TTO Troubleshooting:

Verifying the quality of the epoxy bond for the TTO thermometer shoes (4084-580) and heater shoes (4084-585)

In the Thermal Transport Option (TTO), a small gold-plated copper shoe is used as the interface between the pre-packaged Cernox thermometer sensor (or resistive heater) and the lead of the customer's sample. The model used for analyzing the thermal data from the hot and cold thermometers relies on fast thermal equilibration times within the shoe assemblies. This means that the epoxy bond between the thermometer (or heater) and the shoe is critical.

In order to verify the integrity of your current set of thermometers and heater for TTO, we recommend simply measuring one of the nickel alloy standard samples that was delivered with your TTO. In the graph below, we show a good data set (left), indicating that the epoxy bonds are intact, and a bad data set (right) indicating a problem with at least one of the epoxy bonds. Notable anomalies in the data on the right side are:

1) higher scatter in the thermal conductivity data (top plot in upper graphs)
2) higher estimated error in the thermal conductivity (Cond. Std. Dev.)
3) time constants tau1 and tau2 from the thermal model that are very close in value and sometimes degenerate; degeneracy of tau1 and tau2 implies that the model was unsuccessful in resolving two distinct time constants for the heat diffusion and is a general sign of a breakdown of the model
4) in the .RAW file (bottom graph), a clear deviation of the data from the curve fit is seen in the middle and at the end of the measurement. This will lead to systematic errors in the reported value of the thermal conductivity and Seebeck coefficient since these are based on the extrapolated value of Delta_T = T_hot – T_cold
5) in some cases, the Delta_T fit may still look good but the Seebeck data (Delta_V in .RAW file) will not be well fit by the model. This is because the Seebeck fitting takes the tau1 and tau2 values obtained in the Delta_T fit and does a linear regression to account for a background voltage drift and offset. The Seebeck data will be a measure of the true thermal time constants of the material that is responsible for the Seebeck voltage (the sample and shoe), so it will not reflect the same tau1 and tau2 values. See Figure 2 which shows a good Delta_T fit (upper panel) and poor Delta_V fit (lower panel) at T~240 K on the QD nickel alloy sample. This is a different data set than in Figure 1.

NOTE: if you observe similar features when measuring a research sample, the cause is likely a poor thermal link between the leads and the sample as this will create the same long thermal diffusion time for the shoe assembly. Poor contact is most often due to an epoxy bond that has low cross-sectional area or is too thick, or the epoxy did not bond sufficiently to the sample. Please contact an applications specialist at apps@qdusa.com if you would like to discuss sample mounting. To verify that the shoes are working properly, measure the nickel alloy standard sample which is monolithic and hence does

not have any issues with heat diffusion between its leads and the main body of the sample.

Quantum Design uses a special glass-filled epoxy for the bond between the sensors and the shoes, so if there is a bond failure we strongly urge you to contact your local Quantum Design service representative for a replacement shoe assembly instead of attempting to repair it yourself. For instance, gluing the sensor with 7031 varnish does NOT produce a robust bond over multiple thermal cycles.

