Synthesis, Spectroscopic and X-Ray Structure Characterisation of Bis(tetramethylammonium), Bis(tetraethylammonium) and Bis(tetrapropylammonium) Tetrathiotungstates

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The three new tetraalkylammonium tetrathiotungstates $((Me)_4N)_2[WS_4]$ (**1**), $((Et)_4N)_2[WS_4]$ (**2**) and $((nPr)_4N)_2[WS_4]$ (**3**) were prepared *via* a direct salt substitution using $(NH_4)_2[WS_4]$ as starting material. Compound **1** crystallises in the chiral orthorhombic space group $P2_12_12_1$ with $a = 8.9433(4)$, $b = 15.5658(9)$ and $c = 37.279(2)$ Å. Compound **2** crystallises in space group $P2_1/n$ with lattice parameters $a = 16.6695(12)$, $b = 9.3415(6)$, $c = 16.9965(13)$ Å and $\beta = 117.185(15)°$. The third compound **3** crystallises in space group $C2/c$ with the lattice parameters $a = 32.440(2)$, $b = 13.8453(6)$, $c = 15.0563(10)$ Å and $\beta = 109.19(7)°$. The structures of all compounds consist of slightly distorted $[WS_4]^{2-}$ tetrahedra and tetraalkylammonium cations which are packed in different ways. One interesting observation is that the disorder of parts of the alkyl groups decreases with increasing chain length. The IR and Raman spectra show the vibrations of the $[WS_4]^{2-}$ tetrahedron with a slight shift with increasing alkyl chain length. The most prominent IR-band of the $[WS_4]^{2-}$ tetrahedra is broad but not split, indicating that the distortion of the tetrahedra is small.

Key words: Crystal Structure, Tetrathiotungstates, IR and Raman Spectroscopy