Differential Scanning Calorimetry (DSC) Practical Training Course

2020
Part 2
Maintenance



Discovery DSC2500 DSC250 DSC25





Training Course Agenda

- Online help
- Cleaning a contaminated cell
- Lid cleaning and alignment
- Baseline verification
- Gripper fingers cleaning and checking
- Autosampler calibration
- Prepair Indium sample
- Cooling accessory use and maintenance
- Purge gases & flow rates
- Troubleshooting
- Video's
- Finding part numbers

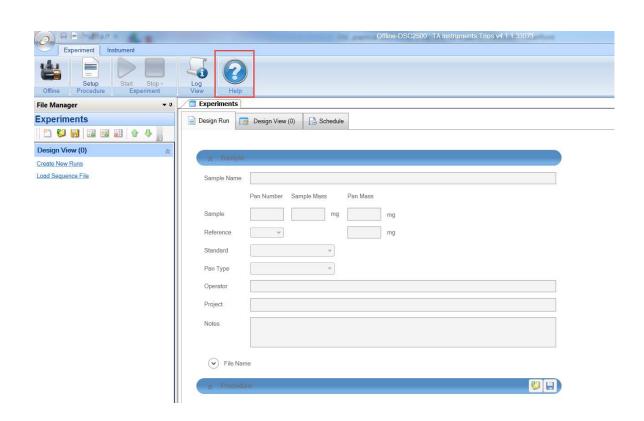


Trios Online Help

Select help function



in the software or press F1.

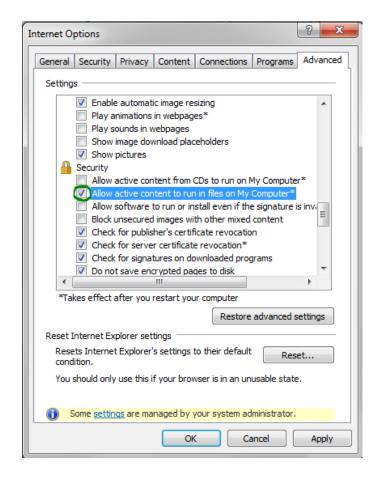




Trios Online Help

If the TRIOS Online help does not work you may need to enable "Allow active content to run in files on My

Computer*".





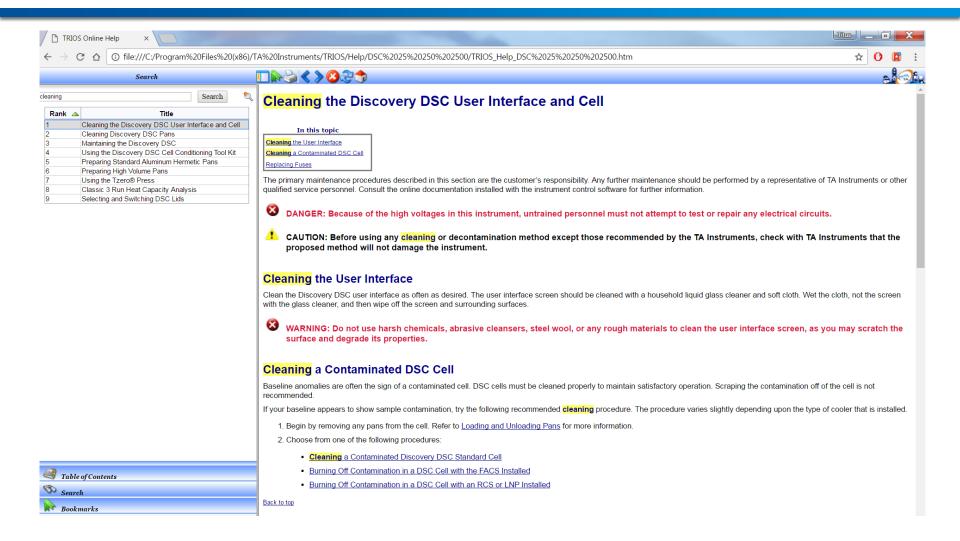
The help function is very useful



•Also checkout the Desktop manual









Gently clean the sensor platform with the fiber brush





Also clean the sensor base and the silver block





Clean the Lid area





After cleaning the cell



Wear safety glasses and use vacuum cleaner to remove fibers

Avoid damaging the silver cell with the vacuum cleaner



After cleaning the cell

Only if vacuum cleaner is not available!



Disconnect purge line from the back of the instrument and blow out the fibers

Make sure that the lid surfaces are clean

After cleaning the cell

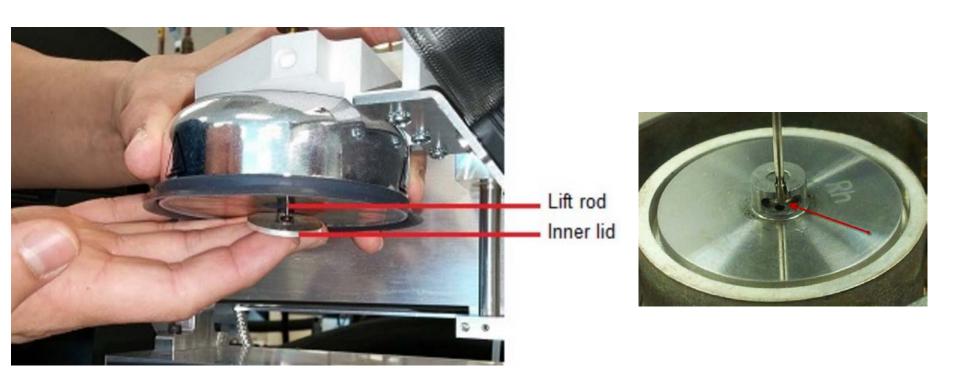
Use cotton swab and solvent to clean the cell



- Heat the cell to 200°C for 5 minutes to drive off any remaining solvent
- Replace the reference pan with a freshly made pan

Removing and installing the lid

Remove the inner lid by lifting it up and sliding it off of the lift rod





Cleaning the lid

Standard lid is coated with Rhodium



- Only use a tissue and solvent to clean this lid
- High temperature lid is not coated
- High temperature lid can also be cleaned with glass fiber brush

- Open the lid with the cell at ambient temperature
- Remove the Autolid arm cover by pulling out the edges and rotating it to the left

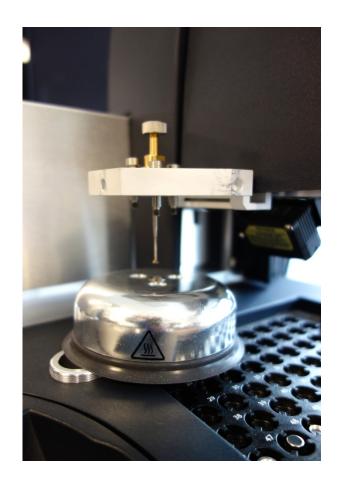






Loosen the three screws and remove the outer lid (heat shield) by sliding it down over the lift rod







