Netzsch 404C DSC User Guide

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Table of Contents

Introduction

The high temperature Netzsch Differential Scanning Calorimeter (DSC) is designed to quantitatively measure the energy absorbed or released by samples as they are heated; anywhere from room temperature up to 1400 °C. The DSC can measure many thermodynamic properties of samples including the:

(**ΔC_p**) Specific Heat as a function of temperature;
(**ΔH**) Transition & Reaction Enthalpies;
(**T_M**) Temperatures of Melting & Phase Transformations;
(**T_G**) Temperature and energy change of Glass Transition & Crystallization;

Tests are performed with an inert reference held in a shared chamber with the sample. Both reference and sample are exposed to the same heating rates and environment. Thermodynamic properties can then be calculated by comparison.

The DSC can heat from room temperature to 1400° C with tests usually performed in argon, although other gases may be used. It is recommended to avoid reducing atmospheres.

The instrument is not designed to handle sample decomposition or volatility and should not be used with reactive samples.

Administrative

TEMPO Access & Safety Training

Users seeking to obtain access to the TEMPO facility are required to complete a suite of safety requirements which can be found on the "Access and Safety Training" Gauchospace website. Instructions for reaching this website can be found in the appendix. All requirements must be satisfied before entering the TEMPO facility.

Once the TEMPO and MRL safety requirements have been completed users may request daytime access through the website "**https://www.mrl.ucsb.edu/access**".

All users are required to wear safety glasses, long pants and close-toed shoes whenever working in any MRL lab. This applies for **all** instrument use including logging onto a computer to retrieve data. If users are working with liquids then a lab coat is also required.

Accessing the DSC

The lab is open Monday through Friday from 8 AM - 5 PM. Users do not need to be present while the DSC is running but it is good practice to verify the DSC is operating correctly before leaving.

Time may be reserved using the FBS system at **https://ucsb.fbs.io/**. After reserving a time slot, you may log in at the beginning of your scheduled slot. Starting the timer will power on the monitor and allow you to perform your measurement. Access to the DSC, through the FBS website, is granted to users after they complete instrument training.

Before using the DSC, users should write down their name, 13 digit recharge number, advisor's name, and the start time on the paper log. On completion of the run the end time should also be noted. The paper log serves as a backup and allows for comments in the event of an error.

Training

Self-training on the instrument is available, after speaking with TEMPO staff. Users choosing to self-train are given a sample and some parameters to perform a measurement on. For users interested in a guided training session, the training schedule is sent out quarterly via e-mail.

To enroll in a training session, users can sign up through the TEMPO Instrument Training and Resources page on Gauchospace.

Safety & Housekeeping

The furnace is capable of achieving very high temperatures. Users must take proper precautions to prevent damage to themselves, other users and the lab. **Do not open or attempt to open the furnace without verifying that the interior has cooled down to at least 100 °C.**

All samples must be labeled with the owner's name and their essential composition. Samples should not be left in the lab unless they are actively running in the DSC. Users should avoid leaving their samples in the instrument if they are not preparing or performing a measurement.

Acknowledgements

In any publications based on research done with MRL Facility instruments (ie in the TEMPO lab) or with help from MRL staff please acknowledge support from the National Science Foundation. Acknowledgements should be stated as:

> **"The MRL Shared Experimental Facilities are supported by the MRSEC Program of the NSF under Award No. DMR 1720256; a member of the NSF-funded Materials Research Facilities Network (www.mrfn.org)"**

Acknowledgements such as these allow the MRL to obtain funding from the NSF and aid the NSF when they justify their requests for funding from congress.

Basic Differential Calorimetry

Process and Theory

Each measurement uses two lidded crucibles made from the same material and of approximately identical masses. One crucible serves as an empty and inert reference and the other holds the sample. The crucibles sit on separate sensors, connected by a thermocouple. When heated in a shared environment any difference in heating rates produces an electrical signal via the thermocouple which is captured by the instrument.

In theory the use of a pair of virtually identical crucibles shouldn't produce a signal as there would be no difference in heating between them but the presence of even minor defects may produce a signal. Fluctuations in the placement of the crucible on the sensor as well as thermal conductivity properties of the sensor head itself result in the appearance of a voltage difference.

Although there isn't a practical means to achieve zero resting voltage, measurements may be improved by first collecting a baseline of the two empty crucibles. This baseline can then be used to correct the data collected during measurement of a material. The baseline correction must match the parameters of the sample measurement exactly, including heating program, gas type and crucibles.

In events of phase transitions the relative difference in temperatures becomes more dramatic and produces visible peaks in the signal. The transition is often accompanied by a shift in specific heat capacity (Cp) and, when plotted as a function of time, is seen by a shift in the slope of the line.

