



THERMAL CONDUCTIVITY METER DTC-300



Getting Started Guide

Revision E Issued April 2020

Notice

The material contained in this manual is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. TA Instruments also reserves the right to revise this document and to make changes without notice.

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Introduction

Important: TA Instruments Manual Supplement

Please click the [TA Manual Supplement](#) link to access the following important information supplemental to this Getting Started Guide:

- TA Instruments Trademarks
- TA Instruments Patents
- Other Trademarks
- TA Instruments End-User License Agreement
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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

NOTE: A NOTE highlights important information about equipment or procedures.

CAUTION: A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

MISE EN GARDE: UNE MISE EN GARDE met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *DTC-300 Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Regulatory Compliance

Safety Standards

For Canada

CAN/CSA-C22.2 No. 61010-1 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010 Particular requirements for laboratory equipment for the heating of materials.

For European Economic Area

(In accordance with Council Directive 2006/95/EC of 12 December 2006 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN 61010-1:2012 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN 61010-2-010:2003 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For United States

UL61010-1:2012 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

UL61010A-2-010:2002 Particular requirements for laboratory equipment for the heating of materials + Amendments.

Electromagnetic Compatibility Standards

For Australia and New Zealand

AS/NZS CISPR 11:2007 Industrial, Scientific and Medical (ISM) Equipment - Radio-frequency Disturbance Characteristics - Limits and Methods of Measurement.

For Canada

ICES-003 Issue 5, February 2004 Information Technology Equipment (ITE) - Interference-Causing Equipment Standard - Limits and Methods of Measurements.

For the European Economic Area

(In accordance with Council Directive 2004/108/EC of 15 December 2004 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1:2006 Electrical Equipment for Measurement, Control, and Laboratory Use - EMC Requirements - Part 1: General Requirements.

Emissions: Meets Class A Requirements per CISPR 11.

Immunity: Per Table 1 - Basic Immunity Test Requirements.

For the United States

Code of Federal Regulations (CFR) Title 47 - Telecommunication, Chapter I - Federal Communications Commission (FCC), Part 15 - Radio Frequency Devices, Subpart B - Unintentional Radiators for a Class A Digital Device (FCC regulation pertaining to radio frequency emissions).

Supplier's Declaration of Conformity

47 CFR § 2.1077 Compliance Information

Unique Identifier: 854030.901–.906

Responsible Party:

TA Instruments
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www.tainstruments.com

FCC Compliance Statement:

This device complies with Part 15 of the FCC rules. Operation is subject to the following two conditions: (1) This device may not cause harmful interference, and (2) this device must accept any interference received, including interference that may cause undesired operation.

Safety

Instrument Symbols

The following label is displayed on the instrument for your protection:

Symbol	Explanation
	<p>This symbol indicates that a hand crush hazard is present. Take care to keep hands clear of the Test Stack during loading and unloading of samples.</p> <p>Ce symbole indique la présence d'un risque de broyage de main. Prenez soin d'éloigner vos mains de la cheminée d'essai pendant la mise en place et la décharge des échantillons.</p>
	<p>This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn to come in contact with this hot surface.</p> <p>Ce symbole indique la présence possible d'une surface chaude. Prenez soin de ne pas toucher cette zone ou de laisser un matériau susceptible de fondre ou de brûler entrer en contact avec cette surface chaude.</p>

Please heed the warning labels and take the necessary precautions when dealing with these areas. This *Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Warnings

WARNING: The operator of this instrument is advised that if the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.

AVERTISSEMENT: L'utilisateur de cet instrument est prévenu qu'en cas d'utilisation contraire aux indications du manuel, la protection offerte par l'équipement peut être altérée.

WARNING: Always unplug the instrument before performing any maintenance.

AVERTISSEMENT: Débranchez toujours l'instrument avant de procéder à la maintenance.

WARNING: No user serviceable parts are contained in the DTC-300. Maintenance not specified in the DTC-300 Getting Started Guide and repairs must be performed by TA Instruments or other qualified service personnel only.

AVERTISSEMENT: Aucune pièce du DTC-300 n'est réparable par l'utilisateur. Les travaux de maintenance non spécifiés dans le guide de démarrage du DTC-300 et les réparations doivent être effectués uniquement par TA Instruments ou tout autre personnel d'entretien qualifié.

WARNING: Do not raise the guard furnace until it is below 50°C. Failure to do so may result in personal injury and/or damage to the instrument.

AVERTISSEMENT: Ne soulevez pas la fournaise de protection tant qu'elle n'est pas inférieure à 50 ° C. Dans le cas contraire, vous risquez de vous blesser et / ou d'endommager l'instrument.

CAUTION: Do not power off the chiller during instrument operation. The top stack, guard furnace, and bottom stack must be below 50°C. Failure to do so may result in damage to the instrument.

Electrical Safety

Supply voltage: 100 to 120VAC or 200 to 240VAC (Instrument Dependent, Reference the Voltage Rating on the Back Panel)

Mains frequency: 50 to 60 Hz

Current Rating: 10A

Fuse type: 2X T 10A L 250V

DANGER: Because of the high voltages in this instrument, maintenance and repair of internal parts must be performed by TA Instruments or other qualified service personnel only.

DANGER: À cause de la présence de tensions élevées dans cet instrument, la maintenance et la réparation des pièces internes doivent être effectuées uniquement par TA Instruments ou tout autre personnel d'entretien qualifié.

WARNING: Only use a mains power cable that meets the ratings displayed on the back on the instrument being installed/reinstalled.

AVERTISSEMENT: Utilisez uniquement un câble du secteur électrique qui répond à la capacité nominale affichée à l'arrière de l'instrument installé/réinstallé.

Lifting the Instrument

WARNING: Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

AVERTISSEMENT: Demandez à deux personnes de soulever et/ou de porter l'instrument. L'instrument est trop lourd pour qu'une seule personne le manipule en toute sécurité.

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Chapter 1:

Introducing the DTC-300

Overview

The Discovery Thermal Conductivity (DTC-300) instrument is used for measuring the thermal conductivity of materials in the range 0.1 to 30 W/(m.K). These include some metals and ceramics, polymers, composites, glass, rubber, and carbon products. The DTC-300 can also be used to test thin samples like paper products and polymeric films. Non-solids of high viscosity can be tested using a special container to hold the material.

Standard thermal resistance range is 0.002 to 0.02 m²K/W. Optional ranges are 0.0005 to 0.01 and 0.01 to 0.05 m²K/W.

The DTC-300 is configured to test a 50.8 mm diameter sample. The instrument operates from -20 to 300°C—a suitable chiller/circulator is required to test from -20 to 20°C. Chiller setpoint temperatures must be a minimum of 15°C below the desired sample temperature.

Input power options include 100–120 VAC or 200–240 VAC. Please check your instrument and make certain that you have received the option you ordered.

The DTC-300 system (shown below) is comprised of the test stack and electronic enclosure. The instrument is PC-controlled and the operational software is also used to generate tabular and graphical data.

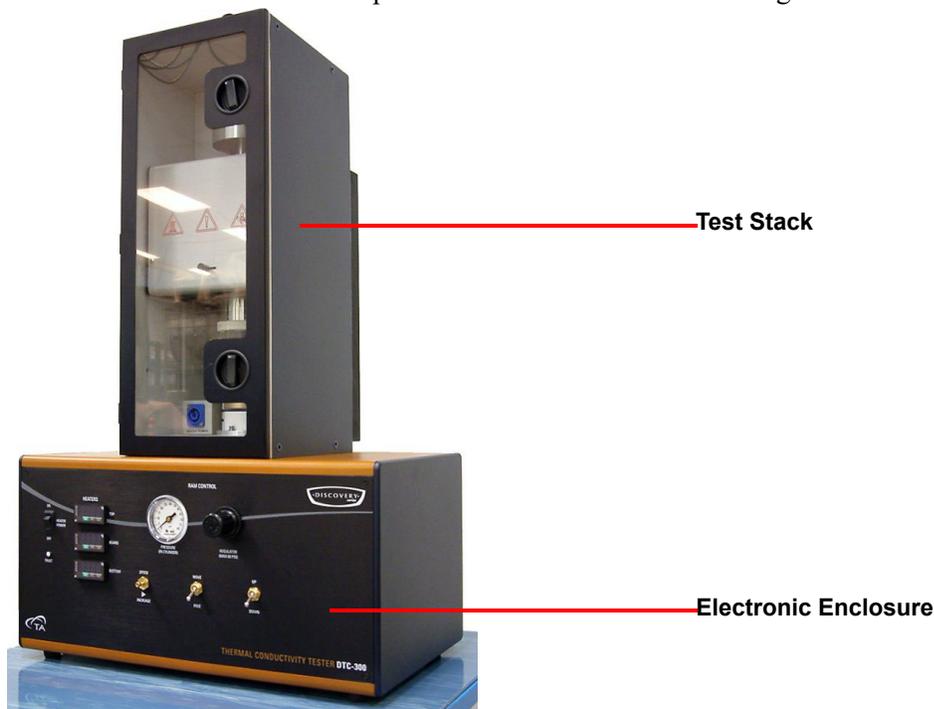


Figure 1 DTC-300 system.

DTC-300 System Components

A DTC-300 system has two major components: DTC-300 electronic enclosure and the test stack.

Electronic Enclosure

The DTC-300 electronic enclosure contains the system electronics used to read and display the output of the temperature sensors.

DTC-300 Front Panel

The heater control for each of the heaters is sensed and automatically adjusted by the software, and can also be adjusted manually using the PID temperature controllers located on the front of the electronic enclosure. The various parameter settings are preset at the factory and should not be adjusted or manipulated without first consulting TA. These parameters are re-initialized at the beginning of each test to ensure proper starting conditions. An independent over-temperature monitor is employed as a safety for the device. The fault indicator on the front panel lights when an over-temperature condition is detected. Pressure applied to the sample can be adjusted using the air pressure regulator and monitored on the built-in pressure gauge. The movement of the test stack is controlled with the **UP/DOWN**, **FREE/MOVE**, and **SPEED** switches.

Refer to Figure 2 below for the locations of knobs, switches, and indicators on the DTC-300 front panel and refer to [Table 1](#) for a description of their functions.



Figure 2 DTC-300 front panel.

Table 1: DTC-300 Front Panel

Indicator	Function
HEATER POWER switch	Powers the heaters ON and OFF
FAULT indicator	Indicates (lights) when an over-temperature condition is detected
Heater temperature display: TOP, GUARD, BOTTOM	Displays current temperature and setpoint for each heater
SPEED/INCREASE knob	Controls the speed at which the top stack moves up or down
MOVE/FREE switch	Activates the top stack pneumatics
UP/DOWN switch	Moves the top stack up/down
Air pressure gauge	Air pressure gauge for monitoring air pressure applied to the top stack
Air pressure regulator knob	Air pressure regulator allows you to adjust the air pressure to the top stack

DTC-300 Back Panel

The DTC-300 back panel has five ports. Figure 3 below shows the rear connections and Table 2 provides a description of the function of each port.

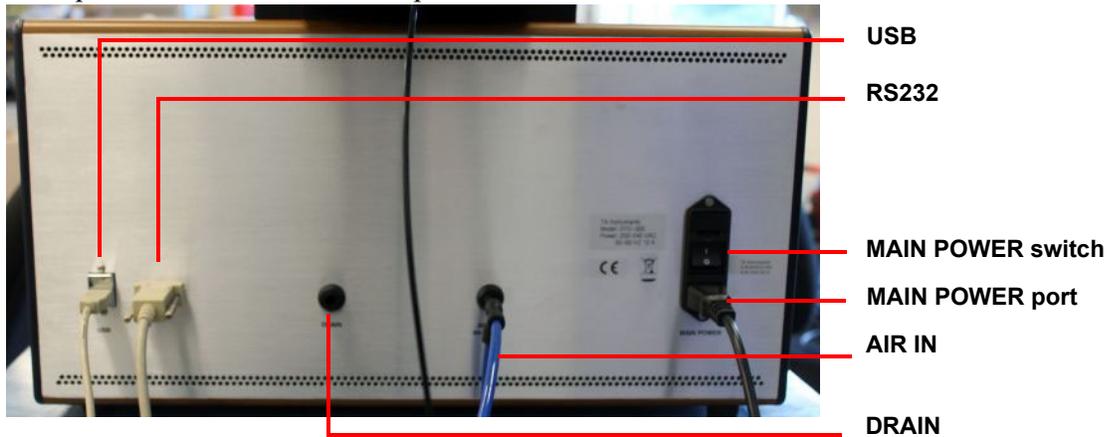


Figure 3 DTC-300 rear panel connections.

