Accessories

UV VIS Spectrophotometer
SPECORD® 200 / 205 / 210 / 250

Analytik Jena AG
Kundendienst
Konrad-Zuse-Str. 1
07745 Jena

Phone:	Hotline:	+ 49 (3641) 77-7407
	Fax:	+ 49 (3641) 77-7449
e-mail:	service@analytik-jena.de	

General information about **Analytik Jena AG**
on the internet: http://www.analytik-jena.de

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Publication No.: 221:426.23

Edition – July 2005

Technical documentation made by:
Analytik Jena AG

This documentation describes the state of this product at the time of publishing. It need not necessarily agree with future versions of the product.

Subject to change!

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Contents

1 Introduction

This manual contains the description of the following devices

- SPECORD® 200, from S/N 222 U (or V W X) 100
- SPECORD® 205
- SPECORD® 210
- SPECORD® 250.

If the descriptions equally refer to all three models, only the collective term SPECORD® will be used.

In this manual, the following symbols are used to refer you to warnings and notes:

Danger!
Danger messages must be strictly observed to prevent personal injury.

Caution!
Caution messages refer you to procedures which, if not observed, could result in damage to the equipment.

Warning! Dangerous electric voltage!

Warning! Emission of UV radiation!

Disconnect power cable!

Note
This note must be followed to obtain correct measurement results.

For easier navigation within the manual, the manual uses the following system:

- Chapters and illustrations are numbered consecutively.
- Every illustration has its own caption.
- Steps of operation are consecutively numbered.
- Cross-references are marked by an arrow, e.g. → Section "Introduction" p. 1.

1.1 Safety notes

Handling of the accessories is subject to the safety instructions given for the SPECORD® basic unit (→ User's Manual SPECORD®, Section Safety Notes).

For all accessories of the SPECORD® Series, the following safety instructions are applicable:

Intended use!

The SPECORD®, including original accessories, may only be used for the applications described in this manual. The manufacturer cannot assume any liability for any other application, including that of individual modules or single parts. This also applies to all service or repair work that is not carried out by authorized service personnel. All warranty claims shall be forfeited.

Transportation and storage!

Accessories not in use should always be stored in the provided packaging!

For transportation, always use the provided original packaging!

Risk of corrosion!

Do not set up the device near aggressive vapors, e.g. strongly corrosive acid or caustic vapors! The vapors might corrode the connections, mechanical and optical components of the device.

Avoid exposing the sample compartment and the accessories to strongly corrosive substances! For the analysis of such samples, use vapor-tight stoppered cells.

Observe specific instructions!

Accessory-specific instructions are given in the chapter describing its use.

Caution! Contamination!

Never touch any optical mirrors of accessory units!

Additionally, observe the operating instructions and safety notes of any supplied system components of other manufacturers (e.g. fluid thermostats

1.2 Care of accessories

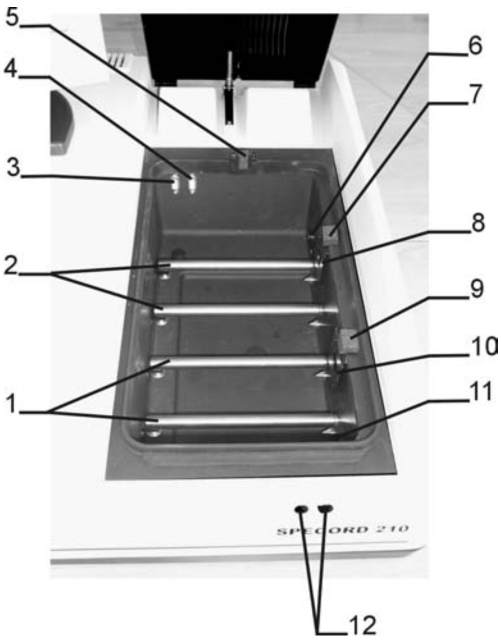
The accessories are largely maintenance-free.

The following rules apply to all accessories:

- Avoid any contamination by handling sample substances carefully.
- Wipe off spilt samples or reagents immediately with an absorbent cloth or cleaning tissue.
- Do not expose accessories to corrosive atmospheres to prevent corrosion.
- If provided, observe the additional instructions on the care of individual accessory units.

1.3 Basics of SPECORD alignment

1.3.1 Sample compartment of SPECORD®



1	Sample beam path with two carrying rails
2	Reference beam path with two carrying rails
3	Connector for sipper and cell changer
4	Connector (reserve)
5	Stopper for duct of optical fiber of measuring probe or electrical cables
6	Duct for coolant tubes
7	Receptacle for cells with turbid samples
8	Mount for cell holder
9	Receptacle for cells with turbid samples
10	Mount for cell holder
11	Duct for drain tube (not visible)
12	Ducts for sample-aspirating tubes (sipper)

Fig. 1-1 Sample compartment

Sample (1) and reference beam (2) enter the sample compartment through the left-hand wall.

The mounts for the cell holders (8), (10) are arranged in front of the exit windows in the right-hand sample compartment wall.

The two pairs of carrying rails serve to accommodate the accessories, which easily clamp to the rods.

The receptacles for the cells (7), (9) with turbid samples are located directly in front of the detector at the right-hand sample-compartment wall.

Two 9-pin connectors (3), (4) allow the connection of electrically driven accessories, such as the automatic temperature-controlled cell changer or the sipper system.

The optical fibers of the measuring probe or the cables of the Peltier temperature-controlled cell holder or the cell changer are threaded through the recess in the rear wall of the sample compartment (5).

The three ducts (6,11) in the right-hand sample compartment wall are used for threading through the sample drain tube and the coolant tubes for the thermostat.

The ducts in the front side of the device cover (12) are provided for threading through the aspirating tubes of the sipper system.

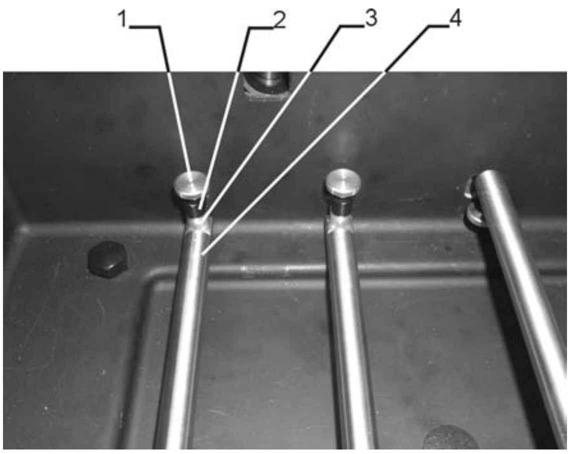
1.3.2 Adjustment of zeroth order

In zeroth order adjustment, white, undispersed light passes through the sample compartment. This high-intensity beam is particularly suitable for the alignment of accessories.

Open the Service Check utility of WinASPECT® software by following this procedure:

1. On the Windows taskbar, click the **Start** button. In the **Programs** folder, open the WinASPECT folder. There, click on **Service xxx**. This will bring up the **Specord xxx - Service Check** dialog box.
2. In this dialog box, click on the **Zeroth Order** button and choose the halogen lamp **(HL – Lamp)**.

1.3.3 Changing the position of the carrying rails



1	Knurled screw	3	Adapter
2	Support in sample compartment wall	4	Carrying rail

Fig. 1-2 Carrying rails mounted in bottom position

To use the cell carousel, you have to mount the carrying rails in the bottom position. For that, you need two adapters, which are included in the equipment of the cell carousel.

1. Unscrew one of the carrying rails in the sample beam path (front pair) and remove the rod from the sample compartment.
2. Screw one adapter (3) onto the carrying rail.
3. Put the carrying rail onto the bottom, cone-shaped journal in the right-hand sample compartment wall.

4. Hold the carrying rail with adapter from the bottom against the support (2) and fasten the carrying rail by means of the knurled screw. Fasten the knurled screw hand-tight so that the carrying rail cannot be moved.
5. Repeat the above procedure for the second carrying rail.

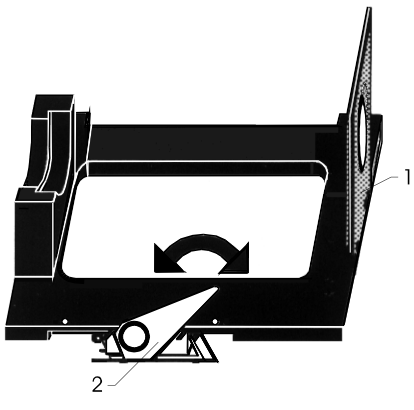
2 Universal holder

2.1 Description and use

The universal holder accommodates the following accessory units:

- Holder for 100 mm cylindrical cell
- Holder for cells of up to 50 mm pathlength
- Thermostatted cell holder for accommodation and temperature control of a cell of up to 10 mm pathlength (may be fitted with magnetic stirrer)
- Thermostatted cell holder for accommodation and temperature control of a cell of up to 50 mm pathlength
- Solid sample holder for accommodation of films and sample plates of up to 25 mm pathlength and a minimum diameter of ≥ 20 mm
- Holder for round cells
- Holder for semi-microcells and microcells

2.2 Description



1 Mounting plate for cell or solid sample holders
2 Clamp lever

Fig. 2-1 Universal holder

2.3 Using the universal holder

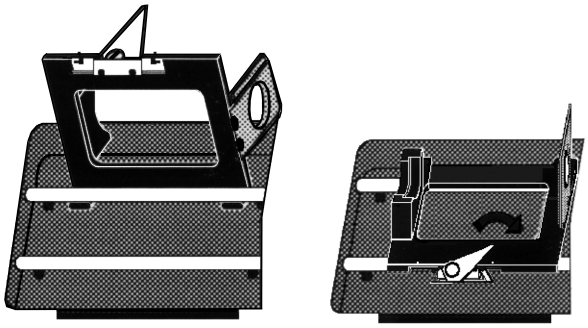


Fig. 2-2 Attaching and clamping the universal holder

1. Put the universal holder with its hook onto the rear of the carrying rails allocated to sample or reference channel. Then, slide it to the right-hand wall of the sample compartment as far as it will go.
2. Throw clamp lever (2) to the right to clamp the universal holder.
3. After you have attached and clamped the universal holder, slide the cell holder or the solid sample holder onto its mounting plate.

Note

It is necessary to position the universal holder at the right-hand wall of the sample compartment if the inserted sample shall be analyzed in the position where the beam cross-section is minimal.
This position ensures optimum signal-to-noise ratio.

If you want to arrange the sample in a different position, you can slide the universal holder to another place on the carrying rail and clamp it there.

You may also mount it inversely so that the plate (1) that accommodates the cell or solid sample holder is on the left-hand side.

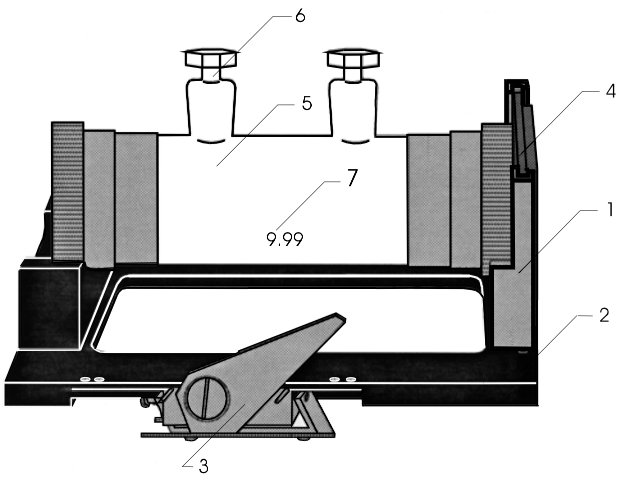
3 100 mm cylindrical cell (absorption tube)

3.1 Description and use

The cylindrical cell serves for measuring liquid samples requiring a pathlength of 100 mm. The effective pathlength of the cell (in cm) is engraved to the cell body.

The 100mm cylindrical cell comes with a special holder (1) for cylindrical cells.

3.2 Description



1	Holder for cylindrical cell	5	100mm cylindrical cell
2	Universal holder	6	Filling port with stopper
3	Clamping lever	7	Effective pathlength of the cell
4	Mounting plate of universal holder		

Fig. 3-1 100 mm cylindrical cell

3.3 Inserting the 100mm cylindrical cell

1. Put the universal holder (2) with its hook onto the rear of the two front carrying rails. Slide it to the right-hand wall of the sample compartment as far as it will go.
2. Throw clamping lever (3) over to the right to clamp the universal holder.
3. Slide holder (1) for cylindrical cells from top onto mounting plate (4) of the universal holder.
4. Insert the 100mm cylindrical cell (5) flush right in holder (1) for cylindrical cells and slide the complete universal holder (2) assembly to the right-hand sample compartment wall.

3.4 Care and transportation of cylindrical cell

Store and carry the cylindrical cell only in the original packing.

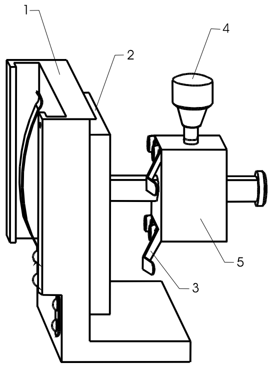
Clean the cylindrical cell after every use.

4 Solid sample holder

4.1 Description and use

The holder is suited for solids having a minimum diameter of 20 mm and a maximum pathlength of 25 mm.

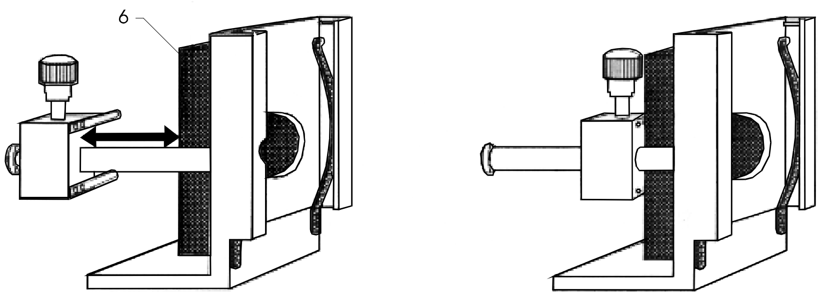
The size of samples should not exceed 80 mm x 140 mm.



1	Guide
2	Mechanical stop
3	Spring clip
4	Knurled screw
5	Support block

Fig. 4-1 Solid sample holder

4.2 Using the holder and inserting the sample



6 Sample

Fig. 4-2 Solid sample in holder slide

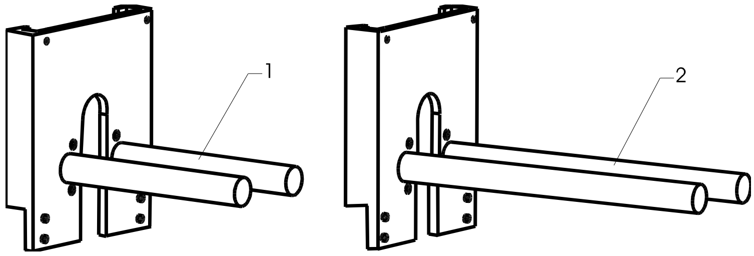
1. Slide the solid sample holder with its guide from top onto the mounting plate (Fig. 1-1 / 10).
2. Loosen knurled screw (4) and push support block (5) to the left stop.
3. Insert sample (6) into holder so that it rests on mechanical stop (2).
4. Push support block (5) towards the sample so that spring clips (3) safely hold the sample.
5. Fasten knurled screw (4).

5 Holder for cylindrical cells

5.1 Description and use

These holders serve for the accommodation of cylindrical cells without and with temperature control (thermocell) having an outside diameter of 22 mm and a length of up to 100 mm.

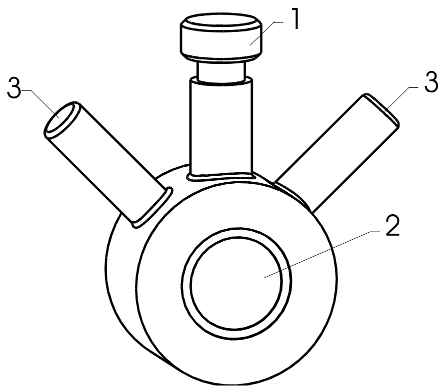
5.2 Description



1 Support rods for cells of up to 50 mm length
2 Support rods for cells of up to 100 mm length

Fig. 5-1 Holders for cylindrical cells

5.3 Using the holder for cylindrical cells



1 Fill-in port
2 Window
3 Connectors for thermostatting tubes

Fig. 5-2 10 mm thermocell (cylindrical)

Slide the holders from to onto the mounting plate for cell holders (Fig. 1-1 / 10).

Put the cylindrical cell onto the support rods. If the thermostatted cell is being used, the thermostatting tubes must be threaded outside the sample compartment through the provided holes in the same way as with the thermostatted cell holder (→ see Section “Thermostatted cell holder”).

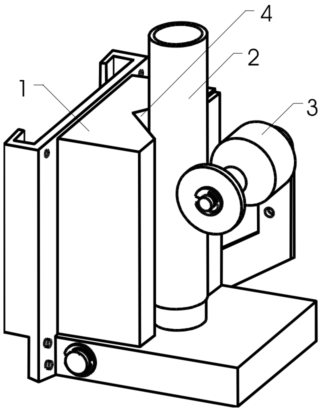
6 Holder for round cells

6.1 Description and use

This holder accommodates round cells or ampoules (cell test) or test tubes.

Cell diameter	12 mm to 20 mm
Cell height	40 mm to 70 mm
Minimum filling level	20 mm

6.2 Description



1	Support
2	Round cell
3	Spring rocker
4	Vee-guide

Fig. 6-1 Holder for round cells

6.3 Using the holder for round cells

1. Plug the holder for round cells from top onto the mounting plate for cell holders (User's Manual, Instrument Description, Fig. 8, Item 4) in the front beam path.
2. Carefully withdraw the spring rocker (3).
3. Slide the round cell (2) down along the vee-guide (4) between support and spring rocker.
4. Carefully let spring rocker return to hold the cell.

Note

Some manufacturers of cell tests provide round cells with a line mark. Position round cells of this type in such a way that the line mark is aligned to the sample beam axis.

If this mark is missing, take several measurements and turn the cell between the measurements each to avoid pathlength errors in high-precision measurements.

7 Holder for semi-microcells and microcells

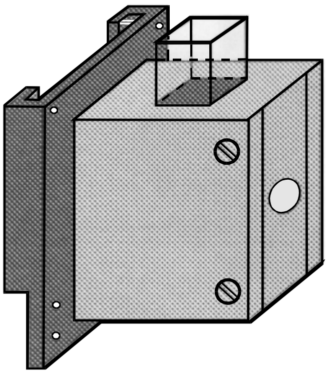
7.1 Description and use

Semi-micro and microcells have the standard outside width of 12.5 mm, but a reduced inside width or filling aperture of 1 to 4 mm. This requires accurate alignment of the cell to the sample and, if necessary, to the reference beam of the SPECORD®.

Three cell holders are available that meet this requirement for precise alignment to the beam:

1. The non-adjustable holder with a fixed mask of 2.5 mm diameter for semi-microcells of glass or quartz and a beam height of 8.5 mm.
2. The non-thermostatted holder for glass or quartz microcells with black sidewalls. This holder is adjustable both vertically and at right angles to the beam axis. The beam heights possible are 8.5 mm and 15 mm.
3. The non-thermostatted holder for glass or quartz microcells with black sidewalls. This holder is adjustable at right angles to the beam axis. The beam height is 8.5 mm.

7.2 Holder for semi-microcells

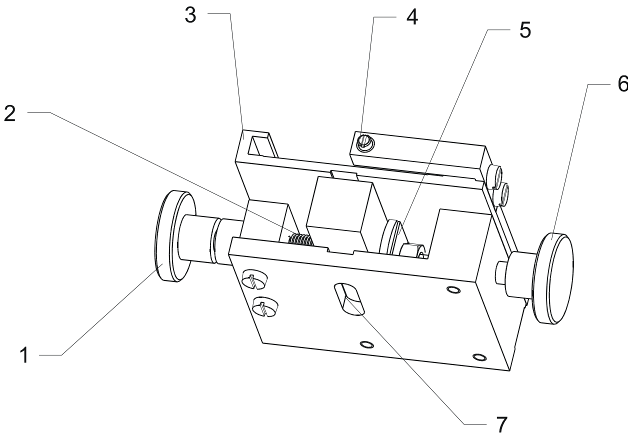


Pathlength of semi-microcell	max.	10	mm
Height of beam center	8.5	mm	
External width of cell	12.5	mm	
Minimum filling level	15	mm	

Fig. 7-1 Holder for semi-microcells

1. Slip this holder onto the mount for cells (Fig. 1-1/ 10) or the universal holder (→ Section "Universal holder" p. 11) and push it down as far as it will go.
2. When you insert the semi-microcell, take care that the leaf spring definitely presses the cell against the left-hand wall.

7.3 Adjustable holder for microcells, non-thermostatted, (8.5 mm / 15 mm beam height)



1	Handle
2	Spring
3	Holder
4	Screw for vertical adjustment
5	Thrust pad
6	Knurled screw
7	Slit position

Fig. 7-2 Holder for microcells (adjustable) with beam heights of 8.5 mm and 15 mm (adjustable)

Technical Data

Pathlengths of microcell	up to 10 mm
Height of beam center	8.5 and 15 mm
Outside cell dimensions	12.5 x 12.6 x 45 mm³

Installing the cell holder in the SPECORD®

The cell holder contains a 6.5 mm high insert, which is to be inserted in an opening below the cell, when using cells of 8.5 mm beam height.

1. To insert a cell, pull out handle (1) a little bit and insert the cell from top. Shift thrust pad (5), if necessary, by turning knurled screw (6).
2. From top, slide down holder (3) onto the mount for cells (Fig. 1-1 / 10) as far as it will go.

Alignment of cell

When you use the holder for the first time or when it is misaligned, it is advisable to realign the cell to the beam path.

First, look at the passage of undispersed "white" light through the cell:

1. Adjust zeroth order in the beam path (→ Section "Adjustment of zeroth order", p. 8).
2. Look through the slit (7) to see how the light is incident on the cell aperture. Turn knurled screw (6) until the light appears centered on the cell aperture.

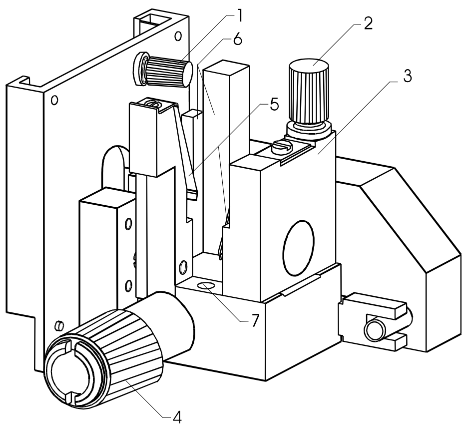
Fine adjustment is done by taking an energy measurement:

3. In the device driver (menu command: **Measurement / Set Parameters**), create a parameter record with e.g. the following parameters:

Wavelength:	500nm
Integration time:	0.1s
Slit:	2nm
Correction:	No
Cycle Mode: Manual:	20
Display:	Energy

4. Align the cell holder at right angles to the beam path by taking repeated measurements and turning knurled screw (6) each to shift the cell mount until you obtain the maximum energy.
5. Turn screw (4) to adjust the cell holder vertically until you obtain maximum energy.

7.4 Adjustable holder for microcells, non-thermostatted (8.5 mm beam height)



1 Fastening screw
2 Knurled screw for fastening the rider
3 Rider
4 Knurled screw for horizontal adjustment
5 Leaf springs
6 Cell guide
7 Locating holes

Fig. 7-3 Holder for microcells (adjustable)

Technical Data

Pathlengths of microcell	1, 2, 5, 10	mm
Height of beam center	8.5	mm
Width of cell	12.5	mm

Inserting the holder

1. Slightly unscrew knurled screw (1) on the holder to avoid jamming when you insert the holder.
2. Slip this holder onto the mount for cells or the universal holder and push it down as far as it will go. The universal holder must be moved as far as possible to the right-hand wall of the sample compartment.
3. Tighten knurled screw (1) to fix the holder in the optical path of the SPECORD®.
4. Adjust the rider (3) using knurled screw (2) to suit the pathlength of the cell to be used. For this, unscrew rider (3) completely by means of knurled screw (2) and

insert it in the appropriate locating hole (7). Then screw it down again using knurled screw (2).

5. Insert either an empty cell in the holder or a cell filled with solvent. Leaf spring (5) will press the cell against cell guide (6).
6. In the device driver (menu command: **Measurement / Set Parameters**), create a parameter record with e.g. the following measurement parameters:

Wavelength:	500nm
Integration time:	0.1s
Slit:	2nm
Correction:	No
Cycle Mode: Manual:	20
Display:	Energy

7. Adjust the cell holder transversely to the beam by activating a transmission measurement program. Run repeated measurements and move the cell holder by turning knurled screw (4) until you obtain a maximum transmission value.

8 Thermostatted cell holder

8.1 Description and use

The thermostatted cell holder accommodates a 10mm pathlength cell of 12.5 x 12.5 x 45 (L x W x H in mm) with a minimum filling level of 25 mm. You may optionally insert a magnetic stirrer in its bottom plate.

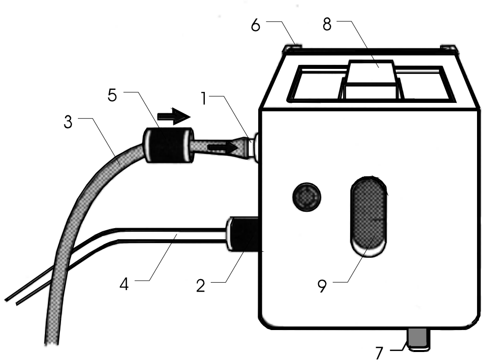
Temperature control is by an external fluid thermostat.
Observe the operating instructions provided with the thermostat!

The thermostatted cell holder is not suitable for the accommodation of HPLC cells (→ Section "Adjustable cell holder, thermostatted" p. 29).

You can place the thermostatted cell holder in the sample beam while working with the cell changer in the reference beam. Both devices may be operated from the same thermostat or cryostat. For this mode, a corresponding set of tubes is available.

You can insert flow cells in the thermostatted cell holder used in combination with the sipper system.

8.2 Description



1 Inlet connector
2 Outlet connector
3 Thermostatting tube (inlet)
4 Thermostatting tube (outlet)
5 Sleeve nut
6 Guide
7 Knurled screws for magnetic stirrer
8 Cell
9 Aperture

Fig. 8-1 Thermostatted cell holder

8.3 Using the thermostatted cell holder

8.3.1 Connection and installation in sample compartment

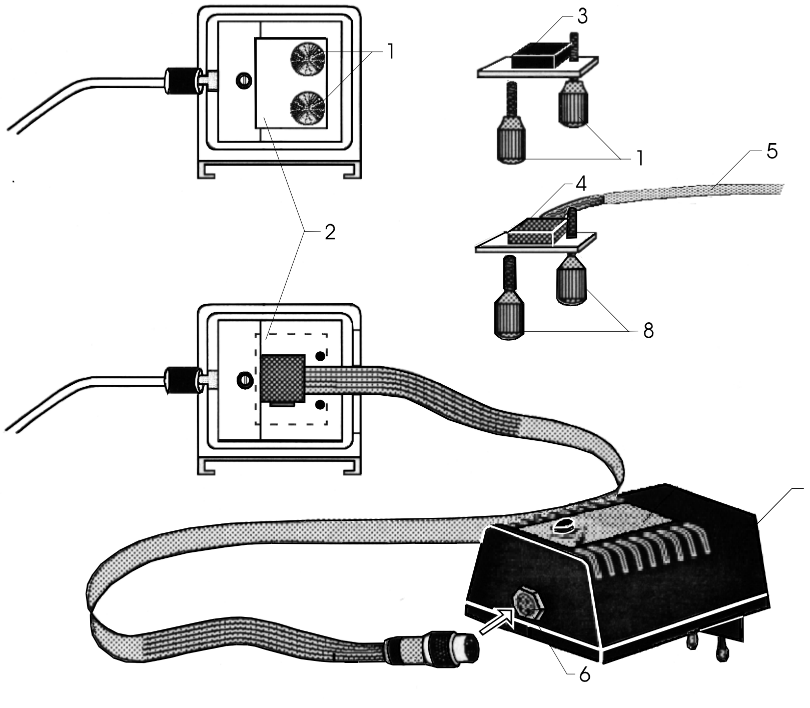
1. Thread the coolant tubes from the interior of the sample compartment through the tubes in the right-hand sample compartment wall of the SPECORD® (Fig. 1-1 / 6). The tube ducts in the wall are provided with steps to achieve a higher light-tightness. If the tube is caught on these steps, try to loosen it by slightly turning it. When it becomes visible in the outer opening, you can bend it towards the opening e.g. by means of a pen.

