

Perkin Elmer Jade DSC --- User's Guide

Version 1.0

Edited by Dr. Hartmut G. Hedderich

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The following guide describes the use of the Perkin Elmer Jade DSC. The guide is intended to assist instrument users after the initial training session.

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The Perkin-Elmer Jade DSC can perform the following measurements between -80°C and 445°C:

- Measure melting and crystallization points
- Measure glass points and transition points
- Enthalpy and molar heat capacity at constant pressure



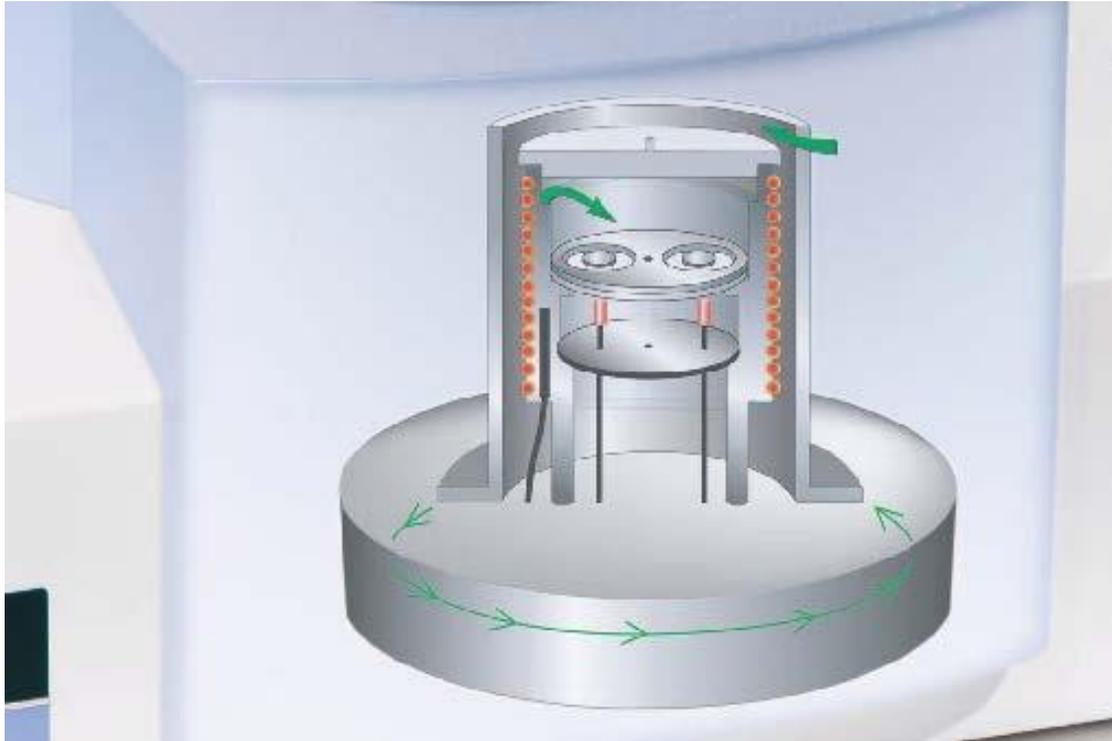
1. Hardware

The instruments consists of the actual DSC unit, an intracooler unit, a nitrogen purge gas cylinder and a computer. **The cooling unit always needs to be turned on independent of the temperature range! The purge gas always needs to be turned on during measurements!**

The sample is put in a sample pan. See below for sample pan models and requirements. If you are interested in measuring thermodynamic data, the weight of the sample needs to be known with high precision!

Open the oven by removing the main lid and the oven lid. The pan is then placed on a sensor disk which is very durable and provides superior instrument response. The sample is put on the left sensor, the reference (an empty pan) is put on the right sensor. Close the oven with its lid. Put the main instrument lid back in place.