Table 2: DTC-300 Back Panel

Port	Function
USB	Provides communications to the Temperature Measurement System
RS232	Provides communication to the PID Heater Control System
DRAIN	Removes condensing water (when needed)
AIR IN	Provides the air or inert gas used to operate the DTC-300 pneumatics
MAIN POWER port	Provides power to the instrument
MAIN POWER switch	Powers the DTC-300 ON (I) / OFF (O)

Test Stack

The sample to be tested is held under a reproducible compressive load between two polished metal surfaces (the “test stack”), each controlled at a different temperature. The lower contact surface is part of a calibrated heat flux transducer. As heat flows from the upper surface through the sample to the lower surface, a uniaxial temperature gradient is established in the stack. By measuring the temperature difference across the sample along with the output from the heat flux transducer, thermal conductivity of the sample can be determined when the sample thickness is known.



Figure 4 Test stack on the DTC-300.

Instrument Specifications

The table below contains the technical specifications for the DTC-300.

Table 3: DTC-300 Technical Specifications

Item/Area	Specifications
Dimensions	Depth 71 cm (28 in) Width 64 cm (25 in) Height 94 cm (37 in)
Weight	44.5 kg (98 lbs)
Power ^{ab}	100–120V (for 110V instrument) or 200–240 VAC (for 220V instrument) 50/60 Hz 10A max current supply Supply voltages lower than indicated limit may result in a degradation of performance.
Fuse Type	2X T 10A L 250V
Operating Environmental Conditions	Temperature: 15 to 35°C Relative humidity: 5 to 80% (non-condensing) Maximum Altitude 2000 m (6560 ft) Locate instrument in a ventilated space (hood, etc.) if noxious gases or vapors are generated during the heating of samples. The degree of protection for this instrument according to EN 60529 is IP20.
Purge Gas System Requirements	Nitrogen Purge Gas^c 1.5 psi (0.1 bar) nitrogen gas furnace flush is required for operating at temperatures below 20°C.
Pneumatic System Requirements	Compressed Air/Inert Gas^d Maximum inlet pressure to the instrument: 60 psi (4.1 bar) Typical operating pressure: 40 psi (2.8 bar). The flow rate is less than 1 L/day. Compressed inert gas or air from a gas cylinder may be used. A proper gas cylinder pressure regulator/reducing regulator is required.
Cooling System Requirements	Chiller/Circulator or Cooling Water and Drain^e Permissible coolant temperatures are between -40 and 20°C, depending upon testing considerations. Excessively cold water may result in “sweating” and possible corrosion of cooled metal surfaces. A nitrogen furnace cavity “flush” is required for sub-ambient operation. Warm water may not permit required cooling and stack temperature gradients during testing.

- a. This instrument is supplied with a power cord approximately 2.4 m (7.75 ft) long. The 220V instrument power cord comes supplied with a USA NEMA 6-15P style plug; the 110V instrument power cord comes supplied with a USA NEMA 5-15P style plug. The TA Representative installing the instrument will provide the appropriate power cable for the geographical location where the instrument is being installed. Ensure that the mains assigned do not also supply power to noise generating equipment nearby, such as motors, welders, transformers, etc.

- b. An independent GROUND is required on the power connection where the instrument is being installed. Improper grounding may cause severe damage for which supplier will not accept responsibility.
- c. The instrument is supplied with a 1.22m (4 ft) length of 3.175 mm (.125 in) tube and a 3.175 mm (.125 in) to 6.35 mm (.25 in) expander fitting to connect the purge gas to the instrument. The customer is required to make all hose connections. A flow meter is required for control of the furnace chamber “flush” gas (not supplied).
- d. The customer is required to supply either compressed air, dried and filtered, to 10 microns, or laboratory grade inert gas (i.e.N₂ from a high pressure cylinder) for operating the pneumatic system.
- e. Operating below the minimum chiller coolant level will result in erratic operation. A chiller/circulator is recommended for this instrument. Consult the chiller/circulator manual for specifications concerning appropriate fluid types. The instrument is supplied with insulated hoses to attach to a chiller/circulator.

Table 4: DTC-300 Performance Specifications

Item/Area	Specifications
Test Method	ASTM E1530
Thermal Conductivity Range	0.1 – 40W/mK
Thermal Resistance Range	Low-Range: 0.0005 – 0.010 m ² K/W Mid-Range: 0.002 – 0.020 m ² K/W Hi-Range: 0.01 – 0.05 m ² K/W
Accuracy	± 3%
Reproducibility	± 2%

Chapter 2:

Installing the DTC-300 System

Unpacking/Repacking

You may wish to retain all of the shipping hardware, the plywood, and boxes from the instrument in the event you wish to repack and ship your instrument.

Installing the System

Before shipment, the DTC-300 is inspected both electrically and mechanically so that it is ready for operation upon proper installation. Installation involves the following procedures:

- Inspecting the system for shipping damage and missing parts
- Choosing a location for instrument installation
- Installing the instrument
- Connecting the utilities

It is strongly recommended that you have your DTC-300 system installed by a TA Instruments Service Representative; call for an installation appointment when you receive your instrument.

CAUTION: To avoid mistakes, read this entire chapter before you begin installation.

MISE EN GARDE: Pour éviter de commettre des erreurs, lisez tout le chapitre avant de commencer l'installation.

Inspecting the System

When you receive your DTC-300 system, look over the instrument and shipping container carefully for signs of shipping damage, and check the parts received against the enclosed shipping list.

- If the instrument is damaged, notify the carrier and TA Instruments immediately.
- If the instrument is intact but parts are missing, contact TA Instruments.

Choosing a Location

Because of the sensitivity of DTC-300 experiments, it is important to choose a location for the instrument using the following guidelines. The DTC-300 system should be:

In

- A temperature and humidity controlled area.
- A clean, vibration-free environment.
- An area with ample working and ventilation space.

On

- A stable, non-flammable work surface.

Near

- A power outlet for the specific voltage of the instrument.
- Compressed lab air and purge gas supplies with suitable regulators and filtering where needed.
- Cooling water/coolant (stable chiller/circulator preferred).

Away from

- Dusty environments.
- Exposure to direct sunlight.
- Direct air drafts (fans, room air ducts).
- Poorly ventilated areas.
- Electrically noisy areas or areas prone to mechanical vibrations.

NOTE: Do not place equipment against walls or cabinets that might impede air flow. Leave at least 7.5 cm (3 in) clearance between the back of the instrument and any objects.



WARNING: For safety, position the equipment in a manner that allows access to the power cord for emergency disconnection.

AVERTISSEMENT: Par mesure de sécurité, placez l'équipement de sorte qu'il permette d'accéder facilement au cordon d'alimentation en cas de débranchement d'urgence.



WARNING: Protect power and communications cable paths. Do not create tripping hazards by laying the cables across access ways.

AVERTISSEMENT: Protégez les chemins de câble électriques et de câbles de télécommunication. Ne créez pas de risques de déclenchement en posant des câbles sur les voies d'accès.

Moving the Instrument



WARNING: Use two people to lift and/or carry the instrument. The instrument is too heavy for one person to handle safely.

AVERTISSEMENT: Demandez à deux personnes de soulever et/ou de porter l'instrument. L'instrument est trop lourd pour qu'une seule personne le manipule en toute sécurité.

Lifting the instrument should always be done with two people. With each person standing on either side of the instrument, place hands between the feet on the bottom of the instrument and lift upwards, keeping the instrument in an upright position.

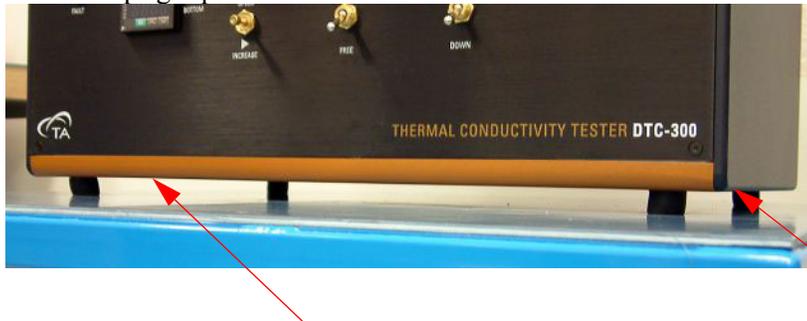


Figure 5 Lifting the instrument.

Connecting the Utilities

Connecting the Power

Plug the power cord from the **MAIN POWER** port on the back of the DTC-300 to an electrical outlet rated in accordance with the power input information on the back of the instrument.



WARNING: Only use a mains power cable that meets the ratings displayed on the back of the instrument being installed/reinstalled.

AVERTISSEMENT: Utilisez uniquement un câble du secteur électrique qui répond à la capacité nominale affichée à l'arrière de l'instrument installé/réinstallé.

CAUTION: Whenever plugging or unplugging power cords, handle them by the plugs, not by the cords.

MISE EN GARDE: Chaque fois que vous branchez ou débranchez les cordons d'alimentation, tenez-les par les fiches et non par les cordons.

Connecting the Air Supply



WARNING: The supply pressure may not be more than 60 psi (4.14 bar)

AVERTISSEMENT: La pression d'alimentation ne peut pas être supérieure à 60 psi (4,14 bar)

- 1 Connect a compressed air supply line to the **AIR IN** connector on the back of the DTC-300 electronic enclosure. To make the connection, firmly push the air line into the connector until it stops. Gently pull on the hose to ensure it is secure in the port.

NOTE: To remove a line, simultaneously push the small ring at the opening towards the connector while pulling the line away from the connector.

- 2 If a hose barb and clamp are supplied, affix the hose barb to an appropriately sized and pressure rated tube, and then insert the hose barb into the connector on the rear of the DTC-300 in the same fashion as described above.
- 3 Set the air pressure initially at 40 psi (2.8 bar) for the operation. The supply air pressure may not be more than 60 psi (4.14 bar).
- 4 Check the air pressure gauge on the front panel and, if necessary, turn the air pressure regulator knob clockwise or counterclockwise to adjust the pressure to the air cylinder. If the regulator knob does not move, make sure the locking ring is not engaged.
- 5 After setting the pressure, lock the knob in position by engaging the locking ring.

Connecting the Drain Hose

The drain is a hose connected to the **DRAIN** port to permit any condensation to be released from the inner collection pan. It should be placed into an open drain (no back pressure). Liquid should be minimal (if any). Connection is in the same manner as the air hose.

Connecting the Coolant

A refrigerated circulator must be connected directly to the heat sink inlet and outlet connections on the rear of the test stack. The flow direction through the heat sink is important. Set the refrigerated circulator temperature at 0–5°C for the sample testing range of 20–100°C, and set the refrigerated circulator temperature at 20°C for the sample testing range of 100–300°C. The minimum sample temperature is approximately 20°C above the coolant temperature.

For -20°C to the 20°C tests, the refrigerated circulator must be set at -40°C to -35°C. For sub-ambient temperature measurements, the sub-ambient insulation ring is used in place of the guard furnace.

The test chamber door must be closed to prevent moisture in the surrounding air from condensing on cold surfaces and purged with nitrogen.

CAUTION: Never operate the DTC-300 without coolant flowing through the heat sink.