2. Connect the thermostatting tubes (3, 4) between the tube connectors (1, 2) of the thermostatted cell holder and the corresponding connectors on the external fluid thermostat. The tubes have an inside diameter of 4 mm and a wall thickness of 1mm. The tubes are transparent.
3. Slide the thermostatted cell holder with its guide (6) from top onto the mount for cell holders (Fig. 1-1 / 10) or the mounting plate of the universal holder.

8.3.2 Inserting the magnetic stirrer

Caution! Incorrect line voltage may damage the unit!

Make sure the operating voltage of the power supply unit (7) agrees with the available line voltage.



1	Knurled screws	5	Flat cable for connector
2	Bottom plate	6	Connector
3	Metal block	7	Power supply with speed control
4	Coil of magnetic stirrer	8	Knurled screws

Fig. 8-2 Inserting the magnetic stirrer coil

Attach the magnetic stirrer before you insert the cell holder in the sample compartment.

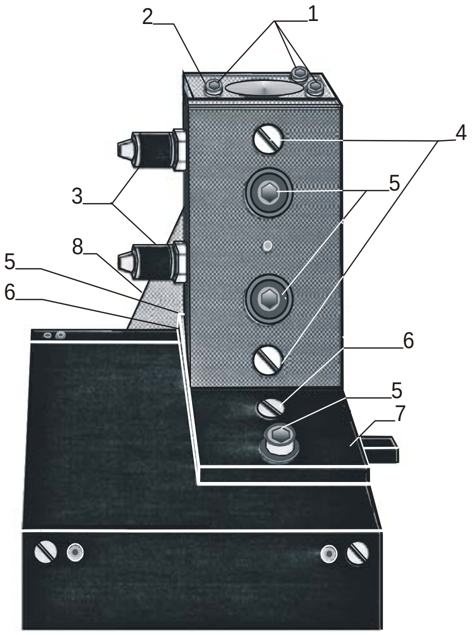
1. Loosen knurled screws (1) on the underside of the thermostatted cell holder and remove bottom plate (2).
2. Remove metal block (3).
3. Put the coil of the magnetic stirrer (4) into the square opening at the bottom of the thermostatted cell holder taking care that the flat cable (5) is flush with the bottom plate.
4. Tighten knurled screws (8).

5. Without twisting the flat cable, thread it under the rear carrying rails and through the stopper in the rear wall of the sample compartment (Fig. 1-1 / 5).
6. Push plug and cable through under the sample compartment cover. Lay the cables in the groove in the SPECORD®. Take care that the sample compartment cover does not squeeze the cables.
7. Plug the plug of the flat cable (5) into the connector (6) of the power supply with speed control (7).
8. Connect the power supply to a power outlet.
9. Insert the thermostatted cell holder with magnetic stirrer exactly in the same way as the thermostatted cell holder without stirrer (see above).
10. Put a stirring magnet into the cell and insert the cell in the holder.
11. Turn the speed control knob to adjust the stirring frequency.

9 Adjustable cell holder, thermostatted

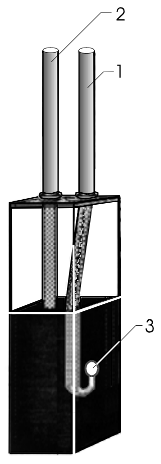
The adjustable thermostatted cell holder accommodates a cell of 10 mm pathlength and the following external dimensions: 12.5 x 12.5 x 35 (L x W x H in mm). The height of beam transmission is 15 mm.

The holder is designed for the accommodation of cells with very small window apertures (e.g. 1mm on HPLC cells). It provides defined location and hold in, and alignment to the beam. With such small apertures, even minimum misalignment of the cell may impair the signal-to-noise ratio.



1	Screws
2	Lid
3	Tube connector with sleeve nut
4	Screws for vertical adjustment
5	Screws to fasten the cell holder (4th screw obscured)
6	Screws for horizontal adjustment (2nd screw obscured)
7	Top part of adjustable cell holder
8	Lever

Fig. 9-1 Adjustable thermostatted cell holder



1	Sample feed
2	Sample aspiration
3	Beam aperture

Fig. 9-2 HPLC cell

9.1 Inserting the cell

1. Unscrew three screws (1) and remove lid (2).
2. Insert the cell and screw down lid (2) again.
3. Put the adjustable cell holder onto the two front carrying rails of the sample compartment (Fig. 1-1 / 1).
4. Push it to the right as far as it will go and clamp it by throwing over lever (8) to the left.

9.2 Aligning the cell

For rough orientation, it is advisable to observe the passage of the sample beam through the cell:

1. Adjust the zeroth order of the monochromator grating as described in → Section "Adjustment of zeroth order", p. 8. In this mode, undispersed "white" light is passing through the sample compartment, which can be observed very well.
2. You can check the illumination of the cell by means of a white screen (e.g. a strip of paper). Align the cell so that the sample beam is centered on the aperture of the cell.

Fine adjustment is done by taking an energy measurement:

3. In the device driver (menu command: **Measurement / Set Parameters**), create a parameter record with e.g. the following parameters:

Wavelength:	500nm
Integration time:	0.1s
Slit:	2nm
Correction:	No
Cycle Mode: Manual:	20
Display:	Energy

4. Loosen the four screws that fix the cell holder in position (5), until the top part of the adjustable cell holder (7) can be moved smoothly.
5. Successively, turn the screws for vertical adjustment (4) and the screws for horizontal adjustment (6).
6. Check the adjustment by taking an energy measurement. In these measurements, take care to always keep the sample compartment cover closed.
7. Repeat the adjustment procedure until the energy value of the sample beam I(M) has reached its maximum.
8. Secure the optimum adjustment by retightening the four screws that fix the cell holder in position (5).

10 6-cell changer, thermostatted

10.1 Description and use

The six-cell changer is an automatic sample changing system that is connected to and controlled through the SPECORD®. Temperature control is by means of a water thermostat.

It consists of two separable modules

- a thermostatted cell holder
- a driving unit with stepping motor drive.

The cell holder has six positions for the accommodation of 10-mm pathlength cells of the following outer dimensions: 12.5 x 12.5 x 45 (L x W x H in mm).

The cell holder is equipped with a quick-lock tube coupling allowing drip-free connection and disconnection of the thermostatting tubes.

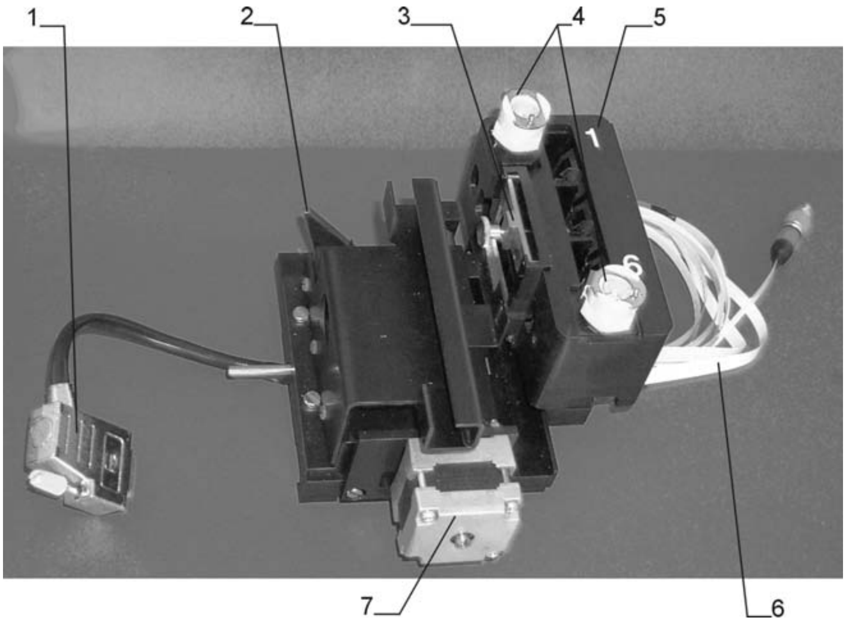
The stepping motor drive of the driving unit moves the cell holder within approx. 1 s to the next position. In combination with an optocoupler defining the initial and the end points of the motion range, it ensures exact and reproducible alignment of the cells to the sample beam.

Optional cell changer equipment

The 6-cell changer can be equipped optionally with magnetic stirrers.

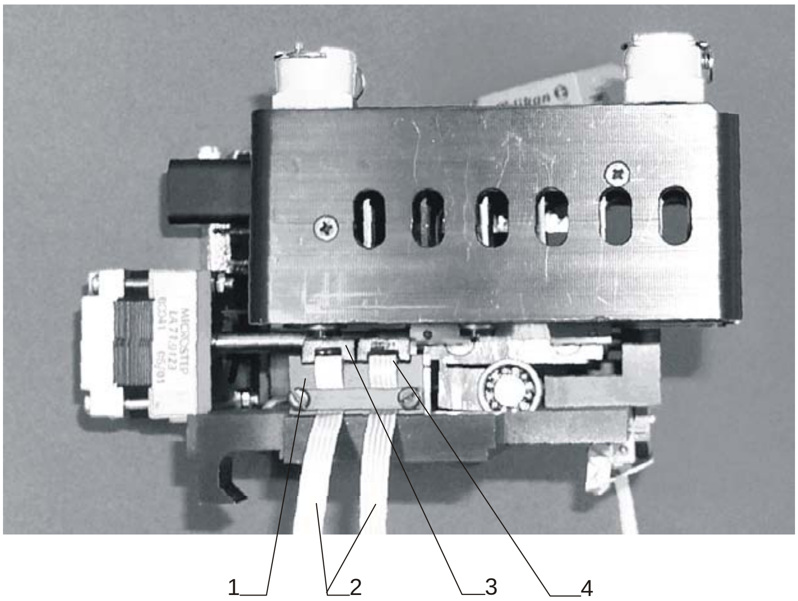
In addition, a cell changer is available, which is designed for the accommodation of cells of 1, 2 and 5cm pathlength. However, this cell changer neither can be thermostatted nor equipped with magnetic stirrers.

10.2 Design



1	Interface cable	5	Cell holder with thermostatting jacket
2	Clamping lever	6	Connector for magnetic stirrer
3	Support for cell holder	7	Driving unit with stepping motor
4	Quick-lock couplings for coolant inlet		

Fig. 10-1 Thermostatted cell holder and driving unit

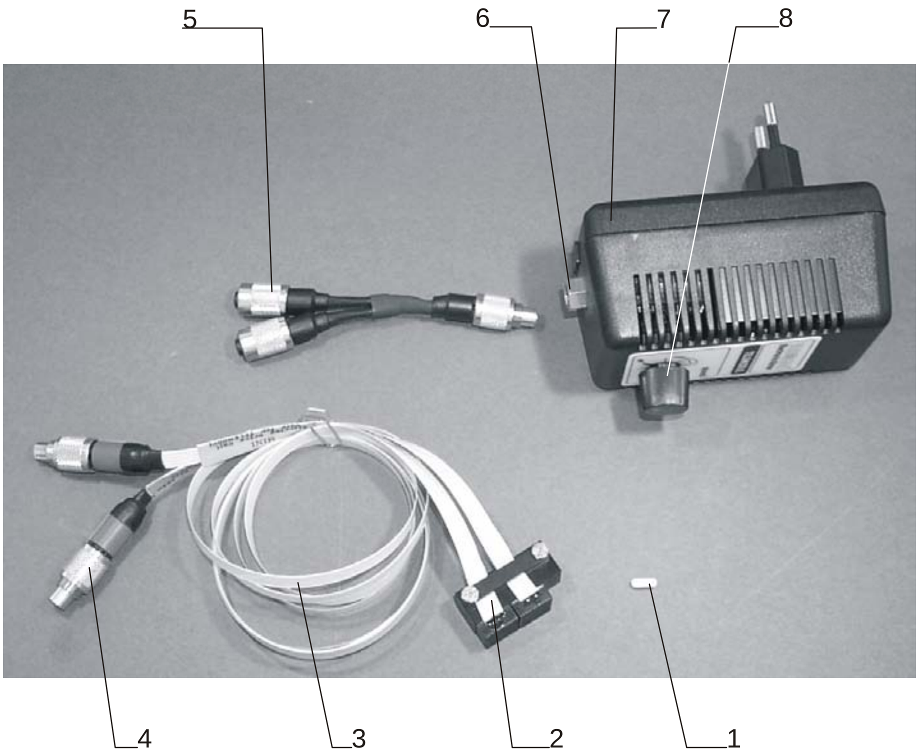


1	Holder for magnetic coils of stirrer	3	Magnetic coil under measuring position
2	Flat cable	4	Magnetic coil under the next position

Fig. 10-2 Thermostatted cell holder with magnetic stirrer

10.3 Installation and insertion in sample compartment

10.3.1 Installation of magnetic stirrers



1	Stirring magnet	5	Adapter
2	Carrier for stirrer coils	6	Connector on power supply
3	Flat cable	7	Power supply
4	Connectors of flat cable	8	Speed control

Fig. 10-3 Installation of magnetic stirrer

Caution! Incorrect line voltage may damage the unit!

Make sure the operating voltage of the power supply unit (7) agrees with the available line voltage.

The driving unit may optionally be fitted with a stirrer containing two magnets.

One magnet is located underneath the measuring place, the other one underneath the place before the measuring position.

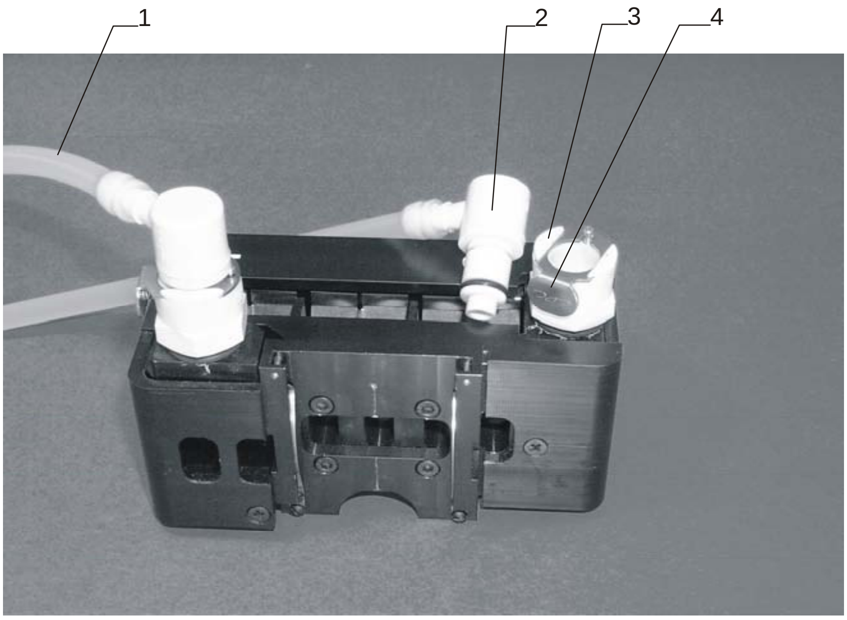
The magnetic stirrers are to be mounted outside the sample compartment.

1. Lift the cell holder off the driving unit.
2. Put the carrier (Fig. 10-3 / 1) for the stirrer coils onto the provided place (Fig. 10-2) and fasten it by means of the screws from the bottom.
3. Connect the connectors (Fig. 10-3 / 4) to the adapter (Fig. 10-3 / 5). Plug the adapter plug into the corresponding socket (Fig. 10-3 / 6) of the power supply (Fig. 10-3 / 7).
4. Reattach the cell holder to its support on the driving unit.

5. Put the stirring magnets (Fig. 10-3 / 1) into the cells.
6. Connect the power supply (Fig. 10-3 / 7) to a power outlet and turn the speed control knob (Fig. 10-3 / 8) to adjust the stirring speed.

The magnetic stirrer is operating.

10.3.2 Connection to a thermostat



1	Thermostatting tube	3	Female tube connector
2	Male tube connector	4	Locking lever

Fig. 10-4 Cell holder with quick-lock tube coupling

The cell holder is connected to an external fluid thermostat through tubes with quick-lock couplings. The cell holder is provided with female tube connectors.

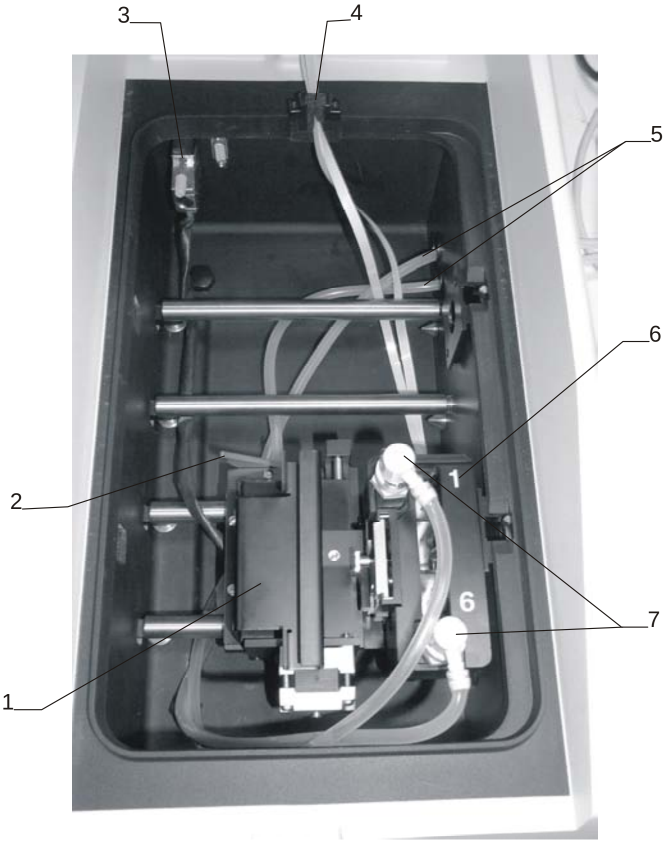
1. Connect the male connectors with the thermostatting tubes.
2. Push the male connectors into the female connectors until you can hear them clicking in.
3. For the connection to a fluid thermostat (after insertion in the SPECORD®), it may be necessary to use tube adapters to compensate for any differences in tube width.

Disconnecting the quick-lock tube couplings

4. Depress the locking lever (4) and remove the male connector (2) from the quick-lock tube coupling.

When you work with the device without cell changer, you may also leave the tubes with the quick-lock tube couplings in the tube ducts.

10.3.3 Installation in sample compartment



1	Driving unit	5	Ducts for thermostatting tubes
2	Clamping lever	6	Cell holder
3	Interface cable	7	Quick-lock tube couplings
4	Flat-cable duct with plastic cover		

Fig. 10-5 Installation of cell changer

The description below refers to the use of a thermostatted cell changer with magnetic stirrer. If you do not intend to install these components, skip the corresponding item.

Caution! Lay thermostatting tubes properly!

Lay the thermostatting tubes in the sample compartment so that they neither hinder the movement of the cell changer.

1. Lay the thermostatting tubes underneath the front and rear carrying rails. Thread the tubes through the provided tube ducts in the right-hand sample compartment wall (Fig. 10-5 / 6).
   The tube ducts in the wall are provided with steps to achieve a higher light-tightness. If the tube is caught on these steps, try to loosen it by slightly turning it. When it becomes visible in the outer opening, you can bend it towards the opening e.g. by means of a pen.
2. Replace the plastic foam stopper inserted in the recess of the sample compartment wall by the supplied plastic foam strips.

3. Thread the cable of the magnetic stirrer under the rear carrying rails and push it through between the two plastic foam strips in the recess in the sample compartment. In doing so, take care that the cover of the sample compartment is not hindered by projecting plastic foam when closing the cover and make sure it closes light-tight.
4. Thread the cable through underneath the cover of the sample compartment. Make sure the cable is not squeezed when the cover is closed.
5. Connect the ribbon cable to the power supply (Fig. 10-3 / 7) by means of the adapter (Fig. 10-3 / 5).
6. With the clamping lever pointing to the back, place the 6-cell changer onto the front carrying rails. Push the 6-cell changer to the right-hand sample compartment wall as far as it will go. Clamp it to the carrying rails by throwing the clamping lever to the left.
7. Thread the interface cable through underneath the rear carrying rails and connect ists plug to the **left** connector in the back wall of the sample compartment.

Adjustment of the cell changer

The cell changer can be adjusted computer-controlled to optimally position the cells in the beam.

Adjustment is necessary

- At the first use of the cell changer
- After wavelength calibration
- After relocation of the SPECORD®
- When working with microcells.

1. Install the empty cell changer in the sample compartment.
2. On the **Accessories** tab, activate the **6cell-changer** option.
3. If you intend to use standard cells (1cm x 1cm) or semi-microcells for the following analyses, perform the adjustment without any cells being inserted.
   If you intend to use microcells in the analyses, insert a microcell filled with water in every of the six positions.
4. Start automatic adjustment by a click on the **[Adjustment]** button.

10.4 Operation

10.4.1 Settings in Measurement Parameters window

The cell changer is controlled via WinASPECT® software. In the **device driver** dialog box (menu command: **Measurement / Set Parameters**), choose the **6-cell changer** option on the **Accessories** tab.

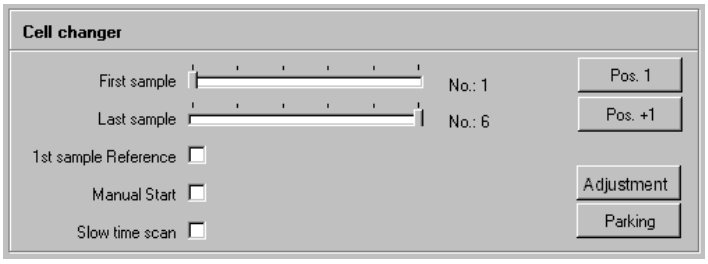


Fig. 10-6 Accessories tab – Cell changer

For the control of the cell changer via WinASPECT®, the following options are available:

Parameter / Button	Description
First sample	Position of the first sample on the cell changer.
Last sample	Position of the last sample on the cell changer.
1st sample reference	To be activated, if the first place on the cell changer holds the reference. In this case, it is not necessary to take a separate reference measurement. The reference measurement is then integrated in the normal sample measurement process.
Manual start	If activated, a small dialog box appears: "Start cycle [OK]". The actual sample measurement is started only after you confirmed the request. You can use this option for kinetic measurements. The measurement in the respective cell will then be started only after you added a starting substance.
Slow time scan	Activate this option, if in cyclic measurement mode you want to measure all samples on the cell changer successively first. Deactivate the Slow time scan option, if in cyclic measurements you want to measure all cycles of one sample first before the cell changer moves to the next cell position.
Interval	Accessible only, if the **slow time scan** option has been activated. The interval time is the time from the beginning of a cycle to the beginning of the next cycle. Hence, it also includes the time required for sample measurement. Enter the desired interval time in the textbox. From the list box beside, select the unit of time.
[Pos.1]	Cell changer moves to the first position.
[Pos. +1]	Cell changer moves to the next position.
[Adjustment]	Starts the adjustment of the cell changer.
[Parking]	Cell changer moves to the parking position with the cell block being centric above the base plate. In this position, the cell changer can be easily removed from and installed in the SPECORD® as well as packed.

10.4.2 Measurements with the 6-cell changer

Measurement without reference cell in cell changer

1. Place the reference cell onto the cell position in the beam path.
2. Start the reference measurement by a click on the **[Reference]** toolbar button or by activating menu command **Measurement / Reference**.
3. Load the cell changer beginning with the position selected as “First sample“.
   Place the other cells continuously onto the following cell positions up to the position defined as “Last sample“.
4. Start the sample measurement with the corresponding **[Start Measurement]** toolbar butto or menu command **Measurement / Measurement**.

Measurement with reference cell in cell changer

1. Place the cell containing the reference onto the position selected as “First sample“.
   Place the other cells continuously onto the following cell positions up to the position defined as "Laset sample".
2. Start the sample measurement with the corresponding **[Start Measurement]** toolbar butto or menu command **Measurement / Measurement**.

10.4.3 Operating modes of cell changers

Beside the normal operating mode, you can operate the cell changers in **Slow time scan** mode.

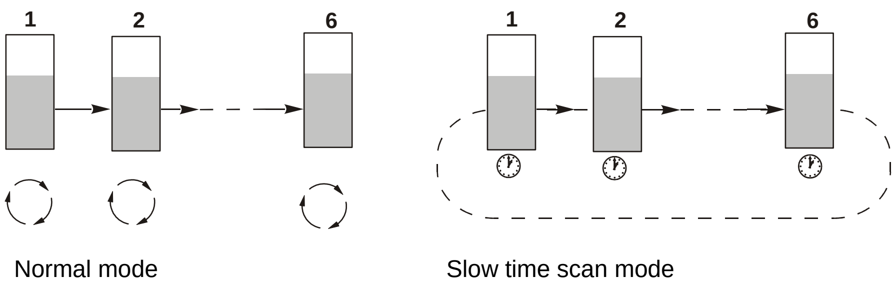


Fig. 10-7 Cell changer -Operating modes

In normal mode, first all cycles of a measurement are executed for the sample on a defined cell position, before the measurement cycles of the sample on the next position are started. For kinetic measurements, this means that the complete reaction-kinetic measurement is taken on one sample first, before the next kinetic reaction is started (requires **Manual start** check box to be activated!).

In **Slow time scan** mode, the reaction-kinetic measurement can be optimized by measuring the samples cyclically in a time-shared mode. This means that in this mode the samples are measured successively in every cycle beginning with the sample on position 1, before the next cycle is executed for all samples.

For the **Slow time scan** mode, observe the following notes regarding parameter selection:

Tab	Settings
Mode	Measuring mode: Time Scan In this mode, it is not possible to subdivide the total measuring time in several periods with different data points. Therefore, enter the total measuring time here. The entries in the **Data Points** text box will not be taken into account. Instead, enter the interval time on the Accessories tab.
Accessories	Activate the **Interval** check box and enter the desired interval time. If you fail to do so, the measurements will be taken at the fastest possible cycle time until the end of the total measuring time. Any activation of the **Manual start** check box will be disregarded. Instead, the measurement of the first sample begins after you confirmed the query "Start time scan?", whereas the measurement of all following samples will be started automatically.

11 8-cell changer

D This cell changer is an automatic sample changing system. The driving unit with stepper motor is controlled by WinASPECT® software.

The cell changer has eight cell positions for holding 10-mm pathlength cells of the following outer dimensions 12.5 x 12.5 x 45 (L x W x H in mm).

The stepper motor drive of the driving unit conveys the cell holder within approx. one second to the next position. Together with a reflection coupler that defines the initial point of the motion range, the stepper motor drive provides exactly reproducible alignment of the cells to the sample beam.

The 8-cell changer is optionally available with temperature control (via an external thermostat) and/or magnetic stirrer. Magnetic stirrer and thermostat connectors must be factory-fitted and cannot be retrofitted.

In addition, a Peltier temperature-controlled 8-cell changer is available (→ Section "Peltier temperature-controlled 8-cell changer", p. 79).

11.1 Removal of transport lock on non-temperature-controlled 8-cell changer

The non-temperature-controlled cell changer is protected in transport by a red plastic foam pad under the cell block.

Transportsicherung /
Transport lock

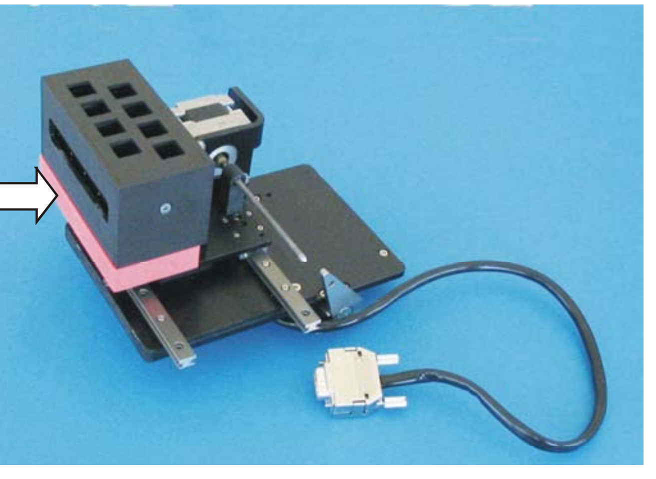
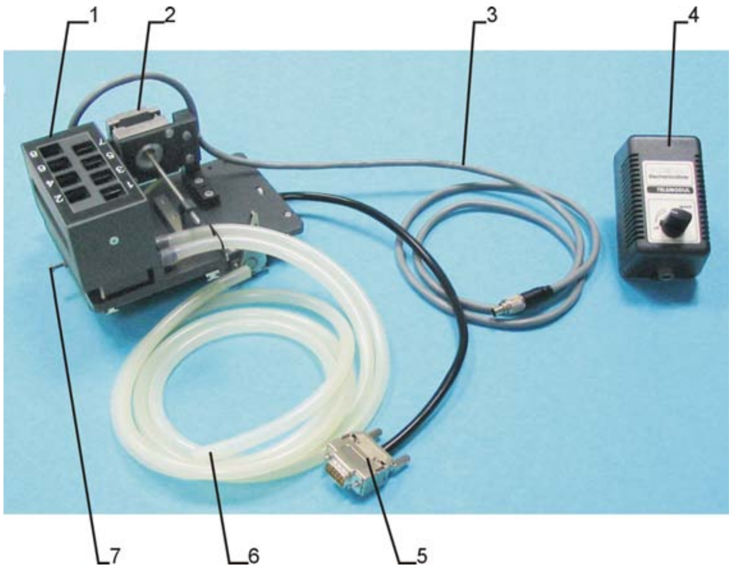


Fig. 11-1 Transport lock on non-temperature-controlled 8-cell changer

Make sure to remove the red plastic foam pad from underneath the cell block before using the cell changer the first time.

