USE OF THE DIFFERENTIAL SCANNING CALORIMETRY DSC Q2000.

		Date
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PURPOSE

This SOP describes how to operate and use the differential scanning calorimetry DSC Q2000 (TA) located in the CAF facility.

SCOPE

This SOP describes procedures to be followed by staff and students when using the DSC Q2000 in the CAF facility.

RISKS.

DO complete the CAF Blackboard safety test, official training and ONE run supervised by the CAF technician before using the equipment. For future runs you will need to book the instrument.

See the attached Risk assessment of the instrument.

You must have your own Risk assessment of the chemical products you are going to analyse.

Please if you want to analyse toxic, explosive, flammable, longer term health hazards chemicals and corrosive samples talk with the CAF technician before running your samples (check the MSDS). Take also the MSDS of the likely degradation products into account. We may need to take extra safety measures.

PROCEDURE.

PRECAUTIONS

- Check the **sample compatibility** with your sample **pan** at temperatures, atmospheres and times you plan to use the instrument. See Appendix.

- Do not run a DSC sample beyond its degradation temperature. This may damage the instrument and is the most common reason for the autosampler to fail.

- Do run a **TGA first** if you do not know the temperature range for degradation.

- The **operating temperature range** is (-90,400°C). However, please talk with the CAF technician if you need to go beyond 250 °C, we may be able to use an alternative instrument. **More information** about the instrument specifications and some theory/applications is found in the blue binder close to the instrument,

- Do **not let anything drop into** the autosampler carrousel or cell. If anything does notify the responsible CAF technician and wait until he can assist you. Please **clean any spillage** on the instrument surrounding area.

- If you encounter an Error message please STOP the action you are doing and look for technical assistance. There is a paper with a list of common errors next to the PC. <u>The only errors you can select OK an</u>d continue working as usual is:

  - *"The requested operating requires elevation"* (it occurs since you are using the software without admin rights).

  - "*A cooling ramp may exceed cooling capacity. Run continuing".* This is error doesn't require any attention. The sample will carry on running. The software has a safe guard that warns you of temperature ramps above 10 °C/min, but our machine is fine up to 150 °C/min.

- Do not attempt to use if signage indicates that the instrument is out of use.

- Please for **overnight sequences** first look for technical assistance.

INSTRUMENT STARTUP

- Please be careful **since other experiments may be running**. If you are in doubt as to this point, look for technical assistance. Runs from multiple users can be added to the sequence. Check the instrument status at the top bar of the software.

- The **instrument and the computer** should always be **on**, if not please ask for technical assistance.

- The **cooling system** is in standby, check that it is in Event mode (it should be always switched on too). If the cooler is OFF look for technical assistance.

- Open the N_2 **(g) regulator.** It is located on the wall, behind the cooling system. You need to put the black gas tap vertical. Check that the flow is already adjusted to 10 psi (the black mark), it takes a couple of minutes to reach this value. If it is not adjusted, please use the white screw to regulate it. Allow the gas to run for 2 minutes.

- **Log on** to the DSC CAF user, password: Tgacaf19. If somebody has signed out you will need to sig in to the PC:

  Username: SCPC9017\dsccaf   Password: Tgacaf19

  Do not log on to Rdg-home with your individual logon as the instrument specific software will not function.

- To **open the instrument software**, on the computer double click on the "TA Instrument explorer" and then on the DSQ2000 icon, if they are not already open. The computer is also used for the TGA, which has a similar software.

- From the top menu bar on the software select →**Control**→**Event**→On

- Allow the system to **stabilise for 10 minutes**. After this period, the green light of the cooling will be on and the sig1 (top menu bar) of the software will indicate -88˚C (approx.). The main T (cell T) should remain at 40 ˚C.

  > CAUTION: If the main T falls below 40 ˚C you have to select Control > Go to Standby T.

- During this time **prepare your samples** and reference pan in the appropriate manner.

SAMPLE PREPARATION. For more information see printed ppt slides next to the PC. For liquid samples first look for technical assistance.

You will **need an empty pan as a reference**. First you will start with the reference pan. You need to write all the weights down. Do not mark your pans. Ideally the difference between sample and reference pans should be less than 1mg.
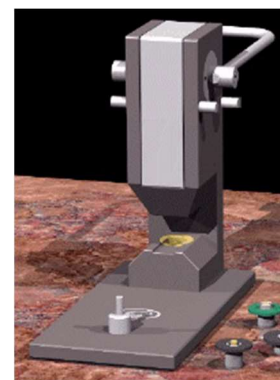
- Select **DSC sample pan appropriate** for your sample. For more information please see the document: "MOST COMMON DSC PANS", there is a copy next to the instrument.

Figure 1. Tzero press and dyes.

- **Clean** the press dies and the bench.

- **Weigh your pan and pan lid** (you can tare the press upper die to use it as a clean platform while weighing). Do a pin hole on the lid if needed.

