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Operating Manual

CPD 030 Critical Point Dryer and coating rate measuring device

Version 1.0



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Fundamentals

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1 Introduction

1.1 Operating manual

BAL-TEC AG in FL-9496 Balzers is the manufacturer of the CPD 030 critical point dryer, which is used for critical point drying of biological specimens with minimal deformation of the specimens.

This operating manual enables the user to operate the CPD 030 critical point dryer safely and in a correct manner.

1.1.1 Applicability range and system identification code

The description in this operating manual refer to the CPD 030 critical point dryer with the f011owing device designation:

CPD 030 critical point dryer

In the event of questions or correspondence with BAL-TEC AG it is helpful and accelerates the inquiry, if the user has the precise system identification code available, as specified on the identification plate of the system. This information defines the system including any customer-specific adaptations.

Note: For questions related to diagnosis (e.g. in case of faults), please mention the detailed error message shown on the screen.

1.1.2 Structure of operating manual

The operating manual is intended primarily as reference. The information included is divided into four parts, according to tasks and topics:

Part 1 Fundamentals

The part "Fundamentals" gives the necessary fundamental knowledge which every user of the CPD 030 critical point dryer must have.

Chapter 1 Introduction

Chapter 2 Safety

Chapter 3 Description and specification

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Part 2 Normal operation

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The part "Normal operation" contains the information required for regular operation of the CPD 030 critical point dryer. With this information the user can operate the system after it is ready for operation.

- **Basic Operation** Chapter 4.
- Performing a critical drying process Chapter 5.

Part 3 Special Operation

The part "Special operation" describes all work not performed during normal operation such as installation, start-up of system, maintenance, trouble shooting and special functions.

- Chapter 6 Unpacking, inspection and installation
- CPD 030 Maintenance Chapter 7

Part 4 Appendix

The "Appendix" contains general information, which always has to be accessible to every user. This concerns information related to the use of the operating manual (indexes) as well as data concerning the CPD 030 critical point dryer itself (technical data).

Chapter 8 **Appendix**

1.1.3 Target audience and mandatory reading

The operating manual is intended for authorized users of the CPD 030 critical point dryer. Exclusively the customer determines who is authorized as a user.

The requirements on the users vary with respect to the tasks to be performed. We therefore distinguish different user roles. One user may assume one or several of these user roles, if the user meets the specified requirements.

Note: The definition of the user roles with the requirements on the corresponding roles can be found in section "Users and their responsibilities".

Mandatory reading

Regardless of the user role assumed, every user is obligated to read chapter "Safety".

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1.1.4 Access aids

This operating manual facilitates fast access to desired information by means of the f011owing access aids:

Table of contents

 Due to the task- and topic- based structure of the operating manual complete information on a certain topic can be found easiest by means of the table of contents.

Index

• Information on a certain task or a special topic can be found fastest by means of the index in the appendix of this operating manual.

Notes on left margin

• Within the chapters of the operating manual the notes on the left margin of each page may be used for fast orientation.

1.2 Presentation convention

The operating manual contains abbreviations and marks to designate text sections or notes. This section explains the presentation conventions valid for the entire operating manual.

1.2.1 Warnings and notes on use

Please observe the significance of the f011owing warnings and notes on use:



Warning: Hazard or unsafe handling which may result in injuries that could be fatal.

Example: Danger of electric shock when in contact with a high tension line.



Caution: Danger or unsafe handling, which can cause only slight injuries but considerable damage to product, property or the environment

Example: Danger of early wear when running the engine with defective cylinder lubrication.

Note: Information on use which will help user to be able to technically use the CPD 030 critical point dryer efficiently or understand the workings and characteristics of the product.

Example: Unpack the instrument and check for exterior damage. Check that the main unit is still in its blue shrink wrapping.

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1.2.2 Symbols and Abbreviations

In this operating manual the f011owing symbols and abbreviations are used.

Symbols

Warnings and notes on use are marked by means of the above symbols.

Abbreviations

Abbreviations are only used for dimension units.

1.2.3 Position numbers and legends

For precise identification of parts or elements the operating manual includes several figures. Text items refer to the figures via position numbers or legends.

Position numbers

Position numbers refer to the overview figures. They designate the parts of the CPD 030 critical point dryer.

Legends

Legends are comprised of small letters and refer to items in the figures immediately above the corresponding text.

1.3 Customer documentation

Although the operating manual is an important document it is merely a part of the customer documentation. This section shows how the entire customer documentation is comprised.

1.3.1 Scope of the documentation

The entire documentation of the CPD 030 critical point dryer is comprised as f011ows:

- Operating manual for CPD 030 critical point dryer
- Documents (CE-certificate etc.)

1.3.2 Location of documentation

The operating manual is delivered as

printed document on paper

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The supplementary technical documents are delivered as

printed document on paper

Note: The EMC measurement protocol and further technical documents are available from the manufacturer upon request.

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2 Safety



Warning: The CPD 030 critical point dryer can be operated simply and safely, if the system is operated according to the operating manual. Hazards can however be caused by the system, if safety notes are not observed.

2.1 Safety concept of BAL-TEC AG

The CPD 030 critical point dryer has only few operational hazards and can be operated simply and safely by every trained user. Poorly instructed users however may cause damage and may even incur injuries through improper use.

As a result of a hazard analysis a safety concept has been established which shall protect the user of the CPD 030 from hazards and incorrect use.

The CPD 030 Critical Point Dryer has been developed in accordance with VDE 0411 regulations concerning electronic measuring devices and controls as well as the regulations of the elimination of radio interference f011owing VDE 0875.

The specimen pressure chamber has been tested by the Swiss Federal Laboratories for Materials Testing and Research in Duebendorf at a pressure of 200 bar with 15.000 pressure alterations (EMPA test report no. 183.723, dating from 21. Aug. 1985). In addition, safe dimensions of the specimen pressure chamber have been confirmed by the Swiss Control Association for Pressure Installations for an operating pressure of 150 bar (confirmation of 1. June 1983).

The specimen pressure chamber is secured with a bursting pane against exceeding the permissible working pressure of 150 bar.

For safe operation it is absolutely necessary to set up and connect the device according to the instuctions in section 6.3.

Besides the implementation of protective measures the core points of this concept regulate authorization for use of the CPD 030 critical point dryer and the responsibility of the individual users. Read the summary of these points in the f011owing chapters.

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2.1.1 Purpose of use of the CPD 030 critical point dryer

The use in accordance with regulations of the CPD 030 critical point dryer consists of critical point drying of biological or solid specimens with minimal deformation of the specimens.

Typical areas of application are:

SEM specimens

Small biological or solid specimens for subsequent SEM analysis

Any other application without written approval of the manufacturer shall constitute a violation. The manufacturer refuses to take any responsibility for damage caused thereby.

2.1.2 Users and their responsibilities

We designate all persons who work with or at the CPD 030 critical point dryer as users. Because the requirements on the users vary with respect to the individual tasks we distinguish different user roles. The user may assume one or several of these user roles, if s/he meets the specified requirements.

Customer

The "Customer" is the contractual partner of the manufacturer and is authorized for legally binding signature of contracts.

The "Customer" procures the CPD 030 critical point dryer and is responsible that the system is used in accordance with regulations. S/He determines, who is authorized to use the system.



Caution: Danger of wrong operation by poorly trained users. The customer is responsible that only users that have been instructed in detail and have been authorized (by the customer) operate the CPD 030 critical point dryer.

Operator

The "Operator" is an employee who has been trained to operate the CPD 030 critical point dryer and is familiar with the critical point drying technique. S/He operates the system ready for use and can recognize and rectify errors.