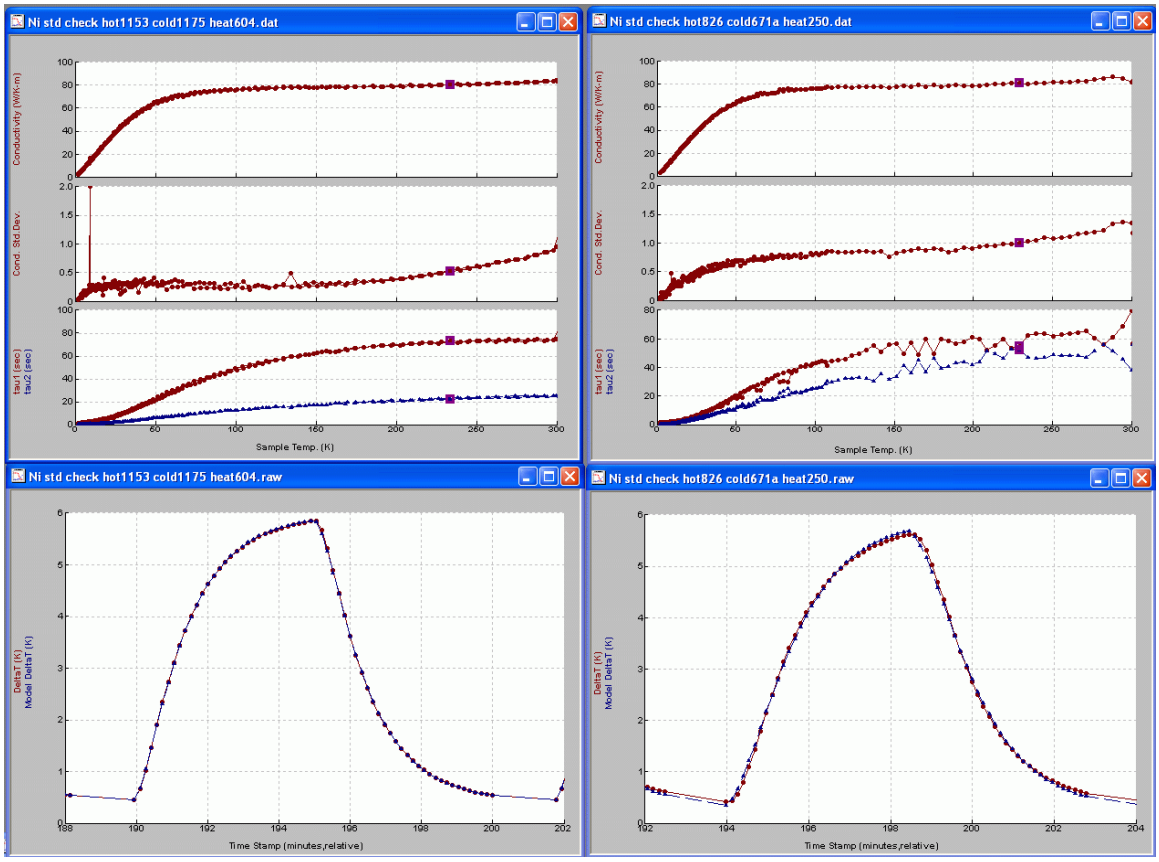

Figure 1: example of a TTO temperature dependence measurement on a TTO nickel alloy standard sample using all good sensor shoes (left) and using a hot thermometer shoe with a degraded epoxy bond (right). See body text for more details. In this case, a careful visual inspection under a microscope revealed that there was a gap between the Cernox sensor and the shoe.

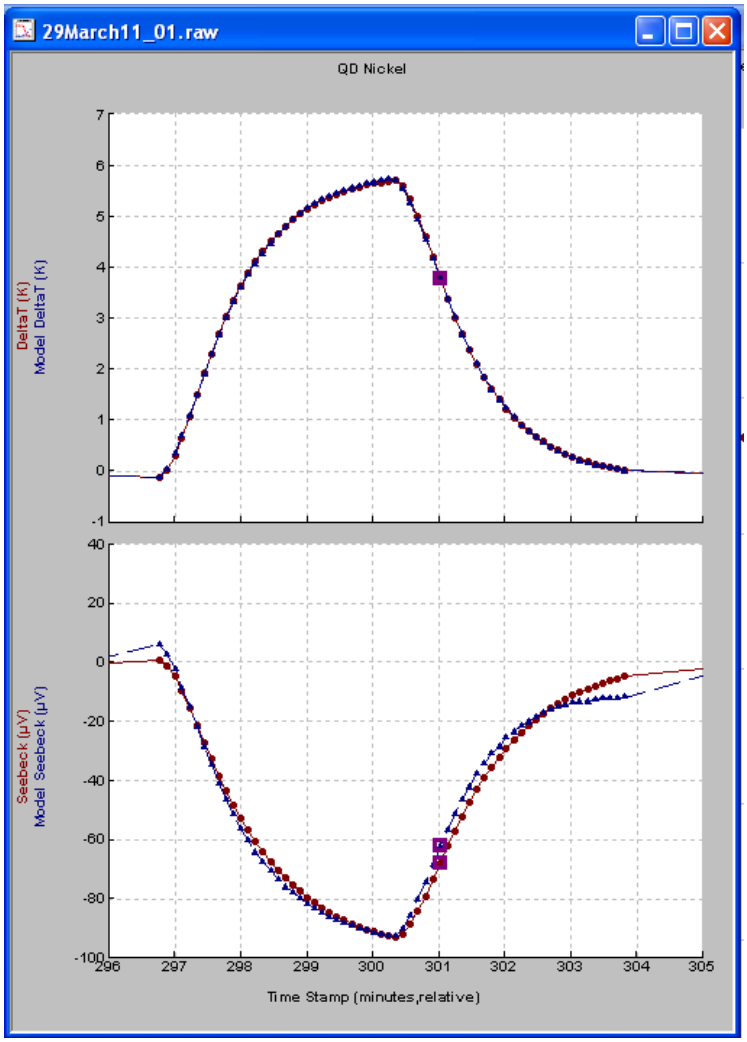

Figure 2: data on QD nickel alloy sample near 240 K showing a good thermal fit (upper) and poor Seebeck fit (lower). The Seebeck.Std.Dev. value throughout this data set is about 2x higher here than in a typical data set on the QD standard sample.