

# DSC Q Series 操作訓練

**2017.11.07**

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# DSC 機型介紹

DSC  
Q20/Q10



DSC  
Q200/Q100



DSC  
Q2000/Q1000



# DSC 開機順序

- 開電腦
- 開氮氣, 出口壓力15~20 psi
- 開DSC電源,約兩分鐘後,Ready燈亮或LCD螢幕出現TA圖樣
- 執行 Instrument Explorer
- 點選DSC圖示,開啟連線控制視窗
- 執行Control \ Go to standby temperature,讓DSC升溫到40度
- 開啟RCS電源開關,將下方開關撥到Manual.
- 約五六分鐘後,Cooling 綠燈亮,RCS開始冷卻降溫
- 執行開機烘乾CELL的除水氣程序
- Equilibrate at 300°C
- Isothermal for 30min
- 完成後便可正常做實驗

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg.dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 22.37°C

### Experiment

#### Standard Sequence

Sequence No. 12  
Run 1:

Summary Procedure Notes

Procedure Summary  
 Mode: Standard  
 Test: Custom

Sample Information  
 Sample Name: ITRI PET  
 Pan Type: Tzero Aluminum  
 Sample Size: 6.280 mg Pan No. 1 Ref. 1  
 Pan Mass: 0.000 mg (Sample) 0.000 mg (Reference)  
 Comments:

Data File Name: \\Ta-lab\ta\Data\DSC\LA WRENCE\TRIM\ITRI PET.001  
 Network Drive

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	22.37 °C
Heat Flow	0.173 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	21.25 °C
Heater Temperature	20.99 °C
Sample Purge Flow	50.01 mL/min

#	Running Segment Description
1	↕↑ Equilibrate at 0.00 °C
2	↕↘ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↕↘ Ramp 10.000 °C/min to 0.00 °C
5	↕↑ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↕↘ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

01 97.90 min. Append Apply Cancel Help

# DSC 操作介面說明

Summary | Procedure | Notes

Procedure Summary

Mode: Standard

Test: Custom

Sample Information

Sample Name: ITRI PET

Pan Type: Tzero Aluminum

Sample Size: 6.280 mg

Pan No.: 1 | Ref.: 1

Pan Mass: 0.000 mg (Sample) | 0.000 mg (Reference)

Comments:

Data File Name: W:\a-lab\ta\Data\DSC\LA WRENCE\TRIN\TRI PET.001

Network Drive

01 97.90 min. | Append | Apply | Cancel | Help

確認正確的  
實驗模式

樣品盤重量  
(Q2000)

路徑與檔名  
請勿用中文

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 22.60 °C

### Experiment

Standard Sequence

Sequence No. 12  
Run 1:

Summary Procedure Notes

Procedure Information

Test: Custom

Notes:

Method Name: Editor... Advanced... Post Test...

#	Segment Description
1	↕ Equilibrate at 0.00 °C
2	↘ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↘ Ramp 10.000 °C/min to 0.00 °C
5	↕ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↘ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	22.60 °C
Heat Flow	0.164 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	21.69 °C
Heater Temperature	21.23 °C
Sample Purge Flow	49.99 mL/min

#	Running Segment Description
1	↕ Equilibrate at 0.00 °C
2	↘ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↘ Ramp 10.000 °C/min to 0.00 °C
5	↕ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↘ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

### Advanced Parameters

Data Sampling Interval: 0.20 s/pt

Load Temperature Range 0.00 to 0.00 °C

OK Cancel Help

### Post Test Parameters

Method End Conditions

Unload Temperature Range 35.00 to 45.00 °C

Delay Time 0.00 min

Discard pan in waste bin

OK Cancel Help

### Method

Method Contents

Name: [ ]

#	Segment Description
1	↕ Equilibrate at 0.00 °C
2	↘ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↘ Ramp 10.000 °C/min to 0.00 °C
5	↕ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↘ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

OK Cancel Help

Segment list

- ↕ Jump
- ↕ Equilibrate
- ↕ Initial temperature
- ↘ Ramp
- ↕ Isothermal
- ↕ Step
- ↕ Increment temperature
- ↕ Repeat
- ↕ Repeat until
- ↕ Abort next segment on limit
- ↕ Sampling interval

開儲啓存

Temperature (°C)

01 97.90 min. Append Apply Cancel Help

Ready

Completed Standard Seg 0 in Run 1 15:41:22

TA

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Start  
Stop  
Reject  
Hold  
Resume  
Lid  
Store  
Gas  
Event  
Air Cool  
PCA Shutter  
Go To Standby Temp  
Autosampler  
LNCS  
Shutdown Instrument

Complete Temp: 22.95°C

Summary Procedure Notes

Notes

Operator: RICO

Extended Text

Mass Flow Control Settings

Sample: #1 - Nitrogen Flow Rate: 50 mL/min

Auto Analysis

Autoanalyze

Analysis Macro

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	22.95 °C
Heat Flow	0.158 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	22.11 °C
Heater Temperature	21.59 °C
Sample Purge Flow	49.98 mL/min

#	Running Segment Description
1	↑ Equilibrate at 0.00 °C
2	↘ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↗ Ramp 10.000 °C/min to 0.00 °C
5	↑ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↘ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

01 97.90 min Append Apply Cancel Help

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg.dsc]

Control Experimental Calibrate Tools View Window Help

Wizard... Ctrl + W  
Modify Running Method... Ctrl + M

Sequence File  
Sequence Run

Standard Sequence

Sequence No. 12  
Run 1

Notes

Operator: RICO

Extended Text

Mass Flow Control Settings

Sample: #1 - Nitrogen Flow Rate: 50 mL/min

Auto Analysis

Autoanalyze

Analysis Macro

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	23.21 °C
Heat Flow	0.155 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	22.36 °C
Heater Temperature	21.84 °C
Sample Purge Flow	49.98 mL/min

#	Running Segment Description
1	↑ Equilibrate at 0.00 °C
2	↓ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↑ Ramp 10.000 °C/min to 0.00 °C
5	↑ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↓ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

01 97.90 min. Append Apply Cancel Help



# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Calibration Wizard  
Analysis...  
AutoCalibration

Autosampler...  
Cell/Temperature Table...  
Indium Tracking  
Report...  
Mass Flow Control...  
Touchscreen

Experiment  
Standard Sequence

Sequence No. 12  
Run 1

Procedure Notes

RICO

Mass Flow Control Settings  
Sample #1 - Nitrogen Flow Rate 50 mL/min

Auto Analysis  
 Autoanalyze  
Analysis Macro

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	23.39 °C
Heat Flow	0.152 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	22.54 °C
Heater Temperature	22.02 °C
Sample Purge Flow	49.99 mL/min

#	Running Segment Description
1	↑ Equilibrate at 0.00 °C
2	↘ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↘ Ramp 10.000 °C/min to 0.00 °C
5	↑ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↘ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

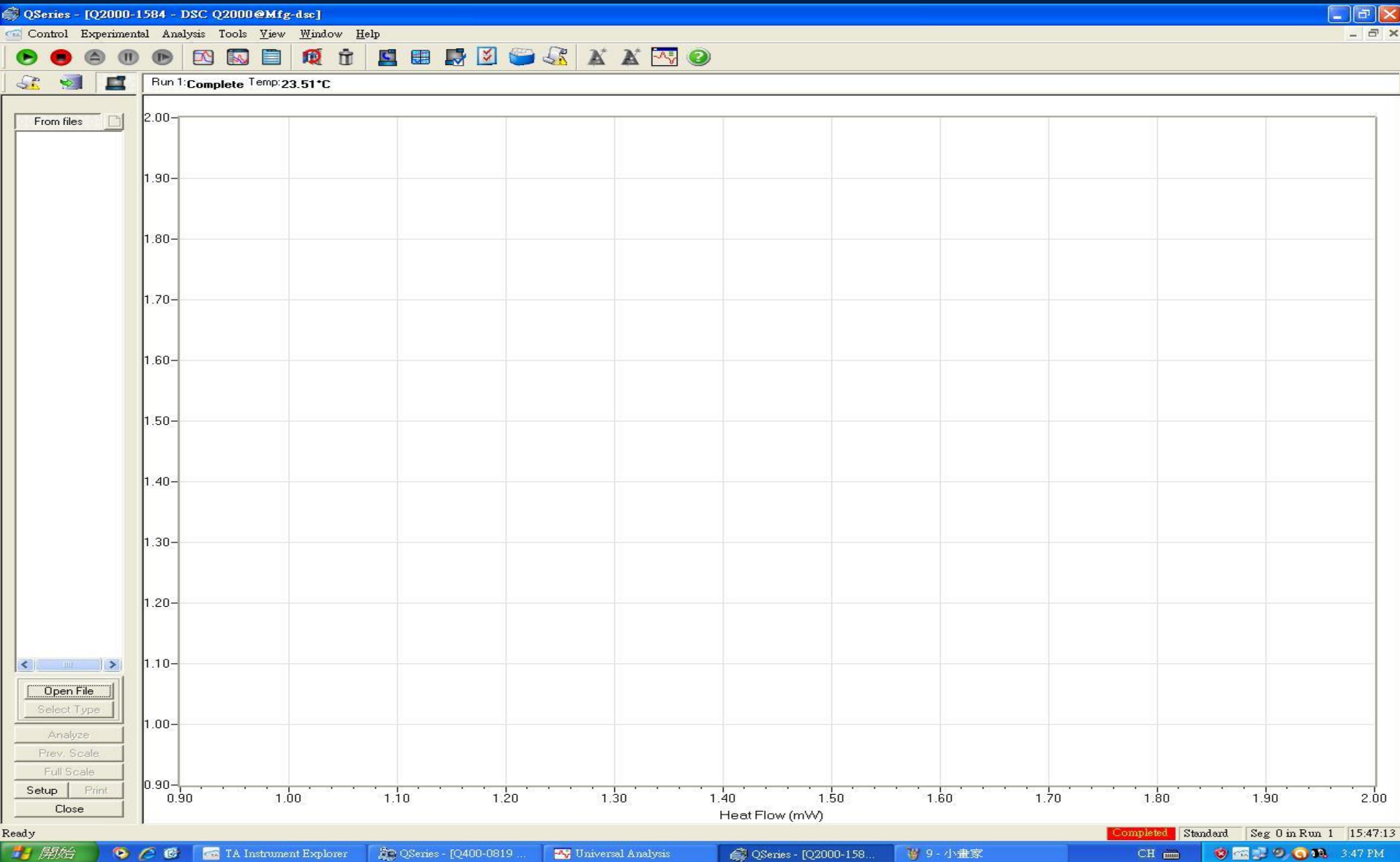
Heat Flow (mW)

Temperature (°C)

01 97.90 min

Append Apply Cancel Help

# DSC 操作介面說明



# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 23.61 °C

**Experiment**  
Standard Sequence

Sequence No. 12  
Run 1:

Notes  
Operator: RICO  
Extended Text

Mass Flow Control  
Sample

Auto Analysis  
 Autoanalyze  
Analysis Macro

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	23.61 °C
Heat Flow	0.149 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	22.75 °C
Heater Temperature	22.23 °C
Sample Purge Flow	50.01 ml/min

**Autosampler Calibration - Reset auto**

Calibration | Cycle Test | Motor Test

**Reset auto**

Current Values	Down / Up	Saved Values	Home
13600	Down / Up	N/A	<input type="checkbox"/>
7085	Left / Right	N/A	<input type="checkbox"/>
0	Extend / Retract	N/A	<input type="checkbox"/>
0	Grip/Release	N/A	<input type="checkbox"/>

Tray  Arm  Optical  Electrical

Start Stop Save Close Help

01 97.90 min. Append Apply Cancel Help

Heat Flow (mW)

Temperature (°C)

Completed Standard Seg 0 in Run 1 15:47:49

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 23.71 °C

Experiment  
Standard Sequence

Sequence No. 12  
Run 1:

Notes  
Operator: RICO  
Extended Text

Mass Flow Control Settings  
Sample: #1 - Nitrogen

Auto Analysis  
 Autoanalyze  
Analysis Macro

Signal Value

Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	23.71 °C
Heat Flow	0.137 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	22.85 °C
Heater Temperature	22.34 °C
	0.00 mL/min

Calibration Set: 0) Not set

Cell Constant (Tuesday, June 07, 2011, 16:57:12)

Cell Constant: 1.0320      Onset Slope: -114.105 mW/°C

Temperature Table

	Observed Temperature °C	Correct Temperature °C
1	155.47	156.60
2	417.75	419.53
3		
4		
5		

MDSC Cp Constant (Total): 1.071  
MDSC Cp Constant (Reversing): 1.100  
Standard Cp Constant (Direct): 1.000

Reset this Set    Reset All    OK    Close    Help

Heat Flow vs. Temperature (°C) graph

01 97.90 min    Append    Apply    Cancel    Help

Completed Standard Seg 0 in Run 1 15:48:34

實測值

理論值

# DSC 操作介面說明

The screenshot displays the QSeries software interface for a DSC instrument. The main window shows the 'Experiment' tab with 'Run 1: Complete' and a temperature of 24.67°C. A 'Calibration Report' dialog box is open, displaying the following information:

**Calibration Report**

**Last Run Calibration**

Kcell 1.0320  
Cpconst Standard: 1.000  
Calib -114.1049  
TempCal 2 pts 155.47 156.60, 417.75 419.53  
TzeroDt -90.0860 -0.0006 -1.1227  
TzeroDtz -90.0860 0.0097 3.9962  
InstCalFile Tzero: \\Ta-lab\ta\Data\DSC  
CALIBRATION\Z702\_RCS (90L\_08\_05\_2011 17\_  
34\_54\_TZR  
InstCalFile Baseline: \\Ta-lab\ta\Data\DSC  
\20110805.001  
InstCalFile Sapphire: \\Ta-lab\ta\Data\DSC  
\20110805.002  
InstCalDate Tzero 2011-08-05 Time 17:34:54  
TempRange -90.08 to 396.58 °C at 19.99 °C/min Heat  
Only  
AutoZero Delta T Offset 0.000 uV  
AutoZero Delta T0 Offset 0.000 uV  
MultiptCal 0  
Description: 0) Not set  
AutoCellConst Calibration Date 2011-06-07 16:57:12

**Multi Point Calibration Information**  
Multi Cal Index 0

Buttons: Print, Close, Help

The background interface includes a 'Signal' table with the following data:

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	24.67 °C
Heat Flow	0.137 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Sample Temperature	23.82 °C
Reference Temperature	23.30 °C
Flow	49.99 mL/min

At the bottom right, a graph shows Heat Flow (mW) vs Temperature (°C) with a scale from -10.0 to 0.0 mW and 0.90 to 2.00 °C.

# DSC 操作介面說明

The screenshot displays the QSeries software interface for a DSC instrument. The main window is titled "QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]". The interface is divided into several sections:

- Control Panel:** Includes buttons for "Control", "Experimental", "Calibrate", and "Tools". A "Run 1: Com" indicator is visible.
- Experiment Section:** Shows "Standard Sequence" and "Sequence No. 12". A "Run 1:" indicator is present.
- Menu:** A "Tools" menu is open, listing options such as "RT Plot", "User Preferences...", "Instrument Preferences...", "Data Transfer...", "File Utility...", "Instrument Setup...", "Register as the Master Controller", "Unregister Master Controller", "Print Setup...", "Controller License...", "Instrument License...", and "Instrument Diagnostics".
- Parameters:** A "Sample" dropdown is set to "#1 - Nitrogen" and "Flow Rate" is set to "50 mL/min".
- Auto Analysis:** Includes an "Autoanalyze" checkbox and an "Analysis Macro" dropdown.
- Running Segment Description Table:**

#	Running Segment Description
1	↕ Equilibrate at 0.00 °C
2	↗ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↘ Ramp 10.000 °C/min to 0.00 °C
5	↕ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↗ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3
- Graph:** A plot of "Heat Flow (mW)" versus "Temperature (°C)". The y-axis ranges from -10.0 to 10.0, and the x-axis ranges from 0.90 to 2.00. The plot area is currently blank.
- Status Bar:** Shows "01 97.90 min" and buttons for "Append", "Apply", "Cancel", and "Help".

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 23.99°C

Help Topics...  
Getting Started...  
About Q Advantage...

### Experiment

Standard Sequence

Sequence No. 12  
Run 1:

Summary Procedure Notes

Notes

Operator: RICO

Extended Text

Mass Flow Control Settings

Sample: #1 - Nitrogen Flow Rate: 50 mL/min

Auto Analysis

Autoanalyze

Analysis Macro

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	23.99 °C
Heat Flow	0.145 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	23.14 °C
Heater Temperature	22.62 °C
Sample Purge Flow	50.02 mL/min

#	Running Segment Description
1	↕ Equilibrate at 0.00 °C
2	↗ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↘ Ramp 10.000 °C/min to 0.00 °C
5	↕ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↗ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

01 97.90 min

Append Apply Cancel Help

# DSC 操作介面說明

DSC Online Help - Windows Internet Explorer

Contents Index Search - Search - GO Powered By RoboHelp

TA

## Welcome to DSC Online Help

Congratulations! You are using the latest software available from TA Instruments – Waters LLC for control of our Q Series™ analyzers



Introduction

Monitoring Data

Analyzing Data

Maintaining the Instrument

Troubleshooting

Preparing the Instrument

Calibrating the Instrument

Setting Up the Experiments

See also: [Trademarks and Patents](#)

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開始 TA Instrument Ex... QSeries - [Q400-0... Universal Analysis QSeries - [Q2000-... E - 小畫家 DSC Online Help - ... CH 3:50 PM

