

Calibration made easy for the DSC82x, DSC1, and the STAR^e Software

The STAR^e software and METTLER TOLEDO DSC's offer a variety of ways to calibrate your instrument. When the instrument was installed the service engineer verified that the calibration was correct and made any necessary adjustments to insure your DSC meets factory specifications. METTLER TOLEDO recommends you come up with a calibration schedule that meets your personal needs. Your local METTLER TOLEDO sales or technical representative will be happy to help you determine a calibration schedule that is appropriate for your needs.

First off METTLER TOLEDO has three major calibrations which are performed on the DSC: Temperature, Heat Flow, and Tau Lag. These are performed with two types of calibration, fully automatic (TOTAL CALIBRATION) and manual (SINGLE CALIBRATION). The Total Calibration uses Indium and Zinc in the same crucible (see METTLER TOLEDO file "IndiumZincPrep.pdf") and takes about three hours to complete. This calibration performs a two point adjustment for temperature and Tau Lag, but only a single point adjustment for heat flow. We commonly refer to this as a course calibration, however this is the only calibration we recommend for Tau Lag and it only needs to be performed once a year or whenever the furnace characteristics change (i.e new DSC sensor, new furnace PT100, new furnace, large change in furnace mass). Once the total calibration is completed we strongly recommend you still perform the manual calibration which is described in greater detail below.

For all general purposes manual calibration of the DSC is the best way to calibrate the instrument on a regular basis, and after a Total Calibration has been performed. First you must prepare your standard in separate crucibles. You can use as many standards, and whichever standards you like. METTELR TOELDO typically uses Indium and Zinc, but also offer Lead and Tin. You can use low temperature standards the same way as we will describe below. For sample preparation of indium see METTELR TOELDO file "Indiumprep.pdf". Once you have your samples prepared you must run the standards in the DSC taking into account any special needs of the standard for preparation such as pre-melting. For Indium and Zinc you can use the standard method "Check exo[^]In" and "Check exo[^]Zn". You can run as many standards as you would like and you can run the standard as many times as you would like for averaging, but keep in mind certain standards susceptibility to oxidation which could effect your end calibration results. Once you have obtained all of the curve values for Onset and normalized heat flow of each standard we can begin the calibration procedure.

Going to the Routine Window of your DSC be sure the instrument is in the Standby mode. You can only make an adjustment of the DSC while in the standby mode. Select “Calib/Adjust” “Single Temp...” as shown in figure 1 below and you will see the window below in figure 2.

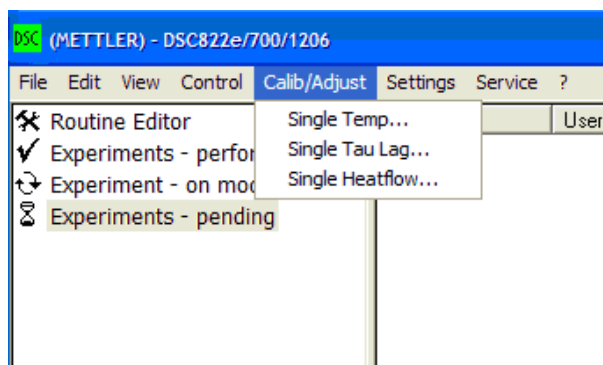


Figure 1

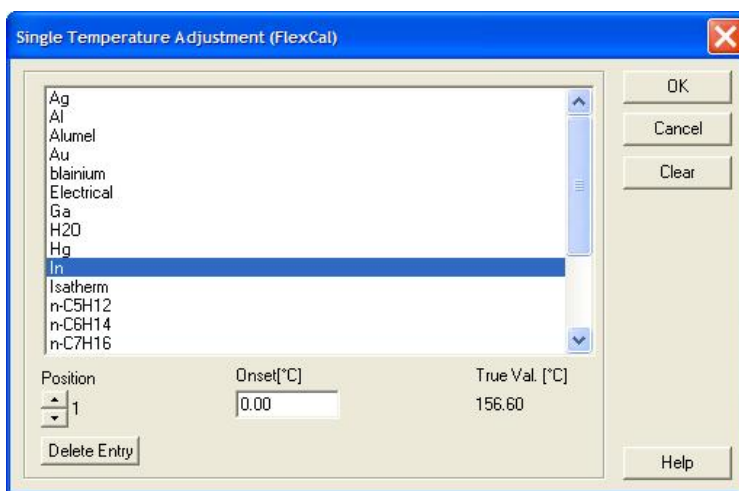


Figure 2

Select the first standard you are going to use, in this case Indium from the list, and you will see the True Val, or theoretical value of the standard. In the white box names Onset, enter the onset value of the measured curve. Select the Position down key as indicated in Figure 3 on the next page. Now you can enter your next standard and repeat the above steps. If you choose you can enter multiple Indium standards in positions 1 through 3 and the software will average the values for you or you can enter multiple standards. Once you have entered all of your standards be sure the position is on the very last standard you entered. The software will only calibrate the values up to the selected position, so if you leave the position on position 1 and choose O.K. it will only perform a single point calibration. Once you have the position on the last standard entered choose OK and the window displayed in figure 4 will pop up.