Replace this pad for any relocation of the cell changer.

11.2 Design of 8-cell changer



1 Cell block with insulation
2 Stepper motor drive
3 Connecting cable for connection to stirrer control unit
4 Stirrer control unit
5 Connecting cable for connection to SPECORD®
6 Water tubes for connection to thermostat

Fig. 11-2 8-cell changer

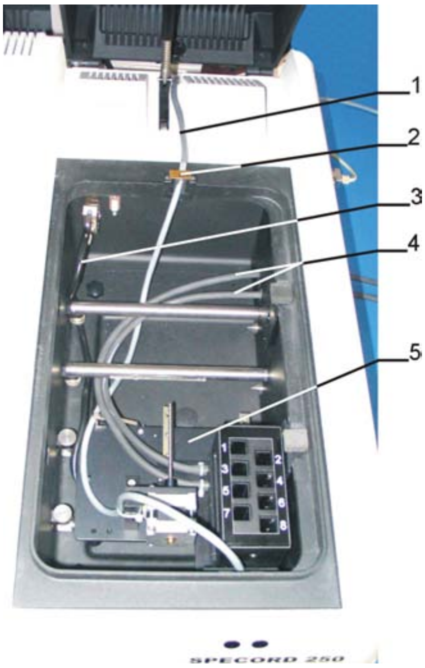
11.3 Installation of 8-cell changer

This section describes the installation of the 8-cell changer with connection to a fluid thermostat and a magnetic stirrer. If your cell changer does not have these additional components, you can skip this part of the description.

Caution! Lay cables and tubes properly!

Lay all connection cables and tubes for connection to SPECORD®, fluid thermostat and/or magnetic stirrer so that they do not project into the beam in the sample compartment.

Lay the connection tubes to the fluid thermostat tension-free to ensure freedom of motion of the cell block of the cell changer.



1 Connection cable for control unit of stirrer
2 Recess in sample compartment wall with plastic foam stopper
3 Connection to SPECORD®
4 Water tubes
5 8-cell changer

Fig. 11-1 8-cell changer installed in sample compartment of SPECORD®

1. Mount the carrying rails in their bottom position (→ Section "Changing the position of the carrying rails" p. 8).
2. Replace the plastic foam stopper (2) in the rear recess of the sample compartment by the plastic foam strips supplied along with the accessory.
3. Thread the cable of the magnetic stirrer under the rear carrying rails and push it through between the two plastic foam strips in the recess in the sample compartment. In doing so, take care that the cover of the sample compartment is not hindered by projecting plastic foam when closing the cover and make sure it closes light-tight.
3. Thread the cable through underneath the cover of the sample compartment. Make sure the cable is not squeezed when the cover is closed.
4. Connect the cable to the magnetic stirrer. Conect the control unit of the magnetic stirrer to the power outlet.
5. Thread the thermostat tubes through underneath the rear carrying rails. Thread the tubes through the provided ducts in the right-hand sample compartment wall (4). The ducts in the sample compartment wall are provided with steps to increase the light tightness. If the tube hooks to these steps when threading it through, try to release it by slightly turning it. When it becomes already visible at the other end of the duct, bend it to the opening e.g. by means of a pen.
6. Connect the tubes to a thermostat. We recommend using quick-lock couplings that allow drip-free connection and disconnection of the thermostat.
4. Put the cell changer with the clamping lever facing the back onto the relocated carrying rails. Push the cell changer to the righ-hand sample compartment wall as far as it will go. Clamp it to the carrying rails by throwing the clamping lever over to the left.

5. Thread the interface cable to the back underneath the rear carrying rails and connect the plug to the **left** connector on the back wall of the sample compartment.

Adjustment of the cell changer

The cell changer can be adjusted computer-controlled to optimally position the cells in the beam.

Adjustment is necessary

- At the first use of the cell changer
- After wavelength calibration
- After relocation of the SPECORD®.

1. Install the empty cell changer in the sample compartment.
2. On the **Accessories** tab, activate the 6cell-changer option.
3. If you intend to use standard cells (1cm x 1cm) or semi-microcells for the following analyses, perform the adjustment without any cells being inserted.
   If you intend to use microcells in the analyses, insert a microcell filled with water in every of the eight cell positions.
4. Start automatic adjustment by a click on the **[Adjustment]** button.

11.4 Operation of the 8-cell changer

11.4.1 Software settings

The cell changer is controlled by WinASPECT® software. On the **Accessories** tab of the measurement parameter window, choose the **8-cell changer** option.

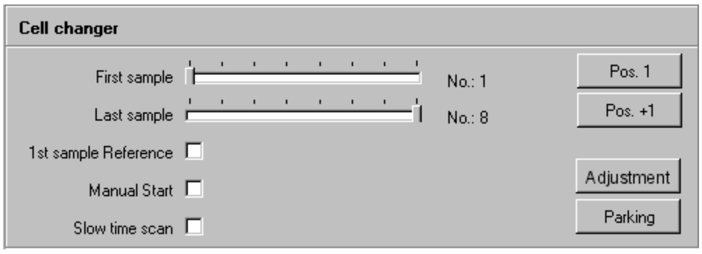


Fig. 11-2 8-Cell changer options for the SPECORD®

Parameter / Button	Description
First sample	Position of the first sample on the cell changer.
Last sample	Position of the last sample on the cell changer.
1st sample reference	To be activated, if the first place on the cell changer holds the reference. In this case, it is not necessary to take a separate reference measurement. The reference measurement is then integrated in the normal sample measurement process.
Start manually	If activated, a small dialog box appears: "Start cycle [OK]". The actual sample measurement is started only after you confirmed the

	request. You can use this option for kinetic measurements. The measurement in the respective cell will then be started only after you added a starting substance.
Slow time scan	Activate this option, if in cyclic measurement mode you want to measure all samples on the cell changer successively first. Deactivate the Slow time scan option, if in cyclic measurements you want to measure all cycles of one sample first before the cell changer moves to the next position.
Interval	Accessible only, if the **slow time scan** option has been activated. The interval time is the time from the beginning of a cycle to the beginning of the next cycle. Hence, it also includes the time required for sample measurement. Enter the desired interval time in the textbox. From the list box beside, select the unit of time.
[Pos.1]	Cell changer moves to the first position.
[Pos. +1]	Cell changer moves to the next position.
[Adjustment]	Starts the adjustment of the cell changer).
[Parking]	Cell changer moves to the parking position with the cell block being centric above the base plate. In this position, the cell changer can be easily removed from and installed in the SPECORD® as well as packed.

11.4.2 Measurements with the 8-cell changer

The measurement procedure with the 8-cell changer is analogous to that of the 6-cell changer (→ Section "

Measurements with the 6-cell changer", p. 38).

11.5 Use of two 8-cell changers

You can also use two 8-cell changers in combination. This way it is possible to analyze every sample against a specific reference (synchronous operation) or to increase the number of samples analyzed within a measurement series (staggered operation).

To this end, install the second cell changer analogously to the first one clamping it to the carrying rails (in bottom position) in the reference beam path (→ Section "Installation of 8-cell changer" p. 42).

Align the two 8-cell changers in the same way as a standard cell changer.

If the cell changers shall be temperature controlled by an external thermostat, connect the two cell changers with each other by a short piece of tube. Lay a connection tube each from every cell changer to the thermostat.

Caution! Ensure correct connections in sample compartment!

Connect the cable of the cell changer in the **rear reference beam path** to the **right-hand connector** in the sample compartment.
Connect the cable of the cell changer in the **front sample beam path** to the **left-hand connector**.

The software settings for the combined use of two 8-cell changers are to be selected on the **Accessories** tab, after having activated the **2 x 8-Cell changer** option.

The major functions available are the same as those for a single 8-cell changer (see Section "Operation of the 8-cell changer" p. 44).

Same as for the use of a single 8-cell changer, the options "slow time scan" and the manual start of measurements are selectable for the combined use of two 8-cell changers.

11.5.1 Staggered operation of two 8-cell changers for more than 8 samples

In staggered operation with two cell changers, up to 14 samples can be analyzed in one measurement series. In this mode, all samples are analyzed against the same reference; Sample and reference beam path are interchanged by computer control so that samples can also be measured in the reference beam path. Irrespective of this, in the further description below, the rear cell changer will always be referred to as "cell changer in the reference beam path" and the front cell changer as "cell changer in the sample beam path".

In staggered operation, the eighth cell position of the cell changer in the reference beam path and the first cell position of the cell changer in the sample beam path remain blank.

In the analysis, first the cell changer in the sample beam path will drive to the first position and stay there while the cell changer in the reference beam path will successively move its cell positions into the beam. When all samples of the cell changer in the reference beam path have been analyzed, this cell changer will stop in its eighth position. Afterwards, the cell changer arranged in the sample beam path will successively move its samples into the beam.

Measurement parameters for staggered operation

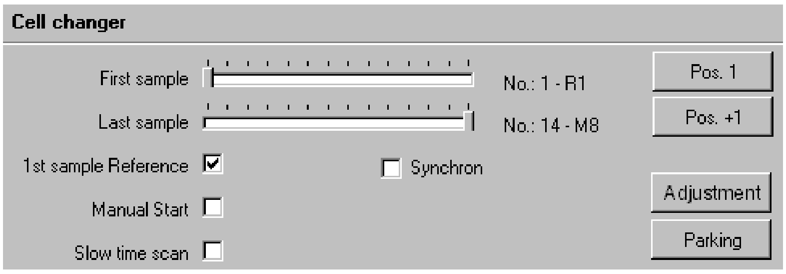


Fig. 11-3 Software settings for the combination of two 8-cell changers in staggered operation

In staggered operation, the **Synchronous** option must always be **deactivated**.

Using the **First sample** and **Last sample** slider buttons, you can set up to 14 cell positions. Beside the slider bar, the number of the sample and its position on the two changers is displayed. **R** denotes the cell changer in the reference beam path and **M** denotes the cell changer in the sample beam path, e.g. **R1** – first position of the cell changer in the reference beam path.

In the presentation of the currently selected measurement parameters on the WinASPECT® workplace, the assignment of cell positions is displayed according to your choice:

Accessory	2 x 8-cell changer
First sample	1
Last sample	14
Cell changer	R.S.S.S.S.S.S.0 0.S.S.S.S.S.S.S
1st sample Reference	

Fig. 11-4 Measurement parameter presentation on WinASPECT® workplace

Beginning with the first position of the cell changer in the reference beam path, load the cells continuously on the following cell positions. In the measurement parameter display, the following abbreviations are used for this:

R Reference

S Sample

0 Blank cell position

Taking measurements in staggered operation

In staggered operation, the reference measurement can be performed in one of the following ways:

1. The reference is not measured separately before taking sample measurements, but in the sample measurement process. The first cell position on the cell changer holds the reference cell.
   In the measurement parameters on the **Accessories** tab, activate the **1st sample reference** option.
2. The reference is measured before samples are analyzed.
   In the measurement parameters on the **General** tab, activate the **Reference** option, and on the **Accessories** tab, the **1st sample reference** option.
   For the reference measurement, place the reference cell in the sample beam path and keep the reference beam path blank.
3. Measurements with strongly absorbing reference.
   In the case of strongly absorbing references, it is useful to use the reference to additionally attenuate the reference signal for sample measurements and to create a balanced energy ratio between reference and sample beam path.
   In the measurement parameters on the **General** tab, activate the **Reference** option, and on the **Accessories** tab, deactivate the **1st sample reference** option.
   The reference measurement is taken with blank sample and reference beam paths. For the sample analysis, place a reference cell each on the eighth position in the reference beam path and on the first position in the sample beam path.

11.5.2 Synchronous operation of two 8-cell changers

In synchronous operation of the two cell changers, every sample is analyzed against a sample-specific reference. Hence, you can simultaneously analyze a maximum of eight samples with their corresponding references. To this end, load the samples in the front cell changer (in the sample beam path) while placing the corresponding references in the cell changer positioned in the reference beam path.

Measurement parameters for synchronous operation

The following measurement parameters must be selected specifically for the synchronous operating mode:

General tab	
Correction	Select the **Reference** option.
Accessories tab – 2 x 8-cell changer selected	
1st sample reference	To be deactivated
Synchronous	To be activated

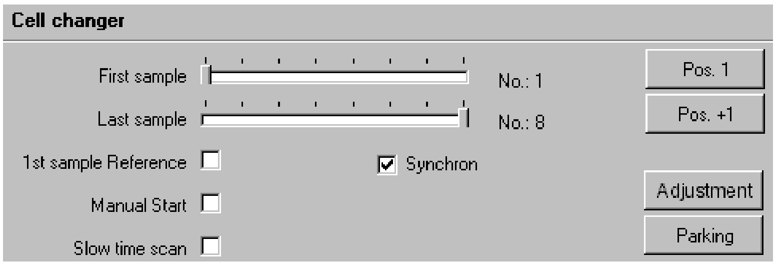


Fig. 11-5 Software settings for the combination of two 8-cell changers in synchronous mode

Taking measurements in synchrounous operation

1. Take the reference measurement with blank cell changers.
2. Load the cell changer. Place the samples in the cell positions on the front cell changer and the references in the corresponding positions on the rear cell changer.
3. Start the sample measurement.

12 Cell carousel

12.1 Description and use

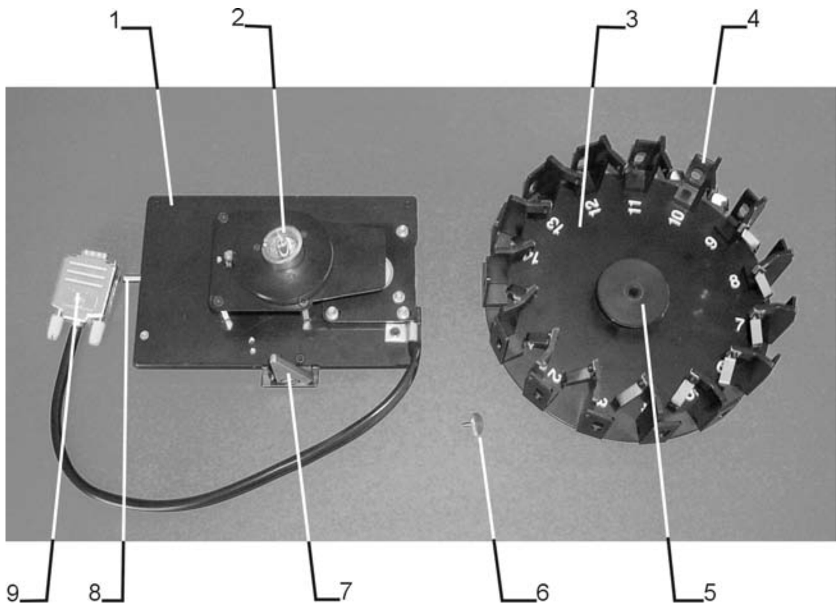
The cell carousel is an automatic sample changing system connected to and controlled by the SPECORD®.

It consists of two separable modules

- a cell holder
- a driving unit with stepper motor.

The cell carousel contains 15 places for 10-mm pathlength cells of the following outer dimensions: 12.5 x 12.5 x 45 (L x W x H in mm).

12.2 Design



1	Driving unit	6	Knurled screw
2	Tray support with guiding slot	7	Clamping lever
3	Sample tray	8	Stop bolt
4	Cell holder	9	Connection cable to SPECORD®
5	Handle		

Fig. 12-1 Cell carousel – Driving unit and sample tray

12.3 Installation in sample compartment

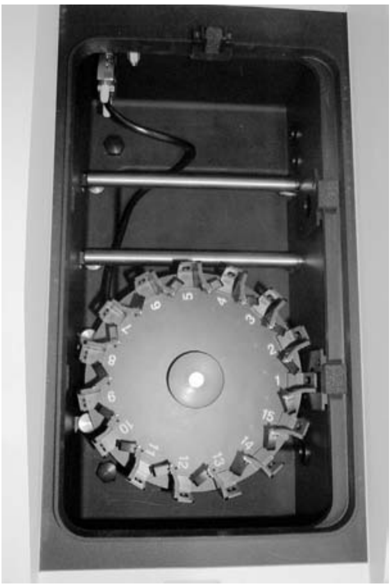


Fig. 12-2 Cell carousel installed in sample compartment

1. First, mount the carrying rails in the sample compartment in their bottom positions (→Section "Changing the position of the carrying rails" p. 8).
2. With clamping lever (Fig. 12-1/ 7) facing the rear side, place the driving unit (Fig. 12-1/ 1) of the cell carousel onto the two front remounted carrying rails in the sample compartment.
3. Push the driving unit to the right-hand sample compartment wall so that the stop bolt touches the right-hand wall.
4. Fasten the driving unit to the carrying rails by throwing the clamping lever down left as far as it will go. If the clamping lever points upward, it will hinder the motion of the sample tray.
5. Put the sample tray (Fig. 12-1 / 3) onto the corresponding support (Fig. 12-1 / 2) of the driving unit. In doing so, slightly turn the tray so that it engages in the guiding slot.
6. Fix the sample tray to the driving unit by means of the knurled screw (Fig. 12-1 / 6).
7. Thread the connection cable underneath the rear carrying rails so that it does not project into the beam path. Insert the plug of the connection cable into the **left** connector in the sample compartment wall.

12.4 Operation

The cell carousel is controlled via WinASPECT® software. In the **Device Driver** dialog box, on the **Accessories** tab, choose the **Carousel** option.

Set the operating parameters of the cell carousel analogously to the cell changer. Here, the maximum number of used cells is 15 (→ Section "6-cell changer, thermostatted, Operation", p.36).

13 Peltier temperature-controlled cell holders/cell changers

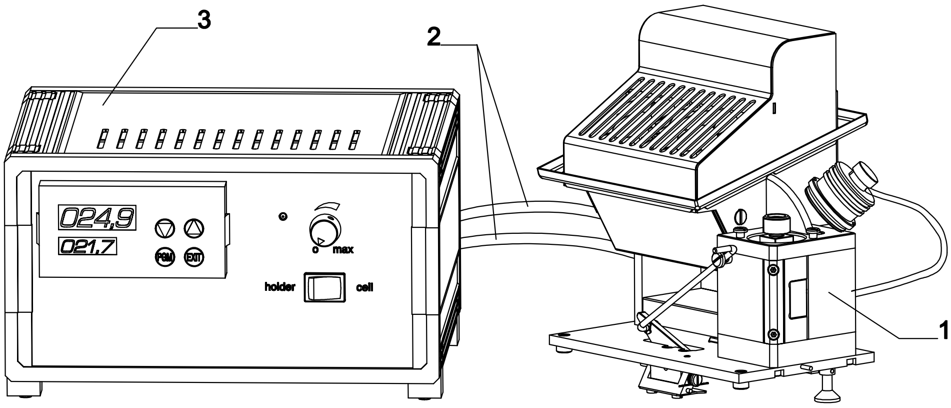
The following cell holders and cell changers with Peltier temperature control are available:

- Peltier temperature-controlled cell holder, air-cooled
- Peltier temperature controlled cell holder with heat exchanger
- Peltier temperature-controlled 6-cell changer with heat exchanger
- Peltier temperature-controlled 8-cell changer with heat exchanger.

Temperature control is through a separate temperature control unit fitted to the corresponding cell holder or cell changer, respectively. However, the connection and the operation of each of these units is the same. For a description, refer to Section "Connection and operation of temperature control unit" p. 90.

The heat exchanger used as counter cooler for cell holders/cell changers is described in Section "Connection and operation of the heat exchanger", p. 94

13.1 Peltier temperature-controlled cell holder, air-cooled



1 Peltier temperature-controlled cell holder
2 Electric cables between control unit and cell holder
3 Temperature control unit

Fig. 13-1 Peltier temperature-controlled 1x cell holder with control unit

The Peltier temperature-controlled cell holder permits the temperature of cells of 12.5 mm x 12.5 mm x 45 mm - 46 mm size (L x W x H) and 10 mm pathlength to be controlled.

Temperature control of the cell holder is performed via a separate control unit. The rear side of the Peltier elements is air-cooled through a heat-transfer system with cooling fins. The controlling sensor used is a Pt100 measuring sensor located at the outer bottom corner of the cell block. In addition to the controlling sensor, the cell holder contains two further Pt100 sensors for optional monitoring of either the holder or the cell temperature.

The cell sensor is specially designed for standard cells with round PTFE stopper. It may remain in the cell during the analytical measurement.

Caution! Risk of distorted results!

When using other cells, the measuring sensor may project into the sample beam thus distorting the measurement results.

By standard, the cell holder is equipped with a magnetic stirrer, which ensures fast and even temperature distribution within the cell. The stirring speed is adjusted on the temperature control unit.

Note

The sample is optimally mixed up, if you use stirring magnets of 3 mm diameter and a length of 6 – 8 mm.

13.1.1 Safety notes

The Peltier temperature-controlled cell holder has been made and tested in compliance with the following standards and directives:

- DIN EN 61010-1 (IEC 1010-1)
- 73/23/EEC
- 89/336/EEC

It was delivered by **Analytik Jena AG** in perfect condition. To keep this condition and ensure safe operation, please observe the safety notes and the notes marked by symbols.

Warning! Risk of electric shocks!

Disconnect the power pug, before opening the instrument or removing any covers to avoid that components carrying line voltage or high tension become accessible.

Earth conductor required!

Only connect the device to a power outlet with earth conductor to ensure operation as per **Protection Class I** (earth-conductor connection). Do not make the protection ineffective by the use of extension cables without earth conductor.

Correct fusing!

Only use fuses of the specified type and rating (→ Section “Technical data of Peltier temperature-controlled cell holder, air-cooled”, p. 56).

Warning! Do not operate the equipment in explosion-risk rooms!

Warning! Risk of burns!

Before replacing the cell after operation at higher temperatures, wait until it has sufficiently cooled down!
There is the risk of burns.

Caution! Accumulation of heat!

Accumulation of heat might result in overheating and faults on the device. Make sure that you do not cover the ventilation slots of the control device and of the heat exchanger of the cell holder!

Caution! Handling liquids!

Take care that no liquids can get into the control device to avoid damage of the device. Particularly do not place any glasses or other vessels containing liquids on the device. When working with an aqueous system at temperatures below the freezing point there is the risk of cell damage by the expansion of ice.

13.1.2 Technical data of Peltier temperature-controlled cell holder, air-cooled

Principle	Thermoelectric heating and cooling
Cooling at rear panel of TEC	Air-cooled
Guaranteed regulated temperature range at 25 °C ambient temperature	+10...+60 °C on models with Type A heat transfer -5...+105 °C on models with Type B heat transfer
Block temperature adjusting range	-20 - +105 °C
Setting accuracy	0.1 degree
Reading accuracy	0.1 degree
Regulating accuracy	+/- 0.1 degree

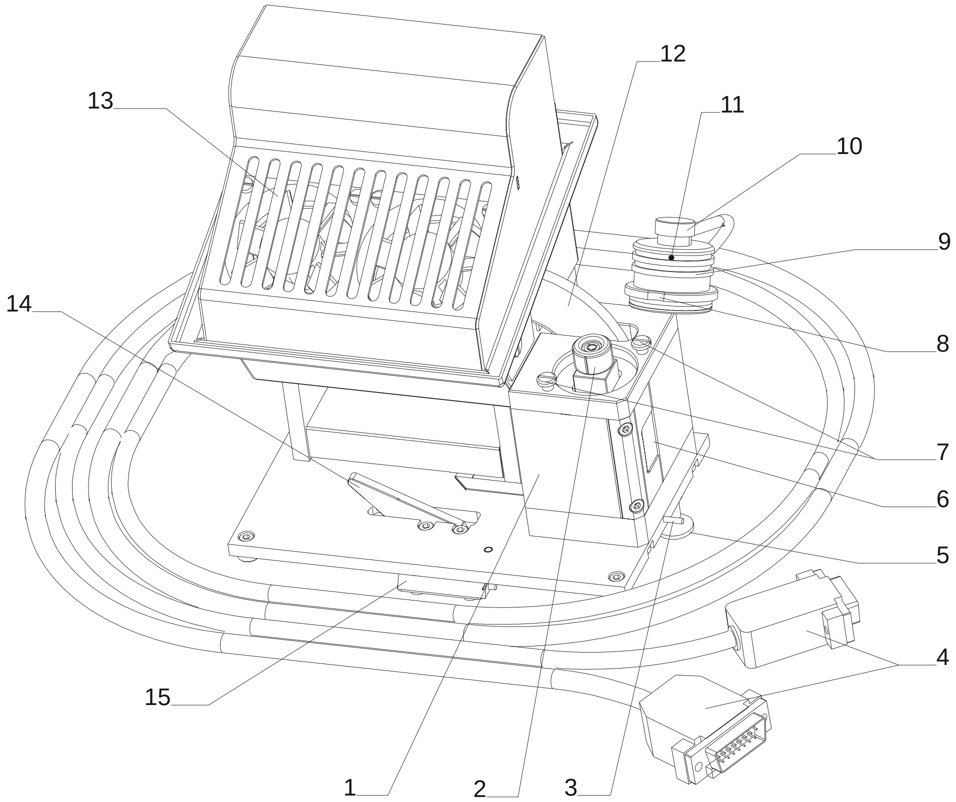
Overview 13-1 Technical data of Peltier temperature-controlled cell holder

Technical data of control unit

Weight of control unit	2.5 kg
Dimensions (W x H x D)	225 x 130 x 200 mm^3
Line voltage	100 - 240 V
Frequency	50 ... 60 Hz
Power consumption	75 VA
Line fuses	2 x T 2.5 A/H 250V, Type 19181 (Wickmann)
EMC (jamming and noise immunity) acc. to DIN EN 61326 and 61326/A1	The device may be installed and operated in all areas.
Fire resistance of the control unit case acc. to UL94	HB / 1,6
Enclosure Protection	IP 20
Data interface	RS232 port
Working temperature range	+15°C...+35°C
Transport and storage temperature range	-40°C...+60°C
Relative humidity	up to 90% (at +30°C)

Overview 13-2 Other technical data of Peltier temperature-controlled cell holder

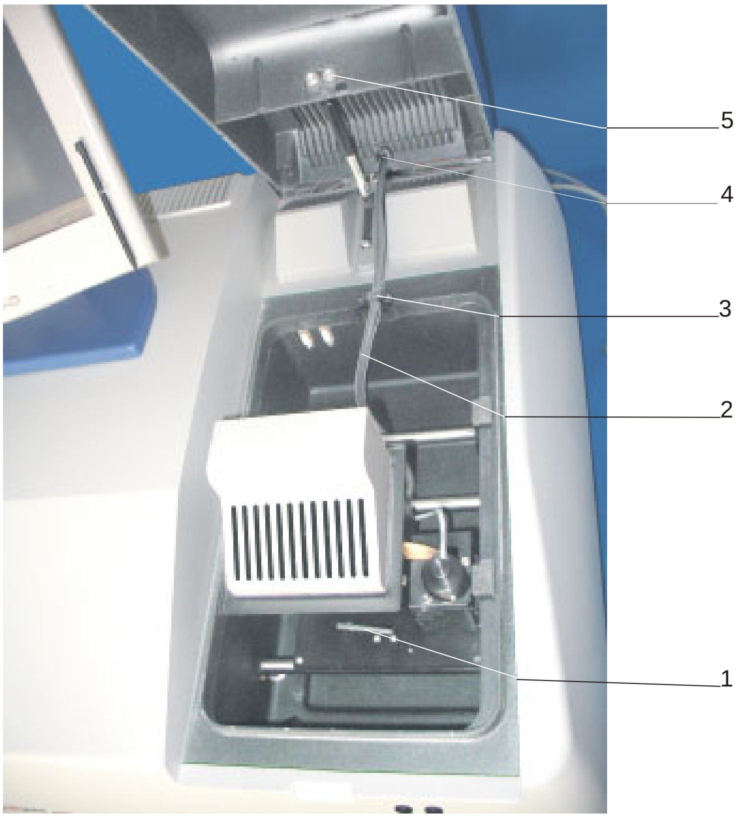
13.1.3 Design of Peltier-temperature-controlled cell holder, air-cooled



1	Cell block with insulation	9	Cover cap
2	Cell with cell temperature sensor	10	Contact cap
3	Spacing pin	11	Lock screw for contact cap
4	Connectors for connection to the control device	12	Heat transfer device
5	Right guide rail stop	13	Heat-exchanger housing with intake slots
6	Quartz window	14	Clamping lever
7	Lock screws	15	Pressure plate
8	Recess		

Fig. 13-2 Peltier temperature-controlled cell holder, air-cooled

13.1.4 Installation of Peltier temperature-controlled cell holder in SPECORD®



1 Clamping lever
2 Electrical cables to temperature control unit
3 Recess with plastic foam stopper
4 Hole with slot in sample compartment cover
5 Knurled screws for fastening the cover prop

Fig. 13-3 Installation of the PTC 100 in the sample compartment

1. The air cooling juts out through an opening in the sample compartment cover. Remove the knurled nuts from the sample compartment cover and remove the cover that closes the opening.
   Carefully retain the cover and the knurled nuts.
2. Slightly incline the cell holder backward to put it onto the front carrying rails of the sample compartment with the cell mount facing the detector.
   Push the cell holder to the right-hand sample compartment wall as far as it will go.
   Throw the clamping lever (1) to the left to fasten the cell holder to the carrying rails.
3. Replace the plastic foam stopper in the rear recess (3) of the sample compartment by the plastic foam strips supplied along with the accessory.
4. Thread the electrical cables (2) to the back underneath the carrying rails. Put the cables between the plastic foam strips in the rear sample compartment wall (3).
   Take care that the cover of the sample compartment is not hindered by projecting plastic foam when closing the cover and make sure it closes light-tight.