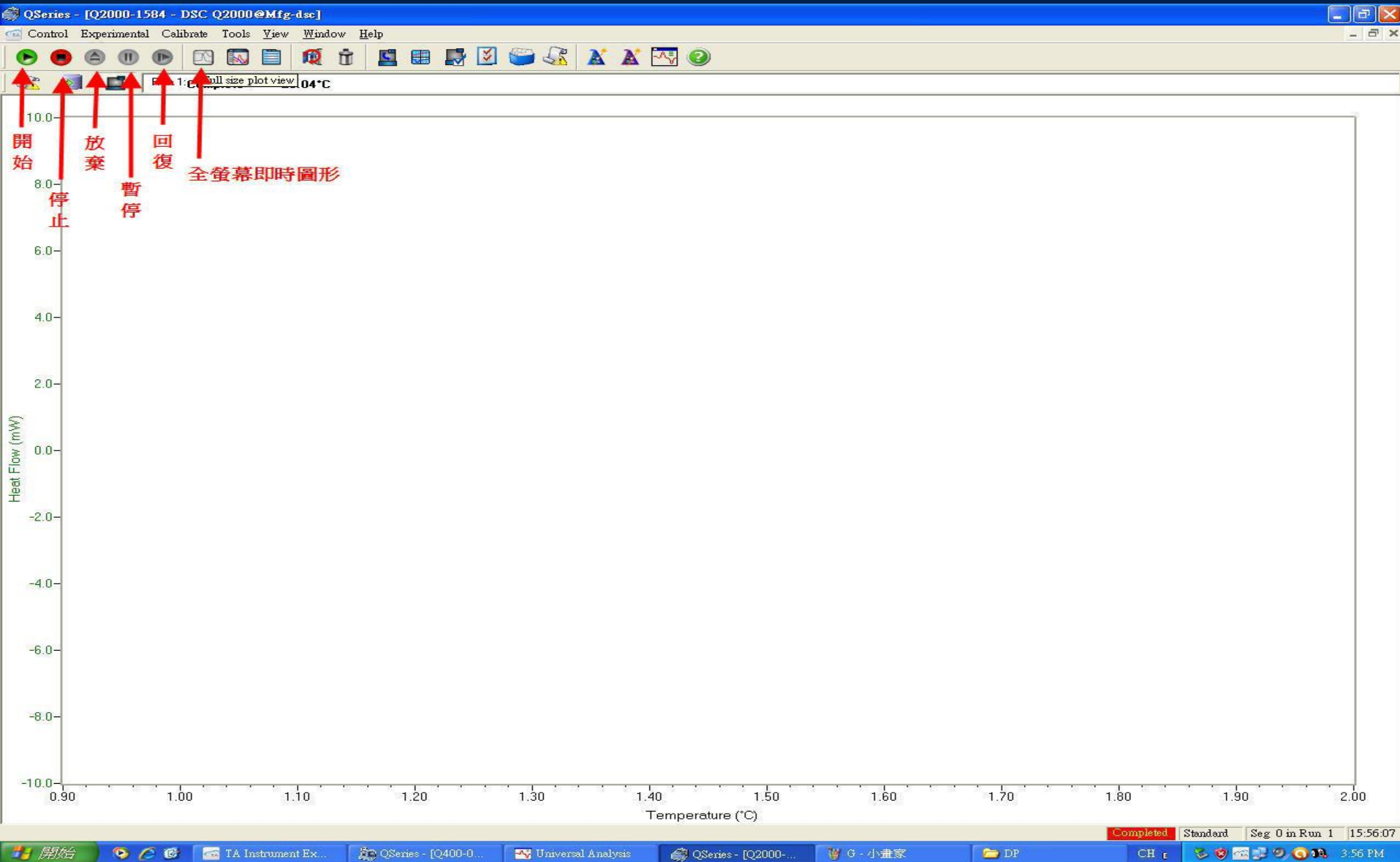




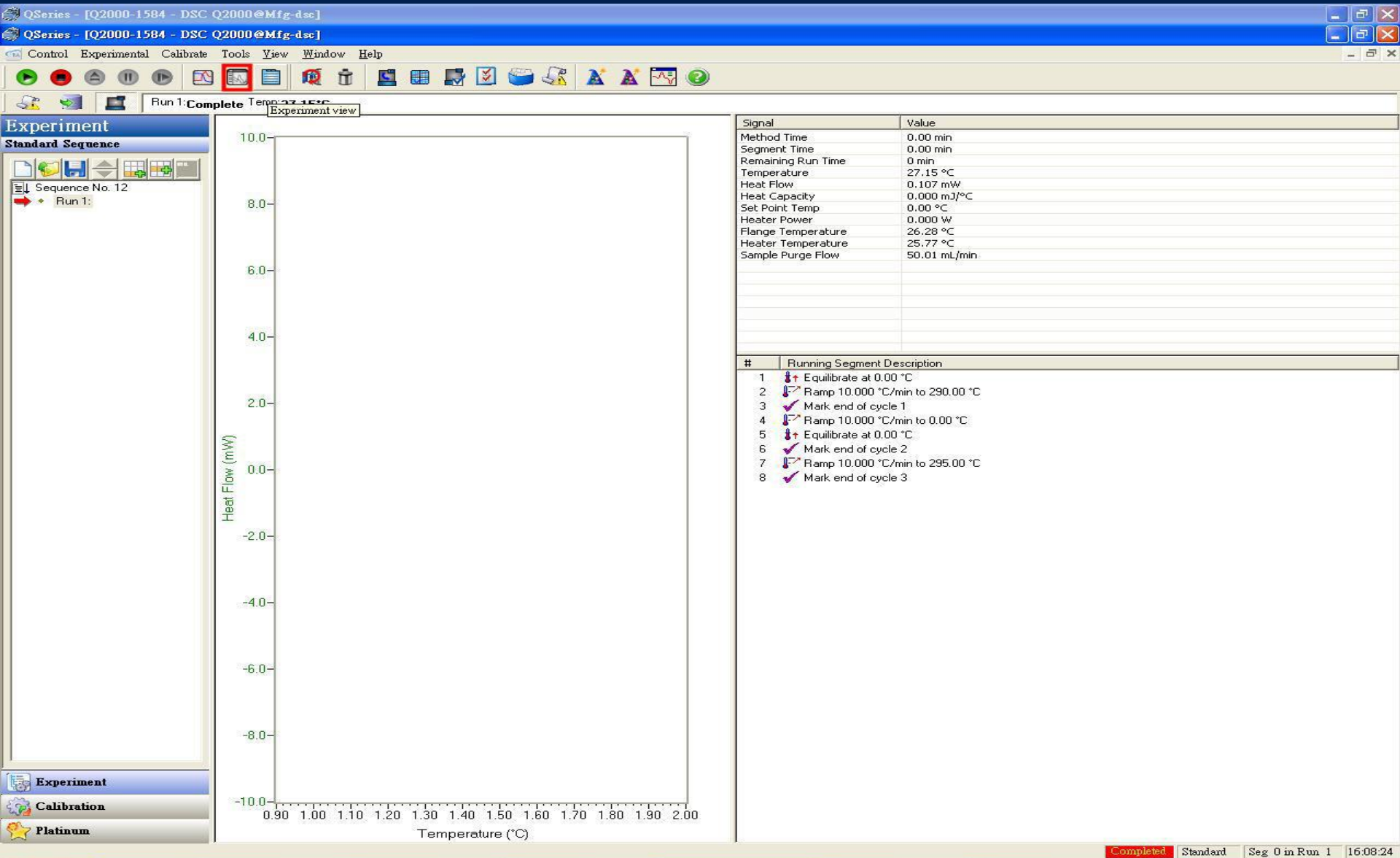
# DSC 操作介面說明



# DSC 操作介面說明



# DSC 操作介面說明



# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 22.37 °C

**Reset 自動進樣** **LNCS 充填**

Experiment  
Standard Sequence

Sequence No. 12  
Run 1:

Summary Procedure Notes

Sample Information  
Sample Name: ITRI PET  
Pan Type: Tzero Aluminum  
Sample Size: 6.280 mg Pan No. 1 Ref. 1  
 Pan Mass: 0.000 mg (Sample) 0.000 mg (Reference)  
Comments

Data File Name: WTa-lab\ta\Data\DSC\LA WRENCE\TRIN\ITRI PET.001  
 Network Drive

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	22.37 °C
Heat Flow	0.173 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	21.25 °C
Heater Temperature	20.99 °C
Sample Purge Flow	50.01 mL/min

#	Running Segment Description
1	↕ Equilibrate at 0.00 °C
2	↗ Ramp 10.000 °C/min to 290.00 °C
3	✓ Mark end of cycle 1
4	↘ Ramp 10.000 °C/min to 0.00 °C
5	↕ Equilibrate at 0.00 °C
6	✓ Mark end of cycle 2
7	↗ Ramp 10.000 °C/min to 295.00 °C
8	✓ Mark end of cycle 3

01 97.90 min Append Apply Cancel Help

Heat Flow (mW)

Temperature (°C)

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 27.36 °C

### Experiment Standard Sequence

Sequence No. 12  
Run 1:

### Notes

Operator: RICO  
Extended Text:

### Mass Flow Control Settings

Sample: #1 - Nitrogen Flow Rate: 50 mL/min


### Auto Analysis

Autoanalyze  
Analysis Macro:

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	27.36 °C
Heat Flow	0.105 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	26.48 °C
Heater Temperature	25.97 °C
Sample Purge Flow	50.00 mL/min

#	Running Segment Description
1	↑ Equilibrate at 0.00 °C

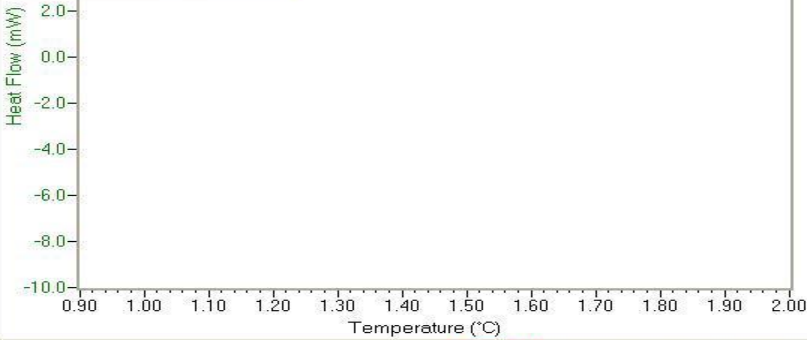
### Instrument Settings [Q2000-1584 - DSC Q2000@Mfg-dsc]



DSC Name: Q2000-1584  
Serial Number: 2000 - 1584  
 Remanufactured  
Location: Mfg-dsc  
IP Address: 192 . 168 . 171 . 113  
Net Mask: 255 . 255 . 255 . 0  
Default Gateway: 0 . 0 . 0 . 0  
A/S Serial No.: 2475

Apply New Settings

< Back Finish Cancel Help

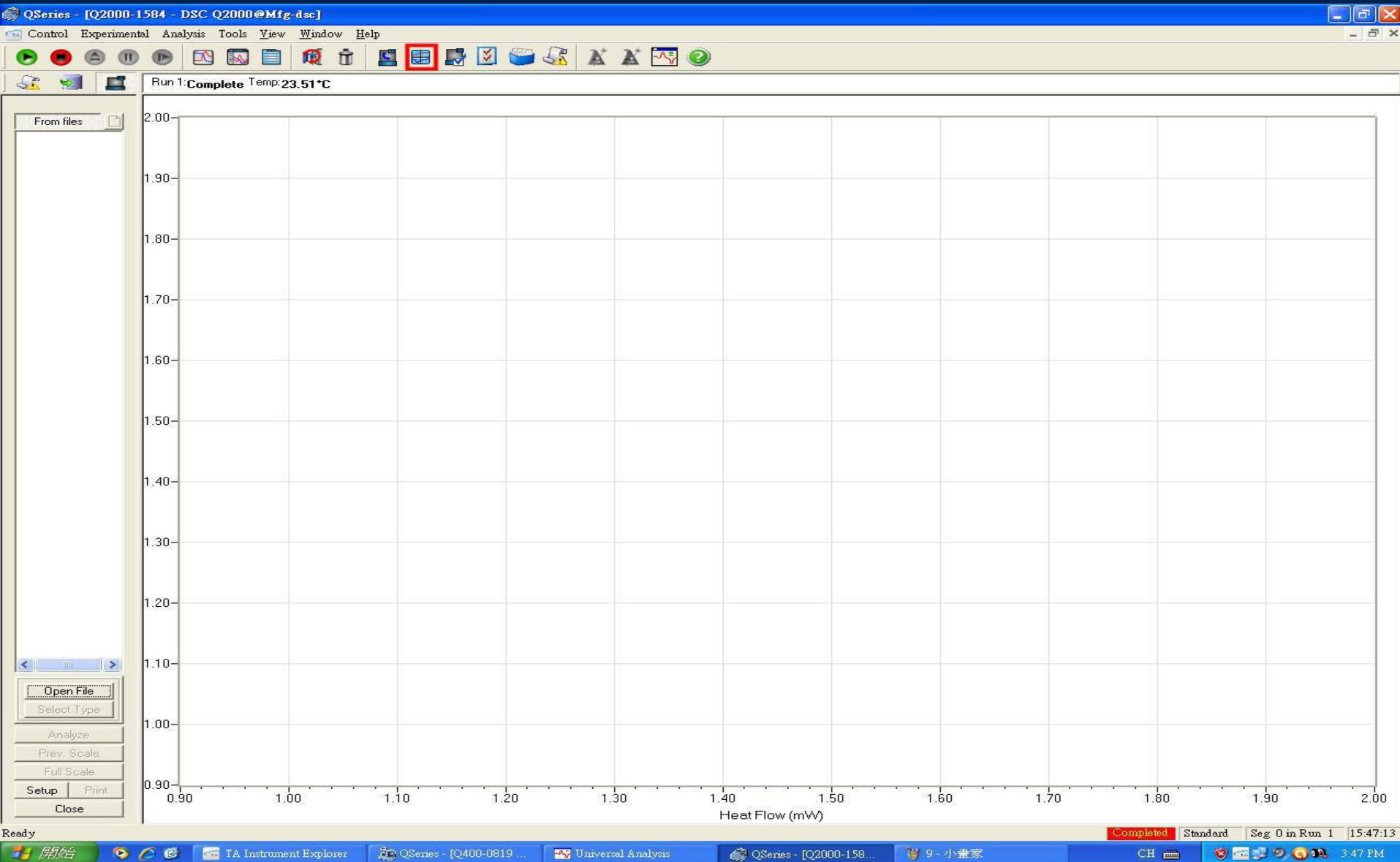


Heat Flow (mW) vs Temperature (°C)

01 97.90 min. Append Apply Cancel Help

Ready Completed Standard Seg 0 in Run 1 16:09:41

# DSC 操作介面說明



# DSC 操作介面說明

The screenshot displays the QSeries software interface for a DSC experiment. The main window shows the 'Experiment' tab with 'Standard Sequence' selected. The 'Run 1: Complete' status is shown at the top, along with the current temperature of 26.22°C. The 'Instrument Preferences' dialog box is open, showing settings for 'MFC Purge', 'LCD Signals', 'DSC', 'Cooler', and 'Auto Sampler'. The 'Gas #1' and 'Gas #2' are both set to 'Nitrogen'. A red arrow points to the 'Gas #1' dropdown menu. The 'Stop experiment when flow rate deviates from the set value' checkbox is checked. The 'Mass Flow Control Settings' section shows 'Sample #1 - Nitrogen' and 'Flow Rate 50 mL/min'. The 'Auto Analysis' section has 'Autoanalyze' unchecked. The 'Analysis Macro' field is empty. The 'Signal' table on the right lists various parameters and their values. The 'Running Segment Description' table shows the current segment is 'Equilibrate at 0.00 °C'. The 'Heat Flow (mW)' vs 'Temperature (°C)' plot is visible at the bottom right, showing a flat line at 0.00 mW. The 'Ready' status is shown at the bottom left. The Windows taskbar at the bottom shows the system tray with the time 4:02 PM and the date 16:02:49.

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 26.22°C

Experiment  
Standard Sequence

Sequence No. 12  
Run 1:

Summary Procedure Notes

Notes  
Operator: RICO  
Extended Text

Mass Flow Control Settings  
Sample #1 - Nitrogen Flow Rate 50 mL/min

Auto Analysis  
 Autoanalyze  
Analysis Macro

Instrument Preferences

MFC Purge LCD Signals DSC Cooler Auto Sampler

Gas #1 Nitrogen  
Gas #2 Nitrogen

Stop experiment when flow rate deviates from the set value

確定 Cancel Apply Help

Signal

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	26.22 °C
Heat Flow	0.117 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	25.37 °C
Heater Temperature	24.84 °C
Sample Purge Flow	49.99 mL/min

Running Segment Description

#	Running Segment Description
1	Equilibrate at 0.00 °C
2	min to 290.00 °C
3	min to 0.00 °C
4	min to 295.00 °C

Heat Flow (mW)

Temperature (°C)

01 97.90 min Append Apply Cancel Help

Ready

Completed Standard Seg 0 in Run 1 16:02:49

開始 TA Instrument Ex... QSeries - [Q400-0... Universal Analysis QSeries - [Q2000-... J - 小畫家 DP CH E 4:02 PM

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 26.28 °C

### Experiment

Standard Sequence

Sequence No. 12  
Run 1:

Notes

Operator: RICO

Extended Text

Mass Flow Control Settings

Sample: #1 - Nitrogen Flow Rate: 50 mL/min

Auto Analysis

Autoanalyze

Analysis Macro

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	26.28 °C
Heat Flow	0.116 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	25.42 °C
Heater Temperature	24.90 °C
Sample Purge Flow	49.99 mL/min

#	Running Segment Description
1	Equilibrate at 0.00 °C
2	Heat to 290.00 °C
3	Hold at 290.00 °C
4	Heat to 0.00 °C
5	Hold at 0.00 °C
6	Heat to 295.00 °C
7	Hold at 295.00 °C

### Instrument Preferences

MFC Purge | LCD Signals | **DSC** | Cooler | Auto Sampler

Transition Direction

Exotherm Down  **Exotherm Up**

Heat Flow Selection: **Heat Flow T4P (mW)**

Standby Temperature: 40.00 °C

Data Mode: Normal Gain

PC & Operation: Single

Enable Diagnostic Signals  AutoLid II installed

Enable Multiple Calibration Sets

確定 Cancel Apply Help

01 97.90 min Append Apply Cancel Help

Heat Flow (mW)

Temperature (°C)



# DSC 操作介面說明

The screenshot displays the QSeries software interface for a DSC instrument. The main window shows the 'Experiment' tab with 'Standard Sequence' selected. The 'Run 1: Complete' status is shown at the top, with a temperature of 26.34 °C. The 'Instrument Preferences' dialog box is open, showing the 'Cooler' tab. The 'Cooler Selection' dropdown is set to 'RCS (90)'. The 'Activate secondary purge when lid is opened' checkbox is checked. The 'Between Runs' section has 'Leave RCS On' and 'Leave LNCS On' checked, and 'Autofill LNCS if below' set to 40%.

**Signal**

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	26.34 °C
Heat Flow	0.115 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	25.48 °C
Heater Temperature	24.96 °C
Sample Purge Flow	50.01 mL/min

**Running Segment Description**

#	Running Segment Description
1	Equilibrate at 0.00 °C
2	Heat to 290.00 °C
3	Hold at 290.00 °C
4	Cool to 0.00 °C
5	Hold at 0.00 °C
6	Heat to 295.00 °C
7	Hold at 295.00 °C

**Heat Flow (mW) vs Temperature (°C)**

Temperature (°C)	Heat Flow (mW)
0.90	0.00
1.00	0.00
1.10	0.00
1.20	0.00
1.30	0.00
1.40	0.00
1.50	0.00
1.60	0.00
1.70	0.00
1.80	0.00
1.90	0.00
2.00	0.00

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 26.45 °C

### Experiment

Standard Sequence

Sequence No. 12  
Run 1:

Summary Procedure Notes

Notes

Operator: RICO

Extended Text

Mass Flow Control Settings

Sample: #1 - Nitrogen Flow Rate: 50 mL/min

Auto Analysis

Autoanalyze

Analysis Macro

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	26.45 °C
Heat Flow	0.114 mW
Heat Capacity	0.000 mJ/°C
Set Point Temp	0.00 °C
Heater Power	0.000 W
Flange Temperature	25.60 °C
Heater Temperature	25.07 °C
Sample Purge Flow	50.01 mL/min

#	Running Segment Description
1	↑ Equilibrate at 0.00 °C
2	↘ Ramp 10.000 °C/min to 290.00 °C
3	↘ Ramp 10.000 °C/min to 0.00 °C
4	↘ Ramp 10.000 °C/min to 295.00 °C
5	↘ Ramp 10.000 °C/min to 0.00 °C

### Edit User Preferences

General | Data | Procedure | Sequence | Status

Background: TA Instruments, Inc.

