

Procedures for operation of the TA Instruments DSC

Purpose and Scope:

This document describes the procedures and policies for using the MSE TA Instruments DSC. The scope of this document is to establish user procedures. Instrument maintenance and repair are outside the scope of this document.

Responsibilities:

This document is maintained by the department Lab manager. The Lab Manager is responsible for general maintenance and for arranging repair when necessary. If you feel that the instrument is in need of repair or is not operating correctly please notify the Lab Manager immediately. The Lab Manger will operate the instruments according to the procedures set down in this document and will provide instruction and training to users within the department. Users are responsible for using the instrument described according to these procedures. These procedures assume that the user has had at least one training session.

Warnings and precautions:

- **DO NOT HEAT YOUR SAMPLE TO DECOMPOSITION TEMPERATURES. TGA MUST ALWAYS BE PERFORMED FIRST!**
- Please do not eat or drink while operating this instrument.
- **NEVER** touch the robotic arm.
- Never attempt to operate the instrument without nitrogen flow.
- Do not place a usb drive in any of the usb ports of the computer.
- Do not change any of the user preferences or instrument preferences.
- Notify the Lab Manger of any issues, do not attempt to 'fix' a problem.
- The analysis software for the DSC and TGA are available for free on the TA Instruments web page at:

[Installation of Universal Analysis from the Advantage Software Package](#)

- You will only need to download the "Universal Analysis" portion. There are also some training power-point videos available to further instruction on analysis.
- You are responsible for providing and caring for your own consumables for this instrument. T-Zero aluminum pans and lids are available from TA Instruments. Reference pans are provided.
- Before starting please verify that there is plenty of N₂ in the cylinder and that the flow rate is at 50 mL/min.

Background

Differential Scanning Calorimetry is used to measure heat flow in or out of a material. From this information a number of thermal properties can be determined; T_g, melting temperature, crystallization temperature, oxidative induction time, % cure, etc. This method uses an empty sample pan as a reference that cancels the heat flow to and from the pan itself.

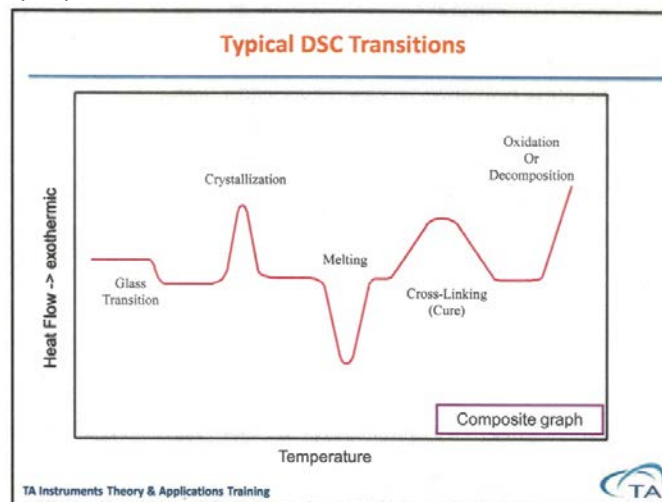


Fig. 1 Typical transitions: TA Instruments training 02/2014

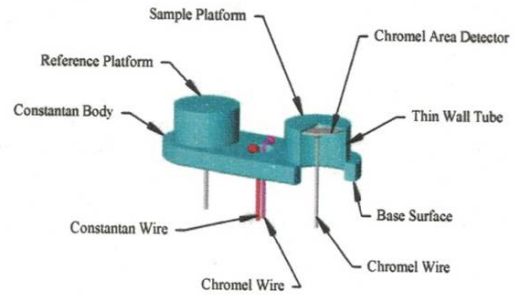
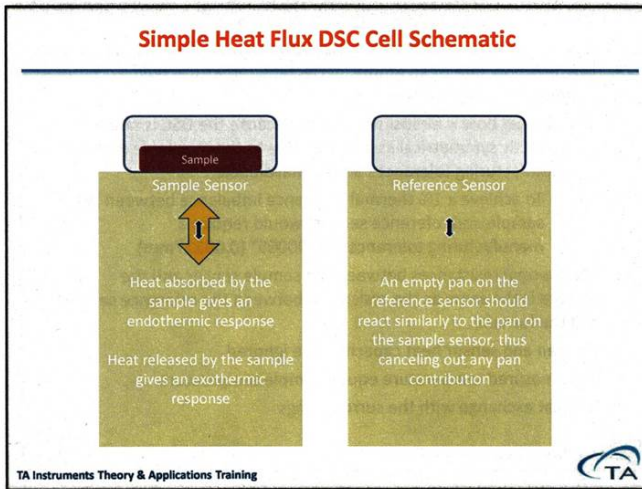