The "Operator" is responsible that errors that he cannot rectify him/herself, are immediately reported to the technician for rectification.



Caution: Danger of damage to the system. For any work that the operator is not familiar with or that is not described in detail in the operating manual, the operator is obligated to call the technician.

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Technician

The "Technician" is a professional with mechanical and/or electrotechnical basic training. He's able to install the CPD 030 critical point dryer, connect it to the energy supply and start up the system. In addition, he performs maintenance and repairs on the system.

The "Technician" is responsible that the system is in a flawless state for operation after installation, maintenance or repair.

2.1.3 Operation modes

The CPD 030 critical point dryer has the f011owing operation modes:

Off

In the operation mode "off" the CPD 030 critical point dryer is switched off and disconnected from the energy supply (mains) by means of the main switch.

Normal operation

We designate as "normal operation" the modes in which the system is either waiting in standby-mode or in normal operation.

Special operation

We refer to "special operation" when work deviating from the tasks in "normal operation" is carried out on the system. This can be installation, maintenance or repair work.

2.2 Operational hazards and protective measures.

The manufacturer has minimized the consequence of existing operational hazards by design and safety measures. However, a user who is deficiently trained may cause damage or may even be injured through improper use.

Pay special attention to all warnings and safety notes included in the description of activities and the f011owing general safety regulations.

2.2.1 Operational hazards to persons

Observe the f011owing operational hazards and the possible countermeasures:



Warning: Danger of electric shock when touching parts under voltage. Disconnect the system from mains supply and disconnect the mains connector before performing any work.



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2.2.2 Protective measures

For protection from remaining hazards of the CPD 030 critical point dryer different measures were implemented into the system design. The most obvious measure is a completely closed casing.

2.2.3 General safety regulations

Generally, the f011owing safety regulations apply to the handling the CPD 030 critical point dryer:

- Every user is responsible for his own health.
- Only instructed users, authorized by the customer are allowed to work at or with the system.
- For all activities at the CPD 030 the users must wear protective clothing prescribed in the respective environment.
- It is strictly prohibited to alter or remove protective equipment or covers of any kind.
- Every user must be trained with safe handling of the process gases and fluids that are utilized in the CPD 30 unit.
- After every repair the user (technician) must verify a flawless state of the system by a test run.
- After repair full operation of the system can only be guaranteed, if original spare parts according to the part lists are used.
- Careful operation and preventive maintenance of the CPD 030 reduces maintenance costs and assures reliable operation.
- By means of frequent checks and prompt rectification of even small damages considerable damage can often be avoided.
- Observation of this operating manual and all notes installed on the system serves your own safety.

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3 Description and specification

This chapter is directed toward all users of the CPD 030 critical point dryer. The chapter shows the design, it describes the function and designates the components of the system.

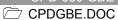
3.1 Introduction

Biological specimens intended for investigation in a scanning electron microscope must be dried before being introduced into the microscope (if the microscope does not have a cold stage) to allow the specimen to be imaged. If specimens such as cell suspensions are dried, tangential forces caused by molecular linkage affect the miniscule structures and cause deformation of the specimen.

Critical point drying with the CPD 030 is one method to prevent the damaging effects of the above mentioned forces by bringing the liquid into the gas phase without freezing. The deforming forces can be avoided by executing this process above the "critical point" of the liquid where the phase boundary between gas and liquid phase no longer exists.

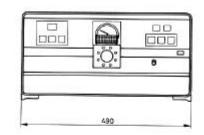
This first section of the manual contains specifications for the CPD 030 critical point dryer. However familiar you may be with this type of instrumentation, we suggest that you first inspect the instrument for any signs of damage incurred during shipping (see section 6 Unpacking, inspection and installation).

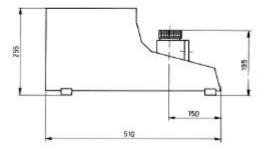
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3.2 Technical specification

Dimensions





Specimen chamber:

Usable volume Ø 40 mm x 36 mm

Fluid filling approx. 70 ml

Weight approx. 33 kg

Connection data

Electrical:

Voltage 230 V / 240 V / 115 V

Frequency 50 / 60 Hz

Power consumption 220 VA

Main fuse F1 / F2 4 A slow blowing (230 V / 240 V)

5 A slow blowing (115 V)

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Transitional fluid:

Inlet M 12 x 1.5

Outlet Ø 6 mm (R1/8")

Gas outlet Ø 6 mm (R1/8")

Highest permissible pressure

Safety bursting membrane approx. 150 bar

Refrigerator

Cooling capacity 85W

Refrigerant R12

Quantity 120 g

Temperature measurement

COOLING range adjustable 2°C to 12°C

HEATING range adjustable 30°C to 45°C

Operating parameters

Cool down time approx. 2.0°C/min.

Heating time NORMAL approx. 3.2°C/min.

Heating time SLOW approx. 0.9°C/min.

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3.3. Description of equipment

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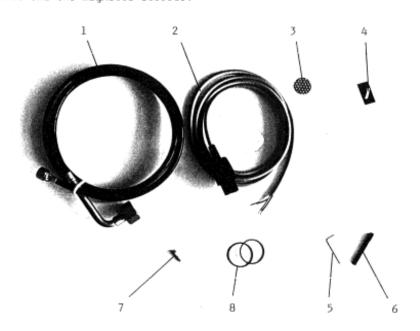
The CPD 030 as shipped consists of the f011owing equipmen

- Housing of the unit with built-in specimen pressure chamber
- Cooling unit for cooling of the specimen chamber
- Continuously contr011ed heater for warming up the specimen chamber
- Temperature sensor with digital temperature display
- Magnetic valves for media inlet as well as for the media and gas outlet
- Manually operated gas dosing valve for contr011ed gas outlet,
- Non-return valve in the media inlet line
- Pressure gauge for the pressure indication in the specimen chamber
- Safety burst membrane
- Magnetic stirring device
- Operating and display panel
- Electrical part for the supply and control of the cooling unit, the specimen chamber heater, the temperature measuring device, the magnetic stirrer.

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- Set of accessories, including
 - 1 Connecting hose, BU 011 471-T for gas pressure bottle
- 2 Power cable without connector plug, 2 m, B 8010 054 87
- 3 Aperture disk DN 30 mm, BU 011 618
- 4 Stirrer, B 8010 114 76
- 5 Pin wrench 1/16, N 5701 144
- 6 "Lampenzieher", B 4683 981 Al
- 7 Poral Filter, BU 011 546-R
- 8 O-ring (two) for screw-on cover of specimen chamber, B 8010 114 75

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Operation and Display Panel (Overview)



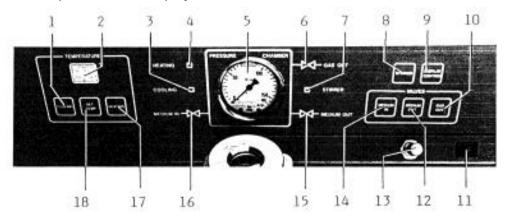
- 1 Brief operating instructions
- 2 Specimen pressure chamber
- 3 Operation and display panel

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Operation and Display Panel

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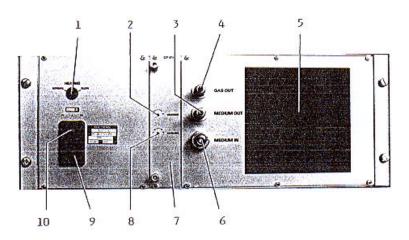


