

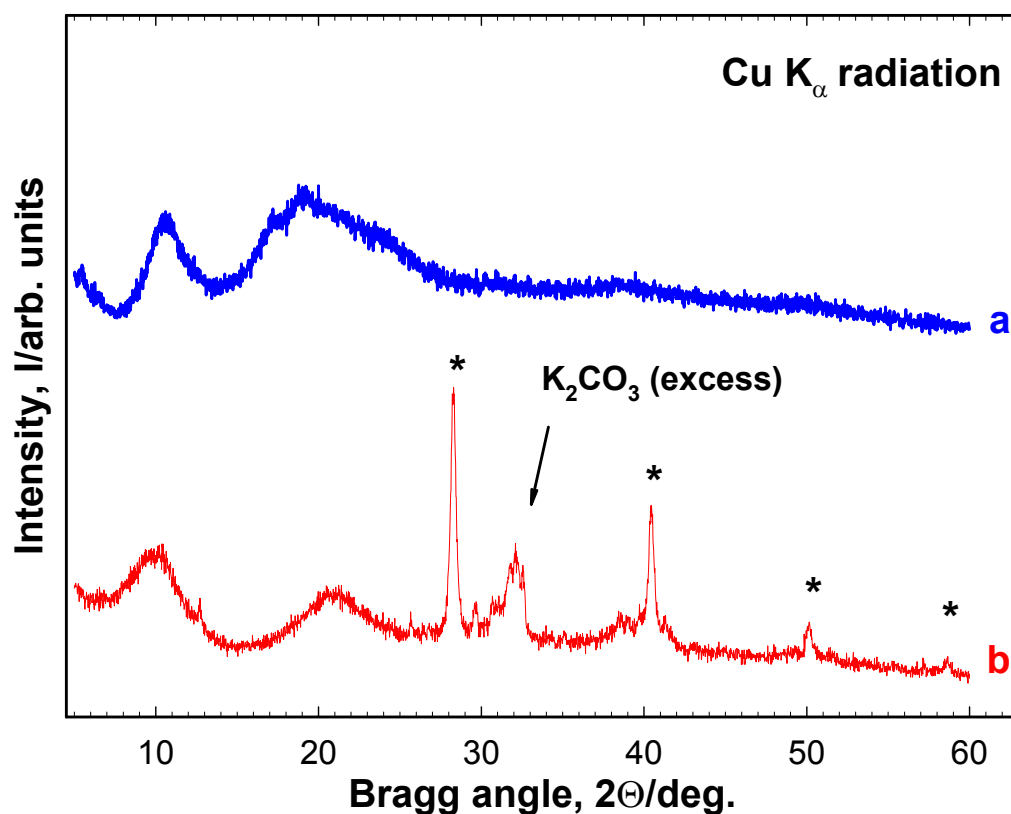
## Solvent-free mechanochemical synthesis of two Pt complexes: *cis*-(Ph<sub>3</sub>P)<sub>2</sub>PtCl<sub>2</sub> and *cis*-(Ph<sub>3</sub>P)<sub>2</sub>PtCO<sub>3</sub>

Viktor P. Balema,<sup>\*a</sup> Jerzy W. Wiench<sup>a</sup>, Marek Pruski<sup>a</sup>, and Vitalij K. Pecharsky<sup>\*ab</sup>

