



DISCOVERY XENON FLASH DXF 200+



Getting Started Guide

Revision C Issued February 2024

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
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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 *DXF 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.

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).

NOTE: This equipment has been tested and found to comply with the limits for Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interference when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with the instruction manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at own expense.

Supplier's Declaration of Conformity

47 CFR § 2.1077 Compliance Information

Unique Identifier: 858001.901

Responsible Party:

TA Instruments
159 Lukens Drive
New Castle, DE 19720
302-427-4000
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 when placing the Environmental Module onto the Source Module.</p> <p>Ce symbole indique qu'il existe un risque d'écrasement des mains. Faites attention lorsque vous placez le module environnemental sur le module source.</p>
	<p>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 system.</p> <p>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.</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.

Electrical Safety

DANGER: This pulse source operates from a high voltage power supply. Do not override the safety interlocks as they prevent contact with potentially lethal voltages.

DANGER: Cette source d'impulsion fonctionne à partir d'une alimentation électrique haute tension. N'annulez pas les dispositifs de sécurité car ils empêchent tout contact avec les tensions potentiellement mortelles.

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 furnace conditioning procedure on page 62 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 procédure de conditionnement de la fournaise à la page 44 pour la méthode utilisée pour sécher l'équipement avant utilisation.

Liquid Nitrogen Safety

Liquid nitrogen is used to cool the furnace below ambient temperature on the EM-200+ Environmental Module. Because of its low temperature (-196°C [-321°F]), liquid nitrogen may burn the skin. When you work with liquid nitrogen, use the following precautions:

NOTE: Please adhere to your company's safety guidelines for handling liquid nitrogen.

WARNING: Liquid nitrogen boils rapidly when exposed to room temperature. Be certain that areas where liquid nitrogen is used are well ventilated to prevent displacement of oxygen in the air.

AVERTISSEMENT: L'azote liquide bout rapidement lorsqu'il est exposé à la température ambiante. Assurez-vous que les zones où l'azote liquide est utilisé sont bien aérées pour éviter le déplacement de l'oxygène dans l'air.

WARNING: Cryogenic blow back possible if dewar is rapidly filled with liquid nitrogen. Fill partially and allow for initial boil-off prior to filling completely.

AVERTISSEMENT: Possibilité de projections de gaz très froid si le Dewar est rapidement rempli avec de l'azote liquide. Remplir partiellement et permettre une évaporation initiale avant de remplir complètement.

- 1 Wear goggles or a face shield, thermally insulated gloves large enough to be removed easily, and a rubber apron. For extra protection, wear high-topped, sturdy shoes, and leave your pant legs outside the tops.
- 2 Transfer the liquid slowly to prevent thermal shock to the equipment. Use containers that have satisfactory low-temperature properties. Ensure that closed containers have vents to relieve pressure.

Thermal Safety

DANGER: Source instrument surfaces can be hot enough to cause discomfort when in contact with the skin during a sample run. If you are conducting a subambient test, cold could also cause injury. After running any type of experiment, you must allow the DXF system to return to near room temperature before you touch the inner furnace surfaces. Always use the available software monitor to display temperature even after the termination of the test cycle.

DANGER: Les surfaces de l'instrument source peuvent être assez chaudes pour provoquer un malaise au contact de la peau pendant l'analyse d'un échantillon. Si vous effectuez un essai à basse température, le froid peut également provoquer des blessures. Après avoir effectué un type d'expérience quelconque, vous devez laisser le système DXF revenir à la température quasi ambiante avant de toucher les surfaces internes de la four. Utilisez toujours le moniteur logiciel disponible pour afficher la température même après la fin du cycle d'essai.

WARNING: In the event of a power failure, do NOT open the furnace.

AVERTISSEMENT: En cas de coupure de courant, N'ouvrez PAS le four.

If the power has not come back on after a power failure, do NOT open the furnace. The temperature controllers will not be displaying a temperature so you will not know what the temperature is inside the furnace.

The test is over once a power failure occurs. This is no recovery method to restart the test at that point, but all data is saved up until the power failure occurs.

Do not press any buttons on the front panel after a power failure, just start a test normally. If the test was running under vacuum, verify the vacuum.

Chemical Safety

WARNING: Do not use hydrogen, oxygen, or any other explosive gas in the DXF system. Only inert gas, such as nitrogen or argon, should be used.

AVERTISSEMENT: N'utilisez pas d'hydrogène, d'oxygène ou tout gaz explosif dans le système DXF. Seuls les gaz inertes comme l'azote ou l'argon doivent être utilisés.

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 Discovery Xenon Flash

Overview

Thermal diffusivity (α) is the thermophysical property defined as a ratio of the thermal conductivity and the volumetric heat capacity. The thermal diffusivity in SI units is cm^2/s . The most popular method used for measuring thermal diffusivity is the flash method. It has the advantage of being fast while providing values with excellent accuracy and reproducibility. The flash diffusivity method involves uniform irradiation of a small, disc-shaped specimen over its front face with a very short pulse of energy. The time-temperature history of the rear face is recorded through high-speed data acquisition from a solid-state optical sensor with very fast thermal response, and thermal diffusivity is determined from the time-dependent thermogram of the rear face. Thermal conductivity can be calculated as a product of the thermal diffusivity, the specific heat, and the density of the material. A Discovery Xenon Flash (DXF) system automatically determines the thermal conductivity using the measured (or separately entered) specific heat capacity and thermal diffusivity, with separately-entered density data.

The DXF system is comprised of a DXF Pulse Source, Environmental Module, IR Detector Assembly, and associated software. The DXF with EM 200+ is shown below.

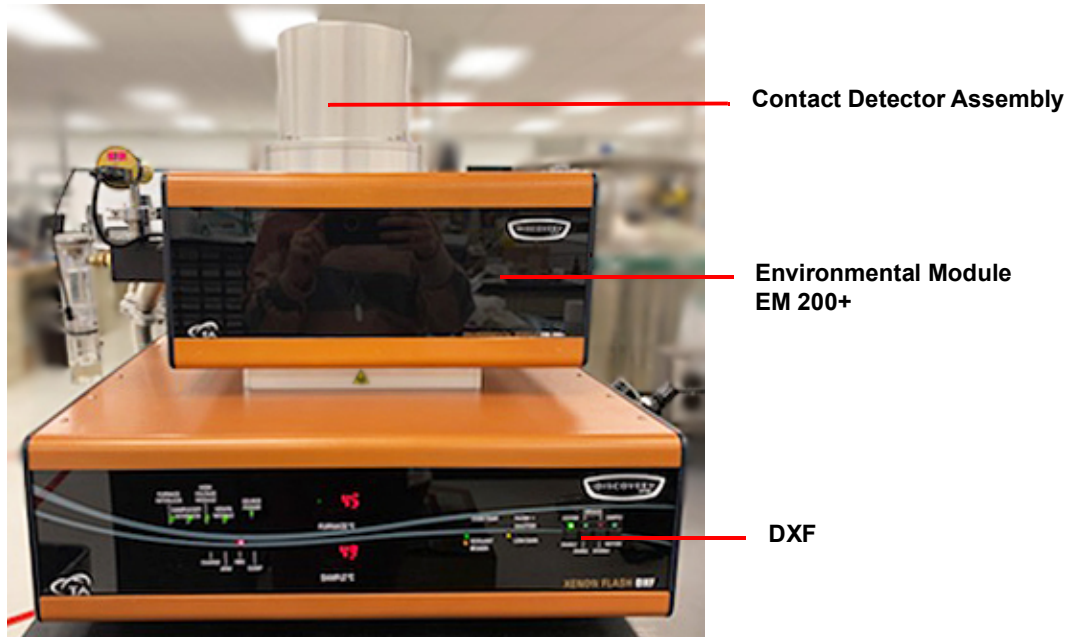


Figure 1 DXF Source Module with EM 200+ and Detector Assembly.

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 necessary information.
- Stores experimental data.
- Allows data analysis of results.

NOTE: For technical reference information, theory of operation, and other information associated with the DXF system not found in this manual, see the online help associated with the instrument control software.

DXF System Components

A DXF system has three major components: the DXF Pulse Source module, the Environmental Module with Detector Assembly, and the controller.

Pulse Source Module

The Discovery Xenon Flash (DXF) covers the most commonly needed cryogenic to 900°C temperatures with a range of Environmental Modules, and employs a High Speed Xenon-Pulse Delivery Source (HSXD). The reflective optic configuration effectively harnesses the power of a Xenon flash tube, and, with the aid of proprietary wave guides, delivers it to the sample inside the Environmental Module. The DXF produces a maximum pulse width of 650 μ s while uniformly concentrating sufficient power from the flash source directly on the entire face of the sample. This optimized energy gathering scheme and the broad light spectrum allows samples as large as 25 mm in diameter to be illuminated with sufficient energy.



Figure 2 DXF Pulse Source Module.

Table 1: DXF Front Panel LED Indicators

Indicator	Function
FURNACE INTERLOCK	GREEN indicates that the EM is sitting correctly on top of the DXF. RED indicates that EM is sitting incorrectly and must be adjusted.
SAMPLE/DETECTOR INTERLOCK	GREEN indicates that the Detector Head is closed and ready for use, and that the detector is secured correctly to the EM. RED indicates that the Detector Head is open or the detector is not secured.
HIGH VOLTAGE MODULE	GREEN indicates that the high voltage module is closed and ready for use. RED indicates that the module is open.
XENON MODULE	GREEN indicates that the xenon module is closed and ready for use. RED indicates that the module is open.
SOURCE POWER	ON indicates that the xenon control board is powered.
CHARGE/ARM	ON / Blinking indicates that the lamp is charging.
FIRE	ON indicates that the fire command was executed.
DUMP	ON indicates that the laser system is in dump mode (not charging and not firing).
FURNACE °C	Indicates the current temperature of the furnace.
SAMPLE °C	Indicates the current temperature of the sample.
OVER TEMP	ON indicates one of the following: <ul style="list-style-type: none">• The EM has gone over its temperature limit• One of the thermocouples is broken or not connected As a result, the furnace is disabled and will not heat.
COOLANT	ON indicates that the internal bypass is in place. Not in use.
POWER	ON indicates the DXF system is powered.
FILTER 1	ON indicates Filter1 is in and ready for use.
SHUTTER	ON indicates that the shutter is open and ready for use.
LOW GAIN	ON indicates the detector amplifier has low gain values.
SYSTEM ENABLE switch	ON indicates that the system is ready to execute all acquisition functions.
FURNACE ENABLE switch	ON indicates that the furnace is available and ready to heat/control the temperature parameters.

Table 1: DXF Front Panel LED Indicators

Indicator	Function
FURNACE DISABLE switch	ON indicates one of the following: <ul style="list-style-type: none">• The furnace is unable to heat• Heat to the furnace was stopped• Any of the conditions for enabling the furnace were violated
SAMPLE MOTION switch	Not in use.

Environmental Modules and Detector Assembly

The Environmental Module includes a resistance-heated and liquid nitrogen cooled furnace, providing temperature control of the sample operation in dry air or inert gas, or in vacuum. The EM also includes a user-replaceable multi-sample tray holder (Autosampler), supporting a variety of sample sizes. and a contact detection system.

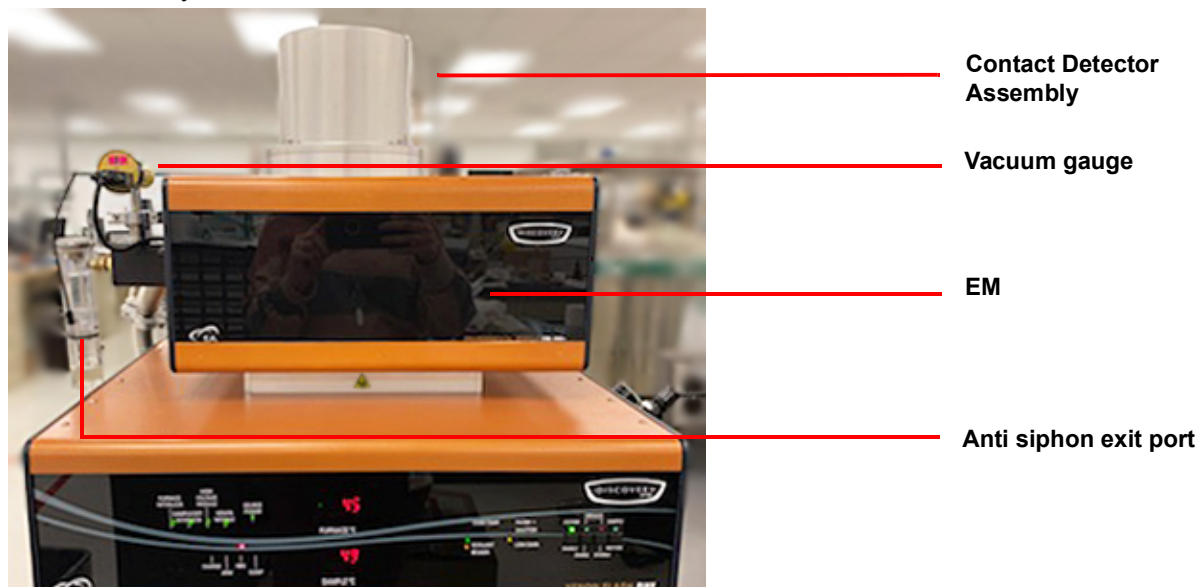


Figure 3 EM and Contact Detector Assembly.