Display Confirmation Message On

- Stop run
- Reject run
- Delete sequence run(s)
- Prompt to save run sequence
- Use WinUA for plot view

Buttons: 確定, Cancel, Apply, Help

01 97.90 min. Append Apply Cancel Help

Heat Flow (mW) vs Temperature (°C)

Y-axis: -10.0 to 2.0 mW  
X-axis: 0.90 to 2.00 °C

Completed Standard Seg 0 in Run 1 16:04:10

Ready

Windows Taskbar: 開始, TA Instrument Ex..., QSeries - [Q400-0..., Universal Analysis, QSeries - [Q2000-..., M - 小畫家, DP, CH E, 4:04 PM

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 26.62°C

### Experiment

Standard Sequence

Sequence No. 12  
Run 1:

Level:	Critical	Warning	Status	Month:	Sequence/Run	All	All
Category:	Status	Calibration	Autosampler	Data File Acceptor	Miscellaneous	Power Supply	Taring
Log Time	Run	Category	Description				
29-Feb-2012 18:26:48	00-00	Miscellaneous	Shutdown instrument. (911)				
29-Feb-2012 18:18:32	00-00	Power Supply	Power supply interface error. (412)				
29-Feb-2012 17:55:19	00-00	DFA	\\T a-lab\ta\data\DSC\LAWRENCE\DSC-drying-022912.001				
29-Feb-2012 17:55:19	00-00	DFA	Run 1 completed and saved.				
29-Feb-2012 17:21:19	04-00	Run Status	Run 1 started.				
29-Feb-2012 17:21:16	04-00	Calibrate	Multi-point Cal set (0) Activated				
29-Feb-2012 17:21:16	00-00	Run Status	Air cool turned off. (837)				
29-Feb-2012 17:19:45	00-00	Run Status	Go to standby temperature enabled. (1025)				
29-Feb-2012 17:17:42	00-00	Autosampler	Autosampler reset complete.				
29-Feb-2012 17:17:42	00-00	Miscellaneous	License file options loaded: MDSC(R)				
29-Feb-2012 17:17:20	00-00	Miscellaneous	Instrument rebooted.				
29-Feb-2012 17:17:17	00-00	Calibrate	Tzero sapphire calibration filename: \\T a-lab\ta\data\DSC\20110805.002 (872)				
29-Feb-2012 17:17:17	00-00	Calibrate	Tzero empty cell calibration filename: \\T a-lab\ta\data\DSC\20110805.001 (871)				
29-Feb-2012 17:17:17	00-00	Calibrate	Tzero coefficient calibration filename: \\T a-lab\ta\data\DSC\CALIBRATION\2702_RCS (90)_08_05_2011 17_34_54.TZR (870)				
29-Feb-2012 17:17:17	00-00	Miscellaneous	Contact established with cell 2702.				
29-Feb-2012 17:17:17	00-00	Autosampler	Cell contact established.				
29-Feb-2012 17:17:17	00-00	Calibrate	Multi-point Cal set (0) Activated				
29-Feb-2012 17:17:06	00-00	Miscellaneous	License file options loaded: MDSC(R)				
28-Feb-2012 21:13:23	00-00	Miscellaneous	Shutdown instrument. (911)				
28-Feb-2012 21:10:31	00-00	Autosampler	Reference pan #1 unloaded				
28-Feb-2012 21:09:56	00-00	Autosampler	Sample pan #3 unloaded				
28-Feb-2012 21:09:45	00-00	Power Supply	Power supply interface error. (412)				
28-Feb-2012 21:05:33	00-00	DFA	\\T a-lab\ta\data\DSC\LAWRENCE\DSC-CPC-26-022812.001				
28-Feb-2012 21:05:33	00-00	DFA	Run 3 completed and saved.				
28-Feb-2012 20:46:24	03-00	Run Status	Run 3 started.				
28-Feb-2012 20:46:21	03-00	Run Status	Air cool turned off. (837)				
28-Feb-2012 20:46:11	03-00	Run Status	Lid closed. (845)				
28-Feb-2012 20:46:11	03-00	Autosampler	Sample pan #3 loaded				
28-Feb-2012 20:46:05	03-00	Power Supply	Power supply interface error. (412)				
28-Feb-2012 20:46:04	03-00	Power Supply	Power supply interface error. (412)				
28-Feb-2012 20:45:52	03-00	Power Supply	Power supply interface error. (412)				
28-Feb-2012 20:45:47	03-00	Run Status	Air cool turned on. (838)				
28-Feb-2012 20:45:47	03-00	Calibrate	Multi-point Cal set (0) Activated				
28-Feb-2012 20:45:47	03-00	Run Status	Air cool turned off. (837)				
28-Feb-2012 20:45:47	03-00	Autosampler	Sample pan #2 unloaded				
28-Feb-2012 20:45:28	03-00	Power Supply	Power supply interface error. (412)				
28-Feb-2012 20:41:22	03-00	DFA	\\T a-lab\ta\data\DSC\LAWRENCE\DSC-CPC-23-022812.001				
28-Feb-2012 20:41:22	03-00	DFA	Run 2 completed and saved.				
28-Feb-2012 20:22:13	03-00	Run Status	Run 2 started.				
28-Feb-2012 20:22:10	03-00	Run Status	Air cool turned off. (837)				
28-Feb-2012 20:22:01	03-00	Run Status	Lid closed. (845)				
28-Feb-2012 20:22:01	03-00	Autosampler	Sample pan #2 loaded.				

Ta-lab hosts 51 of 179 entries

Print... Clear Log... Save View Html

Error Help... Help Close

Ready

Completed Standard Seg 0 in Run 1 16:05:13

開始 TA Instrument Ex... QSeries - [Q400-0... Universal Analysis QSeries - [Q2000-... N - 小畫家 DP CH E 4:05 PM

# DSC 操作介面說明

Advantage Instrument Messages - Mozilla Firefox

檔案 (F) 編輯 (E) 檢視 (V) 歷史 (S) 書籤 (B) 工具 (T) 說明 (H)

file:///C:/Program Files/TA Instruments/Thermal Advantage/Qseries/Help/QInstErr/QInstErr.htm

Contents Index Search

## Message 67

### Flange over temperature. Run terminated.

**Problem:**

The flange temperature is outside the instrument's operating limit. This can occur under the following circumstances:

- If a sample starts to burn during the experiment and raises the temperature beyond the limit.
- If the cooler is not on, or hasn't been on long enough to equilibrate (e.g., flange temperature is too high).
- If the cooler is not operating correctly (e.g., has run out of liquid nitrogen).

**Solution:**

1. Try running the sample in an inert atmosphere and/or to a lower temperature.
2. Turn the cooler on at least 20 minutes prior to starting the run.
3. Check the operation of the cooler.

Call TA Instruments for service, if the problem persists.

General Instrument Messages

- ? What are General Instrument Mess
- ? Message 10
- ? Message 21
- ? Message 60
- ? Message 62
- ? Message 63
- ? Message 66
- ? Message 67
- ? Message 73
- ? Message 75
- ? Message 78
- ? Message 80
- ? Message 81
- ? Message 91
- ? Message 96
- ? Message 97
- ? Message 98
- ? Message 99
- ? Message 601
- ? Message 603
- ? Message 605
- ? Message 606
- ? Message 674
- ? Message 675
- ? Message 676
- ? Message 677
- ? Message 678
- ? Message 679
- ? Message 680
- ? Message 681
- ? Message 682
- ? Message 684

# DSC 操作介面說明

QSeries - [Q2000-1584 - DSC Q2000@Mfg-dsc]

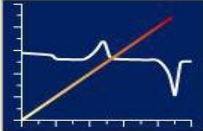
Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 26.82°C

### Experiment

#### Standard Sequence

Sequence No. 12  
Run 1:



This wizard helps you set up and start typical DSC experiments. The types of experiments you can run depend on the Q Series DSC and cooling accessory you are using. Options which do not apply for your DSC system (DSC and cooling accessory) are automatically "grayed out" so they cannot be inadvertently selected.

Conventional DSC  
These experiments measure heat flow into or out of a material as a function of temperature or time.

Modulated DSC ® (MDSC ®)  
These experiments provide the same heat flow information as conventional DSC, but also separate that heat flow into heat capacity related (reversing) and kinetic (nonreversing) components.

Heat Flow Selection: Heat Flow T4P (mW)

Cooler Type: RCS (90) (Applies to all runs)

Next > Cancel Help

Ready

Completed Standard Seg 0 in Run 1 16:06:21

# DSC 校正種類

TA instruments 提供三種不同的Q系列DSC：

- Q20：僅提供傳統之單項熱流計算，我們稱之為T1熱流式。
- Q200：不僅提供單項的T1熱流式，還可延伸拓展計算入加熱爐的熱阻及熱容四項熱流影響，我們稱之為T4熱流式。
- Q2000：提供了T1及T4熱流式之外，並且還計算入樣品盤帶來之影響，我們稱之為T4P熱流式。
- 校正週期：建議每季校驗一次，最長不超過半年，但仍依照您的使用頻率與承擔風險而定。

# DSCQ20 T1校正操作步驟

## T1模式所需執行的校正步驟

T1模式的校正適用於DSC Q20，所需進行的步驟有：

- 基線校正「Baseline」。
- 溫度校正「Indium」。

## 基線校正 (Baseline)

DSC爐子是由純銀物質組成，所提供的熱傳導誤差相當微小。為了消除樣品及參考品平台端的熱傳導誤差，我們建議您進行基線校正。

不論你現在所使用的儀器模式或是爐子形式，DSC基線校正實驗都會以類似的方式執行。依照下列步驟會收集及分析校正資料。

T1熱流式基線校正操作步驟<sup>👉</sup>如下簡介：

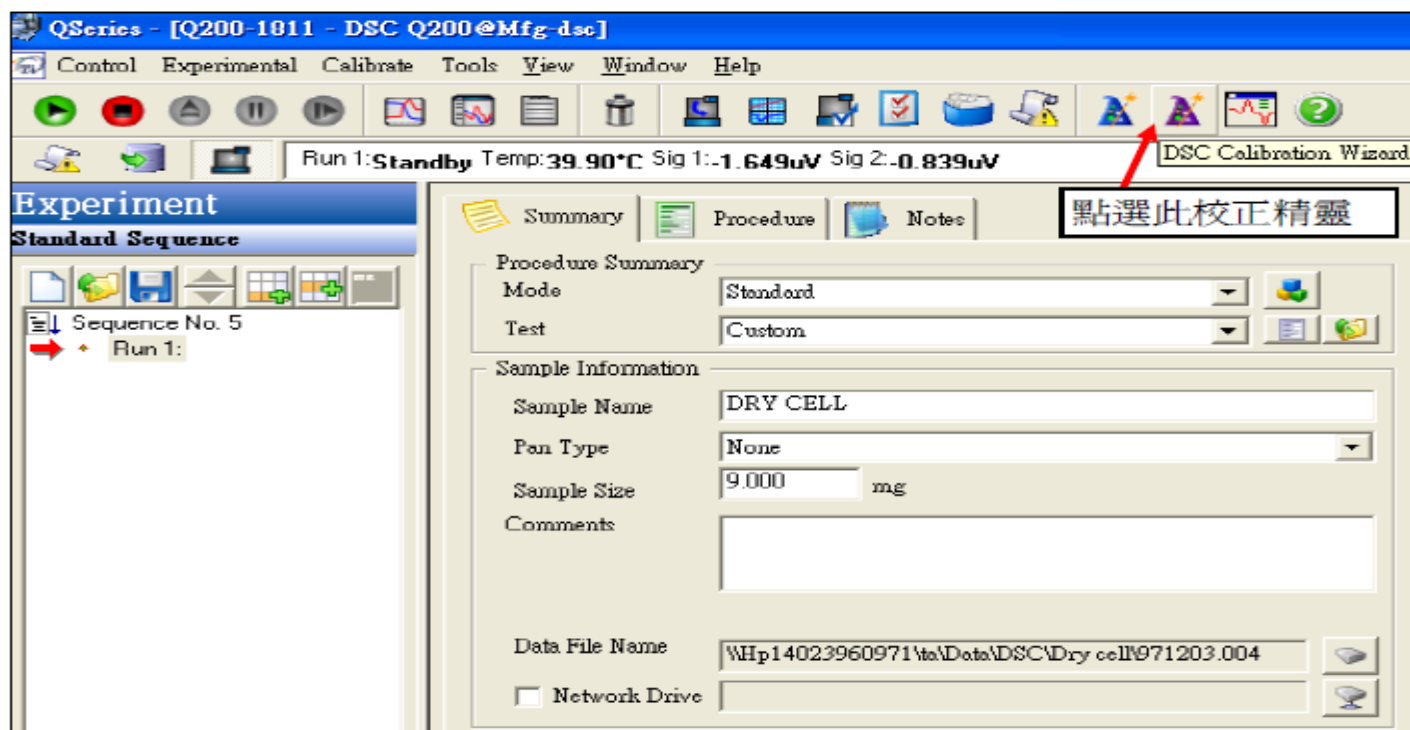
步驟如下：

# DSCQ20 T1熱流基線校正操作步驟

1. 確定Purge gas、Air Cool氣體管線已開啟與冷卻配件(如：RCS) 開機妥善。
2. 開啟DSC儀器。
3. 進入控制軟體TA Instruments，選取校正模式或點選控制頁面上的校正精靈按鈕



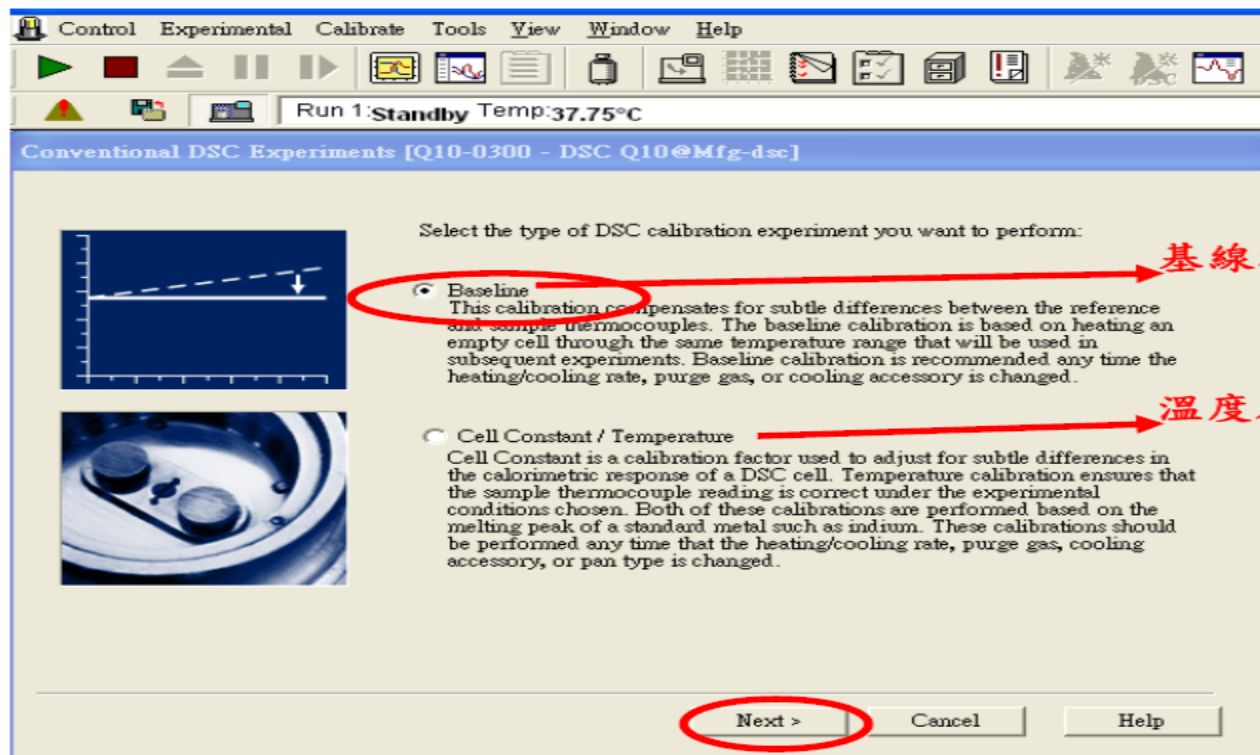
。如下圖所示。





# DSCQ20 T1熱流基線校正操作步驟

4. 根據您的儀器模式來選擇您所需的校正模式「Q20/Heat Flow T1」  
「Q200 or Q2000/Heat Flow T4 or T4P」及對應的冷卻配件「RCS或LNCS」。  
在此選項中亦可選擇您需要的功能。  
在T1熱流式基線校正內控制軟體進入校正精靈後會顯示下列選項讓您選擇；  
首先先選取第一項：**基線校正「Baseline」**。



基線校正選項

溫度及爐子常數校正

# DSCQ20 T1熱流基線校正操作步驟

- 選項一：基線校正「[Baseline calibration](#)」。
- 選項二：選擇爐子常數及溫度校正「[Cell constant calibration](#)」。

校正的週期性取決於客戶對本身實驗的數據可靠度，當您發現實驗數據異常時，我們建議您重新校正您的儀器以釐清問題來源。

通常校正建議每季校正一次，僅在校正完成後軟體未記錄校正檔案時才建議您重新載入校正檔案。

5. 編輯您的校正參數及範圍。

- 編輯溫度範圍。

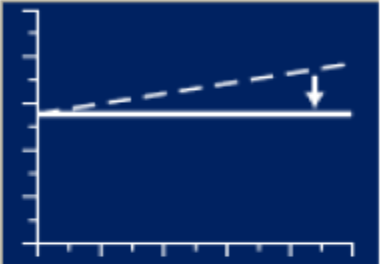
[T1熱流式基線校正](#)的溫度範圍建議：「 $30^{\circ}\text{C} \sim 300^{\circ}\text{C}$ 」。

升溫速率為「 $10^{\circ}\text{C} / \text{min}$ 」。

設定完成後按下「Next」鍵，跳至下一個視窗檢視校正所設定的參數。

# DSCQ20 T1熱流基線校正操作步驟

Experimental Parameters: Baseline [Q10-0300 - DSC Q10@Mfg-dsc]



Baseline calibration involves heating the cell through the entire temperature range using the same ramp rate and purge gas that will be used for subsequent experiments. Typically, an empty cell (e.g., no pans) is used for this calibration.

Enter the desired parameters:

Start temperature  Use current  °C

Heating rate:  °C/min

Final temperature:  °C

1. 設定起始溫度  
2. 設定升溫速率  
3. 設定結束溫度

4. 設定完成之後，按下 Next 進入下一步驟繼續設定

# DSCQ20 T1熱流基線校正操作步驟



Sample Information

Sample Name

Sample Size  mg **不可輸入重量**

Comments


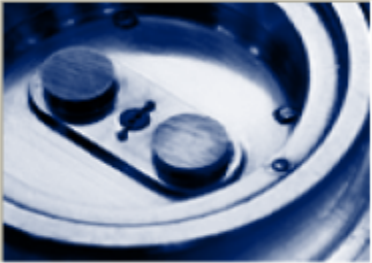
Data File  

Start Remotely

**設定存檔路徑  
路徑與檔案名稱  
不可使用中文**

# DSCQ20 T1熱流式線校正操作步驟

Sample Information [Q10-0300 - DSC Q10@Mfg-dsc]

Notes **1.輸入操作者姓名**

Operator

Pan Type

Extended Text

Mass Flow Control Settings

Sample  Flow Rate  mL/min

**2.設定氣體流量**

< Back **Next >** Cancel Help



**注意：爐內不應放置任何物品。**

# DSCQ20 T1熱流基線校正操作步驟

6. 再次確認DSC爐內不放置任何物品後，蓋上爐蓋，按下「Next」之後，即可進行Baseline校正。

- 確認氣體安裝妥當。
- 確認冷卻配件妥當。
- 已放置好樣品。

按下「Start Run」，即進行Baseline校正。

Purge Gas

✓ Be sure that your purge gas is connected and properly regulated. Purge Gas is recommended for all DSC experiments.

