

Getting Started Guide

Revision A Issued January 2016

Notice

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

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

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

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

©2016 by TA Instruments — Waters LLC 159 Lukens Drive New Castle, DE 19720

Introduction

Important: TA Instruments Manual Supplement

Please click the <u>TA Manual Supplement</u> 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-2 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-2 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-2.
	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-2 system.
	Ce symbole indique que vous devez lire entièrement ce guide de démarrage pour obte- nir 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-2.

Please heed the warning labels and take the necessary precautions when dealing with these areas. This *Get*ting *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

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

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

DANGER: Risque de choc électrique. Présence de tensions élevées dans cet instrument. La maintenance et la réparation des pièces internes doivent être effectuées uniquement par TA Instruments ou tout autre personnel d'entretien qualifié.

WARNING: Protective earthing is provided through the mains power cord. Use of a grounded mains power outlet is required.

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

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 unplug the instrument before you examine or replace the fuses.

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

Liquid Nitrogen Safety

The Detector Assembly uses the cryogenic (low-temperature) agent, liquid nitrogen, for cooling. Because of its low temperature $(-196^{\circ}C [-321^{\circ}F)]$), 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-2 system to return to near room temperature before you touch the inner cell surfaces. Always use the available software monitor to display temperature even after the termination of the test cycle.

DANGER: Les surfaces de l'instrument 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 DLF-2 revenir à la température quasi ambiante avant de toucher les surfaces internes de la cellule. 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 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-2, 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 the chiller is setup properly and functioning after a power failure.

Chemical Safety

WARNING: Do not use hydrogen, oxygen, or any other explosive gas in the DLF-2 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 DLF-2. Seuls les gaz inertes comme l'azote ou l'argon doivent être utilisés.

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.

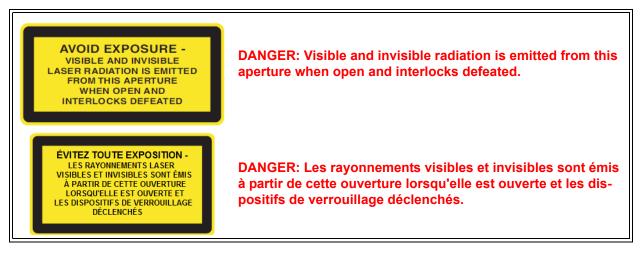
This product is Class I/1, with Class IV/4 and IIIA/3R embedded lasers.

LASER RADIATION AVOID DIRECT EYE EXPOSURE CLASS 3R LASER PRODUCT WAVELENGTH: 650 nm DURATION: CW POWER: <3 mW	DANGER: Avoid direct eye exposure to laser radiation.
RAYONNEMENT LASER ÉVITEZ L'EXPOSITION DIRECTE DES YEUX PRODUIT LASER DE CLASSE 3R LONGUEUR D'ONDE: 650 mm DURÉE: CW PUISSANCE: <3 Mw	DANGER: Évitez l'exposition directe des yeux au ray- onnement 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.



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

Introduction	
Important: TA Instruments Manual Supplement	
Notes, Cautions, and Warnings	
Regulatory Compliance	
Safety Standards Electromagnetic Compatibility Standards	
Safety	
Instrument Symbols	
Warnings	
Electrical Safety	
Liquid Nitrogen Safety	
Thermal Safety	7
Chemical Safety	
Laser Safety	8
Table of Contents	. 10
Chapter 1: Introducing the Discovery Laser Flash	. 12
Overview	. 12
DLE 2 Sustan Common outs	12
DLF-2 System Components Pulse Source Module	. 13
Environmental Modules	
EM-1600	15
Instrument Specifications	. 18
Chapter 2: Installing the System	. 20
Unpacking/Repacking	. 20
	20
Installing the System	
Inspecting the System	
Choosing a Location	
In On	
Near	
Away from	
Moving the Instrument.	
Connecting the Environmental Module to the Pulse Source Module	
DLF-2 Connections	
EM-1600 Connections	24
Connecting the Environmental Module to the Pulse Source	
Connecting the IR Detector Assembly to the Environmental Module	
Inserting the Removable Lower Window Assembly and Attaching the Detector	27
Connecting the Wand	28
Setting Up System Communication with the Controller	
Connecting the Utilities	
Setting Up the Vacuum System	
Attaching the Anti-Siphon Exit Port	33
Connecting Coolant and Gas Lines	34