1	Cooling	Key to	o activate cooling
2	°C		Temperature display
3	Cooling		Indicator that cooling is activated
4	Heatir	ng	Key to activate heating
5 ber	Pressure		Pressure display in specimen cham-
6	Gas o	out	Valve is open when lit
7	Stirre	r	Stirrer on, when lit
8	Stirre	r	Key to activate stirrer
9	Displa	ay	Key for display checkout
10	Gas Out	Key to	o open gas outlet valve
11		Mains	s Switch
12	Medium Out	Key to	o open MEDIUM OUT valve
13			ally operated metering valve to se gas
14	Medium In	Key to	o open MEDIUM IN valve
15	Medium Out	Valve	is open, when lit
16	Medium In	Valve	is open, when lit
17	Heating	Heating is active when lit	
18	Set Temp.	Key for nominal temp. Display	

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Rear View

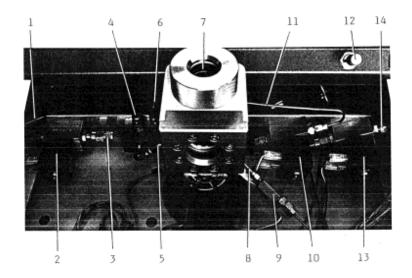


1		Heating	Normal Speed or slow speed
2		Setpoint high	Upper temperature set point
3		Medium Out	Outlet for transition liquid
4		Outlet for gas	eous transition medium
5		Cooling unit	
6		Medium In	Inlet for transition liquid
7		CP 010	Control board
8		Set point low	Lower temperature set point
9	Power	connector	
10	Fuses	and mains sw	vitch



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Supply and Drain System



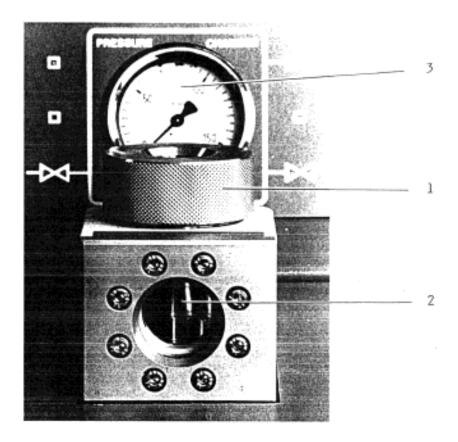
1	Media inlet line		
2	Magnetic valve for media inlet		
3	Non-return valve		
4	Burst membrane		
5	Supply line to specimen pressure chamber		
6	Pressure line to pressure gauge		
7	Specimen pressure chamber		
8	Outlet line of pressure chamber		
9	Filter housing with poral filter		
10	Magnetic valve for media outlet		
11	Media outlet line		
12	Dosing valve (manual) for gas outlet		
13	Magnetic valve for gas outlet		
14	Gas outlet line		

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Specimen Pressure Chamber

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- 1 Screw-on cover for specimen pressure chamber opening
- 2 View port to monitor liquid level
- 3 Pressure gauge

Specimen holders or specimen baskets (see section 9) can be inserted into the specimen pressure chamber. It can be closed with a screw-on cover (1). The front sight glass (2) allows to observe the level of the transition liquid as well as the achievement of the critical point.

A cooling coil connected with the cooling unit and a heater built into the specimen pressure chamber are designed to set the temperature necessary for the critical point drying process. The control is carried out using a temperature sensor.

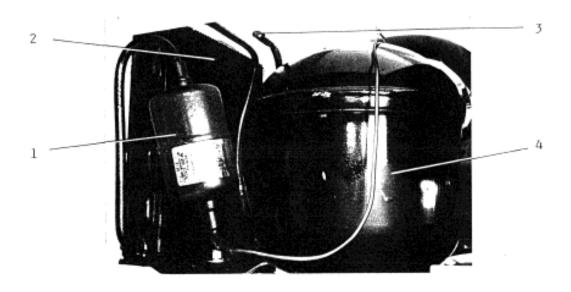
A magnetic stirrer system is built into the bottom of the specimen pressure chamber to mix the liquids during the media exchange. The magnetic stirring rod lies within the specimen chamber.

A burst membrane protects the specimen pressure chamber from exceeding the permissible maximum pressure of approx. 150 bar.



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Cooling Unit



- 1 Dryer
- 2 Condenser, air cooled
- 3 Filler socket for cooling medium
- 4 Refrigerating compressor

Hermetically sealed cooling unit containing, a cooling compressor (4) with a cooling power of 85 W and an air-cooled liquifier (2). Cooling medium R 134a (CFC-free) filling amount 120 g.

The operating temperature of the specimen pressure chamber can be set for a range between 2°C to 12°C. The maximum permissible ambient temperature for a perfect function of the cooling unit is 30°C.

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Normal operation

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4. Basic Operation

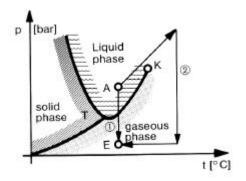
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This chapter is directed toward users who operate the CPD 030 critical point dryer in normal (standard) operation.

As precondition for this work, the user must be familiar with the chapter 3 Description and specification.

4.1 Crtical Drying Method

Drying water-containing biological specimens in air or under vacuum can drastically alter their structures or even destroy them completely. They must therefore be dried by a gentler method. One well-known method is "Critical Point Drying".



K= Critical Point

A= Initial state

E =Final state

T=Triple point

① Air-drving / vacuum-

Phase diagram

The surface tension of the water in a specimen at the point at which it changes from the liquid phase to the gaseous phase ① can destroy a delicate specimen.

By increasing the pressure and temperature of the specimen it is possible to dry it without crossing a phase boundary ②. This is possible because once the critical point has been passed, the density of the "liquid" and the density of the "gas" are the same. The critical point for water is 228,5 bar and 374°C. However, this high pressure and extreme temperature would normally destroy a biological specimen. For this reason the specimen must first be treated in a suitable transitional fluid such as CO2 whose critical point of 73,8 bar and 31°C is considerably more advantageous.

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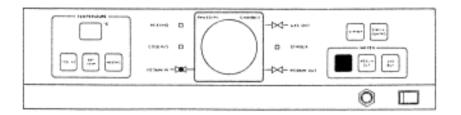
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4.2 Basic Functions

4.2.1. Cooling of the specimen pressure chamber

- Carefully close specimen pressure chamber with screw-on cover prior to operation of the unit.
- Switch on the unit with main switch, located on the right hand side of the middle mask of the unit.



- Press key COOLING:

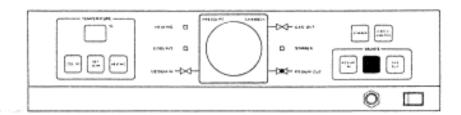
Cooling unit starts up. LED "COOLING" lights up.

Actual temperature of the specimen pressure chamber is shown on the temperature display.

At a room temperature of 21°C the specimen pressure chamber is cooled down to 10°C in about 7,5 minutes and subsequently falls below this temperature to 3° to 4°C.

The cooling unit automatically keeps the temperature of the specimen pressure chamber below 10° C.

4.2.2 Filling transition liquid into the specimen chamber



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If CO2 is used as the transition liquid, open the shut-off valve of the CO2 gas pressure bottle.

Press key "MEDIUM IN":

Medium inlet valve opens. LED "MEDIUM IN" valve lights up.

Manometer shows the pressure in the specimen pressure chamber (identical to the pressure in the CO2 gas pressure bottle).

- Fill specimen pressure chamber up to the upper edge of the front sight glass with liquid CO2.
- Press key "MEDIUM IN" again:

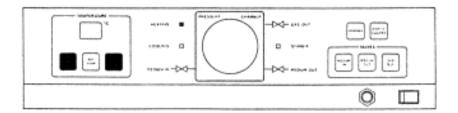
Medium inlet valve shuts. LED of valve "MEDIUM IN" goes off. The CO2-level in the specimen pressure chamber might rise a little.

