Electronic Supplementary Material (ESI) for Dalton Transactions.

Supplementary material for:

Antimony(I)→Pd(II) complexes with (μ-Sb)Pd_2 coordination framework

Monika Kořenková, Martin Hejda, Theresa Block, Filip Uhlík, Roman Jambor, Aleš Růžička, Rainer Pöttgen and Libor Dostál

Table of contents:

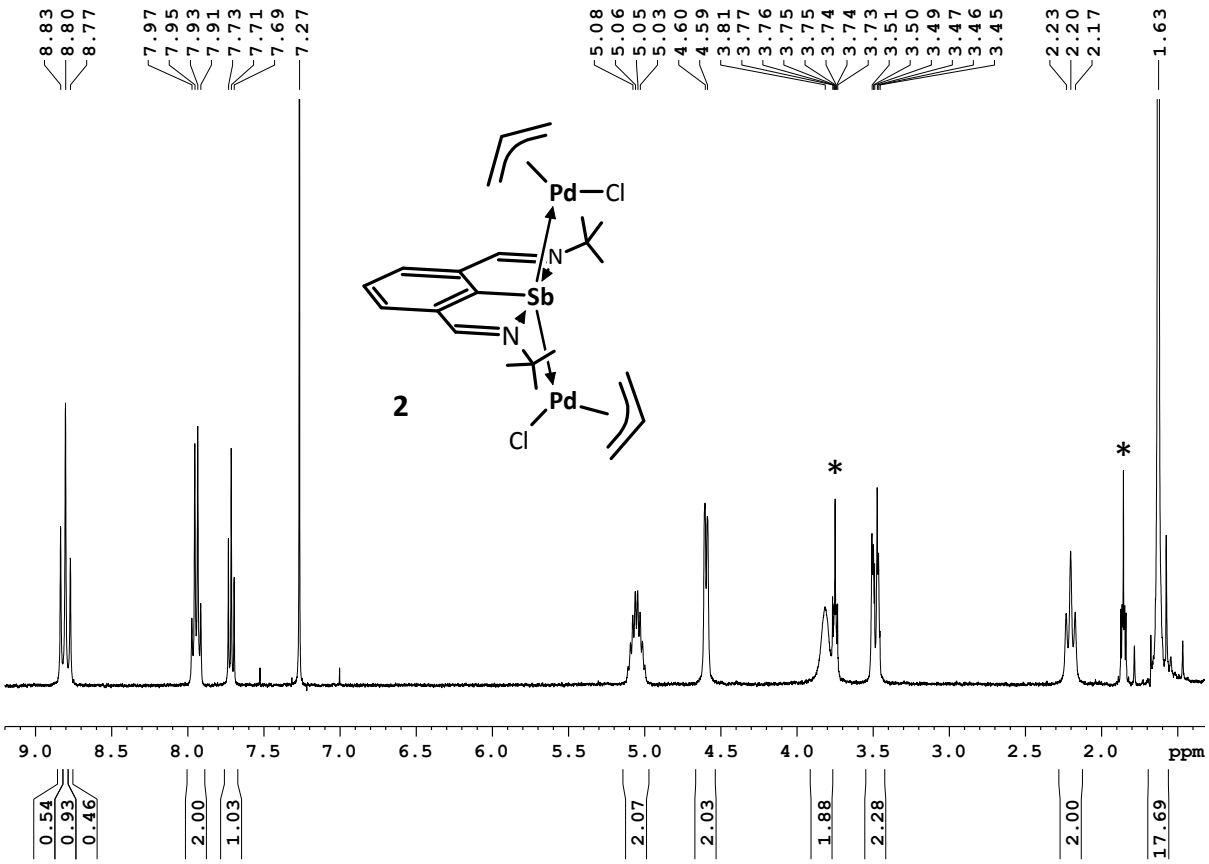

Figure S1: ^{1}H NMR of compound **2** (500.20 MHz, $CDCl_3$, 294 K). * - traces of THF.

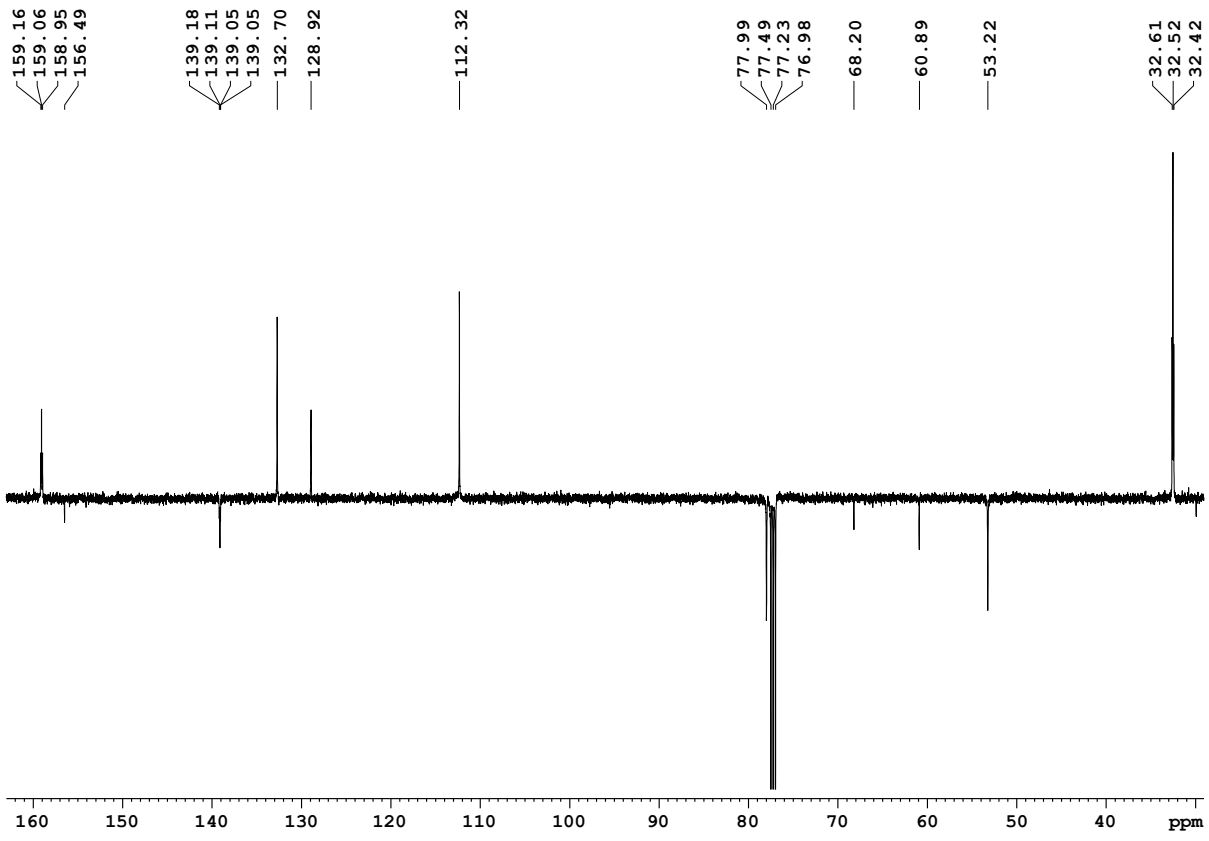

Figure S2: ^{13}C{^{1}H} APT NMR of compound **2** (125.78 MHz, $CDCl_3$, 294 K).