Instrument Specifications

The tables found below contain the technical specifications for the DXF and EM.

Table 2: DXF 200+ System Technical Specifications

Item/Area	DXF 200+ Specifications
Dimensions	Depth 66.04 cm (26 in) Width 62.87 cm (24.75 in) Height 57.79 cm (22.75 in)
Weight	83 kg (183 lbs)
Power ^a	System supply voltage: 198–242 VAC (rated for 6A) 50/60 Hz.
Operating environmental conditions	Temperature: 15 to 35°C Relative humidity: 5 to 80% (non-condensing) Installation Category II Pollution Degree 2 Maximum altitude: 2000 m The degree of protection for this instrument according to EN 60529 is IP20.
Pulse Source	High speed Xenon module

- a. This instrument is supplied with a power cord 2.4 m (7.75 ft) long, rated for 10A and 250V and a USA NEMA 6-15P style plug. The TA Representative installing the instrument will provide the appropriate power cable for the geographical location where the instrument is being installed. Connect the instrument, computer, and monitor to wall outlets on the same circuit and make sure that the mains assigned do not also supply power to noise generating equipment nearby, such as motors, welders, transformers, etc.

Table 3: EM Technical Specifications

Item/Area	EM 200+
Thermocouple Measurement	Type E
Thermal Diffusivity	Accuracy** $\pm 4\%$
Measurement	Repeatability** $\pm 2\%$
Temperature Range	-175 to 200°C
Atmosphere	Air, inert gas, vacuum
Number of Specimens	4 to 12 (size dependent)
Sample Dimension	8 mm to 25.4 mm
Sample Shape	Round disk, square plate (with tray)
Detector Assembly	Thermoelectric Contact Detector
Vacuum	10mTorr with standard pump, 0.1mTorr with Turbo pump

**Using ideal, well-defined, well-behaved 12.7 mm diameter samples of known properties

Chapter 2:

Installing the DXF 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 DXF is inspected both electrically and mechanically so that it is ready for operation upon proper installation. Only limited instructions are given in this manual; consult the online documentation for additional information. Installation involves the following procedures:

- Inspecting the system for shipping damage and missing parts
- Choosing a location for instrument installation
- Connecting the Environmental Module to Pulse Source Module
- Setting up system communication with the controller (computer)
- Connecting the utilities
- Installing the tray
- Installing the pins
- Performing calibrations

It is strongly recommended that you have your DXF 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 DXF 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 DXF experiments, it is important to choose a location for the instrument using the following guidelines. The DXF 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.
- The controller.
- Compressed lab air and purge gas supplies with suitable regulators and filtering where needed.
- Cooling water/coolant (stable chiller/circulator preferred).
- Vacuum pump and vacuum connections.

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.

CAUTION: Drying out the instrument may be needed if it has been exposed to humid conditions. Refer to the furnace conditioning procedure on page 62 for the method used to dry out the equipment before use. It is important to be certain that the instrument ground is adequately connected to the facilities ground for safe operation.

MISE EN GARDE: Le séchage de l'instrument peut être nécessaire s'il a été exposé à des conditions humides. Reportez-vous à la procédure de conditionnement de la fournaise à la page 62 pour la méthode utilisée pour sécher l'équipement avant utilisation. Il est important de s'assurer que la

masse de l'instrument est correctement connectée à la masse de l'installation pour un fonctionnement sûr.

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 communication 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 EM, place hands on the outside bottom corners of the EM and lift upwards, keeping the module in an upright position. Use the same procedure to move the DXF.



WARNING: Hand crush hazard is present. Do not place hands between the Environmental Module and the DXF.

AVERTISSEMENT: Un risque d'écrasement des mains est présent. Ne placez pas les mains entre le module environnemental et le DXF.



Place hands on the corners of the EM



Place hands on the outside of the DXF feet

Figure 4 Lifting the EM (top) and lifting the DXF (bottom).

Connecting the Environmental Module to the Pulse Source Module

NOTE: Refer to [Appendix A: Connection Diagram](#) for a diagram of all instrument connections.



WARNING: Hand crush hazard is present. Do not place hands between the Environmental module and the DXF.

AVERTISSEMENT: Un risque d'écrasement des mains est présent. Ne placez pas les mains entre le module environnemental et le DXF.

Before making any connections, place the Environmental Module on top of the DXF so that they are stacked; make sure to align the 4 pins on the bottom of the EM with the corresponding holes on the top of the DXF. Allow an additional 28 cm (11 in) clearance to the right of the instrument for opening the Autosampler on the EM.

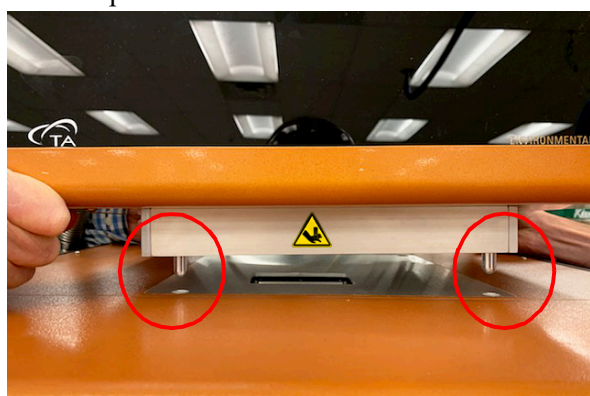


Figure 5 Pins on the bottom of the Environmental Module (EM-200+ shown).

To connect the Environmental Module, you will need access to the DXF instrument back panel and the Environmental Module back panel.

NOTE: Connect all cables before connecting the power cords to outlets. Tighten the screws on all cables.

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.

DXF Back Panel

The DXF back panel has nine ports; the table below provides a description of the function of each port. Refer to [Figure 6](#) for an illustration of rear connections. Ports not described and not labeled are not used.

Table 4: DXF Back Panel Ports and Functions

Port	Function
EM CONTROL Port	Provides communication between the DXF and the EM module
AUTOSAMPLER Port	Provides communications between the autosampler electronics and DXF
DETECTOR Port	Provides communication between the DXF and the Detector Assembly
DAQ Port	Provides communication between Data Acquisition board inside the DXF and the controller
T/C Ports	Connects the sample and furnace thermocouples in the EM to the DXF
FLASH CONTROL Port	Provides communication between the flash control board and the controller
TEMP CONTROL Port	Provides communication between the unit Temperature Control and the controller
EM POWER Port	Provides power to the EM
SYSTEM POWER Switch	Provides power to the DXF/EM and works as a built-in circuit breaker for safety

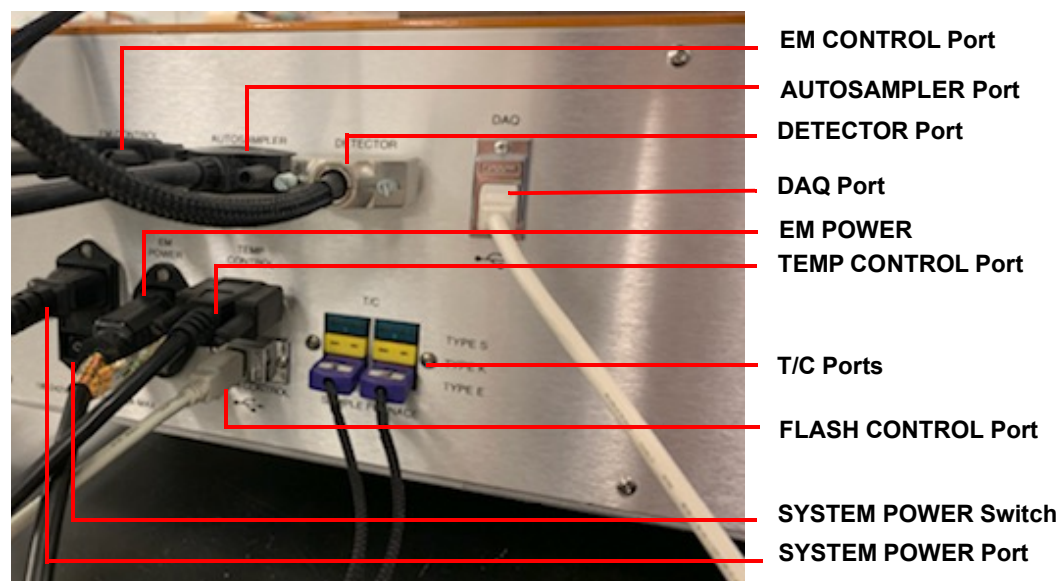


Figure 6 DXF rear panel connections.

EM Back Panel

The EM back panel has 13 ports; the table below provides a description of the function of each port. Refer to [Figure 8](#) for an illustration of rear connections.

Port	Function
DETECTOR PURGE GAS OUTLET	Provides inert gas to EM Detector Assembly.
PURGE GAS INLET	Provides inert gas to the EM Furnace Core and Chamber Backfill.
DETECTOR CONTROL	Provides power and control commands to the Detector Head Assembly.
VACUUM GAUGE	Provides power to the vacuum gauge and communication to the controller.
CRYOGENIC COOLANT INLET	Entry port for cryogenic coolant to cool furnace below ambient temperature.
CRYOGENIC COOLANT OUTLET	Exhaust port for cryogenic coolant (typically nitrogen gas in normal operation).
EM COMM	Provides communication between the computer and the EM.
AUTOSAMPLER	Provides communication between the DXF and the EM.
EM CONTROL	Provides communication between the DXF and the EM.
CRYOGENIC VALVE CONTROL	Provides power to the Cryogenic Coolant Control Solenoid Valve.
EM POWER INLET	Powers the EM.
SAMPLE TC	Provides EM Sample Temperature to the DXF.
FURNACE TC	Provides EM Furnace Temperature to the DXF.

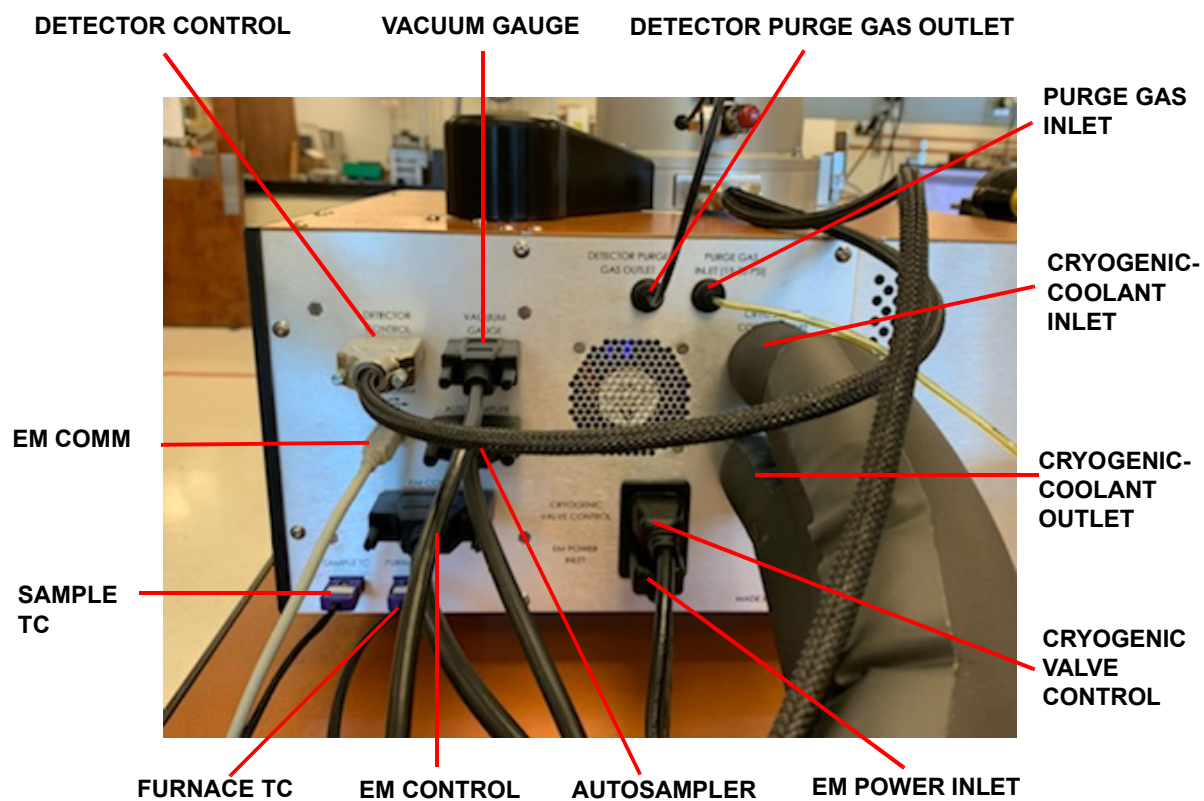


Figure 7 Environmental Module rear panel connections.