4.2.3. Filling up the Medium-Outlet Tube

Press key MEDIUM OUT:

Medium outlet valve opens. LED of valve "blind schematic" symbol MEDIUM OUT lights up.

- Allow the level of the liquid medium in the specimen pressure chamber to drop to 2 to 3 mm below the upper edge of the front sight glass.
- Close medium outlet valve by pressing key MEDIUM OUT



Press key COOLING again.

Cooling unit switches off. LED of the "blind schematic" symbol COOLING goes off.

4.2.4. **Heating of Specimen Pressure Chamber**

Press key HEATING:

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Heater installed in the specimen pressure chamber switches on. LED of the "blind schematic" symbol HEATING lights up.

The rising temperature of the specimen pressure chamber is shown on temperature display.

With the rising temperature the pressure in the specimen pressure chamber increases: display on manometer.

During the warmup of the transition liquid CO2, part of this liquid is transformed into gas. Due to that, the level of the liquid drops.

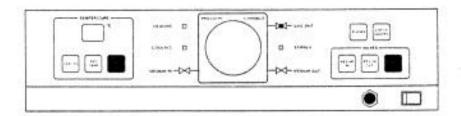
Having reached the critical temperature and the critical pressure (31°C and 73,8 bar for CO2) the liquid CO2 is transformed into the gaseous phase. The change of the physical state can be observed through the front sight glass.

4.2.5. **Releasing Gaseous Transition Medium**

Having reached the critical point of the transition liquid, the gaseous medium is released from the specimen pressure chamber.

Check if manually operated gas dosing valve METERING VALVE is closed.

Carefully turn the operating knob of the gas dosing valve clockwise to its stop. If the valve is closed too tightly, the valve seat can be damaged.



Press key GAS OUT:

Magnetic valve for gas outlet opens. LED of "blind schematic" symbol GAS OUT lights up

- Open gas dosing valve METERING VALVE
- Carefully turn operating knob anti-clockwise.
- If a flow meter (recommended accessory) has been fitted (see section 1.5.), open gas dosing valve just enough to let the ball of the flow meter float in the upper half of the measuring tube. Re-adjust gas dosing valve if the pressure decreases.

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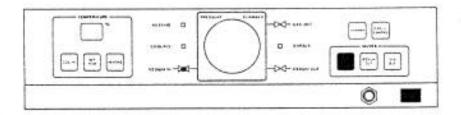
Decrease of pressure in the specimen pressure chamber can be observed on the manometer. Atmospheric pressure should be reached in about 10 minutes. After that the specimen pressure chamber can be opened.

If the pressure drops too fast, the gaseous transition medium will condense due to the cooling caused by expansion. This will become visible by the formation of fog in the specimen pressure chamber. In this case a previously dried specimen can no longer be used.

- After the gaseous transition medium has been released, switch off HEATING and close medium outlet valve.
- Press key GAS OUT:

Magnetic valve for the gas outlet closes. LED of valve "blind schematic" symbol GAS OUT goes off.

4.2.6. Switching off unit



- Close shut-off valve of gas bottle.
- Open screw-on cover of specimen pressure chamber.
- Open medium inlet valve by pressing key MEDIUM IN: The gas contained in the connection tube escapes through the open specimen pressure chamber.
- Close medium inlet valve by pressing again key MEDIUM IN.
- Close specimen pressure chamber with srew-on cover.
- Switch off main switch, located on the right hand side of the middle mask of the unit.
- Press key HEATING:
- The heater is switched off.

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- LED of "blind schematic" symbol HEATING goes of
- Reduce pressure in gas connection tube from gas pressure container to the unit to atmospheric pressure prior to switching off the unit.



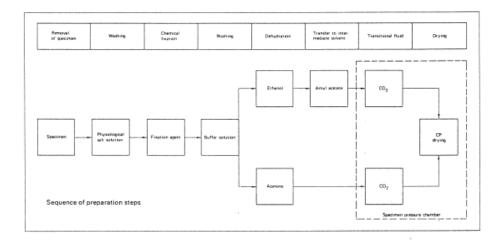
5. Performing a critical drying process

This chapter is directed toward users who operate the CPD 030 critical point dryer in normal (standard) operation.

As precondition for this work, the user must be familiar with part 1 Fundamentals and chapter 4. Basic Operation.

5.1 Preparation of Specimen

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- Remove the specimen from the organ or the culture medium.
- Wash in a physiological salt solution.
- Fix chemically with a suitable fixing agent.
- Wash out fixing agent with a suitable buffer solution.
- Dehydrate the chemically fixed specimen with acetone or ethanol. If ethanol is used as a dehydration medium, which does not mix with the transition liquid CO2, it must first be transformed step by step into the intermedium amylacetate.

NOTE: The specimen to be critical point dried is prepared in the same way as a sample for ultramicrotomy.

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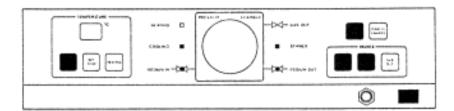


5.2. Insertion of Specimen into Pressure Chamber

- Transfer prepared specimen into the dehydration medium acetone, or possibly into intermedium amylacetate, in a suitable specimen holder (see section ACCESSORIES).
- Fill the specimen pressure chamber with the liquid (in which the prepared specimen is already contained) to such a level that the specimen is completely covered with liquid after insertion.
- Quickly insert the specimen holder into the specimen pressure chamber.
- Tightly close the specimen pressure chamber with the screw-on cover.

The prepared sample must always remain wetted until the critical point drying process is carried out. Otherwise the structure of the sample would be damaged after beginning to dry.

5.3 Transfer the Specimen into Transition Liquid



- Switch on mains switch
- Press key COOLING to pre-cool chamber
- Wait until the pre-selected "lower" temperature has been reached.
- Open shut-off valve of the CO2 gas bottle
- Fill the transition liquid into the specimen pressure chamber
- Press key MEDIUM IN: medium inlet valve opens.
- Fill specimen pressure chamber up to the upper edge of the front sight glass.
- Press key MEDIUM IN again: medium inlet valve shuts.

Mixing the two liquid media

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Press key STIRRER: magnetic stirrer is switched on. The magnetic stirrer should not be used if the specimens are very delicate.

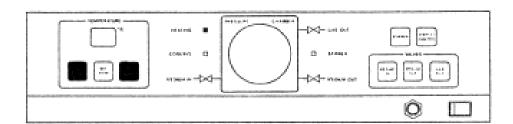
Drain media mixture from specimen pressure chamber

- Press key MEDIUM OUT: medium outlet valve opens.
- Drain media mixture from specimen pressure chamber until the specimen is just still covered by the liquid.
- Press key MEDIUM OUT again: medium outlet valve closes.
- Repeat procedure several times. Let the transition liquid in and let the media mixture out as described above several times until the liquid initially brought into the specimen pressure chamber (acetone or amylacetate) has completely been replaced be the transition liquid CO2. Experience suggests that this process has to be repeated six to eight times.
- Press key STIRRER to switch off the magnetic stirrer.

Fill specimen pressure chamber with transition medium

- Press key MEDIUM IN: medium inlet valve opens.
- Fill specimen pressure chamber with transition medium CO2 to a level approx. 2-3 mm below the upper edge of the front sight glass.
- Press key MEDIUM IN again: medium inlet valve closes.

5.4 **Dry Specimens**



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Check if all valves are closed.

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 LED's of "valve-blindschematic" symbols MEDIUM IN, MEDIUM OUT and GAS OUT must be off.