SPECORD® with recess underneath the sample compartment cover

5. Thread the cables through underneath the sample compartment cover and put the cable into the recess so that they are not squeezed when you close the cover.

SPECORD® without recess underneath the sample compartment cover

5. Remove the knurled screws from the cover prop (5).
   Fold the cover backward.
   Put the connecting plug of the electrical cables (2) through the slit between sample compartment cover and SPECORD® basic device.
   Screw on the cover prop again.
   Remove the cover cap from the opening in the sample compartment cover (4).
   Put the electrical cable through the slit in the opening.
   Slip the annular stopper over the electrical cables and insert it with the slit on top into the opening (Fig. 13-4).

Fig. 13-4 Sample compartment cover with cables threaded through

6. Connect the cell holder to the temperature control unit (see Section "Connection and operation of temperature control unit", p. 90)

Inserting the cell

1. Turn the cover cap (Fig. 13-2 / 9) until the opposite recesses (Fig. 13-2 / 8) are in line with the lock screws (Fig. 13-2 / 7).
2. Pull the cover cap upward and carefully put the cell into the block.
3. Finally, reattach the cover cap to the cell block.

Note

Closing the cell block prevents the cell from being steamed up when working at temperatures below room temperature. Besides, this ensures the temperature constancy over the entire temperature range as specified.
You need not lock the cap unless you use the cell-temperature sensor. To lock the cap, slightly twist it.

Monitoring the cell internal temperature

Caution! Fragile sensor!

Do not use force when attaching the sensor. It is not necessary to press on the sensor, as it receives sufficient pressure through the contact pins in the cover cap.

Caution! Flashing temperature display!

If the temperature display (Fig. 13-17 /2 and 3) on the temperature-control unit is flashing, either the cell sensor is not properly connected to the connector on the control unit or the sensor is defective.

1. Close a standard cell with round stopper by means of the supplied cell sensor.
2. Put the cell into the cell block and close it by means of the cover cap (the grooves are facing the lock screws).
3. Lock the cover cap by slightly twisting it.
4. On the temperature-control unit, switch the holder/cell temperature selector to “cell“.

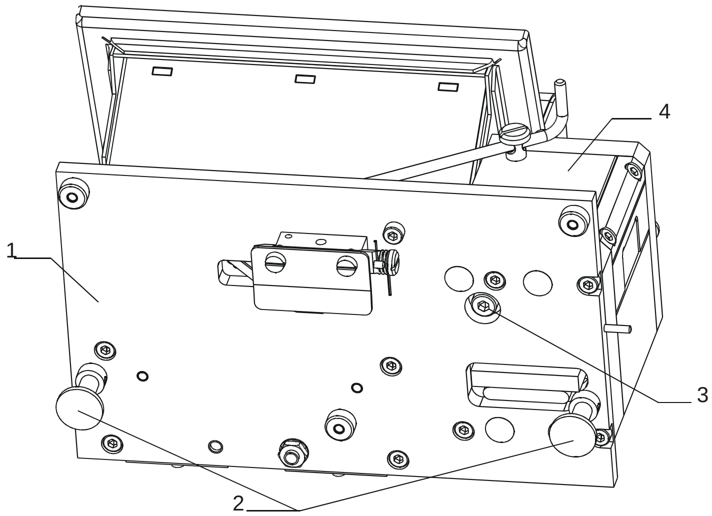
Extending the immersion depth of the measuring sensor

The contact cap (Fig. 13-2 / 10) is fixed in the cover cap with a grub screw. By loosening this screw, you can vary the immersion depth of the contact pins in the cover cap by maximally 4 mm. This allows the cell sensor to be used in a certain range also for non-standard cells.

13.1.5 Care

The cell holder is largely maintenance-free.

- Handle sample substances carefully to avoid contamination especially inside the cell block.
- If despite all care sample substance is spilt, instantly wipe it away with absorbent cloth or paper.
- If despite all care the cell holder should become contaminated (e.g. by sample substance), you may clean the interior of the cell block with ethanol or water plus a dash of a detergent. On the bottom of the base plate (Fig. 13-5 / 1) there is a M4 Allen screw (Fig. 13-5 / 3) for draining the washing liquid.



1	Base plate	3	Draining screw with gasket
2	Guide rail stop	4	Cell block with insulation

Fig. 13-5 Bottom of Peltier temperature-controlled 1x cell holder

The part of the cell sensor immersing in the cell is made of Teflon and thus largely chemically resistant. The upper part of the sensor consists of an anodized aluminum cap with an opening for the gold-plated conductive tracks. These materials, too, are as far as possible corrosion-resistant.

Caution! Ingress of solvents in the sensor!

Avoid immersing the sensor completely in solvents, as they might enter the interior of the sensor.

13.2 Peltier temperature-controlled cell holder with heat exchanger

The Peltier temperature-controlled cell holder allows the temperature control of 10-mm pathlength cells of the following dimensions: 12.5 x 12.5 x 45 - 46 (L x W x H in mm).

Temperature control of the cell holder is performed by a separate control unit. The rear side of the Peltier elements is cooled by a connected heat exchanger. The sensor used for the control is a Pt100 measuring sensor located at the outer bottom corner of the cell block. Beside the measuring sensor for temperature control, two additional Pt100 sensors are used for the optional measurement of the temperature of the holder or the cell.

A heat exchanger is used for the counter cooling of the Peltier elements.

The cell sensor is specially designed for standard cells with round PTFE stopper. This sensor can be left in the cell when taking the optical measurement.

Caution! Risk of getting faulty results!

When using other types of cell, the measuring sensor may project into the the sample beam and thus distort the results of measurement.

The standard equipment of the cell holder includes a magnetic stirrer in order to obtain fast and homogeneous temperature adjustment in the cell.
The stirring speed can be adjusted on the temperature control unit.

Note

Samples are optimally mixed when using stirring magnets of 3mm diameter and 6 to 8 mm length.

13.2.1 Safety notes

The Peltier temperature-controlled cell holder has been made and tested in compliance with the following standards and directives:

- DIN EN 61010-1 (IEC 1010-1)
- 73/23/EEC
- 89/336/EEC.

It was delivered by **Analytik Jena AG** in perfect condition. To keep this condition and ensure safe operation, please observe the safety notes and the notes marked by symbols.

Warning! Risk of electric shocks!

Disconnect the power plug before opening the unit or removing any covers in order to avoid that components carrying line voltage or high tension become accessible.

Only connect the power cable to a power outlet having an earth conductor to ensure operation of the unit according to **Protection Class I** (earth-conductor connection). The protection must not be made ineffective by the use of extension cables without earth conductor.

Caution! Correct fusing!

Only use fuses of the specified type and rating. (→ Section "Technical data of Peltier temperature-controlled cell holder with heat exchanger", p.64).

Warning! Do not operate the equipment in explosion-risk rooms!

Warning! Hot surface!

Before replacing any cells after operation at higher temperatures, wait until they have cooled down sufficiently!
There is the risk of burns.

Caution! Accumulation of heat!

Accumulation of heat might result in overheating and faults on the device. Make sure that you do not cover the ventilation slots of the temperature control unit and of the heat exchanger!

Caution in handling liquids!

Take care to ensure that no liquids enter the temperature control unit to avoid any damage to the unit.

Particularly do not place any glasses or other vessels containing liquids on the device.

When working with an aqueous system at temperatures below the freezing point there is the risk of cell damage by the expansion of ice.

13.2.2 Technical data of Peltier temperature-controlled cell holder with heat exchanger

Principle	Thermoelectric heating and cooling
Cooing at rear panel of TEC	Water-cooled by connection of an external thermostat
Guaranteed regulated temperature range at 25 °C ambient temperature	-10 ... +15 °C
Block temperature adjusting range	-20 ... +105 °C
Setting accuracy	0.1 degree
Reading accuracy	0.1 degree
Regulating accuracy	+/- 0.1 degree

Overview 13-3 Technical data of Peltier temperature-controlled cell holder with heat exchanger

Technical data of control unit

Weight of control unit	2.5 kg
Dimensions (B x H x T)	225 x 130 x 200 mm^3
Line voltage	100 - 240 V
Line frequency	50 ... 60 Hz
Power consumption	75 VA
Line fuses	2 x T 2.5 A/H 250V, Type 19181 (Wickmann)
EMC (jamming and noise immunity) acc. to DIN EN 61326 and 61326/A1	The device may be installed and operated in all environments.
Fire resistance of the control unit acc. to UL94	HB / 1,6
Enclosure Protection	IP 20
Data interface	RS232 port
Operating temperature range	+15°C...+35°C
Transport and storage temperature range	-40°C...+60°C
Relative humidity	Up to 90% (at +30°C)

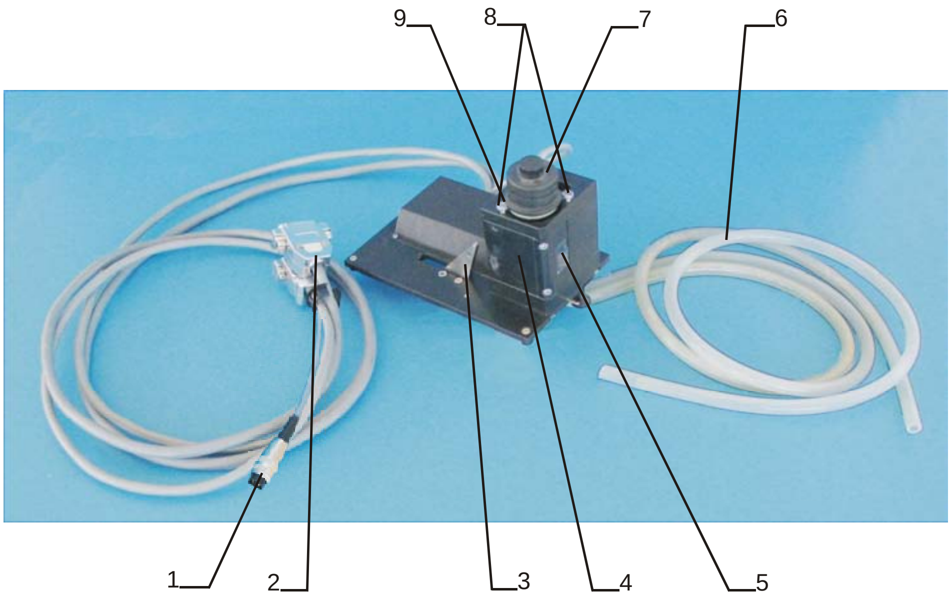
Overview 13-4 Technical data of temperature control unit

Technical data of heat exchanger WC 600 / WC 601

Weight without coolant	3.2 kg
Dimensions (W x H x T)	225 x 175 x 200 mm^3
Line voltage	220 - 240 V
Line frequency	50 Hz
Power consumption	50 VA
Line fuses for WC601	2 x T 0.5 A/H 250V, Type 19181 (Wickmann)
Line fuses for WC601	2 x T 1.6 A/H 250V, Type 19181 (Wickmann)
EMC (jamming and noise immunity) as per DIN EN 61326 and 61326/A1	The device can be installed and operated in all environments.
Fire resistance of the case of the heat exchanger according to UL94	HB / 1,6
Protection Type	IP 20
Coolant	Approximately 0.4 l distilled water with an addition of 4 ml isopropanol
Maximum delivery head	1.2 m
Operating temperature range	+15°C ... +35°C
Transport and storage temperature range	-40°C...+60°C
Relative humidity	up to 90% (at +30°C)

Overview 13-5 Technical data of heat exchanger for PTC 600 / PTC 601

13.2.3 Design of Peltier temperature-controlled cell holder with heat exchanger



1 Connector for connection to heat exchanger
2 5-pole and 9-pole connector for connection to temperature control unit
3 Clamping lever
4 Cell block with insulation
5 Quartz window
6 Tubes for connection to heat exchanger
7 Cover cap
8 Lock screws
9 Grooves

Fig. 13-6 Peltier temperature-controlled cell holder with heat exchanger

13.2.4 Installation of the cell holder in the SPECORD®

Lay connection cables tension-free!

Connection cables and coolant tubes must be laid tension-free. Rule out tensile stress to electrical cables and bending of coolant tubes.

Coolant tubes and connection cables must not project into the beam paths.

Installing the holder in the sample compartment

1. Slightly incline the cell holder backward to put it onto the front carrying rails of the sample compartment with the cell mount facing the detector.
Push the cell holder to the right-hand sample compartment wall as far as it will go.
Throw the clamping lever (3) to the left to fasten the cell holder to the carrying rails.
2. Thread the coolant tubes through underneath the rear carrying rails. Put the tubes into the provided ducts in the right-hand sample compartment wall.
The tube ducts are provided with steps to achieve a higher light-tightness. If the tube is caught on these steps, try to loosen it by slightly turning it. When it becomes visible in the outer opening, you can bend it towards the opening e.g. by means of a pen.

Connect the tubes to a thermostat. We recommend using quick-lock couplings that allow drip-free connection and disconnection of the thermostat.

3. Replace the plastic foam stopper in the rear recess of the sample compartment by the plastic foam strips supplied along with the accessory.
4. Thread the electrical cables (2) to the back underneath the carrying rails. Put the cables between the plastic foam strips in the rear sample compartment wall (3). Take care that the cover of the sample compartment is not hindered by projecting plastic foam when closing the cover and make sure it closes light-tight.

SPECORD® with recess underneath the sample compartment cover

5. Thread the cables through underneath the sample compartment cover and put the cable into the recess so that they are not squeezed when you close the cover.

SPECORD® without recess underneath the sample compartment cover

5. Remove the knurled screws from the cover prop.
   Fold the cover backward.
   Put the connecting plug of the electrical cables (2) through the slit between sample compartment cover and SPECORD® basic device.
   Screw on the cover prop again.
   Remove the cover cap from the opening in the sample compartment cover.
   Put the electrical cable through the slit in the opening.
   Slip the annular stopper over the electrical cables and insert it with the slit on top into the opening (Fig. 13-4, p. 59).
6. Connect the temperature control unit and the heat exchanger (see Sections "Connection and operation of temperature control unit", p. 90 and "Connection and operation of the heat exchanger", p. 94).

Installing a second cell holder

The second cell holder is installed largely in the way described above. However, the recess in the rear sample compartment wall is too small for allowing the electrical cables of two cell holders to be threaded through. Therefore, the accessory is supplied along with a different right-angled bracket.

1. Undo the right-angled bracket mounted in the recess of the sample compartment. In its place, mount the supplied bracket. Put the two supplied plastic foam strips into the recess.
2. Install the second cell holder in the reference beam path.
3. Lay the electrical cables as described above.
4. Connect the cell holders to each other by means of a short piece of tube. Connect one water tube each to every cell holder. Establish the connections to the heat exchanger as described above.
5. Connect the electrical cables to the temperature control units.

Inserting the cell

1. Turn the cover cap (Fig. 13-6 / 7) until the opposite grooves of the cover cap (Fig. 13-6 / 9) are in line with the lock screws (Fig. 13-6 / 8).
2. Pull the cover cap upward and carefully put the cell into the block.
3. Finally, reattach the cover cap to the cell block.

Note

Closing the cell block prevents the cell from being steamed up when working at temperatures below room temperature. Besides, this ensures the temperature constancy over the entire temperature range as specified.

You need not lock the cap unless you use the cell-temperature sensor. To lock the cap, slightly twist it.

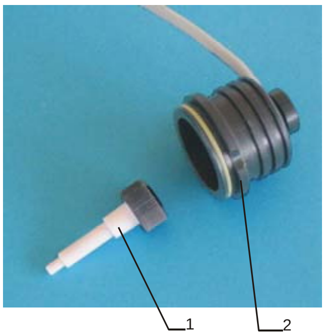
Monitoring the cell internal temperature

Caution! Fragile measuring sensor!

Do not use force when attaching the sensor. It is not necessary to press on the sensor, as it receives sufficient pressure through the contact pins in the cover cap.

Caution! Flashing temperature display!

If the temperature display (Fig. 13-17 / 2 and 3) on the temperature control unit is flashing, either the cell sensor is not properly connected to the corresponding connector ort he sensor is defective.



1 Cell sensor
2 Cover cap of cell holder

Fig. 13-7 Cell temperature measuring sensor PT100

1. Close a standard cell with round stopper by means of the supplied cell sensor.
2. Put the cell into the cell block and close it by means of the cover cap (the grooves are facing the lock screws).
3. Lock the cover cap by slightly twisting it.
4. On the temperature-control unit, switch the holder/cell temperature selector to “cell“.

Extending the immersion depth of the measuring sensor

The contact cap is fixed in the cover cap with a grub screw. By loosening this screw, you can vary the immersion depth of the contact pins in the cover cap by maximally 4 mm. This allows the cell sensor to be used in a certain range also for non-standard cells.

13.3 Peltier temperature-controlled 6-cell changer

13.3.1 Use

The cell changer is an automatic sample changing system. Its stepper motor driving unit is driven by the software. Temperature control of the cell holder is performed via a separate temperature-control unit. The coolant circuit of the heat exchanger keeps the rear side of the Peltier elements at a temperature near the ambient temperature.

The controlling sensor used is a Pt100 measuring sensor located in the upper part of the cell block. In addition to the controlling sensor, the cell holder contains two further Pt100 sensors for optional monitoring of either the holder or the cell temperature.

The cell holder contains six positions for cells having a pathlength of 10 mm and external dimensions of 12.5 x 12.5 x 45 (L x W x H in mm).

The cell sensor is specially designed for standard cells with round PTFE stopper. It may remain in the cell during the analytical measurement.

Note

When using other cells, the immersed measuring sensor may project into the sample beam thus distorting the measurement results.

The stepper-motor drive of the driving unit moves the cell holder to the next sampling place within approximately 1 second. In combination with a reflected-light optocoupler, which defines the starting point of the motion range, the stepper drive ensures exactly reproducible alignment of the cells to the measuring beam.

1 Temperature control unit with status indicator for heat exchanger
2 Heat exchanger
3 Peltier temperature-controlled 6-cell changer

Fig. 13-8 Peltier temperature-controlled 6-cell changer with SPECORD® 210

13.3.2 Safety notes

Electric shock!

The temperature-control unit and the heat exchanger are electrically powered. At several parts inside the system, **extremely hazardous electrical** voltages are accessible.

To ensure Protection Class I (protective earth connection) of the device, connect the power plug of the device only to a power outlet with protective earth conductor.

Connect control unit, heat exchanger and SPECORD® to the same phase of the line power supply (e.g. to a multiple socket power outlet).

Before connecting the devices to the power outlet, make sure the line voltage supplied agrees with the operating voltage specified on the rating plate at the rear panel of the devices. Operation of the devices with any other operating voltage than that specified may result in their destruction.

Only use fuses of the specified type (→ Section "Technical data of Peltier temperature-controlled 6-cell changer", p.71).

Do not operate the device in explosion-risk rooms!

Type label and warning labels!

Observe the type label and the warning labels on the devices!

Accumulation of heat!

Make sure that you do not cover the ventilation slots in the bottom plate and on the rear panel of the device as well as on the front panel of the heat exchanger.

Heat accumulation might result in overheating and faults on the equipment.

Ingress of water!

Take care to ensure that no liquids can get into the control unit to avoid any damage to the device.

Do not place the heat exchanger or containers with liquids onto the control unit.

The control unit complies with the requirements of Protection Type IP 20.

The PTC 600 / PTC 601 Peltier temperature-controlled 6-cell changer was produced and tested in compliance with the following standards and regulations:

- DIN EN 61010-1 (IEC 1010-1)
- 73/23/EC
- 89/336/EC

13.3.3 Technical data of Peltier temperature-controlled 6-cell changer

Operating principle	Thermoelectric heating and cooling
Cooling of rear panel of TEC	Water-cooled
Guaranteed regulated temperature range at 25°C ambient temperature	PTC 600 with heat exchanger WC 600: +10°C...+60°C PTC 601 with heat exchanger WC 601: -5°C...+105°C
Block temperature setting range	PTC 600 with heat exchanger WC 600: +5°C - +65°C PTC 601 with heat exchanger WC 601: -20°C...+105°C
Setting accuracy	0.1 degree
Reading accuracy	0.1 degree
Regulating accuracy	+/– 0.1 degree

Overview 13-6 Technical data of PTC 600 / PTC 601

Technical data of control unit

Weight	2.5 kg
Dimensions (W x H x T)	225 x 130 x 200 mm^3
Line voltage	100 - 240 V
Line frequency	50 ... 60 Hz
Power consumption	150 VA
Line fuses	2 x T 2.5 A/H 250V, Type 19181 (Wickmann)
EMC (jamming and noise immunity) as per DIN EN 61326 and 61326/A1	The device can be installed and operated in all environments.
Fire resistance of the case of the control unit according to UL94	HB / 1,6
Protection Type	IP 20
Data interface	RS232 port
Operating temperature range	+15°C...+35°C
Transport and storage temperature range	-40°C ... +60°C
Relative humidity	up to 90% (at +30°C)

Overview 13-7 Technical data of control device for PTC 600 / PTC 601

Technical data of heat exchanger WC 600 / WC 601

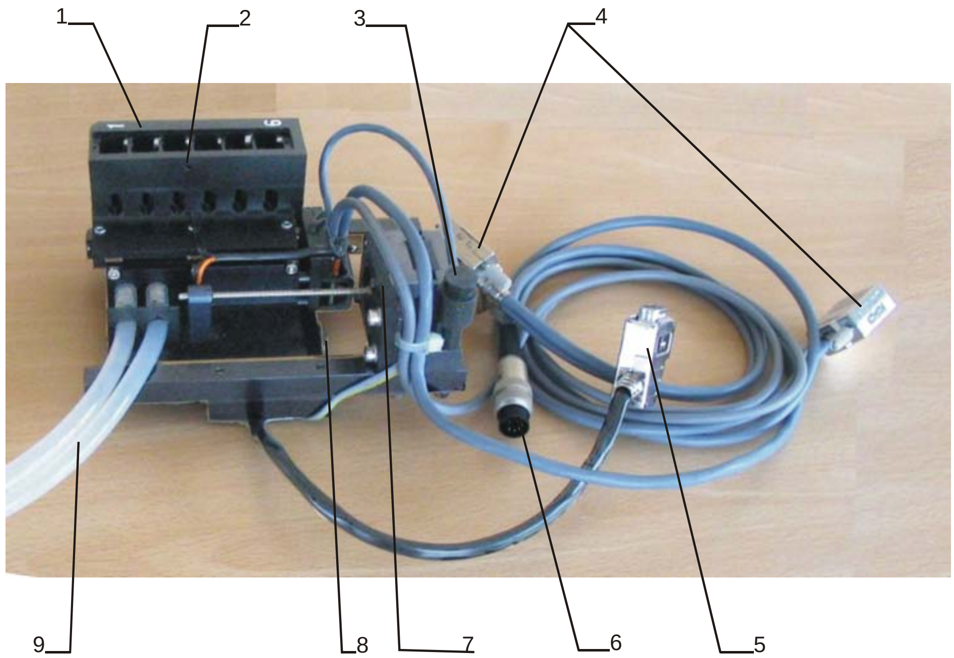
Weight without coolant	3.2 kg
Dimensions (W x H x D)	225 x 175 x 200 mm^3
Line voltage	220 - 240 V
Line frequency	50 Hz
Power consumption	50 VA
Line fuses for WC600	2 x T 0.5 A/H 250V, Type 19181 (Wickmann)
Line fuses for WC600	2 x T 1.6 A/H 250V, Type 19181 (Wickmann)
EMC (jamming and noise immunity) as per DIN EN 61326 and 61326/A1	The device can be installed and operated in all environments.
Fire resistance of the case of the heat exchanger according to UL94	HB / 1,6
Protection Type	IP 20
Coolant	Approximately 0.4 l distilled water with an addition of 4 ml isopropanol
Maximum delivery head	1.2 m
Operating temperature range	+15°C ... +35°C
Transport and storage temperature range	-40°C...+60°C
Relative humidity	up to 90% (at +30°C)

Overview 13-8 Technical data of heat exchanger for PTC 600 / PTC 601

Pay attention to any different technical data on the type label!

If the data given above are different from that on the type label, the data on the type label are valid.

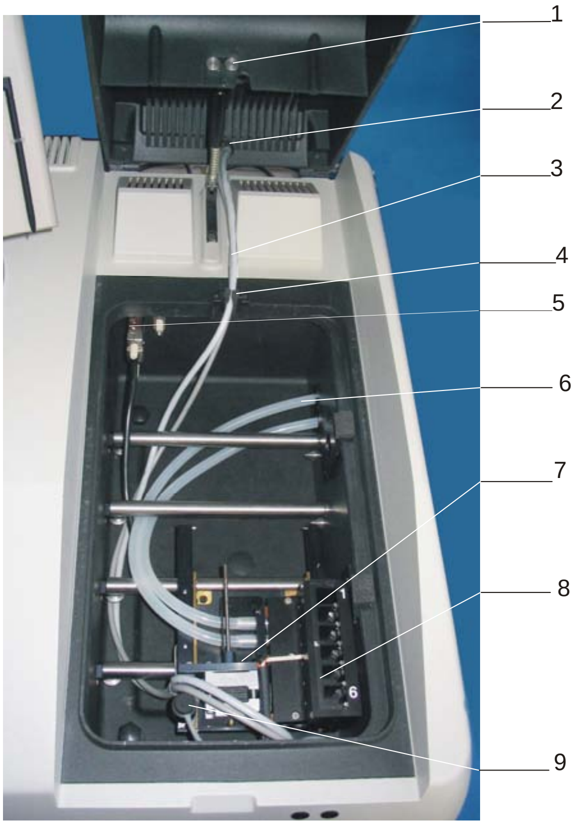
13.3.4 Design of Peltier temperature-controlled 6-cell changer



1 Cell block with insulation
2 Block sensor
3 Connector for cell sensor in storage receptacle
4 Connectors for connection to temperature control unit
5 Connector for connection to SPECORD® S600
6 Connector for connection to heat exchanger
7 Stepper motor drive with lead screw
8 Knurled screw for clamping the unit to the carrying rails
9 Water tubes for connection to the heat exchanger

Fig. 13-9 Design of Peltier temperature-controlled 6-cell changer

13.3.5 Installation of the Peltier-temperature-controlled 6-cell changer



1 Knurled screws for fastening the cover prop
2 Hole with slot in sample compartment cover
3 Electric cables from cell changer to temperature-control unit and to heat exchanger
4 Recess with plastic foam stopper in sample compartment wall
5 9-pin connector for connection to SPECORD®
6 Coolant tubes
7 Knurled screw for fastening the cell changer to the carrying rails
8 Peltier temperature-controlled 6 cell changer
9 Connector of temperature sensor in storage cavity

Fig. 13-10 Installation of Peltier temperature-controlled 6-cell changer in the SPECORD®

Lay connection cables tension-free!

The connecting cables and the coolant tubes must be laid without tension and in a way that they do not hinder the movement of the cell changer while in operation. Make sure that the electric cables are not tensioned and the coolant tubes are not folded.

Take care to ensure that the coolant tubes and the connecting cables do not project into the light path of the sample or reference beam path.

1. Replace the plastic foam stopper in the recess in the rear sample compartment wall (4) by the two plastic foam strips supplied along with the accessory.
2. Turn the knurled screw (7) clockwise as far as it will go.
3. Put the cell changer from top onto the front carrying rails of the sample compartment so that its guideways rest on the carrying rails. Then, push the cell changer to the right-hand sample compartment wall as far as it will go.

4. Clamp the cell changer to the carrying rails by turning the knurled screw counterclockwise. Fastening the knurled screw hand-tight will do to lock the cell changer reliably!
5. Thread the cable with connector to the SPECORD® (5) underneath the rear carrying rails and connect it to the left connector on the sample compartment wall.
6. Lay the coolant tubes underneath the rear carrying rails. Thread them through the provided ducts in the right-hand sample compartment wall (6).
   The tube ducts are provided with steps to achieve a higher light-tightness. If the tube is caught on these steps, try to loosen it by slightly turning it. When it becomes visible in the outer opening, you can bend it towards the opening e.g. by means of a pen.
7. Thread the electrical cables (3) to the back underneath the rear carrying rails. Put the cables between the two plastic foam strips in the recess in the rear sample compartment wall (4).
   Take care that the cover of the sample compartment is not hindered by projecting plastic foam when closing the cover and make sure it closes light-tight.

