Solvothermal Synthesis, Polychalcogenides

The reaction of manganese(II)-chloride-tetrahydrate, caesium triselenide and elemental selenium or tellurium in 1,2-ethanediamine (en) under solvothermal conditions leads to the formation of two new isostructural compounds $[\text{Mn}(\text{en})_3]\text{Se}_3$ (1) and $[\text{Mn}(\text{en})_3]\text{TeSe}_2$ (2). The compounds crystallize in the orthorhombic space group $Pbcn$ with the lattice parameters $a = 1149.39(9)$, $b = 1506.83(11)$, $c = 935.96(6)$ pm for 1 and $a = 1184.1(2)$, $b = 1495.3(2)$, $c = 949.8(1)$ pm for 2. Their crystal structures are built up of $[\text{Mn}(\text{en})_3]^{2+}$ cations and $\text{Se}_3^{2-}$ or $\text{TeSe}_2^{2-}$ anions, respectively. Each cation is surrounded by six next neighbouring anions, and vice versa. Between the cations and the anions hydrogen bonding is observed. The thermal behaviour was investigated using differential thermal analysis, thermogravimetry as well as X-ray powder diffraction. Completely different properties were found. Compound 1 decomposes in two distinct endothermic steps, while compound 2 shows only one endothermic peak. The weight loss for 1 corresponds roughly to the emission of all en molecules, whereas the weight loss for 2 is significantly lower. The final products are composed of MnSe$_2$ and elemental Se or Te, respectively, and an unknown crystalline phase which is different for the two samples.