Cooling Accessory

✓ The Q Series DSC cells generally require a cooling accessory to be connected regardless of the type of experiment being run. The QCA is the only exception. It is removed before initiating heating ramp experiments.

Loading the Sample

✓ Position the sample pan into the cell. [Typically, an empty cell (e.g., no pans) is used for this calibration.] Position a reference pan, of the same type, into the cell (back position). Cover the cell with the appropriate lids.

Append Run Start Run Finish Cancel Help

# DSCQ20 T1熱流基線校正操作步驟

7. 按下「Start Run」，開始Baseline校正後會自行跳到此視窗。「如下圖」。

- 在控制軟體右下方可見Baseline的校正訊號。

5. 當Baseline校正結束後，需執行校正分析軟體。  
如右圖上方所示。

按下校正分析軟體快捷鍵

量測到的基線訊號

Signal	Value
Method Time	0.00 min
Segment Time	0.00 min
Remaining Run Time	0 min
Temperature	40.81 °C
Delta T	-60.182 uV
Heat Flow	-21.489 mW
Set Point Temp	40.00 °C
Heater Power	41.530 mW
Flange Temperature	-73.88 °C
Heater Temperature	39.57 °C
Sample Purge Flow	43.99 mL/min
Diagnostic Sig 1	-60.182 uV
Diagnostic Sig 2	-53.215 uV
Diagnostic Sig 3	39.95 °C

Running Segment Description

- 1 Sampling Interval 0.10 s/pt
- 2 Ramp 10.000 °C/min to 250.00 °C

01 28.00 min    Apply    Apply    Cancel    Help

Ready    Calibration | Seg 0 in Run 1 | 14:00:34

# DSCQ20 T1熱流基線校正操作步驟

- 進入校正分析模式後，選取欲分析的檔案。「如下圖所示」。

The screenshot displays the QSeries software interface for a DSC Q10. The main window shows a graph of Heat Flow (mW) versus temperature, with a 'Run 1: Standby Temp: 40.85°C' label. An 'Open Calibration File' dialog box is open, showing a file list with the following items:

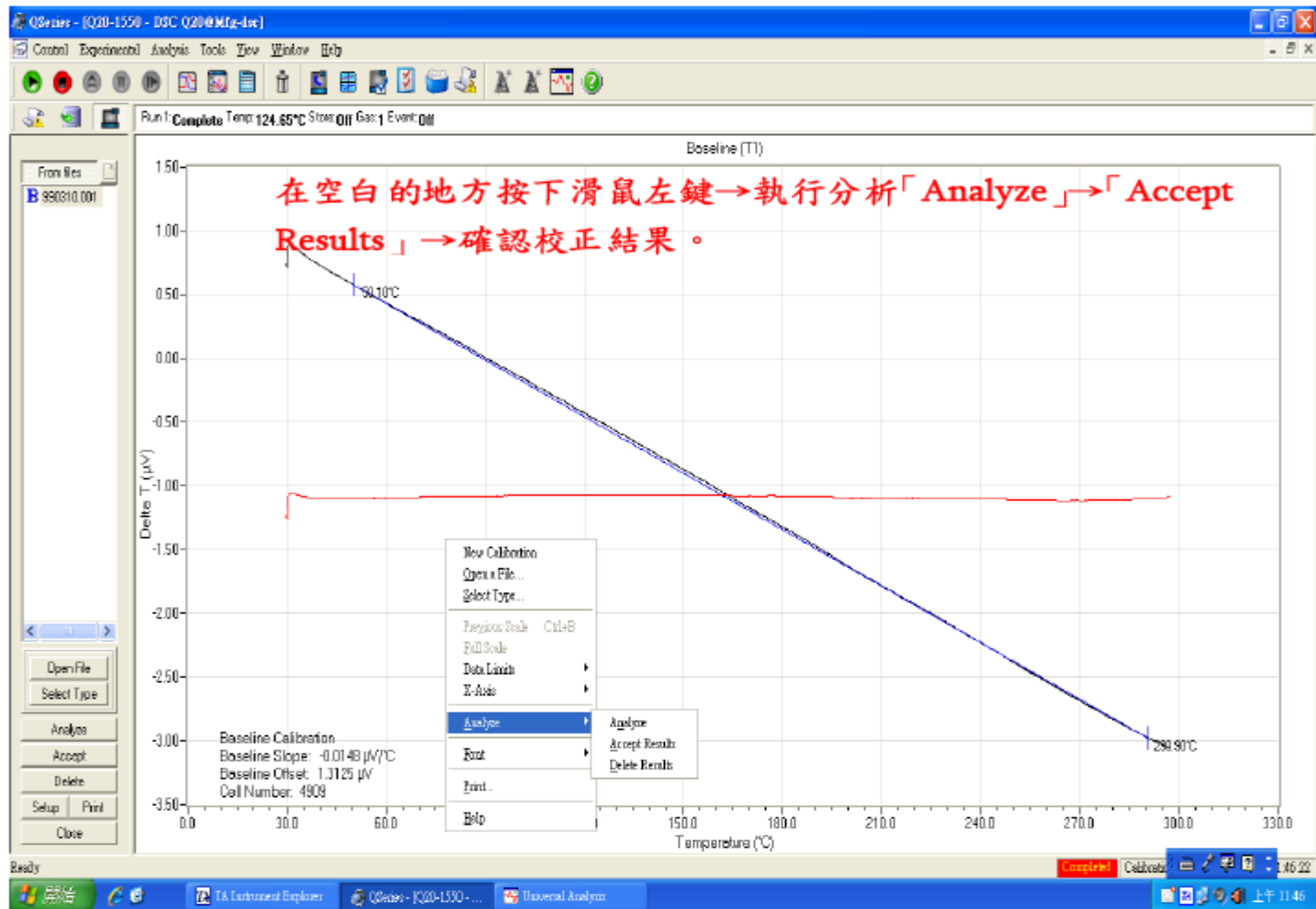
- Baseline 980219
- Dry Cell condition 980219
- Replication

The 'Open File' button in the dialog is highlighted with a red box. A red text annotation '選取欲分析的檔案' (Select the file to be analyzed) is placed over the file list. The software interface includes a menu bar (Control, Experimental, Analysis, Tools, View, Window, Help) and a toolbar with various icons. The status bar at the bottom shows 'Ready', 'Stand by', 'Calibration', 'Seg 0 in Run 1', and the time '14:00:56'.



# DSCQ20 T1熱流基線校正操作步驟

- 選取完您所執行的校正檔後，圖形便會出現在右方的視窗中。



# DSCQ20 T1溫度與爐子常數校正操作步驟

## T1模式

### 溫度及爐子常數校正 (Cell constant & temperature)

爐子常數是已知的標準材料融熔熱和標準材料做量測實驗所得的融熔熱的比值。實驗量出的熱是用尖峰面積積分計算出來的，並以樣品重量做常態化計算。這種校正的形式是用來校正儀器的溫度。在溫度校正實驗中所收集的資料會儲存在溫度校正表裡。

溫度校正建議使用適當的已知熱性質的標準物質（例:錳，鉛等等）。

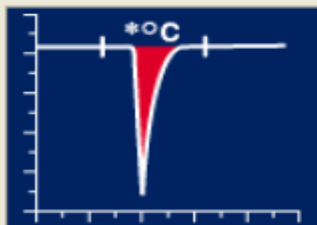
Note :

類型	毫克數
金屬或化學熔點	<5mg
高分子T <sub>g</sub> 或T <sub>m</sub>	10mg
複合物或聚摻物	>10mg

# DSCQ20 T1溫度與爐子常數校正操作步驟

1. 準備標準品樣品。放置於樣品平台。
2. 製作一個空的同型式的參考品盤。放置於參考品平台。
3. 再次進入控制軟體TA Instruments，請選取校正模式或點選控制頁面上的校正精靈按鈕。軟體會執行到以下視窗。

## Conventional DSC Experiments [Q10-0300 - DSC Q10@Mfg-dsc]



Select the type of DSC calibration experiment you want to perform:

- Baseline  
This calibration compensates for subtle differences between the reference and sample thermocouples. The baseline calibration is based on heating an empty cell through the same temperature range that will be used in subsequent experiments. Baseline calibration is recommended any time the heating/cooling rate, purge gas, or cooling accessory is changed.
- Cell Constant / Temperature  
Cell Constant is a calibration factor used to adjust for subtle differences in the calorimetric response of a DSC cell. Temperature calibration ensures that the sample thermocouple reading is correct under the experimental conditions chosen. Both of these calibrations are performed based on the melting peak of a standard metal such as indium. These calibrations should be performed any time that the heating/cooling rate, purge gas, cooling accessory, or pan type is changed.



選取此爐子係數及溫度校正選項並按下一步繼續設定實驗條件。

Next >

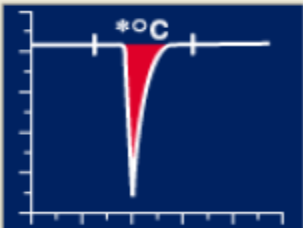
Cancel

Help

# DSCQ20 T1溫度與爐子常數校正操作步驟

4. 選取校正金屬。「如Indium銻金屬」。
5. 設定溫度範圍。一般建議從標準金屬熔點前50度開始平衡，再做升溫檢測，並在標準熔點後50度結束實驗。設定完成後，按下「Next」進入下一設定。

Experimental Parameters: Cell Constant [Q10-0300 - DSC Q10@Mfg-dsc]



Cell constant and temperature calibrations involve heating a high purity, metal standard (e.g., indium) through its melting peak using the same ramp rate, purge gas and pan type that will be used for subsequent experiments.

Enter the desired parameters:

Standard	<input type="text" value="Indium"/>	1. 設定標準金屬
Start temperature	<input type="text" value="Indium"/>	Premelt 2. 設定起始溫度
Heating rate:	<input type="text" value="10.00"/>	°C/min 3. 設定升溫速率
Final temperature:	<input type="text" value="180.000"/>	°C 4. 設定結束溫度

# DSCQ20 T1溫度與爐子常數校正操作步驟



# DSCQ20 T1溫度與爐子常數校正操作步驟

Sample Information [Q10-0300 - DSC Q10@Mfg-dsc]

Sample Information

Sample Name: Indium

Sample Size: 0.000 mg **1.輸入 indium 的重量**

Comments:

Data File: C:\ATA\Data\DSC\TA Service\980219\Baseline 980219... **2.由此按下選取存檔路徑**

Start Remotely

< Back Next > Cancel Help

Signal

- Method Time
- Segment Time
- Remaining Run Time
- Temperature
- Delta T
- Heat Flow
- Set Point Temp
- Heater Power

Enter Desired Data File Name

搜尋位置(S): 980219

- Baseline 980219
- Dry Cell condition 980219
- Replication

檔案名稱(N): Indium.980219.001

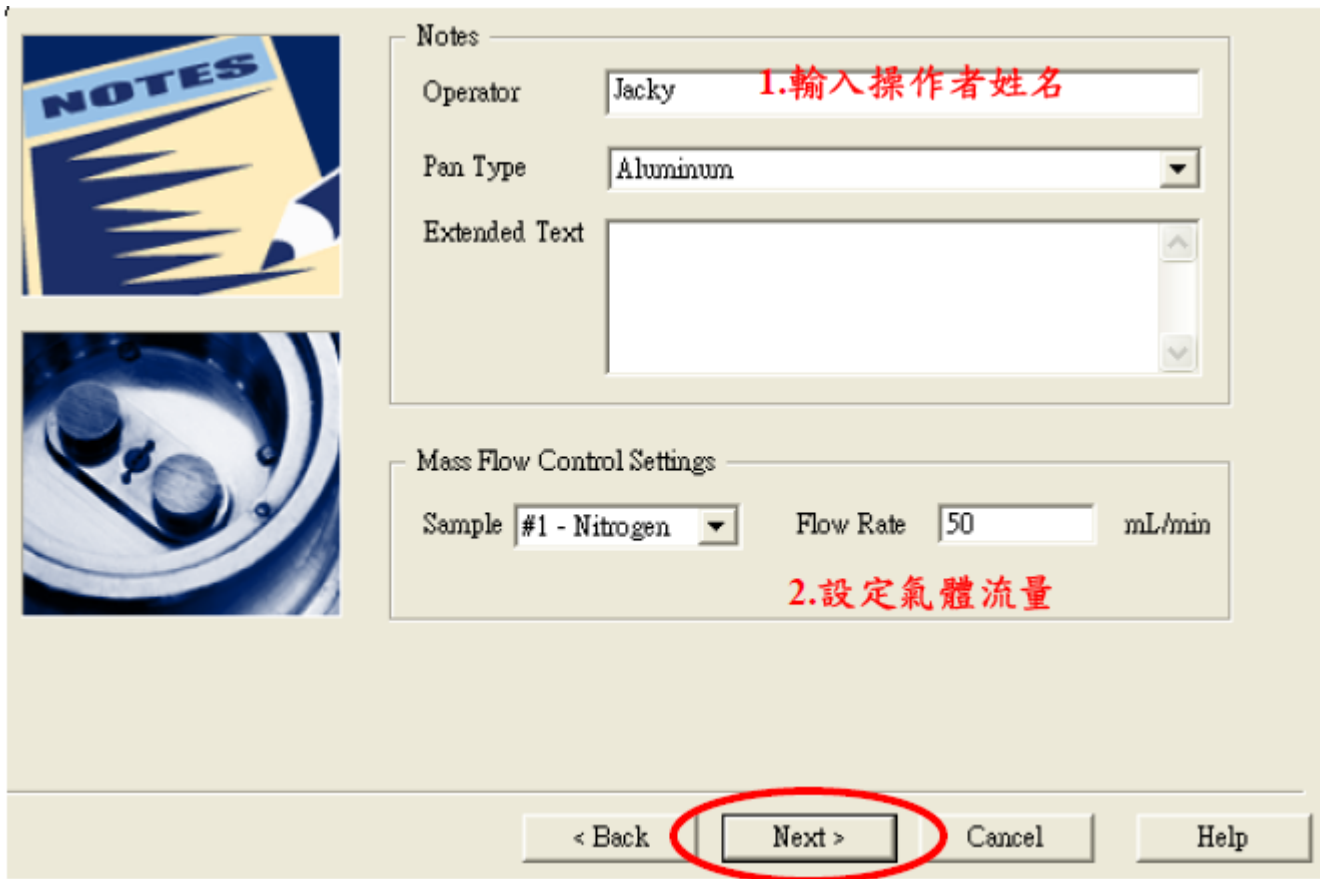
檔案類型(T): Data files (\*.\*)



- 標準金屬的製作過程不可使用雙手，以免外在因素的干擾影響數據。
- 標準金屬的質量需量測到0.01mg為主，以免影響吸放熱數值計算。

# DSCQ20 T1溫度與爐子常數校正操作步驟

進入此視窗之後，輸入使用者及氮氣流量後按下「Next」進入下一設定。



Notes

Operator  1.輸入操作者姓名

Pan Type

Extended Text

Mass Flow Control Settings

Sample  Flow Rate  mL/min

2.設定氣體流量

< Back **Next >** Cancel Help

# DSCQ20 T1溫度與爐子常數校正操作步驟

再次確認以下事項；氣體已準備妥當、冷卻系統已妥當、樣品已放置妥當。  
按下「Start Run」，開始校正。

**Purge Gas**

✓ Be sure that your purge gas is connected and properly regulated. Purge Gas is recommended for all DSC experiments.

**Cooling Accessory**

✓ The Q Series DSC cells generally require a cooling accessory to be connected regardless of the type of experiment being run. The QCA is the only exception. It is removed before initiating heating ramp experiments.

**Loading the Sample**

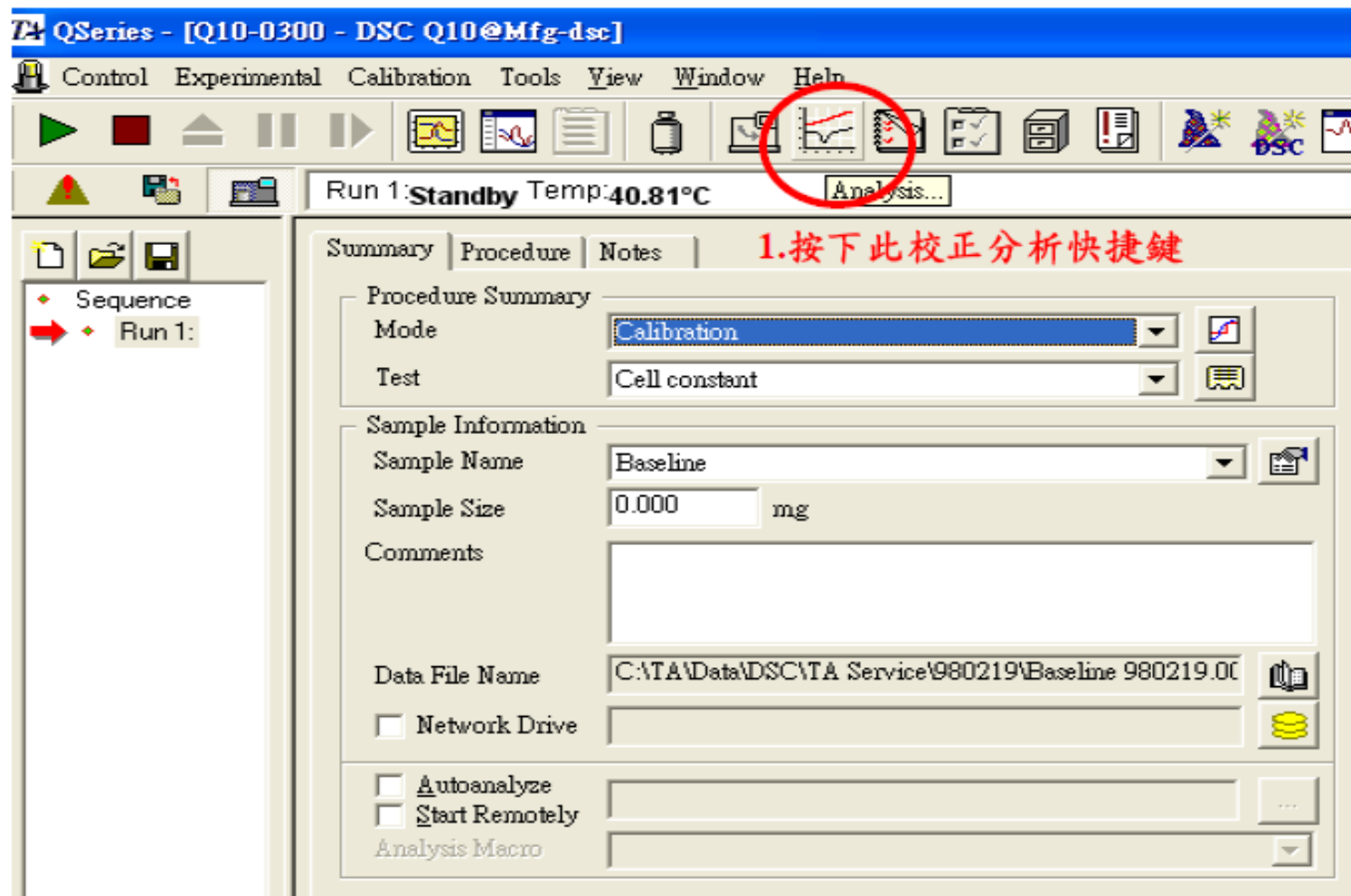
✓ Position the sample pan into the cell. [Typically, an empty cell (e.g., no pans) is used for this calibration.] Position a reference pan, of the same type, into the cell (back position). Cover the cell with the appropriate lids.

