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STANDARD CLEANING AND CALIBRATION PROCEDURE

FOR TGA-50(H) AND TGA-51(H)

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INTRODUCTION

The following procedure is to be used for the maintenance and calibration of the TGA-50(H) and TGA-51(H). Because the TGA measures the weight loss of materials by thermal separation, some of the evolved material can be deposited onto the active parts of the weighing assembly (e.g. quartz rod, hanging pan, sample cell). This can cause erroneous readings in subsequent analyses by showing excessive or exaggerated weight losses which are not indicative of those particular samples.

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

1. A supply of Oxygen (O_2) or air.
2. A supply of 90/10 mixture of N_2/H_2 (Optional).
3. Certified Curie Point standards (SRM-761)
4. Calibrated mg weight set (P/N 201-52724-01)
5. TGA calibration magnet (P/N 220-96401-00)

CLEANING

Method 1 (Standard)

This cleaning method requires O_2 or air to reduce the built up organic residues in the furnace and on the active parts [e.g. quartz rod, Platinum (Pt) cell holder, 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°C/min.
4. Set hold temp. to 700°C/min.
5. Set hold time to 120 min.

6. Set PID's to 10, 10, 10.
7. Start the run (with empty Pt cell).
8. When the run is complete the inside of the furnace and the purge inlet tube should have a clean, white appearance. The quartz rod and Pt cell and holder should also be clean.

Method 2 (Optional)

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

CALIBRATION

Since the TGA measures the change in weight of a sample over a given temperature range, the instrument must be calibrated for both temperature and weight signals. *Weight calibration is not necessary if the TGA analysis is to be performed in percent weight loss only.* The temperature will be calibrated using magnetic transition or Curie point standards. These standard reference materials (SRM) are available from the National Institute of Standards and Technology (N.I.S.T.) as SRM-761. The weight signal will be calibrated using a mg weight kit with certifications traceable to N.I.S.T. If traceability is of no concern then the weight kit supplied with the TGA can be used.

Temperature Calibration

Temperature calibration of the TGA-50 is relatively easy to perform but sample preparation is a little bit more involved. Once the sample is made, however, it can be reused. The materials needed are a TGA calibration magnet (P/N 220-96401-00) and the SRM-761 Curie point standard kit available from N.I.S.T. This kit contains one metal and four alloy standards which are magnetically permeable. When subjected to a magnetic field the sample's apparent weight changes due to a alteration in the sample's structural order brought on by a thermal change. In this case the transition will appear as a weight loss as the temperature increases, and a weight gain as the temperature decreases. When the 1st derivative curve is displayed, the weight loss and weight gain will appear as negative and positive peaks respectively. This is where the Curie point result will be taken.

Sample Configuration

To prepare for the test, dismount the Pt cell and pan from the quartz rod. Mount the TGA calibration magnet on the thermocouple by slightly spreading apart the leads and "cradling" the magnet so that the flat side is horizontal to the

axis of the quartz rod. Now hang the Curie point standard from the quartz rod and position it as close to the magnet as possible without touching it (about 1mm). The magnet should appear to draw the standard down towards it. Close the furnace and allow the weight signal to settle out. Zero the weight signal and set the temperature calibration constants (in this case Permanorm 3 and Mumetal) to Null:

1.	Temp Gain	=	1.000	Temp Offset	=	0.0
2.	T1 Expected	=	259.1	T1 Measured	=	259.1
3.	T2 Expected	=	381.6	T2 Measured	=	381.6
4.	SIG Gain	=	1.000			
5.	SG Expected =	10		SG Measured	=	10

Now set the run parameters to the following:

1.	Heating Rate:	10°C/min.
2.	Hold Temp:	100°C above Curie pt.
3.	Hold Time:	0 min.
4.	PID's:	10, 10, 10.
5.	ATM:	N_2 at 30-40 ml/min.
6.	Sampling Interval:	1.0 sec.

When the run is over, display the 1st derivative curve (smooth it if necessary), scale it and determine the peak temperature of the derivative curve for the apparent weight loss. Once two or more Curie point standards have been run the data can be entered into the TGA calibration function (FUNC 7) using the standard method:

1. Enter Calibration Function (Press FUNC, go to 7, and press ENT).
2. Press ENT 3 times.
3. Enter Measured T1 temp (from sample run) using the or arrow keys and press ENT.
4. Enter Measured T2 temp (from sample run) using the or arrow keys and press ENT.
5. Re-analyze Permanorm 3 and Mumetal standards. Temp should be within ± 5.0°C.

Weight Calibration

Performing the weight calibration for the TGA-50(H) or TGA-51(H) is quick and very easy. Again, this calibration does not need to be performed if weight losses will only be analyzed in percent.

Use the mg weight set supplied with the TGA or, if necessary, any mg weight set traceable to N.I.S.T. Although this calibration uses one weight measurement, it is really a two point measurement since the reference point (or first point) is

Zero. Keep in mind that the calibration weight range should be just above the actual sample weight. That is, if the calibration weight used is 50mg then the sample weight should not be more than 50mg but just under. This should be performed at ambient temperature using the following method:

1. Enter the Calibration Function and set the SIG Gain to 1.000.
2. Set the SG Expected and SG Measured to the desired mg weight.
3. Close the furnace (empty cell on hanging pan), allow to settle.
4. Zero out the weight signal.
5. Lower the furnace and place calibration weight on pan assembly.
6. Close furnace and allow the weight to settle (about 1 min.).
7. Record weight reading and enter it into the SG Measured under Calibration Function.
8. Weight calibration is complete.

BASELINE

Since Thermogravimetric Analyzers measure the weight loss of materials as a function of temperature and time, a good baseline is necessary for optimum weight loss sensitivity. The ideal TGA baseline will be flat and parallel to the x-axis with no buoyancy effect at the start. This is never the case with low sample mass-high sensitivity instruments, as there are many things which can affect the baseline.

The main de-stabilizing effect is buoyancy. Buoyancy is seen as the false increase in the weight signal at the start of a run and is caused by atmospheric disturbance. Before a run is started the sample and the atmosphere inside the furnace are in a stable state. The temperature is steady, and the purge atmosphere is slowly introduced into the furnace chamber. When the run is started, however, the heat energy being pumped into the furnace begins to warm the stable internal atmosphere. Since hot air rises, so also does the purge gas which begins to circulate. This tends to push down slightly on the sample pan assembly which the instrument sees as a weight gain.

De-stabilizing baseline effects can be minimized several ways. Using large sample masses will help reduce the buoyancy effect simply by being more massive and resistant to any atmospheric "push." Lower temperature gradients will also reduce atmospheric disturbance. Finally, slightly higher purge rates can also act as an opposing force to thermally induced atmospheric currents.

Finally, most of the unstable baseline effects can be eliminated from sample data by performing a baseline subtraction. This is done by running a blank or empty sample cell under the same program and atmospheric conditions as the sample and then subtracting the baseline file from the raw sample data file. This procedure is covered on pp. 3-97/98 in the TA-50WSI users manual.

One important note about TGA operation is that the instrument must always be run with a purge gas flowing into the back of the instrument. The reason for this is that it eliminates the chance of any evolved materials drifting up into the detector housing, which can damage the detector. As the purge gas enters the rear of the instrument it is passed through the regulator and then into the detector housing. From there it is directed down the purge inlet tube and into the furnace area. This keeps positive pressure within the detector housing and eliminates back drafting of potentially damaging evolved materials.

CONCLUSION

The TGA-50(H) and TGA-51(H) are both highly sensitive instruments which require proper care and attention. The operation is simple and, with a little time and patience, can easily be temperature and weight 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.