DISCOVERY LASER FLASH
DLF-2800

Getting Started Guide

Revision C Issued April 2020

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.

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Introduction

Important: TA Instruments Manual Supplement

Please click the TA Manual Supplement link to access the following important information supplemental to this Getting Started Guide:

- TA Instruments Trademarks
- TA Instruments Patents
- Other Trademarks
- TA Instruments End-User License Agreement
- 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.

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *DLF 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.

IEC 60825-1 Safety of laser products, Part 1: Equipment classification and requirements.

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.

ANSI Z136.1-2007 American National Standard for Safe Use of Laser

21CFR 1040.10- Code of Federal Regulations, title 21, part 1040-Performance Standard for Light-Emitting Products, #.10- Laser Products

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 interface 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 interface in which case the user will be required to correct the interface at own expense.

Safety

Instrument Symbols

The following labels are displayed on the instrument for your protection:

Symbol	Explanation
	This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn to come in contact with this hot surface. 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.
	This symbol indicates that exposure to laser radiation is possible if not used correctly. Follow instructions for use as detailed in the DLF Getting Started Guide. Ce symbole indique l'exposition à un rayonnement laser est possibles s'il est mal utilisé. Suivez les instructions d'utilisation détaillées dans le Guide de démarrage du DLF.
	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 DLF 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 DLF.

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'opérateur de cet instrument est informé que si l'équipement est utilisé de manière non spécifiée dans ce manuel, la protection fournie par l'équipement peut être altérée.

WARNING: Always turn off power to the instrument before performing any maintenance.

AVERTISSEMENT: Toujours éteindre l'instrument avant d'effectuer toute maintenance.

Electrical Safety

You must turn off power to the instrument before doing any maintenance or repair work; voltages as high as 240 VAC are present in the instrument.

DANGER: Risk of electric shock. High voltages are present in the instrument and power cart. There are no user-servicable parts within the instrument or power cart. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

DANGER: Risque de choc électrique. Des tensions élevées sont présentes dans l'instrument et le chariot de puissance. Il n'y a pas de pièces pouvant être réparées par l'utilisateur dans l'instrument ou le chariot d'alimentation. L'entretien et la réparation des pièces internes doivent être effectués uniquement par TA Instruments ou un autre personnel 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.

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: Always turn off power to the instrument before you examine or replace the fuses in the DLF-2 or the EM-2800 Furnace Module.

AVERTISSEMENT: Mettez toujours l'instrument hors tension avant d'examiner ou de remplacer les fusibles du DLF-2 ou du module de four EM-2800.

Liquid Nitrogen Safety

The Detector Assembly uses the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. 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: 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 DLF system to return to near room temperature before you touch the inner sample holder surfaces. Always use the available software monitor to display temperature even after the termination of the test cycle.

DANGER: Les surfaces des instruments peuvent être suffisamment chaudes pour provoquer des inconforts lorsqu'ils sont en contact avec la peau lors d'un essai. Si vous effectuez un test sous-ambiant, le froid pourrait également causer des blessures. Après avoir exécuté tout type d'expérience, vous devez autoriser le système DLF à revenir à une température ambiante proche avant de toucher les surfaces internes du support d'échantillon. Utilisez toujours le moniteur logiciel disponible pour afficher la température même après la fin du cycle de test.

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. There is no recovery method to restart the test at that point, but all data is saved up until the power failure occurs.

Do not attempt to circumnavigate the standard test sequence or press any buttons on the front panel after a power failure. Power off and back on all components (PC, DLF, and Environmental Module) to reset them to a known state, and then start a test normally. If the test was running under vacuum, verify that the vacuum level is appropriate. Refill the detector with liquid nitrogen, and check that furnace cooling is set up properly and functioning after a power failure.

Chemical Safety

WARNING: Do not use hydrogen, oxygen, or any other explosive gas in the DLF system. Only inert gas, such as nitrogen or argon, should be used for the pneumatic port, and due to high temperature, only argon for the furnace purge (above 1700°C nitrogen combined with graphite particles might result in poisonous gas).

AVERTISSEMENT: Ne pas utiliser d'hydrogène, d'oxygène ou de tout autre gaz explosif dans le système DLF. Seuls les gaz inertes, tels que l'azote ou l'argon, doivent être utilisés pour le port pneumatique, et en raison de la température élevée, seul l'argon pour la purge du four (au-dessus de 1700 ° C, l'azote combiné avec des particules de graphite peut produire un gaz toxique).

WARNING: If you are using samples that may emit harmful gases, vent the gases by placing the instrument near an exhaust.

AVERTISSEMENT: Si vous utilisez des échantillons qui émettent des gaz nocifs, ventilez les gaz en plaçant l'instrument près d'un échappement.

Laser Safety

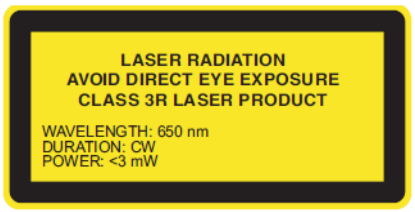
This instrument is a Class I/1 Laser product which, during normal operation, does not permit human access to laser radiation.

Class I/1 Laser Products are accompanied by the following label:

This product complies with IEC 60825-1, with FDA performance standards for laser products.

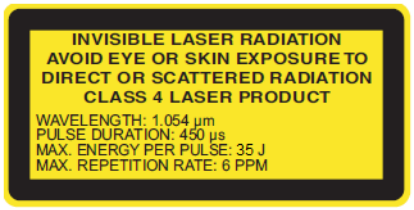
21 CRF 1040.10 except for deviation pursuant to Laser Notice No.50.

This product is Class I/1, with Class IV/4 and IIIA/3R embedded lasers.



DANGER: Avoid direct eye exposure to laser radiation.

DANGER: Évitez l'exposition directe des yeux au rayonnement laser.

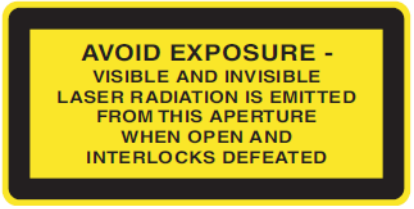


DANGER: Invisible radiation. Avoid direct eye or skin exposure to laser radiation.

DANGER: Rayonnement invisible. Évitez l'exposition directe des yeux ou de la peau au rayonnement laser.

The system is fully interlocked between all component modules to prevent accidental access to high voltages and laser radiation.

Any attempt to defeat the safety interlock elements of this system is a violation of Safety Standards which this product complies with and the protection provided by the product may be impaired.



DANGER: Visible and invisible radiation is emitted from this aperture when open and interlocks defeated.

DANGER: Les rayonnements visibles et invisibles sont émis à partir de cette ouverture lorsqu'elle est ouverte et les dispositifs de verrouillage déclenchés.

CAUTION: Use of controls or adjustments or performance of procedures other than those specified herein may result in hazardous radiation exposure.

MISE EN GARDE: L'utilisation de commandes, de réglages ou l'application de procédures autres que celles indiquées dans le présent document peuvent entraîner une exposition dangereuse aux rayonnements.

Table of Contents

Chapter 1:

Introducing the Discovery Laser 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 Laser Flash (DLF) system automatically determines the thermal conductivity using the measured specific heat capacity and thermal diffusivity, with separately-entered density data.

The DLF-2800 system is comprised of a DLF Pulse Source, Environmental Module, Power Cart, Computer, and associated software.

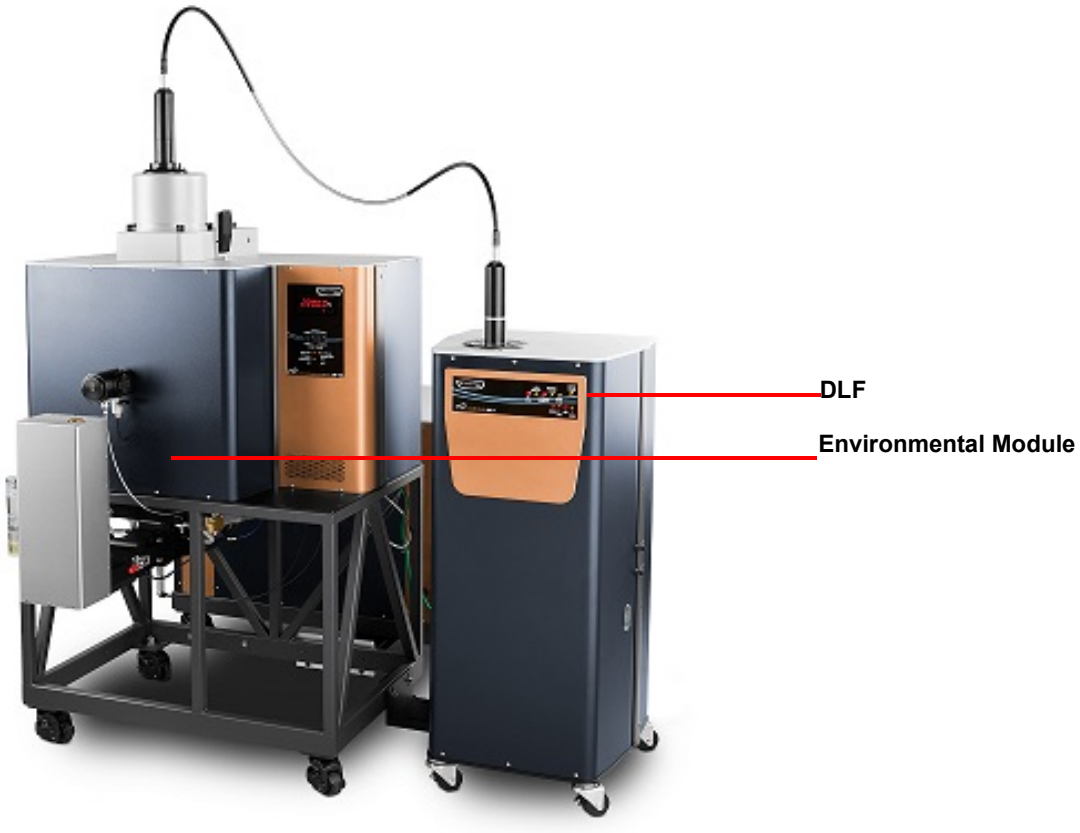


Figure 1 DLF system with EM-2800.

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 DLF system not found in this manual, see the online help associated with the instrument control software.

DLF System Components

A DLF system has three major components: the DLF Pulse Source module, the Environmental Module, and the Power Cart.

Pulse Source Module

The Discovery Laser Flash (DLF) is a Source Module employing a custom laser pulse source to provide a collimated, monochromatic energy pulse to specimens. The laser radiation is delivered via a proprietary fiber optic pulse delivery wand, which ensures homogenized laser pulse energy and produces very high repeatability. The laser is fully shielded and completely safety interlocked for laser radiation and high voltage shock prevention. The laser source is physically separated from the Environmental Module furnace and heating elements so that high specimen temperatures can safely be achieved and maintained.

Figure 2 DLF Pulse Source Module.

Table 1: DLF LED Indicators

Indicator	Location	Function
EM Wand	Front panel	GREEN indicates that the wand located on the EM is properly installed and ready for use. RED indicates that the wand is not installed.
Laser Coolant	Front panel	GREEN indicates that the embedded laser cooling system is operating properly and is ready for use. RED indicates that the coolant is not flowing.
External Interlock	Front panel	GREEN indicates that all of the safety interlocks in the EM are engaged and the system is ready for use. RED indicates that at least one of the EM interlocks is not engaged.
High Voltage Covers	Front panel	GREEN indicates that the high voltage module is closed and ready for use. RED indicates that the module is open.
Laser Wand	Front panel	GREEN indicates that the wand located on the DLF is properly installed and ready for use. RED indicates that the wand is not installed.
Source Power	Front panel	**ON** indicates that the embedded laser control board is powered.
Charge/Arm	Front panel	**ON** indicates that the capacitors are charging. **Blinking** indicates the charge condition for the capacitors is being verified prior to triggering.
Fire	Front panel	**ON** indicates that the command was executed.
Dump	Front panel	**ON** indicates that the embedded laser system is in dump mode (not charging and not firing).

Environmental Module (EM) 2800

The EM-2800 employs graphite heaters and a high-purity graphite muffle and sample holder, supporting continuous RT to 2800°C operation in argon purge and is vacuum tight to 50 mTorr. The EM-2800 includes a six-sample (nominally 12.7 mm diameter) holder.

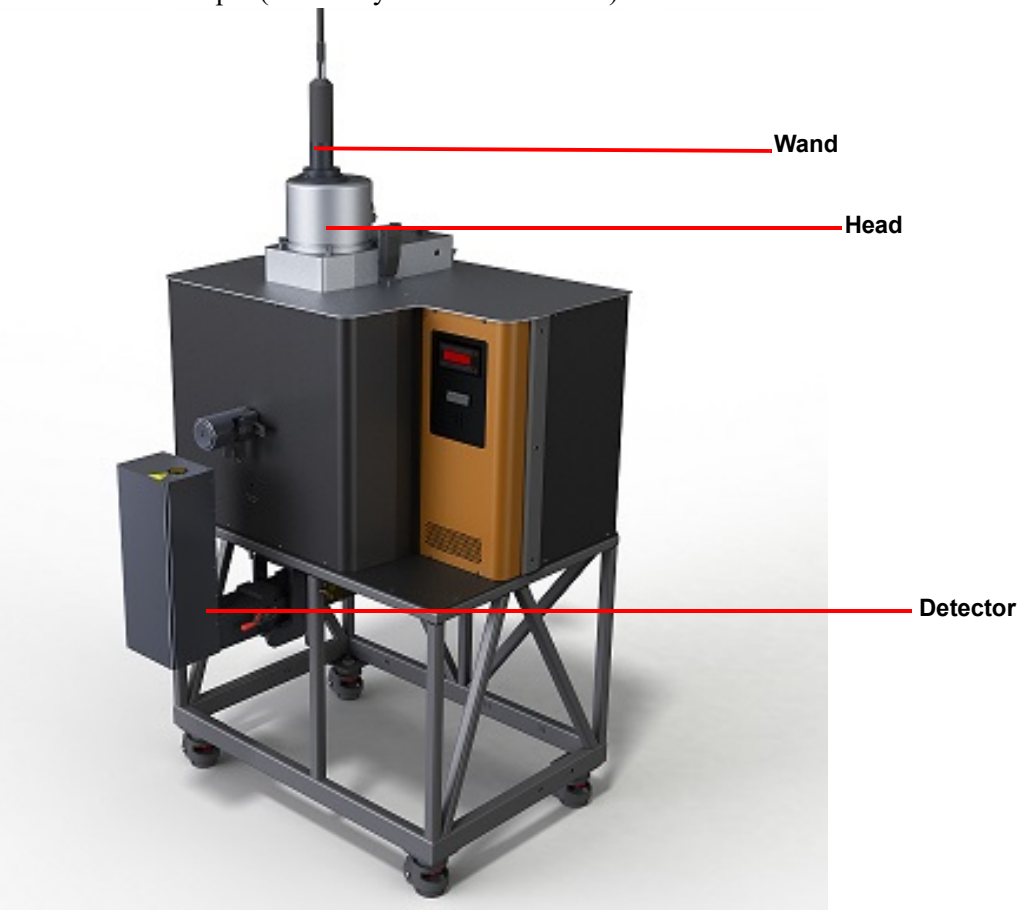


Figure 3 EM-2800.

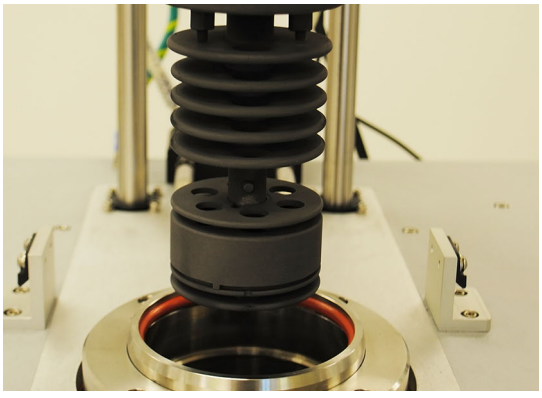


Figure 4 EM-2800 six-sample holder.

Table 2: EM-2800 LED Indicators

Indicator	Location	Function
°C	Front panel	Furnace temperature
System Enable switch	Front panel	**ON** indicates that the system is ready to execute all acquisition functions.
Furnace Enable switch	Front panel	**ON** indicates that the furnace is available and ready to heat/control the temperature parameters. **OFF** indicates one of the following: • The furnace is unable to heat • Heating of the furnace was stopped • Any of the conditions for enabling the furnace were violated
Furnace Diable switch	Front panel	Pressing the switch will disable the furnace and prevent heating.
Sample Motion switch	Front panel	**ON** indicates that the Autosampler is in motion. **OFF** indicates that the Autosampler system is not in motion.
Low Gain	Front panel	**ON** indicates the detector amplifier is operating in low gain mode **OFF** indicates the detector is operating in high gain mode.
OT Alarm	Front panel	**ON** indicates one of the following: • The EM has gone over the temperature limit • One of the thermocouples is broken or not connected As a result, the furnace is disabled and will not heat.
Coolant Flow	Front panel	**ON** indicates that the coolant is flowing. **OFF** indicates that the coolant is not flowing and as a result, the furnace is disabled and will not heat.
Aux	Front panel	Not currently used.
12V Power	Front panel	**ON** indicates that the system is powered on.
Shutter Open	Front panel	**ON** indicates that the shutter is open.
F1 In	Front panel	**ON** indicates that Filter 1 has been inserted.
F2 In	Front panel	**ON** indicates that Filter 2 has been inserted.

Table 2: EM-2800 LED Indicators, Continued

12 VDC Power	Back panel	**ON** indicates that the system is powered on.
Head Open	Back panel	**ON** indicates that the head is open and as a result the furnace is disabled and will not heat, nor will the DLF laser fire.
Detector	Back panel	**ON** indicates that the detector is connected and ready for use.
Shutter	Back panel	**ON** indicates that the shutter is open
Gas Pressure	Back panel	**ON** indicates that the gas pressure for both pneumatic and furnace purge is greater than 40 psi and the instrument will function correctly.
Coolant Flow	Back panel	**ON** indicates that the coolant is flowing. **OFF** indicates that the coolant is not flowing, and as a result the furnace is disabled and will not heat.
Furnace Plate OT	Back panel	**ON** indicates that the temperature of the plate above the furnace has gone over the temperature limit and as a result, the furnace is disabled and will not heat.
12V Analog	Back panel	**ON** indicates that the 12V analog power is functioning correctly.

