DISCOVERY LASER FLASH DLF-1





Getting Started Guide

Revision L Issued March 2025

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 <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-1 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.

Supplier's Declaration of Conformity

47 CFR § 2.1077 Compliance Information

Unique Identifier: 853001.901

Responsible Party:

TA Instruments 159 Lukens Drive New Castle, DE 19720 302-427-4000 www.tainstruments.com

FCC Compliance Statement:

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

Declaration of Conformity

E AMA E AMA E AMA E AMA

The undersigned, representing: TA Instruments – Waters LLC 159 Lukens Drive New Castle, Delaware USA

Herewith declare the product: Discovery Laser Flash DLF-1/EM-1200 P/N 853001.90x (x=1,2)

Is in conformity with the provisions of the following directives:

2014/30/EU	Electromagnetic compatibility
2014/35/EU	Electrical equipment for use within specified voltage limits
2015/863/EU	Restriction of Hazardous Substances in EEE

And conforms to the following standards:

EMC	EN 61326-1: 2006
Safety	EN 61010-1: 2010
	EN 61010-2-010: 2003
	IEC 60825-1: 2001
Quality	ISO 9001: 2015

I hereby declare that the equipment named above has been designated to comply with the relevant sections of the above-referenced specifications. This unit complies with all applicable Essential Requirements of the Directives.

Date of Validity: October 6, 2022

Name of Authorized Signatory: Position held in company: Location: Joyce Yoon Senior Director of Manufacturing New Castle, DE

Signature:

920332.026 Rev. F

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CE

Safety

Instrument Symbols

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

Symbol	Explanation
	This symbol indicates that a hand crush hazard is present. Take care when placing the top module onto the bottom module.
	Ce symbole indique la présence d'un risque de broyage de main. Prenez soin de placer le module supérieur sur le module inférieur.
	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.

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

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.

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: Source instrument surfaces can be hot enough to cause discomfort when in contact with the skin during a sample run. If you are conducting a subambient test, cold could also cause injury. After running any type of experiment, you must allow the DLF-1 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 source peuvent être assez chaudes pour provoquer un malaise au contact de la peau pendant l'analyse d'un échantillon. Si vous effectuez un essai à basse température, le froid peut également provoquer des blessures. Après avoir effectué un type d'expérience quelconque, vous devez laisser le système DLF-1 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 press any buttons on the front panel after a power failure, just start a test normally. If the test was running under vacuum, verify the vacuum. 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-1 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-1. Seuls les gaz inertes comme l'azote ou l'argon doivent être utilisés.

Laser Safety

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





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.



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 comply 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.

Lifting the Instrument

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

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

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Chapter 1:

Introducing the Discovery 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-1) system automatically determines thermal conductivity using the measured (or separately entered) heat capacity and thermal diffusivity, with separately-entered density data.

The DLF-1 system is comprised of a DLF-1 Pulse Source, Environmental Module, IR Detector Assembly, and associated software. The DLF-1 with EM-1200 is shown below.



Figure 1 DLF-1 system with EM-1200 and IR Detector Assembly.

Your controller is a computer that performs the following functions:

- Provides an interface between you and the analysis instrument.
- Enables you to set up experiments and enter necessary information.
- Stores experimental data.
- Allows data analysis of results.

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

DLF-1 System Components

A DLF-1 system has three major components: DLF-1 Embedded Laser Pulse Source, the Environmental Module with Detector Assembly, and the controller.

Pulse Source Module

The Discovery Laser Flash (DLF-1) Source Module uses a custom Class I/1 Nd:glass Laser Pulse Source to provide collimated, monochromatic energy pulse with a 300 μ s to 400 μ s pulse width. It has full-time pulse mapping and recording capability that allows precise pulse shape and pulse width correction calculations, as well as determination of the t₀. The DLF-1 is ideal for measuring thermal conductivity, thermal diffusivity or heat capacity of samples at temperatures up to 1200°C, or for users who require the monochromatic pulse inherent with an embedded laser source in a compact bench-top footprint.



Figure 2 DLF-1 Pulse Source Module.