Table 5: EM Side Panel

Port	Function
PURGE FLOW CONTROL	Controls the purge gas flow into the Furnace Core (sample area).
CORE PURGE ON/OFF	Turns ON and OFF the purge gas to the Furnace Core (sample area).
CHAMBER BACKFILL ON/OFF	Turns on and off the purge gas backfill to the Furnace Chamber.
PURGE GAS OUTLET	Purge gas exit port for both the core and chamber purge; the anti-siphon exhaust bubbler attaches to this port.



Figure 8 Environmental Module side panel connections.

Connecting the Environmental Module to the Pulse Source

To connect the DXF to the EM, follow the instructions below. Refer to [Figure 6](#) and [Figure 8](#) above for connection ports, or the diagram located in the [Appendix](#).

- 1 Connect the **EM CONTROL** cable from the back of the DXF to the back of the EM.
- 2 Connect the **AUTOSAMPLER** cable from the back of the DXF to the back of the EM.
- 3 Connect the **EM POWER INLET** cable from the back of the EM to the back of the DXF.
- 4 Connect the **FURNACE** thermocouple cable into the Type E (violet) T/C port on the back of the DXF, and then connect the other end of the cable to the back of the EM.
- 5 Connect the **SAMPLE** thermocouple cable into the Type E (violet) T/C port on the back of the DXF, and then connect the other end of the cable to the back of the EM.

- 6 Connect the power cable into the back of the DXF and into a power strip and then plug into a wall power outlet.

Connecting the Detector Cable to the Detector Head Assembly

- 1 Connect the **DETECTOR** cable to the connector on the back of the Detector Assembly and to the **DETECTOR CONTROL** connector on the back of the EM and the **DETECTOR** connector on the back of the DXF.

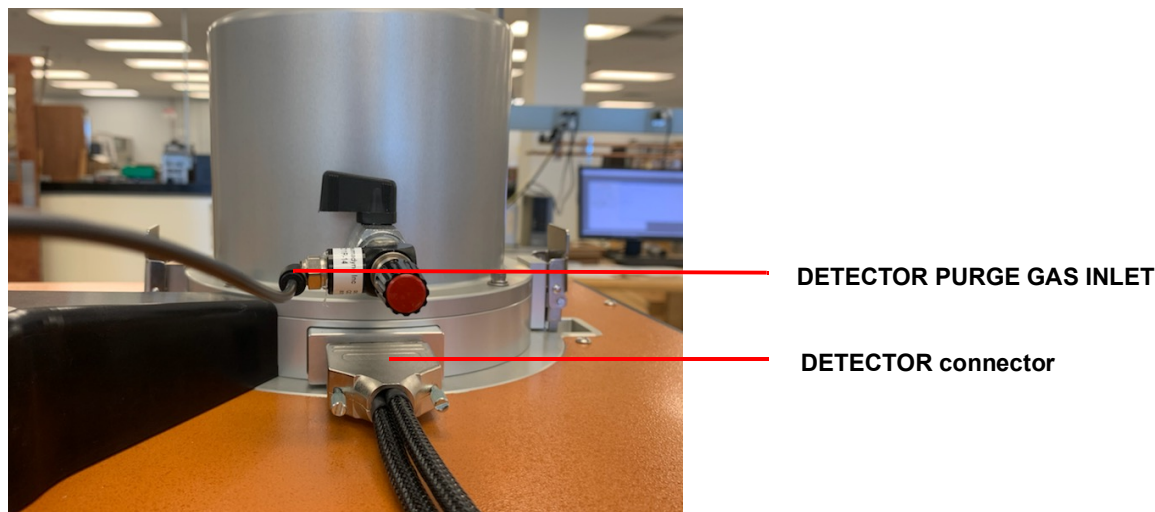


Figure 9 Rear of the Detector Assembly.

- 2 Connect the **DETECTOR PURGE GAS OUTLET** cable from the back of the EM to the back of the Detector Assembly.

Setting Up System Communication with the Controller (Computer)

- 1 Place the computer and monitor to the side of the unit and connect the keyboard and mouse.
- 2 Connect a USB cable from the **DAQ** port on the back of the DXF to the controller.
- 3 Connect a USB cable from the **FLASH CONTROL** port on the back of the DXF to the controller.
- 4 Connect an RS232 cable from the **TEMP CONTROL** port on the back of the DXF to the controller.
- 5 Connect a USB cable from the **EM COMM** port on the back of the EM to the controller.
- 6 Connect the monitor cable to the monitor port on the computer.
- 7 Connect the power cords to the computer and monitor and plug them into the same wall outlets on the same circuit that the DXF is plugged into.
- 8 Turn on the computer and monitor.

Connecting the Utilities

Connecting the Power

Plug the power cord from the **SYSTEM POWER** on the back of the DXF to an electrical outlet rated in accordance with the power input information on the back of the instrument.

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.

Setting Up the Vacuum System

The vacuum system is an accessory available for purchase at TA Instruments. If you are not using a vacuum system, close the valve with a blanking plate.

- 1 Place the vacuum pump on the floor below the left side of the unit.
- 2 Place the o-ring with centering ring between the QF-25 flange on the instrument and the QF-25 flange on the vacuum hose.
- 3 Clamp the flanges together with a QF-25 clamp.
- 4 Connect the QF-25 flange on the opposite end of the hose to the QF-25 flange on the mechanical vacuum pump.
- 5 Connect the anti-siphon exit port to the quick-connect fitting.
- 6 Connect the vacuum gauge cable from the **VACUUM GAUGE** port on the back of the EM to the vacuum.

Connecting Cryogenic Cooling and Purge Gas Lines

Cryogenic Cooling

- 1 Connect the end of the liquid nitrogen inlet hose Swagelok® fitting to the **CRYOGENIC COOLANT INLET** fitting on the back of the EM and connect the other end to the liquid port on the liquid nitrogen dewar.
- 2 Connect the plug from the liquid nitrogen inlet hose to the **CRYOGENIC VALVE CONTROL** receptacle on the back of the EM.
- 3 Connect one end of the liquid nitrogen outlet hose with Swagelok fitting to the **CRYOGENIC COOLANT OUTLET** fitting on the back of the EM and connect the other end to the Swagelok fitting on the liquid nitrogen exhaust container.

NOTE: Remember to open the valve on the liquid port on the liquid nitrogen dewar.

Purge Gas

- 4 Insert the 1/8-inch tube into the **PURGE GAS INLET** fitting on the back of the EM. Connect the other end of the hose to the inert gas supply.
- 5 Close the **PURGE GAS FLOW** needle valve and turn the **CORE PURGE** and **CHAMBER BACK-FILL** valves to the **OFF** position on the side of the EM.
- 6 Open the inert gas supply unit.
- 7 Check all gas lines and connections for leakage.

Installing the Tray

- 1 Unlock the latch on both sides of the Detector Assembly and raise the Assembly up away from the EM. Then swing the Detector Head to the left, away from the furnace.

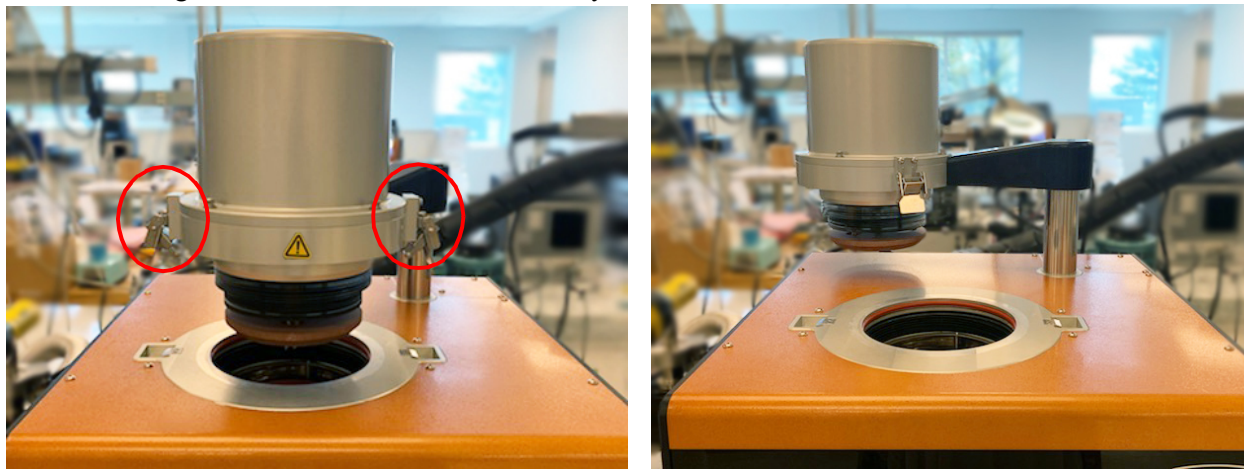


Figure 10 Move the Detector Head away from the furnace.

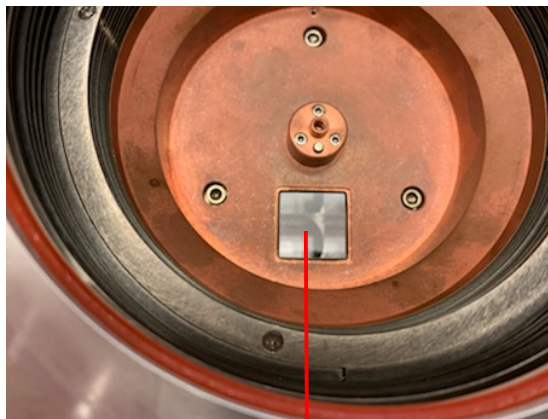
Swagelok® is a registered trademark of Swagelok Company.



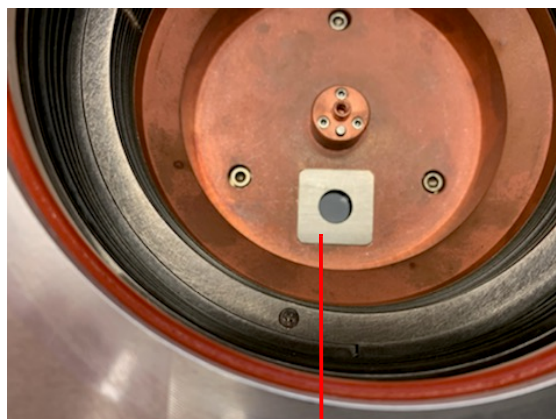
WARNING: Sample plate will be hot. Use caution when loading and unloading samples. Check the sample temperature on the front panel; 60°C and below is safe to handle.

AVERTISSEMENT: la plaque d'échantillonnage sera chaude. Soyez prudent lors du chargement et du déchargement des échantillons. Vérifiez la température de l'échantillon sur le panneau avant; 60 ° C et moins est sûr à manipuler.

- 2 Select the shade appropriate to the sample size, then insert shade over the light pipe, shiny side facing down.



Light pipe



Shade

Figure 11 Left: Light pipe. Right: Shade inserted over the light pipe.

- 3 Align the notch on the tray with the pin until the tray is firmly seated.



Figure 12 Left: Pin. Right: Notch on tray.

- 4 Secure the tray by installing the washer and screw using a 5/64 hex wrench.

Calibrating the Tray Position

This calibration should be performed after the tray is installed or as needed.

- 1 Open the software by double-clicking the FlashLine icon on the computer desktop.
- 2 From the **Setup** menu, click **Select Diagnostic Mode**.
- 3 From the **Diagnostics** menu, click **Autosampler**.

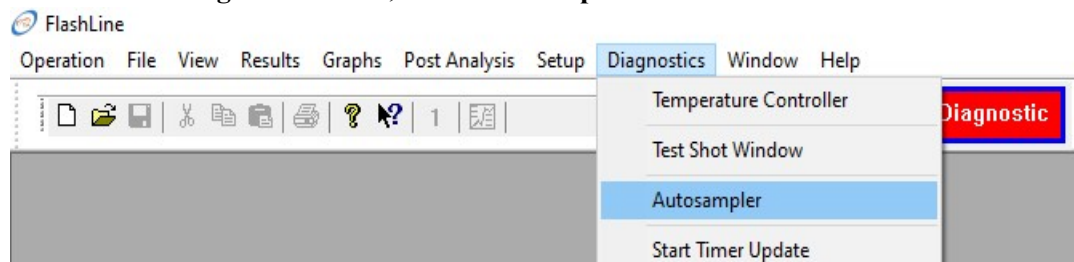


Figure 13 Diagnostics menu.

- 4 Select the tray and then click **OK** to confirm.

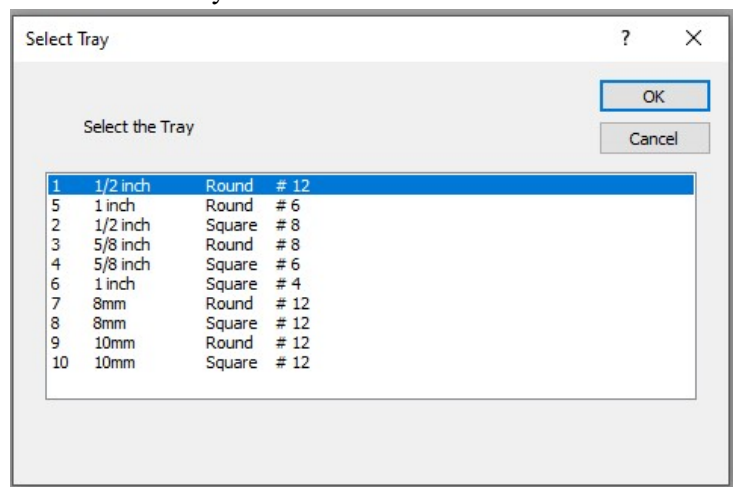


Figure 14 Select the tray.