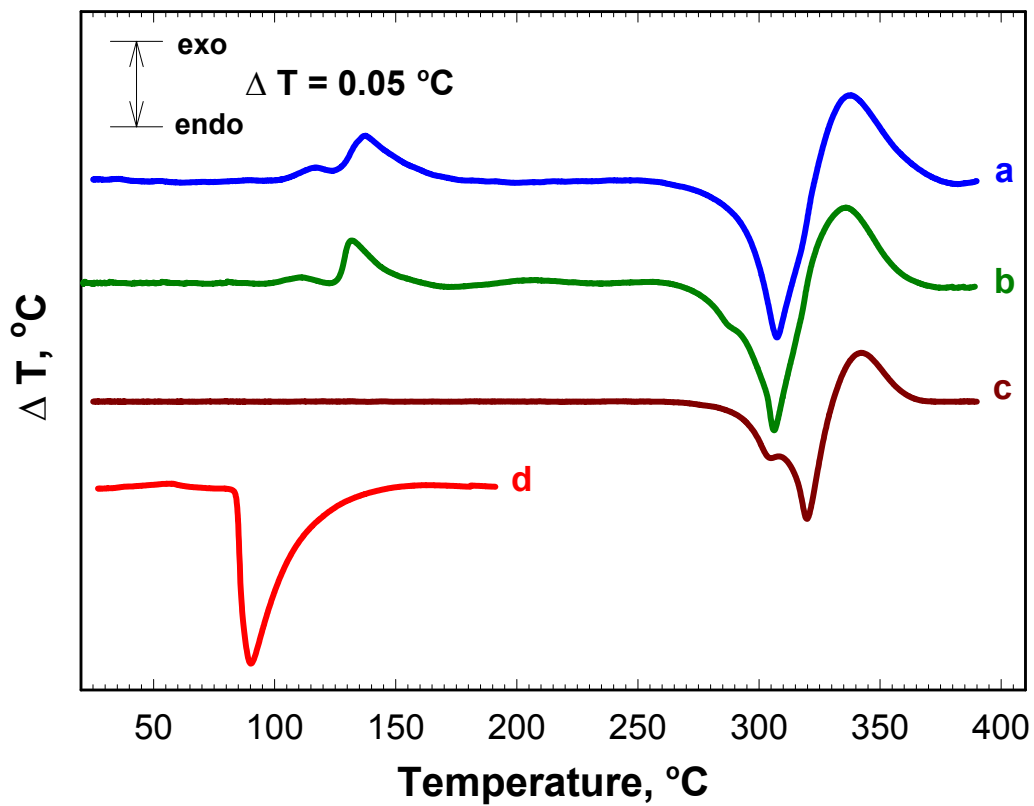
<sup>a</sup> Ames Laboratory, Iowa State University, Ames, IA 50011-3020, U.S.A. E-mail: balema@ameslab.gov

<sup>b</sup> Department of Materials Science and Engineering, Iowa State University, Ames, IA 50011, U.S.A. E-mail: vitkp@ameslab.gov