Chapter 3: Operating the System	35
Using the DLF-2	35
Before You Begin	
Startup and Shutdown Procedures	
Starting the DLF-2 System.	
Shutting Down the DLF-2 System	36
Running a Discovery DLF-2 Experiment	37
Preparing the Sample	37
Sample Dimensions	. 37
Transparent or Translucent Materials	
High Reflectance Samples Loading and Unloading the Sample	
Loading Samples into the EM-1600	
Unloading Samples from the EM-1600	
Preparing the Instrument	41
Evacuating the System	
Starting an Experiment	44
Stopping an Experiment	45
Chapter 4: Maintaining the System	46
Cleaning the System	46
General Cleaning Practices	46
General Optics Cleaning Practices	
Cleaning the Removable Lower Window Assembly	
Cleaning the Carousel Control Mounting Plate Window	48
Replacing the DLF-2 Fuses	49
Replacing the EM-1600 Fuses	51
Changing the Laser Cooling Fluid in the DLF-2	52
Draining the Fluid	52
Filling the DLF-2	53
Calibrating the Autosampler	55
Replacement Parts	61

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 cm²/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-2) system automatically determines the thermal conductivity using the measured specific heat capacity and thermal diffusivity, with separately-entered density data.

The DLF-2 system is comprised of a DLF-2 Pulse Source, Environmental Module, and associated software. The DLF-2 with EM-1600 is shown below.



Figure 1 DLF-2 system with EM-1600.

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-2 system not found in this manual, see the online help associated with the instrument control software.

DLF-2 System Components

A DLF-2 system has two major components: the DLF-2 Pulse Source module and the Environmental Module.

Pulse Source Module

The Discovery Laser Flash (DLF-2) 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.



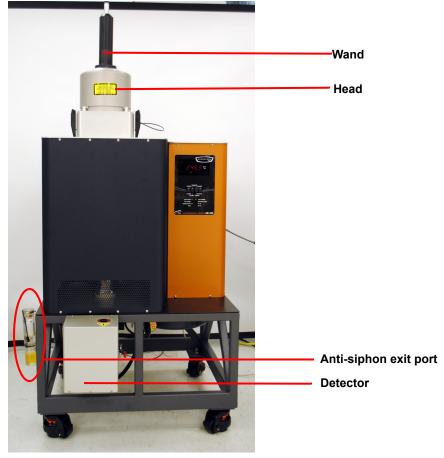
Figure 2 DLF-2 Pulse Source Module.

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 cool- ing 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 inter- locks in the EM are engaged and the system is ready for use. RED indicates that at least one of the EM inter- locks 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-2 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 Modules

EM-1600

The EM-1600 employs $MoSi_2$ heaters and a high-purity alumina muffle and sample holder, supporting continuous RT to 1600°C operation in air or inert gas purge and is vacuum tight to 50 mTorr. The EM-1600 includes a six-sample (nominally 12.7 mm diameter) holder.





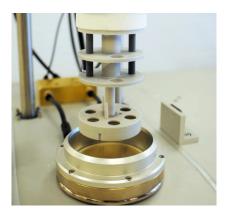


Figure 4EM-1600 six-sample holder.

Table 2: EM-1600 LED Indicators

Indicator	Location	Function
°C	Front panel	Sample 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 fur- nace 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.
Water 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. NOTE: Not used in the EM-1600.

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-2 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 and ready for use.
Gas Pressure	Back panel	ON indicates that the gas pressure is greater than 40 psi and the instrument pneumatics will function correctly.
Water 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 func- tioning correctly.

Instrument Specifications

The tables found below contain the technical specifications for the DLF-2 and EM-1600.

Item/Area	Specifications	
Dimensions DLF-2 (DxWxH) EM-1600 (DxW)	43 cm (17 in) x43 cm (17 in) x 91 cm (36 in) 53 cm (21 in) x 63 cm (25 in); H 114 cm (45 in) closed and 144 cm (57 in) open Complete system minimum lab space requirements (DxWxH): 81 cm (32 in) x116 cm (46 in) x 175 cm (69 in) open	
Weight DLF-2 EM-1600	71 kg (156 lbs) 124 kg (274 lbs)	
Power ^a	System supply voltage: 200–240 VAC (rated for 15 A) 50 or 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	
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	
Thermocouple Measurement	Type S	
Thermal Diffusivity	Accuracy ^b \pm 5%	
Measurement	Repeatability ^c $\pm 2\%$	
Temperature Range	RT to 1600°C	
Atmosphere	Air, inert, vacuum	
Number of Samples	6	
Sample Dimension	12.7 mm (d) x up to 6 mm (t)	
Sample Shape	Round disk	

Table 3: Technical Specifications

a. The DLF-2 is supplied with a power cord 2.4 m (7.75 ft) long. It uses a US-style NEMA 6-15P plug with an IEC 60320 C13 connector rated for 10A and 250 VAC.

The EM-1600 is supplied with a power cord 2.5 m (8.2 ft) long. It uses a US-style NEMA 6-15P plug with an IEC 60320 C19 connector rated for over 15A and 250 VAC.