Power Cart

The main function of the Power Cart is to provide the power to the furnace. The Power Cart communicates with the EM2800 temperature measurement to deliver the power needed to meet temperature control requirements. To ensure safe and reliable operation, the Power Cart monitor powers parameters, coolant flow, and coolant temperature. To ensure common protective ground, the Power Cart provides power to all components of the system, including the environmental module, laser source, computer, and monitor.

Figure 5 Power Cart.

Table 3: Power Cart LED Indicators

Indicator	Location	Function
Power (Green)	Front panel	**ON** indicates that the system is powered on.
Furnace Enable (Green)	Front panel	**ON** indicates that power is ready and available for furnace control. **OFF** indicates that power is not available.
Coolant Flow OK (Green)	Front panel	**ON** indicates that coolant supply and return meet minimum flow requirement. **OFF** indicates that flow is turned off or there is not sufficient flow.
Transformer Over Temp (Red)	Front panel	**ON** indicates that transformer reached maximum-allowed temperature (power to the furnace turned off to protect the system). **OFF** indicates that transformer temperature is within the allowed temperature (normal operation).
Coolant Shutoff (Red)	Front panel	**ON** indicates that the shutoff solenoid that controls coolant to the system is closed. **OFF** indicates that the solenoid is open.
Low Purge (Amber)	Front panel	**ON** indicates that low purge is active. **OFF** indicates that low purge is shut off.
High Purge (Amber)	Front panel	**ON** indicates that high purge is active. **OFF** indicates that high purge is shut off.
AUX #1 (Amber)	Front panel	**ON** indicates that the power to Auxiliary #1 outlet is turned on (future feature).
AUX #2 (Amber)	Front panel	**ON** indicates that the power to Auxiliary #2 outlet is turned on (future feature).


POWER
FURNACE ENABLE
COOLANT FLOW OK
TRANSFORMER OVER TEMP
COOLANT SHUTOFF
LOW PURGE
HIGH PURGE
AUX #1
AUX #2
EM2800
PROTECTIVE
CONDUCTOR

Instrument Specifications

The tables found below contain the technical specifications for the DLF instrument.

Table 4: Technical Specifications

Item/Area	Specifications
Dimensions DLF (DxWxH) EM-2800 (DxWxH) Power Cart (DxWxH)	 43 cm (17 in) x43 cm (17 in) x 91 cm (36 in) 53 cm (21 in) x 76 cm (30 in); H 127 cm (50 in) closed and 158 cm (62 in) open 58 cm (23 in) x81 cm (32 in) x 66 cm (26 in) Complete system minimum lab space requirements (DxWxH): 230 cm (90 in) x 168 cm (66 in) x 188 cm (74 in)
Weight DLF EM-2800 Power Cart	 71 kg (156 lbs) 202 kg (445 lbs) 193 kg (426 lbs)
Power[a]	System supply voltage: 200–240 VAC (rated for 15 A) 50 or 60 Hz Furnace supply voltage: 200-240 VAC (rated for 80 A) 50 or 60 Hz
Cooling	House water supply: Minimum water flow 1.5 GPM with 25°C inlet and maximum pressure of 80 psi. Or Circulator/Cooler: Minimum capacity 10KVA at 40°C coolant return and maximum pressure of 80 psi.
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	Laser module 3% Nd Phosphate Glass
Energy	35 J max
Pulse duration	450 us
Wavelength	1.054 µm
Pulse rate (diagnostic mode only)	1 pulse/min max
Temperature Measurement	Back port: Full range (RT to 2800°C) pyrometer Front port: Limited range (900 to 2800°C) dual color pyrometer
Thermal Diffusivity	Accuracy[b] ± 5%

Table 4: Technical Specifications

Item/Area	Specifications
Measurement	Repeatability[c] ± 2%
Temperature Range	RT to 2800°C
Atmosphere	Argon Grade 5, vacuum (maximum temperature 2000°C)
Number of Samples	6
Sample Dimension	12.7 mm (d) x up to 6 mm (t)
Sample Shape	Round disk

a. The DLF, EM-2800, Computer, and Monitor are plugged with supplied power cords to designated outlets at the back of the Power Cart.

The EM-2800 furnace is connected to the Power Cart with supplied high current cords.

The Power Cart is connected to the wall outlet with power cords in a protective conduit.

The Customer is responsible for providing the appropriate rated power connection wall-mounted box (see Site Preparation Guide for more details). Make sure that the mains assigned do not also supply power to noise generating equipment nearby, such as motors, welders, transformers, etc.

b. Using ideal, well-defined, well-behaved samples of known properties

c. Using ideal, well-defined, well-behaved samples of known properties

Chapter 2:

Installing the 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 system 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 Power Cart to the power grid (must be done by a certified electrician)
- Connecting the Power Cart to the Environmental Module (must be performed by TA Instruments Service personnel)
- Connecting the Environmental Module to Pulse Source Module
- Connecting the IR Detector Assembly to the Environmental Module
- Setting up system communication with the controller
- Preparing and connecting the utilities

It is required that you have your 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.

Inspecting the System

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

In

- A temperature-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 and current 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).
- 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 prone to mechanical vibrations.

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 15 cm (6 in) clearance between the back of the instrument and any objects.

WARNING: For safety, position the equipment in a manner that allows access to the wall outlet for emergency shutoff.

AVERTISSEMENT: Pour plus de sécurité, positionnez l'équipement de manière à permettre l'accès à la prise murale pour la déconnexion 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

The DLF, EM-2800, and Power Cart can be wheeled to their location. The DLF has standard locking brake wheels on the front two wheels, while the EM-2800 and Power Cart wheels can be locked into place. To secure the EM-2800 and Power Cart, turn the red wheel located on each foot until the foot lowers. Continue turning the red wheel until the foot is tight against the floor and the wheel can spin freely.

Figure 6 EM-2800 foot raised (left); EM-2800 foot lowered (right).

Connecting the Power Cart to the Power Grid

DANGER: Risk of electric shock. High voltages are present in the instrument and power cart. There are no user-servicable parts within the instrument or power cart. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

DANGER: Risque de choc électrique. Des tensions élevées sont présentes dans l'instrument et le chariot de puissance. Il n'y a pas de pièces pouvant être réparées par l'utilisateur dans l'instrument ou le chariot d'alimentation. L'entretien et la réparation des pièces internes doivent être effectués uniquement par TA Instruments ou un autre personnel qualifié.

The conduit on the back of the power cart must be connected to a dedicated utility panel with a dedicated main power disconnect switch. The length of the power line conduit is 7 feet, allowing for 3 feet behind the power cart and for the required easy access to the main power disconnect switch. The utility panel must have two circuit breakers for the following power connections:

System supply voltage: 200–240 VAC (rated for 15 A), 50 or 60 Hz.

Furnace supply voltage: 200–240 VAC (rated for 80 A), 50 or 60 Hz.

A label (which is provided with the instrument) indicating the purpose of the switch must be placed in a visible location on the switch cover:.

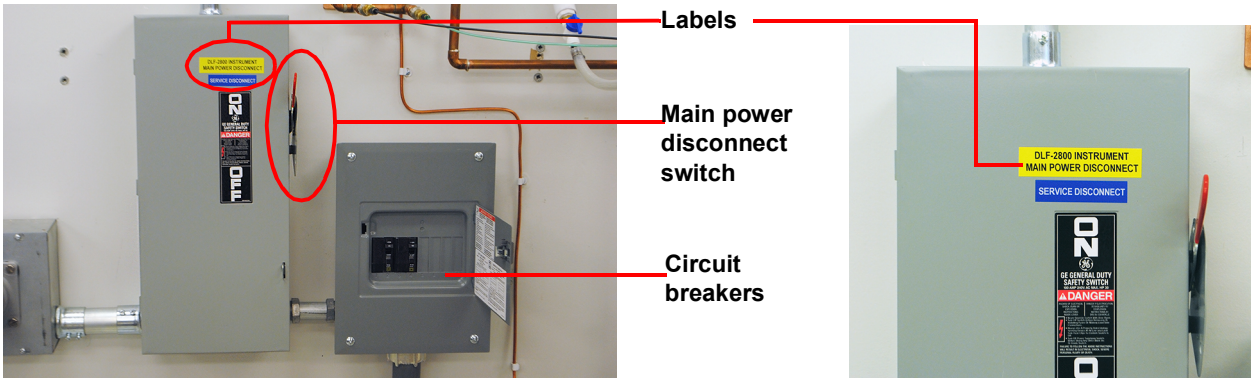


Figure 7 Left: Main power disconnect switch and circuit breakers; Right: Supplied label.

A certified electrician must connect the power cart to the power grid.

The supply voltage must be measured and the appropriate furnace transformer tap–200, 208, 220, and 240 VAC–must be configured.

Refer to the site perparation guide for more information.

Connecting the Environmental Module to the Pulse Source Module

To connect the Environmental Module, you will need access to the DLF 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 the back panel connectors.

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.

DLF Port Connections

The table below provides a description of the function of each DLF port. Refer to Figure 8 for an illustration of rear connections. Ports not described and not labeled are not used.

Table 5: DLF Ports

Port	Location	Function
Main power	Back panel	Powers the DLF
DAQ	Back panel	Provides communication between the DLF and the PC controller.
EM Interlock	Back panel	Connects the DLF to the EM to ensure safe operation of the laser.
Fill/Drain	Right side panel	Coolant drain and refill.
Vent	Right side panel	Provides venting during coolant refill and pressure during coolant drain.

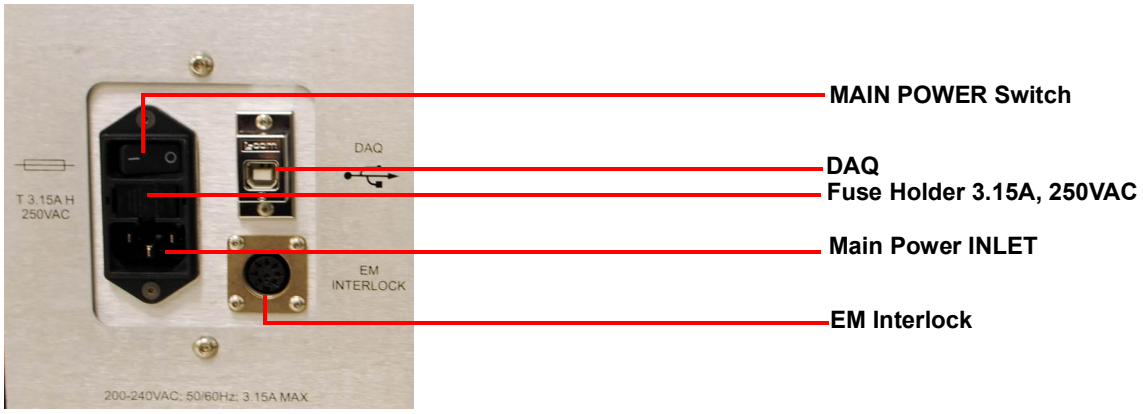


Figure 8 DLF back panel connections.

EM-2800 Port Connections

The table below provides a description of the function of each EM-2800 port. Refer to Figure 9, Figure 10, and Figure 11 for illustrations of rear connections.

Table 6: EM-2800 Ports

Port	Location	Function
Interlock	Back–Data Panel	Connects the EM to the DLF to ensure safe operation of the laser.
Shaft T/C	Back–Data Panel	Connects carousel shaft thermocouple to the measurement system.
Temp Control	Back–Data Panel	Provides temperature-related communication between data acquisition and the PC controller.
DAQ	Back–Data Panel	Provides communication between data acquisition and the PC controller.
Furnace Control	Back–Data Panel	Provides communication between data acquisition and furnace control.
Autosampler	Back–Data Panel	Provides communication between data acquisition and the Autosampler.
Detector	Back–Data Panel	Provides communication between data acquisition and the detector.
Furnace Control	Back–IEC Panel	Provides communication between data acquisition and furnace control.
Pyrometer	Back–IEC Panel	Provides communication between pyrometer and temperature control.
Power Cart	Back–IEC Panel	Provides communication between the power cart and the EM2800.
Vacuum Gauge	Back–IEC Panel	Provides power and communication to the vacuum gauge.
Main Power	Back–IEC Panel	Powers the EM.
Coolant Inlet	Back–Right Panel	Supplies coolant to the EM.
Coolant Outlet	Back–Right Panel	Removes effluent coolant from the EM.
Furnace Purge Inlet	Back–Right Panel	Provides inert gas to the furnace.
Gas Inlet Pneumatics	Back–Right Panel	Provides gas for system pneumatics.
Purge Gas Inlet	Back–Head, Top plate manifold	Provides inert gas to the furnace.
Purge Gas Inlet (#2)	Front of the EM at the bottom of the front port	Keeps the port window clean and provides inert gas to the furnace.
Purge Gas Inlet (#3)	Back of the EM at the bottom of the back port	Keeps the port window clean and provides inert gas to the furnace.

Table 6: EM-2800 Ports

Port	Location	Function
Purge Gas Outlet	Left of EM	Removes gas from the EM.
H1 & H2	Back–Head, Top plate manifold	Connect coolant to the head (H1–inlet, H2–outlet)
Detector	Front of the EM at the back of detector	Provides communication between data acquisition and the detector.
Limited Range Dual Color Pyrometer	Front of the EM at the bottom of the front port	Fiber optics connection to the dual color pyrometer.
Vacuum Gauge	Left of EM	Provides power and communication to the vacuum gauge.

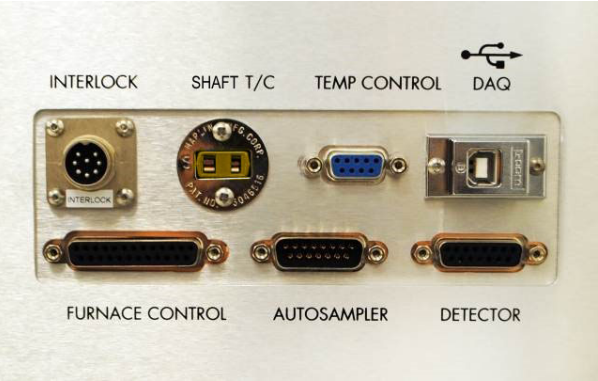


Figure 9 Environmental Module-2800 Data Panel connections.

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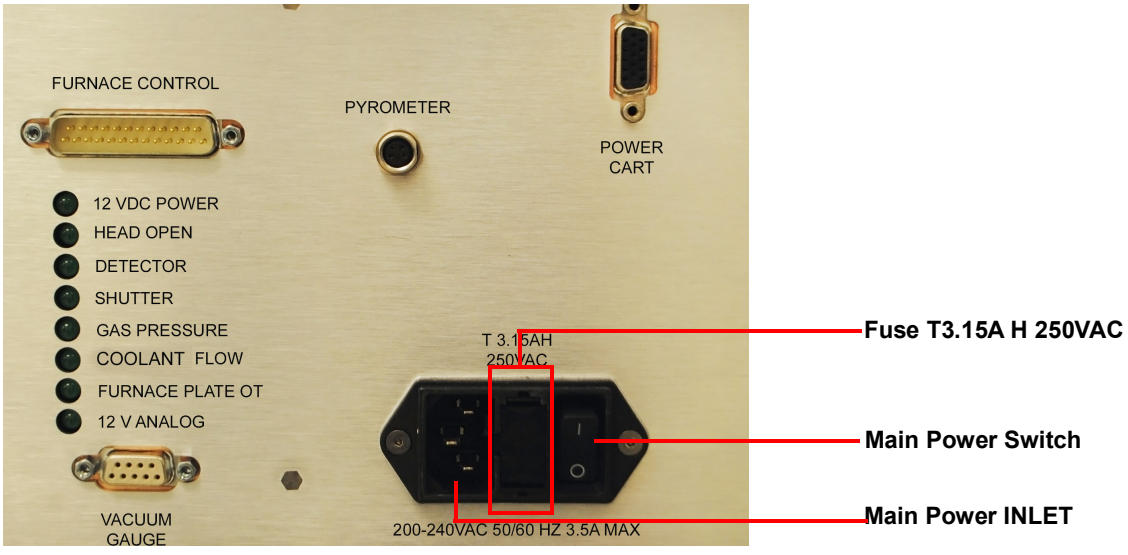

Figure 10 Environmental Module-2800 IEC Panel connections.

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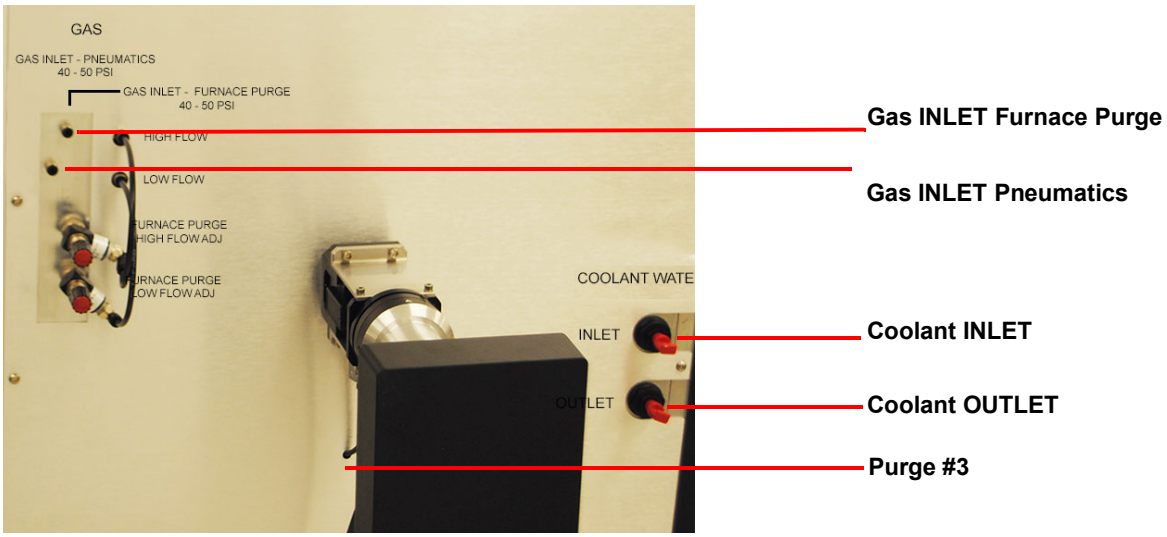

Figure 11 Environmental Module-2800 back-right panel connections.