MISE EN GARDE: N'utilisez jamais le DTC-300 en l'absence du fluide caloporteur dans le dissipateur de chaleur.

- 1 Insert the plastic fittings attached to the coolant hoses (supplied with the instrument) into the two connectors. The connectors are lock-in-place fittings with shut-offs built into them. Press the male fitting into the female. To remove, depress on the release button and pull the male fitting from the connector.
- 2 Connect a chiller or city water supply directly into the **COOLANT** inlet and outlet connectors on the rear of the instrument.

Chapter 3:

Operating the DTC-300

Using the DTC-300

All of your DTC-300 experiments will have the following general outline. In some cases, not all of these steps will be performed.

- Preparing the sample
- Loading the sample
- Starting the analysis

To obtain accurate results, follow these procedures carefully.

Before You Begin

Before you set up an experiment, ensure that the DTC-300 system has been installed properly. Make sure you have:

- Connected all coolant and pneumatic/purge gas lines
- Connected the communication cables from the rear of the instrument to the control computer
- Powered up the unit
- Become familiar with instrument operations
- Become familiar with the software

Startup and Shutdown Procedures

Starting the DTC-300 System

The power switch is located on the back panel of the DTC-300. The power switch is used to turn the DTC-300 system on and off.

To power on the system:

- 1 Check that the power cord is plugged into the **MAIN POWER** socket on the rear panel of the DTC-300 and connected to an electrical outlet in accordance with the electrical ratings on the back panel of the instrument.
- 2 Set the **HEATER POWER** switch on the front panel to the OFF (0) position.
- 3 Set the DTC-300 **MAIN POWER** switch to the ON (I) position.
- 4 The temperature controllers on the front panel indicate that main power to the instrument is active.

Shutting Down the DTC-300 System

Before you decide to power down your system, consider the following:

- All of the components of your thermal analysis system are designed to be powered on for long periods.
- The electronics of the DTC-300 perform more reliably if power fluctuations caused by turning units on and off are minimized.
- The over-temperature protective system is only active while the instrument is powered on.

For these reasons, turning the system and its components on and off frequently is discouraged. Therefore, when you finish running an experiment on your instrument and wish to use the thermal analysis system for some other task, it is recommended that you leave the instrument on.

CAUTION: Do not power off the chiller during instrument operation. The top stack, guard furnace, and bottom stack must be below 50°C. Failure to do so may result in damage to the instrument.

To power down your system, set the DTC-300 **MAIN POWER** switch to the OFF (0) position.

Running a DTC-300 Experiment

Sample Requirements

Test samples must be prepared with smooth and flat surfaces on both sides. Dense, rigid samples must be tested with thermal compound applied to both surfaces, to minimize the thermal resistance of the interface between the sample and the instrument. This might not be appropriate for porous materials, since the thermal compound may penetrate the sample, thereby changing its properties. Thin, flexible samples, like polymeric films and papers, should also be tested without thermal compound.

NOTE: In cases when the sample needs to be tested without using thermal compound, a special calibration must be performed, in the same conditions.

Samples may be up to 20 mm (0.75 in) thick. Unless the sample material is a product having a fixed thickness, e.g. plastic film or paper, the optimum sample thickness for obtaining high test accuracy can be calculated from an estimate of the sample thermal conductivity:

$$d < 50 * \lambda \text{ [mm]}$$

$$\text{and } d > 0.5 * \lambda \text{ [mm]}$$

In these expressions, enter for λ the expected thermal conductivity in W/mK.

Low-Temperature Testing

Loading the Sample



WARNING: Keep fingers away from the test section when lowering the upper stack with the pneumatic controls. Serious personal injury may occur if fingers are accidentally pinched between the upper and lower stack parts.

AVERTISSEMENT: Éloignez vos doigts de la section d'essai lorsque vous abaissez la cheminée supérieure à l'aide de commandes pneumatiques. De graves blessures corporelles risquent de se produire si les pièces de la cheminée supérieure et inférieure pincent vos doigts par accident.

The following is the procedure for applying thermal heat sink compound and loading it into the instrument:

NOTE: Thermal heat sink compound is difficult to remove from clothing and skin, so take care when handling the compound. Plastic gloves may be worn during application.

NOTE: The stack **MOVE/FREE** switch may be used at any time to remove the downward pressure on the sample, ie. to move or rotate the sample after the upper stack has been lowered. Place the toggle switch in the **FREE** position to remove the load. Always keep the switch in the **MOVE** position while testing.

- 1 Turn the instrument on by pressing the main power switch on the rear panel. Check that coolant is flowing through the heat sink.
- 2 Check that the air pressure reading on the gauge is 30–50 psi. If necessary, adjust the air pressure.
- 3 Accurately measure and record the thickness of the sample that will be tested.
- 4 Thoroughly clean the sample faces, top sample plate, and bottom sample plate using isopropyl alcohol and lint-free wipes.
- 5 Apply a small volume of thermal compound (approximately .25” diameter and .25” high) to the center on each face of the sample.
- 6 Make sure the **SPEED/INCREASE** switch is in the off position. Then put the sample onto the bottom stack and lower the top stack slowly using the **SPEED/INCREASE** switch until the stacks press against the sample.
- 7 Assure that the sample is in line with the top and bottom sample plates and continue to increase the pressure using the **SPEED/INCREASE** switch until thermal compound no longer squeezes out from the side of the sample.
- 8 Wipe away any excess compound from around the sample.
- 9 Lower the guard furnace, latch it in place with the spring loaded retaining pin, and begin testing.

Testing the Sample

- 1 Turn the instrument on by pressing the main power switch on the rear panel. Check that coolant is flowing through the heat sink.
- 2 Set the **HEATER POWER** switch to **ON**.
- 3 Raise the heater assembly with the **UP/DOWN** switch. The **MOVE/FREE** switch must be in the **MOVE** position.
- 4 Accurately measure the sample thickness and, if appropriate, apply a thin layer of sink compound (supplied with the instrument) to **both** sides of the sample.

NOTE: Contact TA Instruments if you would like to obtain calibration samples.

- 5 Place the sample on the lower surface plate of the test column and align the sample.
- 6 Lower the heater assembly with the **UP/DOWN** switch. (When a dead weight mechanism is provided instead of the pneumatic system, unlock the shaft and gently lower the heater assembly until it rests on the sample. Then place a weight on the support tray to obtain the desired compression of the sample).
- 7 Release the spring-loaded locking pin and lower the guard furnace. Lock the furnace in the lowest position.
- 8 Close the front door and secure the latches.
- 9 Input the testing information and follow the on-screen programming.

High-Temperature Testing



WARNING: During high-temperature calibration or a multiple sample test, do not raise the guard furnace to load the next sample until the furnace is below 50°C.

Loading the Sample



WARNING: Keep fingers away from the test section when lowering the upper stack with the pneumatic controls. Serious personal injury may occur if fingers are accidentally pinched between the upper and lower stack parts.

AVERTISSEMENT: Éloignez vos doigts de la section d'essai lorsque vous abaissez la cheminée supérieure à l'aide de commandes pneumatiques. De graves blessures corporelles risquent de se produire si les pièces de la cheminée supérieure et inférieure pincent vos doigts par accident.

The following is the procedure for applying thermal heat sink compound and loading it into the instrument:

NOTE: Thermal heat sink compound is difficult to remove from clothing and skin, so take care when handling the compound. Plastic gloves may be worn during application.

NOTE: The stack **MOVE/FREE** switch may be used at any time to remove the downward pressure on the sample, i.e. to move or rotate the sample after the upper stack has been lowered. Place the toggle switch in the **FREE** position to remove the load. Always keep the switch in the **MOVE** position while testing.

- 1 Turn the instrument on by pressing the main power switch on the rear panel. Check that coolant is flowing through the heat sink.
- 2 Check that the air pressure reading on the gauge is 30–50 psi. If necessary, adjust the air pressure.
- 3 Accurately measure and record the thickness of the sample that will be tested.
- 4 Thoroughly clean the sample faces, top sample plate, and bottom sample plate using isopropyl alcohol and lint-free wipes.
- 5 Apply a small volume of thermal compound (approximately .25” diameter and .25” high) to the center on each face of the sample.
- 6 Make sure the **SPEED/INCREASE** switch is in the off position. Then put the sample onto the bottom stack and lower the top stack slowly using the **SPEED/INCREASE** switch until the stacks press against the sample.
- 7 Assure that the sample is in line with the top and bottom sample plates and continue to increase the pressure using the **SPEED/INCREASE** switch until thermal compound no longer squeezes out from the side of the sample.
- 8 Wipe away any excess compound from around the sample.
- 9 Lower the guard furnace, latch it in place with the spring loaded retaining pin, and begin testing.

Testing the Sample

- 1 Turn the instrument on by pressing the main power switch on the rear panel. Check that coolant is flowing through the heat sink.
- 2 Set the **HEATER POWER** switch to **ON**.
- 3 Raise the heater assembly with the **UP/DOWN** switch. The **MOVE/FREE** switch must be in the **MOVE** position.
- 4 Accurately measure the sample thickness and, if appropriate, apply a thin layer of sink compound (supplied with the instrument) to **both** sides of the sample.

NOTE: Contact TA Instruments if you would like to obtain calibration samples.

- 5 Place the sample on the lower surface plate of the test column and align the sample.
- 6 Lower the heater assembly with the **UP/DOWN** switch. (When a dead weight mechanism is provided instead of the pneumatic system, unlock the shaft and gently lower the heater assembly until it rests on the sample. Then place a weight on the support tray to obtain the desired compression of the sample).
- 7 Release the spring-loaded locking pin and lower the guard furnace. Lock the furnace in the lowest position.
- 8 Close the front door and secure the latches.
- 9 Input the testing information and follow the on-screen programming.

Sub-Ambient Temperature Testing

Loading the Sample



WARNING: Keep fingers away from the test section when lowering the upper stack with the pneumatic controls. Serious personal injury may occur if fingers are accidentally pinched between the upper and lower stack parts.

AVERTISSEMENT: Éloignez vos doigts de la section d'essai lorsque vous abaissez la cheminée supérieure à l'aide de commandes pneumatiques. De graves blessures corporelles risquent de se produire si les pièces de la cheminée supérieure et inférieure pincent vos doigts par accident.

- 1 Move the guard furnace to the **UP** position and lock it in place.
- 2 Place the bottom cooling block insulation around the bottom cooling block.
- 3 [Load the sample.](#)
- 4 Separate the two halves of the sub-ambient insulation ring.
- 5 Place the half without the clasps, in and around the stack.
- 6 Rotate the half until the left clip is exposed.
- 7 Place the front half on the stack and clamp it onto the left clip.
- 8 Rotate the halves back around until the right clip is exposed. Clamp the right clasp.
- 9 Close the front door and secure the latches.

- 10 Turn the chiller on to the desired temperature. For a -20°C approximate mean sample testing temperature, the chiller should be set to -40°C or colder.

Testing the Sample

- To setup the DTC-300 to test at sub-ambient temperatures the following items are needed:
 - The sub-ambient insulation ring
 - A tank of dry inert gas
 - A chiller and coolant capable of sub-ambient temperature operation, (-35 to -40°C).
 - If coolant needs to be emptied out of the chiller, then a funnel and/or an empty container(s) are necessary.
- 1 Prepare a sufficient amount of empty containers to handle the coolant to be removed from the chiller bath. Empty the coolant into the containers using a funnel. The below figure shows a chiller with a drain plug being emptied into a container. It is recommended to mark the container “Used Coolant” to avoid any confusion at a later time.
 - 2 The coolant inside of the DTC-300 stack and lines also need to be emptied. Disconnect both coolant lines from the back of the chiller. Place one line into a container and blow through the other line using an external air supply. A small amount of coolant left in the chiller or unit is acceptable.
 - 3 Re-connect the coolant lines to the chiller. **COOLANT OUT** from the unit to **COOLANT IN** on the chiller. **COOLANT IN** from the unit to the **COOLANT OUT** on the chiller.
 - 4 Fill the chiller bath with the adequate coolant specified by the manufacturer of the chiller being used for testing temperatures near -20°C (chiller temperature set at -35 to -45°C).
 - 5 Open the front cover of the unit. Lift the guard heater to the top locking position.
 - 6 Load the sample. Refer to the section [Loading the Sample](#) for instructions.

