

## Electronic Supplementary Information of :

### **Deactivation Processes of Homogeneous Pd Catalysts using *in situ* Time Resolved Spectroscopic Techniques**

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**Table 1: EXAFS analysis results of (Xantphos)Pd(C<sub>5</sub>H<sub>9</sub>)OTf in acetone (room temperature).<sup>a</sup>**

Abs-Sc <sup>b</sup>	N	R (Å)	$\sigma^2$ (Å <sup>-2</sup> )	E <sub>0</sub> (eV)
<b>Pd-P</b>	2.2	2.32	0.008	3.7
<b>Pd-C</b>	3.0	2.22	0.015	13.3
<b>Pd-C</b>	2.2	3.03	0.027	-2.1
<b>Pd-C</b>	3.3	3.67	0.036	-7.8

<sup>a</sup> Fit: R-space,  $3.13 < k < 12.3$ ,  $1.0 < R < 4.0$ ;  $k^0$ -weighted V.I.<sup>b</sup>=0.07, V.A.<sup>b</sup>=0.04,  $k^3$ -weighted V.I.=0.25, V.A.=0.14. <sup>b</sup> Abbreviations: Abs = Absorber, Sc = Scatterer, V.I. = Variance in Imaginary Part, V.A. = Variance in Absolute Part.

**Table 2: EXAFS analysis results of (Xantphos)Pd(C<sub>5</sub>H<sub>9</sub>)OTf in acetone, reaction with piperidine (room temperature).<sup>a</sup>**

Abs-Sc <sup>b</sup>	N	R (Å)	$\sigma^2$ (Å <sup>-2</sup> )	E <sub>0</sub> (eV)
<b>Pd-P</b>	2.0	2.37	0.018	-9.9
<b>Pd-C</b>	3.1	2.05	0.005	9.6
<b>Pd-C</b>	1.9	3.13	0.002	-3.9
<b>Pd-Pd</b>	1.0	2.70	0.221	-6.9

<sup>a</sup> Fit: R-space,  $3.2 < k < 12$ ,  $1.2 < R < 3.5$ ;  $k^0$ -weighted V.I.<sup>b</sup>=0.06, V.A.<sup>b</sup>=0.02,  $k^3$ -weighted V.I.=0.40, V.A.=0.21. <sup>b</sup> Abbreviations: Abs = Absorber, Sc = Scatterer, V.I. = Variance in Imaginary Part, V.A. = Variance in Absolute Part.

**Figure 1:** Fourier Transform of EXAFS data of (Xantphos)Pd(C<sub>5</sub>H<sub>9</sub>)OTf in acetone (dotted line) and of the reaction of (Xantphos)Pd(C<sub>5</sub>H<sub>9</sub>)OTf with piperidine after about 5 minutes (solid line).