The temperature of each crucible is measured by a sensor connected to a thermocouple which translates temperature differences into a voltage potential. The temperature difference is measured in microvolts (μV). This means the data accuracy and precision is influenced by the thermal contact of the crucibles to the sensors and of the sample to its crucible. Variations between crucibles may also affect data points.

Quantitative energy measurements require a calibration to convert the difference in temperature to energy. This may be accomplished with the use of a single standard run under the same conditions as the intended sample. Sapphire standards are often employed. Typically standards should be used with similar dimensions to the sample and with well documented heating capacities at the target temperature range.

Thermal Lag

Optimizing a measurement in the DSC requires that users understand the role of thermal lag in the instrument. **Thermal lag,** in this context, is the difference in heating rate between two materials in a shared environment. Two identical materials, e.g. crucibles, would be expected to heat at the same rate when placed in a shared environment. The introduction of a sample material to only one of the crucibles will alter its properties. The sample containing crucible now is part of an equilibrium relationship with the material which causes it to heat at a different rate.

The difference in heating rate is what produces the electrical signal interpreted by the instrument and although it is ultimately responsible for the data obtained by the user, too much

of a good thing can ruin the data. Measurements which take place over long temperature ranges lead to an accumulation of thermal lag to a degree that may lead to any resultant data being meaningless. If a sample is heated over a continuous 800 °C period then once the furnace has reached its final value the sample containing crucible may have only just reached 725 °C. This would then mean that a melting point observed at 800 °C could in fact be occurring at a temperature of 725 °C.

Hardware, Crucibles, & Gas

Furnace and Temperature Sensors

The DSC 404C furnace is capable of reaching 1400° C and employs a passive cooling system using air flow. After a measurement the sample chamber can take up to three hours to cool to room temperature. Since the slowest part of the cool down is from 200° C to room temperature, users with many samples may save time by starting a little warmer than room temperature. (This is not recommended for C_p measurements.) **The furnace should NOT be raised unless the sample temperature is below 100° C!**

Prior to DSC measurements all samples must be tested for decomposition temperatures or volatility. These events lead to the degradation of the sensor head and may result in the complete failure of the instrument. The cost of replacement for the sensor head is $8,000 and may be assigned to users demonstrating negligence in the use of the device.

The sensor head is very sensitive to contact and may shift with forceful loading of the crucibles. It is recommended that the green fencing around the furnace be used to stabilize your hands when loading crucibles.

Sample Crucibles

Crucibles are available as platinum-rhodium or alumina (Al_2O_3). Other DSC crucibles such as quartz, graphite or ZrO_2 may be purchased from other providers. Either alumina or Pt-Rh will work for most measurements. **Each crucible should have a lid and be preheated to the maximum planned measurement temperature before use**. Pre-heating may be done with a torch or inside a clean furnace. The pre-heating will help to remove dust or other particles that may have settled on your crucible as well as remove any adsorbed moisture prior to the measurement.

TEMPO sells Al_2O_3 crucibles for $11 each. They are similar in appearance to the TGA crucibles but are **not** the same size and are **not** compatible. The DSC crucibles are a 6.8 mm outer diameter. The crucible lid should sit squarely on top of the crucible without sample contact. The lid serves to keep the heat of the reaction inside longer and improves measurement sensitivity. It also prevents the exposure of the instrument's internals to any unintended volatility or decomposition. Optimal sample dimensions are 6 mm x 1.0 mm thick for platinum and graphite crucibles. For Al_2O_3crucibles or platinum crucibles with Al_2O_3 liners, the sample dimensions should be 5.2 mm x 1.0 mm thick.

Each crucible will have advantages and disadvantages. Users must research the appropriate crucible type for their samples. Platinum crucibles will typically display much better heat transfer however they will alloy with most metal samples, ruining the pan, sample and data.

They also tend to soften and stick to the sample carrier if they are hot for too long, although they will not melt. Platinum crucibles should be avoided at temperatures above 1000 °C. If a platinum crucible must be used at high temperatures a sapphire disk can be placed between the crucible and sensor head and may be purchased from Netzsch. In the event a platinum crucible is needed and the sample contains metals, alumina liners may be used.

Alumina crucibles are cheaper than their platinum counterparts and may typically be used with most samples. While cheaper and more robust they do undergo a phase transition at about 800 °C, causing the crucible to turn translucent. Measurements looking for quantitative data around that temperature should opt for another crucible choice. Specific heat capacity measurements that require heating through that region may find increasing error as they move above the transition temperature.

Extreme care must be taken to ensure that no crucible/sample reaction will occur before running the sample in the DSC. One of the major causes of measuring head (sensor) death is crucible/sample reaction. The sensor replacement cost is approximately $8,000. If in doubt, heat the sample in a crucible in a lab furnace before trying it in the DSC. A guide on crucible selection can be found on the TEMPO Instrument Training and Resources page on Gauchospace as well as in the appendix of this manual.

