[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 \(\alpha = 24.50\ \text{ppm}\degree\text{C}^{-1}\) [2]; 30.00 ppm\degree\text{C}^{-1} [3]; 31.9 ppm\degree\text{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\degree\text{C} [2], and 250\degree\text{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 MoK\(_\alpha\)1,2 Radiation. Measurements were made from 30\degree\text{C} to 400\degree\text{C}, in steps of 25\degree\text{C} beginning from 50\degree\text{C}. A final measurement was then taken again at 30\degree\text{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

\[a = 3 \times 10^{-7}T^2 + 0.0001T + 7.8435 \quad \text{with} \quad R = 0.9809.\]

The thermal expansion coefficient at room temperature was found to be lower than what was seen previously, with a value of 21.7 ppm\degree\text{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\degree\text{C}. Lead oxide only formed at 325\degree\text{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.


Keywords: thermal expansion; lead nitrate; variable temperature powder x-ray diffraction