DISCOVERY SDT
SIMULTANEOUS THERMAL ANALYZER

Getting Started Guide

Revision A Issued May 2017

Notice

The material contained in this manual, and in the online help for the software used to support this instrument, 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.

TA Instruments may have patents, patent applications, trademarks, copyrights, or other intellectual property covering subject matter in this document. Except as expressly provided in written license agreement from TA Instruments, the furnishing of this document does not give you any license to these patents, trademarks, copyrights, or other intellectual property.

TA Instruments Operating Software, as well as Module, Data Analysis, and Utility Software and their associated manuals and online help, are proprietary and copyrighted by TA Instruments. Purchasers are granted a license to use these software programs on the module and controller with which they were purchased. These programs may not be duplicated by the purchaser without the prior written consent of TA Instruments. Each licensed program shall remain the exclusive property of TA Instruments, and no rights or licenses are granted to the purchaser other than as specified above.

TA Instruments can accept no liability for loss or damage, however caused, arising from the faulty or incorrect use of its products.TA Instruments shall not be liable for any damages caused by interactions between exogenous materials (e.g. chemicals) and parts of the instrument. This includes interactions of gaseous, liquid or solid materials with parts of the instrument.

TA Instruments — Waters LLC
159 Lukens Drive
New Castle, DE 19720

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
- TA Instruments Offices

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.

Regulatory Compliance

Safety Standards

For Canada

CAN/CSA-C22.2 No. 61010-1-12 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010-04 Particular requirements for laboratory equipment for the heating of materials.

CAN/CSA-C22.2 No. 61010-2-081-04 Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes.

For European Economic Area

(In accordance with Council Directive 2014/35/EU of the European Parliament and of the Council of 26 February 2014 on the harmonization of the laws of the Member States relating to the making available on the market of electrical equipment designed for use within certain voltage limits.)

EN 61010-1:2010 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

EN 61010-2-010:2014 Particular requirements for laboratory equipment for the heating of materials.

EN 61010-2-081:2015 Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes.

For United States

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

UL61010-2-010:2015 Particular requirements for laboratory equipment for the heating of materials.

UL 61010-2-081:2015 Particular requirements for automatic and semi-automatic laboratory equipment for analysis and other purposes.

Electromagnetic Compatibility Standards

For Australia and New Zealand

AS/NZS CISPR11:2015 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

For Canada

ICES-001 Issue 4 June 2006 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

For the European Economic Area

(In accordance with Council Directive 2014/30/EC of the European Parliament and of the Council of 26 February 2014 on the harmonization of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1: 2013 Electrical equipment for measurement, control, and laboratory use - EMC requirements - Part 1: General requirements, Table 3 - Immunity test requirements for equipment used in controlled EM environments, Emission requirements for Group 1, Class A equipment.

For the United States

CFR Title 47 Telecommunication Chapter I Federal Communications Commission, Part 15 Radio frequency devices (FCC regulation pertaining to radio frequency emissions).

CAUTION: This equipment is not intended for use in residential environments and may not provide adequate protection to radio reception in such environments.

MISE EN GARDE: Cet appareil n'a pas destiné à être utilisé dans des environnements résidentiels et ne peut pas fournir une protection adéquate à la réception radio dans de tels environnements.

Safety

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.

Instrument Symbols

The following labels are displayed on the Discovery SDT system for your protection:

Symbol	Explanation
	This symbol indicates that you should read this Getting Started Guide for important safety information. This guide contains important warnings and cautions related to the installation, operation, and safety of the Discovery SDT system. Ce symbole indique que vous devez lire entièrement ce guide de démarrage pour obtenir d'importantes informations relatives à sécurité. Ce guide contient d'importants avertissements et mises en garde relatifs à l'installation, à l'utilisation et à la sécurité du système Discovery SDT.
	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 come in contact with this hot surface. 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.
	High Voltages are present in this instrument. If you are not trained in electrical procedures, do not remove the cabinet covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel. Si vous n'êtes pas formé aux procédures électriques, ne déposez pas les couvercles de l'armoire sauf indications spécifiques contenues dans le manuel. La maintenance et la réparation des pièces internes doivent être effectuées uniquement par TA Instruments ou tout autre personnel d'entretien qualifié.

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

Electrical Safety

You must unplug the instrument before doing any maintenance or repair work; voltages as high as 120/240 VAC are present in the instrument.

WARNING: Risk of electric shock. High voltages are present in this instrument. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

AVERTISSEMENT: Risque de choc électrique. 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: Protective earthing is provided through the mains power cord. Use of a grounded mains power outlet is required.

AVERTISSEMENT: Mise à la terre de protection est assurée par le cordon d'alimentation secteur. Utilisation d'une prise d'alimentation secteur terre est nécessaire.

WARNING: After transport or storage in humid conditions, this equipment could fail to meet all the safety requirements of the safety standards indicated. Refer to the CAUTION on page 24 for the method used to dry out the equipment before use.

AVERTISSEMENT: Après le transport ou le stockage dans des conditions humides, cet équipement pourrait ne pas répondre à toutes les exigences de sécurité des normes de sécurité indiquées. Reportez-vous à la section ATTENTION à la page 24 pour connaître la méthode utilisée pour sécher l'équipement avant utilisation.

Chemical Safety

WARNING: Do not use hydrogen or any other explosive gas in the SDT furnace.

AVERTISSEMENT: N'utilisez pas d'hydrogène ou tout autre gaz explosif dans le four SDT.

WARNING: Oxygen can be used as a purge gas in the SDT. However, the furnace must be kept clean so that volatile hydrocarbons, which may combust, are removed.

AVERTISSEMENT: L'oxygène peut être utilisé comme gaz de drainage dans le SDT. Toutefois le four doit rester propre pour que les hydrocarbures volatils, qui peuvent brûler, soient éliminés.

WARNING: If you are using samples that may emit harmful gases, attach a compatible tube to the purge gas exit to transfer the gas to an exhaust or other suitable protective device.

AVERTISSEMENT: Si vous utilisez des échantillons qui émettent des gaz nocifs, fixez un tuyau compatible à la sortie du gaz de drainage pour transférer le gaz vers un échappement ou vers tout autre dispositif de protection approprié.

WARNING: The SDT furnace assembly contains a layer of refractory ceramic fiber (RCF) insulation. This insulation is completely encapsulated within the furnace can, which is not meant to be disassembled.

AVERTISSEMENT: L'ensemble de four SDT contient une couche d'isolant en fibre céramique réfractaire (RCF). Cette isolation est complètement encapsulée à l'intérieur du four, ce qui n'est pas destiné à être démonté.

Mechanical Safety

WARNING: Keep your fingers and all other objects out of the path of the furnace when it is moving. The seal is very tight.

AVERTISSEMENT: Écartez vos doigts et tous les autres objets du chemin du four lorsqu'il est en mouvement. L'étanchéité est très hermétique.

Lifting the Instrument

The Discovery SDT is a heavy instrument. In order to avoid injury, particularly to the back, please follow this advice:

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.

Table of Contents

Chapter 1:

Introducing the Discovery SDT

Overview

Your Simultaneous DSC-TGA Discovery instrument is an analysis instrument capable of performing both differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) at the same time.

The SDT measures the heat flow and weight changes associated with transitions and reactions in materials over the temperature range ambient to 1500°C. The information provided differentiates endothermic and exothermic events which have no associated weight change (e.g., melting and crystallization) from those which involve a weight change (e.g., degradation). Furthermore, performing both DSC and TGA measurements at the same time, on the same instrument and same sample, offers greater productivity and removes experimental and sampling variables as factors in the analysis of data.

Figure 1 Discovery SDT650 with optional Autosampler.

The Simultaneous DSC-TGA works in conjunction with a controller and associated software to make up a thermal analysis system.

Your controller is a computer that performs the following functions:

- Provides an interface between you and the analysis instrument
- Enables you to set up experiments and enter parameters
- Stores experimental data
- Runs data analysis programs.

NOTE: For technical reference information, theory of operation, and other information associated with the SDT not found in this manual, see TRIOS software Online Help.

SDT System Components

The Discovery SDT is comprised of the following major hardware components:

- The sample and reference balance assemblies, which provide the precise measurement of heat flow and sample weight.
- The heating system, or furnace, which controls the sample atmosphere and temperature.
- The cabinet, where the system electronics and mechanics are housed.
- The optional Autosampler, which loads and unloads the sample to and from the balance.

Balance Assembly

The SDT balance assembly is a null balance system consisting of separate sample and reference balance meter movements. Each meter movement consists of a beam upon which pans are placed, counterweights that are internal to the balance housing, and a balance position sensor.

- The balance meter movement is a taut-band meter movement to which a beam and counterweight are attached.
- The beam is a ceramic rod attached to the meter movement. The ceramic rod has a platform at the furnace end upon which is a platinum liner bonded to a thermocouple. The thermocouple is a type R thermocouple that has an upper measurement temperature of 1500°C.
- The balance position sensor is comprised of two LED-Photodiode modules mounted on a printed circuit board that detect the null position of the meter movement.
- The cylindrical counterweights mechanically balance the weight of the sample or reference pans and beams.