Fig. 2 and 3: Schematics of sample pan and chamber configuration: TA instruments Training: 02/2014

DSC operates under the following assumptions:

(modified from TA Instruments Theory and Applications Training 02/2014)

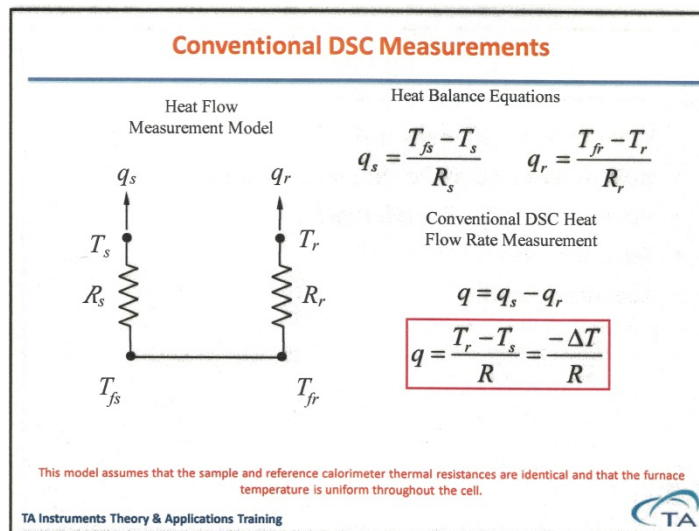


Fig. 4 Schematic and equation for basic DSC measurement: TA Instruments training 02/2014

- The heat flow rate of an empty sample pan is zero
- Thermal resistance between the sample sensor and the furnace is the same as the resistance between the reference sensor and the furnace.
- The pan and sensor heat capacities are ignored
- The measured temperature equals the sample temperature
- No heat is exchanged with the surroundings.
- The sample material is making good contact with the bottom of the sample pan.

Many parameters must be considered when preparing your sample and when interpreting your data. Sample prep is very important. The sample must make good contact with the surface of the sample pan. The bottom of the pans are very precisely machined to be flat (manufacturing tolerance of 0.00127mm). So sample prep should be done in as clean an environment as possible. There is already a prepared empty reference pan in the auto changer. Please do not remove this pan! Contact the Lab Manger if you think there is a problem.

The following figures are presented as examples taken from the TA instruments training manual. Please keep in mind that these are idealized representations and may not be typical results.

