



to 1MHz every 2 minutes, throughout the entire freeze-drying process. One of the plain vials close to the TVIS vials had a type-K thermocouple placed at the bottom centre of the vial, to provide a representative temperature for the TVIS vial during the relatively steady state conditions of the annealing stage (i.e. when the solution is not experiencing any changes in state). A full load of vials was then placed on a single shelf of a Virtis Advantage Plus benchtop Freeze-dryer, with the cluster of solution-filled vials at the centre of an array of empty vials, and a freeze drying protocol with an annealing step used to dry the solution. Scanning electron microscopy (SEM) images of the freeze-