Loosen the nut and remove the lift rod



Verify that the liftrod is straight by rolling it on the table



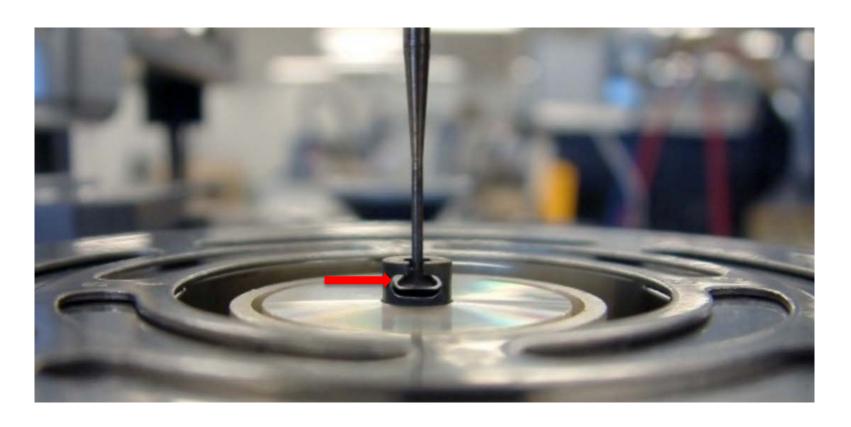
Part number lift rod: 972633.001

Re-install the lift rod and mount the inner lid



Discovery auto lid height calibration

Close the cell with the lid mounted



Adjust the height so the lift rod is not touching the lid

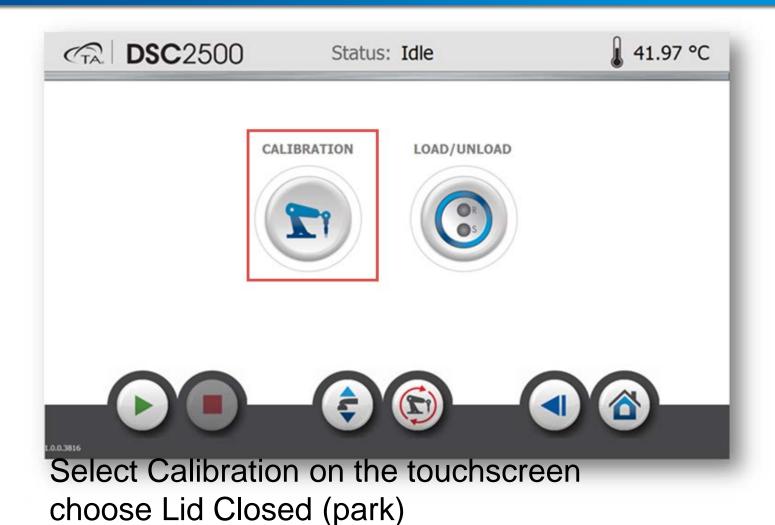


Autolid calibration

Select Autosampler on the touchscreen



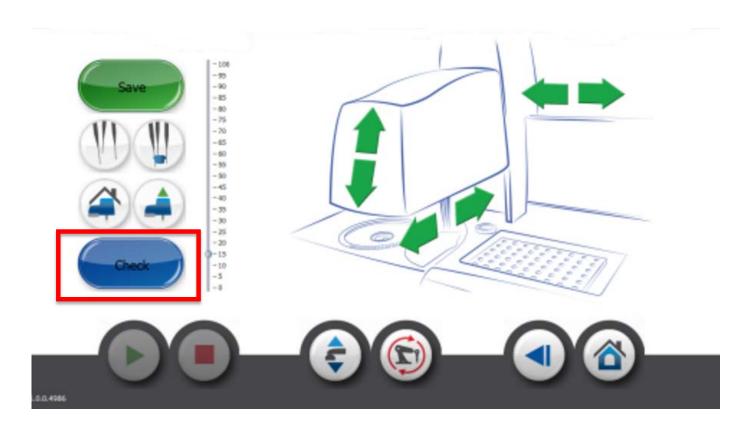






Discovery auto lid height calibration

Use the touchscreen and verify the center position of the lid



Adjust if needed and the new position



Discovery auto lid calibration reassembly

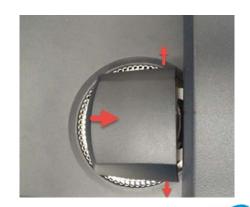
Remove the inner lid and install the outer lid again





• Install the innerlid and lid arm cover by pulling out the edges





Verify that the lid cover fits properly

Do a reset autosampler to home the autosamplers

sensors





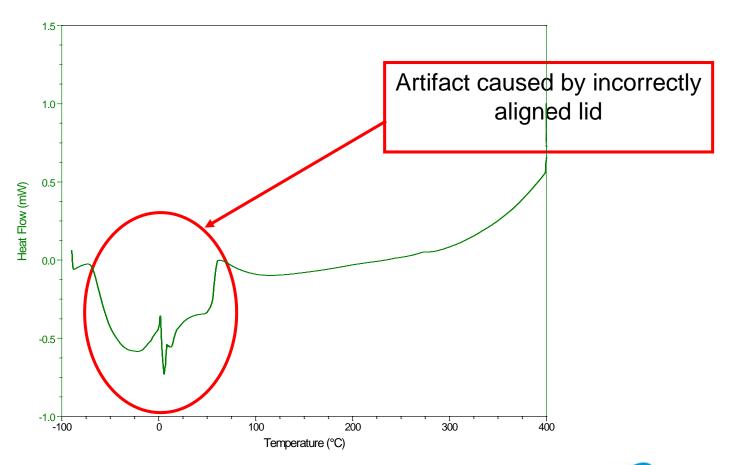


After cleaning

- Verify the performance by running a baseline check
- Verify enthalpy and temperature with Indium
- If results are not good, repeat previous procedures
- Use extended cleaning step if needed



If the lid is not correctly positioned, the data will be noisy and have artifacts



Extensive cleaning

Rhodium lid

- Equilibrate the furnace at 400°C
- Switch to air purge or open the furnace at 400°C

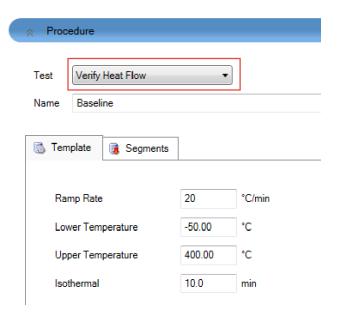
Silver lid

- Equilibrate at 500°C or 550°C
- Switch to air purge or open the furnace at 400°C
- Do not stay long at high temperatures (max. 5 min.)
- Clean with brush and cotton swab at room temp.
- Check baseline and Indium
- Do not calibrate a contaminated cell!