Unknown Sample Testing

To perform a thermal conductivity measurement on an unknown sample:

- 1 Check that the **HEATER POWER** switch on the front panel is **OFF** (no red is visible on the switch). Activate the instrument by pressing the main power switch next to the power input socket in the rear panel. Check that there is coolant flowing through the heat sink.
- 2 Check the air pressure reading on the gauge. If necessary, adjust the air pressure.
- 3 Release the latches and open the front door. Raise the guard furnace and lock it in the elevated position, thus providing access to the test stack. Raise the upper heater assembly by toggling the **UP/DOWN** switch.
- 4 Determine which stack spacer should be used to obtain the desired reference temperatures for the upcoming test, and place the appropriate spacer in the instrument’s stack.
- 5 Measure and record the sample’s thickness and apply a small amount of thermal compound to both of its faces.
- 6 Place the sample on the exposed surface of the bottom heater assembly and align it with the lower stack.
- 7 Lower the top heater assembly with the **UP/DOWN** switch. Check to make sure that the sample is centered between the two metallic surfaces and realign it, if necessary. Remove any excess thermal

compound from the edges of the sample with a cloth or tissue paper. (If the sample needs to be realigned, first place the **FREE/MOVE** switch to the **FREE** position. This removes the air pressure from the cylinder and eliminates the compressive load on the specimen. Realign the sample and then place the switch back in the **MOVE** position.)

- 8 Release the spring-loaded locking pin and lower the guard furnace. Lock the furnace in the lowest position. Close the front door and secure the latches.
- 9 Input the testing information and follow the on-screen programming.

Special Sample Testing

Thin Film (Multiple) Sample Testing

Thin samples such as polymeric films, paper products, and elastomeric insulating pads often have a thermal resistance that falls below the minimum value of $0.0005 \text{ m}^2\text{K/W}$ that is recommended for accurate testing with the DTC-300. By stacking several layers of the sample material, the thermal resistance can be raised above the $0.0005 \text{ m}^2\text{K/W}$ limit and the thermal conductivity may then be calculated. A special software is offered for this application.

The stack should contain as few layers as possible, preferably less than 7, as each additional layer introduces another physical interface in the stack. The method relies on the fact that the interface resistance between individual specimen layers is small compared to the thermal resistance of the material itself, and can therefore be ignored. For thin, conforming materials of relatively low thermal conductivity, this is a valid assumption.

For testing thin samples of higher thermal conductivity (ceramic, metals, graphite, etc.), refer to the section "[Testing the Sample](#)."

Select the appropriate testing option below:

- Test one layer of material, using the [Testing the Sample](#) procedure. If the thermal resistance of this specimen is within the calibration range of the instrument, there is no need to use the thin films technique, and the result of the test can be reported. If the thermal resistance of one layer is lower than the minimum value of the instrument's calibration range, the testing process can continue by testing two, three, four and five layers of material, stacked. It is recommended to have at least four different numbers of layers of material tested.

NOTE: No thermal compound should be used between the layers of samples or between the samples and the instrument. Since the thermal resistance of these interfaces will be considered negligible, the number of samples stacked should be reduced to the minimum necessary. Testing up to five or six layers is usually sufficient. In cases when the layers cannot be stacked without sliding, and only for non-porous materials, a very small amount of thermal compound may be placed between the layers.

NOTE: When using the thin films technique, the thermal conductivity values displayed on the screen at the completion of each test are not to be considered as the final results of the measurements. The thermal resistance of each stack tested and its thickness will be used in the program for calculating the material's thermal conductivity.

NOTE: When choosing the names for these individual tests, pick related names, so that it will be easy to identify which files to use when performing the analysis (for example: Film1 - one layer; Film2 - two layer; Film3 - three layers. etc.).

- For very thin samples having a tendency to stick to each other under compression, though minimizing the thermal resistance between them, higher numbers of layers can be tested together (for example: four, five, six, seven, etc.), forming a new sample that may be treated as a solid piece, with its thermal resistance being within the instrument's operating range. The total thickness of the test sample should be considered for the thermal conductivity calculations, paying attention to use the value of the thickness measured under compression, and not the sum of the individual layers' thicknesses.

- The sample can be stacked on a reference material and tested together with it. It is recommended that one of the reference materials used for the instrument's calibration should be considered for this particular application. The reference material has to be tested first by itself, and then together with the sample. The difference between the two thermal resistance values measured represents the sample's thermal resistance. Knowing the thermal resistance and the thickness, the thermal conductivity can be calculated.

Non-Solid Sample Testing

The DTC-300 can be used to measure the thermal conductivity of non-solid samples with optional test cells, available as instrument accessories, if needed. This allows testing of pastes, gels, liquids, polymers through-the-melt, and other highly viscous substances in the range 0.1 to 1.0 W/(m.K).

Each cell has its own construction for the containment of the sample to be tested during operation. The paste cell and polymer melt cell are described in the following sections, but the liquid cell follows the same basic operating principle.

Before testing non-solid samples with the paste cell, the DTC-300 must be calibrated with reference materials placed inside the cell. Regular instrument calibrations, obtained without the paste or melt cell, cannot be used when testing non-solids with the cell. Calibration references for the paste and melt cells are solid samples of known thermal conductivity similar to those used for normal equipment calibration.

Paste Cell Testing

The paste cell consists of two thermally conducting metal plates separated by a set of insulating rings. The inner ring is a spacer to keep the metal plates at a fixed distance. The outer ring is primarily a container for the specimen material. Each cell is provided with two sets of rings including a 0.2" (5.08 mm) spacer and a 0.4" (10.16 mm) spacer.

If the expected thermal conductivity of the sample material is between 0.1 and 0.7 W/(m.K), use the 0.2" spacer in the test cell. If the expected thermal conductivity is between 0.4 and 1.0 W/(m.K), use the 0.4" spacer.

Follow the [procedure for a normal test without the cell](#), except that, in this case, the paste cell with test sample must be placed in the DTC-300. Follow the instructions below for installing non-solid material in the cell.

- 1 The paste cell consists of 4 separate components: The bottom plate, outer ring, inner ring and top plate. Select a set of rings based on the expected λ of the sample material. Accurately measure and record the thickness of the inner ring (spacer).
- 2 Hold the outer ring with its rim toward the bottom. Insert the bottom metal plate (flange side facing up) in the outer ring and slide the plate all the way down until the flange comes to rest against the lower rim. Insert and lower the inner ring (spacer) until it reaches the metal plate.
- 3 Fill the space inside the inner ring with non-solid sample material until it rises slightly above the level of the inner ring. Put slightly more material in the center of the cell than toward the edges. Stir the material to make sure that there are no bubbles and voids.
- 4 Insert the upper metal plate (the one with the small holes along the perimeter) in the cell and push it down until it makes contact with the spacer.

NOTE: The holes in the plate are drilled at a small angle. The side of the plate where the holes are farthest apart is the top. The below images show the top of the plate on the left and the bottom of the plate on the right.

- 5 As the plate is pushed down, excess sample material is expelled from the cell through the small bleed holes. Remove the excess sample material.

NOTE: If the top surface of the metal plate ends up below the top of the outer ring, you have chosen the wrong outer ring. The top metal surface must always be above the outer ring.

- 6 Hold the cell by the outer ring and apply thermal compound to the exposed top and bottom metal surfaces.
- 7 Place the cell on the bottom plate of the DTC-300.
- 8 The specimen thickness entered under "Sample Information" of the DTC-300 operating program is the thickness of the spacer measured in step 1 above. Be sure that the calibration selected for testing with the paste cell is one obtained with the cell in the test chamber and not one for testing solid specimens without the cell.

Polymer Melt Cell Testing

The polymer melt cell is used to test a polymeric material from a solid through a molten/semi-molten state. The cell is comprised of several components, which when assembled will contain the molten polymer, preventing damage to the instrument. The maximum temperature of use is 300°C.

Loading a Sample into the Polymer Melt Cell

The polymer melt cell incorporates 6 parts: the cell containment ring, two consumable split seal rings, an inner spacer ring (also a consumable part), and both a top and a bottom plate.

The top and bottom plates are made of a stainless steel alloy to prevent any contamination of/interaction with the specimen being tested. The faces of the plates will be in direct contact with the specimen and also with the stack assemblies of the conductivity meter. Any scratches or gouges in the surfaces can lead to a decreased accuracy of the results. These parts are re-usable.

The split rings and the inner spacer ring are made from a dense insulation material. They are fragile, and must be treated with care. They can be re-used with some care, but are considered consumable and may require replacement after testing. Inspect each part to guarantee the channels in the split rings and the grooves in the spacer rings do not show any signs of damage prior to loading the cell. They are low conductive materials to prevent any radial heat issues from developing during the test.

The outer containment ring is used to hold the entire assembly together. It is made from an aluminum alloy, and can be used repeatedly. The split rings are a snug fit inside the containment ring, so exercise caution when sliding them inside.

- 1 Begin with the outer containment ring. Place it with the lip on the bottom.
- 2 Next, locate the two split seal rings. The top of the rings is machined flat, while the bottom of the rings has an offset shoulder machined into the insulation. Place the rings together, and carefully insert them into the outer containment ring.
- 3 The bottom shoulder should align with the inside of the lip on the containment ring, and should not extend beyond the ring; it should be flush with the bottom of the containment ring. Carefully press down on the split rings to seat them in the containment ring. Once the split rings are in place, take the split ring assembly and place it onto the bottom plate. The bottom plate has a groove milled into the face. The groove must point upwards. The groove is used to properly position the inner spacer ring.
- 4 Gently press down on the split rings to seat the bottom plate. The bottom plate will extend approximately 1/8" (3 mm) below the bottom of the split rings.

NOTE: For the testing of polymers through the melt, it has been found from experimentation that once re-solidified, the molten polymer tends to adhere to the surfaces of the split ring. In an attempt to reduce this and extend the life of the split rings, a very thin layer of heat transfer compound is spread in the groove of the split rings. This helps during the removal process.

- 5 Next, insert the inner spacer ring into the groove in the bottom plate. Note that there are four grooves milled into the top. These must be exposed (up) to permit the excess molten material to flow into the split rings. Rotate the inner spacer ring so that the grooves in the top are between the cuts in the split rings.
- 6 Load the sample into the cell. It must fit inside the inner spacer ring. The specimen diameter must be 1.5" (38 mm). The specimen must be 0.2" in thickness. This will seat it even with the top of the spacer ring, so that the top plate will contact it properly.
- 7 Place the top plate onto the specimen surface. It should slide between the split rings and rest on the specimen.
- 8 Load the polymer melt cell into the conductivity meter. Apply a small volume of thermal compound (approximately .25" diameter and .25" high) to the center one each face of the sample. Then insert the cell into the instrument, centering the cell plates on the top and bottom stacks.

Unloading the Sample



WARNING: Do not raise the guard furnace until it is below 50°C. Failure to do so may result in personal injury and/or damage to the instrument.

Open the front door and raise the guard furnace and lock it in the elevated position. To raise the top stack assembly, place the **MOVE/FREE** switch to the **FREE** position, and the **UP/DOWN** switch to **UP**. Gently “break” the seal between the upper stack and the sample, then switch the **MOVE/FREE** switch to **MOVE**. Remove the sample, and clean the contact surfaces with soft lint-free paper or cloth to remove any leftover heat sink compound. If necessary, use laboratory alcohol to remove excessive compound.