Switch off cooling of the specimen pressure chamber

Press key COOLING: cooling unit switches off.

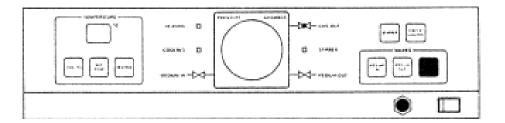
Warm up specimen pressure chamber.

- Press key HEATING: heater of the specimen pressure chamber is switched on.
- Temperature and pressure in the specimen pressure chamber rise: After reaching a temperature of approx. 20°C, the level of the transition liquid will slowly begin to drop until the critical point has been reached due to the transition of the liquid into the gaseous state.

Reaching the Critical Point

- The critical point of CO2 is reached at a temperature of 31°C and a pressure of 73,8 bar: the specimen begins to dry.
- Raise temperature and pressure slightly above the critical values prior to releasing the gas from the specimen pressure chamber.

5.5. Release Gaseous Transition Medium



- Check if the manually operated gas dosing valve METERING VALVE is closed.
- Press key GAS OUT to open magnetic outlet-valve.
- Carefully turn operating knob anti-clockwise.
- Watch flow meter (accessories): open gas dosing valve until indicator ball floats in the upper half of the flow meter measuring tube.

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- Readjust gas dosing valve with decreasing of pressure in the specimen pressure chamber.
- Pressure decrease to atmospheric pressure should take approx. up to 10 minutes.

If the gas is released too quickly the gaseous transition medium condensates (mist-formation). In this case the specimen gets wet again and becomes unusable.

5.6. Remove Dried Specimen

Switch off heater of the specimen pressure chamber

Press key HEATING: heater is switched off

Open specimen pressure chamber

- After reaching atmospheric pressure the screw-on cover of the specimen pressure chamber can be opened.

Close gas-outlet valve

- Press key GAS OUT: magnetic outlet-valve closes.
- Shut manually gas-dosing valve METERING VALVE by turning the operating knob clockwise.

Remove specimen from specimen pressure chamber

- Remove specimen holder with dried specimen from pressure chamber for further processing.
- Since the dried specimen is highly hygroscopic, it has to be coated as quickly as possible with a thin metal- or carbon film to protect it from atmospheric humidity.
- If this is not possible, it is recommended to keep the specimen in a desiccator until it is processed further.

5.7. End Critical Point Drying Process

- Close shut-off valve of gas pressure bottle
- Release pressure from gas connection hose

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- Press key MEDIUM IN: inlet valve opens and gas escapes from the connection hose through the open specimen pressure chamber.
- Press key MEDIUM IN again: inlet valve closes.
- Switch off mains switch.

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Special Operation

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6 Unpacking, inspection and installation

This chapter is directed to the technician who installs the CPD 030 critical point dryer and performs the initial operational set-up.

The premise for this work is that the technician is familiar with the chapter 2 Safety and 3 Description and specification

6.1 Unpacking

As soon as your critical point dryer and accessories have been delivered they should be checked for damage in transit.



- 1) Examine the exterior of the casing for evidence of rough handling.
- 2) Unpack the instrument and check for exterior damage.
- 3) Keep all packing materials for any evidence of miss-handling that may be required or for possible future transport.
- 4) When removing packing check that there may be no rattling when the unit is gently shaken..
- 5) Check all the items off against the delivery note and record the serial numbers in the Appendix for future reference.
- 6) Report any evidence of damage both to the carriers and to BAL-TEC or our local agent immediately. We advise you that no responsibility can be accepted for damage in transit, if such a report is not made within a period of 5 working days.

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6.2 Warranty



BAL-TEC cover all equipment made by BAL-TEC against defects in materials and workmanship for one year from date of shipment. BAL-TEC will not warrant performance of the equipment if accessories or consumables from other manufacturers are used.

BAL-TEC reserves the right to determine and will not allow a warranty claim for, defects which have been caused by the user's modification, misuse or other abnormal conditions of operation.



Static damage to internal components caused by inadequate handling during servicing or damage caused by using the incorrect power is not covered.

BAL-TEC will repair or replace, at its discretion products which prove to be defective during the warranty period provided that they are returned, carriage pre-paid, to BAL-TEC.

No other warranty is expressed or implied. BAL-TEC is not liable for consequential damage to equipment or material.

6.3 Installation

Required space for setting up the unit: width 490 mm, depth 660 mm.

To allow adequate intake of cooling air required for the refrigeration unit and due to the inflexibility of the high pressure gas hose, the wall distance has to be at least 150 mm.

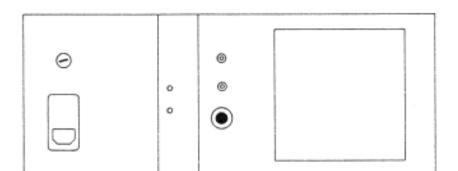
6.3.1. Connection of CO2-Container

For a correct implementation of critical point drying it is absolutely necessary to use a pressurized gas container with a pipe (feed-pipe), that the medium (CO2) can reach the specimen pressure chamber in liquid form.

In order to prevent accidents the CO2-pressurized gas container must be securely fastened in an upright position.

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- Insert seal DN10/6,5 x 2 (BU 011 646) in compression nut M 12 x 1,5 of high pressure gas hose (BU 011 873).
- Screw high pressure gas hose with compression nut M 12 x 1,5 onto connection MEDIUM IN.
- Connect other end of high pressure gas hose to gas pressure container.

High pressure gas hose BU 011 873 and seal BU 011 646 are included in the accessory set of the unit.

6.3.2. Connection of Medium Outlet

- Premounted hose nipple BU 016 448-T is already screwed onto connection MEDIUM OUT.
- Mount PVC tube DN 5/10 mm onto hose nipple.
- Lead other end of tube into a sealable container preferably filled with activated carbon.

Vapours of Acetone or Ethanol/Amylacetate used to dehydrate and transfer the specimen into the transitional fluid respectively, must not be allowed to freely diffuse into the workroom.

6.3.3. Connection of Gas-Outlet

The use of a flow meter BU 011 102 -T (accessory) to control the dosed gas outlet from the specimen pressure chamber is highly recommended.

Screw connecting tube of flow meter BU 011 1O2-T (accessory) onto outlet GAS OUT.

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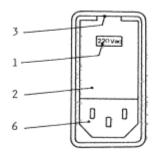
Put flow meter on CPD 030 unit.

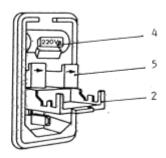
If no flow meter is available, control of dosed gas outlet can be done via tube and water container:

- Premounted hose nipple BU 016 448-T is already screwed onto connection GAS OUT.
- Mount PVC tube DN 5/10 mm onto hose nipple.
- Dip other end of tube into water-filled container: Exhaust of gas can be contr011ed by the bubbles formed and regulated accordingly.

6.3.4. Electrical Connection

Check assigned connecting voltage for the unit of the mains connection and, if necessary, adjust required connecting voltage, observing, the f011owing procedure:

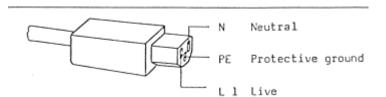




- Push cover (2) by inserting e.g. a screw-driver into the upper slot (3).
- Remove voltage selector (4) and reinsert so that the desired connecting voltage is shown at the display (1).
- Pull out fuse holder (5) and insert fuses with the required value:
 4 AT for 220 V respectively 5 AT for 115 V.
- Reinsert fuse holder (5) and close cover (2).