5 Click **Start Tray Calibration** and then click **Yes**. The tray moves to the Home position.

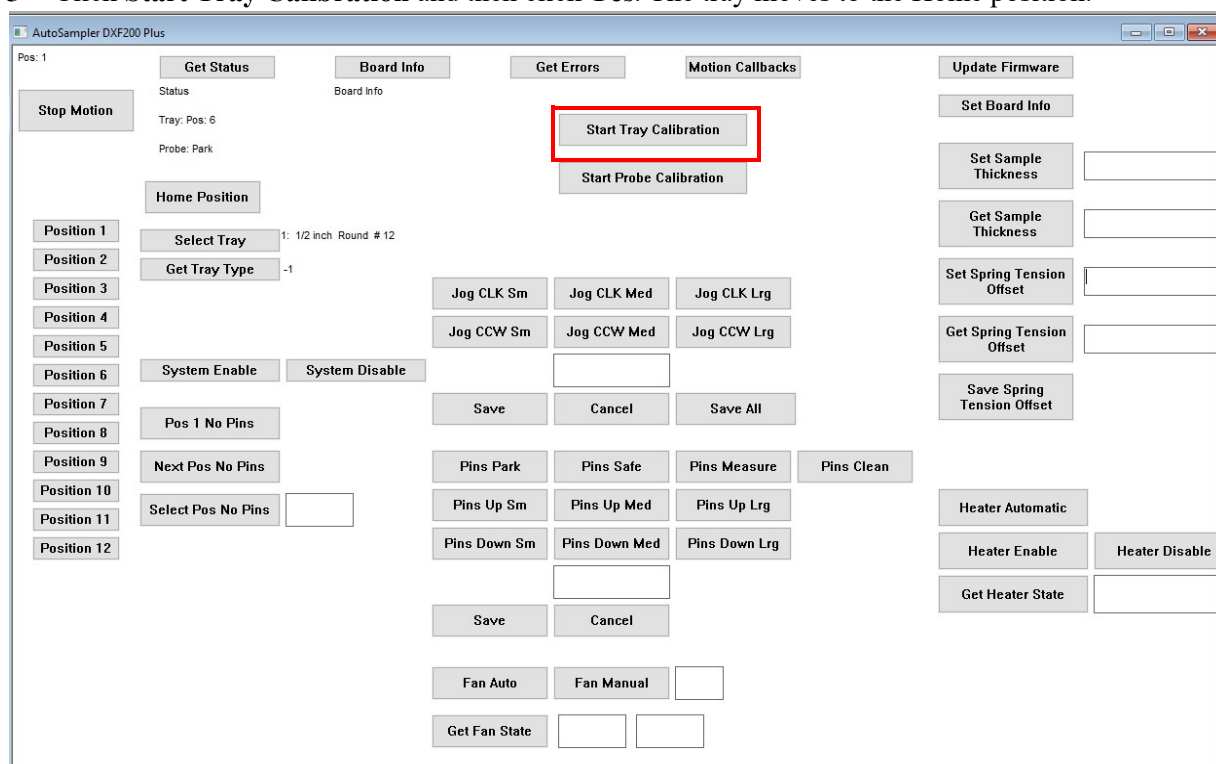


Figure 15 Start Tray Calibration.

6 Move position 1 of the tray (marked by a notch) so that the position 1 round opening on the tray aligns with the center of the shade. Use the **Jog CLK Lrg** (to move the tray in large increments), **Jog CLK Med** (to move the tray in medium increments), or **Jog CLK Sm** (to move the tray in small increments) buttons to move the tray.

7 Click **Save** to save the calibration to that particular tray or **Save All** to save the calibration to all trays.

NOTE: To ensure accurate calibration, the tray should be calibrated in the clockwise (CLK) motion. Use counterclockwise (CCW) to correct an overshoot target.

Verifying the Tray Position Calibration

Click **Pos 1 No Pins** from the **Autosampler** window to automatically move the tray first to the Home position and then back to position 1.

Installing the Pins

- 1 Remove the 4 screws on the Detector cover with a 9/64 hex wrench.

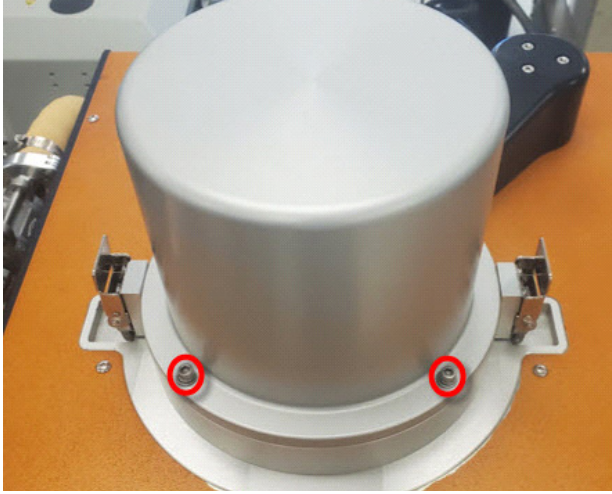


Figure 16 Remove screws.

- 2 Using a 7/64 hex wrench, remove the metal retaining plate screw and set the plate and screw aside.

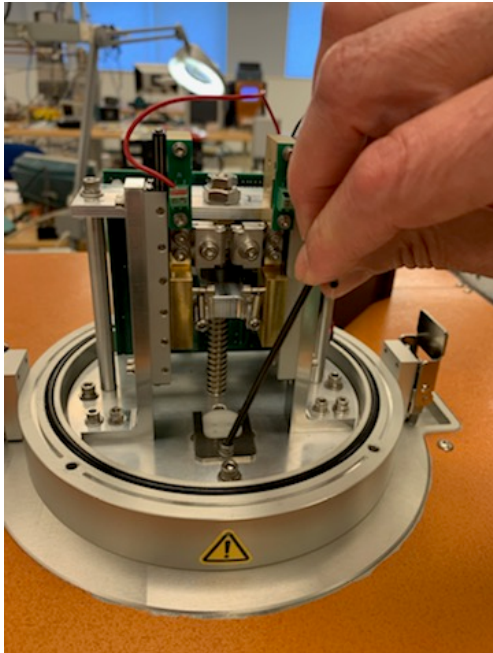


Figure 17 Remove the retaining plate.

- 3 Using tweezers, remove the heat shield (two pieces).

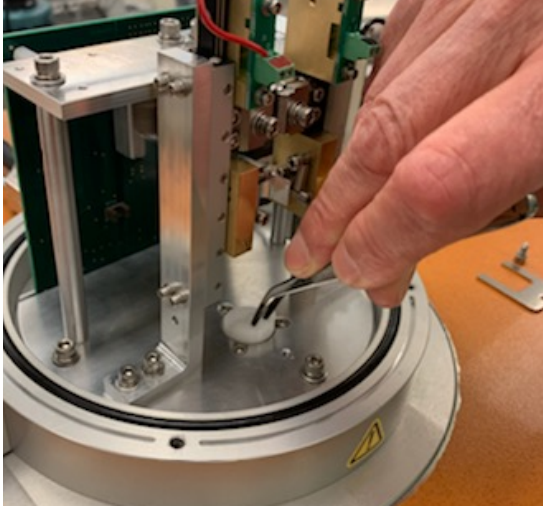


Figure 18

- 4 Using a 7/64 hex wrench, loosen the clamps that hold the Detector pins in place.

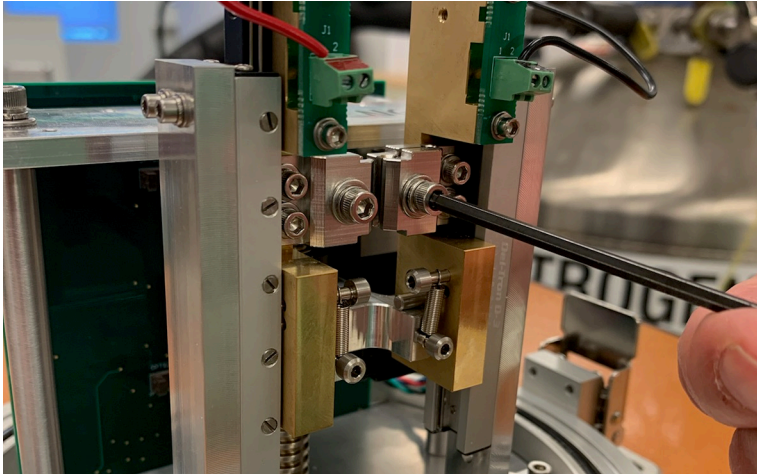


Figure 19 Loosen clamps.

- 5 Using a small flathead screwdriver, loosen the innermost screws that hold the lead for the detector pin.

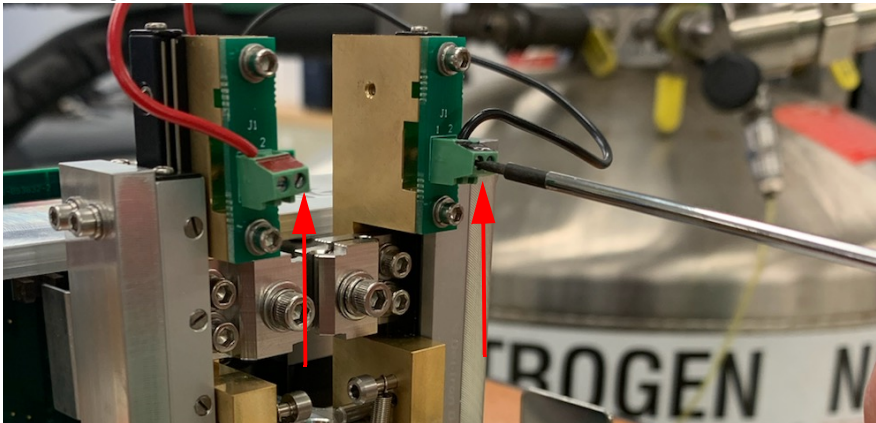


Figure 20 Loosen lead screws.

- 6 Carefully remove the pins from the box.



- 7 Insert the pins, making sure to match the leads on the pin with the leads on the terminal blocks. Orient the detector pins as shown in the image below (left).

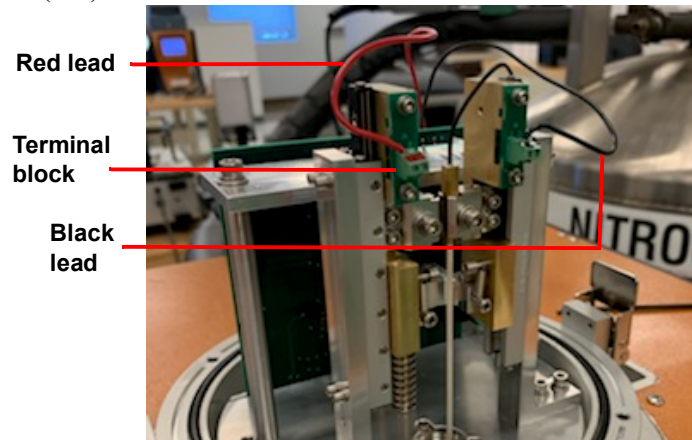
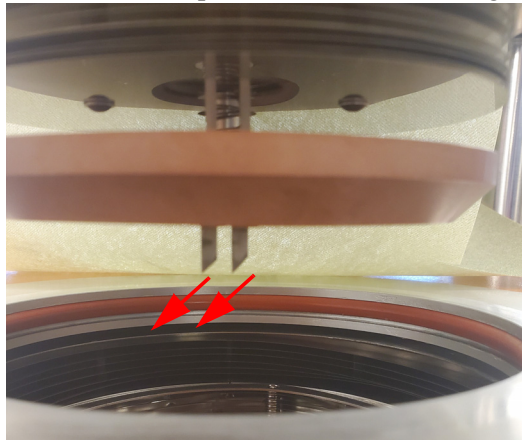


Figure 21 Left: Pin orientation. Right: Pin installed.

NOTE: The above orientation is recommended for best results with 1/2-inch samples; however, other orientations can be used.

- 8 Tighten the clamps that were loosened in [step 4](#).

- 9 Using a small flathead screwdriver, lock the pin leads into place.