Indicator	Function
FURNACE INTERLOCK	GREEN indicates that the EM is sitting correctly on top of the DLF-1. RED indicates that EM is sitting incorrectly and must be adjusted.
SAMPLE/DETECTOR INTERLOCK	GREEN indicates that the Autosampler is closed and ready for use, and that the detector is secured correctly to the EM. RED indicates that the Autosampler is open or the detector is not secured.
HIGH VOLTAGE MODULE	GREEN indicates that the high voltage module is closed and ready for use. RED indicates that the module is open.
LASER MODULE	GREEN indicates that the embedded laser module is closed and ready for use. RED indicates that the module is open.
SOURCE POWER	ON indicates that the embedded laser control board is powered.
CHARGE/ARM	ON / Blinking indicates that the lamp is charging.
FIRE	ON indicates that the fire command was executed.
DUMP	ON indicates that the embedded laser system is in dump mode (not charging and not firing).
FURNACE °C	Indicates the current temperature of the furnace.
SAMPLE °C	Indicates the current temperature of the sample.
OVER TEMP	 ON indicates one of the following: The EM has gone over its temperature limit One of the thermocouples is broken or not connected As a result, the furnace is disabled and will not heat.
COOLANT	 ON indicates that the coolant system is ready. If the LED indicator is not lit, one of the following may have occurred: Coolant line pressure is under 2PSI Coolant system is not connected As a result, the furnace is disabled and will not heat.
POWER	ON indicates the DLF-1 system is powered.
FILTER 1	ON indicates Filter1 is in and ready for use.
SHUTTER	ON indicates that the shutter is open and ready for use.
FILTER 2	ON indicates Filter2 is in and ready for use.
LOW GAIN	ON indicates the detector amplifier has low gain. values.

 Table 1:
 DLF-1 Front Panel LED Indicators

Indicator	Function	
ALIGNMENT	ON indicates that the embedded laser diode alignment tool is turned on and is ready for use.	
SYSTEM ENABLE switch	ON indicates that the system is ready to execute all acquisition functions.	
FURNACE ENABLE switch	ON indicates that the furnace is available and ready to heat/ control the temperature parameters.	
FURNACE DISABLE switch	 ON indicates one of the following: The furnace is unable to heat Heat to the furnace was stopped Any of the conditions for enabling the furnace were violated 	
SAMPLE MOTION switch	ON indicates that the Autosampler is in motion. OFF indicates that the Autosampler system is ready and the samples are in a steady state.	

Table 1: DLF-1 Front Panel LED Indicators

Environmental Modules and IR Detector Assembly

EM-1200

The EM-1200 includes a resistance-heated furnace, providing RT to 1200°C operation in air or inert gas, or RT to 1100°C in vacuum to $50x10^{-3}$ torr. The EM-1200 includes a multi-sample indexed holder (Autosampler), supporting a variety of sample sizes. Adapters are available for smaller cylindrical and/or square samples. The module includes an LN₂-cooled IR Detector.



Figure 3 EM-1200 and IR Detector Assembly.

Instrument Specifications

The tables found below contain the technical specifications for the DLF-1 and EM-1200.

Item/Area	Specifications
Dimensions	Depth 89 cm (35 in) Width 115 cm (45 in) Height 76 cm (30 in)
Weight	92 kg (175 lbs)
Power ^a	System supply voltage: 198–242 VAC (rated for 6A) 50/60 Hz
Operating environmental conditions	Temperature: 15 to 35°C Relative humidity: 5 to 80% (non-condensing) Installation Category II Pollution Degree 2 Maximum altitude: 2000 m The degree of protection for this instrument according to EN 60529 is IP20.
Pulse Source	High speed, embedded class IV/4 laser module

Table 2: DLF-1 Technical Specifications

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

Table 3: EM-1200 Technical Specifications

Item/Area	Specifications
Thermocouple Measurement	Type S
Thermal Diffusivity	Accuracy** $\pm 4\%$ (worst case)
Measurement	Repeatability** $\pm 2\%$ (worst case)
Temperature Range	RT to 1200°C (air or inert gas); RT to 1100°C (vacuum)
Atmosphere	Air, inert gas, vacuum
Number of Specimens	4/2
Sample Dimension	12.7/25.4 mm (d) x up to 6 mm (t)
Sample Shape	Round disk, square plate (with adapters)
IR Detector Assembly	LN ₂ InSb cooled detector

**Using ideal, well-defined, well-behaved 12-mm diameter samples of known properties

Chapter 2:

Installing the DLF-1 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 DLF-1 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
- Connecting the utilities

It is strongly recommended that you have your DLF-1 system installed by a TA Instruments Service Representative; call for an installation appointment when you receive your instrument.