Baseline verification template

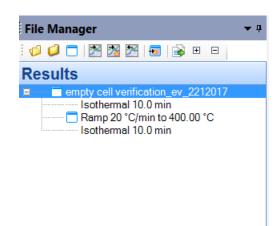
Use the template to run a baseline verification

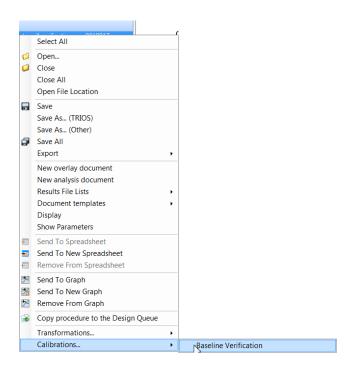


Analysis is done automatically when template is used

Baseline Verification Analysis

- You can manually select baseline verification analysis
- Open the file and right click on the file
- Select Calibrations, Baseline Verifications

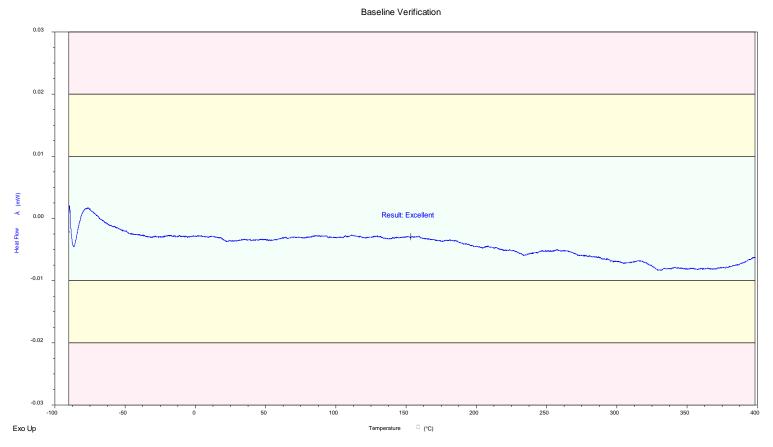






Baseline Verification

 A good baseline is in the green or yellow range (without startup hook)



If you recive "Check Tzero cal" have a look at the Delta T and Delta Tzero signals first



Verify baseline

The full Tzero calibration is usually done during maintenance by TA Instruments and should not be needed in between the annual visits.

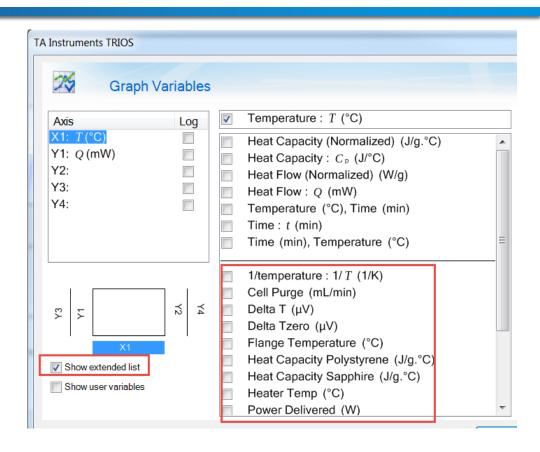
Send datafile to TA Instruments for advise if you are in doubt if the result is good!



Diagnostic Signals

Select Graph Variables

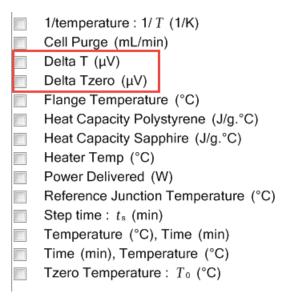
Show extended list



More signals are available

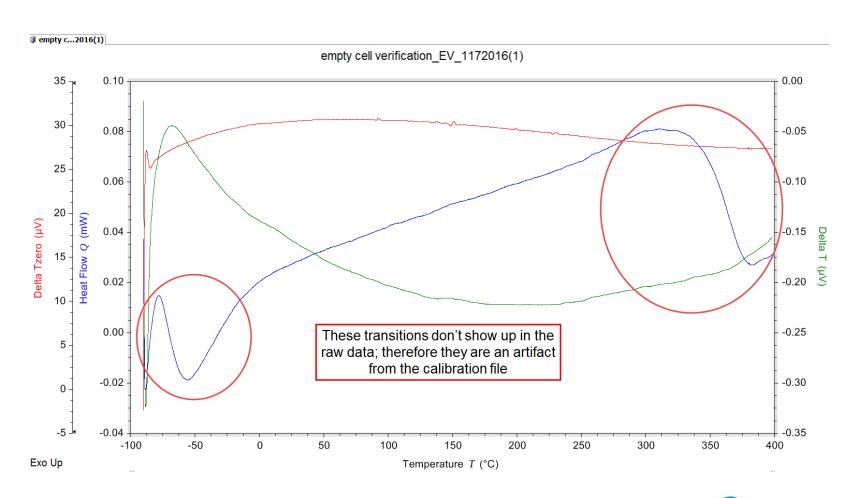


Delta T and Delta T zero can be helpful to diagnose baseline issues



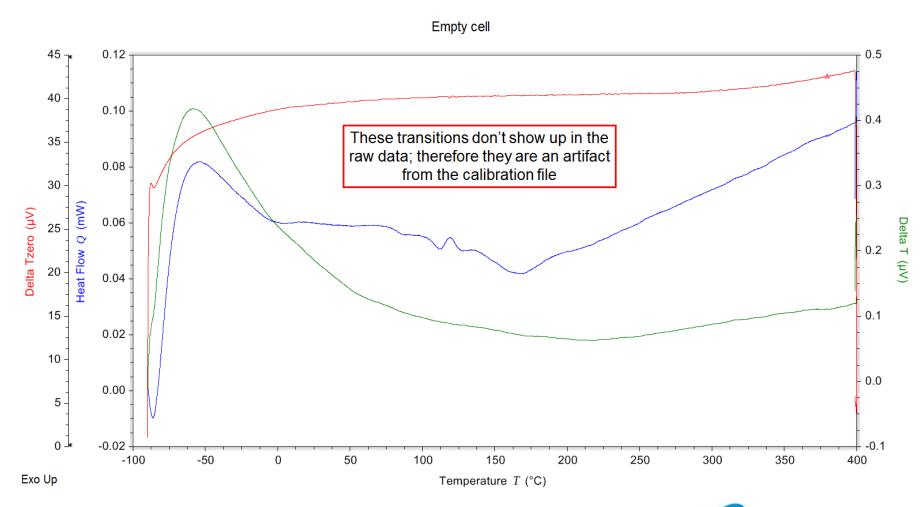


DSC empty cell with Diagnostic signals

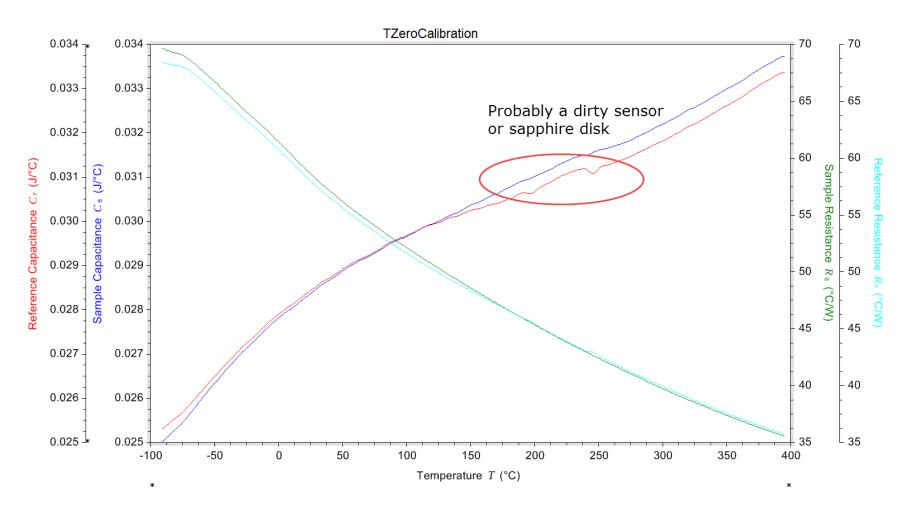




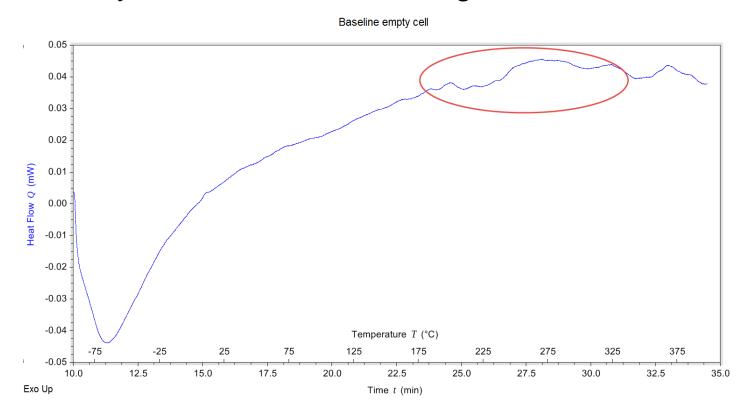
Another example



Bad Tzero calibration



- Baseline after previous bad Tzero calibration
- Why is the Baseline not so good?