The TA Representative installing the instrument will provide the appropriate power cable for the geographical location where the instrument is being installed. The Customer is responsible for providing the appropriately-rated plug. 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.

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 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 strongly recommended 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 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 prone to mechanical vibrations.

NOTE: Allow free air to circulate around both 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 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

Both the DLF-2 and EM-1600 can be wheeled to their location. The DLF-2 has standard locking brake wheels on the front two wheels, while the EM-1600 wheels can be locked into place. To secure the EM-1600, 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.

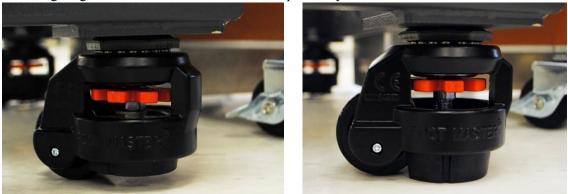


Figure 5 EM-1600 foot raised (left); EM-1600 foot lowered (right).

Connecting the Environmental Module to the Pulse Source Module

To connect the Environmental Module, you will need access to the DLF-2 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.

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-2 Connections

The table below provides a description of the function of each DLF-2 port. Refer to Figure 6 for an illustration of rear connections. Ports not described and not labeled are not used.

Table	4:	DLF-2	Ports
-------	----	-------	-------

Port	Location	Function
Main power	Back panel	Powers the DLF-2
DAQ	Back panel	Provides communication between the DLF-2 and the PC controller.
EM Interlock	Back panel	Connects the DLF-2 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 pres- sure during coolant drain.

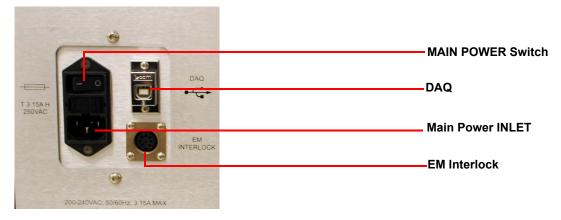


Figure 6 DLF-2 back panel connections.

EM-1600 Connections

The table below provides a description of the function of each EM-1600 port. Refer to <u>Figure 7</u>, <u>Figure 8</u>, and <u>Figure 9</u> for illustrations of rear connections.

Port	Location	Function
Interlock	Back–Data Panel	Connects the EM to the DLF-2 to ensure safe opera- tion of the laser.
Sample T/C	Back–Data Panel	Connects sample 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.
Auto Sampler	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.
Vacuum Gauge	Back–IEC Panel	Provides power and communication to the vacuum gauge.
Main Power	Back–IEC Panel	Powers the EM.
Coolant Inlet	Back–Fan Panel	Supplies coolant to the EM.
Coolant Outlet	Back–Fan Panel	Removes effluent coolant from the EM.
Gas Inlet	Back–Fan Panel	Provides inert gas to the EM.
Purge Gas Inlet, purge gas	Back–Head, Top plate manifold	Provides inert gas to the furnace.
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.
Purge Gas Outlet	Left bottom of EM	Removes gas from the EM.
Vacuum Gauge	Bottom center of EM	Provides power and communication to the vacuum gauge.

Table 5: EM-1600 Ports

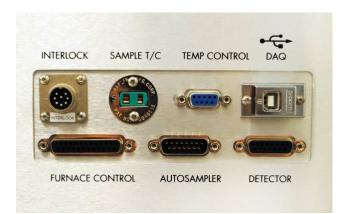


Figure 7 Environmental Module-1600 Data Panel connections.



Figure 8 Environmental Module-1600 IEC Panel connections.

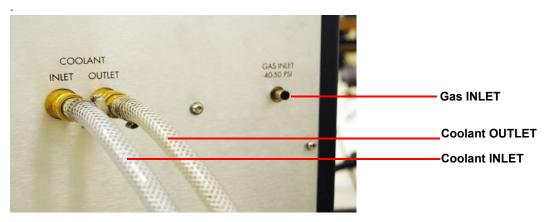


Figure 9 Environmental Module-1600 Fan Panel connections.

Connecting the Environmental Module to the Pulse Source

EM-1600

To connect the DLF-2 to the EM-1600, follow the instructions below. Refer to <u>Figure 6</u>, <u>Figure 7</u>, and <u>Figure 8</u> 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 laser interlock cable into Interlock port on the back of the EM and the other end into the port underneath the DLF-2.
- **3** 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.
- 4 Connect the power cable into the back of the DLF-2 (Data Panel) and then plug into a wall power outlet.

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. Insert the bottom optics assembly into the slot as shown in the figure below and then push the pneumatic actuation switch up.