Connecting the power cable:

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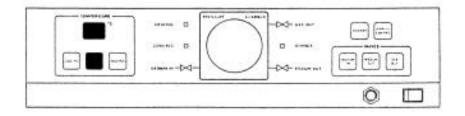
- Fit locally required mains plug to power cable, considering the f011owing figure of the power cable socket (L I + N + PE).
- Connect power cable to wall socket.

6.3.4 PREPARATION OF UNIT

6.3.4.1. Function check

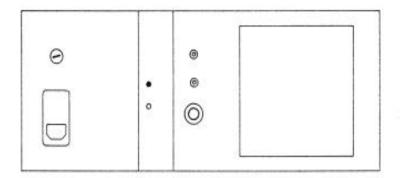
- Connect power cable
- Switch on main switch, on the right hand side of the middle mask.
- Check Display Panel by pressing key DISPLAY CONTROL:
- All 6 LED's of the "blind schematic" symbols must light up while the button is being pressed.
- Checking Magnetic Stirrer, by inserting magnetic stirrer B 8010 114 76 (included in the set of accessories) into specimen pressure chamber and pressing the key STIRRER:
- LED STIRRER of "blind schematic" lights up and inserted magnetic stirrer rotates.
- Switch off stirrer by pressing key STIRRER again.

6.3.4.2. Setting "Upper" Nominal Temperature



Front View

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Rear view of unit

Press key SET TEMP

The pre-set "upper" nominal temperature appears on the temperature display while the key SET TEMP is being pressed.

- Make adjustment with potentiometer HEATING located at the rear panel of the unit.

"Upper" nominal temperature for CO2 as transition liquid is 40 C.

6.3.4.2. Setting "Lower " Nominal Temperature

Press key COOLING

The cooling unit starts up, LED COOLING of "blind schematic" lights up.

The actual temperature of the specimen pressure chamber is shown on the temperature display

Press key SET TEMP

The "lower" nominal temperature is shown on the temperature display, while key SET TEMP is pressed.

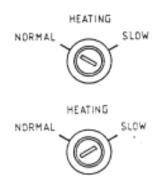
"Lower" nominal temperature for CO2 as transition liquid at a pressure greater 50 bar in the gas bottle is 10 C.

- Make necessary adjustment with potentiometer COOLING (lower) at the rear panel of the unit.

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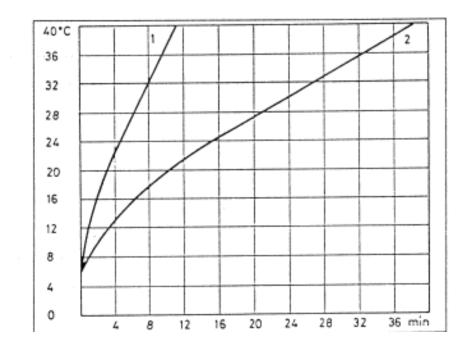
6.3.4.3. Selecting Warm-up Speed



Switch is located on rear panel of unit

The warm-up speed of the specimen pressure chamber can be selected with switch HEATING at the rear panel of the unit:

- Switch to position NORMAL: for normal sensitive specimens (Increase of temperature approx. 3.2°C/min)
- Switch position SLOW: for extreme sensitive specimens (Increase of temperature approx. 0.9°C/min)



Warm-up characteristics of the specimen pressure chamber:

1 Normal Speed

2 Slow Speed

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7 CPD 030 Maintenance

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This chapter is directed toward the technician who maintains CPD 030 critical point dryer and performs repairs.

Note: As a precondition the technician must be familiar with part Fundamentals and part Normal operation

Prior to opening the housing of the unit for maintenance work it is crucial to close gas bottle, disconnect the gas line and disconnect the mains cable.

If the housing of the unit has to be opened for maintenance work, first remove the lower sight mask after having loosened the three screws at the front of the bottom of the unit. Then the front plate has to be pushed out of the spring clips from below.

7.1. Basic Maintenance

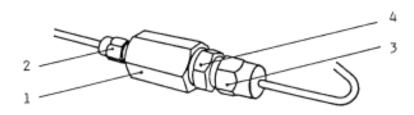
7.1.1. Screw-on Cover of Specimen Pressure Chamber

- Periodically check seal (B 8010 114 75) of screw-on cover and replace if necessary.

7.1.2. Poral Filter in Medium-Inlet

The poral filter (BU 011 546 -R) built into the medium inlet for the transition liquid MEDIUM IN has to be cleaned periodically in an ultrasonic bath. After closing the shut-off valve of the gas pressure bottle and removing the connecting hose (hex-wrench SW 17), the poral filter can be screwed out anti-clock wise with the "Lampenzieher" (B 4683 981 A1) which is part of the tool-set.

7.1.3. Poral Filter in Medium-Outlet



The poral filter (BU 011 546-R) built into the filter housing (1) of the outlet line of the specimen pressure chamber (see section



7.7.) has to be cleaned periodically in an ultrasonic bath. After loosening the screw joints (2) and (3) (hex-wrench SW 8 and SW 12) remove filter housing from outlet line. After loosening screw-joint (4) (hex-wrench SW 14 and SW 17), screw out poral filter from filter housing with the "Lampenzieher" mentioned in section above.

7.1.4. Pinhole Diaphragm in Medium-Inlet

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 Clean pinhole diaphragm (BU 016 994) installed in inlet ME-DIUM IN, if it has been plugged up with dirt. After removing the connecting hose and the poral filter (see section 5.2.) screw out pinhole diaphragm from medium inlet (screwdriver no. 4) and clean it in ultrasonic bath.

7.1.5. Pinhole Diaphragm in Medium-Outlet

Clean pinhole diaphragm (BU 015 541) installed in outlet ME-DIUM OUT if it has been plugged up with dirt and is no longer permeable for the liquid medium from the specimen pressure chamber. After removing the connecting hose (hex-wrench SW 14) from medium outlet, remove pinhole diaphragm and clean it in ultrasonic bath.

7.1.6. Checking Non-Return Valve

Periodically check whether there is no leak in the medium inlet- and outletsystem for the specimen pressure chamber.

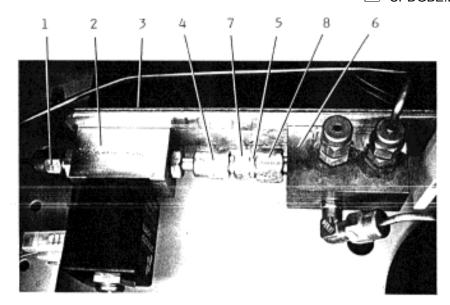
- Cool specimen pressure chamber to 10° C
- Fill with CO2 up to approx. 2 mm below the upper edge of the front sight glass.
- Warm up specimen pressure chamber

Level of liquid drops by a few mm due to physical reasons.

- After reaching the critical point, cool specimen pressure chamber again to 10° C.
- If there is no leak in the system, the liquid has to reach its original level again.

If this check shows that there is a leak in the system, it can be due to a leaking non-return valve.

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In order to remove the non-return valve, proceed as f011ows:

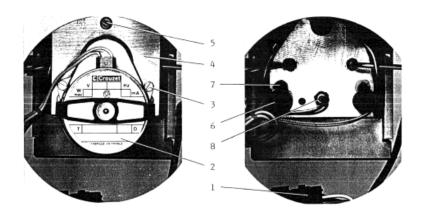
- Loosen screw joint (1) of medium inlet line with inlet valve (2) (hexwrench SW 12).
- Loosen fixing screws (3) of inlet valve (hex-wrench SW 8).
- Loosen screw joint (4) between inlet valve and non-return valve
 (5) (hexwrench SW 12).
- Screw out non-return valve from connecting block (6) (hex-wrench SW 14).
- Open non-return valve by loosening screw joint (7 and 8) (hex-wrench SW 12 and SW 14).
- Remove valve piston from non-return valve and check for damage or contamination.
- If the seal has been damaged, the complete non-return valve (BU 016 546) has to be replaced.
- Prior to refitting the new non-return valve, the two copper seals (B 4119 513 -K) built into the connecting block (6) have to be replaced.
- Reassemble in reverse order.