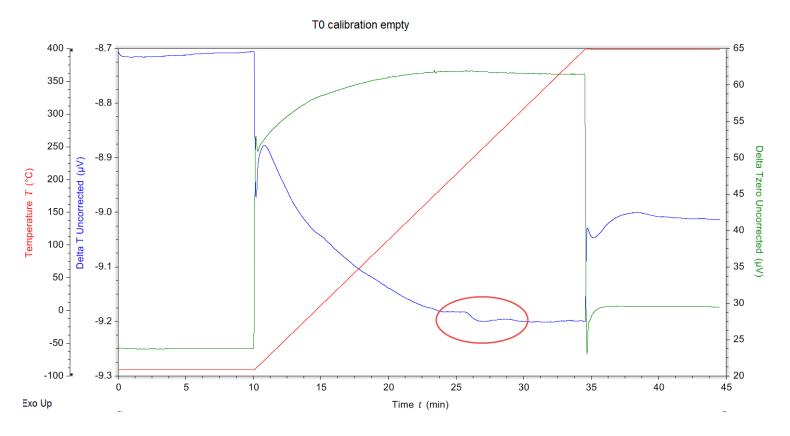


Investigate calibration files with diagnostic signals



Value of Diagnostic Signals

Empty cell calibration run from previous T0 calibration

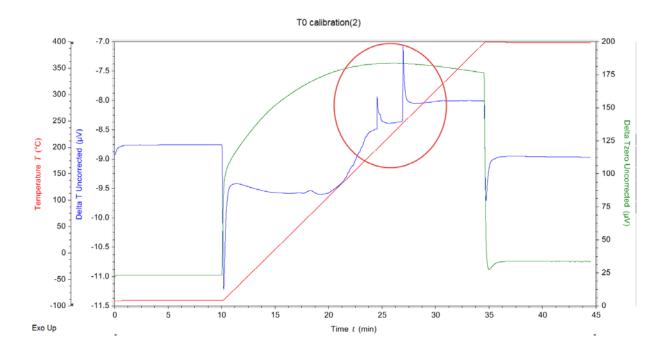


Steps from dirty sensor



Value of Diagnostic Signals

Sapphire run from previous T0 calibration



- Steps from dirty sensor
- This was also visible in the empty cell calibration run
- Don't calibrate a dirty cell



Cleaning gripper fingers

- Gripper fingers can get dirty if a
 - Dirty sample pan was loaded
 - Sample pan that opened during a run was unloaded
- Select to load sample pan 54 and stop the arm above the tray
- Gently clean the gripper fingers with a tissue and some solvent
- If solvent is not enough then a fine sandpaper can be used

Avoid the use of Acetone
Be careful not to bend the gripper fingers



Checking gripper fingers

- Open the lid and stop the autosampler in the open position
- Carefully rotate the gripper fingers and check if the tips are not bent
- Reset the autosampler

Bent gripper fingers can only be replaced by a TA instruments Engineer



- Only calibrate if the gripper fingers are cleaned
- Calibrations to be checked are:
 - 1.Autosampler tray position 1
 - 2. Gripper closed
 - 3.Sample
 - 4.Reference
- Do not save a position that is not good



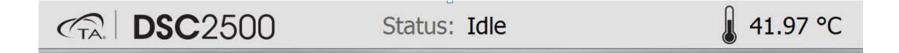
- Place a sapphire disk on tray position 1
- Reset Autosampler via the touchscreen