SPECORD® with recess underneath the sample compartment cover

8. Thread the cables through underneath the sample compartment cover and put the cable into the recess so that they are not squeezed when you close the cover.

SPECORD® without recess underneath the sample compartment cover

8. Remove the knurled screws from the cover prop (5).
   Fold the cover backward.
   Put the connecting plug of the electrical cables (3) through the slit between the sample compartment cover and the SPECORD® basic device.
   Screw on the cover prop again.
   Remove the cover cap from the opening in the sample compartment cover (2).
   Put the electrical cable through the slit in the opening.
   Slip the annular stopper over the electrical cables and insert it with the slit on top into the opening (Fig. 13-4 p. 59).
9. Connect the temperature control unit and the heat exchanger (see Sections "Connection and operation of temperature control unit", p. 90 and "Connection and operation of the heat exchanger", p. 94).

Adjustment of the Peltier temperature-controlled 6-cell changer

The cell changer can be adjusted computer-controlled to optimally position the cells in the beam.

Adjustment is necessary

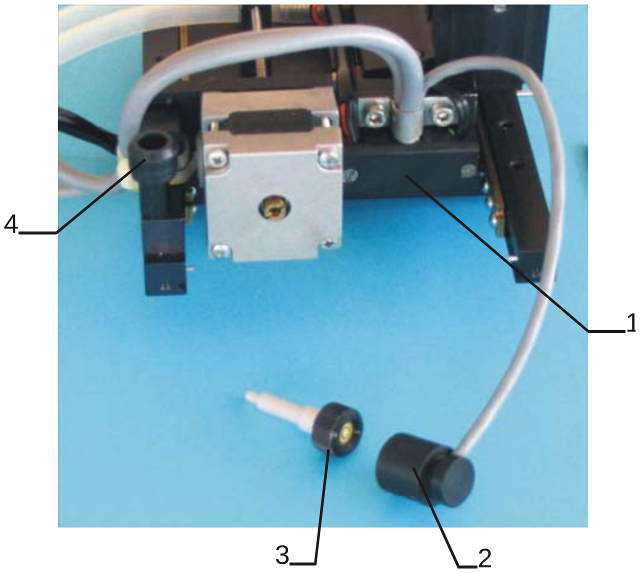
- At the first use of the cell changer
- After wavelength calibration
- After relocation of the SPECORD®
- When working with microcells.

1. Install the empty cell changer in the sample compartment.
2. On the **Accessories** tab of the measurement parameter window, activate the **6-cell changer (Peltier)** option.
3. If you intend to use standard cells (1cm x 1cm) or semi-microcells for the following analyses, perform the adjustment without any cells being inserted.

If you intend to use microcells in the analyses, insert a microcell filled with water in every of the six cell positions.

4. Start automatic adjustment by a click on the **[Adjustment]** button.

Monitoring the cell internal temperature



1	Cell changer
2	Connector for cell sensor
3	Cell sensor
4	Storage receptacle for connector

Fig. 13-11 Cell measuring sensor for the Peltier temperature-controlled 6-cell changer

1. Close a standard cell with round stopper with the supplied cell measuring sensor (Fig. 13-11 / 3) in place of the standard stopper.
2. Put the cell onto any desired position. Take the connector (Fig. 13-11 / 2) from the storage receptacle (Fig. 13-11 / 4) and plug the connector onto the cell measuring sensor.
3. On the temperature-control unit, switch the selector switch to “cell“.
4. If you no longer need the cell sensor, disconnect it from the connector and plug it into the storage receptacle.

Fault indication!

If the temperature displays (**Fig. 13-17**, 2 and 3) on the control unit are flashing, either the cell measuring sensor has not been connected properly to the connector, or the sensor is defective.

Operation without heat exchanger

When operating the Peltier temperature-controlled 6-cell changer without heat exchanger or with the heat exchanger switched off, cooling is electronically suppressed, heating however is possible. In this case, if the ambient temperature is constant and the selected nominal temperature is about 5 degrees above sample compartment temperature, the selected nominal temperatures can be kept to an accuracy of 0.1 – 0.2 degrees at increased settling time. However, the setting accuracy specified in Section "Technical data of Peltier temperature-controlled 8-cell changer", p.81 refers to the operation of the cell changer with heat exchanger.

Operation with purging of sample compartment

Below a certain cell block or cell temperature (dew point) and depending on room temperature and relative humidity, the water contained in the ambient atmosphere condenses at the cell walls and the cell block. This will result in a distortion of measurement results.

For the beginning of condensation (dew-point temperature), the following relation applies:

$$\vartheta_K = \left(\frac{\text{relative humidity}}{100}\right)^{0.1247} * (109.8 + \vartheta_R) - 109.8$$

ϑ_K - Dew-point temperature in °C

ϑ_R - Room temperature in °C

At a room temperature of 20°C and a relative humidity of 60%, the dew-point temperature is 12°C. Condensation can be prevented by purging the sample compartment with dry gas.

1. Thread a tube as thick as possible (inside diameter of at least 6 mm) through the front duct provided for the drain tube into the sample compartment (Fig. 4-1 / 11, p .4-1).
2. Lay the tube mouth in the middle of the front sample compartment wall. To prevent instable temperatures, the gas must not be blown directly onto the cell changer.
3. Purge the sample compartment with 800 – 1000 l/h of dry gas, e.g. air, nitrogen or argon.

Note

Before starting analyses at low temperatures, it is advisable to pre-dry the sample compartment. Heat up the sample compartment by letting the Peltier temperature-controlled cell changer operate for about ten minutes at +80°C.

Avoid opening the sample compartment unnecessarily long and thus the inflow of humid room air, when running analyses at low temperatures.

13.3.6 Settings in WinASPECT® software

The cell changer is controlled by WinASPECT® software. On the **Accessories** tab of the measurement parameter window, choose the **6-cell changer (Peltier)** option.

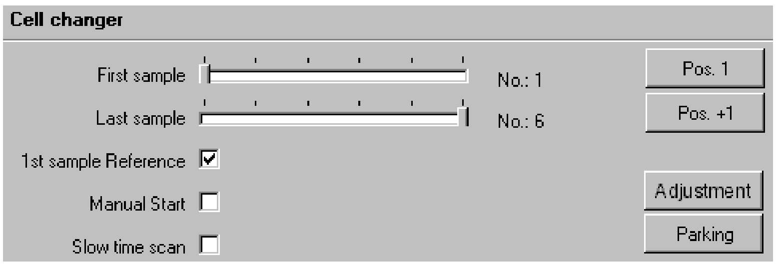


Fig. 13-12 Options for Peltier temperature-controlled 6-cell changer for SPECORD®

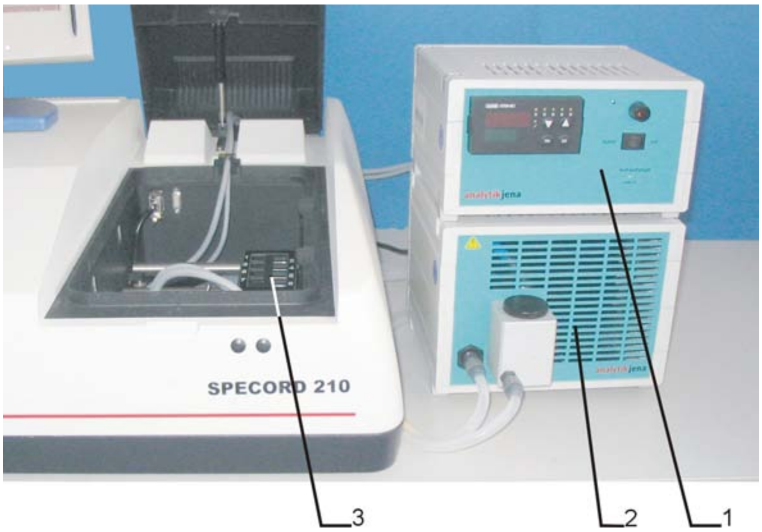
Option / button	Description
First sample	Position of the first sample on the cell changer.
Last sample	Position of the last sample on the cell changer.

1st sample Reference	The sample placed on the 1st position selected above is treated as reference. In this case, it is not necessary to run the reference measurement separately. The reference measurement will be performed within the normal sample measurements.
Start manually	Before every measurement of a cell, a message box appears „Start cycle [OK]“. The actual sample measurement is started only after you confirmed this prompt. This option may be used, for instance, for kinetic measurements. The measurement in the respective cell will be started only after you added the starting substance.
Slow time scan	Activate this checkbox, if you intend to record a slow kinetic reaction in cyclic measurements in all samples in a staggered mode (→ Section "**Fehler! Verweisquelle konnte nicht gefunden werden.**", p. **Fehler! Textmarke nicht definiert.**).
[Pos.1]	Cell changer moves to the first position.
[Pos. +1]	Cell changer moves to the next position.
[Adjustment]	Starts the adjustment of the cell changer.
[Parking]	Cell changer moves to the parking position with the cell block being centric above the base plate. In this position, the cell changer can be easily removed from and installed in the SPECORD® as well as packed.

13.3.7 Measurement procedure

1. Choose the measurement parameters according to your analytical needs.
2. Switch on the temperature control unit and the heat exchanger at the power switches located at the back panels each.
3. Adjust the desired temperature on the temperature control unit (→ Section "Settings on temperature control unit", p. 91).
4. The measurement procedure is analogous to that with the water-thermostatted 6-cell changer (→ Section "
5. Measurements with the 6-cell changer", p.38).

13.4 Peltier temperature-controlled 8-cell changer



1 Temperature control unit
2 Heat exchanger
3 Peltier temperature-controlled 8-cell changer

Fig. 13-1 Peltier temperature-controlled 8-cell changer

The cell changer is an automatic sample changing system. Its driving unit with stepper motor drive is controlled by software. Temperature control of the cell holder is performed via a separate temperature-control unit. The coolant circuit of the heat exchanger keeps the rear side of the Peltier elements at a temperature near the ambient temperature.

The controlling sensor used is a Pt100 measuring sensor located in the upper part of the cell block. In addition to the controlling sensor, the cell holder contains two further Pt100 sensors for optional monitoring of either the holder or the cell temperature.

The cell holder contains eight positions for cells having a pathlength of 10 mm and external dimensions of 12.5 x 12.5 x 45 (L x W x H in mm).

The cell sensor is specially designed for standard cells with round PTFE stopper. It may remain in the cell during the analytical measurement.

Note

When using other cells, the immersed measuring sensor may project into the sample beam thus distorting the measurement results.

Optionally, the 8-cell changer may be factory-equipped with a magnetic stirrer. The stirring speed is adjusted at the temperature control unit.

You can also use two Peltier temperature-controlled 8-cell changers in combination to increase the number of samples that can be measured to a maximum of 14 samples or to analyze every sample against a specific reference. The second cell changer needs a separate control unit. Countercooling is perfomed via a common heat exchanger.

13.4.1 Safety notes

Electric shock!

The temperature-control unit and the heat exchanger are electrically powered. At several parts inside the system, **extremely hazardous electrical** voltages are accessible.

To ensure Protection Class I (protective earth connection) of the device, connect the power plug of the device only to a power outlet with protective earth conductor.

Connect control unit, heat exchanger and SPECORD® to the same phase of the line power supply (e.g. to a multiple socket power outlet).

Before connecting the devices to the power outlet, make sure the line voltage supplied agrees with the operating voltage specified on the rating plate at the rear panel of the devices. Operation of the devices with any other operating voltage than that specified may result in their destruction.

Only use fuses of the specified type (→ Section "Technical data of Peltier temperature-controlled 8-cell changer", p.81).

Disconnect the power cable, before replacing any fuses.

Do not operate the device in explosion-risk rooms!

Type label and warning labels!

Observe the type label and the warning labels on the devices!

Accumulation of heat!

Make sure that you do not cover the ventilation slots in the bottom plate and on the rear panel of the device as well as on the front panel of the heat exchanger.

Heat accumulation might result in overheating and faults on the equipment.

Ingress of water!

Take care to ensure that no liquids can get into the control unit to avoid any damage to the device.

Do not place the heat exchanger or containers with liquids onto the control unit.

The control unit complies with the requirements of Protection Type IP 20.

The Peltier temperature-controlled 8-cell changer was made and tested in compliance with the following standards and directives:

- DIN EN 61010-1 (IEC 1010-1)
- 73/23/EEC
- 89/336/EEC

13.4.2 Technical data of Peltier temperature-controlled 8-cell changer

Operating principle	Thermoelectric heating and cooling
Cooling of rear panel of TEC	Water-cooled
Guaranteed regulated temperature range at 25°C ambient temperature*	PTC 800 with heat exchanger WC 600: 10°C...+60°C PTC 801 with heat exchanger WC 601: -5°C...+ 105°C
Block temperature setting range	PTC 800 with heat exchanger WC 600: +5°C...+65°C PTC 801 with heat exchanger WC 601: -20°C...+105°
Setting accuracy	0.1 degree
Reading accuracy	0,1 degree
Regulating accuracy	+/- 0.1 degree

*) Temperatures below room temperature may result in steaming up of cells.

Overview 13-9 Technical data of Peltier temperature-controlled 8-cell changer

Technical data of temperature control unit

Weight	2.5 kg
Dimensions (W x H x T)	225 x 130 x 200 mm³
Line voltage	100 - 240 V (-15% / +10%)
Line frequency	50 ... 60 Hz
Power consumption	150 VA
Line fuses	2 x T 2.5 A/H 250V, Type 19181 (Wickmann)
EMC (jamming and noise immunity) as per DIN EN 61326 and 61326/A1	The device can be installed and operated in all environments.
Fire resistance of the case of the control unit according to UL94	HB / 1,6
Protection Type	IP 20
Data interface	RS232 port
Operating temperature range	+15°C...+35°C
Transport and storage temperature range	-40°C...+60°C
Relative humidity	Up to 90% (at +30°C)

Overview 13-10 Technical data of temperature control unit PTC 800 / PTC 801

Technical data of heat exchanger

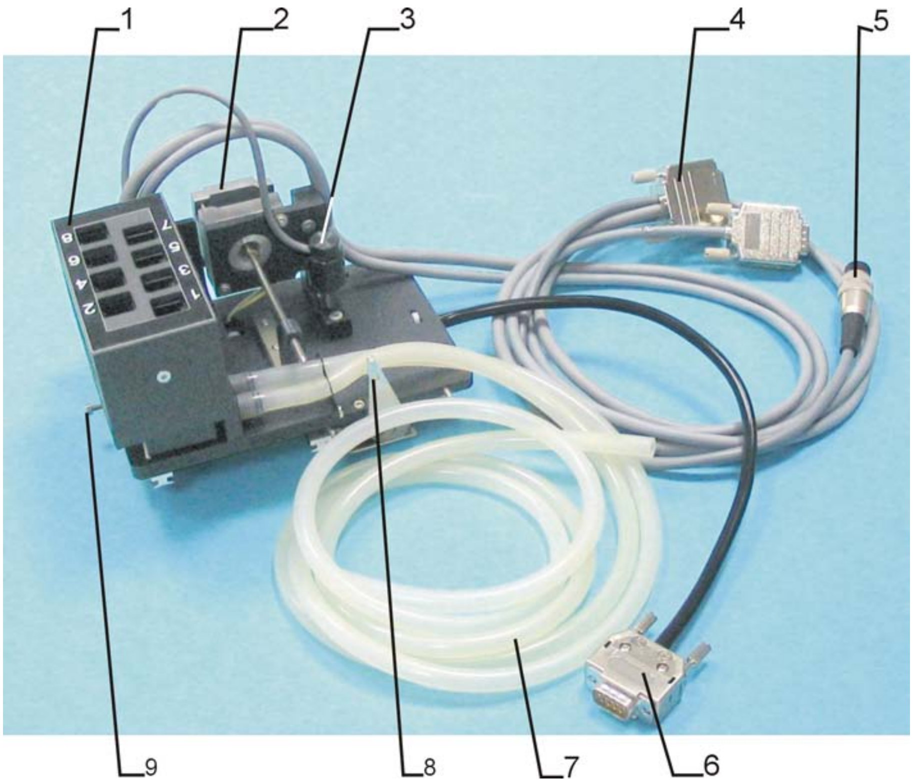
Weight without coolant	3.2 kg
Dimensions (W x H x D)	225 x 175 x 200 mm^3
Line voltage	220 - 240 V (-15% / +10%)
Line frequency	50 Hz
Power consumption	50 VA
Line fuses for WC 600	2 x T 0.5 A/H 250V, Type 19181 (Wickmann)
Line fuses for WC 601	2 x T 1.6 A/H 250V, Type 19181 (Wickmann)
EMC (jamming and noise immunity) as per DIN EN 61326 and 61326/A1	The device can be installed and operated in all environments.
Fire resistance of the case of the heat exchanger according to UL94	HB / 1,6
Protection Type	IP 20
Coolant	Approximately 0.4 l distilled water with an addition of 4 ml isopropanol
Maximum delivery head	1.2 m
Operating temperature range	+15°C...+35°C
Transport and storage temperature range	-40°C...+60°C
Relative humidity	Up to 90% (at +30°C)

Overview 13-11 Technical data of heat exchanger

Pay attention to any different technical data on the type label!

If the data given above are different from that on the type label, the data on the type label are valid.

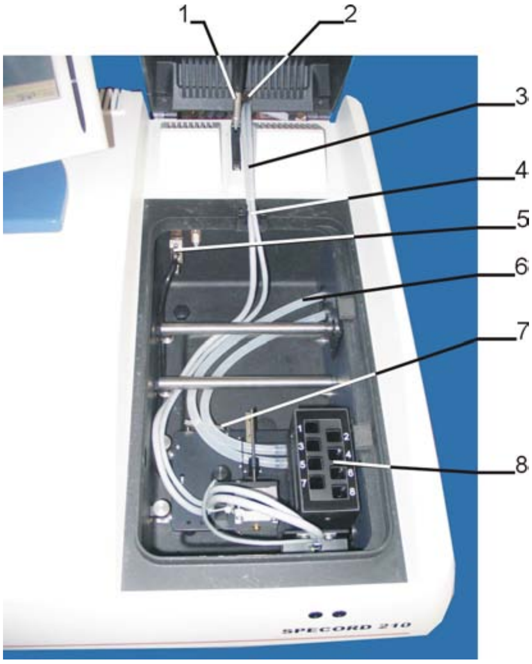
13.4.3 Design of Peltier temperature-controlled 8-cell changer



1 Cell block with insulation
2 Stepper motor drive
3 Connector for cell sensor in storage receptacle
4 Connectors for connection to temperature control unit
5 Connector for connection to heat exchanger
6 Connector for connection to SPECORD®
7 Water tubes for connection to heat exchanger
8 S600 clamping lever

Fig. 13-13 Design of Peltier temperature-controlled 8-cell changer

13.4.4 Installation of Peltier temperature-controlled 8-cell changer



1 Cover prop
2 Opening with slit in sample compartment cover
3 Connection cables to temperature control unit
4 Recess in rear sample compartment wall
5 Connector to SPECORD®
6 Water connection tubes to heat exchanger
7 Clamping lever
8 Cell block with insulation

Fig. 13-2 Installation of Peltier temperature-controlled 8-cell changer

Lay connection cables tension-free!

The connecting cables and the coolant tubes must be laid without tension and in a way that they do not hinder the movement of the cell changer while in operation. Make sure that the electric cables are not tensioned and the coolant tubes are not folded.

Take care to ensure that the coolant tubes and the connecting cables do not project into the light path of the sample or reference beam path.

1. Replace the plastic foam stopper in the recess in the rear sample compartment wall (4) by the two plastic foam strips supplied along with the accessory.
2. Remove the carrying rails from their top position and mount them in their bottom position (→ Section “Changing the position of the carrying rails“, p. 8).
3. Place the cell changer onto the relocated carrying rails with its clamping lever facing the back. Push the cell changer to the right-hand sample compartment wall as far as it will go. Clamp it to the carrying rails by throwing the clamping lever to the left.
4. Thread the connection cable to the SPECORD® to the back underneath the rear carrying rails and connect it to the left connector in the rear sample compartment wall.

5. Put the electrical cables (3) into the recess in the rear sample compartment wall (4) and close the recess again using the plastic foam strips. Fix the plastic foam strips by attaching the supplied metal bracket. Make sure that the recess is closed light-tight.

SPECORD® with recess underneath the sample compartment cover

6. Thread the cables through underneath the sample compartment cover and put the cable into the recess so that they are not squeezed when you close the cover.

SPECORD® without recess underneath the sample compartment cover

6. Remove the knurled screws from the cover prop.
   Fold the cover backward.
   Put the connecting plug of the electrical cables (3) through the slit between the sample compartment cover and the SPECORD® basic device.
   Screw on the cover prop again.
   Remove the cover cap from the opening in the sample compartment cover (2).
   Put the electrical cable through the slit in the opening.
   Slip the annular stopper over the electrical cables and insert it with the slit on top into the opening (Fig. 13-4 p. 59).
7. Lay the thermostatting tubes underneath the rear carrying rails. Thread them through the provided ducts in the right-hand sample compartment wall.
   The tube ducts are provided with steps to achieve a higher light-tightness. If the tube is caught on these steps, try to loosen it by slightly turning it. When it becomes visible in the outer opening, you can bend it towards the opening e.g. by means of a pen.
   Connect the tubes to the thermostat. We recommend using quick-lock couplings that allow drip-free connection and disconnection of the thermostat.
8. Connect the cell changer to the temperature control unit (→ Section "Connection and operation of temperature control unit", p. 90).
9. Connect the heat exchanger (→ Section "Connection and operation of the heat exchanger", p. 94).

Installing a second cell changer

Caution! Correct cable connections in sample compartment!

Connect the cell changer installed in the **rear reference beam path** to the **right connector** in the sample compartment.
Connect the cell changer installed in the **front sample beam path** to the **left connector**.

The second Peltier temperature-controlled 8-cell changer is installed largely in the way described above. However, the recess in the rear sample compartment wall is too small for allowing the electrical cables of two cell changers to be threaded through. Therefore, the accessory is supplied along with a different right-angled bracket.

1. Undo the right-angled bracket mounted in the recess of the sample compartment. In its place, mount the supplied bracket. Put the two supplied plastic foam strips into the recess.
2. Install the second 8-cell changer in the reference beam path.
3. Lay the electrical connection cables as described above. Additionally fasten the supplied metal bracket above the recess in the sample compartment wall to avoid that the cables slip out of the recess.

4. Connect the second 8-cell changer to the right connector in the sample compartment.
5. Both 8-cell changers use the same heat exchanger. Connect the two 8-cell changers to each other by means of a short piece of tube. Connect one water tube each to the heat exchanger and to every 8-cell changer. Establish the connections to the heat exchanger as described above.
6. Connect the 7-pole connectors of the cell changers with the enclosed y cable. Connect the plug of the y cable with the heat exchanger.
7. Connect the electrical cables to the temperature control units.

Adjustment of the Peltier temperature-controlled 8-cell changer

The cell changer can be adjusted computer-controlled to optimally position the cells in the beam.

Adjustment is necessary

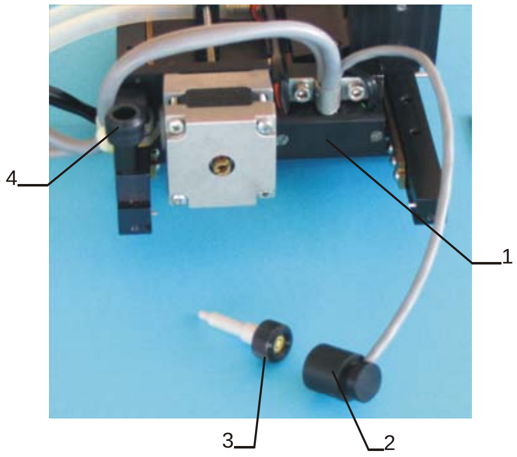
- At the first use of the cell changer
- After wavelength calibration
- After relocation of the SPECORD®
- When working with microcells.

1. Install the empty cell changer in the sample compartment.
2. On the **Accessories** tab of the measurement parameter window, activate the **8-cell changer (Peltier)** option.
3. If you intend to use standard cells (1cm x 1cm) or semi-microcells for the following analyses, perform the adjustment without any cells being inserted.
   If you intend to use microcells in the analyses, insert a microcell filled with water in every of the six positions.
4. Start automatic adjustment by a click on the **[Adjustment]** button.

Note

The adjustment for two 8-cell changers is to be performed in the same way.

Monitoring the cell internal temperature



1 Cell sensor
2 Connector for cell sensor

Fig. 13-14 Cell measuring sensor for the Peltier temperature-controlled 8-cell changer

1. Close a standard cell with round stopper with the supplied cell measuring sensor (Fig. 13-14 / 1) in place of the standard stopper.
2. Put the cell onto any desired cell position. Take the connector (Fig. 13-14 / 2) from the storage receptacle (Fig. 13-13 / 3) and plug the connector onto the cell measuring sensor.
3. On the temperature-control unit, switch the selector switch to “cell“.
4. If you no longer need the cell sensor, disconnect it from the connector and plug it into the storage receptacle (Fig. 13-13 / 3).

Fault indication!

If the temperature displays (**Fig. 13-17**, 2 and 3) on the control unit are flashing, either the cell measuring sensor has not been connected properly to the connector, or the sensor is defective.

Operation without heat exchanger

When operating the Peltier temperature-controlled 6-cell changer without heat exchanger or with the heat exchanger switched off, cooling is electronically suppressed, heating however is possible. In this case, if the ambient temperature is constant and the selected nominal temperature is about 5 degrees above room compartment temperature, the selected nominal temperatures can be kept to an accuracy of 0.1 – 0.2 degrees at increased settling time. However, the setting accuracy specified in Section “Technical data of Peltier temperature-controlled 8-cell changer” on p. 81 refers to the operation of the cell changer with heat exchanger.

Operation with purging of sample compartment

Below a certain cell block or cell temperature (dew point) and depending on room temperature and relative humidity, the water contained in the ambient atmosphere condenses at the cell walls and the cell block. This will result in a distortion of measurement results.

For the beginning of condensation (dew-point temperature), the following relation applies:

$$\vartheta_K = \left(\frac{\text{relative humidity}}{100}\right)^{0.1247} * (109.8 + \vartheta_R) - 109.8$$

ϑ_K - Dew-point temperature in °C

ϑ_R - Room temperature in °C

At a room temperature of 20°C and a relative humidity of 60%, the dew-point temperature is 12°C. Condensation can be prevented by purging the sample compartment with dry gas.

1. Thread a tube as thick as possible (inside diameter of at least 6 mm) through the front duct provided for the drain tube into the sample compartment (Fig. 4-1 / 11, p .4-1).
2. Lay the tube mouth in the middle of the front sample compartment wall. To prevent instable temperatures, the gas must not be blown directly onto the cell changer.
3. Purge the sample compartment with 800 – 1000 l/h of dry gas, e.g. air, nitrogen or argon.

Note

Before starting analyses at low temperatures, it is advisable to pre-dry the sample compartment. Heat up the sample compartment by letting the Peltier temperature-controlled cell changer operate for about ten minutes at +80°C.

Avoid opening the sample compartment unnecessarily long and thus the inflow of humid room air, when running analyses at low temperatures.

13.4.5 Settings in WinASPECT® software

The cell changer is controlled by WinASPECT® software. On the **Accessories** tab of the measurement parameter window, choose the **8-cell changer** option.