Atmosphere for Tests

The DSC can work in several gases with argon as its default. The systems setup uses Argon on Purge 1 or clean dry air on Purge 2. Other suitable gasses are nitrogen and oxygen. Nitrogen is available in the lab but may form nitrosyl compounds at temperatures beyond 700°C. Users interested in using oxygen would need to supply their own gas.

For tests requiring extremely high sensitivity helium serves as a completely inert gas with excellent thermal conductivity. Users interested in helium would need to supply their own gas.

Sample Preparation

Samples must be tested for decomposition or volatility in the TGA prior to use and the data and results confirmed by a TEMPO staff member. Decomposition can lead to deposit buildup within the device and degrade instrument sensitivity. Some compounds may react with the sensor head damaging or destroying it. The use of a crucible lid helps to safeguards the device. Users should consider four basic principles during sample preparation:

1. Selecting the right crucible.
2. Making and maintaining good thermal conduct between the sample and crucible.
3. Preventing contamination of the outer surfaces of the crucibles.
4. The influence of the atmosphere surrounding the sample.

Sample Shape

Ideally samples are shaped as flat disks, powders or pellets; as long as the sample maintains good thermal contact with the crucible floor. Powders may undergo sintering during the heating process and then deform, losing crucible contact. Thin films may also display curling or curvature upon heating, causing a similar loss. A sample of minimal thickness and maximum flat surface area is desired.

Method Development

Basic Measurement Requirements

Although the DSC is functionally quite simple, a good measurement requires some variation in methodology. Some basic guidelines for measurement criteria are:

Thermal Phase Transitions:

- Sample & Crucible mass
- Temperature Calibration file
- Sample measured as Sample

Specific Heat Capacity (C_p):

- Sample & Crucible mass
- Temperature Calibration file
- Baseline Correction
- Heat Capacity Standard measured as Sample + Correction
- Sample measured as Sample + Correction

Transition Enthalpy:

- Sample & Crucible mass
- Temperature Calibration file
- Sensitivity file
- A Baseline Correction measurement.
- Sample measured as Sample+Correction

Quantitative Data of Phase Transitions:

- Sample & Crucible mass
- Temperature Calibration file
- Sensitivity File.
- A Baseline Correction measurement.
- Sample measured as Sample+Correction

Phase Transition Measurements

Measurements looking to determine the onset temperature of phase transitions are typically robust and least likely to be affected by experimental errors. Users looking to obtain high accuracy data should consider performing multiple exploratory runs and attempt to narrow the temperature range over which they measure.

A general form for phase transition measurements is to first ramp to a value at least 60 °C below the expected transition temperature and then hold at that temperature to equilibrate. Once the contents of the furnace are equilibrated users may ramp the temperature over their anticipated range, to at least 60 °C beyond their expected melting point.

Heat Capacity Measurements

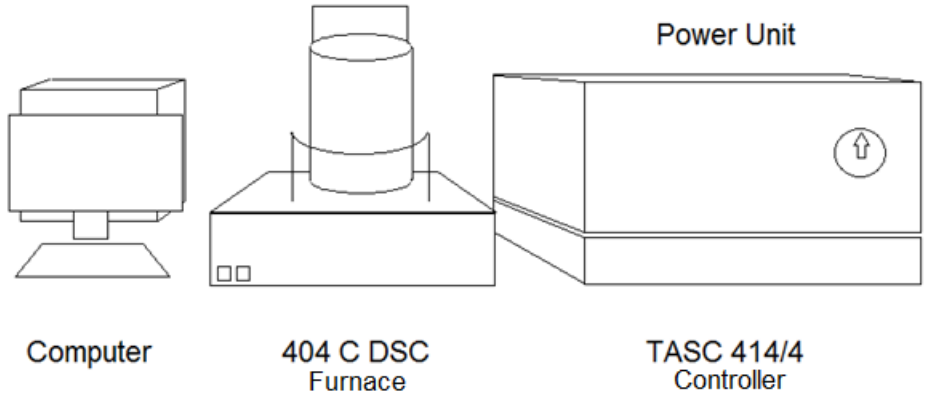
Similar to phase transition measurements, heat capacity measurements may also be subject to thermal lag induced error. Users desiring to measure heat capacity over large ranges of temperature may want to consider measuring heat capacity using a series of ramping periods with intermittent isothermal intervals (or in steps) to allow for equilibrium in between. This method helps to reduce the accumulation of thermal lag over long measurements.

Possible Issues

Theoretically a first order phase transition, such as melting or recrystallization, can be measured as many times as desired however this depends on two principles which may not always hold true.

1. Upon cooling the sample returns to its original state.
2. The sample does not demonstrate any evaporation, sublimation, reaction or other decomposition during the measurement.

Hardware



Instrument Components

Using the Instrument

Powering the Instrument On

The instrument should be given about 15 minutes to warm up after powering on. Each instrument component is powered on by physical switches located on their respective cases.