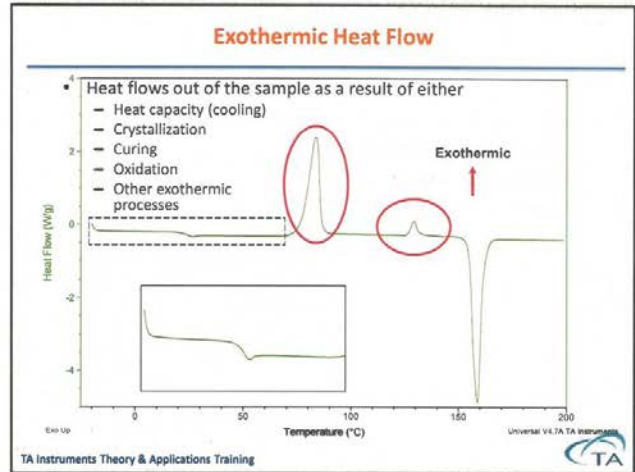
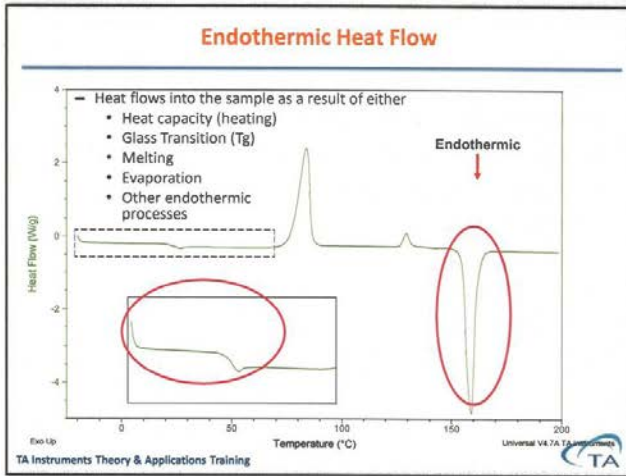


Fig. 5 Endothermic vs Exothermic heat flow: TA Instruments training 02/2014

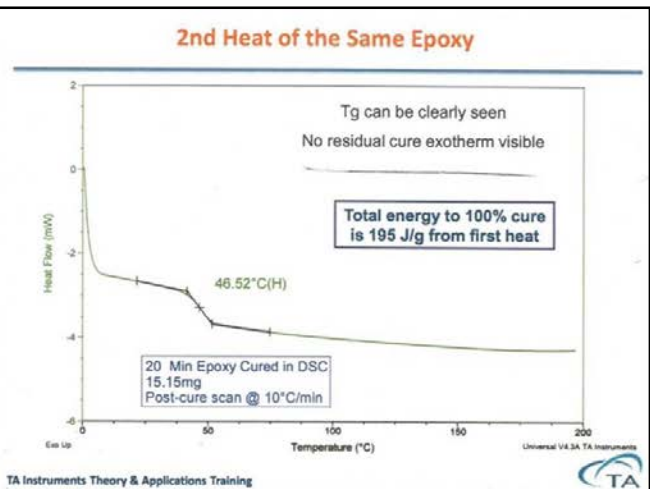
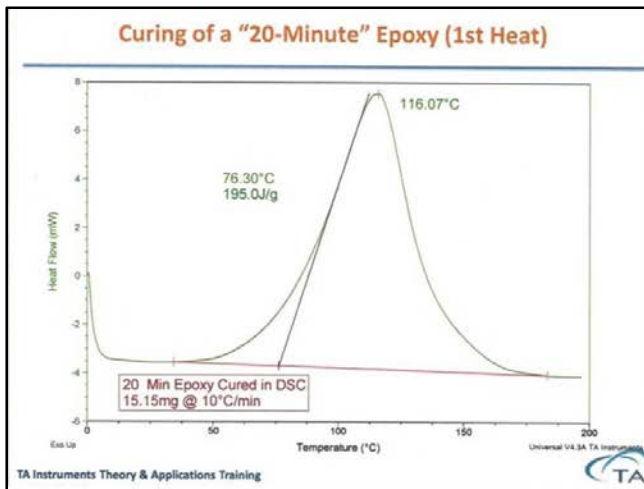