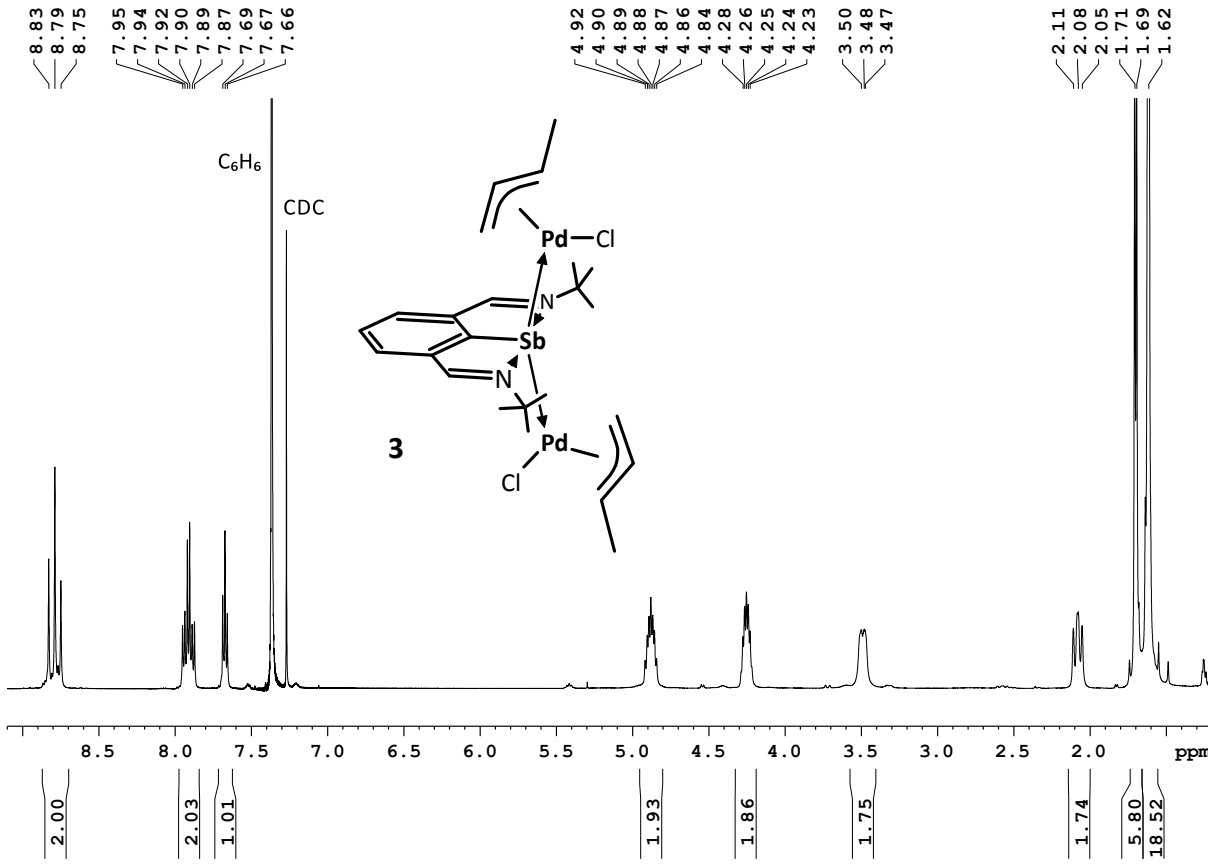

Figure S3: ^{1}H NMR of compound **3**.C_6H_6 (500.20 MHz, $CDCl_3$, 294 K).

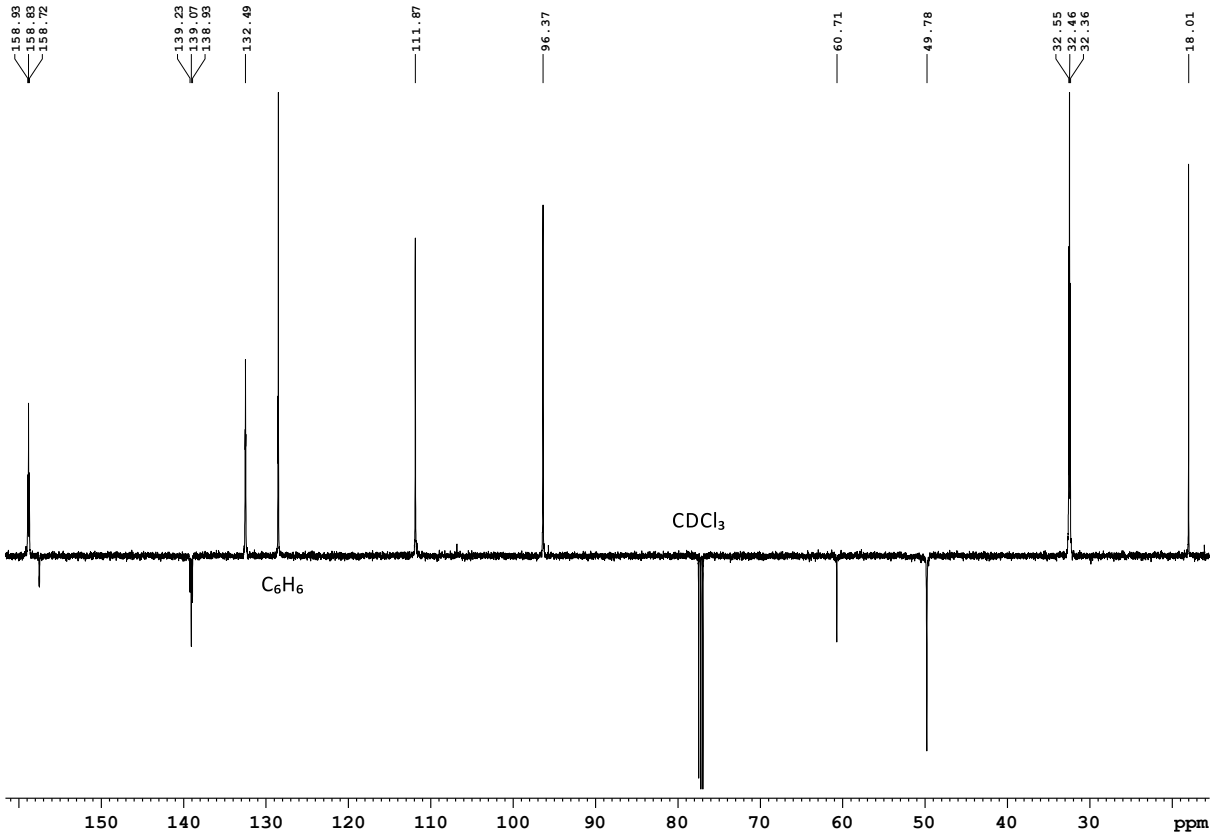

Figure S4: $^{13}C\{^{1}H\}$ APT NMR of compound **3**.C_6H_6 (125.78 MHz, $CDCl_3$, 294 K).

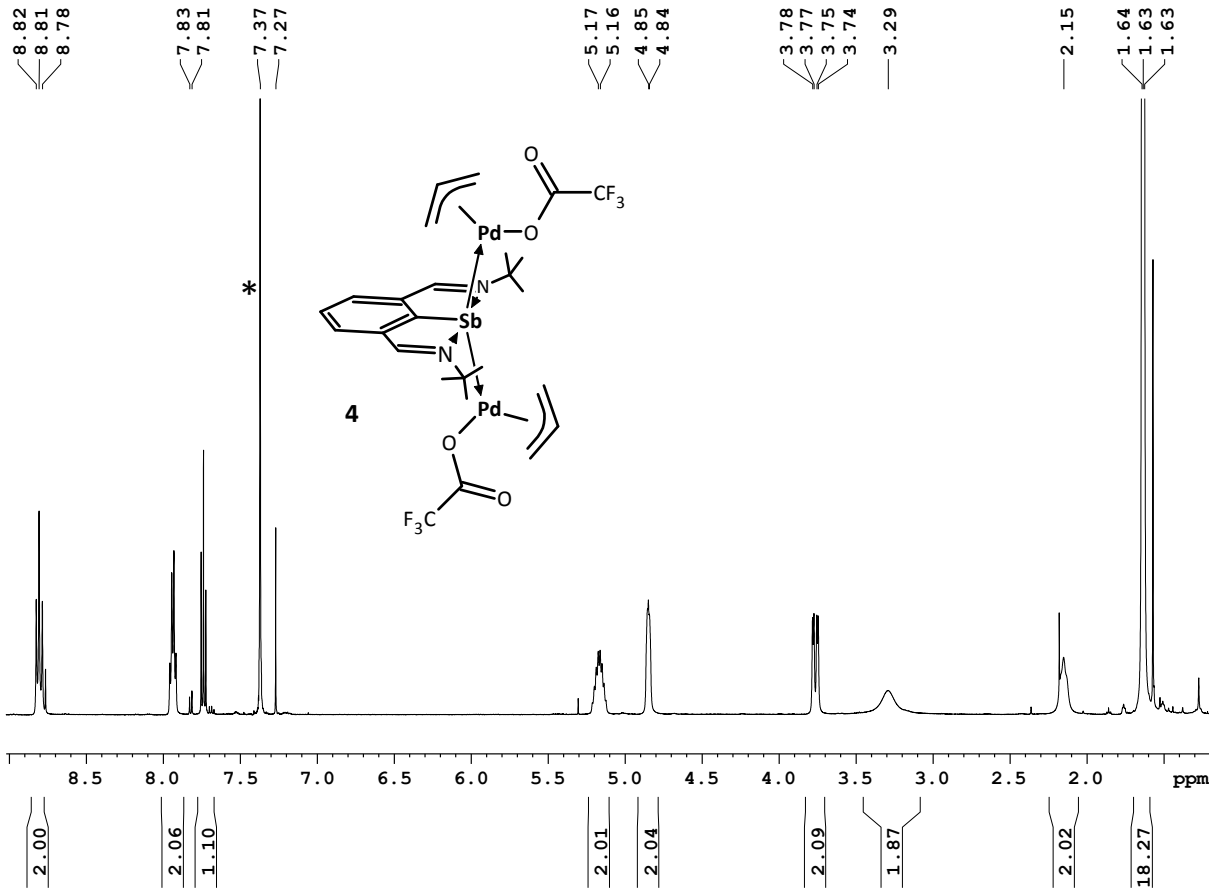

Figure S5: ^{1}H NMR of compound **4** (500.20 MHz, $CDCl_3$, 294 K). * - C_6H_6 (since benzene solvate single crystals of **4** were dissolved in $CDCl_3$).

