Ref.: SSI-TA-JG-011

STANDARD CLEANING AND CALIBRATION PROCEDURE FOR DSC-50 AND DSC-50Q

by Joseph F. Greenbeck, Jr. And Courtney W. Taylor

INTRODUCTION

The following procedure is to be used for the care and maintenance of the DSC-50 and DSC-50Q. It is recommended that from time to time the DSC furnace be cleaned of excess material which may be deposited on the detector through normal use or from runaway samples that spill out of the cell. This procedure should be performed at one-week intervals, but this can be adjusted to suit the requirements of the instrument.

The following materials and supplies are necessary to perform the cleaning and calibration.

1. A bottle of 90% Nitrogen / 10% Hydrogen.
2. Melt point standards (e.g. Indium, Tin, Zinc).

CLEANING

The cleaning method detailed below requires a gas mixture of 90% Nitrogen and 10% Hydrogen to reduce the various residues which have been built up in the furnace area and on the detector.

The procedure is relatively simple and goes as follows:

1. Connect purge gas (90/10 mix) 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.
5. Set hold time for 120 min.
6. Set PID's to 10, 10, 10.
7. Allow cooling water to circulate.
8. Start the run (there should be no cells on the detector).

9. When the run is complete, the detector should have a dull gray or silvery appearance.

CALIBRATION

To ensure the integrity of data gathered on any analytical instrument, periodic calibrations should be performed. The DSC-50 is no exception to this rule. The frequency of calibration depends on how often the instrument is used and what samples are being analyzed. If many samples (10-20) are being run per day, then a daily calibration could be required. If fewer samples (less than 5) are run, then a weekly calibration schedule can be maintained. It is, however, up to the individual thermal analyst.

There are two ways to calibrate the DSC-50. The first is the standard method where certain high purity metals are run through their melting points. The peak temperature and peak areas are then determined and programmed into the instrument. The second method is a software correction routine where several materials with known melt points are analyzed and stored as a file. The sample data file is then corrected using the calibration file. This procedure is covered in the TA-50WSI instruction manual (pp. 3-108) and will not be covered here.

The following standard method will be explained using Indium (In) and Tin (Sn) as calibration standards. First, enter the calibration program under the function menu on the front panel of the instrument. Cursor through and set all calibration constants to Null. For example, if using In and Sn:

1.	Temp Gain	=	1.000	Temp Offset	=	0.0
2.	T1 Expected	=	156.6	T1 Measured	=	156.6
3.	T2 Expected	=	231.9	T2 Measured	=	231.9
4.	SIG Gain	=	1.000			
5.	SG Expected	=	-28.5	SG Measured	=	-28.5

Next, determine the Melt Point (Tm) of In and Sn under the following conditions:

1. Heating Rate: 10°C/min
2. Hold Temp: 180°C (In) or 250°C (Sn)
3. Hold Time: 0 min
4. PID's: 10, 10, 10

5. ATM: N_2 at 20-40 ml/min
6. Sampling interval: 1.0 sec.
7. Sample: Approximately 2 mg weighed to 3 significant digits. Enter into information screen.
8. Reference: Empty Al cell with a lid.

Now determine the Tm and Heat (in J/g) of the In sample and the Tm of the Sn sample using the *Windows* software and enter into the calibration constants.

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. Press ENT 2 times.
6. Enter measured Heat (from In sample run) using the or arrow keys and press ENT.
7. Re-analyze In and Sn samples. Temp should be ± 0.3°C and the Heat should be ± 0.5 J/g.

BASELINE

The ideal baseline for any DSC is a line parallel to the X-axis with no positive or negative drift throughout the instrument's entire temperature range. In reality, however, this is not generally the case. The DSC baseline will typically drift in one direction or another in varying degrees. Some of the causes of this are variances in individual furnace construction, differing heating rates, atmospheric purge rates, wear and tear associated with age, residual material deposited on the detector, or various combinations of these. Baselines with peaks in either direction can be considered dirty and should be cleaned.

The Shimadzu DSC-50 baseline drift specification is ± 1.0 mW full range. That is, the signal baseline will not drift more than 1.0 mW in either direction of the X-axis between ambient and 725°C. If the baseline drifts in the positive or negative direction but is linear, the balance adjust knob can be used to position the curve closer to the X-axis. This is simply done by determining which direction the baseline is drifting and adjusting the balance knob in the opposite direction. This may take two or three runs to nail down. Baselines which curve slightly can be dealt with by using the baseline correction routine in the TASYS software. This

is done simply by running two blank cells on the detector using the same analysis parameters as the sample. Subtracting the Blank baseline file from the Sample file will help flatten or correct the Sample run.

CONCLUSION

Care and calibration are an important part in the general operation of the DSC-50. The frequency of the above procedures will vary from user to user, but the general rule of thumb for calibration is once a week. Obviously, if care is taken not to spill sample on the detector or if the type of material being tested is "clean," then cleaning will be performed less often. In any case, a clean detector will last much longer and give good, consistent results.