1. The power unit is controlled by a red dial on the front of the case. It must be rotated to on.
2. The TASC 414/4 switch is located on the right side at the back of the case.
3. The 404 C DSC switch is located on the left side at the back of the case.

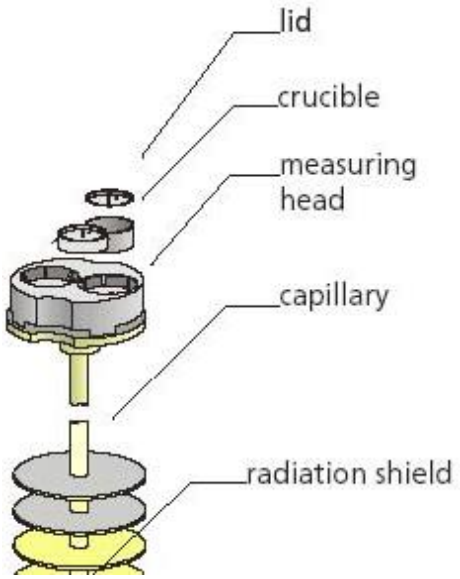
Operating the Furnace

The DSC furnace is raised and lowered using the two arrow buttons located on the front of the device in combination with the safety button on the ride side panel of the furnace base. The safety button and arrow button must both be pressed to move the furnace. Users should confirm that the vacuum indicator (to the right of the arrow buttons) does not have a red light on. If the furnace is under vacuum it should not be opened.

Once the furnace has been raised, and the crucibles loaded the furnace may be lowered again. **Prior to fully lowering the furnace users should visually confirm that it will not contact the measuring head**. Once the furnace has been fully lowered a green light should appear in the arrow button of the furnace base. If the light does not turn on check to make sure that the furnace is fully lowered. **Users should not raise the furnace unless it is below 100 °C**.

Loading and Unloading Crucibles

The measurement head contains two wells for the placement of each crucible. The rear well holds the reference crucible and the front holds the sample crucible. Each is placed in the center of its well and should sit flatly on the floor. The diameter of the sensor well is slightly larger than that of the crucibles so users should pay attention to positioning.



The sensor head is sensitive to misalignments which may throw off the instrument calibration. Users loading crucibles should try to minimize straining or bumping the measurement head as much as possible to avoid this. The green wire guard in front of the sample chamber is there for users to steady their hands during placement.

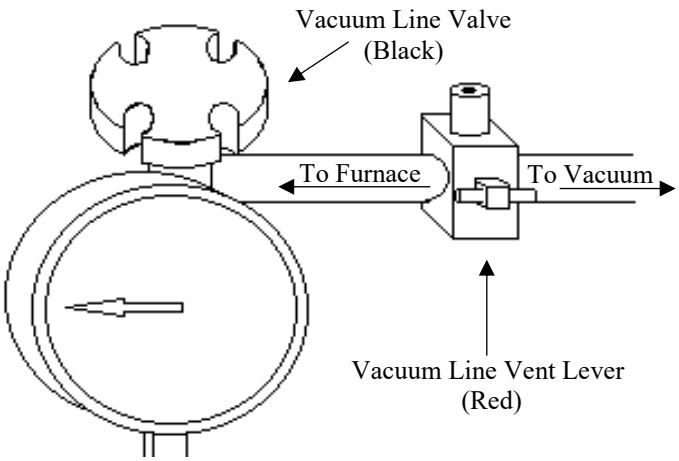
For users performing quantitative measurements positioning of the crucible should be consistent throughout the series.

Evacuating and Backfilling Procedure

Heat transfer from the furnace to the sample is directly affected by its gas environment. Most measurements benefit from a pure and uncontaminated environment which can be achieved by evacuating and backfilling the furnace using a high purity gas. The default configuration uses ultra-high purity (**UHP**) argon as the Purge 1 gas and a dried air on Purge 2.

The vacuum pump has a line connecting to the right-back side of the DSC furnace unit and is fitted with a manometer, black handwheel and a small valve with a red lever. Before purging the sample chamber users should first inspect the front of the furnace and look for a green LED light on the downward arrow, indicating that the furnace is fully lowered.

1. On top of the DSC furnace, **above the sample chamber**, turn the arrow shaped black lever to the right to close the sample chamber vent. The pipe attached to the exhaust vent should not show noticeable flow.
2. Turn on the red vacuum pump located on the shelf **above** the DSC controller.
3. Rotate the red lever until it is parallel to the tubing, as shown in the adjacent diagram.
4. Slowly open the vacuum line valve by rotating the black hand wheel. Opening the valve rapidly may displace the crucibles in the furnace. The manometer will show the pressure decreasing. Note that the manometer is initially resting at a 0 reading.