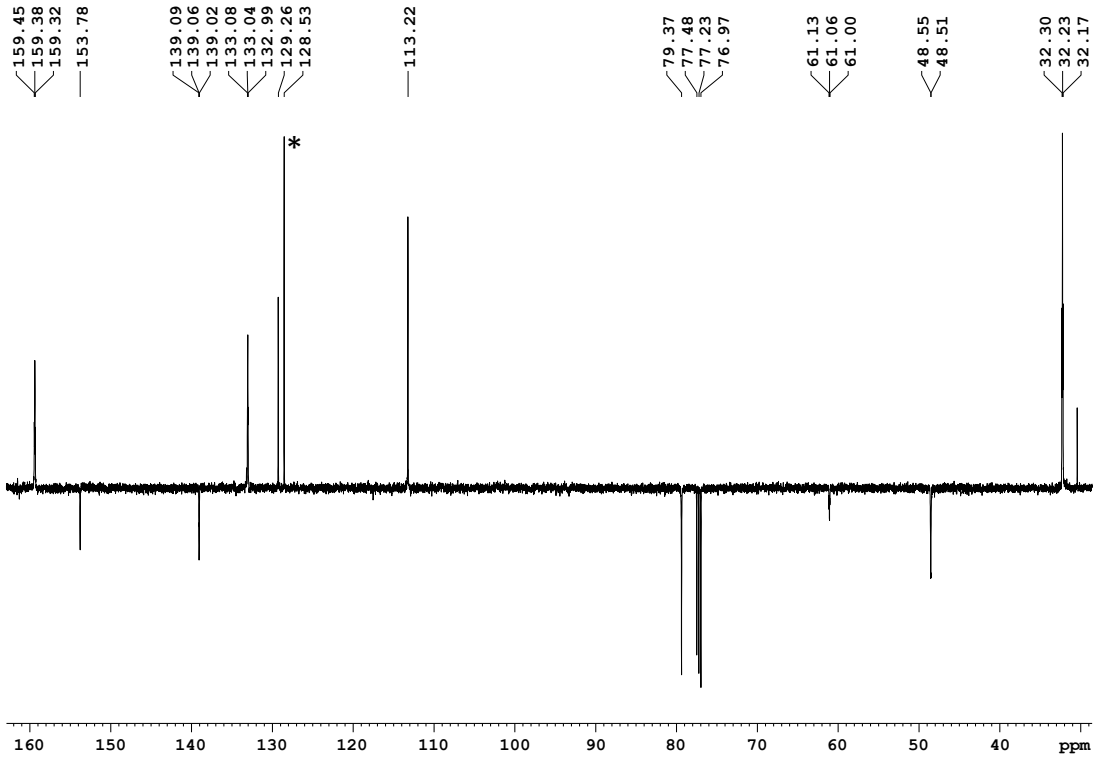

Figure S6: $^{13}C\{^{1}H\}$ APT NMR of compound **4** (125.78 MHz, $CDCl_3$, 294 K). * - C_6H_6 (since benzene solvate single crystals of **4** were dissolved in $CDCl_3$).

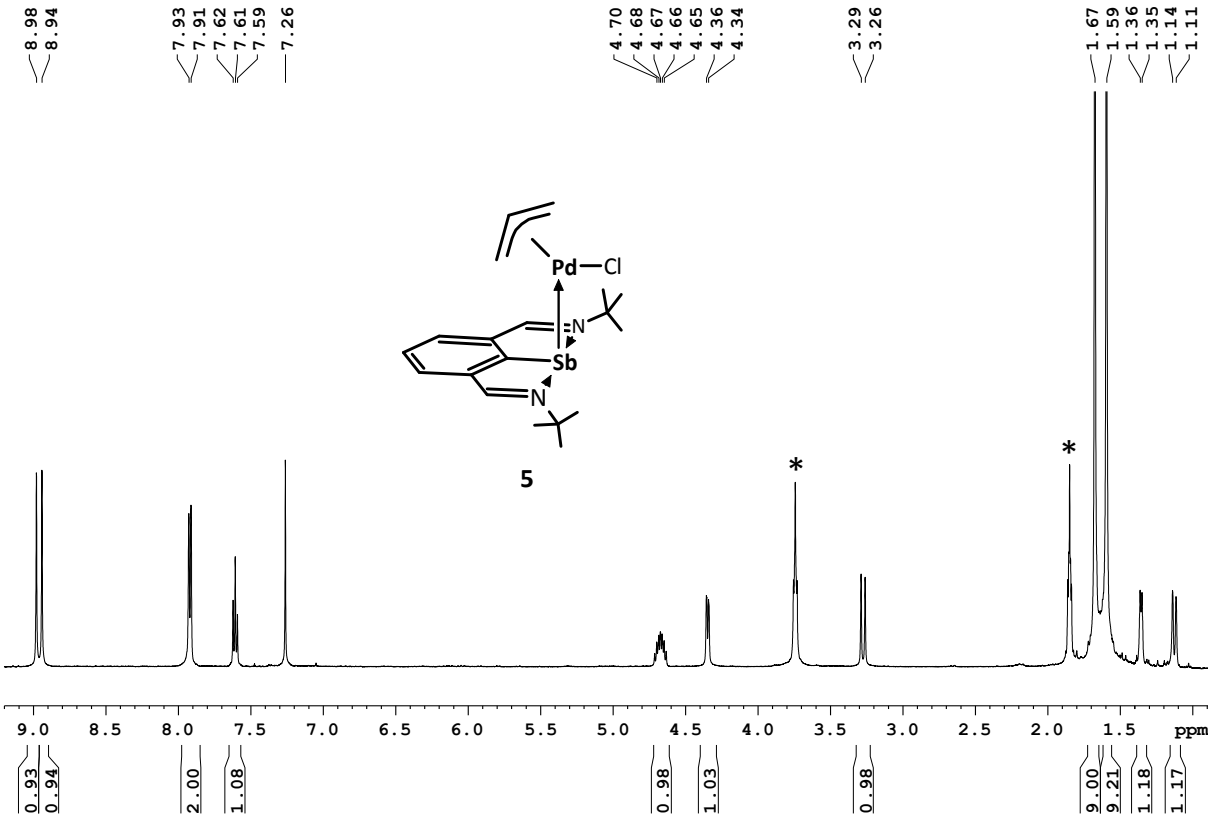

Figure S7: ^{1}H NMR of compound **5** (500.20 MHz, $CDCl_3$, 294 K). * - traces of THF.