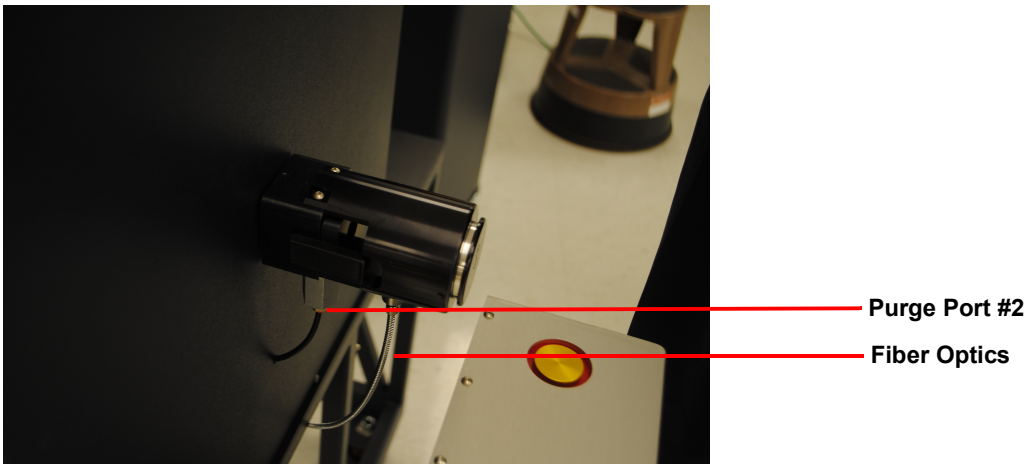

Figure 12 Front purge port and Pyrometer fiber optics.

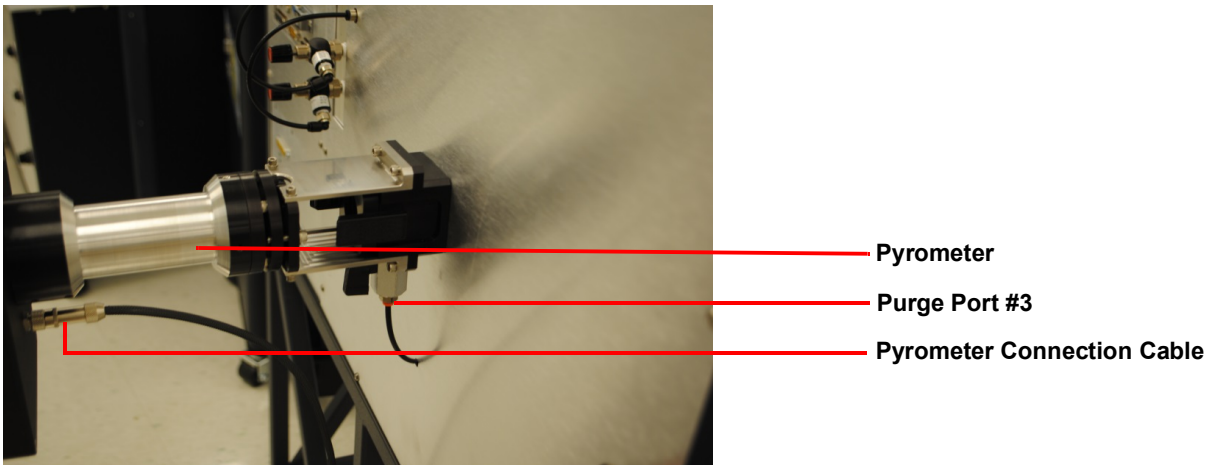

Figure 13 Back purge port and pyrometer.

Environmental Module Connections

To connect the DLF to the system, follow the instructions below. Refer to Figure 8, Figure 9, Figure 10, Figure 11, Figure 12, and Figure 13 above for connection ports.

1 Connect the large attached cable from the back of the EM Head to the Autosampler port on the back of the EM. Connect the smaller attached thermocouple cable from the back of the EM Head to the Sample T/C port on the back of the EM.

2 Connect one end of the Furnace Control cable into the Furnace Control port on the Data (top) Panel of the EM and connect the other end to the Furnace Control port on the IEC (bottom) Panel of the EM.

3 Insert and secure the front pyrometer port, and connect the purge line and fiber optics (Figure 12).

4 Attach the pyrometer to the back port (Figure 13).

5 Connect one end of the pyrometer cable to the pyrometer and the other end to the EM-2800 back IEC panel.

Connecting the IR Detector Assembly to the Environmental Module

Inserting the Removable Lower Window Assembly and Attaching the Detector

1 Insert the Removable Lower Window assembly into the bottom optics assembly all the way into the slot as shown in the figure below and then push the pneumatic actuation switch up (the pneumatics will work once connecting the system to the gas supply. See "Gas Connections" on page 42).

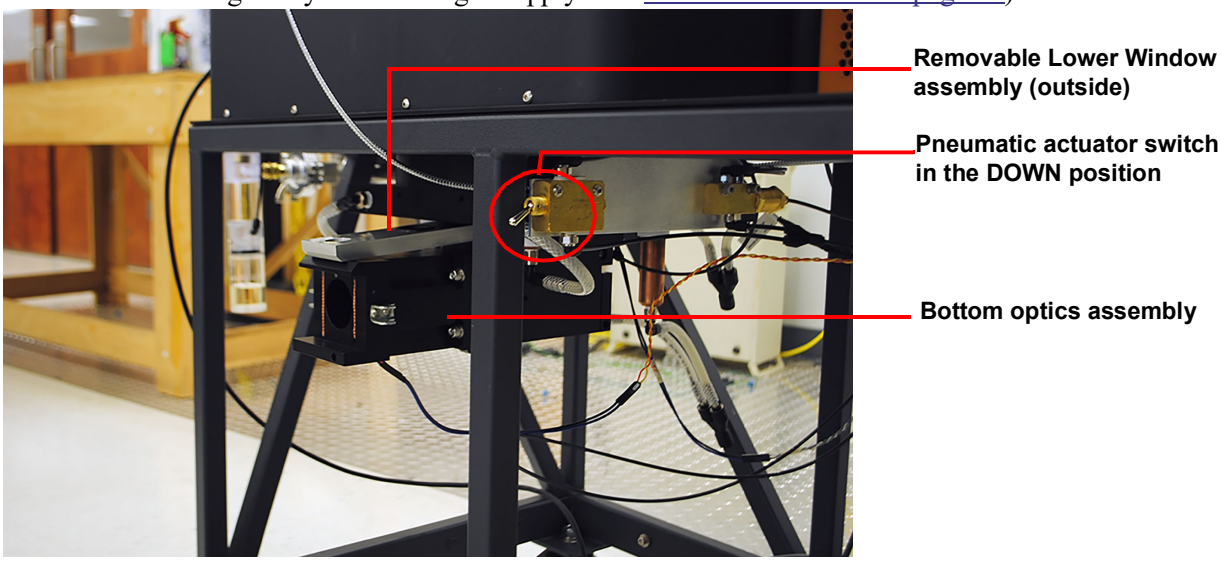

Figure 14 Bottom optics assembly.

2 Attach the Detector to the bottom optics assembly by aligning the front notch and pin and securing both latches simultaneously.

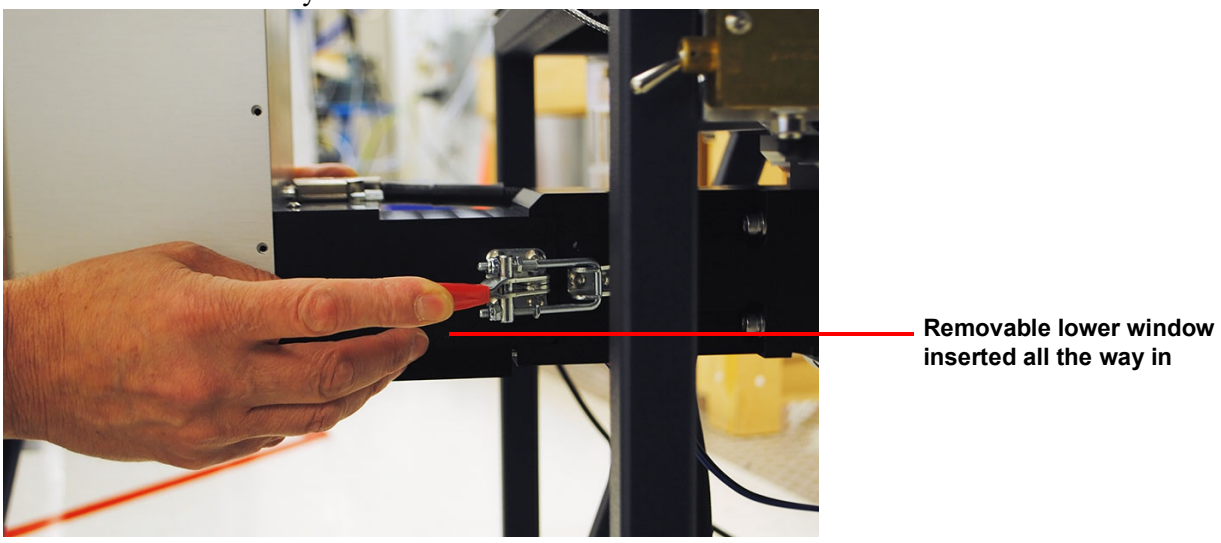

Figure 15 Attach Detector.

3 Connect one side of the Detector cable to the back of the detector, and the other end to the back of the EM (Figure 9).

NOTE: There is no need to remove the detector when removing the Removable Lower Window assembly for cleaning.

Installing the Graphite Carousel

1 Raise the head on the EM.

2 Slide the carousel cover up on the shaft and rotate it to secure from sliding down

3 Attach the samples holder (bottom of the carousel) to the shaft using the supplied graphite nut (finger tight).

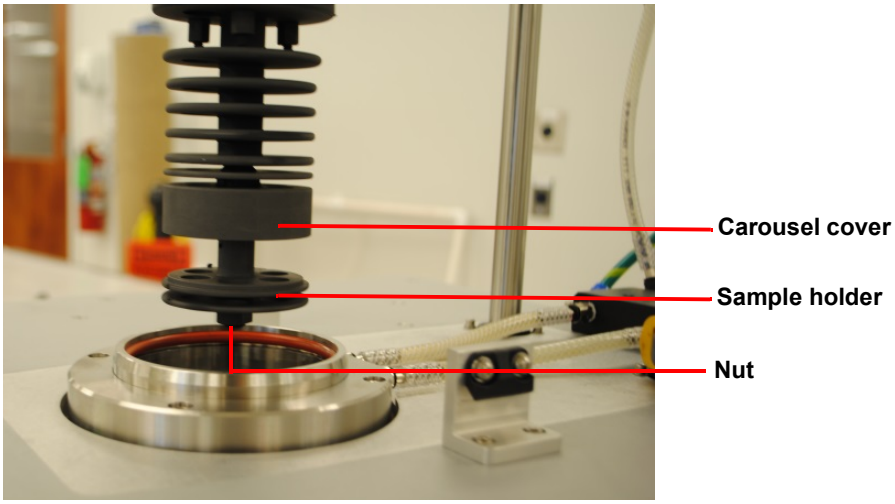

Figure 16 Attach the samples holder.

4 Lower the carousel cover to rest on the sample holder, and rotate until the tab on the cover fits the slot in the holder.

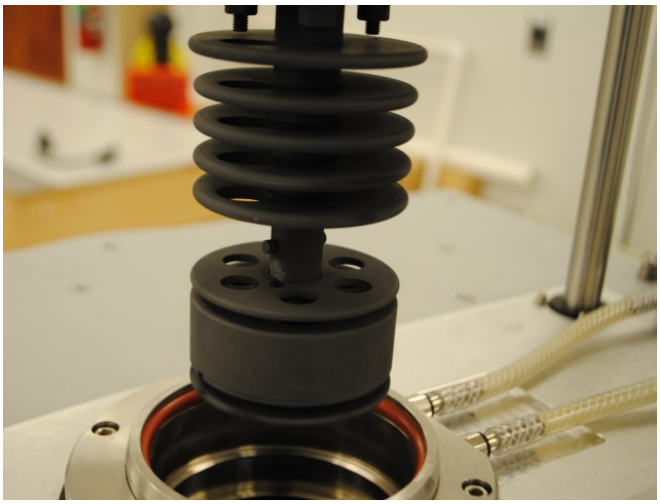

Figure 17 Close the carousel.

5 When placing samples, insert stiff paper or index card under the carousel to prevent items from falling into the furnace tube.

Figure 18 Carousel inserted.

System Connections

The connection of the power cart to the wall outlet must be performed by certified electrician.

The connection of the furnace power conductors from the power cart to the EM-2800 must be preformed by TA service personnel.

1 To align the power cart to the EM-2800, extend the bracket at the front bottom of the power cart to approximately 9" and connect it to the back of the EM-2800 (the final distance depends on the power conductors).

2 Apply supplied silver paste to the fingers at the bottom of the furnace. Then connect the power cart flat front power conductors to the fingers.

3 Connect the ground cables already attached to the power cart (at the bottom right of the front panel) to the underside of the EM-2800.

4 To secure the DLF and protect the fiber optics, extend the bracket attached to the left side of the DLF to approximately 5" and connect it to the right side of the EM-2800 or the power cart (depending on your desired layout).

5 Connect the DLF and EM-2800 power inlets using supplied power cables to the designated outlets at the back of the power cart.

6 Connect one end of the power cart communication cable to the back of the EM-2800 IEC panel and the other end to the front of the power cart panel.

Setting Up System Communication with the Controller

1 Place the computer and monitor to the side of the unit and connect the keyboard and mouse.

2 Connect two USB cables between the back of the controller and the DAQ/USB ports on the back of the EM and the DLF and the front of the Power Cart.

3 Connect an RS232 cable from the Temp Control port on the back of the EM to the controller.

4 Connect the supplied power cords to the computer and monitor and plug them into the designated outlets

on the back of the Power Cart.

5 Turn on the computer and monitor.

NOTE: If the computer was not configured by TA Instruments, please contact TA Instruments Service.

Attaching the Wand to the EM-2800

NOTE: Attaching the wand to the DLF must be done by TA service personnel.

1 Connect one end of the laser interlock cable into Interlock port on the back of the EM and the other end into the back of the DLF.

2 Remove the protective cover from the wand opening on the EM-2800 and from the end of the wand.

3 Align the interlock magnet on the wand with the hole on the top plate

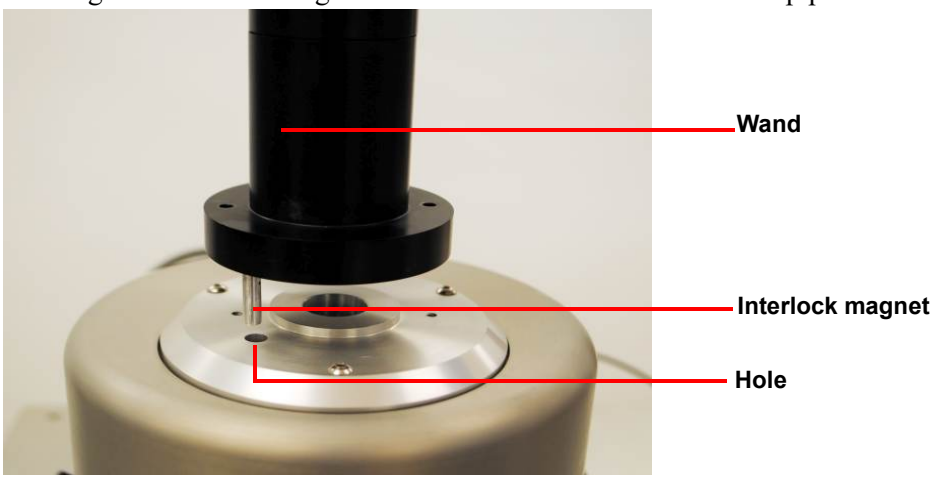

Figure 19 Secure the wand to the EM-2800.

4 Using a 7/62 Allen key, tighten the 2 screws to secure the wand to the Head. Be certain that the wand sits flush on the adjustment plate prior to tightening the screws.

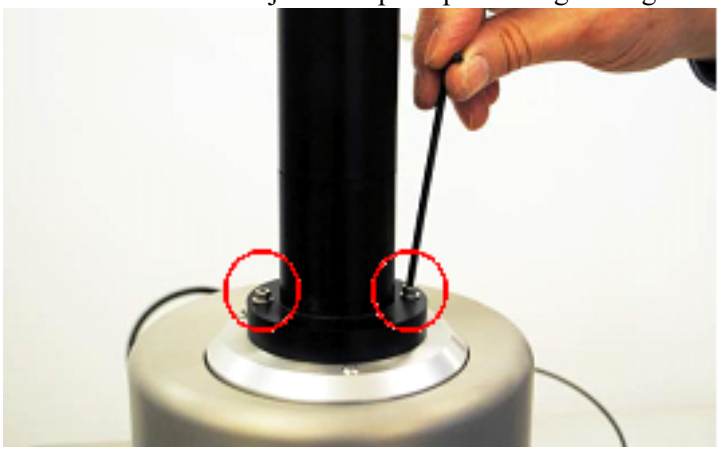

Figure 20 Tighten EM-2800 wand screws

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Table 7: Power Cart Ports (Figure 21 and Figure 22)

Port	Location	Function
EM2800	Front-Right	Communication between Power Cart and EM2800
USB	Front-Right	Communication between the Power Cart and the computer
Protective Conductor	Front Right	Connects the EM2800 ground to the Power Cart ground
Heater Coolant Return	Front-Left	Connection between EM2800 and Power Cart
Furnace Coolant Return	Front-Left	Connection between EM2800 and Power Cart
Furnace Coolant Supply	Front-Left	Connection between EM2800 and Power Cart.
Heater Coolant Supply	Front-Left	Connection between EM2800 and Power Cart
Power Inlet	Back-Left	Connects the system to the facilities power line
Accessories Power	Back-Left	Turns power to the outlets on and off
EM2800	Back-Left	EM2800 power source
DLF2	Back-Left	DLF2 power source
Computer	Back-Left	Computer power source
Monitor	Back-Left	Monitor power source
Power Cart Electronics Switch	Back-Left	Turns power to the Power Cart electronics on and off
Coolant Return #1	Back-Right	Connection to the facilities cooler drain/return
Coolant Return #2	Back-Right	Connection to the facilities cooler drain/return
Coolant Supply	Back-Right	Connection to the facilities cooler drain/return

Figure 21 Power Cart front panel electrical (left) and water (right) connections.

