$(EDT\text{-}TTF\text{-}I_2)_2PbI_3{\bullet}H_2O$: an ambient pressure metal with a β' donor slab topology

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$[(C_2H_5)_4N]PbI_3$

0.97 g (3.4 mmol) of $[(C_2H_5)_4N]I$ in 5 mL of aqueous hydriodic acid solution (57%) is added under stirring to a solution of 1.52 g of PbI_2 (3.3 mmol) in 6 mL of aqueous hydriodic acid solution (57%). A white precipitate appears and is dissolved by heating under reflux. Slow cooling down to 5°C affords pale yellow needles. After filtration, the crystals are washed with diethylether and dried under vacuum (2.14 g, 3.0 mmol, 90%), mp > 315°C. Anal Calcd for $C_8H_{20}NI_3Pb$: C, 13.38; H, 2.81; N, 1.958. Found C, 13.15; H, 2.61; N, 1.93. EDX Calcd (normalised weight): I, 64.8; Pb, 35.2. Found: I, 65.7; Pb, 34.3.

Partial X-Ray structural elucidation confirms the face-sharing connection between the PbI_6 octahedra in the unidimensional polymeric anion $[PbI_3^-]$. Crystal data. $C_{24}H_{60}I_9N_3Pb_3$, M = 2154.42, hexagonal, a = 18.954(2), c = 8.1838(8) Å, U = 2546.2(5) $Å^3$, T = 293(2) K, space group $P6_3/m$ (no. 176), Z = 2, μ(Mo-K_α) = 15.4 mm^{-1}, 3867 reflections measured, 1789 unique (R_{int} = 0.1165) which were used in all calculations. The final $wR(F^2)$ was 0.1928 (all data).

X Ray powder diffraction pattern :