Running a Conductivity Test

After the sample is loaded into the test stack, the operating test software must be programmed. Access the analysis software to create or choose the test type as well as enter sample and instrument information.

- 1 Open the software.
- 2 Click **Operation**. Select **Startup Information** from the **Operation** menu

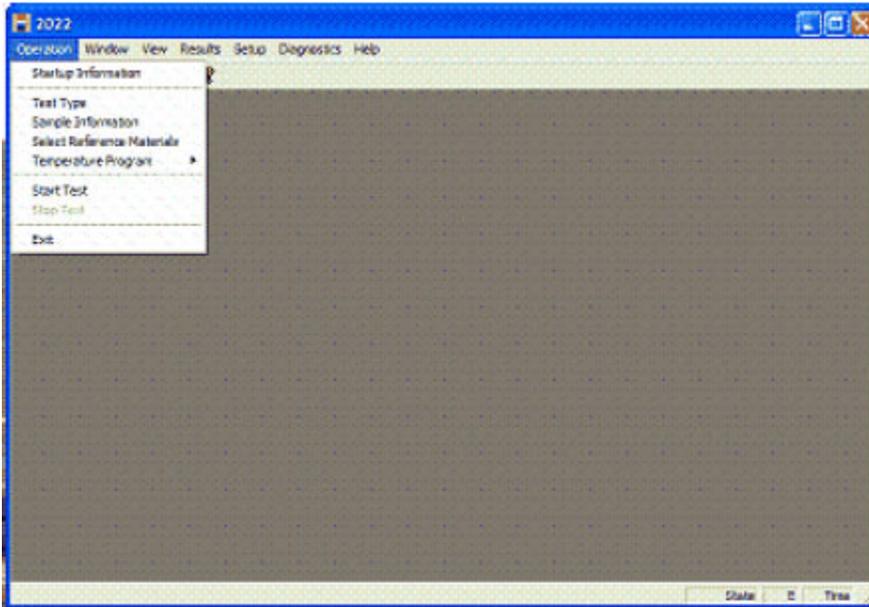


Figure 6 Operation menu.

- 3 Click **OK** on the Selected "2022" pop-up box that displays.



Figure 7 Selected "2022" pop-up box.

- 4 Click **Yes** on the Using Controller Parameters File pop-up box that displays.



Figure 8 Using Controller Parameters File pop-up box.

- 5 The Test Type window displays. Select **Conductivity** and then click **OK**

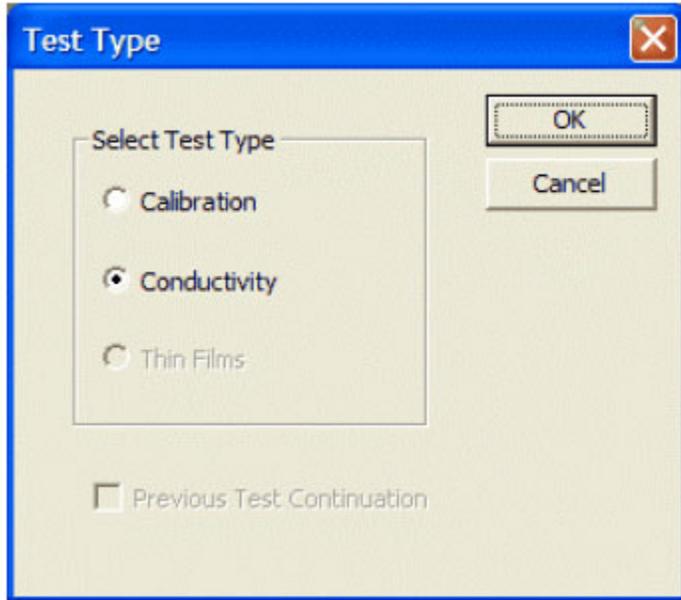


Figure 9 Test Type window.

- 6 The Sample Information window displays. Enter the information and click **OK**.

- **Title:** The test title appears on results and graphs
- **Sample/File ID:** Must be unique for each test; the software creates the result and graph files from this entry
- **Operator:** Enter your name or initials
- **Test Number:** Temperature and Command diagnostic files are created using this number, which TA Instruments personnel use to determine possible errors. Must be unique for each test.
- **Length (mm):** Enter the thickness of the sample.

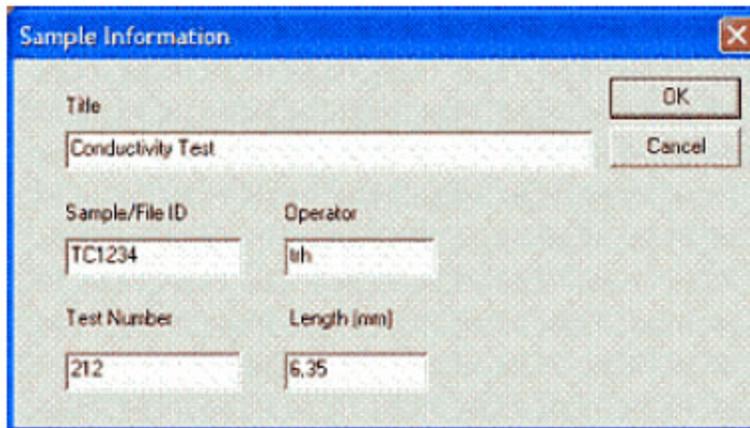


Figure 10 Sample Information window.

- 7 The Temperature Program Titles window displays. This window allows you to choose an existing temperature program or create a new program. Choose a temperature program and then click **OK**, or click **New** to create a new program.

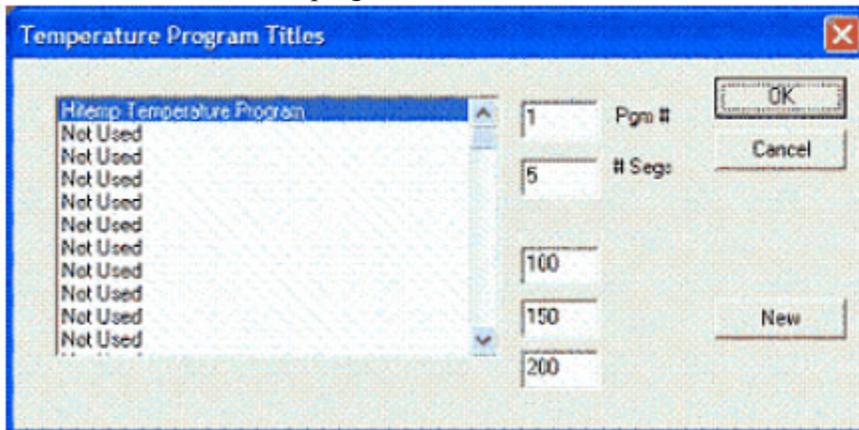


Figure 11 Temperature Program Titles window.

- 8 The Temperature Program window displays. If a saved temperature program is selected, the entry fields will show temperature points to be used for this test. Click **OK**.

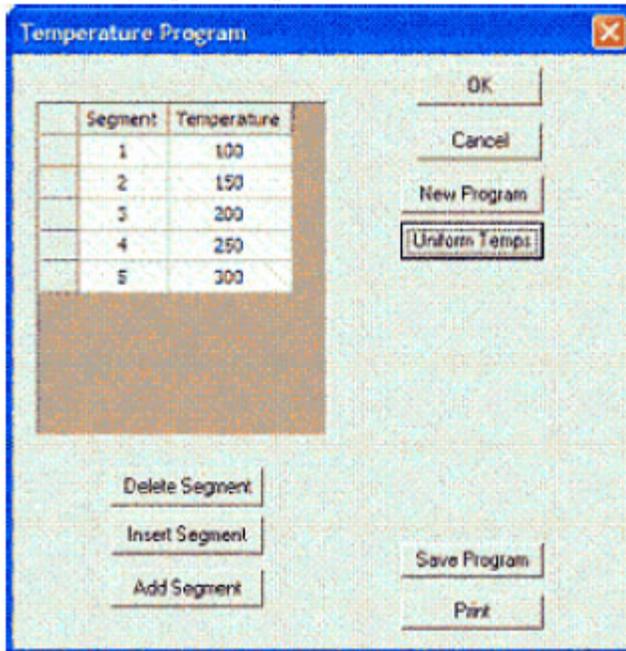


Figure 12 Temperature Program window.

- 9 The Calibration Titles window displays. Select the calibration file to use for the conductivity test and then click **OK**.

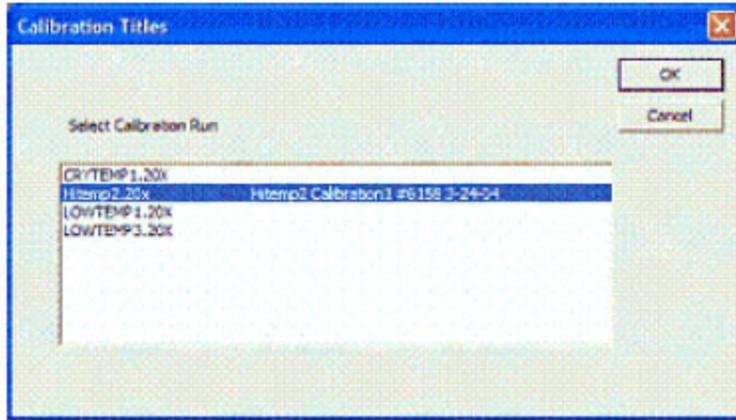


Figure 13 Calibration Titles window.

- 10 The Safety Temperature window displays. Enter the abort temperature. This is a safety feature that will stop the test if any heater reaches the temperature value entered in this window. Click **OK**.

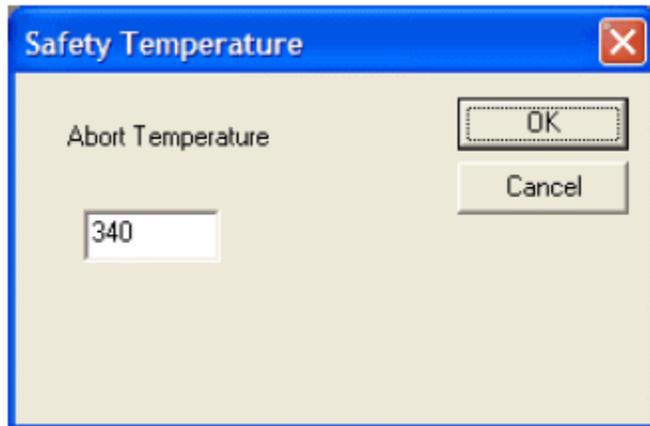


Figure 14 Safety Temperature window.

- 11 A Start Test dialog box displays. Click **Yes** to start the test. If **No** is selected, then the test must be started by clicking **Start Test** in the **Operation** menu.
- 12 You are prompted to make sure the hardware and chiller are on. Click **OK**.

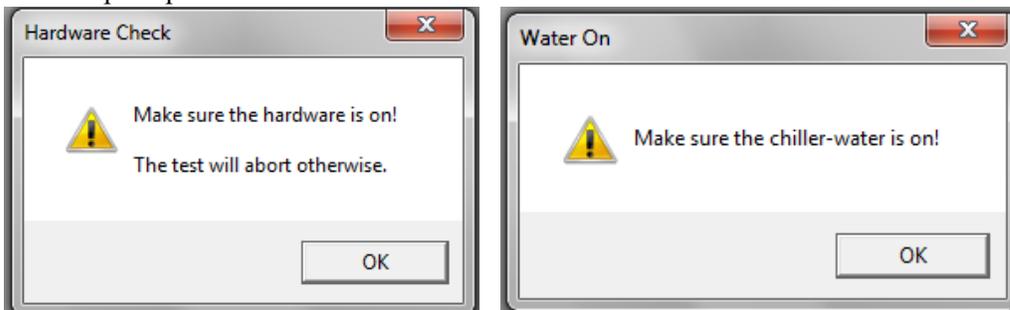


Figure 15 Hardware Check pop-up box and Water On pop-up box.