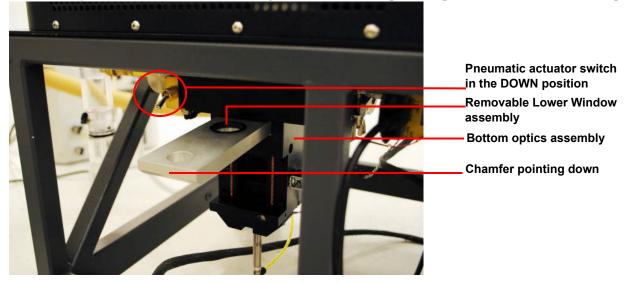
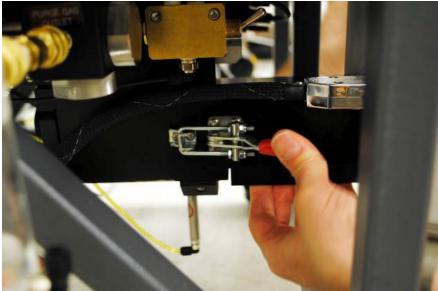


Figure 10 Bottom optics assembly.

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





3 Connect the cable to the Detector.

NOTE: The above steps can also be followed for removing the Removable Lower Window assembly for cleaning.

Connecting the Wand

Attach the wand to the EM-1600

- 4 Remove the protective cover from the wand opening on the EM-1600 and from the end of the wand.
- 5 Align the interlock magnet on the wand with the hole on the top plate

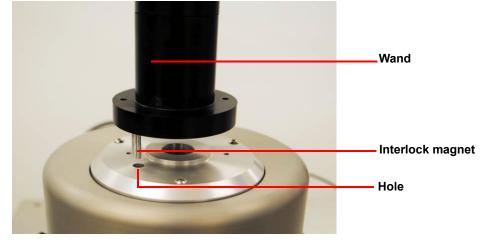


Figure 12 Secure the wand to the EM-1600.

6 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 13 Tighten EM-1600 wand screws.

Inserting the Carousel

- 1 Raise the head on the EM.
- 2 Carefully lower the carousel into the furnace tube, making sure not to bend the thermocouple tip. Raise the carousel stem over the thermocouple, into the baffle tube and into the collet.

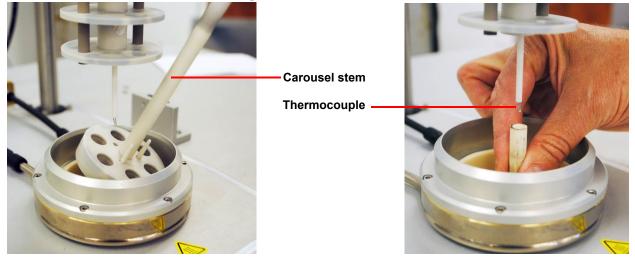


Figure 14 Insert the carousel (left); Raise the carousel stem over the thermocouple (right).

3 Insert stiff paper or index card under the carousel to prevent items from falling into the furnace tube.

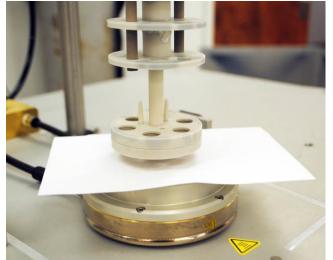


Figure 15 Carousel inserted.

4 Raise the carousel stem into the collet until the mark on the tube is aligned with the bottom edge of the collet, and tighten the collet by hand.

5 Using a wrench at the top to hold the collet shaft in place, use a second wrench to tighten the collet.

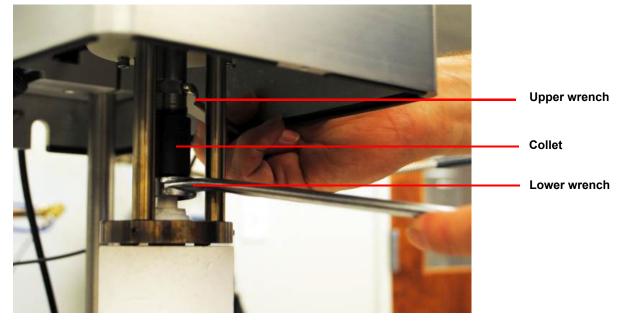


Figure 16 Tighten collet.

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 ports on the back of the EM and the DLF-2.
- 3 Connect an RS232 cable from the Temp Control port on the back of the EM to the controller.
- 4 Connect the monitor cable to the monitor port on the computer.
- 5 Connect the power cords to the computer and monitor and plug them into the same power source (common ground) that the EM and DLF-2 are plugged into.
- 6 Turn on the computer and monitor.

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

Connecting the Utilities

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.

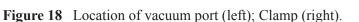
- 1 Place the vacuum pump on the floor below the unit.
- 2 Place the o-ring with centering ring between the QF-16 flange on the instrument and the QF-16 flange on the vacuum hose.