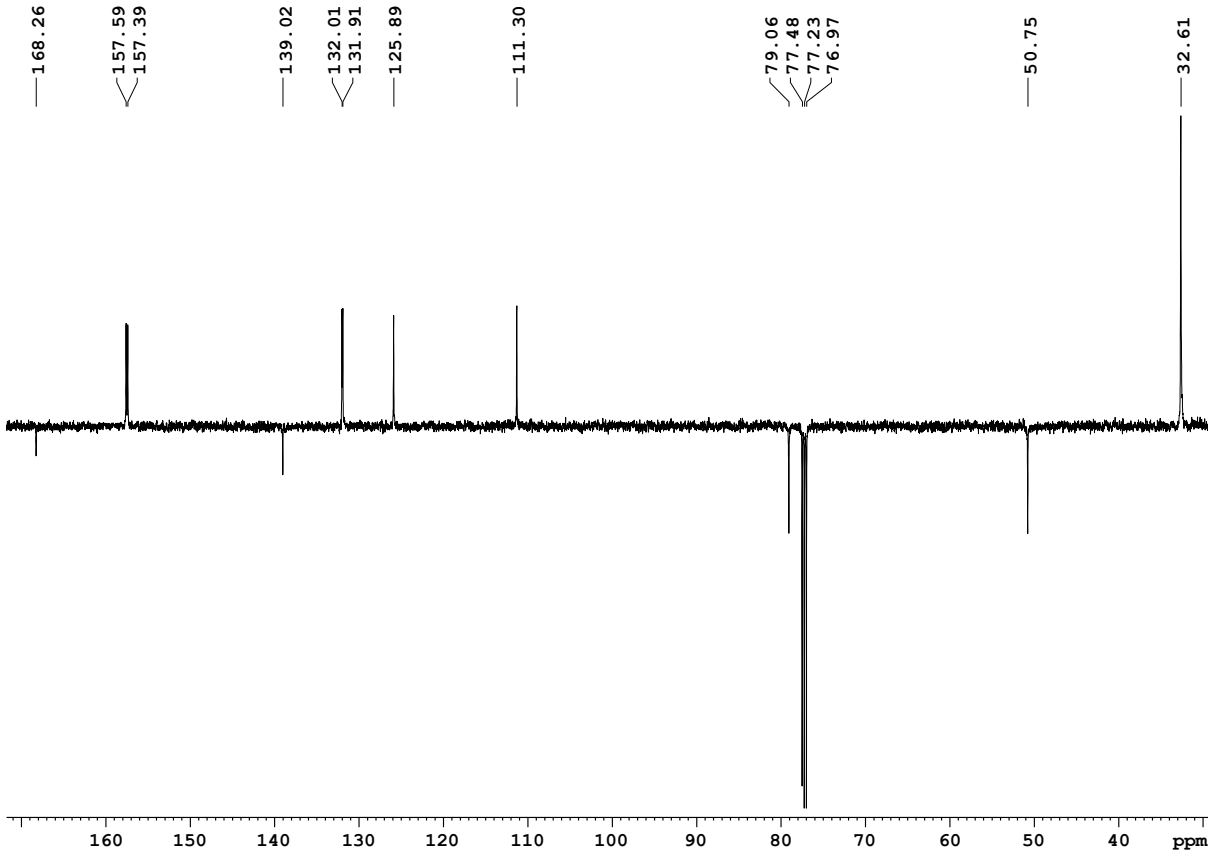

Figure S8: ^{13}C{^{1}H} APT NMR of compound **5** (125.78 MHz, $CDCl_3$, 294 K).

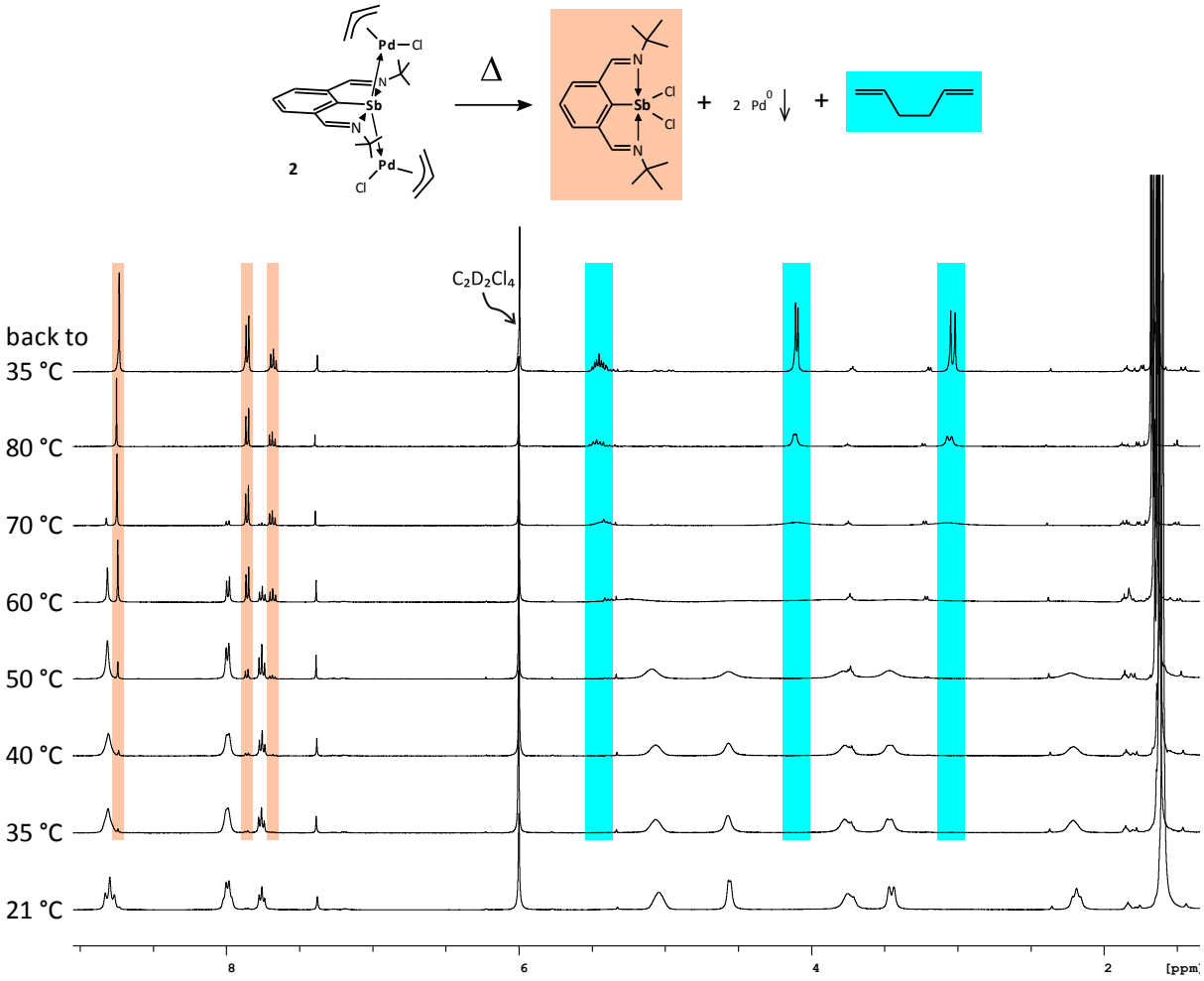

Figure S9: Stacked plot of result of **VT-^{1}H NMR** (500.20 MHz) measurement of freshly prepared NMR sample of compound **2** in $C_2D_2Cl_4$.

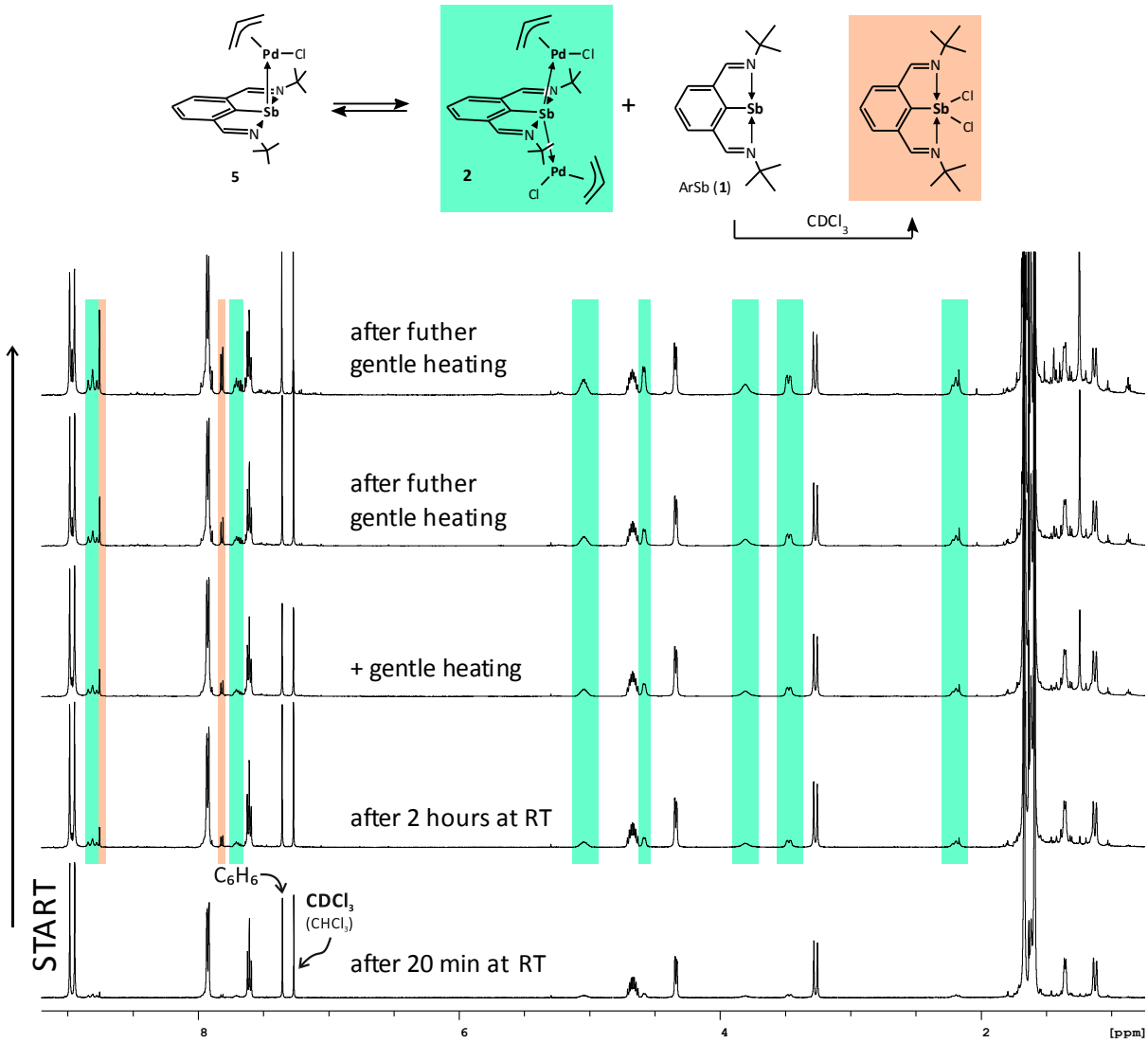

Figure S10: Stacked plot of ^{1}**H NMR** (500.20 MHz) spectra showing decomposition of compound **5** in $CDCl_3$ (gentle heating means heating the NMR tube up for couple of seconds outside the NMR spectrometer with heat gun set to 60 °C).