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

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

Inspecting the System

When you receive your DLF-1 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 DLF-1 experiments, it is important to choose a location for the instrument using the following guidelines. The DLF-1 system should be:

In

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

On

• A stable, non-flammable work surface.

Near

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

Away from

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

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

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

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

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

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

Moving the Instrument

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

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

Lifting the instrument should always be done with two people. With each person standing on either side of the EM, place hands on the outside bottom corners of the EM and lift upwards, keeping the module in an upright position. Use the same procedure to move the DLF-1.



of the EM



Figure 4 Lifting the EM (top) and lifting the DLF-1 (bottom).

Connecting the Environmental Module to the Pulse Source Module

Before making any connections, do the following:

• If using a DLF-1 with EM-1200 system and testing large diameter (25.4 mm) samples, insert the expander assembly into the opening of the DLF-1 (between the EM-1200 and the DLF-1 platform). The expander assembly is not needed for 1/2 in. (12.7 mm) samples.



Figure 5 Inserting the expander assembly.

• Place the Environmental Module on top of the Pulse Source so that they are stacked; make sure to align the 4 pins on the bottom of the EM with the corresponding holes on the top of the Pulse Source. Allow an additional 28 cm (11 in) clearance to the right of the instrument for opening the Autosampler on the EM.



Figure 6 Pins on the bottom of the Environmental Module (EM-1200 shown).

To connect the Environmental Module, you will need access to the DLF-1 instrument back panel and the Environmental Module back panel.

NOTE: Connect all cables before connecting the power cords to outlets. Tighten the screws on the 24 V/CAN/interlock cable.

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-1 Back Panel

The DLF-1 back panel has ten ports; the table below provides a description of the function of each port. Refer to Figure 7 for an illustration of rear connections. Ports not described and not labeled are not used.

Port	Function
EM CONTROL Port	Provides communication between the DLF-1 and the EM module
AUTOSAMPLER Port	Provides communications between the autosampler electron- ics and DLF-1
DETECTOR Port	Provides communication between the DLF-1 and the Detec- tor Assembly
DAQ Port	Provides communication between Data Acquisition and the controller
T/C Ports	Connects the sample and furnace thermocouples in the EM to the DLF-1
FLASH CONTROL Port	Provides communication between the laser control board and the controller
TEMP CONTROL Port	Provides communication between the unit Temperature Con- trol and the controller
EM POWER Port	Provides power to the EM
SYSTEM POWER Switch	Provides power to the DLF-1/EM – off (0) / on (I) – and works as a built-in circuit breaker for safety
SYSTEM POWER Port	Powers the DLF-1

Table 4:DLF-1 Back Panel



Figure 7 DLF-1 rear panel connections.

EM-1200 Back Panel

The EM-1200 back panel has five ports; the table below provides a description of the function of each port.Refer to <u>Figure 8</u> for an illustration of rear connections.

Table 5:	EM-1200	Back Panel
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Port	Function	
COOLANT INLET	Supplies coolant to the EM	
COOLANT OUTLET	Removes effluent coolant from the EM	
Filter and Shutter Pneumatics (already attached)	Opens and closes the shutter and filter	
INTERLOCK LED Indicators	 7 green LED indicators surrounding the EM CONTROL Port indicate system readiness. From left to right: (1) EM Power - ON indicates that the EM module is connected 	
	• (2) Autosampler - ON indicates that the Autosampler is closed	
	• (3) Detector - ON indicates that the detector is in the correct position on top of the EM module	
	• (4) Actuators Gas - ON indicates that the pressure of the filters and shutter is greater than 40 PSI	
	• (5) Gas Inlet - ON indicates that the gas purge inlet is greater than 40 PSI and instrument will function correctly	
	• (6) Coolant - ON indicates that the pressure of the outlet cooling line is greater than 2 PSI and the furnace will function correctly	
	• (7) Thermal Switch - ON indicates that the cast temper- ature of the EM module is less than 63°C and the furnace will function correctly	
GAS INLET	Provides inert gas to the EM	
EM POWER Cable	Powers the EM	
EM CONTROL Port	Provides communication between the DLF-1 and the EM	
VACUUM GAUGE Port	Provides power to the vacuum gauge and communication to the controller	
Head Cooling Hose Ports (already attached)	Coolant conduit to keep the heat from the EM from damaging the electronics in the head	



COOLANT INLET COOLANT OUTLET EM CONTROL Port INTERLOCK LED Indicators Filter and Shutter Pneumatics GAS INLET EM POWER Cable Filter and Shutter Pneumatics LED Indicators VACUUM GAUGE Port

Head Cooling Hose Ports

Figure 8 Environmental Module-1200 rear panel connections.