- 13 You are warned not to start any programs while the test is running. Click **OK**.

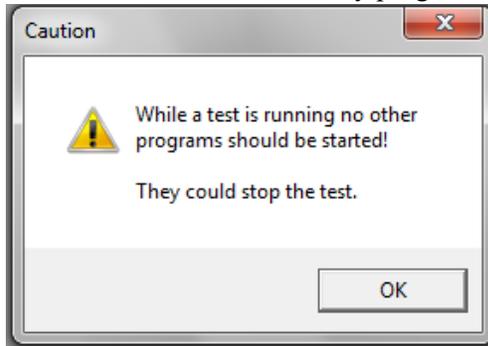


Figure 16 Caution pop-up box.

Stopping a Test

If for some reason you need to discontinue the analysis, you can stop it at any point by clicking the **Operation** menu in the software and then selecting **Stop Test**.

Analyzing a Completed Test

Test data is analyzed using a post-test auxiliary program. The software can also be used to review older tests.

Reviewing the Results

- 1 Click **Results** and select **Conductivity**.

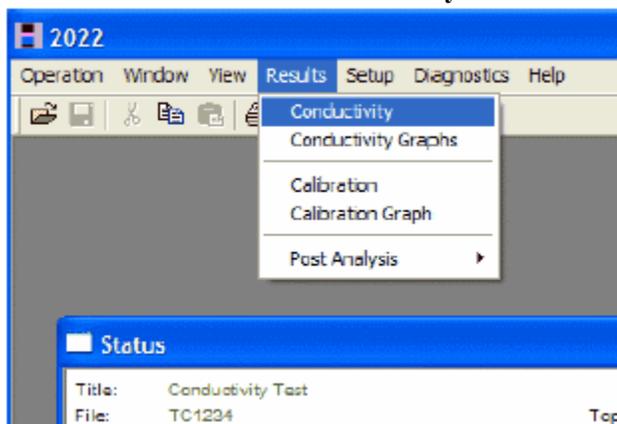


Figure 17 Results menu.

- 2 A window displays the test information and the Conductivity results. Scroll up or down to see all of the information.
- 3 View the graph results by clicking **Results > Conductivity Graphs**. Right-click the graph to display a menu that allows you to change some of the parameters.

Reanalyzing the Test Results

In some cases, there is a need to reanalyze the results of a thermal conductivity test, using a different calibration file. For example, if the instrument has two calibrations, one for a lower thermal resistance range, and one for a higher range, both calibrations being performed with the same heat flow meter module (reference calorimeter), it could happen that the thermal resistance of the unknown sample falls outside the region of the calibration file chosen, but would have been well within the range of the other file. Instead of re-running the test using the second calibration file, the results can be reanalyzed, saving you considerable time.

- 1 Click **Results > Post Analysis > Reanalyze a Conductivity Test**.
- 2 When prompted, select the Conductivity test file to be analyzed.
- 3 In the Sample Information window, modify the sample information, if needed.

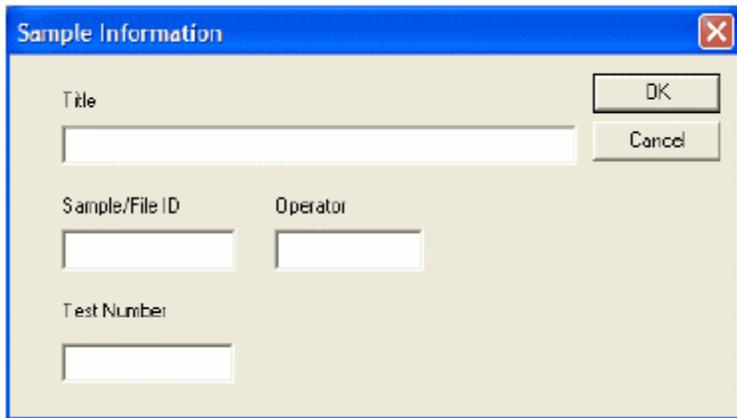


Figure 18 Sample Information window.

- 4 If the Sample/File ID field is left unchanged, you may be asked to overwrite the existing file. Click **Yes** to overwrite the file or **No** to terminate reanalysis.
- 5 The reanalyzed Conductivity test results display.

Manually Creating a Conductivity Test

This option may be used to simulate a test, but also to modify an erroneous entry into the test program (the sample's thickness, for example).

- 1 Click **Results > Post Analysis > Manual Conductivity Entry**.
- 2 When prompted, select the calibration file with which to analyze the newly created Conductivity test.
- 3 In the Sample Information window, enter the sample information and then click **OK**.

- 4 The Setpoint window displays. Enter the number of setpoints for the created Conductivity test. and click **OK**.

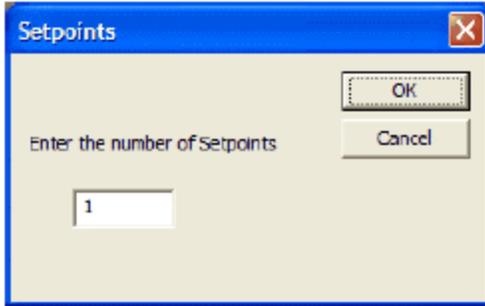


Figure 19 Enter setpoints.

- 5 Enter the temperatures and thickness for all setpoints.

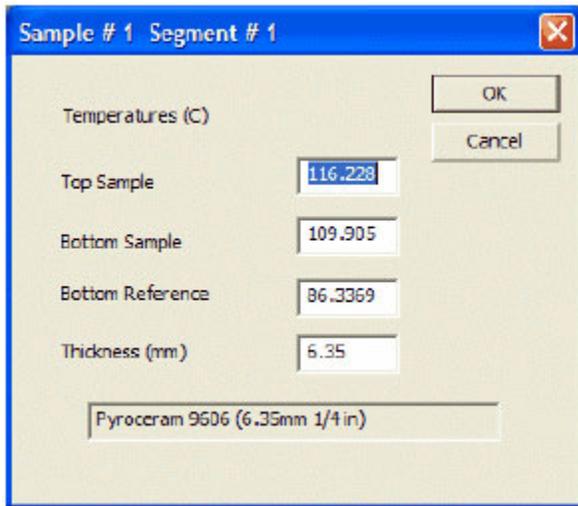


Figure 20 Setpoint information.

- 6 A Conductivity Changes window displays, asking if you would like to create a new file to save the changes. Click **Yes** to display the newly created Conductivity test results. Click **No** to terminate the created Conductivity test.

Chapter 4:

Calibrating the DTC-300

NOTE: At least three reference specimens are required to calibrate the instrument. Available references span the specimen resistance range (R_s) from 0.0005 to 0.05 m²K/W. The references should be within the thermal resistance range of the stack provided and cover any expected test specimen resistance.

Calibration Procedures for Low Temperatures (20°C to 100°C)

NOTE: Make certain to use the copper spacer and insulation for the lower heat sink.

- 1 Check that the **HEATER POWER** switch on the front panel is **OFF** (no red is visible on the switch).
- 2 Activate the instrument by pressing the main power switch next to the power input socket in the rear panel.
- 3 Check that there is coolant flowing through the heat sink.
- 4 Check the air pressure reading on the gauge. If necessary, adjust the air pressure.
- 5 Open the front door. Raise the guard furnace and lock it in the elevated position, thus providing access to the test stack.
- 6 Raise the upper heater assembly by toggling the **UP/DOWN** switch.
- 7 Determine which stack spacer should be used to obtain the desired reference temperatures for the upcoming test, and place the appropriate spacer in the instrument's stack.
- 8 Measure and record the thickness of the first calibration reference you plan to test. Apply a small volume of thermal compound (approximately .25" diameter and .25" high) to the center on each face of the sample.
- 9 Place the calibration reference on the exposed surface of the bottom heater assembly and align the reference with the lower stack.
- 10 Lower the top heater assembly with the **UP/DOWN** switch. Check to make sure that the reference is centered between the two metallic surfaces and realign the reference if necessary. Remove any excess thermal compound from the edges of the reference with a cloth or tissue paper. (If the reference needs to be realigned, first place the **FREE/MOVE** switch to the **FREE** position. This removes the air pressure from the cylinder and eliminates the compressive load on the reference. Realign the reference and then place the switch back in the **MOVE** position.)



WARNING: Keep fingers away from the test stack when activating the DOWN switch. Severe personal injury may result otherwise.

AVERTISSEMENT: Éloignez vos doigts de la cheminée d'essai lors de l'activation de l'interrupteur BAS. Il peut en résulter de graves blessures dans le cas contraire.

- 11 Release the spring-loaded locking pin and lower the guard furnace. Lock the furnace in the lowest position.
-

- 12 Close the front door.
- 13 Input the calibration information and follow the on-screen programming.
- 14 When prompted, change the calibration reference to the appropriate one and resume the calibration test.
- 15 Continue until all calibration references being used have been tested.

Calibration Procedures for High Temperatures (100°C to 300°C)

NOTE: Make certain to use the Vespel spacer and no foam insulation cover for the lower heat sink.

- 1 Check that the **HEATER POWER** switch on the front panel is **OFF** (no red is visible on the switch).
- 2 Activate the instrument by pressing the main power switch next to the power input socket in the rear panel.
- 3 Check that there is coolant flowing through the heat sink.
- 4 Check the air pressure reading on the gauge. If necessary, adjust the air pressure.
- 5 Open the front door. Raise the guard furnace and lock it in the elevated position, thus providing access to the test stack.
- 6 Raise the upper heater assembly by toggling the **UP/DOWN** switch.
- 7 Determine which stack spacer should be used to obtain the desired reference temperatures for the upcoming test, and place the appropriate spacer in the instrument's stack.
- 8 Measure and record the thickness of the first calibration reference you plan to test. Apply a small volume of thermal compound (approximately .25" diameter and .25" high) to the center on each face of the sample.
- 9 Place the calibration reference on the exposed surface of the bottom heater assembly and align the reference with the lower stack.
- 10 Lower the top heater assembly with the **UP/DOWN** switch. Check to make sure that the reference is centered between the two metallic surfaces and realign the reference if necessary. Remove any excess thermal compound from the edges of the reference with a cloth or tissue paper. (If the reference needs to be realigned, first place the **FREE/MOVE** switch to the **FREE** position. This removes the air pressure from the cylinder and eliminates the compressive load on the reference. Realign the reference and then place the switch back in the **MOVE** position.)



WARNING: Keep fingers away from the test stack when activating the DOWN switch. Severe personal injury may result otherwise.

AVERTISSEMENT: Éloignez vos doigts de la cheminée d'essai lors de l'activation de l'interrupteur BAS. Il peut en résulter de graves blessures dans le cas contraire.

- 11 Release the spring-loaded locking pin and lower the guard furnace. Lock the furnace in the lowest position.
- 12 Close the front door.
- 13 Input the calibration information and follow the on-screen programming.
- 14 When prompted, change the calibration reference to the appropriate one and resume the calibration test.

15 Continue until all calibration references being used have been tested.

Calibration Procedures for Sub-Ambient Temperatures (-20°C to 20°C)

NOTE: Make certain to use the copper spacer and insulation for the lower heat sink.

- 1 Check that the **HEATER POWER** switch on the front panel is **OFF** (no red is visible on the switch).
- 2 Activate the instrument by pressing the main power switch next to the power input socket in the rear panel.
- 3 Check that there is coolant flowing through the heat sink.
- 4 Check the air pressure reading on the gauge. If necessary, adjust the air pressure.
- 5 Open the front door. Raise the guard furnace and lock it in the elevated position, thus providing access to the test stack.
- 6 Raise the upper heater assembly by toggling the **UP/DOWN** switch.
- 7 Determine which stack spacer should be used to obtain the desired reference temperatures for the upcoming test, and place the appropriate spacer in the instrument's stack.
- 8 Measure and record the thickness of the first calibration reference you plan to test. Apply a small volume of thermal compound (approximately .25" diameter and .25" high) to the center on each face of the sample.
- 9 Place the calibration reference on the exposed surface of the bottom heater assembly and align the reference with the lower stack.
- 10 Lower the top heater assembly with the **UP/DOWN** switch. Check to make sure that the reference is centered between the two metallic surfaces and realign the reference if necessary. Remove any excess thermal compound from the edges of the reference with a cloth or tissue paper. (If the reference needs to be realigned, first place the **FREE/MOVE** switch to the **FREE** position. This removes the air pressure from the cylinder and eliminates the compressive load on the reference. Realign the reference and then place the switch back in the **MOVE** position.)