Further possibilities for a leak in the system, see section xxx

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7.1.8. Exchanging of Heating Cartridges

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If the preselected upper nominal temperature (40°C, if CO2 is being used) is not reached, one of the two heating cartridges, built into the specimen pressure chamber, can be defective.

- Check by measuring ohm-value of heating cartridges (25 ohm) or by examining for intact passage. For that, the connector plug (1) placed in front of the specimen pressure chamber inside the housing of the unit, has to be unpluged. The two heating cartridges are connected with the plug pins no. 6 and 7 and no. 5 and B respectively.

In order to replace a defective heating cartridge, proceed as f011ows:

- After loosening the two screws (3), remove the motor (2) of the magnetic stirrer.
- Remove the assembly plate (4) after loosening the three screws (5).
- Unscrew the three spacer elements (6).
- Pull defective heating cartridge (7) out of the specimen pressure chamber.
- Cut the connecting cable between defective heating cartridge and connector plug.
- Connect cable ends of connector plug and new heating cartridge (B 8010 055 27) with cable terminal.
- Coat new heating cartridge with heat-conductive paste (B 2753 420 -W) prior to inserting it.
- Reassemble parts in reverse order.

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7.1.9. Exchange of Temperature Sensor

Failure of the temperature display may be due to a disruption within the Pt 100 temperature sensor built into the specimen pressure chamber or in its connecting cable.

Check by measuring the ohm-value (approx. 120 ohm at room temperature) or by examining for intact passage. For that the connector plug (section 5.7. Pos. 1) placed in front of the specimen pressure chamber inside the housing of the unit has to be unpluged. The Pt 100 temperature sensor is connected to the plug pins no. 3 and 4.

To exchange the defective temperature sensor (section 5.7. Pos. 8) proceed as f011ows:

- Remove motor (2) of magnetic stirrer and assembly plate (4)
- Pull temperature sensor (8) out of the specimen pressure chamber after loosening the cable screw joint (hex-wrench SW 8).
- Cut connecting cable between defective temperature sensor and connector plug.
- Connect cable ends of connector plug and new temperature sensor (B 8010 115 63) by soldering.
- Coat temperature sensor with heat-conductive paste (B 2753 420-W) prior to inserting it.
- Reassemble parts in reverse order.

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7.2. TROUBLE SHOOTING

Problem	Cause	Remedy
After switching on mains switch, no function on unit is active.	Fuse F 1 and/or F 2 is,defective.	Disconnect power cable. open cover of fuse com partment (see section 1.6.) and replace defective fuse. Make sure correct line voltage is selected! Fuse values: for 220 V 4 AT for 115 V 5 AT
After switching on unit and pressing key COOLING, the cooling unit doesn't start up.	Fuse on power board of slide-in unit CP 010 is defective.	Disconnect power cable. Loosen screws of CP 010 slide-in unit, pull it out of unit and exchange fuse on power board. Fuse value: for 220 V and 115 V 4 AT
After switching on cooling unit temperature of specimen pressure chamber doesn't drop to pre-selected lower nominal temperature or possibly even rises slowly.	Cooling unit over- heated due to a high room temperature and switched off.	Make sure that room temperature is below 30°C.



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Problem	Cause	Remedy
	Cooling unit over- heated and switched off, because of insuf- ficient supply of cool- ing air.	Make sure minimum wall distance of 150 mm is maintained and ventillation slots on unit are not covered up.
	Cooling unit is leak- ing coolant and does not cooling effectively anymore.	Contact BAL-TEC supplier for repair of cooling device (refrigerator).
Temperature display remains at constant value, despite of COOLING or HEAT- ING	Temperature sensor may be disconnected or defective.	Replace sensor and connection cable (see spares list)
Bursting pane, built in for safety, breaks.	Permissable pressure of 150 bar has been exceeded, because too much transition liquid has been filled in and warmed up.	Open housing of unit. Unscrew holder of bursting pane and re- place pane (BU 011 980). Red side of pane must be on the outside.



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Problem	Cause	Remedy
MEDIUM IN valve is	Shut-off valve of gas	Open valve.
open, but no transi-	bottle is still closed.	
tion liquid flows into		
specimen prepara-	Gas bottle without a	Use bottle with
tion chamber	standpipe (riser) has been connected!	standpipe, otherwise only gaseous medium can be obtained.
	Diaphragm in ME- DIUM IN line has been clogged up.	F011ow procedure in maintenance section to exchange diaphragm
	Temperature in specimen chamber is too high.	Temperature has to be below 10°C before filling in the liquid transition medium.
MEDIUM OUT valve is open, but transi- tion liquid does not flow out of specimen preparation cham- ber	Diaphragm in ME- DIUM OUT line has been clogged up.	F011ow procedure in maintenance section to exchange diaphragm
GAS OUT valve is open, but pressure does not drop in specimen preparation chamber	Manually operated gas dosing valve is closed.	Open gas dosing valve ccw as describe in section basic operation.
	Poral filter in outline is clogged up.	F011ow procedure in maintenance section.

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Problem	Cause	Remedy
Level of transition liquid recedes con- siderably (small drop of level is nor- mal), when speci- men preparation chamber is warmed	Pipe connections are not screwed tightly.	Verify that connections are tight. Check leak tightness by applying soap water on a brush to the connections.
up.	Non return valve in inlet line is not tight due to a defective seal or contamination.	F011ow procedure in maintenance section for repair.
	Valve MEDIUM OUT is not tight due to a defective seal, damage on valve piston or contamination on seal surface.	

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Problem	Cause	Remedy
Critical pressure is not reached when chamber is warmed up.	Upper temperature has not been reached.	Make sure HEATING is on, wait until temperature reaches 31°C. If temperature does
		not rise, check heater cartridges (Mainte- nance section)
	Screw-on cover is not tight.	Verify that cover is screwed on tightly.
	Seal on screw-on cover is defective.	Check seal, change, if any damage can be observed.
	Seal on sight glass is defective.	Check seal, change if any damage can be observed.
	Non-return valve leaks	F011ow procedure specified above.
	MEDIUM OUT valve leaks	F011ow procedure specified above.
	GAS OUT valve leaks	F011ow procedure specified above.
	Tube fittings are leaking.	F011ow procedure specified above.

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8 Appendix

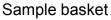
8.1 Spare parts, accessories and consumables



Specimen Container with 6 chambers 0 10 x 14 mm and 3 chambers 4 x 6 x 14 mm to accept up to 9 different specimens. All specimen chambers can be closed with a common cover of fine wire mesh. For suspensions, filter paper can be used instead of the wire mesh.

To reduce the quantity of transition liquid used for the media exchange those specimen chambers that are not being used can be filled with Al-inserts 0 9,8 x 12 mm (BU 011 555).

Order No. BU 011 127-T



 \emptyset 16 mm, height 12 mm, made of stainless steel, with screw-on cover of fine wire mesh.

Order No. B 8010 170 37



Sample basket as above, but \emptyset 23 mm, height 12 mm Order No. B 8010 170 38.

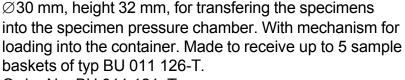
Sample basket



 \varnothing 10 mm, height 21 mm, made of stainless steel, with snap-on lid of fine wire mesh.