- Only for the sample: Remove your lid and keep it. Tare your pan without the lid. Then **add enough amount of sample** to cover the bottom of the pan and gently tap the pan to remove the out (if solid) Weigh your sample, you should have approximately 2-10mg.

> CAUTION:
> To prevent balance contamination, please when adding your sample into the bottom of the pan, do it on a piece of aluminium foil, on the bench. Do not contaminate the outside of the pan and clean any spillage.
> Always use tweezers to pick up your pans.
> You should only use the small metallic platform to keep any clean parts (lid, dyes...). Do not contaminate this plate.
> Clean any spillage once you finish.

- **Put the lid** on the pan in the correct direction.

Figure 2. Tzero Pan with hermetic lid.

- **Load correct dies** for the press for the type of pan you are using. The pan boxes are colour coded to the die mounts (see also help menu).

- **Crimp your pan using the press:** Put the pan on the bottom dye and make sure it is correctly seated on the groove. Then lower the handle very gently and put it back in the standby position.

  > CAUTION: Press does not require large force to operate. Take care not to overload

- **Check** that the pan is not damaged or bent, a symmetric sealed must be achieved see Figure 2. `Tzero Pan with hermetic lid.`

LOADING YOUR SAMPLES

> CAUTION: DO NOT use sample positions #1 and #R1 as this contains the indium verification sample and reference.

- **Ensure the autosampler is NOT** in the process of loading or unloading a sample. You can check the status of the run at the top bar of the software (next to the temperature signal) o at the bottom timer.

- Remove the autosampler **lid**.

- Place your prepared DSC pan in a free position on the sample holder **# 2-49**. It must perfectly sit on the groove.

- Place your reference DSC pan in a free position #**R2-4.**

RUNNING A SAMPLE IN A CUSTOM PROGRAMME: You need to create one run per sample.

- Select the **EXPERIMENT tab** on the left hand menu bar.

- If no other experiments are running **select 'Run 1'** and delete the other runs, otherwise click on 'Append' to create new run. As default settings this run will have the programme parameters last used on the system.

- Enter details under **'Summary'** tab

  Mode → Standard

Test	→ Custom
Sample name	→ As chosen by user
Pan type	→ Tzero aluminium hermetic (these are the standard ones)
Sample size	→ As measured by user (only the sample mass, not the pan mass)
Pan number	→ Enter pan position you selected
Ref	→ Enter pan position you selected
Pan mass	→ (Select tick box) As measured by user (only mass of the pan + the lid, not the sample)
Comments	→ As required by user. It is displayed next to the file when you open it.
Data File name	→ C\TA\DATA\DSC\USERS DATA\YOUR SUPERVISOR'S FOLDER\YOUR OWN FOLDER (**Each user should create his own folder).** You save one file per sample.

- Enter details under '**Procedure' tab** .

Test	→Custom
Notes	→ not needed
Method	→You can open, save or create a new method. You can also modify the current method without saving the changes.
	To create a method select Editor and double click on the desired segment of the list to add it to your method.
	Common method:

> data storage on
> zero heat flow at the selected T (sets the absolute value of the heat flow signal to zero at the end of a five minute isothermal period. It makes the runs more reproducible. Compulsory for heat capacity measurements).
> Mark end of cycle 0. *This segment only marks to help you with the software analysis, it does not* repeat anything.
> Ramp: __ ˚C/min to final T __˚C
> Mark end of cycle 1
> Data storage off
> For T > 250 ˚C talk first look for technical assistance.
> If you want to use other segments, please first talk with the CAF technician. Some segments are not allowed.

You can save the method file using the diskette icon and click OK.

See printed documents in the binder next to the instrument for more detailed recommendations for a particular kind of data collection, e.g. Tg measurement

Please **do not change**:
> Advanced parameters (0.10 s/pt, load T range 18-20˚C)
> Post test parameters (Unload T range 35-45˚C, delay time 0min. discard pan in waste bin not selected)

It used to be 0.50s/pt (0.10 provides higher resolution and changes a little bit the area as a result) and load/unload T range of 40˚C.

- Enter details under 'Notes' tab:
  → Only complete Operator name
  → Don't change any other option (N_2 flow rate must be 50 mL/min)

- Click on 'Apply' to save details.

- If you plant **to run more than one sample** and you think that you'll have time to prepare another sample while the instrument is running sample 1, then right click on the run 1 and select **copy and append.** Then you only need to repeat the previous procedure for the new sample. You can create more runs while the instrument is running following this procedure.

- **Select run (1)** of your sequence list. The red arrow should appear opposite Run 1, if not move the mouse courser to the left of "Run 1" and click.

