

## User Manual

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# Low-Temperature Effusion Cell (LTEC)

## Top Filament (TF)



These are the original English instructions

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EN

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When devices are used for applications with technical safety requirements, the relevant instructions must be followed.

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## Component Modifications

| Year | Type | Modifications |
|------|------|---------------|
|      |      |               |
|      |      |               |
|      |      |               |
|      |      |               |

## Document Revisions

| Date     | Version Number | Document Changes |
|----------|----------------|------------------|
| 15-05-22 | 1.0            | Initial draft    |
|          |                |                  |
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|          |                |                  |

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# 1 PREFACE

## 1.1 Purpose of These Instructions

Thank you for purchasing this Low Temperature Effusion Cell. The purpose of this document is to make you familiar with the features and functions of the source, so that you can safely operate it as an end-user.

This documentation should therefore be regarded as an integral part of the Low Temperature Effusion Cell (LTEC). These instructions are intended for the following product models:

| Source <sup>1</sup> | Crucible Vol. | Add-on <sup>2</sup> | TC    | Flange | IVL/OD <sup>3</sup> |
|---------------------|---------------|---------------------|-------|--------|---------------------|
| LTEC-TF             | 13            | W                   | C / K | 63CF   | L215D60,5           |
| LTEC-TF             | 23            | X                   | C / K | 35CF   | L215D38             |
| LTEC-TF             | 23            | W                   | C / K | 63CF   | L215D60,5           |
| LTEC-TF             | 40            | W                   | C / K | 100CF  | L292                |
| LTEC-TF             | 60            | W                   | C / K | 100CF  | L292D63             |

## 1.2 Using this manual

To make best use of this manual:

- **Read the entire manual first.** Do not attempt to operate or perform maintenance of any kind on the product before you have thoroughly reviewed this manual.
- **Pay close attention to all safety information!** All MBE applications include inherent hazards and require strict adherence to safety standards. Read *Operator Safety* in this preface for critical safety information. Also see *Safety Hazards* in this preface to learn how safety hazards are indicated in this manual.
- **Use this manual as a tool for putting your own knowledge into practice.** This manual does not cover the theory, principles, or best practices for any particular MBE application. It aims to provide useful information to help you achieve your own objectives.
- **Remember that many DCA products are configurable.** It is not possible to address all aspects of all configurations in a single manual. If you are not finding the information you need, please consult *Additional Resources* in this preface, or contact us using the *Technical Support* information.
- **Refer to all graphics in context.** The graphics in this manual may not exactly match your product. Graphics are intended to illustrate only the features relevant to the topic at hand. Any optional, configurable, or missing features are identified, if contextually relevant.

## 1.3 Explanation of Safety Warnings



Indicates a hazardous situation which, if not avoided, could result in death or serious injury.

<sup>1</sup> LTEC – Low-Temperature Effusion Cell, TF – Top Filament,

<sup>2</sup> W – water cooling, S -shutter, X - None

<sup>3</sup> Custom IVL available

## **⚠ CAUTION**

Indicates a hazardous situation which, if not avoided, could result in minor or moderate injury.

## **NOTICE**

Indicates information considered important, but not hazard-related.

### **1.4 Intended User**

This manual is intended for trained UHV/MBE professionals, technicians, and persons under the direct guidance of such individuals. If you are not among these groups, please do not attempt to use the information in this manual to operate the equipment.

Read and understand this manual and its safety instructions before using this product. Failure to do so can result in serious injury or component/system damaged.

The manufacturer is not liable for cases of material damage or personal injury caused by incorrect handling or non-compliance with the safety instructions. In such cases, the warranty will be voided.

### **1.5 Obtaining Documentation and Information**

#### **1.5.1 Additional Resources**

In addition to these user instructions, the following related documentation should be consulted:

- TKD Lambda Power Supply User Manual
- Eurotherm 3500 Series PID Controller User Manual
- DCA Software Control User Manual (if applicable)
- Test report

#### **1.5.2 List of Component Manuals Ordering Documentation**

Documentation, user instructions and technical information can be requested by contacting DCA Instruments Oy at [info@dca.fi](mailto:info@dca.fi).

#### **1.5.3 Other languages**

This is the English user manual. Manuals in other languages are available upon request.

#### **1.5.4 Documentation Feedback**

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We appreciate your comments.

#### **1.5.5 Technical Support and Service**

For other service-related questions, information, technical assistance, or ordering user instructions, please contact the manufacturer:

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## 2 CRITICAL INFORMATION

This chapter provides important safety information, product cautions and a summary of important notes about your DCA component/source.

### 2.1 Safety Precautions

Carefully study this user guide and any related documentation listed in 1.5.1. Only properly trained personnel should operate or maintain the product.

The manufacturer cannot be held liable for damage resulting from errors, unintended or unprofessional use of the Effusion Cell.

The manufacturer is not liable for cases of material damage or personal injury caused by incorrect handling or non-compliance with the user guide. In such cases, the warranty will be voided.

#### 2.1.1 Personal protective Equipment

The Effusion Cell should be handled per general UHV practices. Powder-free protective gloves should be used to avoid contamination.

#### 2.1.2 Installation safety information

Inspect the Effusion Cell for damage before installation. If there is any visible damage, do not install the Effusion Cell and notify the manufacturer within one week of receiving the product.

#### 2.1.3 Maintenance safety information

All cleaning and maintenance tasks shall only be carried out by qualified and skilled personnel. To ensure the optimal performance of the Effusion Cell, regular maintenance shall be carried out by qualified personnel in accordance with the manufacturer's instructions.

Maintenance and inspection should be done on a regular basis. If there is any visible damage, a strong odour, or excessive overheating of components, stop using the Effusion Cell.