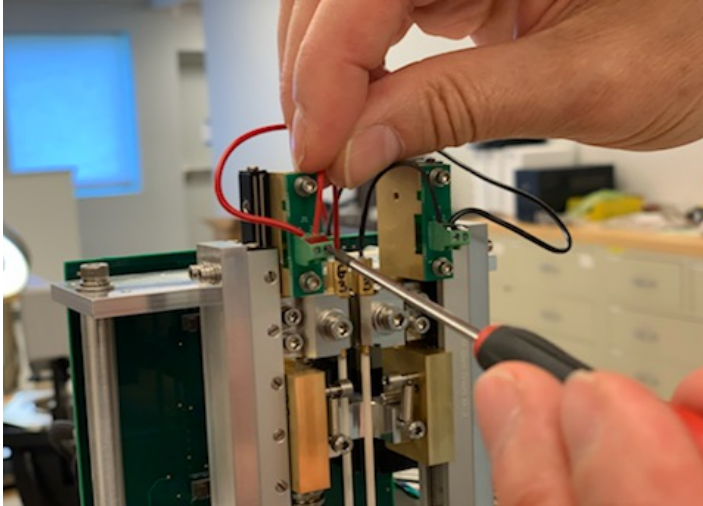


Figure 22 Lock the pin leads.

- 10 Verify pin orientation (see [Figure 21](#)).

- 11 Carefully insert the two pieces of the white heat shield, placing the half with the holes to the front.

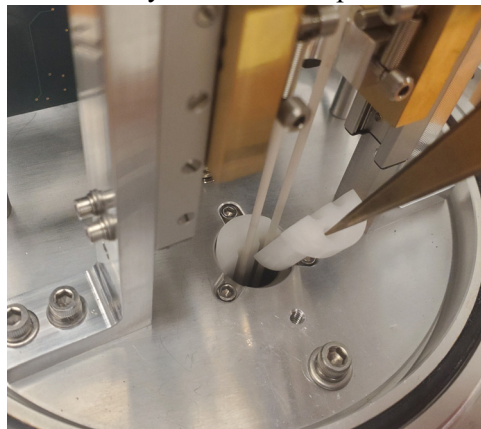


Figure 23 Install the heat shield.

- 12** Place the retaining plate around the heat shield and tighten the screw using a 7/64 hex wrench. Make sure that the pins are not touching the heat shield.

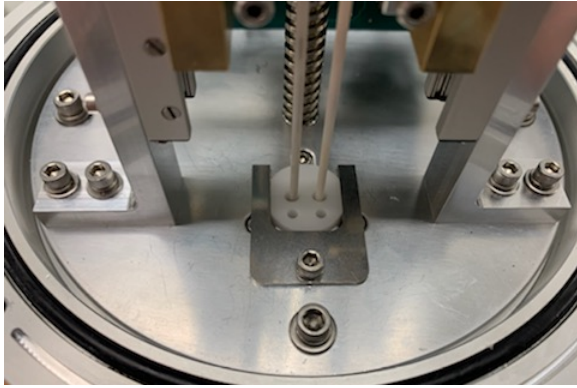


Figure 24 Install the retaining plate.

- 13** Place the Detector Head cover back on and secure the four screws with a 9/64 hex wrench.

Calibrating Pin Height

This calibration should be performed after tray calibration and installing the pins and before running tests or loading samples.

- 1 Open the software by double-clicking the FlashLine icon on the computer desktop.
- 2 From the **Setup** menu, click **Set Diagnostic Mode**.
- 3 From the **Diagnostics** menu, click **Autosampler**.

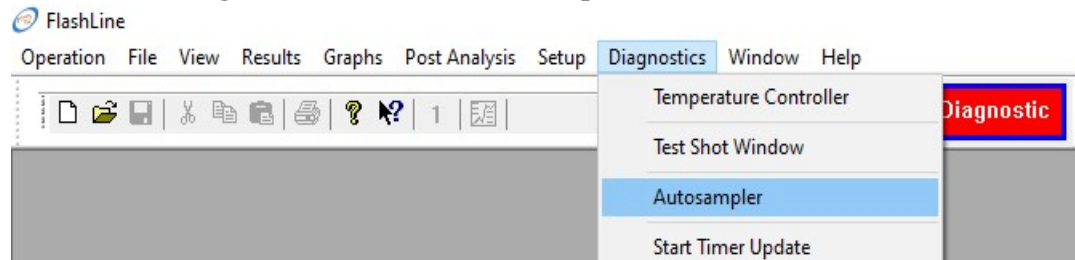


Figure 25 Diagnostics menu.

- 4 Open the Detector Head cover and verify that the tray is in position 1. Install the 10 mm spacer into position 1 of the tray.
- 5 Remove the Detector Head to gain access to the Detector pins. See [step 1](#) of “Installing the Pins.”

6 From the Autosampler window, click **Start Probe Calibration**, and then click **Yes** to start this calibration.

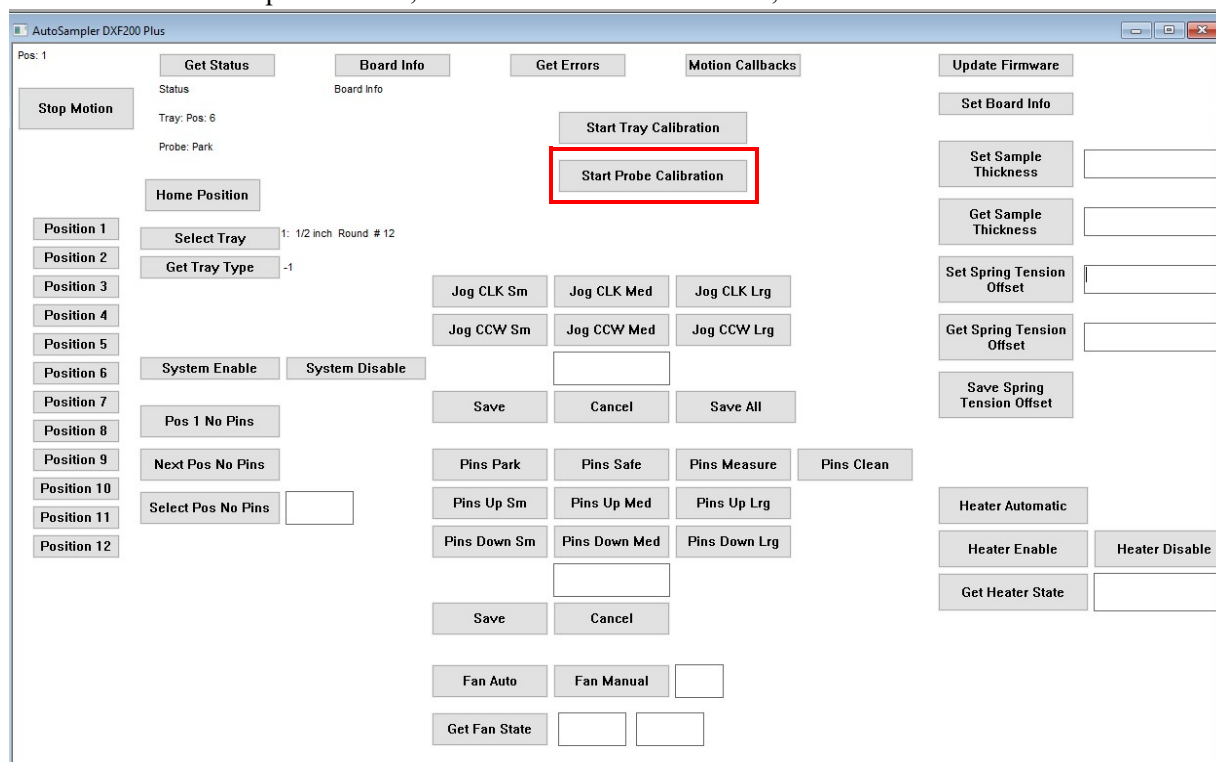


Figure 26 Start Probe Calibration.

- 7 Adjust the pin height using the **Pins Up** and **Pins Down** buttons. “Sm,” “Med,” and “Lrg” indicate the size of the incremental movements of the pins ([Figure 27](#)). The pins should be adjusted until a piece of paper fits in the gap between the pin on the brass block and lift carriage ([Figure 28](#)). This gap should exist on both the left and right detector pins.

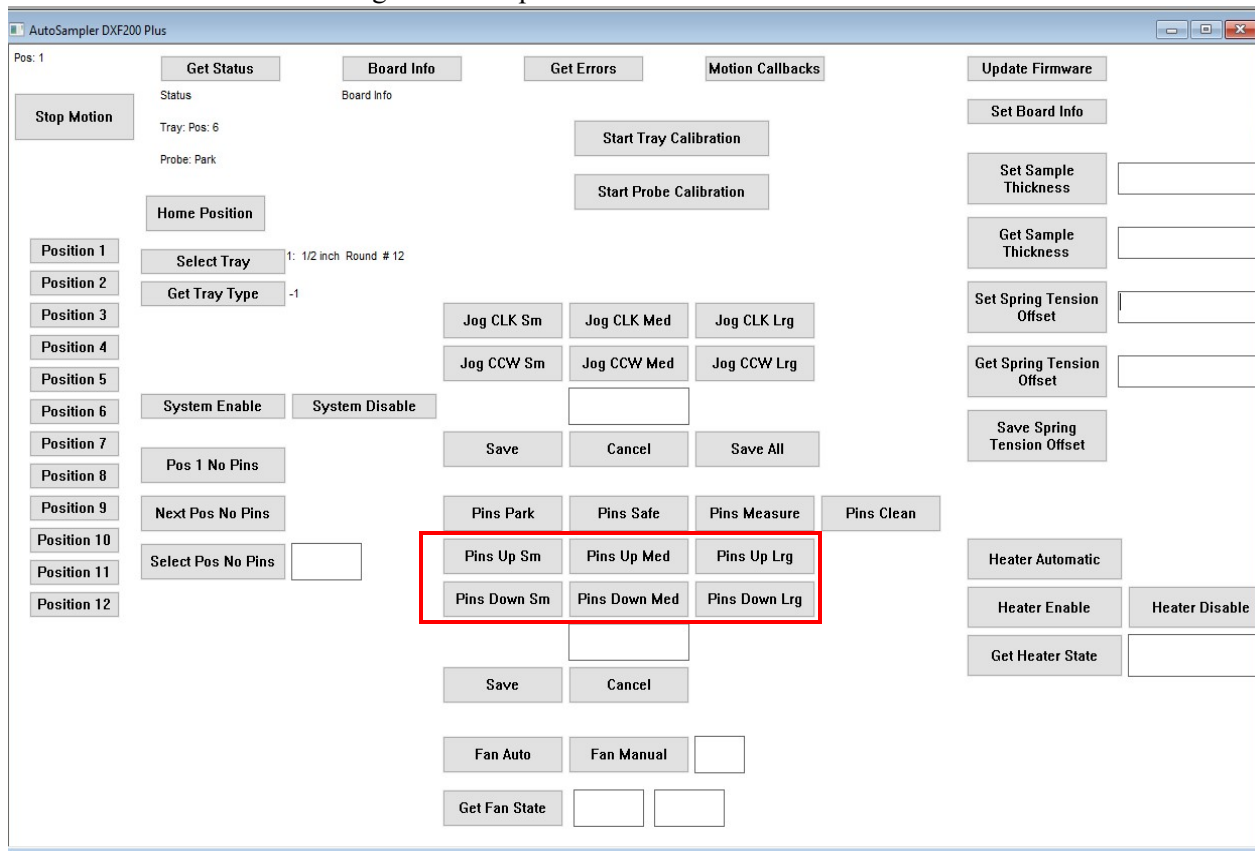


Figure 27 Pins Up and Pins Down buttons.

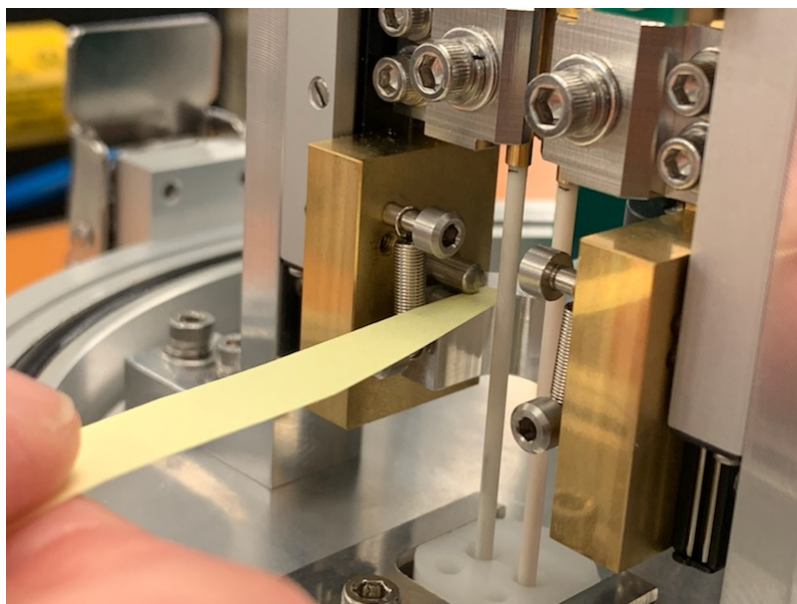


Figure 28 Gap between the pin on the brass block and lift carriage.

- 8 Click the **Save** button located below the Pins Up and Down buttons to save the calibration.
- 9 Click **Pins Park**.

Verifying Pin Height

- 10 From the **Diagnostics** menu, click **Autosampler**. ([Figure 25](#))
- 11 The Autosampler window displays ([Figure 27](#)). Enter **10 mm** in the **Set Sample Thickness** field.
- 12 Enter **0** in the **Set Spring Tension Offset** field.
- 13 Click **Pins Measure**.
- 14 Check the gap between the pins on the brass blocks and lift carriages ([Figure 28](#)).
- 15 Click **Pins Park**.
- 16 Reinstall the Detector Head cover.
- 17 Open the Detector Head and remove the 10 mm spacer from position 1 of the tray.