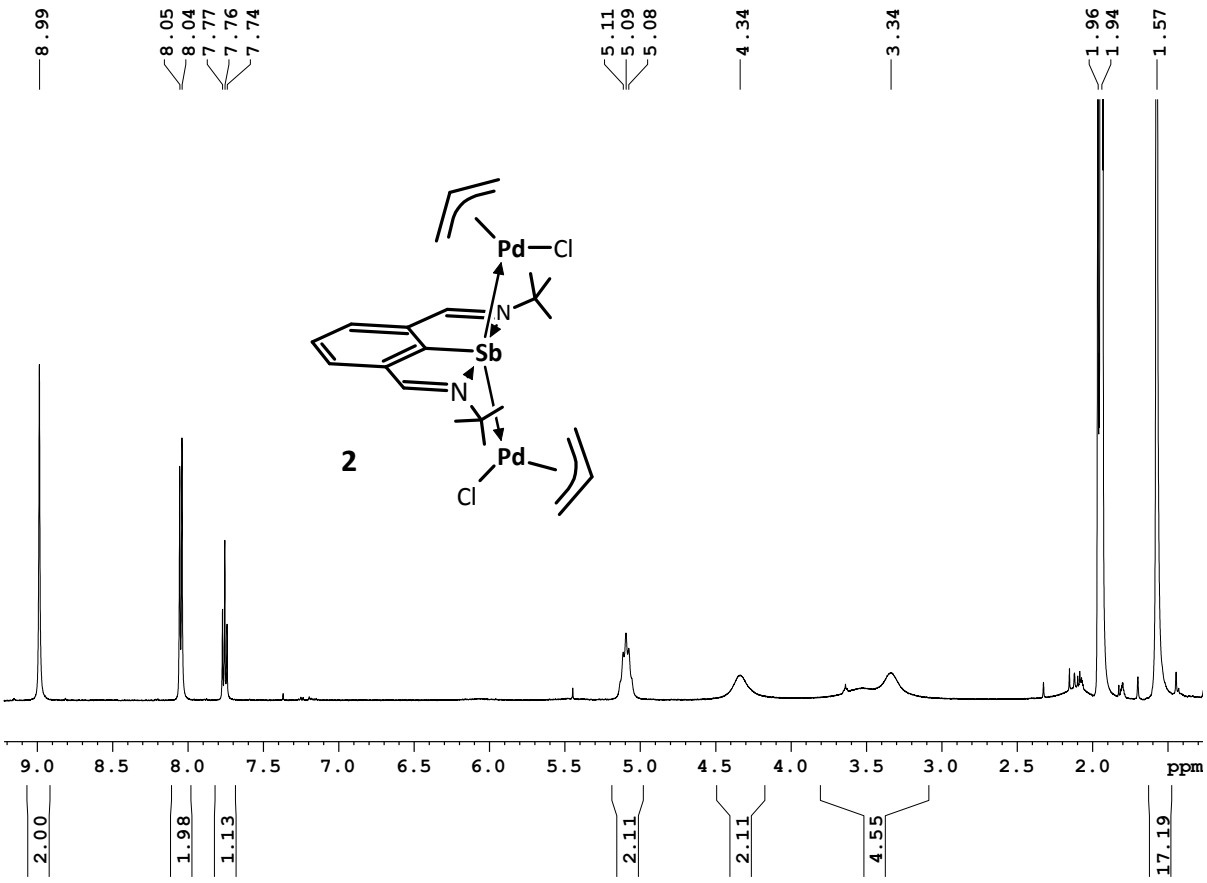

Figure S11: ^{1}H NMR of compound **2** (500.20 MHz, CD_3CN, 294 K).

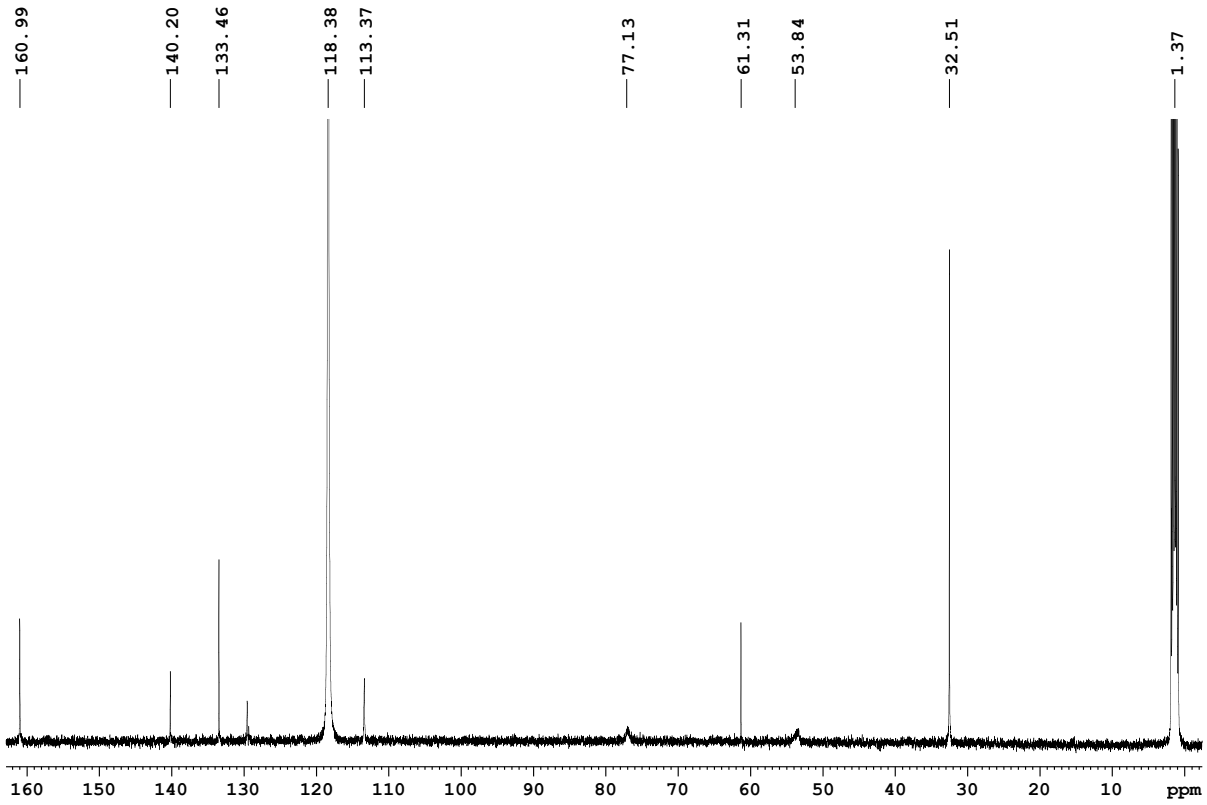

Figure S12: ^{13}C{^{1}H} NMR of compound **2** (125.78 MHz, CD_3CN, 294 K).