Figure 17 O-ring on hose

3 Clamp the flanges together with a QF-16 clamp.





- 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.

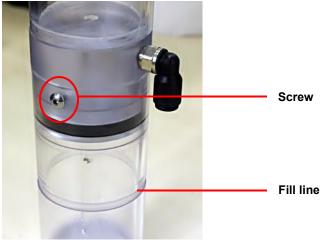


Figure 19 Anti-siphon exit port screw and fill line.

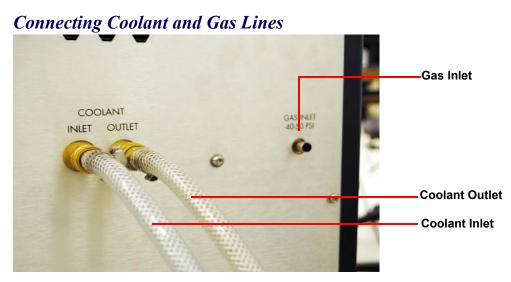
2 Close the port by putting the 2 screws back in place.

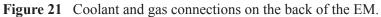
NOTE: Overly tightening the screws will cause the port's acrylic to crack.

3 Connect the port into the PURGE GAS OUTLET on the front of the DLF-2.



Figure 20 Anti-siphon exit port attached to the DLF-2.





Coolant

NOTE: Coolant circulator: It is recommended that you use TA Instruments ThermoCube Chiller (P/N 201786.001) or another circulator that meets the minimum requirements of 1 liter/minute fluid flow and has a cooling capacity of 300W at 25°C. The circulator temperature set point should be room temperature.

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 coolant source.

NOTE: 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.

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

Gas

- 4 Push the 1/8" supply tube onto the INERT GAS INPUT 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.
- 6 Set the gas supply pressure between 40–50 psi.
- 7 Check all air lines and connections for leakage.

Chapter 3:

Operating the System

Using the DLF-2

All of your DLF-2 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-2 system and the controller have been installed properly. Make sure you have:

- Made all necessary cable connections from the Environmental Module to the DLF-2
- 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 DLF-2 System

The power switches are located on the back panel of the DLF-2 and EM-1600. The power switches are used to turn the DLF-2 and EM-1600 system on and off.

To power on the system:

- 1 Check all connections between the DLF-2, the Environmental Module, the Detector Assembly, and the controller. Make sure each component is plugged into the correct connection port.
- 2 Set the DLF-2 and EM MAIN POWER switches to the ON (I) position.
- **3** The temperature controller and LED indicators on the front panels of the DLF-2 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-2 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 DLF-2, 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-2 and EM MAIN POWER switches to the OFF (0) position.

Running a Discovery DLF-2 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 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 $< 1 \mu m$ 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 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-1600

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 tube.
- 3 Raise the lid, rotate it slightly, and rest it on the pins, and then load the samples into the holder cavities, using the Sample Removal Tool underneath the holder cavity to move the sample into place without damaging the graphite coating. Positions are counted from the cavity bracketed by two notches (position #1) counterclockwise. 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.

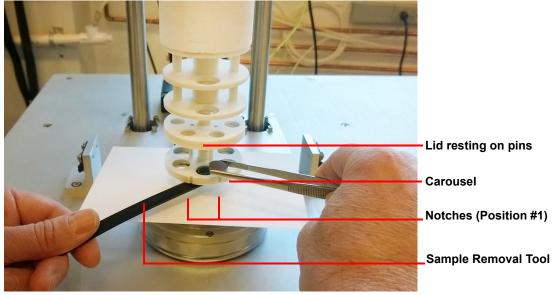


Figure 22 Sample holder.

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-1600

To unload samples from the EM-1600, follow steps 1 and 2 above for loading samples. Then use the sample removal tool to push the samples up out of the carousel cavity. Use tweezers to remove the sample.

Preparing the Instrument

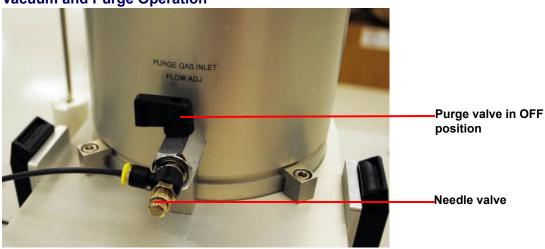
Evacuating the System

All DLF-2/EM-1600 systems are equipped with vacuum and 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 thermocouple gauge tube/vacuum meter assembly for determination of the vacuum level.



Vacuum and Purge Operation

Figure 23 Purge and needle valves.

- 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 valve.
- 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.