5. Allow the furnace to purge for approximately fifteen minutes. A red light should display on the front of the furnace at the vacuum panel which indicates when a vacuum has been established.
6. Close the line by using the handwheel to close the vacuum line valve. Turn off the pump and turn the red level until it is perpendicular, to vent the line.
7. On the front of the module press the button for the purge gas you intend to use.
8. Allow the furnace some time to backfill until the pressure gauge indicates a return to atmospheric pressure levels.
9. Once the sample has returned to approximately atmospheric levels, reopen the vent on top of the sample chamber using the arrow shaped black lever. Gas flow should remain on during this point and for the remainder of the measurement.

Users should perform about three iterations of evacuation and backfilling to insure a pure gas environment. Once finished users must make sure the gas continues to flow and that the black arrow valve on top of the furnace is turned to open again. This is the same valve referenced in step 1 of the evacuating procedure. This can be verified by placing a hand in front of the exhaust port to feel for air flow.

Warning: Failure to open the sample chamber valve before running a measurement may destroy the instrument and pose a danger to lab users.

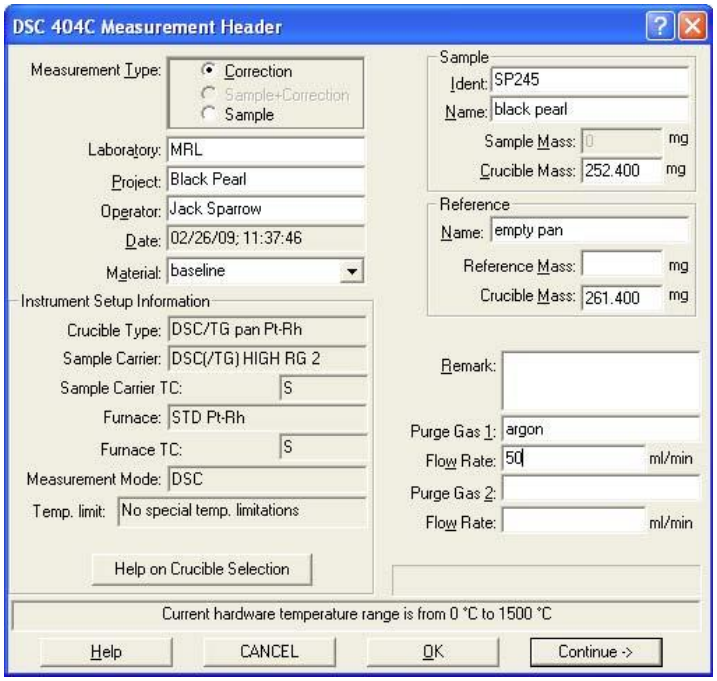
Software

The DSC uses two applications, one for measurements and the other for analysis. The icons of both programs, DSC 404C and Proteus Analysis, are located on the desktop of the computer. Data may be accessed by transferring it to the TEMPO network hard drive, also located on the desktop.

The DSC 404C program is used for instrument communication and measurements. Sample data and method programming are entered through this software. All data is saved in a temporary file until the completion of the measurement making the computer sensitive to memory use during collection. Users should avoid use of the computer during measurement to prevent data loss.

To start a new measurement, open the DSC 404C software and from the top menu select new measurement. This will open the DSC 404C Measurement Header to begin designing a measurement program.

DSC 404C Measurement Header



Users may enter the relevant information for their sample in this window. This information will be stored with the data collected during measurement and users may reference it during post-run analysis. The Proteus analysis software will also use information entered here to perform various calculations. At a minimum the measurement header will require the Measurement Type, Sample Identity, Sample Name, Reference and Sample Crucible Mass as well as a Sample Mass.

Measurement Type

When designing a measurement, the "**Correction**" option functions as a baseline. Selecting a run type of "Correction" will allow users to run a measurement of the empty sample and reference crucibles. Users may then run their sample, using the same crucibles, as the "**Sample+Correction**" option and the correction (baseline) will be automatically removed from the data. The correction measurement data is retained and may be viewed in the analysis program Proteus. Without a recent correction file the Sample+Correction option will be grayed out and not selectable.

Correction runs must follow the same temperature program and use the same calibration files that the sample measurement will be run under. As the hardware in the furnace can change with use it is better that the baseline be created as close to the time of sample measurement as is feasible.

When doing C_p measurements, Netzsch suggests not using a Baseline Correction for more than three consecutive measurements without rechecking. It is also suggested, at least for critical measurements, the Baseline be done twice. Comparison of the two baselines should differ by no more than 0.3 µV at the point of widest variation. If the difference exceeds 0.3 µV then a third baseline should be run and the comparison between the baselines performed again.

Measurements looking for onset temperatures of thermal events generally do not require corrections and may run using the measurement type "**Sample**".

Sample Information

Many of the fields, such as laboratory and project are for user reference and do not need to filled in. The material field is included in this and whatever entry is used does not affect future measurements or calculations. The **gas flow rate** field also does not affect any system settings but does allow users to note what parameters they chose to run at for future reference. The default setting for gas flow rate is approximately 75 cc/min.