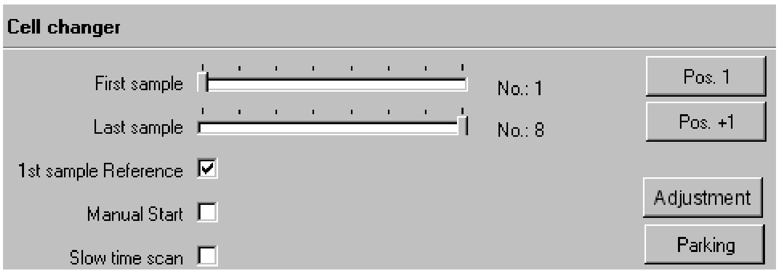


Fig. 13-15 Options for Peltier temperature-controlled 8-cell changer for SPECORD®

Option / button	Description
First sample	Position of the first sample on the cell changer.
Last sample	Position of the last sample on the cell changer.
1st sample - reference	The sample placed on the 1st position selected above is treated as reference. In this case, it is not necessary to run the reference measurement separately. The reference measurement will be performed within the normal sample measurements.
Start manually	Before every measurement of a cell, a message box appears “Start cycle [OK]“. The actual sample measurement is started only after you confirmed this prompt. This option may be used, for instance, for kinetic

	measurements. The measurement in the respective cell will be started only after you added the starting substance.
Slow time scan	Activate this checkbox, if you intend to record a slow kinetic reaction in cyclic measurements in all samples in a staggered mode (→ "**Fehler! Verweisquelle konnte nicht gefunden werden.**", p. **Fehler! Textmarke nicht definiert.**).
[Pos.1]	Cell changer moves to the first position.
[Pos. +1]	Cell changer moves to the next position.
[Adjustment]	Starts the adjustment of the cell changer.
[Parking]	

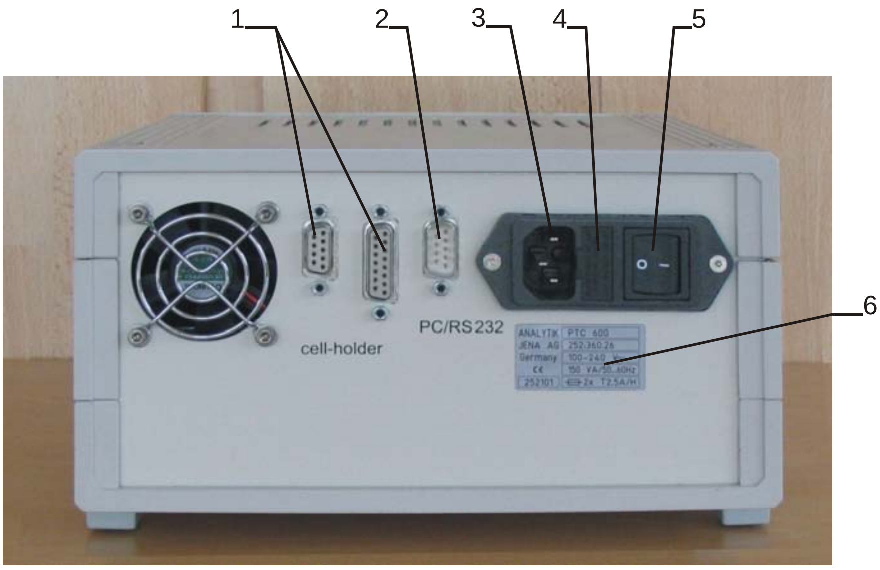
When two Peltier temperature-controlled 8-cell changers have been installed, they can be used in two modes. In synchronous mode, every sample will be analyzed against a specific reference. In staggered mode, the number of samples in a measurement series increases to a maximum of 14.
For this, in the measurement parameters on the **Accessory** tab, the **2x 8-cell changer** must be selected. The possible modes are described in Section "Use of two 8-cell changers", p.45).

13.4.6 Measurement procedure

1. Choose the measurement parameters according to your analytical needs.
2. Switch on the temperature control unit and the heat exchanger at the power switches located at the back panels each.
3. Adjust the desired temperature on the temperature control unit (→ Section "Settings on temperature control unit", p. 91).
4. The measurement procedure is analogous to that with the water-thermostatted 6-cell changer (→ Section "
5. Measurements with the 6-cell changer", p. 38).

13.5 Connection and operation of temperature control unit

13.5.1 Connecting the temperature control unit

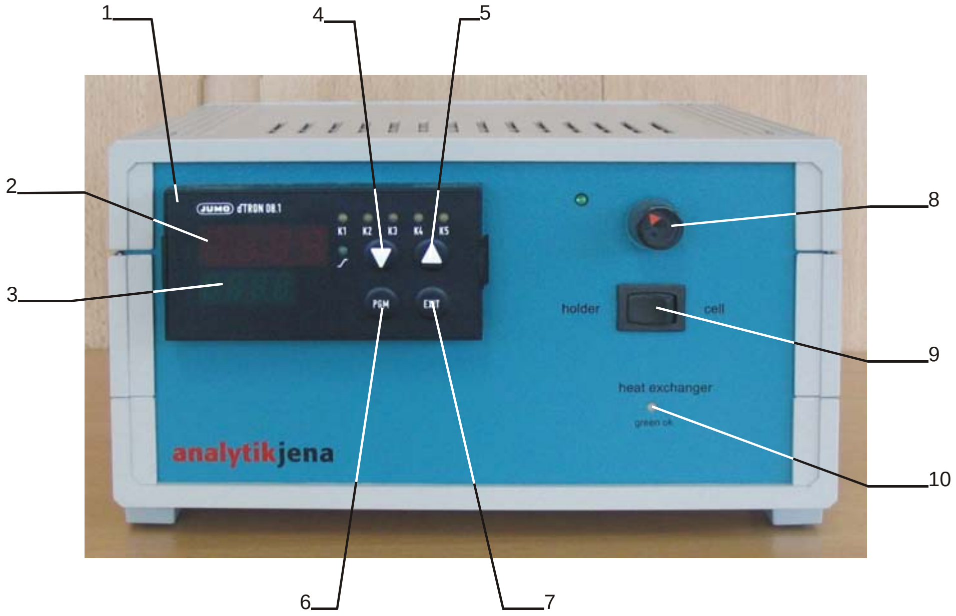


1 9-pole and 15-pole connectors
2 RS232 port
3 Power input connector
4 Fuse holder for line fuse
5 Power switch
6 Type label

Fig. 13-16 Back panel of temperature control unit

1. Connect the 9-pole and the 15-pole connectors of the cell holder/cell changer to the corresponding connectors (Fig. 13-16 / 1) on the back panel of the temperature control unit.
2. Connect the power cable to the power input connector (Fig. 13-16 / 3) and then to a power outlet.

13.5.2 Settings on temperature control unit



1	Control module
2	Block temperature display
3	Temperature display of holder / cell
4	Decrement button
5	Increment button
6	PGM button
7	EXIT button
8	Speed control for stirring magnets (only for cell holders / cell changers with magnetic stirrer)
9	Holder/cell temperature selector switch
10	Status lamp for heat exchanger (not used on air-cooled Peltier temperature-controlled cell holders)

Fig. 13-17 Front panel of temperature control unit

Indicators for heat exchanger (does not apply to air-cooled Peltier temperature-controlled cell holders)

The indicator LED (Fig. 13-17 / 9) located on the front panel of the control unit indicates the three possible states of the heat exchanger

LED off No heat exchanger available or the heat exchanger has not been connected to the control unit via the 6-pin connector.

LED red The heat exchanger is connected to the control unit via the 6-pin connector, but has not been switched on.

LED green The heat exchanger is connected to the control unit via the 6-pin connector and has been switched on.

Pre-selecting the temperature on the temperature-control unit

Caution!

In selecting the nominal temperature, consider that this value always refers to the block temperature. Depending on the temperature in the sample compartment, the type of cell used as well as the type and volume of the sample material, different end values of the temperature inside the cell may result and also different times for reaching these values (Fig.13-18).

1. Press the PGM button (Fig. 13-17 / 6). On the cell holder or cell temperature display (Fig. 13-17 / 3), it appears “SP 1“.

2. Press the decrement button (Fig. 13-17 / 4) to reduce the pre-selected temperature or the increment button (Fig. 13-17 / 5) to increase it.

3. Confirm the pre-selected temperature by pressing the PGM button (Fig. 13-17 / 6). After two seconds, the selected value will automatically be stored.

Note

The nominal temperature can only be varied within the permissible temperature range.

4. Exit the programming mode by pressing the EXIT button (Fig. 13-17 , 7).

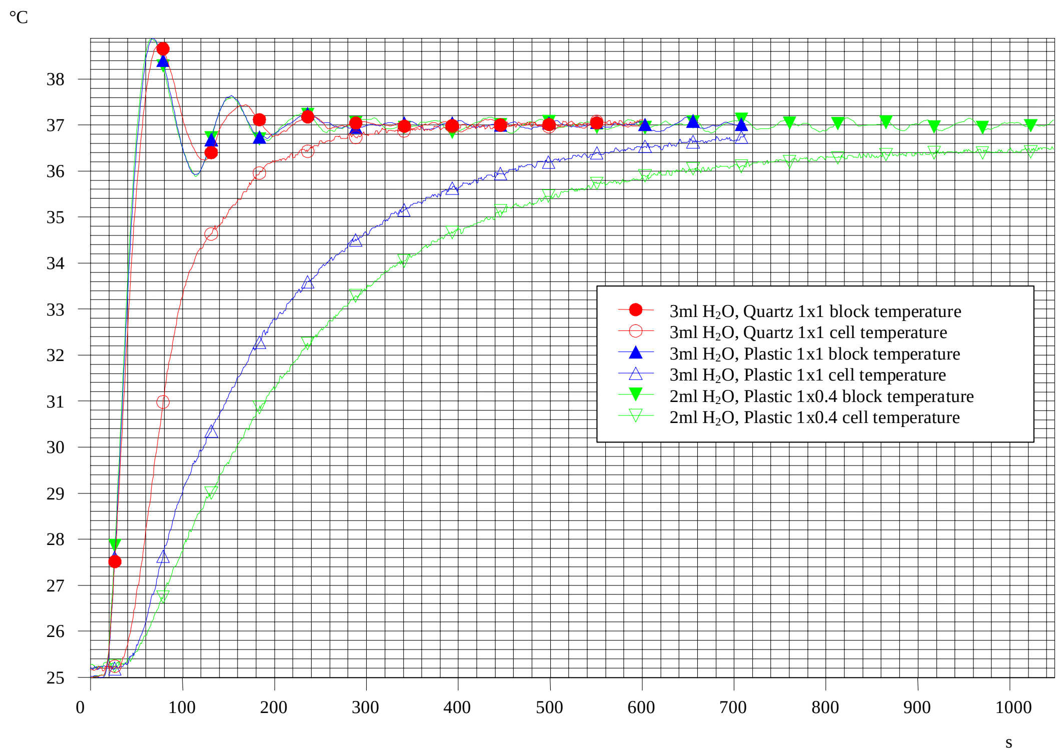


Fig.13-18 Temperature distribution in cell block and in cell for different types of cells (measured on air-cooled, Peltier temperature-controlled cell holder)

13.5.3 Replacing fuses on the temperature control unit

Replace any blown fuses on the temperature control unit by following this procedure:

1. Disconnect the power cable.
2. Open the fuse holder by pulling its lid.
3. Replace the defective fuses considering the technical data of the cell holder or the cell changer you are using.
4. Close the fuse holder again.
5. Reconnect the power cable.
6. Switch the SPECORD® on again.

13.6 Connection and operation of the heat exchanger

Heat accumulation!

Always keep ventilation slits free!

The warning "⚠ Danger! Heat accumulation! Keep free slits!" is located at the front panel of the heat exchanger.

1 Screw cap
2 Compensating tank
3 Coolant tube connectors

Fig. 13-19 Heat exchanger, front panel

1 Power switch
2 Fuse holder for line fuse
3 Power input connector
4 7-pole connector for temperature control unit
5 Type label

Fig.13-20 Heat exchanger, rear panel

13.6.1 Preparing the coolant

In a separate vessel, mix approximately 400 ml of distilled water with 4 to 5 ml of isopropanol.

13.6.2 Connecting the heat exchanger

1. Connect the 7-pin connector of the cell holder / changer (with the appropriate connector (Fig.13-20 / 4) on the rear panel of the temperature-control unit.

2. Connect the power cable to the power input connector (Fig.13-20 / 3) and then to a power outlet.
3. Slip the tubes from the cell holder/changer over the appropriate tube connectors of the heat exchanger (Fig. 13-19 / 3) and lock them in place by turning the knurled nuts anti-clockwise.
4. Remove screw cap (Fig. 13-19 / 1) by turning it anti-clockwise.
5. Switch on the heat exchanger with power switch (Fig.13-20 / 1).
6. Fill the prepared coolant through the opening in the compensating tank (Fig. 13-19 / 2) until the filling level is approximately 1.5 cm below the top edge of the compensating tank and the coolant circuit is free of air bubbles (no bubbles rising in the tank and a noise-free circulation of the coolant).
7. Close the compensating tank hand-tight with the screw cap.

Note

When installing the heat exchanger, please consider that the maximum delivery head of the device is approximately 1.2 m.

Depending on the place of installation of the heat exchanger, the laying of the tubes and for technical reasons it may happen, that air cushions are forming when filling the coolant system, which disappear only after several minutes. Briefly switching the device on and off may accelerate the removal of these air cushions.

13.6.3 Replacing fuses on the heat exchanger

Replace any defective fuses on the heat exchanger by following this procedure:

1. Disconnect the power cable.
2. Open the fuse holder by pulling its lid.
3. Replace the defective fuses considering the technical data of the cell holder or the cell changer you are using.
4. Close the fuse holder again.
5. Reconnect the power cable.
6. Switch the SPECORD® on again.

14 Cassette sipper

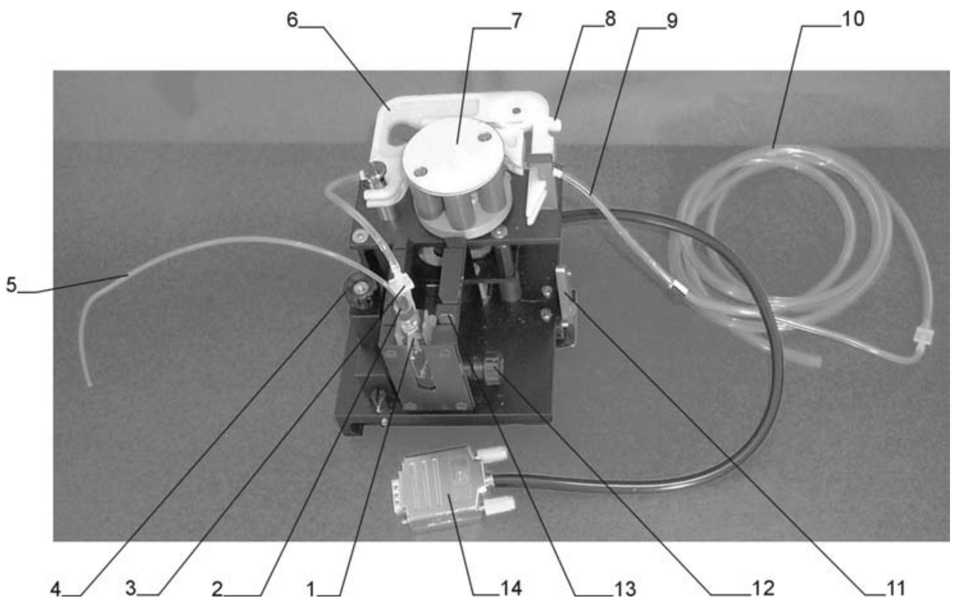
14.1 Intended use

The cassette sipper rationalizes manual laboratory work in laboratories with medium sample batches. Sample feed is computer controlled. A peristaltic pump transfers the sample for the measurement into the flow cell. After the measurement, the sample is pumped into the waste bottle.

Sample feed can be manual or automatic by an autosampler.

The cassette sipper can also aspirate sample and reference simultaneously. In this case, equip the pump with two tube cassettes and place the reference cell in the adjustable cell holder in the reference beam of the SPECORD®.

14.2 Design of cassette sipper



1	Flow cell	8	Eccentric
2	Inlet connector of flow cell	9	Ready-made pump tube with stoppers
3	Outlet connector of flow cell	10	Drain tube
4	Vertical adjusting screw	11	Clamping lever
5	Sample aspirating tube	12	Horizontal adjusting screw
6	Tube cassette	13	Cell retaining spring
7	Pump rollers	14	Connecting plug

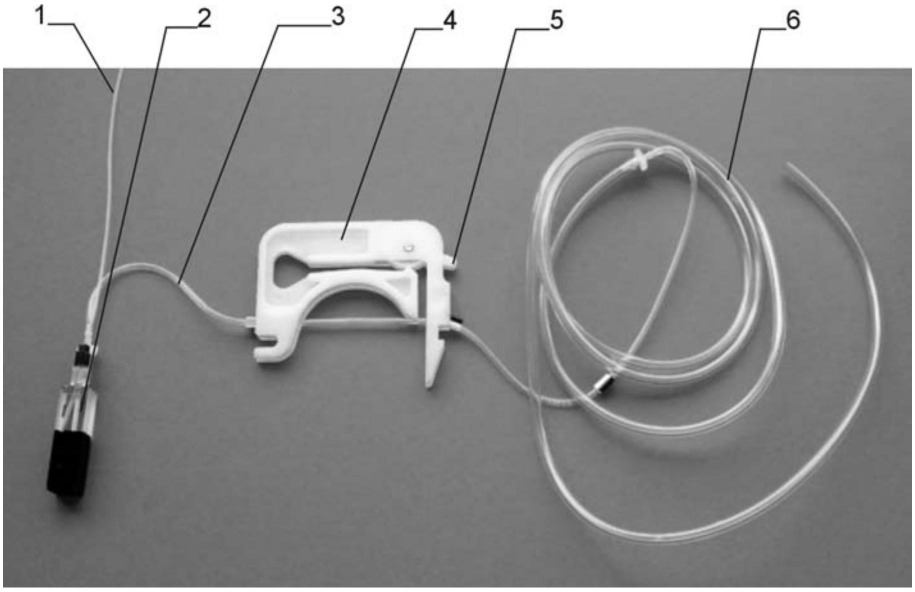
Fig. 14-1 Design of cassette sipper

The cassette sipper consists of:

- Adjustable holder for flow cells of 1, 2, and 5 cm pathlength
- Peristaltic pump
- Tube set.

14.3 Mounting the cassette sipper

14.3.1 Mounting the tube set



1	Sample aspirating tube	4	Tube cassette
2	Flow cell	5	Eccentric
3	Pump tube	6	Drain tube

Fig. 14-2 Tube set for cassette sipper

Mount the tube set as illustrated in Fig. 14-2:

1. Fasten the pump tube in the tube cassette by tensioning it between two stoppers.
2. Fix the sample aspirating tube to the flow cell. To this end, slip the Tygon® tube end over the inlet connector of the cell (observe the marking indicating the direction of flow on the cell). The black Viton® gasket must rest on the inlet connector and the Teflon tube project into the cell. This is to prevent that a sample reservoir forms at the inlet connector, which would cause measuring errors by carry-over.
3. Fix the pump tube with the tube adapter and the short silicon tube to the outlet connector of the cell. In doing so, observe the proper direction of the tube cassette. If the direction is wrong, the pumping direction will be wrong, too.
4. Use the tube adapter to interconnect pump tube and drain tube.

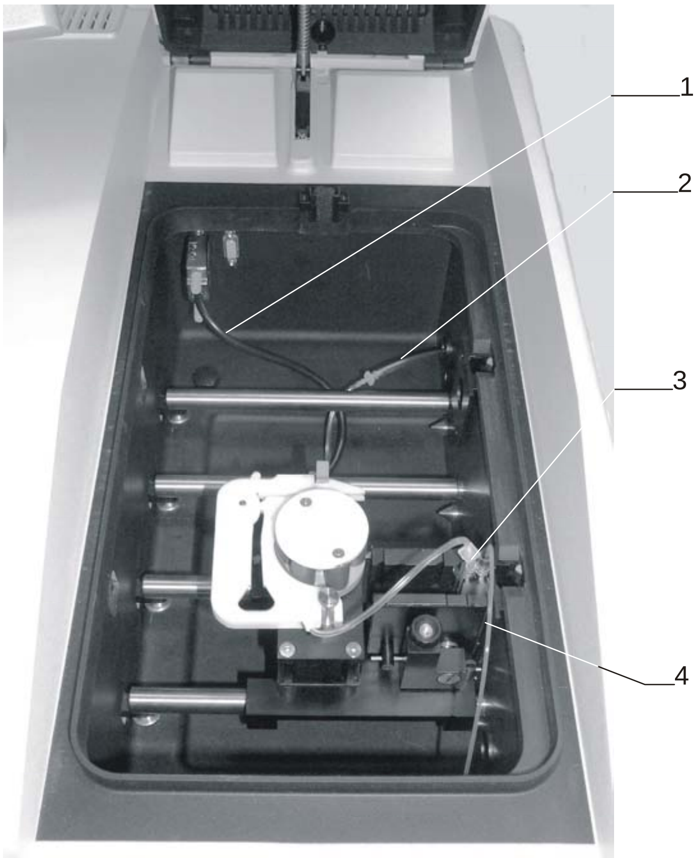
Insert the tube set in the sample holder:

5. Plug the cell retaining spring onto the place that corresponds to the pathlength of the cell.
6. Insert the cell in the cell holder with the outlet connector facing the pump.
7. Fasten the tube cassette to the pump rollers (Fig. 14-1).
8. Press the pump tube to the rollers with the eccentric so that it is fully squeezed.

Note

After longer use, it may happen that the pump tube section tensioned in the cassette sipper loosens its elasticity. If the tube did not additionally become porous, you can tension the second tube section between the middle and the third stopper in the tube cassette.

14.3.2 Mounting the sipper in the sample compartment



1 Interface cable
2 Drain tube
3 Flow cell
4 Sample aspirating tube

Fig. 14-3 Cassette sipper mounted in SPECORD®

1. Thread the drain tube through one of the provided ducts (Fig. 1-1 / 6 or 11) in the sample compartment to the collecting bottle set up outside. Lay the tube under the carrying rails so that it cannot jut into the beams when taking measurements.
   The ducts in the sample compartment wall are provided with steps to increase the light tightness. If the tube hooks to these steps when threading it through, try to release it by slightly turning it. When it becomes already visible at the other end of the duct, bend it to the opening e.g. by means of a pen.
2. Place the cassette sipper system onto the carrying rails in the sample beam of the sample compartment so that the cell holder faces the detector. In this position, clamping lever (Fig. 1-1 / 11) points to the back.
3. To position the flow cell as close to the detector as possible, push the sipper system to the right against the sample compartment wall.
4. Clamp the sample holder by means of the clamping lever.

5. Thread the sample aspirating tube through one of the openings in the front wall (Fig. 1-1 / 12).
   Take care not to fold the tube. The tube has a particularly smooth surface to avoid gas nucleation centers. At tube folds, however, gas bubbles may form. When these bubbles get into the sample cell, they will strongly distort the results.
6. Lead the interface cable for connection to the SPECORD® underneath the rear carrying rails to the connectors in the sample compartment wall and plug its plug into one of these connectors.

14.3.3 Adjusting the flow cell

For the first use of the cassette sipper and after a change of the flow cell, it is advisable to adjust the cell in the beam.

1. First, check the passage of non-dispersed "white" light through the cell. To this end, set the zeroth order of light in the optical path (→ Section Adjustment of zeroth order, p. 8.)
2. For visual observation, insert an approximately 10 mm wide strip of paper in the cell cavity for turbid samples and, from top, look at the beam passing through. Successively turn the screws for vertical (Fig. 14-1 / 4) and horizontal adjustment (Fig. 14-1 / 12).

Then, carry out an energy measurement for fine adjustment:

3. Activate the menu command **Measurement / Set Parameters** to create a parameter data record with e.g. the following settings:

Wavelength:	500nm
Integration time:	0.1s
Slit:	2nm
Correction:	No
Cycle automatic:	5
Display:	Energy

4. Successively turn the vertical and horizontal adjusting screws and, after every adjustment, measure the energy with the **sample compartment cover closed**. Repeat the procedure until the energy of the sample beam I(M) has reached its maximum value.

14.4 Sample measurement

14.4.1 Settings on the PC

In the device driver, choose the following accessory-specific measurement parameters:

1. In WinASPECT® software, open the device driver.
2. Choose the general measurement parameters (slit, wavelength, ordinate display, etc.).
3. As correction mode, choose either **Reference** or **Special** (correction based on a stored file).
4. On the **Accessory** tab, choose the **Sipper** option.

This will bring up the following options for the use of the sipper:

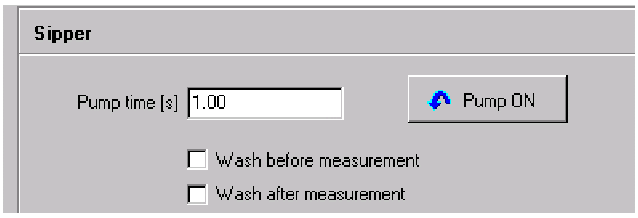


Fig. 14-4 Device driver, Accessories tab with selected Sipper option

For the control of the sipper via WinASPECT®, the following options are available:

Option / Button	Description
Pump time [s]	The pump time is the time needed to optimally transfer the sample into the cell. Because of the rinse effect, approximately 2/3 of the sample should flow through the cell and 1/3 of the sample be before the cell. Adjusting range: 0 ... 300 s
Wash before measurement	Performs a wash step before sample measurement. The wash time is 1.5 x **Pump time**. At the end of the entry procedure, the time will be rounded by software to tenths of a second.
Wash after measurement	Performs an additional wash step after sample measurement.
[Pump ON / OFF]	Click on the **Pump ON** button if you want to check the aspiration process or determine the optimum pump time. The label of this button changes depending on the switching state of the pump.

14.4.2 Performing a sample measurement

Reference measurement

If this Measurement parameter option was selected in the device driver, take a reference measurement. Feed the sample as described in the Section Sample measurement below.

- Start the reference measurement by a click on the corresponding button on the WinASPECT® desktop. Then, follow the procedure described below under Sample measurement.

Sample measurement

Without additional wash steps

- Hold the sample to be analyzed to the aspirating tube.
- Start the measurement by a click on the Measurement start button.

The sample is being aspirated and the measurement started.

With activated "Wash before measurement" option

- On the WinASPECT® desktop, click on the measurement start button.

This will bring up a message window prompting you to hold the wash vessel to the aspirating tube.

- Bring the wash solution to the aspirating tube.
- Start the wash process with **[OK]**.

When the wash process is finished, a message window appears prompting you to prepare the sample to be analyzed.

- Bring the sample to the aspirating tube.
- Start the measurement with **[OK]**.

The sample is being aspirated and the measurement started.

With activated "Wash after measurement" option

- Bring the sample to be analyzed to the aspirating tube.
- Start the measurement by a click on the measurement start button.

The sample is being aspirated and the measurement started.

When the measurement process is finished, a message window appears prompting you to prepare the wash solution.

- Bring the wash solution to the aspirating tube.
- Start the wash process with **[OK]**.

14.5 Care and maintenance

Maintenance

Maintenance on the cassette sipper is restricted to replacing the pump tubes (→ Section “Mounting the tube set”, p. 98).

Pump tubes of other materials or with different inside diameters can be ordered from our Service Department.

Care

Observe the following advice on device care:

- Avoid any contamination on the sample holder. Wipe off spilt samples or reagents instantly with an absorbent cloth or tissue. Remove sticking dirt with a soft cloth moistened with a commercial detergent.
- When you finish using the sipper, fill the flow cell with distilled water. When the cell dries out, sample residues may remain stuck to the flow cell.
- When you finished working with the sipper, detach the tube cassette from the pump rollers thus slackening the pump tube. In this way, the elasticity of the pump tube is maintained longer.

15 APG 53/100 XYZ Autosampler

15.1 Intended use

The APG 53/100 is an XYZ autosampler. Used in combination with a sipper, it serves to transfer the sample to the flow cell in the spectrophotometer. The autosampler is controlled by the PC.

The APG is supplied with a sample rack for 53 sample cups (APG 53) and equipped with a magnetic stirrer. In addition, an upgrade kit is available for a sample rack with 100 sample cups without stirrer (APG 100).

For cleaning the flow-through system, rinse liquid can be aspirated from a rinse vessel.

15.2 Safety notes

Warning!

Electric shock!

Prior to opening the device, switch it off and disconnect the power plug!

Take care that no liquids get onto cable connectors or into the electrical system of the device!

Proper earth conductor!

Connect the power cable only to a properly installed power outlet with earthing contact to ensure protection as per Protective Class I (protective multiple earthing). Do not use an extension cable without earth conductor, as this would make the protection ineffective. If you use multiple socket connectors, make sure the permissible earth current is not exceeded.

Make sure the power cables are free from defects.

Correct line voltage!

Prior to connecting the XYZ autosampler to a power outlet, make sure the operating voltage specified on the rating plate on the rear panel agrees with the local line voltage. Operation on other than the specified line voltage may destroy the device.

The device may be converted for use on other line voltages only by service staff!

Risk of accidents!

Mind the motion range of the guiding arm of the uptake tube! While the device is in operation, there is the risk of accidents!

Do not put your fingers into the guiding aperture of the arm while the device is operating!

Observe the operating manual!

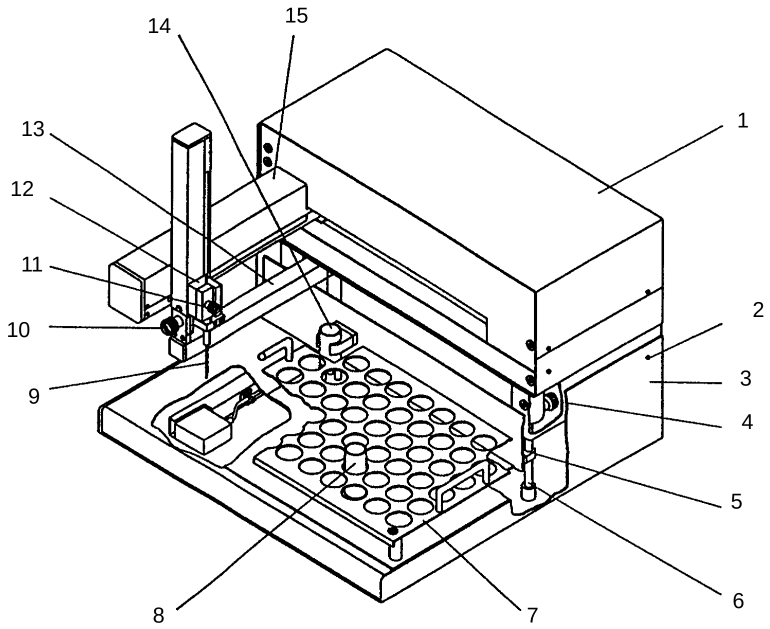
Additionally, observe the instructions and advice given in the accompanying operating manual provided by the manufacturer!