### CAUTION

**Equipment Modifications** to the Effusion Cell are not permitted. Any alterations made to the Effusion Cell without written permission from the manufacturer will void the warranty. Unauthorized alterations may lead to hazardous situations.

See the Appendix for an overview of the spare parts. You can always order spare parts by contacting the manufacturer.

Only clean the component with a lint-free cleanroom compatible cloth and suitable solvent (e.g., ethanol or IPA).

The Effusion Cell may feature safety marks in areas that pose a risk of injury or with other important instructions. Always replace missing or damaged safety marks immediately. If you replace safety marks with new ones, make sure that you place the new safety marks in the same places.

**2.1.4 Repair and modification Safety information**

**⚠ CAUTION**

**DO NOT attempt to repair components without permission and explicit instructions from the manufacturer. Contact DCA Instruments if the component requires repair. Do not attempt to modify the component before consulting DCA Instruments.**

**2.1.5 Safe Disposal**




Do not dispose of electric equipment, accessories, and packaging together with household waste material (only for EU countries). In observance of European Directive 2012/19/EC on waste of electric and electronic equipment and its implementation in accordance with national law, electric equipment that have reached the end of their life shall be collected separately and returned to an environmentally compatible recycling facility.

To safely dispose of the effusion cell, remove the crucible, chemically/mechanically remove any material contaminants from the construction, and remove any ceramic insulators. The remaining cell construction may then be safely disposed of as metal scrap

**2.2 Graphical Symbols**

Table 1 below provides safety information relevant to the Effusion Cell. The labels are located as close as possible to the relevant area it is applicable to.

**Table 1 Explanation of safety information relating to the system and the location of safety labels.**

| Symbol  | Warning / Caution   | Hazard Location                            |
|---|---|--|
|  | <p><b>ELECTRICITY</b></p> <p>May cause electric shock or burn.</p> <p>Trained personnel only.</p> <p>Disconnect and switch off main power before performing any maintenance.</p>  | <p>Electronic racks -<br/>Power supply</p> |
|  | <p><b>HOT SURFACE</b></p> <p>Contact with skin may cause burns.</p> <p>Do not touch during bakeout.</p> <p>Do not operate at high temperatures without water cooling.</p> <p>Ensure the source has cooled to room temperature before performing any maintenance.</p>  | <p>Effusion Cell</p>                       |
|  | <p><b>CAUTION</b></p> <p>Trained personnel only.</p> <p>Do not start operation without checking all connections first.</p> <p>Consult the Material Safety Data Sheet before loading or depositing any materials and follow all safety measures listed.</p> <p>Handle the component according to general UHV practices</p> | <p>Effusion Cell</p>                       |

### 3 INTRODUCTION

The component model number is based on the included features, refer to Fig. 3-1 for more information.

