Ref.: SSI-TA-JG-014

# STANDARD CLEANING AND CALIBRATION PROCEDURE

# FOR TMA-50 AND TMA-50H

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## INTRODUCTION

The following procedure is to be used for the maintenance and calibration of the TMA-50 and TMA-50H. The TMA, or Thermomechanical Analyzer, is a versatile instrument which measures the coefficient of thermal expansion (CTE), penetration, or tensile strength (mainly of thin films and limp fibers) of various materials. The performance of these tests is done through the use of a magnetic force coil which applies positive or negative loads to the sample, and a linear variable displacement transformer which measures the expansion or contraction of the sample. The TMA is almost always used to measure samples well before their point of degradation. However, some materials will be degraded both purposely and accidentally, leaving deposits in and on the measuring surfaces which must be cleaned.

The following materials and supplies will be needed for cleaning and calibration:

1. A supply of Oxygen ($O_2$).
2. A supply of 90:10 mixture of $N_2/H_2$ (Optional).
3. Certified melt point standards (Indium, Tin, etc.).
4. Certified thermal expansion standards (Borosilicate glass, etc.).
5. 0 - 1 inch Micrometers.
6. Aluminum crimp cells (P/N 201-52943-00)

## CLEANING

Method 1 (Standard)

This cleaning method uses $O_2$ or air to reduce the built up organic residues in the furnace and on the active parts (e.g. quartz probe, sample platform, etc.) of the balance assembly. This simple procedure is as follows:

1. Connect oxidative purge gas to the rear of the instrument.
2. Set flow rate to 50 ml/min.
3. Set heating rate to 20℃/min.
4. Set hold temp. to 700℃/min.
5. Set hold time to 120 min.
6. Set PID's to 10, 10, 10.
7. Lift the probe using the micrometer adjustment so that it does not contact the sample platform to allow complete exposure of the probe tip.
8. Start the run.

When the run is complete, the inside of the furnace should have a clean white appearance. The quartz support tube, quartz probe and sample platform should have a clean opaque appearance.

Method 2 (Optional)

This optional method uses a mixture of 90% $N_2$ and 10% $H_2$ and can be used to clean the TMA if the residue is mostly inorganic, or if the $O_2$ method does not sufficiently clean the furnace and active measuring parts. The same instrument parameters should be used as in Method 1.

## CALIBRATION

Since the TMA-50 measures the change in dimension (displacement) of a sample over a given temperature range the instrument must be calibrated for both temperature and displacement signals. The temperature will be calibrated using pure metal melt point standards. These standard reference materials (SRM) are available from the National Institute of Standards and Technology (N.I.S.T.). The displacement signal will be calibrated using any solid material of known length as determined by the micrometers.

Temperature Calibration

The method for temperature calibration of the TMA-50 is relatively easy to perform and like the DSC-50, pure metal melt point standards can be used. However, the way in which the melt point is determined will be different. This method, similar to parallel plate rheometry, uses a pure metal with a known melting point sandwiched between two flat "platens."

The upper platen, carrying a force load transferred by the TMA expansion probe, tries to flatten the sandwiched material which resists this action due to its natural crystalline structure. As the temperature increases, however, the material melts, begins to flow and is displaced by the compressive action from the upper platen. This causes the upper platen to move downward towards the base platen which is detected by the LVDT.

Like the other Series 50 thermal analysis instruments there are two methods of temperature calibration. The first is the standard method which will be covered here. The other method is a software calibration. This procedure is discussed in the TA-50WSI instruction manual (pp. 3-108) and will not be covered here.

To perform the standard temperature calibration method on the TMA-50 set all calibration constants (FUNC 7) to null:

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| 1. | Temp Gain | = | 1.000 | Temp Offset | = | 0.0 | |
| 2. | T1 Expected | = | 154.0 | T1 Measured | = | 154.0 | |
| 3. | T2 Expected | = | 230.0 | T2 Measured | = | 230.0 | |
| 4. | SIG Gain (TMA) | = | 1.000 | | | | |
| 5. | SG1 Expected | = | 1000 | SG1 Measured | = | 1000 | |
| 6. | SG2 Expected | = | 100 | SG2 Measured | = | 100 | |
| 7. | Chuck Offset | = | 0.0 | | | | |

Place a small amount of powdered sample (about 2mg of Indium, Tin, etc.) in an Aluminum sample cell and cover with a lid. The lid may have to be trimmed to prevent any binding against the cell wall. With the furnace in the up position and the sample probe resting on the sample platform with -1.0 grams of force, zero out the signal. Lower the furnace and raise the sample probe from the sample platform and mount the sample cell directly beneath the probe. Lower the probe onto the center of the cell lid. Again, close the furnace and allow the signal to settle out.

Now set the run parameters to the following:

1. Heating Rate: 10℃/min.
2. Hold Temp: 25℃ above onset temp.
3. Hold Time: 0 min.
4. PID's: 10, 10, 10

5. ATM: $N_2$ at 50 ml/min.
6. Sampling Interval: 1.0 sec.
7. Sample Load: -1.0 grams

Once the run has been completed the data can be analyzed. The point at which the probe begins to fall is called the onset point. This point is determined (using Tangent Temp/Time analysis) by extrapolating two lines tangent to either slope and finding their intersect on the temperature scale. Once two onset temperatures have been determined, the data can be entered into the TMA-50 calibration function (FUNC 7) using the standard method:

1. Enter Calibration Function (Press FUNC, go to 7, and press Enter).
2. Press Enter 3 times.
3. Enter Measured T1 temp using the and arrow keys and press ENT.
4. Enter Measured T2 temp using the and arrow keys and press ENT.
5. Press FUNC key to return to main screen.
6. Re-analyze samples to confirm calibration.