Furnace

The SDT wire-wound furnace is a resistance heater wound on alumina ceramic, which allows sample zone temperatures as high as 1500°C with heating rates up to 100°C/min. The housing is mounted to a furnace base that opens and closes the furnace for sample loading and unloading.

Optional Discovery SDT Autosampler

The optional Discovery SDT Autosampler allows you to place multiple samples on the platform for automatic loading and run sequencing. Using the standard pan tray, up to 30 pans can be accommodated. Experiments are performed as normal, but samples can be run on a continual basis.

See "Discovery SDT Sampling System" on page 19 for the pans that can be used with the Discovery SDT Autosampler:

To calibrate the sample tray, refer to "Calibrating the Discovery SDT" on page 33 of this manual and TRIOS Online Help.

Hi-Res™ SDT

The TA Instruments Hi-Res™ SDT technique differs from alternative control techniques in that the heating rate of the sample material is dynamically and continuously modified in response to changes in the rate of decomposition of the sample so as to optimize both weight change resolution and time of analysis. This SDT technique allows the use of high heating rates in baseline regions where no weight changes are occurring, but automatically slows the heating rate during weight changes. Once the weight change(s) are complete, the system returns to the selected ramp heating rate. Typical Hi-Res ramps often take the same or less time to complete than a comparable constant heating rate experiment run at a lower heating rate, while providing improved resolution.

Some of the benefits provided by the Hi-Res option are:

- Improved transition resolution
- Faster scans (sample and condition dependent)
- Enhanced signature analysis capability
- Transition temperatures closer to isothermal values
- Increased method programming versatility

Modulated SDT

TA Instruments Modulated SDT is an innovative option that is used with the Discovery SDT. It encompasses both modulated TGA (MTGA) and modulated DSC (MDSC). The MTGA option is used to study the same decomposition or volatilization properties as conventional SDT. However, MTGA provides unique capabilities that increase the amount of information obtained from a single SDT experiment, thereby improving the quality of interpretation.

These unique capabilities include:

- continuous determination of activation energy
- verification of single kinetic mechanism
- verification of first-order kinetic model.

MTGA is an enhancement of SDT that provides the same information as traditional SDT, plus new information that permits unique insights into the behavior of the weight loss reaction.

Specifically, MTGA provides an alternative way to obtain kinetic information about one or more weight losses, in a shorter period of time than the multiple heating rate approach.

In addition, MTGA provides continuous determined values for activation energy throughout the weight loss reaction, not just at specific reaction levels. The ability to obtain activation energy continuously allows you to follow changes in the activation energy during the reaction, as a function of temperature or conversion. The calculation of activation energy is “model free” – no knowledge about the form of the kinetic equation is required.

The assumption of a first-order kinetic model (a reasonable assumption for many decomposition reactions), permits the calculation of natural logarithm of the pre-exponential factor in the same manner as the continuous determination of activation energy.

The MDSC option is used to study the same heat flow properties as conventional SDT. However, MDSC provides unique capabilities that increase the amount of information obtained from a single SDT experiment thereby improving the quality of interpretation.

These unique capabilities include:

- Continuous determination of heat capacity.
- Separation of heat capacity dependent heat flow signals from kinetic heat flow signals.

See TRIOS online help for more detailed information.

Other Discovery SDT Accessories

Gas-blending Module

The gas-blending module allows for the connection of four gasses and the blending together of these gases in pairs.

Mass Spectrometer Interface Kit

The mass spectrometer interface kit provides a connection between the Discovery Mass Spectrometer and the Discovery SDT for the analysis of sample-evolved gases.

The Discovery SDT User Interface

The Discovery SDT includes an integrated user interface display for local operator control. The functions of the user interface change depending upon the view displayed. This section briefly describes the basic layout of these functions.

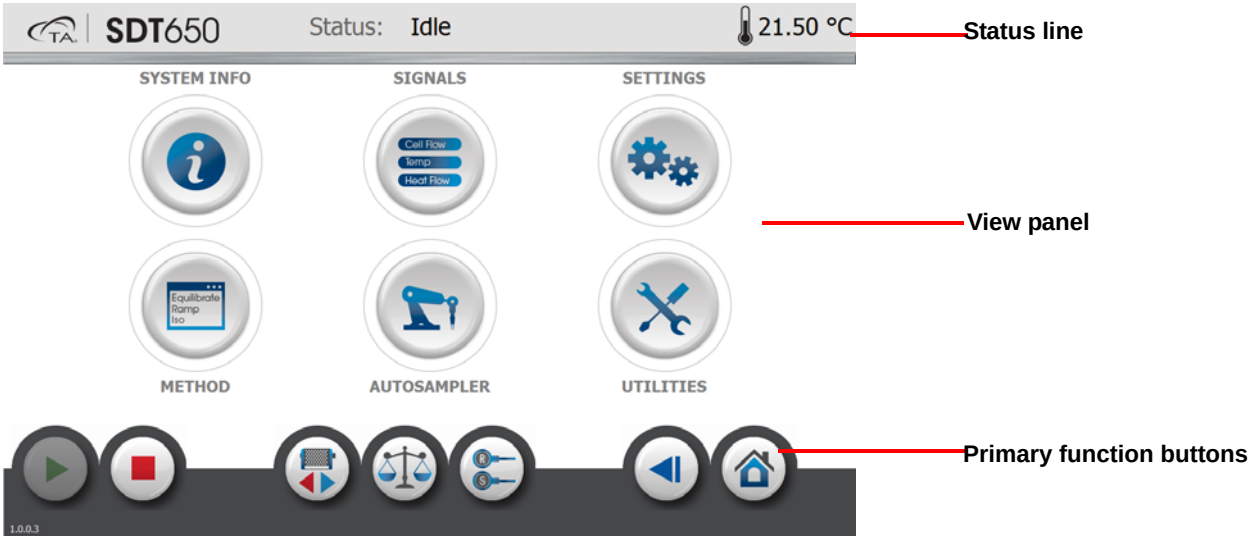

Figure 2 Discovery SDT user interface display.

The instrument user interface has three basic sections:

- The status line along the top of the display indicates the current instrument status and sample temperature.
- The primary function buttons allow for easy access of common functions.
- View panel allows for real-time instrument status and associated actions.

Primary Function Buttons

Use the following buttons for the main functions of the instrument.

Table 1: Primary Function Buttons on the User Interface

Key Name	Description
Start 	Begins the currently-programmed experiment. This is the same function as **Start** on the instrument control software. **Start** (with an Autosampler) automatically loads the selected sample pan and closes the furnace, if necessary, before beginning the experiment.
Stop 	If an experiment is running, this button ends the method normally, as though it had run to completion; i.e., the method-end conditions go into effect and the data that has been generated is saved. This is the same function as **Stop** on the instrument control software. If an experiment is not running (the instrument is in a standby or method-end state), the **Stop** button will halt any activity (air cool, all mechanical motion, etc.). If an Autosampler sequence is in progress, **Stop** will halt the sequence.
Furnace Open/Close 	Toggles between opening and closing the SDT furnace.
Tare 	Zeros the weight of an empty sample and reference pan.
Pan Load/Unload 	Displays the Autosampler screen and allows the pans to load and unload.
Back 	Returns to the previously used screen.
Home 	Returns to the Main screen.

View Panel

The view panel provides real-time instrument status and additional functionality pertinent to the selected operation. A list of available functions is described below.

Table 2: View Panel Functions on the User Interface

Button Name	Description
System Info 	Displays instrument information such as the serial number, IP configuration, and network configuration.
Signals 	User can select signals to display the real-time signal data generated directly from the instrument.
Settings 	Displays options for manual controls, such as Event and Air Cool.
Autosampler 	Displays options for Autosampler control, including Calibration, Loading/Unloading pans, and Reset.
Method 	Provides a summary of the sample and method information for the current run and allows the user to manually advance the method.
Utilities 	Displays system health and shut down button.

Additional Function Buttons

Button Name	Description
Help 	The **Help** button can be found on the lower right side of some screens, and displays information regarding use of the currently displayed touch-screen.
Motion Stop 	Displayed while the Autosampler or furnace is in motion. Pressing this button will stop the motion.

Instrument Specifications

The tables found below contain the technical specifications for the Discovery SDT units and sampling system.

Discovery SDT Instrument Characteristics

Table 3: Discovery SDT Technical Specifications

Item/Area	Specifications
Dimensions	Depth: 64 cm (25 in) Width: 66 cm (26 in) Height: with Autosampler: 53 cm (21 in) without Autosampler: 28 cm (11 in)
Weight	with Autosampler: 40 kg (88 lbs) without Autosampler: 33 kg (73 lbs)
Mains Power	100–240 VAC 47–63 Hz 1200 W
Operating environmental conditions	Temperature: 15 to 30°C Relative humidity: 5 to 80% (non-condensing) Installation Category II Pollution Degree 2 Maximum altitude: 2000 m (6560 ft) The degree of protection for this instrument according to IEC 529 is IP20.
Temperature range	Type R thermocouple: Ambient +5°C to 1500°C
Thermocouple	Type R thermocouple: Platinum-Platinum/13% Rhodium
Heating rate	100°C/minute to 1000°C 25°C/minute to 1500°C

Discovery SDT Sampling System

The following tables contain the specifications associated with the SDT sample pans, balance mechanism, furnace, and purge gases.