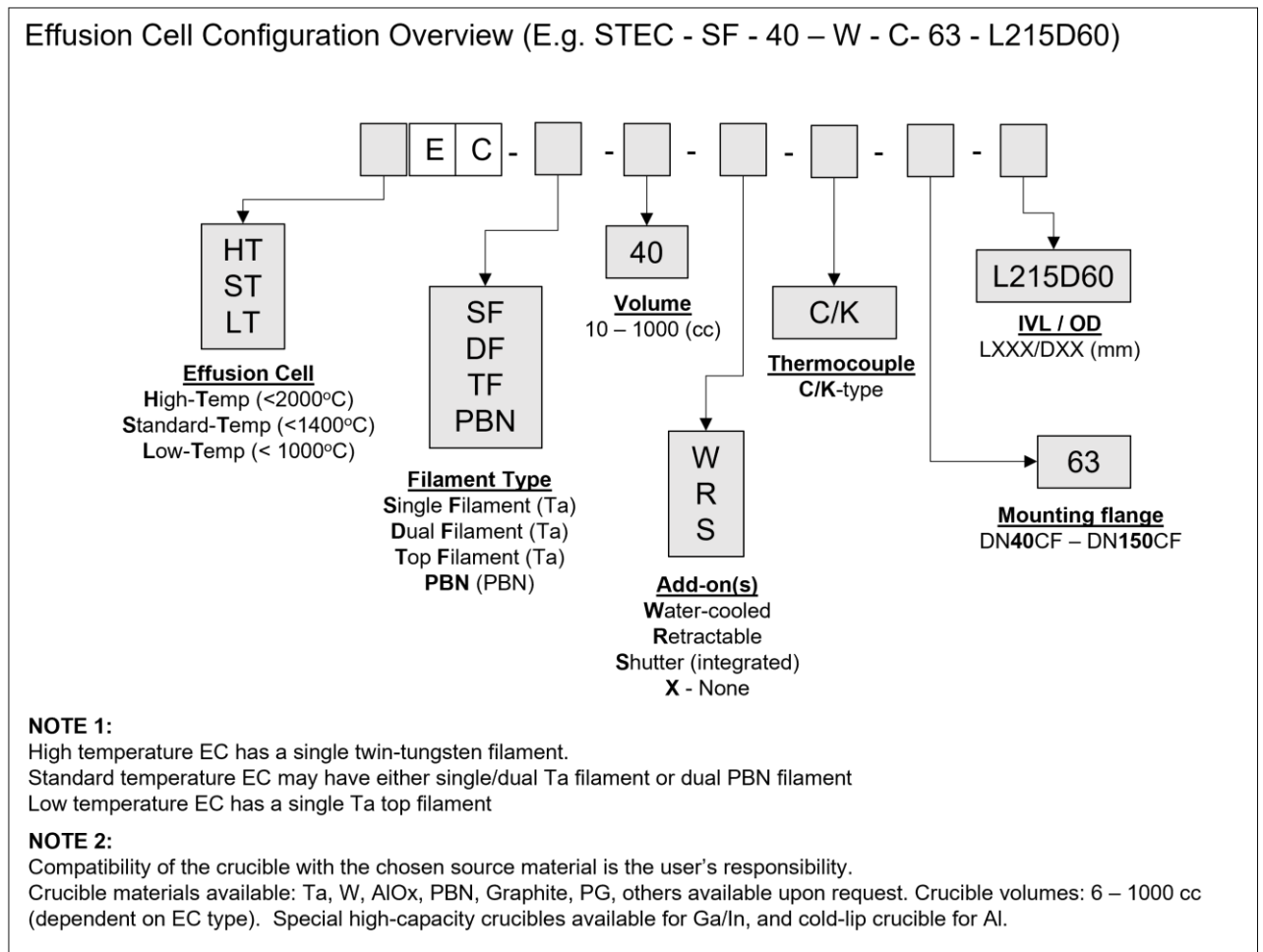


Fig. 3-1 Configuration overview of effusion cell source and possible product models.

#### 3.1 LTEC Product Description

The **Low Temperature Effusion Cell (LTEC)** (Fig. 3-1) is used to deposit materials with a high vapour pressure for molecular beam epitaxy (MBE) e.g. Li, Mg, Ca, Sr, Ba, Bi, K, Sb. The design is aimed at achieving steady flux and excellent film uniformity across the substrate. The LTEC is comprised of an internal heating assembly (Ta filament), thermocouple, mounting flange with power and TC feedthroughs and may also include a water-cooled shroud and/or shutter assembly.

##### 3.1.1 Filament design

The LTEC features a patented flat foil Tantalum filament that minimizes the temperature difference between the crucible and the filament. The filament for the LTEC only surrounds the top section of the crucible. This is designed to create a temperature gradient so that the material surface is used for deposition. This design is also suitable for materials with a high thermal expansion coefficient. The maximum continuous operating temperature is 1000 °C.

##### 3.1.2 Temperature control

The LTEC temperature is measured using a K/C-type thermocouple mounted to the crucible support cup. The TC provides feedback to a Eurotherm PID controller, which regulates the output from the DC power supplies to provide temperature stability better than ± 0.1 °C.

The K-type TC may be exchanged for a C-type TC – refer to the test report for the TC provided.

### 3.1.3 Water-cooling

The LTEC can be provided with or without an integrated water-cooling shroud. With the water-cooling shroud, the LTEC can be heated up to 500 °C without water.

### 3.1.4 Crucible

## ⚠ WARNING

Using materials with incompatible crucibles may result in damage to the LTEC. This will require the LTEC to be sent to DCA for inspection and repair. It is the user’s responsibility to ensure the appropriate crucible material is selected and that it is compatible with the material loaded.

The LTEC shall not be operated outside of the scope set out in this user manual. Interlocks may be implemented to protect the component from error conditions that may occur, and these should be enabled during normal operation.

The LTEC shall only be used with the original accessories supplied with the product. The accessories supplied are listed in Table 2 below.

Table 2 Summary of accessories included with the source and their intended use.

| Accessory                        | Description   |
|----------------------------------|---|
| DC Power supply                  | TDK Lambda: GEN40-19 (13cc - 40cc), GEN80-19 (100-200cc)                |
| Eurotherm PID Controller         | Single-loop 3508 – regulates the temperature. Includes DCA Programming. |
| LEMO® Power cable                | Connects the LTEC to the power supply, bakeable.                        |
| Thermocouple cable               | C-type, connects the LTEC and the ET controller, bakeable.              |
| Water-cooling lines <sup>4</sup> | Bakeable supply and return lines for the cooling shroud.                |

## 3.2 Product Standard Features

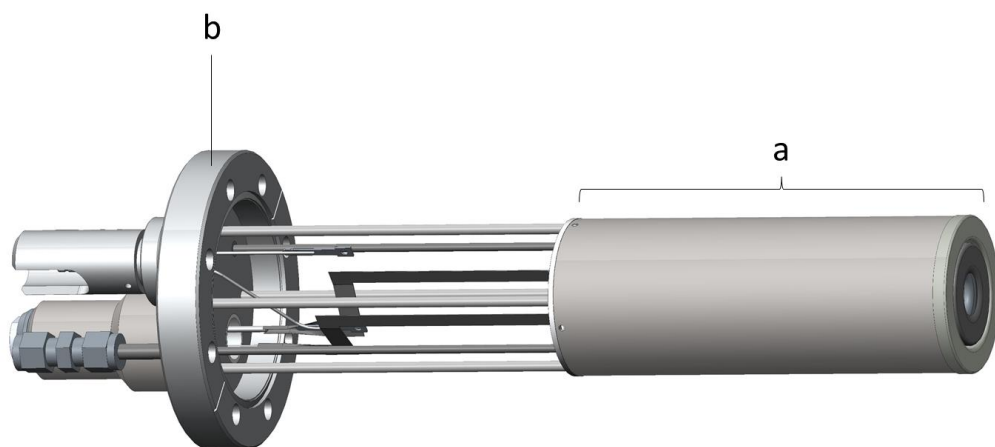


Fig. 3-2 Standard-temperature effusion cell (LTEC) side view. Parts labelled described in Table 3.

<sup>4</sup> If equipped with a cooling shroud.

