Electronic Supporting Information

Direct Synthesis of MoS₂ or MoO₃ via Thermolysis of a Dialkyl Dithiocarbamato Molybdenum(IV) Complex

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Experimental

Synthesis of Mo(DTC)₄

 $Mo(DTC)_4$ was prepared using the method detailed in Lewis et al (Chem. Mater. 2015). Briefly, molybdenum hexacarbonyl (1.0 eq., 3.7 mmol) and tetraethylthiuram disulphide (2.0 eq.,7.4 mmol) were mixed in 40 mL acetone and then heated under reflux at 80 °C for 2 h. The product was then left to cool down to room temperature and stored in freezer overnight to allow full crystallisation to occur. The dark purple crystalline material produced was isolated by vacuum filtration and rinsed with pentane (3 × 20 mL). Anal. Calc. for C₂₀H₄₀N₄S₈Mo: C, 34.9; H 5.9; N, 8.1%. Found: C, 34.5; H 5.8; N, 8.0%. FT-IR (solid) v_{max} /cm⁻¹: 2973 (w), 2927 (w), 2867 (w), 1516 (m), 1495 (m), 1454 (m), 1435 (m), 1376 (m), 1350 (m), 1274 (m), 1205 (m), 1149 (m), 1090 (m), 1068 (m), 987 (m).

Solventless Thermolysis

Thermolysis was carried out by heating the $Mo(DTC)_4$ powder (placed in a ceramic boat and positioned in the centre of a Carbolite MTF furnace) to 450°C and kept at this temperature for one hour under argon or air to produce MoS_2 or MoO_3 respectively. The final products (black/grey powders) were collected after the system was cooled to room temperature.