Table 4: Sample Pan Options for 30-Pan Tray

Item/Area	Specifications
Types of pans available	• Platinum and alumina (Al_20_3) SDT cups • Aluminum, gold, graphite, and copper DSC pans **NOTE**: DSC pans and the 40 µl ceramic and platinum cups are not compatible with the Autosampler.
Volume capacity	• Platinum: 40 µL; 110 µL (recommended for TGA-DTA studies) • Alumina: 40 µL; 90 µL (recommended for DSC-TGA studies)
Number of pans per tray	30 Pans

Table 5: Balance Mechanism

Item/Area	Specifications
Weighing capacity (sample)	200 mg
Dynamic weighing range	200 mg
Accuracy	$\leq \pm$ 0.5% of value or 100 µg, whichever is greater

Table 6: Operating Parameters

Item/Area	Specifications
Heat Flow Accuracy (DSC)	Better than ±2% (based on metal melting standards)
Heat Flow Precision (DSC)	Better than ±2% (based on metal melting standards)
Temperature Accuracy	±1°C (based on metal melting standards)
Temperature Precision	±0.5°C (based on metal melting standards)
ΔT Sensitivity (DTA)	0.001°C (200 to 1300°C)
Furnace Purge Gases	Helium, nitrogen, air, argon, oxygen
Flow Rate	20 to 500 mL/min, (100 mL/min is typical during experiments) Regulated by the Gas Delivery Module with gas switching capability.

NOTE: The SDT balance mechanism is sensitive to changes in the surrounding room temperature. For optimum accuracy, you must regulate the ambient temperature.

WARNING: The reactive gas input can be used to allow introduction of a “reactive” gas at modest concentration in the area of the sample. The reactive gas purge line is made of stainless steel. The reactive gas tube in the furnace is Inconel®. Hence, only gases which do not react with stainless steel, Inconel®, or platinum (or the alumina furnace tube) should be used as reactive purge gas. (Remember that platinum is a catalyst, allowing reactions to take place which might not occur in its absence.)

AVERTISSEMENT: L'entrée de gaz réactif peut être utilisée pour permettre l'introduction d'un gaz "réactif" à une concentration modeste dans la zone de l'échantillon. La conduite de purge de gaz réactif est en acier inoxydable. Le tube de gaz réactif dans le four est Inconel®. Par conséquent, seuls les gaz qui ne réagissent pas avec l'acier inoxydable, l'Inconel® ou le platine (ou le tube du four à alumine) doivent être utilisés comme gaz de purge réactif. (Rappelez-vous que le platine est un catalyseur, permettant des réactions qui pourraient ne pas se produire en son absence.)

Chapter 2:

Installing the Discovery SDT System

Unpacking/Repacking the SDT

The instructions needed to unpack and repack the instrument are found as separate unpacking instructions in the shipping box. You may wish to retain all of the shipping hardware, pallets, and boxes from the instrument in the event you wish to repack and ship your instrument. For future reference, these instructions can be found at http://www.tainstruments.com/wp-content/uploads/963024000.pdf

WARNING: Have an assistant help you unpack this unit. Do not attempt to do this alone.

AVERTISSEMENT: Faites-vous aider par une personne pour dépoter cet appareil. N'essayez pas de le faire tout seul.

Installing the Discovery SDT System

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.

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

1. Inspecting the system for shipping damage and missing parts
2. Choosing a location for instrument installation
3. Preparing the Discovery SDT for installation, which includes removing the shipping bracket and unpacking the balance
4. Setting up system communication between the instrument and computer (controller)
5. Connecting purge gas lines, accessories, and power cable
6. Installing the Autosampler tray
7. Leveling the instrument
8. Closing up the balance and conditioning the balance

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

Inspecting the System

When you receive your Discovery SDT 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 Discovery SDT experiments, it is important to choose a suitable location for the instrument using the following guidelines. The Discovery SDT system should be:

In

- A temperature-controlled area. Temperatures should be in range 20 to 35°C.
- A clean, vibration-free environment, preferably on the ground floor in the building. It should be located away from pumps, motors, or other devices which produce vibrations.
- An area with ample working and ventilation space.

On

- A stable work surface. A marble table is recommended for optimum performance. Isolation mounts on a standard lab bench are not recommended.

Near

- A power outlet (100–240 VAC, 47–63 Hz, 1200 W).
- Your TA Instruments controller.
- Compressed lab air and purge gas supplies with suitable regulators and filters, if required.

Away from

- Dusty environments.
- Exposure to direct sunlight.
- Direct air drafts (fans, room air ducts).
- Poorly ventilated areas.
- Noisy or mechanical vibrations.
- High traffic areas, where constant movements from passing personnel could create air currents or mechanical disturbances.

NOTE: Allow free air to circulate around the enclosures. Do not place equipment against walls or cabinets that might impede air flow. Leave at least 7.5 cm (3 in) clearance around the Discovery SDT.

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.

CAUTION: Drying out the instrument may be needed, if it has been exposed to humid conditions. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation.

Run the following procedure to dry out the instrument:

Ramp at 10°C/min to 400°C.
Isothermal for 30 min.

MISE EN GARDE: Il peut être nécessaire de sécher l'instrument s'il a été exposé à des conditions humides. Il est important de s'assurer que la masse de l'instrument est correctement connectée à la terre des installations pour un fonctionnement sécurisé.

Exécutez la procédure suivante pour sécher l'instrument:

Rampe à 10 ° C / min à 400 ° C.
Isotherme pendant 30 min.

Connecting the Discovery SDT to the Controller

To connect the SDT system, you will need access to the Discovery SDT instrument's back panel.

NOTE: Connect all cables before connecting the power cord to an outlet.

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.

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.

The Discovery SDT back panel has fifteen ports; the table below provides a description of the function of each port.

Table 7: Discovery SDT Back Panel

Port	Function
Power Entry Module (power cord)	Provides power and protective earthing to the system.
Fuses	Provides overcurrent protection.
REACTIVE GAS	Port for introducing an additional gas directly into the vicinity of the sample and reference. External flow regulation required. Maximum pressure is 140 kPa gauge (20 psig).
GAS 1	Gas inlet port controlled by the gas delivery module. Used for the sample and balance purge gas. Maximum pressure is 140 kPa gauge (20 psig).
GAS 2	Gas inlet port controlled by the gas delivery module. Used for the secondary sample purge gas. Maximum pressure is 140 kPa gauge (20 psig).
COOLING GAS	Provides the furnace with air for post-run cooling. Maximum pressure is 140 kPa gauge (20 psig).
RS-232 Port	Provides communications with external accessories.
CAN Communications Port	Provides communications with external accessories.
Ethernet	Provides communication between controller and SDT instrument.
USB 2.0 Port	Provides communications with external accessories.
Micro USB 2.0 Port	Provides communications for external accessories.
SD memory card slot	For TA Instruments Service use only.
Audio Jack	External headset.
EVENT	Capable of providing a general purpose relay contact closure.
24 VDC OUT	Not used.

WARNING: If connecting oxygen, it should ONLY be connected to the GAS 2 and REACTIVE GAS ports.

AVERTISSEMENT: En cas de connexion de l'oxygène, il ne doit être connecté aux ports GAS 2 et gaz réactif.

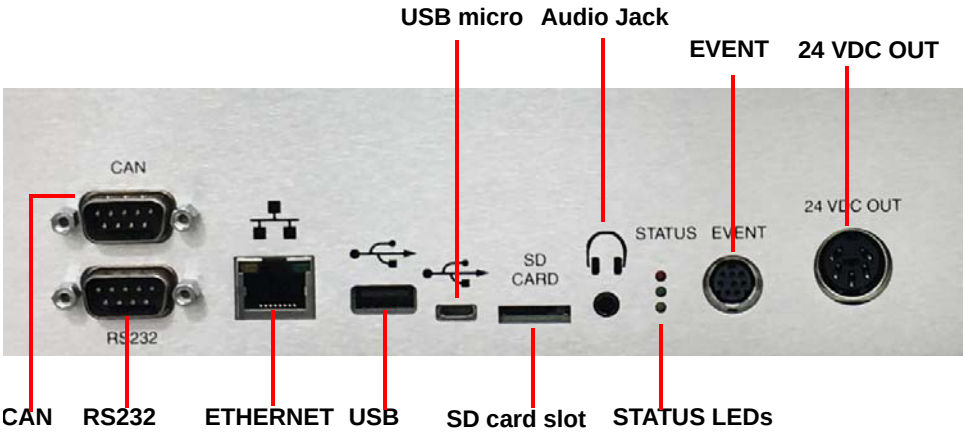

Figure 3 Discovery SDT back panel connections.