Chapter 3:

Operating the DXF

Using the DXF

All of your DXF 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 software; therefore, they will not all be covered in detail here.

- Preparing the sample
- Selecting the appropriate sample tray and holder
- Loading the sample and closing the Detector Head
- Evacuating the system and setting the purge gas flow rate
- Creating or choosing the test procedure and entering sample and instrument information through the instrument control software
- Starting the experiment

To obtain accurate results, follow these procedures carefully.

Before You Begin

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

- Made all necessary cable connections from the Environmental Module to the DXF
- Connected the instrument with the controller
- Connected all coolant and gas lines
- Connected the vacuum system (if applicable)
- Powered up the unit
- Become familiar with controller operations
- Viewed the software's Demo mode by going to **Setup > Select Demo Mode**

Startup and Shutdown Procedures

Starting the DXF System

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

To power on the system:

- 1 Check all connections between the DXF, the Environmental Module, the Detector Assembly, and the controller. Make sure each component is plugged into the correct connection port.
- 2 Set the DXF **SYSTEM POWER** switch to the ON (**I**) position.
- 3 The temperature controllers on the front panel indicate that power is turned on. After the proper power up sequence, the instrument user interface appears and the keypad buttons are lit; this indicates that the instrument is ready for use.

Shutting Down the DXF 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 DXF and the controller 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 with low purge to keep the system dry.

To power down your system, set the DXF **SYSTEM POWER** switch to the OFF (**0**) position.

Running a Discovery DXF Experiment

Preparing the Sample

The method is applicable for testing homogeneous, solid, and opaque materials. However, in real life, materials greatly deviating from the above can be tested with special sample preparations, precautions, and analytical corrections. It is incumbent on the user to ascertain if a new material can be tested in an “as received form” or if special preparation is needed.

Sample Dimensions

It is extremely important to machine samples to exact dimensions. A sample must clear and fit the holder (or the insert) opening. If the sample diameter is equal or slightly bigger than the opening, then there will be an additional heat transfer from the tightly fitted sample to the holder during the measurement, which may negatively affect the preciseness of the thermal diffusivity measurement. If the sample diameter is much smaller than the opening, then the light beam can bypass the sample and interact directly with the IR detector, causing temperature signal distortion, or even the detector destruction.

The thermal diffusivity calculation depends on square of the specimen thickness. Uncertainty in the specimen thickness value due to rough, or not completely flat surfaces will result in uncertainty (error) in the thermal diffusivity determination. It is therefore very important to machine a sample with the tolerance as high as possible (recommended tolerance ± 1 mil). The tolerance on the lateral surfaces can be lower (± 5 mil).

Samples that are too thick are difficult to test due to weak temperature response signal and slow and sluggish response to the flash affected by heat losses from the sample. Samples that are too thin may not represent bulk material, and potentially increase measurement error due to severe finite pulse time effect. The thickness should be designed the way the sample experimental halftime should be within the range from 40 ms to 2 s. (See Theory of the Flash Method in online help for a more detailed explanation of the relationship between the sample thickness and the thermal diffusivity of the material.) More than one iteration of trial and error may be needed to find the optimal sample thickness for a particular material. Examples of thicknesses for different materials:

- Stainless steel = 1.6 mm
- Pyroceram = 2 mm
- Graphite, alumina, and molybdenum = 3.2 mm
- Copper and aluminum = 5 mm

NOTE: Testing samples of irregular shape or uneven thickness is not recommended, due to possible errors in the thermal diffusivity and the specific heat determination and/or possible damage to the instrument.

Transparent or Translucent Materials

The method requires that all of the energy from the flash be absorbed on one face of the sample. For materials that are transparent or translucent, the energy from the light pulse will travel to different depths in the sample (depending on opacity) or may completely pass through in a completely transparent sample. To prevent this, a very thin and opaque layer of high thermal diffusivity material must be deposited on the faces of the samples. For best results, one should select a coating that has a high reflectivity (low emissivity), such as gold or platinum. Conversely, this type of coating is not well suited to absorb the energy pulse, and therefore it is customary to put a second, highly absorbing graphite coat on top of it.

It is critical to keep both coatings at minimal thickness. This reduces the contribution of the layers to the total transmission time as compared to the contribution of the sample.

Best results are obtained with vacuum sputtering of a gold, silver, nickel, or platinum layer with $< 1 \mu\text{m}$ thickness. This coating is then over-coated using an aerosol spray coating of graphite.

Please note that:

- In all cases the coating material must be selected to safely withstand any temperature within the test parameters, and the graphite coating must be applied in a thin layer.
- Avoid scratching the coated surfaces after curing; even tiny amounts of porosity will severely affect the data. Prevent any dust from settling on the coated surfaces.
- Some materials that generally require coating for opacity are: glass, quartz, alumina, zirconia, silicon carbide, silicon nitride, calcium fluoride, zinc selenide, etc.

High Reflectance Samples

In instances where the material has a highly reflective surface, it may be difficult to absorb sufficient energy from the flash to produce a good signal on the opposite face. A thin coating of graphite spray usually remedies this situation.

For specific heat capacity testing it is imperative that both the unknown sample and the reference have identical emissivities over the spectral range of the High-Speed Xenon Source. To ensure this, always coat both the sample and the reference with graphite coating.

Applying Graphite Spray

Electrically conductive samples

The following is the procedure for coating electrically conductive samples with graphite spray:

- 1** Place the samples onto a plain sheet of paper. Locate them in one line close to each other.
- 2** Using a heat gun, gently warm the samples.
- 3** Hold the aerosol can of graphite spray approximately 8–10 inches from the samples. Using a single, fluent motion spray the exposed surfaces from left to right.
- 4** Allow one minute for the coating to dry on the samples.
- 5** Rotate each sample 90°.
- 6** Following the same path as in step 3, apply a second coating across first pass.
- 7** Allow a minimum of 3 minutes for the samples to dry.

NOTE: Polish the non-graphite coated side before each test.

- 8** After drying, store the samples on a soft surface (ex: tissue paper).

Non-conductive samples

- 1** Coat one side of the sample with silver paint; make sure to only coat the flat surface, not the sides. If the sample is semi-transparent, silver paint can be applied to both sides.
- 2** Allow the paint to dry.
- 3** Place the samples onto a plain sheet of paper. Locate them in one line close to each other.
- 4** Coat the bottom with thermal graphite:
 - a** Using a heat gun, gently warm the samples.
 - b** Hold the aerosol can of graphite spray approximately 8–10 inches from the samples. Using a single, fluent motion spray the exposed surfaces from left to right.
 - c** Allow one minute for the coating to dry on the samples.
 - d** Apply a second coating across the first pass.
 - e** Allow a minimum of 3 minutes for the samples to dry.
 - f** After drying, store the samples on a soft surface (ex: tissue paper)

Loading and Unloading the Sample

- 1 Unlock the latch on both sides of the Detector Assembly and raise the Assembly up away from the EM. Then swing the Detector Head to the left, away from the furnace.

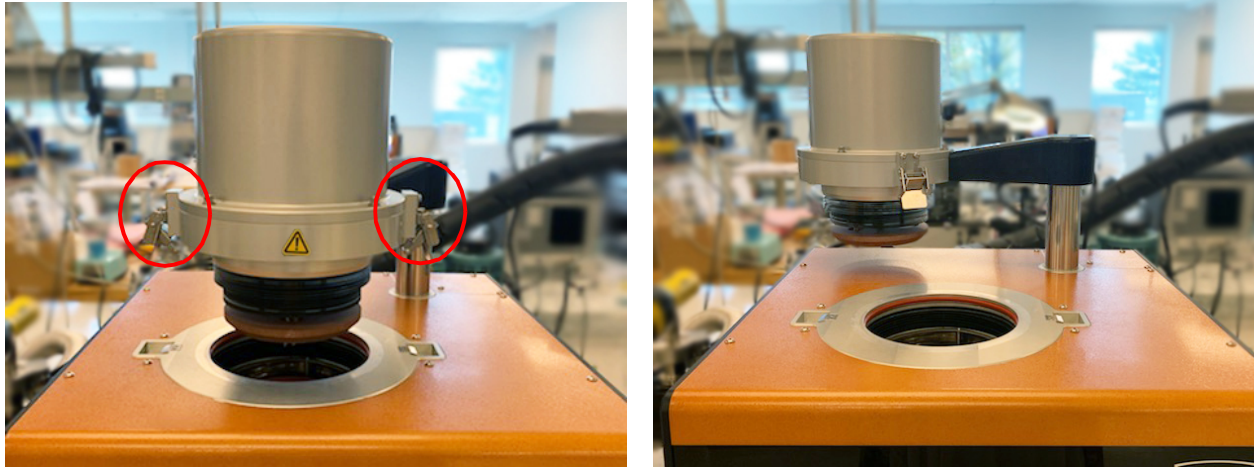


Figure 29 Raise the Detector Head.



WARNING: Sample plate will be hot. Use caution when loading and unloading samples. Check the sample temperature on the front panel; 60°C and below is safe to handle.

AVERTISSEMENT: la plaque d'échantillonnage sera chaude. Soyez prudent lors du chargement et du déchargement des échantillons. Vérifiez la température de l'échantillon sur le panneau avant; 60 ° C et moins est sûr à manipuler.

- 2 Once open, load the samples into the sample plate, graphite-coated side facing down toward the energy source, polished side facing up toward the measurement pins. Positions are numbered.



Figure 30 Loading the sample into the sample plate.

- 3 After all samples have been loaded, carefully close the Detector Assembly back into the EM (position your right hand as shown in the image below to ensure the measurement head is closed safely) and secure the latches. Note that the Detector Head is difficult to lower when pressing down only on the Detector Head. Instead, press down the Detector Head while simultaneously pressing down on the Detector arm, as shown below.

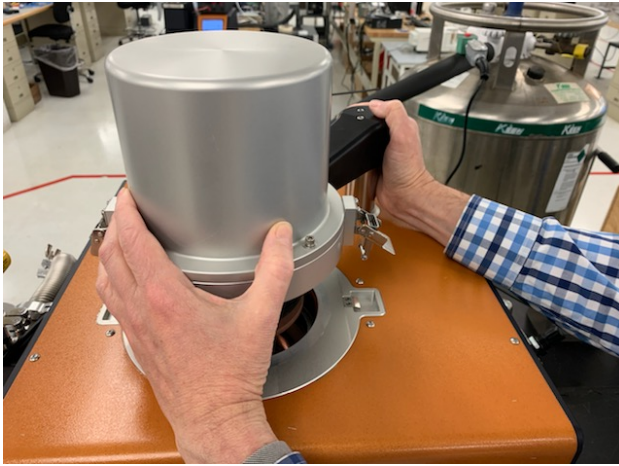


Figure 31 Lowering the Detector Head.

The system is now ready for evacuation and purging.

Preparing the Instrument

Evacuating the System

Some systems are equipped with vacuum capabilities; all are equipped with purge capabilities. Purge gas exits through the anti-siphon exit port assembly.

CAUTION: Never place any type of valve between the EM exhaust and the anti-siphon exit port assembly.

MISE EN GARDE: Ne placez jamais un type de vanne quelconque entre l'échappement EM et l'ensemble de l'orifice de sortie anti-siphon.

The EM is equipped with a vacuum meter assembly for determination of the vacuum level.

Vacuum and Purge Operation

- 1 Verify all vacuum connections.
- 2 Check the oil level in the anti-siphon exit port. Add vacuum pump oil as needed to reach the level marked on the outside of the reservoir.
- 3 Close all purge valves.



Core Purge Valve

Figure 32

- 4 Turn on the vacuum pump.
- 5 Evacuate the system to at least 50 mTorr.

Back-fill with Purge Gas

- 1 Turn off the vacuum pump.
- 2 Follow the procedure for [Vacuum and Purge Operation](#).
- 3 Enable the core purge gas flow to the EM chamber.
- 4 Monitor the flow using the anti-siphon exit port assembly. Flow should be continuous, roughly 4–5 bubbles/second. Adjust with the Purge Flow Control valve ([Figure 8](#)) on the side of the EM.

NOTE: The Chamber Backfill valve and the Detector Purge Gas valve are used to backfill the system and release vacuum. Only the Core Purge valve should stay in the On position during a test. The Purge Flow Control valve controls the Core Purge flow rate.

Starting an Experiment

Access the instrument control software to create or choose the test procedure as well as enter sample and instrument information through the instrument control software.

- 1 To open the software, double-click the FlashLine icon on the computer desktop.
- 2 From the **Operation** menu, click **Startup Information**.