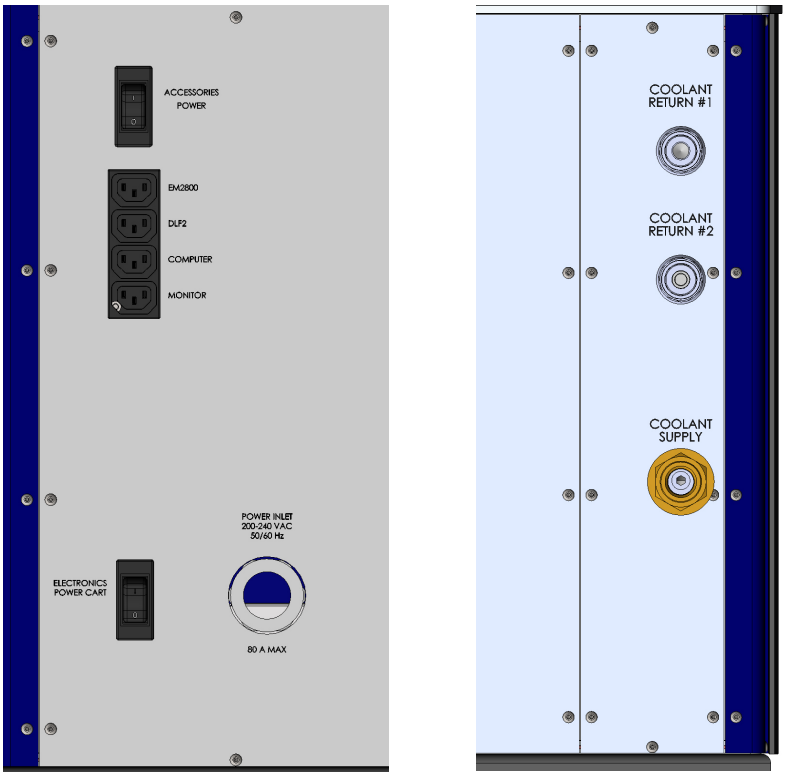


Figure 22 Power Cart back panel electrical (left) and water (right) connections.

Connecting the Utilities

Setting Up the Vacuum System

The vacuum system is an accessory available for purchase at TA Instruments. In order to protect internal graphite parts, it is required that you evacuate the system of oxygen and moisture prior to heating the system to high temperatures.

1 Place the vacuum pump on the floor next to 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.

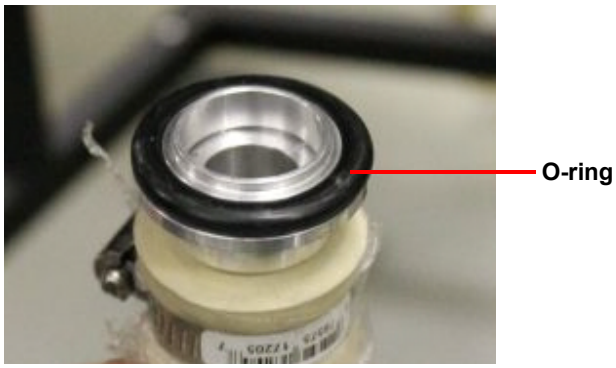


Figure 23 O-ring on hose

3 Clamp the flanges together with a QF-25 clamp.

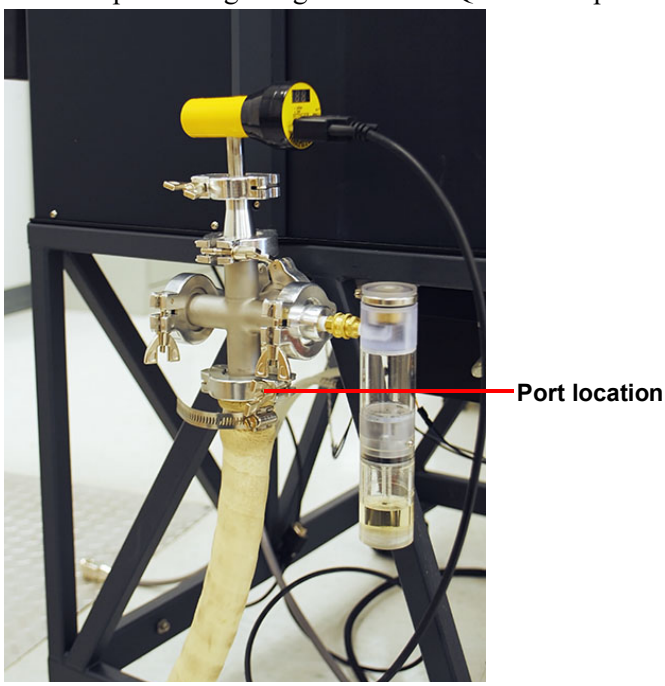


Figure 24 Location of vacuum port.

4 Connect the QF-25 flange on the opposite end of the hose to the QF-25 flange on the mechanical vacuum pump.

Attaching the Anti-Siphon Exit Port

1 Open the anti-siphon exit port by unscrewing the 2 screws with an Allen key. Then fill the port with vacuum oil up to the line indicated in the figure below.

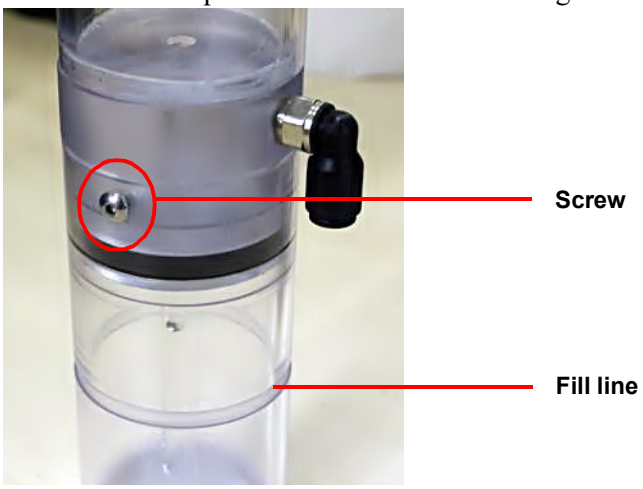


Figure 25 Anti-siphon exit port screw and fill line.

2 Close the port by putting the 2 screws back in place.

NOTE: Overly tightening the screws will cause the port's acrylic to crack.

3 Connect the port into the PURGE GAS OUTLET on the left side of the EM-2800.

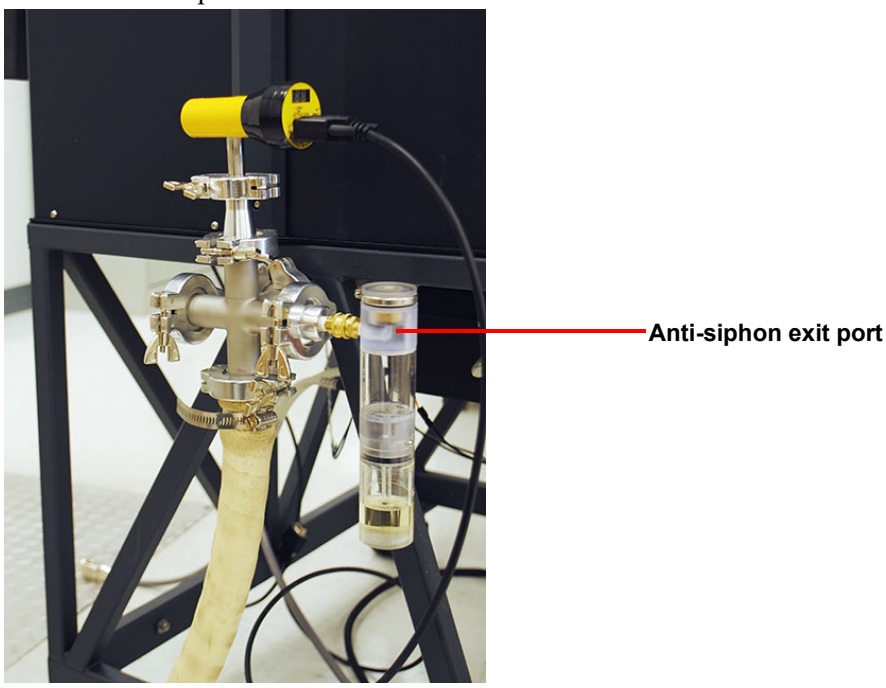


Figure 26 Anti-siphon exit port attached to the EM-2800

Connecting Coolant and Gas Lines

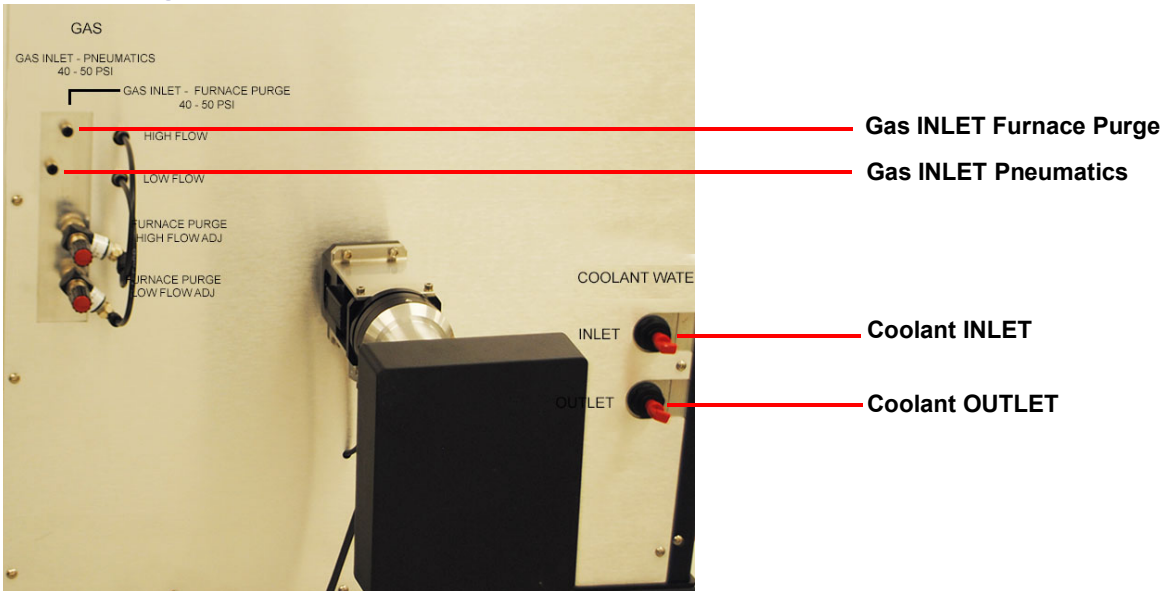


Figure 27 Coolant and gas connections on the back of the EM.

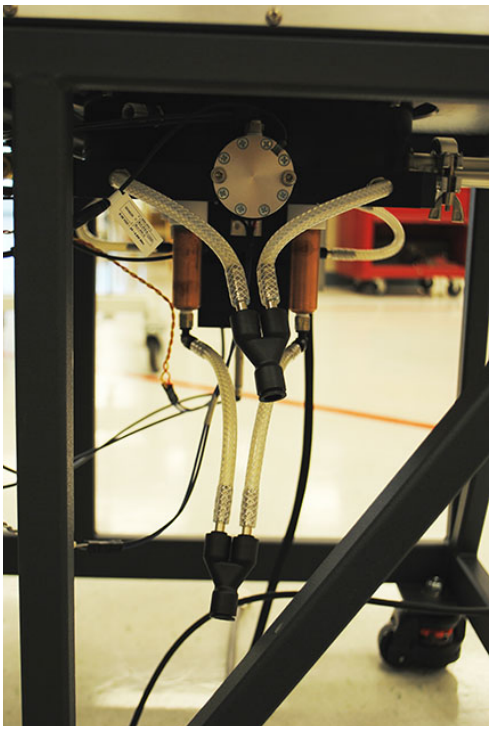


Figure 28 Coolant lines under the EM-2800.

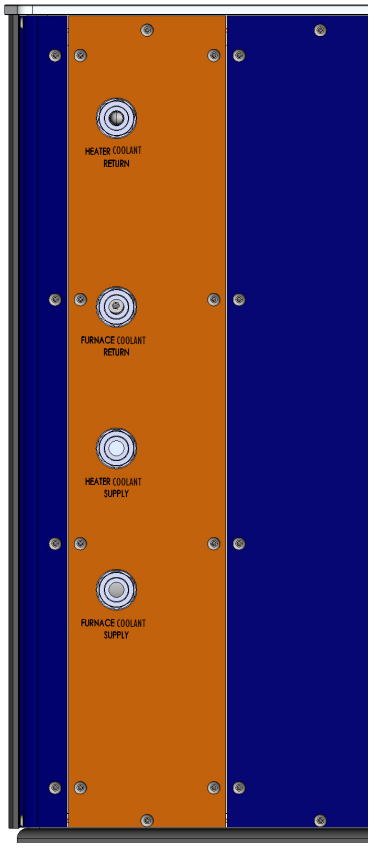


Figure 29 Power Cart front coolant connections.

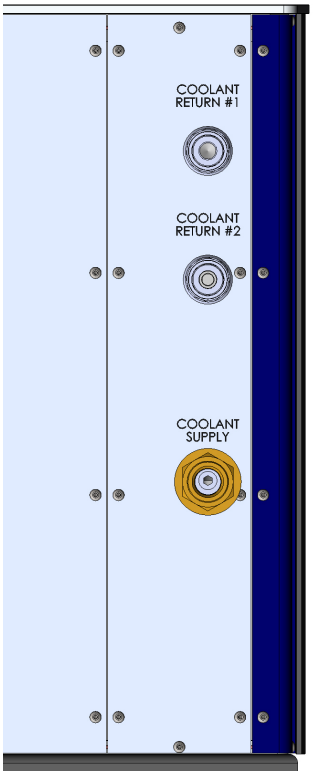


Figure 30 Power Cart back coolant connections.

Coolant Connections

NOTE: The system requires supplied house water of at least 1.5 GPM at 25°C (maximum pressure 80 psi). Water circulator should have capacity of 10KVA at 40°C coolant return.

NOTE: All of the cooling lines have 3/8" ID except the supply inlet at the back of the Power Cart, which uses 1/2" ID. To attach the quick disconnect, pull back on the outer ring of the female connector and insert the mating male connector. Release the outer ring and then gently pull on the hose. It should remain fixed in place.

NOTE: The system is equipped with normally open valve that insure coolant flow in case of lost of power while the furnace is hot. Make sure to close source coolant when making coolant connections or when the system is turned off.

1 Connect one end of the coolant line with quick-disconnect fitting to the COOLANT INLET fitting on the back of the EM and connect the other end to the Furnace Cooling Supply on the front of the Power Cart.

2 Connect one end of the coolant line with quick-disconnect fitting to the COOLANT OUTLET fitting on the back of the EM and connect the other end to the Furnace Cooling Return on the front of the Power Cart.

3 Connect one end of the coolant line with quick-disconnect fitting to the bottom Y shape fitting on the bottom of the EM (see Figure 28) and the other end to the Heater Cooling Supply on the front of the Power Cart.

4 Connect one end of the coolant line with quick-disconnect fitting to the top Y shape fitting on the bottom of the EM (see Figure 28) and the other end to the Heater Cooling Return on the front of the Power Cart.

5 Connect one end of the coolant line with quick-disconnect fitting to the Coolant Supply on the back of the Power Cart and the other end to the coolant source.

6 There are 2 coolant return ports at the back of the Power Cart. Both of them need to be connected to the return/drain. They can be connect separately or joined with a Y fitting that accept two 3/8" and one 1/2" to connect to the return/drain.

7 Check all water lines and connections for leakage. Open the drain and then the source.

Gas Connections

There are two gas ports at the back of the EM-2800. Use Argon for the FURNACE port and inert gas for the PNEUMATIC port. See Figure 27.

NOTE: The system is equipped with normally open valve that insure gas flow in case of lost of power while the furnace is hot. Make sure to close source gas supply (Argon) when making purge gas connections or when system is turned off and furnace purge is not required.

8 Use the 1/8” supplied tube onto the GAS INLET fittings on the back of the EM. Connect the other end of the tube to the Argon and inert gas supply.

9 Use 3 manual shutoff valves to turn off the Argon purge into the furnace. The 3 valves are located on the back of the head, back pyrometer port, and front pyrometer port.

10 Set the gas supply pressure between 40–50 psi.

11 Check all air lines and connections for leakage.

Chapter 3:

Operating the System

Using the DLF

All of your DLF 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
- Loading the sample and closing the EM
- 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 DLF system and the controller have been installed properly. Make sure you have:

- Made all necessary cable connections between the Environmental Module, the DLF, and the Power Cart
- Connected the instrument with the controller
- Connected all coolant and gas lines
- Connected the vacuum system
- 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 DLF System

The main power switch to the system should be on the facility power outlet box connected to the Power Cart.

The power switches are located on the back panel of the Power Cart, the DLF, and the EM-2800. The power switches are used to turn the system on and off.

To power on the system:

1 Check all connections between the Power Cart, DLF, the Environmental Module, the Detector Assembly, and the controller. Make sure each component is plugged into the correct connection port.

2 Set the Power Cart electronics and accessories switches to the ON (**I**) position. Then set the DLF and EM MAIN POWER switches to the ON (**I**) position.

3 The temperature controller and LED indicators on the front panels of the Power Cart, DLF, and EM 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.

4 Turn on the PC power.

Shutting Down the DLF 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 Power Cart, DLF, EM, 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.