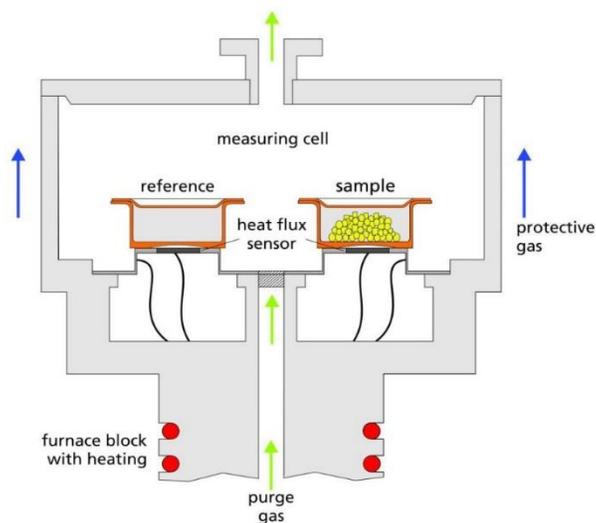
The following diagram shows the inside of the furnace area:



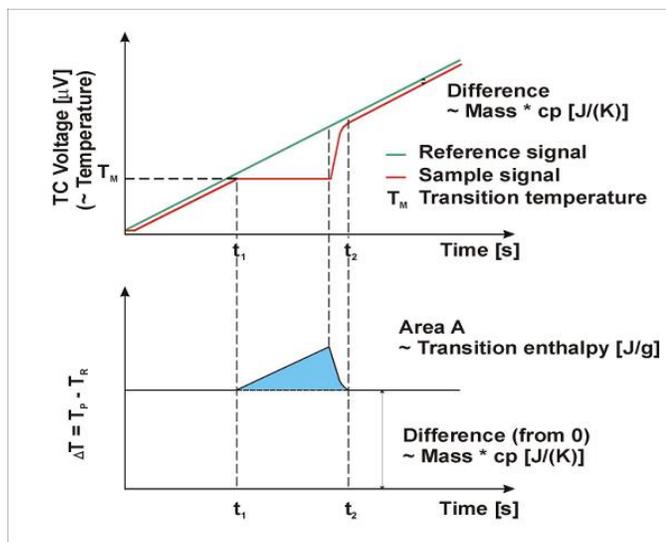
The purge gas enters the furnace area directly above the sample pan. It is pre-heated to avoid any disturbances in the actual signal. Highly sensitive thermocouples are placed below the pans and measure the temperature difference between the sample and reference materials. Tight temperature control is maintained by a platinum resistance-sensing device.

2. Principles

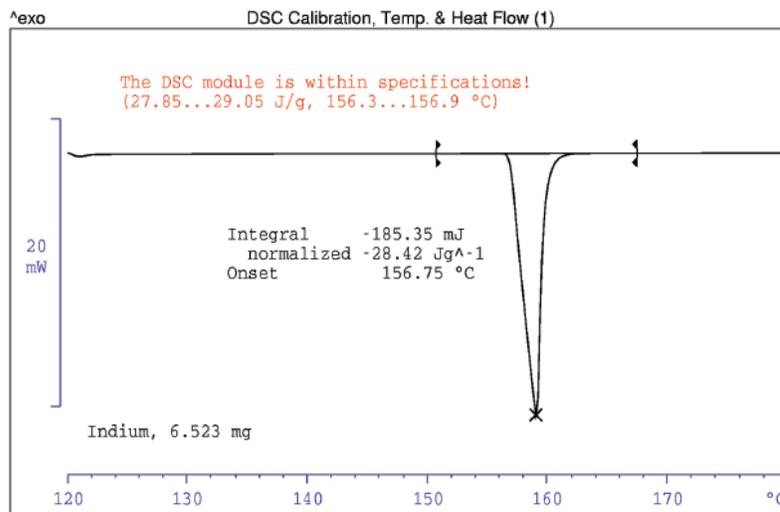
A differential scanning calorimeter (DSC) measures the energy difference between a substance and a reference material as a function of temperature or time, with both sample and reference following a controlled temperature program.



The temperature difference caused by the difference in the heat flow to the sample and reference is measured versus time:



In a calibrated instrument we change the x-axis from time to temperature. Then we plot heat flow versus temperature:



3. Pans

DSC pans come in a variety of different materials and tops.