Fig. 6 Epoxy cure studies: TA Instruments training 02/2014

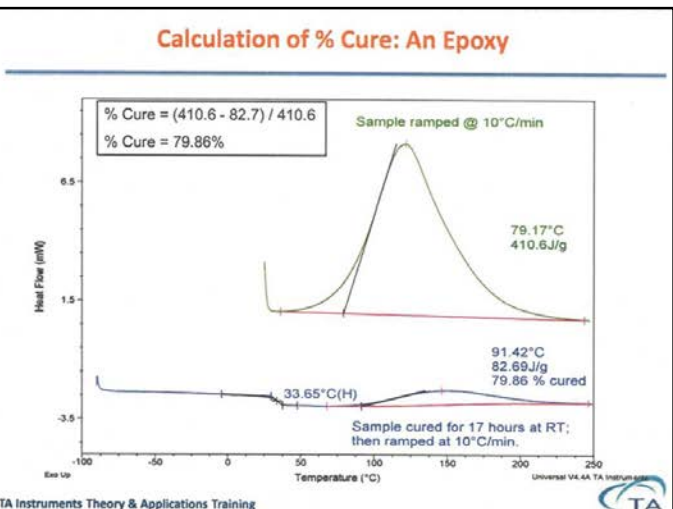
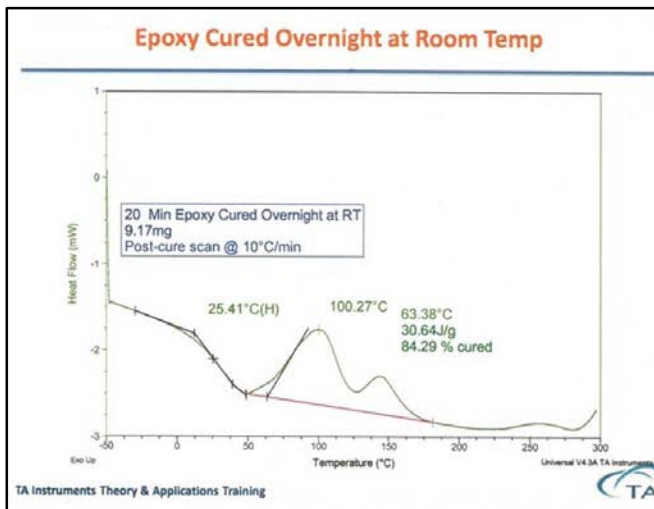


Fig. 7 Epoxy cure studies: TA Instruments training 02/2014

The following parameters should be considered to optimize your measurement:

(modified from TA Instruments Theory and Applications Training 02/2014)

- Determine which pan type is best. The standard pan for our instrument is the T-Zero pan. If your sample loses about 0.5% or more due to volatile materials you should consider the hermitically sealed pans.
- What is the maximum temperature. Run TGA on your sample **AT THE SAME RATE** you plan to run your DSC measurements. Decomposition temperature is lower at lower rates.
 - **Decomposition will contaminate (and ultimately ruin) the sample chamber.**
 - **DSC results are of little value once the sample has lost 5% of its weight due to decomposition (not desolvation)**
- To determine volatile content you can heat about 5 - 10 mg of simple at 10 C/min. Again, only AFTER TGA.
- This is a kinetic measurement so consider rate. If you intend to do comparative studies, all conditions must remain the same. A sample measured at a rate of 20 C/min will not return the same results as a sample run at 10 C/min.
- Try to keep the sample thin.
- The sample should cover as much of the bottom of the pan as possible.
- If you are comparing a number of samples keep the sample masses as much the same as possible.
- Using your own tools and materials will reduce contamination.
- If you are measuring a liquid, you will only need a very small amount. Liquids can spill out of the sample pan either during pressing or during the scan. If this happens while in the chamber the chamber can become contaminated and create odd behavior both in the robotic operation and in the results. Please take care not to let this happen.



Fig. 8 MSE DSC Instrument

Procedures

- Place an empty T-Zero pan on the scale and tare
- Place your sample in the pan. It is very important that the sample make good contact with the surface of the pan. Good thermal contact is crucial to a good measurement.
- Note the weight of the sample (1 – 10 mg)
- Put the top die in place and put the pan in the bottom die.
- Place the lid on the pan and put the assembly in the press.
- Bring the press lever down carefully.
- No pressure is necessary. Please be gentle.