Connecting the Environmental Module to the Pulse Source

To connect the DLF-1 to the EM-1200, follow the instructions below. Refer to Figure 7 and Figure 8 for connection ports, or the diagram located in the <u>App endix</u>.

- 1 Connect the EM CONTROL cable from the back of the DLF-1 to the back of the EM.
- 2 Connect the AUTOSAMPLER cable from the back of the head plate on the EM to the back of the DLF-1.
- 3 Connect the **EM POWER** cable from the back of the EM to the back of the DLF-1. Make sure to align the pins in the head of the power cable with the pins in the DLF-1 power cable port.
- 4 Insert the **FURNACE** thermocouple cable into the right T/C port on the back of the DLF-1, and then connect the other end of the cable to the right side of the EM (behind the vacuum gauge).



Figure 9 Furnace thermocouple connection on the EM.

5 Connect the **SAMPLE** thermocouple cable from the back of the Autosampler on the EM to the left T/ C port on the back of the DLF-1.

6 Connect the power cable into the back of the DLF-1 into a power strip and then plug into a wall power outlet.

Connecting the IR Detector Assembly to the Environmental Module

Inserting the Vacuum Window Assembly

- 1 Remove the 3 screws holding the Detector mounting block to the EM.
- 2 Remove the block to expose the optical conduit for the vacuum window assembly.



Figure 10 IR Detector Assembly mounting block removed to expose optical conduit.

3 Lower the vacuum window assembly into the conduit.

NOTE: The above steps can also be followed for removing the vacuum window assembly.

Connecting the Detector Assembly

- 4 Replace the Detector mounting block.
- 5 Re-install the 3 screws.
- 6 Align the (2) cylindrical magnets on the bottom of the IR Detector Assembly with the corresponding holes on the top of the Detector Assembly mounting block.



Figure 11 IR Detector Assembly and IR Detector Assembly mounting block.

7 Secure the latches on either side of the IR Detector Assembly by hooking the latch at the bottom and then pressing the top.





8 Plug in the detector assembly cable from the Detector to the DLF-1.

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 a USB cable from the DAQ Port on the back of the DLF-1 to the controller.
- 3 Connect a USB cable from the FLASH CONTROL port on the back of the DLF-1 to the controller.
- 4 Connect an RS232 cable from the **TEMP CONTROL** port on the back of the DLF-1 to the controller.
- 5 Connect the monitor cable to the monitor port on the computer.
- 6 Connect the power cords to the computer and monitor and plug them into the same power strip that the DLF-1 is plugged into.
- 7 Turn on the computer and monitor.

Connecting the Utilities

Connecting the Power

Plug the power cord from the **SYSTEM POWER** on the back of the DLF-1 to an electrical outlet rated in accordance with the power input information on the back of the instrument.

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

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

Setting Up the Vacuum System

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

- 1 Place the vacuum pump on the floor below the left side of the unit.
- 2 Place the o-ring with centering ring between the QF-16 flange on the instrument and the QF-16 flange on the vacuum 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.
- 5 Connect the anti-siphon exit port to the quick-connect fitting.
- 6 Connect the vacuum gauge cable from the VACUUM GAUGE port on the back of the EM to the vacuum.

Connecting Coolant and Gas Lines

Coolant

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 Screw the Quick Connect Cap onto the **GAS INLET** fitting on the back of the EM. Connect the other end of the hose to the inert gas supply.



Figure 14 Inert gas connection.

- 5 Close the **PURGE GAS FLOW** needle valve.
- 6 Open the inert gas supply unit.
- 7 Check all air lines and connections for leakage.