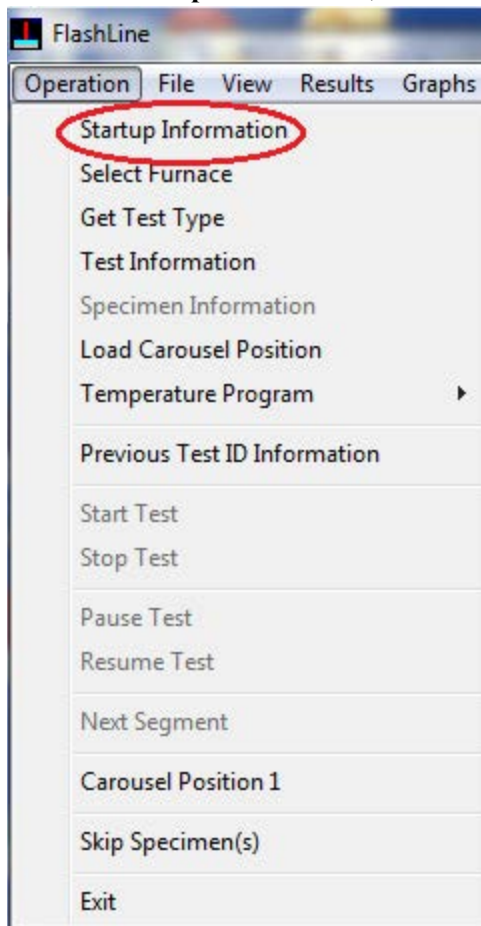


Figure 33 Operation > Startup Information.

- 3 Click **OK** to confirm your selection. The Test Information window displays.

4 Enter the test parameters and then click **Start Test**.

Test Information

Test Title:

Test ID: Sequence Number:

Operator: Date:

Use Information From Previous Test:

Pins / Autosampler Control:

Cancel: Help:

Common Sample ID: Common Sample Title: Common Thickness (cm): Common Tension %: Use Expansion: ☐

Position	Sample ID	Sample Title	Thickness (c...	Diameter (c...	Tensio...	Weight ...
1			0.0000	1.270	50	0.0000
2			0.0000	1.270	50	0.0000
3			0.0000	1.270	50	0.0000
4			0.0000	1.270	50	0.0000
5			0.0000	1.270	50	0.0000
6			0.0000	1.270	50	0.0000
7			0.0000	1.270	50	0.0000

?	Test	Temps	Ramp	Repeat	Power	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11	S12
	Segment	(°C)	°C/Min	Points	Level	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt
	1		5.00	3	500.0												
	2		5.00	3	450.0												
	3		5.00	3	450.0												
	4		5.00	3	450.0												
	5		5.00	3	450.0												
	6		5.00	3	450.0												
	7		5.00	3	450.0												

Uniform: Temperatures: Ramp Rate: Repeat Points: Power Level: Acq Rate: Acquisition Params:

Clear Program: Add Segment: Delete Segment: Insert Segment: List Programs: Save Program: Safety Temperature (C):

Start Test: ☐ Specific Heat/Conductivity ☐ Capsule(s) ☐ Show Acquisition Params

Figure 34 Test Information window.

Top section of the Test Information window:

- **Test Title:** The entered information is added to the test information file for future reference.
- **Test ID:** The name of the test saved in the Instrument Data folder. The Test ID number advances automatically. Add a short description (ex. 123_Stainless_Steel) for future reference.
- **Operator:** Enter the name or initials of the person running the test for future reference.
- **Use Information from Previous Test:** Fills most fields with information from the previously saved test.
- **Pins/Autosampler Control:** Opens a window that allows command of the tray and pins position, verify tray and pin position, and can be used to lower pins for cleaning and rotate the tray for sample loading.
- **Common Sample ID, Common Sample Title, Common Sample Thickness, Common Tension %:** Sets every sample position with the value entered in the Position 1 fields.

NOTE: When only one sample is used, it must be set in position 1. Samples can not be skipped—the second sample must be tested in position 2, third sample in position 3, etc. Only the samples with information entered in the Sample ID column will be tested. If only 4 out of 12 samples tested, the rest of the positions' Sample ID text (Sample ID 5 to 12) must be cleared.

- **Use Expansion:** Allows the inclusion of sample expansion due to temperature changes to the sample thickness.

- **Sample ID:** Enter Sample identification for each position.
- **Sample Title:** Enter a sample description text for each position.
- **Thickness (cm):** Enter the sample thickness for each position.
- **Diameter (cm):** Set automatically based on the selected tray. Verify that it matches the tested sample.
- **Tension %:** Can be set from 0% to 100%, depending on the desired contact between measurement pins and the sample.
- **Weight (g):** Enter the sample weight for each sample (not required; use for future reference)

Bottom section of the Test Information window:

- **Test Segment:** Identifies the segment number.
- **Temps (°C):** Temperature at which the test is run for each segment.
- **Ramp °C/Min:** Heating rate to the set point temperature of each segment.
- **Repeat Points:** The number of shots to each sample at each segment.
- **Power Level:** Shot energy at each segment; this can be modified for each sample (see S1–S12 Vlt)
- **S1–S12 Vlt:** The voltage for each segment for each sample position. This can be modified individually or by right-clicking on the column and selecting the desired option.
- **Uniform:** Use the **Temperatures**, **Ramp Rate**, **Repeat Points**, and **Power Level** buttons to set the same value for all sample positions.
- **Acq Rate:** Allows the setting of uniform data acquisition rate. Default is 15,000 points/sec, maximum available is 500,000 points/sec. High sample rate should only be used when necessary as it will create large data files that will take a long time to store and analyze.
- **Acquisition Params:** Opens a window that allows you to change the acquisition rate and time for each sample (starting with sample 1) and each segment. Follow the menu options to complete the process.
- **Clear Program:** Clears the test parameter fields.
- **Add Segment, Delete Segment, Insert Segment:** **Add Segment** adds a segment to the bottom of the Test Segment list. **Delete Segment** removes the currently selected Test Segment; **Insert Segment** inserts a segment above the currently selected segment.
- **List Programs:** Displays a list of previously saved tests.
- **Save Program:** Saves test parameters, which can be reused in subsequent tests.
- **Safety Temperature (C):** Set by default to the temperature limit of the instrument, this can be changed to a lower temperature if the sample will be damaged (deform, melt, etc.) at certain temperature.
- **Specific Heat/Conductivity:** Selecting this box will include Specific Heat and Conductivity in the calculated results files. Selection required when setting up parameters.
- **Cp/Cond Setup:** This setting is for specifying Specific Heat (Cp) and Conductivity parameters. Selecting the box opens a window to assign density and Specific Heat to sample 1 and density to the rest of the samples.
- **Capsule(s):** Selecting this box opens a window for setting special sample holders (capsules) for each sample. Selecting the holder results in setting applicable parameters for the capsule type.
- **Show Acquisition Parameters:** Selection this box displays acquisition parameters for confirmation purposes after the Start button is selected.

- **Start Test:** Once all of the above fields are filled and the system is ready for test, click the **Start Test** button to start the test process. Follow the window instructions for selecting relevant options and for enabling the system and furnace.

Stopping an Experiment

If for some reason you need to discontinue the experiment, you can stop it at any point by clicking the **Stop Test** icon from the FlashLine application screen. Then confirm that you intend to stop the experiment.

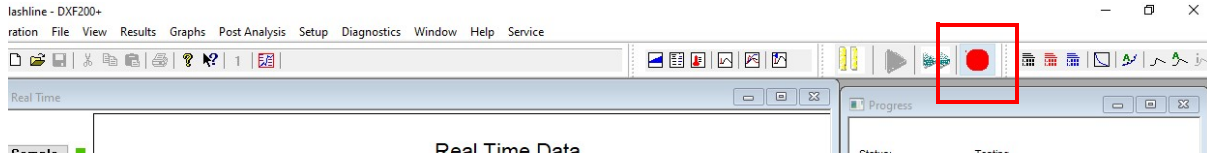


Figure 35 Stop Test.

Chapter 4:

Maintaining the DXF/EM

The primary maintenance procedures described in this chapter are the customer's responsibility. Any further maintenance should be performed by a representative of TA Instruments or other qualified service personnel. Consult the online documentation installed with the instrument control software for further information.

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

DANGER: À cause de la présence de tensions élevées 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.

General Cleaning Practices

- Use protective eye glasses
- Use finger coats or gloves
- Always hold optical components (mirrors, lenses, windows, etc.) from the edges; never touch the center after cleaning
- If strongly burned films remain on the components (like entry laser lens 120377) after the cleaning process, use weak acid (like vinegar) to try to dissolve the residual layers
- Always use distilled water and acetone at the end of cleaning process
- Use a thin layer of vacuum grease to seal all O-ring parts of the EM module whenever it is necessary to reassemble the block containing O-rings

Recommended Cleaning Supplies

- Compressed air duster can or dry nitrogen from the bottle which supply the purge gas for instrument- with flexible plastic, movable/bend nozzle (never use compress air from industrial compressor lines; it contains a large amount of water and residual oil)
- Acetone and propanol-laboratory grade-with droplets pump attach to bottles
- Dust- and lint-free optical tissue or fabric
- Cotton swabs, cleaning brushes, mini vacuum cleaner
- Scouring pads (such as Scotch-Brite™), detergent, acetic acid (vinegar), distilled water

Cleaning Maintenance Schedule

How often cleaning should be performed depends on the following factors: how often the instrument is used, the maximum temperature of the tests, if it is being used in vacuum or purge gas, and the nature of the samples. Tests that measure only thermal diffusivity are more tolerant with contamination of the optics than tests that measure diffusivity and specific heat. For only diffusivity, multiply with 3 the number of tests below.

The cleaning schedule below is recommended for the best results of the instrument:

- After each test run in vacuum at max. temperature 200°C
- After 5 tests run in vacuum below 200°C
- After 10 tests run at max. temperature of 200°C in an inert purge gas atmosphere
- After 20 tests run at max. temperature of 200°C in an inert purge gas atmosphere
- In the case of testing volatile or reactive materials, cleaning is recommended after each test run

The maximum temperature of the DXF 200+ is 200°C. The DXF 200+ uses contact pins to detect temperature changes. The DXF 200+ pins must be carefully cleaned before each test.

Periodically, a check test should be run to verify the cleanliness of the optical path of the instrument. Use a thermographite reference sample 0.300 cm thick, room temperature test, 500 volts charging, and 3 shots. The instrument is considered clean if the signal (max. temperature of thermo diagram) is larger than 200 millivolts.

If, after cleaning the instrument and running a check test, the signal of thermo diagram is still smaller than 200 millivolts, contact TA service.

Maintenance Procedures

Cleaning the Light Pipe Assembly

NOTE: The sample is directly above the Xenon pulse distribution light pipe. It is therefore important to protect the light pipe from dirt and possible damage. Periodic cleaning of the top surface of the light pipe is recommended. If dirt or other substances build up on it, it will adversely affect the signal for the temperature rise of the top surface of the sample, and yield erroneous results.

- 1 Using two people, lift the EM module from the top of DXF module. The EM can simply be moved a few inches back, remaining on top of the DXF.

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.

- 2 Again, using two people, from the front of the module, lift and incline the EM module at a 45° angle.
- 3 Push the assembly while twisting it clockwise, then pull the assembly to remove it from the bottom of the EM.



Figure 36 Remove the light pipe assembly.

- 4 Inspect the assembly for debris, dust, and smoke. Using a clean, oil-free optical-grade air source, blow out all the dust/debris that is on the surface of the light pipe. Clean all outer surfaces of the light pipe using a lint-free optical-grade cloth and high-purity acetone. A small nozzle vacuum may also be used to clean dust from the surface of the light pipe.

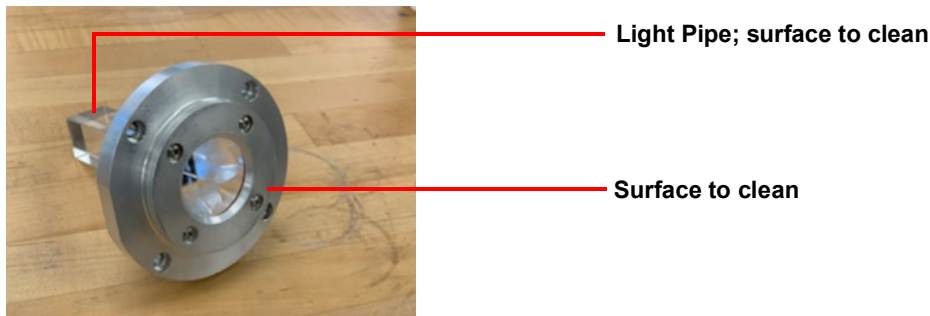


Figure 37 Light Pipe Assembly.

NOTE: Take care not to scratch the surface of the light pipe.

- 5 Reassemble the parts back into EM module.

Clean the Xenon Lamp Assembly Optics

- 6 Using the compress air duster, clean the exit window to remove any possible dust that may be contaminating the window.



Figure 38 Left: Location of the exit window. Right: Exit window.

- 7 Realign the EM module on top of DXF module in original working position.

Cleaning the Detector Pins (DXF 200+ only)

The Detector pins can be worn down over time after repeated contact. Debris from the sample can also accumulate on the tips. Checking and cleaning the pins before each test is important because they must make good contact with the surface of the sample to obtain accurate results. If the pins are not making good contact or if the tips are not the correct shape (see [step 5](#) below), you will see excess noise on the signal.

- 1 Unlock the locking clips on either side of the Detector Head.