Do not fold the sample tube!

Take care that the sample tube is not folded! Folds in the tube will hinder sample flow.

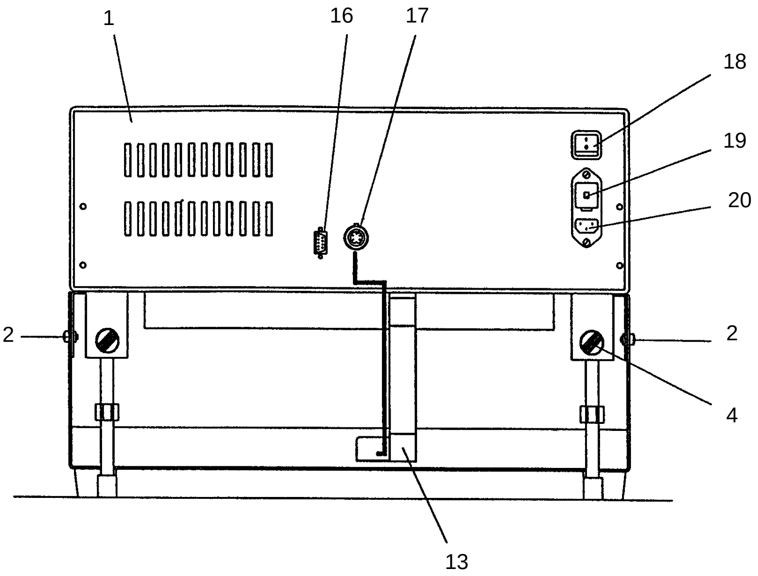
Do not reel up the sample tube in narrow bends!

15.3 Design and installation of the APG 53/100



1	XYZ autosampler basic unit
2	Screws (4x) for fastening the base covering
3	Base covering for the accommodation of the sample rack and for covering the motion range of the magnetic stirrer bracket
4	Knurled screws (4x) for fastening the feet of the device in the foot sockets
5	Plastic fork (2x) for fastening the base covering to the feet of the device
6	Foot of basic unit
7	Sample tray for the accommodation of 53 sample cups and 1 rinse vessel
8	Sample cup
9	Aspirating tube
10	Knurled screw for fastening the stirrer bracket to the Z-dome
11	Knurled screw for fixing the aspirating tube
12	Tube holder
13	Stirring bracket carrying the movable magnetic stirrer underneath the current sample cup
14	Rinse vessel
15	Guiding arm

Fig. 15-1 APG 53 (front view)



1	XYZ autosampler, basic unit
2	Screws (4x) for fastening the base covering to the device
4	Knurled screw (4x) for fastening the feet of the device in the foot sockets
13	Stirrer bracket carrying the movable magnetic stirrer underneath the current sample cup
16	Connector for connection to SPECORD®
17	Diode connector for connection of magnetic stirrer
18	Power switch assembly
19	230V/115V line voltage selector
20	Appliance mains input connector

Fig. 15-2 Connections of APG53 (rear panel)

Installation of XYZ autosampler

When installing the autosampler, additionally observe the description provided by the manufacturer.

1. Put the XYZ autosampler on its rear side.
2. Remove the red transport lock. To this end, unscrew the two screws located on the underside of the device.

Further installation procedure for the APG 53 model

3. Push the four feet into the foot sockets and fasten the feet with knurled screws (4). Put the XYZ autosampler upright on the table and check its stability. Readjust the feet, if necessary.
4. Slide the stirrer bracket onto the underside of the Z dome into the grooves.
   Align the bracket so that it is centered.
   Tighten the knurled screw (10).
   If mounted properly, the middle of the stirrer plate is exactly underneath the tube holder. The bracket points to the back and projects under the basic device.

5. Put the base covering from the front onto the basic device and fasten it by means of the four bolts (2).
   The stirrer bracket is now under the base covering without touching it.
6. Put the sample rack onto the base covering. The rack rests on the conical plug and the stop strips on the base covering.
7. Insert the rinse vessel, if required.

Further installation procedure for the APG 100 model

Use the upgrade kit for the installation of the APG 100.

3. Push the four longer feet into the foot sockets and fasten the feet with the knurled screws (4).
   Put the XYZ autosampler upright on the table and check its stability. Readjust the feet, if necessary.
4. In place of the stirrer bracket of the APG 53, slide the adapter with the duct for the sample tube onto the underside of the Z dome into the grooves.
   Tighten the knurled screw (10).
5. Put the base covering from the front onto the basic device and fasten it by means of the four bolts (2).
6. Put the sample rack onto the base covering. The rack rests on the conical plug and the stop strips on the base covering.

15.4 Connecting the XYZ autosampler to the SPECORD®

1. Set up the autosampler to the right of the SPECORD® keeping a minimum spacing of 10 cm.
2. Use the provided cable to interconnect the 15-pin connector (16) of the XYZ autosampler and the connector labeled “APG” on the right side of the SPECORD®.
3. Verify that the operating voltage setting of the XYZ autosampler (label on (19)) agrees with the available line voltage.
4. Connect a power cable to mains input connector (20). Then, connect this cable to a power outlet.
5. Put the sipper into the sample compartment and connect it.

Inserting the sample tube

Before adjusting the sample tube, make sure the autosampler is switched off!

The sample tube is supplied ready for use. The tube consists of

- an MFA tube for sample transfer,
- the tube connection to the flow cell (silicone tube with black stopper) and
- a piece of tube for stabilization (black) and improved guidance of the MFA tube.

1. Shift the MFA tube within the black guiding tube so that the following tube length juts out from the guiding tube:

APG 53	80 mm
APG 100	100 mm

In the following adjustment, take care that the MFA tube is not displaced within the guide tube and the above lengths be kept.

2. Slip the Tygon® tube piece of the sample tube over the inlet connector of the flow cell.
   In connecting the tube observe the directions of flow possibly marked on the flow cell.
3. Thread the MFA tube together with the tube for stabilization from inside through one of the ducts (Fig. 1-1 / 12) in the front wall of the sample compartment to the outside.
4. For the time being, clamp the guide tube only loosely in the tube holder (12) of the XYZ autosampler.
   Insert a sample cup in the sample tray.
5. With the **autosampler switched off (!)** move the guide arm (15) of the tube holder until the sample tube is in the middle above the sample cup. Move the tube holder (12) down as far as it will go.
6. Push the sample tube down until it is about 1 – 2 mm above the bottom of the cup. When using a magnetic stirrer, put the stirrer magnet first into the cup. Adjust the immersion depth so that the MFA tube does not touch the magnet.
7. Make sure that the black guide tube does not immerse in the sample.
   If the guide tube immerses in the sample, push it up. Then, move down the MFA tube within the guide tube until its end is again about 1 – 2 mm above the bottom of the cup or the stirrer magnet.
8. Tighten the knurled screw (11) on the tube holder (12).

Switching on the XYZ autosampler

Caution! Correct order of switch-on!

Make sure to follow the above order in switching on the system to ensure that the autosampler is moved to the correct starting position. If you should have switched off the autosampler in the mean time, restart the software once more after switching on the autosampler.

Switch on autosampler and SPECORD® in the following order:

1. Switch on autosampler (18, Fig. 15-2).
2. Switch on PC and SPECORD®
3. Start WinASPECT® software and initialize the SPECORD®.
4. Activate the device driver (measurement parameter dialog box).

15.5 Sample measurement

In the Device Driver, choose the following accessory-specific measurement parameters:

1. In WinASPECT®, open the Device Driver.
2. Choose the general measurement parameters (slit, wavelength, ordinate display, etc.)
3. Under **Correction**, choose the **Reference** option or use a saved file (**Special** option).
4. On the **Accessories** tab, choose either **APG 53** or **APG 100** depending on the sample rack used (53 or 100 samples).

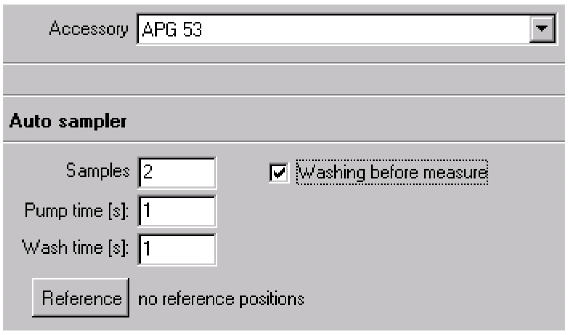


Fig. 15-3 Accessories tab – APG 53 selected

Parameter / Button	Description
Samples	Number of samples placed on the sample rack (including reference and wash solution). Setting range for APG 53: 1-53 Setting range for APG 100: 1-100 Sampling always starts with rack position 1. Make sure to fill the tray consecutively without leaving any empty places in between.
Pump time [s]	The pump time is the time needed for optimum transfer of the sample to the cell. Setting range: 0 ... 300 s
Wash before every sample	Executes a cleaning step between two sample measurements. For that, the aspiration tube dips into the wash vessel. When you enter a wash time, this step includes the aspiration of the wash solution and rinsing of the sipper system.
Wash time	Pump time for the wash solution. Setting range: 0 ... 300 s
[Reference]	Use of one or several references on the sample rack. After a click on **[Reference]**, a small dialog box appears showing the list of samples. Mark the tray position(s) accommodating the reference(s). The selected positions appear beside the [Reference] button. Maximum number of selectable references: 10

Taking the sample measurement

1. Load the samples on the sample rack and insert the rack in the autosampler.
2. Start the measurement by a click on the corresponding toolbar button on the WinASPECT® desktop.

The measurement is started immediately. The reference measurements are performed and considered according to the options selected in the measurement parameters.

15.6 Finding the appropriate pump time

To find the appropriate pump time, use a liquid having a viscosity similar to that of the sample to be analyzed.

1. Put a vessel containing the test liquid in the sample holder.
2. With the **sampler switched off (!)**, move the guide arm of the device over the vessel and push the aspiration tube holder down as far as it will go so that the aspiration tube dips into the test liquid.

To ascertain the optimum pump time, use the parameter options of the sipper.

3. In the Device Driver, on the **Accessories** tab, choose the **Sipper** option.
4. Choose the desired **Pump time** and save the measurement parameters.
5. Start a measurement by a click on the corresponding button on the WinASPECT® desktop.
6. Watch whether the liquid has flown through the cell. The pump time is optimum, if about 2/3 of the sample volume has flown through the cell and 1/3 of the sample is in the aspirating tube before the cell. That way, the cell can be rinsed with the sample before the measurement and carry-over errors be minimized.
7. Enter the found optimum pump time in the measurement parameters of the XYZ sampler.

16 Fiber coupling for measuring probes

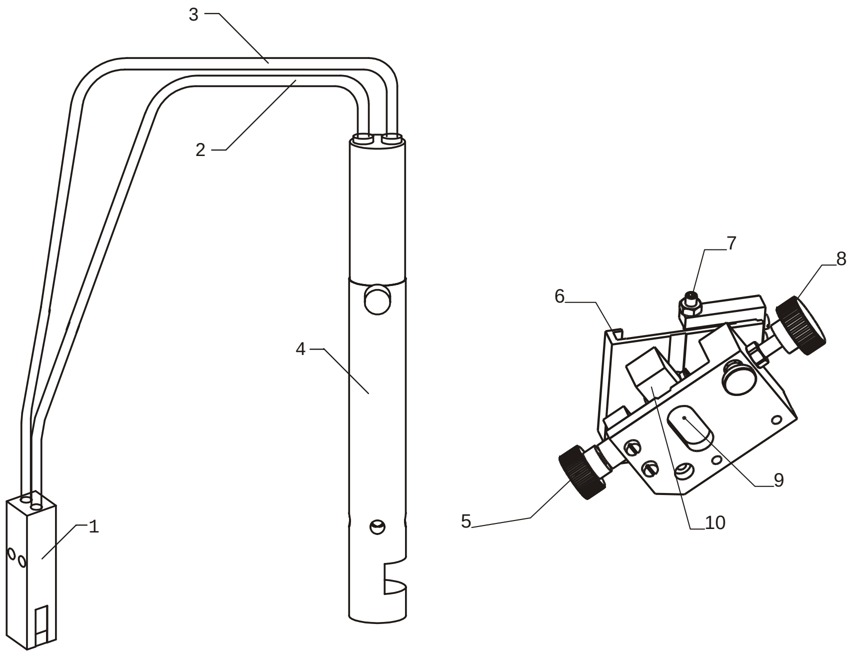
16.1 Description and use

The fiber coupling provides optical connection of measuring probes with their optical fibers to the SPECORD®. It allows samples to be analyzed outside the sample compartment. The operating range of the optical fibers is from 220 nm to 1100 nm.

Caution! Aging of optical fibers by exposure to UV radiation!

Please note that optical fibers may be damaged in the long term if used at wavelengths below 220 nm. That is why you should use them only in the wavelength range above 220 nm.

16.2 Design and function of fiber coupling with measuring probe



1	Adapter for coupling light in/out	6	Holder
2	Optical fiber for coupling light in	7	Setscrew for vertical adjustment
3	Optical fiber for coupling light out	8	Knurled screw
4	Measuring probe (schematic)	9	Slit position
5	Spring-loaded handle	10	Adapter (1) - schematic

Fig. 16-1 Fiber-coupling adapter with measuring probe

The fiber coupling consists of adapter (1), which couples out the light from the sample compartment and sends it into the optical fiber (2). The optical fiber transmits the light to the measuring probe (4). A second optical fiber (3) transmits the light attenuated by the sample in the measuring probe back to the adapter (1). The adapter couples the attenuated light into the sample beam.

Caution! Extraneous light!

Like with all other measurements, too, take care that no extraneous light enters the sample compartment. For that, it is necessary to make sure that the measuring probe does not capture any extraneous light. Among others, use the facilities for shielding extraneous light provided by the probe manufacturer.

16.3 Design and function of the fiber coupling with SMA connectors

In this case, the adapter (Fig. 16-1 / 1) contains two optical fibers that are equipped with SMA connectors each.

16.4 Installation of the adapter in the sample compartment

Caution! Do not squeeze the optical fibers!

When you close the sample compartment cover, take care that the optical fibers are not squeezed.

1. Slide the adapter holder onto the cell-holder mount of the sample beam in the sample compartment wall (Fig. 1-1 / 10).
2. Put the adapter through at the back underneath the sample compartment cover. Remove the lid from the rear circular opening of the sample compartment cover. From the back, put the adapter through this opening.
   Close the opening again with the lid.
3. Insert the adapter in the adapter holder.
4. Put the optical fibers between the plastic strips in the opening for the optical fibers (Fig. 1-1 / 5) in the sample compartment.
5. Close the sample compartment cover taking care that the optical fibers rest in the groove of the device cover under the sample compartment cover. The optical fibers must not be squeezed by the cover.

16.5 Alignment of fiber coupling

If the fiber coupling is used for the first time, the adapter with its holder will have to be aligned in the beam path.

If you use optical fibers with SMA connectors, first connect the fibers with the measuring device to be used for the analysis.

To do this, proceed as described in Section “Adjustable holder for microcells, non-thermostatted (8.5 mm beam height)” p. 23.

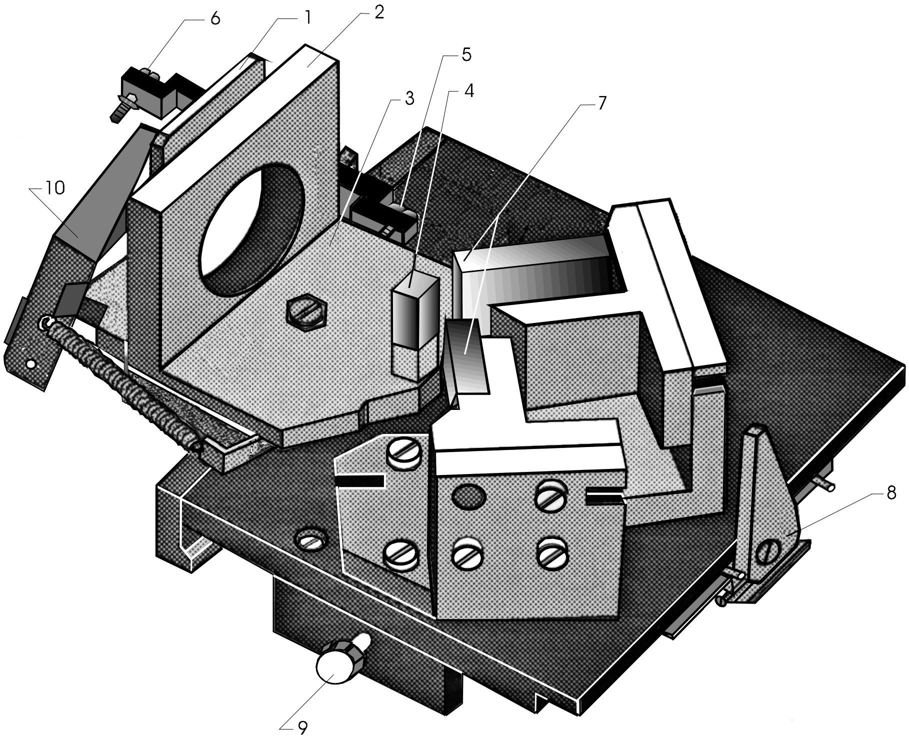
17 Absolute Reflectance Attachment

17.1 Description and use

With this accessory, you can determine the absolute reflectance of reflecting layers. The layers must be homogeneous, smoothly polished and have a constant thickness.

This accessory accommodates samples of a size between 40 x 40 mm and 70 x 70 mm and a thickness of 1 ... 20 mm. The angle of reflection is 7°.

17.2 Design



1	Sample	6	Stop screw for "V" beam configuration
2	Sample locating plate	7	Fixed mirrors
3	Carrier for swivel mirror	8	Clamping lever
4	Swivel mirror	9	Stop screw to sample compartment wall
5	Stop screw for "W" beam configuration	10	Sample holder

Fig. 17-1 Absolute reflectance attachment

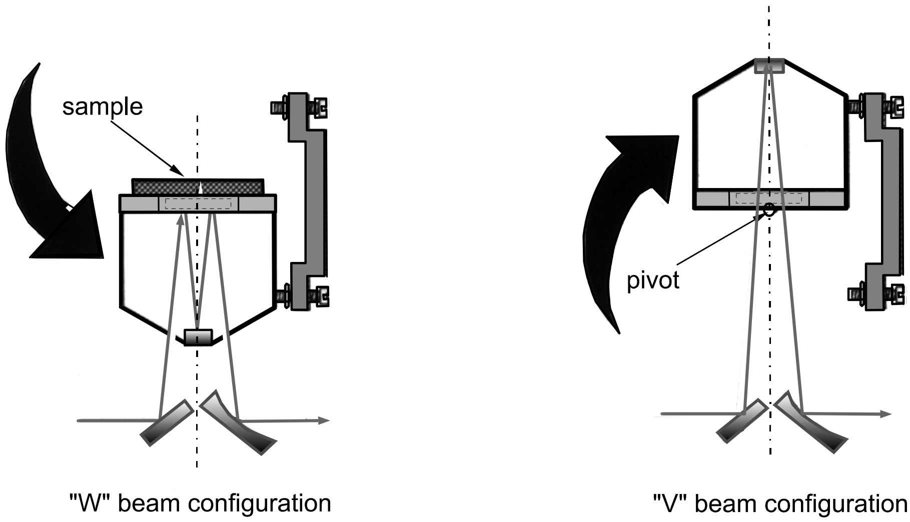
17.3 Installation in sample compartment

Caution! Contamination of mirrors!
Do not touch any mirror surfaces!

1. Put the accessory onto the front carrying rails of the sample compartment with its clamping lever (8) pointing frontward.
2. Push the accessory to the right-hand wall of the sample compartment as far as it will go. Secure it in position by throwing over clamping lever (8) to the right.

17.4 Operating principle

The absolute reflectance of the sample is determined in two steps:



Reference measurement Sample measurement

Fig. 17-2 Beam geometry in Absolute Reflectance Attachment

Reference measurement

- Without any sample inserted turn carrier (3) with swivel mirror (4) up to the stop of the **"V" beam configuration**.

Sample measurement

- Turn carrier (3) with swivel mirror (4) by 180° in the position for "**W" beam configuration**.
- Withdraw sample holder (10) from locating plate (2).
  Put sample (1) to locating plate and let sample holder return until it touches the sample.

The measurement is taken at a reflecting angle of 7°.

17.5 Adjustment

The Reflectance Attachment has been adjusted for the use on SPECORD® 200/205/210. You can use it on these SPECORD® models without any additional adjustment.

If you want to use a reflectance attachment already available, it is advisable to check the adjustment of this unit. For that, follow this procedure:

1. Place the reflectance attachment onto the two front carrying rails in the sample compartment. Push it to the right-hand wall of the sample compartment as far as it will go. Clamp it in this position by means of clamping lever (Fig. 17-1 / 8).
2. Set "V" beam configuration.
3. Adjust the zeroth order of the monochromator (→ Section Adjustment of zeroth order, p. 8).
4. Turn stop screw (Fig. 17-1 / 6) to adjust the unit so that the beam passes through the center of the cell receptacle for turbid samples thus being incident on the center of the detector surface. Put a white screen, e.g. a white paper strip, in the cell receptacle for turbid samples.
5. To adjust the unit in "W" beam configuration, put a flat mirror against the sample locating plate (Fig. 17-1 / 2). Then, proceed as described above for the adjustment of the "V" beam configuration. To adjust the unit, turn stop screw (Fig. 17-1 / 5).

17.6 Measurements with the Absolute Reflectance Attachment

1. Create the desired measurement parameter record in WinASPECT® software.
   For the reflectance measurements, choose the following accessory-specific options:

Measurement parameter	**Option to be activated**
Correction	Reference
Display	Reflectance
Accessory	Reflectance / fixed angle

2. Take the reference measurement. For that, set the reflectance attachment to the "V" beam configuration shown above.
3. When the reference measurement is finished, turn the unit to set it for "W" beam configuration.
4. Withdraw sample holder (10) from sample locating plate (2), insert the sample (1) and let the sample holder return to the sample.
5. Take the sample measurement.

Calculation of reflectance

The measured reflectance R is calculated from the (sample measurement) / (reference measurement) ratio.

$$R = \frac{r_{Sample}}{r_{Reference}}$$

$$r_{Reference} = \frac{I_{RS}}{I_{reference\,beam}}$$

$$r_{Sample} = \frac{I_{PS1} \times I_{RS} \times I_{PS2}}{I_{Reference\,beam}}$$

R_{Sample} – Sample measured in W configuration

$r_{Reference}$ - Reference measurement in V configuration

I_{RS} – Intensity after reflection at the reference mirror

$I_{Reference\ beam}$ – Intensity in reference beam path

I_{PS1} – Intensity after the first reflection at the sample

I_{PSl2} – Intensity after the second reflection at the sample

The result of the measurement is:

$R = I_{PS1} \times I_{PS2}$

If the sample is sufficiently homogeneous, it applies $I_{PS1} = I_{PS2,.}$ The absolute reflectance of the sample R_{abs} resulting from this is:

$R_{abs} = \sqrt{R}$.

In the graph and in the result table, R_{abs} is presented in percent.

17.7 Care and maintenance

Caution! Contamination of reflectance attachment!

Take care not to damage the mirrors by contamination. Observe the instructions on the care of mirrors.

- Keep all mirrors of the reflectance attachment free of dust and grease!
  Don't touch mirror surfaces with bare fingers!
- Remove dust particles on mirrors using a soft, clean and grease-free brush.
- Carefully wipe off traces of grease from mirrors using a cotton swab soaked with distilled water and curd soap. Don't exert any pressure in wiping.
  Then, wipe with a new cotton swab soaked with distilled water only.
  Finally, dab metal mounts dry.
- Carry and store the absolute reflectance attachment only in a closed box.

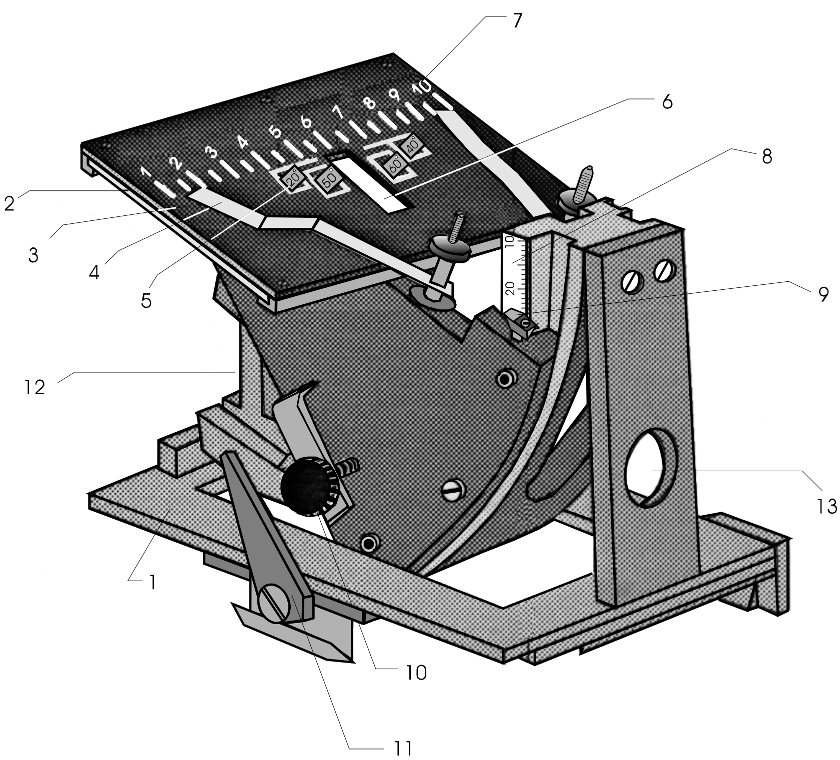
18 Variable Angle Reflectance Attachment

18.1 Description and use

With this accessory, you can determine pathlength and refractive index of solid samples. This is done by measuring the reflectance at different angles of reflection over a defined wavelength range.

For the determination of pathlength, the interferences that occur during the measurement are utilized by evaluating the number of interference maxima appearing in the defined wavelength region. Prior to sample measurement, you must take a reference measurement inserting the provided mirror in place of the sample.

18.2 Design and operating principle



1	Base plate	7	Scale for reproducible sample positioning
2	Table	8	Scale for adjusting the angle of reflection
3	Large tabletop	9	Index for reading the set angle of reflection
4	Sample holding clip	10	Knurled screw for locking the angular setting
5	Marks indicating the illuminated sample area for defined angles of reflection	11	Clamping lever
		12	Beam entrance
6	Measuring aperture	13	Beam exit

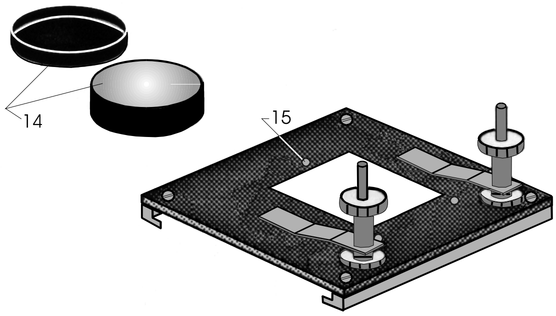
Fig. 18-1 Reflectance attachment with large sample table

The accessory has a base plate (1) that accommodates imaging and reflecting optics as well as the sample table.

The sample table can be swiveled horizontally to vary the **angle of reflection in the range 11° – 60°**. The optical path of the measuring beam is the same for all angles that can be set.

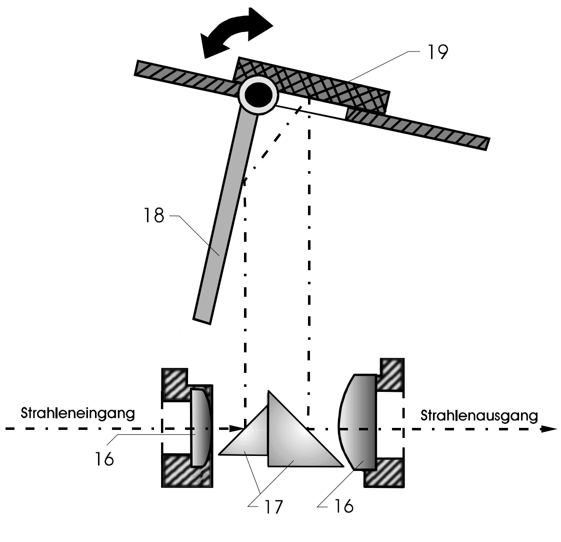
You can slide either a large tabletop with small measuring aperture – preferably for small samples – or a small tabletop with large measuring aperture – preferably for larger samples – onto the table. The samples are clamped to the tabletop. The large tabletop rigidly lies on the table during the measurement. The marks on the tabletop provide reproducible positioning of the sample.