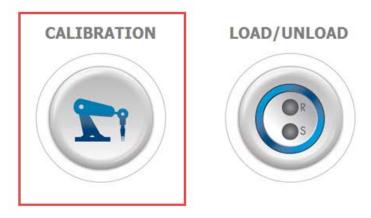


Select Autosampler on the touchscreen





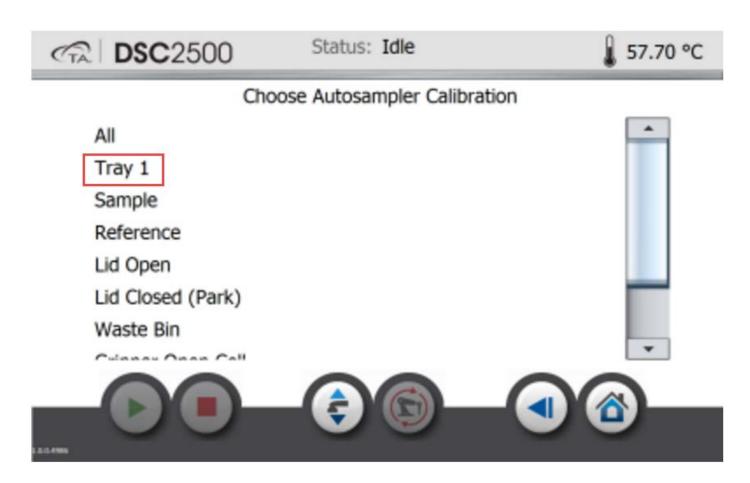






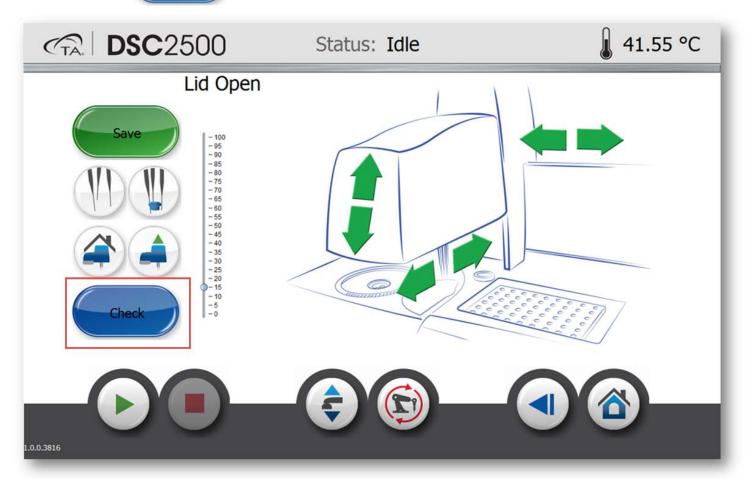


Select Tray 1



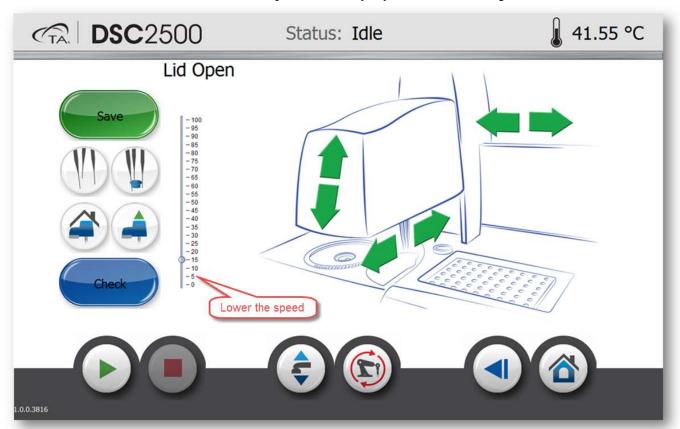


Select to verify the position





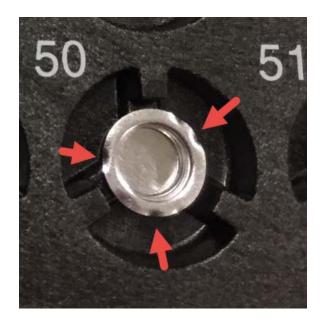
Set slider bar to a low speed (5) and adjust with the arrows



Save the new position when satisfied

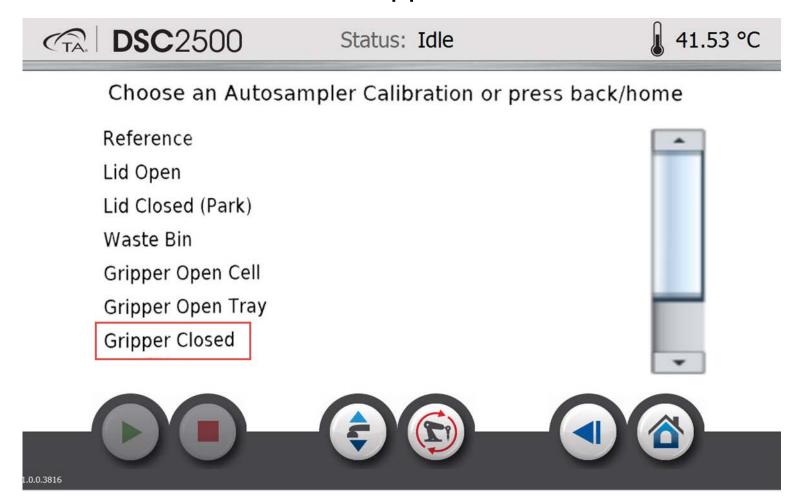


Frequently used (reference) pans can show dents

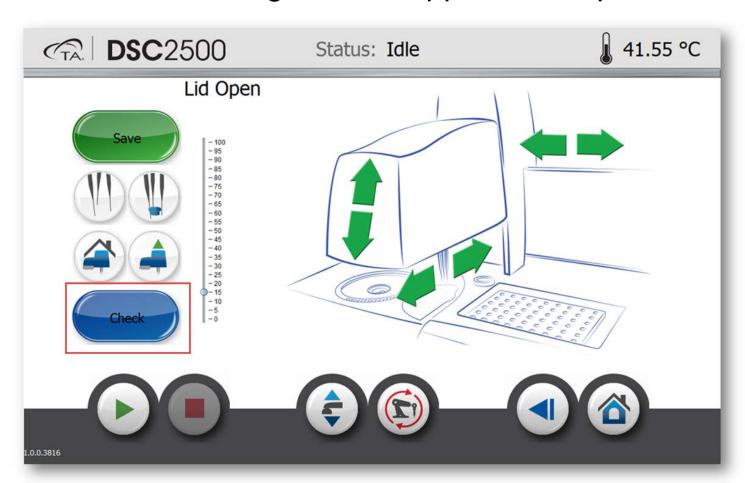


- This is an indication that the grippers are set too tight
- Adjust the gripper closed position

Scroll down and select Gripper closed

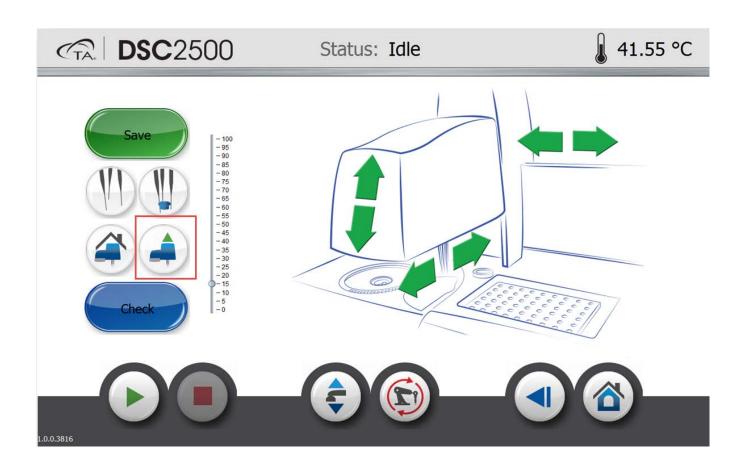


Select Check to grab the sapphire from position 1



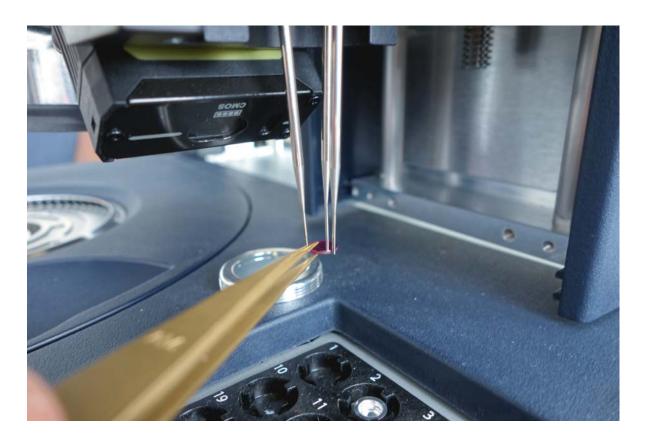


Raise the gripper arm





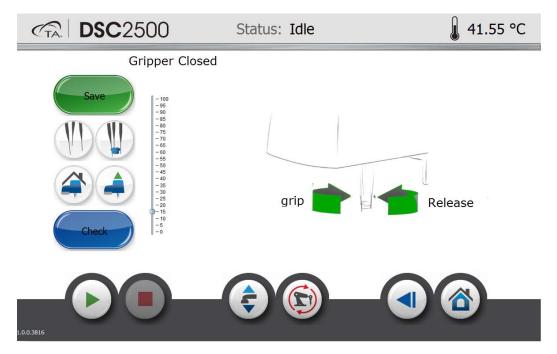
Check with tweezers how well the sample is held