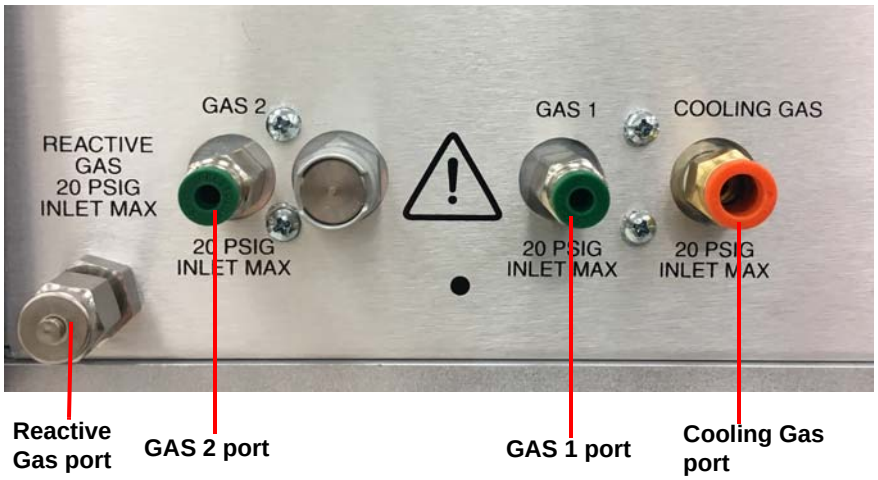

Figure 4 Discovery SDT back panel–Gas connections.

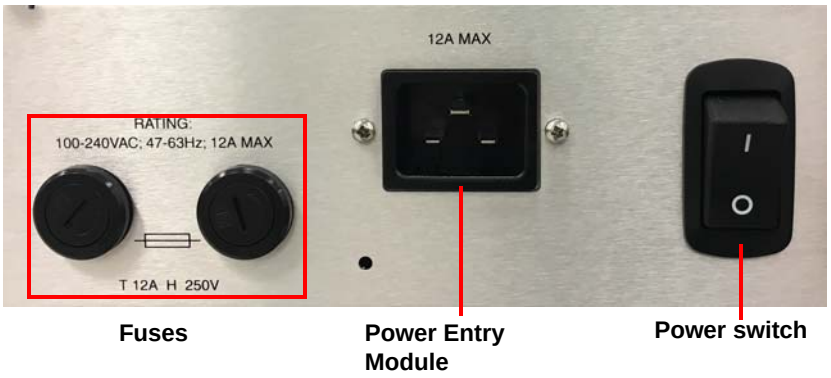

Figure 5 Discovery SDT back panel: Fuses, Power Entry Module, and power switch.

Setting Up System Communication

In order to connect the instrument to a network, you will need to connect the controller computer to a router, then connect the computer to a LAN.

Refer to the TRIOS Software Installation Instructions for more details.

Connecting Purge Gas Lines

You can control the sample atmosphere during SDT experiments by connecting purge gases to the system. The SDT is equipped with a Gas Delivery Module (GDM) to control the flow rate of the sample purge gas. Up to two different gases may be connected to the instrument to facilitate gas switching. Follow these instructions to connect the purge lines. Refer to the figure below to locate the purge lines.

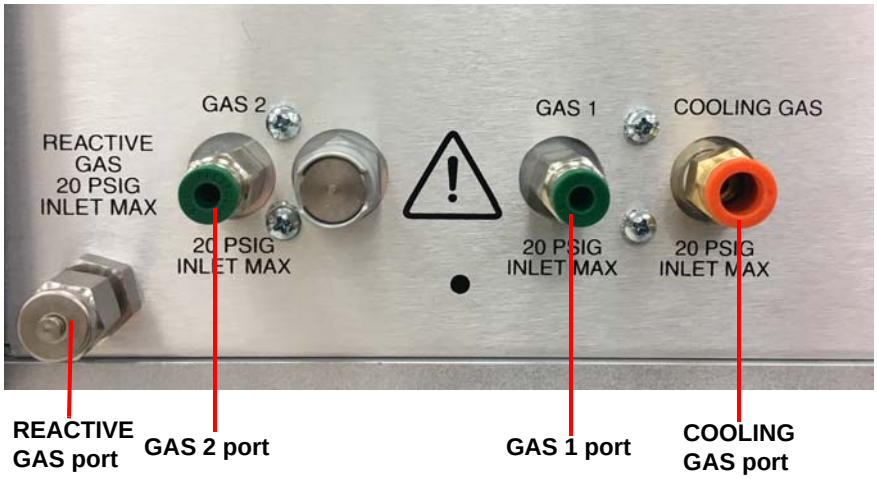

Figure 6 Gas connections on the back of the Discovery SDT.

CAUTION: Do not use any liquid in the purge lines.

MISE EN GARDE: N'utilisez aucun liquide dans les conduites de gaz de drainage.

WARNING: Use of flammable or explosive gas as a purge gas is dangerous and is not recommended for this instrument.

AVERTISSEMENT: L'utilisation d'un gaz inflammable ou explosif comme gaz de drainage est dangereuse et n'est pas recommandée pour cet instrument.

NOTE: If you are using a "house" laboratory supply rather than bottled gas, it is highly recommended that you install an external drier and a 5-µm filter.

1. Locate the **GAS 1** port on the back of the Discovery SDT. The **GAS 1** port is used to purge the sample and balance areas. As such, an inert gas such as nitrogen should be used in this port.
2. Locate the **GAS 2** port. The **GAS 2** port is also used to purge the sample area and is used when a purge gas other than **GAS 1** is desired or when gas switching during an experiment is needed.

3 Connect the primary gas line to the **GAS 1** port using 1/8-inch O.D. tubing. Teflon® TFE tubing is recommended and is supplied in the instrument shipping accessory kit. If desired, connect a second gas to the **GAS 2** port. The flow rate is controlled through the Mass Flow Controller settings chosen using the instrument control software. Refer to Figure 6 for reference.

WARNING: If connecting oxygen, it should ONLY be connected to the GAS 2 port.

AVERTISSEMENT: Tout raccordement de l'oxygène doit s'effectuer UNIQUEMENT sur l'orifice à gaz 2.

NOTE: The flow rate is controlled through settings chosen using the instrument control software.

4 Make sure that the pressure of your purge gas source is regulated between 70 and 140 kPa gauge (10 and 20 psig, respectively).

CAUTION: The GAS 1 and GAS 2 lines feeds into a pressure relief valves that are set to 172 kPa gauge (25 psig). The source pressure setting should not go above this value.

MISE EN GARDE: Les conduites de gaz 1 et 2 alimentent une soupape de détente de pression réglée à 172 kPa (pression manométrique) (25 psig). Le réglage de la pression à la source ne doit pas dépasser cette valeur.

5 Specify the connected gas and set the balance flow rate to 100 mL/min on the **Discovery SDT** > **General** page of the TRIOS **Options**.

6 Set the sample flow rate to 100 mL/min through the Control Panel options within TRIOS instrument control software.

NOTE: If you are using laboratory (house) purge gas, rather than bottled purge gas, it is highly recommended that you install an external drier and a 5 µm filter in the gas line prior to the instrument.

CAUTION: Corrosive gases cannot be used with this instrument.

MISE EN GARDE: Les gaz corrosifs ne peuvent pas être utilisés avec cet instrument.

WARNING: Use of a flammable gas as a purge gas is dangerous and is not recommended for this instrument. For a list of the purge gases that can be used with the SDT instrument, see Chapter 1.

AVERTISSEMENT: L'utilisation d'un gaz inflammable comme gaz de purge est dangereuse et n'est pas recommandée pour cet instrument. Pour une liste des gaz de purge pouvant être utilisés avec l'instrument SDT, voir le chapitre 1.

Reactive Gas Purge Line

The reactive gas purge fitting is used to expose the sample to an additional gas, other than that used through the Gas Delivery Module. Use the following steps to install the reactive gas purge line.

CAUTION: Corrosive gases cannot be used with this instrument.

MISE EN GARDE: Les gaz corrosifs ne peuvent pas être utilisés avec cet instrument.

WARNING: Use of a flammable gas as a purge gas is dangerous and is not recommended for this instrument. For a list of the purge gases that can be used with the SDT instrument, see Chapter 1.

AVERTISSEMENT: L'utilisation d'un gaz inflammable comme gaz de purge est dangereuse et n'est pas recommandée pour cet instrument. Pour une liste des gaz de purge pouvant être utilisés avec l'instrument SDT, voir le chapitre 1.

1. Remove the cap from the reactive gas purge fitting.
2. Install the nut and ferrule supplied in the accessory kit.
3. Connect the desired gas to an external flow meter. Flow is controlled via this flow meter and not by using the instrument control software. In addition, the flow rate for the reactive gas purge is not stored as a signal in the data analysis files.
4. Connect one end of a 1/8-inch O.D. compression tube to the flow meter and the other end to the reactive gas purge fitting.
5. Regulate the flow meter to a recommended 20 mL/min.

NOTE: The sample purge must be used at all times (100 mL/min recommended). This prevents the reactive gas from diffusing back into the balance.

Connecting the Air Cool Line

Use the following steps to install the Cooling Gas line:

1. Locate the **COOLING GAS** fitting on the rear of the Discovery SDT cabinet, marked with a 140 kPa gauge (20 psig) warning label.
2. Make sure your compressed air is regulated to 140 kPa gauge (20 psig) and is free of water and vapors.
3. Connect the 1/4-inch O.D. tubing from the compressed air source to the **COOLING GAS** fitting.

