

Supplementary Info

Coordination Geometry of Lead Carboxylates – Spectroscopic and Crystallographic Evidence

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Detailed Crystallographic Data Fitting

Because of the large a-axis dimension, initial unit-cell determinations using Mo- $k\alpha$ ($\lambda = 0.71073$ Å) failed because of poor resolution of the virtually overlapping diffraction spots. Data collected using Cu- $k\alpha$ ($\lambda = 1.54178$ Å) allowed proper resolution of the diffraction pattern and thereby allowed successful unit cell measurement and subsequently successful integration of the two-dimensional frame data. The high absorption of lead and the thin plate habit of the crystal conspired to prevent sufficient absorption correction of the Cu- $k\alpha$ diffraction data that resulted in a structure with high electron density excursions leading to level A alerts in the checkCIF report.¹ Fortunately, frame data, collected from the same crystal in the same spatial orientation, using Mo- $k\alpha$ could be integrated using the orientation matrix derived from the Cu- $k\alpha$ data, resulting in downgrading the alert level to B thus strongly suggesting that the electron density outliers in the final density map are artificial and caused by less than ideal absorption corrections.

Unit cell parameters were obtained from 36 data frames, $0.3^\circ \omega$, from three different sections of the Ewald sphere. The systematic absences in the diffraction data are consistent with $P2/c$ (No. 13) and Pc (No. 7). Solution in the centrosymmetric space group option yielded chemically reasonable and computationally stable results of refinement. The structures were solved using direct methods and refined with full-matrix, least-squares procedures on F^2 . All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were treated as idealized contributions. The carboxylate C-O distances were restrained to be similar. The oxygen atomic displacement parameters were constrained to be the same for chemically equivalent atoms per carboxylate anion. Anisotropic parameters were treated to global three-dimensional rigid-bond restraints. Although several absorption correction methods for the data-set were explored and with redundant multiscan correction yielding the lowest residuals, the high absorption coefficient of lead and the consistent thin-plate crystal habit conspired to form artifacts, the largest being 1 Å from the lead atom, in the final electron density difference map leading to level B and C alerts in the checkCIF report.¹ Scattering factors are contained in the SHELXTL 6.12 program library.²

1. Check.def File Version 5/2/14, <http://checkcif.iucr.org>.
2. G. M. Sheldrick, *Acta Crystallogr. A*, 2008, 64, 112 - 122.