Chapter 3:

Operating the DLF-1

Using the DLF-1

All of your DLF-1 experiments will have the following general outline. In some cases, not all of these steps will be performed. The majority of these steps are performed using the instrument control software. The instructions needed to perform these actions can be found in the online help in the instrument control software; therefore, they will not all be covered in detail here.

- Preparing the sample
- Selecting the appropriate sample adapter and holder
- 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-1 system and the controller have been installed properly. Make sure you have:

- Made all necessary cable connections from the Environmental Module to the DLF-1
- 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-1 System

The power switch is located on the back panel of the DLF-1. The power switch is used to turn the DLF-1 system on and off.

To power on the system:

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

Shutting Down the DLF-1 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-1 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, set the DLF-1 SYSTEM POWER switch to the OFF (0) position.

Running a Discovery DLF-1 Experiment

Preparing the Sample

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

Sample Dimensions

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

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

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

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

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

Transparent or Translucent Materials

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

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

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

Please note that:

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

High Reflectance Samples

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

For specific heat capacity testing it is imperative that both the unknown sample and the reference have identical emissivities over the spectral range of the High-Speed 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

1 On the EM Autosampler, press upward on the top of the latch to unhook it from the locking section, and gently pull the head sideways out of the furnace.

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

AVERTISSEMENT: Les cartouches sont chaudes. Soyez prudent lors de la mise en place et de la décharge des échantillons. Vérifiez la température du four sur le panneau avant; si elle est égale ou inférieure à 60° C, alors vous pouvez le manipuler en toute sécurité.

2 Once open, load the samples into the cartridges. Positions are counted from left to right, and the number of positions depends on holder configuration. Positions run consecutively for sample loading. Cartridges can be removed from alumina support rods and loaded outside of the head. All cavities must be filled, even if the samples are not being tested.



Figure 15 Loading the sample into Position 2 of the cartridge.

NOTE: <u>Figure 15</u> above shows Alumina cartridges. These may not be the cartridges provided with your instrument.

3 After all samples have been loaded and the cartridges positioned on the alumina rods, push the head back into the EM and secure the latch. The system is now ready for evacuation and purging.

Preparing the Instrument

Evacuating the System

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

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

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

The EM is equipped with a thermocouple gauge tube/vacuum meter assembly for determination of the vacuum level.

Vacuum and Purge Operation

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



3 Close the purge valve.

- **4** Turn on the vacuum pump.
- 5 Evacuate the system to at least 100 mTorr.

Back-fill with Purge Gas

- 1 Turn off the vacuum pump.
- 2 Follow the procedure for vacuum operation.
- 3 Enable the purge gas flow to the EM chamber.
- 4 Monitor the flow using the anti-siphon exit port assembly. Flow should be continuous, roughly 4–5 bubbles /second. Adjust with the built-in flow valve on the side of the EM.

Filling Detector with 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 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 online help for experimental instructions.

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



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

Figure 16 Operation > Startup Information.

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

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

1		_	Sequence	Number	Jun 30,	2010					Cancel
cimen ID	Common S	pecimen Tit	le _	Common	n Thickness	s (cm)		Use Expa	ansion		
Specime	n ID			Specin	nen Title			Thickness (cr	m) Diam	eter (cm)	Weight (g)
								0.0000	1.270)	0.0000
								0.0000	1.270)	0.0000
								0.0000	1.270)	0.0000
								0.0000	1.270)	0.0000
								0.0000	1.270)	0.0000
								0.0000	1.270)	0.0000
	-		-								
Temperature	Ramp	Repeat	Power	Point 1	Point 2	Point 3	Point	4 Point 5	Point 6	-	
Temperature (°)	Ramp (C°/Min.)	Repeat Points	Power Level	Point 1 Level	Point 2 Level	Point 3 Level	Point Leve	4 Point 5 Level	Point 6 Level		Jniform
Temperature (°)	Ramp (C°/Min.) 5.0	Repeat Points 3	Power Level 500.0	Point 1 Level	Point 2 Level	Point 3 Level	Point	4 Point 5 Level	Point 6 Level		Uniform Temperatur
Temperature (°)	Ramp (C°/Min.) 5.0 5.0	Repeat Points 3 0	Power Level 500.0 0.0	Point 1 Level	Point 2 Level	Point 3 Level	Point Leve	4 Point 5 Level	Point 6 Level		Uniform Temperatur
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Figure 17 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 DLF-1