NOTE: Nitrogen may also be used as a cooling gas. Whichever gas is chosen as a cooling gas, it should be clean and dry.

NOTE: Air Cool will operate only when the furnace temperature is below 600°C.

Connecting the Power Cable

NOTE: A <HAR>-marked (harmonized) power cable with an IEC 60320 C19 style connector meeting the standards of the country of installation is required for the European Economic Area.

Install the power cable as follows:

1 Make sure the power switch is in the Off (**O**) position, as shown in the figure below.

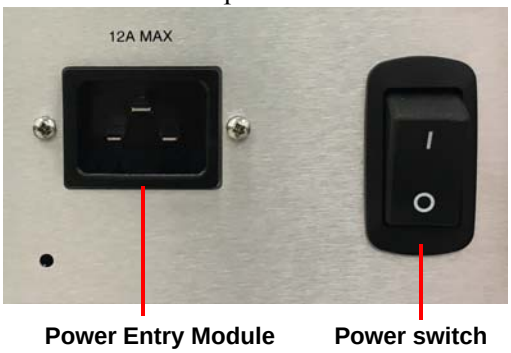

Figure 7 Power Entry Module and power switch.

2 Plug the power cable into the Power Entry Module (shown above).

3 Plug the power cable into the wall outlet.

Installing the Autosampler Tray

When you receive your SDT, the Autosampler tray is shipped in the accessory box, separate from the instrument. After unpacking the instrument and installing the instrument completely (see instructions in this chapter), you will be ready to run samples using the Autosampler.

1 Reset the Autosampler to move the gantry out of the way (if equipped and needed to gain access).

2 Locate the mounting hole and slot on the sample tray. Orient the tray so that the slot is on the right.

3 Place the tray into the opening on top of the balance housing cover and align the two mounting pins with the hole and slot on the tray.

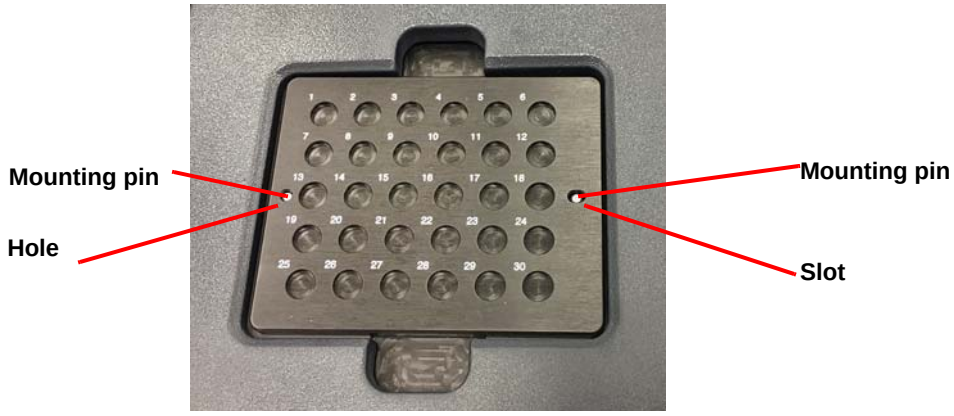

Figure 8 Mounting pins.

Sample cups can now be placed on the tray.

Starting the Discovery SDT System

The power switch is located at the back panel of the instrument. It is part of the assembly called the power entry module, which also contains the power cable connection and fuses. The power switch is used to turn the SDT system on and off.

To power on the system:

1 Check the connection between the SDT, Ethernet switch, and the controller. Make sure each component is plugged into the correct connection port.

2 Set the power switch to the ON **(I)** position.

3 After the proper power up sequence, the instrument user interface displays; this indicates that the instrument is ready for use.

NOTE: Allow the Discovery SDT to warm up for 30 minutes before performing an experiment in order to allow time for the temperature-controlled measurement circuitry to stabilize.

CAUTION: Drying out the instrument may be needed, if it has been exposed to humid conditions. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation.

Run the following procedure to dry out the instrument:

Ramp at 10°C/min to 400°C
Isothermal for 30 min.

MISE EN GARDE: Il peut être nécessaire de sécher l'instrument s'il a été exposé à des conditions humides. Il est important de s'assurer que la masse de l'instrument est correctement connectée à la terre des installations pour un fonctionnement sécurisé.

Exécutez la procédure suivante pour sécher l'instrument:

Rampe à 10 ° C / min à 400 ° C.
Isotherme pendant 30 min.

Shutting Down the Discovery SDT 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 SDT perform more reliably if power fluctuations caused by turning units on and off are minimized.

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.

NOTE: Make sure to remove all samples and pans from the beams before initiating shut down.

To ensure proper shutdown of the instrument, it is recommended that you execute a shutdown from the user interface or TRIOS software before turning off the power to the instrument. To shut down, perform one of the following options:

- Select **Shutdown** from the instrument user interface **Utilities** menu.
- Select **Shutdown** from the **Instrument** menu within the TRIOS software.

A confirmation message displays. Select **Yes** to continue. All communication to the instrument halts while the instrument saves any unsaved data. Once this procedure is complete, the instrument user interface screen goes blank (black), indicating that it is safe to turn off the power to the instrument.

To power down your system, set the power switch to the OFF (**O**) position.

Chapter 3:

Use, Maintenance, & Diagnostics

Using the Discovery SDT

All of your SDT experiments will have the following general outline. In some cases, not all of these steps will be performed. The majority of these steps are performed using the instrument control software. The instructions needed to perform these actions can be found in the online help in the instrument control program; therefore, they will not all be covered in detail here.

- Calibrating the instrument
- Selecting the mode and signals to save
- Selecting the desired pan size
- Selecting the sample purge and secondary gas flow rates
- Creating or choosing the test procedure and entering experiment information in TRIOS software
- Selecting and taring two empty sample pans on the sample and reference beams
- Loading the sample
- Closing the furnace
- Starting the experiment
- Unloading the sample at the end of the experiment

To obtain accurate results, follow procedures carefully and check calibration periodically (once a month).

Before You Begin

Before you set up an experiment, ensure that the Discovery SDT system and the controller have been installed properly. Make sure you have:

- Connected the instrument with the controller
- Connected all gas lines
- Installed any desired options
- Powered up the Discovery SDT
- Become familiar with controller operations
- Calibrated the Discovery SDT, if necessary

Calibrating the Discovery SDT

To obtain accurate experimental results, you should calibrate the instrument upon initial installation. For the best results, however, you should recalibrate periodically. At a minimum, the SDT should be recalibrated anytime the beam set, experimental heating rate, or purge gas is changed.

Several types of calibration are required for the SDT:

- Autosampler calibration: Calibration of the optional Autosampler
- TGA weight calibration: Calibration of the TGA weight signal
- DTA baseline calibration: Calibration of the Delta T signal
- Temperature calibration: Calibration of the temperature signal
- DSC heat flow calibration: Calibration of the heat flow signal
- Dual sample calibration: Calibration of the dual sample weight signals
- MDSC calibration: Calibration of the Reversing Heat Capacity signal.

A brief description of each calibration is provided below. Autosampler calibration is performed via the instrument user interface. All other calibrations are performed through TRIOS software. For more details on performing each type of calibration, refer to the instructions in TRIOS software Online Help.

NOTE: Please make sure that you run your experiments with the same gas that you used to calibrate the system. For example, if you calibrate using nitrogen, make your runs with nitrogen.

Autosampler Calibration

Perform an Autosampler calibration after initial installation of the system, and calibrate periodically thereafter as needed.

If the Autosampler fails to pick up a sample pan correctly during an automatic loading procedure, the Autosampler may need to be calibrated, or the instrument is not level.

To perform an Autosampler calibration, access the Autosampler panel on the user interface. Refer to TRIOS software Online Help for detailed instructions.

TGA Weight Calibration

Weight calibration is required upon initial installation of the system. After the initial weight calibration, weight calibration should then be done periodically (once a month is recommended).

Weight calibration is performed in TRIOS by accessing **Calibration** from the File Manager and then opening the **Calibration Setup** tab. This procedure involves making two runs: an empty beam run and a run with the calibration weights.

See TRIOS software Online Help for more information on weight calibration of either type.

DTA Signal Setup

DTA Signal Setup is based on analyzing Delta T data collected from a baseline run conducted over the temperature range expected in subsequent experiments. This experiment usually utilizes the same empty beam baseline run obtained for TGA Weight Calibration.

NOTE: DTA signal setup is performed automatically when a Weight Calibration is run.

The DTA signal setup is corrected by a linear (slope and offset) function of the sample temperature. This results in shifting and rotating the baseline so that the calibrated portion is near 0°C.

NOTE: The DTA signal setup is not required when using the SDT as a DSC-TGA (i.e, when not saving the Delta T signal). Calibration allows you to collect quantitative Heat Flow data instead of just qualitative DTA data. However, the shape of the DTA baseline is valuable as a quick verification that the SDT beams are correctly positioned and that DSC heat flow calibration can be successfully completed.