Append Run **Start Run** Finish Cancel Help



# DSCQ20 T1溫度與爐子常數校正操作步驟

當校正結束後，按下校正分析軟體來分析校正溫度圖譜。「如圖所示」。



# DSCQ20 T1溫度與爐子常數校正操作步驟

The screenshot displays the QSeries software interface for DSC Q10 calibration. The main window shows a graph with 'Heat Flow (mW)' on the x-axis (0.90 to 2.00) and temperature on the y-axis (0.90 to 2.00). A status bar at the top indicates 'Run 1: Complete Temp: 40.82°C'. An 'Open Calibration File' dialog box is open, showing a search path of '990219' and a list of files including 'Baseline 990219', 'Dry Cell condition 990219', 'Indrum 990219', 'Indrum 990219', and 'Replication'. The 'Indrum 990219' file is selected. The dialog box also shows the file name 'Indrum 990219' and the file type 'Calibration Files (\*.\*)'. The 'Open File' button in the bottom-left corner of the main window is circled in red.

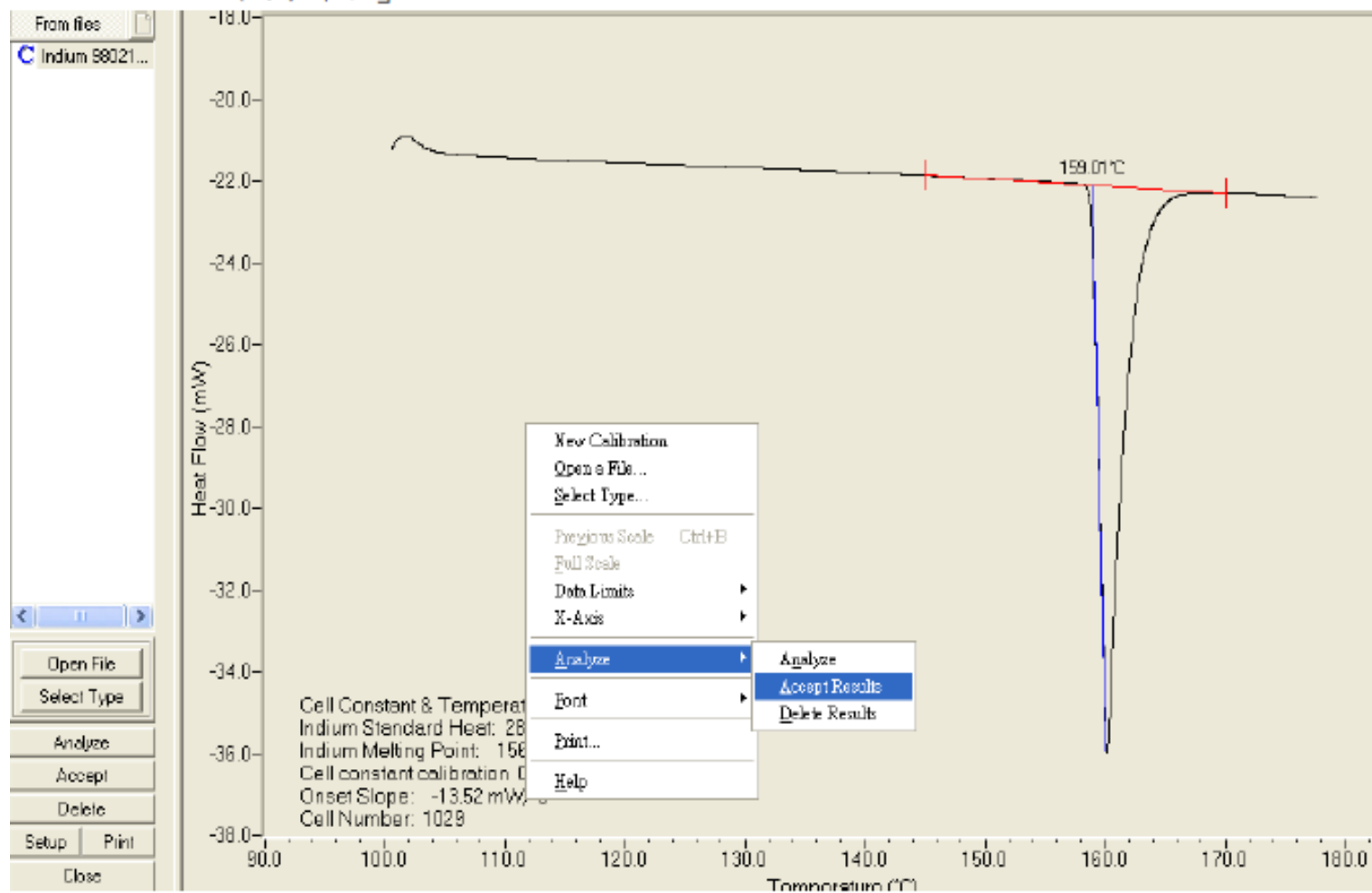
2. 開啟校正檔案

3. 選取檔案

# DSCQ20 T1溫度與爐子常數校正操作步驟

選取完您所執行的校正檔後，圖形便會出現在控制軟體右方的視窗中。

「如下圖所示」。

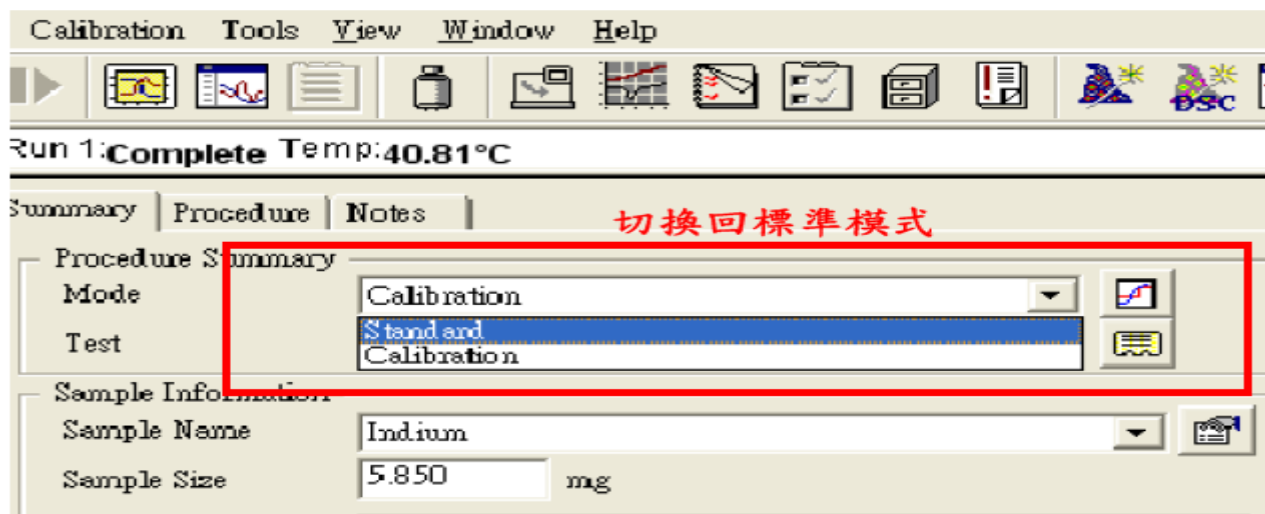


# DSCQ20 T1溫度與爐子常數校正操作步驟

在空白的地方按下滑鼠左鍵→執行分析「Analyze」→「Accept Results」→確認校正結果，此時儀器會將所量測得的溫度與標準溫度間的誤差計算出來，套用到往後的實驗中。

校正項目	校正目的	規格值
Baseline	爐子基本情況	不可有異常吸放熱Peak
Indium	溫度校正	Onset slope $\geq -10\text{mw}/^{\circ}\text{C}$
		156.6°C $\pm 4^{\circ}\text{C}$
Cell constant	吸放熱數值補償	0.95-1.3

結束校正後，請將操作模式切換回標準模式，如下圖所示；



# DSCQ200/2000 T4 之 T-zero 校正操作步驟

## T4模式所需執行的校正步驟

T4模式的校正適用於DSC Q200 及 DSC Q2000，所需進行的步驟有：

- 熱阻、熱熔及藍寶石校正「T-Zero calibration」。
- 溫度校正「Indium」。

## 熱阻熱熔及藍寶石校正（T-Zero calibration）

DSC爐子是由純銀物質組成，所提供的熱傳導誤差相當微小。為了消除樣品及參考品平台端的熱傳導誤差，我們建議您進行基線校正。

不論你現在所使用的儀器模式或是爐子形式，DSC基線校正實驗都會以類似的方式執行。依照下列步驟會收集及分析校正資料。

T4熱流式校正需花費約莫一天的時間，為避免錯誤的誤差影響校正值，請仔細並正確的依循以下步驟；

# DSCQ200/2000 T4 之 T-zero 校正操作步驟

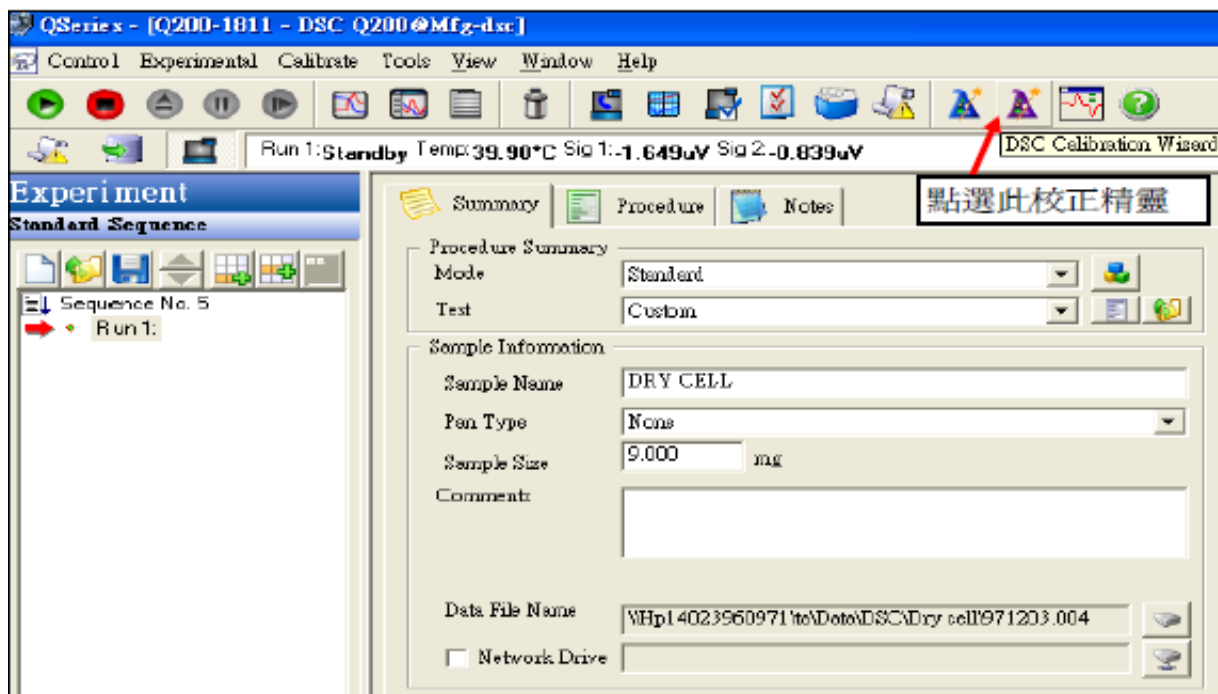
T4熱流式基線校正操作步驟如下簡介：

步驟如下：

1. 確定Purge gas、Air Cool氣體管線已開啟與冷卻配件(如：RCS) 開機妥善。
2. 開啟DSC儀器。
3. 進入控制軟體 [TA Instruments](#)，選取校正模式或點選控制頁面上的校正精靈按鈕。



。如下圖所示。



# DSCQ200/2000 T4 之 T-zero 校正操作步驟

## 4. 根據您的儀器模式來選擇

「Q200 or Q2000 / Heat Flow T4 or T4P」及對應的冷卻配件「RCS或LNCS」。

在此選項中亦可選擇您需要的功能。

在T4熱流式基線校正內控制軟體會顯示下列選項讓您選擇；

首先先選擇校正模式及冷卻系統配件；「如下圖所示」。



DSC calibration should be performed using the same conditions (purge gas, sample pan type, and cooling accessory) that will be used in subsequent experiments on your sample materials. In addition, the calibration sequence performed depends on the type of heat flow that will be stored in your subsequent experiments. Selection of the highest level heat flow for your Q Series DSC is recommended.

Heat Flow Signals:

Heat Flow T4 (mW)

Heat Flow T1 (mW)

Heat Flow T4 (mW)

Heat Flow T4P (mW)

Cooling Unit:

RCS (90)

使用下拉式選單選取欲校正的熱流模式

# DSCQ200/2000 T4 之 T-zero 校正操作步驟



DSC calibration should be performed using the same conditions (purge gas, sample pan type, and cooling accessory) that will be used in subsequent experiments on your sample materials. In addition, the calibration sequence performed depends on the type of heat flow that will be used in your experiments. Selection of the highest level heat flow for your Q Series DSC is recommended.

Heat Flow Signals:

Heat Flow T4 (mW)

Cooling Unit:

RCS (90)

FACS

CCS

RCS (40)

RCS (90)

Quench cooler

設定冷卻系統配件

5. 當您選定校正模式及冷卻配件後，即可按下「Next」進入下一個步驟。此時會顯示下圖的畫面及選項供您選擇欲校正的項目；



# DSCQ200/2000 T4 之 T-zero 校正操作步驟



This wizard helps you setup and start experiments used to calibrate for cell resistance & capacitance differences, heat flow (cell) constant, and temperature. Select from one of the calibration options.

If your instrument is equipped with an autosampler, it will be disabled during this calibration.

Cell Resistance & Capacitance, Cell Constant, and Temperature Calibration:

Cell resistance and capacitance calibration compensates for subtle differences in thermal resistance and capacitance between the reference and sample platforms in the DSC sensor. The calibration is based on two experiments - one run with an empty cell, and the second run with equal weight sapphire disks on the sample and reference platforms. This calibration is recommended any time the purge gas or cooling accessory used in subsequent experiments are changed.

Cell Constant & Temperature Calibration

Cell constant is a calibration factor used to adjust for subtle differences in the calorimetric response of a DSC cell. Temperature calibration ensures that the sample thermocouple reading is correct under the experimental conditions chosen. Both of these calibrations are performed on the melting peak of a standard metal such as indium. These calibrations should be performed any time that the heating/cooling rate, purge gas, or cooling accessory are changed.

Load Saved Cell Resistance & Capacitance Calibration File to Instrument

This option allows you to send a saved cell resistance and capacitance (Tzero) calibration results file to the instrument.

- 選項一：熱阻、熱融、爐子係數及溫度校正。
- 選項二：爐子係數及溫度校正。
- 選項三：讀取校正檔案。此選項僅在校正完成後軟體未記錄校正檔案時才建議您重新載入校正檔案。

# DSCQ200/2000 T4 之 T-zero 校正操作步驟

## 6. 設定校正參數；「如下圖所示」。



Cell resistance & capacitance calibration involves two experiments. The first experiment involves heating an empty cell through the widest temperature range that will be used in subsequent experiments.

Conduct experiment

1. Remove any pans from cell and cover cell.
2. Enter the desired test parameters:

Calibration range: Tzero Heat only cal

Lower temperature: -90.00 °C

Upper temperature: 400.00 °C

Ramp rate: 20.000 °C/min

Operator: Rico

Comment: Tzero Calibration - Baseline Run

Data File Name: \\Lotus1\ta\Data\DSC\980217.001

Network Drive

Purge Gas: #1 - Nitrogen Flow Rate: 50 mL/min

1. 設定起始溫度  
2. 設定終止溫度  
3. 設定升溫速率


4. 設定存檔路徑

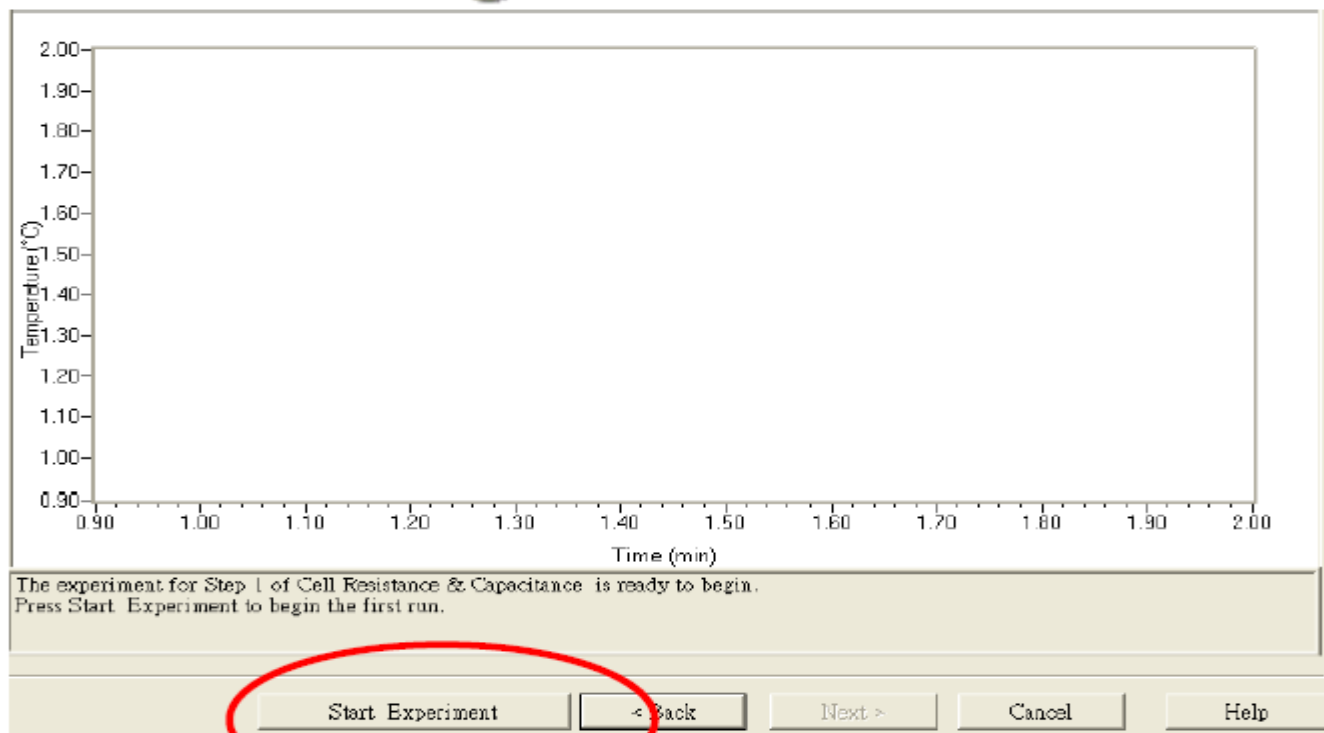
5. 設定氣體流量

Enter existing T Zero Baseline data file:

# DSCQ200/2000 T4 之 T-zero 校正操作步驟

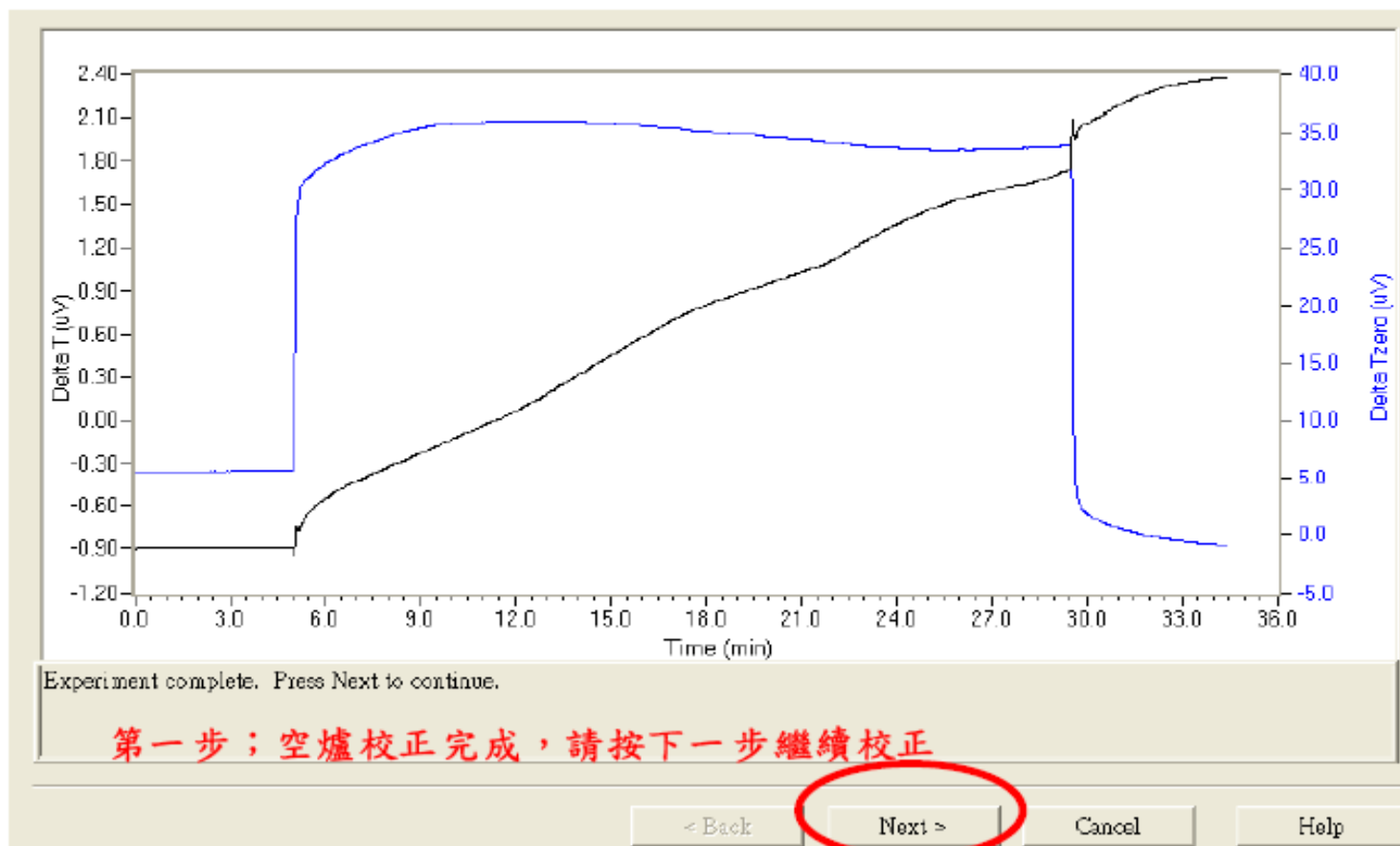
- 在設定完欲校正的溫度範圍及其他參數後，按下「Next」鍵，檢視所設定的校正參數，完成後執行到下圖所示畫面視窗，按下「Start Experiment」開始校正。

再次確認DSC爐內不放置任何物品後，蓋上爐蓋，按下「Start Experiment」之後，即可進行T4校正  **注意：爐內不應放置任何物品。**



# DSCQ200/2000 T4 之 T-zero 校正操作步驟

8. 當第一步將空子的原始的情況校正完成後，控制軟體會將圖譜顯示於視窗內，如下圖所示。此時儀器會要求您繼續藍寶石「Sapphire」熱阻及熱熔校正。



# DSCQ200/2000 T4 之 T-zero 校正操作步驟

## 藍寶石校正 (Sapphire)

使用藍寶石來進行更精確的熱阻及熱熔校正。藍寶石將附在您的儀器配件盒中。建議您使用精密的天平來測量寶石的重量。

校正步驟如下；

當您從上一個校正步驟「T-zero Calibration-Baseline Run」結束之後，便會來到下圖所示視窗，進入藍寶石校正程序。請參照下列步驟執行校正。

1. 測量藍紅寶石重量並紀錄於保存盒上，應量測到小數點後三位數為準。
2. 將透明的 (Clear) 寶石放置於樣品平台上方，紅色的 (Red) 放於參考品平台上方，**不可放置於樣品盤內**，並於控制軟體內輸入重量。如下圖；

# DSCQ200/2000 T4 之 T-zero 校正操作步驟



The second calibration experiment involves heating the cell with two equal weight sapphire disks on the sample and reference platforms through the entire temperature range under the same conditions as the previous experiment. The sapphire disks are placed directly on the cell platforms (i.e., no sample pans are used).

Conduct experiment

1. Place sapphire 1 on sample platform in cell.

2. Enter weight of sapphire 1:  mg **1.輸入透明寶石重量**

3. Place sapphire 2 on reference platform in cell.

4. Enter weight of sapphire 2:  mg **2.輸入紅色寶石重量**

5. Cover cell.

6. Sapphire Data File Name:

Network Drive

**3.設定存檔路徑**

7. Comment:

Enter existing T Zero Sapphire data file:

**3.按下 Next 繼續下一頁設定**

Post-Test ...

< Back

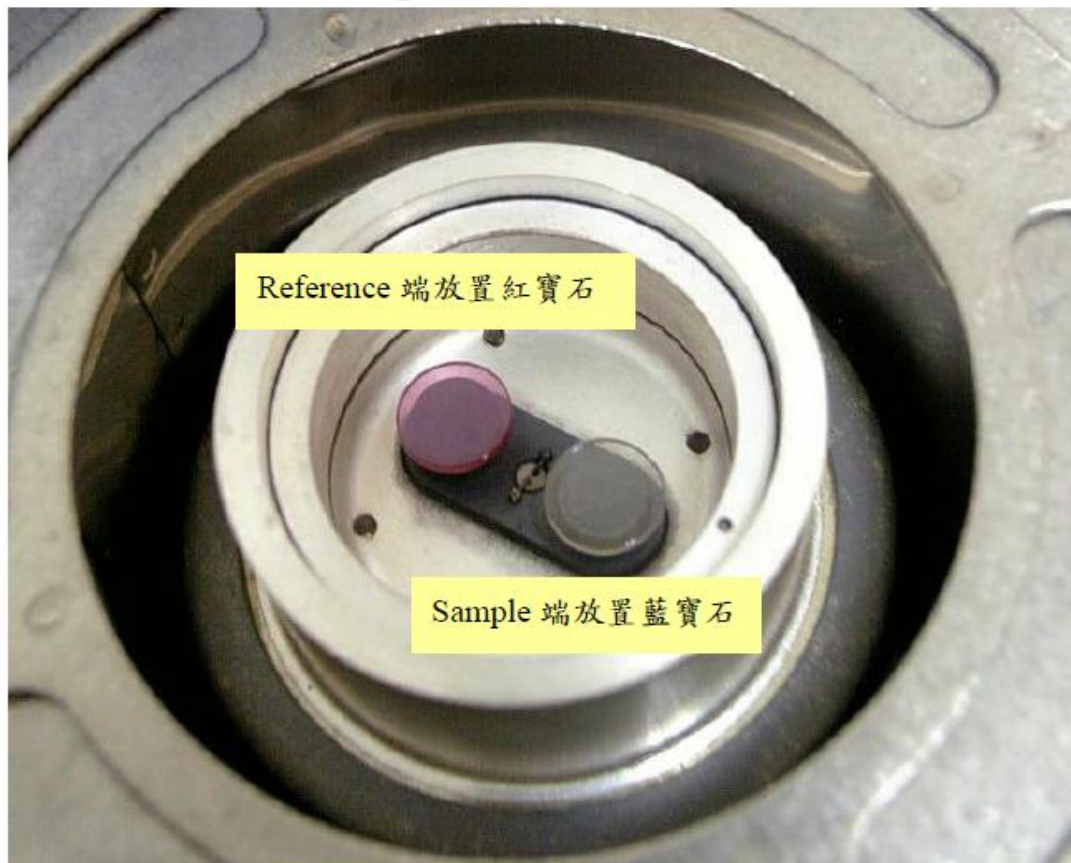
Next >

Cancel

Help

# DSCQ200/2000 T4 之 T-zero 校正操作步驟

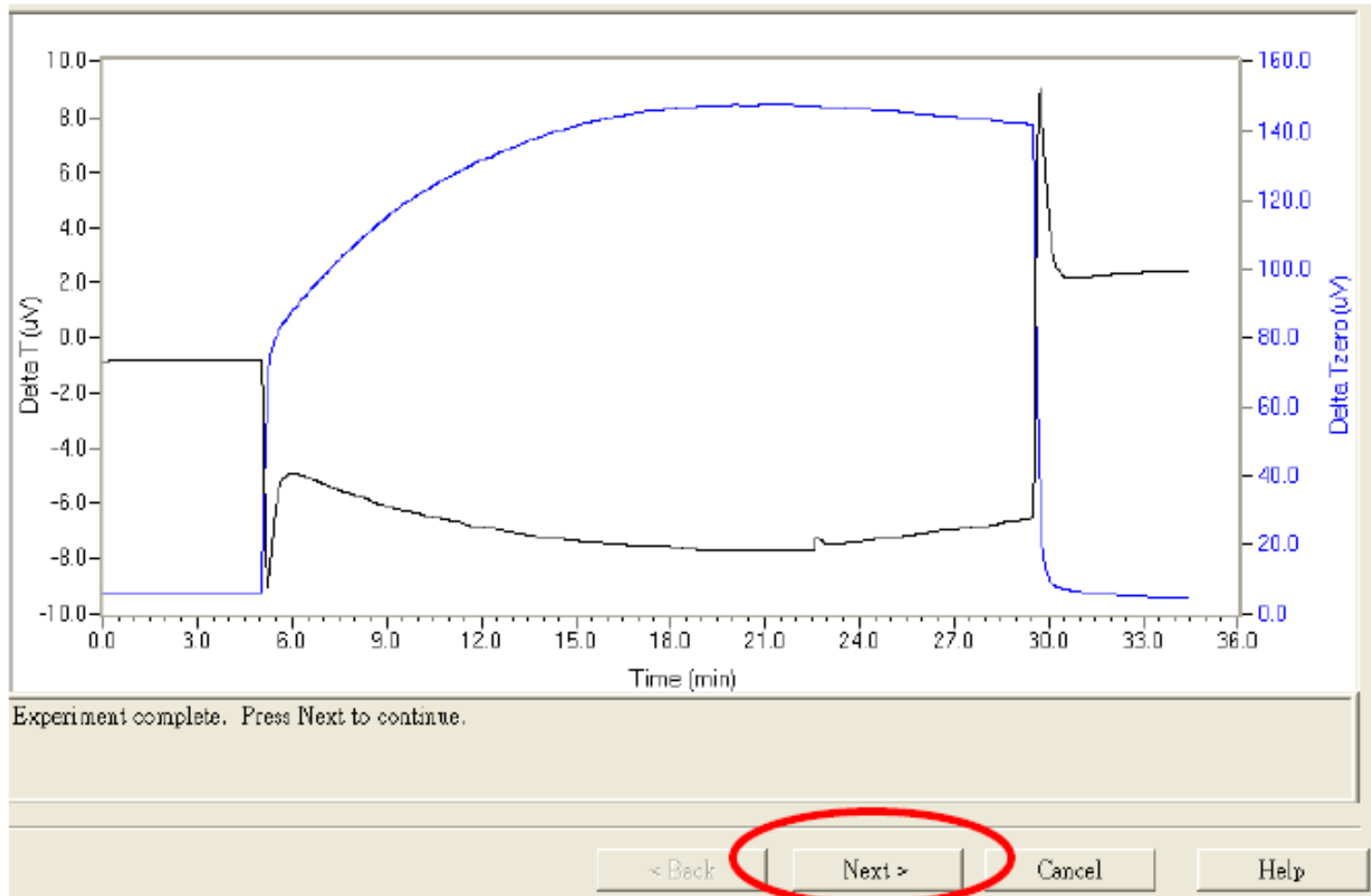
3. 確認藍紅寶石已經放置平穩完整接觸實驗平台後，蓋上爐蓋即可進行 Sapphire 校正。「如下圖所示」。



確認紅藍寶石已完整接觸平台時，將爐蓋蓋上，按下「Start」開始校正。

# DSCQ200/2000 T4 之 T-zero 校正操作步驟

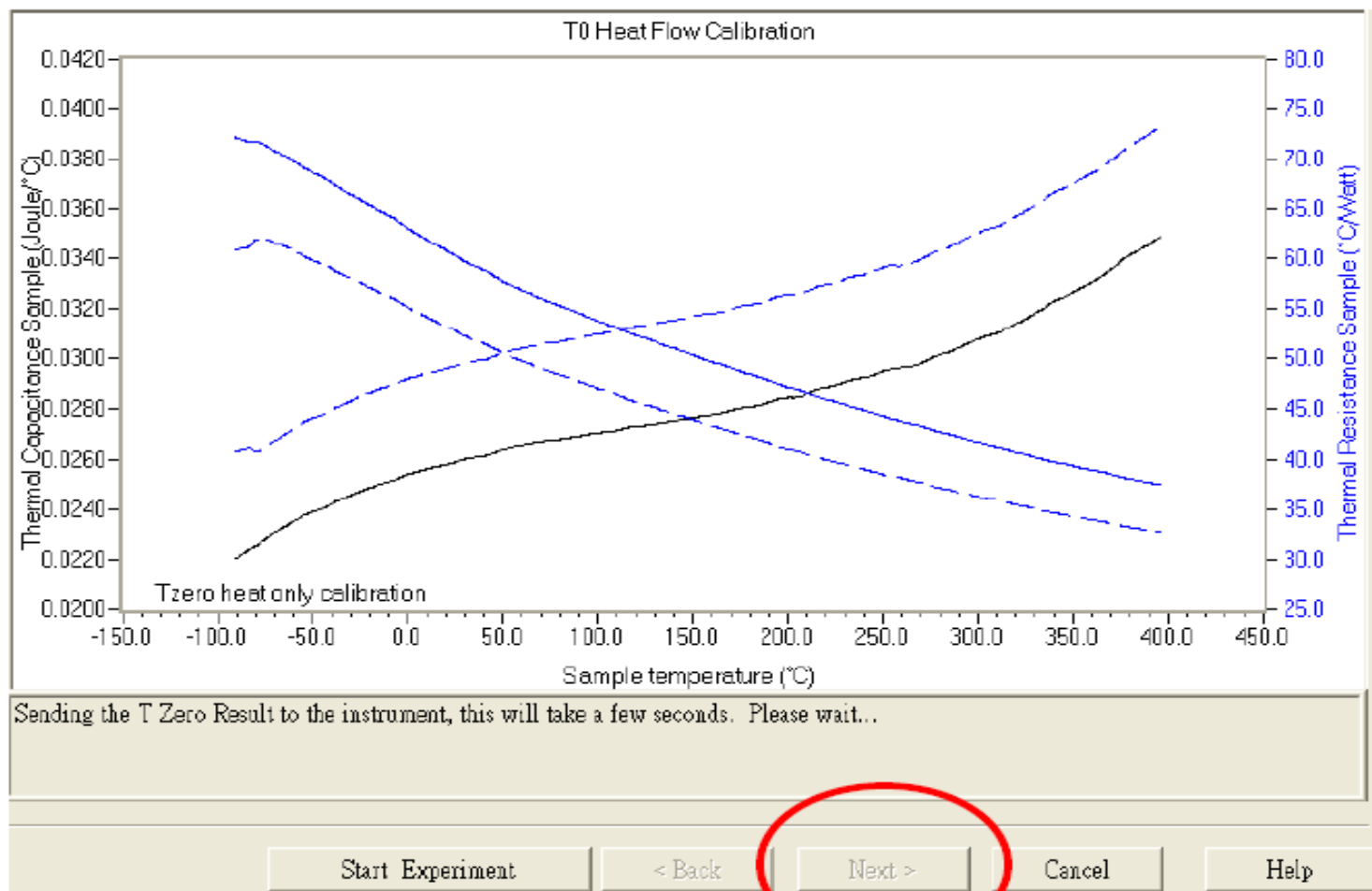
- 當藍寶石校正完成後，軟體會跳到下圖視窗，並等待您進行校正分析。  
按下「Next」以進行校正分析。