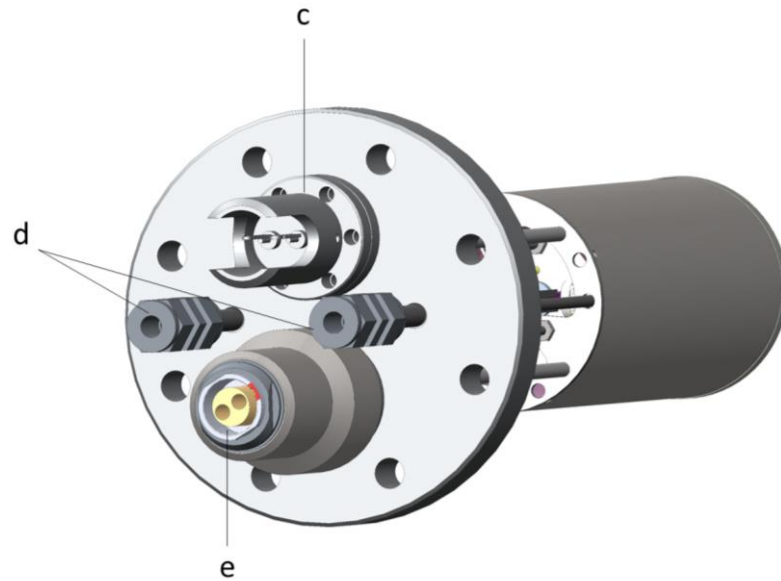


Fig. 3-3 Fig. 3 3 Standard-temperature effusion cell (LTEC) base flange view. Parts labelled described in Table 3.

Table 3 Description of parts labelled Fig. 3-2 and Fig. 3-3.

| Part | Description   |
|------|---|
| a    | Water-cooling shroud                                  |
| b    | Mounting flange                                       |
| c    | Thermocouple feedthrough                              |
| d    | 6mm Swagelok® water-cooling inlet/outlet <sup>5</sup> |
| e    | LEMO power feedthrough                                |

Only use the LTEC within the specified performance limits as described in Table 4 below.

Table 4 Standard features for the Effusion Cell.

| Feature                                   | LTEC                                    |
|---|---|
| Volume (cc)                               | 13-60 cc                                |
| Max. filling level                        | 70%                                     |
| Max. operating temperature                | 1000°C (PBN) / 1400°C (metal crucibles) |
| Max. outgassing temperature (max. 30 min) | 1100°C (PBN) / 1500°C (metal crucibles) |
| Temperature stability                     | Better than $\pm 0.1^\circ\text{C}$     |
| Max. temperature ramp rate                | Up to 100 °C/min                        |
| Thermocouple                              | K-type                                  |
| In-vacuum length                          | Check test report                       |
| Mounting flange <sup>6</sup>              | Check test report                       |

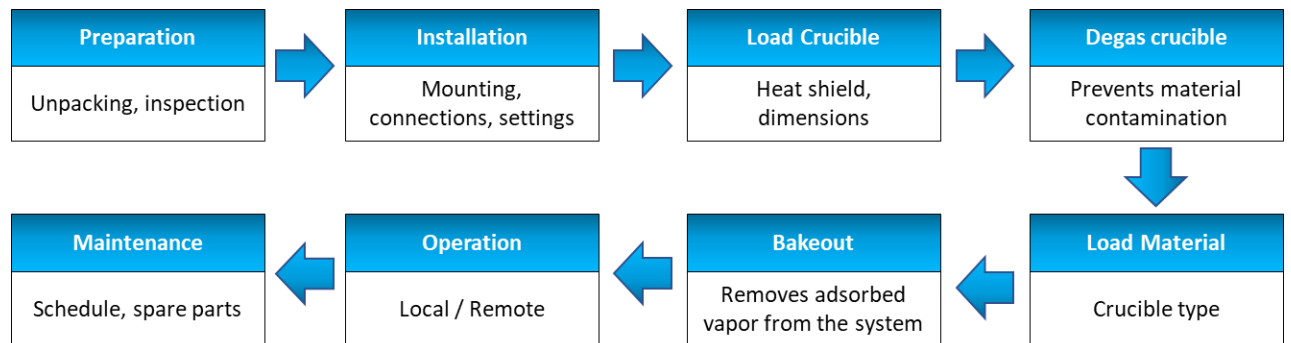
<sup>5</sup> If equipped with water-cooling shroud.

<sup>6</sup> Check test report if equipped with retractable mounting

|                                 |                   |
|---------------------------------|-------------------|
| Cooling water flow <sup>7</sup> | > 0.5 l/min       |
| Mounting flange gasket          | Check test report |
| Weight                          | < 5 kg            |

### 3.3 Operational Flow Chart

The operational flow chart gives an overview of the processes for the proper installation, operation, and servicing of the LTEC source. Each process is described in the following chapters of this manual.



<sup>7</sup> If equipped with water-cooling shroud

## 4 PREPARATION

### 4.1 First Inspection

When the Effusion Cell is first received, first check the external shipping box for any obvious signs of physical damage. Secondly, inspect the status of any impact or shock sensors attached to the shipping box. If any kind of mishandling of the box during transportation is suspected, please contact DCA immediately.

If the shipping box has been cleared after the initial inspection, the source can then be carefully unpacked from the shipping box. The source is shipped with a protective tube to shield the in-vacuum parts. This is attached to the mounting flange with bolts and an O-ring for sealing. After removing the packaging material, move the source with the protective tube to a clean area (e.g., cleanroom or similar contamination free space). In the clean area, the protective transfer tube may be removed.

After unpacking the source from the shipping container and removing the protective tube, inspect for any signs of damage that may have occurred during the shipment. Notify DCA immediately if any damage is found.

If the component will not be mounted to the vacuum system immediately, it should be stored within the protective tube. The shipping container and protective tube should be stored safely so that the component may be shipped back to DCA for repair/maintenance if needed.

### **NOTICE**

Handle the source in a clean area with powder free gloves to prevent contamination.