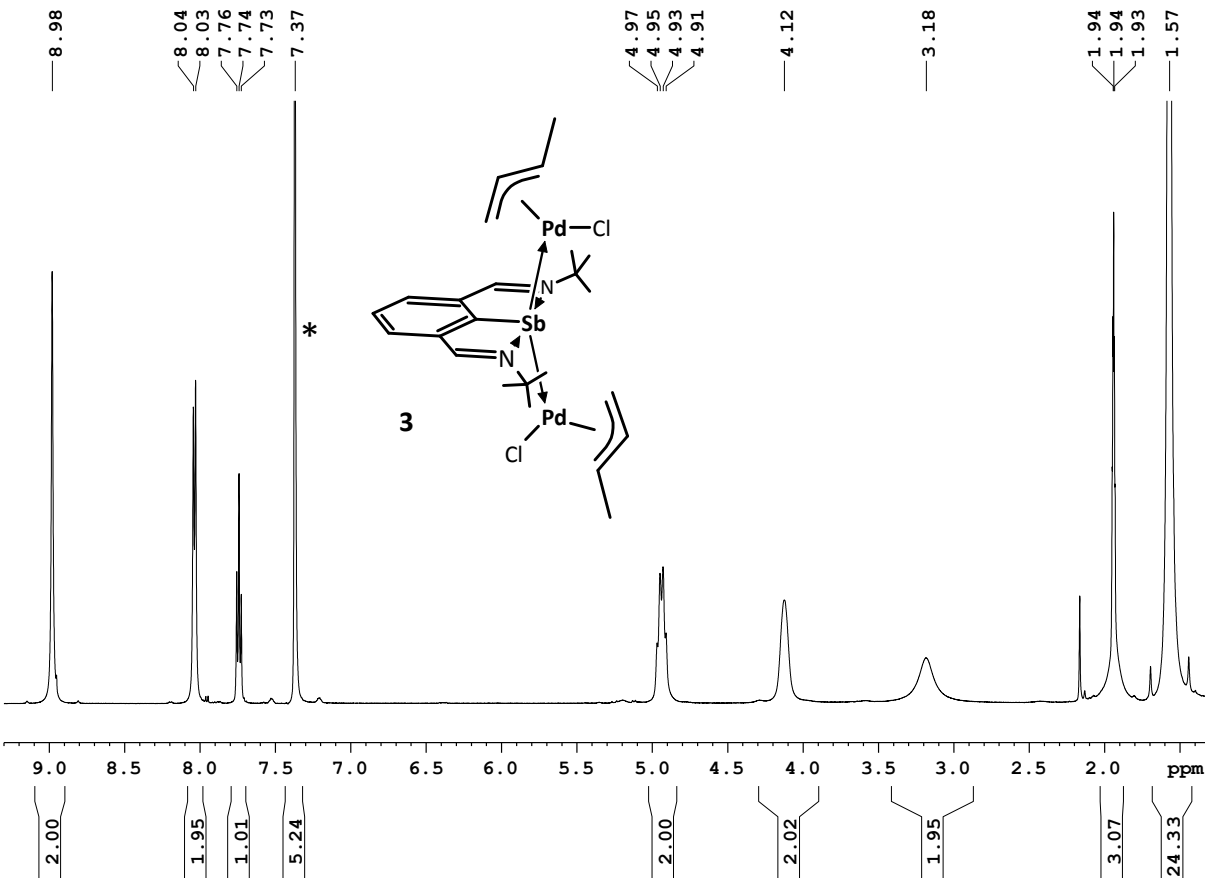

Figure S13: ^{1}H NMR of compound **3** (500.20 MHz, CD_3CN, 294 K). * - benzene (since benzene solvate single crystals of **3** were dissolved in CD_3CN).

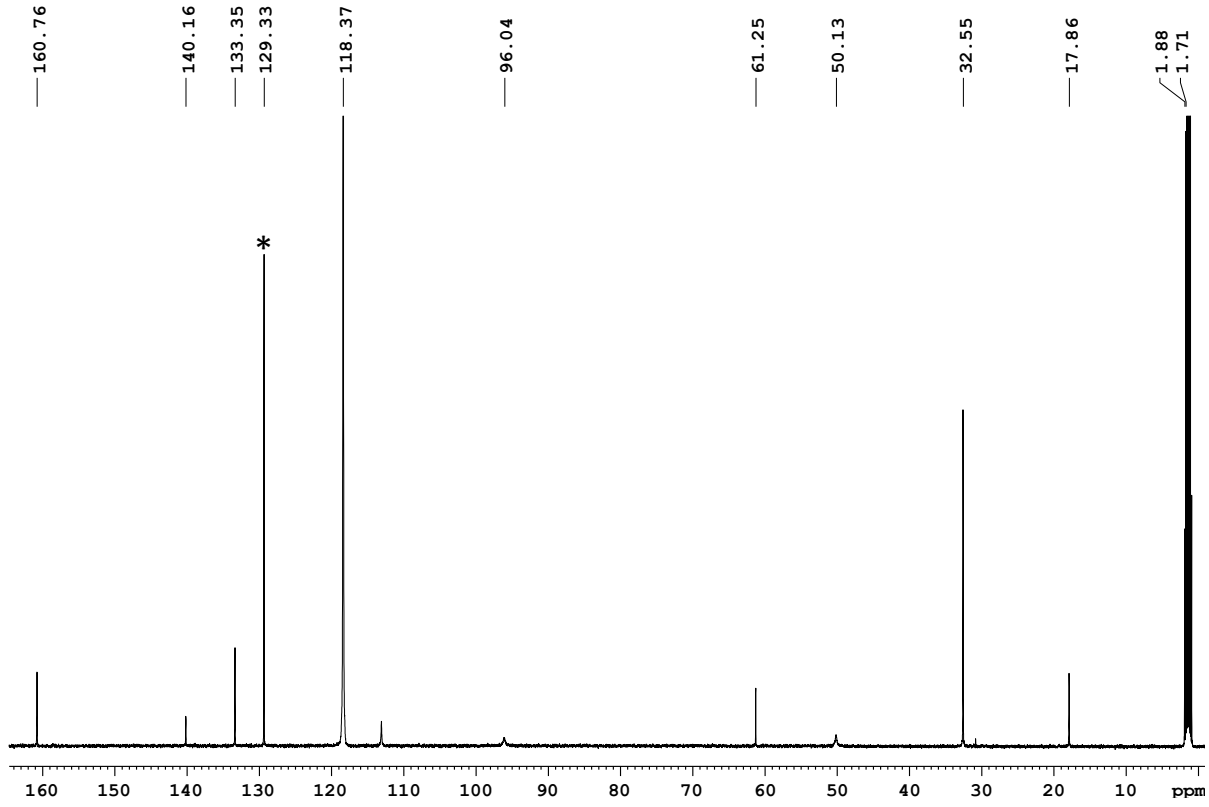

Figure S14: ^{13}C{^{1}H} NMR of compound **3** (125.78 MHz, CD_3CN, 294 K).

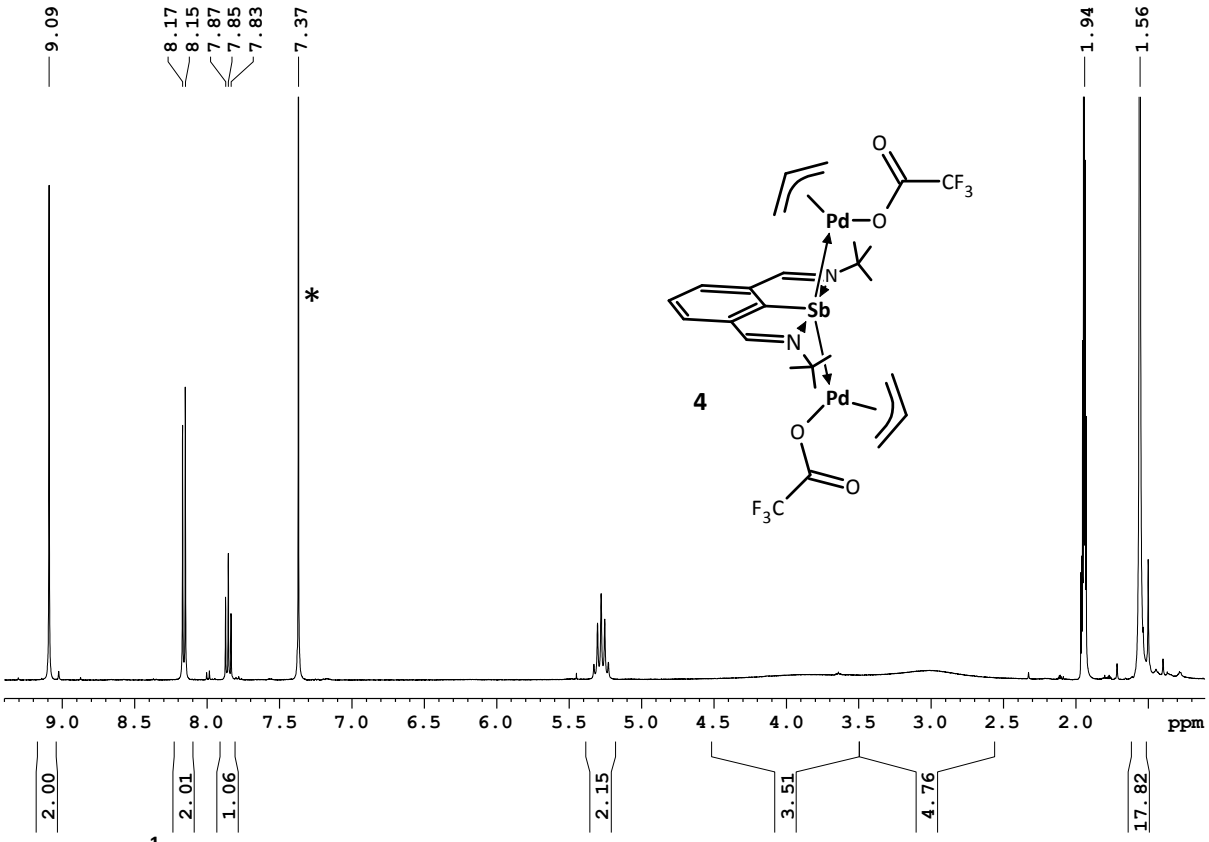

Figure S15: ^{1}H NMR of compound **4** (500.20 MHz, CD_3CN, 294 K). * - benzene (since benzene solvate single crystals of **4** were dissolved in CD_3CN).