Temperature Calibration

Temperature calibration is based on evaluation of the melting endotherm(s) of a high purity metal standard(s). The recorded onset of melting of this standard(s) is compared with the literature value and the difference is calculated for temperature calibration. Up to five standards may be used. Zinc (419°C) is supplied in the SDT accessory kit. Other suitable standards are tin (232°C), aluminum (661°C), silver (961°C), gold (1064°C) and nickel (1455°C). If you use one known standard and observed melting point, the entire curve is offset, or shifted, to the actual melting point. If you use multiple standards, the temperature is corrected by a cubic spline fit. The multiple-point temperature calibration is more accurate than the one-point calibration.

See TRIOS software Online Help for more information

DSC Heat Flow Calibration

SDT heat flow calibration is based on analyzing the heat capacity curve for sapphire over a selected temperature range and the heat of fusion of high purity zinc metal. Three experimental runs are required: one using an empty alumina cup (90 µL) for both the reference and sample (baseline run) and one using a sapphire standard (supplied in the SDT Accessory Kit) as the sample. The measured heat capacity for sapphire is compared with the literature value at multiple temperatures across the range and mathematically fitted to generate a heat flow calibration curve.

This calibration curve may be further refined by a third experiment that measures the heat of fusion of high purity zinc wire (supplied in the accessory kit). The heat of fusion is measured and the cell constant is calculated using the known value of the heat of fusion of zinc (108.7 J/g) and the equation “Cell Constant = Known Value/Measured Value.” The calculated value of the cell constant is then entered into the software.

Dual Sample Calibration

Dual Calibration is an alternative method for providing reference data that allows both pans for use with samples. A baseline run is made with tared, empty pans on both sides and the weight and temperature profiles observed for each side are stored as cubic spline functions of temperature. These stored profiles then provide historical reference data for subsequent Dual Sample runs. For optimum results the same cups should be used in subsequent dual sample experiments.Dual Sample Calibration is automatically performed during a heat flow calibration. No separate calibration is required.

MDSC Reversing Heat Capacity Calibration

A heat capacity calibration curve is generated by running a sapphire sample over a desired temperature range using appropriate modulated conditions. The collected Reversing Heat Capacity curve is calibrated against the true value of the heat capacity of sapphire over the experimental temperature range.

Running a Discovery SDT Experiment

All of your SDT experiments will have the following general outline. In some cases, not all of these steps will be performed. See TRIOS software Online Help for anything not covered in this manual.

1 Attaching and setting up external accessories and/or environmental conditions as required, such as the purge gas

2 Selecting the test type and signals to be saved

3 Selecting the pan size and material

4 Taring the empty sample and reference pans

5 Loading the sample into the pan

6 Entering experiment information in TRIOS software; this includes both sample and instrument information.

7 Creating or selecting the experimental procedure using TRIOS software

8 Starting the experiment

Selecting the Pan Size and Material

Choose a pan based on the desired temperature range and application. See "Discovery SDT Sampling System" on page 19 for the pans that can be used with the Discovery SDT Autosampler.

CAUTION: Using platinum pans at high temperatures (i.e., above 800°C) increases the likelihood that they may stick to the platinum sensor in the sample and reference platforms. This can cause damage to the sensor when the cup is removed, and may result in the need to replace the sample and/or reference beam assembly. To prevent this from occurring, you can (1) use a ceramic pan, or (2) place some finegrain alumina powder between the platinum pan and the sensor before you start a TGA-DTA experiment.

MISE EN GARDE: L'utilisation de casseroles de platine à des températures élevées (c'est-à-dire au-dessus de 800 ° C) augmente la probabilité qu'elles puissent adhérer au capteur de platine dans l'échantillon et / ou les plates-formes de référence. Cela peut endommager le capteur lorsque la coupelle est enlevée et peut entraîner la nécessité de remplacer l'échantillon et l'assemblage du faisceau de référence. Pour éviter que cela ne se produise, vous pouvez (1) utiliser une casserole en céramique, ou (2) placer une poudre d'alumine fine grain entre la casserole de platine et le capteur avant de commencer une expérience TGA-DTA.

Taring the Sample Pan(s)

Taring of all sample pans on the tray must be done before the sample is loaded to ensure that the balance gives you an accurate reading. You should tare the SDT pans before each experiment with the furnace closed, even if you use the same pan in consecutive experiments.

NOTE: A sample pan of the same size and type that will be used for your experiments is required on the reference side for proper operation.

When you tare a pan, the SDT reads the weight of the empty pan and then stores the weight as an offset, which is subtracted from subsequent weight measurements. For optimum accuracy, the weight reading must be stable before it is accepted as an offset. When you use the automatic tare procedure, the SDT will determine when the weight reading is sufficiently stable.

Because the SDT has two balance assemblies, taring is done for both the reference and sample SDT pans.

NOTE: Always use tweezers to handle SDT pans.

Manual Taring

1. Open the furnace.
2. Place clean, empty pan(s) on the platform for the front sample balance assembly, making sure it is seated properly.
3. Place an empty SDT pan on the platform for the back reference balance assembly, making sure that it is seated properly.
4. Close the furnace to protect the pans from air currents.
5. Specify the pan type in TRIOS software. Only pans of the same type may be run in the same sequence.
6. Press the **Tare** button on the user interface. Alternatively, select **Manual Tare** from the Autosampler control panel.

NOTE: The instrument will not tare if the furnace is open, if the temperature is changing too rapidly (i.e., if the temperature causes the weight to change by more than 3.0 µg in a 10-second period), or while the instrument is in calibration mode.

Automatic Taring

1. Click on the **Instrument** tab and select **Tare**, or navigate to the Tare screen on the User Interface.
2. Select the desired pans, then select **Start** from the user interface or from TRIOS to initiate the tare procedure. The pan will automatically be loaded and the furnace closed to make the measurement. When the tare procedure is complete, the furnace automatically opens and the Autosampler unloads the pan.

Loading the Sample Pans

CAUTION: Spilling sample material on the platform could cause permanent contamination of the platform. If this occurs, the contaminated balance beam(s) would need to be replaced. Therefore, remove the pans from the beams when loading samples.

MISE EN GARDE: Le fait de déverser du matériau d'échantillon sur la plate-forme pourrait causer une contamination permanente de la plate-forme. Dans ce cas, il faudrait remplacer le (s) faisceau (s) d'équilibre contaminé (s). Par conséquent, retirez les casseroles des poutres lors du chargement des échantillons.

1. If operating the SDT manually, remove the sample pan from the front (sample) platform. Place the sample material in the sample cup and replace the pan onto the platform.
2. If using an Autosampler, place the sample material in the cup and position the cup at the appropriate position on the Autosampler tray.

NOTE: If the weight is out of range (<Range or >Range), the furnace will not close. This feature prevents damage to the balance beams.

Dual Sample Operation

The Discovery SDT can be operated in dual sample mode. This mode provides comparative TGA-DTA information for two samples simultaneously. When running dual sample experiments, signals are captured in a single data file for sides A (sample) and B (reference). Dual sample mode is selected on the General page of TRIOS Options.

NOTE: The signals saved assume that Weight A is from the sample (front) platform and Weight B is from the reference (back) platform.

When running dual sample experiments, follow these recommendations:

- Perform a heat flow calibration, if necessary. This calibration includes the collection of the appropriate dual sample calibration curves.
- Enter the names of the two samples in the “Sample Name” field (e.g., Sample 1/Sample 2).
- Position two clean SDT pans on the Autosampler tray in the desired positions. Then proceed to the TRIOS Tare screen to tare the two pans. (For optimum results the sample pans used for dual sample calibration should be used.)
- When dual sample mode is selected, a different list of saved signals is available for storage in the data file. These signals include Temperature Difference (°C), Temperature Difference (°C/mg), Weight (mg), and Weight Percent (%) for both side A (sample beam side) and side B (reference beam side). It is important to select the normalized signals (°C/mg and % signals), if these signals are desired for display and analysis within the instrument control software.

NOTE: Only six signals can be selected for display at any one time on the instrument touch screen. Therefore, in order to select the dual sample signals, two of the standard default signals must be unchecked.

- The absolute accuracy of the weight and temperature difference results obtained in dual sample operation is not as good as those obtained in single sample operation. Therefore, single sample operation is recommended for optimum results. In addition, it is recommended that the dual sample mode be used only for ramp experiments.
- The data for both samples (A and B) are contained in a single data file. You can separate the data by selecting the corresponding signal group (A or B) within the instrument control software.

Using the Purge Gases

Balance Purge

The balance purge is regulated by an internal Gas Delivery Module and a gas switching accessory. The GDM continuously monitors and controls the gas flow rate and allows the flow rate to be stored in the data files, if desired. The balance purge is used in all SDT experiments. Typical flow rate is 100 mL/min.

Sample Purge

The sample purge is regulated by an internal Gas Delivery Module and a gas switching accessory. The GDM continuously monitors and controls the gas flow rate and allows the flow rate to be stored in the data files, if desired, and the gas switching accessory allows the sample purge gas to be changed in the middle of an experiment.

The sample purge is used in all SDT experiments. Typical flow rate is 100 mL/min.

Reactive Gas Purge

The reactive purge provides additional experimental flexibility for introduction of a more reactive gas into the sample region.