Release the grip tension if needed



Adjust with the arrows



- Sapphire sample should just be held
- save the new setting

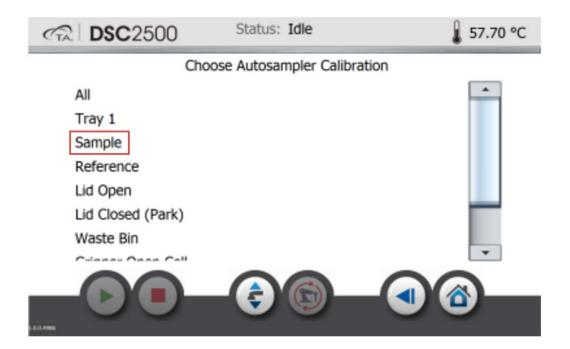


Autosampler cell and reference calibration

Select Sample position and (



Sapphire on tray position 1 will be used for this





Autosampler cell and reference calibration

Press



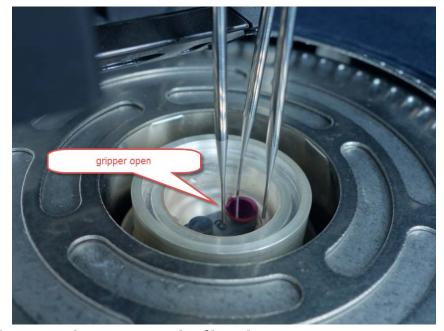
Adjust with the arrows



- Observe the position of sapphire with grippers open
- Use grip/ release buttons after every correction



Make adjustments in the down position



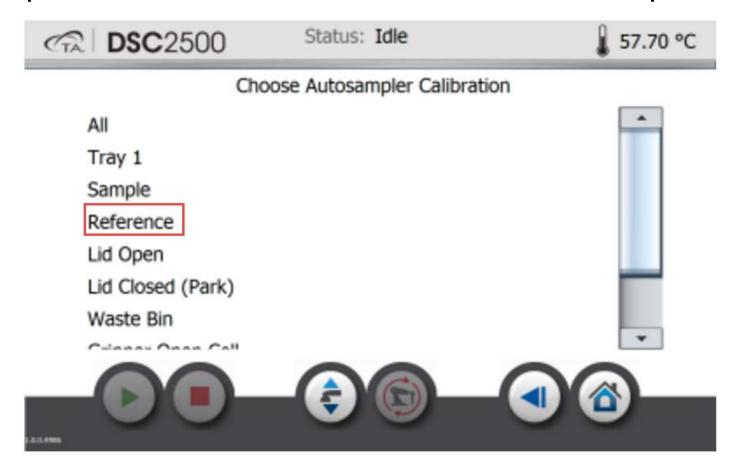


the new position when satisfied



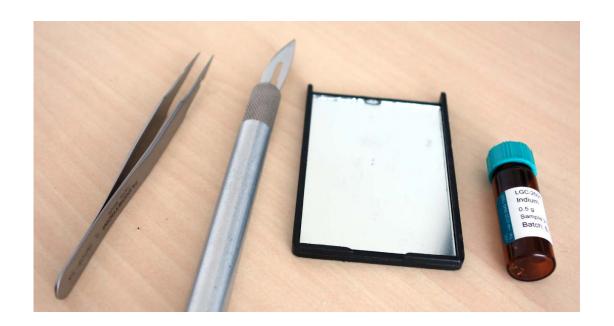
Autosampler cell and reference calibration

Repeat the same calibration for the Reference position





Clean tweezers, scalpel and a flat (glass) surface with ethanol





Cut a piece of Indium (3-5 mg)





Press it flat with the cleaned, rear part of the tweezers





- The Indium sample will often stick to the glass
- Scrape it off and flatten it again with the side of the scalpel





- Weigh the sample pan and lid
- Make a note of the weight (for T4P mode only)



Tare the balance



Pick up the Indium sample with the sharp tip of the scalpel









- Place Indium sample in center of the pan
- Make a note of the weight
- Recommended sample weight is 3-5 mg

Close the lid with the matching die set





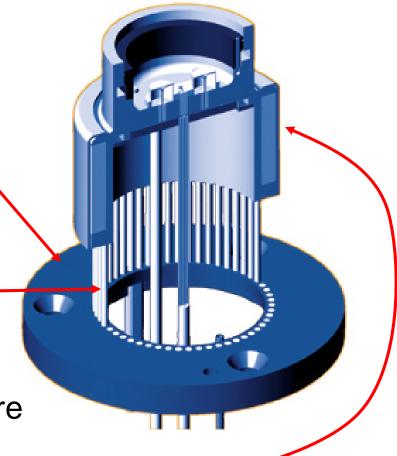
- Place the pan on the auto sampler tray
- Setup calibration or verification run

Coolers

Cooling device sits on flange

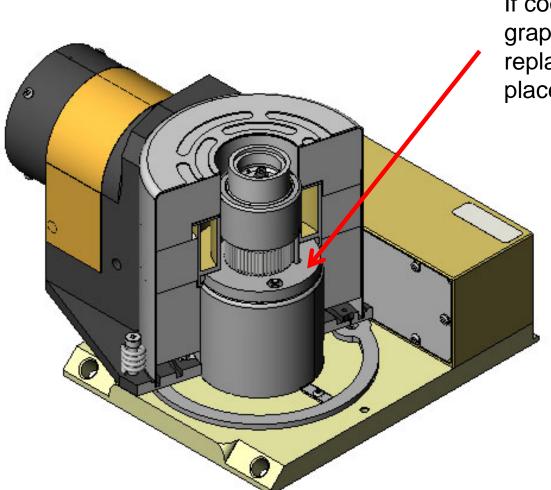
 Nickel rods transfer cooling to furnace / cell

 Furnace works with / against cooling to control the temperature





Cooler and Cell Cut-away



If cooler is removed, graphite disk needs to be replaced before cooler is placed back on the cell

RCS maintenance

- RCS heat exchanger needs to be cleaned on regular basis
- Switch off the RCS
- Remove side panel from RCS (without moving the RCS)







RCS maintenance

Use soft brush and vacuum cleaner to remove dust





Conditioning RCS/LNCS

- Done each time the RCS/LNCS cooling head is installed on the cell or if the system ran without purge gas
- Two step procedure

Drying system

- Run with cooler installed and turned off
- System is held isothermally at 75°C for 30 minutes

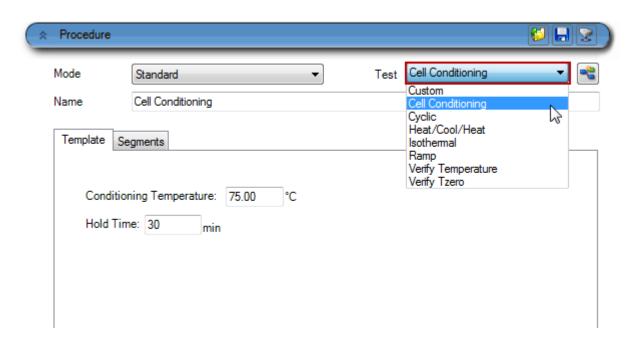
Stabilizing system

- Run with cooler on
- Baseline is cycled



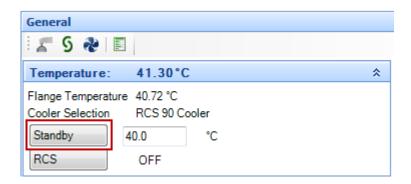
Conditioning RCS/LNCS

The Discovery DSC has a template for the conditioning



Optimizing Performance of Discovery DSC-RCS

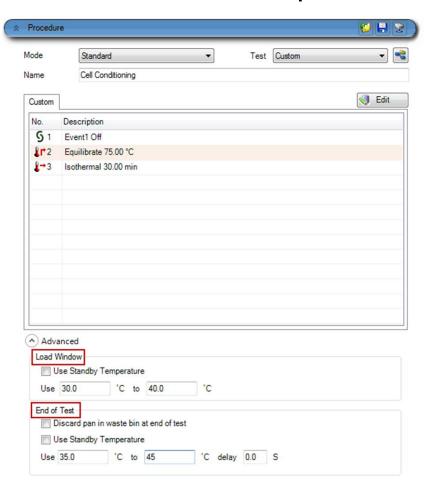
- Always make sure that cell is well above ambient between experiments!
- Leave standby temperature at 40° C