The standard pan material is aluminum. We use aluminum pans with crimped tops for the analysis of polymer materials and other organic substances. A crimped top can withstand an internal pressure of up to about 5psi, it will open if the pressure goes higher. It is important to set the maximum temperature for polymers to below their decomposition point. If you go above the decomposition point the released gases will open the crimped seal!

Metal salts cannot be put into aluminum pans! Most metal salts will react with the aluminum when they reached liquid state. The pan will be dissolved and the instrument will be damaged! For metal salts iron, stainless steel or gold pans are the best choice. If your sample releases toxic gases (i.e. HCl, HBr, Hg, etc.) a high pressure pan needs to be used. Those pans have a screw on top that withstands pressures of several bar.

Please contact the PIC of the DSC for any further information and advice regarding DSC pans.

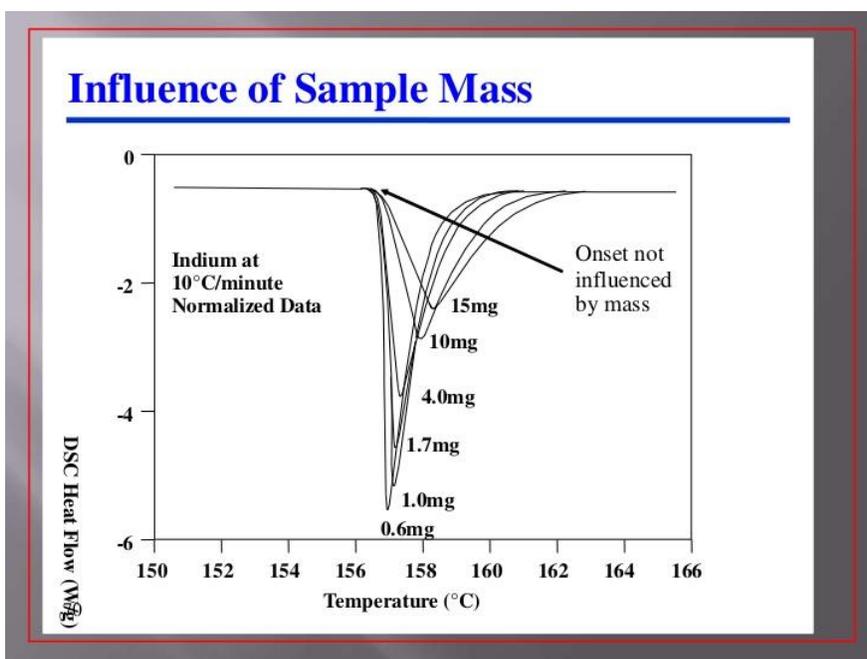
4. Heating Rates and Sample Mass:

Heating rates depend on what kind of data you are interested to collect. Melting points usually give a huge signal, since you need to add energy until all material goes from the solid to the liquid state before the temperature increases again. Transition or glass points usually give a much smaller signal since the energy difference between 2 phases is rather small.

Usually, you want to measure transition points with a smaller heating rate (5°C/min - 10°C/min). Melting points can be measured with 10°C/min or even 20°C/min.

Both heating rate as well as sample mass have a direct influence on the strength and shape of the corresponding sample.

(a) Influence of sample mass:



Under the same experimental conditions a larger sample mass will have a broader curve as depicted above (melting point of indium). The onset of all curves (melting point) is exactly the same. Since the enthalpy is mass dependent, the areas under the curves are different!

5. Startup

Turn on the DSC unit by using the on/off switch at the left back side of the instrument. Turn on the intracooler unit. Open the main gas cylinder valve and the small release valve. Do NOT change the pressure regulator setting! Log into your account and start the software by double-click on the Pyris software icon.

A grey bar will appear on the top of the screen. There are 2 options – the left one (Start Pyris) is an offline version of the software, it does not need an actual DSC connected to the computer. This part can be used for any post processing of saved data. The right button (Jade DSC) starts the data collection program. Push the button to connect the computer to the DSC and start the program.

6. Software

There are two window within the software main window – method editor and instrument viewer. Depending on which window is active the pull down menus on the top will change!

(a) Method Editor:

The method editor allows the user to write a program or method on how to run a specific sample. There are 4 tabs: *Sample Info*, *Initial State*, *Program* and *View Program*.