#### **To handle the source safely:**

1. Do not disconnect cables from components before switching off the power.
2. Handle outer vacuum sections only using UHV cleanroom compatible gloves.
3. Avoid handling in-vacuum parts that may be contaminated with material.

#### **Storing the source**

If the source is not to be mounted to the system immediately, keep it stored inside the protective tube within the shipping container. This should be stored in a dry, well-ventilated area.

If the source in-vacuum parts are contaminated with toxic material, ensure they are sealed to prevent the toxic material from being released into the surrounding area/packing material. Ensure suitable labelling is used to warn of toxic materials being present.

Store the component out of reach of unauthorized persons and do not allow persons unfamiliar with the component or these instructions to operate it.

### 4.2 Continuity Test

If possible, check the following before removing from the shipping container:

- Resistance of thermocouple and LEMO feedthroughs
- Power cables and TC cable resistance

The filament is insulated from the LTEC body and so the resistance between the LEMO connector and the LTEC body should be infinite. Similarly, the thermocouple wires are electrically insulated from the LTEC body, and so the resistance between the thermocouple contacts and the cell body should be infinite.

Check the filament and thermocouple resistances according to the test report. In general, the resistances should be according to the following:

- LEMO - outer LTEC body: Grounded,  $R = 0 \Omega$
- TC - outer LTEC body resistance: infinite,  $R > 1k \Omega$
- TC contacts resistance:  $0.5 - 1 \Omega$
- LEMO filament resistance:  $< 1 \Omega$

## **NOTICE**

The contact resistances may change somewhat during prolonged use.

## 5 INSTALLATION

### 5.1 Mounting the Source

The following tools and accessories are required during this procedure:

- 2 x 13mm wrench<sup>8</sup> or 2x 10mm wrench
- 2 x 14mm wrenches<sup>9</sup>
- Bolts and nuts according to the system design
- Gasket for the mounting flange. Check the mounting flange size from section 3.2.

#### **NOTICE**

It is recommended that only silver coated gaskets are used in UHV systems containing Arsenic.

Before mounting the effusion cell into the vacuum system, check that the in-vacuum length of the source is correct and according to the system design. Ensure that the in-vacuum part can freely move into the vacuum system during installation. To avoid any damage to the source or to the vacuum system, insert the cell slowly and stop immediately if any unwanted friction occurs. The cell may be installed in any orientation.

Remove any used gasket and place a new gasket over the top of the cell and slide down carefully to the mounting flange.

Using a crisscross pattern, tighten the flange with suitable bolts and nuts. In some cases, stud bolts may be required.

### 5.2 Water Connections

The DCA LTEC may be equipped with or without water cooling. If the LTEC is equipped with a water-cooling shroud, follow the instructions in this section.

The LTEC has a cooling coil with two Swagelok 6 mm connectors, one for the supply and one for the return. Because the water-cooling shroud has a coil construction, the water inlet can be connected to either of the 6 mm Swagelok connectors. Fig. 5-1 shows how to connect the waterlines to the Swagelok connectors.

#### 5.2.1 Required Flow

The required flow is summarised in Table 5. A pressure difference of about 2 bars is recommended. The water inlet temperature should be above ambient dew point to avoid condensation on the cooling lines.

#### **NOTICE**

If delivered by DCA the water lines are bakeable up to 200 C.

Table 5 Required cooling water flow rate for the LTEC Source.

| Source | Required flow rate |
|--------|--------------------|
| LTEC   | Min. 0.5 l/min     |

<sup>8</sup> 2x 13 mm wrench if the mounting flange size is DN63CF or bigger

<sup>9</sup> If equipped with water cooling shroud.

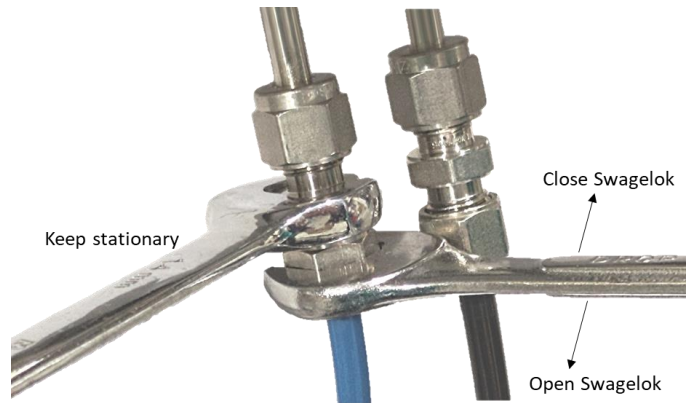


Fig. 5-1 Image showing how to connect and secure the Swagelok connectors.

## NOTICE

If delivered by DCA the water lines are bakeable up to 200 C.

Before starting the water-cooling, ensure the air is purged from the cooling lines to avoid any kind of damage to the cooling water circulation system.

### 5.3 Electrical Connections

## ⚠ WARNING

**High voltage hazard! Do not touch bare power cables or the LEMO connectors while operating the source.**

If the temperature control package is supplied by DCA, the LEMO, TC, and PSU control cables are labelled according to the Eurotherm control loop number. The Loop # is used to identify the source and the connections between the different devices.

## NOTICE

When inserting or removing the electrical cables, always handle the connector and not the cable to avoid unintentional damage. Check the connector pin positions when inserting and avoid forcing the contacts if there is resistance.

Perform the following connections and ensure the correct loop # is used:

1. Connect the power cable with LEMO connector to the LTEC and power supply.
2. Connect the TC cable to the LTEC TC feedthrough and the Eurotherm PID Controller.
3. Connect the Eurotherm PID control unit to the power supply using the control cable provided, (phoenix connector the ET and the DB25 connector to the PSU).

## NOTICE

If delivered by DCA the power cables and thermocouple wires are bakeable up to 200 °C.

