SUPPORTING INFORMATION FOR

The Construction of a Functional Layered Solid using the Tetrakis(imidazolyl)borate Coordinating Anion

Barton H. Hamilton, Kathryn A. Kelly, Todd A. Wagler, Matthew P. Espe and Christopher J. Ziegler*.

Department of Chemistry, University of Akron,
Akron, Ohio 44325

Crystal Structure Experimental Details

A colorless plate-like crystal of $C_{12}H_{14.65}BN_9O_{4.32}Pb$, approximate dimensions 0.02 x 0.18 x 0.23 mm^3, was used for X-ray crystallographic analysis. The X-ray intensity data were measured at 100K (Bruker KRYO-FLEX) on a Bruker SMART APEX CCD-based X-ray diffractometer system equipped with a Mo-target X-ray tube (λ = 0.71073) operated at 2000 watts power. The detector was placed at a distance of 5.009 cm. from the crystal.

A total of 1497 frames were collected with a scan width of 0.3... in and an exposure time of 20 sec./frame. The total data collection time was 10.90 hours. The frames were integrated with the Bruker SAINT software package using a narrow-frame integration algorithm. The integration of the data using a triclinic unit cell yielded a total of 9837 reflections to a maximum 2θ angle of 66.13° (0.65 resolution), of which 5991 were independent (redundancy 1.64, completeness = 86.7%, R_{int} = 2.70%, R_{sig} = 5.04%) and 5391 (90.0%) were greater than 4σ(F). The final cell constants of a = 8.5337(8) , b = 10.7463(9) , c = 11.2476(10) , α = 74.6840(10)¡, β = 79.2840(10)¡, γ = 67.0120(10)¡, volume = 911.84(14) ^{3}, are based upon the refinement of the XYZ-centroids of 5744 reflections above 20 σ(I). Analysis of the data showed negligible decay during data collection. The data were corrected for absorption with the SADABS program (ratio of minimum to maximum apparent transmission: 0.690050).

The structure was solved and refined using the Bruker SHELXTL (Version 6.1) Software Package, using the space group P-1, with Z = 2 for the formula unit $C_{12}H_{14.65}BN_9O_{4.32}Pb$. The final anisotropic full-matrix least-squares refinement on F^2 converged at R1 = 3.16%, wR2 = 7.21% and a goodness-of-fit of 1.075. The largest peak on the final difference map was 2.690 $e^-/^3$. The calculated density for $C_{12}H_{14.65}BN_9O_{4.32}Pb$ is 2.084 g/cm^3 and F(000) is 543 e^-.

Solid-State NMR Experimental Details

Solid-state NMR spectra were obtained on a Varian Unityplus-200 (4.7T) spectrometer using Doty Scientific supersonic and standard MAS probes. All samples were packed into 7 mm silicon nitride rotors with Kel-F end caps. ^{15}N Bloch decay spectra were acquired using a 200 sec relaxation delay and a spinning speed of 2.7 kHz. ^{207}Pb spectra were acquired using a Hahn-echo sequence with a 5 sec relaxation delay and a spinning speed of 8 kHz. ^{15}N and ^{207}Pb chemical shifts were referenced to solid $^{15}NH_4{}^{15}NO_3$ (δ_{NH_4} = 0 ppm) and 0.5 M $Pb(NO_3)_2$ (δ_{Pb} = -2941 ppm) as an external reference, respectively. A 55 kHz decoupling field strength was used in all experiments.