Institute Operation Startup Information Select Fumace Test Information Sample Information Sample Information Sample Information Sample Information Startup Information Sample Information Startup Information Start Fest Previous Test ID Information Start Test Paue Test Net Segment Autosampler Position 1 Vacuum Reading Skip Sample(s) Exit	Vacuum Gauge
Select Furnace Test Information Load Autosampler Position Temperature Program Previous Test ID Information Start Test Stop Test Pause Test Resume Test Next Segment Autosampler Position 1 Vacoum Reading Skip Sample(s)	
Previous Test ID Information Start Test Stop Test Pause Test Resume Test Nest Segment Autosampler Position 1 Vacuum Reading Skip Sample(s)	
Stop Test Pause Test Resume Test Nets Segment Autosampler Position 1 Vacuum Reading Skip Sample(s)	
Pause Test Resume Test Next Segment Autosampler Position 1 Vacuum Reading Skip Sample(s)	
Resume Test Nest Segment Autosampler Position 1 Vacuum Reading Skip Sample(s)	
Next Segment Autosampler Position 1 Vacuum Reading Skip Sample(s)	
Autosampler Position 1 Vacuum Reading Skip Sample(s)	
Vacuum Reading Skip Sample(s)	
Skip Sample(s)	
1 Vacuum Reading (Terr)	

Figure 24 Location of the vacuum gauge (top); View the vacuum reading, furnace temperature, and sample temperature in the software by clicking **Operation** > **Vacuum Reading** (bottom).

Back-fill with Purge Gas

- 1 Open the Purge Gas Inlet valve and enable flow to the EM chamber.
- **2** Turn off the vacuum pump.
- 3 Monitor the flow using the anti-siphon exit port assembly. Flow should be continuous, roughly 4–5 bubbles /second. Adjust with the built-in needle valve at the back of the EM head.

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.



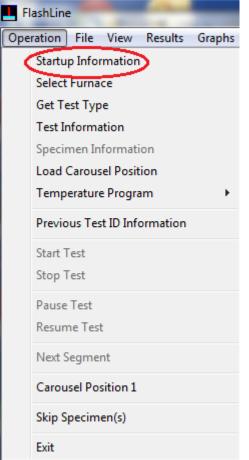


Figure 25 Operation > Startup Information.

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.

Test	Title									Information I Previous Test			Cancel
Test	ID	321			Sequence	Number							L
Oper	ator				Date		Jun 30,	2010					
C	ommom Spe	cimen ID	Common S	pecimen Tit	ile	Commo	n Thicknes:	s (cm)		Use Expa	ansion		
Posi	ition	Specime	en ID			Specir	nen Title			Thickness (cr	n) [)iameter (cm)	Weight (g)
1	1									0.0000	1	.270	0.0000
:	2									0.0000	1	.270	0.0000
:	3									0.0000	1	.270	0.0000
	4									0.0000	1	.270	0.0000
Ę	5									0.0000	1	.270	0.0000
(6									0.0000	1	.270	0.0000
	Test	Temperature	Ramp	Repeat	Power	Point 1	Point 2	Point 3	Point	4 Point 5	Poin	t6 ▲	
	Segment	(°)	(C°/Min.)	Points	Level	Level	Level	Level	Level	Level	Lev	/el	Jniform
	1		5.0	3	500.0								Temperature
	2		5.0	0	0.0								
	3		5.0	0	0.0								Ramp Rate
	4		5.0	0	0.0								Depart Daint
	5		5.0	0	0.0								Repeat Point
	6		5.0	0	0.0								Power Leve
	7		5.0	0	0.0							L	
	8		5.0	0	0.0							-	
	Clear Progra	m Add Segr	ment Delet	e Segment	Insert 9	iegment	List Progr	ams S	ave Proj	gram Safet		re (C) 2840	

Figure 26 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-1600 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.

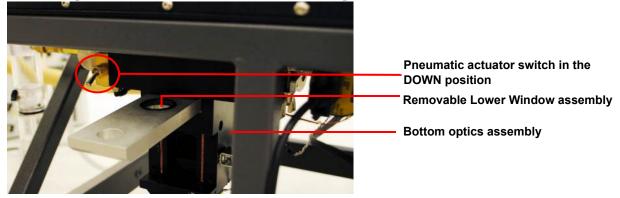
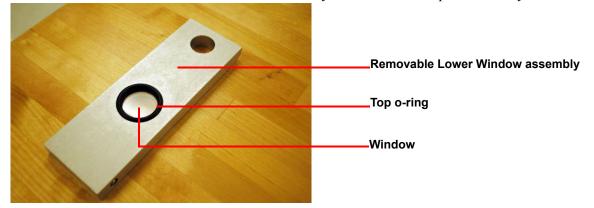
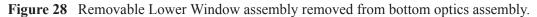


Figure 27

3 Remove the Removable Lower Window assembly from the 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 headcover clamps. Turn the clamps away from the head and then remove the head from the instrument.