Order No. BU 011 126-T

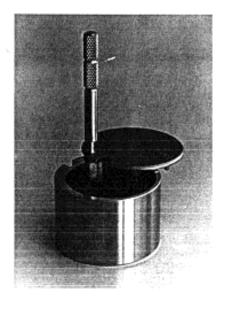
Specimen transfer container



Order No. BU 011 121-T



397-LEC (3)



Specimen container

with insert for 8 cover glasses \emptyset 18 mm. The insert can be exchanged with one for 9 cover glasses \emptyset 12 mm or for 7 cover glasses 22 x 22 mm. The cover allows the transfer of the specimens in the dehydration liquid (e.g. aceton) in spite of a fine wire mesh at the bottom of the container.

Order No. BU 011 128 -T



Insert for specimen container for 9 cover glasses Ø12 mm, Order No. BU 016 775 -T

for 8 cover glasses Ø18 mm, Order No. BU 016 776 -T

for 7 cover glasses 22x22 mm, Order No. BU 016 777 -T



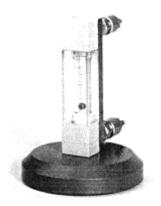
Specimen holder

for 6 specimen grids \emptyset 2,3 mm, suitable for drying cell suspensions deposited on coated grids. Order No. BU 011 122 -T

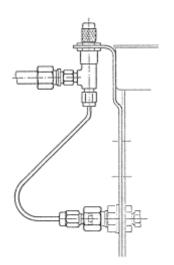
Specimen holder as above, but for specimen grids $\emptyset 3$ mm. Order No. BU 011 123 -T

CFD 030 GBL





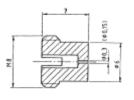
Flow meter with connecting hose to the gas outlet GAS OUT of the CPD 030 critical point dryer; for the contr011ed outlet of the gaseous transition medium after the critical point drying process Order No. BU 011 102 -T

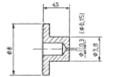


Dosing valve complete

with mounting bracket and connecting parts for adding it to the media inlet. For a contr011ed inlet of the transition medium if extremely sensitive specimens have to be critical-pointdryed.

Mounting of this accessory is possible without changes of the unit. Order No. BU 011 129 -T





Pinhole diaphragm for media inlet

for screwing into the medium inlet line.

Opening of 150 um, Order No. BU 016 994 (already built into basic unit)

Opening of 300 um, Order No. BU 016 995

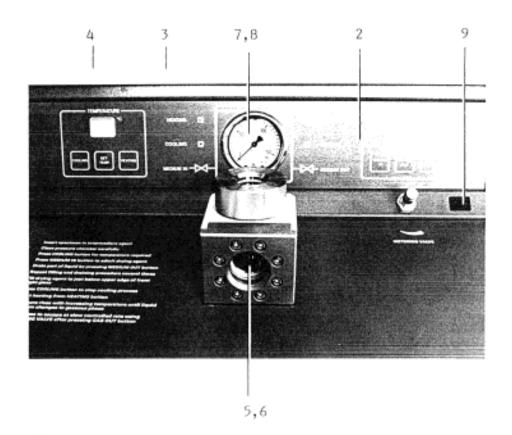
Pinhole diaphragm for media outlet for inserting into medium outlet line.

Opening of 150 um, Order No. BU 015 541 (already built into basic unit)

Opening of 300/um, Order No. BU 015 543



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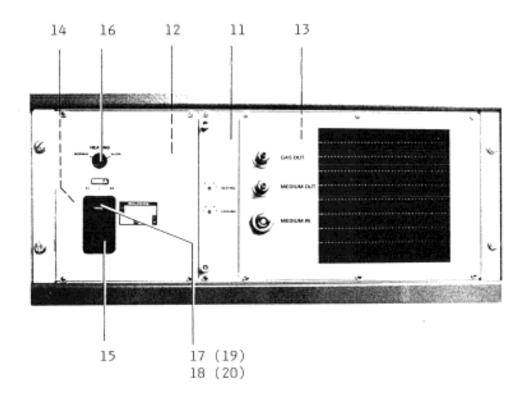
	Description	Item	Order No.
1	Electrical part	1	BU B00 130 E/2
1	Operating and display board	2	BU 005 072-T
1	Cooling unit	3	B 8010 117 45
1	Ventilator, 24 V DC	4	B 8010 119 14
1	Specimen pressure chamber I.	5	BU 800 130 E/3
1	Supply and drain line system	6	BU 800 130 E/5
1	Pressure gauge, 160 bar	7	B 8010 115 57
1	Seal Cu	8	B 8010 115 58

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ELECTRICAL PART

	Description	Item	Order nr.	
	CP 010 power board	11	BU 005 094 -T	
1	Connection board	12	BU 005 074 -T	
1	Ribbon cable, 20-pol.	13	BU 005 668 -T	
1	Trafo, 165 VA	14	B 8010 055 13	
1	Filter, 4 A/250 V	15	B 8010 055 14	
1	Switch	16	B 8010 055 81	
2	Fuse, 5 x 20, 4 AT	17	B 4666 448	(for 220 V)
2	Fuse holder, 5 x 20 mm	18	B 8010 055 B0	(for 220 V)
2	Fuse, 6,3 x 32,5 AT	19	B 4666 550	(for 115 V)
2	Fuse holder, 6,3 x 32 mm	20	B 8010 055 78	(for 115 V)
1	Mains cable, 1,5 m	21	B 8010 054 87	

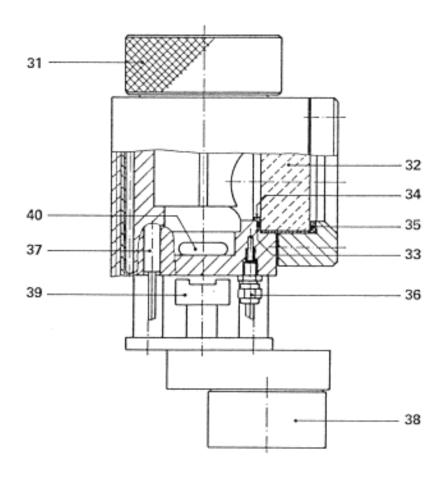


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SPECIMEN PRESSURE CHAMBER

	Description	Item	Order nr.
1	Cover compl. 31		BU 800 130 EA
1	Sight glass, Ø40 x 20 mm	32	BU 015 435-X
1	Seal	33	BU 011 799
1	Support ring	34	BU 011 798
1	Thrust washer	35	BU 011 912
1	Resistor, PT 100	36	B 8010 115 63
2	Heating cartridge	37	B 801.0 055 27
1	Motor, 24 V, 50 Hz	38	B 8010 114 04
1	Magnet, 14-22-16	39	B 8010 114 66
1	Stirrer rod, 5 x 20 mm	40	B 8010 114 76

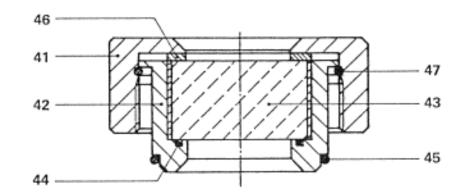


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SCREW-ON COVER

Description	Item	Order nr.
Cover	41	BU 011 413
	• •	
Sealing ring holder	42	BU 011 407
Sight glass, Ø34,5 x 20 mm	43	BU 015 434-X
O-Ring, Ø28,3 x 1,7B mm	44	B 8010 114 74
O-Ring, Ø37,82 x 1,78 mm	45	B 8010 114 75
Thrust washer	46	BU 011 405
Retaining ring	47	B 8010 115 4B





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