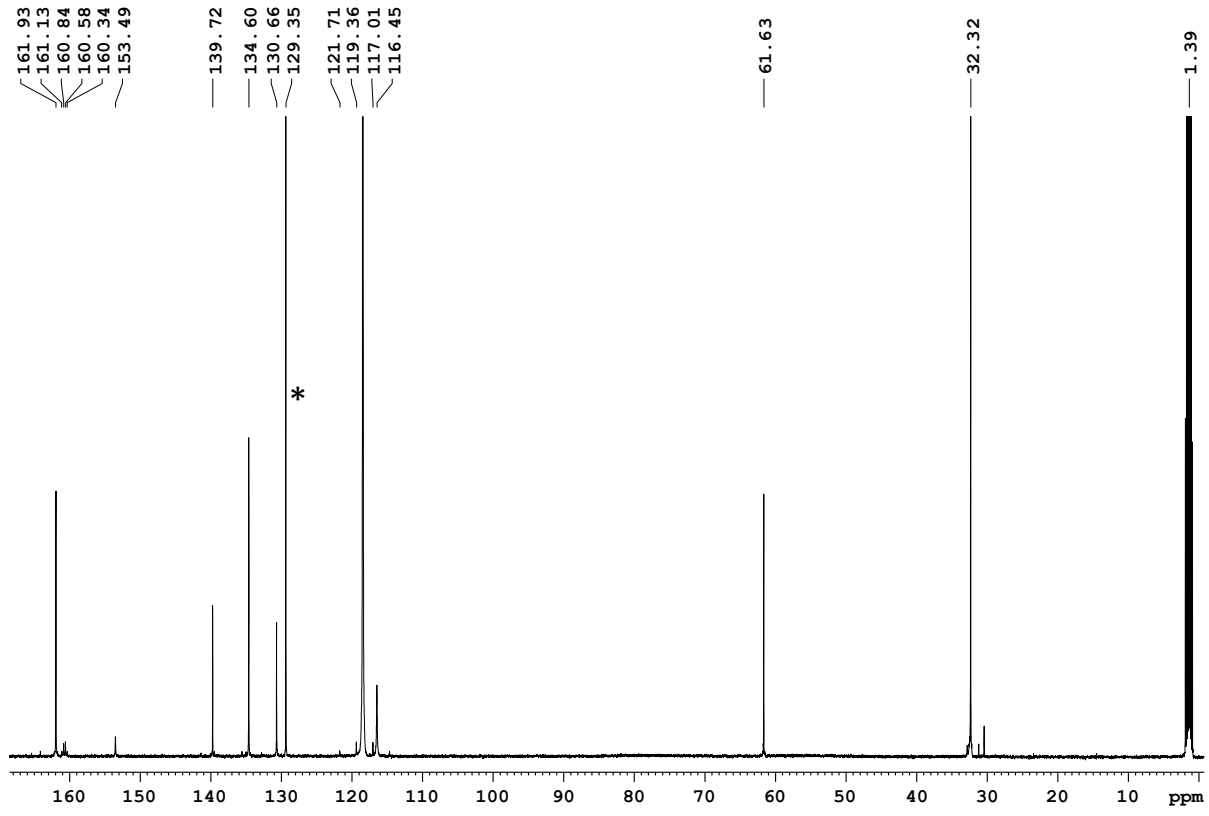

Figure S16: $^{13}C\{^{1}H\}$ NMR of compound **4** (125.78 MHz, CD_3CN, 294 K). * - benzene (since benzene solvate single crystals of **4** were dissolved in CD_3CN).

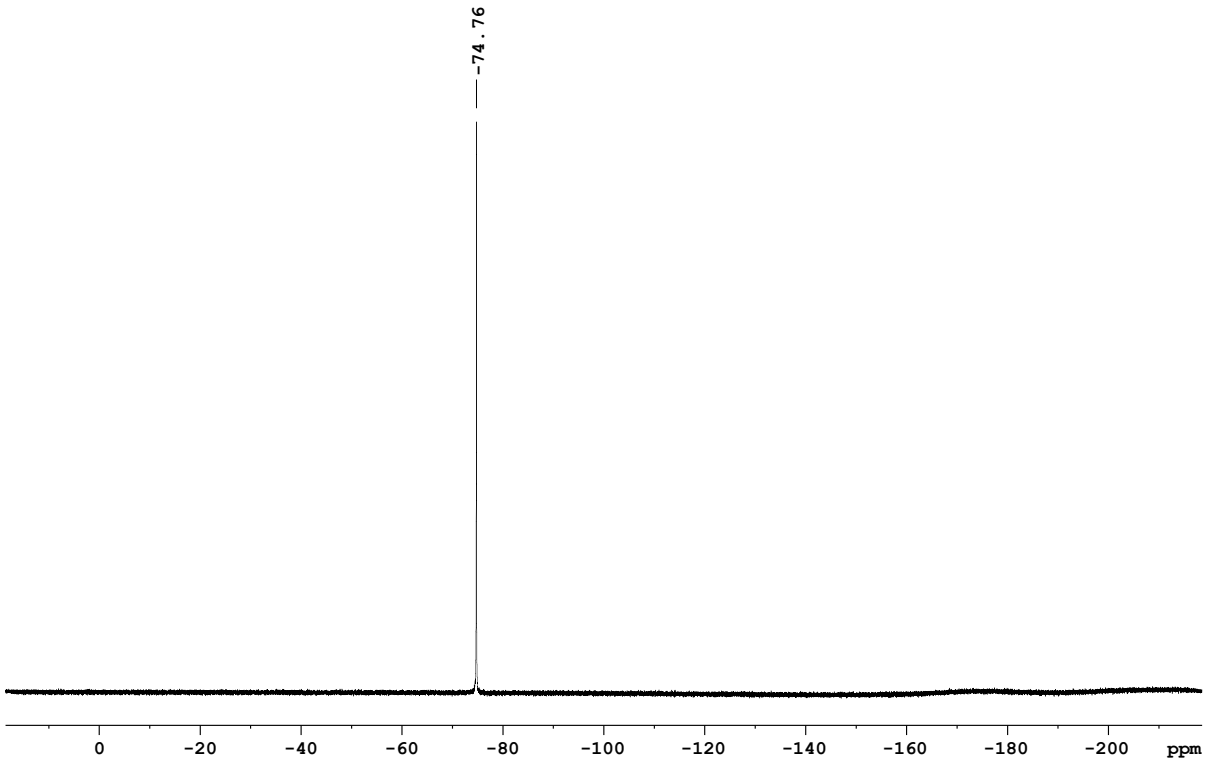

Figure S17: ^{19}F NMR of compound **4** (376.50 MHz, CD_3CN, 294 K).

