Solvent-free mechanochemical synthesis of two Pt complexes: cis-(Ph₃P)₂PtCl₂ and cis-(Ph₃P)₂PtCO₃

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Supplementary information

Fig. 2. The x-ray powder diffraction pattern of powders obtained during ball-milling of PtCl₂ with two equivalents of Ph₃P for one hour (a), and cis-(Ph₃P)₂PtCl₂ I with 2.5 equivalents of anhydrous K₂CO₃ for 6.5 hours (b). The asterisks in (b) indicate Bragg peaks of one of the reaction products - KCl. The excess of K₂CO₃ 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 PtCl₂ with two equivalents of Ph₃P for one hour (a); cis-(Ph₃P)₂PtCl₂ 1 ball-milled for two hours (b); crystalline 1 (c), and crystalline Ph₃P (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 PtCl$_2$ with two equivalents of Ph$_3$P for one hour. As temperature increases from 30 to 180°C, the characteristic amorphous diffraction pattern (broad halos observed between ~3.5 – 6.5 and 8 – 12.5° 2Θ on 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 PtCl₂ ball-milled for one hour (a); crystalline PtCl₂ (Alfa Aester) (b); cis-(Ph₃P)₂PtCl₂ 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.