# DSCQ200/2000 T4 之 T-zero 校正操作步驟

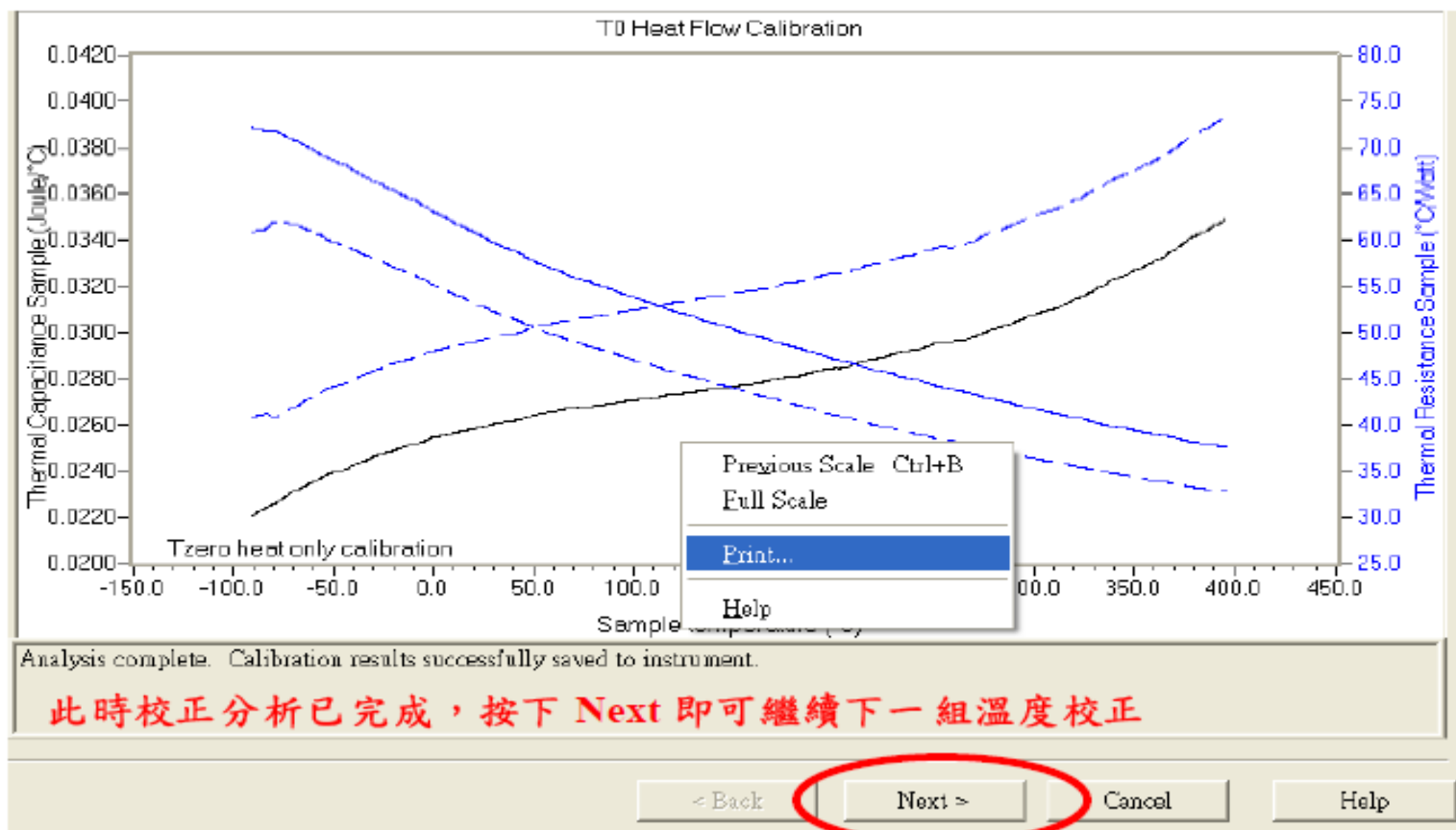
5. 校正圖譜會以下圖型式表現出來，此時按下「Next」以利分析軟體計算校正值。



按下Next鍵之後，分析軟體會自動進行計算，此計算結果大約會花費1分鐘。

# DSCQ200/2000 T4 之 T-zero 校正操作步驟

6. 當分析軟體計算完成後，若結果符合規格，則軟體會將結果自動儲存起來，若結果超出規格值，則結果則不予接受。若想列印校正結果，可在空白處按下右鍵→「Print」→即可列印校正結果。



# DSCQ200/2000 溫度與爐子常數校正操作步驟

## T4模式

### 溫度及爐子常數校正 (Cell constant & temperature)

爐子常數是已知的標準材料融熔熱和標準材料做量測實驗所得的融熔熱的比值。實驗量出的熱是用尖峰面積積分計算出來的，並以樣品重量做常態化計算。這種校正的形式是用來校正儀器的溫度。在溫度校正實驗中所收集的資料會儲存在溫度校正表裡。

溫度校正建議使用適當的已知熱性質的標準物質（例:錫，鉛等等）。

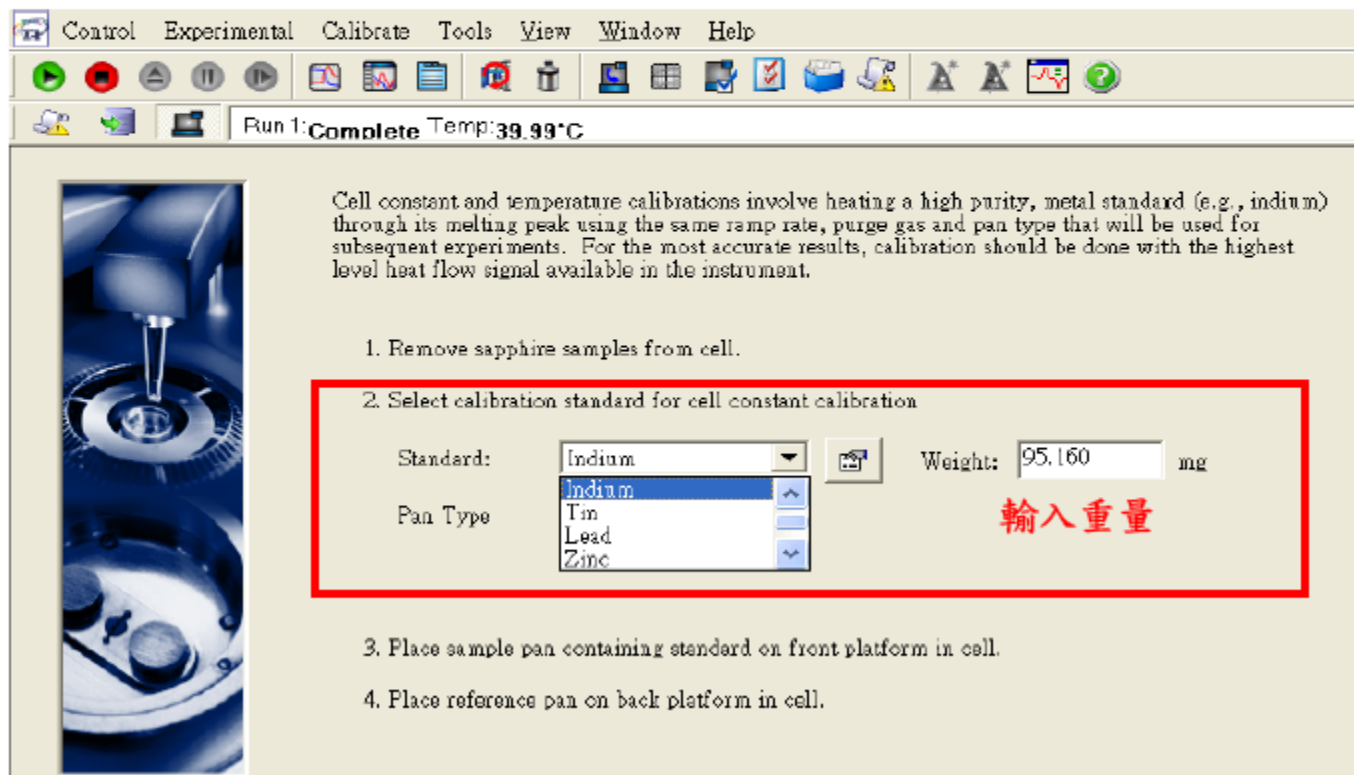
Note :

類型	毫克數
金屬或化學熔點	<5mg
高分子T <sub>g</sub> 或T <sub>m</sub>	10mg
複合物或聚摻物	>10mg

# DSCQ200/2000 溫度與爐子常數校正操作步驟

開始進入溫度校正的程序。

1. 準備標準品樣品。放置於樣品平台。
2. 製作一個空的同型式的參考品盤。放置於參考品平台。
3. 當我們結束T-zero及藍寶石校正之後便會來到以下所示視窗。
4. 選取校正金屬。「如Indium銻金屬」。並輸入重量。



Control Experimental Calibrate Tools View Window Help

Run 1: Complete Temp: 39.93°C

Cell constant and temperature calibrations involve heating a high purity, metal standard (e.g., indium) through its melting peak using the same ramp rate, purge gas and pan type that will be used for subsequent experiments. For the most accurate results, calibration should be done with the highest level heat flow signal available in the instrument.

1. Remove sapphire samples from cell.
2. Select calibration standard for cell constant calibration

Standard: Indium Weight: 95.160 mg

Pan Type: Tin

Lead

Zinc

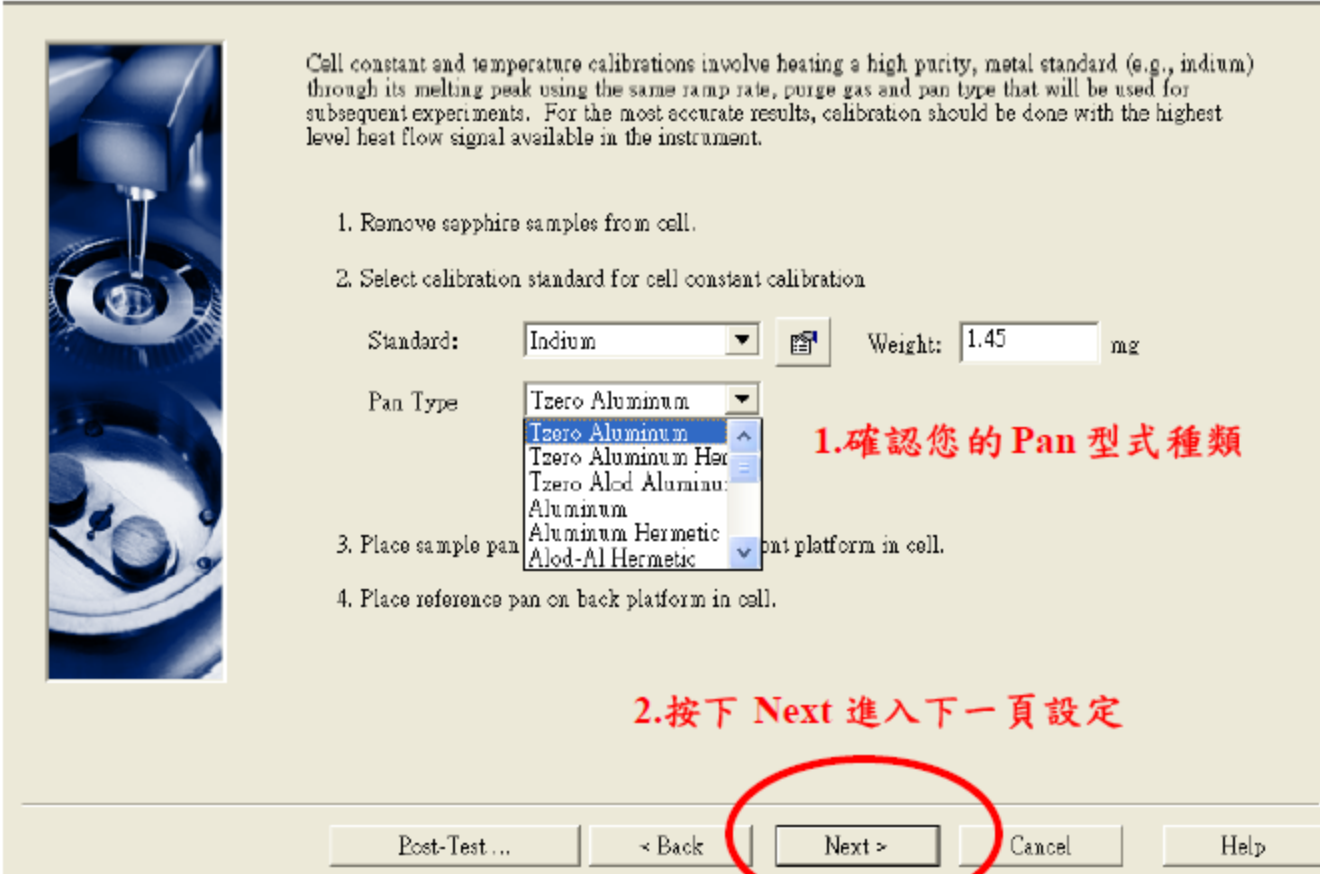
輸入重量

3. Place sample pan containing standard on front platform in cell.
4. Place reference pan on back platform in cell.

# DSCQ200/2000 溫度與爐子常數校正操作步驟

## 5. 確認您的Pan型式及設定溫度範圍。

一般建議從標準金屬熔點前50度開始平衡，再做升溫檢測，並在標準熔點後50度結束實驗。設定完成後，按下「Next」進入下一設定。



Cell constant and temperature calibrations involve heating a high purity, metal standard (e.g., indium) through its melting peak using the same ramp rate, purge gas and pan type that will be used for subsequent experiments. For the most accurate results, calibration should be done with the highest level heat flow signal available in the instrument.

1. Remove sapphire samples from cell.
2. Select calibration standard for cell constant calibration

Standard: Indium Weight: 1.45 mg

Pan Type: Tzero Aluminum

3. Place sample pan on front platform in cell.
4. Place reference pan on back platform in cell.

**1. 確認您的 Pan 型式種類**

**2. 按下 Next 進入下一頁設定**

Post-Test ... < Back **Next >** Cancel Help

# DSCQ200/2000 溫度與爐子常數校正操作步驟

6. 設定實驗條件。如下圖。

Conduct experiment

1. Enter the desired test parameters for cell constant and temperature calibration.

Start Temperature:  Use current  Premelt  
100.00 °C 1. 設定起始溫度

Heating Rate: 10.000 °C/min 2. 設定升溫速率

Final Temperature: 180.00 °C 3. 設定結束溫度

Data File Name: \\Lotus\sta\Data\DSC\980217.003 4. 設定存檔路徑

Network Drive

Operator: Rico

Purge Gas: #1 - Nitrogen Flow Rate: 50 mL/min 5. 設定氮氣流量

2. Cover Cell.

Enter existing Cell Constant data file:

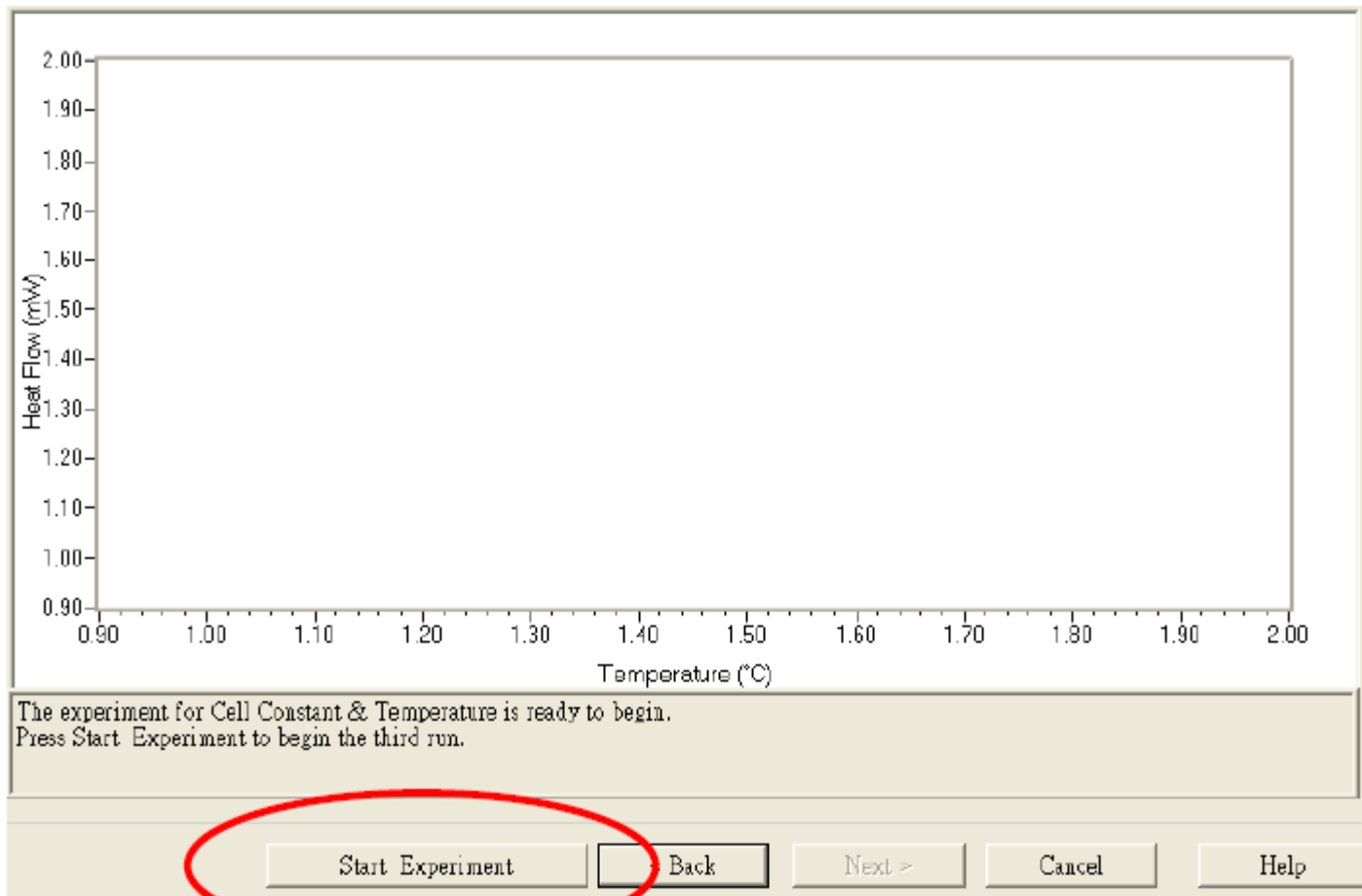
Post-Test ... < Back Next > Cancel Help

# DSCQ200/2000 溫度與爐子常數校正操作步驟

7. 再度確認所有參數皆設定完成後，按下「Next」進入下一設定。
8. 執行到下圖視窗後，用鑷子取出標準金屬「Indium」約3mg左右即可，秤重後使用固態盤製作，並放入樣品量測平台上；另準備一個相同形式的空盤放入量測平台的參考品平台上，蓋上爐蓋準備實驗。