The flow rate of the reactive gas purge is also typically 100 mL/min.

The reactive gas purge rate is controlled using an optional external flow meter and is not a stored signal. The reactive gas purge is not used in most SDT experiments.

Starting an Experiment

Before you start the experiment, ensure that the Discovery SDT is connected with the controller and you have entered all necessary information through the instrument control software.

Start the experiment by selecting **Start** on the instrument control software or touching the **START** button on the instrument user interface. If using an Autosampler, when you start the instrument, the system automatically loads the sample pan and closes the furnace if necessary, and then runs the experiment to completion.

NOTE: Once the experiment is started, operations are best performed at the computer keyboard. The SDT is very sensitive to motion and might pick up the vibration caused by touching a button on the instrument touch screen.

If multiple runs are in the sequence and the instrument is outfitted with an Autosampler, the procedure repeats for the next run until the run sequence completes.

Stopping an Experiment

If you need to discontinue the experiment, you can stop it at any point by selecting **Stop** through the instrument control software or by touching the **STOP** button on instrument user interface.

NOTE: See "Shutting Down the Discovery SDT System" on page 31 for information on shutting down the instrument.

Maintaining the Instrument

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. Consult TRIOS software Online Help for further information.

DANGER: Because of the high power circuitry in this instrument, untrained personnel must not attempt to test or repair any electrical circuits.

DANGER: À cause de la présence de circuits électriques de grande puissance dans cet instrument, le personnel non formé ne doit pas essayer de tester ou de réparer les circuits électriques.

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.

Conditioning the Balance

Balance conditioning is required for the Discovery SDT in order to achieve optimum performance from your instrument and keep the balance housing dry. Perform a balance conditioning when the instrument is first installed and whenever the balance housing is opened.

In order to ensure an inert atmosphere, the balance housing will need to be flushed of air. This could take up to 12 hours.

To maintain a dry balance after conditioning, use a balance purge rate of 100 mL/min.

Cleaning the Furnace

After extended use, the inside of the furnace may become coated with sample residue and therefore require cleaning. Periodic cleaning is recommended for optimum operation and performance. It is recommended that you clean the furnace by first removing the cover and then heating it to approximately 1000°C in air at a ramp rate of 20°C/minute. Keep the time allowed for this cleaning to a minimum. Leaving the furnace at high temperatures for extended periods of time may shorten the life of your furnace.

See TRIOS software Online Help for furnace cleaning instructions.

Cleaning the Pans

The SDT platinum and ceramic sample pans are designed to be reusable. However, they must be thoroughly cleaned between experiments. This is typically accomplished by “burn-off” of residue with a propane torch. In some cases, soaking the pans in an appropriate solvent provides another alternative. Care must be taken not to deform the pan during cleaning, or the SDT automatic pan pick-up process will not work.

Cleaning the User Interface Screen

You can clean the SDT user interface screen as often as you like. The instrument should first be turned off in order to prevent activating the touch screen switches, which can result in unexpected instrument behavior. The user interface screen should be cleaned with a household liquid glass cleaner and soft cloth. Wet the cloth (not the user interface screen) with the glass cleaner, and then wipe off the screen and surrounding surfaces.

WARNING: Do not use harsh chemicals, abrasive cleansers, steel wool, or any rough materials to clean the user interface screen, as you may scratch the surface and degrade its properties.

AVERTISSEMENT: N'utilisez pas de produits chimiques agressifs, de nettoyants abrasifs, de la laine d'acier ou tout autre matériau rugueux pour nettoyer l'écran de l'interface utilisateur, car vous pourriez égratigner sa surface et dégrader ses propriétés.

Replacing Fuses

WARNING: Always unplug the instrument before you examine or replace the fuses.

AVERTISSEMENT: Débranchez toujours l'instrument avant d'examiner ou de remplacer les fusibles.

The Discovery SDT contains internal fuses that are not user serviceable. If any of the internal fuses blows, a hazard may exist. Call your TA Instruments service representative.

The only customer-replaceable fuses are in the fuse holder located on the back panel of the instrument. To check or change these fuses:

1. Turn the instrument off and remove the power cord.
2. Next to the instrument power entry module, there are two standard fuse holders. Use a flat-blade screwdriver to remove the fuse carriers.

Figure 9 Remove fuse carriers.

3 Remove old fuses from the fuse carriers and replace the fuses only with the type and rating indicated on the instrument's rear panel.

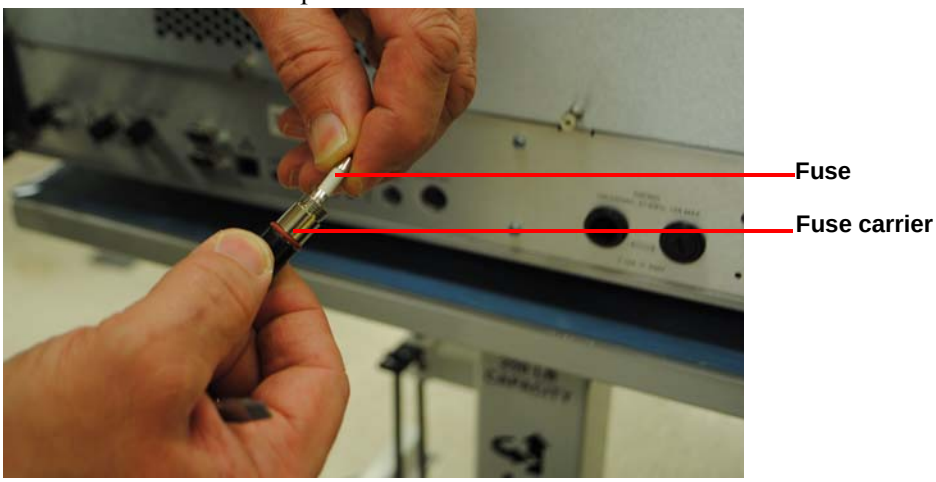

Figure 10 Remove fuse.

4 Place the fuse carriers back into the fuse holders and lock in place with the screwdriver.

Replacing a Beam

1 Loosen the six screws (three on the left side of the meter housing and three on the right side) on the meter housing and lift to remove it.

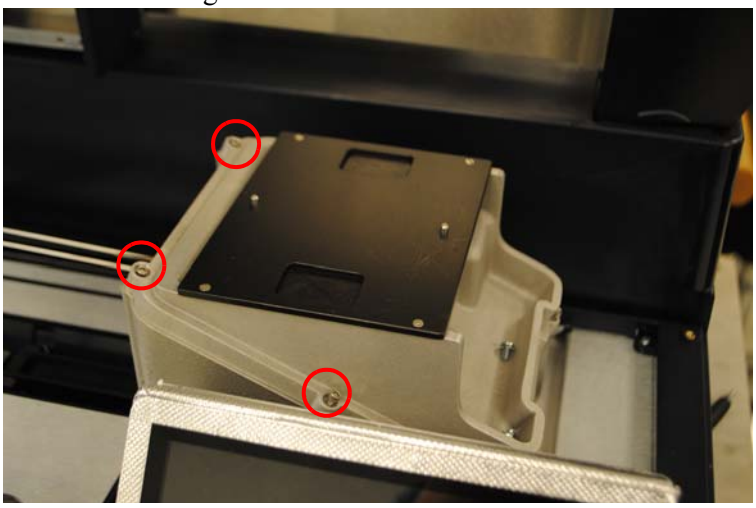

Figure 11 Screws on the meter housing (3 of 6 shown).

2 Lift up the IR shield.

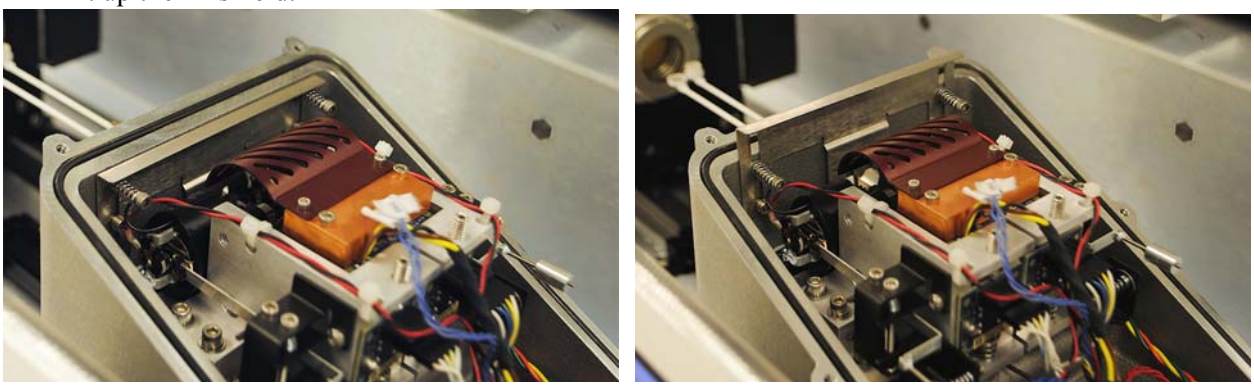

Figure 12 Left: IR shield lowered; Right: IR shield raised.