To power down your system, close the controller software and then set the DLF and EM MAIN POWER switches to the OFF (**0**) position. Then turn off the accessories and Power Cart electronics. The last step is to turn off the power switch on the facilities outlet box.

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 irregular 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). Measure the thickness of the samples before any sputter or graphite coatings are applied.

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 µm 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 Laser Source. To ensure this, always coat both the sample and the reference with graphite coating.

Applying Graphite Spray

The following is the procedure for coating 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 before coating the other side. Repeat steps 2 through 6.

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

Loading and Unloading the Sample

Loading Samples into the EM-2800

1 On the EM head, press both latches simultaneously sideways to unhook it from the top plate, and gently pull the head up out of the furnace.

WARNING: Autosampler may 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: Le passeur d'échantillons automatique peut être chaud. Soyez prudent lors de la mise en place et de la décharge des échantillons. Vérifiez la température du échantillon sur le panneau avant; si elle est égale ou inférieure à 60° C, alors vous pouvez le manipuler en toute sécurité.

2 Insert a stiff paper or index card under the carousel to prevent samples from falling into the furnace.

3 Raise the lid, rotate it slightly, and rest it on the shoulder of the carousel shaft, and then load the samples into the holder cavities using tweezers (Figure 31). Positions are counted from the cavity marked by a notch (position #1) counterclockwise (Figure 32). All holder cavities must be filled, even if the samples are not being tested. Once all of the samples are loaded, lower the lid back into its resting position.

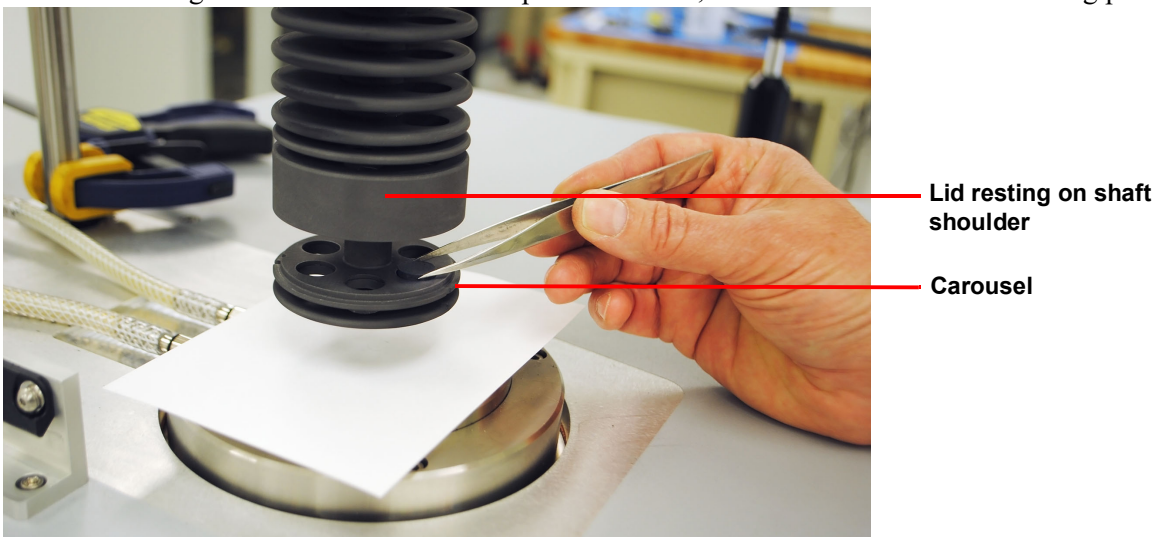


Figure 31 Sample holder.

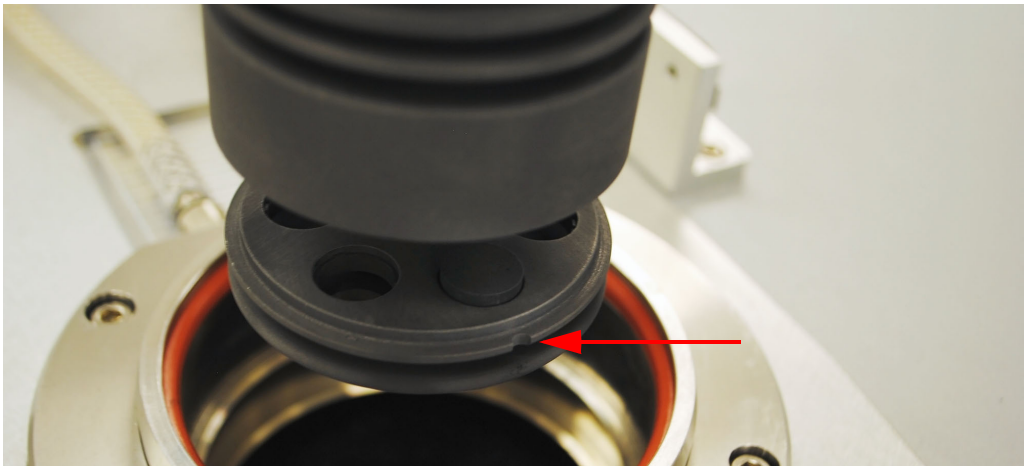


Figure 32 Notch in sample holder indicating Position 1.

4 After all samples have been loaded and lid lowered back into position, push the head back into the EM and secure the latches. The system is now ready for evacuation and purging.

Unloading Samples from the EM-2800

To unload samples from the EM-2800, follow steps 1 and 2 above for loading samples. Then use tweezers to remove the samples from the carousel cavity.

Preparing the Instrument

Evacuating the System

All DLF/EM-2800 systems are equipped with vacuum and purge capabilities. Purge gas exits through the anti-siphon exit port assembly. See Figure 26.

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 vacuum meter assembly for determination of the vacuum level.

Vacuum and Purge Operation

Purge ports are located in the following locations:

- Head of the EM-2800

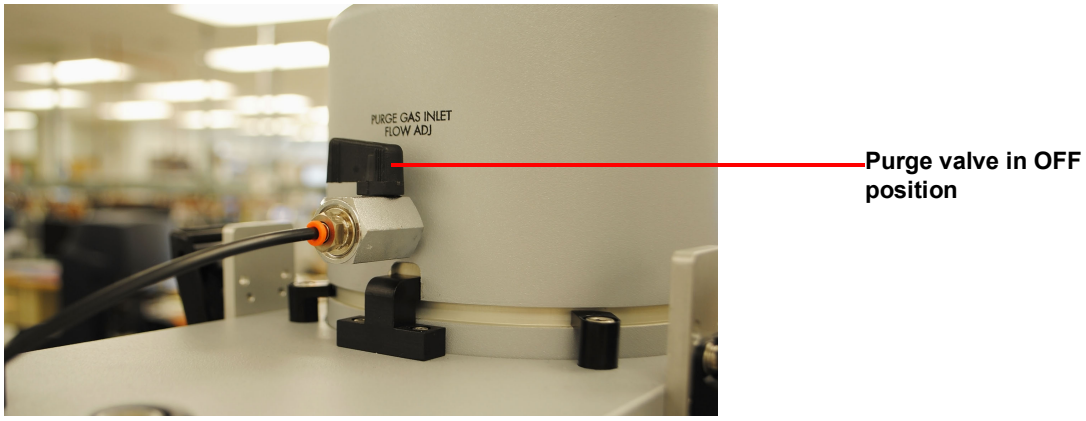


Figure 33

- Front of pyrometer

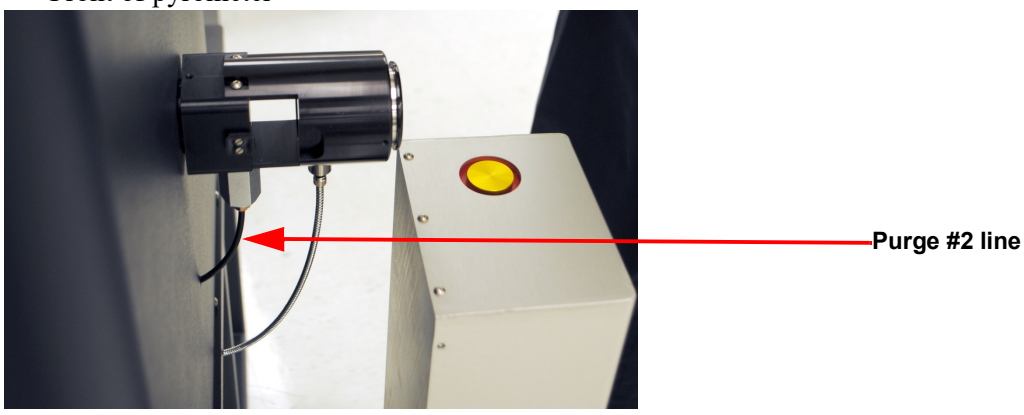


Figure 34

- Back of pyrometer

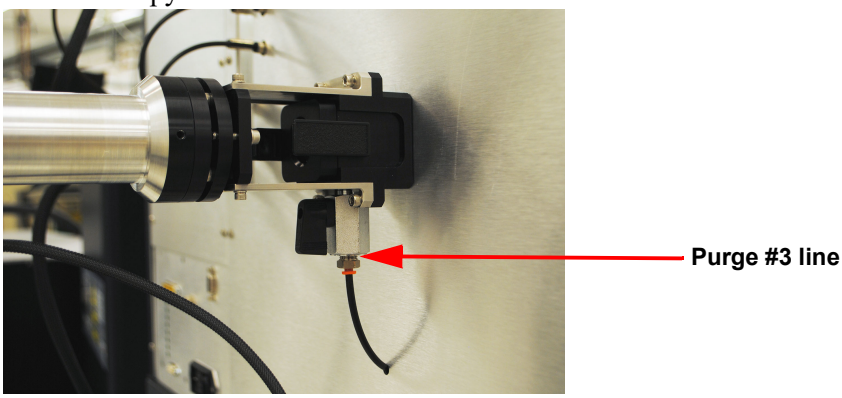


Figure 35

Procedure

1 Verify all vacuum connections.Verify the vacuum pump is prepared as per the manufacturer's specification.

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 the purge valves at the back of the head, front pyrometer, and back pyrometer ports with a quarter-turn.

4 Turn on the vacuum pump.

5 Evacuate the system to at least 50 mTorr. This may require several vacuum/purge cycles depending upon the conditioning of the furnace.

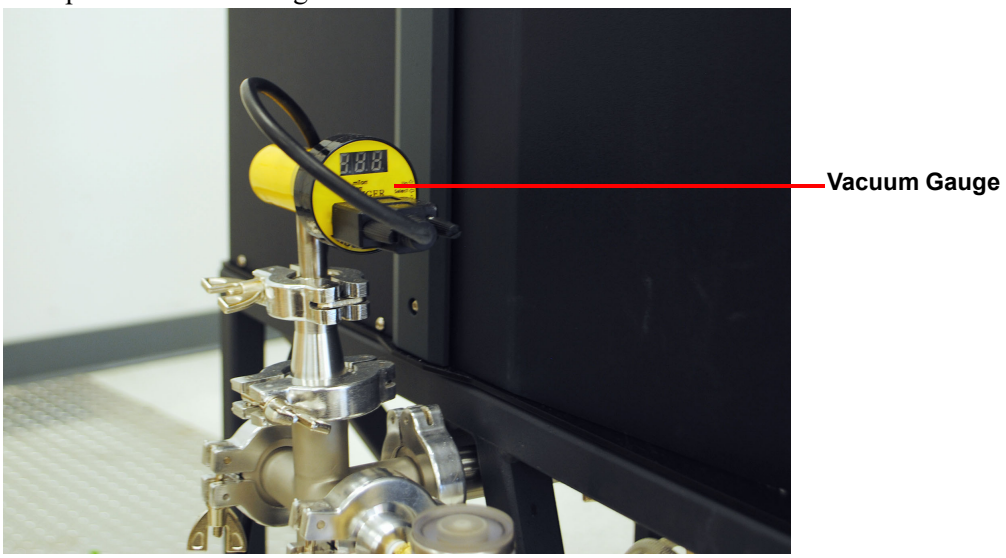


Figure 36 Location of the vacuum gauge.

Back-fill with Purge Gas

1 Turn off the vacuum pump.

2 Open the Purge Gas Inlet valves and enable **High flow** to the EM chamber.

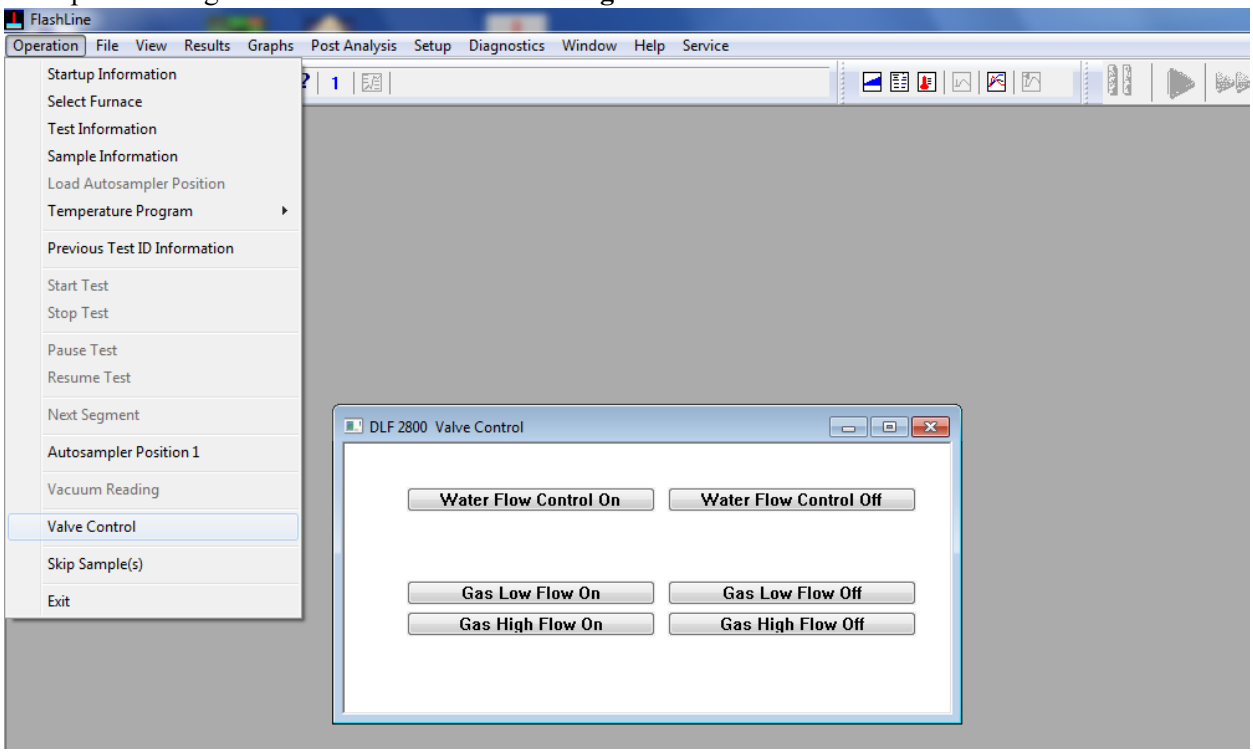


Figure 37 High flow enabled.

3 Monitor the flow using the anti-siphon exit port assembly; once it starts to bubble, turn off the **High flow** in the software. Flow should be continuous, roughly 4– 5 bubbles /second. Adjust with the built-in needle valve at the back of the EM.

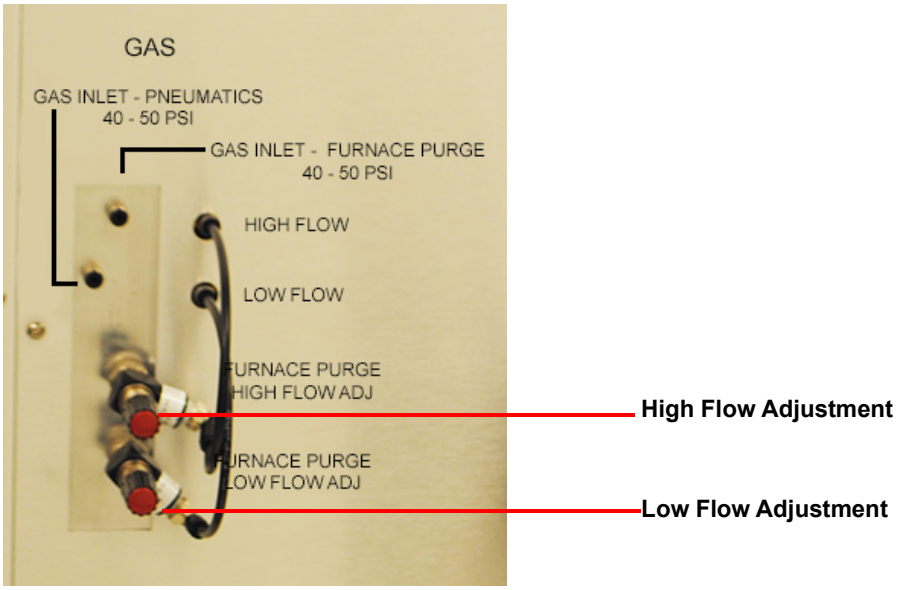


Figure 38

Filling the Detector with Liquid Nitrogen

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

1 Remove the dewar plug.

2 Place the liquid nitrogen funnel (supplied) into the dewar.

3 Fill the detector with liquid nitrogen.

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.

4 Remove the funnel and replace the dewar plug.

5 Allow the dewar to thermally stabilize for 5–10 minutes.

NOTE: Liquid nitrogen may be added while the test is running (if needed), but should not be added while a measurement is in progress.

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. Consult FlashLine Help for experimental instructions.