Sample Info allows the user to put in some general info like Sample ID, Operator ID and Comment. Here is also the place to choose the directory and name for the save file. Most importantly, the weight of the sample can be entered. **A very precise sample weight is necessary to calculate correct thermodynamic constants!**

Initial State lists method name, sample ID, name for saved data file as well as information on how to run the data (action occurs immediately) and that the purge gas is turned on.

Program is the heart of the method editor. Here the user defines the temperature program, the way how the sample will be treated. Initial temperature, final temperature and heating rate will be defined for each step of the program. The simplest program contains one line: "Heat (Cool) from temperature 1 to temperature 2 at ramp rate degree Celsius per minute". Further command lines can be added with **Add Item** and **Insert Item** buttons. A **Delete Step** button lets the user remove lines from the program. The temperature program can do two things: heat/cool or hold temperature for a certain amount of time (isothermal). It is useful for multistep programs to start with an isothermal to stabilize the temperature and then switch between temperature scans and isothermals. It is absolutely necessary to have isothermals when working with temperature in the -50°C to about 20°C range. The system needs longer to stabilize low temperatures. **End Condition** is a function which tells the program what to do when the scan is finished – go to load temperature, hold temperature or go to any other temperature.

View Program displays the whole program for the user to check if everything is correct.

Place your sample in the oven if you haven't done so already! Make sure that the purge gas is running (the display should show a 20ml/min value) and hit the **Start/Stop** button on the right hand side panel.

(b) Instrument Viewer:

There is always a default instrument viewer window open (cannot be closed out) that shows the actual output of the DSC. Once the program starts, the window shows the temperature changes until the actual starting temperature for the scan is reached. Now a new instrument viewer window (with filename displayed) will be opened that shows the actual data of the scan. Once the scan is finished the default instrument viewer will be active again.

As long as the oven hasn't reached the end condition the method editor and the program are locked. No new scan can be started! **Please do NOT open the actual oven during the cool down period. It will be hot! Besides burning your fingers, you can damage the oven itself due to reaction with oxygen!**

Data Evaluation – the program can measure and calculate melting/crystallization points as well as glass points or transition points. Furthermore enthalpy ΔH and specific heat at constant pressure ΔC_p can be calculated if correct sample weights were entered! There are two options within the program to calculate those values.

The *Instrument Viewer* window that contains the data needs to be active! Under the "**Calc**" tab the menu options **Peak Area** and **Tg** are now accessible.

Peak Area will calculate the melting point and the area under the curve (ΔH). The user defines the left and right temperature boundaries of the curve. Activate **onset** and **%area curve** buttons. The program will automatically calculate and display the results on the instrument viewer window.

Tg will calculate the transition or glass point as well as the specific heat at constant pressure. In order to get correct results a tangent needs to be defined and put onto each of the 2 sides of the curve. Do not cross the tangent lines! Again the program will calculate the transition point (half extrapolated C_p) automatically.

Saving Data – the program will save the data (**Save Data**) in a program-specific format. All graphs can also be saved in that format (**Save MultiCurve**). It is useful to have those data sets in case the user wants to perform further evaluation of the data with the offline software tool.

In order to get the data in a text format the user needs to export the data to an ASCII or CSV formatted file. The text file will contain time stamp, heat flow and temperature values. A complete set of the calibration data and calibration formulas can be added to the save files.

7. Known Problems/Maintenance

With the software upgrade we performed in 2021, there are no known software or hardware problems with the instrument!

Once a DSC is installed and the original calibration is performed, the instrument should be working within its limits as long as it is not moved. Unfortunately, over time users spilled material in the oven, some corrosion occurred due to opening the hot oven, etc. For that reason it is necessary to regularly check and redo the calibration. The PIC will do this every 3 months to make sure the instrument works within its stated parameters.

8. Turn Off/Shut Down DSC

Once the user is done with all experiment and the data is saved, the software can be closed. The program will automatically disconnect the DSC. Right-click on the grey bar and choose exit program. This will close the software.

Turn off the power to the DSC and the cooling unit! **Never forget to turn off the cooling unit or ice build-up on the cold finger will damage the instrument!** Close the gas cylinder main valve!

9. Notes