WARNING: Keep fingers away from the test stack when activating the DOWN switch. Severe personal injury may result otherwise.

AVERTISSEMENT: Éloignez vos doigts de la cheminée d'essai lors de l'activation de l'interrupteur BAS. Il peut en résulter de graves blessures dans le cas contraire.

- 11 Release the spring-loaded locking pin and lower the guard furnace. Lock the furnace in the lowest position.
- 12 Close the front door.
- 13 Input the calibration information and follow the on-screen programming.
- 14 When prompted, change the calibration reference to the appropriate one and resume the calibration test.
- 15 Continue until all calibration references being used have been tested.

Calibration Procedure with the Paste Cell

The calibration procedure is identical to the normal procedure without the cell except that, in each case, the calibration reference is located inside the cell and the entire reference/cell assembly must be placed in the DTC-300 test chamber instead of just the reference alone. Following is a description of how the calibration reference are placed inside the cell:

- 1 Measure and record the thickness of the calibration reference.
- 2 Select a set of rings whose inner spacer is thinner than the height of the reference.
- 3 Hold the outer ring with its rim toward the bottom.
- 4 Insert the bottom metal plate (flange side facing up) in the outer ring and slide the plate all the way down until the flange comes to rest against the lower rim. Insert and lower the inner ring (spacer) until it reaches the metal plate.
- 5 Apply thermal compound to both sides of the calibration reference.
- 6 Insert the calibration reference in the cell inside the spacer. The top surface of the reference must be above that of the spacer.
- 7 Insert the upper metal plate (the one with the small holes along the perimeter) in the cell and push it down until it makes contact with the calibration reference. If the top surface of the metal plate ends up below the top of the outer ring, you have chosen the wrong outer ring. The top metal surface must always be above the outer ring.
- 8 Hold the cell by the outer ring and apply thermal compound to the exposed top and bottom metal surfaces.
- 9 Place the cell on the bottom plate of the DTC-300.

Reanalyzing and Modifying a Calibration

Reanalyzing a Calibration

If, during the review of the calibration results, one point on the graph is found questionable, being far from the linear curve that could be drawn through the other points, several causes may be considered:

- An erroneous thickness of this particular reference material was entered into the program
- The reference material in question is chipped, badly scratched or otherwise damaged
- The reference material was not properly placed in the instrument, for example:
 - The compression on the specimen was not on
 - Too much or too little thermal compound was used, compared with the other samples
 - The guard may not have been lowered for that particular run
- The wrong spacer was placed in the lower stack of the instrument

If an erroneous thickness of the piece was entered into the program, the calibration file may be modified and then reanalyzed, to show the corrected results:

NOTE: Although the software allows changes to be made to the temperatures measured during the test, this is **NOT RECOMMENDED**. Only the thickness of the sample should be modified, in case it was wrongly entered into the program.

- 1 In the software, click **Results > Post Analysis > Reanalyze a Calibration**.
- 2 Select the calibration file to be reanalyzed.
- 3 Modify the thickness of all necessary samples and then click **OK**.

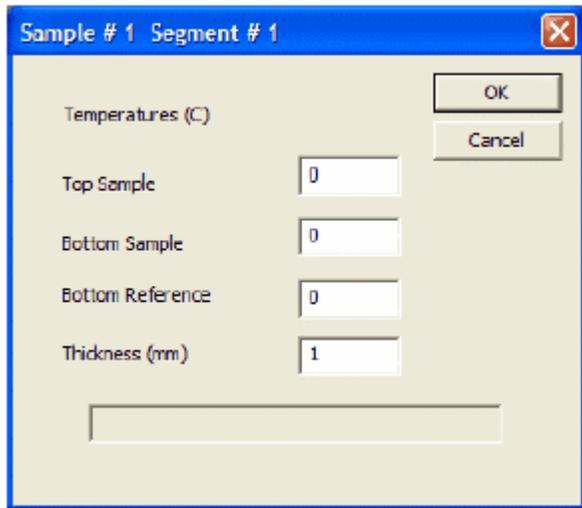


Figure 21 Setpoint information.

- 4 A Calibration Changes window displays, asking if you would like to create a new file to save the changes. Click **No** to terminate the reanalysis, or click **Yes** to continue.
- 5 A Sample Information window displays. Update the information, if needed.
- 6 If the Sample/File ID field is left unchanged, a window displays asking to overwrite the file. Click **Yes** to overwrite the file or **No** to terminate reanalysis.
- 7 Reanalyzed calibration results display.

Modifying a Calibration

In some cases, a particular point from the calibration curve has to be removed, due to any of the reasons listed in the beginning of the previous paragraph. Other times, the calibration needs to be extended, having either more temperature points, or reference materials, added. In all these situations, the calibration file may be modified.

- 1 In the software, click **Results > Post Analysis > Modify a Calibration File**.
- 2 A Calibration Modify window displays. Select **Individual Values** (to modify the temperatures and/or thickness on all of the necessary samples and segments. Click **OK**..

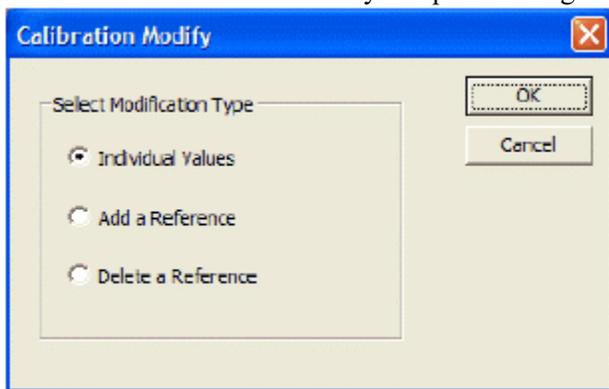


Figure 22 Calibration Modify.

- 3 Modify each sample temperature and thickness, as needed.
- 4 A Calibration Changes window displays, asking if you would like to create a new file to save the changes. Click **No** to terminate the modification, or click **Yes** to continue.
- 5 A Sample Information window displays. Update the information, if needed.
- 6 If the Sample/File ID field is left unchanged, a window displays asking to overwrite the file. Click **Yes** to overwrite the file or **No** to terminate modification.
- 7 Modified calibration results display.

Adding a Reference

The **Add a Reference** option allows you to add one or more reference pieces to the calibration.

- 1 In the software, click **Results > Post Analysis > Modify a Calibration File**.
- 2 A Calibration Modify window displays. Select **Add a Reference**.
- 3 The Select Reference Material window displays.

- 4 Highlight the reference to be added. Click **Another** to continue adding reference. Click **Finished** to continue.

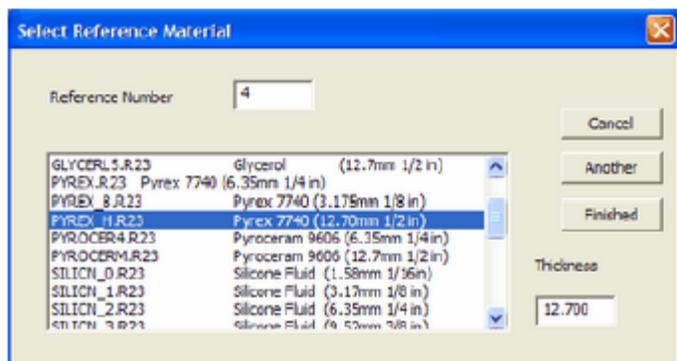


Figure 23 Select Reference Material.

- 5 Enter three temperatures and a thickness for the number of segments that the original calibration had.

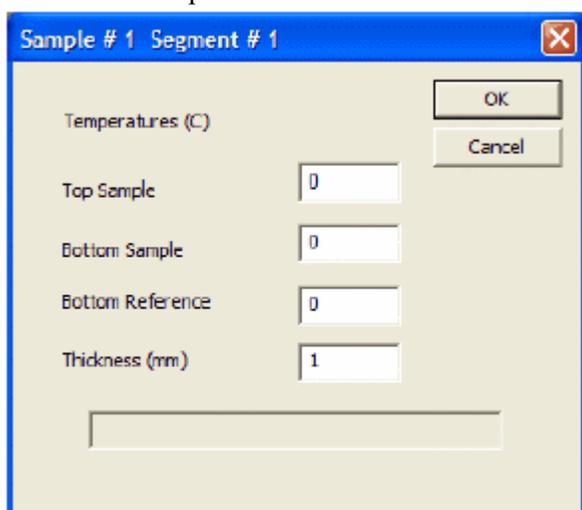


Figure 24 Setpoint information.

- 6 A Calibration Changes window displays, asking if you would like to create a new file to save the changes. Click **No** to terminate the reanalysis, or click **Yes** to continue.
- 7 A Sample Information window displays. Update the information, if needed.
- 8 If the Sample/File ID field is left unchanged, a window displays asking to overwrite the file. Click **Yes** to overwrite the file or **No** to terminate modification.
- 9 Modified calibration results display.

Deleting a Reference

The **Delete a Reference** option allows you to delete one or more reference pieces of the calibration.

NOTE: The calibration must always have at least 3 references.

- 1 In the software, click **Results > Post Analysis > Modify a Calibration File**.
- 2 A Calibration Modify window displays. Select **Delete a Reference**.

- 3 The Reference Delete window displays. Highlight the reference to be deleted and click **OK**.

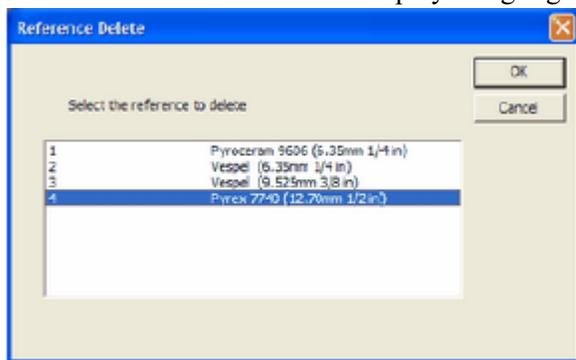


Figure 25 Reference Delete.

- 4 A Sample Information window displays. Update the information, if needed.
- 5 If the Sample/File ID field is left unchanged, a window displays asking to overwrite the file. Click **Yes** to overwrite the file or **No** to terminate modification.
- 6 Modified calibration results display.

Manually Creating a Calibration File

- 1 In the software, click **Results > Post Analysis > Create a Calibration File**.
- 2 The Calibration: Number of Samples and Segments window displays. Enter the number of samples (at least 3) and segments to be used to create the calibration.

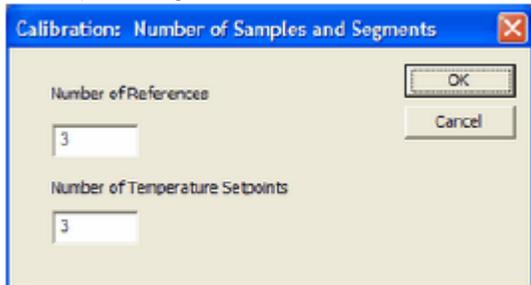
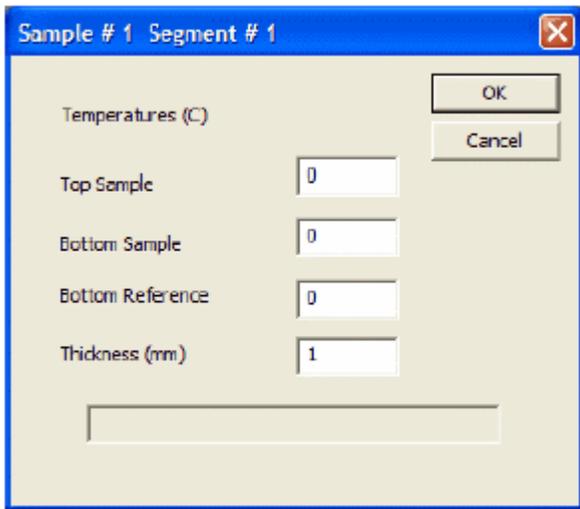


Figure 26 Calibration: Number of Samples and Segments.