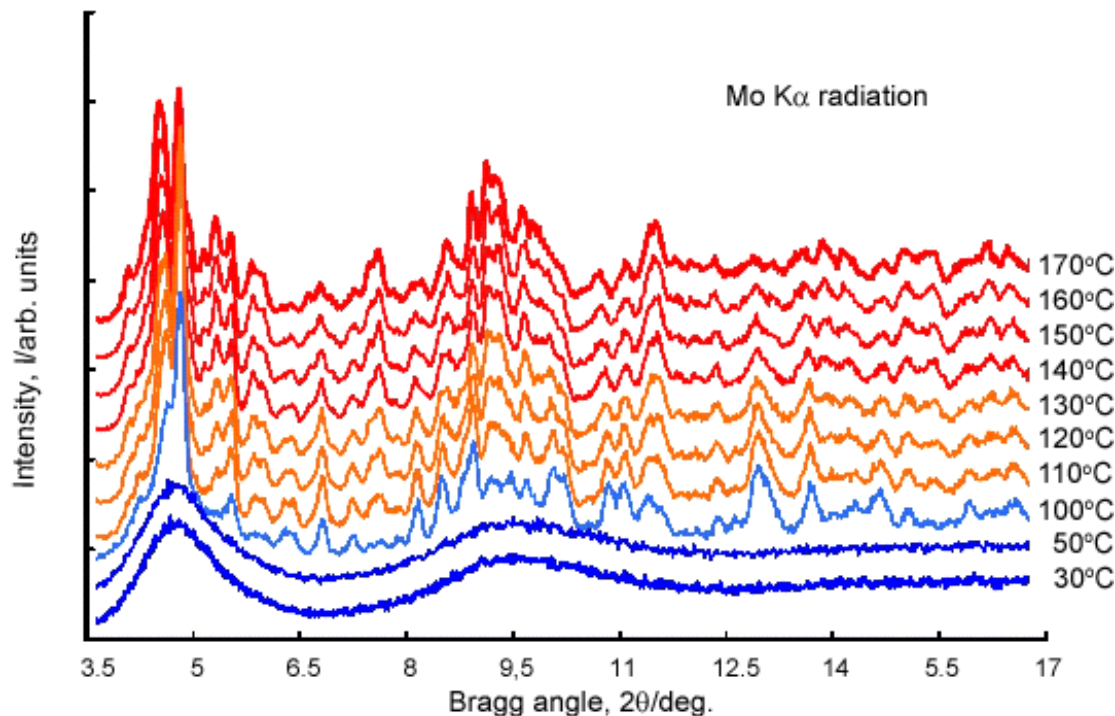
### Supplementary information



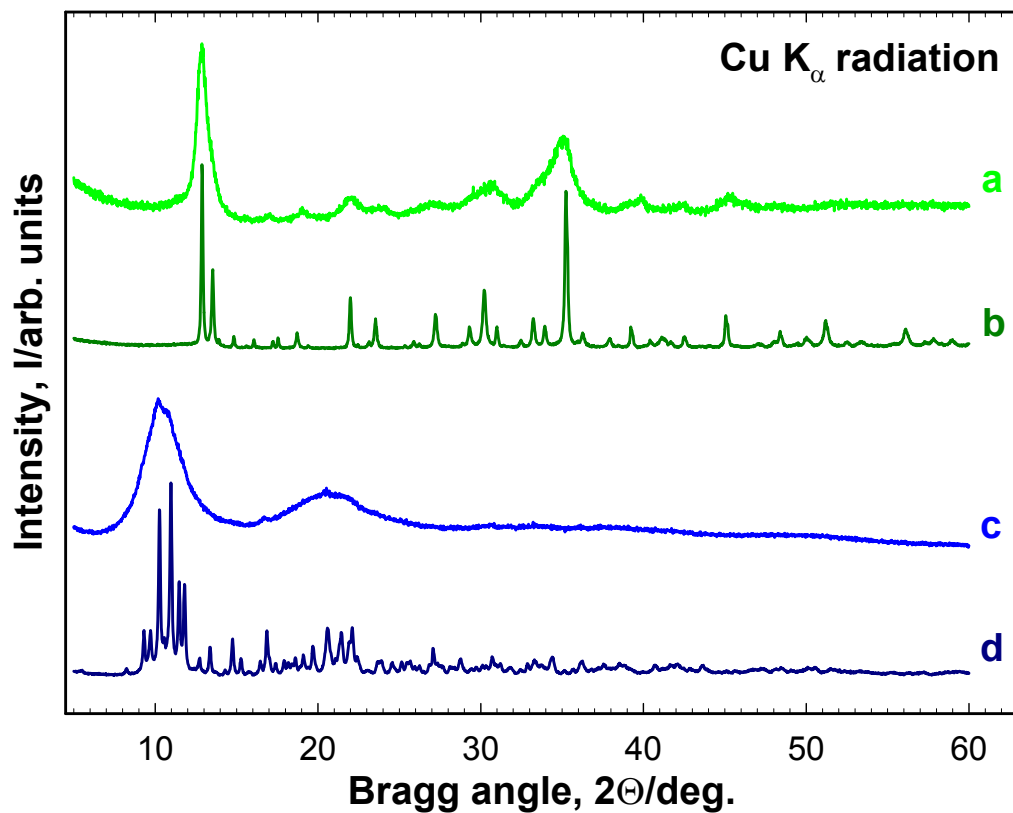
**Fig. 2.** The x-ray powder diffraction pattern of powders obtained during ball-milling of PtCl<sub>2</sub> with two equivalents of Ph<sub>3</sub>P for one hour (**a**), and *cis*-(Ph<sub>3</sub>P)<sub>2</sub>PtCl<sub>2</sub> **1** with 2.5 equivalents of anhydrous K<sub>2</sub>CO<sub>3</sub> for 6.5 hours (**b**). The asterisks in (**b**) indicate Bragg peaks of one of the reaction products - KCl. The excess of K<sub>2</sub>CO<sub>3</sub> is detected in the x-ray diffraction pattern as a group of Bragg peaks between ~30 and 33° 2θ as indicated by the arrow.



**Fig. 3.** The DTA traces of powders obtained during ball-milling of  $\text{PtCl}_2$  with two equivalents of  $\text{Ph}_3\text{P}$  for one hour (**a**); *cis*- $(\text{Ph}_3\text{P})_2\text{PtCl}_2$  **1** ball-milled for two hours (**b**); crystalline **1** (**c**), and crystalline  $\text{Ph}_3\text{P}$  (Aldrich) (**d**).



**Fig. 4.** The results of the *in-situ* high temperature x-ray powder diffraction study of the powder prepared by ball-milling of  $\text{PtCl}_2$  with two equivalents of  $\text{Ph}_3\text{P}$  for one hour. As temperature increases from 30 to 180°C, the characteristic amorphous diffraction pattern (broad halos observed between  $\sim 3.5 - 6.5$  and  $8 - 12.5^\circ 2\theta$  on  $\text{Mo K}\alpha$  radiation) transforms into that of a crystalline material. The temperature, where the appearance of the crystalline phase becomes visible from x-ray powder diffraction (100°C), is identical to that of the onset of the first exothermic event in the DTA trace of the same material (see Fig.3).



**Fig. 5.** The x-ray powder diffraction patterns of PtCl<sub>2</sub> ball-milled for one hour (**a**); crystalline PtCl<sub>2</sub> (Alfa Aester) (**b**); *cis*-(Ph<sub>3</sub>P)<sub>2</sub>PtCl<sub>2</sub> **1** ball-milled for two hours (**c**); and crystalline **1** (**d**). Considerable broadening of Bragg peaks in the patterns of ball-milled compounds indicates substantial loss of crystallinity in the mechanically processed samples.