Users should confirm that their crucible type is correct for their intended measurement and that the sample carrier field has "DSC(/TG) HIGH RG 2" written in. If something else is displayed then please inform the MRL staff before using the instrument.

Once the appropriate fields have been filled users may press Continue to move to the next step and select a temperature calibration. Selecting OK will end the measurement configuration.

Temperature Calibration & Sensitivity Files

When designing a measurement, users will be required to specify a **Temperature and Sensitivity Calibration** file. The **Temperature Calibration** selection window will automatically open to the correct folder and the most recent temperature calibration file should be chosen.

Temperature Calibration File

Multiple calibration files may be present, each attenuated to specific temperature ranges and testing conditions. For users measuring thermal events the default temperature calibration file (labeled Newest) will be appropriate. Users should select the temperature calibration file that covers their desired temperature range and was performed in similar conditions including:

- Crucible type
- Gas environment
- Heating rate

If unable to find an appropriate temperature calibration for the intended measurement, please speak with the MRL staff about having one made. Currently the DSC has calibration files designed for use with Al_2O_3 crucibles which are appropriate for most phase transition measurements. Samples that are incompatible with alumina may be run in the Pt-Rh crucibles however these pans often alloy with the metal standards used to make the calibration files. Users will need to purchase alternative standards for use with the Pt-Rh pans.

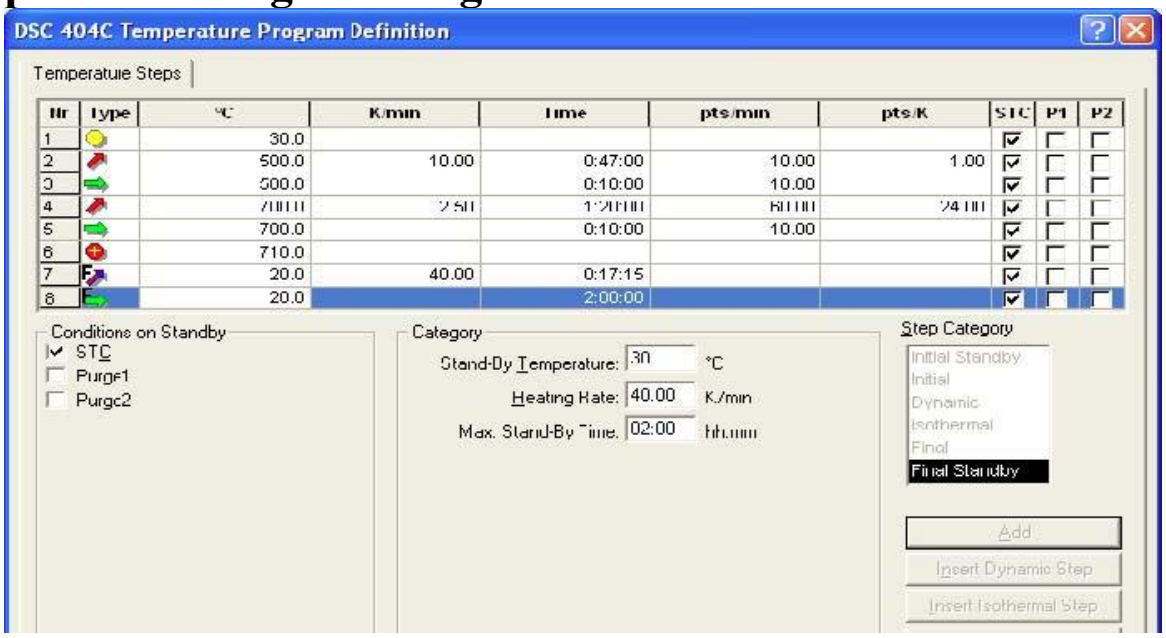
Sensitivity File

A **sensitivity calibration** file is required if the user wants quantitative energy information from their measurement. For phase transition measurements, which only look at temperature-event relationships, the Senzero file may be selected.

Quantitative measurements require a sensitivity file that matches the measurements parameters as closely as possible. When a sensitivity file is selected users may measure energy changes in mW/mg and integrate peak areas in joules/gram.

The creation of a sensitivity calibration file is an involved process that requires measurements of five to six standards. Users planning on doing quantitative measurements should verify that an appropriate sensitivity file is already present. If a new sensitivity file is needed please speak with the MRL staff at least a week in advance of the intended measurement.

Temperature Programming



Temperature programs are composed of, at minimum:

- The Initial Standby/Initial
- An Isothermal Step
- A Dynamic Step
- The Final Emergency Setting

The **initial** step sets the temperature value that data begins recording at. Once the measurement program is started the instrument will attempt to reach this initial value but will not record any data until it stabilizes at that point. The lowest recommended starting temperature is 40 °C due to low heat limitations of the furnace heating element.