1 To open the software, double-click the FlashLine icon on the computer desktop.

2 Click **Operation** > **Startup Information**.

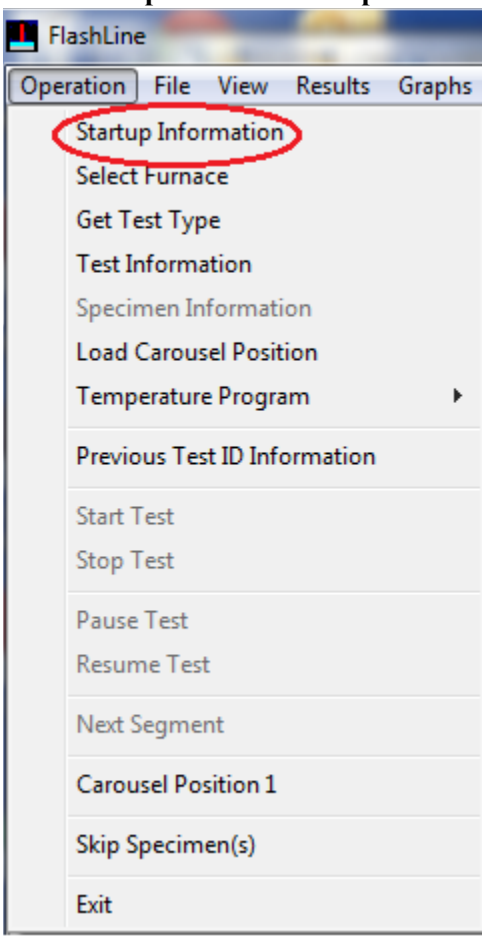


Figure 39 Operation > Startup Information.

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

4 Enter the test parameters and then click **Start Test**. Refer to FlashLine Help for details on entering test parameters in the Test Information window.

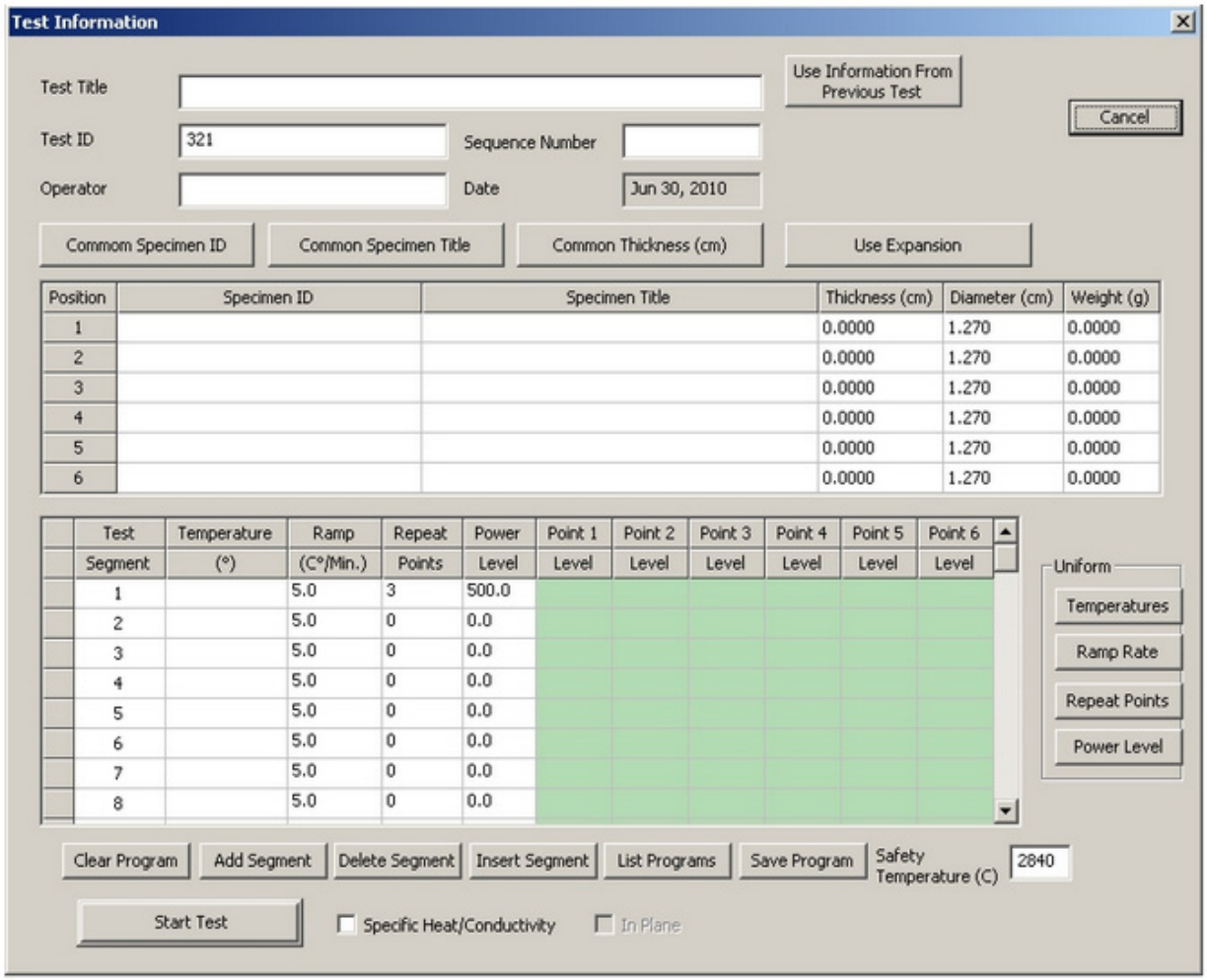


Figure 40 Test Information window.

Stopping an Experiment

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

Chapter 4:

Maintaining the System

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 [le fabricant], vérifiez auprès de TA Instruments que la méthode proposée n'endommagera pas l'instrument.

Cleaning the System

General Cleaning Practices

Use a thin layer of vacuum grease to seal all O-ring parts of the EM-2800 module whenever it is necessary.

General Optics 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
- Remove optics from the instrument prior to cleaning.
- Always use 99% Isopropyl Alcohol and, if needed, acetone during the cleaning process
- If strongly burned films remain on the components after the cleaning process, use weak acid (like vinegar) to try to dissolve the residual layers

Cleaning the Removable Lower Window Assembly

1 Detach the detector from the instrument.

2 Put the pneumatic actuation switch in the down (off) position.

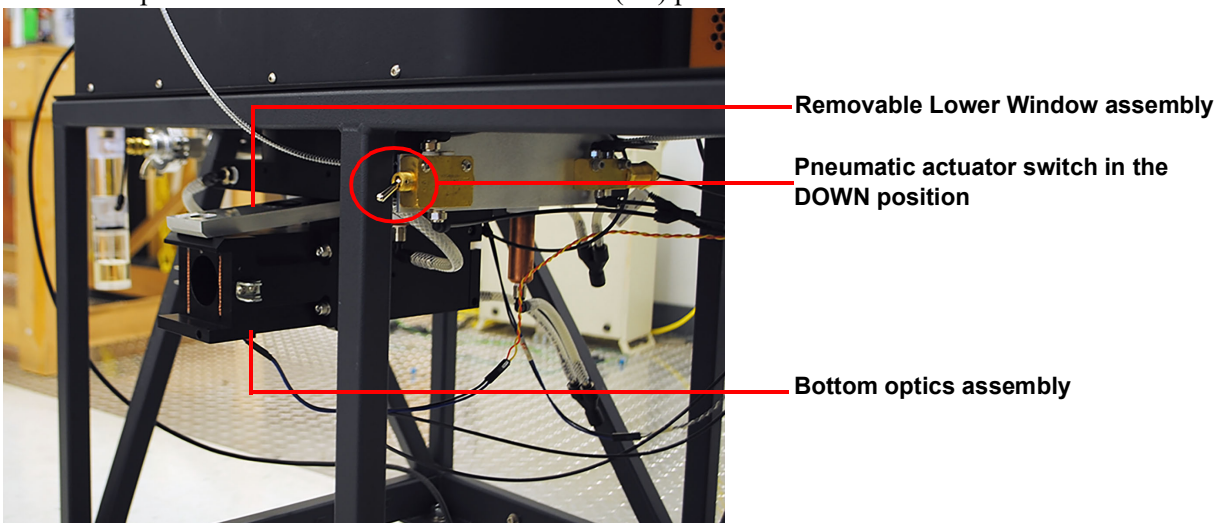


Figure 41

3 Remove the Removable Lower Window assembly from the bottom optics assembly.

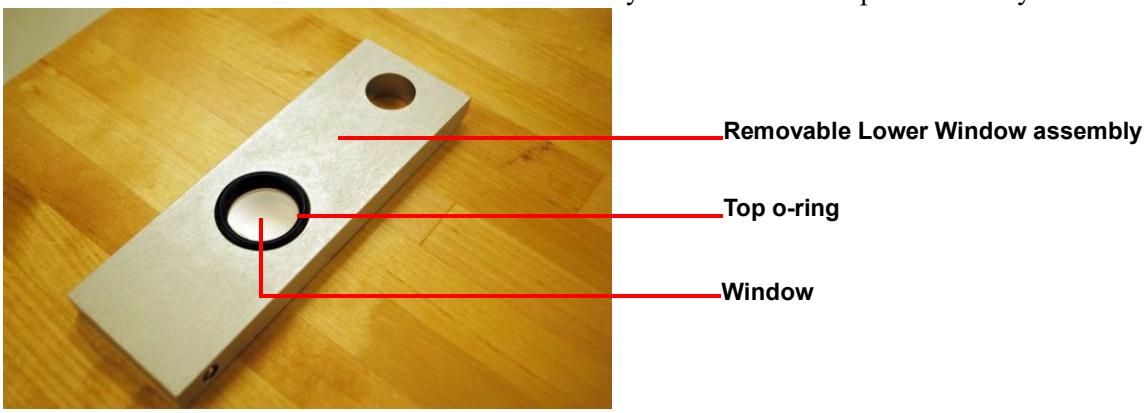


Figure 42 Removable Lower Window assembly removed from bottom optics assembly.

4 Remove the top o-ring and the window from the Removable Lower Window assembly. The bottom o-ring can remain in place.

5 Clean the window and top o-ring with alcohol.

6 Place the window back into the Removable Lower Window assembly. Grease the top o-ring, and then place the o-ring back on top of the window.

7 Insert the Removable Lower Window assembly back into the bottom optics assembly.

8 Put the pneumatic actuation switch back in the up (on) position.

9 Re-attach the detector.

Cleaning the Carousel Control Mounting Plate Window

1 Turn off the instrument.

2 Using a hex key, loosen the screws in the head cover clamps. Turn the clamps away from the head and then remove the head from the instrument.

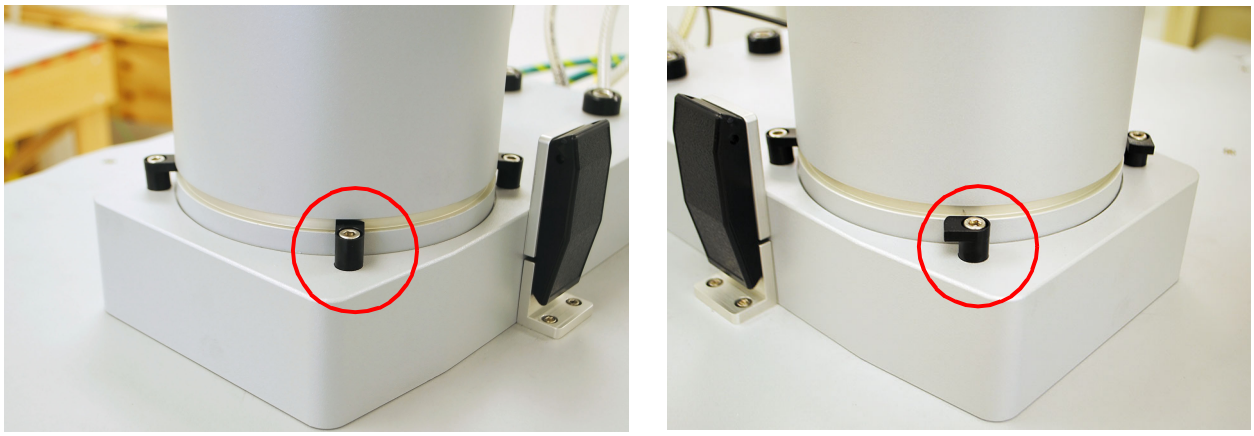


Figure 43 head cover clamp securing the head (left); clamp turned away from the head (right).

3 Using a hex key, loosen the window holder and then rotate it away from the window.

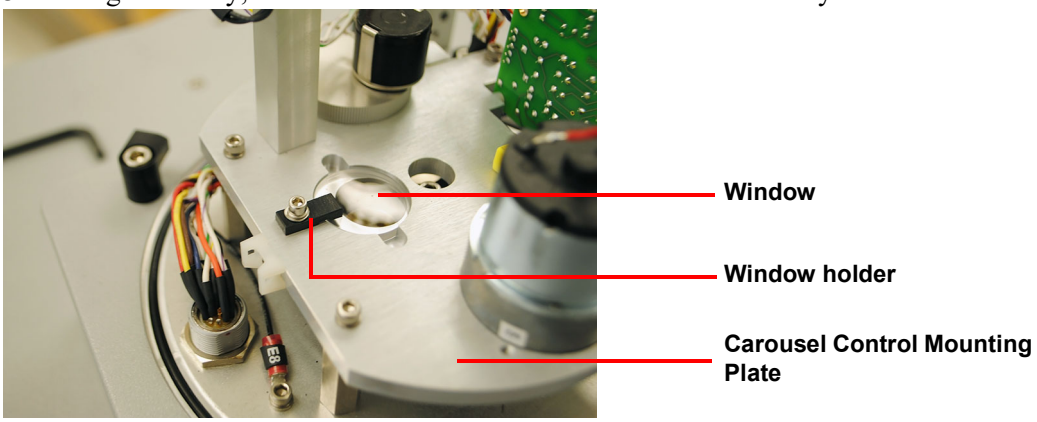


Figure 44 Window holder securing the window in place.

4 Using tweezers, remove the window from the Carousel Control Mounting Plate and clean it with alcohol. Replace the window when finished.

5 Reattach the head. Make sure that the cutout on the back of the head aligns with the stationary clamp under the purge gas port. Secure the head by turning all of the head cover clamps back into their original positions; tighten the screws.

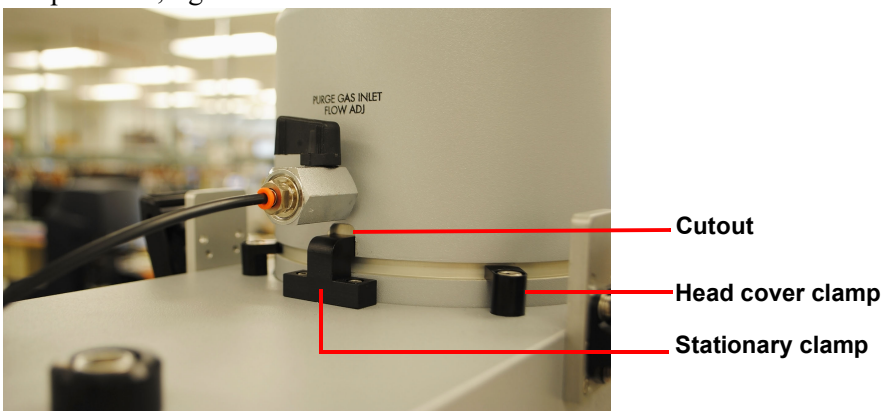


Figure 45 Cutout aligned with stationary clamp.

Cleaning the Windows in the Front and Back Pyrometers

1 Unlatch the window holder and remove it from the pyrometer.

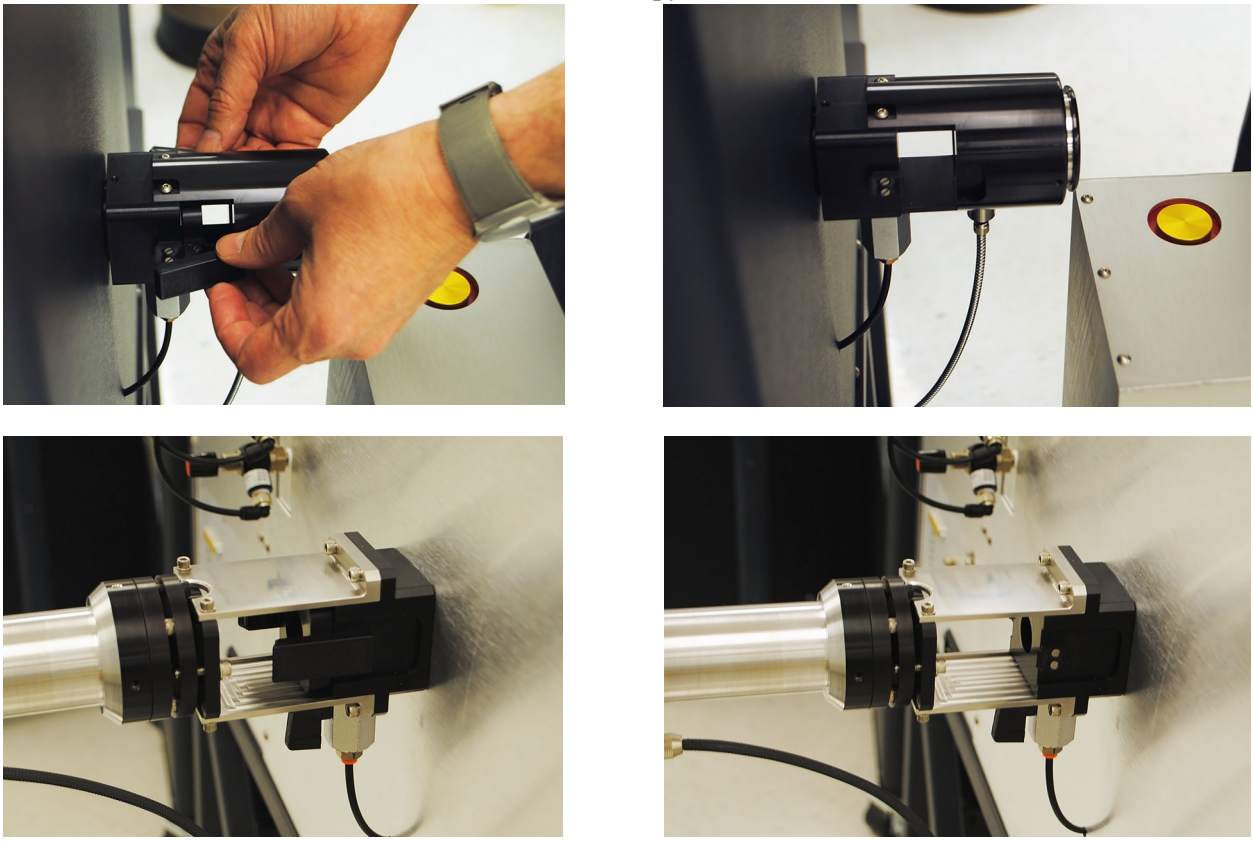


Figure 46 **Top left**: Unlatching the front pyrometer port; **Top right**: Window holder removed from front pyrometer port.
Bottom left: Back pyrometer port; **Bottom right**: Window holder removed from the back pyrometer port.

