H(4A)	4924	-2180	8452	37
H(5A)	5459	-453	7299	37
H(7A)	1478	-226	10705	53
H(7B)	2619	-1380	11326	53
H(7C)	1106	-2011	10828	53
H(8A)	4946	-3414	9807	50
H(8B)	3262	-3784	9365	50
H(8C)	3400	-3902	10453	50
H(9A)	8376	-372	5334	59
H(9B)	8453	215	4285	59
H(9C)	6833	-537	4716	59
H(10A)	4903	3907	4517	59
H(10B)	4553	2195	4230	59
H(10C)	6066	3028	3734	59
H(11A)	9154	2885	5878	60
H(11B)	7961	4362	5577	60
H(11C)	9163	3445	4825	60