[MS20-P08] The Decomposition of Lead Nitrate

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Nitrates are often used in many synthetic procedures, including solid state synthesis [1]. This work includes the thermal behaviour and decomposition temperature of lead nitrate, a possible source of lead in such reactions. The thermal expansion coefficient has been determined by a number of sources prior to this study, and one source determined it to be non-linear [2]. Previous coefficients of thermal expansion include α = 24,50 ppm°C^{-1} [2]; 30,00 ppm°C^{-1} [3]; 31,9 ppm°C^{-1} [4]. The methods for determination of these values were by X-Ray diffraction, dilatometric and interferometric methods respectively. Previously reported decomposition temperatures include 380°C [2], and 250°C [5]. Disorder in the crystal structure of lead nitrate has been reported to form permanently upon grinding, although this should be reduced with time [6]. The mentioned properties were probed by Variable Temperature – Powder X-Ray Diffraction (VT-PXRD) and were measured in situ by a Bruker D8 Diffractometer fitted with an MRI TC-wide range temperature chamber, using MoKα1,2 Radiation. Measurements were made from 30°C to 400°C, in steps of 25°C beginning from 50°C. A final measurement was then taken again at 30°C. The cell parameter was determined by Rietveld refinement using Topas [7]. The structure determined by Nowotny and Heger was used as the starting model [8]. Refinements were performed sequentially using the refined of the preceding scan. It was found that the temperature dependence of the unit cell parameter in lead nitrate was non-linear, thus confirming previous observations qualitatively [2]. The quadratic describing this dependence is as follows: $a=3\times10^{-7}T^2+0.0001T+7.8435$ with R = 0.9809. The thermal expansion coefficient at room temperature was found to be lower than what was seen previously, with a value of 21.7ppm°C^{-1}. Several anomalous peaks were observed associated with Bragg planes, namely (311); (222); (004); (331); (042); and (422). Upon heating, all anomalous peaks saw a reduction in intensity, with reflections (004), (331) and (042) losing their anomalous peak entirely above 275°C. Lead oxide only formed at 325°C. A correlation between these phenomena has not yet been established. Investigations are in progress to determine if the anomalous peaks relate to a distinct phase or with disordered NO_3 groups.

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