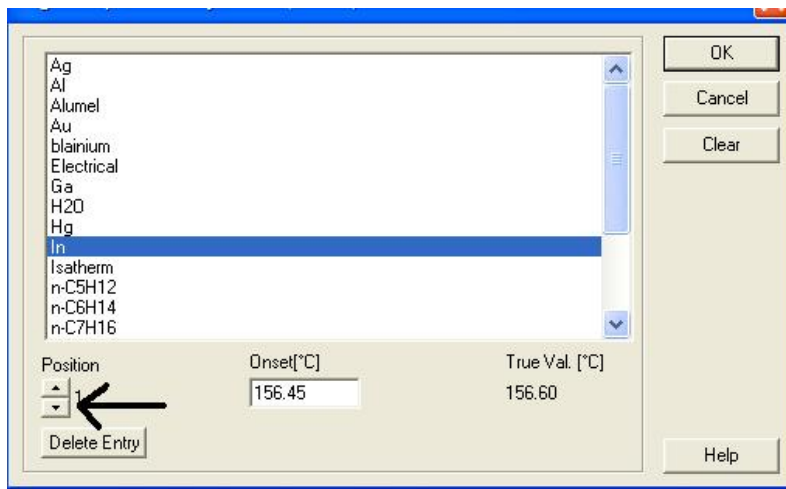


Figure 3

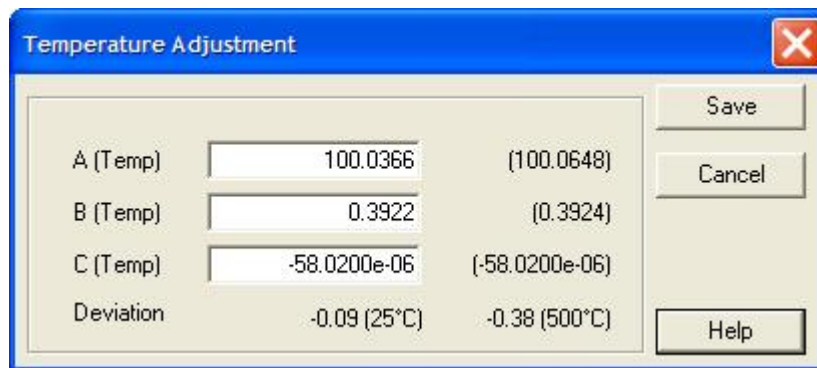


Figure 4

Figure 4 shows the calibration adjustment overview for you to review. You can see the A, B, and C adjustment values for the furnace PT100. In the right hand grey column are the old values, and in the white boxes are the new values. At the bottom of the screen you can also see the deviations across the temperature scale. If at this point you see some drastic changes or deviation, simple cancel and start over. If everything looks good, then choose “save”, you have no completed the temperature calibration.

Next we are going to calibrate the “heat flow” which works in a similar way. Go to “Calib/Adjust” “Single Temp...” as shown in figure 1 above and you will see the window below in figure 5. The heat flow calibration is slightly different than the temperature calibration as you must choose the purge gas and crucible type before commencing with the standards calibration. ****IMPORTANT- You must choose “Air” and “Aluminum Standard 40ul” regardless of whether you are using air or nitrogen as purge gas in order to keep the integrity of the FlexCal® Routine!*** Choose Air and then the Aluminum standard 40ul. Now you can proceed to the calibration material just as you did for the temperature calibration.