- 3 The Select Reference Material window displays. Select the reference material to be used and then click **Finished**.

- 4 Enter three temperatures and a thickness for the number of segments that the calibration is being created with.



The image shows a dialog box titled "Sample # 1 Segment # 1". It has a blue title bar with a close button (X) in the top right corner. The main area is light beige and contains the following elements:

- A label "Temperatures (C)" is positioned above the input fields.
- Four input fields are arranged vertically:
 - "Top Sample" with the value "0"
 - "Bottom Sample" with the value "0"
 - "Bottom Reference" with the value "0"
 - "Thickness (mm)" with the value "1"
- Two buttons, "OK" and "Cancel", are located on the right side of the dialog.
- At the bottom, there is a long, empty rectangular input field.

Figure 27 Setpoint information.

- 5 A Calibration Changes window displays, asking if you would like to create a new file to save the changes. Click **No** to terminate the calibration creation, or click **Yes** to continue.
- 6 A Sample Information window displays. Fill in the information and click **OK**.
- 7 Calibration results display.

Chapter 5:

Maintaining the DTC-300

The primary maintenance procedures described in this section are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel.

Cleaning the Test Stack Surface

Clean the stack surfaces with a lint-free cloth and isopropyl alcohol. Stronger solvents could cause issues with the surfaces of the faces contacting the sample.

CAUTION: Before using any cleaning or decontamination method except those recommended by TA Instruments, check with TA Instruments that the proposed method will not damage the instrument.

MISE EN GARDE: Avant d'utiliser une méthode de nettoyage ou de décontamination autre que celle recommandée par TA Instruments, vérifiez auprès de TA Instruments que la méthode proposée n'endommagera pas l'instrument.

Uninstalling and Installing the Stack Spacer

Uninstalling the Stack Spacer

- 1 Release the latches and open the front door.
- 2 Lift the guard heater to the top locking position.
- 3 Remove the bottom cooling block insulation, if installed.
- 4 Remove any samples in the stack.
- 5 Clean both the top and bottom stack surfaces with a lint-free cloth and a small amount of isopropyl alcohol.
- 6 Remove the two screws under the bottom stack.

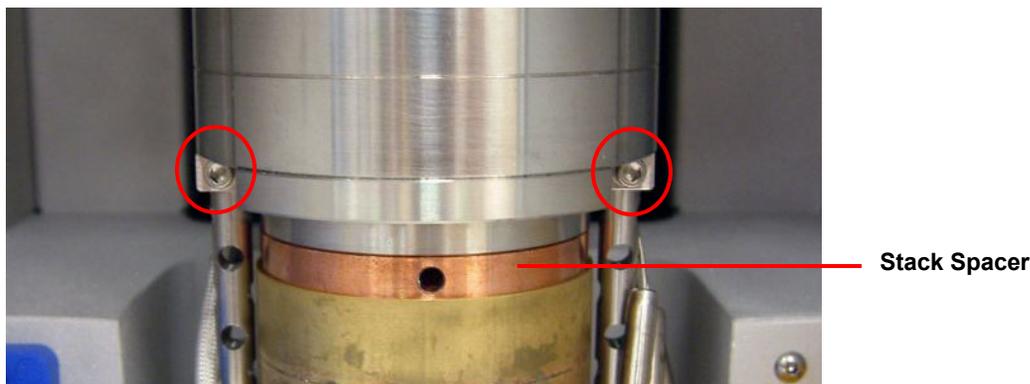


Figure 28 Remove screws.

- 7 Carefully lift the bottom stack off of the securing posts. **NOTE:** The thermocouples are secured to the bottom plate, **DO NOT** lift the bottom stack more than the thermocouples will allow.

CAUTION: Damage can occur if care is not taken when lifting the bottom stack.

MISE EN GARDE: Des dégâts peuvent survenir si vous ne faites pas attention lors du levage de la cheminée inférieure.

- 8 Using a hex key, loosen the stack spacer, then remove it from under the bottom stack.
- 9 Temporarily place the bottom stack back on the securing posts.

Installing the Stack Spacer

- 1 Clean both the top and bottom stack surfaces with a lint-free cloth and a small amount of isopropyl alcohol.
- 2 Lift the bottom stack off of the securing posts and insert the stack spacer using a hex key.
- 3 Place the bottom stack back onto the securing posts. Make sure that all of the bottom stack components are situated correctly.
- 4 Place a clean sample onto the bottom stack surface or simply put the two stack faces together.
- 5 Lower the top stack to apply pressure to the bottom stack. Make sure that the bottom stack is correctly aligned and proper contact between all surfaces is maintained. If a problem is found, release the pressure to the bottom stack.
- 6 Replace the two retaining screws directly under the bottom stack.

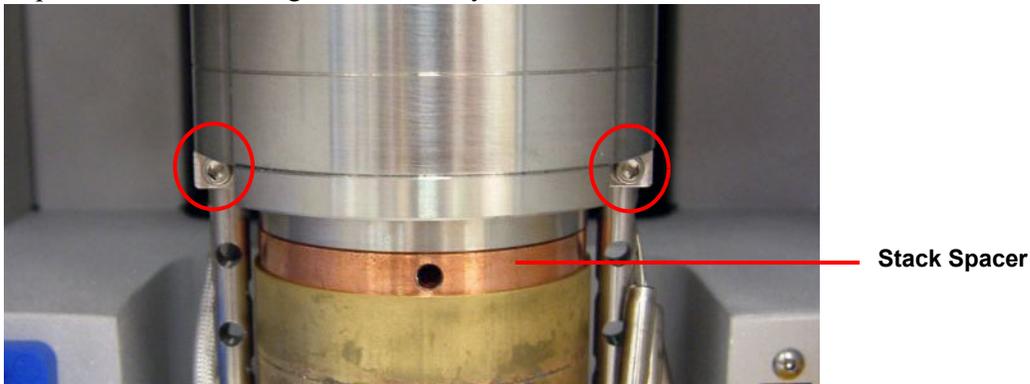


Figure 29 Replace screws.

- 7 Raise the top stack and remove the sample.

Uninstalling and Installing the Bottom Stack

Uninstalling the Bottom Stack

- 1 Release the latches and open the front door.
- 2 Lift the guard heater to the top locking position.
- 3 Remove the sub-ambient insulation ring or bottom cooling block insulation, if installed.
- 4 Remove any samples in the stack.
- 5 Clean both the top and bottom stack surfaces with a lint-free cloth and a small amount of isopropyl alcohol.
- 6 Remove the two screws under the bottom stack. Unplug the bottom heater connector from the left side of the bottom stack.

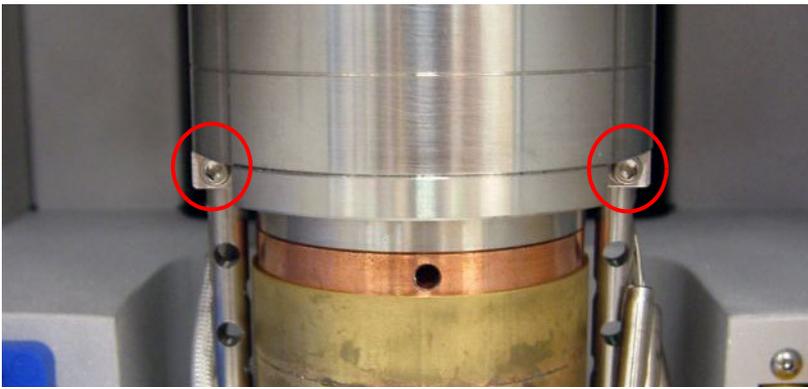


Figure 30 Remove screws.

- 7 Unplug the three thermocouple connectors from the right side of the bottom stack.
- 8 Carefully lift the bottom stack off of the securing posts. Lift the bottom stack off of the support posts and remove it from the unit.

NOTE: Take care not to scratch or otherwise damage the top and bottom sample plates when moving the bottom stack in and out of the instrument.

Installing the Bottom Stack

- 1 Place the bottom stack assembly back onto the securing posts. Make certain that the proper stack spacer is on top of the lower heat sink, Vespel for high temperature testing and Copper for sub-ambient testing.
- 2 Carefully plug the 3 thermocouples into the connectors. Be certain that the proper thermocouple is connected to each female plug on the instrument.
- 3 Plug in the bottom heater connector.
- 4 Make sure that all bottom stack components are correctly aligned.
- 5 Place a clean sample onto the bottom stack surface.
- 6 Replace the two screws directly under the bottom stack, but do not tighten them. Slowly lower the top stack to apply pressure to the bottom stack. Make sure that the bottom stack is correctly aligned and proper contact between all surfaces is maintained. If a problem is found, release the pressure to the bottom stack.

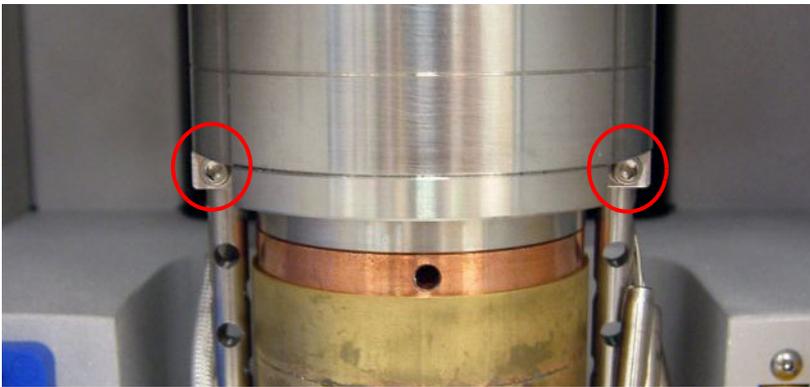


Figure 31 Replace screws.

- 7 Tighten the screws, making sure that pressure remains on the bottom stack.
- 8 Raise the top stack and remove the samples.

Replacement Parts

Replacement parts for the DTC-300 are listed below. Refer to the tables below when ordering parts.

Table 5: Consumables

Part Number	Description
854135.001	Heat Transfer Compound, jar

Table 6: Accessories

Part Number	Description
854100.901	Paste Cell, 50.8 mm OD
854101.901	Liquid Cell, 50.8 mm OD
202422.001	Chiller / Circulator
854108.901	Stack Module, Low Range
854111.901	Stack Module, High Range

Table 7: References

Part Number	Description
854117.901	Stainless Steel Reference, 50.8 mm OD x 19.05 mm thick
854118.901	Stainless Steel Reference, 50.8 mm OD x 12.7 mm thick
854119.901	Stainless Steel Reference, 50.8 mm OD x 6.35 mm thick
854129.901	Vespel Reference, 50.8 mm OD x 6.35 mm thick
854131.901	Vespel Reference, 50.8 mm OD x 3.175 mm thick
854132.901	Vespel Reference, 50.8 mm OD x 9.525 mm thick
854153.001	Vespel Reference 50.8 mm OD x 19.05 mm thick
854151.001	Vespel Reference 50.8 mm OD x 4.76 mm thick
854152.001	Vespel Reference 50.8 mm OD x 12.7 mm thick
854153.901	Vespel Reference 50.8 mm OD x 19.05 mm thick