Optimizing Performance of Discovery DSC-RCS

Define a load and unload temperature



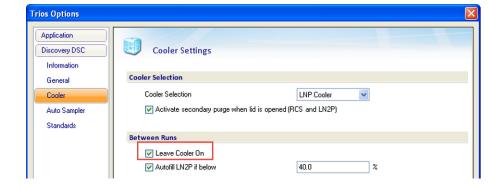


Optimizing Performance of Discovery DSC-RCS

Run in event mode (switch on front panel RCS)



- Leave RCS on between runs
- Requires constant supply of cell & base purge





Purge Gas

- Set pressure on regulator to 1.4 Bar (140kPa)
- Cell purge: 50 ml/min N2
- RCS base purge: 300-350 ml/min (mandatory)
- RCS base purge is controlled by the inlet pressure
- RCS base purge flow is a signal on the touch screen
- RCS secondary purge: 9 l/min (optional and active only when auto lid open for sample loading and / or during cell conditioning



Purge Gas Selection

- Gas 1 is always Nitrogen
- Gas 1 is used for cell and base purge
- Helium is used as Gas 2 for low temperature operation with LN2P (below -100°C)
- Cooling gas is Nitrogen or air if a FACS is used





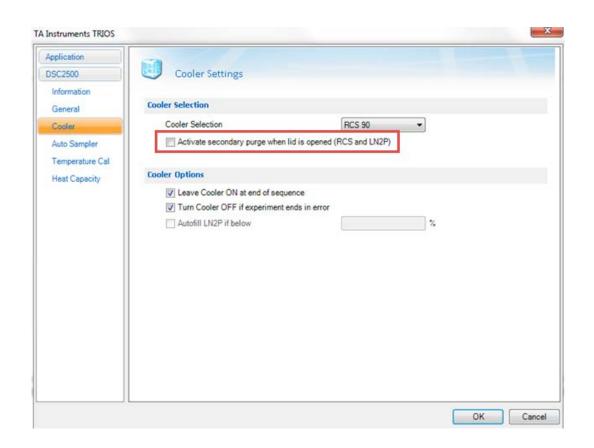
Secondary Purge

- Used in high humidity conditions
- Used if sample is loaded below room temperature
- When activated, purges area around heat exchanger & cell when the lid is open
- Helps to ensure moisture doesn't get in this area



Secondary Purge

Instrument Options



Troubleshooting

Topics:

- Notifications log
- Troubleshooting with online help
- Diagnostic signals
- Value of Diagnostic signals
- System health
- Video's
- Part numbers

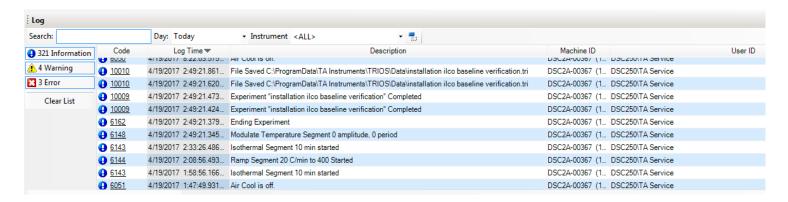


Notifications Manager

 The notifications log shows all messages from instrument and controller



Every step is logged

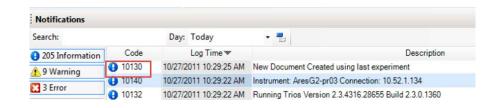


Save and send by email for support



Notifications Manager

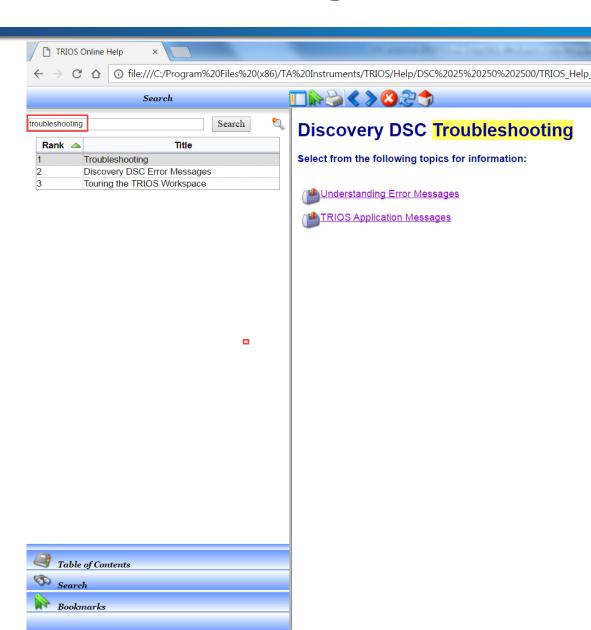
Click on "Code" for more information



Code explanation can also be found in the Online help

Troubleshooting with Online help

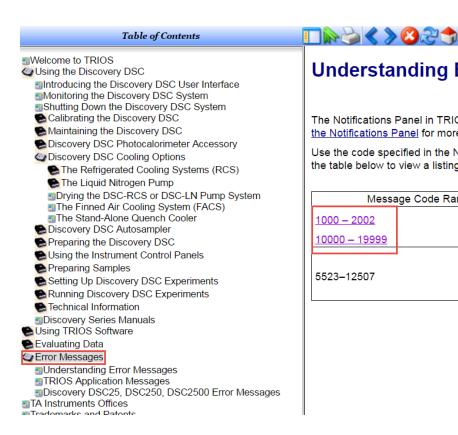






Troubleshooting with Online help

Online help shows detailed information per message



Understanding Error Messages in TRIOS

The Notifications Panel in TRIOS displays a current list of messages, along with the relative code the Notifications Panel for more information on understanding and using this panel.

Use the code specified in the Notifications Panel and the table below to determine the category of the table below to view a listing of the messages, as well as possible solutions to error messages.

Message Code Range		Message Category
1000 – 2002		TRIOS Application messages
<u> 10000 – 19999</u>		
5523–12507		Discovery DSC messages
		(includes Autosampler, PCA, and GDM errors)



Troubleshooting with Online help

TRIOS Application Error Messages

Refer to the table below for the list of TRIOS messages and possible solutions to error messages.