## 5.4 Check the Eurotherm Settings

### NOTICE

Refer to the Eurotherm 3500 Series User Manual for detailed operating instructions.

If delivered by DCA, follow the PID control unit (Eurotherm) instructions in this chapter. This chapter will give a short introduction to the basic features of the Eurotherm temperature control unit as it is in DCA's default configuration. You can operate the Eurotherm from the front panel or using the serial communication port.

In a standard DCA set up, the voltage is controlled. In voltage control, the current is determined by the resistance of the component. Depending on the power supply it is normally possible to also limit the current. Refer to the power supply manual to do this.

The maximum power supply voltage can be limited from the Eurotherm setting OPHi. For example, setting the OPHi parameter to 80% for a 60-18 power supply means the maximum voltage is limited to 48 V.

It is advisable to set the OPHi to a level which is slightly above what is needed for the maximum temperature of the source. If this feature has not been set at the factory, you can set it using the data from the test report or during the first degas of the source. Refer also to the Eurotherm manual provided.

During normal operation it is recommended to use the automatic PID control with a suitable temperature ramp for heating and cooling. The PID settings are typically pre-set by DCA and are specified in the supplied test report. A typical temperature ramp for an empty cell is 100 °C/min. The suitable ramp rate with a certain crucible or certain source material depends greatly on the material qualities, typically the rates are between 30 °C/min and 60 °C/min.

### NOTICE

Before operation it is recommended to familiarize yourself with the following operations also from the Eurotherm control panel:

- Setting a ramp rate (**RR**)
- Setting a **OPHi**
- Setting a setpoint (**SP**)
- Switching between auto mode and manual mode
- Tuning the PIDs
  - Setting PID-values manually
  - Turning Auto-Tune on

### ⚠ CAUTION

The user should be aware that unsuitable PID control coefficients can lead to severe power oscillations which might damage the source. If the power oscillates during the first heating, autotune the PID parameters at a lower temperature. The autotuning can be repeated at a mid-point of the operating temperature range to achieve optimum stability across the entire range.

## 6 INSERTING THE CRUCIBLE

While holding the cell in an upright position, place the crucible inside. Pay special attention to the fit of the crucible. The lip of the crucible should rest on the top edge of the LTEC. Leaving a gap between the crucible lip and the cell top edge leads to extra heat loss and during deposition runs material may start to excessively condense on the crucible lip. Depending on the crucible material you may also risk breaking the crucible lip due to thermal stress.



Fig. 6-1 Inserting the crucible into the LTEC-TF. Part descriptions given in Table 6.

Table 6 LTEC parts labelled in Fig. 6-1.

| Part | Description        |
|------|--------------------|
| a    | Crucible (conical) |

### **NOTICE**

DCA recommends that the crucible is degassed prior to loading material.

## 7 OUTGASSNG (without material)

Prior to shipment, the LTEC is degassed and thoroughly tested. The test report supplied with the source provides the power vs. temperature and specifications for the source. If mounting the source directly after receiving it, it is not necessary to degas the empty LTEC prior to loading the crucible. However, if it has been stored for a prolonged period before use, degassing the source before loading the crucible is recommended. Similarly, degassing the crucible prior to loading the material is at the user's discretion.

### 7.1 First Heating

During the first heating test you should be able to confirm the correct control settings between the Eurotherm and power supply. The Eurotherm control signal is in the range 0-10 V, with 10 V corresponding to the highest power supply output i.e. the maximum voltage of the power supply. For example, for a 60-18 power supply you should see 3 V from the power supply for a 5 % Eurotherm control signal.

The power supply has two control settings 0-5 V and 0-10 V. The power supply is usually set to the 0-10 V control range at the DCA factory. If your supply is set to 0-5 V, you will find that the voltage from the power supply is doubled compared to the control signal. You can change the power supply control setting according to the instructions given in the power supply manual.

Before performing the first heat test, check the following:

- Electrical connections are in the correct position – TC and LEMO cables
- Water-cooling lines are connected, and cooling is running at the recommended flow rate
- Crucible is loaded correctly (with/without source material)
- Power supplies are switched on
- Eurotherm settings are correct

#### 7.1.1 PID Values Not Confirmed

If the PID values of the Eurotherm control unit were not previously tested, start heating the LTEC manually. Set the ET to manual mode and apply 1-2 % power at first to confirm the PSU and ET show the expected response. Continue to increase the power slowly, heat the cell in manual mode to 200 °C. Here you can switch from manual mode to auto mode and tune the PIDs if the power is oscillating.

#### 7.1.2 PID Values Confirmed

It is recommended to start heating the LTEC with a lower temperature ramp rate, for example 30 °C/min up to 200 °C, after which the ramp rate can be increased to 60 °C/min. As an extra safety measure, it is recommended to check the water outlet temperature during the first degas.

Degas the LTEC at about 100 °C higher temperature than the maximum temperature you intend to use. **The maximum degassing temperature for the cell is 1100 °C for the maximum period of 30 min.**

## CAUTION

**Never exceed the maximum degassing temperatures given for the cell.**

## **8 LOADING MATERIAL**

Remove the cell from the vacuum system, refer to section 5.1 for the tools required.

It is important not to overfill the crucible. Consider the angle at which the cell is mounted, take into account thermal expansion and that some materials are liquid during evaporation.

A filling level of  $2/3$  volume is considered safe. When using granules or pellets, consider melting the charge once and refilling to  $2/3$  level for maximum amount of source material.

Degas the material according to the instructions for the material and following necessary safety instructions.

## 9 OPERATION

### **⚠ CAUTION**

It is the user's responsibility to determine the safe operating procedure for each material. Damage caused to the effusion cell by crucible brakeage will be considered misuse and not be covered by the warranty.