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

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

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

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

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

General Cleaning Practices

- Use protective eye glasses
- Use finger coats or gloves
- Always hold optical components (mirrors, lenses, windows, etc.) from the edges; never touch the center after cleaning
- If strongly burned films remain on the components (like entry lens 120377) after the cleaning process, use weak acid (like vinegar) to try to dissolve the residual layers
- Always use distilled water and acetone at the end of cleaning process
- Use a thin layer of vacuum grease to seal all O-ring parts of the EM1200 module whenever it is necessary to reassemble the block containing O-rings

Recommended Cleaning Supplies

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

Cleaning Maintenance Schedule

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

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

- After each test run in vacuum at max. temperature over 800°C
- After 5 tests run in vacuum below 800°C
- After 3 tests run at max. temperature of 1200°C in an inert purge gas atmosphere
- After 5 tests run at max. temperature of 1000°C in an inert purge gas atmosphere
- After 10 tests run at max. temperature of 800°C in an inert purge gas atmosphere
- After 20 tests run at max. temperature of 500°C in an inert purge gas atmosphere
- In the case of testing volatile or reactive materials, cleaning is recommended after each test run

Periodically a check test should be run to verify the cleanliness of the optical path of the instrument. Use a thermographite reference sample 0.300 cm thick, room temperature test, 1000 Volts charging laser, and 3 shots. The instrument is considered clean if the signal (max. temperature of thermo diagram) is larger than 250 millivolts.

If, after cleaning the instrument and running a check test, the signal of thermo diagram is still smaller than 250 millivolts, contact TA service.

Maintenance Procedures

Cleaning the Optics

All parts included into optical path of the laser or detector should be cleaned and maintained as dust and smoke free components:

- Optics A: The exit window from laser, made from fused silica (hard material)
- Optics B: The entry lens into the furnace, made from fused silica (hard material)
- Optics C: The hot window inside the furnace, made from sapphire (hard and strong material)
- Optics D: The vacuum window on top of the furnace, made from sapphire (hard and strong material)
- Optics E: The optical lenses in the detector block, made from germanium (soft coating-scratches easily)



Figure 18 Optical path.

This cleaning process includes optics **B**, **C**, and **D** and will not take more than 20–30 minutes.

- 1 Exit from the software program, power off the instrument, disconnect the vacuum hose, and remove the bubbler from its port.
- 2 Remove the detector and empty it of residual LN_2 content.
- **3** Visually inspect the optics (E) in the bottom detector block. It should be free of scratches, dust or lint. If is necessary, use a compressed air can to dust off all the unwanted dust or lint from the surface of optics.

NOTE: If optics E is scratched, contact TA Service.

4 Using a hex key, unscrew the 3 screws from the bottom detector block, and then remove it from the top of the EM..



Figure 19 Remove the bottom detector block.

5 Using a hex key, unscrew the two screws from the vacuum window assembly and then remove it from the top of the EM.



Figure 20 Remove the vacuum window assembly.

6 Use tweezers to extract the optics (C). If it is smoked, should be removed and cleaned using isopropyl alcohol or acetone. If the layer of smoke is persistent, use vinegar, scouring pads, detergent, or distilled water to clean the surface exposed to the thermal process.



Figure 21 Extract and clean the optics.

- 7 With help of flash light, inspect the optical path of the detector. It should be free of debris or fiber material that comes from the furnace insulation. If contamination exists, it should be removed using tweezers, cotton swab, or (for the best results) a vacuum cleaner with small diameter suction port (< 10mm OD).
- 8 Using two people, lift the EM module from the top of DLF-1 module. The EM can simply be moved a few inches back, remaining on top of the DLF-1.
- 9 Again, using two people, from the front of the module, lift and incline the EM module at a 45° angle.
- 10 Using a hex key, unscrew the three screws from the entry lens assembly and then remove it from the bottom of the EM.



Figure 22 Remove the entry lens assembly.

11 Inspect the assembly for debris, dust, and smoke on the top of the entry laser lens. With an air duster, blow out all the dust/debris that is inside the block. For persistent smoke, use a cotton swab moist with acetone. If a remnant layer of smoke film is still there, using a hex key, unscrew the three screws from the assembly and extract lens B. Use scouring pads, detergent, vinegar, or distilled water to clean the surface exposed to the thermal process.