Message Code	Message Text	Explanation of Message	Solutions to Error
1000	Printing Information threw exception : <error></error>	Calibration tab printing failure.	Check the printer is connected, switched on, and ready to print.
1001	Sending Proc Content Changes threw exception : <error></error>	Unexpected software error.	Please contact TA Instruments for assistance.
1002	The instrument's address has not been specified. Please set the address in its Property window and try again.	No IP entered, connection to the instrument could not be effected.	Check that the correct IP has been specified in the connection dialog.
1003	Failed Connecting to the Instrument.	Initial communications with the instrument could not be established.	Check that the IP has been correctly specified, all network cables are connected and that the instrument is switched on.
1004	CloseLiveDataThread liveData null or filename empty	Software operation informational message.	Please inform TA Instruments.
1005	CloseLiveDataThread - live doc already closing	Software operation informational message.	Please inform TA Instruments.
1006	The instrument interface has been changed.	Software operation error message.	Please contact TA Instruments for assistance.
1007	Compiler Error: <error></error>	Software operation error message.	Please contact TA Instruments for assistance.
1008	LoadDataFile(Binary section): <filename> <error></error></filename>	Software operation error message.	Please contact TA Instruments for assistance.
1009	OnDragOver threw exception : <error></error>	Software operation informational message.	Please inform TA Instruments.
1010	OnDragDrop threw exception : <error></error>	Software operation informational message.	Please inform TA Instruments.



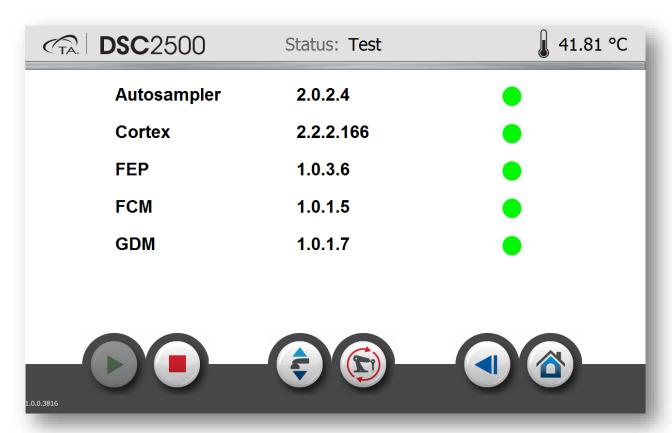
System health

This function checks the circuits boards



System Health (Benefit for Service)

Reboot if an indicator is red

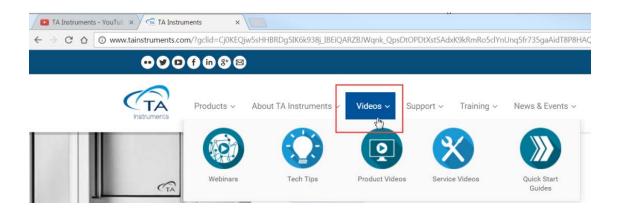


Contact TA Service if problem persists



Video's

Video's are available www.tainstruments.com



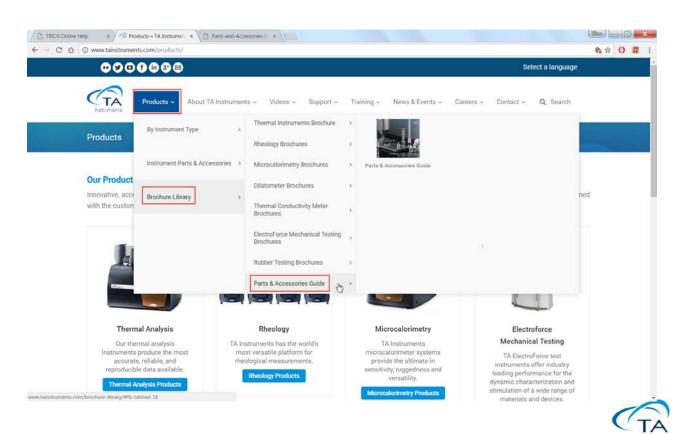
And on YouTube





Part numbers

- Part numbers in cover of accessory box
- Part numbers labeled on parts
- Part numbers on TA Instruments.com



TAINSTRUMENTS.COM

Part numbers

935012.001	Dual Liquid Light Guide for PCA (-50 °C to 80 °C operation)
	Note: Requires RCS for subambient use.
972734.901	Discovery PCA Trigger Adapter: an adapter to convert USB communication from the Discovery products to RS232 communication for the PCA. This adapter is required for the Discovery DSC to communicate with the PCA and should be added to PCA orders for the Discovery DSC.
935014.901	PCA/UV External Filter Adapter Accessory Kit (allows use of external filters with the PCA P/N: 935000.901)
935009.005	Blank Filter Holder (for the Omnicure S-2000 with external filter adapter)
935009.001	Filter, External, 320 - 480 nm (for the Omnicure S-2000 with external filter adapter)
935009.002	Filter, External, 320 - 390 nm (for the Omnicure S-2000 with external filter adapter)
935009.004	Filter, External, 400 - 500 nm (for the Omnicure S-2000 with external filter adapter)
935009.003	Filter, External, 365 nm (for the Omnicure S-2000 with external filter adapter)
935013.001	PCA Air Filter Kit (pkg. of 10)
935018.901	PCA Longpass Filter, 390 nm (pkg. of 2)
935019.901	PCA Longpass Filter, 490 nm (pkg. of 2)
935015.901	Filter, Neutral Density, 1 %
935016.901	Filter, Neutral Density, 10 %
935021.001	Lamp, 100 W, Mercury (for the Novacure 2100)
935021.002	Lamp, 200 W, Mercury (for the Omincure S-2000)
935051.901	Universal Optical Accessory Kit
200700.001	Radiometer (R2000) (for use with either PCA)
	Tzero DSC Sample Press and Die Sets/Sample Pans and Lids
901600.901	Tzero Sample Press and Die Sets Kit

901608.902	DSC Hermetic Die Set (WHITE color) (for use in the Tzero Sample Press)		
901608.903	DSC Tzero Die Set (BLACK color) (for use in the Tzero Sample Press)		
901608.904	DSC Tzero Hermetic Die Set (BLUE color) (for use in the Tzero Sample Press)		
901608.905	DSC High Volume Die Set (YELLOW color) (for use with the Tzero Sample Press and standard series high volume pans and lids)		
972800.901	Tzero Powder Sample Preparation Kit		
901670.901	Tzero Low-Mass Pans (pkg. of 100)		
901671.901	Tzero Lids (pkg. of 100)		
901683.901	Tzero Pans (pkg. of 100)		
901684.901	Tzero Hermetic Lids (pkg. of 100)		
901685.901	Tzero Hermetic Pinhole Lid (75 micron diameter pinhole) (pkg. of 50)		
901697.901	Tzero Alodined Pans (pkg. of 100)		
901698.901	Tzero Hermetic Alodined Lids (pkg. of 100)		
	Standard Series DSC Sample Pans and Lids Note: (*) Indicates the pans and lids work with the Tzero or Standard DSC Press.		
900760.901	Classic Aluminum Pans* (pkg. of 200) (higher sidewall compared to standard aluminum sample pans)		
900786.901	Standard Aluminum Pans* (pkg. of 200)		
900779.901	Standard Aluminum Lids* (pkg. of 200)		
900793.901	Standard Aluminum Hermetic Pans* (pkg. of 200)		
900794.901	Standard Aluminum Hermetic Lids* (pkg. of 200)		
900860.901	Hermetic Lids with Pinhole* (pkg. of 50)		
900796.901	Standard Coated Aluminum Hermetic Pans* (pkg. of 200)		
900790.901	Standard Coated Aluminum Hermetic Lids* (pkg. of 200)		



Thank You

The World Leader in Thermal Analysis, Rheology, and Microcalorimetry