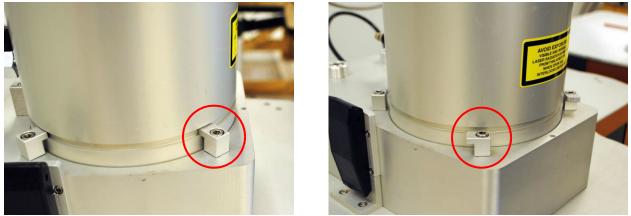


Figure 29 Headcover clamp securing the head (left); clamp turned away from the head (right).

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

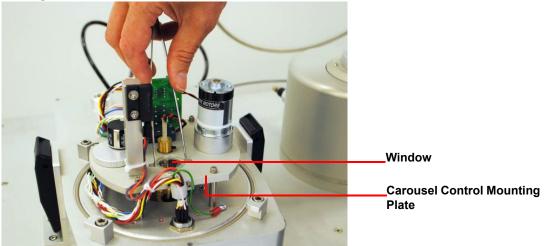


Figure 30 Remove window.

4 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 headcover clamps back into their original positions; tighten the screws.



Figure 31 Cutout aligned with stationary clamp.

Replacing the DLF-2 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-2 contains internal fuses that are not user serviceable. If any of the internal fuses blows, a hazard may exist. Call your TA Instruments service representative.

The only customer-replaceable fuses are the fuses located in the fuse holder on the back panel of the instrument. 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 32 Remove the fuse carrier.

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



Figure 33 Remove the fuses.

4 Place the fuse carrier back into the fuse holder.

Replacing the EM-1600 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 EM-1600 contains internal fuses that are not user serviceable. If any of the internal fuses blows, a hazard may exist. Call your TA Instruments service representative.

The only customer-replaceable fuses are the fuses located in the fuse holders on the back panel of the instrument. 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 (one fuse per holder). Use a flat-blade screwdriver to remove each of the fuse carriers.



Figure 34 Remove the fuse carriers.

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



Figure 35 Remove the fuses.

4 Place the fuse carriers back into the fuse holders.

Changing the Laser Cooling Fluid in the DLF-2

Draining the Fluid

1 Insert the drain tube into the Fill/Drain port on the side of the DLF-2.



Figure 36 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 37 Fill/Drain and Air Pump tubes connected.

4 Pump the Air Pump until the fluid has emptied from the DLF-2. Drain until no more liquid is coming out of the tube.



Figure 38 Draining the fluid.

Filling the DLF-2

1 Attach the funnel bracket onto the top of the DLF-2 by aligning the screws with the holes and tightening the screws by hand.

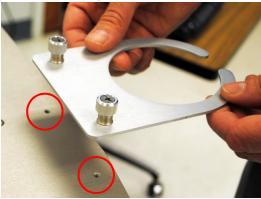


Figure 39 Attach the bracket.

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 40 Funnel and funnel tube on the DLF-2.

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-2 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-2 and EM are on, and the detector is attached to the unit.

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

Elashline	
Operation File View Results Graphs Post Analysis	Setup Diagnostics Window Help
	Select CommPort
1	Select Demo Mode
	Select Diagnostic Mode
	Select Autosampler Cartridge Select In Plane Samples

Figure 41 Setup > Select Diagnostic Mode.

2 A confirmation message displays, click Yes.

Diagnostic Mode	?
Are you sure yo	u want to put the instrument in Diagnostic Mode?
	Yes <u>N</u> o

Figure 42 Diagnostic Mode confirmation.

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

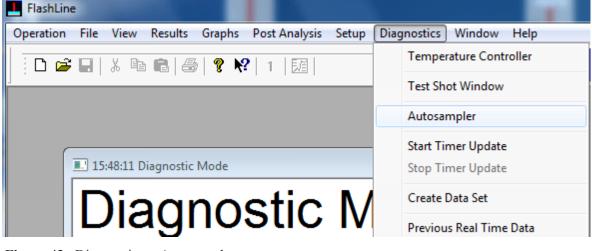


Figure 43 Diagnostics > Autosampler.

4	The Autosample	r Control	window	displays.	Click System Enable.
---	----------------	-----------	--------	-----------	----------------------

Position 1	1858	-1	Get Positio	n -1					
Position 2	2199	-1	Set Positio	n					
Position 3	2529	-1							
Position 4	2861	-1	Jog	Jog	Med				
Position 5	3198	-1	Calib 1	Calib 2	Calib 3	Calib 4	Calib 5	Calib 6	Save-File
Position 6	3527	-1	-1	-1	-1	-1	-1	-1	Read-File
	•								
System	0 Enable	Sy	stem Disable						Edit-File
Alignment	Laser On	Aligr	nment Laser Off					Position	Calibration
Open S	hutter	C	lose Shutter				_		
Filter	1 In		Filter 1 Out						
Filter	2 In		Filter 2 Out	1					

Figure 44 System Enable.