The small tabletop permits the sample on the table to be gradually shifted and measured, for instance, for testing its homogeneity. Here, the marks on the table permit the sample to be positioned reproducibly.



14 Mirror for reference measurement and protective cap
15 Point support to protect the sample surface from scratches

Fig. 18-2 Mirror and small tabletop



16 Quartz lens
17 Reflecting mirror
18 Plane mirror
19 Sample

Fig. 18-3 Optical path in reflectance attachment (front view)

18.3 Installation in sample compartment

1. Put the accessory onto the two rear carrying rails of the sample compartment with the clamping lever pointing frontward.
2. Push it to the right-hand wall of the sample compartment as far as it will go. Secure it by throwing clamping lever (11) to the right.

18.4 Adjusting the angle of reflection

Loosen knurled screw (10) to set the sample table to the desired angular position. Retighten knurled screw (10) to lock the sample table in position.

Note

In the angular range from 11° to 15°, the beam is partly masked thus impairing the signal-to-noise ratio.

18.5 Measuring with the reflectance attachment

1. Create the desired measurement parameter record in WinASPECT® software.
   For the reflectance measurements, choose the following accessory-specific options:

Measurement parameter	Option to be activated
Correction	Reference
Display	Reflectance
Wavelength range	Sufficiently large to get a sufficient number of interference maxima, if you intend to carry out a layer thickness measurement.
Accessory	Reflectance (variable)

2. Place the provided reference mirror onto the tabletop (3) and clamp it with the sample holding clips (4). Carry out the reference measurement with this mirror.
3. Replace the reference mirror by the sample to be measured. Carry out the sample measurement.

18.6 Calculating the reflectance

The measured reflectance R is calculated from the (sample measurement) / (reference measurement) ratio.

$$R = \frac{r_{Sample}}{r_{Reference}}$$

$$r_{Reference} = \frac{I_{RS}}{I_{reference\,beam}}$$

R_{Sample} – Sample measured in W configuration

$r_{Reference}$ - Reference measurement in V configuration

I_{RS} – Intensity after reflection at the reference mirror

$I_{Reference\ beam}$ – Intensity in reference beam path

$$r_{Sample} = \frac{I_{PS}}{I_{Reference\,beam}}$$

I_{PS} – Intensity after the reflection at the sample

The result of the measurement is the relative reflectance of the sample relative to the reference mirror R_{rel}:

$$R_{rel} = R = \frac{I_{PS}}{I_{RS}}$$

In the graph and in the result table, R_{rel} is presented in percent.

18.7 Calculating pathlength and refractive index

If the refractive index of the sample is know, you can calculate the pathlength of the sample using the following formula:

$$d = \frac{m \cdot \frac{\lambda_2 \cdot \lambda_1}{2(\lambda_2 - \lambda_1)}}{\sqrt{n^2 - \sin^2 \Theta}} \qquad ; \qquad \lambda_2 > \lambda_1$$

d	Thickness of sample
m	Number of interference maxima after zeroth order maximum (see Example)
n:	Refractive index of sample
Θ :	Adjusted angle of reflection
λ_1:	Wavelength of zeroth interference maximum
λ_2:	Wavelength of m[th] interference maximum

Wavelengths λ_1 and λ_2 must be determined as accurately as possible. For that, in WinASPECT® software, use the **Peaklist** function of the **Data Handling** menu.

Example: Measuring curve

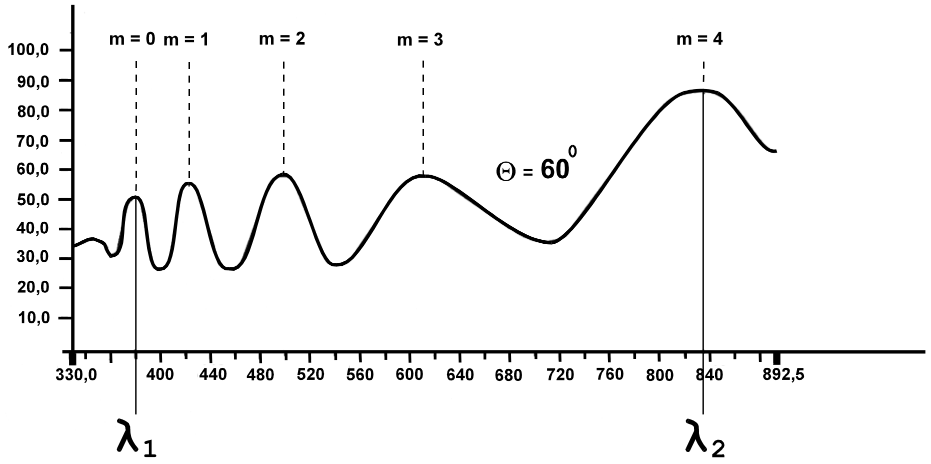


Fig. 18-4 Example of interferences at an angle of reflection Θ = 60°

The curve shown above has been scanned at an angle of reflection of 60°.
It contains maxima of zeroth to fourth order.

18.8 Care and maintenance

Caution! Contamination of reflectance attachment!

Take care not to damage the mirrors by contamination. Observe the instructions on the care of mirrors.

- Keep all mirrors of the reflectance attachment free of dust and grease! Don't touch mirror surfaces with bare fingers!
- Remove dust particles on mirrors using a soft, clean and grease-free brush.
- Carefully wipe off traces of grease from mirrors using a cotton swab soaked with distilled water and curd soap. Don't exert any pressure in wiping. Then, wipe with a new cotton swab soaked with distilled water only. Finally, dab metal mounts dry.
- Carry and store the absolute reflectance attachment only in a closed box.

18.9 Technical data

Angle of reflection	11° – 60°
Graduation of angular scale	1°
Accuracy of angular adjustment	0.5°
Vignetting of beam	in angular range 11° ... 15°
Minimum sample size	12 mm x 10 mm
Maximum sample thickness	30 mm
Illuminated sample area	2.5 mm x 6 mm to 2.5 mm x 12 mm, depending on adjusted angle of reflection
Reference sample	Aluminized mirror with protective coating
Dimensions	165 mm x 115 mm x 135 mm
Size of large tabletop	115 mm x 80 mm
Size of small tabletop	70 mm x 80 mm
Weight	ca. 2 kg

19 Integrating Sphere

19.1 Description and use

The integrating sphere is suitable for the measurement of transmittance and diffuse reflectance of scattering solid or liquid samples on these spectrophotometers.

In diffuse reflectance measurements, the measuring geometry of the sphere is 8°/d.

Consider that due to the effect of the sphere the level of the measured signal drops to a small percentage compared to the optical path without sphere. This means that you can take measurements at a high accuracy only with an absorbance of up to about 2 A.

The integrating sphere with a sphere diameter of 75 mm consists of two Spectralon® hemispheres with apertures for beam entrance and exit, beam reflecting optics and holders for samples and cells for transmittance and diffuse reflectance measurements. It is inserted in the optical path of the SPECORD® sample compartment. For loading powdery samples, it may be removed from the sample compartment and reproducibly reinserted.

Caution! Observe the following conditions of use!

With its uncovered mirrors and high-reflectivity Spectralon® components, the integrating sphere is a very sensitive optical device. Especially when handling powdery samples, take care to avoid that powder or other dirt particles get into the sphere and thus reduce their reflectivity. A decrease in reflectivity of the Spectralon® units from 99% to 98% will result in a reduction to half of the standard efficiency of the integrating sphere. The Spectralon® inserts and the gloss trap, too, should be treated with the same care, as the efficiency of the unit will also be reduced by a decrease in the reflectivity of these components.

Unpacking

Carefully remove the sphere body from the transport and storage box taking care not to touch the mirrors. Hold the sphere body only at the side plates.

Storage

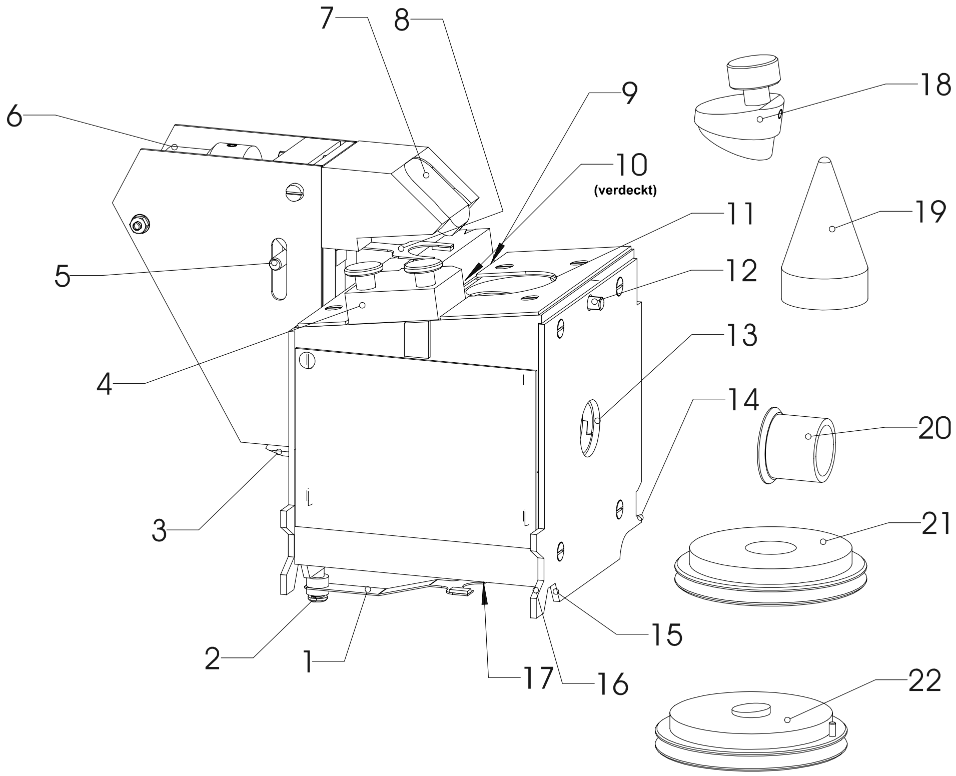
In periods of non-use of the integrating sphere, store it along with all its accessory items in the corresponding box. Though the mirrors carry a protective coating of SiO_2, you should protect them from contamination. Besides, take care that you remove the reflector from the SPECORD®, if you do not use the integrating sphere. Otherwise, its bright aluminum surface might be affected.

Cleaning

Despite all care it may become necessary to clean the mirrors, particularly mirror 3 (Fig. 19-1 / 7). For that, only use dry, dust-free and oil-free compressed air or nitrogen.

You may also clean the Spectralon® locating surfaces. For that, use dry and clean compressed air, then rinse the surfaces with distilled water and finally blow them dry. To remove sticky dirt from these surfaces, you may also use fine-grained abrasive paper (grain size 220 – 240). Subsequently wash the surfaces with distilled water and then dry them with compressed air.

19.2 Design of integrating sphere



1	Retaining clip 1 for reflectance sample	12	Knurled locating pin
2	Guide pin for retaining clip 1	13	Aperture to detector angle
3	Mirror 1	14	Locating surface 1 for horizontal mounting
4	Adjustable stop for cells	15	Locating surface for vertical mounting
5	Handle for retaining clip	16	Locating surface 2 for horizontal mounting
6	Mirror 2	17	Aperture for reflectance measurements
7	Mirror 3	18	Spectralon® insert in place of gloss trap
8	Retaining clip 2 for transmission samples	19	Gloss trap
9	Guide for cells	20	Reflector
10	Aperture for transmittance measurements	21	Holder for powdery samples
11	Aperture for gloss trap	22	Spectralon® insert for reflectance aperture

Fig. 19-1 Integrating sphere with accessory items

The integrating sphere is shown in that position it is inserted into the SPECORD® for reflectance measurements of powdery samples (vertical arrangement). With this arrangement, the reflectance sample is located underneath the sphere body.

The sphere body may also be inserted swiveled by 90° (horizontal arrangement) for transmittance measurement of solid or liquid samples in cells.

It is placed with its locating surfaces (14), (15) or (16) onto the carrying rails in the sample compartment of the SPECORD®.

19.3 Measurements with the integrating sphere

In transmittance or diffuse reflectance measurements, the sample is brought into direct contact with the sphere. In this way, the sample presents a part of the inner sphere surface thus affecting the efficiency of the sphere.

So, for instance, in transmittance measurement, the radiation is weakened by the absorption in the sample. As the sample itself, however, is part of the sphere, the sphere efficiency changes depending on the reflective or reflectance properties of the sample. This means that the efficiency of the sphere depends on sample properties. This dependence results in a non-linear functional relation between sample absorbance and the measured signal.

For high-precision measurements, it is therefore advisable to take this non-linear relation into account by calibrating the system by analogy with quantitative analysis.

For the calibration, certified standards should be used.

19.4 Transmittance measurements

The integrating sphere causes even illumination of the detector surface in the spectrometer independent of any influence (scatter, deflection) of the beam through the sample. In this way, the accuracy of results is improved.

For the analysis of solid transparent samples of a large optical pathlength, measurements with the integrating sphere are the only way to avoid the systematic measuring error otherwise caused by the influences on the beam.

Fig. 19-2 illustrates the optical path in transmittance measurements of a scattering sample.

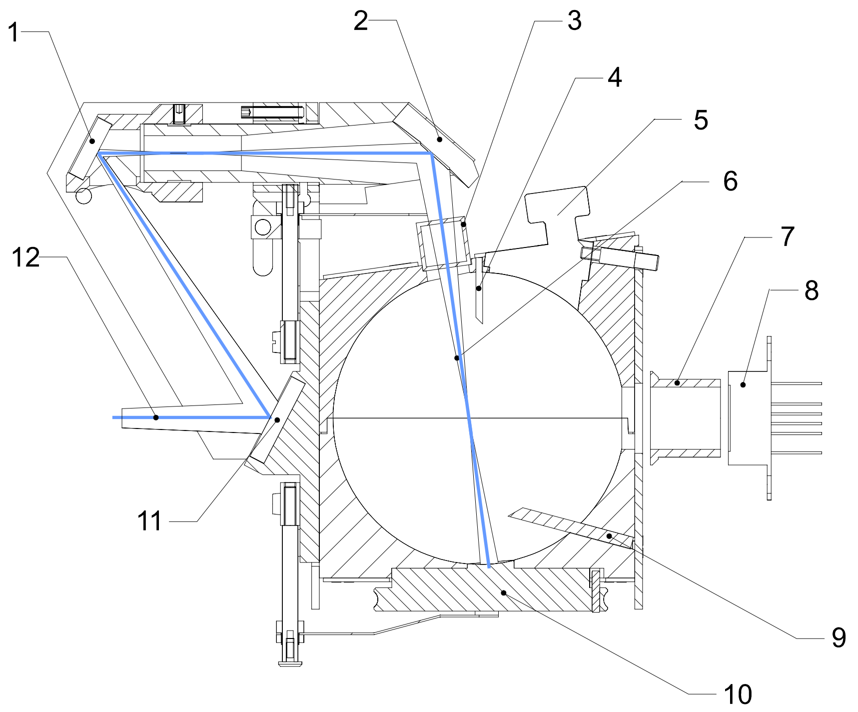


Fig. 19-2 Optical path in transmittance measurements

1	Mirror 2	7	Reflector
2	Mirror 3	8	Detector of SPECORD®
3	Cell with scattering sample	9	Baffle plate 2
4	Baffle plate 1	10	Spectralon® insert for reflectance aperture
5	Spectralon® insert in place of gloss trap	11	Mirror 1
6	Non-scattered radiation	12	Beam path of SPECORD®

Baffle plate 1 (Fig. 19-2 / 4) prevents the direct light of the sample from being incident on the detector surface. Baffle plate 2 (Fig. 19-2 / 9) prevents the light not scattered by the sample from being incident on the detector of the SPECORD® after the first reflection by the SPECORD® insert (Fig. 19-2 / 10).

If you want to measure samples in cells, you can vary the height of beam transmission through the cell by adjusting the stop (Fig. 19-1 / 4). In this way, you can set the following beam heights: 8.5 and 15 mm.

For the transmittance measurement of larger solid samples, you may remove the stop (Fig. 19-1 / 4) and the knurled screw on the Spectralon® insert (Fig. 19-1 / 18). You can place the sample onto the thus arising smooth surface.

Preparing the measurement

1. First, insert reflector (Fig. 19-1 / 20) as far as it will go into the round aperture in the middle of the rear mount for the cell holder. The flange of the reflector should rest on the mount.
2. Insert the Spectralon® insert (Fig. 19-1 / 18) in aperture (Fig. 19-1 / 11). Screw in locating pin (Fig. 19-1 / 12) to ensure correct position of the insert.
3. Adjust stop (Fig. 19-1 / 4) to the desired beam transmission height.

4. If required by the type of sample, unscrew the stop (Fig. 19-1 / 4) and the knurled screw on the Spectralon® insert (Fig. 19-1 / 18).
5. Place the integrating sphere onto the two front carrying rails and push it to the right as far as it will go. Make sure to position it reproducibly against the stop for every measurement. Insert the unit in the way appropriate for the type of sample to be analyzed:

Position for transmittance measurement of solid samples

Install the sphere body either with the carrier of mirrors 2 and 3 (Fig. 19-1 / 6 and 7) pointing upward (vertical installation) or with the carriers of the two mirrors rotated by 90° and pointing to the front side of the instrument (horizontal installation).

Position for the measurement of liquid samples in cells

Install the sphere body with the carrier of the two mirrors rotated by 90° and pointing to the front side of the instrument (horizontal installation).

Transmittance measurement procedure

1. In the Device Driver dialog of WinASPECT® software, create the desired measurement parameter record.
   Choose the following accessory-specific options for the transmittance measurement:

Measurement parameter	Option to be activated
Correction	Reference
Slit	As wide as possible to obtain a high energy level.

2. Insert the two Spectralon® inserts for the gloss trap (Fig. 19-1 / 18) and the reflectance aperture (Fig. 19-1 / 22) in the sphere body.
3. Carry out the reference measurement. For that, you may insert a standard.
4. Insert the sample and fix it by means of retaining clip 2 (Fig. 19-1 / 8) by moving it towards the sample with the handle (Fig. 19-1 / 5) until it is slightly clamped. Make sure to install the sphere body in the SPECORD® exactly in the same way as you did for the reference measurement. Then, carry out the sample measurement.

If you want to take very accurate transmittance measurements at a defined wavelength, first calibrate the system using several certified standards same as in **Quantitative Analysis**. Then, as you are used to do from concentration determination in Quantitative Analysis, measure the unknown sample based on the found calibration curve.

19.5 Reflectance measurements

The colored appearance or the gray tone of an opaque body is due to its wavelength-dependent back reflection (reflectance). Reflectance measurements can be performed with different illumination and measurement geometries depending on the surface structure of the sample.

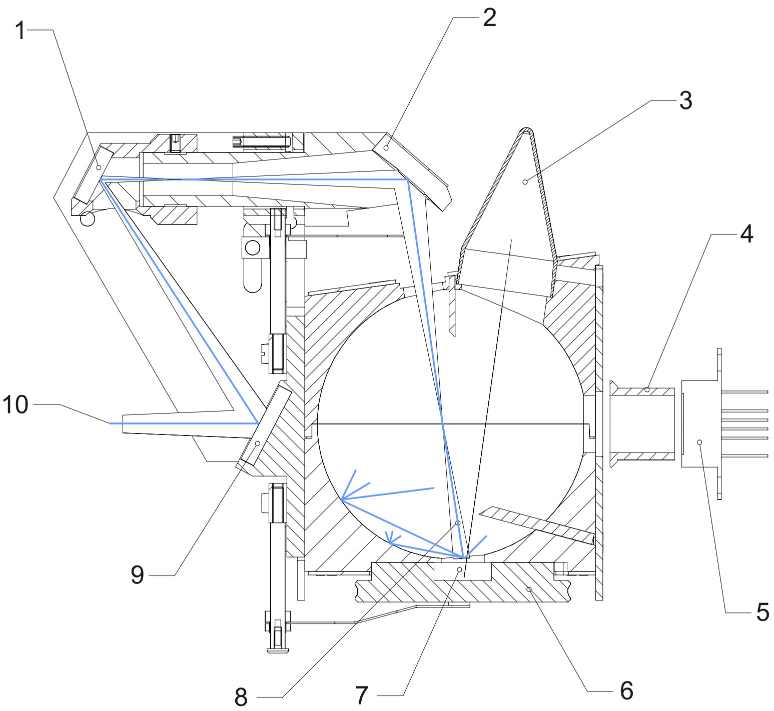
To preclude the effect of the surface structure as far as possible, the sample surface is diffusely illuminated via the inside wall of the integrating sphere. The sphere captures the radiation reflected by the sample and sends it diffusely to the detector of the SPECORD®.

The measurement geometry of 8°/d (d = diffuse) means, that the surface of the sample is illuminated at an angle of 8° relative to the surface normal and the light reflected by the sample surface into the integrating sphere is diffusely sent to the detector.

The reflectance of the sample is the ratio of the radiation reflected from its surface and the radiation reflected from a completely matt white surface of a standard (e.g. a Spectralon® standard) under the same optical conditions.

The integrating sphere is preferably used for reflectance measurements of textured (rough, grained, etc.) surfaces, such as cellulose, leather or fabrics and of samples with azimuthal gloss, i.e. a gloss that varies while rotating the sample about its surface normal.

Fig. 19-3 illustrates the optical path in reflectance measurements.



1	Mirror 2	6	Spectralon® insert for reflectance aperture
2	Mirror 3	7	Reflectance aperture for reflecting powder sample
3	Gloss trap	8	Diffuse reflectance
4	Reflector	9	Mirror 1
5	Detector	10	Optical path of SPECORD®

Fig. 19-3 Optical path in reflectance measurements

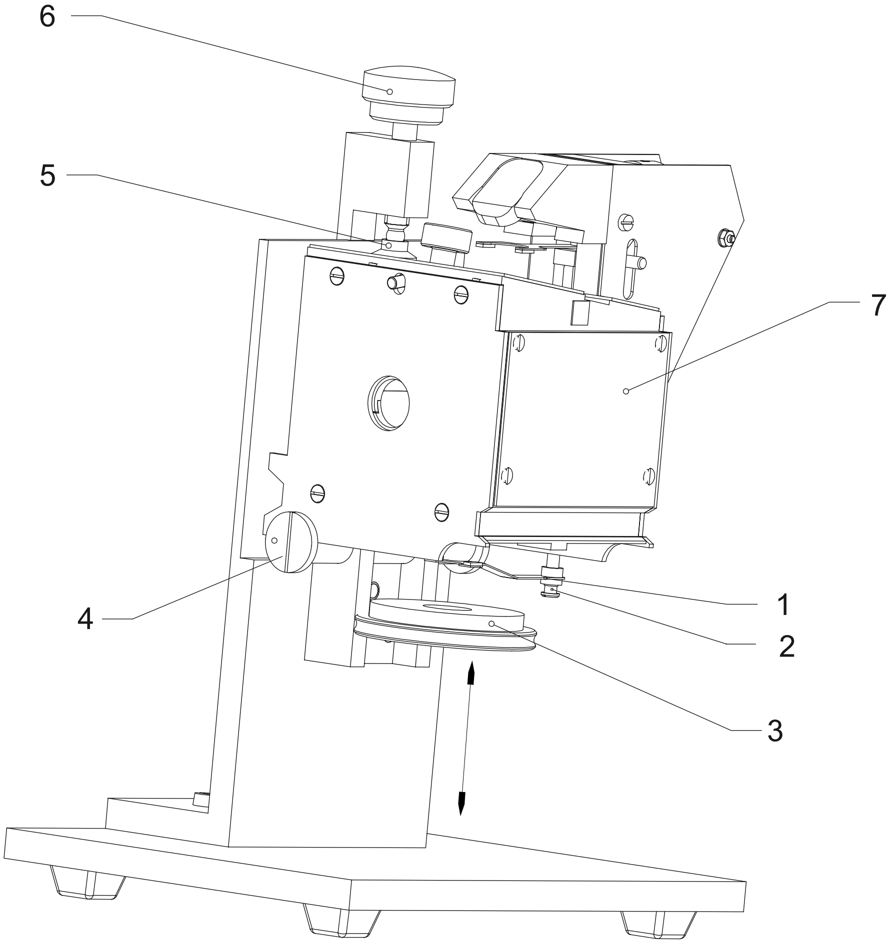
Preparing the measurements

5. First, insert reflector (Fig. 19-1 / 20) as far as it will go into the round aperture in the middle of the rear mount for the cell holder. The flange of the reflector should rest on the mount.

6. You can take reflectance measurements of solid and powder samples. For the measurement of solid samples it is advisable to insert the sphere body in such a way that the carrier of mirrors 2 and 3 (Fig. 19-1 / 6 and 7) points to the back and aperture (Fig. 19-1 / 17) is well visible. For the measurement of powder samples, insert the sphere in such a way that the samples are always at the bottom of the sphere.

7. The samples do not always scatter back 100% of the incident radiation; a significant portion is also reflected specularly as by the surface of a mirror. To eliminate this portion, insert the gloss trap (Fig. 19-1 / 19) in aperture (Fig. 19-1 / 11) after having removed locating pin (Fig. 19-1 / 12).

Loading powder samples



1	Retaining clip	5	Thrusting disk
2	Guiding pin	6	Knurled screw
3	Sample holder for powder samples	7	Integrating sphere
4	Cylindrical support for integrating sphere		

Fig. 19-4 Integrating sphere with sample-loading fixture

Uncompressed powder samples must be loaded outside the SPECORD®. For that, a special holding fixture (Fig. 19-4) is provided.

8. Fill the sample into the holder for powder samples (Fig. 19-1 / 21). Slightly compress the sample and smooth the surface.

9. Place the sphere onto the support (Fig. 19-4 / 4). Turn knurled screw (Fig. 19-4 / 6) to move the thrusting disk (Fig. 19-4 / 5) towards the sphere body. Fasten the knurled screw only as much as is necessary to hold the sphere body reliably.

10. Put the sample holder (Fig. 19-4 / 3) into the recess located at the underside. Slightly clamp the sample holder by means of retaining clip (Fig. 19-4 / 1) by pushing down the clip on the guiding pin (Fig. 19-4 / 2).
11. Carefully insert the sphere body in the SPECORD® and move it to the right as far as it will go against the cell mount in the sample compartment (Fig. 1-1 / 10).

Reflectance measurement procedure

The measurement procedure is similar to that of transmittance measurements.

If you want to measure the diffuse reflectance as relative quantity in order to compare two only slightly different surfaces, it will do to perform a reference measurement with a purely white Spectralon® standard and subsequently take the sample measurement.

Insert the reflector into the round aperture in the rear mount for the cell holder as described above.

1. In the Device Driver dialog of WinASPECT® software, create the desired measurement parameter record.
   For the reflectance measurements, choose the following accessory-specific options:

Measurement parameter	Option to be activated
Correction	Reference
Slit	As wide as possible to obtain a good energy level
Accessory	Integrating Sphere

2. Insert the two Spectralon® inserts for gloss trap (Fig. 19-1 / 18) and reflectance aperture (Fig. 19-1 / 22) in the sphere body.
3. Carry out a reference measurement. For that, you may use a standard.
4. If you have to analyze a powder sample, fill it into the corresponding holder (Fig. 19-1 / 21) outside the SPECORD®. Accommodate the sphere body in the sample-loading fixture (Fig. 19-4). Attach the holder for powder samples to the sphere body.
5. Make sure to install the sphere body in the SPECORD® exactly in the same way as you did for the reference measurement.
6. By taking two measurements – with and without gloss trap – you can determine the percentages of scattered and reflected radiation.

If you want to take very accurate diffuse reflectance measurements at a defined wavelength, first calibrate the system using several certified standards same as in **Quantitative Analysis**. Then, as you are used to do from concentration determination in Quantitative Analysis, measure the unknown sample based on the found calibration curve.

19.6 Technical Data

Spectral range:	380 – 1100 nm
Sphere material:	Spectralon®
Sphere diameter:	75 mm
Light entrance aperture:	∅ 10 mm
Reflectance aperture:	∅ 12 mm
Light exit aperture:	∅ 16 mm
Dimensions (L x W x H):	150 mm x 105 mm x 145 mm
Weight:	1.0 kg
Transmittance cells for liquid samples:	Width 12.5mm Pathlength up to 10 mm
Solid samples for transmittance measurement (L x W x H):	up to 250 mm x 85 mm x 15 mm
Samples for diffuse reflectance	Diameter up to 50 mm Depth up to 20 mm
Sample holder for powder samples	Diameter 16 mm Depth 5 mm

20 Figures