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## A new approach to the study of processes of thermal decomposition and formation of nanoalloys: double complex salt [Pd(NH<sub>3</sub>)<sub>4</sub>][PtCl<sub>6</sub>]

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**Supplementary Figure S1.**  $k^2$ -weighted EXAFS-functions of (a) the Pd K-edges and (b) the Pt L<sub>3</sub>edges during thermal decomposition of  $[Pd(NH_3)_4][PtCl_6]$  in He atmosphere.



**Supplementary Figure S2.** TG/DTG/DSC/MS curves for the decomposition of [Pd(NH<sub>3</sub>)<sub>4</sub>][PtCl<sub>6</sub>] in He atmosphere at the heating rate of 10K min<sup>-1</sup> [J. Therm. Anal. Calorim., 2016, 123, 1183-1195].



Supplementary Figure S3. PXRD pattern of  $[Pd(NH_3)_4][PtCl_6]$  in He flow ( $\lambda = 0.68894$  Å) at 260°C.



**Supplementary Figure S4.** Calculated XRD patterns of Pd, Pt, PdPt alloy and experimental PXRD pattern of the PdPt nanoalloy ( $\lambda = 0.68894$  Å).





**Supplementary Figure S5.** The first order derivate of XANES (d $\mu$ /dE, where  $\mu$  is a linear absorption coefficient and E is a photon energy) at (a) the Pd K-edge and (b) the Pt L<sub>3</sub>-edge for [Pd(NH<sub>3</sub>)<sub>4</sub>][PtCl<sub>6</sub>] during thermal decomposition in He atmosphere. (c) Example of linear combination fit for the Pd K-edge XANES spectrum at 230°C by the combination of EXAFS spectra of Pd foil and that measured at 210°C (determined by EXAFS mainly as Pd(NH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>).



**Supplementary Figure S6.** Comparison of XANES spectra of the metal foil and the final product of the thermal decomposition of  $[Pd(NH_3)_4][PtCl_6]$  at the highest temperature: (a) XANES spectra at the Pd K-edge, (b) XANES spectra at the Pt L<sub>3</sub>-edge.