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

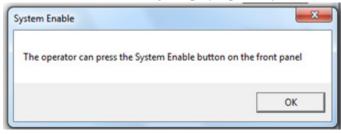


Figure 45 System Enable message.

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

- I Autosampler Control Position 1 1858 -1 Get Position -1 Position 2 2199 -1 Set Position Position 3 2529 -1 Jog Med Jog Position 4 2861 -1 Position 5 3198 Calib 1 Calib 2 Calib 3 Calib 4 Calib 5 Calib 6 Save-File -1 -1 -1 -1 -1 -1 -1 Position 6 3527 -1 **Read-File** 0 Edit-File System Enable System Disable Alignment Laser On Alignment Laser Off **Position Calibration** Open Shutter **Close Shutter** Filter 1 In Filter 1 Out Filter 2 In Filter 2 Out
- 7 In the Autosampler Control window, click Alignment Laser On.

Figure 46 Alignment Laser On.

8 The red alignment laser turns on.

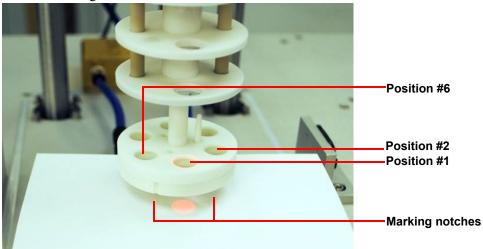


Figure 47 Alignment laser is on.

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

Position 1	1858	-1	Get Positi	on 1864	1				
Position 2	2199	-1	Set Positi	on					
Position 3	2529	-1							
Position 4	2861	-1	Jog	Jog	Med				
	3198	-1	Calib 1	Calib 2	Calib 3	Calib 4	Calib 5	Calib 6	Save-File
Position 5	3190								

Figure 48 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.

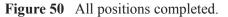
11 A Calibration confirmation message displays. Click Yes.

Calibration		×
Are you sure you want to	start the Calibrati	on Process?
Yes	<u>N</u> o	Cancel

Figure 49 Calibration confirmation.

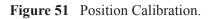
12 The position displays under the button. Move the Autosampler to position two using the **Jog** or **Jog Med** buttons. Once the laser is aligned, click **Calib 2**. Continue until all positions have been completed.

Position 1	1858	-1	Get	Positio	n 3526					
Position 2	1	2197	_	Positio						
Position 3	2529	2528								
Position 4	2861	2861		Jog	Jog	Med				
Position 5	3198	3197	Ca	lib 1	Calib 2	Calib 3	Calib 4	Calib 5	Calib 6	Save-File
Position 6	3527	3527		1865	2197	2528	2861	3196	3526	
	3								-	Read-File



13 Save the data to the position file:

bration.							
3197	Calib 1	Calib 2	Calib 3	Calib 4	Calib 5	Calib 6	Save-File
3527	1865	2197	2528	2861	3196	3526	Read-File
		F				Position C	Edit-File
					1		
	Alignme Clos	3197 Calib 1 3527 1865 System Disable	3197 Calib 1 Calib 2 3527 1865 2197 System Disable Alignment Laser Off Close Shutter	System Disable Alignment Laser Off	System Disable Alignment Laser Off	System Disable Alignment Laser Off Close Shutter	3197 Calib 1 Calib 2 Calib 3 Calib 4 Calib 5 Calib 6 3527 1865 2197 2528 2861 3196 3526 System Disable Alignment Laser Off Position C Position C



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

Autosampler	Calibration Calculatio	n	? ×
			OK Cancel
Position	Original Value	Calibration Value	
1	1865	1865	
2	2197	2197	These are the values
3	2528	2528	that will be written to the file for future
4	2861	2861	use. They can be modified in the
5	3196	3196	boxes to the left.
6	3526	3526	
		Save to File	D

Figure 52 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.

Autosampler Con	trol								
		_							
Position 1	1862	1862	Get Positi	ion 1862					
Position 2	2197	2197	Set Positi	on					
Position 3	2528	2528							
Position 4	2861	2861	Jog	Jog	Med				
Position 5	3196	3197	Calib 1	Calib 2	Calib 3	Calib 4	Calib 5	Calib 6	Save-File
Position 6	3526	3527	1862	2197	2528	2861	3196	3526	
	L								Read-File
	0								

Figure 53 New values displayed.

Replacement Parts

Part Number	Description
201242.001	EM-1600 Fuse, T12.0A H 250VAC
201242.004	DLF-2 Fuse, T3.15A H 250VAC
201786.001	ThermoCube Chiller
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
856654.001	Sample Removal Tool

Replacement parts for the DLF-2 are listed below. Refer to the table below when ordering parts.