Length Calibration

Calibrating the length signal is relatively simple. Use a sample with a known length dimension. This can be determined with a good set of calibrated 0-1 inch micrometers. The micrometers should read in millimeters to three decimal places. The sample should not be any longer than 2.500mm and the measuring surfaces should be flat, smooth, and parallel. This calibration should be performed at ambient temperature in the following manner:

1. Measure sample length (using micrometers) in mm to three decimal places.
2. Enter calibration function (FUNC 7) and enter through to SIG GAIN (TMA).
3. Set SIG GAIN to 1.000 and press ENT.
4. Enter sample length into SG1 Expected and SG1 Measured.
5. With the furnace in the down position and the sample probe resting on the sample platform (-1.0g force) zero out the TMA signal.
6. Raise the sample probe, mount the sample directly beneath the probe and then lower the probe onto the sample. Allow the signal to settle.
7. Record the TMA signal on the LCD screen. Return to the calibration function (FUNC 7) and enter the value into SG1 measured.
8. Once this value is entered into calibration, the measured TMA signal value on the main screen will reflect the actual sample length.

## BASELINE

The ideal TMA baseline, like any other thermal analysis instrument, would be a line parallel to the X-axis. This is never the case and even more so with the TMA since *everything* expands or contracts with a change in temperature and that includes the active measuring components of the TMA system. The standard material of these measuring components is Fused Quartz. Fused Quartz has the lowest CTE of any material which makes it best suited for TMA measuring applications. For high temperature applications (TMA-50H) Alumina parts are used because of their resistance to temperatures up to and above 1500℃.

The TMA-50 Baseline varies from instrument to instrument and will drift in both positive and negative directions. This variation is due mainly to thermal inconsistencies throughout the active measuring area. Another cause is the amount of force that is applied by the expansion probe. In the case of Alumina, the baseline will appear to drift even more due to its higher CTE compared to Quartz.

To correct for baseline drift the standard baseline correction routine may be used. This is performed by making a blank run (no sample on platform) under the exact same conditions as the sample and then subtracting out the baseline from the sample file. This routine is used only when looking for qualitative changes in samples where the elimination of baseline drift is desirable. It is not the intended method for obtaining quantitative expansion data. This routine is covered (pp. 3-97) in the TA-50WSI instruction manual.

For obtaining accurate quantitative dimensional changes, Total Expansion Correction analysis function should be used. This software function not only eliminates normal baseline drift but also accounts for other instrumental inconsistencies (background drift). This background drift interferes with the TMA's ability to accurately measure the true dimensional changes of the sample. This method eliminates both baseline and background drift by measuring a sample with a known CTE. The difference between the true CTE and the measured CTE is saved as a calibration file, which is then added or subtracted to the unknown sample file which has been run under the same sampling conditions. That is, if the measured CTE of the calibration standard is less than what it should be then the difference is added to the unknown sample file. If the measured CTE is more than what it should be then it will be subtracted from the unknown sample file.

The Total Expansion Correction routine is performed in the following manner:

1. Perform an analysis of the unknown sample.
2. Perform an analysis of the NIST calibration standard (SRM-731 Borosilicate glass) using the same analysis parameters as the unknown sample.
3. Open the calibration sample file in the Analysis program and create a Total Expansion Correction file.
4. Open the Manipulate pull down menu. Select "Expansion[Total]Correction".
5. The "Reference Data" window opens. Select the "Add" button.
6. The "Approximate Function" window opens. Select the "Standard" button.
7. The "Browse" window opens. Highlight the SRM-731 Borosilicate Glass file and Click on the "OK" button.
8. The "Approximate Function" window re-opens displaying the certified NIST SRM-731 coefficient of thermal expansion data. The "Reference File" box located above the CTE data will be empty. Click on the "Files" button.
9. The data files screen appears. Highlight the TMA file to be corrected (in this case it would be the SRM-731 file) and Click on the "Open" button.
10. The "Approximate Function" window re-opens with the objective file name displayed in the "Reference File" box. Click on the "OK" button.
11. You have now created a Total Expansion Correction file. Give it a name and save it as a .TXP file.
12. Close out the SRM-731 file and open the Unknown sample file.
13. Open the Manipulate window in the Analysis program and select "Total Expansion Correction."
14. The "Reference Data" window opens. Highlight the named correction file and Click on "OK." Total Expansion Correction is now performed on the objective file and subsequent analysis can be performed.

The above procedure covers the use of the Total Expansion Correction function.

A new Total Expansion Correction file must be made whenever there is a change in sample type or a change in the program parameters. More information regarding the methodology behind this function can be found in the TASYS help screens.

## CONCLUSION

The TMA-50 is a highly sensitive instrument which requires proper care and attention. The operation is simple and, with a little time and patience, can easily be temperature and length calibrated. Obviously a clean instrument will provide the user with quality data for a longer time than one that is not properly maintained. The frequency of cleaning, however, will vary from user to user and depend on the type of samples tested. The general rule of thumb is to clean and calibrate the instrument once a month.