- Move the lever up and back to its original position.
 - Remove the pan and place in a position in the auto chamber.
 - Take great care not to touch the robotic arm. It is very easy to bump it out of alignment.
 - Repeat for each sample.
 - **(This will be demonstrated during training).**
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- If the software isn't already open, open instrument explorer and select the **Q20 DSC**. The last user's information will be displayed.
 - In the summary tab select <Standard Mode> <Custom Test> and if you already have a completed procedures file you can open it here as well by clicking on the file icon. This is usually done during your training.
 - Type in the sample name
 - Select the correct sample pan position and reference pan position
 - Select the icon to the right of "Data file name" and name the unique file.
 - Note: If you are running more than one sample you can name the first one and TA will add .00X to each subsequent run. Or you can provide a unique file name for each one.
 - If necessary, select <Apply>
 - If you have not created a procedures file, select the second tab, <Procedure>
 - Under method, select <Editor> To the right of the screen will be a list of steps you can select. The most common is simply the rate and temperature. Standard would be 10 C/min to a temperature just past melting temperature. **If you do not know what that temperature is you MUST RUN TGA to determine decomposition temperature. Do not run a sample to or past a materials decomposition temperature as this can damage the instrument.**
 - Select <Advanced> This is where you can define what temperature to load the next sample at. Enter a 10 degree range with the center range being the temperature you want to start your run at.
 - Select OK then open the <End> conditions button. Enter a 10 degree range of temperatures to unload the completed sample.
 - You can also choose to have the sample pan discarded.
 - Check the <Notes> tab and enter your name in the appropriate field. Make sure that the flow rate is 50 mL / min. **DO NOT CHANGE THIS FLOW RATE.**

To run more than one sample:

- After you have completed the first run parameters right click on the <Run> (left side of screen) and select <Copy and Append> Do this for every run.
- Go back and in each summary page type in the sample name, the sample weight, the pan number and if desired, provide a unique file name.
- Select <Apply>
- Double click the top run.
- Select <Run> at the top of the page.

Run Icon

The screenshot shows a software interface for programming and operating a thermal analysis instrument. The main window is titled "Experiment" and displays a "Standard Sequence" with "Run 1" selected. A status bar at the top indicates "Run 1 Complete Temp: 39.99°C Gas: 1".

On the left, a sidebar contains "Experiment", "Calibration", and "Platinum" buttons. A callout box points to a "Run Icon" in the top toolbar.

The central area is divided into three tabbed windows: "Summary", "Procedure", and "Notes". The "Summary" tab is active, showing "Procedure Summary" with fields for "Mode" (Standard), "Test" (Custom - \\2013labview-1\ta\QProcedures\DS), "Sample Information" (Sample Name, Pan Type: Tzero Aluminum Hemetic, Sample Size: 15.100 mg, Pan No. 1, Ref. 4), "Data File Name" (\\2013labview-1\ta\Data\DSC\Data.001), and a "Network Drive" checkbox.

On the right, a "Relevant Signals" table lists various parameters and their values:

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	39.99 °C
Heat Flow	-0.020 mW
Set Point Temp	40.00 °C
Heater Power	2.311 W
Flange Temperature	33.84 °C
Heater Temperature	40.47 °C
Sample Purge Flow	49.99 mL/min

A callout box labeled "Gas Flow" points to the "Sample Purge Flow" value. Below the table is a "Segment Description" table:

#	Running Segment Description
1	Ramp 20.000 °C/min to 50.00 °C
2	Air Cool: On

A callout box labeled "Segment Description" points to this table. At the bottom right, a graph plots "Heat Flow (mW)" on the y-axis (ranging from -8.0 to 2.0) against "Temperature (°C)" on the x-axis (ranging from 34.0 to 48.0). The graph shows a downward-sloping curve. A callout box labeled "Graph of most recent or current run" points to the graph.

At the bottom of the window, a status bar shows "Ready", "01 1.50 min", and buttons for "Append", "Apply", "Cancel", and "Help".

Fig. 9 Programming and operations software

When all of your measurements have been completed you can collect your data from the WORKGROUP computer. No other action is necessary. Please leave the computer on.