# DSCQ200/2000 溫度與爐子常數校正操作步驟



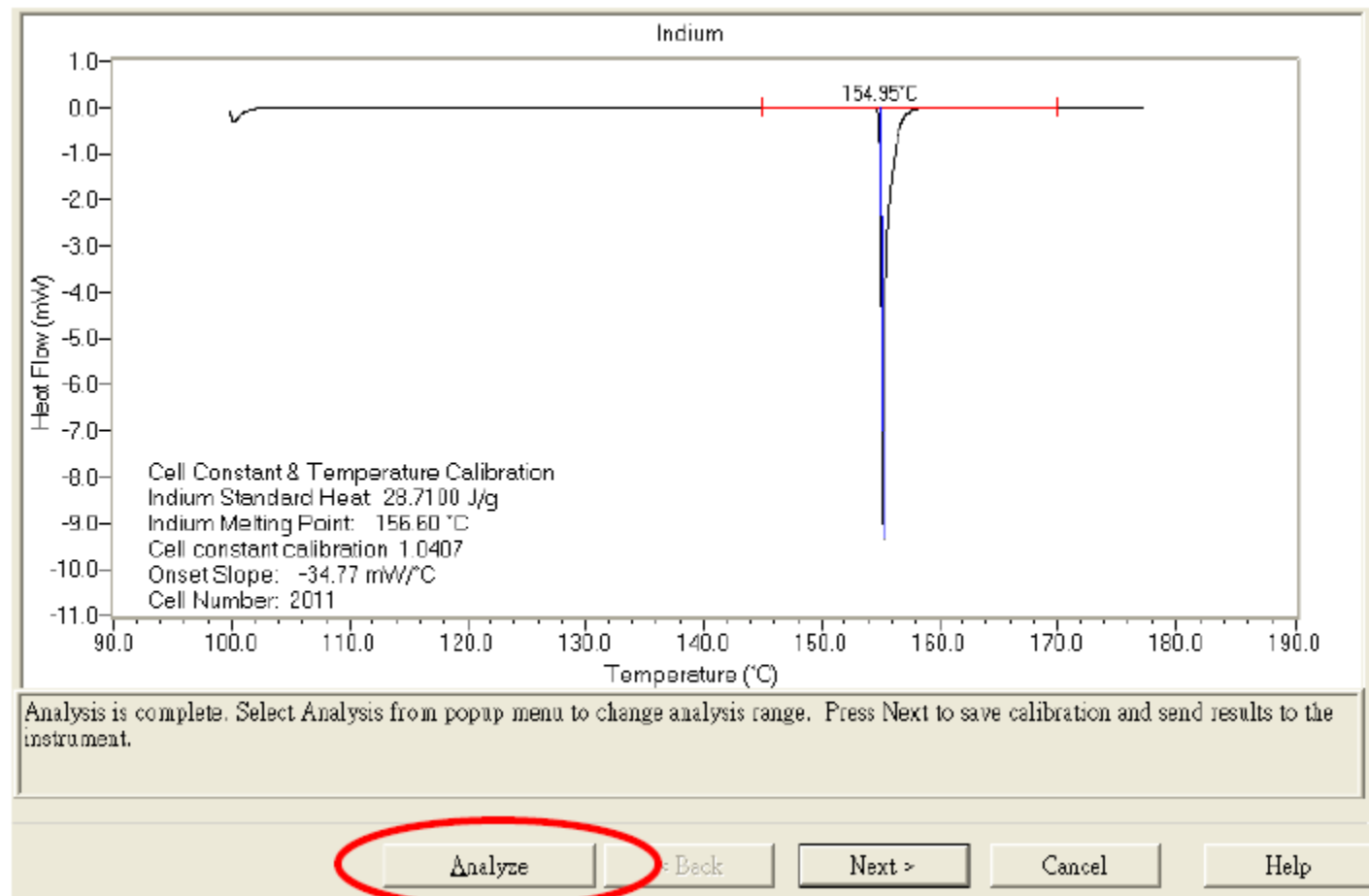
按下「Start Experiment」，開始校正。



# DSCQ200/2000 溫度與爐子常數校正操作步驟

選取完您所執行的校正檔後，圖形便會出現在控制軟體右方的視窗中。

按下「Analyze」以分析校正結果，「如下圖所示」。



# DSCQ200/2000 溫度與爐子常數校正操作步驟

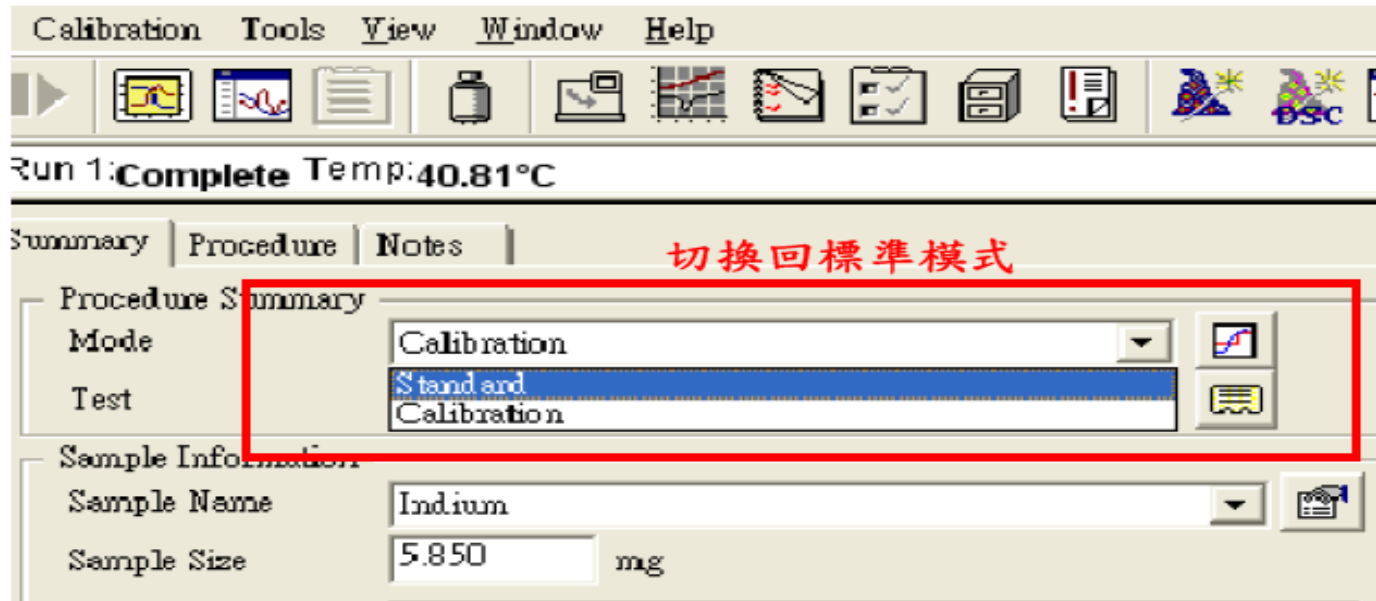
當顯示如下圖所示文字時，代表已經完成分析校正值，按下「Next」鍵以執行儲存校正資料。確認校正結果，此時儀器會將所量測得的溫度與標準溫度間的誤差計算出來，套用到往後的實驗中。

Analysis complete. Calibration results successfully saved to instrument.

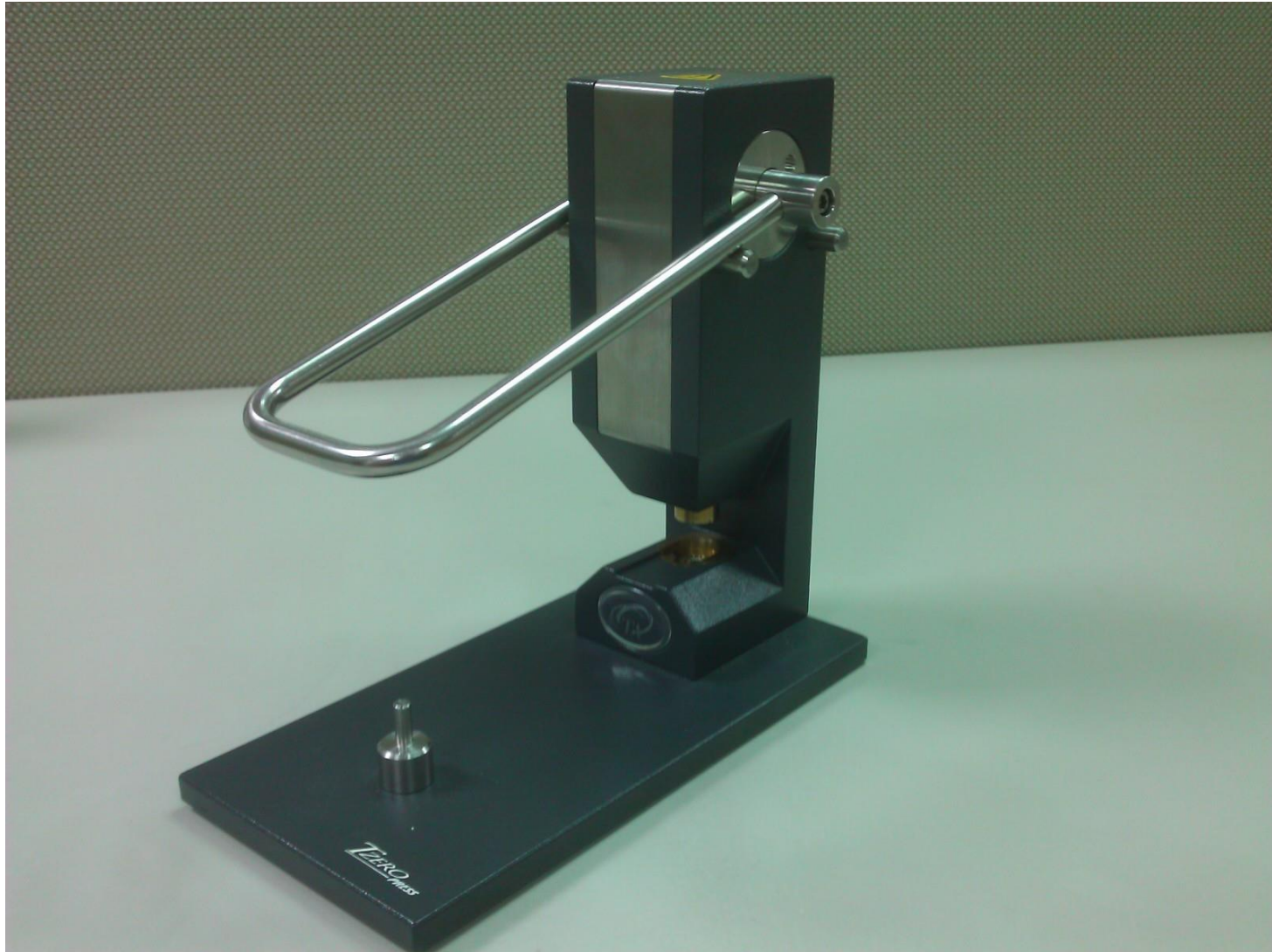
校正項目	校正目的	規格值
Baseline	爐子基本情況	不可有異常吸放熱Peak
Indium	溫度校正	Onset slope $\geq -10\text{mw}/^\circ\text{C}$
		156.6°C $\pm 4^\circ\text{C}$
Cell constant	吸放熱數值補償	0.95-1.3

# DSCQ200/2000 溫度與爐子常數校正操作步驟

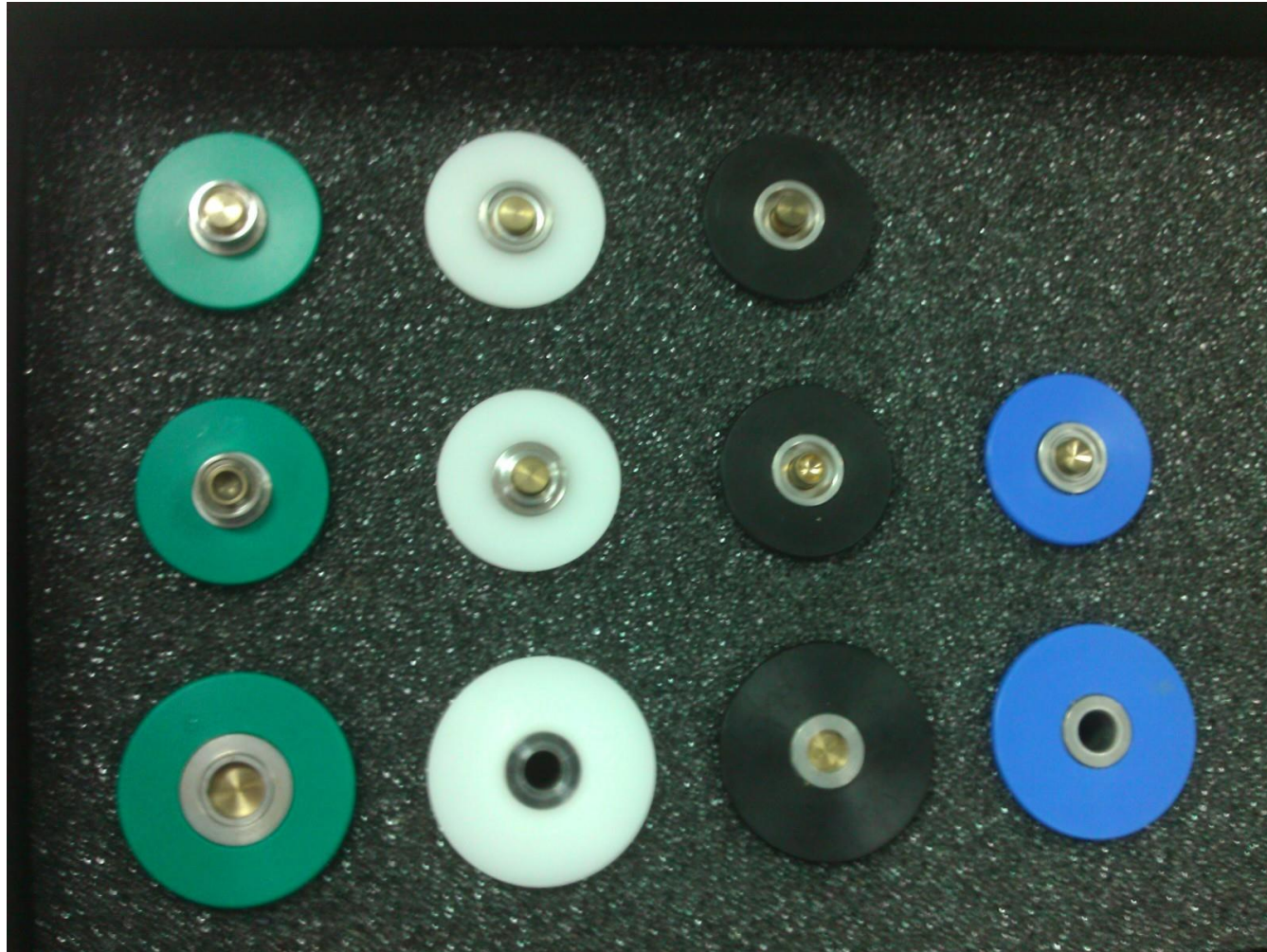
結束校正後，請將操作模式切換回標準模式，如下圖所示；



# T-Zero DSC 壓片機



# T-Zero 壓片機 模具組



# T-Zero 樣品盤



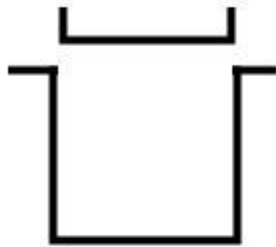
# T-Zero 樣品盤



# T-Zero 樣品盤

固態 (非密閉)

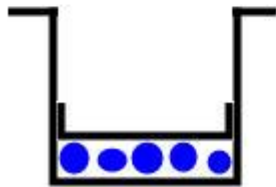
T-zero Lid



T-zero pan

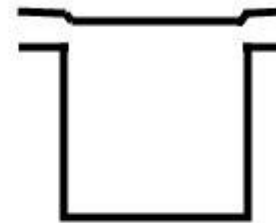


黑色模具



液態 (密閉)

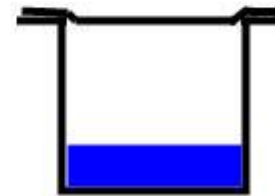
T-zero Hermetic Lid



T-zero pan



藍色模具





# DSC CELL 汙染與清潔

DSC最常見的問題就是爆盤而造成汙染。

汙染會造成基線異常, T-zero熱電偶損傷甚至斷裂。

發現汙染後須立刻停止實驗, 並進行 Clean Cell的動作。

## 清潔Cell的步驟

- 首先可先用酒精或溶劑將大部分的汙染物擦拭清除。
- 接著將DSC加熱到400~450度, 將汙染物燒至裂解, 升溫速率可用10~20°C/min, 此時請用N2 Purge。
- 讓Cell 冷卻到40度以下後, 用玻璃纖維刷將汙染物刷除乾淨。
- 用吹球將刷下的汙染物及玻纖刷碎屑吹除乾淨, 或用吸塵器吸乾淨。
- 清潔Cell後因Cell的狀況與熱傳已改變, **請務必先完成所有校正**, 然後方可測試樣品。
- <https://www.youtube.com/watch?v=cclJXrbUICA>

# DSC 關機順序

- 關機步驟：
  - 等待DSC回到40度
  - 將RCS撥回EVENT後,關掉RCS電源
  - 執行關機前烘乾CELL的除水氣程序
  - Equilibrate at 85°C
  - Isothermal for 30min
  - 完成後,執行Control \ Shutdown Instrument
  - 出現Shutdown視窗,按Start
  - 此時視窗自動關閉,DSC進行關機前參數回存動作
  - 等待DSC前方Ready燈熄滅或LCD螢幕提示可以關機後,便可關閉DSC電源
  - 關電腦
  - 關氮氣

# UA分析軟體實際演練說明

# 維修工時合約與PLUS合約

## 維修工時合約

### 1. 維護方式:

- a. 全年度無限次數維修工時費與交通費用一律免費。
- b. 所有維修零組件和耗材享有10%折扣。
- c. 提供一次年度證書校正，或年度保養。
- d. 設備服務時間: 周一至周五，早上08:30~下午17:30
- e. 回應時間:(以接到叫修時間為準)  
24小時內電話回應查尋問題所在。

# 維修工時合約與PLUS合約

## PLUS合約

### 1. 維護方式:

- a. 全年度無限次數維修工時費與交通費用一律免費。
- b. 非耗材之電子零組件部分損壞,一律免費更換。
- c. 消費性零組件和耗材享有10%折扣。
- d. 免費網路線上e-Training訓練課程。
- e. 設備服務時間: 周一至周五, 早上08:30~下午17:30
- f. 回應時間:(以接到叫修時間為準)

24小時內電話回應查尋問題所在.

優先安排維修行程至現場查看及檢修. (以工作時間為主)

# 頂級維修與應用合約

## 1. 維護方式:

- a. 全年度無限次數維修工時費與交通費用一律免費。
- b. 非耗材之電子零組件部分損壞,一律免費更換。
- c. 消費性零組件和耗材享有10%折扣。
- d. 年度PMV機台性能保養與校正.
- e. 設備服務時間:周一至周五,早上08:30~下午17:30
- f. 回應時間:(以接到叫修時間為準)

24小時內電話回應查尋問題所在.

優先安排維修行程至現場查看及檢修. (以工作時間為主)

## 2. 應用技術協助:

- a. 由TA專業應用人員到廠,針對需求提供專業技術諮詢與建議.  
總計五個工作天.
- b. 全年度無限次數e-mail專業應用技術諮詢.

# 保固政策說明

- A. 從安裝當天起12月計算或是運送到客戶端起18個月計算，任何條件成立即開始計算保固時間。
- B. 下列物品為安裝時若有破損才可以免費保固更換：
1. Glassware and Quartz Parts
  2. Thermocouples
  3. Gaskets/O-Rings
  4. CDs/DVDs
- C. 下列為90天保固期：
1. 在全部保固政策之內沒有特別註明的維修替代品零件
  2. 維修人員修理配件
  3. 零件重新維修品
- D. 下列為限制性保固一年。(若因人為使用不當或腐蝕則不予與保固)
1. Replacement TGA, SDT, DMA, TMA, ETC, and FCO furnaces and furnace cores

## 現場DSC操作問題 Q & A



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# Thank You

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