The LTEC may be operated locally from the Eurotherm PID controller in the electronic rack or remotely from the PC using the DCA control software.

For local operation using the ET controller, refer to section 5.4 and the Eurotherm 3500 Series manual.

### 9.1 Remote Operation – Text Recipe Editor

For remote operation consult the DCA Control Software User Manual.

The DCA Control Software includes a Text Recipe Editor, which is used to program the source for a particular process, this includes datalogging, ramping, dwell, and cooling. Below is an **example** recipe, this should be used as a reference only and should not be considered part of the source instructions for safe operation, as this strongly dependent on the source material.

*Set LTEC Datalogging Header*

*Set LTEC Datalogging ON*

*Set LTEC Manual*

*Pause 1*

*Set LTEC Ramprate 50.0*

*Set LTEC Setpoint 200.0*

*Pause 1*

*While LTEC Temperature < 200.0*

*Set LTEC Output +0.1*

*Pause 10*

*While end*

*Set LTEC Auto*

*Set LTEC Ramprate 50.0*

*Set LTEC Setpoint 200.0*

*Pause 10*

*Set LTEC Setpoint 500.0*

*Pause 100*

*While LTEC Temperature < 980.0*

*Pause 300*

*Set LTEC Setpoint +100.0*

*Pause 150*

*While end*

*Pause 3600*

*Set LTEC Setpoint 10.0*

*Wait until LTEC Temperature < 500*

*Set LTEC Datalogging OFF*

## 10 BAKEOUT

The LTEC is bakeable up to 200 °C. If delivered by DCA, the water-cooling lines, LEMO power cables, and thermocouple cable are also bakeable to 200°C. These can therefore be left in place for bakeout.

### **⚠ CAUTION**

**Before bakeout make sure to completely purge the LTEC cooling lines.**

If possible, DCA recommends keeping the LTEC at a higher temperature than the bakeout temperature to minimise possible contamination, for example 300-400 °C (depending on the source material). The maximum temperature without water cooling is 500 °C if the LTEC is equipped with a water-cooling shroud.

## **11 MAINTENANCE**

When using a copper gasket, the gasket should be changed at least once every 12 months.

The water-cooling shroud of the LTEC can be cleaned periodically if there is a significant amount of deposit.

Inspect all electrical cables regularly for any damage and exchange if any fraying or exposed wiring is observed.

## 12 TROUBLESHOOTING

| PROBLEM   | PROBABLE CAUSE  | RECOMMENDED ACTION  |
|---|---|---|
| Crucible is stuck inside the EC                                   | <ul style="list-style-type: none"> <li>• A metal crucible is micro welded to the support cup.</li> <li>• Crucible is damaged and the evaporated material is gluing it to the support cup</li> <li>• Evaporated material goes through the grain boundaries of the crucible and is gluing the crucible to the support cup.</li> </ul> | <p>In any case <b>DO NOT TRY TO ROTATE THE CRUCIBLE</b>. This might damage the whole cell construction.</p> <p>Take a flat headed screwdriver and gently rotate it between the crucible lip and the heat shield (after removing the top heat shield). This applies a force perpendicular to the cell axis and the crucible should get loosen from the cup. If not, contact DCA.</p> |
| No current flow   | <ul style="list-style-type: none"> <li>• The power supply contacts are loose or damaged</li> <li>• The LEMO connectors are damaged</li> <li>• The filament assembly is damaged</li> </ul>   | <p>Check all contacts. Measure the resistance of the filament assembly at the feedthrough. An infinite resistance implies a damaged feedthrough or filament assembly. Check the cell visually if possible.</p>  |
| Sudden drop in filament resistance                                | <ul style="list-style-type: none"> <li>• Short circuit in the filament assembly</li> <li>• Crucible break</li> </ul>  | <p>Measure the resistance of the filament at the feedthrough and filament resistance to the cell body. Compare the measured to values to the expected ones (test report) Check the cell visually if possible.</p>   |
| The EC is leaking   | <ul style="list-style-type: none"> <li>• The power feedthrough, TC feedthrough or water-cooling assembly might be leaking.</li> <li>• The cell flange may also be damaged.</li> </ul>   | <p>Leak test carefully the power and TC feedthroughs and the water cooling tubes. Pay special attention to welded seams. Water may seal the leak in the cooling coil, so dry it out to see the leak with Helium. If possible, check a used gasket for signs of a damaged knife edge.</p>  |
| The cooling water outlet is hot                                   | <ul style="list-style-type: none"> <li>• The water flow rate is too low.</li> <li>• The water inlet temperature is too high.</li> </ul>   | <p>Check the cooling water set-up, make sure there is water flow and compare the conditions to the specified required values.</p>   |
| The apparent maximum voltage is less than the power supply rating | <ul style="list-style-type: none"> <li>• There is a OPHi setting in the Eurotherm</li> <li>• The voltage has been manually limited using the dial on the power source front panel</li> <li>• There is a short circuit in the filament assembly</li> </ul>   | <p>Change the Eurotherm setting OPHi. Do this with care and consideration since the power limit might have been set in the factory to protect the cell from overheating. Consult the DCA factory if necessary. Check the resistance and compare it to previous results to make sure you do not have a short circuit in the filament assembly</p>                                    |