Initial Standby is an alternative to the initial step. This step allows users to instruct the furnace to heat to a given temperature and then hold that for a set of time prior to recording data. This performs similar to an unrecorded isothermal step but may be used to heat treat the sample prior to data recording. Either initial or initial standby may be used. Most measurements will use the initial step.

The **isothermal step** holds the sample chamber at its current temperature for a designated period of time. These are often used to allow the sample temperature to equilibrate before or

after a dynamic step. Preceding or following a dynamic step with isothermals of 10 to 15 minutes will increase measurement accuracy and reduce artifacts in the data.

Dynamic steps act as ramp rates or heating rates. A target temperature is selected, as well as a heating rate. Note that the target temperature is in °C and the heating rate is in K/min. Ramping speed can be changed based on interest in a specific temperature range. For ranges containing data of interest a ramping rate of 10-20 K/minute is recommend. A faster rate may be set, such as 40 K/minute, for regions without interest. The instrument is capable of heating beyond 40 K/minute however this stresses the heating elements and may cause the sample chamber to overshoot the desired temperature. It is recommended that each dynamic step be preceded by an isothermal to allow the temperature of the sample and sample chamber to equilibrate.

Isothermal and dynamic steps also require a pts/min parameter which tells the instrument how often to record a data point during the step. **Data acquisition rates** can be changed based on interest in a specific temperature range. It may be entered as either points per degree or per minute. 60 points per minute is generally appropriate for sections of interest. The system has a limit of 24000 data points per measurement that it will notify you of, should your program go beyond that.

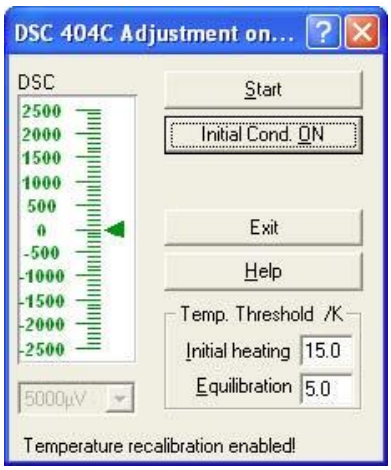
The **Final** step sets a safeguard temperature for the measurement. This will auto-populate with a value 10 °C higher than your highest temperature. If the instrument reaches this temperature it will automatically terminate the sequence as an effort to prevent damage to the instrument and sample.

Final Standby programs the DSC with instructions on what to do once the sample has finished. The instrument will drop to the stand-by temperature at the heating rate designated and attempt to hold it there for the given time. As this should always be a cooling step, the heating rate can be set to 40.0 K/min to allow the system to cool quickly and shorten the time needed per measurement.

Possible Issues

In measurements that do not use corrections (baselines) there are often artifacts in the data at points where the furnace heating rate changes so users should set temperature programs with steady heating rates that go at least 40 °C beyond your desired end point. Shorter dynamic steps allow for an increase in measurement accuracy but run the risk of cutting off phase transition events prematurely or missing them altogether.

DSC 404C Adjustment Window



After designing a temperature program and selecting continue, the DSC 404C adjustment window will appear. If a temperature recalibration file was selected the program will apply it at this point and the temperature indicated at the bottom right corner of the software screen will alter to reflect it. Selecting initial conditions on applies the options selected for the first step of the temperature program, i.e. the chosen gas flow and STC option if selected.

The Temp. Threshold /K fields specify an initial heating rate and equilibration range. In the adjacent example the furnace will heat at 15 K/min to within ± 5 K of the 40 °C standby temperature. This standby temperature is specified in your temperature program by the initial standby step.

When ready users may select the start button to begin the measurement. After reaching the initial temperature and equilibrating the programmed measurement will begin and data points will be plotted on the screen as they are collected.

Measurement Conclusion

Once your measurement has finished, the data is automatically saved in the user designated location. Users should wait until the furnace has cooled to a safe temperature of 40 °C before attempting to remove their sample. Users should not turn off any component of the DSC once completing a measurement.