2 Remove the top o-ring and the window from the window holder. The bottom o-ring can remain in place. Clean the window and top o-ring with alcohol.

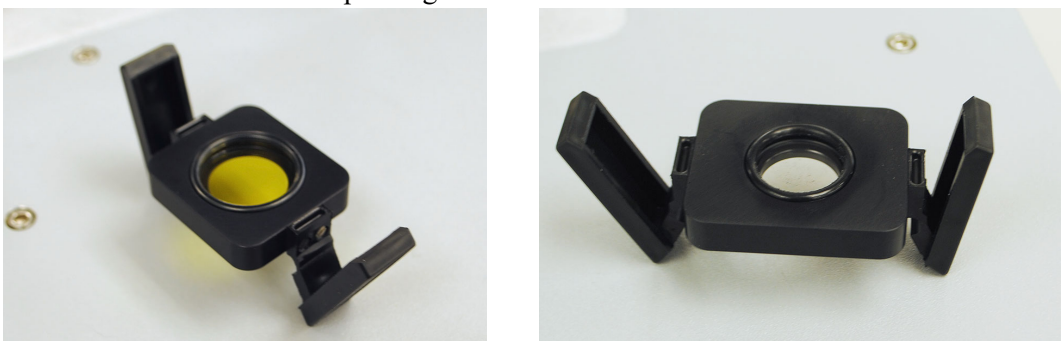


Figure 47 **Left**: Back pyrometer port window holder; **Right**: Front pyrometer port window holder.

NOTE: The back pyrometer port contains a yellow window. Place the yellow window in the back pyrometer port only.

3 Place the window back into the window holder. Grease the top o-ring, and then place the o-ring back on top of the window.

4 Place the window holder back into its appropriate pyrometer port.

5 Secure the latches.

Furnace Conditioning

To restore optimal vacuum performance (<50 mTorr) at room temperature:

1 Vacuum the furnace down to 300 mTorr, then purge with grade 5 purity (99.999%) argon gas (high flow) while vacuuming (flushing) for 3 to 5 minutes.

2 Close the purge and view pressure gauge. Repeat flushing until pressure is below 100 mTorr.

3 Set the temperature of the furnace to 1000°C under vacuum for 2 hours.

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

NOTE: High purge flow can be used for faster vacuum release and while cooling from high temperature.

Replacing the DLF and EM 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 DLF and EM contain 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 the fuses located in the fuse holder on the back panel of the DLF and EM; both units have the same fuse holder and use the same fuse. To check or change these fuses:

1 Turn the instrument off and remove the power cord.

2 The instrument power entry module has two standard fuse holders built in. Use a flat-blade screwdriver to remove the fuse carrier.

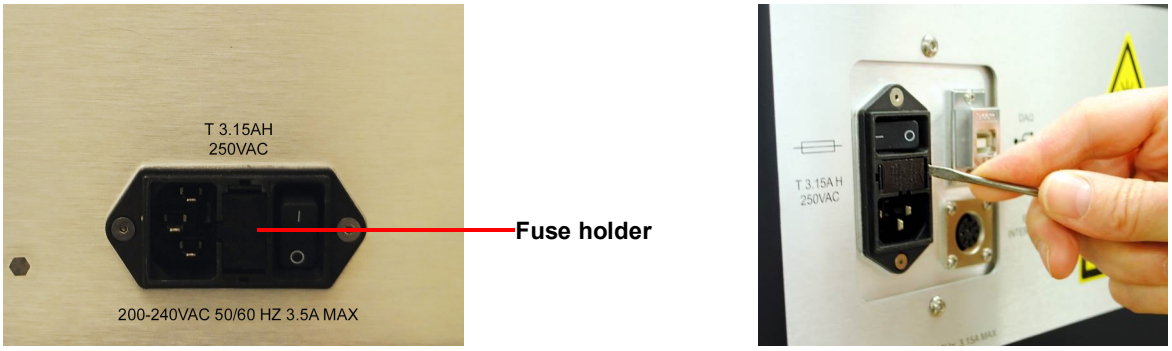


Figure 48 Left: Back of the EM; Right: Back of the DLF.

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

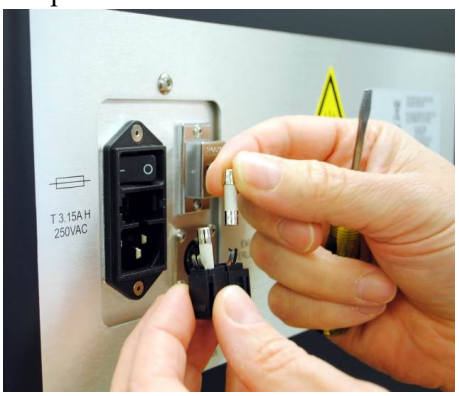


Figure 49 Remove the fuses.

4 Place the fuse carrier back into the fuse holder.

Draining the Fluid

1 Insert the drain tube into the Fill/Drain port on the side of the DLF.

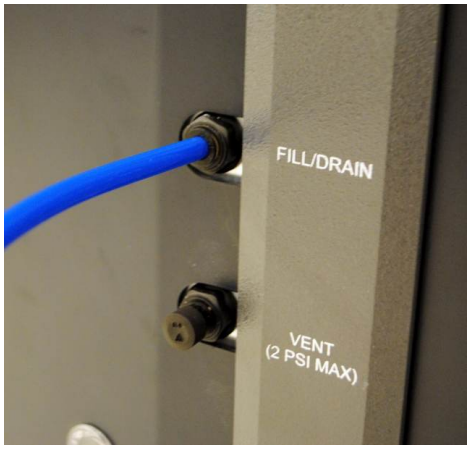


Figure 50 Fill/Drain tube.

2 Place the end of the tube into an empty bottle into which the fluid can drain.

3 Insert the Air Pump tube into the Vent port.

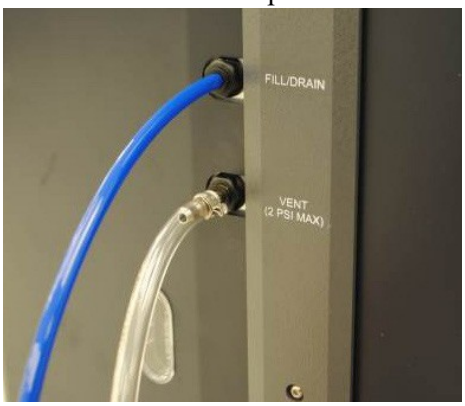


Figure 51 Fill/Drain and Air Pump tubes connected.

4 Pump the Air Pump until the fluid has emptied from the DLF. Drain until no more liquid is coming out of the tube.

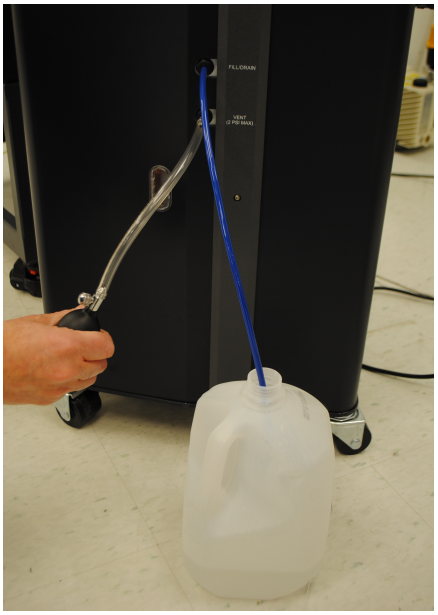


Figure 52 Draining the fluid.

Filling the DLF

1 Attach the funnel bracket onto the top of the DLF by aligning the screws with the holes and tightening the screws by hand.

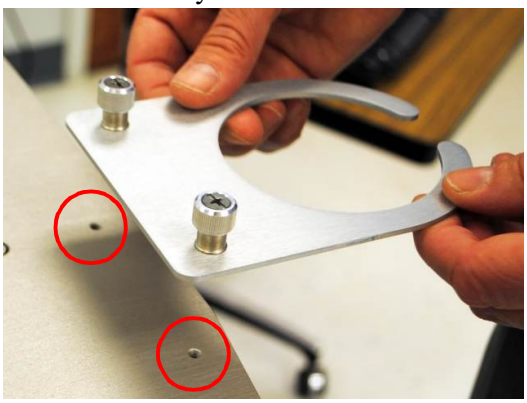


Figure 53 Attach the bracket.

2 Place the funnel into the bracket and attach the funnel tube to the Fill/Drain port. Remove the plug from the Vent port, leaving the port open

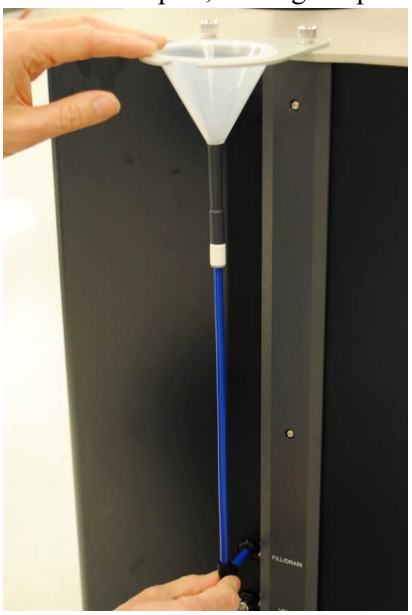
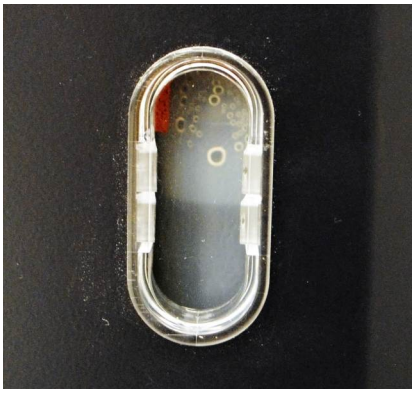


Figure 54 Funnel and funnel tube on the DLF.

NOTE: The Laser Coolant fluid is a mixture of 50%/50% (by volume) of distilled and deionized water/ laboratory-grade ethylene glycol.

3 Monitoring the fluid window, pour the fluid into the funnel. Stop filling the DLF when the fluid has reached the middle of the window (approximately 1.75 Liter).



4 Insert the plugs back into the Fill/Drain and Vent ports. Remove the funnel bracket.

Calibrating the Autosampler

Before beginning, make sure the Head is down and latched, the power to the DLF and EM are on, and the detector is attached to the unit.

1 In the software, click **Setup** and then select **Select Diagnostic Mode**.

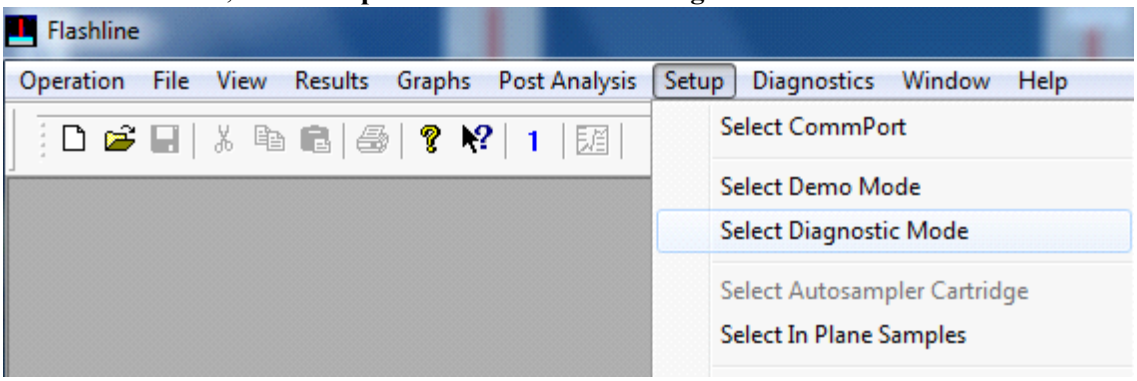


Figure 55 Setup > Select Diagnostic Mode.

2 A confirmation message displays, click **Yes**.

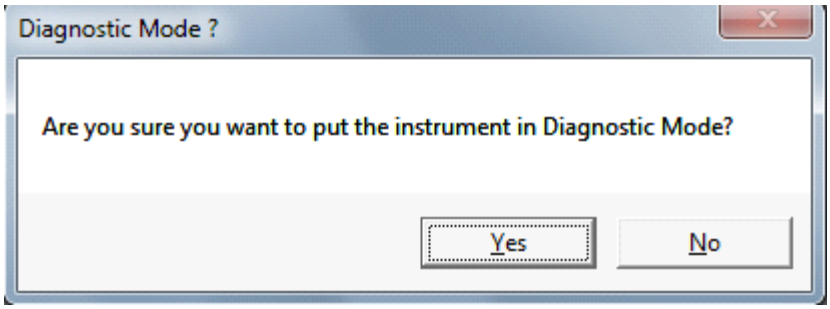


Figure 56 Diagnostic Mode confirmation.

3 Click **Diagnostics** and then select **Autosampler**.

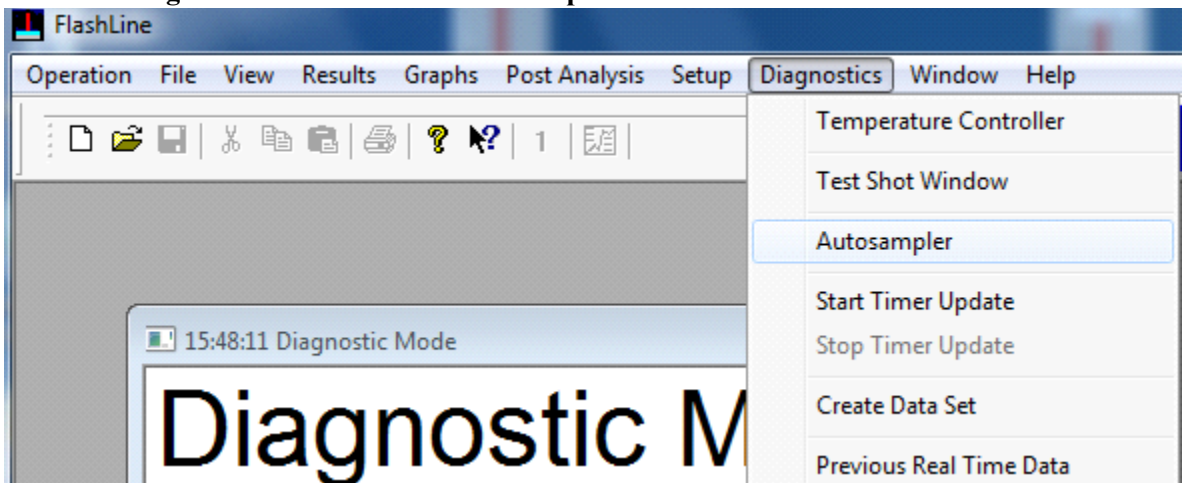


Figure 57 Diagnostics > Autosampler.

4 The Autosampler Control window displays. Click **System Enable**.

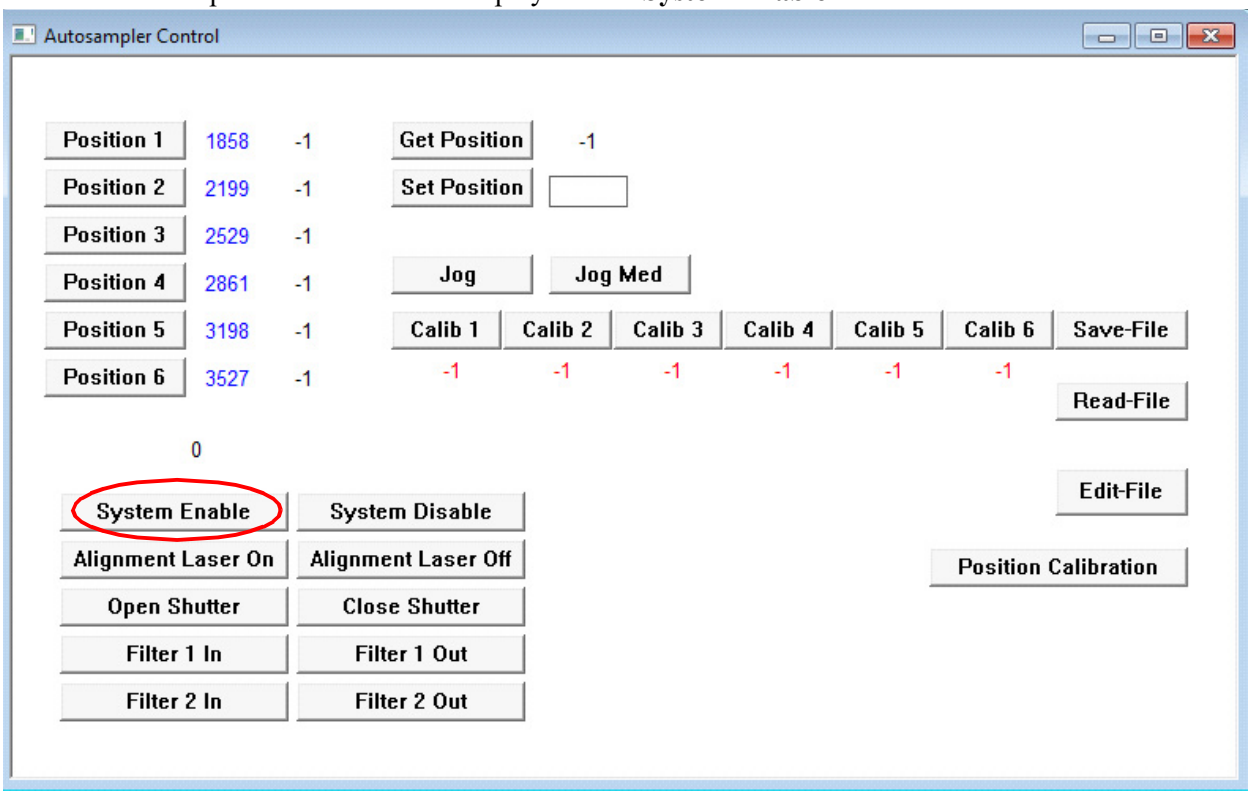


Figure 58 System Enable.