|   |   |   |
|---|---|---|
| <p>Temperature fluctuating</p>  | <p>is</p> <ul style="list-style-type: none"> <li>• The PID parameters are unsuitable</li> <li>• The filament assembly is damaged</li> <li>• The TC wiring is damaged.</li> </ul>  | <p>First switch the control unit to constant power mode (manual mode). Monitor current and voltage values to check if the resistance changes or is different from previous experiments. If the temperature still fluctuates with constant power, there might be a problem with the thermocouple wiring inside or outside of the cell or a filament problem. Contact DCA in this case.</p> <p>If the temperature is not fluctuating with constant power, the PID values need to be re-tuned. Re-tune the PID coefficients using the Eurotherm's autotune function. If you have trouble completing the tuning, add any value to the parameter D (if originally "off") to get it into use and try to retune.</p> |
| <p>Temperature indicated by the thermocouple (matched to power) differs significantly from previous experiments</p> | <ul style="list-style-type: none"> <li>• Crucible usually adds 100 to 200 °C to the temperature achieved with certain power compared to an empty cell (the crucible material and source material load also have an effect)</li> <li>• The thermocouple type is entered wrong to the Eurotherm or software</li> <li>• The thermocouple wiring is damaged/ the filament assembly is damaged.</li> </ul> | <p>Check the conditions of your comparison data/ Check that the Eurotherm and software have the correct thermocouple type entered.</p>  |
| <p>There is no flux</p>   | <ul style="list-style-type: none"> <li>• The crucible is empty</li> <li>• The crucible orifice might be clogged by condensed material.</li> <li>• The shutter is closed</li> <li>• The isolation gate valve is closed</li> </ul>  | <p>If possible, check the cell visually. The clogging might be caused by poor positioning of the crucible, leaving a gap between the cell top edge and the crucible lip.</p> <p>If the crucible seems well positioned, try to evaporate the crucible lip clean by increasing the power to the top compared to the main filament. To avoid the clogging, run the top filament hotter with more power compared to the main filament (applicable to dual-filament cells only).</p>   |

In case further advice or assistance is needed contact the DCA factory, Aerotie 6, 20360, Finland - Tel. +358 2 2382 500 – email: [info@dca.fi](mailto:info@dca.fi)

## 13 WARRANTY

- I. DCA Instruments warrants that the Low Temperature Effusion Cell is free from defects in material and workmanship for a period of 12 months from the completed installation date.
- II. DCA Instruments shall incur no liability under this warranty if
  - a. the allegedly defective goods are not returned prepaid to DCA Instruments within thirty (30) days of the discovery of the alleged defect and in accordance with DCA Instruments' repair procedures; or
  - b. DCA Instruments' tests disclose that the alleged defect is not due to defects in material or workmanship.
- III. DCA Instruments' liability shall be limited to either repair or replacement of the defective goods, at DCA Instruments' option.
- IV. DCA Instruments makes no express or implied warranties regarding the quality, merchantability, or fitness for a particular purpose beyond those that appear in the applicable DCA Instruments user's documentation. DCA Instruments shall not be responsible for consequential, incidental, or punitive damage, including, but not limited to, loss of profits or damages to business or business relations. This warranty is in lieu of all other warranties.

## APPENDIX I - ACCESSORIES, CONSUMABLES, AND SPARE PARTS

For ordering accessories, consumables and/or spare parts, please contact:

DCA Instruments Oy  
 Aerotie 6, 20360, Turku, Finland  
[sales@dca.fi](mailto:sales@dca.fi)  
 +358 2 238 2500

### Supplied accessories/components

| Manufacturer | Item Description | Model                       |
|--------------|------------------|-----------------------------|
| Eurotherm    | PID Controller   | 3500 Series                 |
| TDK-Lambda   | Power supplies   | Genesys Programmable DC PSU |

### Consumables

| DCA #            | Item                      | Description     |
|------------------|---------------------------|-----------------|
| <b>CRUCIBLES</b> |                           |                 |
|                  | 6 cc Ta (9311-660-19)     |                 |
| 11020            | 6 cc PBN (S1)             |                 |
| 11021            | 6 cc PG (S1/)             |                 |
| 11028            | 13 cc PBN (S3)            |                 |
| 12008            | 23 cc AlOx (662-14)       |                 |
| 11029            | 23 cc PBN (S4-2)          |                 |
| 12068            | 23 cc PBN (2185-S-CRUC_B) | For < 1" sample |
| 12248            | 23 cc Ta (662-15)         |                 |
| 12069            | 23 cc W (S4-3_C)          |                 |
| 12906            | 40 cc AlOx (S6-1)         |                 |
| 10032            | 40 cc PBN-PG (S5)         | For Hot lip     |
| 11034            | 40 cc PBN (S6-1)          |                 |
| 11798            | 40 cc PG (S5)             |                 |
| 12088            | 60 cc PBN (8142-651-18)   |                 |
| 12584            | 60 cc PG (635-05)         |                 |

### Spare/replacement parts

| DCA #          | Item | Description |
|----------------|------|-------------|
| <b>GASKETS</b> |      |             |

|                          |                      |                 |
|--------------------------|----------------------|-----------------|
| 10077                    | Gasket DN40CF        | COPPER SILVERED |
| 10081                    | Gasket DN63CF        | COPPER SILVERED |
| 10084                    | Gasket DN100CF       | COPPER SILVERED |
| <b>SCREWS &amp; NUTS</b> |                      |                 |
| 11890                    | Screw Hex Head M4x10 | Ti DIN 912      |
| 12249                    | Screw Hex Head M4x8  | Ti DIN 912      |
| 12535                    | Screw Hex Head M3x6  | Ti DIN912       |
| 12536                    | Screw Hex Head M3x5  | Ti DIN912       |
| 18223                    | Screw Hex Head M4    |                 |
| 11162                    | NUT M2 HEXAGONAL     | Ta DIN934       |
| 11163                    | NUT M3 HEXAGONAL     | Ta DIN934       |
| 11310                    | NUT M5               | Ti DIN 934      |
| 11751                    | NUT M4 DIN934        | Mo              |
| 11792                    | NUT M3               | Mo              |