3 Using the T2 Torx screwdriver (included in the accessory kit), remove the beam receptacle screw from the beam being replaced.

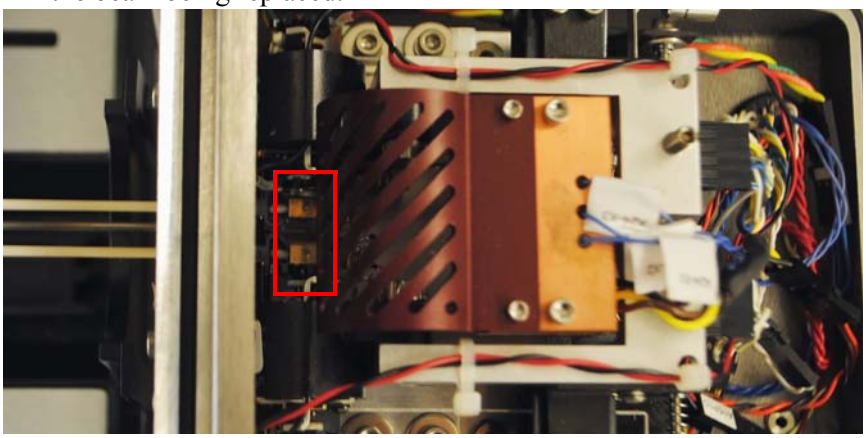

Figure 13 Beam receptacle screws.

CAUTION: Do not place any objects through the red shield protecting the connector wires. Doing so can result in damage to the balance.

MISE EN GARD: Ne placez aucun objet à travers le blindage rouge protégeant les fils du connecteur. Vous risqueriez d'endommager l'équilibre.

CAUTION: DO NOT PRESS DOWN on the beam receptacle screws. Doing so can result in damage to the balance.

MISE EN GARD: NE PAS APPUYER VERS LE BAS sur les vis du réceptacle du faisceau. Vous risqueriez d'endommager l'équilibre.

4 Remove the beam.

5 Follow the instructions in reverse order to install a new beam.

6 Return the shield to the closed (down) position.

7 Replace the meter housing.

Aligning the Beams

1 Raise the beam alignment tool.

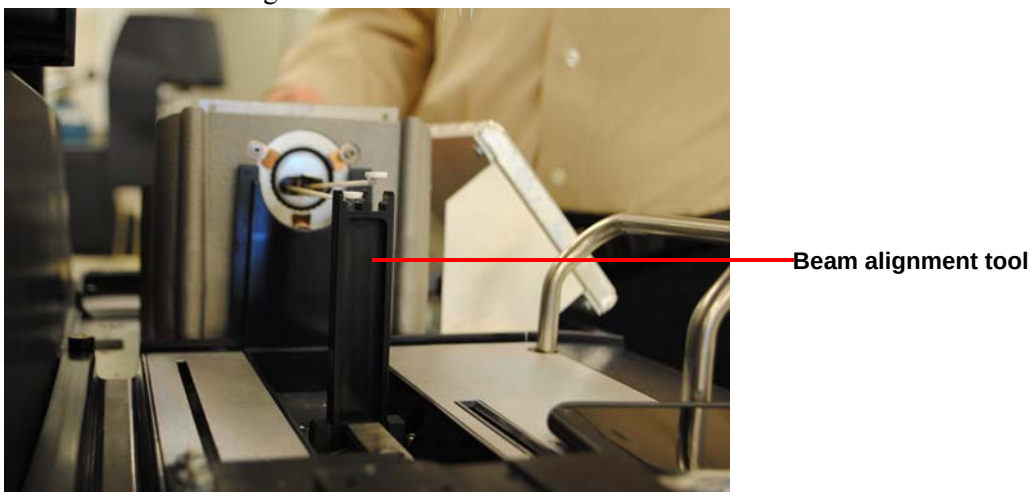

Figure 14 Beam alignment tool.

CAUTION: Do not place any objects through the red shield protecting the connector wires. Doing so will damage the balance.

MISE EN GARD: Ne placez aucun objet à travers le blindage rouge protégeant les fils du connecteur. Vous risqueriez d'endommager l'équilibre.

2 Loosen the balance meter bracket screws on the side of the beam that you are aligning.

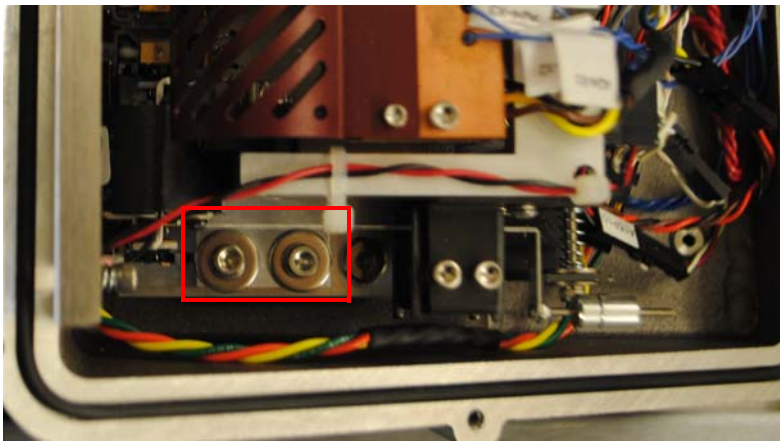

Figure 15 Balance meter bracket screws.

3 To align the beam horizontally, adjust the theta screw (located behind the tare weight) until the beam rests in the alignment tool pocket.

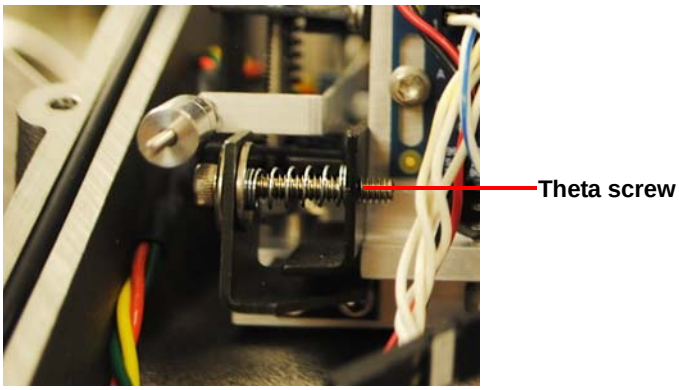

Figure 16 Theta screw.

4 To align the beam vertically, loosen the photo detector board screws (two screws per board, shown below) and then turn the set screw directly above the board to adjust the beam up or down until it rests in the beam alignment tool. The beam should float in the tool when finished..

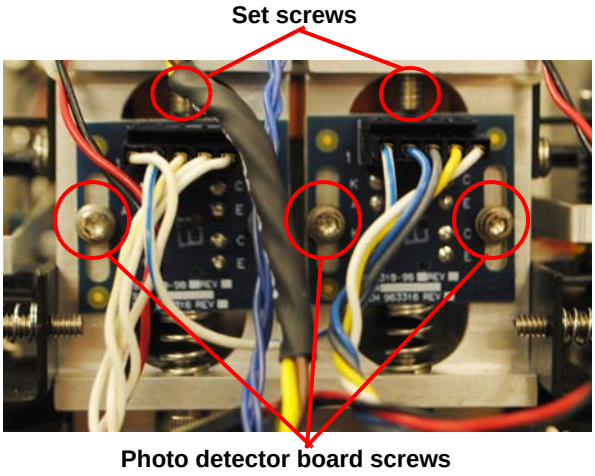

Figure 17 Photo detector board screws (3 of 4 shown) and (2) set screws.

Replacement Parts

Replacement parts for the Discovery SDT are listed below. Refer to the tables below when ordering parts.

Table 8: Fuses, Cords, and Cables*

Part Number	Description
200721.002	Power cord, 240 V
201242.001	Fuse 12-amp time delay, 250 V (T12 A H 250 V)
251470.010	Ethernet cable (10 foot, shielded)
920223.901	Event cable

*Contact your local TA Instruments representative for information on non-US style power cords.

Table 9: Discovery SDT Tools and Parts

Part Number	Description
259508.000	Brass tweezers
259509.000	Spatula, curved, 165 mm long
270226.022	O-ring, furnace tube
963259.901	Furnace assembly
963881.901	SDT type R beam - single

Table 10: Standard Series Pans and Lids

Part Number	Description
963420.001	30-pan tray, Autosampler
960148.901	Platinum sample pans, 40 µL; pkg of 3 (for TGA-DTA studies)
960149.901	Platinum sample pans, 110 µL; pkg of 3 (for TGA-DTA studies)
960072.901	Alumina sample pans, 40 µL; pkg of 3 (for TGA-DTA studies)
960070.901	Alumina sample pans, 90 µL; pkg of 3 (for DSC-TGA studies)
960239.901	Alumina sample lids; pkg of 3 (for DSC-TGA studies)

Table 11: SDT Calibration and Reference Materials

Part Number	Description
915079.903	Sapphire DSC heat flow calibration standard
900905.901	Calcium oxalate
900907.901	Vial of zinc wire for temperature calibration
960014.901	Calibration weight set
960034.901	Aluminum oxide reference material for TGA-DSC studies