5 Once the below message displays, press **System Enable** on the front panel of the instrument.

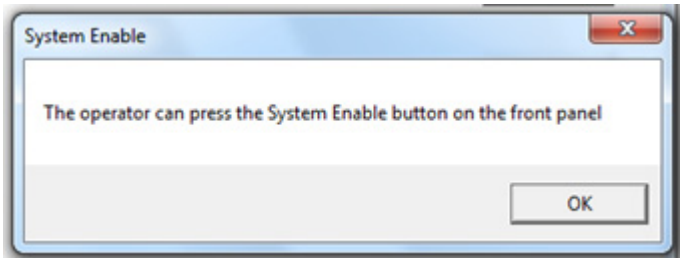


Figure 59 System Enable message.

6 Open the EM Head. Insert stiff paper or index card under the carousel to prevent items from falling into the furnace tube.

7 In the Autosampler Control window, click **Alignment Laser On**.

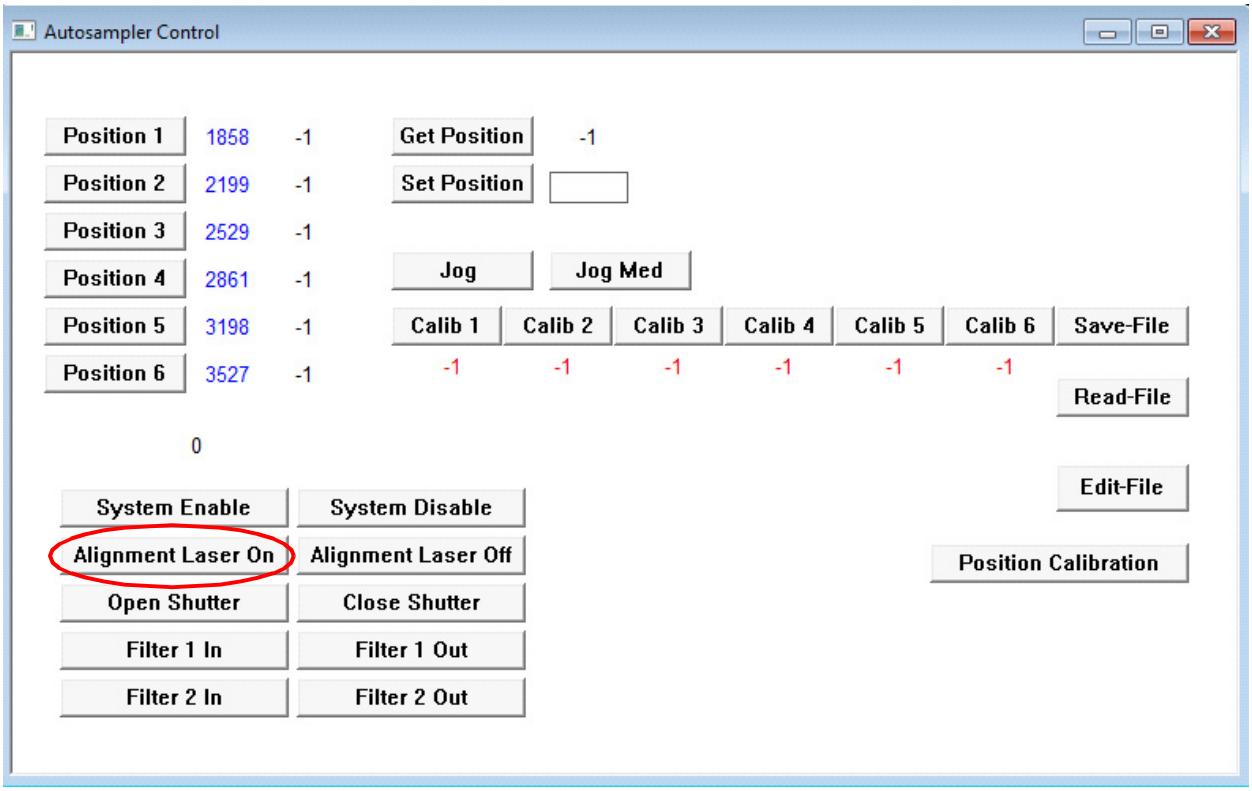


Figure 60 Alignment Laser On.

8 The red alignment laser turns on.

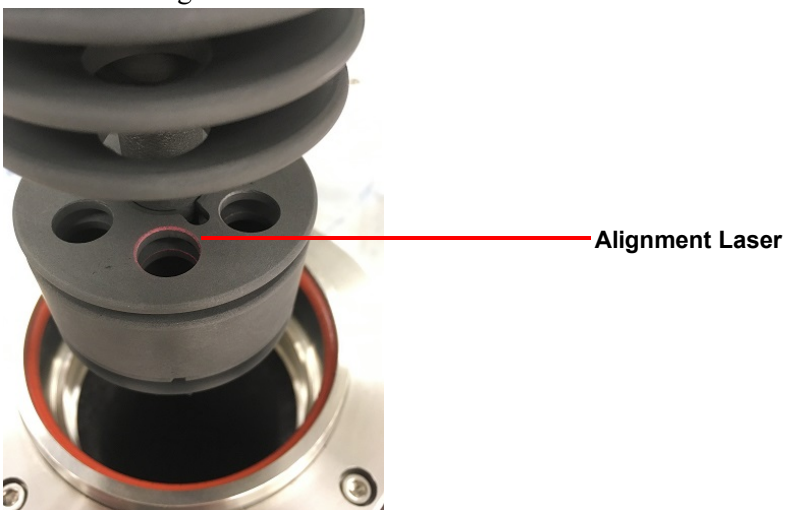


Figure 61 Alignment laser is on.

9 Click **Get Position**. The numbers that display to the right of the button indicate the current location of the Autosampler.

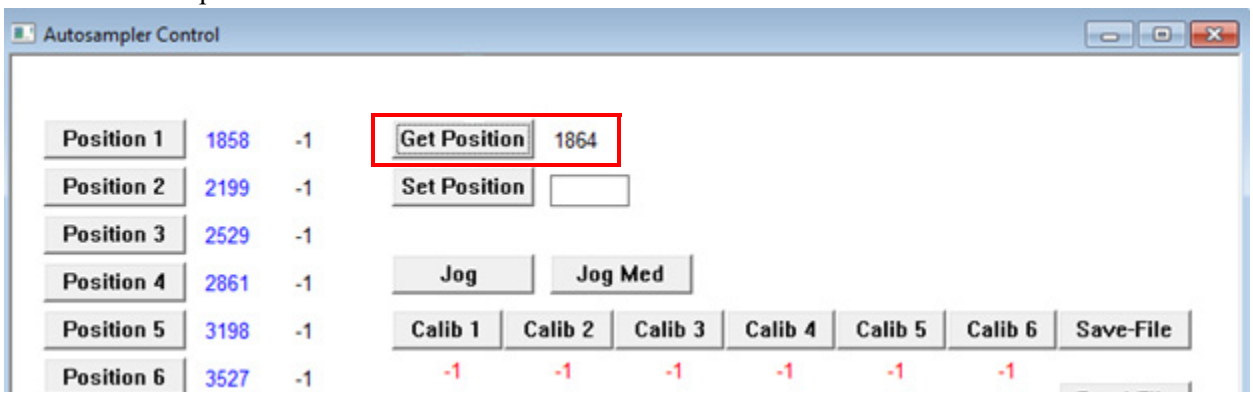


Figure 62 Get Position.

10 Set the first position. Move the Autosampler by clicking **Jog** or **Jog Med** until the laser is aligned with the first position on the carousel. When the laser is centered, click **Calib 1** to save.

NOTE: Use caution to not over rotate the carousel. Set the position with a number thant is lower than the target number, making sure that the carousel turns clockwise during the calibration. There is no way to move the Autosampler back.

11 A Calibration confirmation message displays. Click **Yes**.

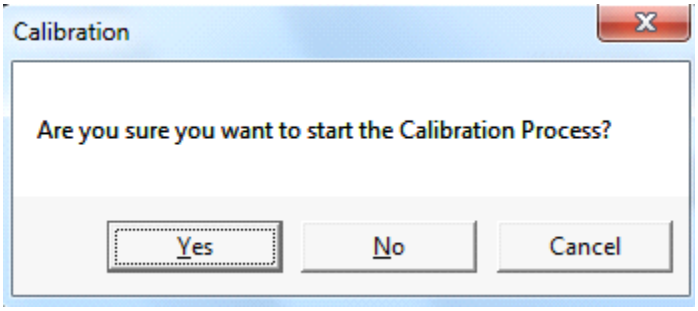


Figure 63 Calibration confirmation.

12 The position displays under the button. Move the Autosampler to position two using the **Jog** or **Jog Med** buttons. Once the laser is aligned, click **Calib 2**. Continue until all positions have been completed.

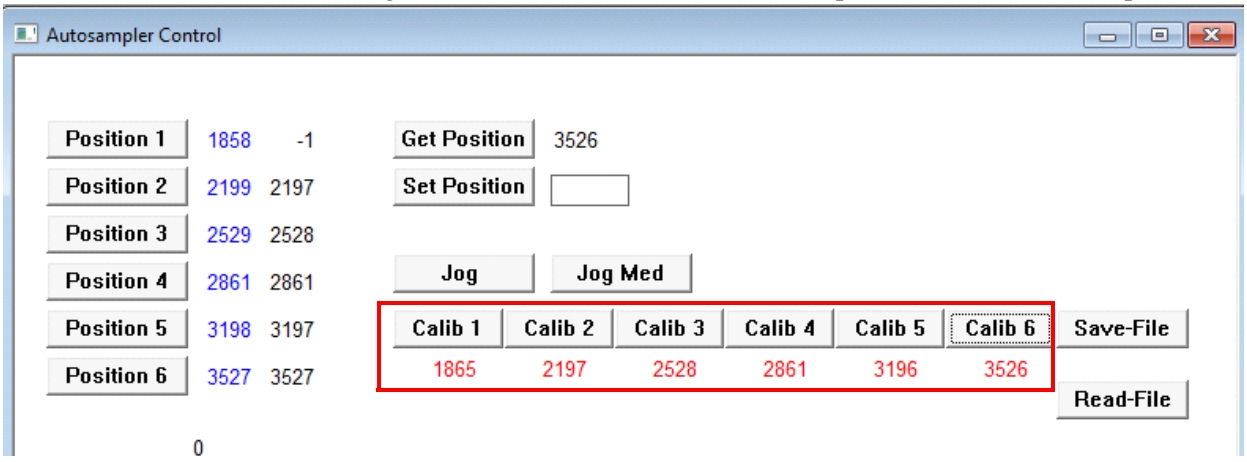


Figure 64 All positions completed.

13 Save the data to the position file:

a Click **Position Calibration**.

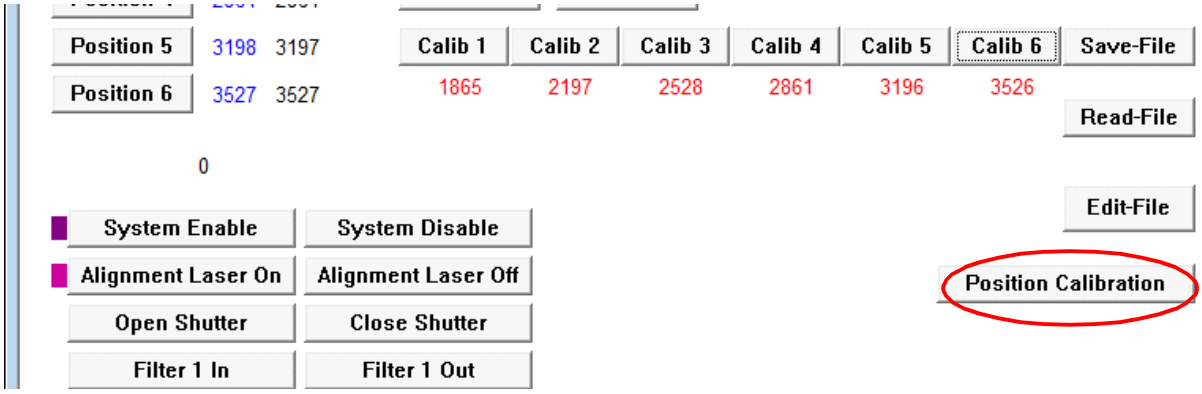


Figure 65 Position Calibration.

b The Autosampler Calibration Calculation window displays. Verify the position values. The calibration values can be modified, if necessary. Click **Save to File**.

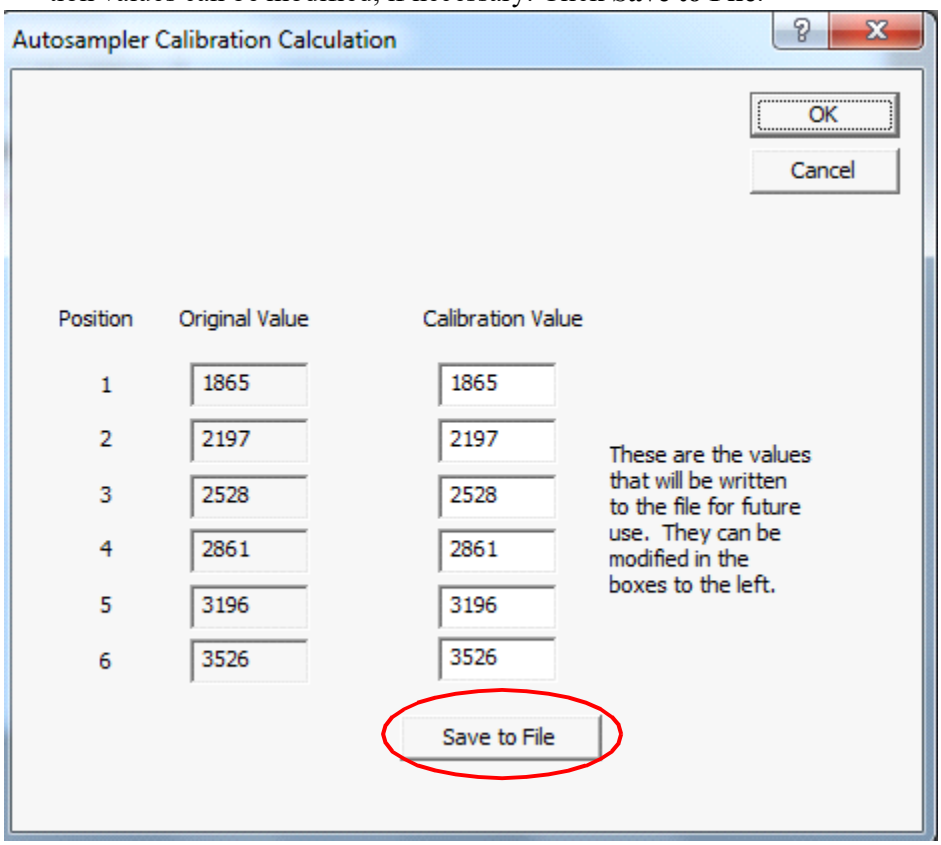


Figure 66 Save to file.

c A confirmation box displays. Click **Yes**.

d The Autosampler Control window displays. The new values can be found in blue next to the positions. The values in black are the previously used calibration values. Verify that the positions are correct.

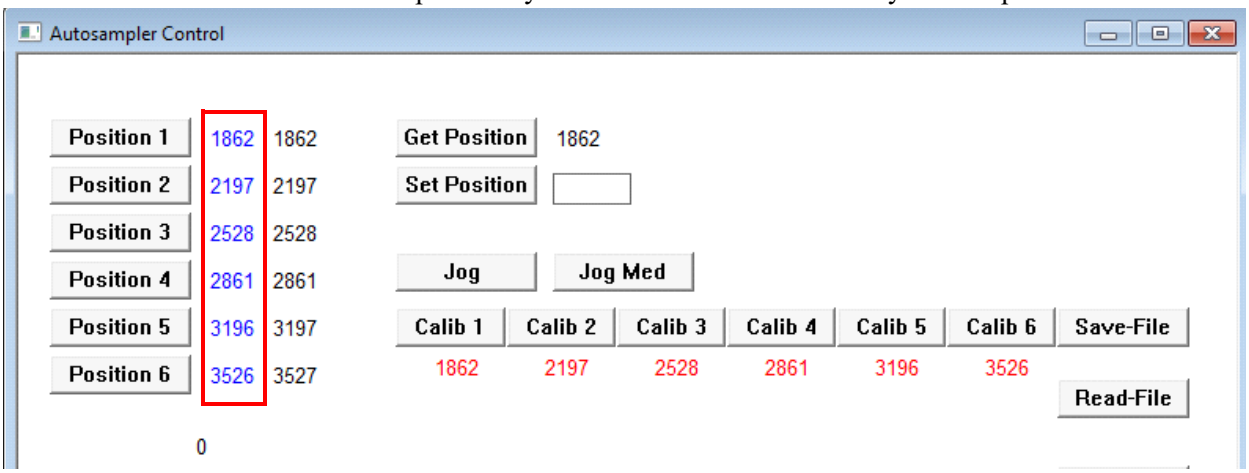


Figure 67 New values displayed.

Replacement Parts

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

Part Number	Description
201242.004	DLF and EM Fuse, T3.15A H 250VAC
202315.001	Moderate Vacuum Module: large capacity two-stage mechanical vacuum pump
202417.001	Graphite Paint
202419.001	Laser Coolant 1 Liter Bottle (2 bottles required for refill)
853135.901	Thermographite Reference, 12.7 mm OD, 3.175 mm thick
853143.901	Stainless Steel Reference, 12.7 mm OD x 1.58 mm thick
853145.901	Electrolytic Iron Reference, 12.7 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
853161.901	Silver Paint - Vial - 1 oz.
853170.901	Entry Window Laser
856098.901	Removable Lower Window