Figure 23 Entry lens assembly and parts.

- 12 Reassemble the parts back into EM module.
- 13 Using the compress air duster, clean the exit window (A) to remove any possible dust that may be contaminating the window.
- 14 Realign the EM module on top of DLF-1 module in original working position.

Conditioning the Furnace

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

- 1 Vacuum the furnace down to 300 mTorr.
- 2 After the gas begins to bubble, purge inert gas (stop the vacuum pump and open the valve for inert gas to pass through the furnace) for more than 1 minute.
- **3** Restart the vacuum to 250 mTorr and purge again with inert gas. Repeat the process a couple of times, gradually going down with vacuum values to 200 mTorr.
- 4 Set the furnace temperature to 700°C (see below for instructions) for two hours under vacuum.

Setting the Furnace Temperature to 700°C

1 From the Setup menu, select Select Diagnostic Mode.



Figure 24 Setup > Select Diagnostic Mode.

2 Enter the password provided by TA Service Personnel.

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

Commport 3				
Initialize	Furnace Tempera	iture 0.0 (C	:)	
Read TC	Sample Temperat	ure 0.0 (C	:)	
Read Setpoint	Low Limit 0.0	Low Limit 0.0		
Catasiat	Hgh Limit 10.0	Hgh Limit 10.0)	
Setpoint	PV 0.0	PV 0.0		
SV Offset	SV 0.0	SV 0.0		
System Enable	ole System Disable		Off	
		Furnace Disable		
Furnace Enable	Furnace Di	sable	Off	
Furnace Enable StandBy Cycle Ramp Setpoint Ramp Rate 5	Furnace Di Enable Cryo Coolin Disable Cryo Coolin	sable g	off	
Furnace Enable StandBy Cycle Ramp Setpoint Ramp Rate 5 Ramp Dwell 30	Furnace Di Enable Cryo Coolin Disable Cryo Coolin	sable g	Off	

Figure 25 Temperature Controller window.

- 4 Click Initialize.
- 5 Click **System Enable** both from the Temperature Controller window and on the front panel of the instrument.
- 6 Click **Furnace Enable** both from the Temperature Controller window and on the front panel of the instrument.
- 7 Enter 700 in the field next to the Setpoint button.
- 8 Click Setpoint.
- **9** After 2 hours at 700°C, stop temperature control by clicking **Furnace Disable** either from the Temperature Controller window or on the instrument front panel.

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

Replacement Parts

Replacement parts for the	DLF-1 are listed below.	Refer to the table below	when ordering parts.
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Part Number	Description
202315.001	Moderate Vacuum Module: large capacity two-stage mechanical vacuum pump
853024.901	In-Plane Test Kit: fixture used to mask the energy pulse for thermal diffusivity determination in the X-Y plane. The kit includes customized software.
202417.001	Graphite Paint
202419.001	Silver Paint - Vial - 1 oz.
853128.001	IR Detector, 12 Hour Dewar
853132.901	Adapter Ring, Molybdenum, 8 mm square specimens
853135.901	Thermographite Reference, 12.7 mm OD, 3.175 mm thick
853142.901	Thermographite Reference, 25.4 mm OD x 3.175 mm thick
853143.901	Stainless Steel Reference, 12.7 mm OD x 1.58 mm thick
853144.901	Stainless Steel Reference, 25.4 mm OD x 1.58 mm thick
853145.901	Electrolytic Iron Reference, 12.7 mm OD x 2.54 mm thick
853146.901	Electrolytic Iron Reference, 25.4 mm OD x 2.54 mm thick
853147.901	OFHC Copper Reference, 12.7 mm OD x 5.08 mm thick
853152.901	Vespel Reference, 12.7 mm OD x 1.27 mm thick
853154.001	Alumina Reference, 12.7 mm OD x 3.175 mm thick
853157.901	Molybdenum Reference, 12.7 mm OD x 3.175 mm thick
120377	Fused silica optical entry lens
113714	Sapphire optical hot window lens
100657	Sapphire optical vacuum window lens

Appendix A: Connection Diagram



 Make sure that the controller, monitor, and DLF-1 instrument are connected with certified power cords/plugs to the same electrical wall outlet circuit. The circuit must be rated for at least 15A.