- To **START** the experiment click the green start icon on the top bar of the instrument software. Sample will be automatically loaded, the method will start when within specified conditions and the sample will be auto-unloaded at end of run. Then the instrument will automatically continue to next run

> CAUTION:
>
> While the instrument is in progress **avoid bumping** the bench and the instrument.
>
> If there is **any problem during** the pan loading process or during the run, please press the **red** stop icon on the instrument software (top bar) to stop the experiment and look for technical assistance.
>
> There is more **troubleshooting info** in the APPENDIX.

- If you already have the results you were expecting, **you can stop** the experiment at any point by selecting the stop red icon on the instrument software (not the reject one). The data will be saved.

- You can **extend the run** adding the desire segments while the instrument is running. On the right of the instrument software you can find the running segment description, right click and select modify running method. Then save changes.

SHUTDOWN PROCEDURE

- At the **completion of the run**, the instrument will unload the pan once the unload T is reached.

- **Remove samples** from autosampler and dispose of in the plastic vial next to the PC.

- From the top menu bar select →**Control**→**Event**→**Off**

- Allow '**temperature' and 'Sig 1 Temperature'** (flange temperature), to return to standby T (40°C). This may require a short wait. These temperature are displayed within 'instrument signals' at the top right of screen. **If this does not work** please select control, go to standby T.

- Fill in the **Excel log book** (desktop)

- Once the instrument has reached the standby T, **you can switch off N2 (g) supply,** closing (horizontal) the black N_2 tap. Don't change the regulator original pressure (10 psi).

- **Leave everything on** (the DSC software, the instrument and the cooler should be always on)

- Don't log off computer. The Windows screen saver automatically appears after 15 minutes

- Please send all your **data by email**, do not save it in a pen drive. Please note that data may be purged from the instrument once a year (after back up) and therefore backing up your data onto your own computer regularly is advisable. For <u>data analysis</u> please see Appendix.

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APPENDIX.

<u>SAMPLE COMPATIBILITY WITH YOUR SAMPLE PAN.</u>

Aluminium pans are normally made of very high-purity metal to prevent unwanted catalytic effects. Corrosive chemicals may react with these pans (especially bases), water can also react with the aluminium used to make these pans.

For food analysis, for example, other more inert pans may be recommended.

For the selection of the pans please see the printed document in the binder close to the PC: "MOST COMMON DSC PANS".

<u>DATA ANALYSIS.</u>

You can use TA UNIVERSAL ANALYSIS SOFTWARE, installed in the desktop of all the Thermal PCs. You can also download the software from TA website: <u>https://www.tainstruments.com/installation-of-universal-analysis-from-the-advantage-software-package/</u>. Ask the CAF technician for a training. You can find a manual in the Desktop too.

You can export your data into Excel selecting File > EXPORT DATA FILE > plot signals or file and plot signals > then select: spreadsheet text file, Unicode, finish and save it adding the ".txt" to the file name. Please be careful since not all the data are exported in the desirable units (such as weight normalised units).
You can also export the plot: File > Export plot > Choose the following options: clipboard, metafile, full size, pen width 3 is (the default). Then select export and paste it wherever you want. You can also remove the head of the picture: right click on the picture and select crop.

To save the graph as a PDF you can select file > save as > analysis and data or export as PDF.

For integration of peaks please see the examples of baseline constructions in the printed document in the binder next to the instrument. See also the printed document "DSC, some theory" for information about baseline types, peak integration, enthalpy calculations and more issues.

There is also very interesting information in the printed document: "Interpreting Unexpected Events and Transitions in DSC Results".

TROUBLESHOOTING. If you face an error please always look for technical assistance.

- If there is **a problem with the autosampler you** need to figure out what happened-i.e., sample degraded during run and caused the pan to move, the lid came off and the pans has changed position, the arm has become sticky because it need to be cleaned, the autosampler tray is dirty...

DO NOT SIMPLY RESET THE SYSTEM WITHOUT determining the cause of the problem! Almost all the failures are due to sample prep or running the T too high.

- PLEASE REFER TO THE printed DSC COMMON ERRORS DOCUMENT for other problems such as "**DATA TRANSFER PROBLEM**".

ADDITIONAL INFORMATION located in the binder next to the PC:

- For the SELECTION OF THE PANS please see the document: "most common DSC pans".

- SAMPLE PREPARATION: For more information see the printed ppt slides: "Basic info for training. DSCQ2000".

- METHOD INFORMATION:

    See the printed ppt slides: "Basic info for training. DSCQ2000".

    There is also very interesting information (especially for Tg) in the printed document: "Interpreting Unexpected Events and Transitions in DSC Results".

    See TA Applications Notes Library (saved in Browser Bookmarks).

- DATA ANALYSIS:

    See the printed document "DSC, some theory" for information about baseline types, peak integration, enthalpy calculations and more issues.

    For integration of peaks please see the document with visual examples of baseline constructions

    There is also very interesting information (especially for Tg) in the printed document: "Interpreting Unexpected Events and Transitions in DSC Results".