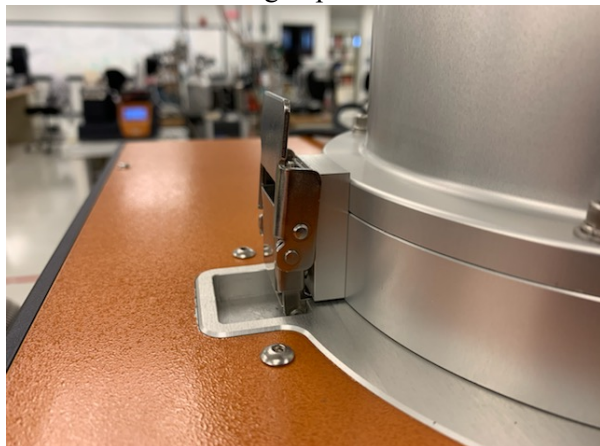


Figure 39 Locking clip in the locked position.

2 Lift the Detector Assembly up.

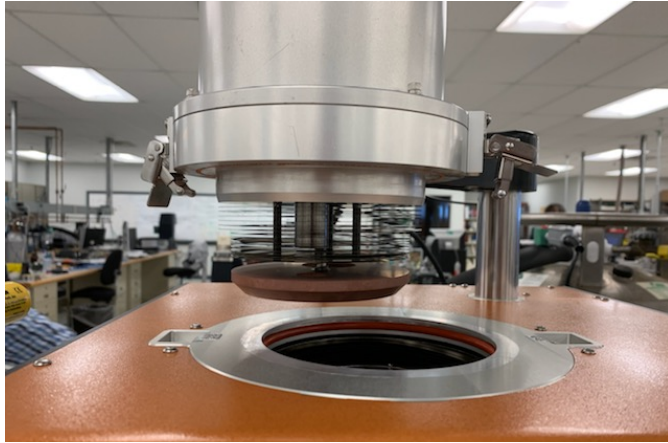


Figure 40 Detector raised.

3 From the FlashLine software Test Information window, click **Pins/Autosampler Control**.

Test Information

Test Title:

Test ID: Sequence Number:

Operator: Date:

Use Information From Previous Test ☐

Pins / Autosampler Control ☐

Cancel Help

Common Sample ID: Common Sample Title: Common Thickness (cm): Common Tension %: Use Expansion: ☐

Position	Sample ID	Sample Title	Thickness (c...	Diameter (c...	Tensio...	Weight ...
1			0.0000	1.270	50	0.0000
2			0.0000	1.270	50	0.0000
3			0.0000	1.270	50	0.0000
4			0.0000	1.270	50	0.0000
5			0.0000	1.270	50	0.0000
6			0.0000	1.270	50	0.0000
7			0.0000	1.270	50	0.0000

?	Test	Temps	Ramp	Repeat	Power	S1	S2	S3	S4	S5	S6	S7	S8	S9	S10	S11	S12
	Segment	(°C)	°C/Min	Points	Level	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt	Vlt
	1		5.00	3	500.0												
	2		5.00	3	450.0												
	3		5.00	3	450.0												
	4		5.00	3	450.0												
	5		5.00	3	450.0												
	6		5.00	3	450.0												
	7		5.00	3	450.0												

Uniform

Temperatures ☐ Ramp Rate ☐ Repeat Points ☐ Power Level ☐ Acq Rate ☐ Acquisition Params ☐

Clear Program Add Segment Delete Segment Insert Segment List Programs Save Program Safety Temperature (C)

Start Test ☐ Specific Heat/Conductivity ☐ Cp/Cond Setup ☐ Capsule(s) ☐ Show Acquisition Params

Figure 41 Access pins and Autosampler control functions.

- 4 The Pins/Autosampler Control window displays. Click **Pins Clean** to lower the pins.

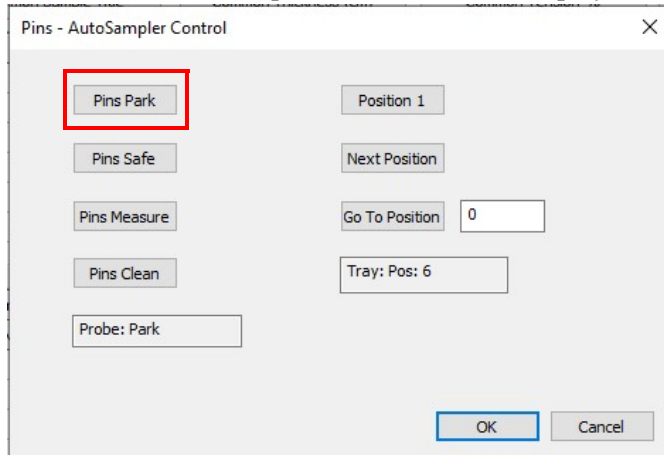


Figure 42 Pins Park.

- 5 Check the pin shape. The slightly pointy shape of the pin can dent or even penetrate the sample. The tip should be slightly flat.



Figure 43 Pin shape.

- 6 Use very fine sandpaper (30 mic grade) to clean the tip of each pin. Slowly and carefully smooth out any rough edges or points. Use a dry, soft cleaning paper to remove any remnants from the tip of the pin.
- 7 Click **Pins Park**.

- 8 Put the Detector back in place, lowering it back into the sample area. Note that the Detector Head is difficult to lower when pressing down only on the Detector Head. Instead, press down the Detector Head while simultaneously pressing down on the Detector arm, as shown below.

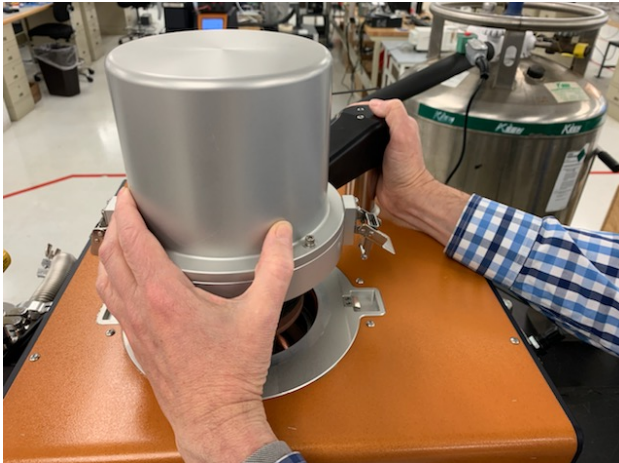


Figure 44 Lowering the Detector Head.

Lock the Detector Head into place. Make sure the clips do not get stuck in the clip groove as shown in the left image below.



Figure 45 Left: Incorrect clip position. Middle: Correct clip position. Right: Clip in the locked position.

Conditioning the Furnace

Vacuum performance dwindles during off time due to moisture absorption into the insulation and contamination of the furnace. To prevent this, the furnace should be kept closed and under positive pressure of inert gas atmosphere. Regardless, after a period of time, an unused furnace may require conditioning in order to restore vacuum performance.

- 1 Vacuum the furnace down to below 300 mTorr.
- 2 Purge inert gas (stop the vacuum pump and open the valves for inert gas to pass through the furnace) for more than 1 minute.
- 3 Restart the vacuum to 250 mTorr and purge again with inert gas. Repeat the process a couple of times, gradually going down with vacuum values to 50 mTorr. If the vacuum valves do not reach 50 mTorr, move to step 4 or leave the system under vacuum over night.
- 4 Set the furnace temperature to 200°C (see below for instructions) for two hours under vacuum.

Setting the Furnace Temperature to 200°C

- 1 From the **Setup** menu, select **Select Diagnostic Mode**.

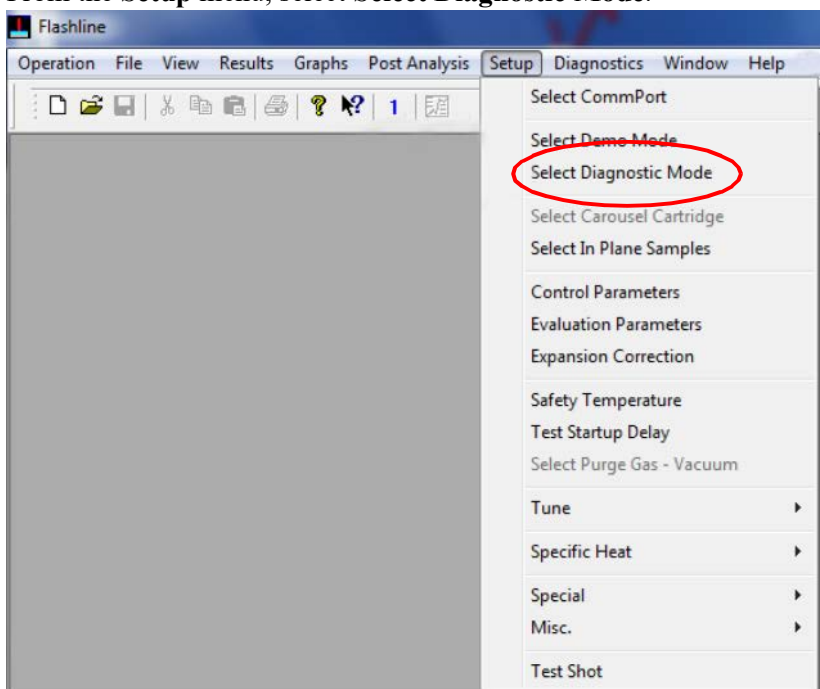


Figure 46 Setup > Select Diagnostic Mode.

- 2 From the **Diagnostics** menu, select **Temperature Controller**. A Temperature Controller window displays.

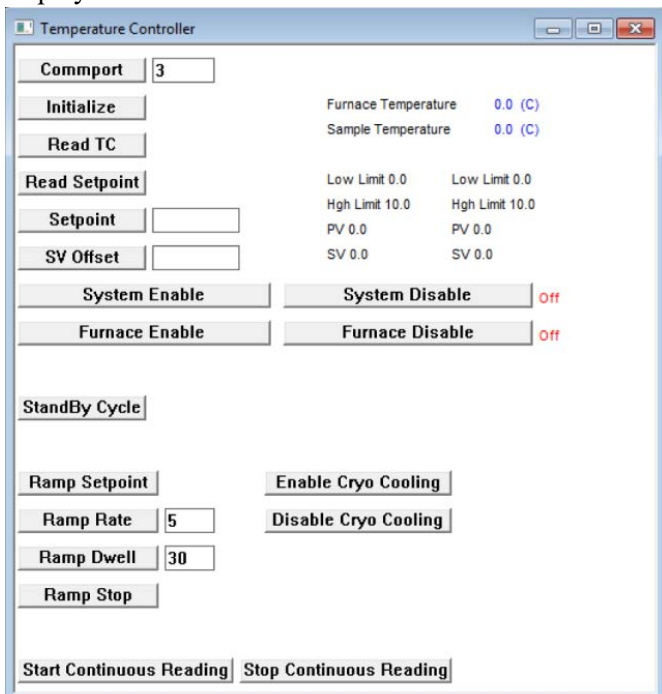


Figure 47 Temperature Controller window.

- 3 Click **Initialize**.
- 4 Click **System Enable** both from the Temperature Controller window and on the front panel of the instrument.
- 5 Click **Furnace Enable** both from the Temperature Controller window and on the front panel of the instrument.
- 6 Enter **200** in the field next to the Setpoint button. Do not click the Setpoint button.
- 7 Enter 10 in the field next to Ramp Rate and click **Ramp Rate**.
- 8 Enter 120 in the field next to Ramp Dwell and click **Ramp Dwell**.
- 9 Click **Ramp Setpoint** to start heating.
- 10 After 2 hours at 200°C, the heating stops and the system cools down.

The next time that the furnace is used for a new test, the vacuum should be below 50 mTorr in less than 30 minutes.

Replacement Parts

Replacement parts for the DXF are listed below. Refer to the table below when ordering parts.

Part Number	Description
202315.001	Moderate Vacuum Module: large capacity two-stage mechanical vacuum pump
853024.901	In-Plane Test Kit: fixture used to mask the energy pulse for thermal diffusivity determination in the X-Y plane. The kit includes customized software.
202417.001	Graphite Paint
202419.001	Silver Paint - Vial - 1 oz.
853135.901	Thermographite Reference, 12.7 mm OD, 3.175 mm thick
853142.901	Thermographite Reference, 25.4 mm OD x 3.175 mm thick
853143.901	Stainless Steel Reference, 12.7 mm OD x 1.58 mm thick
853144.901	Stainless Steel Reference, 25.4 mm OD x 1.58 mm thick
853145.901	Electrolytic Iron Reference, 12.7 mm OD x 2.54 mm thick
853146.901	Electrolytic Iron Reference, 25.4 mm OD x 2.54 mm thick
853147.901	OFHC Copper Reference, 12.7 mm OD x 5.08 mm thick
853152.901	Vespel Reference, 12.7 mm OD x 1.27 mm thick
853154.001	Alumina Reference, 12.7 mm OD x 3.175 mm thick
853157.901	Molybdenum Reference, 12.7 mm OD x 3.175 mm thick
858160.901	Fused silica optical light pipe assembly

Appendix A:

Connection Diagram