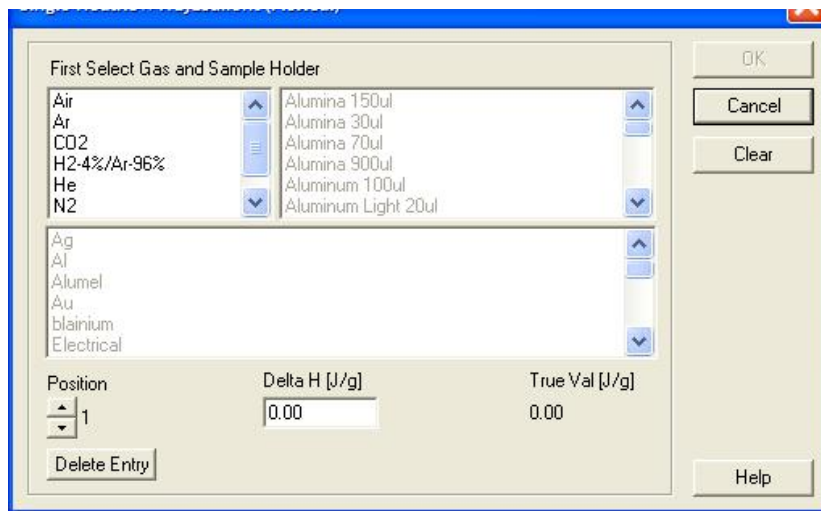


Figure 5

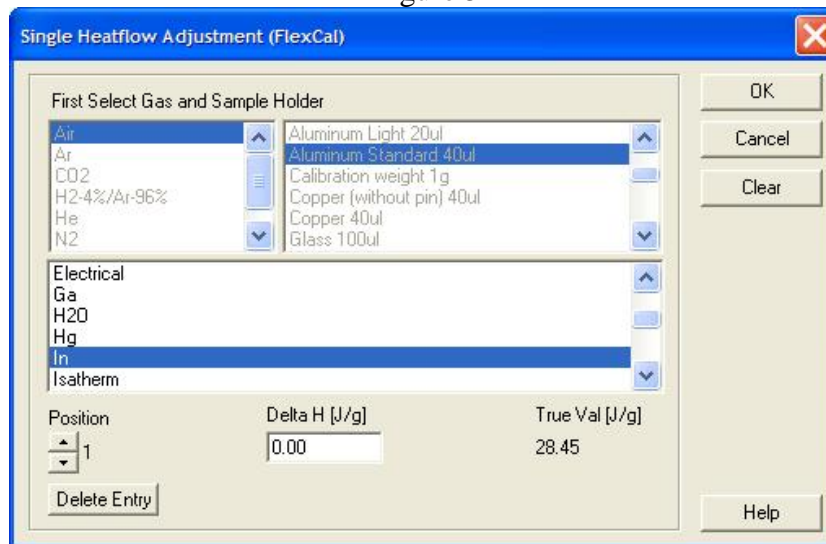


Figure 6

Just like with the temperature calibration once you select your standard, Indium in this case, you will see the “True Value” or theoretical value appear in grey next to the open white box. In the open white box enter the normalized heat flow (as an absolute value). Choose the position down key for the second position and choose the next standard. Now you can enter your next standard and repeat the above steps. If you choose you can enter multiple Indium standards in positions 1 through 3 and the software will average the values for you or you can enter multiple different standards. When you are done entering all of your standards and have the position on the last entered position choose “OK” and you will see the window in figure 7.

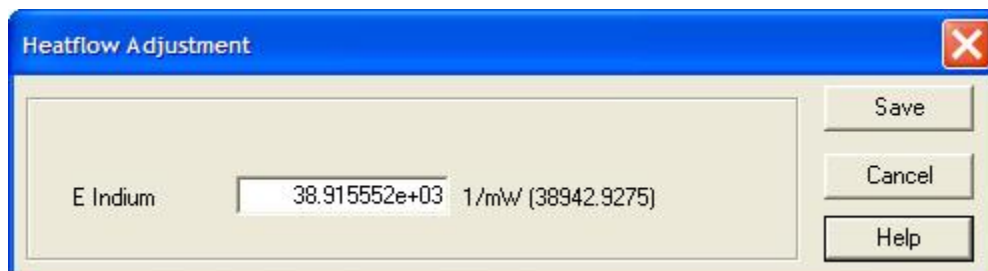


Figure 7

The values displayed in the above window are a calculation of the heat flow calibration. In parenthesis to the right you will see the old value; in the white box is the new value. If this value deviated by a large amount the software will not give you the option to save the calibration, but providing you can save the value choose "Save" and the heat flow calibration is completed.

After you have completed this calibration you should go back and re-run the "Check DSC exo[^]In, and Check DSC exo[^]Zn. If the instrument passes you are ready to continue, if not then using the new runs re-adjust the instrument with the same procedure and continue until both standards pass. If you continue to struggle with the calibration please contact your sales representative or our technical support group for further help.

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