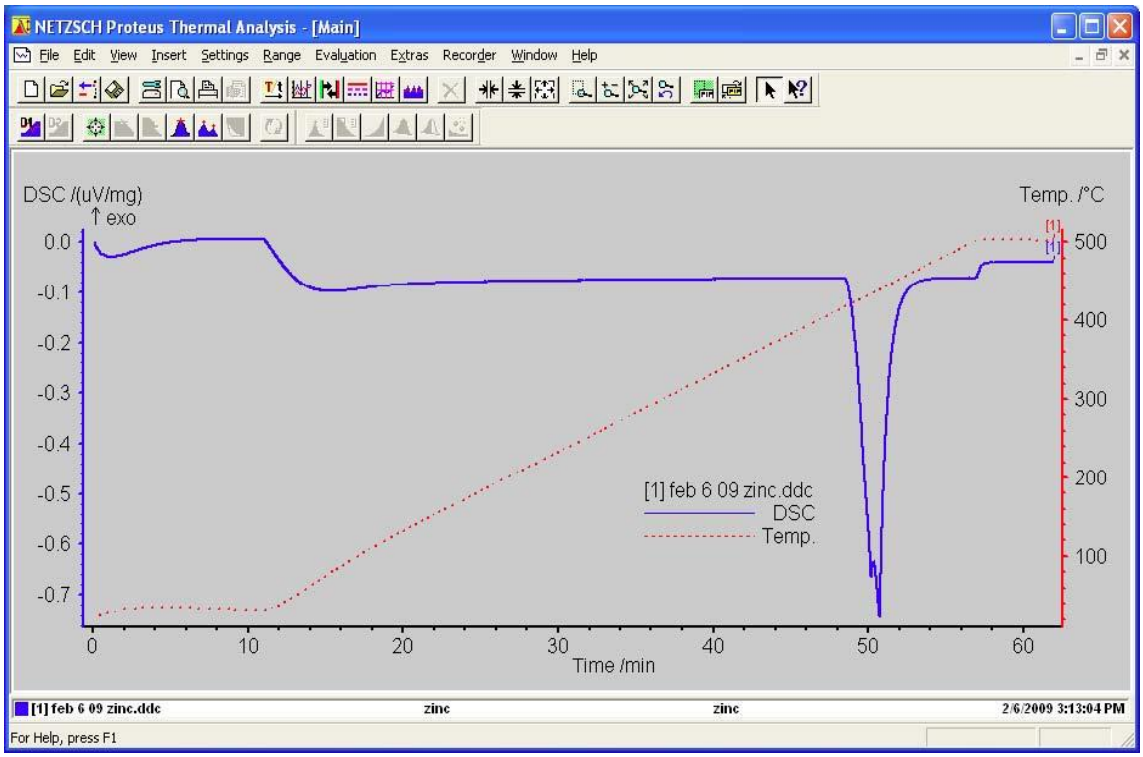
Shutting off the furnace power transformer before the furnace reaches a value below 500 °C will permanently damage the device.

The Proteus Analysis Program

Data analysis can be performed on the Netzsch Proteus Software, located on the desktop. Once a run has completed the data is automatically saved. Users can analyze their data in the Proteus software by double clicking the icon at the center of the desktop. Once open, data files can be selected using the ***File*** option, from the menu at the top of the screen or the folder icon in the toolbar.

The Proteus Interface

An example of a DSC measurement is shown below. The shown sample is heated to a peak of 500°C after a 10-minute equilibration period. The dotted red line shows the temperature over time and the blue shows the signal difference between the reference and tested material in units of µV/mg. Notice the phase transition around 420°C.



Curve Properties & Measurement Configuration

Curves may be right clicked to bring up some menu options for manipulation. Selecting the curve properties option will allow the user to customize the color and appearance of the line while the file properties option will bring up a window containing all of the measurement data stored in the file. This includes the user created temperature program and all the parameters that the measurement was performed under.

Temperature X-Axis

The software automatically displays time along the x-axis when opening new data however users looking for onset temperatures of transitions can display temperature as the x-axis by selecting the  button in the toolbar. This will cut your measurement into segments with each segment corresponding to one step in the temperature program. Isothermal segments will automatically be hidden as these will consist of a flat line.

Hiding Line Segments

Users wishing to high (or unhide) a line segment may right click on the curve and select view segments. This will produce a window listing each segment which users may check or uncheck to display. Alternatively they may right click a segment and select ***Hide this segment***.

Exporting Data

Experimental results may be exported as either an ASCII or ANSI Unicode file. Typically, users will want to select ASCII and then choose the ***.csv*** format. This will export their data as a comma separated values file which may then be opened in excel.

To export the data, select the ***Extras*** option from the toolbar at the top of the Proteus window. In the drop down menu choose ***Export Data*****.** Fill out the dialog box that should pop up and choose where you would like to export your data to.

Retrieving Data

To maintain the integrity of our instrument computers, USB access is disabled. Instead each instrument has access to a shared TEMPO hard drive, with a shortcut present on the desktop. Users may access that hard drive, from the instrument, and make a folder for themselves. They can then copy all data from the instrument computer to the shared hard drive. This hard drive may then be access by the shared computer in TEMPO labs, and data can be retrieved there using a USB device.

Ending Notes

Further Reading

For more information the TEMPO Gauchospace page on differential scanning calorimetry contains several useful documents as well as the appendix to this guide. Users interested in using different operating parameters or using a different method are encouraged to speak with TEMPO staff.

Mettler-Toledo's website also contains a wealth of freely accessible documents and papers on thermal analysis principles and methods.

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