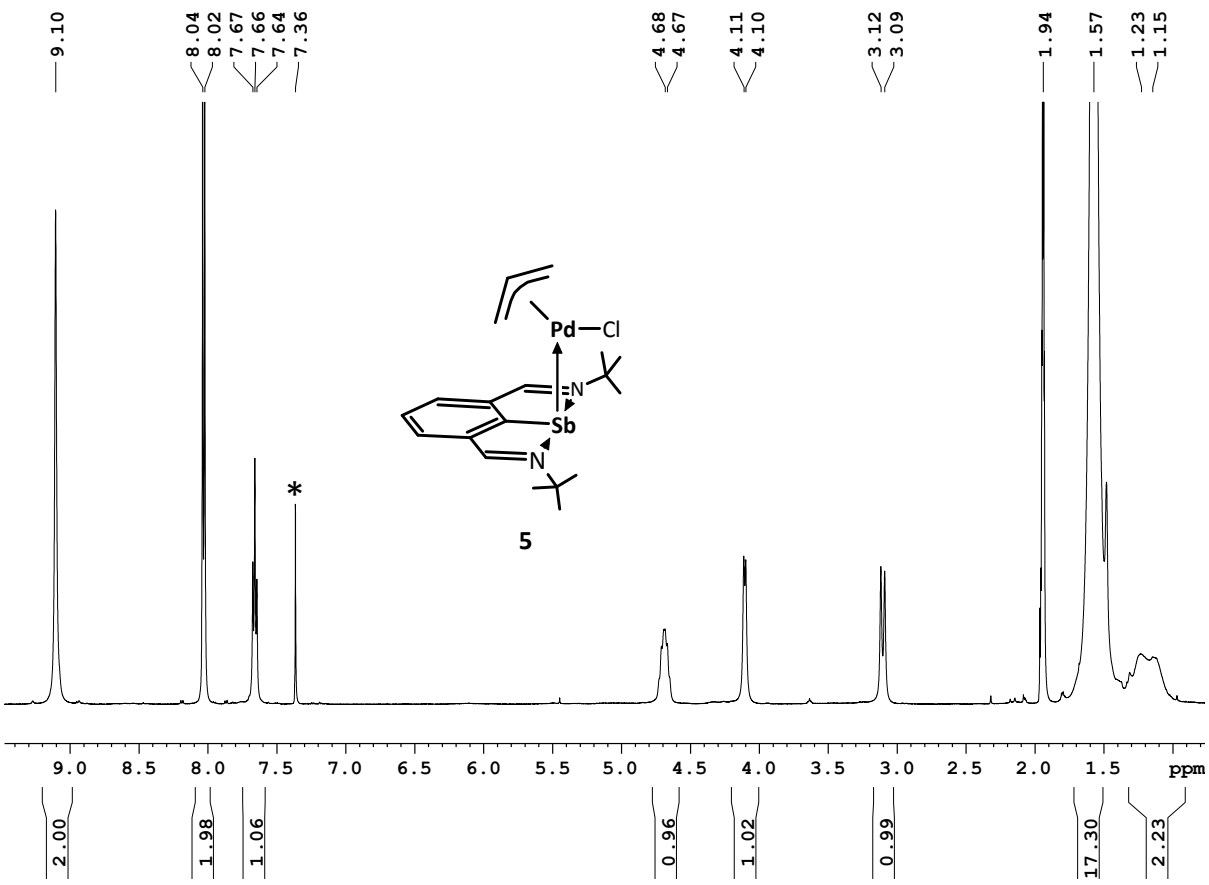

Figure S18: ^{1}H NMR of compound **5** (500.20 MHz, CD_3CN, 294 K). * - traces of benzene.

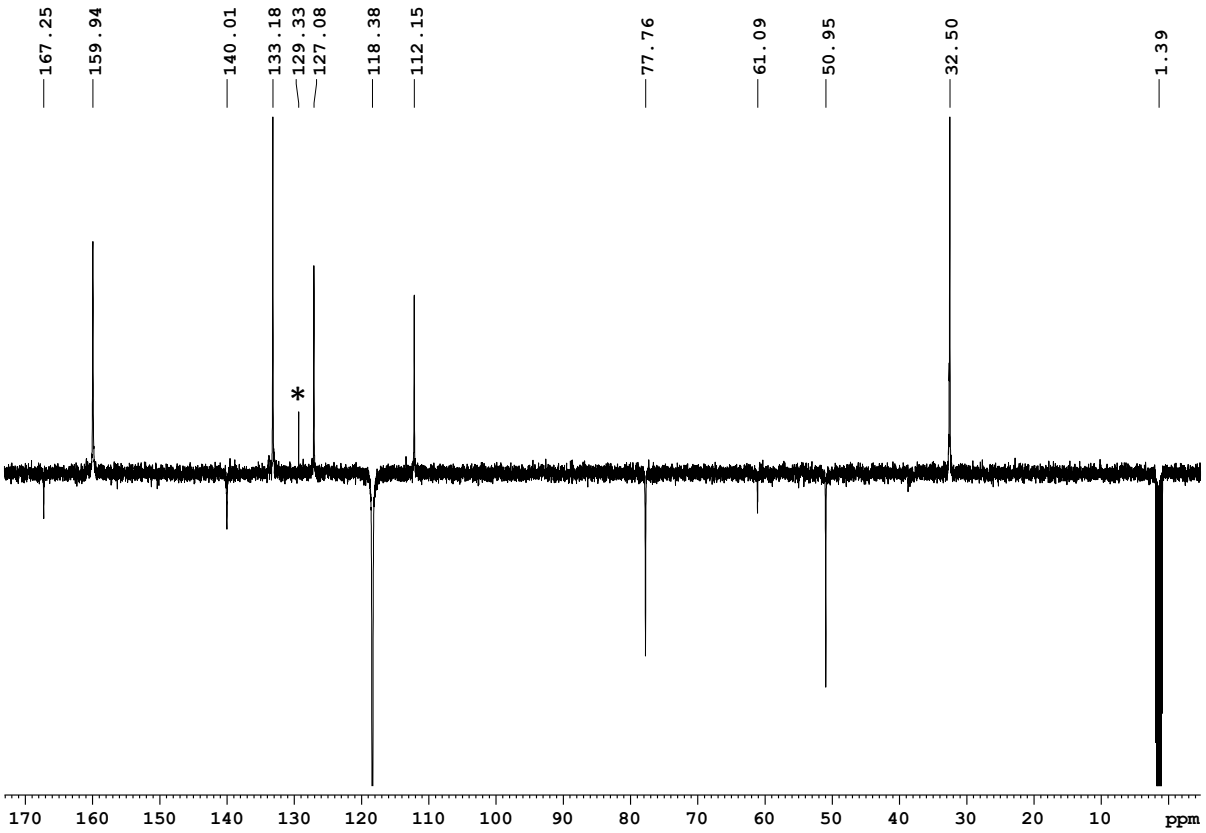

Figure S19: $^{13}C\{^{1}H\}$ APT NMR of compound **5** (125.78 MHz, CD_3CN, 294 K).

Table S1. Crystallographic data for studied compounds **2**-**5**.

	2	**3**	**4**	**5**
chemical formula	$C_{22}H_{33}Cl_2N_2Pd_2Sb$ .2(C_6H_6)	$C_{24}H_{37}Cl_2N_2Pd_2Sb$ .2(C_6H_6)	$C_{26}H_{33}F_6N_2O_4Pd_2Sb$.(C_6H_6)	$C_{19}H_{28}ClN_2PdSb$
Cryst syst	monoclinic	orthorhombic	orthorhombic	monoclinic
Space group	$P2_1/c$	$Pca2_1$	$Pca2_1$	$P2_1/c$
a[Å]	9.9651(10)	26.6878(9)	18.3001(13)	16.2454(7)
b[Å]	22.9000(14)	12.1213(4)	10.5668(6)	16.0559(7)
c[Å]	18.0049(13)	23.2984(8)	18.5561(14)	17.8662(8)
β[°]	118.278(7)	90	90	113.1910(10)
Z	4	8	4	8
μ[mm^{-1}]	1.9	1.8	1.8	2.2
D_x [g cm^{-3}]	1.63	1.61	1.78	1.70
Crystal size [mm]	0.59×0.31×0.26	0.50×0.30×0.10	0.20×0.08×0.07	0.57×0.27×0.17
θ range, [deg]	1-27.5	1-32	1-27.9	1-32
T_{min}, T_{max}	0.579, 0.746	0.543, 0.746	0.466, 0.746	0.584, 0.746
no. of reflns measd	94 388	71 935	30 074	123 966
no. ofunique reflns, R_{int}	53544, 0.022	21184, 0.107	8073, 0.134	13698, 0.083
no. of obsd reflns	6890	13371	5563	8821
no. of params	370	817	473	475
S all data	1.222	1.031	0.998	1.093
final R indices [$I>2\sigma(I)$]	0.024	0.067	0.058	0.058
wR2 indices (all data)	0.051	0.084	0.075	0.069
Δρ, max., min. [e $Å^{-3}$]	0.95, -0.72	0.92, -1.38	0.71, -0.82	1.28, -0.95
CCDC	1908187	1908186	1908185	1908188

$R_{int} = \sum | F_o^2 - F_{o,mean}^2 | / \sum F_o^2$, GOF = $[\sum(w(F_o^2 - F_c^2)^2)/(N_{diffrs} - N_{params})]^{½}$ for all data, $R(F) = \sum | | F_o | - | F_c | | / \sum | F_o |$ for observed data, $wR(F^2) = [\sum(w(F_o^2 - F_c^2)^2)/(\sum w(F_o^2)^2)]^{½}$ for all data.